

(III)

where R_2 and R_3 are as defined above for the compound of formula (I),

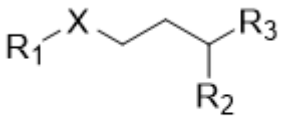
in the presence of at least one agent for initiating a radical addition reaction comprising or consisting of a persulfate and one or several transition metals, ~~in elemental form or in oxidized form~~ said transition metals being selected from Cu, Zn and Fe, in their elemental form or in an oxidized form selected from sulfates, phosphates, chlorides, carbonates, hydroxides, nitrites, nitrates, acetates, alcoholates, hydrogenphosphates and hydrogen sulfates, and their alloys such as brass.

In another aspect, the present disclosure refers to a use of an agent for the initiation of a radical addition reaction for obtaining compounds of formula (I) as described herein, said agent comprising or consisting of a persulfate and one or several transition metals, ~~in elemental or oxidized form~~said transition metals being selected from Cu, Zn and Fe, in their elemental form or in an oxidized form selected from sulfates, phosphates, chlorides, carbonates, hydroxides, nitrites, nitrates, acetates, alcoholates, hydrogenphosphates and hydrogen sulfates, and their alloys such as brass.

Detailed Description

The invention concerns an agent for the initiation of a radical addition reaction and its application in a method for preparing a compound of formula

[Chem I]



(I)

in which

X is selected from S, Se and O;

R_1 is selected from H, alkyl groups, aryl groups and alkylaryl groups;

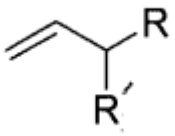
R_2 is selected from H; OH; NR_4R_5; $NHCOR_4$; $OCOR_4$; where R_4 and R_5 are the same or different and are selected from H, alkyl groups, aryl groups, alkylaryl groups; and protecting groups;

R_3 is selected from OH, CH_2OH, COOH, $COOR_6$ where R_6 is selected from alkyl, CN, $CONR_4R_5$ where R_4 and R_5 are as defined above, and COZ where Z represents a halide;

by a radical addition reaction in the presence of such an agent.

According to WO98/32735A1, there is known a method for preparing methionine and hydroxymethylthiobutyric acid (HMTBA), as well as their derivatives, by radical addition of methylthiol to a compound of the formula below

[Chem I]

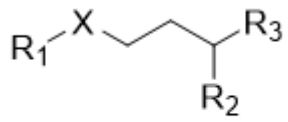


in which R is selected from COOH and the corresponding ester, amide, nitrile and trichloro functions and R′ is selected from OH, NH_2, oxycarbonyl and aminocarbonyl functions and protecting groups. This reaction is initiated in the presence of an initiator agent consisting of azobisisobutyronitrile (AIBN) at a temperature in the range of 50 °C to 60 °C under a pressure in the range of 3.5 bars to 4.8 bars, or by UV irradiation. It leads to yields of at least 80%. The proven lessons of this document, and in particular regarding the above conditions, are limited to obtaining the HMTBA. The use of AIBN or its derivatives in this reaction has several

CLAIMS

1. A method for preparing a compound of formula (I)

[Chem I]



(I)

in which

X is selected from S, Se and O;

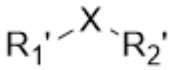
R_1 is selected from H, alkyl groups, aryl groups and alkylaryl groups;

R_2 is selected from H; OH; NR_4R_5; $NHCOR_4$; $OCOR_4$; where R_4 and R_5 are identical or different and are selected from H, alkyl groups, aryl groups, alkylaryl groups; and protecting groups;

R_3 is selected from OH, CH_2OH, COOH, $COOR_6$ where R_6 is selected from alkyl, CN, $CONR_4R_5$ where R_4 and R_5 are as defined above, and COZ where Z represents a halide;

which method comprising the reaction of a compound of formula (II)

[Chem II]



(II)

In which

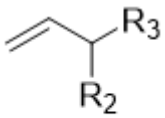
X is selected from S, Se and O;

R_1' is selected from H, alkyl, aryl, alkylaryl, thiol, thioalkyl, thioaryl, thioalkylaryl, selenol, selenoalkyl, selenoaryl and selenoalkylaryl groups, and

R_2' is selected from H, alkyl, aryl and alkylaryl groups;

with a compound of formula (III)

[Chem III]



(III)

where R_2 and R_3 are as defined above for the compound of formula (I),

in the presence of at least one agent for initiating a radical addition reaction comprising or consisting of a persulfate and one or several transition metals, ~~in elemental form or in oxidized form~~ said transition metals being selected from Cu, Zn and Fe, in their elemental form or in an oxidized form selected from sulfates, phosphates, chlorides, carbonates, hydroxides, nitrites, nitrates, acetates, alcoholates, hydrogenphosphates and hydrogen sulfates, and their alloys such as brass.

2. The method according to claim 1, characterized in that the persulfate is in the form of a salt of a cation, preferably selected from sodium, potassium, calcium, lithium and ammonium ions.

~~3. The method according to claim 1 or 2, characterized in that the transition metal(s) are selected from Cu, Zn and Fe, in their elemental form or in an oxidized form selected from sulfates, phosphates, chlorides, carbonates, hydroxides, nitrites, nitrates, acetates, alcoholates, hydrogenphosphates and hydrogen sulfates, and their alloys such as brass.~~

~~4.~~3. The method according to ~~any one of claims 1 to 3~~claim 1 or 2, characterized in that the molar ratio of persulfate to the transition metal(s) ranges from 1 to 1000.

~~5.~~4. The method according to any one of claims 1 to ~~4~~3, characterized in that the persulfate is potassium persulfate or sodium persulfate.

~~6.~~5. The method according to any one of claims 1 to ~~5~~4, characterized in that the molar ratio of compound (II) or of compound (III) to the initiation agent ranges from 2 to 1000.

~~7.~~6. The method according to any one of claims 1 to ~~6~~5, characterized in that the molar ratio of compound (II) to compound (III) ranges from 0.9 to 20, preferably from 1.1 to 3.

~~8.~~7. The method according to any one of claims 1 to ~~7~~6, characterized in that the compound (II) is methylthiol or methylselenol.

~~9.~~8. The method according to any one of claims 1 to ~~8~~7, characterized in that the compound (III) is methyl vinyl glycolate.

~~10.~~9. The method according to any one of claims 1 to ~~9~~8, characterized in that the reaction is carried out at a temperature from -20 °C to 40 °C.

~~11.~~10. A use of an agent for the initiation of a radical addition reaction for obtaining compounds of formula (I) as defined in claim 1, said agent comprising or consisting of a persulfate and one or several transition metals, ~~in elemental or oxidized form~~said transition metals being selected from Cu, Zn and Fe, in their elemental form or in an oxidized form selected from sulfates, phosphates, chlorides, carbonates, hydroxides, nitrites, nitrates, acetates, alcoholates, hydrogenphosphates and hydrogen sulfates, and their alloys such as brass.