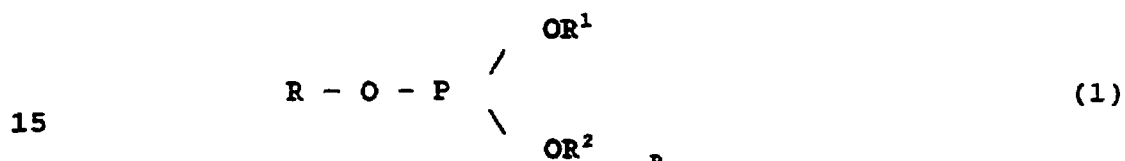


5 PROCESS TO PREPARE A MULTIDENTATE PHOSPHITE COMPOUND

The invention relates to a process to prepare
a multidentate phosphite compound represented by the
10 general formula (1)



in which n is 2-6, R is an n-valent organic group and
R¹ and R² are fused aromatic ring systems with 2 or more
20 rings, which rings are substituted on the ortho
position relative to the oxygen atom, only with
hydrogen, provided substitution is possible. The
phosphite compounds are prepared by first preparing a
phosphorochloridite compound from R¹-OH and R²-OH
25 alcohol compounds and a phosphorus chloride compound.
In a subsequent contacting step the phosphorochloridite
compound is contacted with an alcoholic compound
according to R-(OH)_n to yield the phosphite compound.

Such a process is described in US-A-5235113.
30 This patent publication describes the preparation of a
phosphorochloridite by reacting 3,6-di-tert-butyl-2-
naphthol dissolved in triethylamine and PCl₃ dissolved
in toluene. A bidentate phosphite is subsequently
prepared by contacting the phosphorochloridite compound
35 with 2,2'-biphenyldiol in triethylamine.

Disadvantages of this known process include
the difficulty in preparing the intermediate
phosphorochloridite compound in a high yield when

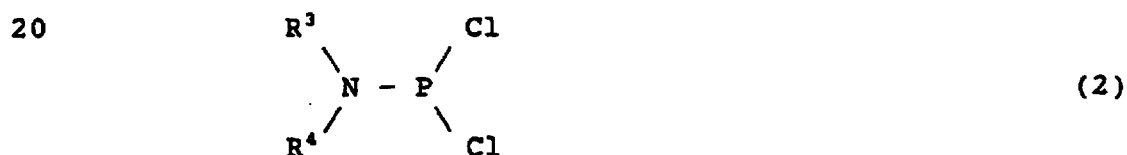
- 2 -

starting from alcohols (R^1OH and R^2OH), in which the steric bulk of the molecule around the hydroxyl-functional group is not sufficiently large. The main product will then be a triorganophosphite compound, in which the organo-groups correspond with the starting alcohol compound. The formation of this compound can be explained by the less steric hinderance around the P-O bond of the phosphite, which makes it possible that three moles of alcohol compounds react with one mole of PCl_3 .

There is a need for a process in which these phosphorochloridite compounds can be prepared in a high yield in order to be able to prepare the class of multidentate phosphite compounds according to formula (1) which presently are difficult to obtain.

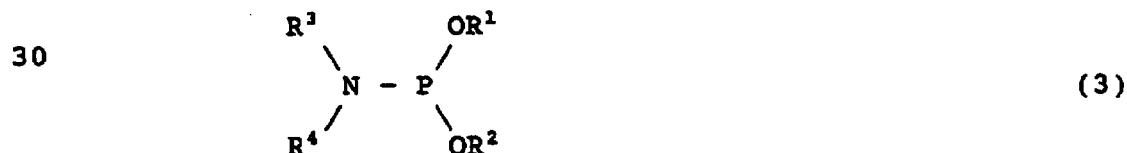
This aim is achieved in that the phosphorochloridite, is prepared by performing the following two steps in a solvent:

a) contacting a compound with the general formula:



in which R^3 and R^4 are C_1 - C_4 alkyl groups, with the R^1OH and R^2OH compounds, and

b) contacting the resulting compound of step (a) which has the formula (3):



with HY , wherein Y represents a halogen atom.

When using the process according to the invention it is possible to prepare the phosphorochloridite compound and thus the phosphite compound according to formula (1) in a high yield.

A method for preparing phosphite compounds directly, in one step, from a dialkyl-N,N'-

- 3 -

dialkylphosphoramite compound and a mono-alcohol is described in Synthesis, 1988, 2, 142. The resulting phosphite compound is however a monodentate phosphite compound. It appeared to be difficult to directly
5 prepare a multidentate phosphite compound in an analogous method by starting from a compound, $R(OH)_n$, with more than one alcohol functionalities ($n > 1$).

Starting alcoholic compounds R^1OH and R^2OH preferably have 10-30 carbon atoms and preferably
10 comprise of between 2-4 fused aromatic rings. Examples are naphthol, anthranol and phenanthrol. All of the carbon atoms adjacent to the carbon atom on which the hydroxyl group is substituted, are substituted, if possible, only with hydrogen. The other carbon atoms of
15 the fused rings may optionally be substituted with other groups, for example alkoxy, alkyl, amine and halogen groups. Preferably R^1 and R^2 are the same group. R^1 and R^2 are preferably 9-phenanthryl or 1-naphthyl groups.

20 Step (a) can be performed in the same manner as described in Synthesis, 1988, 2, 142-144. This article describes the preparation of alkyl and aryl di-tert-butyl phosphates using di-tert-butyl N,N-diethylphosphoramidite as an intermediate compound.
25 This intermediate compound is analogous to the compound as described by formula (3).

The compound according to formula (2) can be obtained by the methods known to the man skilled in the art, for example on an analogous manner as described in
30 the above article.

R^3 and R^4 are C_1 - C_4 alkyl, for example methyl, ethyl, propyl, butyl or tert-butyl. Preferably R^3 and R^4 are the same group.

In Step (a) the compound according to formula
35 (2) is contacted in a suitable solvent with the R^1OH and R^2OH , preferably in the presence of a base. Examples of bases include organic bases, for example a

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trialkylamine with 2 to 12 carbon atoms. Examples of suitable solvents are for example ethers, for example diethyl ether, dioxane or tetrahydrofuran or aromatic solvents for example benzene or toluene. Step (a) is preferably performed at a temperature between -80 and 60 °C, or more preferably about room temperature.

The concentration of the compound according to formula (2) is preferably between 0.01 and 5 mol/l. The molar ratio of R¹OH and R²OH and the compound (2) is preferably stoichiometric. Other ratio's are possible, but a greater effort is then needed to purify the product.

In Step (b) the Y of HY can be F, Cl, Br or I. A preferred HY is HCl. HY can be present in a gaseous form or dissolved in a solvent, for example an ether, for example diethyl ether, dioxane or tetrahydrofuran or aromatics, for example benzene or toluene. The temperature in step (b) is preferably between -80 and 60°C. The molar ratio of HCl and compound (3) is preferably between 0.8 and 5.

The step (b) phosphorochloridite reaction product, is generally obtained dissolved in the reaction mixture as obtained in step (b) having R³R⁴NH.HCl salt present. It can be advantageous to separate the salt from the phosphorochloridite compound before using this compound further. Separation can be performed by, for example, filtration of the reaction mixture.

The thus obtained phosphorochloridite compound is subsequently contacted with an alcoholic compound according to R-(OH)_n in which R is the n-valent organic group of formula (1) and n is 2-6.

The conditions for such a contacting in order to prepare the phosphite compound are generally known and are for example described in the aforementioned US-A-5235113. Generally the contacting is performed in a suitable solvent, for example the solvents as mentioned

- 5 -

as possible solvents for step (a). Preferably the contacting is performed in the presence of a base, for example an organic base, for example alkylamine for example triethylamine. Temperature is preferably
5 between -80 and 100°C.

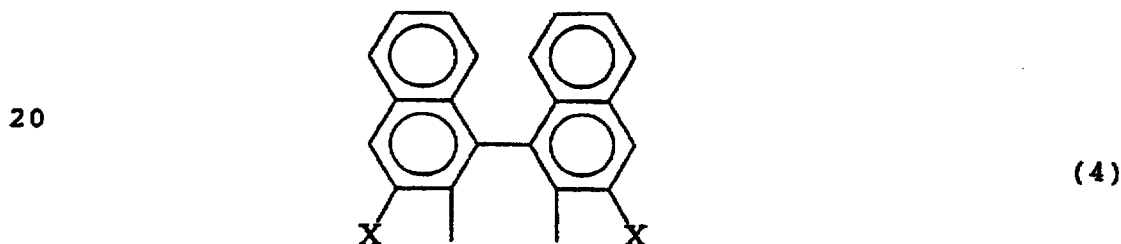
The reaction mixture obtained in step (b), comprising the phosphorochloridite compound and the $R^3R^4NH.HCl$ salt may be used in the preparation of the multidentate phosphite compound. Preferably the
10 phosphorochloridite compound is first separated from the $R^3R^4NH.HCl$ salt before preparing the phosphite compound. This is especially preferred when the $R-(OH)_n$ compound is not very reactive. Separation may be for example performed by filtration.

15 The n-valent group R can be any organic bridging group which are commonly known for multidentate phosphite compounds, for example those groups which are described in US-A-5235113, EP-A-214622 or WO-A-9518089.

20 Preferably the n-valent group has at least two carbon atoms. The n-valent group preferably has less than 40 carbon atoms. The n-valent group may be alkylene groups or divalent aromatic groups. Examples of possible alkylene groups are ethylene, trimethylene,
25 tetramethylene or pentamethylene. Examples of alcohols according to $R-(OH)_n$, which are the building blocks of the n-valent organic group are 2,5-di-t-butylhydroquinone, 2,5-di-t-amylhydroquinone, 2,5-dimethylhydroquinone, 4,6-di-t-butylresorcinol, 4,4'-isopropylidenebisphenol, 4,4'-methylenebis(2-methyl-6-t-butylphenol), 4,4'-oxobis(2-methyl-6-isopropylphenol), 4,6'-butylidenebis (3-methyl-6-t-butylphenol), 2,2'-biphenyldiol, 3,3',5,5'-tetramethyl-2,2'-biphenyldiol, 3,3',5,5'-tetra-t-butyl-2,2'-
30 biphenyldiol, 3,3'-dimethoxy-5,5'-dimethyl-2,2'-biphenyldiol, 3,3'-di-t-butyl-5,5'-dimethoxy-2,2'-biphenyldiol, 3,3'-di-t-butyl-5,5'-dimethyl-2,2'-

biphenyldiol, 2,2'-methylenebis(4-methyl-6-t-butylphenol), 2,2'-methylenebis(4-ethyl-6-t-butylphenol), 2,2'-thiobis(4-methyl-6-t-butylphenol), 2,2'-thiobis(4-t-butyl-6-methylphenol), 2,2'-thiobis(4,6-di-t-butylphenol), 1,1'-thiobis(2-naphthol), catechol, 2,3-dihydroxynaphthalene, 1,8-dihydroxynaphthalene, 1,4-dihydroxynaphthalene, 1,3,5-trihydroxybenzene, 1,1'-methylenebis(2-naphthol), 1,1'-di-2-naphthol, 10,10'-di-9-phenanthrol, ethyleneglycol, 1,3-propanediol, 1,2-butanediol, 1,4-butanediol, pentaerythritol, trans-1,2-cyclohexanediol, cis-1,2-cyclohexanediol, cis-1,2-cyclohexanedimethanol, cis-1,2-cyclododecanediol or the like.

A preferred and novel class of bidentate phosphite compounds according to formula (1) have a group R according to the following formula (4):



25 in which X is hydrogen or an organic group and preferably both X are an organic group and more preferably an alkyl group, an aryl group, a triarylsilyl group, a trialkylsilyl group, a carboalkoxy group, a carboaryloxy group, an aryloxy group, an alkoxy group, an alkylcarbonyl group, an arylcarbonyl group, an amide or a nitrile group. The invention is also directed to this novel bidentate phosphite ligand.

35 The alkyl group is preferably a C₁-C₁₀ alkyl group, for example methyl, ethyl, propyl, isopropyl, butyl, tert-butyl, isobutyl, pentyl or hexyl. An example of a triarylsilyl group is triphenylsilyl and

examples of a trialkylsilyl group are trimethylsilyl and triethylsilyl. Preferred aryl groups have 6 to 20 carbon atoms, for example benzyl, tolyl, naphthyl or phenanthryl. Preferred aryloxy groups have 6 to 12 carbon atoms, for example phenoxy. Preferred alkoxy groups have 1 to 10 carbon atoms, for example methoxy, ethoxy, tert-butoxy or isopropoxy. Preferred alkylcarbonyl groups have 2 to 12 carbon atoms, for example methylcarbonyl, tert-butylcarbonyl. Preferred arylcarbonyl groups have 7 to 13 carbon atoms, for example phenylcarbonyl.

X is most preferably a carboalkoxyl or carboaryloxy groups, $-\text{CO}-\text{O}-\text{R}^3$, in which R^3 is an alkyl group with 1 to 20 carbon atoms or an aryl group with 6-12 carbon atoms. Examples of suitable R groups are methyl, ethyl, propyl, isopropyl, n-butyl, tert-butyl, isobutyl, phenyl, tolyl. Even more preferably both X's are substituted with the same carboalkoxyl group.

R^1 and R^2 are preferably 9-phenanthryl or 1-naphthyl groups.

The phosphite compounds having a bridging group R according to formula (4) as described above can be advantageously used as part of a homogeneous catalyst system also comprising rhodium. Such a catalyst system is preferably used for the hydroformylation reaction of an unsaturated organic compound and especially of an internally unsaturated organic compound to a terminal aldehyde organic compound. High reaction rates and high selectivities to linear aldehyde (terminal aldehyde) products can be achieved when using such catalyst system.

For example it has been found that when using such a catalyst system the reaction of a C_1 - C_6 alkyl 3-pentenoate ester to the C_1 - C_6 alkyl 5-formylvalerate ester proceeds with a high selectivity, yield and reaction rate when compared to state of the art processes, such as for example described in WO-A-

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9518089. The invention is also directed to a process to prepare C₁-C₆ alkyl 5-formylvalerate esters. Preferably the alkyl is methyl or ethyl.

5 Preferably the multidentate phosphite compound according to formula (1) as obtained by the process of the invention is used as a polymer stabilizer.

10 Preferably the multidentate phosphite compound according to formula (1) as obtained by the process of the invention is used as a fire retarding additive.

15 Preferably the multidentate phosphite compound as obtained by the process of the invention is used as part of a homogeneous catalyst system also comprising a metal of Group VIII. The homogeneous catalyst system can be used for the isomerization of olefins. More preferably the homogeneous catalyst system is used in the hydroformylation of an ethylenically unsaturated organic compound to a terminal aldehyde compound. When used as a hydroformylation catalyst the group VIII metal is preferably rhodium or iridium and most preferably rhodium. The molar ratio of multidentate phosphite ligand to rhodium is generally from about 0.5 to 100 and preferably from 1 to 10 and most preferably less than 1.2 (mol ligand/mol rhodium). It has been found that by using a slight excess of ligand, the ligand degradation can be reduced. Preferably the unsaturated compound is an pentenoic acid, its corresponding ester or pentenitrile. The resulting 5-formylvaleric acid, its ester or nitrile are important intermediates in a process to prepare precursors for Nylon-6 and Nylon-6.6.

35 The hydroformylation reaction is preferably performed as described below.

The concentration of rhodium in the reaction mixture may vary from 1 to 5000 ppm rhodium.

Preferably, the concentration is between 50 and 1000 ppm.

The reaction mixture may serve as a solvent, so that as a rule the addition of an additional solvent is not necessary. The reaction mixture is a mixture of the reactants of the hydroformylation, for example the unsaturated organic compound, the aldehyde and/or by-products formed, in particular the by-products with high boiling temperatures. If an additional solvent is added, a saturated hydrocarbon, for example naphtha, kerosine, mineral oil or cyclohexane, or an aromatic compound, for example toluene, benzene, xylene, or an ether, for example diphenylether, tetrahydrofuran, or a ketone, for example cyclohexanone, or a nitrile, for example benzonitrile, texanol® or tetraglyme® (Union Carbide), is suitable for use as additional solvent. A mixture of two or more of these compounds is also suitable for use as additional solvent.

The temperature is generally between room temperature and 200°C, preferably between 50°C and 150°C. The pressure is generally between 0.1 MPa and 20 MPa, preferably between 0.15 MPa and 10 MPa and most preferably between 0.2 MPa and 1 MPa.

The molar ratio of hydrogen and carbon monoxide is generally between 10:1 and 1:10, preferably between 6:1 and 1:2.

The hydroformylation reaction can be carried out in a gas/liquid contactor known to a person skilled in the art. Examples of suitable reactors are a bubble column, screen-plate column or a gas-liquid agitated reactor.

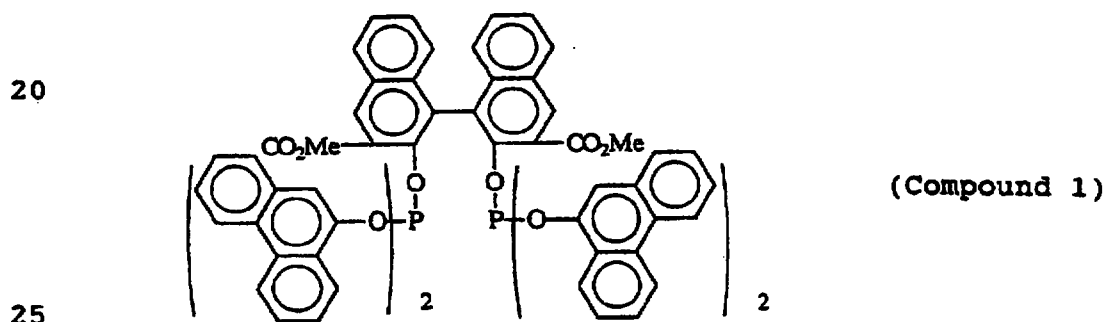
The invention shall be elucidated with the following non-limiting examples.

35 Example I

3.88 g (20 mmol) 9-phenanthrol was dissolved in 250 ml toluene and the water was removed by

- 10 -

azeotropic distillation. Subsequently, 2.3 g triethylamine and 1.74 g (10 mmol) diethylamino-phosphorous dichloride were added at room temperature while stirring. In this way diethylamino diphenanthrene phosphite was synthesized (^{31}P NMR δ 138.7 ppm). To obtain diphenanthrene phosphorous chloride 22 ml of a 1 M HCl solution in diethyl ether was added (^{31}P NMR δ 161.4 ppm). The reaction mixture was filtered and to the filtrate 3 g (30 mmol) triethyl amine and 1.98 g dimethyl 2,2'-dihydroxy-1,1'-binaphthalene-3,3'-dicarboxylate were added. After stirring for 10 minutes the reaction was completed and $\text{NEt}_3\cdot\text{HCl}$ was removed by filtration (Et = ethyl). The solvent was removed and the product was purified by crystallization from acetonitril/toluene (^{31}P NMR δ 126.6 ppm). The yield of Compound 1 was 90%.



Comparative Experiment A

Compound 1 was also intended to be prepared by the synthetic route as described in Example 10 of US-A-5235113 starting from 9-phenanthrol. It was however not possible to obtain the phosphorochloridite intermediate in a high yield (about 5%). Mainly tri(9-phenanthryl)phosphite was obtained. The 5% of the desired product could not be isolated easily. This experiment illustrates that it is difficult to prepare the Compound 1 by the prior art synthetic route.

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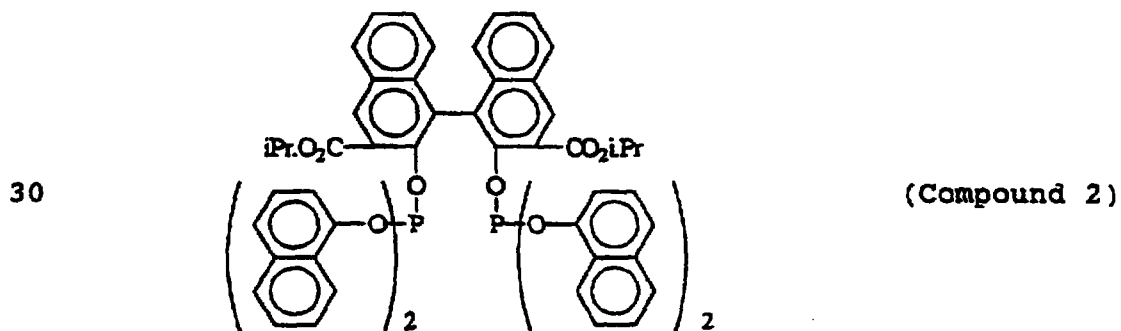
Example II

A 150 ml Hastelloy-C steel autoclave (Parr) was filled under nitrogen with 5.8 mg Rh(acac)(CO)₂ (acac = acetylacetonate) (4.8×10^{-5} mol), 14.0×10^{-5} mol of Compound 1 as ligand (ligand/rhodium ratio (L/Rh) = 2.9 mol/mol) and 60 ml of toluene. Hereafter, the autoclave was closed and purged with nitrogen. Next, the autoclave was brought to a pressure of 1 MPa using carbon monoxide/hydrogen (1:1) and heated to 90°C in approx. 30 min. Subsequently a mixture of 7.44 g (65 mmol) freshly distilled methyl 3-pentenoate and 1.2 gram of nonane topped up to 15 ml with toluene was injected into the autoclave. The composition of the reaction mixture was determined by gas chromatography. After 7 hours of reaction a 90.1% conversion was determined. The selectivity to methyl 5-formylvalerate was 75.1%. The molar ratio of methyl 5-formylvalerate and the sum of methyl 3- and methyl 4-formylvalerate (n/b ratio) was 9.3, and the hydrogenation to methyl valerate was 5.7%.

Example III

Example II was repeated in which the L/Rh was 3.1 with a ligand according to:

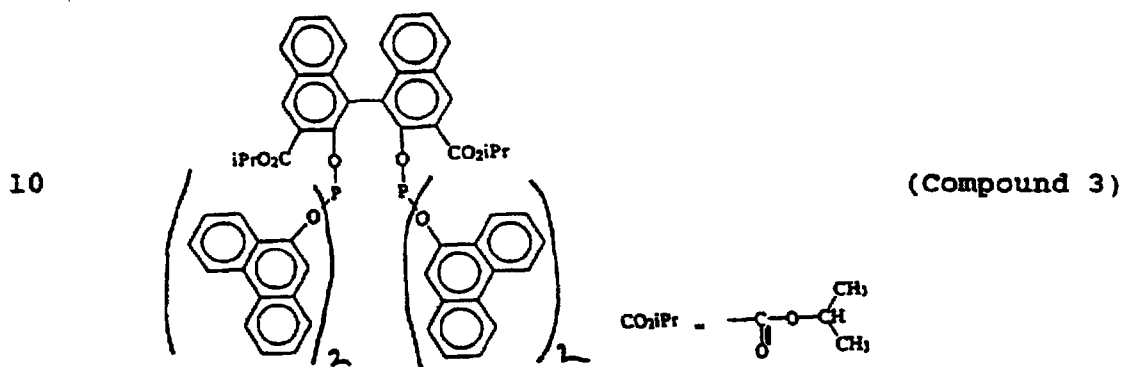
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35 The conversion was 81.8% and the selectivity to methyl 5-formylvalerate was 84.6%.

Example IV

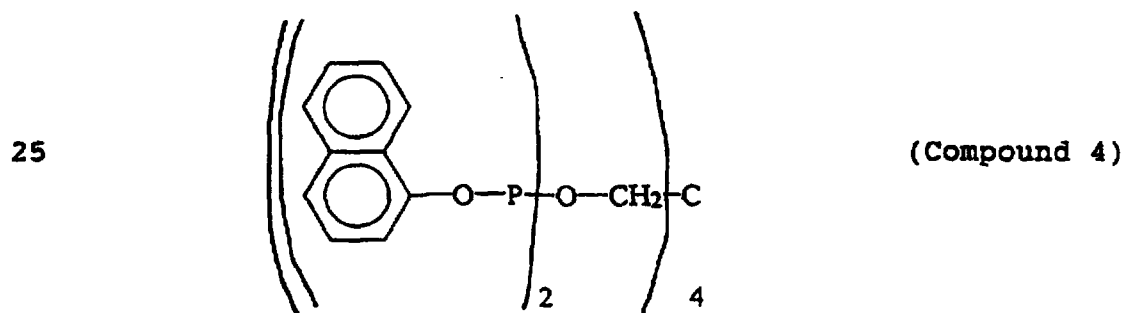
Example I was repeated using as $R-(OH)_n$ compound di-isopropyl 2,2'-dihydroxy-1,1'-binaphthalene-3,3'-dicarboxylate and as $R^1(OH)$ starting compound 9-phenanthrol. A compound according to:



15 was obtained as a slightly yellow coloured powder in a 85 % yield.

Example V

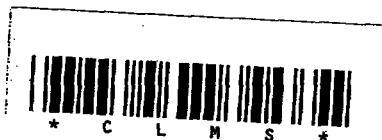
Example I was repeated using as $R-(OH)_n$ compound pentaerythritol and as $R^1(OH)$ starting compound 1-naphthol. A compound according to:



30 was obtained as a yellow coloured oil in a 80 % yield.

Example VI

Example I was repeated using as $R-(OH)_n$ compound diethyl 2,2'-dihydroxy-1,1'-binaphthalene-3,3'-dicarboxylate and as $R^1(OH)$ starting compound 4-chloro-1-naphthol. The yield was about 90%.

C L A I M S

1. Process to prepare a multidentate phosphite compound represented by the general formula (1)

5



10

in which n is 2-6, R is an n -valent organic group and R^1 and R^2 are fused aromatic ring systems with 2 or more rings, which rings are substituted on the ortho position relative to the oxygen atom only with hydrogen, by first preparing a phosphorochloridite compound from a $R^1\text{-OH}$ and $R^2\text{-OH}$ alcohol compound and a phosphorus chloride compound and subsequently contacting the phosphorochloridite compound with an alcoholic compound according to $R - (\text{OH})_n$, characterized in that the phosphorochloridite is prepared by performing the two steps in a solvent:

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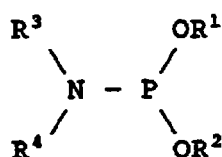
- a) contacting a compound with the general formula:

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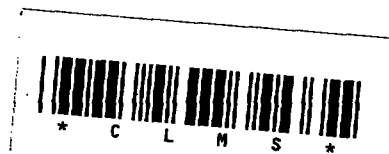
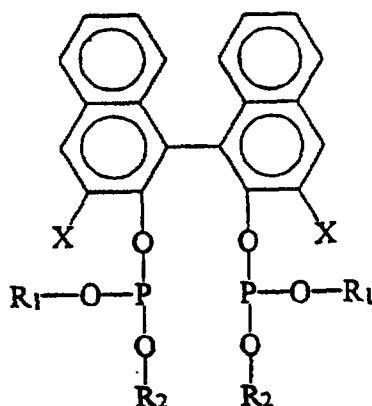
- in which R^3 and R^4 are $\text{C}_1\text{-C}_4$ alkyl groups, with the $R^1\text{OH}$ and $R^2\text{OH}$ compounds, and
b) contacting the resulting compound of step (a) which has the formula (3):



(3)

- 5
- with HY, wherein Y represents a halogen atom.
- 10 2. Process according to claim 1, characterized in that HY is HCl.
3. Process according to any one of claims 1-2, characterized in that R³ and R⁴ are the same alkyl group.
- 15 4. Process according to any one of claims 1-3, characterized in that the HCl is dissolved in a solvent when contacting with the compound according to formula (3).
- 20 5. Process according to any one of claims 1-4, characterized in that gaseous HCl is contacted with the compound according to formula (3) present in the solvent.
- 25 6. Process according to any one of claims 1-5, characterized in that the temperature of step (a) and (b) is between -80°C and 60°C.
- 30 7. Process according to any one of claims 1-6, characterized in that the phosphorochloridite compound is separated from the precipitated R³R⁴NH.HCl salt, which salt is also formed in the process, of the reaction mixture which results from the process.
8. Process according to any one of claims 1-7, characterized in that R¹-OH and R²-OH are one of the alcohols including naphthol, anthranol or phenanthrol.
- 35 9. Process according to any one of claims 1-8, characterized in that group R has between 2 to 40 carbon atoms.
10. Bidentate phosphite compound according to the following general formula:

- 15 -



5

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in which X is hydrogen or an organic group and R^1 and R^2 are fused aromatic ring systems with 2 or more rings, which rings are substituted on the ortho position relative to the oxygen only with hydrogen.

15

11. Bidentate phosphite compound according to claim 10, characterized in that X is an alkyl group, an aryl group, a triaryl silyl group, a trialkylsilyl group, a carboalkoxy group, a carboaryloxy group, an aryloxy group, an alkoxy group, an alkylcarbonyl group, an arylcarbonyl group, a nitrile group or an amide group.

20

12. Bidentate phosphite compound according to claim 11, characterized in that X is a carbo alkoxyl group or carboaryloxy group according to $-CO - O - R^3$, in which R^3 is an alkyl group with 1 to 20 carbon atoms or an aryl group with 6-12 carbon atoms.

25

13. Bidentate phosphite compound according to any one of claims 10-12, characterized in that R^1 and R^2 are 1-naphthyl or 9-phenanthryl groups.

30

14. Process to prepare a bidentate phosphite compound according to claims 10-13 by using the process according to any one of claims 1-9.

35

15. Homogeneous catalyst system comprising a metal of Group VIII and a multidentate phosphite compound as obtained by the process according to any one of claims 1-9 or 14.

16. Homogeneous catalyst system according to claim 15,



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characterized in that the group VIII metal is rhodium.

17. Homogeneous catalyst system according to claims 15-16, characterized in that the phosphite to
5 Group VIII metal molar ratio is between about 1 and 1.2.
18. Use of the homogeneous catalyst system of any of claims 15-17 in the hydroformylation of an ethylenically unsaturated organic compound to an
10 aldehyde compound.
19. Use according to claim 18, characterized in that the ethylenically unsaturated organic compound is a C₁-C₆ alkyl 3-pentenoate.

PCT

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(71) Applicants (for all designated States except US): DSM N.V. [NL/NL]; Het Overloon 1, NL-6411 TE Heerlen (NL). E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US). ✓

(72) Inventors; and

(75) Inventors/Applicants (for US only): SNIJDER, Carina, Sacha [NL/NL]; Voegezenstraat 18, NL-6137 JR Sittard (NL). TEUNISSEN, Antonius, Jacobus, Josephus, Maria [NL/NL]; Van Ostadestraat 42, NL-6165 XM Geleen (NL). HANSEN, Carolina, Bernedette [NL/NL]; Rolandsweg 27A, NL-6136 BT Sittard (NL). SHARIPO, Rafael [US/US]; 1415 Fresno Road, Wilmington, DE 19803 (US). GARNER, James, Michael [US/US]; 815 N. Jackson #3, Wilmington, DE 19803 (US).

(74) Agent: CRAMWINCKEL, Michiel; Octrooibureau DSM, P.O. Box 9, NL-6160 MA Geleen (NL).

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(54) Title: PROCESS TO PREPARE A MULTIDENTATE PHOSPHITE COMPOUND

(57) Abstract

Process to prepare a multidentate phosphite compound according to general formula (1) in which n is 2-6, R is an n-valent organic group and R¹ and R² are fused aromatic ring systems with 2 or more rings, which rings are substituted on the ortho position relative to the oxygen atom only with hydrogen, by first preparing a phosphorochloridite compound from a R¹-OH and R²-OH alcohol compound and a phosphorus chloride compound and subsequently contacting the phosphorochloridite compound with an alcoholic compound according to R-(OH)_n, wherein the phosphorochloridite is prepared by performing the two steps in a solvent: a) contacting a compound with general formula (2) in which R³ and R⁴ are C₁-C₄ alkyl groups, with the R¹OH and R²OH compounds and b) contacting the resulting compound of step (a) which has formula (3) with HY, wherein Y represents a halogen atom.

