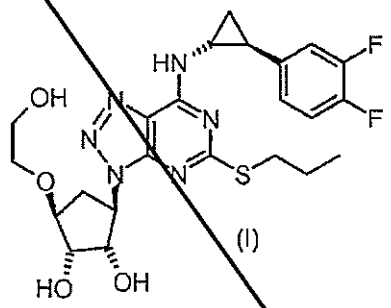
Claims

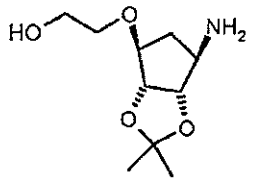
1. Process for preparing a compound of formula (I)



(I)

comprising

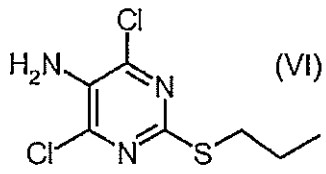
(a) reacting a compound of formula (II)



(II)

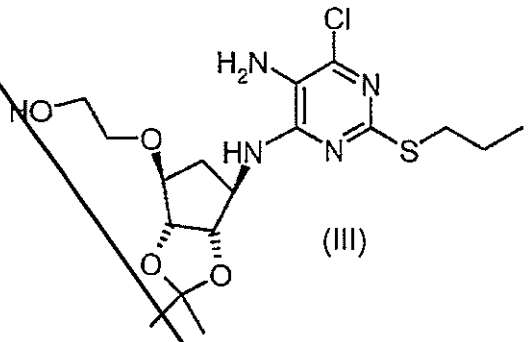
with oxalic acid or dibenzoyl-L-tartaric acid to form the oxalate salt or the dibenzoyl-L-tartrate salt of the compound of formula (II); and

(b) reacting the salt of the compound of formula (II) with a compound of formula (VI)



(VI)

in the presence of a tertiary amine at between 80° and 115°C at an oxygen concentration below 2.0% by volume, to obtain the compound of formula (III)

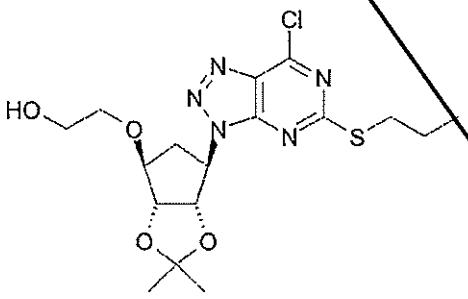


(III)

and isolating the compound of formula (III) in crystalline form;

and

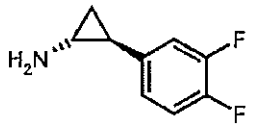
(c) reacting the compound of formula (III) with acetic acid and sodium nitrite at between 0°C and 40°C to obtain the compound of formula (IV)



(IV)

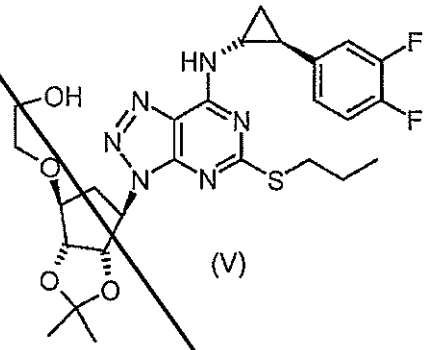
and

(d) reacting the compound of formula (IV) with a compound of formula (VII) at a temperature equal to or below 40°C



(VII)

to obtain the compound of formula (V)



(V)

and

(e) deprotecting the compound of formula (V) using aqueous hydrochloric acid in methanol in a two-phase system to obtain the compound of formula (I).

2. A process according to claim 1, wherein step (a) is carried out in ethanol or in a mixture of water and ethanol.

3. A process according to claim 1 or 2, wherein the compound of formula (II) is reacted with oxalic acid.

4. A process according to claim 3, wherein iso-propylacetate is added to the reaction mixture in step (a).

5. A process according to claim 1 or 2, wherein the compound of formula (II) is reacted with dibenzoyl-L-tartaric acid.

6. A process according to any one of claims 1-5, wherein the compound of formula (VI) is charged in excess in step (b).

7. A process according to claim 6, wherein the tertiary amine used in step (b) is triethylamine.

8. A process according to any one of claims 1-7, wherein the oxygen concentration is below 1.0% by volume in step (b).

9. A process according to claim 8, wherein the oxygen concentration is below 0.5% by volume in step (b).

10. A process according to any one of claims 1-9, wherein the solvent used in step (b) is ethanol, isopropyl alcohol or ethylene glycol.

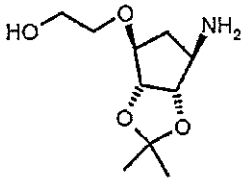
11. A process according to claim 10, wherein the solvent used in step (b) is ethylene glycol.

12. A process according to any one of claims 1-11, wherein step (c) is carried out at 20°C-30°C.

13. A process according to any one of claims 1-12, wherein the product of step (c) is used in the subsequent reaction step without isolation.

14 A process according to any one of claims 1-13, wherein the compound of formula (VII) is added to the reaction mixture in step (d) at a rate that keeps the reaction temperature at or below 30°C.

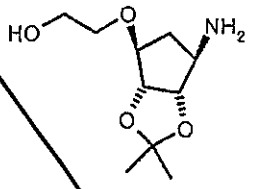
15. A process for preparing an oxalate salt or a dibenzoyl-L-tartrate salt of the compound of formula (II)



(II)

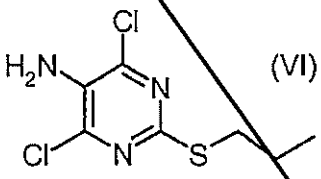
wherein the compound of formula (II) is reacted with oxalic acid or dibenzoyl-L-tartaric acid to form the oxalate salt or the dibenzoyl-L-tartrate salt.

16. Process for preparing a compound of formula (III) wherein an oxalate salt or a dibenzoyl-L-tartrate salt of the compound of formula (II)



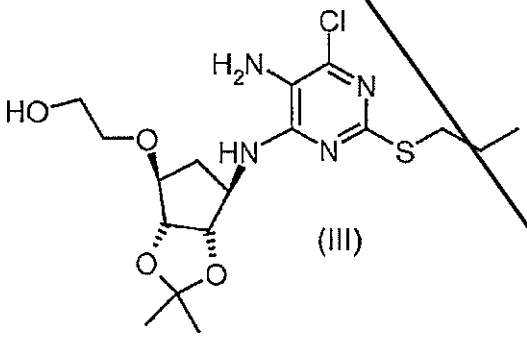
(II)

is reacted with a compound of formula (VI)



(VI)

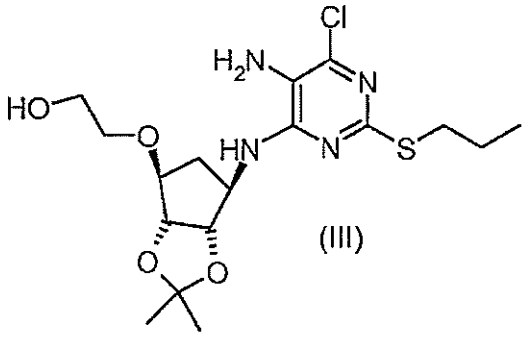
in the presence of a tertiary amine at between 80° and 115°C at an oxygen concentration below 2.0% by volume, to obtain the compound of formula (III)



(III)

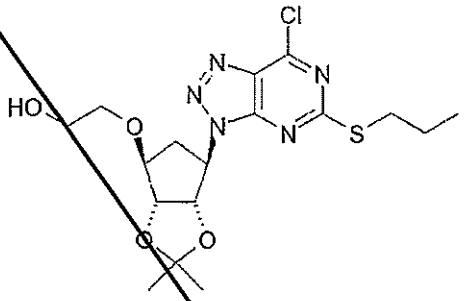
and isolating the compound of formula (III) in crystalline form.

17. Process for preparing a compound of formula (IV) wherein a compound of formula (III)



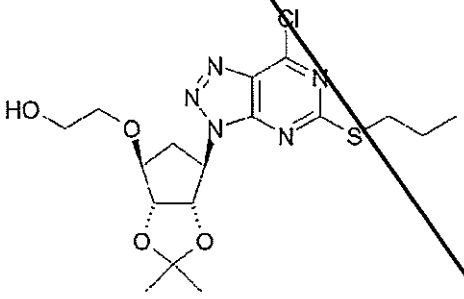
(III)

is reacted with acetic acid and sodium nitrite at between 0°C and 40°C to obtain the compound of formula (IV)



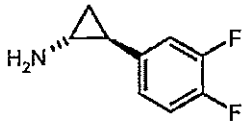
(IV).

18. Process for preparing a compound of formula (V) wherein a compound of formula (IV)



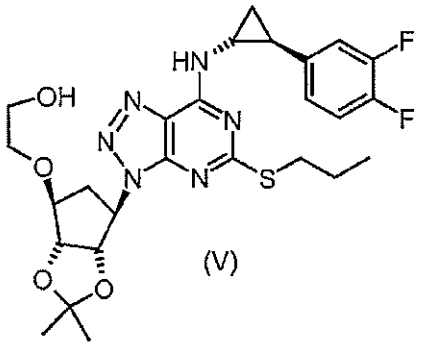
(IV)

is reacted with a compound of formula (VII) at a temperature equal to or below 40°C



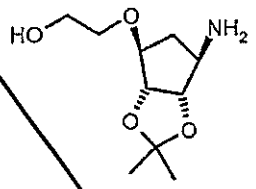
(VII)

to obtain the compound of formula (V)



(V)

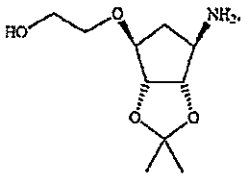
19. A compound selected from the oxalate salt or the dibenzoyl-L-tartrate salt of the compound of formula (II)



(II)

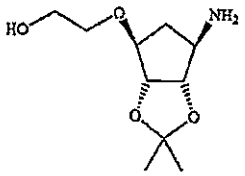
Claims

1. A compound selected from the oxalate salt or the dibenzoyl-L-tartrate salt of the compound of formula (II)



(II)

2. A process for preparing an oxalate salt or a dibenzoyl-L-tartrate salt of the compound of formula (II)



(II)

wherein the compound of formula (II) is reacted with oxalic acid or dibenzoyl-L-tartaric acid to form the oxalate salt or the dibenzoyl-L-tartrate salt.