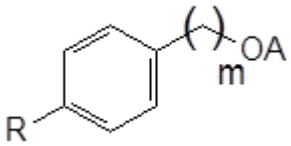
CLAIMS

1. A method for preparing and purifying a compound of the following general Formula (XIX)

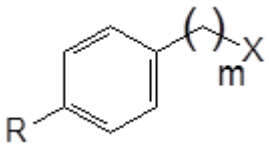


(Formula (XIX)),

wherein m=1, R represents a hydrocarbon group having a linear chain of 2 to 12 carbon atoms and one or more methyl groups as substituents on said linear chain, and A represents a polyoxyethylene residue,

the method comprising the steps of

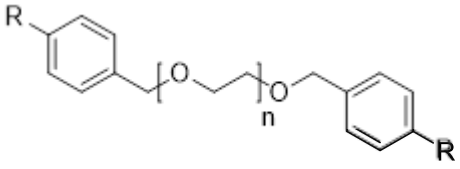
(3) Reacting a compound of the following general Formula (XII)



Formula (XII),

wherein m and R are as defined above and X represents a halogen atom or a hydroxyl group,

with a polyethylene glycol (PEG) of the general formula $HO(CH_2CH_2O)_nH$, wherein n is an integer of n≥2, to produce a compound of said general formula (XIX) and a side product of the following general Formula (XIII)



(Formula (XIII)),

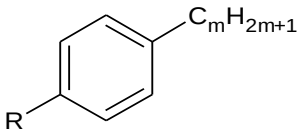
wherein R is as defined above and n is an integer of n≥2;

and

(4) Purifying the compound of said general Formula (XIX) by removing therefrom unreacted polyethylene glycol (PEG) and said side product.

2. The method of claim 1, which further comprises the following step (2) prior to said step (3):

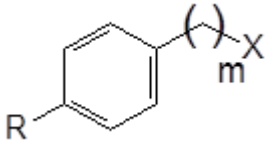
(2) Converting a compound of the following general Formula (XIV),



(Formula (XIV)),

wherein m and R are as defined in claim 1,

into a compound of the following general Formula (XII)

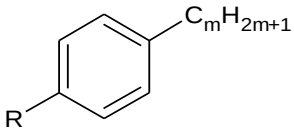


Formula (XII),

wherein m, R and X are as defined in claim 1,

which optionally further comprises the following step (1) prior to said step (2):

(1) Reacting toluene so as to obtain a compound of the following general Formula (XIV)



(Formula (XIV)),

wherein m and R are as defined in claim 1,

optionally wherein in step (1), toluene is reacted with diisobutylene or a compound R-X, wherein R is as defined in claim 1 and X represents a halogen atom.

3. The method of claim 2, wherein:

(a) the converting in step (2) is a radical reaction using AIBN (azobis(isobutyronitrile)) as a radical initiator; and/or

(b) the converting in step (2) uses N-bromosuccinimide (NBS) as a halogenating reagent.

4. The method of any of claims 1 to 3, wherein X is a chlorine, iodine or bromine atom, or wherein X is a bromine atom.

5. The method of any one of the preceding claims, wherein:
(a) said step (4) comprises subjecting a solution of the compound of said general Formula (XIX) in a solvent selected from alkanes, ethers and esters and mixtures thereof to one or more aqueous washes to remove said unreacted polyethylene glycol (PEG) from the compound of said general Formula (XIX), optionally wherein said solvent selected from alkanes, ethers and esters and mixtures thereof is an ester, further optionally wherein said solvent selected from alkanes, ethers and esters and mixtures thereof is ethyl acetate; and/or
(b) the purifying of said compound of said general Formula (XIX) by removing therefrom said side product in step (4) comprises subjecting a solution of said compound of said general Formula (XIX) in a mixture comprising water and a water-miscible organic solvent to one or more washes with a non-polar solvent,
optionally wherein the non-polar solvent comprises an alkane, further optionally wherein the alkane is hexane, cyclohexane or heptane, further optionally wherein the alkane is cyclohexane.

6. The method of any one of the preceding claims, wherein:
(a) in step (3), said polyethylene glycol (PEG) is used both as reactant and as a solvent; and/or
(b) step (3) is performed without employing a solvent or diluent other than said polyethylene glycol (PEG); and/or
(c) no halogenated solvent is used; and/or
(d) said polyethylene glycol (PEG) used in step (3) is characterized in that it is liquid at the reaction temperature of said step (3).

7. The method of any one of the preceding claims, which does not include the use of any preparative chromatographic methods after step (3); and/or which does not include the use of any preparative chromatographic methods.

8. The method of any one of the preceding claims, wherein:
(a) the reacting in step (3) takes place for at least 10 minutes and for not more than 1 hour, or the reacting in step (3) takes place for at least 20 minutes and for not more than 45 minutes, or for 30 minutes; and/or

(b) the reacting in step (3) takes place at a temperature of between 55°C and 65°C, or at a temperature of 60°C; and/or

(c) in step (3), each molar equivalent (eq.) of said compound of said general Formula (XII) is reacted with at least 1.0, at least 2.0, at least 3.0 or at least 4.0 molar equivalents (eq.) of said polyethylene glycol (PEG), or with at least 4.5 molar equivalents (eq.) of said polyethylene glycol (PEG), or with at least 5.0 molar equivalents (eq.) of said polyethylene glycol (PEG); and/or

(d) the method is carried out at a scale which yields at least 100g, at least 1 kg, at least 10 kg, at least 100 kg or at least 1000 kg of said compound of said Formula (XIX); and/or

(e) at least 1, at least 2, at least 3 or at least 4 aqueous washes are carried out after step 3; and/or

(f) 4 to 6 aqueous washes are carried out after step 3.

9. The method of any one of claims 2-8, wherein the compound of formula (XII) obtained in step (2) is precipitated from a solvent, optionally wherein:

(a) the solvent is an alcohol solvent; and/or

(b) the solvent is a polar solvent, optionally wherein the solvent is acrylonitrile; and/or

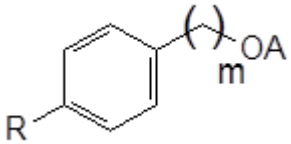
(c) the solvent is an alcohol solvent selected from the group consisting of isopropyl alcohol, ethanol, methanol and butanol or combinations thereof; and/or

(d) the solvent is a secondary alcohol; and/or

(e) the solvent is isopropyl alcohol.

10. The method of any one of claims 2-9, wherein at least one recrystallization of said compound of Formula (XII) is carried out in between step (2) and step (3). optionally wherein the solvent of claim 9 is employed as solvent for the recrystallization between step 2 and step 3.

11. A method of purifying a compound of formula (XIX),



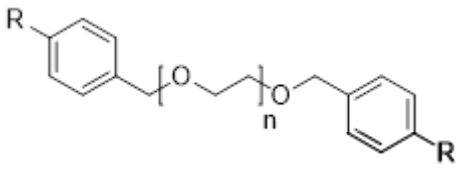
Formula (XIX),

wherein m=1, R represents a hydrocarbon group having a linear chain of 2 to 12 carbon atoms and one or more methyl groups as substituents on said linear chain, and A represents a polyoxyethylene residue,

from a composition comprising the compound of formula (XIX), the method comprising subjecting a solution of said composition in a solvent selected from alkanes, ethers and esters and mixtures thereof to one or more aqueous washes.

12. The method of claim 11, wherein:
(a) said solvent selected from alkanes, ethers and esters and mixtures thereof is an ester, optionally wherein said solvent selected from alkanes, ethers and esters and mixtures thereof is ethyl acetate; and/or
(b) the composition comprises polyethylene glycol (PEG), optionally wherein the purifying of the compound of formula (XIX) comprises removing the polyethylene glycol (PEG) from the composition, further optionally wherein the removing of the polyethylene glycol (PEG) comprises said subjecting of the solution to the one or more aqueous washes.

13. The method of claims 11 or 12, wherein the composition further comprises a compound of the following general Formula (XIII):



(Formula (XIII)),

and wherein R is as defined in claim 11 and n is an integer of n≥2.

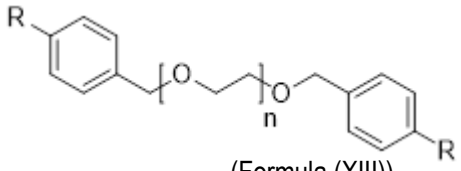
14. The method of claim 13, wherein the purifying of the compound of formula (XIX) comprises removing the compound of formula (XIII) from the composition,
optionally wherein the removing of the compound of formula (XIII) comprises preparing an aqueous solution comprising said compound of said general Formula (XIX) and removing said compound of formula (XIII) by one or more washes of said aqueous solution with a non-polar solvent,
further optionally wherein the non-polar solvent comprises an alkane,
further optionally wherein the alkane is hexane, cyclohexane or heptane,
further optionally wherein the alkane is cyclohexane.

15. The method of any one of claims 1 to 14, wherein:

(a) the degree of purity of the compound of formula (XIX) as obtained by the method, as measured by high-performance liquid chromatography with UV detection at 200 nm, is at least 80%, preferably at least 85%, more preferably at least 90%, and most preferably at least 95%; and/or

(b) the degree of purity of the compound of formula (XIX) as obtained by the method, as measured by high-performance liquid chromatography with detection using an evaporative light scattering detector (ELSD), is at least 99%; and/or

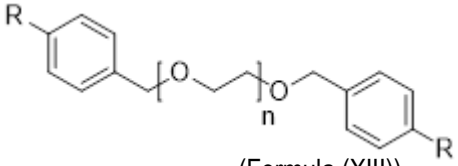
(c) the method further comprises a step of identifying a compound of the following general Formula (XIII):



(Formula (XIII)),

Wherein R represents a hydrocarbon group having a linear chain of 2 to 12 carbon atoms and one or more methyl groups as substituents on said linear chain, and n is an integer of n≥2; and/or

(d) the method further comprises a step of quantifying a compound of the following general Formula (XIII):



(Formula (XIII)),

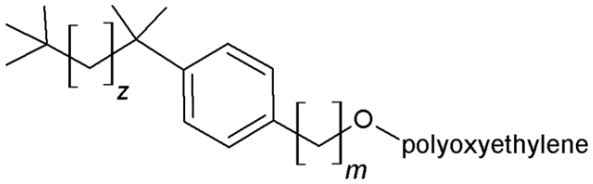
wherein R represents a hydrocarbon group having a linear chain of 2 to 12 carbon atoms and one or more methyl groups as substituents on said linear chain, and n is an integer of n≥2; and/or

(e) A represents a polyoxyethylene residue comprising 4 to 16 oxyethylene units, or 8 to 10 oxyethylene units, or 9 or 10 oxyethylene units; and/or

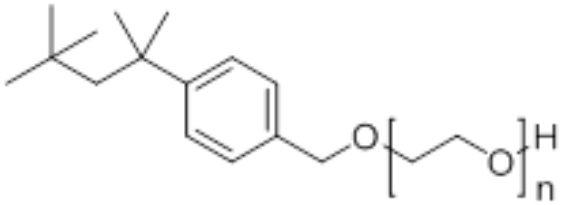
(f) R represents a hydrocarbon group having a linear chain of 2 to 6 carbon atoms and one or more methyl groups as substituents on said linear chain, or a linear chain of 2 to 6 carbon atoms and 2 to 4 methyl groups as substituents on said linear chain, or a linear chain of 4 carbon atoms and 4 methyl groups as substituents on said linear chain; and/or

(g) R represents a 2,4,4-trimethyl-pent-2-yl group; and/or

(h) the compound of Formula (XIX) is the following compound:

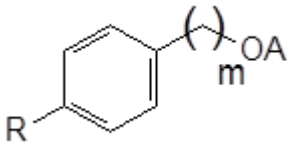


wherein m equals 1 and z is an integer selected from:
z = 1 to 5; and/or
(i) the compound of Formula (XIX) is the following compound:



wherein n is an integer between 4 and 16, preferably wherein n is equal to 8, 9 or 10.

16. A compound of the following general Formula (XIX)



(Formula (XIX)),

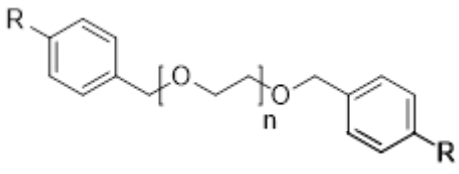
wherein m=1, R represents a hydrocarbon group having a linear chain of 2 to 12 carbon atoms and one or more methyl groups as substituents on said linear chain, and A represents a polyoxyethylene residue,

and wherein said compound is obtainable by the method of any one of the preceding claims.

17. The compound of claim 16, which:
(a) exhibits a degree of purity, as measured by high-performance liquid chromatography with UV detection at 200 nm, of at least 80%, preferably at least 85%, more preferably at least 90%, and most preferably at least 95%; and/or

(b) exhibits a degree of purity, as measured by high-performance liquid chromatography with detection using an evaporative light scattering detector (ELSD), of at least 99%.

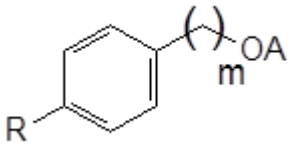
18. Use of a non-polar solvent for extracting and removing a compound of the following general Formula (XIII):



(Formula (XIII)),

wherein R represents a hydrocarbon group having a linear chain of 2 to 12 carbon atoms and one or more methyl groups as substituents on said linear chain and n is an integer of n≥2,

from a composition comprising said compound of Formula (XIII) and a compound of formula (XIX),



Formula (XIX),

wherein m=1, R represents a hydrocarbon group having a linear chain of 2 to 12 carbon atoms and one or more methyl groups as substituents on said linear chain, and A represents a polyoxyethylene residue.

19. The use of claim 18, wherein:
(a) the non-polar solvent is as defined in claim 14; and/or
(b) A is as defined in claim 15; and/or
(c) R is as defined in claim 15; and/or
(d) the compound of Formula (XIX) is as defined in claim 15.