

PARG INHIBITOR**CROSS-REFERENCES TO RELATED APPLICATIONS**

[0001] This application claims the benefit of priority under 35 U.S.C. § 119(e) to U.S. Provisional Application No. 63/584,035 filed September 20, 2023 and U.S. Provisional Application No. 63/605,189 filed December 01, 2023, the contents of each are incorporated by reference in their entirety for all purposes.

**STATEMENT AS TO RIGHTS TO INVENTIONS MADE UNDER
FEDERALLY SPONSORED RESEARCH AND DEVELOPMENT**

[0002] NOT APPLICABLE

10 **REFERENCE TO A "SEQUENCE LISTING," A TABLE, OR A COMPUTER
PROGRAM LISTING APPENDIX SUBMITTED ON A COMPACT DISK**

[0003] NOT APPLICABLE

BACKGROUND

[0004] Cancer is caused by uncontrolled and unregulated cellular proliferation. The consequence of this often-rapid proliferation is a high level of oxidative stress within the tumor which damages DNA and leads to a much-increased mutation rate. Tumor cells therefore engage and rely heavily upon DNA damage repair mechanisms.

[0005] Single-strand breaks (SSBs) are the most common type of lesion arising in cells and PARG (Poly ADP-ribose glycohydrolase) together with PARP (poly ADP-ribose polymerase) is involved along with a number of other proteins in single strand break repair (SSBR) and another repair mechanism called base excision repair (BER).

[0006] One of the earliest events during single strand DNA repair is the binding of PARP (poly ADP-ribose polymerase) to the break and the rapid synthesis of poly ADP-ribose (PAR) on PARP itself. This molecular structure serves as a signal to recruit other DNA repair proteins, initially XRCC1, which will then repair the break (Mortusewicz, Fouquerel et al. 2011). The signal initiated by these PAR chains is short-lived as they are rapidly degraded by the enzyme PARG. When PARP is bound to PAR, its catalytic activity is reduced and

therefore PARG activity helps to restore PARP to its catalytically active form (Curtin and Szabo 2013).

5 [0007] PARG is derived from a single gene with isoforms that reside in the nucleus, mitochondria and cytosol. Another known protein with glycohydrolase activity is ARH3 which is localized to the mitochondria (Mashimo, Kato et al. 2014). Although, known primarily for its direct role in DNA repair, PARG impacts PAR signaling in splicing, transcriptional and epigenetic pathways (Ji and Tulin 2009) (Le May, Iltis et al. 2012) (Dahl, Maturi et al. 2014) (Guastafierro, Catizone et al. 2013) (Caiafa, Guastafierro et al. 2009).

10 [0008] Cancer cells may become reliant upon a specific DNA repair pathway when other mechanisms of DNA repair are non-functional. Tumors carrying mutations in proteins involved in double strand break repair are often more sensitive to PARP inhibitors of SSBR. There is already some evidence that PARG depletion inhibits SSBR and reduces survival of BRCA2-deficient cells (Fathers, Drayton et al. 2012). However, other tumor mutations may give rise to deficiencies in double strand DNA repair mechanisms (so-called "BRCA-ness")
15 thereby sensitizing tumor cells to PARG inhibition.

[0009] PARG depletion has been studied in a number of murine and human model systems. Murine cells that are null or depleted for PARG display an increased sensitivity to experimental and clinical DNA damaging agents. However, as deficiency in PARG doesn't sensitize to all agents (e.g. gemcitabine, camptothecin) this suggests a specificity for PARG
20 function with certain pathways of DNA damage repair and chemo- and radiotherapies (Fujihara, Ogino et al. 2009) (Shirai, Fujimori et al. 2013) (Zhou, Feng et al. 2010) (Zhou, Feng et al. 2011).

[0010] In humans, PARG depletion sensitizes lung, cervical and pancreatic cancer cells to γ - irradiation or experimental DNA damaging agents (e.g. hydrogen peroxide,
25 Methylmethanesulfonate) (Ame, Fouquerel et al. 2009) (Nakadate, Kodera et al. 2013) (Shirai, Poetsch et al. 2013).

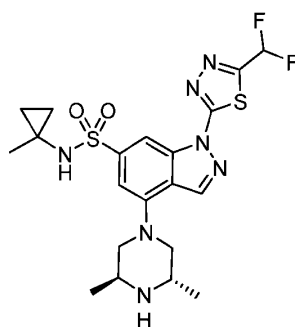
[0011] PARP inhibitors are currently undergoing multiple clinical trials where the concept of synthetic lethality or chemo-sensitization is being explored. Clinical resistance to PARP inhibitors has already been described (Drost and Jonkers 2014) (Barber, Sandhu et al. 2013)
30 and therefore there is a requirement that alternative inhibitors targeting the DNA damage repair machinery are found.

[0012] Although current models show that PARG depletion leads to PARP-dependent effects on DNA repair, recent research has shown a mechanistic differentiation from PARP inhibition. Following a genotoxic stimulus depletion of PARG, in contrast to PARP depletion, leads to a drop in NAD levels. This leads to lung cancer cell death as a result of energy failure (Erdelyi, Bai et al. 2009).

[0013] These results indicate that PARG inhibitors and their polymorphic forms may provide a useful therapy for cancer. The present disclosure addresses these needs and provides related advantages as well.

BRIEF SUMMARY

[0014] The present disclosure provides salt and free base crystalline forms of 1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-((3S,5S)-3,5-dimethylpiperazin-1-yl)-N-(1-methylcyclopropyl)-1H-indazole-6-sulfonamide (Compound 1), represented by the formula:



(Compound 1).

[0015] In some aspects, provided herein is an HCl salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is HCl salt Form I. In some embodiments, the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.

[0016] In some aspects, provided herein is a free base of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is free base Form I. In some embodiments, provided herein is a crystalline form of Compound 1 which is free base Form II. In some embodiments, the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.

[0017] In some aspects, provided herein is a sulfate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is sulfate salt Form I. In some embodiments, the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.

5 [0018] In some aspects, provided herein is a tosylate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is tosylate salt Form I. In some embodiments, provided herein is a crystalline form of Compound 1 which is tosylate salt Form II. In some embodiments, provided herein is a crystalline form of Compound 1 which is tosylate salt Form III. In some embodiments, the crystalline form is a
10 single crystalline form substantially free of other crystalline or amorphous forms.

[0019] In some aspects, provided herein is a besylate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is besylate salt Form I. In some embodiments, provided herein is a crystalline form of Compound 1 which is besylate salt Form II. In some embodiments, the crystalline form is a single crystalline form
15 substantially free of other crystalline or amorphous forms.

[0020] In some aspects, provided herein is a mesylate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is mesylate salt Form I. In some embodiments, provided herein is a crystalline form of Compound 1 which is mesylate salt Form II. In some embodiments, provided herein is a crystalline form of
20 Compound 1 which is mesylate salt Form III. In some embodiments, the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.

[0021] In some aspects, provided herein is a maleate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is maleate salt Form I. In some embodiments, provided herein is a crystalline form of Compound 1 which is
25 maleate salt Form II. In some embodiments, the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.

[0022] In some aspects, provided herein is a citrate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is citrate salt Form I. In some embodiments, the crystalline form is a single crystalline form substantially free of
30 other crystalline or amorphous forms.

[0023] In some aspects, provided herein is a fumarate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is fumarate salt Form I. In some embodiments, provided herein is a crystalline form of Compound 1 which is fumarate salt Form II. In some embodiments, provided herein is a crystalline form of
5 Compound 1 which is fumarate salt Form III. In some embodiments, the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.

[0024] In some aspects, provided herein is a malate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is malate salt Form I. In some embodiments, provided herein is a crystalline form of Compound 1 which is
10 malate salt Form II. In some embodiments, the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.

[0025] In some aspects, provided herein is a succinate salt of Compound 1. In some embodiments, provided herein is a crystalline form of Compound 1 which is succinate salt Form I. In some embodiments, the crystalline form is a single crystalline form substantially
15 free of other crystalline or amorphous forms.

[0026] Each of the provided crystalline forms can be further characterized as described herein.

[0027] In further aspects, provided herein are various solid state forms of Compound 1, comprising at least 50 percent by weight (wt%), 60 wt%, 70 wt%, 80 wt%, 90 wt%, 95 wt%,
20 or 99 wt% of a particular crystalline form as described herein.

[0028] In further aspects, provided herein are pharmaceutical compositions of the crystalline forms of Compound 1 described herein.

[0029] In further aspects, provided herein are pharmaceutical compositions comprising a crystalline form of Compound 1 described herein or pharmaceutical compositions prepared
25 using a crystalline form of Compound 1 described herein. Also provided are pharmaceutical compositions comprising a solid state form as described herein, or a pharmaceutical compositions prepared using a solid state form described herein. Pharmaceutical compositions will include one or more pharmaceutically acceptable excipients.

[0030] In additional aspects, provided herein are methods of inhibiting PARG *in vivo* in a
30 patient, comprising administering to the patient an effective amount of a crystalline form of

Compound 1 described herein. In additional aspects, provided herein are methods of inhibiting PARG *in vivo* in a patient, comprising administering to the patient an effective amount of a solid state form of Compound 1 described herein.

5 [0031] In additional aspects, provided herein are methods of inhibiting PARG *in vivo* in a patient, comprising administering to the patient an effective amount of a pharmaceutical composition described herein, wherein the pharmaceutical composition i) comprises a crystalline form of Compound 1 described herein, ii) is prepared using a crystalline form of Compound 1 described herein, iii) comprises a solid state form as described herein, or iv) is prepared using a solid state form described herein.

10 [0032] In additional aspects, provided herein are methods of inhibiting cell proliferation, *in vitro* or *in vivo*, the method comprising contacting a cell with an effective amount of a crystalline form of Compound 1 described herein. In additional aspects, provided herein are methods of inhibiting cell proliferation, *in vitro* or *in vivo*, the method comprising contacting a cell with an effective amount of a solid state form of Compound 1 described herein.

15 [0033] In additional aspects, provided herein are methods of inhibiting cell proliferation, *in vitro* or *in vivo*, the method comprising contacting a cell with an effective amount of a pharmaceutical composition described herein, wherein the pharmaceutical composition i) comprises a crystalline form of Compound 1 described herein, ii) is prepared using a crystalline form of Compound 1 described herein, iii) comprises a solid state form as
20 described herein, or iv) is prepared using a solid state form described herein.

[0034] In additional aspects, provided herein are methods of treating a cancer resistant to one or more platins or one or more PARP inhibitors in a patient in need thereof, comprising administering to the patient an effective amount of a crystalline form of Compound 1 described herein. In additional aspects, provided herein are methods of treating a cancer
25 resistant to one or more platins or one or more PARP inhibitors in a patient in need thereof, comprising administering to the patient an effective amount of a solid state form of Compound 1 described herein.

[0035] In additional aspects, provided herein are methods of treating a cancer resistant to one or more platins or one or more PARP inhibitors in a patient in need thereof, comprising
30 administering to the patient an effective amount of a pharmaceutical composition described herein, wherein the pharmaceutical composition i) comprises a crystalline form of Compound 1 described herein, ii) is prepared using a crystalline form of Compound 1 described herein,

iii) comprises a solid state form as described herein, or iv) is prepared using a solid state form described herein.

5 **[0036]** In additional aspects, provided herein are methods of treating and/or preventing a homologous recombinant deficient (HRD) cancer in a patient, comprising administering to the patient an effective amount of a crystalline form of Compound 1 described herein. In additional aspects, provided herein are methods of treating and/or preventing a homologous recombinant deficient (HRD) cancer in a patient, comprising administering to the patient an effective amount of a solid state form of Compound 1 described herein.

10 **[0037]** In additional aspects, provided herein are methods of treating and/or preventing a homologous recombinant deficient (HRD) cancer in a patient, comprising administering to the patient an effective amount of a pharmaceutical composition described herein, wherein the pharmaceutical composition i) comprises a crystalline form of Compound 1 described herein, ii) is prepared using a crystalline form of Compound 1 described herein, iii) comprises a solid state form as described herein, or iv) is prepared using a solid state form described herein.

15 **[0038]** In additional aspects, provided herein are methods of treating and/or preventing a cancer in a patient, where the cancer is characterized by a reduction or absence of BRCA1 and/or BRCA2 gene expression, the absence or mutation of BRCA1 and/or BRCA2 genes, or reduced function of BRCA1 and/or BRCA2 proteins, comprising administering to the patient an effective amount of a crystalline form of Compound 1 described herein. In additional
20 aspects, provided herein are methods of treating and/or preventing a cancer in a patient, where the cancer is characterized by a reduction or absence of BRCA1 and/or BRCA2 gene expression, the absence or mutation of BRCA1 and/or BRCA2 genes, or reduced function of BRCA1 and/or BRCA2 proteins, comprising administering to the patient an effective amount of a solid state form of Compound 1 described herein.

25 **[0039]** In additional aspects, provided herein are methods of treating and/or preventing a cancer in a patient, where the cancer is characterized by a reduction or absence of BRCA1 and/or BRCA2 gene expression, the absence or mutation of BRCA1 and/or BRCA2 genes, or reduced function of BRCA1 and/or BRCA2 proteins, comprising administering to the patient an effective amount of a pharmaceutical composition described herein, wherein the
30 pharmaceutical composition i) comprises a crystalline form of Compound 1 described herein, ii) is prepared using a crystalline form of Compound 1 described herein, iii) comprises a solid state form as described herein, or iv) is prepared using a solid state form described herein.

[0040] In additional aspects, the crystalline forms provided herein and pharmaceutical compositions thereof are useful in the treatment of cancer.

[0041] In additional aspects, provided herein are uses of the crystalline forms provided herein and pharmaceutical compositions thereof for the manufacture of a medicament for
5 treating cancer.

BRIEF DESCRIPTION OF THE DRAWINGS

[0042] FIG. 1 shows the X-ray powder diffraction (XRPD) pattern of free base Form I of Compound 1.

[0043] FIG. 2 shows the differential scanning calorimetry (DSC) thermogram as well as
10 the thermal gravimetric analysis (TGA) of free base Form I of Compound 1.

[0044] FIG. 3 shows a representative polarized light microscopy (PLM) image of free base Form I of Compound 1.

[0045] FIG. 4 shows the X-ray powder diffraction (XRPD) pattern of free base Form II of Compound 1.

[0046] FIG. 5 shows the differential scanning calorimetry (DSC) thermogram as well as
15 the thermal gravimetric analysis (TGA) of free base Form II of Compound 1.

[0047] FIG. 6 shows a representative polarized light microscopy (PLM) image of free base Form II of Compound 1.

[0048] FIG. 7 shows the X-ray powder diffraction (XRPD) pattern of HCl salt Form I of
20 Compound 1.

[0049] FIG. 8 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of HCl salt Form I of Compound 1.

[0050] FIG. 9 shows dynamic vapor sorption (DVS) plot of HCl salt Form I of Compound 1.

[0051] FIG. 10 shows a representative polarized light microscopy (PLM) image of HCl salt Form I of Compound 1.

[0052] FIG. 11 shows the X-ray powder diffraction (XRPD) pattern of sulfate salt Form I of Compound 1.

- [0053] FIG. 12 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of sulfate salt Form I of Compound 1.
- [0054] FIG. 13 shows a representative polarized light microscopy (PLM) image of sulfate salt Form I of Compound 1.
- 5 [0055] FIG. 14 shows the X-ray powder diffraction (XRPD) pattern of tosylate salt Form I of Compound 1.
- [0056] FIG. 15 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of tosylate salt Form I of Compound 1.
- [0057] FIG. 16 shows a representative polarized light microscopy (PLM) image of tosylate
10 salt Form I of Compound 1.
- [0058] FIG. 17 shows the X-ray powder diffraction (XRPD) pattern of tosylate salt Form II of Compound 1.
- [0059] FIG. 18 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of tosylate salt Form II of Compound 1.
- 15 [0060] FIG. 19 shows a representative polarized light microscopy (PLM) image of tosylate salt Form II of Compound 1.
- [0061] FIG. 20 shows the X-ray powder diffraction (XRPD) pattern of tosylate salt Form III of Compound 1.
- [0062] FIG. 21 shows the differential scanning calorimetry (DSC) thermogram as well as
20 the thermal gravimetric analysis (TGA) of tosylate salt Form III of Compound 1.
- [0063] FIG. 22 shows dynamic vapor sorption (DVS) plot of tosylate salt Form III of Compound 1.
- [0064] FIG. 23 shows a representative polarized light microscopy (PLM) image of tosylate salt Form III of Compound 1.
- 25 [0065] FIG. 24 shows the X-ray powder diffraction (XRPD) pattern of besylate salt Form I of Compound 1.
- [0066] FIG. 25 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of besylate salt Form I of Compound 1.

- [0067] FIG. 26 shows a representative polarized light microscopy (PLM) image of besylate salt Form I of Compound 1.
- [0068] FIG. 27 shows the X-ray powder diffraction (XRPD) pattern of besylate salt Form II of Compound 1.
- 5 [0069] FIG. 28 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of besylate salt Form II of Compound 1.
- [0070] FIG. 29 shows a representative polarized light microscopy (PLM) image of besylate salt Form II of Compound 1.
- [0071] FIG. 30 shows the X-ray powder diffraction (XRPD) pattern of mesylate salt Form
10 I of Compound 1.
- [0072] FIG. 31 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of mesylate salt Form I of Compound 1.
- [0073] FIG. 32 shows dynamic vapor sorption (DVS) plot of mesylate salt Form I of Compound 1.
- 15 [0074] FIG. 33 shows a representative polarized light microscopy (PLM) image of mesylate salt Form I of Compound 1.
- [0075] FIG. 34 shows the X-ray powder diffraction (XRPD) pattern of mesylate salt Form II of Compound 1.
- [0076] FIG. 35 shows the differential scanning calorimetry (DSC) thermogram as well as
20 the thermal gravimetric analysis (TGA) of mesylate salt Form II of Compound 1.
- [0077] FIG. 36 shows a representative polarized light microscopy (PLM) image of mesylate salt Form II of Compound 1.
- [0078] FIG. 37 shows the X-ray powder diffraction (XRPD) pattern of mesylate salt Form III of Compound 1.
- 25 [0079] FIG. 38 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of mesylate salt Form III of Compound 1.
- [0080] FIG. 39 shows a representative polarized light microscopy (PLM) image of mesylate salt Form III of Compound 1.

- [0081] FIG. 40 shows the X-ray powder diffraction (XRPD) pattern of maleate salt Form I of Compound 1.
- [0082] FIG. 41 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of maleate salt Form I of Compound 1.
- 5 [0083] FIG. 42 shows a representative polarized light microscopy (PLM) image of maleate salt Form I of Compound 1.
- [0084] FIG. 43 shows the X-ray powder diffraction (XRPD) pattern of maleate salt Form II of Compound 1.
- [0085] FIG. 44 shows the differential scanning calorimetry (DSC) thermogram as well as
10 the thermal gravimetric analysis (TGA) of maleate salt Form II of Compound 1.
- [0086] FIG. 45 shows a representative polarized light microscopy (PLM) image of maleate salt Form II of Compound 1.
- [0087] FIG. 46 shows the X-ray powder diffraction (XRPD) pattern of citrate salt Form I of Compound 1.
- 15 [0088] FIG. 47 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of citrate salt Form I of Compound 1.
- [0089] FIG. 48 shows a representative polarized light microscopy (PLM) image of citrate salt Form I of Compound 1.
- [0090] FIG. 49 shows the X-ray powder diffraction (XRPD) pattern of fumarate salt Form
20 I of Compound 1.
- [0091] FIG. 50 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of fumarate salt Form I of Compound 1.
- [0092] FIG. 51 shows a representative polarized light microscopy (PLM) image of fumarate salt Form I of Compound 1.
- 25 [0093] FIG. 52 shows the X-ray powder diffraction (XRPD) pattern of fumarate salt Form II of Compound 1.
- [0094] FIG. 53 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of fumarate salt Form II of Compound 1.

- [0095] FIG. 54 shows a representative polarized light microscopy (PLM) image of fumarate salt Form II of Compound 1.
- [0096] FIG. 55 shows the X-ray powder diffraction (XRPD) pattern of fumarate salt Form III of Compound 1.
- 5 [0097] FIG. 56 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of fumarate salt Form III of Compound 1.
- [0098] FIG. 57 shows a representative polarized light microscopy (PLM) image of fumarate salt Form III of Compound 1.
- [0099] FIG. 58 shows the X-ray powder diffraction (XRPD) pattern of malate salt Form I
10 of Compound 1.
- [0100] FIG. 59 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of malate salt Form I of Compound 1.
- [0101] FIG. 60 shows a representative polarized light microscopy (PLM) image of malate salt Form I of Compound 1.
- 15 [0102] FIG. 61 shows the X-ray powder diffraction (XRPD) pattern of malate salt Form II of Compound 1.
- [0103] FIG. 62 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of malate salt Form II of Compound 1.
- [0104] FIG. 63 shows dynamic vapor sorption (DVS) plot of malate salt Form II of
20 Compound 1.
- [0105] FIG. 64 shows a representative polarized light microscopy (PLM) image of malate salt Form II of Compound 1.
- [0106] FIG. 65 shows the X-ray powder diffraction (XRPD) pattern of succinate salt Form I of Compound 1.
- 25 [0107] FIG. 66 shows the differential scanning calorimetry (DSC) thermogram as well as the thermal gravimetric analysis (TGA) of succinate salt Form I of Compound 1.
- [0108] FIG. 67 shows a representative polarized light microscopy (PLM) image of succinate salt Form I of Compound 1.

DETAILED DESCRIPTION OF THE INVENTION

I. General

[0109] The present disclosure provides crystalline forms of Compound 1. These forms are characterized by an X-ray powder diffraction (XRPD) pattern. Selected crystalline forms are further characterized by a differential scanning calorimetry (DSC), a thermal gravimetric analysis (TGA), a dynamic vapor sorption analysis (DVS), and/or a polarized light microscope (PLM) profile. The present disclosure further provides pharmaceutical compositions of the crystalline forms of Compound 1 described herein. In some embodiments, the present disclosure provides a pharmaceutical composition described herein, wherein the pharmaceutical composition i) comprises a crystalline form of Compound 1 described herein, ii) is prepared using a crystalline form of Compound 1 described herein, iii) comprises a solid state form as described herein, or iv) is prepared using a solid state form described herein.

[0110] The present disclosure further provides methods of inhibiting PARG in vivo in a patient, the method comprising administering to the patient a therapeutically effective amount of a crystalline form of Compound 1 described herein. The present disclosure further provides methods of inhibiting cell proliferation, in vitro or in vivo, the method comprising contacting a cell with an effective amount of a crystalline form of Compound 1 described herein. The present disclosure further provides methods of treating a cancer in a patient in need thereof, the method comprising administering to the patient a therapeutically effective amount of a crystalline form of Compound 1 described herein. The present disclosure further provides methods of treating a cancer resistant to one or more platins or one or more PARP inhibitors in a patient in need thereof, the method comprising administering to the patient an effective amount of a crystalline form of Compound 1 described herein. The present disclosure further provides methods of treating and/or preventing a homologous recombinant deficient (HRD) cancer in a patient, the method comprising administering to the patient a therapeutically effective amount of a crystalline form of Compound 1 described herein. The present disclosure further provides methods of treating and/or preventing a cancer in a patient, where the cancer is characterized by a reduction or absence of BRCA1 and/or BRCA2 gene expression, the absence or mutation of BRCA1 and/or BRCA2 genes, or reduced function of BRCA1 and/or BRCA2 proteins, the method comprising administering to

the individual a therapeutically effective amount of a crystalline form of Compound 1 described herein.

[0111] The present disclosure further provides methods of inhibiting PARG in vivo in a patient, the method comprising administering to the patient a therapeutically effective amount of a pharmaceutical composition described herein. The present disclosure further provides methods of inhibiting cell proliferation, in vitro or in vivo, the method comprising contacting a cell with an effective amount of a pharmaceutical composition described herein. The present disclosure further provides methods of treating a cancer in a patient in need thereof, the method comprising administering to the patient a therapeutically effective amount of a pharmaceutical composition described herein. The present disclosure further provides methods of treating a cancer resistant to one or more platins or one or more PARP inhibitors in a patient in need thereof, the method comprising administering to the patient a therapeutically effective amount of a pharmaceutical composition described herein. The present disclosure further provides methods of treating and/or preventing a homologous recombinant deficient (HRD) cancer in a patient, the method comprising administering to the individual a therapeutically effective amount of a pharmaceutical composition described herein. The present disclosure further provides methods of treating and/or preventing a cancer in a patient, where the cancer is characterized by a reduction or absence of BRCA1 and/or BRCA2 gene expression, the absence or mutation of BRCA1 and/or BRCA2 genes, or reduced function of BRCA1 and/or BRCA2 proteins, the method comprising administering to the individual a therapeutically effective amount of a pharmaceutical composition described herein.

II. Definitions

[0112] “Substantially free” refers to that an amount of 10% or less of another form is present in a particular desired form, preferably 9%, 8.5%, 8%, 7.5%, 7%, 6.5%, 6%, 5.5%, 5%, 4.5%, 4%, 3.5%, 3%, 2.5%, 2%, 1.5%, 1%, 0.5%, or less of another form.

[0113] “Crude” refers to a mixture including a desired compound (*e.g.*, the compound of Formula (I)) and at least one other species (*e.g.*, a solvent, a reagent such as an acid or base, a starting material, or a byproduct of a reaction giving rise to the desired compound).

[0114] “Solvate” refers to a compound provided herein or a salt thereof that binds to a stoichiometric or non-stoichiometric amount of solvent by non-covalent intermolecular forces. Where the solvent is water, the solvate is a hydrate.

5 [0115] “Hydrate” refers to a compound that is complexed to a stoichiometric or non-stoichiometric amount of water. The compounds of the present invention can be complexed with from $\frac{1}{2}$ or 1 to 10 water molecules. For example, the compounds of the present invention can be complexed with $\frac{1}{2}$ water molecule, the compounds of the present invention can be complexed with 1 water molecule, or the compounds of the present invention can be complexed with 2 water molecules.

10 [0116] “Crystalline form” refers to a solid form of a compound wherein the constituent molecules are packed in a regularly ordered, repeating pattern. A crystalline form can include triclinic, monoclinic, orthorhombic, tetragonal, trigonal, hexagonal, and cubic crystal geometries. A crystalline form can include one or more regions, *i.e.*, grains, with distinct crystal boundaries. A crystalline solid can include two or more crystal geometries.

15 [0117] “Amorphous form” refers to a solid form of a compound having no definite crystal structure, *i.e.*, lacking a regularly ordered, repeating pattern of constituent molecules.

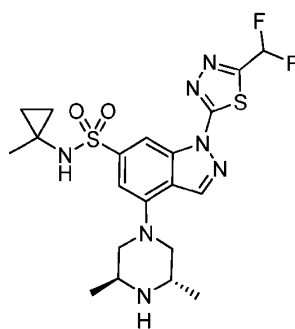
[0118] “FeSSIF” stands for Fed State Simulated Intestinal Fluid. “FaSSIF” stands for Fasted State Simulated Intestinal Fluid. “SGF” stands for Simulated Gastric Fluid.

20 [0119] “About” means a range of values including the specified value, which a person of ordinary skill in the art would consider reasonably similar to the specified value. In some embodiments, the term “about” means within a standard deviation using measurements generally acceptable in the art. In some embodiments, “about” means a range extending to +/- 10% of the specified value.

25 [0120] “Pharmaceutically acceptable salts” as used herein is meant to include salts of the active compounds which are prepared with relatively nontoxic acids or bases, depending on the particular substituents found on the compounds described herein. When compounds disclosed herein contain relatively acidic functionalities, base addition salts can be obtained by contacting the neutral form of such compounds with a sufficient amount of the desired base, either neat or in a suitable inert solvent. Examples of salts derived from
30 pharmaceutically-acceptable inorganic bases include aluminum, ammonium, calcium, copper, ferric, ferrous, lithium, magnesium, manganic, manganous, potassium, sodium, zinc and the

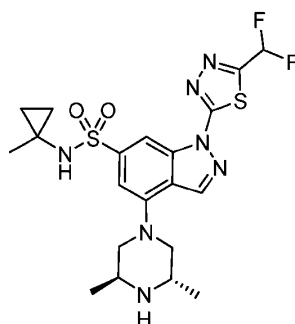
like. Salts derived from pharmaceutically-acceptable organic bases include salts of primary, secondary and tertiary amines, including substituted amines, cyclic amines, naturally-occurring amines and the like, such as arginine, betaine, caffeine, choline, N,N'-dibenzylethylenediamine, diethylamine, 2-diethylaminoethanol, 2-dimethylaminoethanol, ethanolamine, ethylenediamine, N-ethylmorpholine, N-ethylpiperidine, glucamine, glucosamine, histidine, hydrabamine, isopropylamine, lysine, methylglucamine, morpholine, piperazine, piperidine, polyamine resins, procaine, purines, theobromine, triethylamine, trimethylamine, tripropylamine, tromethamine and the like. When compounds of the present invention contain relatively basic functionalities, acid addition salts can be obtained by contacting the neutral form of such compounds with a sufficient amount of the desired acid, either neat or in a suitable inert solvent. Examples of pharmaceutically acceptable acid addition salts include those derived from inorganic acids like hydrochloric, hydrobromic, nitric, carbonic, monohydrogen carbonic, phosphoric, monohydrogen phosphoric, dihydrogen phosphoric, sulfuric, monohydrogen sulfuric, hydriodic, or phosphorous acids and the like, as well as the salts derived from relatively nontoxic organic acids like acetic, propionic, isobutyric, malonic, benzoic, succinic, suberic, fumaric, mandelic, phthalic, benzenesulfonic, p-tolylsulfonic, citric, tartaric, methanesulfonic, and the like. Also included are salts of amino acids such as arginate and the like, and salts of organic acids like glucuronic or galactunoric acids and the like (see, for example, Berge, S.M., et al, "Pharmaceutical Salts", *Journal of Pharmaceutical Science*, **1977**, 66, 1-19). Certain specific compounds of the present invention contain both basic and acidic functionalities that allow the compounds to be converted into either base or acid addition salts.

[0121] "Compound 1" is a chemical compound having an IUPAC name of 1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-((3S,5S)-3,5-dimethylpiperazin-1-yl)-N-(1-methylcyclopropyl)-1H-indazole-6-sulfonamide, and the structure shown below:



(Compound 1).

[0122] “Free base of Compound 1” or “Compound 1 in free base” refers to Compound 1 represented by the formula:



(Compound 1).

[0123] The term “treating” or “treatment” encompasses both disease-modifying treatment and symptomatic treatment, either of which may be prophylactic (i.e., before the onset of symptoms, in order to prevent, delay or reduce the severity of symptoms) or therapeutic (i.e., after the onset of symptoms, in order to reduce the severity and/or duration of symptoms).

[0124] The term “individual” or “patient” or “individual in need thereof” or “patient in need thereof” refers to mammals, which includes primates (especially humans), domesticated companion animals (such as dogs, cats, horses, and the like) and livestock (such as cattle, pigs, sheep, and the like), with dosages as described herein. In some embodiments, the term “individual,” “patient” or “individual in need thereof” or “patient in need thereof” refers to a human. In some embodiments, the human is an adult.

[0125] An “effective amount” or a “therapeutically effective amount” is an amount sufficient to accomplish a stated purpose (e.g. achieve the effect for which it is administered, treat a disease, reduce enzyme activity, reduce one or more symptoms of a disease or condition). An example of an “effective amount” is an amount sufficient to contribute to the treatment, or reduction of a symptom or symptoms of a disease, which could also be referred to as a “therapeutically effective amount.” A “reduction” of a symptom or symptoms (and grammatical equivalents of this phrase) means decreasing of the severity or frequency of the symptom(s), or elimination of the symptom(s).

III. Detailed Description of Embodiments

[0126] Provided herein are various crystalline forms of Compound 1, including free base and salt forms, pharmaceutical compositions comprising the same, methods of their use, and

methods of preparing the salt forms. Certain crystalline forms may be hydrates, solvates, or anhydrous forms of Compound 1. Certain crystalline forms may be hydrates, solvates, or anhydrous forms of Compound 1 in free base. Certain crystalline forms may be hydrates, solvates, or anhydrous forms of Compound 1 in salt forms.

5 [0127] Methods for collection of XRPD data are known in the art, and any such methods can be used for characterizing the crystalline forms described herein. For example, the X-ray powder diffraction patterns described herein can be generated using Cu K α 1 radiation.

[0128] In some embodiments, the crystalline forms described herein are further characterized by a differential scanning calorimetry (DSC) thermogram. In some
10 embodiments, a DSC thermogram is recorded using a sample weight of about 1-3 mg, which is subjected to temperatures ranging from 25°C to 300°C using a ramp of 10°C/min.

[0129] In some embodiments, the crystalline form described herein is further characterized by a thermal gravimetric analysis (TGA). In some embodiments, a TGA thermogram is recorded using a sample weight of about 1-5 mg, which is subjected to temperatures ranging
15 from room temperature to 300°C using a ramp of 10°C/min.

[0130] In some embodiments, the crystalline form described herein is further characterized by a polarized light microscope (PLM) profile.

[0131] In some embodiments, the crystalline form described herein is further characterized by a dynamic vapor sorption (DVS) profile. In some embodiments, a DVS is recorded
20 according to the method as described herein.

A. Free Base Forms of Compound 1

[0132] In some embodiments, the crystalline form is a free base form of Compound 1. In some embodiments, the crystalline form is free base Form I. In some embodiments, the crystalline form is free base Form II.

i. Free Base Form I

[0133] In one embodiment, the present disclosure provides free base Form I of Compound 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0 and 18.3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ). In some
embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD)

pattern comprising peaks at 9.0, 16.4, and 18.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.0, and 18.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.0, 18.3, and 19.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 16.4, 18.0, 18.3, and 19.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 16.4, 18.0, 18.3, 18.9, and 19.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, and 19.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 11.5, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 11.5, 12.7, 13.7, 16.4, 17.2, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 11.5, 12.2, 12.7, 13.7, 16.4, 17.2, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ).

[0134] In some embodiments, free base Form I is characterized by an XRPD pattern comprising three peaks selected from 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an XRPD pattern comprising three or more peaks selected from 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an XRPD pattern comprising four peaks selected from 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an XRPD pattern comprising five peaks selected from 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ).

[0135] In some embodiments, free base Form I is characterized by an XRPD pattern further comprising one or more peaks (such as one, two, or three peaks) selected from 23.2, 23.5, and

24.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes a peak at 24.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes one or two peaks selected from 23.5 and 24.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 22.5, 23.2, 23.5, 24.6, and 27.8 degrees 2θ (± 0.2 degrees 2θ). For example, in some embodiments, free base Form I is characterized by an XRPD pattern comprising peaks at 9.0, 16.4, 18.3, 23.5, and 24.6 degrees 2θ (± 0.2 degrees 2θ). For example, in some embodiments, free base Form I is characterized by an XRPD pattern comprising peaks at 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, 23.5, and 24.6 degrees 2θ (± 0.2 degrees 2θ).

[0136] In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in Table 1.

[0137] In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in Table 1. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in Table 1. In some embodiments, free base Form I is characterized by an

X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 1**. In some embodiments, free
5 base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 1**.

[0138] In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an
10 X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at
15 least five peaks having a relative intensity of at least 20% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at
20 least 20% as listed in **Table 1**.

[0139] In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an
25 X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at
30 least five peaks having a relative intensity of at least 30% as listed in **Table 1**. In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 1**.

- [0140] In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 18.3, and 24.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.3, and 24.6 degrees 2θ (± 0.2 degrees 2θ).
- 5 [0141] In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.3, 23.5, and 24.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 12.7, 13.7, 18.0, 18.9, and 19.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at
- 10 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, 23.5, and 24.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, 19.7, 23.5, and 24.6 degrees 2θ (± 0.2 degrees 2θ).
- [0142] In some embodiments, free base Form I is characterized by an X-ray powder
- 15 diffraction (XRPD) pattern substantially in accordance with FIG. 1.
- [0143] In some embodiments, free base Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 200°C. In some embodiments, free base Form I is further characterized by a differential scanning calorimetry (DSC) thermogram including an endotherm onset temperature of about 199°C and an
- 20 endothermic peak at about 200°C.
- [0144] In some embodiments, free base Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 2.
- [0145] In some embodiments, free base Form I is further characterized by no weight loss upon heating prior to decomposition, as measured by a thermal gravimetric analysis (TGA).
- 25 In some embodiments, free base Form I is further characterized by substantially no weight loss upon heating prior to decomposition, as measured by a thermal gravimetric analysis (TGA). In some embodiments, free base Form I is further characterized by a weight loss of no greater than 5%, 4%, 3%, 2%, or 1% by weight upon heating prior to decomposition, as measured by a thermal gravimetric analysis (TGA).
- 30 [0146] In some embodiments, free base Form I is further characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 2.

[0147] In some embodiments, free base Form I is further characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 3.

[0148] In some embodiments, free base Form I is substantially free of other crystalline or amorphous forms.

5

ii. *Free Base Form II*

[0149] In one embodiment, the present disclosure provides free base Form II of Compound 1. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3 and 18.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, and 18.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 17.1, and 18.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 17.1, 18.7, and 19.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 16.4, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 13.4, 15.6, 16.4, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 13.4, 15.6, 16.1, 16.4, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 13.4, 14.1, 15.6, 16.1, 16.4, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 13.4, 14.1, 15.6, 16.1, 16.4, 17.1, 18.7, 19.5, 19.7, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 13.4, 14.1, 15.6, 16.1, 16.4, 17.1, 17.4, 18.7, 19.5, 19.7, and 20.0 degrees 2θ (± 0.2 degrees 2θ).

[0150] In some embodiments, free base Form II is characterized by an XRPD pattern comprising three peaks selected from 12.3, 15.6, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an XRPD pattern comprising four peaks selected from 12.3, 15.6, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an XRPD pattern comprising five peaks selected from 12.3, 15.6, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ).

[0151] In some embodiments, free base Form II is characterized by an XRPD pattern further comprising one or more peaks (such as one, two, three, four, five, six, or seven peaks) selected from 20.2, 20.7, 23.0, 25.0, 25.7, 26.5, and 31.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an XRPD pattern further comprising a peak at 20.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an XRPD pattern further comprising one or two peaks selected from 20.2 and 20.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 20.2, 20.7, and 25.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 20.2, 20.7, 23.0, and 25.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 20.2, 20.7, 23.0, 25.7, and 26.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 20.2, 20.7, 23.0, 25.0, 25.7, and 26.5 degrees 2θ (± 0.2 degrees 2θ). For example, in some embodiments, free base Form II is characterized by an XRPD pattern comprising peaks at 12.3, 15.6, 17.1, 18.7, and 20.7 degrees 2θ (± 0.2 degrees 2θ). For example, in some embodiments, free base Form II is characterized by an XRPD pattern comprising peaks at 12.3, 15.6, 17.1, 18.7, 19.5, 19.7, 20.2, 20.7, 23.0, and 25.7 degrees 2θ (± 0.2 degrees 2θ).

[0152] In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in Table 2. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in Table 2. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in Table 2. In some embodiments, free base Form II is characterized by an

X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 2**.

[0153] In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 2**.

[0154] In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD)

pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an

5 X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at

10 least ten peaks having a relative intensity of at least 20% as listed in **Table 2**.

[0155] In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an

15 X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at

20 least five peaks having a relative intensity of at least 30% as listed in **Table 2**. In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 2**.

[0156] In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, and 18.7 degrees 2θ (± 0.2

25 degrees 2θ). In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 18.7, and 20.7 degrees 2θ (± 0.2 degrees 2θ).

[0157] In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 17.1, 18.7, and 20.7 degrees 2θ ($\pm$

30 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 19.5, 19.7, 20.2, 23.0, and 25.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, free base

Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 17.1, 18.7, 19.5, 19.7, 20.2, 20.7, 23.0, and 25.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).

[0158] In some embodiments, free base Form II is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 4**.

- 5 **[0159]** In some embodiments, free base Form II is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic peak at about 148 °C, and/or a second endothermic peak at about 198 °C. In some embodiments, free base Form II is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic onset and peak at about 143 °C and 148 °C, respectively, and/or a second
- 10 endothermic onset and peak at about 197 °C and 198 °C, respectively. In some embodiments, free base Form II is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic onset and peak at about 143 °C and about 148 °C, respectively, and/or a second endothermic onset and peak at about 197 °C and about 198 °C, respectively.

- 15 **[0160]** In some embodiments, free base Form II is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 5**.

[0161] In some embodiments, free base Form II is characterized by 15.1% weight loss upon heating to 158 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, free base Form II is characterized by about 15.1% weight loss upon heating to about 158 °C, as measured by a thermal gravimetric analysis (TGA).

- 20 **[0162]** In some embodiments, free base Form II is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 5**.

[0163] In some embodiments, free base Form II is characterized by a polarized light microscope (PLM) profile substantially as shown in **FIG. 6**.

- 25 **[0164]** In some embodiments, free base Form II is substantially free of other crystalline or amorphous forms.

B. Salt Forms of Compound 1

- [0165]** The present disclosure provides various salt forms of Compound 1 including an HCl salt, sulfate salt, tosylate salt, besylate salt, mesylate salt, maleate salt, citrate salt, fumarate salt, malate salt, and succinate salt. The single crystalline forms of the described salts are, in
- 30 certain embodiments, substantially free of other crystalline or amorphous forms. The

crystalline forms of the described salts are, in certain embodiments, substantially free of other crystalline or amorphous forms.

[0166] Particular salts disclosed herein can exist in one or more polymorphic forms.

Individual polymorphic forms of the described salts are identified as Form I, Form II, and

5 Form III.

i. HCl salt of Compound 1 (Form I)

[0167] In some aspects, the crystalline form is an HCl salt of Compound 1. In some embodiments, the crystalline form is HCl salt Form I. In some embodiments, HCl salt Form I is an anhydrate.

10 **[0168]** In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 13.2 and 13.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 13.2 and 17.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 13.2, 13.7, and 17.7 degrees 2θ (± 0.2 degrees 2θ). In some
15 embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 11.7, 13.2, 13.7, and 17.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 11.7, 13.2, 13.7, 17.7, and 18.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 11.7, 13.2, 13.7, 17.2, and 17.7 degrees 2θ (± 0.2 degrees 2θ).
20 20). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 11.7, 13.2, 13.7, 17.2, 17.7, and 18.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 7.0, 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 7.0, 8.0, 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments,
25 HCl salt Form I is characterized by an XRPD pattern comprising peaks at 7.0, 11.7, 12.2, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some
30 embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 7.0,

8.0, 11.7, 12.2, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 7.0, 8.0, 11.7, 12.2, 13.2, 13.5, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ).

- 5 **[0169]** In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising three peaks selected from 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9. In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising three or more peaks selected from 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9. In some
10 embodiments, HCl salt Form I is characterized by an XRPD pattern comprising four peaks selected from 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9. In some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising five peaks selected from 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9.

- [0170]** In some embodiments of HCl salt Form I, the X-ray powder diffraction pattern further includes one or more peaks (such as one, two, three, four, five, six, seven, eight, nine,
15 ten, or eleven peaks) selected from 20.6, 20.9, 21.2, 21.7, 22.3, 24.3, 25.0, 25.3, 25.6, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes a peak at 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes one or two peaks selected from 26.1 and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction
20 pattern further includes one or more peaks selected from 24.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes one or more peaks selected from 20.9, 24.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes one or more peaks selected from 20.9, 24.3, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some
25 embodiments, the X-ray powder diffraction pattern further includes one or more peaks selected from 20.6, 20.9, 24.3, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes one or more peaks selected from 20.6, 20.9, 21.2, 24.3, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some
30 embodiments, the X-ray powder diffraction pattern further includes one or more peaks selected from 20.6, 20.9, 21.2, 24.3, 25.0, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes one or more peaks selected from 20.6, 20.9, 21.2, 21.7, 24.3, 25.0, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the X-ray powder diffraction pattern further includes one

or more peaks selected from 20.6, 20.9, 21.2, 21.7, 24.3, 25.0, 25.3, 25.6, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). For example, in some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 13.2, 13.7, 17.7, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). For example, in some embodiments, HCl salt Form I is characterized by an XRPD pattern comprising peaks at 13.2, 13.7, 17.7, 20.6, 20.9, 21.2, 24.3, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).

[0171] In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 3**.

[0172] In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 3**. In some embodiments, HCl

salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least 10 peaks having a relative intensity of at least 15% as listed in **Table 3**.

[0173] In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least 10 peaks having a relative intensity of at least 20% as listed in **Table 3**.

[0174] In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in

- Table 3.** In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 30% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 30% as listed in **Table 3**. In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least 10 peaks having a relative intensity of at least 30% as listed in **Table 3**.
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- 10 **[0175]** In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.7, 17.7, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.7, 17.7, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an X-ray powder
- 15 diffraction (XRPD) pattern comprising peaks at 13.7, 17.7, 24.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.7, 17.7, 20.6, 20.9, 21.2, 24.3, 25.0, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.7, 17.7,
- 20 20.6, 20.9, 21.2, 21.7, 24.3, 25.0, 25.3, 25.6, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.7, 13.7, 17.2, 17.7, 18.4, 20.6, 20.9, 21.2, 21.7, 24.3, 25.0, 25.3, 25.6, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at
- 25 11.7, 13.7, 15.3, 17.2, 17.7, 18.4, 20.0, 20.6, 20.9, 21.2, 21.7, 24.3, 25.0, 25.3, 25.6, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).
- [0176]** In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.2, 13.7, 17.7, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 20.6,
- 30 20.9, 21.2, 24.3, and 25.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.2, 13.7, 17.7, 20.6, 20.9, 21.2, 24.3, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).

- [0177] In some embodiments, HCl salt Form I is characterized by an X-ray powder diffraction pattern substantially in accordance with FIG. 7.
- [0178] In some embodiments, HCl salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including no endothermic peak prior to decomposition at about 300 °C.
- [0179] In some embodiments, HCl salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 8.
- [0180] In some embodiments, HCl salt Form I is characterized by no weight loss upon heating prior to decomposition, as measured by a thermal gravimetric analysis (TGA). In some embodiments, HCl salt Form I is characterized by no substantial weight loss upon heating prior to decomposition, as measured by a thermal gravimetric analysis (TGA). In some embodiments, HCl salt Form I is characterized by a weight loss of no greater than 5%, 4%, 3%, 2%, 1%, or 0.5% by weight upon heating prior to decomposition, as measured by a thermal gravimetric analysis (TGA).
- [0181] In some embodiments, HCl salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 8.
- [0182] In some embodiments, HCl salt Form I is characterized by a weight gain of about 0.5% after undergoing a dynamic vapor sorption profile cycle from about 0% relative humidity (RH) to about 90% RH at 25 °C.
- [0183] In some embodiments, HCl salt Form I is characterized by a dynamic vapor sorption (DVS) profile substantially as shown in FIG. 9.
- [0184] In some embodiments, HCl salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 10.
- [0185] In some embodiments, HCl salt Form I is substantially free of other crystalline or amorphous forms.

ii. *Sulfate salt of Compound 1 (Form I)*

[0186] In some aspects, the crystalline form is a sulfate salt of Compound 1. In some embodiments, the crystalline form is sulfate salt Form I. In some embodiments, sulfate salt Form I is a hydrate.

- [0187] In one embodiment, the present disclosure provides sulfate salt Form I of Compound 1. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.0, 8.5, 13.2, 15.6, and 16.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, sulfate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 8.8, 12.9, 15.0, 17.2, and 19.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, sulfate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 20.2, 22.1, 24.3, 25.1, and 26.7 degrees 2θ (± 0.2 degrees 2θ).
- [0188] In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.0, 8.5, 13.2, 15.6, and 16.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 8.8, 12.9, 15.0, 19.0, and 24.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.0, 8.5, 8.8, 12.9, 13.2, 15.0, 15.6, 16.1, 19.0, and 24.3 degrees 2θ (± 0.2 degrees 2θ).
- [0189] In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 4**.
- [0190] In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 4**. In some

embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 4**.

[0191] In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 4**.

[0192] In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative

intensity of at least 30% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 4**. In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 30% as listed in **Table 4**.

[0193] In some embodiments, sulfate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 11**.

[0194] In some embodiments, sulfate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including a first endotherm peak 118 °C, a second endotherm peak at 220 °C, an exotherm peak at 231 °C, and/or a third endotherm peak at 279 °C. In some embodiments, sulfate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including a first endotherm peak at about 118 °C, a second endotherm peak at about 220 °C, an exotherm peak at about 231 °C, and/or a third endotherm peak at about 279 °C. In some embodiments, sulfate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including a first endotherm onset and peak at 78 °C and 118 °C, respectively; a second endotherm onset and peak at 206 °C and 220 °C, respectively; an exotherm onset and peak at 227 °C and 231 °C, respectively; and/or a third endotherm onset and peak at 273 °C and 279 °C, respectively. In some embodiments, sulfate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including a first endotherm onset and peak at about 78 °C and about 118 °C, respectively; a second endotherm onset and peak at about 206 °C and about 220 °C, respectively; an exotherm onset and peak at about 227 °C and about 231 °C, respectively; and/or a third endotherm onset and peak at about 273 °C and about 279 °C, respectively.

[0195] In some embodiments, sulfate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 12**.

[0196] In some embodiments, sulfate salt Form I is characterized by 2.8% weight loss upon heating to 130 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, sulfate salt Form I is characterized by about 2.8% weight loss upon heating to about 130 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, sulfate salt Form I is characterized by weight loss not greater than about 10%, 5%, 4%, or 3% by weight upon heating to about 130 °C, as measured by a thermal gravimetric analysis (TGA).

[0197] In some embodiments, sulfate salt Form I is characterized by a thermal gravimetric

[0198] In some embodiments, sulfate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 13.

[0199] In some embodiments, sulfate salt Form I is substantially free of other crystalline or amorphous forms.

iii. *Tosylate salt of Compound 1 (Form I)*

[0200] In some aspects, the crystalline form is a tosylate salt of Compound 1. In some embodiments, the crystalline form is tosylate salt Form I. In some embodiments, tosylate salt Form I is an anhydrate.

[0201] In one embodiment, the present disclosure provides tosylate salt Form I of Compound 1. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.8, 9.6, 14.5, 18.2, and 19.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 4.7, 11.7, 13.7, 15.5, and 15.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 21.4, 22.8, 24.0, 24.3, and 29.3 degrees 2θ (± 0.2 degrees 2θ).

[0202] In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.6, 14.5, 18.2, 19.5, and 24.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 6.8, 21.4, 22.8, 24.0, and 29.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.8, 9.6, 14.5, 18.2, 19.5, 21.4, 22.8, 24.0, 24.3, and 29.3 degrees 2θ (± 0.2 degrees 2θ).

- [0203] In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 5**. In some embodiments,
- 5 tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 5**.
- 10 [0204] In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 5**. In some embodiments, tosylate salt Form I is
- 15 characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 5**. In some
- 20 embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative
- 25 intensity of at least 15% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 5**.
- 30 [0205] In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative

intensity of at least 20% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 5**.

[0206] In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 5**. In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 5**.

[0207] In some embodiments, tosylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 14**.

[0208] In some embodiments, tosylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 231 °C. In some embodiments, tosylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 222 °C and an endothermic peak at about 231°C.

[0209] In some embodiments, tosylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 15**.

[0210] In some embodiments, tosylate salt Form I is characterized by 1.2% weight loss upon heating from 110 °C to 230 °C, as measured by a thermal gravimetric analysis (TGA).

In some embodiments, tosylate salt Form I is characterized by about 1.2% weight loss upon heating from about 110 °C to about 230 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, tosylate salt Form I is characterized by a weight loss of no greater than about 10%, 5%, 4%, 3%, or 2% by weight upon heating from about 110 °C to
5 about 230 °C, as measured by a thermal gravimetric analysis (TGA).

[0211] In some embodiments, tosylate salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 15.

[0212] In some embodiments, tosylate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 16.

10 [0213] In some embodiments, tosylate salt Form I is substantially free of other crystalline or amorphous forms.

iv. *Tosylate salt of Compound 1 (Form II)*

[0214] In some aspects, the crystalline form is a tosylate salt of Compound 1. In some embodiments, the crystalline form is tosylate salt Form II. In some embodiments, tosylate salt
15 Form II is anhydrate.

[0215] In one embodiment, the present disclosure provides tosylate salt Form II of Compound 1. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.7, 14.3, 15.4, 17.3, and 19.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form II is characterized by an
20 XRPD pattern further comprising one or more peaks selected from 9.1, 10.2, 13.0, 17.0, and 18.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 20.1, 20.5, 23.8, 24.2, and 26.2 degrees 2θ (± 0.2 degrees 2θ).

[0216] In some embodiments, tosylate salt Form II is characterized by an X-ray powder
25 diffraction (XRPD) pattern comprising peaks at 6.7, 15.4, 17.3, 20.1, and 24.2 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 14.3, 17.0, 19.1, 20.5, and 26.2 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.7, 14.3, 15.4, 17.0, 17.3, 19.1, 20.1, 20.5, 24.2, and 26.2 degrees 2θ (± 0.2 degrees 2θ).

- [0217] In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 6**. In some embodiments,
- 5 tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 6**.
- 10 [0218] In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 6**. In some embodiments, tosylate salt Form II is
- 15 characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 6**. In some
- 20 embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative
- 25 intensity of at least 15% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 6**.
- 30 [0219] In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative

intensity of at least 20% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 20% as listed in **Table 6**.

[0220] In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 6**. In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 6**.

[0221] In some embodiments, tosylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 17**.

[0222] In some embodiments, tosylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 229 °C. In some embodiments, tosylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 223 °C and an endothermic peak at about 229 °C.

[0223] In some embodiments, tosylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 18.

[0224] In some embodiments, tosylate salt Form II is characterized by 1.4% weight loss upon heating from 90 °C to 230 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, tosylate salt Form II is characterized by about 1.4% weight loss upon heating from about 90 °C to about 230 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, tosylate salt Form II is characterized by a weight loss of no greater than about 10%, 5%, 4%, 3%, or 2% by weight upon heating from about 90 °C to about 230 °C, as measured by a thermal gravimetric analysis (TGA).

[0225] In some embodiments, tosylate salt Form II is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 18.

[0226] In some embodiments, tosylate salt Form II is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 19.

[0227] In some embodiments, tosylate salt Form II is substantially free of other crystalline or amorphous forms.

v. *Tosylate salt of Compound 1 (Form III)*

[0228] In some aspects, the crystalline form is a tosylate salt of Compound 1. In some embodiments, the crystalline form is tosylate salt Form III. In some embodiments, tosylate salt Form III is an anhydrate.

[0229] In one embodiment, the present disclosure provides tosylate salt Form III of Compound 1. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.9, 14.5, 15.5, 18.2, and 19.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form III is characterized by an XRPD pattern further comprising one or more peaks selected from 9.7, 14.1, 14.9, 16.0, and 16.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form III is

characterized by an XRPD pattern further comprising one or more peaks selected from 21.2, 21.4, 22.0, 24.0, and 24.4 degrees 2θ (± 0.2 degrees 2θ).

5 [0230] In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 18.2, 19.5, 21.2, 22.0, and 24.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 6.9, 14.5, 15.5, 21.4, and 24.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.9, 14.5, 15.5, 18.2, 19.5, 21.2, 21.4, 22.0, 24.0, and 24.4 degrees 2θ (± 0.2 degrees 2θ).

10 [0231] In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 7**. In some embodiments, tosylate salt Form III is
15 characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 7**.

[0232] In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at
20 least 15% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 7**. In some
25 embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an
30 X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks

having a relative intensity of at least 15% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 7**.

[0233] In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 20% as listed in **Table 7**.

[0234] In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least

four peaks having a relative intensity of at least 30% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 7**. In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 7**.

5 [0235] In some embodiments, tosylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 20**.

[0236] In some embodiments, tosylate salt Form III is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 222 °C. In some embodiments, tosylate salt Form III is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 206 °C and an endothermic peak at about 222 °C.

15 [0237] In some embodiments, tosylate salt Form III is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 21**.

[0238] In some embodiments, tosylate salt Form III is characterized by 2.6% weight loss upon heating from about 110 °C to about 222 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, tosylate salt Form III is characterized by about 2.6% weight loss upon heating from about 110 °C to about 222 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, tosylate salt Form III is characterized by a weight loss loss of no greater than about 10%, 5%, 4%, or 3% by weight upon heating from about 110 °C to about 222 °C, as measured by a thermal gravimetric analysis (TGA).

25 [0239] In some embodiments, tosylate salt Form III is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 21**.

[0240] In some embodiments, tosylate salt Form III is characterized by a weight gain of about 3.2% after undergoing a dynamic vapor sorption profile cycle from about 0% relative humidity (RH) to about 90% RH at 25 °C.

30 [0241] In some embodiments, tosylate salt Form III is characterized by a dynamic vapor sorption (DVS) profile substantially as shown in **FIG. 22**.

[0242] In some embodiments, tosylate salt Form III is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 23.

[0243] In some embodiments, tosylate salt Form III is substantially free of other crystalline or amorphous forms.

5 vi. *Besylate salt of Compound 1 (Form I)*

[0244] In some aspects, the crystalline form is a besylate salt of Compound 1. In some embodiments, the crystalline form is besylate salt Form I. In some embodiments, besylate salt Form I is anhydrate.

[0245] In one embodiment, the present disclosure provides besylate salt Form I of
10 Compound 1. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.8, 14.5, 15.4, 17.3, and 19.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, besylate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 15.0, 15.9, 17.7, 18.3, and 19.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, besylate salt Form I is
15 characterized by an XRPD pattern further comprising one or more peaks selected from 20.3, 20.8, 24.0, 25.2, and 26.0 degrees 2θ (± 0.2 degrees 2θ).

[0246] In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.8, 17.3, 19.1, 24.0, and 25.2 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 14.5,
20 15.4, 17.7, 20.3, and 26.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.8, 14.5, 15.4, 17.3, 17.7, 19.1, 20.3, 24.0, 25.2, and 26.0 degrees 2θ (± 0.2 degrees 2θ).

[0247] In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in
25 Table 8. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in Table 8. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in Table 8. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed
30 in Table 8. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in Table 8.

[0248] In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 8**.

[0249] In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray

- powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 8**. In some embodiments,
- 5 besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 20% as listed in **Table 8**.
- 10 **[0250]** In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 8**. In some embodiments, besylate salt Form I is
- 15 characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction
- 20 (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks
- 25 having a relative intensity of at least 30% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 30% as listed in **Table 8**. In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 30% as
- 30 listed in **Table 8**.
- [0251]** In some embodiments, besylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 24**.

[0252] In some embodiments, besylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 202 °C. In some embodiments, besylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 190 °C and an endothermic peak at about 202 °C.

[0253] In some embodiments, besylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 25.

[0254] In some embodiments, besylate salt Form I is characterized by 0.6% weight loss upon heating from 100 °C to 210 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, besylate salt Form I is characterized by about 0.6% weight loss upon heating from about 100 °C to about 210 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, besylate salt Form I is characterized by a weight loss of no greater than about 10%, 5%, 4%, 3%, 2%, or 1% by weight upon heating from about 100 °C to about 210 °C, as measured by a thermal gravimetric analysis (TGA).

[0255] In some embodiments, besylate salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 25.

[0256] In some embodiments, besylate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 26.

[0257] In some embodiments, besylate salt Form I is substantially free of other crystalline or amorphous forms.

vii. *Besylate salt of Compound 1 (Form II)*

[0258] In some aspects, the crystalline form is a besylate salt of Compound 1. In some embodiments, the crystalline form is besylate salt Form II. In some embodiments, besylate salt Form II is an anhydrate.

[0259] In one embodiment, the present disclosure provides besylate salt Form II of Compound 1. In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 7.6, 15.2, 15.7, 19.2, and 19.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, besylate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 9.0, 10.0, 12.4, 14.7, and 18.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, besylate salt Form II is

characterized by an XRPD pattern further comprising one or more peaks selected from 10.4, 20.1, 22.9, 25.0, and 26.5 degrees 2θ (± 0.2 degrees 2θ).

5 [0260] In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 7.6, 15.2, 19.2, 22.9, and 26.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 15.7, 18.1, 19.7, 20.1, and 25.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 7.6, 15.2, 15.7, 18.1, 19.2, 19.7, 20.1, 22.9, 25.0, and 26.5 degrees 2θ (± 0.2 degrees 2θ).

10 [0261] In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in Table 9. In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in Table 9. In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in Table 9. In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in Table 9. In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in Table 9.

[0262] In some embodiments, besylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 27.

20 [0263] In some embodiments, besylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 165 °C. In some embodiments, besylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 154°C and an endothermic peak at about 165 °C.

25 [0264] In some embodiments, besylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 28

[0265] In some embodiments, besylate salt Form II is characterized by 0.5% weight loss upon heating from 64 °C to 185 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, besylate salt Form II is characterized by about 0.5% weight loss upon heating from about 64 °C to about 185 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, besylate salt Form II is characterized by a weight loss of no

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greater than about 10%, 5%, 4%, 3%, 2%, or 1% by weight upon heating from about 64 °C to about 185 °C, as measured by a thermal gravimetric analysis (TGA).

[0266] In some embodiments, besylate salt Form II is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 28**.

- 5 [0267] In some embodiments, besylate salt Form II is characterized by a polarized light microscope (PLM) profile substantially as shown in **FIG. 29**.

[0268] In some embodiments, besylate salt Form II is substantially free of other crystalline or amorphous forms.

viii. Mesylate salt of Compound 1 (Form I)

- 10 [0269] In some aspects, the crystalline form is a mesylate salt of Compound 1. In some embodiments, the crystalline form is mesylate salt Form I. In some embodiments, mesylate salt Form I is an anhydrate.

[0270] In one embodiment, the present disclosure provides mesylate salt Form I of Compound 1. In some embodiments, mesylate salt Form I is characterized by an X-ray
15 powder diffraction (XRPD) pattern comprising peaks at 14.2, 17.0, 18.0, 18.6, and 19.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 11.0, 11.9, 12.7, 15.1, and 17.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 20.7,
20 21.2, 22.1, 24.1, and 25.0 degrees 2θ (± 0.2 degrees 2θ).

[0271] In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 14.2, 17.0, 18.6, 22.1, and 24.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 18.0, 19.3, 21.2, 21.5, and 24.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt
25 Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 14.2, 17.0, 18.0, 18.6, 19.3, 21.2, 21.5, 22.1, 24.1, and 24.9 degrees 2θ (± 0.2 degrees 2θ).

[0272] In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder
30 diffraction (XRPD) pattern comprising three peaks listed in **Table 10**. In some embodiments,

mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 10**.

[0273] In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 10**.

[0274] In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 10**. In some

embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 10**.

5 [0275] In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 10**. In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least
10 four peaks having a relative intensity of at least 30% as listed in **Table 10**.

[0276] In some embodiments, mesylate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 30**.

[0277] In some embodiments, mesylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 222 °C. In
15 some embodiments, mesylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 218°C and an endothermic peak at about 222°C.

[0278] In some embodiments, mesylate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 31**.

20 [0279] In some embodiments, mesylate salt Form I is characterized by 0.5% weight loss upon heating from 125 °C to 225 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, mesylate salt Form I is characterized by about 0.5% weight loss upon heating from about 125 °C to about 225 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, mesylate salt Form I is characterized by a weight loss of no
25 greater than about 10%, 5%, 4%, 3%, 2%, or 1% by weight upon heating from about 125 °C to about 225 °C, as measured by a thermal gravimetric analysis (TGA).

[0280] In some embodiments, mesylate salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 31**.

30 [0281] In some embodiments, mesylate salt Form I is characterized by a weight gain of about 19% after undergoing a dynamic vapor sorption profile cycle from about 0% relative humidity (RH) to about 90% RH at 25 °C.

[0282] In some embodiments, mesylate salt Form I is characterized by a dynamic vapor sorption (DVS) profile substantially as shown in FIG. 32.

[0283] In some embodiments, mesylate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 33.

- 5 [0284] In some embodiments, mesylate salt Form I is substantially free of other crystalline or amorphous forms.

ix. *Mesylate salt of Compound 1 (Form II)*

[0285] In some aspects, the crystalline form is a mesylate salt of Compound 1. In some embodiments, the crystalline form is mesylate salt Form II. In some embodiments, mesylate salt Form II is an anhydrate.

[0286] In one embodiment, the present disclosure provides mesylate salt Form II of Compound 1. In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.7, 15.4, 17.2, 17.6, and 19.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 10.0, 10.3, 11.4, 15.0, and 16.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 12.1, 20.2, 22.7, 23.2, and 24.1 degrees 2θ (± 0.2 degrees 2θ).

[0287] In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.7, 15.4, 17.5, 19.1, and 23.2 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 9.9, 10.3, 15.0, 17.2, and 20.2 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.7, 9.9, 10.3, 15.0, 15.4, 17.2, 17.5, 19.1, 20.2, and 23.2 degrees 2θ (± 0.2 degrees 2θ).

[0288] In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in Table 11. In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in Table 11. In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in Table 11. In some embodiments, mesylate salt Form II is

characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 11**. In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 11**.

5 [0289] In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 11**. In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 11**. In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least
10 two peaks having a relative intensity of at least 20% as listed in **Table 11**.

[0290] In some embodiments, mesylate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 34**.

[0291] In some embodiments, mesylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 190 °C. In
15 some embodiments, mesylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 176 °C and an endothermic peak at about 190 °C.

[0292] In some embodiments, mesylate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 35**.

20 [0293] In some embodiments, mesylate salt Form II is characterized by 0.2% weight loss upon heating from 140 °C to 205 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, mesylate salt Form II is characterized by about 0.2% weight loss upon heating from about 140 °C to about 205 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, mesylate salt Form II is characterized by a weight loss of no
25 greater than about 10%, 5%, 4%, 3%, 2%, or 1% by weight upon heating from about 140 °C to about 205 °C, as measured by a thermal gravimetric analysis (TGA).

[0294] In some embodiments, mesylate salt Form II is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 35**.

30 [0295] In some embodiments, mesylate salt Form II is characterized by a polarized light microscope (PLM) profile substantially as shown in **FIG. 36**.

[0296] In some embodiments, mesylate salt Form II is substantially free of other crystalline or amorphous forms.

x. *Mesylate salt of Compound 1 (Form III)*

5 [0297] In some aspects, the crystalline form is a mesylate salt of Compound 1. In some embodiments, the crystalline form is mesylate salt Form III. In some embodiments, mesylate salt Form III is a solvate or anhydrate.

[0298] In one embodiment, the present disclosure provides mesylate salt Form III of Compound 1. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.5, 8.9, 17.5, 18.5, and 19.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt Form III is characterized by an XRPD pattern further comprising one or more peaks selected from 6.1, 13.1, 13.8, 14.4, and 19.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt Form III is characterized by an XRPD pattern further comprising one or more peaks selected from 13.3, 18.0, 21.4, 23.6, and 26.0 degrees 2θ (± 0.2 degrees 2θ).

15 [0299] In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.5, 8.9, 18.5, 19.6, and 21.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 6.1, 13.8, 17.5, 19.4, and 23.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 20 6.1, 6.5, 8.9, 13.8, 17.5, 18.5, 19.4, 19.6, 21.4, and 23.6 degrees 2θ (± 0.2 degrees 2θ).

[0300] In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in Table 12. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in Table 12. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in Table 12. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in Table 12. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in Table 12.

30 [0301] In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at

- least 15% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least
- 5 four peaks having a relative intensity of at least 15% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least
- 10 15% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 12**. In some
- 15 embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 12**.
- 20 **[0302]** In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 12**. In some embodiments, mesylate salt
- 25 Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least
- 30 20% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 12**. In some embodiments, mesylate salt

Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 12**.

[0303] In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 12**. In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 12**.

[0304] In some embodiments, mesylate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 37**.

[0305] In some embodiments, mesylate salt Form III is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 173°C. In some embodiments, mesylate salt Form III is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 138 °C and an endothermic peak at about 173 °C.

[0306] In some embodiments, mesylate salt Form III is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 38**.

[0307] In some embodiments, mesylate salt Form III is characterized by 1.5% weight loss upon heating from 80 °C to 180 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, mesylate salt Form III is characterized by about 1.5% weight loss upon heating from about 80 °C to about 180 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, mesylate salt Form III is characterized by a weight loss of no greater than about 10%, 5%, 4%, 3%, or 2% by weight upon heating from about 80 °C to about 180 °C, as measured by a thermal gravimetric analysis (TGA).

[0308] In some embodiments, mesylate salt Form III is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 38**.

[0309] In some embodiments, mesylate salt Form III is characterized by a polarized light microscope (PLM) profile substantially as shown in **FIG. 39**.

- 5 [0310] In some embodiments, mesylate salt Form III is substantially free of other crystalline or amorphous forms.

xi. Maleate salt of Compound 1 (Form I)

- [0311] In some aspects, the crystalline form is a maleate salt of Compound 1. In some embodiments, the crystalline form is maleate salt Form I. In some embodiments, maleate salt
10 Form I is an anhydrate.

- [0312] In one embodiment, the present disclosure provides maleate salt Form I of Compound 1. In some embodiments, maleate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.7, 14.8, 15.5, 18.7, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, maleate salt Form I is characterized by
15 an XRPD pattern further comprising one or more peaks selected from 7.9, 8.3, 9.9, 12.7, and 18.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, maleate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 16.7, 21.2, 22.5, 23.3, and 24.0 degrees 2θ (± 0.2 degrees 2θ).

- [0313] In some embodiments, maleate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 14.8, 15.5, 18.7, 22.5, and 23.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 7.9, 8.3, 8.7, 9.9, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, maleate salt
20 Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 7.9, 8.3, 8.7, 9.9, 14.8, 15.5, 18.7, 20.0, 22.5, and 23.3 degrees 2θ (± 0.2 degrees 2θ).

- [0314] In some embodiments, maleate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 13**. In some embodiments, maleate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 13**. In some embodiments, maleate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern
25 comprising five peaks listed in **Table 13**. In some embodiments, maleate salt Form I is
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characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in Table 13. In some embodiments, maleate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in Table 13.

5 [0315] In some embodiments, maleate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 40.

[0316] In some embodiments, maleate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 195°C. In some embodiments, maleate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 191°C and an
10 endothermic peak at about 195°C.

[0317] In some embodiments, maleate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 41.

[0318] In some embodiments, maleate salt Form I is characterized by no weight loss prior to decomposition, as measured by a thermal gravimetric analysis (TGA). In some
15 embodiments, maleate salt Form I is characterized by no substantial weight loss prior to decomposition, as measured by a thermal gravimetric analysis (TGA). In some embodiments, maleate salt Form I is characterized by a weight loss of no greater than 5%, 4%, 3%, 2%, 1%, or 0.5% by weight upon heating prior to decomposition, as measured by a thermal gravimetric analysis (TGA).

20 [0319] In some embodiments, maleate salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 41.

[0320] In some embodiments, maleate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 42.

25 [0321] In some embodiments, maleate salt Form I is substantially free of other crystalline or amorphous forms.

xii. Maleate salt of Compound 1 (Form II)

[0322] In some aspects, the crystalline form is a maleate salt of Compound 1. In some embodiments, the crystalline form is maleate salt Form II. In some embodiments, maleate salt Form II is an acetone solvate.

- [0323] In one embodiment, the present disclosure provides maleate salt Form II of Compound 1. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 14.3, 15.3, 17.5, 18.3, and 18.8 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, maleate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 9.6, 11.9, 13.3, 14.2, and 19.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, maleate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 17.9, 21.4, 24.0, 24.3, and 24.7 degrees 2θ (± 0.2 degrees 2θ).
- [0324] In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 14.3, 15.3, 17.5, 18.3, and 24.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 11.9, 13.3, 14.2, 18.8, and 21.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.9, 13.3, 14.2, 14.3, 15.3, 17.5, 18.3, 18.8, 21.4, and 24.0 degrees 2θ (± 0.2 degrees 2θ).
- [0325] In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 14**.
- [0326] In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in

Table 14. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 14**.

[0327] In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 20% as listed in **Table 14**.

[0328] In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 30% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 30% as listed in **Table 14**. In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 30% as listed in **Table 14**.

[0329] In some embodiments, maleate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 43**.

[0330] In some embodiments, maleate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic peak at about 142 °C and/or a second endothermic peak at about 162 °C. In some embodiments, maleate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic onset and peak temperature of about 135 °C and 142 °C, respectively, and/or a second endothermic onset and endothermic peak at about 155 °C and 162 °C, respectively. In some embodiments, maleate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic onset and peak temperature of about 135 °C and about 142 °C, respectively, and/or a second endothermic onset and endothermic peak at about 155 °C and about 162 °C, respectively.

[0331] In some embodiments, maleate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 44.

[0332] In some embodiments, maleate salt Form II is characterized by 1.6% weight loss upon heating from 80 °C to 144 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, maleate salt Form II is characterized by about 1.6% weight loss upon heating from about 80 °C to about 144 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, maleate salt Form II is characterized by a weight loss of no greater than about 10%, 5%, 4%, 3%, or 2% by weight upon heating from about 80 °C to about 144 °C, as measured by a thermal gravimetric analysis (TGA).

[0333] In some embodiments, maleate salt Form II is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 44.

[0334] In some embodiments, maleate salt Form II is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 45.

[0335] In some embodiments, maleate salt Form II is substantially free of other crystalline or amorphous forms.

xiii. Citrate salt of Compound 1 (Form I)

[0336] In some aspects, the crystalline form is a citrate salt of Compound 1. In some embodiments, the crystalline form is citrate salt Form I. In some embodiments, citrate salt Form I is an acetone solvate or anhydrate.

[0337] In one embodiment, the present disclosure provides citrate salt Form I of Compound 1. In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.4, 15.8, 17.9, 19.2, and 19.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, citrate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 10.6, 13.1, 15.1, 17.0, and 18.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, citrate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 20.3, 22.6, 23.1, 24.5, and 27.1 degrees 2θ (± 0.2 degrees 2θ).

[0338] In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.4, 15.8, 19.2, 20.3, and 27.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 17.9,

19.5, 22.6, 23.1, and 24.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.4, 15.8, 17.9, 19.2, 19.5, 20.3, 22.6, 23.1, 24.5, and 27.1 degrees 2θ (± 0.2 degrees 2θ).

5 [0339] In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in Table 15. In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in Table 15. In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in Table 15. In some embodiments, citrate salt Form I is
10 characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in Table 15. In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in Table 15.

[0340] In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at
15 least 15% as listed in Table 15. In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in Table 15.

[0341] In some embodiments, citrate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 46.

20 [0342] In some embodiments, citrate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 167 °C. In some embodiments, citrate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 159 °C and an endothermic peak at about 167 °C.

25 [0343] In some embodiments, citrate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 47.

[0344] In some embodiments, citrate salt Form I is characterized by 22% weight loss upon heating from 95 °C to 225 °C, as measured by a thermal gravimetric analysis (TGA). In some
30 embodiments, citrate salt Form I is characterized by about 22% weight loss upon heating from about 95 °C to about 225 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, citrate salt Form I is characterized by a weight loss of no greater than

about 30%, 25%, or 22% by weight upon heating from about 95 °C to about 225 °C, as measured by a thermal gravimetric analysis (TGA).

[0345] In some embodiments, citrate salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 47.

- 5 [0346] In some embodiments, citrate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 48.

[0347] In some embodiments, citrate salt Form I is substantially free of other crystalline or amorphous forms.

xiv. Fumarate salt of Compound 1 (Form I)

- 10 [0348] In some aspects, the crystalline form is a fumarate salt of Compound 1. In some embodiments, the crystalline form is fumarate salt Form I. In some embodiments, fumarate salt Form I is a THF solvate.

[0349] In one embodiment, the present disclosure provides fumarate salt Form I of Compound 1. In some embodiments, fumarate salt Form I is characterized by an X-ray
15 powder diffraction (XRPD) pattern comprising peaks at 4.2, 8.5, 10.6, 18.8, and 19.8 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, fumarate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 12.7, 13.0, 15.9, 17.0, and 19.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, fumarate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 21.1,
20 21.4, 23.4, 25.7, and 27.4 degrees 2θ (± 0.2 degrees 2θ).

[0350] In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 4.2, 10.6, 19.8, 21.4, and 23.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 8.5, 17.0, 18.8, 19.4, and 25.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, fumarate salt
25 Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 4.2, 8.5, 10.6, 17.0, 18.8, 19.4, 19.8, 21.4, 23.4, and 25.7 degrees 2θ (± 0.2 degrees 2θ).

[0351] In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in Table 16. In some embodiments, fumarate salt Form I is characterized by an X-ray powder
30 diffraction (XRPD) pattern comprising three peaks listed in Table 16. In some embodiments,

fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 16**.

[0352] In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 16**.

[0353] In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 16**. In some

embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 16**.

[0354] In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 16**. In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 16**.

[0355] In some embodiments, fumarate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 49**.

[0356] In some embodiments, fumarate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 198°C. In some embodiments, fumarate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 194°C and an endothermic peak at about 198 °C.

[0357] In some embodiments, fumarate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 50**.

[0358] In some embodiments, fumarate salt Form I is characterized by 9.7% weight loss upon heating from 100 °C to 210 °C, as measured by a thermal gravimetric analysis (TGA).

In some embodiments, fumarate salt Form I is characterized by about 9.7% weight loss upon heating from about 100 °C to about 210 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, fumarate salt Form I is characterized by a weight loss of no greater than about 20%, 15%, or 10% by weight upon heating from about 100 °C to about
5 210 °C, as measured by a thermal gravimetric analysis (TGA).

[0359] In some embodiments, fumarate salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 50.

[0360] In some embodiments, fumarate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 51.

10 [0361] In some embodiments, fumarate salt Form I is substantially free of other crystalline or amorphous forms.

xv. *Fumarate salt of Compound 1 (Form II)*

[0362] In some aspects, the crystalline form is a fumarate salt of Compound 1. In some embodiments, the crystalline form is fumarate salt Form II. In some embodiments, fumarate
15 salt Form II is an acetone solvate.

[0363] In one embodiment, the present disclosure provides fumarate salt Form II of Compound 1. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 4.2, 8.5, 10.5, 12.9, and 18.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, fumarate salt Form II is characterized by an
20 XRPD pattern further comprising one or more peaks selected from 15.1, 16.0, 18.6, 19.6, and 20.0 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, fumarate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 21.7, 23.3, 23.6, 26.1, and 27.7 degrees 2θ (± 0.2 degrees 2θ).

[0364] In some embodiments, fumarate salt Form II is characterized by an X-ray powder
25 diffraction (XRPD) pattern comprising peaks at 4.2, 8.5, 12.9, 21.7, and 23.6 degrees 2θ (± 0.2 degrees 2θ). In some embodiments the XRPD pattern further comprises peaks at 10.5, 16.0, 18.9, 20.0, and 26.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 4.2, 8.5, 10.5, 12.9, 16.0, 18.9, 20.0, 21.7, 23.6, and 26.1 degrees 2θ (± 0.2 degrees 2θ).

- [0365] In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 17**. In some embodiments,
- 5 fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 17**.
- 10 [0366] In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 17**. In some embodiments, fumarate salt
- 15 Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least
- 20 15% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks
- 25 having a relative intensity of at least 15% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as
- 30 listed in **Table 17**.
- [0367] In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized

by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 17**. In some

5 embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an

10 X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern

15 comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 20% as listed in **Table 17**.

[0368] In some embodiments, fumarate salt Form II is characterized by an X-ray powder

20 diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least

25 four peaks having a relative intensity of at least 30% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least

30 30% as listed in **Table 17**. In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 17**.

[0369] In some embodiments, fumarate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 52.

5 [0370] In some embodiments, fumarate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic peak at about 144 °C and/or a second endothermic peak at about 200 °C. In some embodiments, fumarate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic onset and peak at about 144°C, and/or a second endothermic onset and peak at about 197 °C and 200 °C, respectively.

10 [0371] In some embodiments, fumarate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 53.

[0372] In some embodiments, fumarate salt Form II is characterized by 2.1% weight loss upon heating from 90 °C to 175 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, fumarate salt Form II is characterized by about 2.1% weight loss upon heating from about 90 °C to about 175 °C, as measured by a thermal gravimetric analysis
15 (TGA). In some embodiments, fumarate salt Form II is characterized by a weight loss of no greater than about 10%, 5%, 4%, or 3% by weight upon heating from about 90 °C to about 175 °C, as measured by a thermal gravimetric analysis (TGA).

[0373] In some embodiments, fumarate salt Form II is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 53.

20 [0374] In some embodiments, fumarate salt Form II is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 54.

[0375] In some embodiments, fumarate salt Form II is substantially free of other crystalline or amorphous forms.

xvi. Fumarate salt of Compound 1 (Form III)

25 [0376] In some aspects, the crystalline form is a fumarate salt of Compound 1. In some embodiments, the crystalline form is fumarate salt Form III. In some embodiments, fumarate salt Form III is an ethyl acetate solvate.

[0377] In one embodiment, the present disclosure provides fumarate salt Form III of Compound 1. In some embodiments, fumarate salt Form III is characterized by an X-ray
30 powder diffraction (XRPD) pattern comprising peaks at 5.7, 7.9, 8.9, 10.6, and 17.4 degrees

20 (± 0.2 degrees 2θ). In some embodiments, fumarate salt Form III is characterized by an XRPD pattern further comprising one or more peaks selected from 10.3, 11.5, 16.1, 17.9, and 18.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, fumarate salt Form III is characterized by an XRPD pattern further comprising one or more peaks selected from 2.1, 21.9, 24.0, 25.1, and 26.9 degrees 2θ (± 0.2 degrees 2θ).

[0378] In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 5.7, 7.9, 8.9, 10.6, and 17.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 11.5, 16.1, 17.9, 18.5, and 25.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 5.7, 7.9, 8.9, 10.6, 11.5, 16.1, 17.4, 17.9, 18.5, and 25.1 degrees 2θ (± 0.2 degrees 2θ).

[0379] In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 18**.

[0380] In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by

- an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 18**. In some
- 5 embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 18**.
- 10 **[0381]** In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a
- 15 Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder
- 20 diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 18**. In some
- 25 embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least
- 30 20% as listed in **Table 18**.
- [0382]** In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized

by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 18**. In some
5 embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by
10 an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 18**. In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 30% as listed in **Table 18**. In some
15 embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 30% as listed in **Table 18**.

[0383] In some embodiments, fumarate salt Form III is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 55**.

[0384] In some embodiments, fumarate salt Form III is characterized by a differential
20 scanning calorimetry (DSC) thermogram including a first endothermic peak at about 113 °C and/or a second endothermic peak at about 164 °C. In some embodiments, fumarate salt Form III is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic onset and peak at about 106 °C and 113 °C, respectively, and/or a second endothermic onset and peak at about 158 °C and 164 °C, respectively.

25 **[0385]** In some embodiments, fumarate salt Form III is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 56**.

[0386] In some embodiments, fumarate salt Form III is characterized by 3.1% weight loss upon heating from 85 °C to 135 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, fumarate salt Form III is characterized by about 3.1% weight loss upon
30 heating from about 85 °C to about 135 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, fumarate salt Form III is characterized by a weight loss of no

greater than about 10%, 5%, or 4% by weight upon heating from about 85 °C to about 135 °C, as measured by a thermal gravimetric analysis (TGA).

[0387] In some embodiments, fumarate salt Form III is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 56**.

- 5 [0388] In some embodiments, fumarate salt Form III is characterized by a polarized light microscope (PLM) profile substantially as shown in **FIG. 57**.

[0389] In some embodiments, fumarate salt Form III is substantially free of other crystalline or amorphous forms.

xvii. Malate salt of Compound 1 (Form I)

- 10 [0390] In some aspects, the crystalline form is a malate salt of Compound 1. In some embodiments, the crystalline form is malate salt Form I. In some embodiments, malate salt Form I is acetone solvate.

[0391] In one embodiment, the present disclosure provides malate salt Form I of Compound 1. In some embodiments, malate salt Form I is characterized by an X-ray powder
15 diffraction (XRPD) pattern comprising peaks at 10.9, 12.4, 13.9, 18.7, and 19.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, malate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 4.1, 8.3, 10.5, 16.0, and 16.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, malate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 20.0, 20.5, 21.1, 22.9,
20 and 24.7 degrees 2θ (± 0.2 degrees 2θ).

[0392] In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 10.9, 12.4, 19.4, 20.0, and 20.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 10.5, 13.9, 21.1, 22.9, and 24.7 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, malate salt
25 Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 10.5, 10.9, 12.4, 13.9, 19.4, 20.0, 20.5, 21.1, 22.9, and 24.7 degrees 2θ (± 0.2 degrees 2θ).

[0393] In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 19**. In some embodiments, malate salt Form I is characterized by an X-ray powder
30 diffraction (XRPD) pattern comprising three peaks listed in **Table 19**. In some embodiments,

malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 19**. In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 19**. In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 19**.

[0394] In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 19**. In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 19**. In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 19**. In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 19**.

[0395] In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 19**. In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 19**. In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 19**.

[0396] In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 19**.

[0397] In some embodiments, malate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 58**.

[0398] In some embodiments, malate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic peak at about 157 °C, and/or a second endothermic peak at about 206 °C. In some embodiments, malate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic onset and peak at about 147 °C and 157 °C, respectively, and/or a second endothermic onset and peak at about 181 °C and 206 °C, respectively. In some embodiments,

malate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including a first endothermic onset and peak at about 147 °C and about 157 °C, respectively, and/or a second endothermic onset and peak at about 181 °C and about 206 °C, respectively.

5 [0399] In some embodiments, malate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 59.

[0400] In some embodiments, malate salt Form I is characterized by 1.9% weight loss upon heating from 75 °C to 157 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, malate salt Form I is characterized by about 1.9% weight loss upon heating from about 75 °C to about 157 °C, as measured by a thermal gravimetric analysis (TGA). In
10 some embodiments, malate salt Form I is characterized by a weight loss of no greater than about 10%, 5%, 4%, 3%, or 2% by weight upon heating from about 75 °C to about 157 °C, as measured by a thermal gravimetric analysis (TGA).

[0401] In some embodiments, malate salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 59.

15 [0402] In some embodiments, malate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 60.

[0403] In some embodiments, malate salt Form I is substantially free of other crystalline or amorphous forms.

xviii. Malate salt of Compound 1 (Form II)

20 [0404] In some aspects, the crystalline form is a malate salt of Compound 1. In some embodiments, the crystalline form is malate salt Form II. In some embodiments, malate salt Form II is an anhydrate.

[0405] In one embodiment, the present disclosure provides malate salt Form II of Compound 1. In some embodiments, malate salt Form II is characterized by an X-ray
25 powder diffraction (XRPD) pattern comprising peaks at 8.2, 12.4, 14.7, 15.8, and 18.3 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, malate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 4.0, 15.6, 16.4, 17.0, and 19.1 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, malate salt Form II is characterized by an XRPD pattern further comprising one or more peaks selected from 21.5,
30 22.2, 22.9, 24.4, and 26.2 degrees 2θ (± 0.2 degrees 2θ).

[0406] In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.2, 14.7, 15.8, 18.3, and 24.4 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 12.4, 19.1, 21.5, 22.9, and 26.2 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, malate salt
5 Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.2, 12.4, 14.7, 15.8, 18.3, 19.1, 21.5, 22.9, 24.4, and 26.2 degrees 2θ (± 0.2 degrees 2θ).

[0407] In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder
10 diffraction (XRPD) pattern comprising three peaks listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder
15 diffraction (XRPD) pattern comprising ten peaks listed in **Table 20**.

[0408] In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative
20 intensity of at least 15% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 15% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 20**. In some
25 embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 15% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by
30 an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 20**. In some embodiments, malate

salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 20**.

[0409] In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 20% as listed in **Table 20**.

[0410] In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in

- Table 20.** In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 30% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 30% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 30% as listed in **Table 20**. In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 30% as listed in **Table 20**.
- 5
- 10 **[0411]** In some embodiments, malate salt Form II is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 61**.
- [0412]** In some embodiments, malate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 183 °C. In some embodiments, malate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 180 °C and an endothermic peak at about 183 °C.
- 15
- [0413]** In some embodiments, malate salt Form II is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with **FIG. 62**.
- [0414]** In some embodiments, malate salt Form II is characterized by 0.5% weight loss upon heating from about 95 °C to about 170 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, malate salt Form II is characterized by about 0.5% weight loss upon heating from about 95 °C to about 170 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, malate salt Form II is characterized by a weight loss of no greater than about 10%, 5%, 4%, 3%, 2%, or 1% by weight upon heating from about 95 °C to about 170 °C, as measured by a thermal gravimetric analysis (TGA).
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- [0415]** In some embodiments, malate salt Form II is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 62**.
- [0416]** In some embodiments, malate salt Form II is characterized by a weight gain of about 1.8% after undergoing a dynamic vapor sorption profile cycle from about 0% relative humidity (RH) to about 90% RH at 25 °C.
- 30

[0417] In some embodiments, malate salt Form II is characterized by a dynamic vapor sorption (DVS) profile substantially as shown in FIG. 63.

[0418] In some embodiments, malate salt Form II is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 64.

- 5 [0419] In some embodiments, malate salt Form II is substantially free of other crystalline or amorphous forms.

xix. Succinate salt of Compound 1 (Form I)

- [0420] In some aspects, the crystalline form is a succinate salt of Compound 1. In some embodiments, the crystalline form is succinate salt Form I. In some embodiments, succinate salt Form I is an acetone solvate.

- [0421] In one embodiment, the present disclosure provides succinate salt Form I of Compound 1. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 10.5, 12.8, 15.0, 18.8, and 19.9 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, succinate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 8.5, 10.7, 12.1, 15.9, and 18.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, succinate salt Form I is characterized by an XRPD pattern further comprising one or more peaks selected from 21.4, 22.8, 23.3, 24.5, and 24.8 degrees 2θ (± 0.2 degrees 2θ).

- [0422] In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.8, 15.0, 18.8, 21.4, and 24.8 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, the XRPD pattern further comprises peaks at 10.5, 19.9, 22.8, 23.3, and 24.5 degrees 2θ (± 0.2 degrees 2θ). In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 10.5, 12.8, 15.0, 18.8, 19.9, 21.4, 22.8, 23.3, 24.5, and 24.8 degrees 2θ (± 0.2 degrees 2θ).

- [0423] In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three, four, five, or seven, ten, or more peaks listed in Table 21. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks listed in Table 21. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks listed in Table 21. In some embodiments, succinate salt Form I is

characterized by an X-ray powder diffraction (XRPD) pattern comprising seven peaks listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising ten peaks listed in **Table 21**.

- 5 **[0424]** In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 15% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 15% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least
- 10 four peaks having a relative intensity of at least 15% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least
- 15 15% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 15% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 15% as listed in **Table 21**. In some embodiments,
- 20 succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 15% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 15% as listed in **Table 21**.
- 25 **[0425]** In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 20% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 20% as listed in **Table 21**. In some embodiments, succinate salt
- 30 Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 20% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in

Table 21. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 20% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least seven peaks having a relative intensity of at least 20% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least eight peaks having a relative intensity of at least 20% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least nine peaks having a relative intensity of at least 20% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least ten peaks having a relative intensity of at least 20% as listed in **Table 21**.

[0426] In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least two peaks having a relative intensity of at least 30% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least three peaks having a relative intensity of at least 30% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least four peaks having a relative intensity of at least 30% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 30% as listed in **Table 21**. In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern comprising at least six peaks having a relative intensity of at least 30% as listed in **Table 21**.

[0427] In some embodiments, succinate salt Form I is characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 65**.

[0428] In some embodiments, succinate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an endothermic peak at about 136 °C. In some embodiments, succinate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram including an onset temperature of about 128 °C and an endothermic peak at about 136 °C.

[0429] In some embodiments, succinate salt Form I is characterized by a differential scanning calorimetry (DSC) thermogram substantially in accordance with FIG. 66.

[0430] In some embodiments, succinate salt Form I is characterized by 2.8% weight loss upon heating from 85 °C to 145 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, succinate salt Form I is characterized by about 2.8% weight loss upon heating from about 85 °C to about 145 °C, as measured by a thermal gravimetric analysis (TGA). In some embodiments, succinate salt Form I is characterized by a weight loss of no greater than about 10%, 5%, 4%, or 3% by weight upon heating from about 85 °C to about 145 °C, as measured by a thermal gravimetric analysis (TGA).

[0431] In some embodiments, succinate salt Form I is characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 66.

[0432] In some embodiments, succinate salt Form I is characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 67.

[0433] In some embodiments, succinate salt Form I is substantially free of other crystalline or amorphous forms.

C. Solid State Forms

[0434] Provided herein are various solid state forms of Compound 1, comprising at least 50 wt% of a particular crystalline form as described herein. In some embodiments, the solid state form comprises at least 60 wt% of a particular crystalline form as described herein. In some embodiments, the solid state form comprises at least 70 wt% of a particular crystalline form as described herein. In some embodiments, the solid state form comprises at least 80 wt% of a particular crystalline form as described herein. In some embodiments, the solid state form comprises at least 90 wt% of a particular crystalline form as described herein. In some embodiments, the solid state form comprises at least 95 wt% of a particular crystalline form as described herein. In some embodiments, the solid state form comprises at least 99 wt% of a particular crystalline form as described herein. In some embodiments, the particular crystalline form is HCl salt Form I. In some embodiments, the particular crystalline form is free base Form I. In some embodiments, the particular crystalline form is free base Form II.

D. Pharmaceutical Compositions

- [0435] Provided herein are pharmaceutical compositions comprising a crystalline form of Compound 1 described herein or pharmaceutical compositions prepared using a crystalline form of Compound 1 described herein. Also provided are pharmaceutical compositions comprising a solid state form as described herein, or a pharmaceutical compositions prepared using a solid state form described herein. Pharmaceutical compositions will include one or more pharmaceutically acceptable excipients. In some embodiments, provided herein are pharmaceutical compositions comprising a crystalline form of Compound 1 described herein and one or more pharmaceutically acceptable excipients. In some embodiments, provided herein are pharmaceutical compositions prepared using a crystalline form of Compound 1 described herein and one or more pharmaceutically acceptable excipients. In some embodiments, provided herein are pharmaceutical compositions comprising a solid state form described herein and one or more pharmaceutically acceptable excipients. In some embodiments, provided herein are pharmaceutical compositions prepared using a solid state form described herein and one or more pharmaceutically acceptable excipients.
- 15 [0436] The pharmaceutical compositions provided herein may be used in the methods disclosed herein; thus, for example, the pharmaceutical compositions can be administered *ex vivo* or *in vivo* to a subject in order to practice the therapeutic methods and uses described herein.
- [0437] Pharmaceutical compositions may be in the form suitable for administration to a patient. The pharmaceutical compositions can be formulated to be compatible with the intended method or route of administration; exemplary routes of administration are set forth herein. Furthermore, the pharmaceutical compositions may be used in combination with other therapeutically active agents or compounds as described herein in order to treat the diseases, disorders and conditions contemplated by the present disclosure.
- 25 [0438] The compositions may be in a form suitable for oral use (for example as tablets, lozenges, hard or soft capsules, aqueous or oily suspensions, emulsions, dispersible powders or granules, syrups or elixirs), for topical use (for example as creams, ointments, gels, or aqueous or oily solutions or suspensions), for administration by inhalation (for example as a finely divided powder or a liquid aerosol), for administration by insufflation (for example as a finely divided powder) or for parenteral administration (for example as a sterile aqueous or oily solution for intravenous, subcutaneous, intramuscular, intraperitoneal or intramuscular dosing or as a suppository for rectal dosing).
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[0439] The compositions may be obtained by conventional procedures using conventional pharmaceutical excipients, well known in the art. Thus, compositions intended for oral use may contain, for example, one or more coloring, sweetening, flavoring and/or preservative agents. Tablets, capsules and the like contain the active ingredient in admixture with non-toxic pharmaceutically acceptable excipients which are suitable for the manufacture of tablets, capsules, and the like. These excipients may be, for example, diluents, such as calcium carbonate, sodium carbonate, lactose, calcium phosphate or sodium phosphate; granulating and disintegrating agents, for example, corn starch, or alginic acid; binding agents, for example starch, gelatin or acacia, and lubricating agents, for example magnesium stearate, stearic acid or talc.

[0440] The tablets, capsules and the like suitable for oral administration may be uncoated or coated by known techniques to delay disintegration and absorption in the gastrointestinal tract and thereby provide a sustained action. For example, a time-delay material such as glyceryl monostearate or glyceryl di-stearate may be employed. The tablets may also be coated by techniques known in the art to form osmotic therapeutic tablets for controlled release. Additional agents include biodegradable or biocompatible particles or a polymeric substance such as polyesters, polyamine acids, hydrogel, polyvinyl pyrrolidone, polyanhydrides, polyglycolic acid, ethylene-vinyl acetate, methylcellulose, carboxymethylcellulose, protamine sulfate, or lactide and glycolide copolymers, polylactide and glycolide copolymers, or ethylene vinyl acetate copolymers in order to control delivery of an administered composition. For example, the oral agent can be entrapped in microcapsules prepared by coacervation techniques or by interfacial polymerization, by the use of hydroxymethyl cellulose or gelatin-microcapsules or poly (methyl methacrylate) microcapsules, respectively, or in a colloid drug delivery system. Colloidal dispersion systems include macromolecule complexes, nanocapsules, microspheres, microbeads, and lipid-based systems, including oil-in-water emulsions, micelles, mixed micelles, and liposomes. Methods for the preparation of the above-mentioned formulations are known in the art.

[0441] Formulations for oral use may also be presented as hard gelatin capsules wherein the active ingredient is mixed with an inert solid diluent, for example, calcium carbonate, calcium phosphate, kaolin or microcrystalline cellulose, or as soft gelatin capsules wherein the active ingredient is mixed with water or an oil medium, for example peanut oil, liquid paraffin, or olive oil.

[0442] Aqueous suspensions contain the active materials in admixture with excipients suitable for the manufacture thereof. Such excipients can be suspending agents, for example sodium carboxymethylcellulose, methylcellulose, (hydroxypropyl)methyl cellulose, sodium alginate, polyvinyl-pyrrolidone, gum tragacanth and gum acacia; dispersing or wetting agents, for example a naturally-occurring phosphatide (e.g., lecithin), or condensation products of an alkylene oxide with fatty acids (e.g., poly-oxyethylene stearate), or condensation products of ethylene oxide with long chain aliphatic alcohols (e.g., for heptadecaethyleneoxycetanol), or condensation products of ethylene oxide with partial esters derived from fatty acids and a hexitol (e.g., polyoxyethylene sorbitol monooleate), or condensation products of ethylene oxide with partial esters derived from fatty acids and hexitol anhydrides (e.g., polyethylene sorbitan monooleate). The aqueous suspensions may also contain one or more preservatives.

[0443] Oily suspensions may be formulated by suspending the active ingredient in a vegetable oil, for example, arachis oil, olive oil, sesame oil or coconut oil, or in a mineral oil such as liquid paraffin. The oily suspensions may contain a thickening agent, for example beeswax, hard paraffin or cetyl alcohol. Sweetening agents such as those set forth above, and flavoring agents may be added to provide a palatable oral preparation.

[0444] Dispersible powders and granules suitable for preparation of an aqueous suspension by the addition of water provide the active ingredient in admixture with a dispersing or wetting agent, suspending agent and one or more preservatives. Suitable dispersing or wetting agents and suspending agents are exemplified herein.

[0445] The pharmaceutical compositions may also be in the form of oil-in-water emulsions. The oily phase may be a vegetable oil, for example olive oil or arachis oil, or a mineral oil, for example, liquid paraffin, or mixtures of these. Suitable emulsifying agents may be naturally occurring gums, for example, gum acacia or gum tragacanth; naturally occurring phosphatides, for example, soy bean, lecithin, and esters or partial esters derived from fatty acids; hexitol anhydrides, for example, sorbitan monooleate; and condensation products of partial esters with ethylene oxide, for example, polyoxyethylene sorbitan monooleate.

[0446] Liquid compositions for parenteral administration (for example as a sterile aqueous or oily solution for intravenous, subcutaneous, intramuscular, intraperitoneal or intramuscular dosing or as a suppository for rectal dosing) are also contemplated. Suitable pharmaceutically acceptable excipients include, but are not limited to, antioxidants (e.g., ascorbic acid and

sodium bisulfate), preservatives (e.g., benzyl alcohol, methyl parabens, ethyl or n-propyl, p-hydroxybenzoate), emulsifying agents, suspending agents, dispersing agents, solvents, fillers, bulking agents, detergents, buffers, vehicles, diluents, and/or adjuvants. For example, a suitable vehicle may be physiological saline solution or citrate buffered saline, possibly supplemented with other materials common in pharmaceutical compositions for parenteral administration. Neutral buffered saline or saline mixed with serum albumin are further exemplary vehicles. Those skilled in the art will readily recognize a variety of buffers that can be used in the pharmaceutical compositions and dosage forms contemplated herein. Typical buffers include, but are not limited to, pharmaceutically acceptable weak acids, weak bases, or mixtures thereof. As an example, the buffer components can be water soluble materials such as phosphoric acid, tartaric acids, lactic acid, succinic acid, citric acid, acetic acid, ascorbic acid, aspartic acid, glutamic acid, and salts thereof. Acceptable buffering agents include, for example, a Tris buffer, N-(2-Hydroxyethyl)piperazine-N'-(2-ethanesulfonic acid) (HEPES), 2-(N-Morpholino)ethanesulfonic acid (MES), 2-(N-Morpholino)ethanesulfonic acid sodium salt (MES), 3-(N-Morpholino)propanesulfonic acid (MOPS), and N-tris[Hydroxymethyl]methyl-3-aminopropanesulfonic acid (TAPS).

[0447] After a pharmaceutical composition has been formulated, it may be stored in sterile vials as a solution, suspension, gel, emulsion, solid, or dehydrated or lyophilized powder. Such formulations may be stored either in a ready-to-use form, a lyophilized form requiring reconstitution prior to use, a liquid form requiring dilution prior to use, or other acceptable form. In some embodiments, the pharmaceutical composition is provided in a single-use container (e.g., a single-use vial, ampoule, syringe, or autoinjector (similar to, e.g., an EpiPen®)), whereas a multi-use container (e.g., a multi-use vial) is provided in other embodiments.

[0448] Formulations can also include carriers to protect the composition against rapid degradation or elimination from the body, such as a controlled release formulation, including liposomes, hydrogels, prodrugs and microencapsulated delivery systems. For example, a time delay material such as glyceryl monostearate or glyceryl stearate alone, or in combination with a wax, may be employed. Any drug delivery apparatus may be used to deliver a composition described herein, including implants (e.g., implantable pumps) and catheter systems, slow injection pumps and devices, all of which are well known to the skilled artisan.

[0449] An effective amount of a crystalline form of Compound 1 described herein for use in therapy is an amount of Compound 1 sufficient to treat or prevent a proliferative condition referred to herein, slow its progression and/or reduce the symptoms associated with the condition.

- 5 [0450] An effective amount of a pharmaceutical composition described herein (i.e., a pharmaceutical composition comprising a crystalline form of Compound 1 described herein, a pharmaceutical composition prepared using a crystalline form of Compound 1 described herein, a pharmaceutical composition comprising a solid state form as described herein, or a pharmaceutical composition prepared using a solid state form described herein), for use in
10 therapy is an amount of Compound 1 sufficient to treat or prevent a proliferative condition referred to herein, slow its progression and/or reduce the symptoms associated with the condition.

- [0451] The size of the dose for therapeutic or prophylactic purposes of a crystalline form of Compound 1 described herein will naturally vary according to the nature and severity of the
15 conditions, the age and sex of the animal or patient and the route of administration, according to well-known principles of medicine. The size of the dose for therapeutic or prophylactic purposes of a pharmaceutical composition described herein will naturally vary according to the nature and severity of the conditions, the age and sex of the animal or patient and the route of administration, according to well-known principles of medicine.

- 20 [0452] In using a crystalline form of Compound 1 described herein for therapeutic or prophylactic purposes it will generally be administered so that a total daily dose of Compound 1 in the range, for example, 0.01 mg/kg to 100 mg/kg body weight is received. In using a pharmaceutical composition described herein for therapeutic or prophylactic purposes it will generally be administered so that a total daily dose of Compound 1 in the range, for
25 example, 0.01 mg/kg to 100 mg/kg body weight is received. Oral administration may also be suitable, particularly in tablet form.

E. Methods of Treatment

[0453] Provided herein are crystalline forms of compounds that function as inhibitors of PARG.

- 30 [0454] The present invention therefore provides a method of inhibiting PARG enzyme activity *in vitro* or *in vivo*, said method comprising contacting a cell with an effective amount

of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein.

[0455] The present invention also provides a method of treating a disease or disorder in which PARG activity is implicated in a patient in need of such treatment, said method comprising administering to said patient a therapeutically effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein. In an embodiment, the disease or disorder is an advanced or metastatic solid tumor. In an embodiment, the disease or disorder is cancer. In an embodiment, the cancer is ovarian, gastric, or breast cancer. In an embodiment, the cancer is lung, cervical, or pancreatic cancer. In an embodiment, the cancer is non-small cell lung cancer (NSCLC). In an embodiment, the cancer is prostate cancer. In an embodiment, the cancer is triple negative breast cancer (TNBC). In an embodiment, the cancer is a homologous recombinant deficient (HRD) cancer.

[0456] Provided herein is a method of inhibiting cell proliferation, *in vitro* or *in vivo*, said method comprising contacting a cell with an effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein.

[0457] Provided herein is a method of treating a proliferative disorder in a patient in need of such treatment, said method comprising administering to said patient a therapeutically effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein. In an embodiment, the proliferative disorder is a solid tumor. In an embodiment, the proliferative disorder is a metastatic solid tumor. In an embodiment, the proliferative disorder is cancer. In an embodiment, the cancer is ovarian, gastric, or breast cancer. In an embodiment, the cancer is lung, cervical or pancreatic cancer. In an embodiment, the cancer is prostate cancer. In an embodiment, the cancer is prostate cancer, colorectal cancer, or endometrial cancer. In an embodiment, the cancer is colorectal cancer. In an embodiment, the cancer is prostate cancer. In an embodiment, the cancer is endometrial cancer. In an embodiment, the cancer is ovarian cancer, gastric cancer, breast cancer, lung cancer, cervical cancer, pancreatic cancer, prostate cancer, colorectal cancer, or endometrial cancer. In an embodiment, the cancer is a homologous recombinant deficient (HRD) cancer.

[0458] Provided herein is a method of treating cancer in a patient in need of such treatment, said method comprising administering to said patient a therapeutically effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein. In an embodiment, the cancer is ovarian, gastric, or breast cancer. In an embodiment, the cancer is lung, cervical or pancreatic cancer. In an embodiment, the cancer is a homologous recombinant deficient (HRD) cancer.

[0459] Provided herein is a method of treating and/or preventing a homologous recombinant deficient (HRD) cancer in a patient comprising administering to the patient a therapeutically effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein. In an embodiment, the patient is in recognized need of such treatment. In an embodiment, the homologous recombinant deficient (HRD) cancer is breast cancer, ovarian cancer, gastric cancer, prostate cancer, lung cancer, cervical cancer, or pancreatic cancer. In an embodiment, the homologous recombinant deficient (HRD) cancer is breast cancer, ovarian cancer, gastric cancer, lung cancer, cervical cancer, pancreatic cancer, prostate cancer, colorectal cancer, or endometrial cancer.

[0460] In some embodiments, provided are methods of treating and/or preventing a cancer in a subject in need thereof, the methods comprising administering to the subject a therapeutically effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, thereby treating the cancer in the subject, wherein the cancer is a homologous recombinant deficient (HRD) cancer, and the cancer is Estrogen Receptor (ER) positive. In an embodiment, the cancer is breast cancer or ovarian cancer.

[0461] In some embodiments, provided are methods of treating and/or preventing a cancer in a subject in need thereof, the methods comprising administering to the subject a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, thereby treating the cancer in the subject, wherein the cancer is an HRD cancer, the cancer is ER positive, and the cancer is optionally Progesterone Receptor (PR) positive. In some embodiments, provided are methods of treating and/or preventing a cancer in a subject in need thereof, the methods comprising administering to the subject a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, thereby treating the cancer in the subject, wherein the cancer is an HRD

cancer, the cancer is ER positive, and the cancer is PR positive. In an embodiment, the cancer is breast cancer or ovarian cancer.

5 [0462] In some embodiments, provided are methods of treating and/or preventing a cancer in a subject in need thereof, the methods comprising administering to the subject a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, thereby treating the cancer in the subject, wherein the cancer is an HRD cancer, the cancer is ER positive, and the cancer is optionally human epidermal growth factor receptor 2 (HER2) negative. In some embodiments, provided are methods of treating and/or preventing a cancer in a subject in need thereof, the methods comprising administering to the subject a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, thereby treating the cancer in the subject, wherein the cancer is HRD cancer, the cancer is ER positive, and the cancer is HER2 negative. In an embodiment, the cancer is breast or ovarian cancer.

15 [0463] In some embodiments, provided are methods of treating and/or preventing a cancer in a subject in need thereof, the methods comprising administering to the subject a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, thereby treating the cancer in the subject, wherein the cancer is an HRD cancer, the cancer is ER positive, the cancer is optionally PR positive, and the cancer is optionally HER2 negative. In an embodiment, the cancer is breast or ovarian cancer.

20 [0464] In some embodiments, provided are methods of treating and/or preventing a breast cancer in a subject in need thereof, the methods comprising administering to the subject a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, thereby treating the cancer in the subject, wherein the cancer is an HRD cancer, the cancer is ER positive, the cancer is optionally PR positive, and the cancer is optionally HER2 negative.

25 [0465] Provided herein is a method of treating and/or preventing a cancer in a patient, where the cancer is characterized by a reduction or absence of BRCA1 and/or BRCA2 gene expression, an absence or mutation of BRCA1 and/or BRCA2 genes, or a reduced function of BRCA1 and/or BRCA2 proteins, comprising administering to the patient a therapeutically effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein. In an embodiment, the cancer is ovarian,

gastric, or breast cancer. In an embodiment, the cancer is lung, cervical or pancreatic cancer.
In an embodiment, the cancer is prostate cancer.

[0466] In an embodiment, the cancer is PARP inhibitor-resistant. In some embodiments, the PARP inhibitor-resistant cancer is resistant to any one or more of niraparib, olaparib, 5 rucaparib, talazoparib, veliparib, AZD5305, and AZD9574. In some embodiments, the PARP inhibitor-resistant cancer is resistant to niraparib. In some embodiments the PARP inhibitor-resistant cancer is resistant to olaparib. In some embodiments, the PARP inhibitor-resistant cancer is ovarian cancer, breast cancer, or pancreatic cancer.

[0467] In an embodiment, the cancer is platin-resistant. In some embodiments, the platin- 10 resistant cancer is resistant to any one or more of cisplatin, carboplatin, satraplatin, heptaplatin, picoplatin, nedaplatin, triplatin, lipoplatin, and oxaliplatin. In some embodiments, the platin-resistant cancer is resistant to cisplatin. In some embodiments, the platin-resistant cancer is resistant to carboplatin.

[0468] Provided herein are methods of treating cancer in a patient in need of such 15 treatment, said method comprising administering to said patient a therapeutically effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, wherein the patient has been determined to be resistant to one or more PARP inhibitors. In some embodiments, said methods comprise administering to said patient a therapeutically effective amount of a crystalline form 20 of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, wherein said patient has been diagnosed as resistant to one or more PARP inhibitors.

[0469] Provided herein are methods of treating cancer in a patient in need of such treatment, said method comprising administering to said patient a therapeutically effective 25 amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein, wherein the patient has been determined to be resistant to platinum based chemotherapeutics. In some embodiments, said methods comprise administering to said patient a therapeutically effective amount of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and 30 described herein, wherein said patient has been diagnosed as resistant to one or more platinum based chemotherapeutics.

[0470] Provided herein is a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein for use in therapy.

[0471] Provided herein is a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein for use in the treatment
5 of a proliferative condition.

[0472] Provided herein is a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein for use in the treatment of cancer. In a particular embodiment, the cancer is human cancer. In an embodiment, the cancer is ovarian, gastric, or breast cancer. In an embodiment, the cancer is lung, cervical or
10 pancreatic cancer. In an embodiment, the cancer is prostate cancer. In an embodiment, the cancer is a homologous recombinant deficient (HRD) cancer.

[0473] Provided herein is a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein for use in the inhibition of PARC enzyme activity.

[0474] Provided herein is a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein for use in the treatment
15 of a disease or disorder in which PARC activity is implicated.

[0475] Provided herein is a use of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein in the manufacture of a
20 medicament for the treatment of a proliferative condition.

[0476] Provided herein is a use of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein in the manufacture of a medicament for the treatment of cancer. Suitably, the medicament is for use in the treatment of human cancers. In an embodiment, the cancer is ovarian, gastric, or breast cancer. In an
25 embodiment, the cancer is lung, cervical or pancreatic cancer. In an embodiment, the cancer is prostate cancer. In an embodiment, the cancer is a homologous recombinant deficient (HRD) cancer.

[0477] Provided herein is a use of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein in the manufacture of a
30 medicament for the inhibition of PARC enzyme activity.

[0478] Provided herein is a use of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein in the manufacture of a medicament for the treatment of a disease or disorder in which PARG activity is implicated.

5 [0479] The present disclosure also contemplates the use of a crystalline form of Compound 1 described herein, or a pharmaceutical composition thereof as defined and described herein in combination with other therapeutically active agents or compounds as described herein in order to treat the diseases, disorders and conditions contemplated by the present disclosure. In some embodiments, the additional therapeutic agent is a chemotherapeutic agent. In some
10 embodiments, the additional therapeutic agent is a MAT2A inhibitor. In some embodiments, the additional therapeutic agent is a PRMT5 inhibitor. In some embodiments, the PRMT5 inhibitor is a MTA-cooperative PRMT5 inhibitor. In some embodiments, the additional therapeutic agent is a cell cycle checkpoint inhibitor. In some embodiments, the additional therapeutic agent is immunecheckpoint inhibitor.

[0480] The term “proliferative disorder” and “proliferative condition” are used
15 interchangeably herein and pertain to an unwanted or uncontrolled cellular proliferation of excessive or abnormal cells which is undesired, such as, neoplastic or hyperplastic growth, whether in vitro or in vivo. Examples of proliferative conditions include, but are not limited to, pre-malignant and malignant cellular proliferation, including but not limited to, malignant neoplasms and tumors, cancers, leukemias, psoriasis, bone diseases, fibroproliferative
20 disorders (e.g., of connective tissues), and atherosclerosis. Any type of cell may be treated, including but not limited to, lung, colon, breast, ovarian, prostate, gastric, liver, pancreas, brain, and skin. Proliferative disorders also include, for example, advanced or metastatic solid tumors.

[0481] The anti-proliferative effects of a crystalline form of Compound 1 described herein,
25 or a pharmaceutical composition thereof as defined and described herein, have particular application in the treatment of human cancers (by virtue of their inhibition of PARG enzyme activity).

[0482] The anti-cancer effect may arise through one or more mechanisms, including but not limited to, the regulation of cell proliferation, the inhibition of angiogenesis (the
30 formation of new blood vessels), the inhibition of metastasis (the spread of a tumor from its origin), the inhibition of invasion (the spread of tumor cells into neighboring normal structures), or the promotion of apoptosis (programmed cell death).

[0483] In a particular embodiment of the disclosure, the proliferative condition to be treated is cancer.

F. Methods of Preparation

[0484] Compound 1 can be prepared, for example, as described in Example 1.

- 5 [0485] The various salt forms described herein can be prepared, for example, as described in the provided Examples. It is understood that there may be more than one crystallization method that will yield the described free base, HCl, sulfate, tosylate, besylate, mesylate, maleate, citrate, fumarate, malate, and succinate forms.

[0486] In one aspect, the present disclosure provides a method for preparing a crystalline form of Compound 1. In one aspect, the present disclosure provides a method for preparing a crystalline salt form of Compound 1. In some embodiments, the method includes:

- 1) forming a first mixture comprising Compound 1 and a first solvent;
- 2) adding an acid to the first mixture to form a second mixture;
- 3) optionally adding a second solvent to the second mixture;
- 15 4) stirring the second mixture;
- 5) isolating the precipitate; and
- 6) drying the precipitate to provide the crystalline form of the salt of Compound 1.

[0487] In some embodiments, the method for preparing a crystalline form of Compound 1 includes:

- 20 1) forming a first mixture comprising Compound 1 and a first solvent;
- 2) adding an acid to the first mixture to form a second mixture;
- 3) optionally adding a second solvent to the second mixture to form a third mixture;
- 4) stirring the second mixture when steps 1) and 2) are conducted, or stirring the third mixture when steps 1) to 3) are conducted;
- 25 5) isolating the precipitate; and
- 6) drying the precipitate to provide the crystalline form of the salt of Compound 1.

[0488] In another aspect, the present disclosure provides a method for preparing a crystalline free base form of Compound 1. In some embodiments, the method includes:

- 1) forming a first mixture comprising Compound 1 and a first solvent;
- 2) optionally adding a second solvent to the first mixture;
- 5 3) stirring the first mixture;
- 4) isolating the precipitate; and
- 5) drying the precipitate to provide the crystalline form of Compound 1.

[0489] In some embodiments, the method for preparing a crystalline free base form of Compound 1 includes:

- 10 1) forming a first mixture comprising Compound 1 and a first solvent;
- 2) optionally adding a second solvent to the first mixture to form a second mixture;
- 3) stirring the first mixture when step 1) is conducted, or stirring the second mixture when steps 1) and 2) are conducted;
- 4) isolating the precipitate; and
- 15 5) drying the precipitate to provide the crystalline form of Compound 1.

[0490] In some embodiments, all steps as described above are performed at room temperature. In some embodiments, all steps as described above are conducted at 10 °C to 40 °C.

[0491] In some embodiments, the first mixture is formed at room temperature. In some
20 embodiments, the first mixture is heated to a temperature of between about 40 °C and about 70 °C. In some embodiments, the first mixture is heated to a temperature of about 50 °C. In some embodiments, the first mixture is heated to a temperature of about 60 °C.

[0492] In some embodiments, the first solvent is selected from the group consisting of acetone, acetonitrile, ethyl acetate, methanol, ethanol, isopropanol, and tetrahydrofuran. In
25 some embodiments, the first solvent is selected from the group consisting of acetone, acetonitrile, ethyl acetate, methanol, ethanol, isopropanol, NMP (N-methyl pyrrolidone), DMSO (dimethylsulfoxide), and tetrahydrofuran.

[0493] In some embodiments, the acid is selected from the group consisting of HCl, sulfuric acid, p-toluenesulfonic acid, benzenesulfonic acid, methanesulfonic acid, maleic acid, citric acid, fumaric acid, L-malic acid, and succinic acid.

5 [0494] In some embodiments, the second solvent is selected from the group consisting of heptane, methyl tert-butyl ether (MTBE), and water. In some embodiments, the second solvent is selected from the group consisting of hexane, heptane, methyl tert-butyl ether (MTBE), and water.

[0495] In some embodiments, provided is a method of preparing HCl salt Form I of Compound 1. In some embodiments, the method includes 1) forming a first mixture
10 comprising Compound 1 and methanol, and heating the first mixture to a temperature of about 50 °C, 2) adding HCl to form a second mixture, 3) adding MTBE to the second mixture, 4) stirring the second mixture at room temperature, 5) isolating the solids, and 6) drying the solids. In some embodiments, the method includes 1) forming a first mixture comprising Compound 1 and methanol, and heating the first mixture to a temperature of
15 about 50 °C, 2) adding HCl to form a second mixture, 3) adding MTBE to the second mixture to form a third mixture, 4) stirring the third mixture at room temperature, 5) isolating the solids, and 6) drying the solids. In some embodiments, the solids are dried under vacuum. In some embodiments, the solids are dried at elevated temperature. In some embodiments, the solids are dried under vacuum at a temperature of about at 50 °C.

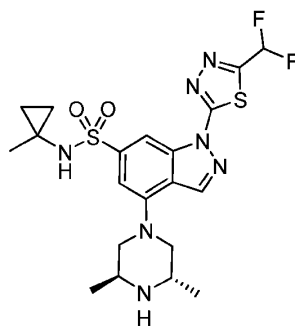
20 [0496] In some embodiments, provided is a method of preparing free base Form I of Compound 1. In some embodiments, the method includes 1) forming a first mixture comprising Compound 1 and an alcohol solvent, 3) stirring the first mixture, 4) isolating the precipitate, and 5) drying the precipitate. In some embodiments, the alcohol solvent is methanol or ethanol. In some embodiments, the method includes slurrying Compound 1 in
25 methanol or ethanol at elevated temperature. In some embodiments, the method includes slurrying Compound 1 in methanol or ethanol at about 50 °C. In some embodiments, the method includes slurrying Compound 1 in ethanol at about 50 °C. In some embodiments, the method includes isolating the solids, e.g. by filtration. In some embodiments, the solids are dried under vacuum. In some embodiments, the solids are dried at elevated temperature. In
30 some embodiments, the solids are dried under vacuum at a temperature of about at 50 °C.

[0497] In some embodiments, provided is a method of preparing free base Form II of Compound 1. In some embodiments, the method includes 1) forming a first mixture

- comprising Compound 1 and DMSO, 2) adding water to the first mixture, 3) stirring the first mixture, 4) isolating the precipitate, and 5) drying the precipitate. In some embodiments, the method includes 1) forming a first mixture comprising Compound 1 and DMSO, 2) adding water to the first mixture to form a second mixture, 3) stirring the second mixture, 4) isolating the precipitate, and 5) drying the precipitate. In some embodiments, the method includes isolating the solids, e.g. by filtration. In some embodiments, the solids are dried under vacuum. In some embodiments, the solids are dried at elevated temperature. In some embodiments, the solids are dried under vacuum at a temperature of about at 50 °C.

G. Non-limiting Exemplary Embodiments

- 10 [0498] **Embodiment 1.** A crystalline form of Compound 1



(Compound 1),

or a salt thereof.

- [0499] **Embodiment 2.** The crystalline form of embodiment 1, wherein the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.
- 15 [0500] **Embodiment 3.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is HCl salt Form I.
- [0501] **Embodiment 4.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.2 and 17.7 degrees 2θ (± 0.2 degrees 2θ).
- 20 [0502] **Embodiment 5.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks selected from 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9.

- [0503] **Embodiment 6.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising four peaks selected from 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9.
- 5 [0504] **Embodiment 7.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks selected from 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9.
- [0505] **Embodiment 8.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.2, 13.7, and 17.7 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0506] **Embodiment 9.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.7, 13.2, 13.7, and 17.7 degrees 2θ (± 0.2 degrees 2θ).
- [0507] **Embodiment 12.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.7, 13.2, 13.7, 17.7, 15 and 18.4 degrees 2θ (± 0.2 degrees 2θ).
- [0508] **Embodiment 11.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.7, 13.2, 13.7, 17.2, 17.7, and 18.4 degrees 2θ (± 0.2 degrees 2θ).
- [0509] **Embodiment 12.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.7, 13.2, 13.7, 17.2, 20 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ).
- [0510] **Embodiment 13.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ).
- 25 [0511] **Embodiment 14.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 7.0, 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ).
- [0512] **Embodiment 15.** The HCl salt Form I, according to embodiment 3, characterized by an XRPD pattern comprising peaks at 7.0, 11.7, 12.2, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, 30 and 20.0 degrees 2θ (± 0.2 degrees 2θ).

- [0513] **Embodiment 16.** The HCl salt Form I, according to any one of embodiments 8 to 15, characterized by an XRPD pattern further comprising one or more peaks selected from 20.9, 24.3, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).
- 5 [0514] **Embodiment 17.** The HCl salt Form I, according to any one of embodiments 8 to 15, characterized by an XRPD pattern further comprising one or more peaks selected from 20.6, 20.9, 21.2, 21.7, 22.3, 24.3, 25.0, 25.3, 25.6, 26.1, 28.9 degrees 2θ (± 0.2 degrees 2θ).
- [0515] **Embodiment 18.** The HCl salt Form I, according to any one of embodiments 8 to 15, characterized by an XRPD pattern further comprising a peak at 28.9 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0516] **Embodiment 19.** The HCl salt Form I, according to any one of embodiments 8 to 15, characterized by an XRPD pattern further comprising peaks at 26.1 and 28.9 degrees 2θ (± 0.2 degrees 2θ).
- [0517] **Embodiment 20.** The HCl salt Form I, according to any one of embodiments 8 to 15, characterized by an XRPD pattern further comprising peaks at 24.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).
- 15 [0518] **Embodiment 21.** The HCl salt Form I, according to any one of embodiments 8 to 15, characterized by an XRPD pattern further comprising peaks at 20.9, 24.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).
- [0519] **Embodiment 22.** The HCl salt Form I, according to any one of embodiments 8 to 20, characterized by an XRPD pattern further comprising peaks at 20.9, 24.3, 25.3, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).
- [0520] **Embodiment 23.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in **Table 3**.
- 25 [0521] **Embodiment 24.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 20% as listed in **Table 3**.
- [0522] **Embodiment 25.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a 30 relative intensity of at least 30% as listed in **Table 3**.

- [0523] **Embodiment 26.** The HCl salt Form I, according to embodiment 3, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 7.
- [0524] **Embodiment 27.** The HCl salt Form I, according to any one of embodiments 3 to 26, characterized by a differential scanning calorimetry thermogram (DSC) comprising no
5 endotherm prior to decomposition.
- [0525] **Embodiment 28.** The HCl salt Form I, according to any one of embodiments 3 to 26, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 8.
- [0526] **Embodiment 29.** The HCl salt Form I, according to any one of embodiments 3 to
10 28, characterized by no weight loss upon heating prior to decomposition, as measured by thermal gravimetric analysis (TGA).
- [0527] **Embodiment 30.** The HCl salt Form I, according to any one of embodiments 3 to 28, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 8.
- [0528] **Embodiment 31.** The HCl salt Form I, according to any one of embodiments 3 to
15 30, characterized by a weight gain of about 0.5 % after undergoing a dynamic vapor sorption cycle from about 0% relative humidity (RH) to about 90% RH at 25 °C.
- [0529] **Embodiment 32.** The HCl salt Form I, according to any one of embodiments 3 to 31, characterized by a dynamic vapor sorption profile substantially as shown in FIG. 9.
- [0530] **Embodiment 33.** The HCl salt Form I, according to any one of embodiments 3 to
20 32, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 10.
- [0531] **Embodiment 34.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is free base Form I.
- [0532] **Embodiment 35.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0 and 18.3 degrees 2 θ ($\pm$ 0.2
25 degrees 2 θ).
- [0533] **Embodiment 36.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks selected from 9.0, 12.7, 13.7,
30 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).

- [0534] **Embodiment 37.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising four peaks selected from 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- 5 [0535] **Embodiment 38.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks selected from 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- [0536] **Embodiment 39.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, and 18.3 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0537] **Embodiment 40.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.0, and 18.3 degrees 2θ (± 0.2 degrees 2θ).
- [0538] **Embodiment 41.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.0, 18.3, and 19.4
15 degrees 2θ (± 0.2 degrees 2θ).
- [0539] **Embodiment 42.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 16.4, 18.0, 18.3, and 19.4 degrees 2θ (± 0.2 degrees 2θ).
- [0540] **Embodiment 43.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 16.4, 18.0, 18.3, 18.9,
20 and 19.4 degrees 2θ (± 0.2 degrees 2θ).
- [0541] **Embodiment 44.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, and 19.4 degrees 2θ (± 0.2 degrees 2θ).
- 25 [0542] **Embodiment 45.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- [0543] **Embodiment 46.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 11.5, 12.7, 13.7, 16.4, 18.0,
30 18.3, 18.9, 19.4, and 19.7 degrees 2θ (± 0.2 degrees 2θ).

- [0544] **Embodiment 47.** The free base Form I of any one of embodiments 39 to 46, characterized by an XRPD pattern further comprising one or more peaks selected from 22.5, 23.2, 23.5, 24.6, and 27.8 degrees 2θ (± 0.2 degrees 2θ).
- 5 [0545] **Embodiment 48.** The free base Form I of any one of embodiments 39 to 46, characterized by an XRPD pattern further comprising one or more peaks selected from 23.2, 23.5, and 24.6 degrees 2θ (± 0.2 degrees 2θ).
- [0546] **Embodiment 49.** The free base Form I of embodiment 34, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 1.
- 10 [0547] **Embodiment 50.** The free base Form I, according to any one of embodiments 34 to 49, characterized by a differential scanning calorimetry thermogram (DSC) comprising a melting point peak at about 200 °C.
- [0548] **Embodiment 51.** The free base Form I, according to any one of embodiments 34 to 49, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 2.
- 15 [0549] **Embodiment 52.** The free base Form I, according to any one of embodiments 34 to 51, characterized by no weight loss upon heating prior to decomposition, as measured by thermal gravimetric analysis (TGA).
- [0550] **Embodiment 53.** The free base Form I, according to any one of embodiments 34 to 51, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 2.
- 20 [0551] **Embodiment 54.** The free base Form I, according to any one of embodiments 34 to 53, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 3.
- [0552] **Embodiment 55.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is free base Form II.
- 25 [0553] **Embodiment 56.** The free base Form II of embodiment 55, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3 and 18.7 degrees 2θ (± 0.2 degrees 2θ).

- [0554] **Embodiment 57.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising three peaks selected from 12.3, 15.6, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- 5 [0555] **Embodiment 58.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising four peaks selected from 12.3, 15.6, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- [0556] **Embodiment 59.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising five peaks selected from 12.3, 15.6, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0557] **Embodiment 60.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, and 18.7 degrees 2θ (± 0.2 degrees 2θ).
- [0558] **Embodiment 61.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 17.1, and 18.7
15 degrees 2θ (± 0.2 degrees 2θ).
- [0559] **Embodiment 62.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 17.1, 18.7, and 19.5 degrees 2θ (± 0.2 degrees 2θ).
- [0560] **Embodiment 63.** The free base Form II of embodiment **55**, characterized by an
20 X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- [0561] **Embodiment 64.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 15.6, 16.4, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- 25 [0562] **Embodiment 65.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 13.4, 15.6, 16.4, 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- [0563] **Embodiment 66.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 13.4, 15.6, 16.1, 16.4,
30 17.1, 18.7, 19.5, and 19.7 degrees 2θ (± 0.2 degrees 2θ).

- [0564] **Embodiment 67.** The free base Form II of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 12.3, 13.4, 14.1, 15.6, 16.1, 16.4, 17.1, 18.7, 19.5, and 19.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 5 [0565] **Embodiment 68.** The free base Form II of any one of embodiments **60 to 67**, characterized by an XRPD pattern further comprising one or more peaks selected from 20.2, 20.7, 23.0, 25.0, 25.7, 26.5, and 31.4 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0566] **Embodiment 69.** The free base Form II of any one of embodiments **60 to 67**, characterized by an XRPD pattern further comprising one or more peaks selected from 20.2, 20.7, 23.0, 25.7, and 26.5 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 10 [0567] **Embodiment 70.** The free base Form I of embodiment **55**, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 4**.
- [0568] **Embodiment 71.** The free base Form II, according to any one of embodiments **55 to 70**, characterized by a differential scanning calorimetry thermogram (DSC) comprising a first endotherm peak at about 148 °C and/or a second endotherm peak at about 198 °C.
- 15 [0569] **Embodiment 72.** The free base Form II, according to any one of embodiments **55 to 70**, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with **FIG. 5**.
- [0570] **Embodiment 73.** The free base Form II, according to any one of embodiments **55 to 72**, characterized by a 15.1% weight loss upon heating to about 158 °C, as measured by
- 20 thermal gravimetric analysis (TGA).
- [0571] **Embodiment 74.** The free base Form II, according to any one of embodiments **55 to 72**, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 5**.
- [0572] **Embodiment 75.** The free base Form II, according to any one of embodiments **55 to 74**, characterized by a polarized light microscope (PLM) profile substantially as shown in
- 25 **FIG. 6**.
- [0573] **Embodiment 76.** The crystalline form of embodiment **1** or embodiment **2**, wherein the crystalline form is sulfate salt Form I.

- [0574] **Embodiment 77.** The sulfate salt Form I of embodiment 76, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.0, 8.5, 13.2, 15.6, and 16.1 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 5 [0575] **Embodiment 78.** The sulfate salt Form I of embodiment 77, characterized by an XRPD pattern further comprising one or more peaks selected from 8.8, 12.9, 15.0, 17.2, and 19.0 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0576] **Embodiment 79.** The sulfate salt Form I of embodiment 77 or 78, characterized by an XRPD pattern further comprising one or more peaks selected from 20.2, 22.1, 24.3, 25.1, and 26.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 10 [0577] **Embodiment 80.** The sulfate salt Form I of embodiment 76, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 11.
- [0578] **Embodiment 81.** The sulfate salt Form I, according to any one of embodiments 76 to 80, characterized by a differential scanning calorimetry thermogram (DSC) comprising a first endotherm peak at about 118 °C, a second endotherm peak at about 220 °C, an
- 15 exotherm peak at about 231 °C, and/or a third endotherm peak at about 279 °C.
- [0579] **Embodiment 82.** The sulfate salt Form I, according to any one of embodiments 76 to 80, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 12.
- [0580] **Embodiment 83.** The sulfate salt Form I, according to any one of embodiments
- 20 76 to 82, characterized by about 2.8% weight loss upon heating to around 130 °C, as measured by thermal gravimetric analysis (TGA).
- [0581] **Embodiment 84.** The sulfate salt Form I, according to any one of embodiments 76 to 82, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 12.
- 25 [0582] **Embodiment 85.** The sulfate salt Form I, according to any one of embodiments 76 to 84, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 13.
- [0583] **Embodiment 86.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is tosylate salt Form I.

- [0584] **Embodiment 87.** The tosylate salt Form I of embodiment **86**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.8, 9.6, 14.5, 18.2, and 19.5 degrees 2θ (± 0.2 degrees 2θ).
- 5 [0585] **Embodiment 88.** The tosylate salt Form I of embodiment **87**, characterized by an XRPD pattern further comprising one or more peaks selected from 4.7, 11.7, 13.7, 15.5, and 15.9 degrees 2θ (± 0.2 degrees 2θ).
- [0586] **Embodiment 89.** The tosylate salt Form I of embodiment **87** or **88**, characterized by an XRPD pattern further comprising one or more peaks selected from 21.4, 22.8, 24.0, 24.3, and 29.3 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0587] **Embodiment 90.** The tosylate salt Form I of embodiment **86**, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 14**.
- [0588] **Embodiment 91.** The tosylate salt Form I, according to any one of embodiments **86** to **90**, characterized by a differential scanning calorimetry thermogram (DSC) comprising a melting point peak at about 231 °C.
- 15 [0589] **Embodiment 92.** The tosylate salt Form I, according to any one of embodiments **86** to **90**, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with **FIG. 15**.
- [0590] **Embodiment 93.** The tosylate salt Form I, according to any one of embodiments **86** to **92**, characterized by about 1.2% weight loss upon heating from about 110 °C to about 20 230 °C, as measured by thermal gravimetric analysis (TGA).
- [0591] **Embodiment 94.** The tosylate salt Form I, according to any one of embodiments **86** to **92**, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 15**.
- [0592] **Embodiment 95.** The tosylate salt Form I, according to any one of embodiments 25 **86** to **94**, characterized by a polarized light microscope (PLM) profile substantially as shown in **FIG. 16**.
- [0593] **Embodiment 96.** The crystalline form of embodiment **1** or embodiment **2**, wherein the crystalline form is tosylate salt Form II.

- [0594] **Embodiment 97.** The tosylate salt Form II of embodiment 96, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.7, 14.3, 15.4, 17.3, and 19.1 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 5 [0595] **Embodiment 98.** The tosylate salt Form II of embodiment 97, characterized by an XRPD pattern further comprising one or more peaks selected from 9.1, 10.2, 13.0, 17.0, and 18.4 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0596] **Embodiment 99.** The tosylate salt Form II of embodiment 97 or 98, characterized by an XRPD pattern further comprising one or more peaks selected from 20.1, 20.5, 23.8, 24.2, and 26.2 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 10 [0597] **Embodiment 100.** The tosylate salt Form II of embodiment 96, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 17.
- [0598] **Embodiment 101.** The tosylate salt Form II, according to any one of embodiments 96 to 100, characterized by a differential scanning calorimetry thermogram (DSC) comprising a melting point peak at about 229 °C.
- 15 [0599] **Embodiment 102.** The tosylate salt Form II, according to any one of embodiments 96 to 100, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 18.
- [0600] **Embodiment 103.** The tosylate salt Form II, according to any one of embodiments 96 to 102, characterized by about 1.4% weight loss upon heating from about 90 °C to about 20 230 °C, as measured by thermal gravimetric analysis (TGA).
- [0601] **Embodiment 104.** The tosylate salt Form II, according to any one of embodiments 96 to 102, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 18.
- [0602] **Embodiment 105.** The tosylate salt Form II, according to any one of embodiments 25 96 to 104, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 19.
- [0603] **Embodiment 106.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is tosylate salt Form III.

- [0604] **Embodiment 107.** The tosylate salt Form III of embodiment **106**, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.9, 14.5, 15.5, 18.2, and 19.5 degrees 2θ (± 0.2 degrees 2θ).
- 5 [0605] **Embodiment 108.** The tosylate salt Form III of embodiment **107**, characterized by an XRPD pattern further comprising one or more peaks selected from 9.7, 14.1, 14.9, 16.0, and 16.9 degrees 2θ (± 0.2 degrees 2θ).
- [0606] **Embodiment 109.** The tosylate salt Form III of embodiment **107** or **108**, characterized by an XRPD pattern further comprising one or more peaks selected from 21.2, 21.4, 22.0, 24.0, and 24.4 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0607] **Embodiment 110.** The tosylate salt Form III of embodiment **106**, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 20**.
- [0608] **Embodiment 111.** The tosylate salt Form III, according to any one of embodiments **106** to **110**, characterized by a differential scanning calorimetry thermogram (DSC) comprising a melting point peak at 222 °C.
- 15 [0609] **Embodiment 112.** The tosylate salt Form III, according to any one of embodiments **106** to **110**, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with **FIG. 21**.
- [0610] **Embodiment 113.** The tosylate salt Form III, according to any one of embodiments **106** to **112**, characterized by about 2.6% weight loss upon heating from about 20 110 °C to about 222 °C, as measured by thermal gravimetric analysis (TGA).
- [0611] **Embodiment 114.** The tosylate salt Form III, according to any one of embodiments **106** to **112**, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 21**.
- [0612] **Embodiment 115.** The tosylate salt Form III, according to any one of 25 embodiments **106** to **114**, characterized by a weight gain of about 3.2% after undergoing a dynamic vapor sorption cycle from about 0 % relative humidity (RH) to about 90 % RH at 25 °C.
- [0613] **Embodiment 116.** The tosylate salt Form III, according to any one of 30 embodiments **106** to **114**, characterized by a dynamic vapor sorption profile substantially as shown in **FIG. 22**.

- [0614] **Embodiment 117.** The tosylate salt Form III, according to any one of embodiments 106 to 116, characterized by a polarized light microscope (PLM) profile substantially as shown in **FIG. 23**.
- 5 [0615] **Embodiment 118.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is besylate salt Form I.
- [0616] **Embodiment 119.** The besylate salt Form I of embodiment 118, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.8, 14.5, 15.4, 17.3, and 19.1 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0617] **Embodiment 120.** The besylate salt Form I of embodiment 119, characterized by an XRPD pattern further comprising one or more peaks selected from 15.0, 15.9, 17.7, 18.3, and 19.6 degrees 2θ (± 0.2 degrees 2θ).
- [0618] **Embodiment 121.** The besylate salt Form I of embodiment 119 or 120, characterized by an XRPD pattern further comprising one or more peaks selected from 20.3, 20.8, 24.0, 25.2, and 26.0 degrees 2θ (± 0.2 degrees 2θ).
- 15 [0619] **Embodiment 122.** The besylate salt Form I of embodiment 118, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 24**.
- [0620] **Embodiment 123.** The besylate salt Form I, according to any one of embodiments 119 to 122, characterized by a differential scanning calorimetry thermogram (DSC) comprising an endothermic peak at about 202 °C.
- 20 [0621] **Embodiment 124.** The besylate salt Form I, according to any one of embodiments 118 to 122, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with **FIG. 25**.
- [0622] **Embodiment 125.** The besylate salt Form I, according to any one of embodiments 118 to 124, characterized by about 0.6% weight loss upon heating from about 100 °C to
25 about 210 °C, as measured by thermal gravimetric analysis (TGA).
- [0623] **Embodiment 126.** The besylate salt Form I, according to any one of embodiments 118 to 124, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 25**.

- [0624] **Embodiment 127.** The besylate salt Form I, according to any one of embodiments 118 to 126, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 26.
- 5 [0625] **Embodiment 128.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is besylate salt Form II.
- [0626] **Embodiment 129.** The besylate salt Form II of embodiment 128, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 7.6, 15.2, 15.7, 19.2, and 19.7 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0627] **Embodiment 130.** The besylate salt Form II of embodiment 129, characterized by an XRPD pattern further comprising one or more peaks selected from 9.0, 10.0, 12.4, 14.7, and 18.1 degrees 2θ (± 0.2 degrees 2θ).
- [0628] **Embodiment 131.** The besylate salt Form II of embodiment 129 or 130, characterized by an XRPD pattern further comprising one or more peaks selected from 10.4, 20.1, 22.9, 25.0, and 26.5 degrees 2θ (± 0.2 degrees 2θ).
- 15 [0629] **Embodiment 132.** The besylate salt Form II of embodiment 128, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 27.
- [0630] **Embodiment 133.** The besylate salt Form II, according to any one of embodiments 128 to 132, characterized by a differential scanning calorimetry thermogram (DSC) comprising an endotherm peak at about 165 °C.
- 20 [0631] **Embodiment 134.** The besylate salt Form II, according to any one of embodiments 128 to 132, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 28.
- [0632] **Embodiment 135.** The besylate salt Form II, according to any one of embodiments 128 to 134, characterized by about 0.5% weight loss upon heating from about 25 64 °C to about 185 °C, as measured by thermal gravimetric analysis (TGA).
- [0633] **Embodiment 136.** The besylate salt Form II, according to any one of embodiments 128 to 134, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 28.

- [0634] **Embodiment 137.** The besylate salt Form II, according to any one of embodiments 128 to 136, characterized by a polarized light microscope (PLM) profile substantially as shown in **FIG. 29**.
- 5 [0635] **Embodiment 138.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is mesylate salt Form I.
- [0636] **Embodiment 139.** The mesylate salt Form I of embodiment 138, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 14.2, 17.0, 18.0, 18.6, and 19.3 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0637] **Embodiment 140.** The mesylate salt Form I of embodiment 139, characterized by an XRPD pattern further comprising one or more peaks selected from 11.0, 11.9, 12.7, 15.1, and 17.5 degrees 2θ (± 0.2 degrees 2θ).
- [0638] **Embodiment 141.** The mesylate salt Form I of embodiment 139 or 140, characterized by an XRPD pattern further comprising one or more peaks selected from 20.7, 21.2, 22.1, 24.1, and 25.0 degrees 2θ (± 0.2 degrees 2θ).
- 15 [0639] **Embodiment 142.** The mesylate salt Form I of embodiment 139, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 30**.
- [0640] **Embodiment 143.** The mesylate salt Form I, according to any one of embodiments 138 to 142, characterized by a differential scanning calorimetry thermogram (DSC) comprising an endotherm peak at about 222 °C.
- 20 [0641] **Embodiment 144.** The mesylate salt Form I, according to any one of embodiments 138 to 142, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with **FIG. 31**.
- [0642] **Embodiment 145.** The mesylate salt Form I, according to any one of embodiments 138 to 144, characterized by about 0.5% weight loss upon heating from about 125 °C to
25 about 225 °C, as measured by thermal gravimetric analysis (TGA).
- [0643] **Embodiment 146.** The mesylate salt Form I, according to any one of embodiments 138 to 144, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with **FIG. 31**.

- [0644] **Embodiment 147.** The mesylate salt Form I, according to any one of embodiments 138 to 146, characterized by a weight gain of about 19 % after undergoing a dynamic vapor sorption cycle from about 0 % relative humidity (RH) to about 90 % RH at 25 °C.
- [0645] **Embodiment 148.** The mesylate salt Form I, according to any one of embodiments 138 to 146, characterized by a dynamic vapor sorption profile substantially as shown in FIG. 32.
- [0646] **Embodiment 149.** The mesylate salt Form I, according to any one of embodiments 138 to 148, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 33.
- 10 [0647] **Embodiment 150.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is mesylate salt Form II.
- [0648] **Embodiment 151.** The mesylate salt Form II of embodiment 150, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.7, 15.4, 17.2, 17.6, and 19.1 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 15 [0649] **Embodiment 152.** The mesylate salt Form II of embodiment 151, characterized by an XRPD pattern further comprising one or more peaks selected from 10.0, 10.3, 11.4, 15.0, and 16.5 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0650] **Embodiment 153.** The mesylate salt Form II of embodiment 151 or 153, characterized by an XRPD pattern further comprising one or more peaks selected from 12.1, 20.2, 22.7, 23.2, and 24.1 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 20 [0651] **Embodiment 154.** The mesylate salt Form II of embodiment 150, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 34.
- [0652] **Embodiment 155.** The mesylate salt Form II, according to any one of embodiments 150 to 154, characterized by a differential scanning calorimetry thermogram (DSC) comprising an endotherm peak at about 190 °C.
- 25 [0653] **Embodiment 156.** The mesylate salt Form II, according to any one of embodiments 150 to 154, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 35.

- [0654] **Embodiment 157.** The mesylate salt Form II, according to any one of embodiments 150 to 156, characterized by about 0.2% weight loss upon heating from about 140 °C to about 205 °C, as measured by thermal gravimetric analysis (TGA).
- 5 [0655] **Embodiment 158.** The mesylate salt Form II, according to any one of embodiments 150 to 156, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 35.
- [0656] **Embodiment 159.** The mesylate salt Form II, according to any one of embodiments 150 to 158, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 36.
- 10 [0657] **Embodiment 160.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is mesylate salt Form III.
- [0658] **Embodiment 161.** The mesylate salt Form III of embodiment 160, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 6.5, 8.9, 17.5, 18.5, and 19.6 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 15 [0659] **Embodiment 162.** The mesylate salt Form III of embodiment 161, characterized by an XRPD pattern further comprising one or more peaks selected from 6.1, 13.1, 13.8, 14.4, and 19.4 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0660] **Embodiment 163.** The mesylate salt Form III embodiment 161 or 162, characterized by an XRPD pattern further comprising one or more peaks selected from 13.3, 18.0, 21.4, 23.6, and 26.0 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 20 [0661] **Embodiment 164.** The mesylate salt Form III of embodiment 160, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 37.
- [0662] **Embodiment 165.** The mesylate salt Form III, according to any one of embodiments 160 to 164, characterized by a differential scanning calorimetry thermogram (DSC) comprising a melting peak at about 173 °C.
- 25 [0663] **Embodiment 166.** The mesylate salt Form III, according to any one of embodiments 160 to 164, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 38.

- [0664] **Embodiment 167.** The mesylate salt Form III, according to any one of embodiments 160 to 166, characterized by 1.5% weight loss upon heating from about 80 °C to about 180 °C as measured by thermal gravimetric analysis (TGA).
- 5 [0665] **Embodiment 168.** The mesylate salt Form III, according to any one of embodiments 160 to 166, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 38.
- [0666] **Embodiment 169.** The mesylate salt Form III, according to any one of embodiments 160 to 168, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 39.
- 10 [0667] **Embodiment 170.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is maleate salt Form I.
- [0668] **Embodiment 171.** The maleate salt Form I of embodiment 170, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 8.7, 14.8, 15.5, 18.7, and 20.0 degrees 2θ (± 0.2 degrees 2θ).
- 15 [0669] **Embodiment 172.** The maleate salt Form I of embodiment 171, characterized by an XRPD pattern further comprising one or more peaks selected from 7.9, 8.3, 9.9, 12.7, and 18.1 degrees 2θ (± 0.2 degrees 2θ).
- [0670] **Embodiment 173.** The maleate salt Form I of embodiment 171 or 172, characterized by an XRPD pattern further comprising one or more peaks selected from 16.7, 20 21.2, 22.5, 23.3, and 24.0 degrees 2θ (± 0.2 degrees 2θ).
- [0671] **Embodiment 174.** The maleate salt Form I of embodiment 170, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 40.
- [0672] **Embodiment 175.** The maleate salt Form I, according to any one of embodiments 170 to 174, characterized by a differential scanning calorimetry thermogram (DSC) 25 comprising an endotherm peak at about 195 °C.
- [0673] **Embodiment 176.** The maleate salt Form I, according to any one of embodiments 170 to 174, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 41.

- [0674] **Embodiment 177.** The maleate salt Form I, according to any one of embodiments 170 to 176, characterized by no weight loss prior to decomposition, as measured by thermal gravimetric analysis (TGA).
- 5 [0675] **Embodiment 178.** The maleate salt Form I, according to any one of embodiments 170 to 176, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 41.
- [0676] **Embodiment 179.** The maleate salt Form I, according to any one of embodiments 170 to 178, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 42.
- 10 [0677] **Embodiment 180.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is maleate salt Form II.
- [0678] **Embodiment 181.** The maleate salt Form II of embodiment 180, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 14.3, 15.3, 17.5, 18.3, and 18.8 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 15 [0679] **Embodiment 182.** The maleate salt Form II of embodiment 181, characterized by an XRPD pattern further comprising one or more peaks selected from 9.6, 11.9, 13.3, 14.2, and 19.3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0680] **Embodiment 183.** The maleate salt Form II of embodiment 181 or 182, characterized by an XRPD pattern further comprising one or more peaks selected from 17.9, 20 21.4, 24.0, 24.3, and 24.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0681] **Embodiment 184.** The maleate salt Form II of embodiment 180, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 43.
- [0682] **Embodiment 185.** The maleate salt Form II, according to any one of embodiments 180 to 184, characterized by a differential scanning calorimetry thermogram (DSC) 25 comprising a first endotherm peak at about 142 °C, and/or a second endotherm peak at about 162 °C.
- [0683] **Embodiment 186.** The maleate salt Form II, according to any one of embodiments 180 to 184, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 44.

- [0684] **Embodiment 187.** The maleate salt Form II, according to any one of embodiments 180 to 186, characterized by about 1.6% weight loss upon heating from about 80 °C to about 144 °C, as measured by thermal gravimetric analysis (TGA).
- [0685] **Embodiment 188.** The maleate salt Form II, according to any one of embodiments 180 to 186, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 44.
- [0686] **Embodiment 189.** The maleate salt Form II, according to any one of embodiments 180 to 188, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 45.
- 10 [0687] **Embodiment 190.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is citrate salt Form I.
- [0688] **Embodiment 191.** The citrate salt Form I of embodiment 190, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.4, 15.8, 17.9, 19.2, and 19.5 degrees 2θ (± 0.2 degrees 2θ).
- 15 [0689] **Embodiment 192.** The citrate salt Form I of embodiment 191, characterized by an XRPD pattern further comprising one or more peaks selected from 10.6, 13.1, 15.1, 17.0, and 18.9 degrees 2θ (± 0.2 degrees 2θ).
- [0690] **Embodiment 193.** The citrate salt Form I of embodiment 191 or 192, characterized by an XRPD pattern further comprising one or more peaks selected from 20.3, 20 22.6, 23.1, 24.5, and 27.1 degrees 2θ (± 0.2 degrees 2θ).
- [0691] **Embodiment 194.** The citrate salt Form I of embodiment 190, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 46.
- [0692] **Embodiment 195.** The citrate salt Form I, according to any one of embodiments 190 to 194, characterized by a differential scanning calorimetry thermogram (DSC) 25 comprising an endothermic peak at about 167 °C.
- [0693] **Embodiment 196.** The citrate salt Form I, according to any one of embodiments 190 to 194, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 47.

- [0694] **Embodiment 197.** The citrate salt Form I, according to any one of embodiments 190 to 196, characterized by about 22% weight loss upon heating from about 95 °C to about 225 °C, as measured by thermal gravimetric analysis (TGA).
- [0695] **Embodiment 198.** The citrate salt Form I, according to any one of embodiments 190 to 196, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 47.
- [0696] **Embodiment 199.** The citrate salt Form I, according to any one of embodiments 190 to 198, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 48.
- 10 [0697] **Embodiment 200.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is fumarate salt Form I.
- [0698] **Embodiment 201.** The fumarate salt Form I of embodiment 200, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 4.2, 8.5, 10.6, 18.8, and 19.8 degrees 2θ (± 0.2 degrees 2θ).
- 15 [0699] **Embodiment 202.** The fumarate salt Form I of embodiment 201, characterized by an XRPD pattern further comprising one or more peaks selected from 12.7, 13.0, 15.9, 17.0, and 19.4 degrees 2θ (± 0.2 degrees 2θ).
- [0700] **Embodiment 203.** The fumarate salt Form I of embodiment 201 or 202, characterized by an XRPD pattern further comprising one or more peaks selected from 21.1, 21.4, 23.4, 25.7, and 27.4 degrees 2θ (± 0.2 degrees 2θ).
- 20 [0701] **Embodiment 204.** The fumarate salt Form I of embodiment 200, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 49.
- [0702] **Embodiment 205.** The fumarate salt Form I, according to any one of embodiments 200 to 205, characterized by a differential scanning calorimetry thermogram (DSC) comprising a melting peak at about 198 °C.
- 25 [0703] **Embodiment 206.** The fumarate salt Form I, according to any one of embodiments 200 to 205, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 50.

- [0704] **Embodiment 207.** The fumarate salt Form I, according to any one of embodiments 200 to 206, characterized by about 9.7% weight loss upon heating from about 100 °C to about 210 °C, as measured by thermal gravimetric analysis (TGA).
- 5 [0705] **Embodiment 208.** The fumarate salt Form I, according to any one of embodiments 200 to 206, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 50.
- [0706] **Embodiment 209.** The fumarate salt Form I, according to any one of embodiments 200 to 208, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 51.
- 10 [0707] **Embodiment 210.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is fumarate salt Form II.
- [0708] **Embodiment 211.** The fumarate salt Form II of embodiment 210, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 4.2, 8.5, 10.5, 12.9, and 18.9 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 15 [0709] **Embodiment 212.** The fumarate salt Form II of embodiment 211, characterized by an XRPD pattern further comprising one or more peaks selected from 15.1, 16.0, 18.6, 19.6, and 20.0 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0710] **Embodiment 213.** The fumarate salt Form II of any one of embodiments 211 or 212, characterized by an XRPD pattern further comprising one or more peaks selected from 20 21.7, 23.3, 23.6, 26.1, and 27.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0711] **Embodiment 214.** The fumarate salt Form II of embodiment 210, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 52.
- [0712] **Embodiment 215.** The fumarate salt Form II, according to any one of embodiments 210 to 214, characterized by a differential scanning calorimetry thermogram 25 (DSC) comprising a first endotherm peak at about 144 °C, and/or a second endotherm peak at about 200 °C.
- [0713] **Embodiment 216.** The fumarate salt Form II, according to any one of embodiments 210 to 214, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 53.

- [0714] **Embodiment 217.** The fumarate salt Form II, according to any one of embodiments 210 to 216, characterized by about 2.1% weight loss upon heating from about 90 °C to about 175 °C, as measured by thermal gravimetric analysis (TGA).
- 5 [0715] **Embodiment 218.** The fumarate salt Form II, according to any one of embodiments 210 to 216, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 53.
- [0716] **Embodiment 219.** The fumarate salt Form II, according to any one of embodiments 210 to 218, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 54.
- 10 [0717] **Embodiment 220.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is fumarate salt Form III.
- [0718] **Embodiment 221.** The fumarate salt Form III of embodiment 220, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 5.7, 7.9, 8.9, 10.6, and 17.4 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 15 [0719] **Embodiment 222.** The fumarate salt Form III of embodiment 221, characterized by XRPD pattern further comprising one or more peaks selected from 10.3, 11.5, 16.1, 17.9, and 18.5 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0720] **Embodiment 223.** The fumarate salt Form III of embodiment 221 or 222, characterized by an XRPD pattern further comprising one or more peaks selected from 2.1, 20 21.9, 24.0, 25.1, and 26.9 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0721] **Embodiment 224.** The fumarate salt Form III of embodiment 221, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 55.
- [0722] **Embodiment 225.** The fumarate salt Form III, according to any one of embodiments 220 to 224, characterized by a differential scanning calorimetry thermogram 25 (DSC) comprising a first endotherm peak at about 113 °C, and/or a second endotherm peak at about 164 °C.
- [0723] **Embodiment 226.** The fumarate salt Form III, according to any one of embodiments 220 to 224, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 56.

- [0724] **Embodiment 227.** The fumarate salt Form III, according to any one of embodiments 220 to 226, characterized by about 3.1% weight loss upon heating from about 85 °C to about 135 °C, as measured by thermal gravimetric analysis (TGA).
- 5 [0725] **Embodiment 228.** The fumarate salt Form III, according to any one of embodiments 220 to 226, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 56.
- [0726] **Embodiment 229.** The fumarate salt Form III, according to any one of embodiments 220 to 228, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 57.
- 10 [0727] **Embodiment 230.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is malate salt Form I.
- [0728] **Embodiment 231.** The malate salt Form I of embodiment 230, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 10.9, 12.4, 13.9, 18.7, and 19.4 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 15 [0729] **Embodiment 232.** The malate salt Form I of embodiment 231, characterized by an XRPD pattern further comprising one or more peaks selected from 4.1, 8.3, 10.5, 16.0, and 16.3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0730] **Embodiment 233.** The malate salt Form I of embodiment 231 or 232, characterized by an XRPD pattern further comprising one or more peaks selected from 20.0, 20.5, 21.1, 22.9, and 24.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 20 [0731] **Embodiment 234.** The malate salt Form I of embodiment 230, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 58.
- [0732] **Embodiment 235.** The malate salt Form I, according to any one of embodiments 230 to 234, characterized by a differential scanning calorimetry thermogram (DSC)
- 25 comprising a first endothermic peak at about 157 °C and/or a second endothermic peak at about 206 °C.
- [0733] **Embodiment 236.** The malate salt Form I, according to any one of embodiments 230 to 234, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 59.

- [0734] **Embodiment 237.** The malate salt Form I, according to any one of embodiments 230 to 236, characterized by about 1.9% weight loss upon heating from about 75 °C to about 157 °C, as measured by thermal gravimetric analysis (TGA).
- [0735] **Embodiment 238.** The malate salt Form I, according to any one of embodiments 230 to 236, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 59.
- [0736] **Embodiment 239.** The malate salt Form I, according to any one of embodiments 230 to 238, characterized by a polarized light microscope (PLM) profile substantially as shown in FIG. 60.
- 10 [0737] **Embodiment 240.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is malate salt Form II.
- [0738] **Embodiment 241.** The malate salt Form II of embodiment 240, characterized by an XRPD pattern further comprising one or more peaks selected from 8.2, 12.4, 14.7, 15.8, and 18.3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 15 [0739] **Embodiment 242.** The malate salt Form II of embodiment 241, characterized by an XRPD pattern further comprising one or more peaks selected from 4.0, 15.6, 16.4, 17.0, and 19.1 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0740] **Embodiment 243.** The malate salt Form II of embodiment 241 or 242, characterized by an XRPD pattern further comprising one or more peaks selected from 21.5, 22.2, 22.9, 24.4, and 26.2 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 20 [0741] **Embodiment 244.** The malate salt Form II of embodiment 240, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 61.
- [0742] **Embodiment 245.** The malate salt Form II, according to any one of embodiments 240 to 244, characterized by a differential scanning calorimetry thermogram (DSC) comprising an endotherm peak at about 180 °C.
- 25 [0743] **Embodiment 246.** The malate salt Form II, according to any one of embodiments 240 to 244, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 62.

- [0744] **Embodiment 247.** The malate salt Form II, according to any one of embodiments 240 to 246, characterized by about 0.5% weight loss upon heating from about 95 °C to about 170 °C, as measured by thermal gravimetric analysis (TGA).
- 5 [0745] **Embodiment 248.** The malate salt Form II, according to any one of embodiments 240 to 246, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 62.
- [0746] **Embodiment 249.** The malate salt Form II, according to any one of embodiments 240 to 248, characterized by a weight gain of about 1.8 % after undergoing a dynamic vapor sorption cycle from about 0 % relative humidity (RH) to about 90 % RH at 25 °C.
- 10 [0747] **Embodiment 250.** The malate salt Form II, according to any one of embodiments 240 to 248, characterized by a dynamic vapor sorption profile substantially as shown in FIG. 63.
- [0748] **Embodiment 251.** The malate salt Form II, according to any one of embodiments 240 to 250, characterized by a polarized light microscope (PLM) profile substantially as
15 shown in FIG. 64.
- [0749] **Embodiment 252.** The crystalline form of embodiment 1 or embodiment 2, wherein the crystalline form is succinate salt Form I.
- [0750] **Embodiment 253.** The succinate salt Form I of embodiment 252, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 10.5, 12.8, 15.0, 18.8, and
20 19.9 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0751] **Embodiment 254.** The succinate salt Form I of embodiment 253, characterized by an XRPD pattern further comprising one or more peaks selected from 8.5, 10.7, 12.1, 15.9, and 18.5 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0752] **Embodiment 255.** The succinate salt Form I of embodiment 253 or 254,
25 characterized by an XRPD pattern further comprising one or more peaks selected from 21.4, 22.8, 23.3, 24.5, and 24.8 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0753] **Embodiment 256.** The succinate salt Form I of embodiment 252, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 65.

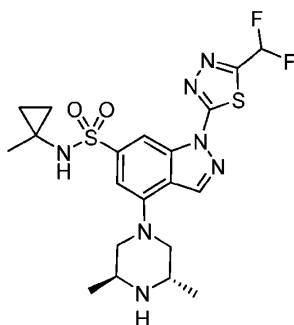
- [0754] **Embodiment 257.** The succinate salt Form I, according to any one of
embodiments **252** to **256**, characterized by a differential scanning calorimetry thermogram
(DSC) comprising an endotherm peak at about 136 °C.
- 5 [0755] **Embodiment 258.** The succinate salt Form I, according to any one of
embodiments **252** to **256**, characterized by a differential scanning calorimetry thermogram
(DSC) substantially in accordance with **FIG. 66**.
- [0756] **Embodiment 259.** The succinate salt Form I, according to any one of
embodiments **252** to **258**, characterized by about 2.8% weight loss upon heating from about
85 °C to about 145 °C, as measured by thermal gravimetric analysis (TGA).
- 10 [0757] **Embodiment 260.** The succinate salt Form I, according to any one of
embodiments **252** to **258**, characterized by a thermal gravimetric analysis (TGA) thermogram
substantially in accordance with **FIG. 66**.
- [0758] **Embodiment 261.** The succinate salt Form I, according to any one of
embodiments **252** to **260**, characterized by a polarized light microscope (PLM) profile
15 substantially as shown in **FIG. 67**.
- [0759] **Embodiment 262.** A pharmaceutical composition comprising a crystalline form
according to any one of embodiments **1** to **261** and at least one pharmaceutically acceptable
excipient.
- [0760] **Embodiment 263.** A solid state form of Compound **1** or a salt thereof, wherein the
20 solid state form comprises at least 50 wt.% of a particular crystalline form, wherein the
particular crystalline form is according to any one of embodiments **1** to **261**.
- [0761] **Embodiment 264.** The solid state form of embodiment **263**, wherein the solid state
form comprises at least 60 wt.% of the particular crystalline form.
- [0762] **Embodiment 265.** The solid state form of embodiment **263**, wherein the solid state
25 form comprises at least 70 wt.% of the particular crystalline form.
- [0763] **Embodiment 266.** The solid state form of embodiment **263**, wherein the solid state
form comprises at least 80 wt.% of the particular crystalline form.
- [0764] **Embodiment 267.** The solid state form of embodiment **263**, wherein the solid state
form comprises at least 90 wt.% of the particular crystalline form.

- [0765] **Embodiment 268.** The solid state form of embodiment 263, wherein the solid state form comprises at least 95 wt.% of the particular crystalline form.
- [0766] **Embodiment 269.** The solid state form of embodiment 263, wherein the solid state form comprises at least 99 wt.% of the particular crystalline form.
- 5 [0767] **Embodiment 270.** The solid state form of any one of embodiments 263 to 269, wherein the particular crystalline form is HCl salt Form I.
- [0768] **Embodiment 271.** The solid state form of any one of embodiments 263 to 269, wherein the particular crystalline form is free base Form I.
- [0769] **Embodiment 272.** A pharmaceutical composition comprising a solid state form of
10 any one of embodiments 263 to 269, and at least one pharmaceutically acceptable excipient.
- [0770] **Embodiment 273.** A pharmaceutical composition prepared by combining a solid state form of any one of embodiments 263 to 269 with at least one pharmaceutically acceptable excipient.
- [0771] **Embodiment 274.** A method of inhibiting PARG *in vivo* in a patient, the method
15 comprising administering to the patient an effective amount of a crystalline form of any one of embodiments 1 to 261.
- [0772] **Embodiment 275.** A method of inhibiting cell proliferation, *in vitro* or *in vivo*, the method comprising contacting a cell with an effective amount of a crystalline form of any one of embodiments 1 to 261.
- 20 [0773] **Embodiment 276.** A method of treating a cancer resistant to one or more platins or one or more PARP inhibitors in a patient in need thereof, the method comprising administering to the patient an effective amount of a crystalline form of any one of embodiments 1 to 261.
- [0774] **Embodiment 277.** A method of treating and/or preventing a homologous
25 recombinant deficient (HRD) cancer in a patient comprising administering to the patient a therapeutically effective amount of a crystalline form of any one of embodiments 1 to 261.
- [0775] **Embodiment 278.** The method of embodiment 277, wherein the patient is in recognized need of such treatment.

- [0776] **Embodiment 279.** A method of treating and/or preventing a cancer in a patient, where the cancer is characterized by a reduction or absence of BRCA1 and/or BRCA2 gene expression, the absence or mutation of BRCA1 and/or BRCA2 genes, or reduced function of BRCA1 and/or BRCA2 proteins, comprising administering to the patient a therapeutically effective amount of a crystalline form of any one of embodiments **1** to **261**.
- [0777] **Embodiment 280.** The method of embodiment **279**, wherein the patient is in recognized need of such treatment.
- [0778] **Embodiment 281.** The crystalline form as defined in any one of embodiments **1** to **261** for use in the treatment of cancer.
- [0779] **Embodiment 282.** Use of a crystalline form as defined in any one of embodiments **1** to **261** in the manufacture of a medicament for treating cancer.
- [0780] **Embodiment 283.** The method of any one of embodiments **276** to **280** or the use of embodiments **281** or **282**, wherein the cancer is breast cancer, ovarian cancer, gastric cancer, lung cancer, cervical cancer, pancreatic cancer, prostate cancer, colorectal cancer, or endometrial cancer.

H. Additional Non-limiting Exemplary Embodiments

- [0781] **Embodiment A1.** A crystalline form of Compound **1**



(Compound **1**), or a salt thereof.

- [0782] **Embodiment A2.** The crystalline form of Embodiment **A1**, wherein the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.
- [0783] **Embodiment A3.** The crystalline form of Embodiment **A1** or Embodiment **A2**, wherein the crystalline form is HCl salt Form I.

[0784] **Embodiment A4.** The crystalline form of Embodiment A3, wherein the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.

5 [0785] **Embodiment A5.** The HCl salt Form I, according to Embodiment A3 or A4, characterized by an X-ray powder diffraction (XRPD) pattern comprising three or more peaks selected from 11.7, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9 degrees 2θ (± 0.2 degrees 2θ).

[0786] **Embodiment A6.** The HCl salt Form I, according to Embodiment A3 or A4, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 11.7, 13.2, 13.7, 17.7, and 18.4 degrees 2θ (± 0.2 degrees 2θ).

10 [0787] **Embodiment A7.** The HCl salt Form I, according to Embodiment A3 or A4, characterized by an XRPD pattern comprising peaks at 7.0, 11.7, 12.2, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0 degrees 2θ (± 0.2 degrees 2θ).

[0788] **Embodiment A8.** The HCl salt Form I, according to any one of Embodiments A3 to A7, wherein the XRPD pattern further comprises one or more peaks selected from 20.6, 20.9, 21.2, 21.7, 22.3, 24.3, 25.0, 25.3, 25.6, 26.1, 28.9 degrees 2θ (± 0.2 degrees 2θ).

[0789] **Embodiment A9.** The HCl salt Form I, according to Embodiment A3 or A4, characterized by an X-ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative intensity of at least 15% as listed in Table 3.

20 [0790] **Embodiment A10.** The HCl salt Form I according to Embodiment A3 or A4, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 13.2, 13.7, 17.7, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).

[0791] **Embodiment A11.** The HCl salt Form I according to Embodiment A10, wherein the XRPD pattern further comprises peaks at 20.6, 20.9, 21.2, 24.3, and 25.3 degrees 2θ (± 0.2 degrees 2θ).

25 [0792] **Embodiment A12.** The HCl salt Form I, according to Embodiment A3 or A4, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 7.

[0793] **Embodiment A13.** The HCl salt Form I, according to any one of Embodiments A3 to A12, characterized by a differential scanning calorimetry thermogram (DSC) comprising
30 no endotherm prior to decomposition.

- [0794] **Embodiment A14.** The HCl salt Form I, according to any one of Embodiments A3 to A12, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 8.
- 5 [0795] **Embodiment A15.** The HCl salt Form I, according to any one of Embodiments A3 to A14, characterized by no weight loss upon heating prior to decomposition, as measured by thermal gravimetric analysis (TGA).
- [0796] **Embodiment A16.** The HCl salt Form I, according to any one of Embodiments A3 to A14, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 8.
- 10 [0797] **Embodiment A17.** The HCl salt Form I, according to any one of Embodiments A3 to A16, characterized by a weight gain of about 0.5 % after undergoing a dynamic vapor sorption cycle from about 0% relative humidity (RH) to about 90% RH at 25 °C.
- [0798] **Embodiment A18.** The HCl salt Form I, according to any one of Embodiments A3 to A16, characterized by a dynamic vapor sorption profile substantially as shown in FIG. 9.
- 15 [0799] **Embodiment A19.** The crystalline form of Embodiment A1 or A2, wherein the crystalline form is free base Form I.
- [0800] **Embodiment A20.** The crystalline form of Embodiment A19, wherein the crystalline form is a single crystalline form substantially free of other crystalline or amorphous forms.
- 20 [0801] **Embodiment A21.** The free base Form I of Embodiments A19 or A20, characterized by an X-ray powder diffraction (XRPD) pattern comprising three or more peaks selected from 9.0, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0802] **Embodiment A22.** The free base Form I of Embodiment A19 or A20,
- 25 characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.0, 18.3, and 19.4 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- [0803] **Embodiment A23.** The free base Form I of Embodiment A19 or A20, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 11.5, 12.7, 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).

- [0804] **Embodiment A24.** The free base Form I of any one of Embodiments A21 to A23, wherein the XRPD pattern further comprises one or more peaks selected from 22.5, 23.2, 23.5, 24.6, and 27.8 degrees 2θ (± 0.2 degrees 2θ).
- 5 [0805] **Embodiment A25.** The free base Form I according to Embodiment A19 or A20, characterized by an X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.3, 23.5, and 24.6 degrees 2θ (± 0.2 degrees 2θ).
- [0806] **Embodiment A26.** The free base Form I according to c Embodiment A25, wherein the XRPD pattern further comprises peaks at 12.7, 13.7, 18.0, 18.9, and 19.4 degrees 2θ (± 0.2 degrees 2θ).
- 10 [0807] **Embodiment A27.** The free base Form I of Embodiment A19 or A20, characterized by an X-ray powder diffraction (XRPD) pattern substantially in accordance with FIG. 1.
- [0808] **Embodiment A28.** The free base Form I, according to any one of Embodiments A19 to A27, characterized by a differential scanning calorimetry thermogram (DSC)
- 15 comprising an endothermic peak at about 200 °C.
- [0809] **Embodiment A29.** The free base Form I, according to any one of Embodiments A19 to A27, characterized by a differential scanning calorimetry thermogram (DSC) substantially in accordance with FIG. 2.
- [0810] **Embodiment A30.** The free base Form I, according to any one of Embodiments
- 20 A19 to A29, characterized by no weight loss upon heating prior to decomposition, as measured by thermal gravimetric analysis (TGA).
- [0811] **Embodiment A31.** The free base Form I, according to any one of Embodiments A19 to A29, characterized by a thermal gravimetric analysis (TGA) thermogram substantially in accordance with FIG. 2.
- 25 [0812] **Embodiment A32.** A pharmaceutical composition comprising a crystalline form according to any one of Embodiments A1 to A31 and at least one pharmaceutically acceptable excipient.
- [0813] **Embodiment A33.** A pharmaceutical composition prepared by combining a crystalline form according to any one of Embodiments A1 to A31 and at least one
- 30 pharmaceutically acceptable excipient.

- [0814] **Embodiment A34.** A solid state form of Compound 1 or a salt thereof, wherein the solid state form comprises at least 50 wt.% of a particular crystalline form, at least 60 wt.% of a particular crystalline form, at least 70 wt.% of a particular crystalline form, at least 80 wt.% of a particular crystalline form, at least 90 wt.% of a particular crystalline form, at least 95 wt.% of a particular crystalline form, or at least 99 wt.% of a particular crystalline form, wherein
- the particular crystalline form is according to any one of Embodiments **A1** to **A31**.
- [0815] **Embodiment A35.** The solid state form of Embodiment **A34**, wherein the particular crystalline form is HCl salt Form I.
- 10 [0816] **Embodiment A36.** The solid state form of Embodiment **A34**, wherein the particular crystalline form is free base Form I.
- [0817] **Embodiment A37.** A pharmaceutical composition comprising a solid state form of any one of Embodiments **A34** to **A36**, and at least one pharmaceutically acceptable excipient.
- [0818] **Embodiment A38.** A pharmaceutical composition prepared by combining a solid
- 15 state form of any one of Embodiments **A34** to **A36** with at least one pharmaceutically acceptable excipient.
- [0819] **Embodiment A39.** A method of inhibiting PARG *in vivo* in a patient, the method comprising administering to the patient an effective amount of a crystalline form of any one of Embodiments **A1** to **A31**, or a pharmaceutical composition of any one of Embodiments
- 20 **A32**, **A33**, **A37** and **A38**.
- [0820] **Embodiment A40.** A method of inhibiting cell proliferation, *in vitro* or *in vivo*, the method comprising contacting a cell with an effective amount of a crystalline form of any one of Embodiments **A1** to **A31**, or a pharmaceutical composition of any one of Embodiments **A32**, **A33**, **A37** and **A38**.
- 25 [0821] **Embodiment A41.** A method of treating a cancer resistant to one or more platins or one or more PARP inhibitors in a patient in need thereof, the method comprising administering to the patient a therapeutically effective amount of a crystalline form of any one of Embodiments **A1** to **A31**, or a pharmaceutical composition of any one of Embodiments **A32**, **A33**, **A37** and **A38**.

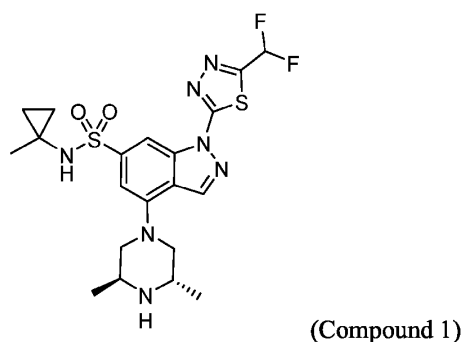
- [0822] **Embodiment A42.** A method of treating a cancer in a patient in need thereof, the method comprising administering to the patient a therapeutically effective amount of a crystalline form of any one of Embodiments A1 to A31, or a pharmaceutical composition of any one of Embodiments A32, A33, A37 and A38.
- 5 [0823] **Embodiment A43.** A method of treating and/or preventing a homologous recombinant deficient (HRD) cancer in a patient in need thereof, the method comprising administering to the patient a therapeutically effective amount of a crystalline form of any one of Embodiments A1 to A31, or a pharmaceutical composition of any one of Embodiments A32, A33, A37 and A38.
- 10 [0824] **Embodiment A44.** A method of treating and/or preventing a cancer in a patient in need thereof, where the cancer is characterized by a reduction or absence of BRCA1 and/or BRCA2 gene expression, the absence or mutation of BRCA1 and/or BRCA2 genes, or reduced function of BRCA1 and/or BRCA2 proteins, the method comprising administering to the patient a therapeutically effective amount of a crystalline form of any one of
- 15 Embodiments A1 to A31, or a pharmaceutical composition of any one of Embodiments A32, A33, A37 and A38.
- [0825] **Embodiment A45.** The crystalline form as defined in any one of Embodiments A1 to A31, or a pharmaceutical composition of any one of Embodiments A32, A33, A37 and A38, for use in the treatment of cancer.
- 20 [0826] **Embodiment A46.** Use of a crystalline form as defined in any one of Embodiments A1 to A31, or a pharmaceutical composition of any one of Embodiments A32, A33, A37 and A38, in the manufacture of a medicament for the treatment of cancer.
- [0827] **Embodiment A47.** The method of any one of Embodiments A41 to A44, or the use of Embodiment A45 or A46, wherein the cancer is breast cancer, ovarian cancer, gastric
- 25 cancer, lung cancer, cervical cancer, pancreatic cancer, prostate cancer, colorectal cancer, head and neck cancer, or endometrial cancer.

IV. Examples

- [0828] The following examples are put forth so as to provide those of ordinary skill in the art with a complete disclosure and description of how to make and use the present invention,
- 30 and are not intended to limit the scope of what the inventors regard as their invention, nor are they intended to represent that the experiments below were performed or that they are all of

the experiments that may be performed. It is to be understood that exemplary descriptions written in the present tense were not necessarily performed, but rather that the descriptions can be performed to generate data and the like of a nature described therein. Efforts have been made to ensure accuracy with respect to numbers used (e.g., amounts, temperature, etc.),
 5 but some experimental errors and deviations should be accounted for.

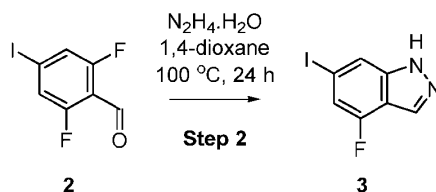
Example 1: Preparation of Compound 1



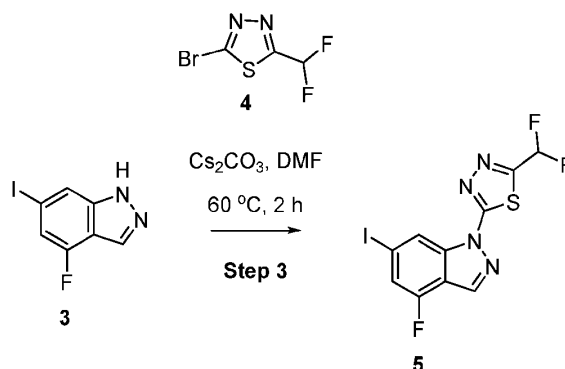
Step 1: Preparation of 2,6-difluoro-4-iodobenzaldehyde (Int 2)



- 10 **[0829]** To a stirred solution of 1,3-difluoro-5-iodobenzene (**Int 1**) (50 g, 208.3mmol, Oakwood Chemical, CAS 2265-91-0, catalogue #024566) in THF (500 mL) was added LDA (80 mL, 625.0 mmol) and DMF (48.3 mL, 625mmol) at -78°C and stirred at -78 °C for 2 h. After complete consumption of starting material, the reaction mixture was diluted with water (500 mL) and extracted with EtOAc (2 x 300 mL), the combined organic phases were washed
 15 with brine solution (200 mL), dried over anhydrous sodium sulphate, filtered and the filtrate was concentrated under reduced pressure to get crude product as an oil. The crude material was purified by column chromatography using silica gel (100-200) and eluted with 20% EtOAc/Hexane as a gradient. The product was eluted with a gradient of 30% EtOAc/Hexane. The purified fractions were concentrated under reduced pressure to afford 2,6-difluoro-4-
 20 iodobenzaldehyde (**Int 2**) (23 g) as a solid. ¹H NMR (500MHz, CHLOROFORM-d) δ: 10.29 (s, 1H), 7.37-7.46 (m, 2H).

Step 2: Preparation of 4-fluoro-6-iodo-1H-indazole (Int 3)

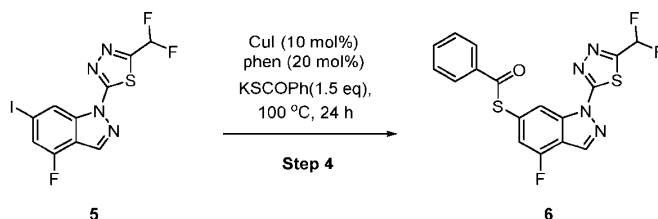
- [0830]** To a stirred solution of 2,6-difluoro-4-iodobenzaldehyde (**Int 2**) (5 g, 18.6 mmol) in 1,4 dioxane (110 mL) was added hydrazine hydrate (18.6 mL, 373.1 mmol) at RT and the resulting mixture was stirred at 100°C for 24 h. The reaction mixture was concentrated under reduced pressure and ice cold water (100 mL) was added. The mixture was stirred for 30 min during which time solid was precipitated out. The mixture was filtered. The solid was washed with water (100 mL), *n*-pentane (50 mL), and dried under vacuum to afford product 4-fluoro-6-iodo-1H-indazole (**Int 3**) (2.3 g) as a solid. MS ESI calculated for $\text{C}_7\text{H}_4\text{FIN}_2$ $[\text{M}+\text{H}]^+$ 262.94., found 262.99. ^1H NMR (CDCl_3 , 400 MHz): 10.12 (s, 1 H), 8.10 (s, 1H), 7.70 (s, 1H), 7.15 (dd, $J = 9$ Hz, 1H).

Step 3: Preparation of 2-(difluoromethyl)-5-(4-fluoro-6-iodo-1H-indazol-1-yl)-1,3,4-thiadiazole (Int 5)

- [0831]** To a stirred solution of 4-fluoro-6-iodo-1H-indazole (**Int 3**) (5 g, 19.0 mmol) in DMF (50 mL) was added cesium carbonate (18.6 g, 57.24 mmol) and 2-bromo-5-(difluoromethyl)-1,3,4-thiadiazole (**Int 4**) (3.8 g, 18.1 mmol, Enamine Stock Building Blocks, CAS 1340313-49-6, catalogue #EN300-108825). The resulting mixture was stirred at 60°C for 2 h. The progress of the reaction was monitored by TLC. The reaction mixture was quenched with ice cold water (50 mL) and stirred for 30 min during which time solid precipitated out. The mixture was filtered. The solid collected was washed with water (100

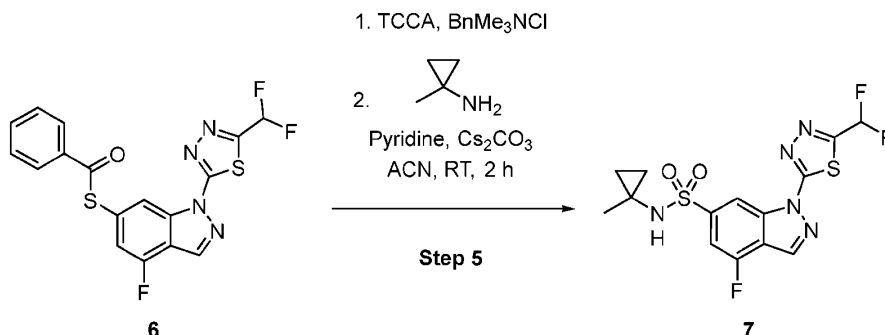
mL) followed by *n*-pentane (100 mL), and dried under vacuum to afford 2-(difluoromethyl)-5-(4-fluoro-6-iodo-1H-indazol-1-yl)-1,3,4-thiadiazole (**Int 5**) (4.2 g) as a solid. MS ESI calculated for $C_{10}H_4F_3IN_4S$ $[M+H]^+$ 396.92., found 396.91. 1H NMR ($CDCl_3$, 500 MHz): 8.87 (s, 1 H), 8.29 (s, 1H), 7.40 (dd, $J = 17$ Hz, 1H), 7.0 (t, $J = 53.5$ Hz, 1H).

5 Step 4: Preparation of S-(1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-fluoro-1H-indazol-6-yl) benzothioate (Int 6)



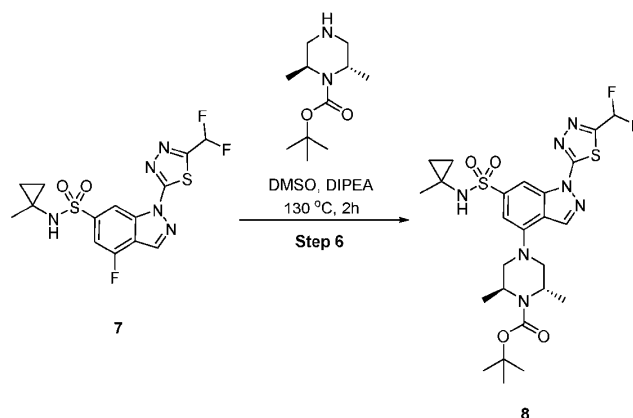
[0832] To a stirred solution of 2-(difluoromethyl)-5-(4-fluoro-6-iodo-1H-indazol-1-yl)-1,3,4-thiadiazole (**Int 5**) (100 mg, 0.25 mmol) in toluene (1 mL) degassed for 5 min, was added CuI (5mg, 0.025 mmol), 1,10-phenanthroline (phen) (11 mg, 0.05 mmol), and potassium thiobenzoate (67 mg, 0.378mmol) at RT. The resulting mixture was stirred at 100 °C for 16 h. The progress of the reaction was monitored by LCMS. The crude mixture was purified by column chromatography using silica gel (100-200) and eluted with 10% EtOAc/Hexane as a gradient. The purified fractions were collected and concentrated under reduced pressure to afford S-(1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-fluoro-1H-indazol-6-yl) benzothioate (**Int 6**) (55 mg) as a solid. MS ESI calculated for $C_{17}H_9F_3N_4OS_2$ $[M+H]^+$ 407.02., found 407.01. 1H NMR ($CDCl_3$, 400 MHz): 8.68 (s, 1 H), 8.39 (s, 1H), 8.03 (d, $J = 7.6$ Hz, 2H), 7.64 (t, $J = 7.2$ Hz, 1H), 7.53 (t, $J = 7.6$ Hz, 2H), 7.27 (s, 1H), 6.99 (t, $J = 53.2$ Hz, 1H).

Step 5: Preparation of 1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-fluoro-N-(1-methylcyclopropyl)-1H-indazole-6-sulfonamide (Int 7)



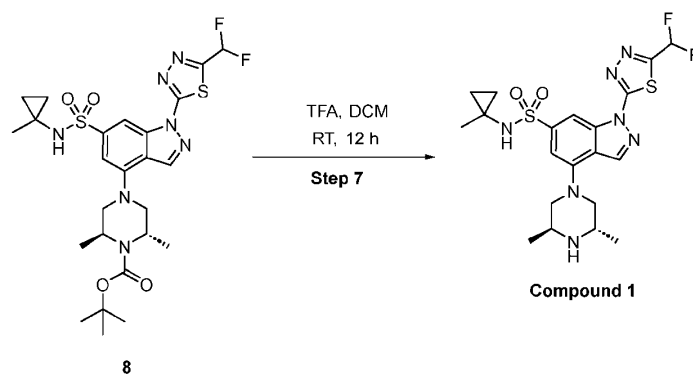
- [0833]** To a stirred solution of S-(1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-fluoro-1H-indazol-6-yl) benzothioate (**Int 6**) (500 mg, 1.23 mmol) in acetonitrile (10 mL) at 0 °C were added a solution of BnMe₃NCl (682 mg, 3.69 mmol), and TCCA (trichloroisocyanuric acid) (370 mg 1.59 mmol) in acetonitrile (40 mL). The reaction mixture was stirred for 20 min. Then a solution of 1-methyl cyclopropane-1-amine (1.71 g, 7.38 mmol, Combi-Blocks, CAS 22936-83-0, catalogue #QH-3639) in pyridine (2.5 mL) and cesium carbonate (198 mg, 0.61 mmol) were added to the reaction mixture at 0 °C and stirred at RT for 2 h. The progress of the reaction was monitored by LCMS. LCMS showed complete consumption of starting material (S-(1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-fluoro-1H-indazol-6-yl) benzothioate) (**Int 6**). The reaction mixture was diluted with water (50 mL) and extracted with EtOAc (2x30 mL). The combined organic layer was washed with brine solution (20 mL), dried over anhydrous sodium sulphate, filtered and the filtrate was concentrated under reduced pressure to obtain crude product. The crude product was purified by column chromatography using silica gel (100-200) and eluted with 5 to 50% EtOAc/hexane as a gradient. The product was eluted at 20% EtOAc/hexane. The purified fractions were collected and concentrated under reduced pressure to afford 1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-fluoro-N-(1-methylcyclopropyl)-1H-indazole-6-sulfonamide (**Int 7**) (90 mg) as a solid. MS ESI calculated for C₁₄H₉F₃N₆O₂S₂ [M+H]⁺ 404.04., found 404.18. ¹H NMR (CDCl₃, 400 MHz): 9.00 (s, 1 H), 8.80 (s, 1H), 8.54 (s, 1H), 7.63 (t, *J*= 48.8 Hz, 2H), 1.10 (s, 3 H), 0.65 (s, 2 H), 0.44 (s, 2H).

Step 6: Preparation of tert-butyl (2S,6S)-4-(1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-6-(N-(1-methylcyclopropyl)sulfamoyl)-1H-indazol-4-yl)-2,6-dimethylpiperazine-1-carboxylate (Int 8)



- 5 **[0834]** To a stirred solution of 1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-fluoro-N-(1-methylcyclopropyl)-1H-indazole-6-sulfonamide (**Int 7**) (80 mg, 0.19 mmol) in DMSO (dimethyl sulfoxide) (2 mL) were added *tert*-butyl (2S,6S)-2,6-dimethylpiperazine-1-carboxylate (85 mg, 0.39 mmol, BLD Pharmatech, CAS 574007-66-2, catalogue #BD233798) and DIPEA (N,N-diisopropyl ethylamine) (0.1 mL, 0.59 mmol) and reaction
- 10 mixture was stirred at 130 °C for 2 h. The reaction mixture was quenched with ice cold water (20 mL) and stirred for 30 min. The obtained solid was filtered, washed with water (10 mL), dried under vacuum and purified by column chromatography over silica gel (100-200) and eluted with 50% EtOAc/hexane as a gradient, purified fractions concentrated under reduced pressure to afford *tert*-butyl (2S,6S)-4-(1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-6-(N-(1-methylcyclopropyl) sulfamoyl)-1H-indazol-4-yl)-2,6-dimethylpiperazine-1-carboxylate (**Int**
- 15 **8**) (110 mg, yield: 92%) as a solid. MS ESI calculated for C₂₅H₃₃F₂N₇O₄S₂ [M+H]⁺ 598.20, found 598.26.

Step 7: Preparation of 1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-((3S,5S)-3,5-dimethylpiperazin-1-yl)-N-(1-methylcyclopropyl)-1H-indazole-6-sulfonamide (Compound 1)



- 5 **[0835]** To a stirred solution of *tert*-butyl (2*S*,6*S*)-4-(1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-6-(*N*-(1-methylcyclopropyl)sulfamoyl)-1*H*-indazol-4-yl)-2,6-dimethyl piperazine-1-carboxylate (**Int 8**) (100 mg, 0.16mmol) in DCM (3 mL) was added trifluoroacetic acid (0.07 mL, 0.98 mmol) at 0 °C and reaction mixture was stirred at RT for 12 h. The reaction mixture was concentrated under reduced pressure, purified by Prep HPLC purification (Prep
- 10 HPLC conditions: MOBILE PHASE – 10 mM ammonium bicarbonate in H₂O: MeCN COLUMN – Inertsil ODS (20X250) mm 5u Flow-18ml/min GRADIENT METHOD- 0/50,9.5/82,9.55/99,11.5/99,11.55/50,14.5/50, SOLUBILITY: CAN, Fraction Volume: 100 mL) to afford 1-(5-(difluoromethyl)-1,3,4-thiadiazol-2-yl)-4-((3*S*,5*S*)-3,5-dimethylpiperazin-1-yl)-*N*-(1-methylcyclopropyl)-1*H*-indazole-6-sulfonamide (**Compound 1**) (18 mg, yield:
- 15 21%) as a solid. MS ESI calculated for C₂₀H₂₅F₂N₇O₂S₂ [M+H]⁺ 498.15, found 498.34. ¹H NMR (DMSO-*d*₆, 400 MHz): δ (ppm) 8.75 (s, 1H), 8.40 (s, 1H), 8.31 (s, 1H), 7.59 (t, *J* = 52.8 Hz, 1H), 7.10 (d, *J* = 1.0 Hz, 1H), 3.37 (br dd, *J* = 11.5, 2.9 Hz, 2H), 3.22-3.30 (m, 2H), 3.08 (br dd, *J* = 11.7, 6.1 Hz, 2H), 1.17 (d, *J* = 6.4 Hz, 6H), 1.08 (s, 3H), 0.58-0.76 (m, 2H), 0.29-0.49 (m, 2H).

20 **Example 2: Free base crystalline Form I of Compound 1**

[0836] The solid prepared in Example 1 was characterized and assigned as the free base Form I (anhydrate) with 0.4% residual acetonitrile as detected by NMR (data not shown). A slurry study was performed using Compound 1 as prepared in Example 1. About 15-25 mg of Compound 1 was weighed into a sample vial and 0.2-0.5 mL of a solvent (water, EtOH,

isopropyl alcohol, MTBE, toluene, n-heptane, cyclohexane, EtOAc, MeCN, isopropyl acetate, acetone, MEK, or MeOH) was added to make a suspension, respectively. All suspensions were stirred at RT for 3 days and 50 °C for 1 day. Then, the solids were filtered and the filter cakes were analyzed by XRPD. The resulting solids were all Form I, and no new form was obtained.

[0837] As the starting material had about 0.4% ACN, some slurry samples were analyzed by NMR to check the residual solvent level. The samples that were slurried in (a) water at RT for 3 days, (b) MeOH at 50 °C for 1 day, or (c) EtOH at 50 °C for 1 day, had no residual solvents. The solids obtained by slurrying Compound 1 in EtOH at 50 °C for 1 day were characterized accordingly, and assigned as free base Form I (anhydrate) (no residual solvent).

[0838] XRPD diffractograms were collected with an X-ray diffractometer. The sample was prepared on a zero-background silicon wafer by gently pressing onto the flat surface. The parameters of XRPD diffraction are given in the table below.

Instrument	Bruker, D8 Advance
Radiation	Cu K α (λ = 1.5418 Å)
Detector	LynxEye XE-T
Scan angle	3-40° (2 θ)
Scan step	0.02° (2 θ)
Scan speed	0.1 s/step
Tube voltage/current	40 kV/40 mA
Divergence slit	0.6 mm
Rotation	30 rpm
Sample holder	Zero-background sample pan

A plot of the XRPD is shown in FIG. 1, and Table 1, below, summarizes selected peaks observed in the XRPD plot.

Table 1: Selected Peaks in the free base of Compound 1 (Form I)

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
6.6	3.1%	19.4	29.3%
9.0	39.5%	19.7	18.0%
11.2	4.5%	20.9	2.6%
11.5	12.5%	21.4	5.0%
12.2	6.8%	21.9	3.2%
12.7	19.9%	22.5	13.5%
13.7	18.5%	23.2	16.5%
16.4	36.8%	23.5	36.8%
16.6	3.0%	24.0	7.8%

17.2	11.5%	24.6	100.0%
18.0	35.6%	27.8	12.3%
18.3	86.2%	33.8	11.5%
18.9	18.9%	35.0	10.1%
19.2	3.3%		

[0839] DSC analysis was performed with a TA Instrument. About 1-3 mg of a sample was placed into an aluminum pan with pin-hole and heated with the parameters in the table below. The data were analyzed using TRIOS.

Instrument	TA, Discovery DSC 250
Sample pan	Aluminum, pin-holed
Temperature range	25 – 300 °C
Heating rate	10 °C/min
Purge gas	N ₂
Flow rate	50 mL/min

DSC analysis determined that the melting point onset and peak at about 199 °C and 200 °C, respectively. A plot of the DSC thermogram is shown in **FIG. 2** (lower trace).

[0840] TGA analysis was performed using a TA Instrument. About 1-5 mg of a sample was loaded onto a pre-tared aluminum pan and heated with the parameters in the table below. The data were analyzed using TRIOS.

Instrument	TA, Discovery TGA 55
Sample pan	Aluminum, open
Temperature range	RT – 300 °C
Heating rate	10 °C/min
Purge gas	N ₂
Flow rate	Balance chamber: 40 mL/min
	Sample chamber: 25 mL/min

The TGA analysis determined that free base Form I of Compound 1 exhibits no weight loss upon heating prior to decomposition. A plot of the TGA thermogram is shown in **FIG. 2** (upper trace).

[0841] The collected crystals were observed under magnification using polarized light microscopy. Light microscopy analysis was performed using an ECLIPSE LV100POL (Nikon, JPN,) microscope. Each sample was placed on a glass slide with a drop of immersion oil and covered with a glass slip. The sample was observed using a 4 – 20× objective with polarized light. Micrometer bar was inserted onto the image as a reference for size. Representative images of the crystals are shown in **FIG. 3**.

Example 3: Free base crystalline Form II of Compound 1

[0842] An amount of Compound 1 as prepared in Example 1 was dissolved in DMSO at RT at a concentration of 100 mg/mL. Anti-solvent (water) was added gradually until the ratio of DMSO to anti-solvent was 1:1 by volume, and the mixture was stirred until precipitation occurred. The solids were isolated and characterized accordingly, and assigned as free base Form II (mono-DMSO solvate). 14% residual DMSO was detected by NMR (data not shown).

[0843] A plot of the XRPD is shown in FIG. 4; and Table 2, below, summarizes selected peaks observed in the XRPD plot.

Table 2: Selected Peaks in the mono-DMSO solvate of Compound 1 (Form II)

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
6.7	1.8%	20.0	7.6%
8.6	6.8%	20.2	35.8%
9.8	0.9%	20.7	43.6%
10.4	3.3%	21.1	1.7%
10.8	4.7%	21.7	12.2%
12.3	57.9%	22.5	1.5%
12.8	2.2%	22.7	14.6%
13.4	9.3%	23.0	23.2%
14.1	7.7%	23.3	7.9%
14.4	1.9%	23.8	0.9%
15.6	53.9%	24.5	1.0%
16.1	8.6%	25.0	16.0%
16.4	10.4%	25.7	26.5%
17.1	39.9%	26.5	19.2%
17.4	6.9%	27.0	10.5%
18.7	100.0%	27.9	12.5%
19.5	28.9%	31.4	15.3%
19.7	23.7%		

[0844] Differential scanning calorimetry (DSC) was performed as described in Example 2. The DSC plot exhibited an endothermic onset and peak at about 143 °C and about 148 °C due to desolvation, suggesting Form II was a mono-DMSO solvate. DSC analysis determined melting point onset and peak at about 197 °C and about 198 °C, respectively. A plot of the DSC thermogram is shown in FIG. 5 (lower trace). After heating to 160 °C by DSC, Form II converted to Form I by XRPD (data not shown).

[0845] TGA was performed as described in Example 2. The TGA analysis determined that free base Form II of Compound 1 exhibits about a 1.5% weight loss upon heating to around 90 °C, and about a 13.6% weight loss upon heating between 90 and 158 °C, corresponding to 14% of residual DMSO by ¹H-NMR (data not shown) and suggesting Form II was a mono-DMSO solvate. A plot of the TGA thermogram is shown in FIG. 5 (upper trace).

[0846] The collected crystals were observed under magnification using polarized light microscopy as described in Example 2. Representative images of the crystals are shown in FIG. 6.

[0847] Alternative preparation of Form II: An amount of Compound 1 as prepared in Example 1 was dissolved in DMSO at RT at a concentration of 100 mg/mL. Anti-solvent (isopropanol) was added gradually until the ratio of DMSO to anti-solvent was 1:1 by volume, and the mixture was stirred until precipitation occurred. The solids were isolated as a mono-DMSO solvate, free base Form II.

Example 4: HCl salt Form I of Compound I

[0848] Compound 1 (105 mg) was added to MeOH (1 mL) at 50 °C. Then 12 M HCl acid (1.1 eq.) was added. The mixture became clear after adding the acid. MTBE (4 ml) was added to the mixture; precipitation occurred. The mixture was stirred at room temperature for about 7h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as HCl salt Form I (mono-HCl salt) (anhydrate).

[0849] A plot of the XRPD is shown in FIG. 7, and Table 3, below, summarizes selected peaks observed in the XRPD plot.

Table 3: Selected Peaks in HCl salt Form I of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
7.0	13.5%	20.6	35.1%
8.0	11.1%	20.9	36.2%
11.3	1.6%	21.2	33.0%
11.7	23.3%	21.7	31.3%
12.2	13.0%	22.0	1.7%
13.2	97.0%	22.3	15.0%
13.5	10.9%	22.8	0.9%
13.7	64.4%	23.4	1.0%
14.0	4.1%	23.6	8.3%

15.3	15.2%	24.0	6.7%
16.1	1.7%	24.3	63.2%
17.2	20.7%	25.0	31.6%
17.7	100.0%	25.3	35.5%
18.4	22.8%	25.6	27.1%
18.9	1.7%	26.1	63.7%
19.2	1.7%	27.2	13.5%
20.0	19.5%	28.9	77.6%

[0850] Differential scanning calorimetry (DSC) was performed as described in Example 2. No melting peak was detected prior to decomposition at around 300 °C by DSC. A plot of the DSC thermogram is shown in FIG. 8 (lower trace).

[0851] TGA was performed as described in Example 2. No weight loss was detected prior to decomposition by TGA. A plot of the TGA thermogram is shown in FIG. 8 (upper trace).

[0852] The hygroscopicity and physical stability of the collected crystals was measured using dynamic vapor sorption (DVS). Moisture sorption/desorption data were collected on a DVS Intrinsic. about 10-50 mg of a sample was placed into a tared sample chamber and automatically weighed. The samples were analyzed with the setting parameters in the table below.

Instrument	SMS, DVS Intrinsic
dm/dt	0.002%/min
Sample size	10-50 mg
Drying/ Measurement temperature	40 °C/ 25 °C
Cycle	Full cycle
Minimum dm/dt stability duration	30 min
Maximum dm/dt equilibrium time	120 min
Save data rate	5 s
Gas and Total flow rate	N ₂ , 200 sccm
Post experiment total flow	200 sccm
RH step size	10% RH
Method	Adsorption: 0, 10, 20, 30, 40, 50, 60, 70, 80, 90
	Desorption: 80, 70, 60, 50, 40, 30, 20, 10, 0

A plot of the DVS measurement is shown in FIG. 9. HCl salt Form I was slightly hygroscopic with 0.5% weight gain after undergoing a DVS cycle from about 0% relative humidity (RH) to about 90% relative humidity (RH) at 25 °C. No form change occurred after DVS testing (data not shown).

[0853] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 10.

[0854] Alternative preparation 1: Compound 1 (405 mg) was added to MeOH (0.6 mL) at 50 °C. Then 12 M HCl acid (1.1 eq.) was added and precipitation occurred in a few minutes. MTBE (2.4 mL) was added into the mixture at 50 °C and stirred for 30 min. Then the mixture was slowly cooled to RT and stirred at RT for about 7 h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The sample was irregular shaped crystals with small size and trace organic solvent.

[0855] Alternative preparation 2: Compound 1 (2.5 g) was added to MeOH (3 mL) at 50 °C. Then 12 M HCl acid (1.1 eq.) was added and precipitation occurred. MTBE (1.25 mL) was added into the mixture, and the mixture was stirred at RT for 2.5 hours. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The sample was irregular shaped crystals with small size, and the crystallinity was high. NMR showed 0.1% residual MeOH (data not shown).

[0856] Alternative trials were conducted to prepare HCl salt on 20 mg scale with 1.2 eq. 12 M HCl acid. The resulting solids were characterized after being dried at 50 °C under vacuum overnight. The procedures and results are summarized in the table below.

Solvent	Temp.	Observation	Result
MeOH (0.5 mL)	RT	The solids were dissolved after adding the acid at 50 °C. No precipitation occurred after cooled to RT. Added 2 mL of MTBE, precipitation occurred and stirred at RT for ~5 h.	HCl salt Form I 0.7% MTBE
Acetone (0.3 mL)	RT	The solids were dissolved after adding the acid at 50 °C. No precipitation occurred after cooled to RT. Precipitation occurred after adding 1.2 mL of n-heptane. Stirred at RT for ~5 h.	HCl salt Form I 1.4% acetone
ACN (0.75 mL)	50 °C	The solids were dissolved at 50 °C, precipitation occurred after adding the acid. Stirred at RT for ~5 h.	HCl salt Form I 1.5% ACN
IPA (1.2 mL)	40 °C	The solids were suspended at 60 °C, the mixture became clear after adding the acid and then precipitation occurred in 1 min. Stirred at RT for ~5 h.	HCl salt Form I 0.1% IPA

Example 5: Sulfate salt Form I of Compound 1

[0857] Compound 1 (20 mg) was added to acetone (0.3 mL) at 50 °C. Then sulfuric acid (1.2 eq.) was added. The solids dissolved after adding the acid. The mixture was cooled to room temperature and water (1.2 mL) was added. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C

overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as sulfate salt Form I (hydrate). No residual solvent was observed by NMR (data not shown).

[0858] A plot of the XRPD is shown in FIG. 11, and Table 4, below, summarizes selected
5 peaks observed in the XRPD plot.

Table 4: Selected Peaks in sulfate salt Form I of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
8.0	60.6%	19.0	19.7%
8.5	75.3%	20.2	15.6%
8.8	33.4%	20.9	1.0%
10.0	7.0%	21.4	7.6%
11.5	7.6%	21.7	8.5%
12.9	37.0%	22.1	15.7%
13.2	100.0%	22.9	8.6%
15.0	46.2%	23.4	5.4%
15.6	59.3%	23.7	1.2%
16.1	47.2%	24.3	26.0%
16.9	8.7%	24.7	2.6%
17.2	10.8%	25.1	16.1%
17.5	3.9%	26.7	17.4%

[0859] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined several thermal events attributed to dehydration, melting, and form transition. DSC analysis determined a first endotherm onset and peak at 78 °C and 118 °C,
10 respectively; a second endotherm onset and peak at 206 °C and 220 °C, respectively; an exotherm onset and peak at 227 °C and 231 °C, respectively; and a third endotherm onset and peak at 273 °C and 279 °C, respectively. A plot of the DSC thermogram is shown in FIG. 12 (lower trace).

[0860] TGA was performed as described in Example 2. The TGA analysis determined that
15 sulfate salt Form I of Compound 1 exhibits about a 2.8% weight loss upon heating to around 130 °C. A plot of the TGA thermogram is shown in FIG. 12 (upper trace).

[0861] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 1. Representative images of the crystals are shown in FIG. 13.

Example 6: Tosylate salt Form I of Compound 1

- [0862] Compound 1 (20 mg) was added to ethyl acetate (0.9 mL) at 50 °C. Then p-toluenesulfonic acid (1.2 eq.) was added, and the solids dissolved after adding the acid. Precipitation occurred within 10 minutes. . The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as tosylate salt Form I (mono tosylate) (anhydrate). 0.2% residual ethyl acetate was observed by NMR (data not shown).
- [0863] A plot of the XRPD is shown in FIG. 14 and Table 5, below, summarizes selected peaks observed in the XRPD plot.

10 **Table 5:** Selected Peaks in tosylate salt Form I of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
4.7	10.3%	20.5	15.1%
6.8	19.0%	21.4	17.9%
9.6	35.8%	22.0	5.9%
11.7	7.9%	22.8	26.7%
13.7	6.8%	24.0	21.8%
14.5	62.4%	24.3	43.1%
15.5	12.5%	25.5	14.1%
15.9	14.6%	29.3	25.7%
18.2	100.0%	30.0	11.1%
19.5	31.4%		

- [0864] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined melting point onset and peak at 222 °C and 231 °C, respectively. A plot of the DSC thermogram is shown in FIG. 15 (lower trace).
- 15 [0865] TGA was performed as described in Example 2. The TGA analysis determined that tosylate salt Form I of Compound 1 exhibits about a 1.2% weight loss upon heating from about 110 °C to about 230 °C. A plot of the TGA thermogram is shown in FIG. 15 (upper trace).
- [0866] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 16.
- 20

Example 7: Tosyalte salt Form II of Compound 1

[0867] Compound 1 (20 mg) was suspended in isopropanol (1.2 mL) at 60 °C. Then p-toluenesulfonic acid (1.2 eq.) was added, and the solids dissolved after adding the acid.

Precipitation occurred within 10 minutes. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as tosylate salt Form II (mono tosylate) (anhydrate). No residual solvent was observed by NMR (data not shown).

[0868] A plot of the XRPD is shown in FIG. 17 and Table 6, below, summarizes selected peaks observed in the XRPD plot.

Table 6: Selected Peaks in tosylate salt Form II of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
6.7	92.8%	18.4	17.6%
7.1	12.9%	19.1	30.4%
9.1	20.7%	19.4	3.5%
9.5	10.8%	20.1	51.8%
10.2	21.0%	20.5	29.8%
13.0	13.1%	22.1	5.6%
14.3	29.2%	23.3	5.4%
14.7	5.8%	23.8	15.3%
15.4	82.9%	24.2	43.7%
15.7	8.6%	24.6	5.0%
16.7	2.8%	24.9	4.0%
17.0	28.3%	26.2	30.4%
17.3	100.0%		

[0869] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined melting point onset and peak at 223 °C and 229 °C, respectively. A plot of the DSC thermogram is shown in FIG. 18 (lower trace).

[0870] TGA was performed as described in Example 2. The TGA analysis determined that tosylate salt Form II of Compound 1 exhibits about a 1.4 % weight loss upon heating from about 90 °C to about 230 °C. A plot of the TGA thermogram is shown in FIG. 18 (upper trace).

[0871] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 19.

Example 8: Tosylate salt Form III of Compound 1

[0872] Compound 1 (20 mg) was added to acetone (0.3 mL) at 60 °C. Then p-toluenesulfonic acid (1.2 eq.) was added, and the solids dissolved after adding the acid. No

precipitation occurred after cooling to room temperature. Heptane (0.9 mL) was added at room temperature, and precipitation occurred. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned
5 as tosylate salt Form III (mono tosylate) (anhydrate). No residual solvent was observed by NMR (data not shown).

[0873] A plot of the XRPD is shown in FIG. 20 and Table 7, below, summarizes selected peaks observed in the XRPD plot.

Table 7: Selected Peaks in tosylate salt Form III of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
4.7	3.0%	19.5	36.4%
6.9	25.3%	20.1	10.1%
9.7	20.7%	20.5	19.4%
11.6	10.0%	20.8	2.1%
13.7	7.5%	21.2	42.6%
14.1	16.6%	21.4	27.4%
14.5	34.8%	22.0	42.1%
14.9	21.7%	22.9	21.8%
15.5	32.9%	24.0	44.1%
16.0	13.5%	24.4	25.7%
16.9	11.7%	24.9	9.0%
18.2	100.0%	25.5	11.9%
19.0	4.6%		

10

[0874] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined melting point onset and peak at 206 °C and 222 °C, respectively. A plot of the DSC thermogram is shown in FIG. 21 (lower trace).

[0875] TGA was performed as described in Example 2. The TGA analysis determined that
15 tosylate salt Form III of Compound 1 exhibits about a 2.6% weight loss upon heating from about 110 °C to about 222 °C. A plot of the TGA thermogram is shown in FIG. 21 (upper trace).

[0876] The hygroscopicity and physical stability of the collected crystals was measured using dynamic vapor sorption (DVS) as described in Example 4. A plot of the DVS
20 measurement is shown in FIG. 22. Tosylate salt Form III was slightly hygroscopic with 3.2% weight gain after undergoing a dynamic vapor sorption cycle from about 0 % relative

humidity (RH) to about 90 % RH at 25 °C, and no form change occurred after DVS testing (data not shown).

[0877] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 23.

5 [0878] The tosylate salt exhibited complex polymorphism.

Example 9: Besylate salt Form I of Compound 1

[0879] Compound 1 (20 mg) and benzenesulfonic acid (1.1 eq.) were added to acetone (0.3 mL) at 50 °C. Precipitation occurred after adding heptane (0.9 mL) at room temperature. The mixture was stirred at room temperature for about 3h. The solid was collected by filtration,
10 dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as besylate salt Form I (mono besylate) (anhydrate). 0.2% residual heptane was observed by NMR (data not shown).

[0880] A plot of the XRPD is shown in FIG. 24 and Table 8, below, summarizes selected peaks observed in the XRPD plot.

15 **Table 8: Selected Peaks in besylate salt Form I of Compound 1**

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
6.8	67.2%	20.3	62.6%
7.5	16.2%	20.8	41.5%
9.1	15.1%	21.3	40.8%
9.6	9.4%	22.0	7.5%
10.6	17.6%	22.7	10.6%
14.5	58.7%	23.2	5.1%
15.0	22.7%	24.0	66.6%
15.4	63.0%	24.6	11.5%
15.9	21.5%	25.2	100.0%
16.8	10.0%	25.6	26.2%
17.3	68.5%	26.0	46.4%
17.7	55.7%	26.9	15.4%
18.3	38.6%	28.2	21.6%
19.1	79.1%	29.7	11.4%
19.6	20.1%	36.0	11.7%

[0881] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined a broad endothermic onset and peak at about 190 °C and 202 °C,

respectively, possibly due to melting and/or release of the residual solvent. A plot of the DSC thermogram is shown in FIG. 25 (lower trace).

[0882] TGA was performed as described in Example 2. The TGA analysis determined that besylate salt Form I of Compound 1 exhibits about a 0.6 % weight loss upon heating from about 100 °C to about 210 °C. A plot of the TGA thermogram is shown in FIG. 25 (upper trace).

[0883] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 26.

Example 10: Besylate salt Form II of Compound 1

10 [0884] Compound 1 (20 mg) and benzenesulfonic acid (1.1 eq.) were added to ethyl acetate (0.9 mL) at 50 °C. Precipitation occurred within 5 minutes. The mixture was stirred at room temperature for about 3h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as besylate salt Form II (mono besylate) (anhydrate). 0.3% residual ethyl acetate
15 was observed by NMR (data not shown).

[0885] A plot of the XRPD is shown in FIG. 27 and Table 9, below, summarizes selected peaks observed in the XRPD plot.

Table 9: Selected Peaks in besylate salt Form II of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
7.6	100.0%	17.3	1.2%
9.0	1.4%	18.1	2.6%
10.0	2.0%	19.2	10.1%
10.4	1.4%	19.7	5.5%
11.4	1.0%	20.1	2.7%
12.1	1.3%	21.7	1.0%
12.4	2.1%	22.4	1.4%
13.0	1.1%	22.9	13.0%
14.7	2.4%	24.4	1.1%
15.2	6.6%	25.0	5.1%
15.7	3.1%	26.5	11.0%
16.2	1.2%	17.3	1.2%

- [0886] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined a broad endothermic onset and peak at 154 °C and 165 °C, respectively. A plot of the DSC thermogram is shown in FIG. 28 (lower trace).
- [0887] TGA was performed as described in Example 2. The TGA analysis determined that besylate salt Form II of Compound 1 exhibits about a 0.5% weight loss upon heating from about 64 °C to about 185 °C. A plot of the TGA thermogram is shown in FIG. 28 (upper trace).
- [0888] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 29.
- 10 [0889] The besylate exhibited polymorphism in addition to the trapped solvents.

Example 11: Mesylate salt Form I of Compound 1

- [0890] Compound 1 (20 mg) was dissolved in acetonitrile (0.75 mL) at 50 °C. Then methanesulfonic acid (1.2 eq.) was added, and precipitation occurred. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as mesylate salt Form I (mono mesylate salt) (anhydrate). No residual solvent was observed by NMR (data not shown).
- 15 [0891] A plot of the XRPD is shown in FIG. 30 and Table 10, below, summarizes selected peaks observed in the XRPD plot.

20 **Table 10: Selected Peaks in mesylate salt Form I of Compound 1**

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
10.1	1.1%	19.3	16.3%
11.0	6.7%	20.3	11.6%
11.9	12.4%	20.7	16.5%
12.7	9.7%	21.2	19.8%
14.2	100.0%	21.5	13.4%
15.1	4.0%	22.1	24.6%
17.0	89.5%	22.5	11.0%
17.5	12.3%	23.5	2.0%
18.0	19.5%	24.1	38.5%
18.6	50.8%	24.9	17.6%

- [0892] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined an endothermic having an onset and peak at 218°C and 222°C,

respectively. A plot of the DSC thermogram for mesylate salt Form I is shown in FIG. 31 (lower trace).

[0893] TGA was performed as described in Example 2. The TGA analysis determined that all mesylate salt Form I of Compound 1 exhibit about a 0.5 % weight loss upon heating from about 125 °C to about 225 °C. A plot of the TGA thermogram for mesylate salt Form I is shown in FIG. 31 (upper trace).

[0894] The hygroscopicity and physical stability of the collected mesylate Form I crystals was measured using dynamic vapor sorption (DVS) as described in Example 4. A plot of the DVS measurement is shown in FIG. 32. Mesylate salt Form I was hygroscopic with 4 % and 19% water uptake at 80 % and 90 % relative humidity (RH), respectively, and became nearly amorphous after DVS testing (data not shown).

[0895] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 33.

Example 12: Mesylate salt Form II of Compound 1

[0896] Compound 1 (20mg) was suspended in isopropanol (1.2 mL) at 60 °C. Then methanesulfonic acid (1.2 eq.) was added, and the solids dissolved after adding the acid. Precipitation occurred after adding heptane (1.2 mL) at room temperature. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as mesylate salt Form II (mono mesylate) (anhydrate). About 0.7 % residual heptane was observed by NMR (data not shown).

[0897] A plot of the XRPD is shown in FIG. 34 and Table 11, below, summarizes selected peaks observed in the XRPD plot.

Table 11: Selected Peaks in mesylate salt Form II of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
8.7	100.0%	17.5	16.3%
9.9	7.5%	18.5	1.6%
10.3	7.6%	19.1	13.4%
10.9	1.3%	19.8	3.5%
11.4	5.9%	20.2	7.0%
12.1	4.6%	20.5	4.1%
15.0	9.4%	21.2	2.5%
15.4	11.1%	22.7	5.4%

15.9	3.7%	23.2	27.1%
16.5	4.9%	24.1	5.6%
17.2	9.8%		

- [0898] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined an endothermic having an onset and peak at 176 °C and 190 °C, respectively. A plot of the DSC thermogram for mesylate salt Form II is shown in **FIG. 35** (lower trace).
- [0899] TGA was performed as described in Example 2. The TGA analysis determined that all mesylate salt Form II of Compound 1 exhibit about a 0.2 % weight loss upon heating from about 140 °C to about 205 °C. A plot of the TGA thermogram for mesylate salt Form II is shown in **FIG. 35** (upper trace).
- 10 [0900] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in **FIG. 36**.

Example 13: Mesylate salt Form III of Compound 1

- [0901] Compound 1 (20 mg) was added to acetone (0.3 mL) at 50 °C. Then methanesulfonic acid (1.2 eq.) was added, and the solids dissolved after adding the acid. No precipitation occurred after cooling to room temperature. Precipitation occurred after adding heptane (0.9 mL) at room temperature. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as mesylate salt Form III (mono mesylate) (anhydrate or solvate). 1.2 % residual heptane was observed by NMR (data not shown).
- 15
- 20
- [0902] A plot of the XRPD is shown in **FIG. 37** and **Table 12**, below, summarizes selected peaks observed in the XRPD plot.

Table 12: Selected Peaks in mesylate salt Form III of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
6.1	19.0%	18.0	13.6%
6.5	100.0%	18.5	63.8%
8.9	48.8%	19.4	35.2%
13.1	18.2%	19.6	76.0%
13.3	17.4%	20.5	5.8%
13.8	25.7%	21.4	50.9%

14.4	18.2%	21.7	10.0%
14.6	9.0%	22.9	7.9%
16.8	5.6%	23.6	19.7%
17.5	39.9%	26.0	11.4%

[0903] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined an endothermic having an onset and peak at 138 °C and 173 °C, respectively. A plot of the DSC thermogram for mesylate salt Form III is shown in **FIG. 38** (lower trace).

[0904] TGA was performed as described in Example 2. The TGA analysis determined that all mesylate salt Form III of Compound 1 exhibit about a 1.5 % weight loss upon heating from about 80 °C to about 180 °C. A plot of the TGA thermogram for mesylate salt Form II is shown in **FIG. 38** (upper trace).

10 [0905] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in **FIG. 39**.

[0906] Mesylate salts exhibited complex polymorphism in addition to hygroscopicity.

Example 14: Phosphate salt of Compound 1 (amorphous)

15 [0907] Compound 1 (20 mg) was added to acetone (0.3 mL) at 50 °C. Then phosphoric acid (1.1 eq.) was added. Precipitation occurred after adding heptane (0.6 mL) at room temperature. The mixture was stirred at room temperature for about 5h. Oil was obtained.

[0908] Compound 1 (20 mg) was added to ethyl acetate (0.9 mL) at 50 °C. Then phosphoric acid (1.1 eq.) was added and precipitation occurred. The mixture was stirred at room temperature for about 5h. Amorphous solid was obtained. .

20 [0909] No crystalline phosphate salt was obtained.

Example 15: Maleate salt Form I of Compound 1

25 [0910] Compound 1 (20 mg) was added to ethyl acetate (0.9 mL) at 50 °C. Then maleic acid (1.1 eq.) was added, and the solids precipitated within 20 minutes. The mixture was cooled to room temperature and stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as maleate salt Form I (mono maleate) (anhydrate). No residual solvent was observed by NMR (data not shown).

[0911] A plot of the XRPD is shown in FIG. 40 and Table 13, below, summarizes selected peaks observed in the XRPD plot.

Table 13: Selected Peaks in maleate salt Form I of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
7.9	2.6%	18.1	1.9%
8.3	3.2%	18.7	6.8%
8.7	5.4%	20.0	5.3%
9.9	1.9%	21.2	1.4%
11.4	0.7%	22.5	10.7%
12.7	1.5%	23.3	5.9%
14.8	10.2%	24.0	1.9%
15.5	100.0%	24.6	1.1%
16.7	1.2%		

[0912] Differential scanning calorimetry (DSC) was performed as described in Example 2.

5 DSC analysis determined an overlapping melting/decomposition onset and peak at 191 °C and 195 °C, respectively. A plot of the DSC thermogram is shown in FIG. 41 (lower trace).

[0913] TGA was performed as described in Example 2. The TGA analysis determined that maleate salt Form I of Compound 1 exhibits no weight loss prior to decomposition. A plot of the TGA thermogram is shown in FIG. 41 (upper trace).

10 [0914] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 42.

Example 16: Maleate salt Form II of Compound 1

[0915] Compound 1 (20 mg) was added to acetone (0.3 mL) at 50 °C. Then acid (1.1 eq.) was added, and the solids dissolved after adding the acid. No precipitation occurred after
15 cooling to room temperature. Precipitation occurred after adding heptane (0.9 mL) at room temperature. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as maleate salt Form II (mono maleate salt) (acetone solvate). 1.3 % residual acetone was observed by NMR (data
20 not shown).

[0916] A plot of the XRPD is shown in FIG. 43 and Table 14, below, summarizes selected peaks observed in the XRPD plot.

Table 14: Selected Peaks in maleate salt Form II of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
8.7	13.6%	18.3	100.0%
9.1	14.7%	18.8	54.8%
9.6	39.0%	19.3	39.4%
11.0	5.8%	20.6	8.1%
11.6	17.2%	21.0	12.2%
11.9	47.2%	21.4	58.3%
13.3	48.8%	22.2	24.8%
13.5	12.7%	22.8	9.7%
13.9	9.3%	24.0	91.1%
14.2	48.2%	24.3	30.9%
14.3	75.8%	24.7	36.7%
15.1	14.2%	24.9	17.5%
15.3	62.4%	25.3	15.7%
17.0	26.6%	27.1	11.7%
17.5	71.3%	29.7	13.4%
17.9	32.0%		

[0917] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined two endothermic peaks due to desolvation and melting. DSC analysis determined a first endothermic onset and peak at 135 °C and 142 °C, respectively, and a second endothermic onset and peak at 155 °C and 162 °C, respectively. A plot of the DSC thermogram is shown in FIG. 44 (lower trace).

[0918] TGA was performed as described in Example 2. The TGA analysis determined that maleate salt Form II of Compound 1 exhibits about a 1.6 % weight loss upon heating from about 80 °C to about 144 °C. A plot of the TGA thermogram is shown in FIG. 44 (upper trace).

[0919] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 45.

[0920] Preparation of a maleate salt in isopropanol yielded a solid with low crystallinity.

Example 17: Tartrate salt of Compound 1

[0921] Compound 1 (20 mg), L-tartaric acid (1.1 eq.), and methanol (50 µL) were added to acetone (0.3 mL) at 50 °C. Precipitation occurred after adding heptane (0.6 mL) at room temperature. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight, and had low crystallinity.

Example 18: Citrate salt Form I of Compound 1

[0922] Compound 1 (20 mg) was added to acetone (0.3 mL) at 50 °C. Then citric acid (1.1 eq.) was added. Precipitation occurred after adding the acid. Heptane (0.3 mL) was added at room temperature, and the mixture was stirred at room temperature for about 3h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized.

- 5 The solids were isolated and characterized accordingly, and assigned as citrate salt Form I (mono citrate) (possible acetone solvate). 3.2 % residual acetone was observed by NMR (data not shown).

[0923] A plot of the XRPD is shown in FIG. 46 and Table 15, below, summarizes selected peaks observed in the XRPD plot.

10 **Table 15:** Selected Peaks in citrate salt Form I of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
3.8	2.8%	17.5	1.4%
7.6	0.5%	17.9	4.6%
9.4	0.8%	18.3	0.6%
9.7	0.7%	18.9	3.1%
10.6	3.8%	19.2	100.0%
11.4	12.5%	19.5	7.7%
11.8	0.7%	20.3	22.5%
12.2	1.6%	20.6	0.5%
12.4	0.5%	20.9	0.4%
13.0	1.0%	21.2	0.9%
13.1	3.7%	21.8	2.1%
13.8	1.8%	22.6	9.5%
13.9	1.4%	23.1	8.7%
14.4	0.5%	23.4	0.8%
15.1	3.5%	23.8	3.1%
15.3	0.4%	24.5	5.9%
15.8	13.4%	24.7	3.1%
17.0	3.8%	27.1	11.2%

- [0924] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined one endothermic onset and peak at about 159 °C and 167 °C, respectively, due to melting, desolvation, or decomposition. A plot of the DSC thermogram is shown in FIG. 47 (lower trace).
- 15

[0925] TGA was performed as described in Example 2. The TGA analysis determined that citrate salt Form I of Compound 1 exhibits about a 22% weight loss upon heating from about 95 °C to about 225 °C. A plot of the TGA thermogram is shown in FIG. 47 (upper trace).

[0926] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 48.

Example 19: Fumarate salt Form I of Compound 1

[0927] Compound 1 (20 mg) was dissolved in tetrahydrofuran (0.2 mL) at 50 °C. Then
5 fumaric acid (1.1 eq.) was added, and precipitation occurred. Additional tetrahydrofuran (0.6 mL) was added at room temperature, and the mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as fumarate salt Form I (mono fumarate) (THF solvate). 5.8 % residual THF was observed by
10 NMR (data not shown).

[0928] A plot of the XRPD is shown in FIG. 49 and Table 16, below, summarizes selected peaks observed in the XRPD plot.

Table 16: Selected Peaks in fumarate salt Form I of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
4.2	34.9%	19.4	19.0%
8.5	29.8%	19.8	96.4%
9.6	3.3%	20.4	3.4%
10.6	40.4%	21.1	19.6%
12.2	2.4%	21.4	100.0%
12.7	11.5%	22.6	3.1%
13.0	8.4%	23.4	60.3%
15.0	7.8%	24.8	6.5%
15.9	16.3%	25.7	28.0%
16.1	7.2%	26.1	15.0%
17.0	25.6%	27.4	17.7%
17.8	5.2%	28.1	11.2%
18.4	2.3%	30.4	11.3%
18.8	27.1%	31.9	11.3%

15 [0929] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined melting point onset and peak at 194 °C and 199 °C, respectively. A plot of the DSC thermogram is shown in FIG. 50 (lower trace).

[0930] TGA was performed as described in Example 2. The TGA analysis determined that fumarate salt Form I of Compound 1 exhibits about a 9.7 % weight loss upon heating from

about 100 °C to about 210 °C. A plot of the TGA thermogram is shown in **FIG. 50** (upper trace).

[0931] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in **FIG. 51**.

5 **Example 20: Fumarate salt Form II of Compound 1**

[0932] Compound 1 (20 mg) was added to acetone (0.3 mL) at 50 °C. Then fumaric acid (1.1 eq.) was added, and precipitation occurred. Additional acetone (0.6 mL) was added at room temperature, and the mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized.

10 The solids were isolated and characterized accordingly, and assigned as fumarate salt Form II (mono fumarate) (acetone solvate). 1.8 % residual acetone was observed by NMR (data not shown).

[0933] A plot of the XRPD is shown in **FIG. 52** and **Table 17**, below, summarizes selected peaks observed in the XRPD plot.

15 **Table 17: Selected Peaks in fumarate salt Form II of Compound 1**

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
4.2	51.7%	19.6	10.7%
8.5	80.4%	20.0	26.4%
10.5	29.8%	21.7	100.0%
12.3	6.5%	22.5	4.9%
12.9	68.1%	23.3	19.8%
15.1	16.4%	23.6	63.2%
16.0	20.7%	24.9	9.9%
17.2	4.5%	25.2	10.4%
17.9	2.7%	26.1	30.4%
18.6	7.6%	27.7	18.4%
18.9	39.8%	28.3	10.9%

[0934] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined a first endotherm having an onset and peak at 144 °C, and a second endotherm having an onset and peak at 197 °C and 200 °C, respectively. A plot of the DSC thermogram is shown in **FIG. 53** (lower trace).

20

[0935] TGA was performed as described in Example 2. The TGA analysis determined that fumarate salt Form II of Compound 1 exhibits about a 2.1 % weight loss upon heating from

about 90 °C to about 175 °C. A plot of the TGA thermogram is shown in FIG. 53 (upper trace).

[0936] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 54.

5 **Example 21: Fumarate salt Form III of Compound 1**

[0937] Compound 1 (20 mg) was added to ethyl acetate (0.9 mL) at 50 °C. Then acid (1.1 eq.) was added, and precipitation occurred. The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as
10 fumarate salt Form III (mono fumarate) (ethyl acetate solvate). 2.1 % residual ethyl acetate was observed by NMR (data not shown).

[0938] A plot of the XRPD is shown in FIG. 55 and Table 18, below, summarizes selected peaks observed in the XRPD plot.

Table 18: Selected Peaks in fumarate salt Form III of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
5.7	100.0%	16.9	5.5%
7.9	55.3%	17.4	42.6%
8.9	46.8%	17.9	40.9%
9.4	2.9%	18.5	25.2%
10.3	8.0%	19.3	5.2%
10.6	50.9%	21.1	20.0%
11.5	40.4%	21.9	19.0%
12.8	2.9%	23.5	3.8%
13.4	7.6%	24.0	9.4%
13.9	3.3%	24.3	5.1%
14.3	5.8%	25.1	34.6%
16.1	37.4%	26.9	12.5%

15 [0939] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined a first endotherm having an onset and peak at 106 °C and 113 °C, respectively; a second endotherm having an onset and peak at 158 °C and 164 °C, respectively; and a third endotherm having an onset and peak at 208 °C and 216 °C,
20 respectively. A plot of the DSC thermogram is shown in FIG. 56 (lower trace).

- [0940] TGA was performed as described in Example 2. The TGA analysis determined that fumarate salt Form III of Compound 1 exhibits about a 3.1 % weight loss upon heating from about 85 °C to about 135 °C. A plot of the TGA thermogram is shown in FIG. 56 (upper trace).
- 5 [0941] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 57.
- Example 22: Malate salt Form I of Compound 1**
- [0942] Compound 1 (20 mg) was added to acetone (0.3 mL) at 50 °C. Then L-malic acid (1.1 eq.) was added, and precipitation occurred within 30 minutes. The mixture was stirred at
- 10 room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as malate salt Form I (mono malate) (acetone solvate). 2.2 % residual acetone was observed by NMR (data not shown).
- [0943] A plot of the XRPD is shown in FIG. 58 and Table 19, below, summarizes selected
- 15 peaks observed in the XRPD plot.

Table 19: Selected Peaks in malate salt Form I of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
4.1	2.4%	19.4	21.4%
8.3	4.4%	20.0	47.7%
10.5	8.4%	20.5	24.4%
10.9	100.0%	21.1	12.2%
12.4	15.5%	21.9	3.1%
13.9	9.2%	22.9	9.4%
16.0	1.8%	24.1	4.7%
16.3	3.5%	24.7	5.7%
18.7	13.5%	24.9	4.3%

- [0944] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined a first endothermic onset and peak at 147 °C and 157 °C,
- 20 respectively, and a second endothermic onset and peak at 181 °C and 206 °C, respectively. A plot of the DSC thermogram is shown in FIG. 59 (lower trace).
- [0945] TGA was performed as described in Example 2. The TGA analysis determined that malate salt Form I of Compound 1 exhibits about a 1.9 % weight loss upon heating from

about 75 °C to about 157 °C. A plot of the TGA thermogram is shown in FIG. 59 (upper trace).

[0946] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 60.

5 **Example 23: Malate salt Form II of Compound 1**

[0947] Compound 1 (20 mg) was added to ethyl acetate (0.9 mL) at 50 °C. Then L-malic acid (1.1 eq.) was added, and precipitation occurred The mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and
10 assigned as malate salt Form II (mono malate) (anhydrate). 0.3 % residual solvent was observed by NMR (data not shown).

[0948] A plot of the XRPD is shown in FIG. 61 and Table 20, below, summarizes selected peaks observed in the XRPD plot.

Table 20: Selected Peaks in malate salt Form II of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
4.0	8.6%	18.3	84.1%
8.2	92.6%	19.1	50.0%
8.8	6.6%	20.2	3.5%
11.4	4.0%	20.7	22.2%
12.4	55.1%	21.5	52.3%
14.7	100.0%	21.8	5.8%
15.0	7.5%	22.2	31.7%
15.6	13.1%	22.9	40.5%
15.8	55.4%	23.6	9.5%
16.4	21.4%	24.4	59.0%
16.7	5.8%	24.9	7.3%
17.0	16.7%	26.2	39.1%
17.5	7.5%	27.6	30.8%
17.8	6.9%	31.1	10.1%

15

[0949] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined one overlapping melting/decomposition onset and peak at 180 °C and 183 °C, respectively. A plot of the DSC thermogram is shown in FIG. 62 (lower trace).

[0950] TGA was performed as described in Example 2. The TGA analysis determined that
20 malate salt Form II of Compound 1 exhibits about a 0.5 % weight loss upon heating from

about 95 °C to about 170 °C. A plot of the TGA thermogram is shown in FIG. 62 (upper trace).

[0951] The hygroscopicity and physical stability of the collected crystals was measured using dynamic vapor sorption (DVS) as described in Example 4. A plot of the DVS measurement is shown in FIG. 63. Malate salt Form II was slightly hygroscopic with 1.8% weight gain after undergoing a dynamic vapor sorption cycle from about 0 % relative humidity (RH) to about 90% RH at 25 °C. No form change occurred after DVS testing (data not shown).

[0952] The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in FIG. 64.

Example 24: Succinate salt Form I of Compound 1

[0953] Compound 1 (20 mg) was added to acetone (0.3 mL) at 50 °C. Then succinic acid (1.1 eq.) was added, and precipitation occurred. Heptane (0.6 mL) was added at room temperature and the mixture was stirred at room temperature for about 5h. The solid was collected by filtration, dried under vacuum at 50 °C overnight and then characterized. The solids were isolated and characterized accordingly, and assigned as succinate salt Form I (mono succinate) (acetone solvate). 3.9 % residual acetone was observed by NMR (data not shown).

[0954] A plot of the XRPD is shown in FIG. 65 and Table 21, below, summarizes selected peaks observed in the XRPD plot.

Table 21: Selected Peaks in succinate salt Form I of Compound 1

2-theta (deg)	Rel. Int.	2-theta (deg)	Rel. Int.
4.2	10.7%	19.9	25.6%
8.5	17.4%	20.2	19.8%
10.5	25.4%	20.6	3.8%
10.7	23.0%	21.2	14.6%
12.1	20.8%	21.4	35.0%
12.8	54.7%	22.3	7.2%
15.0	37.8%	22.8	31.2%
15.9	14.2%	23.3	27.7%
17.1	3.2%	24.0	7.1%
17.8	8.7%	24.5	29.5%
18.5	19.8%	24.8	32.1%
18.8	100.0%	25.8	15.2%
19.0	11.2%	28.2	14.6%

19.6	5.1%
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[0955] Differential scanning calorimetry (DSC) was performed as described in Example 2. DSC analysis determined one endothermic onset and peak at 128 °C and 136 °C, respectively. A plot of the DSC thermogram is shown in **FIG. 66** (lower trace).

- 5 **[0956]** TGA was performed as described in Example 2. The TGA analysis determined that succinate salt Form I of Compound 1 exhibits about a 2.8 % weight loss upon heating from about 85 °C to about 145 °C. A plot of the TGA thermogram is shown in **FIG. 66** (upper trace).

- 10 **[0957]** The collected crystals were observed using polarized light microscopy (PLM) as described in Example 2. Representative images of the crystals are shown in **FIG. 67**.

[0958] A summary of crystalline forms is provided in **Table 22** below.

Table 22: Summary of Crystalline Forms

Solid Form Base/Acid Ratio Solvation	DSC Onset/Peak T(°C), ΔH (J/g)	TGA Wt. loss%/T (°C)	DVS Wt. gain% /90RH	Residual solvent by NMR
Free base Form I N/A Anhydrate	199/200, 93	None	/	None
Free base Form II N/A DMSO solvate	143/148, 119 197/198, 84	1.5/25-90 13.6/90-158	/	14% DMSO
HCl salt Form I 1/1 Anhydrate	No melting prior to decomp.	None	0.5	Trace
Sulfate Form I Ratio not determined Hydrate	78/118, 133 206/220, 30 227/231, 35 (exo) 273/279, 66	2.8/RT-130	/	None
Tosylate Form I 1/1 Anhydrate	222/231, 61	1.2/110-230	/	0.2% EA
Tosylate Form II 1/1 Anhydrate	223/229, 45	1.4/90-230	/	None
Tosylate Form III 1/1 Anhydrate	206/222, 48	2.6/110-222	3.2	None
Besylate Form I 1/1 Anhydrate	190/202, 34	0.6/100-210	/	0.4% Heptane

Solid Form Base/Acid Ratio Solvation	DSC Onset/Peak T(°C), ΔH (J/g)	TGA Wt. loss%/T (°C)	DVS Wt. gain% /90RH	Residual solvent by NMR
Besylate Form II 1/1 Anhydrate	154/165, 21	0.5/64-185	/	0.3% EA
Mesylate Form I 1/1 Anhydrate	218/222, 42	0.5/125-225	18.9	None
Mesylate Form II 1/1 Anhydrate	176/190, 29	0.2/140-205	/	0.8% heptane
Mesylate Form III 1/1 Anhydrate	138/173, 29	1.5/80-180	/	1.2% heptane
Maleate Form I 1/1 Anhydrate	191/195, 142 (overlapped)	None.	/	None
Maleate Form II 1/1 Solvate	135/142, 18 155/162, 12	1.6/80-144	/	1.3% acetone
Fumarate Form I 1/1 Solvate	194/199, 80	9.7/100-210	/	5.8% THF
Fumarate Form II 1/1 Solvate	144/144, 13 197/200, 73	2.1/90-175	/	1.8% acetone
Fumarate Form III 1/1 Solvate	106/113, 16 158/164, 25 208/216, 17	3.1/85-135	/	2.1% EA
Citrate Form I 1/1	159/167, 287 (overlapped, m/d)	22/95-225 Desolvation/ decomposition	/	3.2% acetone
Malate Form I 1/1 Solvate	147/157, 56 181/206, 105 (m/d)	1.9/75-157	/	2.2 % acetone
Malate Form II 1/1 Anhydrate	180/183, 201 (overlapped, m/d)	0.5/95-170	1.8	0.3 % EA
Succinate Form I 1/1 Solvate	128/136, 65	2.8/85-145	/	3.9 % acetone

EA = ethyl acetate; THF = tetrahydrofuran

Example 25: Polymorph Screen of free base Form I and HCl salt Form I of Compound 1

- [0959] Free base Form I was prepared as described in Example 1 and used as a starting material for free base polymorph screen. Polymorph screen was performed by various methods including evaporation, slurry, anti-solvent precipitation, cooling crystallization and mechanical treatment. Form I was obtained from most systems, except anti-solvent

precipitation with DMSO/water or DMSO/IPA, which yielded Form II as described in Example 3.

- [0960]** HCl salt Form I was prepared as described in Example 4 and used as a starting material for HCl salt polymorph screen. Polymorph screen was performed by various methods including evaporation, slurry, anti-solvent precipitation, cooling crystallization and mechanical treatment. No new forms were generated in this study.

Example 26: Solubility of free base Form I and HCl salt Form I of Compound 1

[0961] The solubility of HCl salt Form I and the free base Form I was measured in water, pH 2, pH 8 buffer and bio-relevant media with the target solid loading of 5 mg/mL at 37 °C.

Table 23: Solubility Study

Sample	Media ¹	Solubility (mg/mL) ³			pH			XRPD -24 h
		0.5 h	2 h	24 h	0.5 h	2 h	24 h	
Free base Form I	SGF	> 5	> 5	> 5	1.29	1.28	1.2	/
	pH 2.0	4.77	4.83	4.7	3.73	4.22	4.75	/
	FeSSIF	0.6	1.23	1.37	5.03	5.02	5.02	Sticky
	FaSSIF	0.04	0.05	0.06	6.47	6.48	6.47	Unchanged
	Water	<0.0004	<0.0004	<0.0004	6.73	6.60	6.79	Unchanged
	pH 8.0	0.0003	0.0004	0.0005	8	7.99	8.02	Unchanged
HCl salt Form I ²	SGF	0.16	0.18	0.19	/	/	1.21	Unchanged
	pH 2.0	1.38	1.47	1.85	2.15	2.2	2.18	Unchanged
	FeSSIF	1.52	1.74	1.94	5.08	5.12	5.28	Unchanged
	FaSSIF	0.12	0.15	0.14	6.45	6.42	6.46	Unchanged
	Water	2.24	2.44	3.04	6.39	6.29	6.32	Unchanged
	pH 8.0	0.027	0.042	0.018	8	8.01	7.28	FB Form I + extra peaks

- [0962]** The solubility of free base Form I showed pH dependency, it was the highest in simulated gastric fluid (SGF), > 5 mg/mL, and the lowest in water, < 0.4 µg/mL. During the solubility testing, the free base Form I remained unchanged in water, fasted state simulated intestinal fluid (FaSSIF) and pH 8 buffer, while became gel in fed state simulated intestinal fluid (FeSSIF) at 0.5 h.

[0963] HCl salt Form I had lower solubility than the free base in SGF and pH 2 buffer, attributed to common ion effect; but much higher solubility than the free base in other media, especially in water, the solubility was 3 mg/mL with pH shift. HCl salt Form I dissociated to

the free base Form I with extra peaks in pH 8 buffer at 24 h and remained unchanged in other media. Overall, HCl salt Form I showed higher solubility than the free base Form I, except in SGF and pH 2 buffer due to common ion effect.

Example 27: Solid stability of free base Form I and HCl salt Form I of Compound 1

5 [0964] Solid stability of HCl salt Form I and the free base Form I was evaluated at 40 °C/75%RH (open) and 60 °C (capped) conditions for 7 days. No obvious degradation was observed by HPLC and no form changed by XRPD under testing conditions. Hence, the free base Form I and HCl salt Form I were both physically and chemically stable at testing conditions for 7 days.

10 **Table 24: Solid Stability Study**

Sample	Time	40 °C 75%RH		60 °C	
		Purity (Area%)	XRPD	Purity (Area%)	XRPD
Free Base Form I	0 d	99.19	No change	99.19	No change
	7 d	99.15	No change	99.15	No change
HCl salt Form I	0 d	99.47	No change	99.47	No change
	7 d	99.48	No change	99.46	No change

Humidity

[0965] Free base Form I was stored at 92.5% relative humidity (RH) at room temperature for 10 days, and the resulting solids were analyzed by XRPD at 1 and 10 days. The XRPD spectrum remained unchanged. Form I was physically stable under high humidity conditions for 10 days.

[0966] HCl salt Form I was stored at 92.5% relative humidity (RH) at room temperature for 14 days, and the resulting solids were analyzed by XRPD at 1, 7, and 14 days. The XRPD spectrum remained unchanged. HCl salt Form I was physically stable under high humidity conditions for 14 days.

Compression

[0967] Free base Form I (100 mg) was placed in a 0.8 cm diameter die cavity. The punch was inserted into the cavity and compressed with the aid of a benchtop Carver press for 2 min at 2 MPa and 4 MPa, respectively. No form change occurred by XRPD. Crystallinity decreased upon compression at both 2 min at 2 MPa and 4 MPa.

[0968] HCl salt Form I (100 mg) was placed in a 0.8 cm diameter die cavity. The punch was inserted into the cavity and compressed with the aid of a benchtop Carver press for 2 min

at 2 MPa and 4 MPa, respectively. No form change occurred by XRPD. Crystallinity decreased upon compression at both 2 min at 2 MPa and 4 MPa.

Wet Granulation Simulation

5 [0969] Free base Form I (15 mg) was mixed with 5 μ L of water into a vial, then vortexed for about 5 min, the resulting solids were collected and characterized by XRPD. The results showed that the crystal form remained unchanged suggesting free base Form I was physically stable during wet granulation simulation experiment.

10 [0970] HCl salt Form I (15 mg) was mixed with 5 μ L of water into a vial, then vortexed for about 5 min, the resulting solids were collected and characterized by XRPD. The results showed that the crystal form remained unchanged suggesting free base Form I was physically stable during wet granulation simulation experiment.

Mechanical treatment

15 [0971] Mechanical treatment was carried out with dry grinding as well as solvent-drop grinding using free base Form I as the starting material. For dry grinding, Form I (15 mg) was ground into a mortar for 5 minutes and analyzed by XRPD at 2 min and 5 min, respectively. For solvent-drop grinding, Form I (15 mg) was weighed into a mortar and ground for about 3 minutes with solvent-drop stepwise, and then the resulting solids were analyzed by XRPD. After grinding, Form I remained unchanged. The crystallinity of free base Form I slightly decreased after dry grinding for 5 min.

20 [0972] Mechanical treatment was carried out with dry grinding as well as solvent-drop grinding using HCl salt Form I as the starting material. For dry grinding, HCl salt Form I (10 mg) was ground into a mortar for 5 minutes and analyzed by XRPD after 2 minutes and 5 minutes. For solvent-drop grinding, HCl salt Form I (15 mg) was weighed into a mortar and ground for about 2 minutes with solvent-drop stepwise, and then the resulting solids were
25 analyzed by XRPD. After grinding, Form I remained unchanged. The crystallinity of free base Form I decreased after dry grinding.

Conclusion

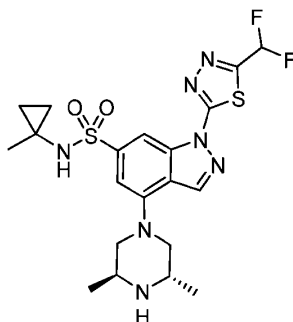
[0973] Free base Form I showed favorable solid-state properties. It may have the residual solvent issue. Crystallinity decreased after grinding and compression.

[0974] HCl salt Form I showed favorable solid-state properties. Crystallinity decreased after grinding and compression. HCl salt Form I showed good solubility in physiologically relevant conditions.

[0975] Although the foregoing invention has been described in some detail by way of illustration and example for purposes of clarity of understanding, one of skill in the art will appreciate that certain changes and modifications may be practiced within the scope of the appended claims. In addition, each reference provided herein is incorporated by reference in its entirety to the same extent as if each reference was individually incorporated by reference. Where a conflict exists between the instant application and a reference provided herein, the instant application shall dominate.

WHAT IS CLAIMED IS:

- 1 **1.** A crystalline form of Compound 1



(Compound 1),

- 3 or a salt thereof.

- 1 **2.** The crystalline form of claim 1, wherein the crystalline form is a single
2 crystalline form substantially free of other crystalline or amorphous forms.

- 1 **3.** The crystalline form of claim 1 or claim 2, wherein the crystalline form
2 is HCl salt Form I.

- 1 4. The crystalline form of claim 3, wherein the crystalline form is a single
2 crystalline form substantially free of other crystalline or amorphous forms.

- 1 5. The HCl salt Form I, according to claim 3 or 4, characterized by an X-
2 ray powder diffraction (XRPD) pattern comprising three or more peaks selected from 11.7,
3 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 19.9 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).

- 1 6. The HCl salt Form I, according to claim 3 or 4, characterized by an X-
2 ray powder diffraction (XRPD) pattern comprising peaks at 11.7, 13.2, 13.7, 17.7, and 18.4
3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).

- 1 7. The HCl salt Form I, according to claim 3 or 4, characterized by an
2 XRPD pattern comprising peaks at 7.0, 11.7, 12.2, 13.2, 13.7, 15.3, 17.2, 17.7, 18.4, and 20.0
3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).

- 1 **8.** The HCl salt Form I, according to any one of claims 3 to 7, wherein
2 the XRPD pattern further comprises one or more peaks selected from 20.6, 20.9, 21.2, 21.7,
3 22.3, 24.3, 25.0, 25.3, 25.6, 26.1, and 28.9 degrees 2θ (± 0.2 degrees 2θ).
- 1 **9.** The HCl salt Form I, according to claim 3 or 4, characterized by an X-
2 ray powder diffraction (XRPD) pattern comprising at least five peaks having a relative
3 intensity of at least 15% as listed in **Table 3**.
- 1 **10.** The HCl salt Form I, according to claim 3 or 4, characterized by an X-
2 ray powder diffraction (XRPD) pattern comprising peaks at 13.2, 13.7, 17.7, 26.1, and 28.9
3 degrees 2θ (± 0.2 degrees 2θ).
- 1 **11.** The HCl salt Form I, according to claim 10, wherein the XRPD pattern
2 further comprises peaks at 20.6, 20.9, 21.2, 24.3, and 25.3 degrees 2θ (± 0.2 degrees 2θ).
- 1 **12.** The HCl salt Form I, according to claim 3 or 4, characterized by an X-
2 ray powder diffraction (XRPD) pattern substantially in accordance with **FIG. 7**.
- 1 **13.** The HCl salt Form I, according to any one of claims 3 to 12,
2 characterized by a differential scanning calorimetry thermogram (DSC) comprising no
3 endotherm prior to decomposition.
- 1 **14.** The HCl salt Form I, according to any one of claims 3 to 12,
2 characterized by a differential scanning calorimetry thermogram (DSC) substantially in
3 accordance with **FIG. 8**.
- 1 **15.** The HCl salt Form I, according to any one of claims 3 to 14,
2 characterized by no weight loss upon heating prior to decomposition, as measured by thermal
3 gravimetric analysis (TGA).
- 1 **16.** The HCl salt Form I, according to any one of claims 3 to 14,
2 characterized by a thermal gravimetric analysis (TGA) thermogram substantially in
3 accordance with **FIG. 8**.
- 1 **17.** The HCl salt Form I, according to any one of claims 3 to 16,
2 characterized by a weight gain of about 0.5 % after undergoing a dynamic vapor sorption
3 cycle from about 0% relative humidity (RH) to about 90% RH at 25 °C.

- 1 **18.** The HCl salt Form I, according to any one of claims **3** to **16**,
2 characterized by a dynamic vapor sorption profile substantially as shown in **FIG. 9**.
- 1 **19.** The crystalline form of claim **1** or claim **2**, wherein the crystalline form
2 is free base Form I.
- 1 **20.** The crystalline form of claim **19**, wherein the crystalline form is a
2 single crystalline form substantially free of other crystalline or amorphous forms.
- 1 **21.** The free base Form I of claim **19** or **20**, characterized by an X-ray
2 powder diffraction (XRPD) pattern comprising three or more peaks selected from 9.0, 12.7,
3 13.7, 16.4, 18.0, 18.3, 18.9, 19.4, and 19.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 1 **22.** The free base Form I of claim **19** or **20**, characterized by an X-ray
2 powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.0, 18.3, and 19.4
3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 1 **23.** The free base Form I of claim **19** or **20**, characterized by an X-ray
2 powder diffraction (XRPD) pattern comprising peaks at 9.0, 11.5, 12.7, 13.7, 16.4, 18.0, 18.3,
3 18.9, 19.4, and 19.7 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 1 **24.** The free base Form I of any one of claims **21** to **23**, wherein the XRPD
2 pattern further comprises one or more peaks selected from 22.5, 23.2, 23.5, 24.6, and 27.8
3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 1 **25.** The free base Form I according to claim **19** or **20**, characterized by an
2 X-ray powder diffraction (XRPD) pattern comprising peaks at 9.0, 16.4, 18.3, 23.5, and 24.6
3 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 1 **26.** The free base Form I according to claim **25**, wherein the XRPD pattern
2 further comprises peaks at 12.7, 13.7, 18.0, 18.9, and 19.4 degrees 2 θ ($\pm$ 0.2 degrees 2 θ).
- 1 **27.** The free base Form I of claim **19** or **20**, characterized by an X-ray
2 powder diffraction (XRPD) pattern substantially in accordance with **FIG. 1**.

- 1 **28.** The free base Form I, according to any one of claims **19** to **27**,
2 characterized by a differential scanning calorimetry thermogram (DSC) comprising an
3 endothermic peak at about 200 °C.
- 1 **29.** The free base Form I, according to any one of claims **19** to **27**,
2 characterized by a differential scanning calorimetry thermogram (DSC) substantially in
3 accordance with **FIG. 2**.
- 1 **30.** The free base Form I, according to any one of claims **19** to **29**,
2 characterized by no weight loss upon heating prior to decomposition, as measured by thermal
3 gravimetric analysis (TGA).
- 1 **31.** The free base Form I, according to any one of claims **19** to **29**,
2 characterized by a thermal gravimetric analysis (TGA) thermogram substantially in
3 accordance with **FIG. 2**.
- 1 **32.** A pharmaceutical composition comprising a crystalline form according
2 to any one of claims **1** to **31** and at least one pharmaceutically acceptable excipient.
- 1 **33.** A pharmaceutical composition prepared by combining a crystalline
2 form according to any one of claims **1** to **31** and at least one pharmaceutically acceptable
3 excipient.
- 1 **34.** A solid state form of Compound 1 or a salt thereof, wherein
2 the solid state form comprises at least 50 wt.% of a particular crystalline form, at least 60
3 wt.% of a particular crystalline form, at least 70 wt.% of a particular crystalline form, at least
4 80 wt.% of a particular crystalline form, at least 90 wt.% of a particular crystalline form, at
5 least 95 wt.% of a particular crystalline form, or at least 99 wt.% of a particular crystalline
6 form, wherein the particular crystalline form is according to any one of claims **1** to **31**.
- 1 **35.** The solid state form of claim **34**, wherein the particular crystalline
2 form is HCl salt Form I.
- 1 **36.** The solid state form of claim **34**, wherein the particular crystalline
2 form is free base Form I.

1 **37.** A pharmaceutical composition comprising a solid state form of any
2 one of claims **34** to **36**, and at least one pharmaceutically acceptable excipient.

1 **38.** A pharmaceutical composition prepared by combining a solid state
2 form of any one of claims **34** to **36** with at least one pharmaceutically acceptable excipient.

1 **39.** A method of treating a cancer in a patient in need thereof, the method
2 comprising administering to the patient a therapeutically effective amount of a crystalline
3 form of any one of claims **1** to **31**, or a pharmaceutical composition of any one of claims **32**,
4 **33**, **37**, and **38**.

1 **40.** A method of treating and/or preventing a homologous recombinant
2 deficient (HRD) cancer in a patient in need thereof, the method comprising administering to
3 the patient a therapeutically effective amount of a crystalline form of any one of claims **1** to
4 **31**, or a pharmaceutical composition of any one of claims **32**, **33**, **37**, and **38**.

1 **41.** A method of treating and/or preventing a cancer in a patient in need
2 thereof, where the cancer is characterized by a reduction or absence of BRCA1 and/or
3 BRCA2 gene expression, the absence or mutation of BRCA1 and/or BRCA2 genes, or
4 reduced function of BRCA1 and/or BRCA2 proteins, the method comprising administering to
5 the patient a therapeutically effective amount of a crystalline form of any one of claims **1** to
6 **31**, or a pharmaceutical composition of any one of claims **32**, **33**, **37**, and **38**.

1 **42.** The crystalline form as defined in any one of claims **1** to **31**, or a
2 pharmaceutical composition of any one of claims **32**, **33**, **37**, and **38**, for use in the treatment
3 of cancer.

1 **43.** Use of a crystalline form as defined in any one of claims **1** to **31**, or a
2 pharmaceutical composition of any one of claims **32**, **33**, **37**, and **38**, in the manufacture of a
3 medicament for the treatment of cancer.

1 **44.** The method of any one of claims **39** to **41**, or the use of claim **42** or **43**,
2 wherein the cancer is breast cancer, ovarian cancer, gastric cancer, lung cancer, cervical
3 cancer, pancreatic cancer, prostate cancer, colorectal cancer, head and neck cancer, or
4 endometrial cancer.