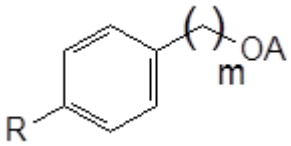
CLAIMS

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

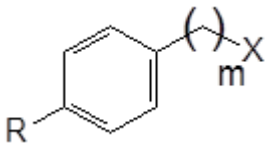


(Formula (XIX)),

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

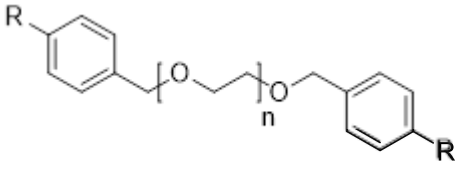
(3) Reacting a compound of the following general Formula (XII)



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)),

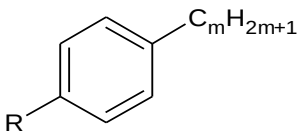
wherein R is as defined above and n is an integer of n≥2;

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):

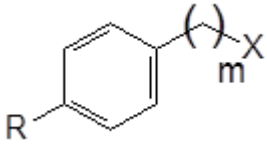
(2) Converting a compound of the following general Formula (XIV),



(Formula (XIV)),

wherein m and R are as defined in claim 1,

into a compound of the following general Formula (XII)

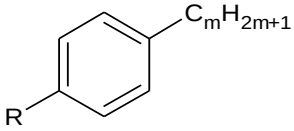


Formula (XII),

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

~~3. The method of claim 2,~~ 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)),

~~W~~wherein m and R are as defined in claim 1~~.~~,

~~4. The method of any one of the preceding claims, wherein in step (3), said polyethylene glycol (PEG) is used both as a reactant and as a solvent.~~

~~5. The method of claim 3 or 4,~~

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.

63. The method of ~~any of~~ claim~~s~~ 2 ~~to 5~~, 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.

74. The method of any of claims 1 to ~~6~~3, wherein X is a chlorine, iodine or bromine atom, or~~.~~

~~8. The method of any of claims 1 to 7,~~ wherein X is a bromine atom.

~~9. The method of any of claims 2 to 8, wherein the converting in step (2) uses N-bromosuccinimide (NBS) as a halogenating reagent.~~

~~10~~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), ~~.~~

~~11. The method of claim 10,~~ optionally wherein said solvent selected from alkanes, ethers and esters and mixtures thereof is an ester, ~~.~~

~~12. The method of claim 11,~~ further optionally wherein said solvent selected from alkanes, ethers and esters and mixtures thereof is ethyl acetate; and/or~~.~~

~~13. The method of any one of the preceding claims, wherein~~(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, ~~.~~

~~14. The method of claim 13,~~ optionally wherein the non-polar solvent comprises an alkane, ~~.~~

~~15. The method of claim 14,~~further optionally wherein the alkane is hexane, cyclohexane or heptane~~.~~

~~16. The method of claim 15,~~ further optionally wherein the alkane is cyclohexane.

~~17~~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).

~~18. The method of any one of the preceding claims, wherein no halogenated solvent is used.~~

~~19~~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~~.~~

~~20. The method of any one of the preceding claims,~~ which does not include the use of any preparative chromatographic methods.

~~21~~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~~.~~

~~22. The method of any one of the preceding claims, wherein~~ the reacting in step (3) takes place for at least 20 minutes and for not more than 45 minutes, or~~.~~

~~23. The method of any one of the preceding claims, wherein the reacting in step (3) takes place~~ for 30 minutes; ~~and/or~~

~~24. The method of any one of the preceding claims, wherein~~ (b) the reacting in step (3) takes place at a temperature of between 55°C and 65°C, or~~.~~

~~25. The method of any one of the preceding claims, wherein the reacting in step (3) takes place~~ at a temperature of 60°C; and/or ~~.~~

~~26. The method of any one of the preceding claims, wherein~~ (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 ~~.~~

~~27. The method of any one of the preceding claims, wherein in step (3), each molar equivalent (eq.) of said compound of said general Formula (XII) is reacted~~ with at least 4.5 molar equivalents (eq.) of said polyethylene glycol (PEG)~~.~~, or

~~28. The method of any one of the preceding claims, wherein in step (3), each molar equivalent (eq.) of said compound of said general Formula (XII) is reacted~~ with at least 5.0 molar equivalents (eq.) of said polyethylene glycol (PEG)~~.~~; and/or

~~29. The method of any one of the preceding claims, wherein said polyethylene glycol (PEG) used in step (3) is characterized in that it is liquid at the reaction temperature of said step (3).~~

~~30. The method of any one of the preceding claims, wherein~~
(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.

~~31~~9. The method of any one of claims 2-~~30~~8, wherein the compound of formula (XII) obtained in step (2) is precipitated from a solvent~~.~~,

~~32. The method of claim 31,~~ optionally wherein:
(a) the solvent is an alcohol solvent~~.~~; and/or

~~33. The method of any one of claims 31-32, wherein~~
(b) the solvent is a polar solvent, optionally wherein the solvent is acrylonitrile; and/or~~.~~

~~34. The method of any one of claims 32-33, wherein~~
(c) the solvent is an alcohol solvent ~~is~~ selected from the group consisting of isopropyl alcohol, ethanol, methanol and butanol or combinations thereof; and/or~~.~~

~~35. The method of any one of claims 32-34, wherein~~

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

~~36. The method of any one of claims 32-35, wherein~~

(e) the ~~alcohol~~ solvent is isopropyl alcohol.

~~37. The method of any one of claims 32-33, wherein the solvent is acetonitrile.~~

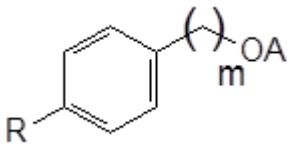
~~38~~10. The method of any one of claims 2-~~37~~9, wherein at least one recrystallization of said compound of Formula (XII) is carried out in between step (2) and step (3). .

~~39. The method of claim 38,~~ optionally wherein the solvent of ~~any one of~~ claim~~s 31 to 37~~ 9 is employed as solvent for the recrystallization between step 2 and step 3.

~~40. The method of any one of the preceding claims, wherein at least 1, at least 2, at least 3 or at least 4 aqueous washes are carried out after step 3.~~

~~41. The method of any one of the preceding claims, wherein 4 to 6 aqueous washes are carried out after step 3.~~

~~42~~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.

4312. The method of claim 4211, wherein:

(a) said solvent selected from alkanes, ethers and esters and mixtures thereof is an ester, ~~.~~

~~44. The method of claim 43,~~optionally wherein said solvent selected from alkanes, ethers and esters and mixtures thereof is ethyl acetate; and/or~~.~~

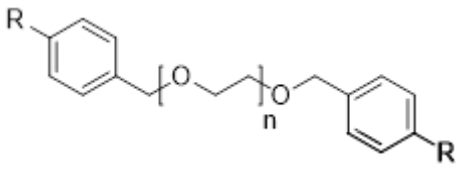
~~45. The method of any of claims 42 to 44, wherein~~

(b) the composition comprises polyethylene glycol (PEG)~~.~~,

~~46. The method of claim 45,~~ optionally wherein the purifying of the compound of formula (XIX) comprises removing the polyethylene glycol (PEG) from the composition, ~~.~~ further optionally

~~47. The method of claim 46,~~ wherein the removing of the polyethylene glycol (PEG) comprises said subjecting of the solution to the one or more aqueous washes.

4813. The method of ~~any one of~~ claims ~~42~~ 11 ~~to 47~~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 ~~42~~ 11 and n is an integer of n≥2.

4914. The method of claim 4813, wherein the purifying of the compound of formula (XIX) comprises removing the compound of formula (XIII) from the composition~~.~~,

~~50. The method of claim 49,~~ 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, ~~.~~

~~51. The method of claim 50,~~

further optionally wherein the non-polar solvent comprises an alkane, .

52. The method of claim 51, further optionally wherein the alkane is hexane, cyclohexane or heptane, .

53. The method of claim 52, further optionally wherein the alkane is cyclohexane.

5415. The method of any one of claims 1 to 5314, wherein:

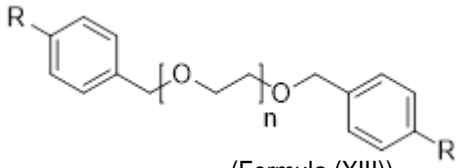
(a) the degree of purity of the compound of formula (XIX) as obtained by the method, as measured by high-performance liquid chromatography with UV detection at 200 nm, is at least 80%, preferably at least 85%, more preferably at least 90%, and most preferably at least 95%; and/or.

55. The method of any one of claims 1 to 54, wherein

(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

56. The method of any one of the preceding claims,

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

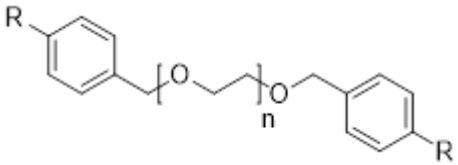


(Formula (XIII)),

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.

57. The method of any one of the preceding claims,

(d) the method further comprisesing 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.

~~58. The method of any one of the preceding claims,~~

(e) ~~wherein~~ A represents a polyoxyethylene residue comprising 4 to 16 oxyethylene units, or .

~~59. The method of any one of the preceding claims, wherein A represents a polyoxyethylene residue comprising~~ 8 to 10 oxyethylene units., or

~~60. The method of any one of the preceding claims, wherein A represents a polyoxyethylene residue comprising~~ 9 or 10 oxyethylene units; and/or.

~~61. The method of any one of the preceding claims,~~

(f) ~~wherein~~ 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.

~~62. The method of any one of the preceding claims, wherein R represents a hydrocarbon group having~~ a linear chain of 2 to 6 carbon atoms and 2 to 4 methyl groups as substituents on said linear chain, .or

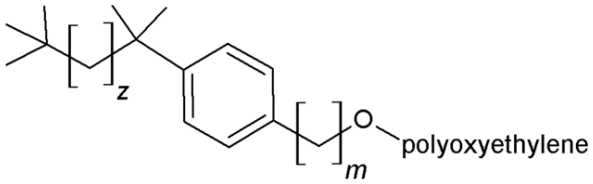
~~63. The method of any one of the preceding claims, wherein R represents a hydrocarbon group having~~ a linear chain of 4 carbon atoms and 4 methyl groups as substituents on said linear chain; and/or.

~~64. The method of any one of the preceding claims, wherein~~

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

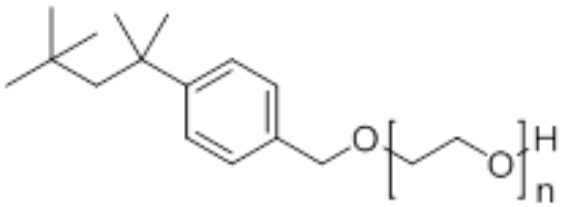
~~65. The method of any one of the preceding claims, wherein~~

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



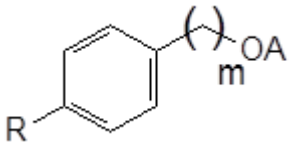
wherein m equals 1 and z is an integer selected from:
z = 1 to 5~~.~~; and/or

~~66. The method of any one of the preceding claims, wherein~~
(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.

~~67~~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.

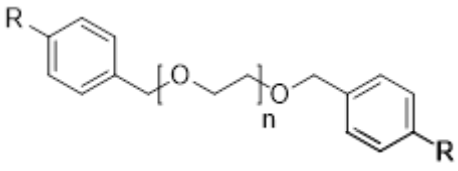
~~68~~17. The compound of claim ~~67~~16, which:

(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

69. The compound of claim 67 or 68,

(b) which 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%.

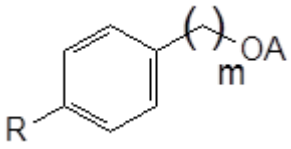
7018. Use of a non-polar solvent for extracting and removing a compound of the following general Formula (XIII):



(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.

7119. The use of claim 7018, wherein:

(a) the non-polar solvent is as defined in any one of claims 51-5314; and/or.

72. The use of claim 70 or 71, wherein

(b) A is as defined in any one of claims 58-6015.; and/or

~~73. The use of any one of claims 70 to 72, wherein~~

(c) R is as defined in ~~any one of~~ claim~~s 61-64~~ 15~~.~~; and/or

~~74. The use of any one of claims 70 to 73, wherein~~

(d) the compound of Formula (XIX) is as defined in ~~any one of~~ claim~~s 65-66~~15.