CLAIMS

1. A crystalline form of 2-((1H-pyrrolo[2,3-b]pyridin-5-yl)oxy)-N-((4-((((1r,4r)-4-hydroxy-4-methylcyclohexyl)methyl)amino)-3-nitrophenyl)sulfonyl)-4-(2-((S)-2-(2-isopropylphenyl)pyrrolidin-1-yl)-7-azaspiro[3.5]nonan-7-yl)benzamide (Compound 1) is an EtOAc solvate, containing about 1 mol of EtOAc per mol, said form is designated as Form A.

2. The crystalline form according to claim 1, wherein

i) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 16.5±0.1° and 24.5 ±0.1°; or

ii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 12.4±0.1°, 16.5±0.1° and 24.5 ±0.1°; or

iii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 12.4±0.1°, 16.5±0.1°, 20.7±0.1° and 24.5 ±0.1°; or

iv) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 10.6±0.1°, 12.4±0.1°, 16.5±0.1°, 20.7±0.1° and 24.5 ±0.1°; or

v) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 10.6±0.1°, 12.4±0.1°, 13.8±0.1°, 16.5±0.1°, 20.7±0.1° and 24.5 ±0.1°; or

vi) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 10.6±0.1°, 12.4±0.1°, 13.8±0.1°, 14.1±0.1°, 16.5±0.1°, 20.7±0.1° and 24.5 ±0.1°; or

vii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 10.6±0.1°, 12.4±0.1°, 13.8±0.1°, 14.1±0.1°, 16.5±0.1°, 17.0±0.1°, 20.7±0.1° and 24.5 ±0.1°; or

viii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 10.6±0.1°, 12.4±0.1°, 13.8±0.1°, 14.1±0.1°, 16.5±0.1°, 17.0±0.1°, 19.5±0.1°, 20.7±0.1° and 24.5 ±0.1°; or

ix) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 6.9±0.1°, 10.6±0.1°, 12.4±0.1°, 13.8±0.1°, 14.1±0.1°, 16.5±0.1°, 17.0±0.1°, 19.5±0.1°, 20.7±0.1° and 24.5 ±0.1°.

3. The crystalline form according to any one of claims 1-2, wherein Form A has an XRPD pattern substantially as shown in **Figure 1A** or **Figure 1E**.

4. The crystalline form according to any one of claims 1-2, wherein Form A is characterized by having two endotherm peaks at about 150 ºC and about 178 ºC by differential scanning calorimetry (DSC).

5. The crystalline form according to any one of claims 1-2, wherein Form A has a DSC thermogram substantially as shown in **Figure 1B**.

6. The crystalline form according to any one of claims 1-2, wherein the crystal system of Form A is triclinic and the space group is P1 having the cell parameters: (a) is about 13.644 Å, (b) is about 14.070 Å, (c) is about 15.012Å, (α) is about 112.0202(3)º, (β) is about 104.6821(3)º, and (γ) is about 93.6507(2)º.

7. A crystalline form of 2-((1H-pyrrolo[2,3-b]pyridin-5-yl)oxy)-N-((4-((((1r,4r)-4-hydroxy-4-methylcyclohexyl)methyl)amino)-3-nitrophenyl)sulfonyl)-4-(2-((S)-2-(2-isopropylphenyl)pyrrolidin-1-yl)-7-azaspiro[3.5]nonan-7-yl)benzamide (Compound 1) is an anhydrate, said form is designated as Form B.

8. The crystalline form according to claim 7, wherein

i) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 14.4±0.1°; or

ii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 14.4±0.1° and 17.5±0.1°; or

iii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 14.4±0.1°, 17.5±0.1° and 18.4±0.1°; or

iv) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 14.4±0.1°, 17.5±0.1°, 18.4±0.1° and 19.6±0.1°; or

v) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values independently selected from the group consisting of º2θ values at 7.2±0.1°, 14.4±0.1°, 17.5±0.1°, 18.4±0.1° and 19.6±0.1°; or

vi) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 6.7±0.1°, 7.2±0.1°, 13.8±0.1°, 14.4±0.1°, 17.5±0.1°, 18.4±0.1° and 19.6±0.1°; or

vii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 6.7±0.1°, 7.2±0.1°, 13.8±0.1°, 14.4±0.1°, 17.5±0.1°, 18.4±0.1° and 19.6±0.1°.

9. The crystalline form according to any one of claims 7-8, wherein Form B has an XRPD pattern substantially as shown in **Figure 2A** or **Figure 2D**.

10. The crystalline form according to any one of claims 7-8, wherein Form B is characterized by having two endotherm peaks at about 187 ºC by differential scanning calorimetry (DSC).

11. The crystalline form according to any one of claims 7-8, wherein Form B has a DSC thermogram substantially as shown in **Figure 2B**.

12. A crystalline form of 2-((1H-pyrrolo[2,3-b]pyridin-5-yl)oxy)-N-((4-((((1r,4r)-4-hydroxy-4-methylcyclohexyl)methyl)amino)-3-nitrophenyl)sulfonyl)-4-(2-((S)-2-(2-isopropylphenyl)pyrrolidin-1-yl)-7-azaspiro[3.5]nonan-7-yl)benzamide is an anhydrate, said form is designated as Form U.

13. The crystalline form according to claim 12, wherein

i) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 11.3±0.1° and 24.3±0.1°; or

ii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 11.3±0.1°, 15.6±0.1° and 24.3±0.1°; or

iii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 11.3±0.1°, 15.6±0.1°, 21.2±0.1° and 24.3±0.1; or

iv) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 11.3±0.1°, 13.5±0.1°, 15.6±0.1°, 21.2±0.1° and 24.3±0.1°; or

v) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 11.3±0.1°, 13.5±0.1°, 15.6±0.1°, 17.0±0.1°, 21.2±0.1° and 24.3±0.1°; or

vi) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 11.3±0.1°, 13.5±0.1°, 15.6±0.1°, 17.0±0.1°, 19.5±0.1°, 21.2±0.1° and 24.3±0.1°; or

vii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 7.0±0.1°, 11.3±0.1°, 13.5±0.1°, 15.6±0.1°, 17.0±0.1°, 19.5±0.1°, 21.2±0.1° and 24.3±0.1°; or

viii) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 7.0±0.1°, 11.3±0.1°, 13.5±0.1°, 15.6±0.1°, 17.0±0.1°, 19.5±0.1°, 20.0±0.1°, 21.2±0.1° and 24.3±0.1°; or

ix) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 7.0±0.1°, 9.4±0.1, 11.3±0.1°, 13.5±0.1°, 15.6±0.1°, 17.0±0.1°, 19.5±0.1°, 20.0±0.1°, 21.2±0.1° and 24.3±0.1°; or

x) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 7.0±0.1°, 9.4±0.1, 11.3±0.1°, 13.5±0.1°, 15.6±0.1°, 17.0±0.1°, 17.5±0.1°, 19.5±0.1°, 20.0±0.1°, 21.2±0.1° and 24.3±0.1°; or

xi) the crystalline form has an X-ray powder diffraction pattern comprising diffraction peaks having °2θ angle values at 7.0±0.1°, 9.4±0.1, 11.3±0.1°, 13.5±0.1°, 15.6±0.1°, 16.1±0.1°, 17.0±0.1°, 17.5±0.1°, 19.5±0.1°, 20.0±0.1°, 21.2±0.1°, 21.6±0.1° and 24.3±0.1°.

14. The crystalline form according to any one of claims 12-13, wherein Form U has an XRPD pattern substantially as shown in **Figure 21A**.

15. The crystalline form according to any one of claims 12-13, wherein Form U is characterized by having one endotherm peak at about 171 ºC by differential scanning calorimetry (DSC).

16. The crystalline form according to any one of claims 12-13, wherein Form U has a DSC thermogram substantially as shown in **Figure 21B**.

17. An amorphous form of 2-((1H-pyrrolo[2,3-b]pyridin-5-yl)oxy)-N-((4-((((1r,4r)-4-hydroxy-4-methylcyclohexyl)methyl)amino)-3-nitrophenyl)sulfonyl)-4-(2-((S)-2-(2-isopropylphenyl)pyrrolidin-1-yl)-7-azaspiro[3.5]nonan-7-yl)benzamide (**Compound 1**).

18. A pharmaceutical composition, comprising (a) a therapeutically effective amount of a crystalline form according to any one of claims 1-16, or an amorphous form of Compound 1 of claim 17, and; (b) one or more one pharmaceutically acceptable excipient.

19. Use of a crystalline form according to any one of claims 1-16, or an amorphous form of Compound 1 of claim 17, or a pharmaceutical composition of claim 18 in the preparation of a medicament for the treatment of disease related to Bcl-2 protein inhibition.

20. The crystalline form according to any one of claims 1-6, obtained by the process comprising any one of the following procedures:

a) dissolving Compound 1 in DCM, removing DCM, charging with EA, to obtain Form A;

b) dissolving Compound 1 in DCM, concentrating, charging with EA, exchanging DCM with EA, MeOH and EA separately, to obtain Form A;

c) dissolving Compound 1 in EA, heating and cooling, to obtain Form A; or,

d) dissolving Compound 1 in THF/EtOAc (1:2, v/v) solvent mixture, evaporating, to obtain Form A.

21. The crystalline form according to any one of claims 7-11, obtained by the process comprising any one of the following procedures:

a) dissolving Compound 1 in acetone, evaporating the solvent, to obtain Form B;

b) heating Form A, Form C, Form O to about 160 ºC and cooling, to obtained Form B;

c) heating Form A stepwise isothermally to about 100 ºC, to obtained Form B;

d) heating Form D or Form J to about 130 ºC and being isothermal, to obtained Form B; or,

c) adding Form K into heptane, heating to about 100 ºC and cooling, to obtain Form B.

22. The crystalline form according to any one of claims 12-16, obtained by the process comprising any one of the following procedures:

a) dissolving Compound 1 in DCM, adding n-heptane in batches and stirring, to obtain Form U; or,

b) dissolving Compound 1 in the mixture of DCM/n-heptane (1:1, v/v) and stirring, to obtain the Form U.

23. The amorphous form according to claim 17, obtained by the process comprising any one of the following procedures:

a) dissolving Compound 1 in DCM, drying, to obtain the amorphous form; or,

b) dissolving Compound 1 in a mixture of solvent containing DCM, drying, to obtain the amorphous form.