CLAIMS

1. A method of preparing crystalline Form A of 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3-carboxamide, comprising:

(a) mixing 2 grams of Compound (I), which is 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3-carboxamide, into 143 mL of THF and 7 mL of water until the Compound (I) is solubilized, thereby forming a solution;

(b) polish filtering the solution;

(c) drying the polish filtered solution to obtain a solid;

(d) suspending the solid in 12 mL of EtOAc to obtain a slurry;

(e) filtering the slurry to obtain a wet cake;

(f) washing the wet cake with 5 mL of EtOAc; and

(g) drying the washed wet cake to obtain crystalline Form A of 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3-carboxamide.

2. The method according to claim 1, wherein the mixing is performed at 25 °C.

3. The method according to claim 2, wherein the polish filtering is performed at 25 °C.

4. The method according to claim 3, wherein the suspending is performed at 60 °C, and the obtained slurry is aged at 60 °C prior to the filtering.

5. The method according to claim 4, wherein the drying in (g) is performed in a vacuum oven at a temperature of from 50 °C to 60 °C.

6. Crystalline Form A of 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3-carboxamide produced by the method according to any one of claims 1 to 5, having a purity of greater than 90 wt%.

7. A method of preparing crystalline Form A of 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3 carboxamide, comprising:

(a) adding 1.9 grams of 1-methylimidazole, 17 mL of ACN, 3.3 grams of ($^2H_3$)methanamine, 17.0 grams of 6-cyclopropaneamido-4-{[2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl]amino}pyridazine-3-carboxylic acid, hemi zinc salt, 17 mL of ACN, and 8.5 mL of NMP to 34 mL NMP, to obtain a solution;

(b) heating the solution;

(c) aging the heated solution;

(d) adding 3.3 g of HOBt and 10.3 grams of EDC HCl to the aged heated solution;

(e) aging the result of (d);

(f) adding 17 mL of ACN to the result of (e), to obtain a slurry;

(g) aging the slurry;

(h) adding 17 mL of water and 51 mL of ACN to the aged slurry;

(i) cooling the result of (h) to obtain a cold slurry;

(j) aging the cold slurry;

(k) filtering the aged cold slurry to obtain crystalline solids;

(1) washing the crystalline solids with 51 mL of 2:1 water:ACN followed by 51 mL of ACN to obtain a wet cake; and

(m) drying the wet cake to obtain crystalline Form A of 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3-carboxamide.

8. The method according to claim 7, wherein in (b), the solution is heated to 65 °C, and wherein in (c), the heated solution is aged for 1 hour at 65 °C.

9. The method according to claim 8, wherein in (e), the aging occurs for 15 minutes.

10. The method according to claim 9, wherein in (g), the slurry is aged for 30 minutes at 65 °C.

11. The method according to claim 10, wherein in (i), the result of (h) is cooled to -10 °C over 2 hours, and wherein in (j), the cold slurry is aged for 12 hours.

12. The method according to claim 11, wherein in (m), the drying is performed in a vacuum oven at a temperature of from 50 °C to 100 °C.

13. A method of preparing crystalline Form A of 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3 carboxamide, comprising:

(a) mixing 10 grams of Compound (I), which is 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3-carboxamide, into 55 mL of NMP, thereby forming a slurry;

(b) heating the slurry to obtain a solution of the Compound (I) in the NMP;

(c) polish filtering the solution to obtain a filtrate;

(d) heating the filtrate;

(e) adding 30 mL of IPA to the heated filtrate;

(f) allowing the temperature of the result of (e) to cool, and then adding seeds of Compound (I) in an amount of 1 wt%;

(g) aging the result of (f);

(h) adding 60 mL of IPA to the result of (g);

(i) cooling the result of (h) to obtain a cold slurry;

(j) aging the cold slurry;

(k) filtering the aged cold slurry to obtain crystalline solids;

(1) washing the crystalline solids twice with 40 mL of IPA each time, to obtain a wet cake; and

(m) drying the wet cake to obtain crystalline Form A of 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine 3-carboxamide.

14. The method according to claim 13, wherein in (b), the slurry is heated to 70 °C.

15. The method according to claim 14, wherein in (c), the polish filtering is performed at a temperature in the range of 62.5 °C to 85 °C.

16. The method according to claim 15, wherein in (d), the filtrate is heated to 70 °C, and wherein in (f), the temperature of the result of (e) is allowed to cool to 70 °C.

17. The method according to claim 16, wherein in (g), the result of (f) is aged for 1 hour at 70 °C.

18. The method according to claim 17, wherein in (h), the IPA is added over one hour.

19. The method according to claim 18, wherein in (i), the result of (h) is cooled to -10 °C over 3.5 hours, and wherein in (j), the cold slurry is aged at -10 °C for 12 hours.

20. The method according to claim 19, wherein in (m), the drying is performed in a vacuum oven at a temperature of from 50 °C to 100 °C.

21. Crystalline Form A of 6-(cyclopropanecarboxamido)-4-((2-methoxy-3-(1-methyl-1H-1,2,4-triazol-3-yl)phenyl) amino)-N-(methyl-d_3)pyridazine-3-carboxamide produced by the method according to any one of claims 13-20, having a purity of greater than 90 wt%.