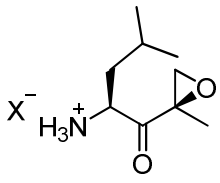
CLAIMS

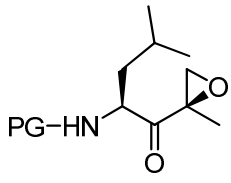
1. A method for the preparation of a crystalline compound of Formula (III),



(III)

wherein X is any suitable counterion, comprising

(i) preparing a solution of a compound of Formula (IV) in an organic solvent, wherein PG is a suitable protecting group



(IV)

(ii) adding a suitable acid;

(iii) bringing the solution to supersaturation to cause formation of crystals; and

(iv) isolating the crystals.

2. A method of claim 1, wherein PG is selected from Boc and Cbz and X is trifluoroacetate.

3. A method of claim 1 or 2, wherein the organic solvent is selected from dichloromethane, ethyl acetate, isopropyl acetate, isobutyl acetate, butyl acetate, propyl acetate, diethyl ether, methyl tert-butyl ether (MTBE), or any combination thereof.

4. A method of claim 3, wherein the organic solvent is selected from dichloromethane, ethyl acetate, MTBE, or any combination thereof.

5. A method of any one of claims 1 to 4, wherein bringing the solution to supersaturation comprises addition of an anti-solvent, allowing the solution to cool, reducing the volume of the solution, or any combination thereof.

6. A method of claim 5, wherein bringing the solution to supersaturation comprises adding an anti-solvent, cooling the solution to ambient temperature or lower, and reducing the volume of the solution.

7. A method of claim 5 or 6, wherein the anti-solvent is hexanes or heptanes.

8. A method of any one of claims 1 to 7, further comprising washing the crystals.

9. A method of claim 8, wherein the washing comprises washing with ~~a liquid selected from~~ an anti-solvent, dichloromethane, ethyl acetate, ~~isopropyl acetate, isobutyl acetate, butyl acetate, propyl acetate, diethyl ether, and methyl tert-butyl ether,~~ or any combination thereof.

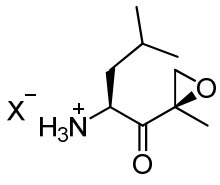
10. A method of claim 9, wherein washing comprises washing with anti-solvent.

11. A method of claim 10, wherein the anti-solvent is ~~hexanes or~~ heptanes.

12. A method of any one of claims 1 to 11, wherein isolating the crystals comprises filtering the crystals.

13. A method of any one of claims 1 to 12, further comprising drying the crystals under reduced pressure.

14. A crystalline compound having a structure of Formula (III)



(III)

wherein X is trifluoroacetate.

15. A crystalline compound of claim 14, having a DSC thermogram substantially as shown in Figure 9.

16. A crystalline compound of claim 14 or 15, having an XRPD pattern substantially as shown in Figure 10.

17. A crystalline compound of any one of claims 14 to 16, having 2θ values 8.84; 15.18; 15.32; 16.20; 16.82; 17.66; 18.26; 19.10; 21.20; 22.58; 23.06; 23.52; 25.32; 26.58; 28.60; 30.08; 30.48; 30.84; 32.20; 36.14; 37.12.