

10. The polymorph form of Claim 8 ~~to~~or 9, wherein the X-ray powder diffraction pattern shown as in Figure 3.

11. The polymorph form of Claim 1, wherein the polymorph form is of Polymorph Form IV and its X-ray powder diffraction pattern having characteristic peaks at diffraction angles 2θ of 6.1° , 8.0° , 14.7° , 17.3° and $18.3^\circ \pm 0.2^\circ$.

12. The polymorph form of Claim 11, wherein its X-ray powder diffraction pattern having characteristic peaks at diffraction angles 2θ of 6.1° , 8.0° , 14.7° , 17.3° , 18.3° , 20.2° , 21.3° and $23.8^\circ \pm 0.2^\circ$.

13. The polymorph form of Claim 11 or 12, wherein the X-ray powder diffraction pattern shown as in Figure 4.

14. A process of preparing a polymorph form of Claim 1, comprising:

a). reactive crystallization of the Icotinib solution with solutions of phosphoric acid in THF, Dioxane, H_2O/THF , or $H_2O/Acetone$ to obtain the polymorph form of any one of Claims 2-4; or

b). reactive crystallization of the Icotinib solution with solutions of phosphoric acid in IPA, Acetone, ACN, 2-Butanone, or EtOH respectively to obtain the polymorph form of any one of Claims 5-7; or

c). at least one step chosen from reverse anti-solvent or anti-solvent crystallization from DMSO/EtOAc, vapor diffusion from DMSO/IPAc or DMSO/MTBE to obtain the polymorph form of any one of Claims 8-10; or

d). at least one step chosen from anti-solvent crystallization from DMF/DCM to obtain the polymorph form of any one of Claims 11-13.

15. The process of Claim 14, wherein:

(a). the temperature of step a), b), c) or d) is room temperature; or

(b). the volume ratio of H_2O and THF, or H_2O and Acetone of step a) ranges from 1:10 to 1:30; or

(c) the molar ratio between phosphoric acid and Icotinib of step a) or b) ranges from 1:1 to 2:1.

16. The process of Claim 14 or 15, wherein the molar ratio between phosphoric acid and Icotinib is 1:1.

17. The process of Claim 14, wherein the step c) or d), comprises:

(i). dissolving Icotinib phosphate in DMSO to obtain a saturated solution, adding EtOAc or to the saturated solution, stirring the resulting solution for at least 2hrs and isolating the polymorph form of any one of Claims 8-10; or

(ii). dissolving Icotinib phosphate in DMSO to obtain a saturated solution, adding the saturated solution into EtOAc, stirring the resulting solution for at least 2hrs and isolating the polymorph form of any one of Claims 8-10; or

(iii). dissolving Icotinib phosphate into DMSO to get a saturated solution in a first container, placing the first container in a second container containing IPAc or MTBE, and inducing precipitation to obtain polymorph form of any one of Claims 8-10; or

(iv) dissolving Icotinib phosphate in DMF to obtain a saturated solution, adding DCM to the saturated solution, stirring the resulting solution for at least 2hrs and isolating the polymorph form of any one of Claims 11-13.

18. A pharmaceutical composition comprising a therapeutically effective amount of polymorph form of any one of Claims 1-13 and a pharmaceutically acceptable excipient, adjuvant or carrier.

19. The pharmaceutical composition of Claim 18, wherein the polymorph form of any one of Claims 1-13 has a purity of $\geq 85\text{wt}\%$.

20. The pharmaceutical composition of Claim 18, wherein the polymorph form of any one of Claims 1-13 has a purity of $\geq 99\text{wt}\%$.

21. The pharmaceutical composition of any one of Claims 18-20, comprising at least one of additional active ingredient.

22. The pharmaceutical composition of any one of Claims 18-21, wherein the pharmaceutical composition is suitable for oral administration.

23. The pharmaceutical composition of any one of Claims 18-22, wherein the pharmaceutical composition is in a form of tablets or capsules.

24. The pharmaceutical composition of any one of Claims 18-23, wherein the composition comprises 0.01wt%-99wt% of the polymorph form of any one of Claims 1-13.

25. The pharmaceutical composition of Claim 24, wherein the composition comprises 10wt%-50wt% of the polymorph form of any one of Claims 1-13.

26. Use of the polymorph form of any one of Claims 1-13 or the pharmaceutical composition of any one of Claims 18-25 in the manufacture of ~~manufacturing~~ a medicament for the treatment or

prevention in mammals of an excessive non-malignant hyperplasia disease, pancreatitis, kidney disease, cancer, angiogenesis or vascular occurrence-related illness, or for the mammalian embryo cell transplantation.

27. The use of Claim 26, wherein the excessive non-malignant hyperplasia disease is benign skin hyperplasia or benign prostatic hyperplasia.

28. The use of Claim 26 or 27, wherein the excessive non-malignant hyperplasia disease, pancreatitis, kidney disease, cancer, angiogenesis or angiogenesis-related disease is selected from: tumor angiogenesis, chronic inflammatory diseases, skin diseases, diabetic retinopathy, premature retinopathy, age-related degeneration stains, hemangioma, glioma, Kaposi Internal tumor , ovarian cancer, breast cancer, lung cancer, pancreatic cancer, lymphoma, prostate, colon and skin tumors and their complications.

29. The use of ~~any one of~~ Claim 28, wherein the skin diseases is psoriasis, scleroderma, or diabetes-induced skin diseases; and the chronic inflammatory diseases is rheumatoid arthritis or atherosclerosis.

~~30. A method for treating a diseases with excessive proliferation of mammalian tissues, comprising administering to a patient with the disease with an therapeutically effective amount of the polymorph form of any of Claims 1-13 and/or the pharmaceutical composition according to any of Claims 18-25.~~

~~31. The method of Claim 30, further comprising administering to the patient at least one additional active ingredient chosen from matrix metalloproteinase inhibitors, vascular endothelial growth factor receptor kinase inhibitors, HER2 inhibitors, vascular endothelial growth factor receptor antibody drugs, or endostatin drugs.~~

~~32. The method of Claim 30 or 31, further comprising administering to the patient an anti-tumor agent selected from: mitotic inhibitors, alkylating agents, anti-metabolites, tumor antibiotics, growth factor inhibitors, cell cycle inhibitors, enzymes, enzyme inhibitors, biological response modifiers, or anti-hormones drugs.~~

~~33. A method for treating a disease related to tyrosine kinase dysfunction, comprising administering to a patient with the disease with a therapeutically effective amount of the polymorph form of any of claims 1-13 and/or the pharmaceutical composition according to any of Claims 18-25.~~

~~34. The method of Claim 33, the disease related to tyrosine kinase dysfunction is selected from brain, lung, liver, bladder, chest, head and neck, esophagus, gastrointestinal tract, breast, ovary, cervix or thyroid tumors and their complications.~~

~~35. The method of Claim 33 or 34, the disease is selected from brain cancer, lung cancer, kidney cancer, bone cancer, liver cancer, bladder cancer, breast, head and neck cancer, esophageal cancer, stomach, colon, rectum cancer, breast cancer, ovarian cancer, melanoma, skin cancer, adrenal cancer, cervical cancer, lymphoma, or thyroid tumors and their complications.~~

10. The polymorph form of Claim 8 or 9, wherein the X-ray powder diffraction pattern shown as in Figure 3.

11. The polymorph form of Claim 1, wherein the polymorph form is of Polymorph Form IV and its X-ray powder diffraction pattern having characteristic peaks at diffraction angles 2θ of 6.1° , 8.0° , 14.7° , 17.3° and $18.3^\circ \pm 0.2^\circ$.

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13. The polymorph form of Claim 11 or 12, wherein the X-ray powder diffraction pattern shown as in Figure 4.

14. A process of preparing a polymorph form of Claim 1, comprising:

a). reactive crystallization of the Icotinib solution with solutions of phosphoric acid in THF, Dioxane, $\text{H}_2\text{O}/\text{THF}$, or $\text{H}_2\text{O}/\text{Acetone}$ to obtain the polymorph form of any one of Claims 2-4; or

b). reactive crystallization of the Icotinib solution with solutions of phosphoric acid in IPA, Acetone, ACN, 2-Butanone, or EtOH respectively to obtain the polymorph form of any one of Claims 5-7; or

c). at least one step chosen from reverse anti-solvent or anti-solvent crystallization from DMSO/EtOAc, vapor diffusion from DMSO/IPAc or DMSO/MTBE to obtain the polymorph form of any one of Claims 8-10; or

d). at least one step chosen from anti-solvent crystallization from DMF/DCM to obtain the polymorph form of any one of Claims 11-13.

15. The process of Claim 14, wherein:

(a). the temperature of step a), b), c) or d) is room temperature; or

(b). the volume ratio of H_2O and THF, or H_2O and Acetone of step a) ranges from 1:10 to 1:30; or

(c) the molar ratio between phosphoric acid and Icotinib of step a) or b) ranges from 1:1 to 2:1.

16. The process of Claim 14 or 15, wherein the molar ratio between phosphoric acid and Icotinib is 1:1.

17. The process of Claim 14, wherein the step c) or d), comprises:

(i). dissolving Icotinib phosphate in DMSO to obtain a saturated solution, adding EtOAc or to the saturated solution, stirring the resulting solution for at least 2hrs and isolating the polymorph form of any one of Claims 8-10; or

(ii). dissolving Icotinib phosphate in DMSO to obtain a saturated solution, adding the saturated solution into EtOAc, stirring the resulting solution for at least 2hrs and isolating the polymorph form of any one of Claims 8-10; or

(iii). dissolving Icotinib phosphate into DMSO to get a saturated solution in a first container, placing the first container in a second container containing IPAc or MTBE, and inducing precipitation to obtain polymorph form of any one of Claims 8-10; or

(iv) dissolving Icotinib phosphate in DMF to obtain a saturated solution, adding DCM to the saturated solution, stirring the resulting solution for at least 2hrs and isolating the polymorph form of any one of Claims 11-13.

18. A pharmaceutical composition comprising a therapeutically effective amount of polymorph form of any one of Claims 1-13 and a pharmaceutically acceptable excipient, adjuvant or carrier.

19. The pharmaceutical composition of Claim 18, wherein the polymorph form of any one of Claims 1-13 has a purity of $\geq 85\text{wt}\%$.

20. The pharmaceutical composition of Claim 18, wherein the polymorph form of any one of Claims 1-13 has a purity of $\geq 99\text{wt}\%$.

21. The pharmaceutical composition of any one of Claims 18-20, comprising at least one of additional active ingredient.

22. The pharmaceutical composition of any one of Claims 18-21, wherein the pharmaceutical composition is suitable for oral administration.

23. The pharmaceutical composition of any one of Claims 18-22, wherein the pharmaceutical composition is in a form of tablets or capsules.

24. The pharmaceutical composition of any one of Claims 18-23, wherein the composition comprises $0.01\text{wt}\%$ - $99\text{wt}\%$ of the polymorph form of any one of Claims 1-13.

25. The pharmaceutical composition of Claim 24, wherein the composition comprises $10\text{wt}\%$ - $50\text{wt}\%$ of the polymorph form of any one of Claims 1-13.

26. Use of the polymorph form of any one of Claims 1-13 or the pharmaceutical composition of any one of Claims 18-25 in the manufacture of a medicament for the treatment or prevention in mammals of an excessive non-malignant hyperplasia disease, pancreatitis, kidney disease, cancer, angiogenesis or vascular occurrence-related illness, or for the mammalian embryo cell transplantation.

27. The use of Claim 26, wherein the excessive non-malignant hyperplasia disease is benign skin hyperplasia or benign prostatic hyperplasia.

28. The use of Claim 26 or 27, wherein the excessive non-malignant hyperplasia disease, pancreatitis, kidney disease, cancer, angiogenesis or angiogenesis-related disease is selected from: tumor angiogenesis, chronic inflammatory diseases, skin diseases, diabetic retinopathy, premature retinopathy, age-related degeneration stains, hemangioma, glioma, Kaposi Internal tumor , ovarian cancer, breast cancer, lung cancer, pancreatic cancer, lymphoma, prostate, colon and skin tumors and their complications.

29. The use of Claim 28, wherein the skin diseases is psoriasis, scleroderma, or diabetes-induced skin diseases; and the chronic inflammatory diseases is rheumatoid arthritis or atherosclerosis.