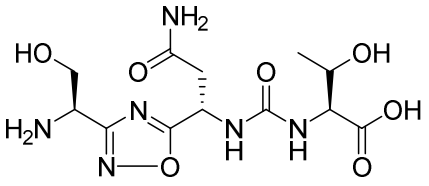
Claims:

1. An anhydrous crystalline compound having the structure of formula (I),



(I).

2. The anhydrous crystalline compound of claim 1, having 2θ values 18.8 ± 0.2, 20.5 ± 0.2, 26.4 ± 0.2, 27.5 ± 0.2, and 32.0 ± 0.2.

3. The anhydrous crystalline compound of claim 1 or 2, having 2θ values 18.8 ± 0.2, 20.5 ± 0.2, 26.1 ± 0.2, 26.4 ± 0.2, 27.5 ± 0.2, 30.6 ± 0.2, and 32.0 ± 0.2.

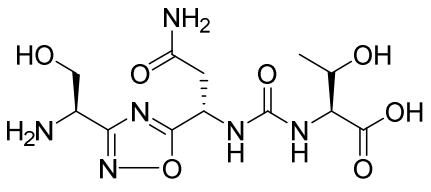
4. The anhydrous crystalline compound of any one of claims 1-3, having 2θ values 15.8 ± 0.2, 16.5 ± 0.2, 18.8 ± 0.2, 20.5 ± 0.2, 25.5 ± 0.2, 26.1 ± 0.2, 26.4 ± 0.2, 27.5 ± 0.2, 30.6 ± 0.2, and 32.0 ± 0.2.

5. The anhydrous crystalline compound of any one of claims 1-4, having 2θ values 15.8 ± 0.2, 16.5 ± 0.2, 18.8 ± 0.2, 20.5 ± 0.2, 21.8 ± 0.2, 22.3 ± 0.2, 24.2 ± 0.2, 25.5 ± 0.2, 26.1 ± 0.2, 26.4 ± 0.2, 27.5 ± 0.2, 30.4 ± 0.2, 30.6 ± 0.2, and 32.0 ± 0.2.

6. The anhydrous crystalline compound of any one of claims 1-5, having 2θ values 10.2 ± 0.2, 15.2 ± 0.2, 15.8 ± 0.2, 16.5 ± 0.2, 17.6 ± 0.2, 18.1 ± 0.2, 18.8 ± 0.2, 20.5 ± 0.2, 21.8 ± 0.2, 22.3 ± 0.2, 22.9 ± 0.2, 24.2 ± 0.2, 25.4 ± 0.2, 26.1 ± 0.2, 26.4 ± 0.2, 27.5 ± 0.2, 28.1 ± 0.2, 28.8 ± 0.2, 29.3 ± 0.2, 30.4 ± 0.2, 30.6 ± 0.2, 32.0 ± 0.2, 33.4 ± 0.2, 33.6 ± 0.2, 34.6 ± 0.2, 35.8 ± 0.2, 36.7 ± 0.2, 37.3 ± 0.2, 38.2 ± 0.2, 38.6 ± 0.2, and 39.2 ± 0.2.

7. A pharmaceutical composition comprising the anhydrous crystalline compound of any one of claims 1-6 and one or more pharmaceutically acceptable excipients.

8. A method for preparing an anhydrous crystalline compound having the structure of formula (I):



(I);

comprising the steps of:

a) providing a mixture comprising a compound of formula (I) and a solvent; and

b) crystallizing the compound of formula (I) from the mixture comprising the compound of formula (I).

9. The method of claim 8, wherein the method comprises the steps of:

a) providing a mixture comprising a compound of formula (I) and a solvent;

b) adding an anti-solvent to the mixture; and

c) crystallizing the compound of formula (I) from the mixture comprising the compound of formula (I).

10. The method of claim 8 or 9, wherein the method comprises the steps of:

a) providing a mixture comprising a compound of formula (I) and a solvent;

b) adding an anti-solvent to the mixture;

c) adding seed crystals to the mixture;

d) adding additional anti-solvent to the mixture; and

e) crystallizing the compound of formula (I) from the mixture comprising the compound of formula (I).

11. The method of any one of claims 8-10, wherein the solvent is selected from acetonitrile, anisole, dichloromethane, ethanol, isopropyl acetate, methyl tert-butyl ether (MTBE), n-heptane, tetrahydrofuran, water, and mixtures thereof.

12. The method of any one of claims 8-10, wherein the solvent is a mixture comprising water.

13. The method of claim 12, wherein the mixture further comprises a solvent selected from ethanol, isopropanol, methanol, tetrahydrofuran, 1,4-dioxane, acetonitrile, and acetone.

14. The method of claim 12, wherein the mixture further comprises a solvent selected from ethanol and isopropanol.

15. The method of any one of claims 8-14, wherein the mixture comprising the compound of formula (I) and the solvent is a reaction mixture.

16. The method of any one of claims 9-15, wherein, the anti-solvent is selected from tetrahydrofuran, methanol, isopropanol, diethyl ether, ethanol, 1,4-dioxane, acetonitrile, and acetone.

17. The method of any one of claims 9-16, wherein the anti-solvent is ethanol.

18. The method of any one of claims 8-17, wherein the mixture further comprises seed crystals.

19. The method of any one of claims 10-18, wherein the seed crystals are added at a weight percentage of the total mixture selected from about 1 wt%, about 2 wt%, about 3 wt%, about 4 wt%, about 5 wt%, about 6 wt%, about 7 wt%, about 8 wt%, about 9 wt%, and about 10 wt%.

20. The method of any one of claims 10-19, wherein the seed crystals are added at a weight percentage of the total mixture selected from about 1 wt%, about 2 wt%, about 3 wt%, about 4 wt%, about 5 wt%, and about 6 wt%.

21. The method of any one of claims 10-20, wherein the seed crystals have 2θ values 18.8 ± 0.2, 20.5 ± 0.2, 26.4 ± 0.2, 27.5 ± 0.2, and 32.0 ± 0.2.

22. The method of any one of claims 10-21, wherein the seed crystals are milled.

23. The method of any one of claims 8-22, wherein the mixture comprising the compound of formula (I) is a solution.

24. The method of claim 23, wherein the solution comprises a crude solid material comprising the compound of formula (I) dissolved in a solvent.

25. The method of claim 23, wherein the solution comprises a reaction mixture.

26. The method of any one of claims 8-25, wherein the mixture comprising the compound of formula (I) is a solution, and the step of crystallizing the compound of formula (I) from the mixture comprises bringing the solution to supersaturation to cause the compound of formula (I) to precipitate out of solution.

27. The method of claim 26, wherein the step of bringing the solution to supersaturation comprises slowly adding an anti-solvent, allowing the solution to cool, reducing the volume of the solution, or any combination thereof.

28. The method of claim 26, wherein the step of bringing the solution to supersaturation comprises slowly adding an anti-solvent.

29. The method of claim 26, wherein the step of bringing the solution to supersaturation comprises cooling the solution to ambient temperature or lower.

30. The method of claim 26, wherein the step of bringing the solution to supersaturation comprises maintaining a solution temperature at ambient temperature or higher.

31. The method of claim 30, wherein the step of bringing the solution to supersaturation comprises maintaining a solution temperature above about 20 °C.

32. The method of claim 30 or 31, wherein the step of bringing the solution to supersaturation comprises maintaining a solution temperature at about 20 °C, about 25 °C, about 30 °C, about 35 °C, about 40 °C, about 45 °C, about 50 °C, about 55 °C, about 60 °C, about 65 °C, or about 70 °C.

33. The method of any one of claims 8-22, wherein the mixture comprising the compound of formula (I) is a slurry.

34. The method of claim 33, wherein the slurry comprises crude solid material comprising the compound of formula (I).

35. The method of claim 33 or 34, wherein the slurry is maintained at ambient temperature or higher.

36. The method of any one of claims 33-35, wherein the slurry is maintained at a temperature above about 20 °C.

37. The method of any one of claims 33-36, wherein the slurry is maintained at a temperature of about 20 °C, about 25 °C, about 30 °C, about 35 °C, about 40 °C, about 45 °C, about 50 °C, about 55 °C, about 60 °C, about 65 °C, or about 70 °C.

38. The method of any one of claims 24, 26-32, and 34-37, wherein the crude solid material comprises 70% to 90% compound of formula (I).

39. The method of any one of claims 8-38, further comprising isolating the crystalline compound.

40. The method of claim 39, wherein isolating the crystalline compound comprises filtering the crystallized compound from the mixture.

41. The method of claim 39 or 40, further comprising drying the crystalline compound under reduced pressure.

42. The method of any one of claims 8-41, wherein the purity of the crystalline form of the compound of formula (I) is selected from about 90%, about 91%, about 92%, about 93%, about 94%, about 95%, about 96%, about 97%, about 98%, and about 99%.

43. The method of any one of claims 8-42, wherein the crystalline compound is the crystalline compound of any one of claims 1-6.