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(54) Title
Solid forms of 1-((S)-4-((R)-7-(6-amino-4-methyl-3- (trifluoromethyl)pyridin-2-yl)-6-chloro-8-fluoro-2-(((S)-1- methylpyrrolidin-2-yl)methoxy)quinazolin-4-yl)-3-methylpiperazin-1-YL)prop-2-en-1-one

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Abstract

Provided herein are solid forms of 1-((S)-4-((R)-7-(6-amino-4-methyl-3-(trifluoromethyl)pyridin-2-yl)-6-chloro-8-fluoro-2-(((S)-1-methylpyrrolidin-2-yl)methoxy)quinazolin-4-yl)-3-methylpiperazin-1-yl)prop-2-en-1-one and methods of their use in the treatment of cancer.

**SOLID FORMS OF 1-((S)-4-((R)-7-(6-AMINO-4-METHYL-3-
(TRIFLUOROMETHYL)PYRIDIN-2-YL)-6-CHLORO-8-FLUORO-2-(((S)-1-
METHYLPYRROLIDIN-2-YL)METHOXY)QUINAZOLIN-4-YL)-3-
METHYLPIPERAZIN-1-YL)PROP-2-EN-1-ONE**

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit of U.S. Provisional Patent Application Number 63/307526, filed 7 February 2022, and is a divisional application of Australian Patent Application No. 2023215488, the disclosures of which are incorporated herein by reference in their entirety and for all purposes.

FIELD OF THE INVENTION

[0002] Provided herein are solid forms of 1-((S)-4-((R)-7-(6-amino-4-methyl-3-(trifluoromethyl)pyridin-2-yl)-6-chloro-8-fluoro-2-(((S)-1-methylpyrrolidin-2-yl)methoxy)quinazolin-4-yl)-3-methylpiperazin-1-yl)prop-2-en-1-one and methods of their use in the treatment of cancer.

BACKGROUND

[0003] Compounds and pharmaceutically acceptable salts thereof described herein are useful in the treatment of various types of cancers, including for example, lung cancer and colorectal cancer.

[0004] It is not possible to predict whether a given molecule will crystallize in one or several crystal forms, whether it will form solvates with different stoichiometries or will combine with other molecules and form stable co-crystals. (Struct Bond (2009) 132: 25–50, 27). The relative stability of varying crystal forms and, for example, the possibility of interconversion between forms or between an amorphous phase and a crystalline phase can significantly and unpredictably affect both physical and chemical properties. Thus, altering the processing, stability, bioavailability, formulation, and/or storage of pharmaceutical compounds. There is no reliable predictability of the solid form and its usefulness as a crystalline solid or amorphous solid.

[0005] Accordingly, there is a need for identifying and isolating stable forms of 1-((S)-4-((R)-7-(6-amino-4-methyl-3-(trifluoromethyl)pyridin-2-yl)-6-chloro-8-fluoro-2-(((S)-1-methylpyrrolidin-2-yl)methoxy)quinazolin-4-yl)-3-methylpiperazin-1-yl)prop-2-en-1-

one.

SUMMARY

[0006] Provided herein are solutions to the problems above and other problems in the art.

[0007] In one aspect provided herein is a compound of formula 1 as described herein.

[0008] In another aspect provided herein is a solid form of the compound of formula 1. In one embodiment, the solid form is Compound A. In one embodiment, the solid form is Compound B. In one embodiment, the solid form is Compound C. In one embodiment, the solid form is Compound D. In one embodiment, the solid form is Compound E. In one embodiment, the solid form is Compound F.

[0009] In another aspect provided herein is a solid form of Compound A corresponding to Compound A Form A as described herein. In one embodiment, is a solid form of Compound A corresponding to Compound A Form B as described herein. In one embodiment, is a solid form of Compound A corresponding to Compound A Form C as described herein. In one embodiment, is a solid form of Compound A corresponding to Compound A Form D as described herein.

[0010] In another aspect provided herein is a solid form of Compound B corresponding to Compound B Form A. Further provided herein is a solid form of Compound C corresponding to Compound C Form A. Further provided herein is a solid form of Compound D corresponding to Compound D Form A. Further provided herein is a solid form of Compound E corresponding to Compound E Form A. Further provided herein is a solid form of Compound F corresponding to Compound F Form A.

[0011] In another aspect provided herein is a method of treating a cancer comprising a KRasG12C mutation in a patient having said cancer, the method comprising administering an effective amount of Compound 1 as described herein or a crystal form corresponding to a Form as described herein of Compound A, Compound B, Compound C, Compound D, Compound E, or Compound F as described herein.

[0012] In still another aspect provided herein is a method of treating a cancer

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comprising a KRasG12C mutation in a patient having said cancer, the method comprising determining if the patient has the mutation and if the patient is determined to have the mutation, then administering an effective amount of Compound 1 as described herein or a crystal form corresponding to a Form as described herein of Compound A, Compound B, Compound C, Compound D, Compound E, or Compound F as described herein.

[0013] The present embodiments can be understood more fully by reference to the detailed description and examples, which are intended to exemplify non-limiting embodiments.

BRIEF DESCRIPTION OF THE FIGURES

[0014] FIG. 1 depicts the XRPD spectrum of Compound A Form A.

[0015] FIG. 2 depicts a TGA thermogram and DSC thermogram overlap for Compound A Form A.

[0016] FIG. 3 depicts a DSC thermogram for Compound A Form A.

[0017] FIG. 4 depicts single crystal XRD of Compound A Form A.

[0018] FIG. 5 depicts the XRPD spectrum of Compound A Form B.

[0019] FIG. 6 depicts a TGA thermogram and DSC thermogram overlap for Compound A Form B.

[0020] FIG. 7 depicts single crystal XRD of Compound A Form C.

[0021] FIG. 8 depicts the XRPD spectrum of Compound A Form D.

[0022] FIG. 9 depicts DSC thermogram for Compound A Form D.

[0023] FIG. 10 depicts a TGA thermogram and DSC thermogram overlap for Compound A Form D.

[0024] FIG. 11 depicts the XRPD spectrum overlay of Compound B Form A.

[0025] FIG. 12 depicts DSC thermogram for Compound B Form A.

[0026] FIG. 13 depicts the XRPD spectrum overlay of Compound C Form A.

[0027] FIG. 14 depicts a TGA thermogram and DSC thermogram overlap for Compound C Form A.

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- [0028] FIG. 15 depicts the XRPD spectrum overlay of Compound D Form A.
- [0029] FIG. 16 depicts a TGA thermogram and DSC thermogram overlap for Compound D Form A.
- [0030] FIG. 17 depicts the XRPD spectrum overlay of Compound E Form A.
- [0031] FIG. 18 depicts the XRPD overlay of slurry competition between Compound A Form A and B.
- [0032] FIG. 19 depicts XRPD of Compound A at varying relative humidity (RH).
- [0033] FIG. 20 depicts single crystal XRD of Compound A Form D.
- [0034] FIG. 21 depicts a view of the crystal packing of Compound A Form D looking down the crystallographic *b*-axis.
- [0035] FIG. 22 depicts a view of the crystal packing of Compound A Form D looking down the crystallographic *c*-axis.
- [0036] FIG. 23 depicts a view of the crystal packing of Form C looking down the crystallographic *b*-axis. Only major components of disorder are shown.
- [0037] FIG. 24 depicts a view of the crystal packing of Form C looking down the crystallographic *c*-axis. Only major components of disorder are shown.
- [0038] FIG. 25 depicts a comparison of crystal packing between hydrated Form D and anhydrous Form C, viewed down crystallographic *b*-axis.
- [0039] FIG. 26 depicts the XRPD spectrum overlay of Compound F Form A.
- [0040] FIG. 27 depicts a TGA thermogram and DSC thermogram overlap for Compound F Form A.

DETAILED DESCRIPTION

[0041] Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by those of ordinary skill in the art to which the invention belongs. See, e.g., Singleton et al., *DICTIONARY OF MICROBIOLOGY AND MOLECULAR BIOLOGY* 2nd ed., J. Wiley & Sons (New York, NY 1994); Sambrook et al., *MOLECULAR CLONING, A LABORATORY MANUAL*, Cold Springs Harbor Press (Cold Springs Harbor, NY 1989). Any methods, devices and materials similar or equivalent to those described herein can be used in the

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practice of this invention.

[0042] The following definitions are provided to facilitate understanding of certain terms used frequently herein and are not meant to limit the scope of the present disclosure. All references referred to herein are incorporated by reference in their entirety.

[0043] As used herein, and unless otherwise specified, the terms “about” and “approximately,” when referring to doses, amounts, or weight percents of ingredients of a composition or a dosage form, mean a dose, amount, or weight percent that is recognized by one of ordinary skill in the art to provide a pharmacological effect equivalent to that obtained from the specified dose, amount, or weight percent. The equivalent dose, amount, or weight percent can be within 30%, 20%, 15%, 10%, 5%, 1%, or less of the specified dose, amount, or weight percent.

[0044] As used herein, and unless otherwise specified, the terms “about” and “approximately,” when referring to a numeric value or range of values used for characterization of a particular solid form described herein (e.g., XRPD peak values) indicate that the value or range of values may deviate from a given value to an extent deemed reasonable to one of ordinary skill in the art while still describing the solid form. In one embodiment, the value of an XRPD peak position may vary by up to $\pm 0.1^\circ 2\theta$ (or ± 0.05 degree 2θ) while still describing the particular XRPD peak.

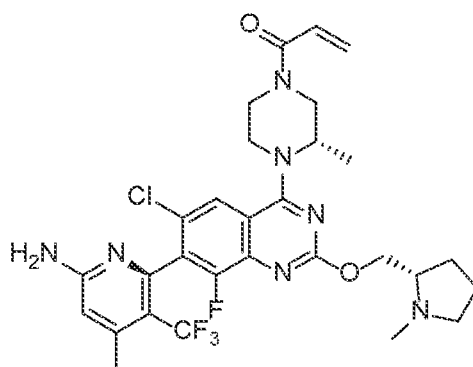
[0045] As used herein, and unless otherwise specified, a crystalline that is “pure,” i.e., substantially free of other crystalline or amorphous solids or other chemical compounds, and contains less than about 10%, 9%, 8%, 7%, 6%, 5%, 4%, 3%, 2%, 1%, 0.5%, 0.4%, 0.3%, 0.2%, 0.1%, 0.05%, or 0.01% of one or more other solid forms on a weight basis. The detection of other solid forms can be accomplished by, for example, diffraction analysis, thermal analysis, elemental combustion analysis and/or spectroscopic analysis. The detection of other chemical compounds can be accomplished by, for example, mass spectrometry analysis, spectroscopic analysis, thermal analysis, elemental combustion analysis and/or chromatographic analysis.

[0046] Unless otherwise specified, the terms “solvate” and “solvated,” as used herein, refer to a solid form of a substance which contains solvent. The terms “hydrate” and “hydrated” refer to a solvate wherein the solvent is water. The terms “solvate” and

“solvated,” as used herein, can also refer to a solvate of a salt, cocrystal, or molecular complex. The terms “hydrate” and “hydrated,” as used herein, can also refer to a hydrate of a salt, cocrystal, or molecular complex.

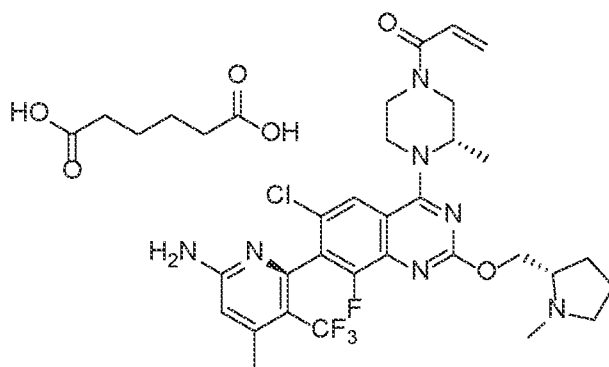
[0047] The term “pharmaceutically acceptable,” refers to a diluent, excipient, or carrier in a formulation compatible with the other ingredient(s) of the formulation and not deleterious to the recipient thereof.

[0048] “Compound 1” refers to a compound having the structure:



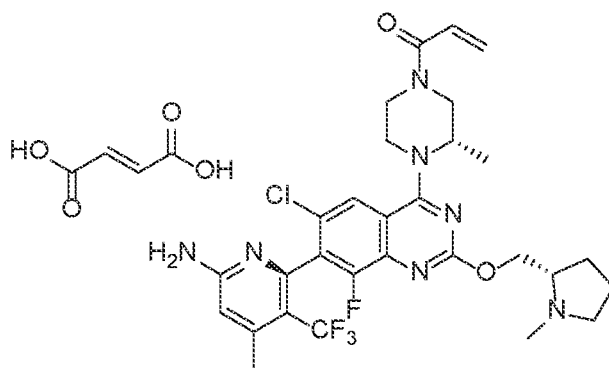
and having the name 1-((S)-4-((R)-7-(6-amino-4-methyl-3-(trifluoromethyl)pyridin-2-yl)-6-chloro-8-fluoro-2-(((S)-1-methylpyrrolidin-2-yl)methoxy)quinazolin-4-yl)-3-methylpiperazin-1-yl)prop-2-en-1-one.

[0049] “Compound A” refers to a compound having the structure:



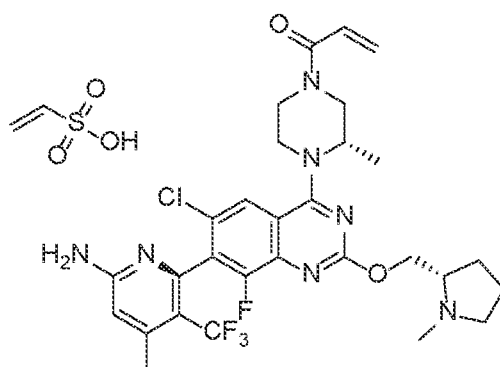
In some embodiments, Compound A is also described as Compound 1 adipate.

[0050] “Compound B” refers to a compound having the structure:



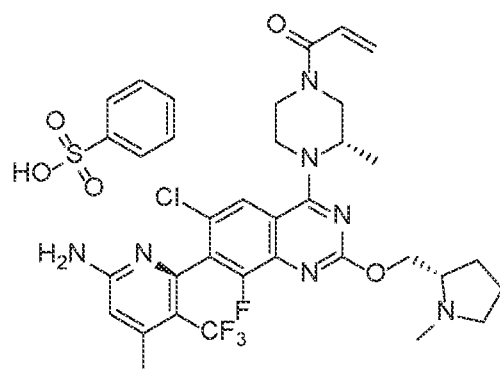
In some embodiments, Compound B is also described as Compound 1 fumarate.

[0051] "Compound C" refers to a compound having the structure:



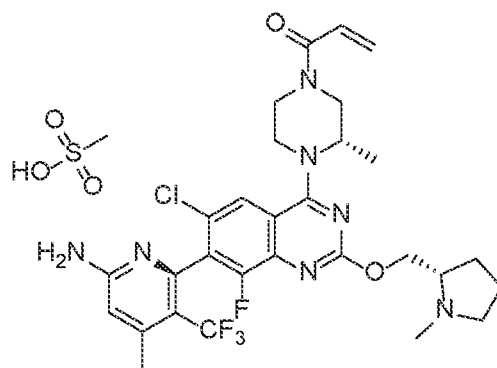
In some embodiments, Compound C is also described as Compound 1 ethylenesulphonate.

[0052] "Compound D" refers to a compound having the structure:



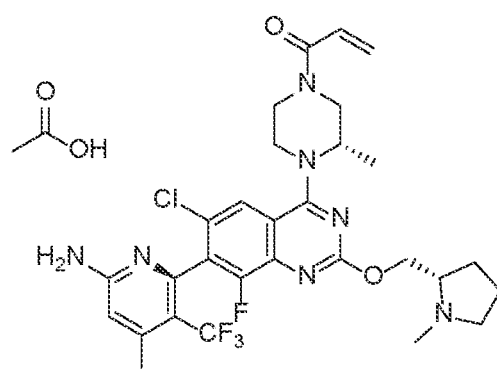
In some embodiments, Compound D is also described as Compound 1 besylate.

[0053] "Compound E" refers to a compound having the structure:



In some embodiments, Compound E is also described as Compound 1 mesylate.

[0054] “Compound F” refers to a compound having the structure:



In some embodiments, Compound F is also described as Compound 1 acetate.

[0055] The term “solid form” refers to a physical form that is not predominantly in a liquid or a gaseous state. A solid form may be a crystalline form or a mixture thereof. In certain embodiments, a solid form may be a liquid crystal. In certain embodiments, the solid form of Compound A is a solid form corresponding to Compound A Form A, Compound A Form B, Compound A Form C, Compound A Form D, Compound B Form A, Compound C Form A, Compound D Form A, or Compound E Form A, or Compound F Form A, or a mixture thereof.

[0056] In one embodiment, the solid form of Compound A is an adipate salt corresponding to Form A. In one embodiment, the solid form of Compound A is an adipate salt corresponding to Form B. In another embodiment, the solid form of Compound A is an adipate salt corresponding to Form C. In still another embodiment, the solid form of Compound A is an adipate salt corresponding to Form D. In one embodiment, the solid form of Compound B is a fumarate salt corresponding to Form A. In another embodiment, the solid form of Compound C is an ethylenesulphonate

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salt corresponding to Form A. In another embodiment, the solid form of Compound D is a besylate salt corresponding to Form A. In another embodiment, the solid form of Compound E is a mesylate salt corresponding to Form A. In another embodiment, the solid form Compound F is an acetate salt corresponding to Form A.

[0057] The term “crystal form” or “crystalline form” refers to a solid form that is crystalline. In certain embodiments, a crystal form of a compound described herein may be substantially free of amorphous solids and/or other crystal forms. In certain embodiments, a crystal form of a compound described herein may contain less than about 1%, less than about 2%, less than about 3%, less than about 4%, less than about 5%, less than about 6%, less than about 7%, less than about 8%, less than about 9%, less than about 10%, less than about 15%, less than about 20%, less than about 25%, less than about 30%, less than about 35%, less than about 40%, less than about 45%, or less than about 50% by weight of one or more amorphous solids and/or other crystal forms. In certain embodiments, a crystal form described herein is pure. In certain embodiments, a crystal form of a compound described herein may be about 99%, 98%, 97%, 96%, 95%, 94%, 93%, 92%, 91%, or 90% pure. In some embodiments, a crystal form is a solid form described herein.

[0058] The term “amorphous” or “amorphous solid” refers to a solid form that not substantially crystalline as determined by X-ray diffraction. In particular, the term “amorphous solid” describes a disordered solid form, i.e., a solid form lacking long range crystalline order. In certain embodiments, an amorphous solid of a compound described herein may be substantially free of other amorphous solids and/or crystal forms. In certain embodiments, an amorphous solid may be pure. In certain embodiments, an amorphous solid of a compound described herein may be about 99%, 98%, 97%, 96%, 95%, 94%, 93%, 92%, 91%, or 90% pure.

[0059] A “patient” or “subject” is defined herein to include animals, such as mammals, including, but not limited to, primates (e.g., humans), cows, sheep, goats, horses, dogs, cats, rabbits, rats, mice, monkeys, chickens, turkeys, quails, or guinea pigs and the like, in one embodiment a mammal, in another embodiment a human. In one embodiment, a subject is a human having or at risk for cancer.

[0060] As used herein, “essentially” refers to at least 90%, at least 95%, at least 98% or at least 99%.

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[0061] In the description herein, if there is a discrepancy between a depicted structure and a name given to that structure, then the depicted structure controls. Additionally, if the stereochemistry of a structure or a portion of a structure is not indicated with, for example, bold wedged, or dashed lines, the structure or portion of the structure is to be interpreted as encompassing all stereoisomers of it. In some cases, however, where more than one chiral center exists, the structures and names may be represented as single enantiomers to help describe the relative stereochemistry.

[0062] Unless otherwise indicated, the terms "a compound of the formula" or "a compound of formula" or "compounds of the formula" or "compounds of formula" refer to any compound selected from the genus of compounds as defined by the formula (including any pharmaceutically acceptable salt of any such compound if not otherwise noted).

[0063] The terms "treat", "treating", and "treatment" refer to clinical intervention designed to alter the natural course of the patient or cell being treated during the course of clinical pathology. Desirable effects of treatment include decreasing the rate of disease progression, ameliorating or palliating the disease state, and remission or improved prognosis. For example, a patient is successfully "treated" if one or more symptoms associated with a cancer described herein are mitigated or eliminated, including, but are not limited to, reducing the proliferation of (or destroying) cancerous cells, decreasing symptoms resulting from the disease, increasing the quality of life of those suffering from the disease, decreasing the dose of other medications required to treat the disease, and/or prolonging survival of patients.

[0064] The term "delaying progression" of a disease refers to deferring, hindering, slowing, retarding, stabilizing, and/or postponing development of a cancer described herein. This delay can be of varying lengths of time, depending on the history of the cancer and/or patient being treated. As is evident to one skilled in the art, a sufficient or significant delay can, in effect, encompass prevention, in that the patient does not develop cancer.

[0065] An "effective amount" is at least the minimum amount required to effect a measurable improvement of a cancer described herein. An effective amount herein may vary according to factors such as the disease state, age, sex, and weight of the

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patient, and the ability of the agent to elicit a desired response in the patient. An effective amount is also one in which any toxic or detrimental effects of the treatment are outweighed by the therapeutically beneficial effects. Beneficial or desired results include results such as eliminating or reducing the risk, lessening the severity, delaying the onset of the disease (including biochemical, histological and/or behavioral symptoms of the disease, its complications and intermediate pathological phenotypes presenting during development of the disease), decreasing one or more symptoms resulting from the disease, increasing the quality of life of those suffering from the disease, decreasing the dose of other medications required to treat the disease, enhancing effect of another medication such as via targeting, delaying the progression of the disease, and/or prolonging survival. In some embodiments, an effective amount of the drug may have the effect in reducing the number of cancer cells; reducing the tumor size; inhibiting (i.e., slow or stop) cancer cell infiltration into peripheral organs; inhibit (i.e., slow or stop) tumor metastasis; inhibiting (i.e., slow or stop) tumor growth; and/or relieving one or more of the symptoms associated with the disorder. An effective amount can be administered in one or more administrations. An effective amount of drug, compound, pharmaceutical composition, or combination therapy described herein can be an amount sufficient to accomplish therapeutic treatment either directly or indirectly. As is understood in the clinical context, an effective amount of a drug, compound, or pharmaceutical composition may or may not be achieved in conjunction with another drug, compound, or pharmaceutical composition, or combination therapy. Thus, an "effective amount" may be considered in the context of administering one or more therapeutic agents, and a single agent may be considered to be given in an effective amount if, in conjunction with one or more other agents, a desirable result may be or is achieved.

[0066] Exemplary solvent abbreviations used herein

Abbreviation	Solvent	Abbreviation	Solvent
MeOH	Methanol	ACN	Acetonitrile
EtOH	Ethanol	DCM	Dichloromethane
IPA	Isopropyl alcohol	EtOAc	Ethyl acetate
MIBK	Methyl isobutyl ketone	IPAc	Isopropyl acetate
MEK	Methyl ethyl ketone	2-MeTHF	2-Methyltetrahydrofuran

Abbreviation	Solvent	Abbreviation	Solvent
NMP	N-Methyl pyrrolidone	THF	Tetrahydrofuran
DMF	Dimethyl formamide	DMSO	Dimethyl sulfoxide
CPME	Cyclopentyl methyl ether	DMAc	Dimethylacetamide
MTBE	Methyl tert-butyl ether	--	--

Polymorphs

[0067] Provided herein are solid forms, formulations comprising such solid forms, and methods of using such solid forms of Compound 1 (e.g. Compound A, B, C, D, or E). In one embodiment provided herein are solid forms of Compound 1. Compound 1 can be a freebase as described herein existing in an amorphous solid form.

[0068] In one embodiment, Compound 1 is a crystalline adipate salt having the formula of Compound A as described herein. In one embodiment, Compound A exists in one or more forms as described herein. In one such embodiment, Compound A comprises Compound A Form A as described herein. In one such embodiment, Compound A comprises Compound A Form B as described herein. In one such embodiment, Compound A comprises Compound A Form C as described herein. In one such embodiment, Compound A comprises Compound A Form D as described herein. In another such embodiment, Compound A consists of a single Form (e.g. A, B, C, D) as described herein. In one embodiment, a compound or pharmaceutical composition described herein comprises two or more forms of Compound A. In one such embodiment, the compound or pharmaceutically acceptable salt thereof described herein comprises a mixture of Compound A Form A and Compound A Form D as described herein. In one embodiment, the compound or pharmaceutically acceptable salt thereof described herein comprises a mixture of Compound A Form A and Compound A Form C as described herein.

[0069] In another embodiment, Compound 1 is a crystalline fumarate salt having the formula of Compound B as described herein. In one such embodiment, Compound B comprises Compound B Form A as described herein.

[0070] In another embodiment, Compound 1 is a crystalline ethylenesulphonate salt having the formula of Compound C as described herein. In one such embodiment, Compound C comprises Compound C Form A as described herein.

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[0071] In another embodiment, Compound 1 is a crystalline besylate salt having the formula of Compound D as described herein. In one such embodiment, Compound D comprises Compound D Form A as described herein.

[0072] In another embodiment, Compound 1 is a crystalline mesylate salt having the formula of Compound E as described herein. In one such embodiment, Compound E comprises Compound E Form A as described herein.

[0073] In another embodiment, Compound 1 is a crystalline acetate salt having the formula of Compound F as described herein. In one such embodiment, Compound F comprises Compound F Form A as described herein.

[0074] Solid forms can be characterized by physical properties such as, for example, stability, solubility and dissolution rate, density, compressibility, hardness, morphology, cleavage, stickiness, solubility, water uptake, electrical properties, thermal behavior, solid-state reactivity, physical stability, and chemical stability) affecting particular processes (e.g., yield, filtration, washing, drying, milling, mixing, tableting, flowability, dissolution, formulation, and lyophilization) which make certain solid forms suitable for the manufacture of a solid dosage form. Such properties can be determined using particular analytical chemical techniques, including solid-state analytical techniques (e.g., X-ray diffraction, microscopy, spectroscopy and thermal analysis), as described herein and are unpredictable. There is no protocol of polymorph screening that can guarantee the identification of all crystal forms of a given molecule.

[0075] Techniques for characterizing crystal forms and amorphous solids include, for example, thermal analysis (e.g., differential scanning calorimetry (DSC), dynamic vapor sorption (DVS), thermal gravimetric analysis (TGA), and hot-stage microscopy), spectroscopy (e.g., infrared, Raman, and solid-state nuclear magnetic resonance), differential scanning calorimetry (DSC), X-ray powder diffractometry (XRPD), single-crystal X-ray diffractometry, vibrational spectroscopy, e.g., infrared (IR) and Raman spectroscopy, solid-state and solution nuclear magnetic resonance (NMR) spectroscopy (including ^1H NMR and ^19F NMR), scanning electron microscopy (SEM), electron crystallography and quantitative analysis, particle size analysis (PSA), surface area analysis, solubility studies, and dissolution studies, ultra-high performance liquid chromatography (UHPLC), and proton nuclear magnetic

resonance spectrum.

[0076] The purity of the solid forms provided herein can be determined by standard analytical methods, such as thin layer chromatography (TLC), gel electrophoresis, gas chromatography, ultra-high performance liquid chromatography (UHPLC), and mass spectrometry (MS).

Compound A: Form A:

[0077] In some embodiments, provided herein is a solid form corresponding to Compound A designated as Form A. Form A is an adipate crystalline solid form of Compound 1. In one embodiment, Form A of Compound A is an anhydrate. In one embodiment, Form A of Compound A is obtained from acetone, THF, or EtOAc. In one embodiment, Form A of Compound A is obtained from acetone. In another embodiment, Form A of Compound A is obtained from THF. In another embodiment, Form A of Compound A is obtained from EtOAc. In one embodiment, the stoichiometric ratio of adipic acid:freebase of Form A of Compound A is 0.9, 1.0, 1.01, 1.02, 1.03, 1.04, 1.05, 1.06, 1.07, 1.08, 1.09, or 1.10. In one such embodiment, the stoichiometric ratio of adipic acid:freebase of Form A of Compound A is 1.06.

[0078] In one embodiment, a solid form provided herein, e.g., Form A of Compound A, is a adipate salt, and is substantially crystalline, as indicated by X-ray powder diffraction pattern (XRPD) measurements. In one embodiment, the XRPD of a solid form provided herein, e.g., Form A of Compound A, is substantially as shown in FIG. 1. In another embodiment, a solid form provided herein, e.g., Form A of Compound A, has one or more characteristic XRPD peaks at approximately 4.8059, 7.506, 8.5157, 9.2973, 9.6474, 11.0365, 11.1828, 11.677, 12.7936, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 16.9847, 18.7038, 18.9135, 19.3881, 19.7517, 19.9746, 20.5178, 21.1487, 21.4666, 21.9673, or $22.1828 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein. In another embodiment, a solid form provided herein, e.g., Form A of Compound A, has at least 3, 5, 10, 15, or 20 characteristic XRPD peaks at approximately 4.8059, 7.506, 8.5157, 9.2973, 9.6474, 11.0365, 11.1828, 11.677, 12.7936, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 16.9847, 18.7038, 18.9135, 19.3881, 19.7517, 19.9746, 20.5178, 21.1487, 21.4666, 21.9673, or $22.1828 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein. In still

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another embodiment, a solid form provided herein, e.g., Form A of Compound A, has at least five, six, seven, eight, nine, ten, eleven, twelve, thirteen, fourteen, fifteen, sixteen, seventeen, eighteen, nineteen, twenty, or all of the characteristic XPRD peaks at approximately 4.8059, 7.506, 8.5157, 9.2973, 9.6474, 11.0365, 11.1828, 11.677, 12.7936, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 16.9847, 18.7038, 18.9135, 19.3881, 19.7517, 19.9746, 20.5178, 21.1487, 21.4666, 21.9673, or $22.1828 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein.

[0079] In still another embodiment, a solid form provided herein, e.g., Form A of Compound A, has at least 10 characteristic XPRD peaks at approximately 4.8059, 7.506, 8.5157, 9.2973, 9.6474, 11.0365, 11.1828, 11.677, 12.7936, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 16.9847, 18.7038, 18.9135, 19.3881, 19.7517, 19.9746, 20.5178, 21.1487, 21.4666, 21.9673, or $22.1828 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein. In another embodiment, a solid form provided herein, e.g., Form A of Compound A, has at least 1, 2, 3, 4, 5, 6, 7, 8, 9, or 10 characteristic XPRD peaks at approximately 9.6474, 11.0365, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 18.7038, 18.9135, or $21.9673 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein.

[0080] In still another embodiment, a solid form provided herein, e.g., Form A of Compound A, has at least 5 characteristic XPRD peaks at approximately 4.8059, 7.506, 8.5157, 9.2973, 9.6474, 11.0365, 11.1828, 11.677, 12.7936, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 16.9847, 18.7038, 18.9135, 19.3881, 19.7517, 19.9746, 20.5178, 21.1487, 21.4666, 21.9673, or $22.1828 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound A, has at least 1, 2, 3, 4, or 5 characteristic XPRD peaks at approximately 9.6474, 11.0365, 15.4059, 16.4193, $18.7038 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein. In one embodiment, a solid form provided herein, e.g., Form A of Compound A, comprises an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 9.6474, 11.0365, 15.4059, 16.4193, $18.7038 (\pm 0.1^\circ 2\theta)$.

[0081] In still another embodiment, a solid form provided herein, e.g., Form A of Compound A, has at least 15 characteristic XPRD peaks at approximately 4.8059,

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7.506, 8.5157, 9.2973, 9.6474, 11.0365, 11.1828, 11.677, 12.7936, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 16.9847, 18.7038, 18.9135, 19.3881, 19.7517, 19.9746, 20.5178, 21.1487, 21.4666, 21.9673, or $22.1828 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound A, has at least 20 characteristic XPRD peaks at approximately 4.8059, 7.506, 8.5157, 9.2973, 9.6474, 11.0365, 11.1828, 11.677, 12.7936, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 16.9847, 18.7038, 18.9135, 19.3881, 19.7517, 19.9746, 20.5178, 21.1487, 21.4666, 21.9673, or $22.1828 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound A, has all of the characteristic XPRD peaks at approximately 4.8059, 7.506, 8.5157, 9.2973, 9.6474, 11.0365, 11.1828, 11.677, 12.7936, 14.5086, 15.4059, 16.1353, 16.4193, 16.6719, 16.9847, 18.7038, 18.9135, 19.3881, 19.7517, 19.9746, 20.5178, 21.1487, 21.4666, 21.9673, and $22.1828 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 1, and as found in Table 1 herein.

[0082] Table 1: Representative XRPD Peaks for Compound A Form A

Pos. [$^\circ 2\theta$]	d-spacing [Å]	Rel. Int. [%]
4.8059	18.37243	12.57
7.506	11.76829	20.27
8.5157	10.37514	6.82
9.2973	9.50454	22.86
9.6474	9.16041	85.05
11.0365	8.01034	57.73
11.1828	7.90589	25.7
11.677	7.57235	11.66
12.7936	6.91391	20.85
14.5086	6.10025	33.73
15.4059	5.7469	100
16.1353	5.48871	40.28
16.4193	5.3944	65.37
16.6719	5.31324	30.23
16.9847	5.21609	18.22
18.7038	4.74037	89.52
18.9135	4.68829	39.33

19.3881	4.57458	19.09
19.7517	4.49117	10.64
19.9746	4.44156	24.31
20.5178	4.32518	24.85
21.1487	4.19755	25.86
21.4666	4.13611	9.25
21.9673	4.04295	43.19
22.1828	4.00415	20.22

[0083] In one embodiment described herein, Form A of Compound A has a TGA thermogram as substantially depicted in FIG. 2, comprising a weight loss of about 2% up to 100 °C.

[0084] In one such embodiment, Form A of Compound A has a DSC thermogram corresponding to FIG. 2, comprising two endotherms at 68.8 °C and 169.1 °C.

[0085] In one embodiment, Form A of Compound A has a DSC thermogram corresponding to FIG. 3, comprising one endotherm at 173.3 °C (peak).

[0086] Single crystal XRD for Compound A Form A is shown in FIG. 4. Absolute stereochemistry was unambiguously determined from the diffraction data and is depicted in FIG. 4.

Compound A: Form B:

[0087] In some embodiments, provided herein is a solid form corresponding to Compound A designated as Form B. Form B is a crystalline solid form of Compound A. In one embodiment, Form B of Compound A is obtained from anti-solvent addition in DCM/toluene, followed by transferring to 5 °C. In one embodiment, Form B of Compound A is an anhydrate. In one embodiment, no form change is observed after air drying at RT overnight. In one embodiment, the stoichiometric ratio of adipic acid:freebase of Form B of Compound A is 1.5, 1.55, 1.6, 1.65, 1.7, 1.8, 1.9, 2, 2.05, or 2.10. In one embodiment, the stoichiometric ratio of adipic acid:freebase of Form B of Compound A is 1.65 or 2.10.

[0088] In one embodiment, a solid form provided herein, e.g., Form B of Compound A, is a adipate salt, and is substantially crystalline, as indicated by X-ray powder diffraction pattern (XRPD) measurements. In one embodiment, the XRPD of a solid

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form provided herein, e.g., Form B of Compound A, is substantially as shown in FIG. 5. In another embodiment, a solid form provided herein, e.g., Form B of Compound A, has one or more characteristic XRPD peaks at approximately 5.2063, 8.8524, 11.1301, 12.3721, 14.63, 15.6885, 17.7161, 18.2857, 19.8008, 21.9253, or 25.1335 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 5, and as found in Table 2 herein. In another embodiment, a solid form provided herein, e.g., Form B of Compound A, has at least 3, 5, 7, 9, or 10 characteristic XRPD peaks at approximately 5.2063, 8.8524, 11.1301, 12.3721, 14.63, 15.6885, 17.7161, 18.2857, 19.8008, 21.9253, or 25.1335 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 5, and as found in Table 2 herein. In still another embodiment, a solid form provided herein, e.g., Form B of Compound A, has at least 5, 6, 7, 8, 9, 10 or all of the characteristic XRPD peaks at approximately 5.2063, 8.8524, 11.1301, 12.3721, 14.63, 15.6885, 17.7161, 18.2857, 19.8008, 21.9253, or 25.1335 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 5, and as found in Table 2 herein.

[0089] In still another embodiment, a solid form provided herein, e.g., Form B of Compound A, has at least 10 characteristic XRPD peaks at approximately 5.2063, 8.8524, 11.1301, 12.3721, 14.63, 15.6885, 17.7161, 18.2857, 19.8008, 21.9253, or 25.1335 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 5, and as found in Table 2 herein. In another embodiment, a solid form provided herein, e.g., Form B of Compound A, has at least 1, 2, 3, 4, 5, 6, 7, 8, 9, or 10 characteristic XRPD peaks at approximately 5.2063, 8.8524, 12.3721, 14.63, 15.6885, 17.7161, 18.2857, 19.8008, 21.9253, or 25.1335 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 5, and as found in Table 2 herein.

[0090] In still another embodiment, a solid form provided herein, e.g., Form B of Compound A, has at least 5 characteristic XRPD peaks at approximately 5.2063, 8.8524, 11.1301, 12.3721, 14.63, 15.6885, 17.7161, 18.2857, 19.8008, 21.9253, or 25.1335 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 5, and as found in Table 2 herein. In still another embodiment, a solid form provided herein, e.g., Form B of Compound A, has at least 1, 2, 3, 4, or 5 characteristic XRPD peaks at approximately 5.2063, 14.63, 17.7161, 21.9253, 25.1335 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 5, and as found in Table 2 herein. In one embodiment, a solid form provided herein, e.g., Form B of Compound A, has an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 5.2063, 14.63, 17.7161, 21.9253, 25.1335 ($\pm 0.1^\circ 2\theta$).

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[0091] Table 2: Representative XRPD Peaks for Compound A Form B

Pos. [2θ]	d-spacing [Å]	Rel. Int. [%]
5.2063	16.96012	100
8.8524	9.98124	13.93
11.1301	7.94318	12.92
12.3721	7.14847	19.39
14.63	6.04991	31.95
15.6885	5.64402	18.78
17.7161	5.00236	19.59
18.2857	4.84781	18.68
19.8008	4.48015	15.37
21.9253	4.0506	22.24
25.1335	3.54035	22.89

[0092] In one embodiment described herein, Form B of Compound A has a TGA thermogram as substantially depicted in FIG. 6, comprising a weight loss of about 2.2% up to 100 °C. No form change may be observed after heating Form B of Compound A under nitrogen to about 90 °C.

[0093] In one such embodiment, Form B of Compound A has a DSC thermogram corresponding to FIG. 6, comprising two broad endotherms at 55.7 °C and 109.6 ° and a small endother at 158.8 °C.

Compound A: Form C:

[0094] In some embodiments, provided herein is a solid form corresponding to Compound A designated as Form C. Form C is a crystalline solid form of Compound A. In one embodiment, Form C of Compound A is an anhydrate. In one such embodiment, Form C of Compound A is an under-occupied variant of Form D. In one embodiment, Form C is a disordered lattice when compared to Form A or Form D of Compound A. In another such embodiment, the asymmetric unit contains one molecule of Compound A with positionally disordered methylpyrrolidine ring and one adipate anion with one COO⁻ group disordered over two sites (approximately 50/50). (See Examples below). In one embodiment, Form C of Compound A is formed by dehydrating Form D of Compound A. In one such embodiment, the percentage of Form D of Compound A after drying is less than 20%, 15%, 12%, 10%, 8%, 6%, 5%, 2%, or 1%.

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[0095] In one embodiment, a solid form provided herein, e.g., Form C of Compound A, is a adipate salt, and is substantially crystalline, as indicated by X-ray powder diffraction pattern (XRPD) measurements. In one embodiment, the XRPD of a solid form provided herein, e.g., Form C of Compound A, is equivalent to Form A of Compound A.

[0096] Single Crystal XRD of Compound A Form C is shown in FIG. 7.

Compound A: Form D:

[0097] In some embodiments, provided herein is a solid form corresponding to Compound A designated as Form D. Form D is a crystalline solid form of Compound B. In one embodiment, Form D of Compound A is a hemihydrate. In one embodiment, Form D of Compound A is formed by exposing Form A or Form C of Compound A as described herein to increasing percentage of relative humidity (rh). In one embodiment, Form D can be dried, thereby reforming Form A and/or Form C of Compound A. In one such embodiment, the percentage of Form D of Compound A after drying is less than 20%, 15%, 12%, 10%, 8%, 6%, 5%, 2%, or 1%.

[0098] In one embodiment, a solid form provided herein, e.g., Form D of Compound A, is a adipate salt, and is substantially crystalline, as indicated by X-ray powder diffraction pattern (XRPD) measurements. In one embodiment, the XRPD of a solid form provided herein, e.g., Form D of Compound A, is substantially as shown in FIG. 8. In another embodiment, a solid form provided herein, e.g., Form D of Compound A, has one or more characteristic XRPD peaks at approximately 7.4529, 9.309, 9.7745, 11.0487, 11.2178, 11.6936, 12.8642, 14.65, 14.9254, 15.2452, 15.5252, 16.0821, 16.3536, 16.6345, 17.1089, 17.3151, 18.6477, 18.7908, 19.5758, 19.8874, 20.2, 20.6308, 21.0403, 21.6587, or $22.0397 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein. In another embodiment, a solid form provided herein, e.g., Form D of Compound A, has at least 3, 5, 10, 15, or 20 characteristic XRPD peaks at approximately 7.4529, 9.309, 9.7745, 11.0487, 11.2178, 11.6936, 12.8642, 14.65, 14.9254, 15.2452, 15.5252, 16.0821, 16.3536, 16.6345, 17.1089, 17.3151, 18.6477, 18.7908, 19.5758, 19.8874, 20.2, 20.6308, 21.0403, 21.6587, or $22.0397 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein. In still another embodiment, a solid form provided herein, e.g., Form D of Compound A, has

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at least 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 14, 15, 16, 17, 18, 19, 20, or all of the characteristic XPRD peaks at approximately 7.4529, 9.309, 9.7745, 11.0487, 11.2178, 11.6936, 12.8642, 14.65, 14.9254, 15.2452, 15.5252, 16.0821, 16.3536, 16.6345, 17.1089, 17.3151, 18.6477, 18.7908, 19.5758, 19.8874, 20.2, 20.6308, 21.0403, 21.6587, or $22.0397 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein.

[0099] In still another embodiment, a solid form provided herein, e.g., Form D of Compound A, has at least 10 characteristic XPRD peaks at approximately 7.4529, 9.309, 9.7745, 11.0487, 11.2178, 11.6936, 12.8642, 14.65, 14.9254, 15.2452, 15.5252, 16.0821, 16.3536, 16.6345, 17.1089, 17.3151, 18.6477, 18.7908, 19.5758, 19.8874, 20.2, 20.6308, 21.0403, 21.6587, or $22.0397 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein. In still another embodiment, a solid form provided herein, e.g., Form D of Compound A, has at least 1, 2, 3, 4, 5, 6, 7, 8, 9, or 10 characteristic XPRD peaks at approximately 15.2452, 11.0487, 18.6477, 18.7908, 9.7745, 11.2178, 16.6345, 16.0821, 16.3536, or $20.6308 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein.

[0100] In still another embodiment, a solid form provided herein, e.g., Form D of Compound A, has at least 5 characteristic XPRD peaks at approximately 7.4529, 9.309, 9.7745, 11.0487, 11.2178, 11.6936, 12.8642, 14.65, 14.9254, 15.2452, 15.5252, 16.0821, 16.3536, 16.6345, 17.1089, 17.3151, 18.6477, 18.7908, 19.5758, 19.8874, 20.2, 20.6308, 21.0403, 21.6587, or $22.0397 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein. In still another embodiment, a solid form provided herein, e.g., Form D of Compound A, has at least 1, 2, 3, 4, or 5 characteristic XPRD peaks at approximately 9.7745, 11.0487, 15.2452, 18.6477, $18.7908 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein. In one embodiment, a solid form provided herein, e.g., Form D of Compound A, has an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 9.7745, 11.0487, 15.2452, 18.6477, 18.7908 ($\pm 0.1^\circ 2\theta$)

[0101] In still another embodiment, a solid form provided herein, e.g., Form D of Compound A, has at least 15 characteristic XPRD peaks at approximately 7.4529, 9.309, 9.7745, 11.0487, 11.2178, 11.6936, 12.8642, 14.65, 14.9254, 15.2452,

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15.5252, 16.0821, 16.3536, 16.6345, 17.1089, 17.3151, 18.6477, 18.7908, 19.5758, 19.8874, 20.2, 20.6308, 21.0403, 21.6587, or $22.0397 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein.

[0102] In still another embodiment, a solid form provided herein, e.g., Form D of Compound A, has at least 20 characteristic XPRD peaks at approximately 7.4529, 9.309, 9.7745, 11.0487, 11.2178, 11.6936, 12.8642, 14.65, 14.9254, 15.2452, 15.5252, 16.0821, 16.3536, 16.6345, 17.1089, 17.3151, 18.6477, 18.7908, 19.5758, 19.8874, 20.2, 20.6308, 21.0403, 21.6587, or $22.0397 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein.

[0103] In still another embodiment, a solid form provided herein, e.g., Form D of Compound A, has all of the characteristic XPRD peaks at approximately 7.4529, 9.309, 9.7745, 11.0487, 11.2178, 11.6936, 12.8642, 14.65, 14.9254, 15.2452, 15.5252, 16.0821, 16.3536, 16.6345, 17.1089, 17.3151, 18.6477, 18.7908, 19.5758, 19.8874, 20.2, 20.6308, 21.0403, 21.6587, or $22.0397 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 8, and as found in Table 3 herein.

[0104] In one embodiment, the single crystal XRD of Compound A Form D is provided in FIG. 21. Crystal packing and other scXRD information for Compound A Form D is as set forth herein (e.g. Examples).

[0105] Table 3: Representative XRPD Peaks for Compound A Form D

Pos. [$^\circ 2\theta$]	d-spacing [Å]	Rel. Int. [%]
7.4529	11.85207	17.22
9.309	9.49265	24.32
9.7745	9.04158	56.55
11.0487	8.00156	64.53
11.2178	7.88129	55.85
11.6936	7.56169	20.16
12.8642	6.87607	13.15
14.65	6.04167	18.46
14.9254	5.93083	7.53
15.2452	5.80712	100
15.5252	5.70301	16.24
16.0821	5.50675	46.63
16.3536	5.41595	41.59

16.6345	5.32509	52.84
17.1089	5.17852	12.68
17.3151	5.11731	9.61
18.6477	4.75451	62.88
18.7908	4.71863	58.63
19.5758	4.53114	16.41
19.8874	4.46084	14.43
20.2	4.39251	32.41
20.6308	4.30174	41.35
21.0403	4.21895	30.63
21.6587	4.09985	15.45
22.0397	4.02983	35.46

[0106] In one embodiment described herein, Compound A Form D has a DSC thermogram as substantially depicted in FIG. 9. In one embodiment, the DSC of Compound A Form D has one endotherm at about 173.4 °C (Peak).

[0107] In one embodiment described herein, Form B of Compound A has a TGA thermogram as substantially depicted in FIG. 10, comprising a weight loss of about 1.4% up to 100 °C.

Compound B: Form A:

[0108] In some embodiments, provided herein is a solid form corresponding to Compound B designated as Form A. Compound B Form A is a fumarate crystalline solid form of Compound B. In one embodiment, Compound B Form A is obtained from acetone and air dried at RT. In one embodiment, Compound B Form A is an anhydrate.

[0109] In one embodiment, no form change was observed before and after heating Compound B Form A to 100 °C under N₂ protection and cooling down and exposed to ambient conditions.

[0110] In one embodiment, a solid form provided herein, e.g., Form A of Compound B, is a fumarate salt, and is substantially crystalline, as indicated by X-ray powder diffraction pattern (XRPD) measurements. In one embodiment, the XRPD of a solid form provided herein, e.g., Form A of Compound B, is substantially as shown in FIG. 11. In another embodiment, a solid form provided herein, e.g., Form A of Compound B, has one or more characteristic XRPD peaks at approximately 6.6796, 7.8754,

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8.4472, 8.724, 9.6832, 12.1433, 13.3393, 14.1386, 14.9097, 15.2963, 17.4837, 18.5763, 19.4416, 20.3394, 21.4642, 22.4341, 23.0009, 23.3901, 23.7124, 24.7816, 26.1995, 26.7708, 27.7302, or $30.9939 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein. In another embodiment, a solid form provided herein, e.g., Form A of Compound B, has at least 3, 5, 10, 15, or 20 characteristic XPRD peaks at approximately 6.6796, 7.8754, 8.4472, 8.724, 9.6832, 12.1433, 13.3393, 14.1386, 14.9097, 15.2963, 17.4837, 18.5763, 19.4416, 20.3394, 21.4642, 22.4341, 23.0009, 23.3901, 23.7124, 24.7816, 26.1995, 26.7708, 27.7302, or $30.9939 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound B, has at least 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, or all of the characteristic XPRD peaks at approximately 6.6796, 7.8754, 8.4472, 8.724, 9.6832, 12.1433, 13.3393, 14.1386, 14.9097, 15.2963, 17.4837, 18.5763, 19.4416, 20.3394, 21.4642, 22.4341, 23.0009, 23.3901, 23.7124, 24.7816, 26.1995, 26.7708, 27.7302, or $30.9939 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein.

[0111] In still another embodiment, a solid form provided herein, e.g., Form A of Compound B, has at least 10 characteristic XPRD peaks at approximately 6.6796, 7.8754, 8.4472, 8.724, 9.6832, 12.1433, 13.3393, 14.1386, 14.9097, 15.2963, 17.4837, 18.5763, 19.4416, 20.3394, 21.4642, 22.4341, 23.0009, 23.3901, 23.7124, 24.7816, 26.1995, 26.7708, 27.7302, or $30.9939 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound B, has at least 1, 2, 3, 4, 5, 6, 7, 8, 9, or 10 characteristic XPRD peaks at approximately 6.6796, 13.3393, 14.9097, 15.2963, 17.4837, 19.4416, 20.3394, 23.0009, 23.7124, or $26.7708 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein.

[0112] In still another embodiment, a solid form provided herein, e.g., Form A of Compound B, has at least 5 characteristic XPRD peaks at approximately 6.6796, 7.8754, 8.4472, 8.724, 9.6832, 12.1433, 13.3393, 14.1386, 14.9097, 15.2963, 17.4837, 18.5763, 19.4416, 20.3394, 21.4642, 22.4341, 23.0009, 23.3901, 23.7124, 24.7816, 26.1995, 26.7708, 27.7302, or $30.9939 \pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound B, has at least 1, 2, 3, 4, or 5 characteristic

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XPRD peaks at approximately 6.6796, 14.9097, 15.2963, 19.4416, 23.7124 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein. In one embodiment, a solid form provided herein, e.g., Form A of Compound B, has an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 6.6796, 14.9097, 15.2963, 19.4416, 23.7124 ($\pm 0.1^\circ 2\theta$).

[0113] In still another embodiment, a solid form provided herein, e.g., Form A of Compound B, has at least 15 characteristic XPRD peaks at approximately 6.6796, 7.8754, 8.4472, 8.724, 9.6832, 12.1433, 13.3393, 14.1386, 14.9097, 15.2963, 17.4837, 18.5763, 19.4416, 20.3394, 21.4642, 22.4341, 23.0009, 23.3901, 23.7124, 24.7816, 26.1995, 26.7708, 27.7302, or 30.9939 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein.

[0114] In still another embodiment, a solid form provided herein, e.g., Form A of Compound B, has at least 20 characteristic XPRD peaks at approximately 6.6796, 7.8754, 8.4472, 8.724, 9.6832, 12.1433, 13.3393, 14.1386, 14.9097, 15.2963, 17.4837, 18.5763, 19.4416, 20.3394, 21.4642, 22.4341, 23.0009, 23.3901, 23.7124, 24.7816, 26.1995, 26.7708, 27.7302, or 30.9939 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein.

[0115] In still another embodiment, a solid form provided herein, e.g., Form A of Compound B, has all of the characteristic XPRD peaks at approximately 6.6796, 7.8754, 8.4472, 8.724, 9.6832, 12.1433, 13.3393, 14.1386, 14.9097, 15.2963, 17.4837, 18.5763, 19.4416, 20.3394, 21.4642, 22.4341, 23.0009, 23.3901, 23.7124, 24.7816, 26.1995, 26.7708, 27.7302, and 30.9939 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 11, and as found in Table 4 herein.

[0116] Table 4: Representative XRPD Peaks for Compound B Form A

Pos. [$^\circ 2\theta$]	d-spacing [Å]	Rel. Int. [%]
6.6796	13.22236	43.06
7.8754	11.21714	22.02
8.4472	10.45905	13.58
8.724	10.12778	20.88
9.6832	9.1266	22.35
12.1433	7.28262	25.77
13.3393	6.63225	31.01

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14.1386	6.25903	12.07
14.9097	5.93701	77.21
15.2963	5.78782	100
17.4837	5.06834	27
18.5763	4.77263	21.29
19.4416	4.5621	66.36
20.3394	4.36271	34.81
21.4642	4.13656	6.92
22.4341	3.95988	12.22
23.0009	3.86356	31.5
23.3901	3.80014	25.7
23.7124	3.74921	52.37
24.7816	3.58982	13.39
26.1995	3.39867	10
26.7708	3.32742	27.07
27.7302	3.21445	12.56
30.9939	2.883	11.13

[0117] In one embodiment described herein, Form A of Compound B has a DSC thermogram as substantially depicted in FIG. 12.

[0118] In one such embodiment, the DSC thermogram comprises one endotherm at about 58.7 °C and an overlapped peak with peak temperature at 157.1 °C.

[0119] In another embodiment, Form A of Compound B is pure. In one such embodiment, Form A of Compound B is substantially free of other solid forms described herein (e.g. amorphous solid). In another embodiment, the purity of Form A of Compound B is no less than about 95%, no less than about 96%, no less than about 97%, no less than about 98%, no less than about 99%, no less than about 99.5%, or no less than about 99.9%.

Compound C: Form A:

[0120] In some embodiments, provided herein is a solid form corresponding to Compound C designated as Form A. Form A of Compound C is a ethylsulphonate crystalline solid form of Compound C. In one embodiment, Form A of Compound C is obtained from ethyl acetate and air dried at RT.

[0121] In one embodiment, no form change was observed before and after heating

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Form A of Compound C to 100 °C under N₂ protection and cooling down and exposed to ambient conditions.

[0122] In one embodiment, a solid form provided herein, e.g., Form A of Compound C, is an ethylenesulphonate salt, and is substantially crystalline, as indicated by X-ray powder diffraction pattern (XRPD) measurements. In one embodiment, the XRPD of a solid form provided herein, e.g., Form A of Compound C, is substantially as shown in FIG. 13. In another embodiment, a solid form provided herein, e.g., Form A of Compound C, has one or more characteristic XRPD peaks at approximately 5.5814, 8.7554, 11.1932, 12.5401, 13.5091, 14.2557, 15.8682, 16.3963, 16.834, 17.5707, 18.9739, 20.2549, 21.3525, 26.5569 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 13, and as found in Table 5 herein. In another embodiment, a solid form provided herein, e.g., Form A of Compound C, has at least 3, 5, 7, 10, or 12 characteristic XRPD peaks at approximately 5.5814, 8.7554, 11.1932, 12.5401, 13.5091, 14.2557, 15.8682, 16.3963, 16.834, 17.5707, 18.9739, 20.2549, 21.3525, 26.5569 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 13, and as found in Table 5 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound C, has at least 5, 6, 7, 8, 9, 10, 11, 12, 13, or all of the characteristic XRPD peaks at approximately 5.5814, 8.7554, 11.1932, 12.5401, 13.5091, 14.2557, 15.8682, 16.3963, 16.834, 17.5707, 18.9739, 20.2549, 21.3525, 26.5569 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 13, and as found in Table 5 herein.

[0123] In still another embodiment, a solid form provided herein, e.g., Form A of Compound C, has at least 10 characteristic XRPD peaks at approximately 5.5814, 8.7554, 11.1932, 12.5401, 13.5091, 14.2557, 15.8682, 16.3963, 16.834, 17.5707, 18.9739, 20.2549, 21.3525, 26.5569 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 13, and as found in Table 5 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound C, has at least 1, 2, 3, 4, 5, 6, 7, 8, 9, or 10 characteristic XRPD peaks at approximately 12.5401, 17.5707, 16.834, 21.3525, 26.5569, 8.7554, 14.2557, 16.3963, 20.2549, 11.1932 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 13, and as found in Table 5 herein.

[0124] In still another embodiment, a solid form provided herein, e.g., Form A of Compound C, has at least 5 characteristic XRPD peaks at approximately 5.5814, 8.7554, 11.1932, 12.5401, 13.5091, 14.2557, 15.8682, 16.3963, 16.834, 17.5707,

18.9739, 20.2549, 21.3525, 26.5569 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 13, and as found in Table 5 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound C, has at least 5 characteristic XPRD peaks at approximately 12.5401, 17.5707, 16.834, 21.3525, 26.5569 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 13, and as found in Table 5 herein. In one embodiment, a solid form provided herein, e.g., Form A of Compound C, has an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 12.5401, 17.5707, 16.834, 21.3525, 26.5569 ($\pm 0.1^\circ 2\theta$).

[0125] Table 5: Representative XRPD Peaks for Compound C Form A

Pos. [$^\circ 2\theta$]	d-spacing [Å]	Rel. Int. [%]
5.5814	15.82116	18.18
8.7554	10.09154	27.49
11.1932	7.89859	22.06
12.5401	7.05308	100
13.5091	6.54927	10.14
14.2557	6.2079	26.79
15.8682	5.58052	19.33
16.3963	5.40194	25.03
16.834	5.26244	71.34
17.5707	5.04343	73.89
18.9739	4.67349	21.79
20.2549	4.38072	23.49
21.3525	4.15796	32.57
26.5569	3.35374	31.51

[0126] In one embodiment described herein, Form A of Compound C has a TGA thermogram as substantially depicted in FIG. 14. In one embodiment, a weight loss of 8.6% up to 100 $^\circ\text{C}$ is observed.

[0127] In one embodiment, Form A of Compound C has a DSC thermogram as substantially depicted in FIG. 14, comprising two endotherms at 81.0 $^\circ\text{C}$ and 149.2 $^\circ\text{C}$ (peak temperature).

[0128] In another embodiment, Form A of Compound C is pure. In one such embodiment, Form A of Compound C is substantially free of other solid forms described herein (e.g. amorphous solid). In another embodiment, the purity of Form A

of Compound C is no less than about 95%, no less than about 96%, no less than about 97%, no less than about 98%, no less than about 99%, no less than about 99.5%, or no less than about 99.9%.

Compound D: Form A:

[0129] In some embodiments, provided herein is a solid form corresponding to Compound D designated as Form A. Form A of Compound D is a crystalline solid form of Compound D. In one embodiment, Form A of Compound D is obtained from evaporation of acetone/n-heptane (1:1 v/v).

[0130] In one embodiment, a solid form provided herein, e.g., Form A of Compound D, is an besylate salt, and is substantially crystalline, as indicated by X-ray powder diffraction pattern (XRPD) measurements. In one embodiment, the XRPD of a solid form provided herein, e.g., Form A of Compound D, is substantially as shown in FIG. 15. In another embodiment, a solid form provided herein, e.g., Form A of Compound D, has one or more characteristic XRPD peaks at approximately 7.3809, 9.2213, 10.7407, 12.7588, 13.6799, 14.654, 15.8145, 17.1195, 18.5979, 19.5937, 20.8941, 21.9068, 24.4114, 25.2558 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 15, and as found in Table 6 herein. In another embodiment, a solid form provided herein, e.g., Form A of Compound D, has at least 3, 5, 7, 10 or 12 characteristic XRPD peaks at approximately 7.3809, 9.2213, 10.7407, 12.7588, 13.6799, 14.654, 15.8145, 17.1195, 18.5979, 19.5937, 20.8941, 21.9068, 24.4114, 25.2558 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 15, and as found in Table 6 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound D, has at least 5, 6, 7, 8, 9, 10, 11, 12, 13, or all of the characteristic XRPD peaks at approximately 7.3809, 9.2213, 10.7407, 12.7588, 13.6799, 14.654, 15.8145, 17.1195, 18.5979, 19.5937, 20.8941, 21.9068, 24.4114, 25.2558 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 15, and as found in Table 6 herein.

[0131] In still another embodiment, a solid form provided herein, e.g., Form A of Compound D, has at least 10 characteristic XRPD peaks at approximately 7.3809, 9.2213, 10.7407, 12.7588, 13.6799, 14.654, 15.8145, 17.1195, 18.5979, 19.5937, 20.8941, 21.9068, 24.4114, 25.2558 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 15, and as found in Table 6 herein. In still another embodiment, a solid form provided

herein, e.g., Form A of Compound D, has at least 1, 2, 3, 4, 5, 6, 7, 8, 9, or 10 characteristic XPRD peaks at approximately 7.3809, 10.7407, 12.7588, 14.654, 15.8145, 17.1195, 18.5979, 21.9068, 24.4114, 25.2558 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 15, and as found in Table 6 herein.

[0132] In still another embodiment, a solid form provided herein, e.g., Form A of Compound D, has at least 5 characteristic XPRD peaks at approximately 7.3809, 9.2213, 10.7407, 12.7588, 13.6799, 14.654, 15.8145, 17.1195, 18.5979, 19.5937, 20.8941, 21.9068, 24.4114, 25.2558 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 15, and as found in Table 6 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound D, has at least 5 characteristic XPRD peaks at approximately 7.3809, 10.7407, 14.654, 18.5979, 25.2558 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 15, and as found in Table 6 herein. In one embodiment, a solid form provided herein, e.g., Form A of Compound D, has an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 7.3809, 10.7407, 14.654, 18.5979, 25.2558 ($\pm 0.1^\circ 2\theta$).

[0133] Table 6: Representative XPRD Peaks for Compound D Form A

Pos. [$^\circ 2\theta$]	d-spacing [Å]	Rel. Int. [%]
7.3809	11.96741	100
9.2213	9.58275	1.19
10.7407	8.23033	18.04
12.7588	6.93266	8.47
13.6799	6.46789	2.68
14.654	6.04005	34.95
15.8145	5.59932	3.01
17.1195	5.17532	3.34
18.5979	4.76713	15.98
19.5937	4.52704	1.74
20.8941	4.24813	2.07
21.9068	4.05397	12.77
24.4114	3.64343	10.56
25.2558	3.52348	15.68

[0134] In one embodiment described herein, Form A of Compound D has a TGA thermogram as substantially depicted in FIG. 16. In one embodiment, a weight loss of

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7.7% was observed up to 170 °C is observed.

[0135] In one embodiment, Form A of Compound D has a DSC thermogram as substantially depicted in FIG. 16, comprising one endotherm at 75.8 °C. In one embodiment, the stoichiometric ratio of benzenesulfonic acid:freebase is 1, 1.01, 1.02, or 1.03.

[0136] In another embodiment, Form A of Compound D is pure. In one such embodiment, Form A of Compound D is substantially free of other solid forms described herein (e.g. amorphous solid). In another embodiment, the purity of Form A of Compound D is no less than about 95%, no less than about 96%, no less than about 97%, no less than about 98%, no less than about 99%, no less than about 99.5%, or no less than about 99.9%.

Compound E: Form A:

[0137] In some embodiments, provided herein is a solid form corresponding to Compound E designated as Form A. Form A of Compound E is a crystalline solid form of Compound E. In one embodiment, Form A of Compound E is obtained from acetone.

[0138] In one embodiment, a solid form provided herein, e.g., Form A of Compound E, is an mesylate salt, and is substantially crystalline, as indicated by X-ray powder diffraction pattern (XRPD) measurements. In one embodiment, the XRPD of a solid form provided herein, e.g., Form A of Compound E, is substantially as shown in FIG. 17. In another embodiment, a solid form provided herein, e.g., Form A of Compound E, has one or more characteristic XRPD peaks at approximately 5.9702, 6.2798, 7.766, 11.9769, 12.4314, 14.8918, 15.5087, 16.002, 17.9217, 18.9264, 20.6365, 22.334, 23.8046, 25.8171, 26.1887 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 17, and as found in Table 7 herein. In another embodiment, a solid form provided herein, e.g., Form A of Compound E, has at least 3, 5, 8, 10, or 12 characteristic XRPD peaks at approximately 5.9702, 6.2798, 7.766, 11.9769, 12.4314, 14.8918, 15.5087, 16.002, 17.9217, 18.9264, 20.6365, 22.334, 23.8046, 25.8171, 26.1887 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 17, and as found in Table 7 herein. In still another embodiment, a solid form provided herein, e.g., Compound E, has at least 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, or all of the characteristic XPRD peaks at approximately 5.9702, 6.2798, 7.766,

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11.9769, 12.4314, 14.8918, 15.5087, 16.002, 17.9217, 18.9264, 20.6365, 22.334, 23.8046, 25.8171, 26.1887 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 17 and as found in Table 7 herein.

[0139] In still another embodiment, a solid form provided herein, e.g., Form A of Compound E, has at least 10 characteristic XPRD peaks at approximately 5.9702, 6.2798, 7.766, 11.9769, 12.4314, 14.8918, 15.5087, 16.002, 17.9217, 18.9264, 20.6365, 22.334, 23.8046, 25.8171, 26.1887 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 17, and as found in Table 7 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound E, has at least 1, 2, 3, 4, 5, 6, 7, 8, 9, or 10 characteristic XPRD peaks at approximately 20.6365, 12.4314, 5.9702, 6.2798, 18.9264, 22.334, 14.8918, 25.8171, 16.002, 23.8046 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 17, and as found in Table 7 herein.

[0140] In still another embodiment, a solid form provided herein, e.g., Form A of Compound E, has at least 5 characteristic XPRD peaks at approximately 5.9702, 6.2798, 7.766, 11.9769, 12.4314, 14.8918, 15.5087, 16.002, 17.9217, 18.9264, 20.6365, 22.334, 23.8046, 25.8171, 26.1887 $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 17, and as found in Table 7 herein. In still another embodiment, a solid form provided herein, e.g., Form A of Compound E, has at least 5 characteristic XPRD peaks at approximately 5.9702, 6.2798, 12.4314, 18.9264, 20.6365, $\pm 0.1^\circ 2\theta$, as depicted in, for example, FIG. 17, and as found in Table 7 herein. In one embodiment, a solid form provided herein, e.g., Form A of Compound E, has an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 5.9702, 6.2798, 12.4314, 18.9264, 20.6365 ($\pm 0.1^\circ 2\theta$).

[0141] Table 7: Representative XRPD Peaks for Compound E Form A

Pos. [$^\circ 2\theta$]	d-spacing [$\text{\AA}$]	Rel. Int. [%]
5.9702	14.79172	72.39
6.2798	14.0631	69.41
7.766	11.37484	23.74
11.9769	7.38342	21.81
12.4314	7.11451	97.62
14.8918	5.94411	41.98
15.5087	5.70906	16.92

16.002	5.53413	36.61
17.9217	4.94544	23.26
18.9264	4.68511	63.17
20.6365	4.30057	100
22.334	3.97739	62.92
23.8046	3.73491	31.31
25.8171	3.44814	36.87
26.1887	3.40005	30.45

[0142] In another embodiment, Form A of Compound E is pure. In one such embodiment, Form A of Compound E is substantially free of other solid forms described herein (e.g. amorphous solid). In another embodiment, the purity of Form A of Compound E is no less than about 95%, no less than about 96%, no less than about 97%, no less than about 98%, no less than about 99%, no less than about 99.5%, or no less than about 99.9%.

Methods of Treating Cancer

[0143] The compounds and solid forms described herein can be administered to the patient in an effective amount (e.g. an amount as described herein) for treating cancer mediated by a KRas^{G12C} mutation. In one such embodiment, the cancer is a solid tumor (e.g. lung cancer, CRC, or pancreatic cancer). It is to be understood that the methods described herein also include treatment with a pharmaceutical composition as described herein comprising a compound (e.g. Compound 1 or a solid form of Compound A, B, C, D, E, or F as described herein. In one embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a cancer, the method comprising administering an effective amount of a solid form corresponding to Compound A, B, C, D, E, or F, or a mixture thereof as described herein) to the patient having cancer.

[0144] In one embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound A as described herein to the patient. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of a solid form of Compound A (e.g. Form A, B, C, or D, or a

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mixture thereof) to the patient as described herein.

[0145] In one embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A as described herein. In one embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form A (e.g. substantially free of another solid form such as Compound A Form B, Compound A Form C, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form A is 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A comprising one or more solid form other than Form A Compound A. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form D. In one such embodiment, the method comprises administering to the patient an effective amount Compound A Form A comprising Compound A Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form D may include Compound A Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%. In another such embodiment, the method comprises administering to the patient an effective amount Compound A Form A further comprising a percentage of Compound A Form C. In one such embodiment, the method comprises administering Compound A Form A comprising Compound A Form C at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form C may include Compound A Form C at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0146] In one embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form D as described herein. In one embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form D (e.g. substantially free of another solid form such as

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Compound A Form A, Compound A Form B, or Compound A Form C. In one such embodiment, the crystalline purity of Compound A Form D is 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A comprising one or more solid form other than Form A Compound A. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A comprising Compound A Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form D may include Compound A Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%. In another such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form C. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A comprising Compound A Form C at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form C may include Compound A Form C at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0147] In one embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form C as described herein. In one embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form C (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form B, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form C is 80%, 85%, 90%, 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form c comprising one or more solid form other than Form A Compound c. In one such embodiment, the method comprises

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administering to the patient an effective amount of Compound A Form C further comprising a percentage of Compound A Form A and/or Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form C comprising Compound A Form A and/or Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form C includes Compound A Form A and/or Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0148] In one embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form B as described herein. In one embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form B (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form C, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form B is 80%, 85%, 90%, 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having lung cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form B comprising one or more solid form other than Form A Compound B. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form B further comprising a percentage of Compound A Form A, Form C, and/or Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form b comprising Compound A Form A, Form C, and/or Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, 10%-50%, or 25%-75%. In another such embodiment, the mixture of Compound A Form B includes Compound A Form A, Form C and/or Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0149] In one embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound B as described herein to the patient. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering

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an effective amount of a solid form of Compound B (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound B Form A to the patient as described herein.

[0150] In one embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound C as described herein to the patient. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of a solid form of Compound C (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound C Form A to the patient as described herein.

[0151] In one embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound D as described herein to the patient. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of a solid form of Compound D (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound D Form A to the patient as described herein.

[0152] In one embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound E as described herein to the patient. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of a solid form of Compound E (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating lung cancer

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mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound E Form A to the patient as described herein.

[0153] In one embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound F as described herein to the patient. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of a solid form of Compound F (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating lung cancer mediated by a KRas^{G12C} mutation in a patient having such a lung cancer, the method comprising administering an effective amount of Compound F Form A to the patient as described herein.

[0154] In such embodiments, the lung cancer is non-small cell lung cancer (NSCLC) comprising KRas^{G12C} mutations. In another embodiment of the methods provided herein, the lung cancer is adenocarcinoma, squamous-cell lung carcinoma or large-cell lung carcinoma. In one such embodiment, the cancer is lung adenocarcinoma. In another such embodiment, the lung cancer is a small cell lung carcinoma. In another embodiment, the lung cancer is small cell lung carcinoma. In still another embodiment, the lung cancer is glandular tumors, carcinoid tumors or undifferentiated carcinomas. The lung cancer can be stage I or II lung cancer. In one embodiment, the lung cancer is stage III or IV lung cancer.

[0155] Further provided herein is the use (UL1) of Compound 1, Compound A, Compound B, Compound C, Compound D, Compound E, Compound F, or a solid form thereof as described herein for the treatment of lung cancer as described herein. Further provided herein is the use (UL2) of Compound A Form A as described herein for the treatment of lung cancer as described herein. Further provided herein is the use (UL3) of Compound A Form C as described herein for the treatment of lung cancer as described herein. Further provided herein is the use (UL4) of Compound A Form D as described herein for the treatment of lung cancer as described herein.

[0156] In one embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a cancer, the method comprising

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administering an effective amount of a solid form corresponding to Compound A, B, C, D, E, or F, or a mixture thereof as described herein) to the patient having cancer.

[0157] In one embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound A as described herein to the patient. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of a solid form of Compound A (e.g. Form A, B, C, or D, or a mixture thereof) to the patient as described herein.

[0158] In one embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A as described herein. In one embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form A (e.g. substantially free of another solid form such as Compound A Form B, Compound A Form C, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form A is 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A comprising one or more solid form other than Form A Compound A. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form D. In one such embodiment, the method comprises administering to the patient an effective amount Compound A Form A comprising Compound A Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form D may include Compound A Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%. In another such embodiment, the method comprises administering to the patient an effective amount Compound A Form A further comprising a percentage of Compound A Form C. In one such embodiment, the method comprises administering Compound A Form A comprising Compound A Form C at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or

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10%-50%. In another such embodiment, the mixture of Compound A Form A and Form C may include Compound A Form C at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0159] In one embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form D as described herein. In one embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form D (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form B, or Compound A Form C. In one such embodiment, the crystalline purity of Compound A Form D is 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A comprising one or more solid form other than Form A Compound A. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A comprising Compound A Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form D may include Compound A Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%. In another such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form C. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A comprising Compound A Form C at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form C may include Compound A Form C at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0160] In one embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of Compound

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A Form C as described herein. In one embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form C (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form B, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form C is 80%, 85%, 90%, 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form c comprising one or more solid form other than Form A Compound c. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form C further comprising a percentage of Compound A Form A and/or Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form C comprising Compound A Form A and/or Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form C includes Compound A Form A and/or Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0161] In one embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form B as described herein. In one embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form B (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form C, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form B is 80%, 85%, 90%, 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having colorectal cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form B comprising one or more solid form other than Form A Compound B. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form B further comprising a percentage of Compound A Form A, Form C, and/or Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form b comprising Compound A Form A, Form C, and/or

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Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, 10%-50%, or 25%-75%. In another such embodiment, the mixture of Compound A Form B includes Compound A Form A, Form C and/or Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0162] In one embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound B as described herein to the patient. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of a solid form of Compound B (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound B Form A to the patient as described herein.

[0163] In one embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound C as described herein to the patient. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of a solid form of Compound C (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound C Form A to the patient as described herein.

[0164] In one embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound D as described herein to the patient. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of a solid form of Compound D (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating colorectal

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cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound D Form A to the patient as described herein.

[0165] In one embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound E as described herein to the patient. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of a solid form of Compound E (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound E Form A to the patient as described herein.

[0166] In one embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound F as described herein to the patient. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of a solid form of Compound F (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating colorectal cancer mediated by a KRas^{G12C} mutation in a patient having such a colorectal cancer, the method comprising administering an effective amount of Compound F Form A to the patient as described herein.

[0167] Further provided herein is the use (UC1) of Compound 1, Compound A, Compound B, Compound C, Compound D, Compound E, Compound F, or a solid form thereof as described herein for the treatment of colorectal cancer as described herein. Further provided herein is the use (UC2) of Compound A Form A as described herein for the treatment of colorectal cancer as described herein. Further provided herein is the use (UC3) of Compound A Form C as described herein for the treatment of colorectal cancer as described herein. Further provided herein is the use (UC4) of Compound A Form D as described herein for the treatment of colorectal cancer as described herein.

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[0168] In one embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a cancer, the method comprising administering an effective amount of a solid form corresponding to Compound A, B, C, D, E, or F, or a mixture thereof as described herein) to the patient having cancer.

[0169] In one embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound A as described herein to the patient. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of a solid form of Compound A (e.g. Form A, B, C, or D, or a mixture thereof) to the patient as described herein.

[0170] In one embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A as described herein. In one embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form A (e.g. substantially free of another solid form such as Compound A Form B, Compound A Form C, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form A is 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A comprising one or more solid form other than Form A Compound A. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form D. In one such embodiment, the method comprises administering to the patient an effective amount Compound A Form A comprising Compound A Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form D may include Compound A Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%. In another such embodiment, the method comprises administering to the patient an effective amount Compound A Form A further comprising a percentage of Compound A Form C. In one such embodiment, the

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method comprises administering Compound A Form A comprising Compound A Form C at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form C may include Compound A Form C at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0171] In one embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form D as described herein. In one embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form D (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form B, or Compound A Form C). In one such embodiment, the crystalline purity of Compound A Form D is 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A comprising one or more solid form other than Form A Compound A. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A comprising Compound A Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form D may include Compound A Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%. In another such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form C. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A comprising Compound A Form C at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form C may include Compound A Form C at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

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[0172] In one embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form C as described herein. In one embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form C (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form B, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form C is 80%, 85%, 90%, 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form c comprising one or more solid form other than Form A Compound c. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form C further comprising a percentage of Compound A Form A and/or Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form C comprising Compound A Form A and/or Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form C includes Compound A Form A and/or Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0173] In one embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form B as described herein. In one embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form B (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form C, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form B is 80%, 85%, 90%, 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having pancreatic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form B comprising one or more solid form other than Form A Compound B. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form B further comprising a percentage of Compound A Form A, Form C, and/or Form D. In

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one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form b comprising Compound A Form A, Form C, and/or Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, 10%-50%, or 25%-75%. In another such embodiment, the mixture of Compound A Form B includes Compound A Form A, Form C and/or Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0174] In one embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound B as described herein to the patient. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of a solid form of Compound B (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound B Form A to the patient as described herein.

[0175] In one embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound C as described herein to the patient. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of a solid form of Compound C (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound C Form A to the patient as described herein.

[0176] In one embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound D as described herein to the patient. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the

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method comprising administering an effective amount of a solid form of Compound D (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound D Form A to the patient as described herein.

[0177] In one embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound E as described herein to the patient. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of a solid form of Compound E (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound E Form A to the patient as described herein.

[0178] In one embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound F as described herein to the patient. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of a solid form of Compound F (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating pancreatic cancer mediated by a KRas^{G12C} mutation in a patient having such a pancreatic cancer, the method comprising administering an effective amount of Compound F Form A to the patient as described herein.

[0179] Further provided herein are methods of treating tumor agnostic cancer comprising a KRas^{G12C} mutation in a patient having such a cancer. In one such embodiment, the method comprising treating tumor agnostic cancer comprising a KRas^{G12C} mutation in a patient having such a cancer by

- (a) determining the absence or presence of a KRas^{G12C} mutation in a sample taken from a patient with a suspected diagnosed cancer; and

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(b) administering to the patient an effective amount of a solid form of Compound A (e.g. Form A, B, C, or D, or a mixture thereof) to the patient as described herein.

[0180] In one embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A as described herein. In one embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form A (e.g. substantially free of another solid form such as Compound A Form B, Compound A Form C, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form A is 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A comprising one or more solid form other than Form A Compound A. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form D. In one such embodiment, the method comprises administering to the patient an effective amount Compound A Form A comprising Compound A Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form D may include Compound A Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%. In another such embodiment, the method comprises administering to the patient an effective amount Compound A Form A further comprising a percentage of Compound A Form C. In one such embodiment, the method comprises administering Compound A Form A comprising Compound A Form C at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form C may include Compound A Form C at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0181] In one embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form D as described herein. In one embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C}

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mutation, an effective amount of pure Compound A Form D (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form B, or Compound A Form C. In one such embodiment, the crystalline purity of Compound A Form D is 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form A comprising one or more solid form other than Form A Compound A. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A comprising Compound A Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form D may include Compound A Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%. In another such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A further comprising a percentage of Compound A Form C. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form A comprising Compound A Form C at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form A and Form C may include Compound A Form C at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0182] In one embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form C as described herein. In one embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form C (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form B, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form C is 80%, 85%, 90%, 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having a tumor

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agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form c comprising one or more solid form other than Form A Compound c. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form C further comprising a percentage of Compound A Form A and/or Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form C comprising Compound A Form A and/or Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, or 10%-50%. In another such embodiment, the mixture of Compound A Form C includes Compound A Form A and/or Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0183] In one embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form B as described herein. In one embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of pure Compound A Form B (e.g. substantially free of another solid form such as Compound A Form A, Compound A Form C, or Compound A Form D. In one such embodiment, the crystalline purity of Compound A Form B is 80%, 85%, 90%, 95%, 97%, 98%, 98.5%, 99%, 99.5%, 99.9%. In another embodiment, the method comprises administering to the patient having a tumor agnostic cancer comprising a KRas^{G12C} mutation, an effective amount of Compound A Form B comprising one or more solid form other than Form A Compound B. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form B further comprising a percentage of Compound A Form A, Form C, and/or Form D. In one such embodiment, the method comprises administering to the patient an effective amount of Compound A Form b comprising Compound A Form A, Form C, and/or Form D at a percentage of about 0.1%-10%, 0.5%-20%, 1%-30%, 1%-40%, 1%-50%, 10%-50%, or 25%-75%. In another such embodiment, the mixture of Compound A Form B includes Compound A Form A, Form C and/or Form D at an amount of less than about 0.5%, 1%, 2%, 5%, 7%, 10%, 15%, 20%, 25%, 40%, 50%, 60%, 75%, 80%, or 90%.

[0184] In one embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer, the method

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comprising administering an effective amount of Compound B as described herein to the patient. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of a solid form of Compound B (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer, the method comprising administering an effective amount of Compound B Form A to the patient as described herein.

[0185] In one embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of Compound C as described herein to the patient. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of a solid form of Compound C (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of Compound C Form A to the patient as described herein.

[0186] In one embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of Compound D as described herein to the patient. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of a solid form of Compound D (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of Compound D Form A to the patient as described herein.

[0187] In one embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of Compound E as described herein to the patient. In one such embodiment is a method of treating a tumor agnostic cancer

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mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of a solid form of Compound E (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of Compound E Form A to the patient as described herein.

[0188] In one embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of Compound F as described herein to the patient. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of a solid form of Compound F (e.g. Form A) to the patient as described herein. In one such embodiment is a method of treating a tumor agnostic cancer mediated by a KRas^{G12C} mutation in a patient having such a tumor agnostic cancer , the method comprising administering an effective amount of Compound F Form A to the patient as described herein.

[0189] Further provided herein is the use (UP1) of Compound 1, Compound A, Compound B, Compound C, Compound D, Compound E, Compound F, or a solid form thereof as described herein for the treatment of tumor agnostic cancer as described herein. Further provided herein is the use (UP2) of Compound A Form A as described herein for the treatment of tumor agnostic cancer as described herein. Further provided herein is the use (UP3) of Compound A Form C as described herein for the treatment of tumor agnostic cancer as described herein. Further provided herein is the use (UP4) of Compound A Form D as described herein for the treatment of tumor agnostic cancer as described herein.

[0190] In one embodiment of the methods and uses described herein, Compound 1 or a solid form described herein (e.g. Compound A Form A/B/C/D, Compound B, Compound C, Compound D, Compound E, or Compound F) is administered as a fixed dose QD administration. In one embodiment, the administration is oral (PO), where Compound 1 or a solid form described herein (e.g. Compound A Form A/B/C/D, Compound B, Compound C, Compound D, Compound E, or Compound F) is formulated as a tablet or capsule. In one embodiment, Compound 1 or a solid form

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described herein (e.g. Compound A Form A/B/C/D, Compound B, Compound C, Compound D, Compound E, or Compound F) is administered at an amount of 5mg-600mg, 5mg-500mg, 5mg-400mg, 5mg-300mg, 5mg-250mg, 5mg-200mg, 5mg-150mg, 5mg-100mg, 5mg-50mg, 5mg-25mg, 25mg-600mg, 25mg-500mg, 25mg-400mg, 25mg-300mg, 25mg-250mg, 25mg-200mg, 25mg-150mg, 25mg-100mg, 25mg-50mg, 50mg-600mg, 50mg-500mg, 50mg-400mg, 50mg-300mg, 50mg-250mg, 50mg-200mg, 50mg-150mg, or 50mg-100mg QD. In another embodiment, Compound 1 or a solid form described herein (e.g. Compound A Form A/B/C/D, Compound B, Compound C, Compound D, Compound E, or Compound F) is administered at an amount of about 5mg, 25mg, 50mg, 100mg, 150mg, 200mg, 250mg, 300mg, 400mg or 500mg. In another embodiment, Compound A Form A or Compound A Form D as described herein is administered at an amount of about 5mg, 25mg, 50mg, 100mg, 150mg, 200mg, 250mg, 300mg, 400mg or 500mg.

Kits:

[0191] Compound 1 and the solid forms described herein (e.g. Compound A, Compound B, Compound C, Compound D, Compound E, and/or Compound F) can be provided as an article of manufacture (i.e a kit) comprising one or more of the compounds described herein for administration. In one embodiment, the kit includes Compound A or a solid form thereof as described herein (e.g. Compound A Form A, B, C, or D) for administration as described herein. In one such embodiment, a kit as described herein comprises Compound A Form A or Compound A Form D as described herein. In another embodiment, the kit includes Compound B Form A. In another embodiment, the kit includes Compound C Form A. In still another embodiment, the kit includes Compound D Form A. In still another embodiment, the kit includes Compound E form A. In still another embodiment, the kit includes Compound F Form A.

[0192] In some instances, the article of manufacture further comprises package insert comprising instructions to treat or delay progression of a solid tumor (e.g. lung cancer, CRC, or pancreatic cancer as described herein). In one such embodiment, the cancer is NSCLC. In one embodiment, the article of manufacture further comprises package insert comprising instructions for using Compound 1 or solid forms described herein (e.g. Compound A, Compound B, Compound C, Compound D, Compound E,

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and/or Compound F) to treat or delay progression of NSCLC in a patient. In one embodiment, the article of manufacture further comprises package insert comprising instructions for using Compound 1 or a solid forms described herein (e.g. Compound A, Compound B, Compound C, Compound D, Compound E, and/or Compound F) to treat or delay progression of pancreatic cancer in a patient. In one embodiment, the article of manufacture further comprises package insert comprising instructions for using Compound 1 or a solid forms described herein (e.g. Compound A, Compound B, Compound C, Compound D, Compound E, and/or Compound F) to treat or delay progression of CRC in a patient.

[0193] In such kits, Compound 1 or a solid forms described herein (e.g. Compound A, Compound B, Compound C, Compound D, Compound E, and/or Compound F) is provided in a container. Suitable containers include, for example, bottles, vials, bags and syringes. The container may be formed from a variety of materials such as glass, plastic (such as polyvinyl chloride or polyolefin), or metal alloy (such as stainless steel or hastelloy). In some instances, the container holds the formulation and the label on, or associated with, the container may indicate directions for use. The article of manufacture or kit may further include other materials desirable from a commercial and user standpoint, including other buffers, diluents, filters, needles, syringes, and package inserts with instructions for use. In some instances, the article of manufacture further includes one or more of another agent (e.g., an additional chemotherapeutic agent or anti-neoplastic agent). Suitable containers for the one or more agents include, for example, bottles, vials, bags and syringes.

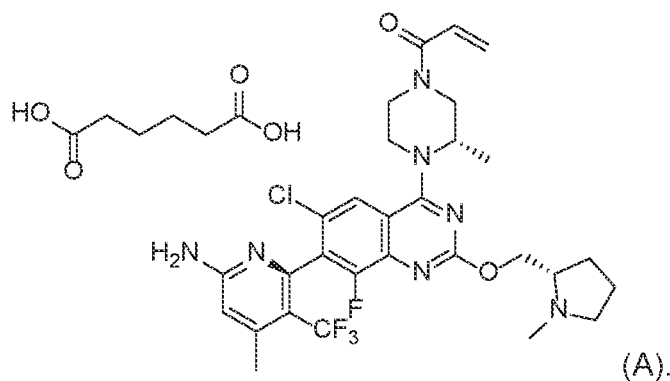
[0194] Any of the articles of manufacture or kits described herein may include instructions to administer Compound 1 or a solid form described herein (e.g. Compound A, Compound B, Compound C, Compound D, Compound E, and/or Compound F) to a patient in accordance with any of the methods described herein.

[0195]

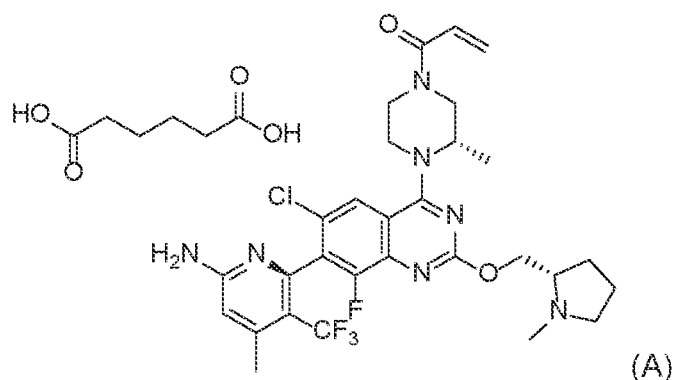
Embodiments:

[0196] Provided below are some exemplary embodiments of the invention.

[0197] Embodiment 1. A compound having the structure:



[0198] Embodiment 2. A crystal form comprising Compound A



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 9.6474, 11.0365, 15.4059, 16.4193, 18.7038 ($\pm 0.1^\circ 2\theta$).

[0199] Embodiment 3. The crystal form of embodiment 2, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 1.

[0200] Embodiment 4. The crystal form of embodiment 2, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 1.

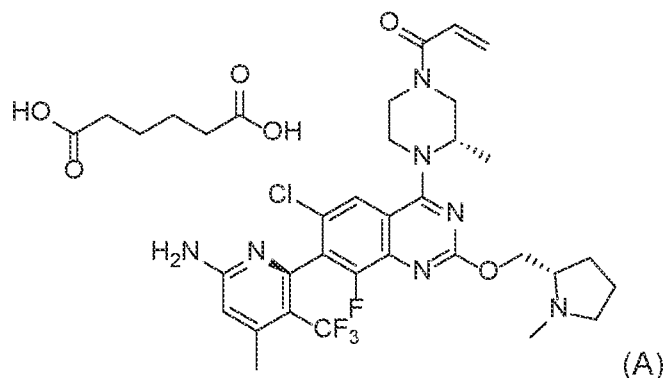
[0201] Embodiment 5. The crystal form of embodiment 2, wherein the crystal form of Compound A has an X-ray powder diffraction pattern substantially as shown in FIG 1.

[0202] Embodiment 6. The crystal form of embodiment 2, wherein the crystal form

of Compound A has a TGA thermograph corresponding substantially to the representative TGA thermogram as depicted in FIG 2.

[0203] Embodiment 7. The crystal form of embodiment 2, wherein the crystal form of Compound A has a DSC thermograph corresponding substantially as depicted in FIG 2.

[0204] Embodiment 8. A crystal form comprising Compound A:



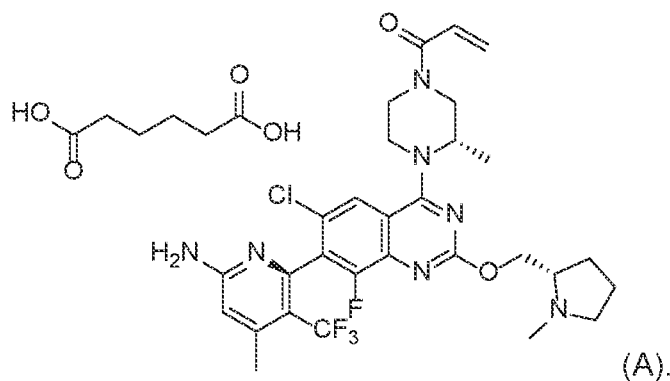
having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 5.2063, 14.63, 17.7161, 21.9253, 25.1335 ($\pm 0.1^\circ 2\theta$).

[0205] Embodiment 9. The crystal form of embodiment 8, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 2.

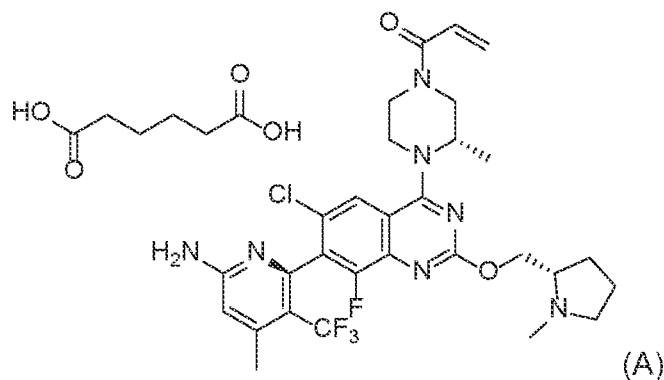
[0206] Embodiment 10. The crystal form of embodiment 8, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 2.

[0207] Embodiment 11. The crystal form of embodiment 8, wherein the crystal form of Compound A has an X-ray powder diffraction pattern substantially as shown in FIG 5.

[0208] Embodiment 12. A crystal form comprising Compound A:



[0209] Embodiment 13. A crystal form comprising Compound A:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 9.7745, 11.0487, 15.2452, 18.6477, 18.7908 ($\pm 0.1^\circ 2\theta$).

[0210] Embodiment 14. The crystal form of embodiment 13, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 3.

[0211] Embodiment 15. The crystal form of embodiment 13, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 3.

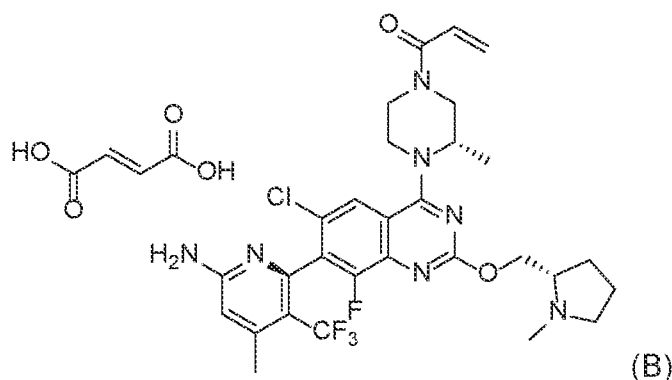
[0212] Embodiment 16. The crystal form of embodiment 13, wherein the crystal form of Compound A has an X-ray powder diffraction pattern substantially as shown in FIG 8.

[0213] Embodiment 17. The crystal form of embodiment 13, wherein the crystal form

of Compound A has a TGA thermograph corresponding substantially to the representative TGA thermogram as depicted in FIG 10.

[0214] Embodiment 18. The crystal form of embodiment 13, wherein the crystal form of Compound A has a DSC thermograph corresponding substantially as depicted in FIG 9.

[0215] Embodiment 19. A crystal form comprising Compound B:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 6.6796, 14.9097, 15.2963, 19.4416, 23.7124 ($\pm 0.1^\circ 2\theta$).

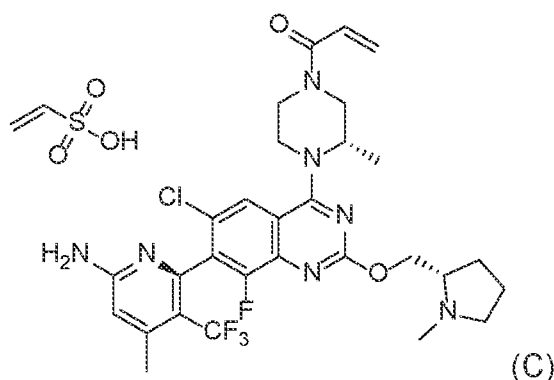
[0216] Embodiment 20. The crystal form of embodiment 19, wherein the crystal form of Compound B has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 4.

[0217] Embodiment 21. The crystal form of embodiment 19, wherein the crystal form of Compound B has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 4.

[0218] Embodiment 22. The crystal form of embodiment 19, wherein the crystal form of Compound B has an X-ray powder diffraction pattern substantially as shown in FIG 11.

[0219] Embodiment 23. The crystal form of embodiment 19, wherein the crystal form of Compound B has a DSC thermograph corresponding substantially as depicted in FIG 12.

[0220] Embodiment 24. A crystal form comprising Compound C:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 12.5401, 17.5707, 16.834, 21.3525, 26.5569 ($\pm 0.1^\circ 2\theta$).

[0221] Embodiment 25. The crystal form of embodiment 24, wherein the crystal form of Compound C has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 5.

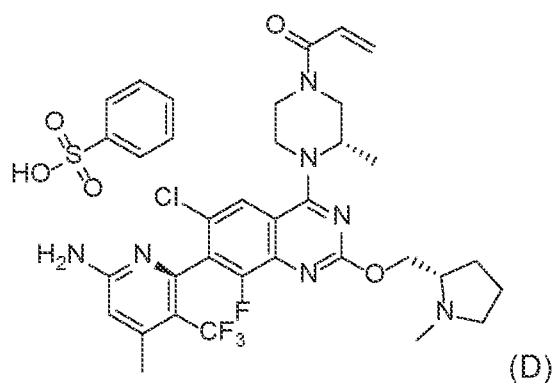
[0222] Embodiment 26. The crystal form of embodiment 24, wherein the crystal form of Compound C has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 5.

[0223] Embodiment 27. The crystal form of embodiment 24, wherein the crystal form of Compound C has an X-ray powder diffraction pattern substantially as shown in FIG 13.

[0224] Embodiment 28. The crystal form of embodiment 24, wherein the crystal form of Compound C has a TGA thermograph corresponding substantially to the representative TGA thermogram as depicted in FIG 14.

[0225] Embodiment 29. The crystal form of embodiment 24, wherein the crystal form of Compound C has a DSC thermograph corresponding substantially as depicted in FIG 14.

[0226] Embodiment 30. A crystal form comprising Compound D:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 7.3809, 10.7407, 14.654, 18.5979, 25.2558 ($\pm 0.1^\circ 2\theta$).

[0227] Embodiment 31. The crystal form of embodiment 30, wherein the crystal form of Compound D has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 6.

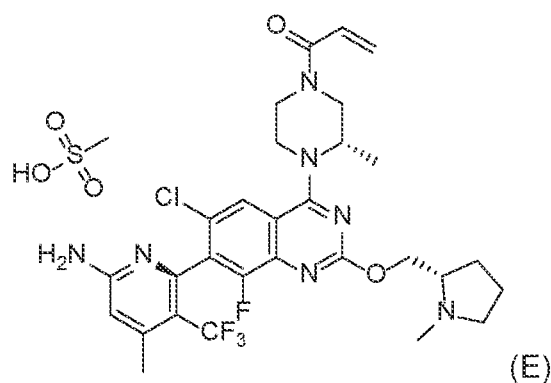
[0228] Embodiment 32. The crystal form of embodiment 30, wherein the crystal form of Compound D has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 6.

[0229] Embodiment 33. The crystal form of embodiment 30, wherein the crystal form of Compound D has an X-ray powder diffraction pattern substantially as shown in FIG 15.

[0230] Embodiment 34. The crystal form of embodiment 30, wherein the crystal form of Compound D has a TGA thermograph corresponding substantially to the representative TGA thermogram as depicted in FIG 16.

[0231] Embodiment 35. The crystal form of embodiment 30, wherein the crystal form of Compound D has a DSC thermograph corresponding substantially as depicted in FIG 16.

[0232] Embodiment 36. A crystal form comprising Compound E:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 5.9702, 6.2798, 12.4314, 18.9264, 20.6365 ($\pm 0.1^\circ 2\theta$).

[0233] Embodiment 37. The crystal form of embodiment 36, wherein the crystal form of Compound E has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 7.

[0234] Embodiment 38. The crystal form of embodiment 36, wherein the crystal form of Compound E has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 7.

[0235] Embodiment 39. The crystal form of embodiment 36, wherein the crystal form of Compound E has an X-ray powder diffraction pattern substantially as shown in FIG 17.

[0236] Embodiment 40. A pharmaceutical composition comprising the solid form of any one of claims 2-39 and at least one pharmaceutically acceptable excipient.

[0237] Embodiment 41. A pharmaceutical composition of embodiment 40, wherein said composition is formulated for oral administration.

[0238] Embodiment 42. A method of treating a cancer comprising a KRasG12C mutation in a patient having said cancer, the method comprising administering an effective amount of a compound of embodiment 1 or a crystal form of any one of embodiments 2-39.

[0239] Embodiment 43. A method for treating a cancer comprising a KRasG12C

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mutation in a patient having said cancer, the method comprising:

determining if the patient has the mutation; and

if the patient is determined to have the mutation, then administering to the patient an effective amount of a compound of claim 1 or a crystal form of any one of embodiments 2-39.

[0240] Embodiment 44. The method of embodiment 42 or 43, wherein the cancer comprises lung cancer, colorectal cancer, or pancreatic cancer.

[0241] Embodiment 45. The method of embodiment 42 or 43, wherein the cancer is lung cancer.

[0242] Embodiment 46. The method of embodiment 45, wherein the lung cancer is non-small cell lung cancer (NSCLC).

[0243] Embodiment 47. The method of embodiment 42 or 43, wherein the cancer is colorectal cancer.

[0244] Embodiment 48. The method of embodiment 42 or 43, wherein the cancer is pancreatic cancer.

[0245] Embodiment 49. A method for inhibiting proliferation of a cell population wherein the cell population comprises a KRasG12C mutation, the method comprising contacting the cell population with a compound of claim 1 or a crystal form of any one of embodiments 2-39.

[0246] Embodiment 50. A method for inhibiting tumor metastasis comprising administering to an individual in need thereof a therapeutically effective amount of the compound of embodiment 1 or a crystal form of any one of embodiments 2-39.

[0247] Embodiment 51. Use of a compound of embodiment 1 or a crystal form of any one of embodiments 2-39 in the manufacture of a medicament for treating a cancer.

[0248] Embodiment 52. The use of embodiment 51, wherein the cancer comprises lung cancer, colorectal cancer, or pancreatic cancer.

[0249] Embodiment 53. The compound of claim 1 or a crystal form of any one of embodiments 2-39 for use in a method of treating cancer.

[0250] Embodiment 54. The compound of embodiment 53, wherein the cancer comprises lung cancer, colorectal cancer, or pancreatic cancer.

Examples:

[0251] The following Examples are presented by way of illustration, not limitation.

Example 1:

[0252] Compound 1 was initially screened with 21 acids and four solvent systems for pharmaceutically acceptable salts and polymorphs. (Table 8). For each solvent of acetone, EtOAc, THF, and MeOH/H₂O (19:1, v/v), freebase and corresponding acid were mixed with a molar charge ratio of 1:1, and stirred at RT overnight. Due to the observation that all the samples were clear solution, all the samples were transferred to stirring at 5 °C overnight. The solids obtained at 5 °C were tested by XRPD. For the clear solutions, anti-solvent (n-heptane for acetone, THF and EtOAc; H₂O for MeOH/H₂O) was added. The mixtures were then stirred at 5 °C for 3 days. The solids obtained were characterized by XRPD, and the other samples were transferred to heating-cooling between 5 - 50 °C. The solids obtained were characterized by XRPD, and the clear solutions were transferred to slow evaporation at RT.

[0253] Table 8:

Solvent	Acetone	THF	EtOAc	MeOH/H ₂ O (19:1, v/v)
Blank	Freebase Form A ^s	Freebase Form A (Low crystallinity) ^s	Amorphous ^s	Gel ^s
H ₃ PO ₄	Amorphous ^s	Amorphous*	Amorphous*	Gel ^s
Acetic acid	Compound E Form A ^s	Compound E Form A ^s	Amorphous ^s	Gel ^s
Maleic acid	Amorphous ^s	Amorphous ^s	Amorphous ^s	Gel ^s
Fumaric acid ^{as}	Compound B Form A**	Low crystallinity*	Low crystallinity ^s	Gel ^s
	Compound B Form A ^s	Low crystallinity*	Low crystallinity ^s	Gel (EtOH)
Succinic acid	Amorphous ^s	Amorphous ^s	Amorphous ^s	Gel ^s
L-Malic acid	Amorphous ^s	Amorphous ^s	Amorphous ^s	Gel ^s

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Adipic acid	Compound A Form A [§]	Compound A Form A*	Adipate Type A [§]	Gel [§]
L-Tartaric acid	Amorphous [§]	Amorphous*	Acid*	Gel [§]
Hippuric acid	Amorphous [§]	Amorphous [§]	Amorphous [§]	Gel [§]
Citric acid	Amorphous [§]	Amorphous*	Acid**	Gel [§]
Glycolic acid	Amorphous [§]	Amorphous [§]	Amorphous [§]	Gel [§]
Malonic acid	Amorphous [§]	Amorphous [§]	Amorphous [§]	Gel [§]
Benzoic acid	Amorphous [§]	Amorphous [§]	Amorphous [§]	Gel [§]
Gentisic acid	Amorphous [§]	Amorphous [§]	Amorphous*	Gel [§]
Oxalic acid	Amorphous [§]	Amorphous [§]	Amorphous**	Gel [§]
R-mandelic acid	Amorphous [§]	Amorphous [§]	Amorphous [§]	Gel [§]
S-mandelic acid	Amorphous [§]	Amorphous [§]	Amorphous [§]	Gel [§]
p-Toluenesulfonic acid	Gel [§]	Amorphous [§]	Amorphous [§]	Gel [§]
Benzenesulfonic acid	Compound D Form A [§]	Amorphous [§]	Amorphous [§]	Gel [§]
Methylsulfonic acid	Compound E Form A [#]	Amorphous [§]	Amorphous [§]	Gel [§]
Ethylsulfonic acid	Amorphous [§]	Amorphous [§]	Compound C Form A [§]	Gel [§]

[§]: Obtained from slurry at 5 °C.

*: Anti-solvent of n-heptane was added and stirred at 5 °C.

** : Anti-solvent of n-heptane was added and stirred at 5 °C. Heating-cooling between 5 °C and 50 °C was performed.

: Limited solids were observed after stirring at 5 °C. During XRPD test, it was observed that the sample may tend to absorb moisture and turn to liquid. The suspension was transferred to -20 °C, but limited solids were still observed. Anti-solvent (n-heptane) was added and the sample was transferred to 5 °C. However, gel was obtained.

§: Anti-solvent was added and stirred at 5°C. Heating-cooling between 5°C and 50 °C was performed, followed by slow evaporation at RT.

§§: For fumaric acid, to try to obtain potential anhydrous/hydrous fumarate hits with good crystallinity, experiments in acetone, THF, and EtOAc were repeated, and one more experiment in EtOH was also set up. However, anhydrous/hydrous fumarate hit with better crystallinity was not obtained.

[0254] As summarized in Table 9, a total of six crystalline hits were obtained. The salt hits were characterized by XRPD, TGA, DSC, HPLC and NMR.

[0255] Table 9: Characterization summary of crystalline salt hits

Salt	Weight Loss in TGA (%)	Endotherm in DSC (°C , peak)	Purity (area%)	Stoichiometry (acid/base)
Compound A Form A	2.0	68.8, 169.1	98.30	1.06
Compound B Form A	0.7	53.8, 156.3	93.56	1.03
Compound C Form A	8.6	81.0, 149.2	95.18	1.04
Compound D Form A	7.7	75.8	96.23	1.03
Compound E Form A	NA	NA	NA	NA
Compound F Form A	9.0	65.5, 125.4	88.42	0.69

[0256] A diverse range of conditions was also explored by carrying out maturation at two different temperatures starting with the amorphous material from a diverse range of solvent/solvent mixtures.

[0257] Amorphous material (25 mg) in HPLC vials was treated with 5 vol (125 µl) of solvent and the samples were placed on a shaker at 50 °C or on a stirrer at 5 °C for 7 days with observations noted upon solvent addition. Any solids were filtered using a fritted filter and initially analysed by XRPD. Any solutions were treated with anti-solvent in increasing solvent:anti-solvent ratios until a maximum ratio of 1:5 had been reached at 25 °C. The samples were then cooled to 5 °C at 0.1 °C/min and held over the weekend. Any solutions from cooling were left to evaporate at RT with the caps removed and any turbid samples were filtered using a fritted filter and initially analysed by XRPD.

[0258] Maturation at both low and high temperature for 7 days starting with the amorphous material resulted in the majority of samples remaining as solutions. The solutions from 50 °C maturation were treated with anti-solvent which largely remained as clear solutions after addition of 1:5 solvent: anti-solvent ratio and cooling to 5 °C. The samples were subsequently evaporated which largely resulted in amorphous, Compound A Form A/D or gums. The solutions obtained from 5 °C maturation were kept at 5 °C for the duration of the project and remained as solutions.

[0259] Table 10: Results and observations from maturation at 5 °C for 7 days starting on amorphous.

Solvent	Upon addition at 5°C	After 5°C maturation	XRPD
Water	Clear Solution	Clear Solution	N/P
Methanol	Clear Solution	Clear Solution	N/P
Ethanol	Clear Solution	Clear Solution	N/P
2-propanol	Clear Solution	Suspension	Insufficient material
1-propanol	Clear Solution	Clear Solution	N/P
Acetone	Clear Solution	Clear Solution	N/P
Ethyl Acetate	Clear Solution	Suspension	Insufficient material
Acetonitrile	Clear Solution	Turbid	Insufficient material
Toluene	Clear Solution	Solution +fine particles	N/P
Isopropyl Acetate	Clear Solution	Solution +fine particles	N/P
TBME	Turbid/ Hazy	Hazy Solution	N/P
2-butanone (MEK)	Clear Solution	Clear Solution	N/P
THF	Clear Solution	Clear Solution	N/P
DMSO	Clear Solution	Clear Solution	N/P
NMP	Clear Solution	Clear Solution	N/P
Diethyl ether	Hazy Solution	Turbid/ Hazy	Amorphous
MIBK	Clear Solution	Turbid/Hazy	Insufficient material
DCM	Clear Solution	Clear Solution	N/P
Xylene	Partially Dissolved	Solution + large particles	N/P
DMI	Clear Solution	Clear Solution	N/P
Cumene	Partially Dissolved	Solution + large particles	N/P
Sulfolane	Clear Solution	Clear Solution	N/P
DMPU	Clear Solution	Clear Solution	N/P
2-Methyl THF	Clear Solution	Clear Solution	N/P
Heptane	Turbid/Hazy	Hazy Solution	N/P
1,4-dioxane	Clear Solution	Clear Solution	N/P
DMF	Clear Solution	Clear Solution	N/P
Nitromethane	Clear Solution	Solution + fine particles	N/P
Chloroform	Clear Solution	Clear Solution	N/P
1-butanol	Clear Solution	Clear Solution	N/P
1-Methoxy-2-propanol	Clear Solution	Clear Solution	N/P
3-Methyl-1-butanol	Clear Solution	Clear Solution	N/P
Anisole	Clear Solution	Clear Solution	N/P
t-butanol/water (1:1)	Clear Solution	Clear Solution	N/P
10% water/ methanol	Clear Solution	Clear Solution	N/P
5% water/EtOH	Clear Solution	Clear Solution	N/P
20% water/ methanol	Clear Solution	Clear Solution	N/P
10% water/EtOH	Clear Solution	Clear Solution	N/P
10% water/IPA	Clear Solution	Clear Solution	N/P
10% water/ACN	Clear Solution	Clear Solution	N/P
10% water/Acetone	Clear Solution	Clear Solution	N/P
PEG 400	Clear Solution	Clear Solution	N/P
Tetralin	Partially Dissolved	Gummy Solution	N/P

Solvent	Upon addition at 5°C	After 5°C maturation	XRPD
Cyclohexane	Thin Suspension	Very Thin Suspension	Amorphous
Acetic Acid	Clear Solution	Clear Solution	N/P
Hexane	Thin Suspension	Turbid	Insufficient material
Ethyleneglycol	Clear Solution	Clear Solution	N/P
Pyridine	Clear Solution	Clear Solution	N/P

[0260] Table 11: Results and observations from anti-solvent addition from 50 °C maturation

Solvent	Antisolvent	Antisolvent added at RT (solvent:anti-solvent ratio)			Upon cooling to 5°C	XRPD
		1:1	1:3	1:5		
Water	3-Methyl-1-butanol	✓	+/-	+/-	Cloudy Solution	Very little solid, poor signal, Form A/D
Methanol	Xylene	✓	✓	✓	Frozen	Gummy, amorphous
Ethanol	Diethyl ether	✓	✓	✓	Clear Solution	Gummy, very poor signal, Form A/D
2-propanol	Diethyl ether	✓	✓	✓	Clear Solution	Gummy
1-propanol	Diethyl ether	✓	✓	✓	Clear Solution	Gummy, partial match Form A/D, extra peaks
Acetone	Diethyl ether	✓	✓	✓	Cloudy Solution	Very Poorly Crystalline
2-butanone (MEK)	Diethyl ether	✓	✓	✓	Clear Solution	Form A/D
THF	Diethyl ether	✓	✓	✓	Clear Solution	Form A/D
DMSO	Ethyl Acetate	✓	✓	✓	Clear Solution	Amorphous
NMP	Ethyl Acetate	✓	✓	✓	Clear Solution	Amorphous
DCM	Ethyl Acetate	x			Turbid/Hazy	Poorly Crystalline, Form A/D
DMI	Ethyl Acetate	✓	✓	+/-	Clear Solution	Amorphous
Sulfolane	Ethyl Acetate	✓	✓	✓	Clear Solution	Clear Solution - after vac drying
DMPU	Ethyl Acetate	✓	✓	✓	Clear Solution	Amorphous
2-Methyl THF	Diethyl ether	✓	+/-	+/-	Partially Cloudy Solution	Form A/D
1,4-dioxane	Diethyl ether	✓	✓	X	Gummy solution	Very little solid, insufficient material
DMF	Diethyl ether	✓	✓	✓	Clear Solution	Very little solid, insufficient material

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Solvent	Antisolvent	Antisolvent added at RT (solvent:anti-solvent ratio)			Upon cooling to 5°C	XRPD
		1:1	1:3	1:5		
Nitromethane	Diethyl ether	✓	✓	+/-	Gummy solution	Gummy, poor signal, Form A/D
Chloroform	Diethyl ether	X			Turbid	Poorly Crystalline, Form A/D
1-butanol	Diethyl ether	✓	✓	✓	Clear Solution	Gummy, amorphous
1-Methoxy-2-propanol	IPA	✓	✓	✓	Clear Solution	Gummy
3-Methyl-1-butanol	IPA	✓	✓	✓	Clear Solution	Gummy
t-butanol/water (1:1)	IPA	✓	✓	✓	Clear Solution	Gummy
10% water/methanol	IPA	✓	✓	✓	Clear Solution	Gummy
5% water/EtOH	Xylene	✓	✓	✓	Frozen	Form A/D
20% water/methanol	IPA	✓	✓	✓	Clear Solution	Very little solid, insufficient material
10% water/EtOH	Xylene	✓	+/-	+/-	Frozen	Very little solid, amorphous
10% water/IPA	Xylene	✓	✓	✓	Frozen	Very Poorly Crystalline
10% water/ACN	Xylene	✓	✓	✓	Frozen	Gummy, amorphous
10% water/Acetone	Xylene	✓	✓	✓	Partially Frozen	Oily Residue
PEG 400	Diethyl ether	✓	+/-	+/-	Biphasic Layers	Clear gel like solution
Acetic Acid	Diethyl ether	✓	✓	✓	Clear Solution	Very little solid, insufficient material
Ethyleneglycol	Diethyl ether	✓	✓	✓	Clear Solution	Amorphous
Pyridine	Diethyl ether	✓	✓	✓	Clear Solution	Gummy, amorphous

Example 2:

[0261] A total of 62 polymorph screening experiments were performed for freebase through different crystallization methods. Compared with the starting material, better crystallinity was observed for the sample obtained by slow evaporation in ACN with adipic acid, and named as Compound A Form A.

[0262] Single crystal data of Compound A Form A was obtained as shown in FIG. 4. According to the single crystal analysis result, Compound A Form A is anhydrous in the lattice, but might contain some surface water.

[0263] Compound A Form A was prepared by slurry freebase and adipic acid in

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EtOAc at RT for 48 hrs and then transferred to 5 °C for 24 hrs to increase the yield. The wet sample was vacuum dried at RT for 3 hrs. No form change was observed before and after drying. A solid was obtained with a yield of 62.2%. As per TGA and DSC, the sample showed a weight loss of 0.6% up to 100 °C, and DSC curve showed two endotherms at 56.0 °C and 168.7 °C (peak temperature). Based on the integrals in NMR spectrum, the stoichiometric ratio of adipic acid:freebase was determined to be 1.06. A peak of EtOAc was observed. Around 0.07 molar EtOAc (0.8 wt%) was detected, which was consistent with TGA weight loss.

[0264] A disordered version of Compound A Form A, designated as Compound A Form C, was identified via single crystal XRD. See FIG. 7. Further description of this form and its interchangeability with Compound A Form A and Compound A Form D is below.

[0265] Compound A Form A was exposed to ambient humidity levels $\geq 40\%$, whereupon Compound A Form D was formed. Compound A Form D is a hemihydrate form of Compound A. For reversibility, Compound A Form D was dehydrated, whereupon Compound A Form D formed Compound A Form C.

[0266] Another form of Compound A was obtained and designated as Form B. Compound A Form B was obtained from anti-solvent addition in DCM/toluene, followed by transferring to 5 °C. No form change was observed after air drying at RT overnight. A weight loss of 2.2% up to 100 °C was observed in TGA, and two broad endotherms at 55.7 °C and 109.6 °C (peak), and one small endotherm at 158.8 °C (peak) were observed in DSC. Based on the integrals, the stoichiometric ratio of adipic acid:freebase was determined to be 1.65.

[0267] VT-XRPD was performed on Compound A Form B for form identification. After heating to 90 °C under N₂ protection, no form change was observed, indicating the TGA weight loss was caused by surface water. Thus, Compound A Form B was speculated to be an anhydrate. No peak of DCM was observed. To further confirm the stoichiometric ratio, three batches of Compound A Form B were re-prepared by anti-solvent addition in DCM/toluene, followed by transferring to 5 °C. Based on the NMR results, the stoichiometric ratio of adipic acid:freebase for Compound A Form B varied from 1.5 to 1.8.

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[0268] Slurry Competition between Compound A Form A and B. Slurry competition experiments between Compound A Form A and B were set up to determine the relationship between these two anhydrates. The saturated solution of Compound A Form A was obtained via slurry of Compound A Form A at target temperature overnight. A mixture of Compound A Form A+B was added into ~0.7 mL of saturated solution of Compound A Form A, and suspensions were observed. The suspensions were stirred at a speed of 750 rpm and tested by XRPD in transmission mode. As summarized in Table 12, in toluene, after a mixture of Compound A Form A+B was stirred at 5 °C, a mixture of Compound A Form A+B was observed after 5 days. After transferring the sample to RT, Compound A Form A was observed after slurry at RT for 18 days (the peak at ~18.1° was possibly caused by stirrer). In IPA, the mixture turned to Compound A Form A at both 5 °C and -20 °C.

[0269] Table 12: Slurry competition between adipate Type A and B

Starting Form	Temperature (°C)	Solvent	Result
Compound A Form A+B	5 to RT	Toluene	Compound A Form A
	5	IPA	Compound A Form A
	-20	IPA	Compound A Form A

[0270] Two adipate forms of anhydrates Compound A Form A and Compound A Form B were observed from polymorph screening of adipate with Compound 1. It should be noted that although Compound A Form A was a mono-salt, the stoichiometry of acid/freebase for different Compound A Form B batches varied from 1.5 to 2.1, suggesting Compound A Form B might be a sesqui- or bis-salt. Slurry competition experiments between Compound A Form A and B in toluene (RT) and IPA (5 °C and -20 °C) resulted in Compound A Form A.

[0271] Reactive Crystallisation with Adipic Acid. Additional steps were investigated to identify whether additional forms of the adipate salt exist when starting from the free form of Compound 1.

[0272] Compound 1 (30 mg) was weighed into HPLC vials with stirrers and dissolved at 50 °C. Adipic acid solution in THF (1M, 1 or 1.5 eq.) was added to each vial and if a solid formed, then an aliquot was taken. The solutions or suspensions were cooled

to 40 °C at 0.5 °C/min when they were seeded with a very small amount of Compound A Form A. The samples were then further cooled to 5 °C at 0.1 °C/min overnight and if a precipitate formed, it was filtered and analysed by XRPD.

[0273] Reactive crystallisation with adipic acid largely resulted in Compound A Form A/D or sticky material which was amorphous by XRPD. The results and observations from reactive crystallisation with adipic acid are summarised below in Table 13.

[0274] Table 13:

Solvent	Acid	Solvent vol.	Obs. After acid addition	Obs. After cooling to 5 °C	XRPD
Water	1 equivalent adipic acid	40*	Became clear	Clear Solution	N/P
10% water/IPA		30	Clear Solution	Clear Solution	Sticky material, amorphous
EtOH		20	Clear Solution	Clear Solution	Form A/D
Ethyl acetate		30	Light Precipitate	White Precipitate	Form A/D
Acetone		20	Clear Solution	Clear Solution	Form A/D
Acetonitrile		30	Clear Solution	White Precipitate	Form A/D
MEK		20	Clear Solution	Clear Solution	Form A/D
THF		5	Clear Solution	White Precipitate, fine	Form A/D
TBME		40*	White Precipitate	White Precipitate	Form A/D (both & A)
Dioxane		10	Clear Solution	Clear Solution	Sticky material, amorphous
Anisole		20	Gel-like suspension at 40 °C	White Precipitate	Form A/D (both & A)
Acetic acid		5	Clear Solution	Clear Solution	Sticky material, N/P
Water	1.5 equivalents adipic acid	40*	Partially Clear	Clear Solution	N/P
10% water/IPA		30	Clear Solution	Clear Solution	Sticky material, amorphous
EtOH		20	Clear Solution	Clear Solution	Form A/D
Ethyl acetate		30	White Precipitate, at 40 °C	White Precipitate	Form A/D (both & A)
Acetone		20	Clear Solution	Clear Solution	Form A/D
Acetonitrile		30	Clear Solution	White Precipitate	Form A/D
MEK		20	Clear Solution	White Precipitate	Form A/D
THF		5	Clear Solution	White Precipitate, fine	Form A/D
TBME		40*	White Precipitate	White Precipitate	Form A/D (both & A)
Dioxane		10	Clear Solution	Clear Solution	Sticky material, amorphous
Anisole		20	Gel-like suspension at 40 °C	White Precipitate	Form A/D (both & A)
Acetic acid		5	Clear Solution	Clear Solution	Sticky material, amorphous

Example 3:

[0275] Instruments and methods

[0276] XRPD. For XRPD analysis, PANalytical X-ray powder diffractometers in reflection mode were used. The XRPD parameters are listed in the table below.

Parameters	Reflection Mode		Transmission Mode
Model	Empyrean	X' Pert ³	X' Pert ³
X-Ray wavelength	Cu, $\text{K}\alpha$,		
	K α 1 (Å): 1.540598		
	K α 2 (Å): 1.544426		
X-Ray tube setting	K α 2/K α 1 intensity ratio: 0.50		
	45 kV, 40 mA		
Scan mode	Continuous		
Scan range ($^{\circ}$ 2 Theta)	3 $^{\circ}$ -40 $^{\circ}$		
Divergence slit	Automatic	1/8 $^{\circ}$	1/2 $^{\circ}$
Scan step time (s)	17.8	46.7	31.6
Step size ($^{\circ}$ 2 Theta)	0.0167	0.0263	0.013
Test Time	5 min 30 s	5 min 4 s	~7 min

[0277] XRPD was also collected as follows. XRPD diffractograms were collected on a Bruker D8 diffractometer using Cu K α radiation (40 kV, 40 mA) and a θ -2 θ goniometer fitted with a Ge monochromator. The incident beam passes through a 2.0 mm divergence slit followed by a 0.2 mm anti-scatter slit and knife edge. The diffracted beam passes through an 8.0 mm receiving slit with 2.5 $^{\circ}$ Soller slits followed by the Lynxeye Detector. The software used for data collection and analysis was *DiffraC Plus* XRD Commander and *DiffraC Plus* EVA respectively.

[0278] Samples were run under ambient conditions as flat plate specimens using powder as received. The sample was prepared on a polished, zero-background (510) silicon wafer by gently pressing onto the flat surface or packed into a cut cavity. The sample was rotated in its own plane.

[0279] Single Crystal X-Ray Diffraction (SCXRD). Data were collected on a Rigaku Synergy Custom MM007-HF using a HyPix-6000 HPAD detector (The Woodlands, TX, USA). Data were collected in a nitrogen gas stream at 90 K using omega scans. Data were integrated and scaled using CrysAlisPro (Rigaku Oxford Diffraction, 2021).

WinGX was used for the solution and refinement of the crystal structures. Solution by iterative methods (SHELXT-2014) produced a complete heavy-atom phasing model. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2018). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2018.

[0280] TGA/DSC. TGA data were collected using a TA Q5000 TGA or TA Discovery TGA5500 from TA Instruments and DSC was performed using a TA Discovery DSC2500 from TA Instruments.

[0281] General Parameters for TGA and DSC testing

Parameters	TGA	DSC
Method	Ramp	Ramp
Sample pan	Aluminum, open	Aluminum, crimped
Temperature	RT - desired temperature	25 °C - desired temperature
Heating rate	10 °C/min	10 °C/min
Purge gas	N ₂	N ₂

[0282] Solution NMR. Solution NMR was collected on Bruker 400M NMR Spectrometer using DMSO-d₆.

Example 4 – Compound A - Amorphous

[0283] Compound A (20 mg) was weighed into HPLC vials and solvent was added in 10 vol aliquots (200 µl) at RT on a stirrer plate until sample dissolution or until a maximum of 50 vol had been added and observations were made between additions. The solutions that were obtained were frozen in a dry ice acetone bath before being placed on the freeze dryer under vacuum.

[0284] Amorphous material of Compound A (mono adipate salt) was successfully prepared through freeze drying. Characterisation of the initial batch of samples were all shown to be amorphous by XRPD and the NMR's contained ca. 1.1 eq. adipic acid with varying quantities of residual solvent, except for acetonitrile/water (1:1) which contained no residual solvent. The purities were low, between 85 – 89 %, except for 1,4-dioxane which had a very low purity of 57.5 %. Static storage at 25 °C / 97 % RH and 40 °C / 75 % RH after 7 days were all shown to have deliquesced.

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[0285] Liquid asset grinding (LAG) screens: Amorphous material (25 mg) in HPLC vials was treated with two ball bearings and solvent (5 μ l, 0.2 vol) and milled on a Fritsch planetary mill fitted with an Automaxion adapter for 2 hours at 500 rpm. Samples that went into solution upon solvent addition were allowed to slowly evaporate with a needle inserted into the cap. The resultant solids from LAG were initially analysed by XRPD.

Example 4 – Compound A Form A

[0286] Compound A Form A could be obtained in different solvent systems of acetone, THF, and EtOAc from screening. Single crystal data of Compound A Form A shows that Compound A Form A is anhydrous.

[0287] Compound A Form A was obtained from THF system and the sample was air dried at RT. No form change was observed before and after drying. A weight loss of 2.0% up to 100 °C was observed in TGA and DSC result showed two endotherms at 68.8 °C and 169.1 °C (peak temperature). Based on the integrals, the stoichiometric ratio of adipic acid:freebase was determined to be 1.06. Peak of THF was observed. Around 0.17 molar THF (1.9 wt%) was detected, which was consistent with TGA weight loss. The purity of Compound A Form A was 98.30 area%. After heating adipate Type A to 100 °C under N₂ protection, cooled down and exposed to ambient conditions, very slight differences were observed before and after heating. Peak of THF was observed in the heated sample. Around 0.15 molar THF (1.7 wt%) was still detected, suggesting the organic solvent might be occluded in the crystal form.

[0288] VH-XRPD Analysis of Compound A. The VH-XRPD analysis performed on Compound A shows small changes between 44 – 46 % RH which remain up until 80 % RH. FIG. 19. The sample then reverts to the original form upon reaching 30 % RH. The sample appears to be a channel-like hydrate system which can exist with a range of occupancies of water from 0 to around 1 eq.

[0289] Single Crystal XRD. X-ray quality crystals were grown from a saturated isopropanol solution followed by the slow vapor diffusion of heptane to deposit the crystal diffracted. A colorless prism 0.100 x 0.100 x 0.100 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 90(2) K using phi and omega scans. Crystal-to-detector distance was 40 mm and exposure

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time was 0.2 seconds per frame using a scan width of 1.0° . Data collection was 99.9% complete to 67.000° in θ . A total of 40537 reflections were collected covering the indices, $-10 \leq h \leq 10$, $-15 \leq k \leq 15$, $-22 \leq l \leq 22$. 12985 reflections were found to be symmetry independent, with an R_{int} of 0.0345. Indexing and unit cell refinement indicated a primitive, triclinic lattice. The space group was found to be P 1 (No. 1). The data were integrated and scaled using CrysAlisPro 1.171.40.51a. Solution by iterative methods (SHELXT-2014) produced a complete heavy-atom phasing model. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2018). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2018. Absolute stereochemistry was unambiguously determined from the diffraction data.

[0290] Table 14: Crystal data and structure refinement for Compound A Form A

Empirical formula	C ₃₅ H ₄₂ Cl F ₄ N ₇ O ₆
Formula weight	768.20
Temperature	90(2) K
Wavelength	1.54184 Å
Crystal system	Triclinic
Space group	P 1
Unit cell dimensions	$a = 8.6289(3) \text{ Å}$ $\alpha = 90.728(2)^\circ$ $b = 12.4836(4) \text{ Å}$ $\beta = 103.055(2)^\circ$ $c = 18.3690(5) \text{ Å}$ $\gamma = 110.167(3)^\circ$
Volume	1800.49(10) Å ³
Z	2
Density (calculated)	1.417 Mg/m ³
Absorption coefficient	1.604 mm ⁻¹
F(000)	804
Crystal size	0.100 x 0.100 x 0.100 mm ³
Theta range for data collection	2.481 to 75.148°.
Index ranges	$-10 \leq h \leq 10$, $-15 \leq k \leq 15$, $-22 \leq l \leq 22$
Reflections collected	40537
Independent reflections	12985 [$R_{\text{int}} = 0.0345$]
Completeness to $\theta = 67.000^\circ$	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.84789

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	12985 / 3 / 974
Goodness-of-fit on F^2	1.089
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0471$, $wR2 = 0.1296$
R indices (all data)	$R1 = 0.0487$, $wR2 = 0.1310$
Absolute structure parameter	0.008(14)
Extinction coefficient	0.0016(4)
Largest diff. peak and hole	0.364 and -0.309 e.Å ⁻³

[0291] Table 15: Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound A Form A. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
C(1)	561(5)	-64(3)	3149(2)	26(1)
C(2)	1896(5)	1047(3)	3157(2)	26(1)
C(3)	2296(5)	2035(3)	3650(2)	28(1)
C(4)	3565(5)	3031(3)	3580(2)	29(1)
C(5)	4519(5)	3096(3)	3048(2)	26(1)
C(6)	4192(5)	2109(3)	2613(2)	26(1)
C(7)	2903(5)	1058(3)	2650(2)	25(1)
C(8)	1564(5)	-816(3)	2294(2)	26(1)
C(9)	-1348(6)	632(3)	3713(2)	33(1)
C(10)	-3268(6)	209(4)	3364(3)	36(1)
C(11)	-3402(6)	-1761(4)	3361(2)	38(1)
C(12)	-1516(6)	-1395(3)	3739(2)	33(1)
C(13)	-1197(6)	-1450(4)	4583(3)	40(1)
C(14)	-5275(6)	-1084(4)	3994(2)	37(1)
C(15)	-5889(6)	-2210(4)	4304(3)	43(1)
C(16)	-7177(8)	-2464(5)	4619(3)	54(1)
C(17)	5723(5)	4189(3)	2868(2)	28(1)
C(18)	7476(5)	4559(3)	3083(2)	29(1)
C(19)	8434(5)	5557(3)	2790(2)	30(1)
C(20)	7546(6)	6130(3)	2338(2)	30(1)
C(21)	5756(5)	5709(3)	2144(2)	29(1)
C(22)	8421(6)	3966(3)	3618(2)	33(1)
C(23)	10347(6)	5972(4)	2929(3)	40(1)
C(24)	2289(6)	-1692(3)	1334(2)	32(1)

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C(25)	1414(5)	-2762(3)	784(2)	31(1)
C(26)	2374(6)	-2759(4)	182(2)	38(1)
C(27)	1654(12)	-2118(8)	-390(5)	33(3)
C(27A)	923(13)	-3304(10)	-570(5)	47(3)
C(28)	-294(7)	-2696(5)	-440(3)	43(1)
C(29)	-1708(6)	-3895(4)	442(3)	41(1)
C(30)	-2114(6)	-1128(3)	927(2)	32(1)
C(31)	-2463(6)	-124(4)	1232(3)	36(1)
C(32)	-1359(6)	1073(4)	1105(2)	34(1)
C(33)	-1876(6)	1973(4)	1450(3)	37(1)
C(34)	-1243(6)	3160(4)	1183(3)	40(1)
C(35)	627(6)	3838(3)	1411(2)	33(1)
C(36)	7653(5)	10613(3)	6751(2)	28(1)
C(37)	7881(5)	9510(3)	6775(2)	28(1)
C(38)	6880(6)	8511(4)	6285(2)	31(1)
C(39)	7240(6)	7533(3)	6375(2)	31(1)
C(40)	8586(5)	7468(3)	6943(2)	30(1)
C(41)	9591(5)	8461(3)	7394(2)	30(1)
C(42)	9308(5)	9499(3)	7325(2)	28(1)
C(43)	10207(5)	11397(3)	7608(2)	29(1)
C(44)	4522(6)	9900(4)	6212(3)	35(1)
C(45)	3459(7)	10323(4)	6626(3)	41(1)
C(46)	5218(7)	12289(4)	6592(3)	41(1)
C(47)	6296(6)	11924(4)	6157(3)	37(1)
C(48)	5729(7)	11975(4)	5316(3)	44(1)
C(49)	2036(7)	11560(4)	5994(3)	40(1)
C(50)	2168(7)	12681(5)	5690(3)	47(1)
C(51)	787(9)	12880(6)	5368(3)	55(1)
C(52)	8898(6)	6387(3)	7130(2)	32(1)
C(53)	10055(6)	6015(3)	6897(2)	34(1)
C(54)	10344(7)	5033(4)	7197(2)	38(1)
C(55)	9368(6)	4466(3)	7672(2)	38(1)
C(56)	8175(6)	4875(4)	7867(2)	35(1)
C(57)	11028(7)	6603(4)	6348(3)	40(1)
C(58)	11690(8)	4619(4)	7040(3)	53(1)
C(59)	12759(6)	12364(3)	8545(2)	33(1)
C(60)	12137(6)	12133(4)	9265(2)	38(1)

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C(61)	11813(7)	13135(5)	9591(3)	50(1)
C(62)	13569(7)	13900(4)	10070(3)	49(1)
C(63)	14632(7)	13137(5)	10203(3)	51(1)
C(64)	12720(8)	11266(5)	10444(3)	51(1)
C(65)	16230(7)	11281(5)	9132(3)	48(1)
C(66)	17137(8)	10602(6)	8843(4)	61(2)
C(67)	16592(8)	9320(6)	8949(3)	60(2)
C(68)	14934(7)	8601(5)	8435(3)	53(1)
C(69)	14320(9)	7353(6)	8609(4)	63(2)
C(70)	15286(7)	6618(5)	8437(3)	51(1)
N(1)	2756(4)	125(3)	2203(2)	26(1)
N(2)	470(4)	-973(3)	2731(2)	26(1)
N(3)	-621(5)	-228(3)	3558(2)	32(1)
N(4)	-4105(5)	-927(3)	3582(2)	36(1)
N(5)	4861(4)	4735(3)	2399(2)	28(1)
N(6)	4866(5)	6245(3)	1694(2)	34(1)
N(7)	-347(5)	-2823(3)	360(2)	33(1)
N(8)	10482(5)	10454(3)	7766(2)	31(1)
N(9)	8879(5)	11544(3)	7140(2)	30(1)
N(10)	6246(5)	10756(3)	6320(2)	33(1)
N(11)	3487(5)	11455(3)	6421(2)	39(1)
N(12)	13474(5)	11960(3)	9876(2)	39(1)
N(13)	7970(5)	5833(3)	7604(2)	33(1)
N(14)	7211(6)	4359(3)	8331(2)	44(1)
O(1)	-5807(5)	-325(3)	4122(2)	46(1)
O(2)	1324(4)	-1797(2)	1889(2)	30(1)
O(3)	-3160(5)	-2117(3)	930(2)	44(1)
O(4)	-798(4)	-932(3)	691(2)	39(1)
O(5)	1267(4)	4690(3)	1112(2)	44(1)
O(6)	1515(4)	3481(3)	1963(2)	45(1)
O(7)	668(5)	10771(3)	5860(2)	48(1)
O(8)	11360(4)	12411(2)	7968(2)	34(1)
O(9)	16640(6)	12316(4)	9040(3)	68(1)
O(10)	15093(5)	10760(3)	9474(2)	47(1)
O(11)	15259(5)	5750(3)	8737(2)	52(1)
O(12)	16106(6)	6985(4)	7918(2)	60(1)
F(1)	5128(3)	2137(2)	2117(1)	34(1)

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F(2)	7403(3)	3077(2)	3890(2)	46(1)
F(3)	9380(4)	3549(2)	3302(2)	48(1)
F(4)	9486(4)	4674(2)	4213(2)	45(1)
F(5)	10905(3)	8438(2)	7938(1)	39(1)
F(6)	10771(5)	5868(3)	5752(2)	64(1)
F(7)	12714(4)	7034(3)	6630(2)	58(1)
F(8)	10602(4)	7471(3)	6067(2)	53(1)
Cl(1)	4020(1)	4261(1)	4170(1)	37(1)
Cl(2)	5988(2)	6287(1)	5777(1)	42(1)

[0292] Table 16: Bond lengths [Å] and angles [°] for Compound A Form A

C(1)-N(2)	1.332(5)	C(14)-C(15)	1.492(7)
C(1)-N(3)	1.361(5)	C(15)-C(16)	1.312(7)
C(1)-C(2)	1.463(5)	C(15)-H(15)	0.9500
C(2)-C(7)	1.408(5)	C(16)-H(16A)	0.9500
C(2)-C(3)	1.413(5)	C(16)-H(16B)	0.9500
C(3)-C(4)	1.374(5)	C(17)-N(5)	1.354(5)
C(3)-H(3)	0.9500	C(17)-C(18)	1.378(6)
C(4)-C(5)	1.400(6)	C(18)-C(19)	1.425(5)
C(4)-Cl(1)	1.742(4)	C(18)-C(22)	1.500(5)
C(5)-C(6)	1.367(5)	C(19)-C(20)	1.377(6)
C(5)-C(17)	1.497(5)	C(19)-C(23)	1.508(6)
C(6)-F(1)	1.341(4)	C(20)-C(21)	1.405(6)
C(6)-C(7)	1.412(5)	C(20)-H(20)	0.9500
C(7)-N(1)	1.368(5)	C(21)-N(6)	1.343(5)
C(8)-N(1)	1.309(5)	C(21)-N(5)	1.351(5)
C(8)-N(2)	1.340(5)	C(22)-F(4)	1.335(5)
C(8)-O(2)	1.352(4)	C(22)-F(2)	1.340(5)
C(9)-N(3)	1.470(5)	C(22)-F(3)	1.342(5)
C(9)-C(10)	1.531(7)	C(23)-H(23A)	0.9800
C(9)-H(9A)	0.9900	C(23)-H(23B)	0.9800
C(9)-H(9B)	0.9900	C(23)-H(23C)	0.9800
C(10)-N(4)	1.460(5)	C(24)-O(2)	1.436(5)

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C(10)-H(10A)	0.9900	C(24)-C(25)	1.517(5)
C(10)-H(10B)	0.9900	C(24)-H(24A)	0.9900
C(11)-N(4)	1.468(6)	C(24)-H(24B)	0.9900
C(11)-C(12)	1.520(7)	C(25)-N(7)	1.518(6)
C(11)-H(11A)	0.9900	C(25)-C(26)	1.524(6)
C(11)-H(11B)	0.9900	C(25)-H(25)	1.0000
C(12)-N(3)	1.477(5)	C(26)-C(27)	1.483(9)
C(12)-C(13)	1.518(6)	C(26)-C(27A)	1.595(10)
C(12)-H(12)	1.0000	C(26)-H(26A)	0.9900
C(13)-H(13A)	0.9800	C(26)-H(26B)	0.9900
C(13)-H(13B)	0.9800	C(27)-C(28)	1.564(11)
C(13)-H(13C)	0.9800	C(27)-H(27A)	0.9900
C(14)-O(1)	1.227(6)	C(27)-H(27B)	0.9900
C(14)-N(4)	1.357(6)	C(27A)-C(28)	1.547(11)
C(27A)-H(27C)	0.9900	C(40)-C(52)	1.494(5)
C(27A)-H(27D)	0.9900	C(41)-F(5)	1.340(5)
C(28)-N(7)	1.489(5)	C(41)-C(42)	1.401(5)
C(28)-H(28A)	0.9900	C(42)-N(8)	1.369(5)
C(28)-H(28B)	0.9900	C(43)-N(8)	1.302(5)
C(29)-N(7)	1.482(6)	C(43)-N(9)	1.337(5)
C(29)-H(29A)	0.9800	C(43)-O(8)	1.358(5)
C(29)-H(29B)	0.9800	C(44)-N(10)	1.469(6)
C(29)-H(29C)	0.9800	C(44)-C(45)	1.528(6)
C(30)-O(4)	1.253(5)	C(44)-H(44A)	0.9900
C(30)-O(3)	1.254(5)	C(44)-H(44B)	0.9900
C(30)-C(31)	1.516(6)	C(45)-N(11)	1.461(6)
C(31)-C(32)	1.524(6)	C(45)-H(45A)	0.9900
C(31)-H(31A)	0.9900	C(45)-H(45B)	0.9900
C(31)-H(31B)	0.9900	C(46)-N(11)	1.457(7)
C(32)-C(33)	1.523(6)	C(46)-C(47)	1.526(7)

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C(32)-H(32A)	0.9900	C(46)-H(46A)	0.9900
C(32)-H(32B)	0.9900	C(46)-H(46B)	0.9900
C(33)-C(34)	1.522(6)	C(47)-N(10)	1.481(5)
C(33)-H(33A)	0.9900	C(47)-C(48)	1.520(6)
C(33)-H(33B)	0.9900	C(47)-H(47)	1.0000
C(34)-C(35)	1.496(6)	C(48)-H(48A)	0.9800
C(34)-H(34A)	0.9900	C(48)-H(48B)	0.9800
C(34)-H(34B)	0.9900	C(48)-H(48C)	0.9800
C(35)-O(5)	1.217(5)	C(49)-O(7)	1.218(6)
C(35)-O(6)	1.308(5)	C(49)-N(11)	1.368(6)
C(36)-N(9)	1.331(5)	C(49)-C(50)	1.491(7)
C(36)-N(10)	1.360(5)	C(50)-C(51)	1.312(8)
C(36)-C(37)	1.455(5)	C(50)-H(50)	0.9500
C(37)-C(42)	1.410(6)	C(51)-H(51A)	0.9500
C(37)-C(38)	1.410(6)	C(51)-H(51B)	0.9500
C(38)-C(39)	1.362(6)	C(52)-N(13)	1.350(5)
C(38)-H(38)	0.9500	C(52)-C(53)	1.380(6)
C(39)-C(40)	1.402(6)	C(53)-C(54)	1.427(6)
C(39)-Cl(2)	1.748(4)	C(53)-C(57)	1.495(6)
C(40)-C(41)	1.372(6)	C(54)-C(55)	1.381(6)
C(54)-C(58)	1.504(7)	C(66)-H(66B)	0.9900
C(55)-C(56)	1.405(7)	C(67)-C(68)	1.492(9)
C(55)-H(55)	0.9500	C(67)-H(67A)	0.9900
C(56)-N(14)	1.339(6)	C(67)-H(67B)	0.9900
C(56)-N(13)	1.349(6)	C(68)-C(69)	1.527(9)
C(57)-F(8)	1.333(5)	C(68)-H(68A)	0.9900
C(57)-F(7)	1.337(6)	C(68)-H(68B)	0.9900
C(57)-F(6)	1.347(5)	C(69)-C(70)	1.507(8)
C(58)-H(58A)	0.9800	C(69)-H(69A)	0.9900
C(58)-H(58B)	0.9800	C(69)-H(69B)	0.9900

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C(58)-H(58C)	0.9800	C(70)-O(11)	1.218(7)
C(59)-O(8)	1.434(5)	C(70)-O(12)	1.308(6)
C(59)-C(60)	1.530(6)	N(6)-H(6A)	0.8800
C(59)-H(59A)	0.9900	N(6)-H(6B)	0.8800
C(59)-H(59B)	0.9900	N(7)-H(7)	1.0000
C(60)-N(12)	1.493(6)	N(12)-H(12)	1.0000
C(60)-C(61)	1.516(6)	N(14)-H(14A)	0.8800
C(60)-H(60)	1.0000	N(14)-H(14B)	0.8800
C(61)-C(62)	1.537(8)	O(6)-H(6)	0.8400
C(61)-H(61A)	0.9900	O(12)-H(12A)	0.8400
C(61)-H(61B)	0.9900	N(2)-C(1)-N(3)	117.0(3)
C(62)-C(63)	1.521(7)	N(2)-C(1)-C(2)	120.0(3)
C(62)-H(62A)	0.9900	N(3)-C(1)-C(2)	123.0(3)
C(62)-H(62B)	0.9900	C(7)-C(2)-C(3)	119.8(3)
C(63)-N(12)	1.488(6)	C(7)-C(2)-C(1)	114.1(3)
C(63)-H(63A)	0.9900	C(3)-C(2)-C(1)	126.0(3)
C(63)-H(63B)	0.9900	C(4)-C(3)-C(2)	119.5(4)
C(64)-N(12)	1.484(6)	C(4)-C(3)-H(3)	120.2
C(64)-H(64A)	0.9800	C(2)-C(3)-H(3)	120.2
C(64)-H(64B)	0.9800	C(3)-C(4)-C(5)	122.2(3)
C(64)-H(64C)	0.9800	C(3)-C(4)-Cl(1)	119.6(3)
C(65)-O(9)	1.242(7)	C(5)-C(4)-Cl(1)	118.3(3)
C(65)-O(10)	1.276(6)	C(6)-C(5)-C(4)	117.3(3)
C(65)-C(66)	1.499(8)	C(6)-C(5)-C(17)	118.0(3)
C(66)-C(67)	1.534(10)	C(4)-C(5)-C(17)	124.3(3)
C(66)-H(66A)	0.9900	F(1)-C(6)-C(5)	118.6(3)
F(1)-C(6)-C(7)	117.8(3)	H(13A)-C(13)-H(13C)	109.5
C(5)-C(6)-C(7)	123.6(3)	H(13B)-C(13)-H(13C)	109.5
N(1)-C(7)-C(2)	125.2(3)	O(1)-C(14)-N(4)	121.8(4)
N(1)-C(7)-C(6)	117.5(3)	O(1)-C(14)-C(15)	120.1(4)

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C(2)-C(7)-C(6)	117.3(3)	N(4)-C(14)-C(15)	118.0(4)
N(1)-C(8)-N(2)	129.6(3)	C(16)-C(15)-C(14)	120.5(5)
N(1)-C(8)-O(2)	118.2(3)	C(16)-C(15)-H(15)	119.8
N(2)-C(8)-O(2)	112.1(3)	C(14)-C(15)-H(15)	119.8
N(3)-C(9)-C(10)	111.3(3)	C(15)-C(16)-H(16A)	120.0
N(3)-C(9)-H(9A)	109.4	C(15)-C(16)-H(16B)	120.0
C(10)-C(9)-H(9A)	109.4	H(16A)-C(16)-H(16B)	120.0
N(3)-C(9)-H(9B)	109.4	N(5)-C(17)-C(18)	123.3(3)
C(10)-C(9)-H(9B)	109.4	N(5)-C(17)-C(5)	110.6(3)
H(9A)-C(9)-H(9B)	108.0	C(18)-C(17)-C(5)	125.9(3)
N(4)-C(10)-C(9)	110.5(4)	C(17)-C(18)-C(19)	118.6(4)
N(4)-C(10)-H(10A)	109.5	C(17)-C(18)-C(22)	122.9(3)
C(9)-C(10)-H(10A)	109.5	C(19)-C(18)-C(22)	118.5(4)
N(4)-C(10)-H(10B)	109.5	C(20)-C(19)-C(18)	117.6(4)
C(9)-C(10)-H(10B)	109.5	C(20)-C(19)-C(23)	119.4(4)
H(10A)-C(10)-H(10B)	108.1	C(18)-C(19)-C(23)	123.0(4)
N(4)-C(11)-C(12)	110.5(3)	C(19)-C(20)-C(21)	120.8(4)
N(4)-C(11)-H(11A)	109.5	C(19)-C(20)-H(20)	119.6
C(12)-C(11)-H(11A)	109.5	C(21)-C(20)-H(20)	119.6
N(4)-C(11)-H(11B)	109.5	N(6)-C(21)-N(5)	117.3(4)
C(12)-C(11)-H(11B)	109.5	N(6)-C(21)-C(20)	121.7(3)
H(11A)-C(11)-H(11B)	108.1	N(5)-C(21)-C(20)	121.0(3)
N(3)-C(12)-C(13)	111.0(4)	F(4)-C(22)-F(2)	106.2(3)
N(3)-C(12)-C(11)	109.3(3)	F(4)-C(22)-F(3)	105.9(4)
C(13)-C(12)-C(11)	112.1(4)	F(2)-C(22)-F(3)	105.9(3)
N(3)-C(12)-H(12)	108.1	F(4)-C(22)-C(18)	112.0(3)
C(13)-C(12)-H(12)	108.1	F(2)-C(22)-C(18)	113.7(4)
C(11)-C(12)-H(12)	108.1	F(3)-C(22)-C(18)	112.5(3)
C(12)-C(13)-H(13A)	109.5	C(19)-C(23)-H(23A)	109.5
C(12)-C(13)-H(13B)	109.5	C(19)-C(23)-H(23B)	109.5

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H(13A)-C(13)-H(13B)	109.5	H(23A)-C(23)-H(23B)	109.5
C(12)-C(13)-H(13C)	109.5	C(19)-C(23)-H(23C)	109.5
H(23A)-C(23)-H(23C)	109.5	C(27)-C(28)-H(28A)	111.2
H(23B)-C(23)-H(23C)	109.5	N(7)-C(28)-H(28B)	111.2
O(2)-C(24)-C(25)	107.2(3)	C(27)-C(28)-H(28B)	111.2
O(2)-C(24)-H(24A)	110.3	H(28A)-C(28)-H(28B)	109.1
C(25)-C(24)-H(24A)	110.3	N(7)-C(29)-H(29A)	109.5
O(2)-C(24)-H(24B)	110.3	N(7)-C(29)-H(29B)	109.5
C(25)-C(24)-H(24B)	110.3	H(29A)-C(29)-H(29B)	109.5
H(24A)-C(24)-H(24B)	108.5	N(7)-C(29)-H(29C)	109.5
C(24)-C(25)-N(7)	110.9(3)	H(29A)-C(29)-H(29C)	109.5
C(24)-C(25)-C(26)	111.6(4)	H(29B)-C(29)-H(29C)	109.5
N(7)-C(25)-C(26)	105.2(3)	O(4)-C(30)-O(3)	123.6(4)
C(24)-C(25)-H(25)	109.7	O(4)-C(30)-C(31)	118.7(4)
N(7)-C(25)-H(25)	109.7	O(3)-C(30)-C(31)	117.8(4)
C(26)-C(25)-H(25)	109.7	C(30)-C(31)-C(32)	117.2(4)
C(27)-C(26)-C(25)	102.2(4)	C(30)-C(31)-H(31A)	108.0
C(25)-C(26)-C(27A)	104.8(5)	C(32)-C(31)-H(31A)	108.0
C(27)-C(26)-H(26A)	111.3	C(30)-C(31)-H(31B)	108.0
C(25)-C(26)-H(26A)	111.3	C(32)-C(31)-H(31B)	108.0
C(27)-C(26)-H(26B)	111.3	H(31A)-C(31)-H(31B)	107.2
C(25)-C(26)-H(26B)	111.3	C(33)-C(32)-C(31)	110.1(4)
H(26A)-C(26)-H(26B)	109.2	C(33)-C(32)-H(32A)	109.6
C(26)-C(27)-C(28)	101.2(6)	C(31)-C(32)-H(32A)	109.6
C(26)-C(27)-H(27A)	111.5	C(33)-C(32)-H(32B)	109.6
C(28)-C(27)-H(27A)	111.5	C(31)-C(32)-H(32B)	109.6
C(26)-C(27)-H(27B)	111.5	H(32A)-C(32)-H(32B)	108.1
C(28)-C(27)-H(27B)	111.5	C(34)-C(33)-C(32)	115.4(4)
H(27A)-C(27)-H(27B)	109.3	C(34)-C(33)-H(33A)	108.4
C(28)-C(27A)-C(26)	97.1(5)	C(32)-C(33)-H(33A)	108.4

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C(28)-C(27A)-H(27C)	112.3	C(34)-C(33)-H(33B)	108.4
C(26)-C(27A)-H(27C)	112.3	C(32)-C(33)-H(33B)	108.4
C(28)-C(27A)-H(27D)	112.3	H(33A)-C(33)-H(33B)	107.5
C(26)-C(27A)-H(27D)	112.3	C(35)-C(34)-C(33)	118.1(4)
H(27C)-C(27A)-H(27D)	109.9	C(35)-C(34)-H(34A)	107.8
N(7)-C(28)-C(27A)	104.3(5)	C(33)-C(34)-H(34A)	107.8
N(7)-C(28)-C(27)	103.0(4)	C(35)-C(34)-H(34B)	107.8
N(7)-C(28)-H(28A)	111.2	C(33)-C(34)-H(34B)	107.8
H(34A)-C(34)-H(34B)	107.1	C(44)-C(45)-H(45A)	109.5
O(5)-C(35)-O(6)	122.3(4)	N(11)-C(45)-H(45B)	109.5
O(5)-C(35)-C(34)	121.7(4)	C(44)-C(45)-H(45B)	109.5
O(6)-C(35)-C(34)	116.0(4)	H(45A)-C(45)-H(45B)	108.1
N(9)-C(36)-N(10)	117.1(3)	N(11)-C(46)-C(47)	110.5(4)
N(9)-C(36)-C(37)	120.0(4)	N(11)-C(46)-H(46A)	109.5
N(10)-C(36)-C(37)	122.8(4)	C(47)-C(46)-H(46A)	109.5
C(42)-C(37)-C(38)	119.1(3)	N(11)-C(46)-H(46B)	109.5
C(42)-C(37)-C(36)	114.5(4)	C(47)-C(46)-H(46B)	109.5
C(38)-C(37)-C(36)	126.3(4)	H(46A)-C(46)-H(46B)	108.1
C(39)-C(38)-C(37)	120.0(4)	N(10)-C(47)-C(48)	110.5(4)
C(39)-C(38)-H(38)	120.0	N(10)-C(47)-C(46)	109.7(4)
C(37)-C(38)-H(38)	120.0	C(48)-C(47)-C(46)	112.3(4)
C(38)-C(39)-C(40)	122.6(4)	N(10)-C(47)-H(47)	108.0
C(38)-C(39)-Cl(2)	120.2(3)	C(48)-C(47)-H(47)	108.0
C(40)-C(39)-Cl(2)	117.2(3)	C(46)-C(47)-H(47)	108.0
C(41)-C(40)-C(39)	116.6(4)	C(47)-C(48)-H(48A)	109.5
C(41)-C(40)-C(52)	118.4(4)	C(47)-C(48)-H(48B)	109.5
C(39)-C(40)-C(52)	124.8(4)	H(48A)-C(48)-H(48B)	109.5
F(5)-C(41)-C(40)	118.5(3)	C(47)-C(48)-H(48C)	109.5
F(5)-C(41)-C(42)	117.9(3)	H(48A)-C(48)-H(48C)	109.5
C(40)-C(41)-C(42)	123.6(4)	H(48B)-C(48)-H(48C)	109.5

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N(8)-C(42)-C(41)	118.2(4)	O(7)-C(49)-N(11)	122.0(5)
N(8)-C(42)-C(37)	123.8(3)	O(7)-C(49)-C(50)	120.2(5)
C(41)-C(42)-C(37)	117.9(4)	N(11)-C(49)-C(50)	117.9(5)
N(8)-C(43)-N(9)	129.6(4)	C(51)-C(50)-C(49)	120.2(6)
N(8)-C(43)-O(8)	119.0(4)	C(51)-C(50)-H(50)	119.9
N(9)-C(43)-O(8)	111.4(3)	C(49)-C(50)-H(50)	119.9
N(10)-C(44)-C(45)	111.2(4)	C(50)-C(51)-H(51A)	120.0
N(10)-C(44)-H(44A)	109.4	C(50)-C(51)-H(51B)	120.0
C(45)-C(44)-H(44A)	109.4	H(51A)-C(51)-H(51B)	120.0
N(10)-C(44)-H(44B)	109.4	N(13)-C(52)-C(53)	123.0(4)
C(45)-C(44)-H(44B)	109.4	N(13)-C(52)-C(40)	111.4(3)
H(44A)-C(44)-H(44B)	108.0	C(53)-C(52)-C(40)	125.5(4)
N(11)-C(45)-C(44)	110.6(4)	C(52)-C(53)-C(54)	118.5(4)
N(11)-C(45)-H(45A)	109.5	C(52)-C(53)-C(57)	122.8(4)
C(54)-C(53)-C(57)	118.7(4)	C(62)-C(61)-H(61A)	110.9
C(55)-C(54)-C(53)	117.8(4)	C(60)-C(61)-H(61B)	110.9
C(55)-C(54)-C(58)	119.1(4)	C(62)-C(61)-H(61B)	110.9
C(53)-C(54)-C(58)	123.1(4)	H(61A)-C(61)-H(61B)	109.0
C(54)-C(55)-C(56)	120.5(4)	C(63)-C(62)-C(61)	105.7(4)
C(54)-C(55)-H(55)	119.7	C(63)-C(62)-H(62A)	110.6
C(56)-C(55)-H(55)	119.7	C(61)-C(62)-H(62A)	110.6
N(14)-C(56)-N(13)	116.5(4)	C(63)-C(62)-H(62B)	110.6
N(14)-C(56)-C(55)	122.6(4)	C(61)-C(62)-H(62B)	110.6
N(13)-C(56)-C(55)	120.9(4)	H(62A)-C(62)-H(62B)	108.7
F(8)-C(57)-F(7)	105.8(4)	N(12)-C(63)-C(62)	106.7(4)
F(8)-C(57)-F(6)	105.8(4)	N(12)-C(63)-H(63A)	110.4
F(7)-C(57)-F(6)	105.5(4)	C(62)-C(63)-H(63A)	110.4
F(8)-C(57)-C(53)	114.3(4)	N(12)-C(63)-H(63B)	110.4
F(7)-C(57)-C(53)	113.8(4)	C(62)-C(63)-H(63B)	110.4
F(6)-C(57)-C(53)	110.9(4)	H(63A)-C(63)-H(63B)	108.6

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C(54)-C(58)-H(58A)	109.5	N(12)-C(64)-H(64A)	109.5
C(54)-C(58)-H(58B)	109.5	N(12)-C(64)-H(64B)	109.5
H(58A)-C(58)-H(58B)	109.5	H(64A)-C(64)-H(64B)	109.5
C(54)-C(58)-H(58C)	109.5	N(12)-C(64)-H(64C)	109.5
H(58A)-C(58)-H(58C)	109.5	H(64A)-C(64)-H(64C)	109.5
H(58B)-C(58)-H(58C)	109.5	H(64B)-C(64)-H(64C)	109.5
O(8)-C(59)-C(60)	107.4(4)	O(9)-C(65)-O(10)	123.6(5)
O(8)-C(59)-H(59A)	110.2	O(9)-C(65)-C(66)	118.6(5)
C(60)-C(59)-H(59A)	110.2	O(10)-C(65)-C(66)	117.7(5)
O(8)-C(59)-H(59B)	110.2	C(65)-C(66)-C(67)	118.0(5)
C(60)-C(59)-H(59B)	110.2	C(65)-C(66)-H(66A)	107.8
H(59A)-C(59)-H(59B)	108.5	C(67)-C(66)-H(66A)	107.8
N(12)-C(60)-C(61)	103.8(4)	C(65)-C(66)-H(66B)	107.8
N(12)-C(60)-C(59)	110.5(4)	C(67)-C(66)-H(66B)	107.8
C(61)-C(60)-C(59)	113.4(4)	H(66A)-C(66)-H(66B)	107.1
N(12)-C(60)-H(60)	109.6	C(68)-C(67)-C(66)	114.2(5)
C(61)-C(60)-H(60)	109.6	C(68)-C(67)-H(67A)	108.7
C(59)-C(60)-H(60)	109.6	C(66)-C(67)-H(67A)	108.7
C(60)-C(61)-C(62)	104.1(4)	C(68)-C(67)-H(67B)	108.7
C(60)-C(61)-H(61A)	110.9	C(66)-C(67)-H(67B)	108.7
H(67A)-C(67)-H(67B)	107.6	C(29)-N(7)-C(28)	111.8(4)
C(67)-C(68)-C(69)	113.2(5)	C(29)-N(7)-C(25)	112.7(3)
C(67)-C(68)-H(68A)	108.9	C(28)-N(7)-C(25)	106.8(3)
C(69)-C(68)-H(68A)	108.9	C(29)-N(7)-H(7)	108.5
C(67)-C(68)-H(68B)	108.9	C(28)-N(7)-H(7)	108.5
C(69)-C(68)-H(68B)	108.9	C(25)-N(7)-H(7)	108.5
H(68A)-C(68)-H(68B)	107.7	C(43)-N(8)-C(42)	113.7(3)
C(70)-C(69)-C(68)	117.6(5)	C(36)-N(9)-C(43)	117.4(3)
C(70)-C(69)-H(69A)	107.9	C(36)-N(10)-C(44)	122.9(3)
C(68)-C(69)-H(69A)	107.9	C(36)-N(10)-C(47)	119.6(3)

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C(70)-C(69)-H(69B)	107.9	C(44)-N(10)-C(47)	114.4(3)
C(68)-C(69)-H(69B)	107.9	C(49)-N(11)-C(46)	128.3(4)
H(69A)-C(69)-H(69B)	107.2	C(49)-N(11)-C(45)	119.1(4)
O(11)-C(70)-O(12)	123.5(5)	C(46)-N(11)-C(45)	111.4(4)
O(11)-C(70)-C(69)	123.1(5)	C(64)-N(12)-C(63)	112.0(4)
O(12)-C(70)-C(69)	113.4(5)	C(64)-N(12)-C(60)	111.5(4)
C(8)-N(1)-C(7)	113.1(3)	C(63)-N(12)-C(60)	104.8(4)
C(1)-N(2)-C(8)	117.9(3)	C(64)-N(12)-H(12)	109.5
C(1)-N(3)-C(9)	124.3(3)	C(63)-N(12)-H(12)	109.5
C(1)-N(3)-C(12)	119.6(3)	C(60)-N(12)-H(12)	109.5
C(9)-N(3)-C(12)	114.9(3)	C(56)-N(13)-C(52)	119.2(4)
C(14)-N(4)-C(10)	120.2(4)	C(56)-N(14)-H(14A)	120.0
C(14)-N(4)-C(11)	128.0(4)	C(56)-N(14)-H(14B)	120.0
C(10)-N(4)-C(11)	111.6(3)	H(14A)-N(14)-H(14B)	120.0
C(21)-N(5)-C(17)	118.6(4)	C(8)-O(2)-C(24)	116.3(3)
C(21)-N(6)-H(6A)	120.0	C(35)-O(6)-H(6)	109.5
C(21)-N(6)-H(6B)	120.0	C(43)-O(8)-C(59)	117.1(3)
H(6A)-N(6)-H(6B)	120.0	C(70)-O(12)-H(12A)	109.5

[0293] Table 17: Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound A Form A. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
C(1)	30(2)	20(2)	27(2)	3(1)	6(2)	6(2)
C(2)	28(2)	20(2)	27(2)	2(1)	4(2)	6(2)
C(3)	31(2)	22(2)	27(2)	0(1)	7(2)	6(2)
C(4)	34(2)	20(2)	29(2)	0(1)	4(2)	7(2)
C(5)	27(2)	20(2)	26(2)	2(1)	1(2)	5(2)
C(6)	28(2)	19(2)	29(2)	2(1)	7(2)	6(2)
C(7)	28(2)	18(2)	26(2)	2(1)	4(2)	5(2)
C(8)	32(2)	17(2)	26(2)	1(1)	5(2)	6(2)
C(9)	38(2)	22(2)	40(2)	1(2)	16(2)	8(2)
C(10)	37(2)	32(2)	39(2)	5(2)	12(2)	10(2)

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C(11)	48(3)	25(2)	37(2)	0(2)	17(2)	5(2)
C(12)	40(2)	24(2)	39(2)	7(2)	19(2)	8(2)
C(13)	43(3)	35(2)	42(2)	11(2)	13(2)	12(2)
C(14)	29(2)	41(2)	31(2)	-2(2)	2(2)	5(2)
C(15)	39(3)	43(2)	37(2)	0(2)	9(2)	1(2)
C(16)	55(3)	53(3)	38(2)	-4(2)	17(2)	-1(3)
C(17)	33(2)	18(2)	28(2)	0(1)	5(2)	5(2)
C(18)	31(2)	20(2)	29(2)	1(1)	2(2)	5(2)
C(19)	31(2)	20(2)	31(2)	-1(1)	4(2)	2(2)
C(20)	36(2)	17(2)	31(2)	1(1)	5(2)	4(2)
C(21)	35(2)	16(2)	29(2)	0(1)	4(2)	5(2)
C(22)	32(2)	24(2)	34(2)	5(2)	2(2)	1(2)
C(23)	35(2)	31(2)	46(2)	9(2)	5(2)	5(2)
C(24)	36(2)	23(2)	36(2)	-1(2)	11(2)	7(2)
C(25)	36(2)	24(2)	32(2)	1(2)	8(2)	11(2)
C(26)	42(3)	39(2)	36(2)	-2(2)	9(2)	17(2)
C(27)	38(5)	42(5)	26(4)	4(3)	10(3)	21(4)
C(27A)	45(6)	58(6)	32(4)	-12(4)	4(4)	15(5)
C(28)	46(3)	58(3)	33(2)	10(2)	10(2)	28(2)
C(29)	38(3)	31(2)	47(2)	-2(2)	11(2)	4(2)
C(30)	35(2)	26(2)	34(2)	4(2)	8(2)	11(2)
C(31)	37(2)	30(2)	42(2)	2(2)	12(2)	12(2)
C(32)	31(2)	29(2)	39(2)	-1(2)	7(2)	9(2)
C(33)	30(2)	31(2)	48(2)	-3(2)	9(2)	9(2)
C(34)	32(2)	30(2)	54(3)	-3(2)	3(2)	12(2)
C(35)	33(2)	24(2)	41(2)	-2(2)	2(2)	12(2)
C(36)	31(2)	26(2)	28(2)	3(1)	10(2)	10(2)
C(37)	32(2)	26(2)	27(2)	2(1)	8(2)	10(2)
C(38)	32(2)	31(2)	26(2)	2(2)	5(2)	9(2)
C(39)	37(2)	24(2)	25(2)	-3(1)	7(2)	6(2)
C(40)	35(2)	25(2)	31(2)	0(2)	10(2)	10(2)
C(41)	30(2)	27(2)	31(2)	1(2)	3(2)	11(2)
C(42)	32(2)	24(2)	27(2)	1(1)	7(2)	11(2)
C(43)	32(2)	23(2)	29(2)	1(1)	6(2)	9(2)
C(44)	31(2)	26(2)	42(2)	-1(2)	2(2)	7(2)
C(45)	44(3)	33(2)	50(3)	7(2)	14(2)	14(2)
C(46)	50(3)	29(2)	39(2)	1(2)	1(2)	16(2)

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C(47)	36(2)	28(2)	42(2)	8(2)	0(2)	11(2)
C(48)	50(3)	46(3)	42(2)	15(2)	12(2)	22(2)
C(49)	45(3)	43(2)	38(2)	-2(2)	15(2)	20(2)
C(50)	54(3)	48(3)	46(3)	3(2)	11(2)	27(2)
C(51)	72(4)	63(3)	37(2)	-4(2)	0(2)	40(3)
C(52)	39(2)	23(2)	28(2)	-2(1)	7(2)	7(2)
C(53)	44(3)	20(2)	38(2)	2(2)	14(2)	11(2)
C(54)	55(3)	24(2)	36(2)	3(2)	15(2)	14(2)
C(55)	53(3)	23(2)	36(2)	4(2)	12(2)	12(2)
C(56)	45(3)	27(2)	28(2)	1(2)	9(2)	7(2)
C(57)	59(3)	28(2)	44(2)	7(2)	26(2)	21(2)
C(58)	79(4)	35(2)	66(3)	17(2)	38(3)	31(3)
C(59)	33(2)	24(2)	35(2)	0(2)	1(2)	8(2)
C(60)	34(2)	38(2)	37(2)	-4(2)	3(2)	12(2)
C(61)	50(3)	53(3)	47(3)	-11(2)	4(2)	26(2)
C(62)	55(3)	40(2)	50(3)	-9(2)	10(2)	16(2)
C(63)	44(3)	40(3)	58(3)	-3(2)	-2(2)	10(2)
C(64)	52(3)	52(3)	51(3)	13(2)	18(2)	19(2)
C(65)	42(3)	63(3)	41(2)	15(2)	11(2)	22(2)
C(66)	46(3)	86(4)	54(3)	-2(3)	19(3)	22(3)
C(67)	57(4)	89(4)	50(3)	-4(3)	9(3)	48(3)
C(68)	53(3)	72(4)	44(3)	2(2)	13(2)	34(3)
C(69)	68(4)	76(4)	65(3)	14(3)	37(3)	36(3)
C(70)	50(3)	61(3)	48(3)	4(2)	18(2)	24(3)
N(1)	28(2)	19(1)	28(2)	1(1)	6(1)	4(1)
N(2)	30(2)	19(1)	28(2)	1(1)	7(1)	5(1)
N(3)	37(2)	18(2)	41(2)	5(1)	17(2)	8(1)
N(4)	36(2)	29(2)	37(2)	2(1)	10(2)	4(2)
N(5)	32(2)	18(1)	30(2)	2(1)	5(1)	6(1)
N(6)	34(2)	21(2)	41(2)	6(1)	2(2)	7(1)
N(7)	37(2)	28(2)	34(2)	0(1)	9(2)	12(2)
N(8)	36(2)	24(2)	30(2)	2(1)	4(1)	11(1)
N(9)	33(2)	26(2)	30(2)	2(1)	6(1)	11(1)
N(10)	31(2)	24(2)	38(2)	5(1)	2(2)	8(1)
N(11)	42(2)	32(2)	45(2)	4(2)	9(2)	16(2)
N(12)	38(2)	35(2)	39(2)	3(2)	8(2)	10(2)
N(13)	39(2)	26(2)	31(2)	0(1)	9(2)	8(2)

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N(14)	57(3)	36(2)	43(2)	11(2)	22(2)	15(2)
O(1)	43(2)	54(2)	47(2)	5(2)	17(2)	19(2)
O(2)	37(2)	18(1)	32(1)	0(1)	11(1)	5(1)
O(3)	46(2)	28(2)	55(2)	8(1)	16(2)	9(1)
O(4)	39(2)	29(1)	49(2)	2(1)	13(1)	11(1)
O(5)	41(2)	29(2)	54(2)	9(1)	0(2)	10(1)
O(6)	33(2)	36(2)	54(2)	14(2)	-1(2)	5(1)
O(7)	41(2)	52(2)	50(2)	-2(2)	9(2)	17(2)
O(8)	36(2)	22(1)	37(1)	0(1)	-3(1)	8(1)
O(9)	63(3)	70(3)	84(3)	37(2)	34(2)	28(2)
O(10)	50(2)	52(2)	47(2)	10(2)	18(2)	23(2)
O(11)	56(2)	59(2)	47(2)	11(2)	23(2)	22(2)
O(12)	79(3)	74(3)	60(2)	28(2)	44(2)	50(2)
F(1)	39(1)	22(1)	39(1)	0(1)	19(1)	3(1)
F(2)	37(2)	34(1)	54(2)	24(1)	1(1)	3(1)
F(3)	48(2)	43(2)	57(2)	11(1)	9(1)	25(1)
F(4)	47(2)	33(1)	38(1)	0(1)	-12(1)	6(1)
F(5)	41(1)	25(1)	46(1)	0(1)	-4(1)	14(1)
F(6)	107(3)	39(2)	56(2)	1(1)	51(2)	17(2)
F(7)	54(2)	55(2)	71(2)	22(2)	30(2)	17(2)
F(8)	77(2)	45(2)	63(2)	31(1)	43(2)	36(2)
Cl(1)	48(1)	22(1)	36(1)	-6(1)	12(1)	5(1)
Cl(2)	55(1)	26(1)	36(1)	-8(1)	0(1)	10(1)

[0294] Table 18: Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound A Form A

	x	y	z	U(eq)
H(3)	1693	2012	4026	33
H(9A)	-784	1352	3506	39
H(9B)	-1128	799	4262	39
H(10A)	-3749	754	3534	43
H(10B)	-3483	176	2811	43
H(11A)	-3566	-1822	2808	45
H(11B)	-4016	-2524	3504	45
H(12)	-1056	-1930	3529	40
H(13A)	32	-1203	4804	60
H(13B)	-1753	-2239	4689	60
H(13C)	-1662	-943	4799	60

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H(15)	-5344	-2747	4274	52
H(16A)	-7726	-1929	4650	64
H(16B)	-7562	-3183	4815	64
H(20)	8150	6819	2154	36
H(23A)	10662	5511	2593	60
H(23B)	10845	5897	3452	60
H(23C)	10776	6779	2831	60
H(24A)	3472	-1624	1575	38
H(24B)	2323	-1000	1070	38
H(25)	1319	-3458	1061	37
H(26A)	3616	-2363	381	46
H(26B)	2149	-3549	-26	46
H(27A)	1917	-2223	-877	39
H(27B)	2077	-1289	-222	39
H(27C)	1338	-3090	-1027	57
H(27D)	401	-4149	-590	57
H(28A)	-924	-2203	-663	52
H(28B)	-779	-3451	-741	52
H(29A)	-2815	-3890	160	61
H(29B)	-1706	-3948	974	61
H(29C)	-1505	-4555	247	61
H(31A)	-2335	-159	1780	43
H(31B)	-3667	-228	1006	43
H(32A)	-147	1205	1337	40
H(32B)	-1491	1138	560	40
H(33A)	-3135	1698	1338	44
H(33B)	-1446	2042	2003	44
H(34A)	-1598	3072	627	48
H(34B)	-1839	3620	1363	48
H(38)	5956	8520	5893	37
H(44A)	4597	9176	6401	42
H(44B)	3956	9741	5670	42
H(45A)	2269	9774	6497	50
H(45B)	3917	10362	7175	50
H(46A)	5735	12357	7138	49
H(46B)	5194	13050	6457	49
H(47)	7501	12464	6336	44

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H(48A)	6429	11710	5059	66
H(48B)	5861	12766	5215	66
H(48C)	4531	11481	5133	66
H(50)	3256	13252	5729	57
H(51A)	-302	12310	5329	66
H(51B)	877	13595	5175	66
H(55)	9504	3795	7867	45
H(58A)	11988	4157	7438	80
H(58B)	12704	5279	7025	80
H(58C)	11255	4151	6555	80
H(59A)	13732	13100	8622	39
H(59B)	13134	11743	8403	39
H(60)	11077	11436	9166	45
H(61A)	10983	12871	9904	60
H(61B)	11374	13550	9188	60
H(62A)	14095	14559	9801	59
H(62B)	13468	14196	10553	59
H(63A)	15587	13411	9956	61
H(63B)	15106	13145	10748	61
H(64A)	13585	11037	10778	76
H(64B)	11764	10580	10189	76
H(64C)	12312	11723	10739	76
H(66A)	18366	10963	9088	74
H(66B)	17005	10680	8299	74
H(67A)	17488	9039	8868	72
H(67B)	16509	9219	9474	72
H(68A)	15055	8626	7912	63
H(68B)	14062	8933	8473	63
H(69A)	14340	7347	9150	76
H(69B)	13117	6984	8326	76
H(6A)	3746	5959	1576	41
H(6B)	5403	6882	1516	41
H(7)	-576	-2158	559	40
H(12)	14118	11567	9654	46
H(14A)	6493	4649	8448	53
H(14B)	7297	3731	8518	53
H(6)	2554	3875	2029	67

H(12A)	16607	6541	7840	90
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Example 5 – Compound A Form D

[0295] The crystal structure of Compound A Form D was determined using X-ray data collected at 100 K on a crystal isolated from the batch. The structure of Compound A Form D was solved and refined in the non-centrosymmetric, monoclinic space group $I2$ with the final $R1[I > 2\sigma(I)] = 3.54\%$. The asymmetric unit was found to contain one molecule of Compound A, one fully ordered adipate anion, one 50 % occupied water molecule (O7), which is positioned on a crystallographic special position (2-fold axis) and one partially occupied water molecule (O8, 20% occupancy) disordered about a special position (2-fold axis). In the structure, the molecule also shows positional disorder with the methylpyrrolidine ring disordered over two sites and modelled with a 64.6:35.4 ratio. The absolute stereochemistry of the compound has been determined with stereocentres C6 and C25 in the *S* configuration. The Flack parameter for this structure is 0.007(8). The XRPD pattern simulated from the crystal structure data (collected at 100 K) is consistent with the reference experimental diffractogram of Form D (collected at RT). Slight differences in the simulated and experimental diffractograms are attributable to lattice variations with temperature and preferred orientation.

[0296] An overlay with calculated diffractogram of hydrated Form D shows that calculated XRPDs of these two Forms are similar, however there are some differences. The simulated diffractogram is consistent with the experimental VH diffractogram of Compound A Form A at 10% RH. Slight differences in the simulated and experimental diffractograms are attributable to lattice variations with temperature and preferred orientation.

[0297] Compound A Form A, C and D are structurally similar and interconvert rapidly depending upon the ambient relative humidity with the majority of water uptake occurring between 40-50 % RH at 25 °C.

[0298] Transitions of Forms of Compound A at varying RH.

% Relative Humidity	XRPD
0% - 44%	Compound A Form A

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49% - 54%	Transition
58% - 76% - 49%	Compound A Form D
44%	Transition
39% - 0% -44%	Compound A Form A
49%	Transition
54% - 76% - 53%	Compound A Form D
48%	Transition
44% - 0%	Compound A Form A

[0299] Single crystal experiments. The single crystal X-ray structure of Compound A Form D was determined at 100 K using a crystal grown by slow evaporation from THF. A crystal of sufficient size and quality for analysis by single crystal X-ray diffraction was isolated with approximate dimensions 0.22 x 0.20 x 0.08 mm.

[0300] The crystals were monoclinic, group *I*2 with the final $R1=[\sum |F_o| - \sum |F_c|] / \sum |F_o| = 3.54\%$. The compound was confirmed as Compound A Form D. The asymmetric unit contained one molecule of Compound A Form D, one fully ordered adipate anion, one 50% occupied water molecule, O7, sitting on a crystallographic special position (2-fold axis) and one partially occupied water molecule, O8, disordered about a special position (2-fold axis). The refined atomic occupancy of O8 is 0.195 (approximately 20 % water). The molecule also showed positional disorder at carbon C27 methylpyrrolidine ring which has been modelled over two sites with a 64.6:35.4 ratio.

[0301] Table 19: Crystal data and structure refinement for Compound A Form D

Empirical formula	C35 H44 Cl F4 N7 O6.50
Formula weight	778.22
Temperature	90(2) K
Wavelength	1.54184 Å
Crystal system	Monoclinic
Space group	<i>I</i> 2
Unit cell dimensions	$a = 20.2510(3) \text{ Å}$ $\alpha = 90^\circ$. $b = 8.73850(10) \text{ Å}$ $\beta = 112.156(2)^\circ$. $c = 22.2870(3) \text{ Å}$ $\gamma = 90^\circ$.
Volume	3652.76(10) Å ³

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Z	4
Density (calculated)	1.415 Mg/m ³
Absorption coefficient	1.598 mm ⁻¹
F(000)	1632
Crystal size	0.200 x 0.120 x 0.100 mm ³
Theta range for data collection	2.516 to 75.031°.
Index ranges	-24<= <i>h</i> <=24, -9<= <i>k</i> <=10, -27<= <i>l</i> <=27
Reflections collected	28840
Independent reflections	6849 [R(int) = 0.0256]
Completeness to theta = 67.000°	100.0 %
Absorption correction	Gaussian
Max. and min. transmission	1.000 and 0.671
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	6849 / 1 / 509
Goodness-of-fit on F ²	1.048
Final R indices [<i>I</i> >2σ(<i>I</i>)]	R1 = 0.0320, wR2 = 0.0899
R indices (all data)	R1 = 0.0323, wR2 = 0.0902
Absolute structure parameter	-0.009(5)
Extinction coefficient	0.00030(8)
Largest diff. peak and hole	1.072 and -0.188 e.Å ⁻³

[0302] Table 20: Atomic coordinates (× 10⁴) and equivalent isotropic displacement parameters (Å² × 10³) for Compound A Form D. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C(1)	3573(1)	5230(3)	6770(1)	23(1)
C(2)	3021(1)	5999(3)	6231(1)	23(1)
C(3)	2279(1)	5675(3)	5985(1)	25(1)
C(4)	1829(1)	6460(3)	5458(1)	25(1)
C(5)	2072(1)	7610(3)	5151(1)	23(1)
C(6)	2777(1)	7969(3)	5421(1)	23(1)
C(7)	3269(1)	7229(3)	5965(1)	22(1)
C(8)	4368(1)	7039(3)	6722(1)	23(1)
C(9)	3956(1)	3494(3)	7706(1)	31(1)
C(10)	4306(1)	1966(3)	7677(1)	32(1)
C(11)	3297(1)	1196(3)	6730(1)	30(1)
C(12)	2936(1)	2738(3)	6713(1)	28(1)

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C(13)	3568(2)	3460(4)	8177(1)	37(1)
C(14)	3642(1)	-507(3)	7667(1)	30(1)
C(15)	4070(2)	-760(4)	8367(1)	40(1)
C(16)	4045(2)	-2070(4)	8646(2)	46(1)
C(17)	1622(1)	8320(3)	4514(1)	23(1)
C(18)	1326(1)	9766(3)	4430(1)	25(1)
C(19)	974(1)	10322(3)	3785(1)	27(1)
C(20)	917(1)	9355(3)	3286(1)	27(1)
C(21)	1231(1)	7892(3)	3405(1)	25(1)
C(22)	1350(1)	10762(3)	4983(1)	31(1)
C(23)	690(2)	11921(4)	3637(1)	38(1)
C(24)	5276(1)	8670(3)	6669(1)	27(1)
C(25)	6079(1)	8610(3)	6908(1)	29(1)
C(26)	6375(1)	9814(3)	6575(2)	36(1)
C(27)	6974(2)	8987(5)	6419(2)	34(1)
C(28)	6658(2)	7409(3)	6254(1)	36(1)
C(26A)	6375(1)	9814(3)	6575(2)	36(1)
C(27A)	6369(4)	9037(10)	6025(4)	36(2)
C(28A)	6658(2)	7409(3)	6254(1)	36(1)
C(29)	6818(1)	6274(3)	7315(1)	35(1)
C(30)	5218(1)	4019(3)	6244(1)	28(1)
C(31)	4551(2)	3104(3)	5891(1)	32(1)
C(32)	4066(1)	3634(3)	5219(1)	30(1)
C(33)	3427(2)	2568(3)	4943(1)	40(1)
C(34)	2995(2)	2726(4)	4220(2)	47(1)
C(35)	2497(1)	4066(3)	3987(1)	32(1)
N(1)	3952(1)	7773(3)	6207(1)	24(1)
N(2)	4228(1)	5819(2)	7016(1)	24(1)
N(3)	3458(1)	3928(3)	7051(1)	27(1)
N(4)	3766(1)	794(3)	7388(1)	31(1)
N(5)	1585(1)	7400(2)	4018(1)	25(1)
N(6)	1200(1)	6941(3)	2921(1)	32(1)
N(7)	6321(1)	7079(3)	6737(1)	29(1)
O(1)	3181(1)	-1430(2)	7363(1)	39(1)
O(2)	5052(1)	7512(2)	7011(1)	26(1)
O(3)	5243(1)	5376(2)	6049(1)	33(1)
O(4)	5704(1)	3432(2)	6711(1)	39(1)

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O(5)	2191(1)	4320(2)	3410(1)	38(1)
O(6)	2414(1)	4910(2)	4440(1)	35(1)
O(7)	5000	7215(5)	5000	84(2)
F(1)	3023(1)	9104(2)	5150(1)	32(1)
F(2)	696(1)	11166(2)	4942(1)	44(1)
F(3)	1665(1)	10118(2)	5567(1)	43(1)
F(4)	1703(1)	12087(2)	5009(1)	45(1)
Cl(1)	922(1)	6035(1)	5145(1)	34(1)

[0303] Table 21: Bond lengths [Å] and angles [°] for Compound A Form D.

C(1)-N(2)	1.332(3)	C(14)-O(1)	1.227(4)
C(1)-N(3)	1.361(3)	C(14)-N(4)	1.365(3)
C(1)-C(2)	1.461(3)	C(14)-C(15)	1.487(4)
C(2)-C(7)	1.409(3)	C(15)-C(16)	1.312(4)
C(2)-C(3)	1.419(3)	C(15)-H(15)	0.9500
C(3)-C(4)	1.368(3)	C(16)-H(16A)	0.9500
C(3)-H(3)	0.9500	C(16)-H(16B)	0.9500
C(4)-C(5)	1.404(3)	C(17)-N(5)	1.347(3)
C(4)-Cl(1)	1.741(2)	C(17)-C(18)	1.381(3)
C(5)-C(6)	1.360(3)	C(18)-C(19)	1.428(3)
C(5)-C(17)	1.501(3)	C(18)-C(22)	1.494(3)
C(6)-F(1)	1.351(3)	C(19)-C(20)	1.367(4)
C(6)-C(7)	1.403(3)	C(19)-C(23)	1.499(4)
C(7)-N(1)	1.366(3)	C(20)-C(21)	1.408(4)
C(8)-N(1)	1.308(3)	C(20)-H(20)	0.9500
C(8)-N(2)	1.336(3)	C(21)-N(6)	1.344(3)
C(8)-O(2)	1.354(3)	C(21)-N(5)	1.351(3)
C(9)-N(3)	1.478(3)	C(22)-F(3)	1.337(3)
C(9)-C(10)	1.525(4)	C(22)-F(2)	1.339(3)
C(9)-C(13)	1.531(3)	C(22)-F(4)	1.351(3)

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C(9)-H(9)	1.0000	C(23)-H(23A)	0.9800
C(10)-N(4)	1.458(4)	C(23)-H(23B)	0.9800
C(10)-H(10A)	0.9900	C(23)-H(23C)	0.9800
C(10)-H(10B)	0.9900	C(24)-O(2)	1.438(3)
C(11)-N(4)	1.458(3)	C(24)-C(25)	1.509(3)
C(11)-C(12)	1.527(4)	C(24)-H(24A)	0.9900
C(11)-H(11A)	0.9900	C(24)-H(24B)	0.9900
C(11)-H(11B)	0.9900	C(25)-N(7)	1.521(4)
C(12)-N(3)	1.471(3)	C(25)-C(26)	1.533(4)
C(12)-H(12A)	0.9900	C(25)-C(26A)	1.533(4)
C(12)-H(12B)	0.9900	C(25)-H(25)	1.0000
C(13)-H(13A)	0.9800	C(26)-C(27)	1.560(5)
C(13)-H(13B)	0.9800	C(26)-H(26A)	0.9900
C(13)-H(13C)	0.9800	C(26)-H(26B)	0.9900
C(27)-C(28)	1.506(5)	N(6)-H(6A)	0.8800
C(27)-H(27A)	0.9900	N(6)-H(6B)	0.8800
C(27)-H(27B)	0.9900	N(7)-H(7)	1.0000
C(28)-N(7)	1.504(3)	O(6)-H(6)	0.8400
C(28)-H(28A)	0.9900	O(7)-H(7X)	1.05(7)
C(28)-H(28B)	0.9900	N(2)-C(1)-N(3)	116.8(2)
C(26A)-C(27A)	1.399(9)	N(2)-C(1)-C(2)	119.7(2)
C(26A)-H(26C)	0.9900	N(3)-C(1)-C(2)	123.5(2)
C(26A)-H(26D)	0.9900	C(7)-C(2)-C(3)	118.9(2)
C(27A)-C(28A)	1.550(9)	C(7)-C(2)-C(1)	114.4(2)
C(27A)-H(27C)	0.9900	C(3)-C(2)-C(1)	126.6(2)
C(27A)-H(27D)	0.9900	C(4)-C(3)-C(2)	119.8(2)
C(28A)-N(7)	1.504(3)	C(4)-C(3)-H(3)	120.1

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C(28A)-H(28C)	0.9900	C(2)-C(3)-H(3)	120.1
C(28A)-H(28D)	0.9900	C(3)-C(4)-C(5)	122.3(2)
C(29)-N(7)	1.480(3)	C(3)-C(4)-Cl(1)	119.84(18)
C(29)-H(29A)	0.9800	C(5)-C(4)-Cl(1)	117.85(17)
C(29)-H(29B)	0.9800	C(6)-C(5)-C(4)	116.8(2)
C(29)-H(29C)	0.9800	C(6)-C(5)-C(17)	119.0(2)
C(30)-O(4)	1.241(3)	C(4)-C(5)-C(17)	123.8(2)
C(30)-O(3)	1.271(3)	F(1)-C(6)-C(5)	118.3(2)
C(30)-C(31)	1.511(4)	F(1)-C(6)-C(7)	117.51(19)
C(31)-C(32)	1.520(3)	C(5)-C(6)-C(7)	124.2(2)
C(31)-H(31A)	0.9900	N(1)-C(7)-C(6)	117.7(2)
C(31)-H(31B)	0.9900	N(1)-C(7)-C(2)	124.6(2)
C(32)-C(33)	1.523(4)	C(6)-C(7)-C(2)	117.7(2)
C(32)-H(32A)	0.9900	N(1)-C(8)-N(2)	129.6(2)
C(32)-H(32B)	0.9900	N(1)-C(8)-O(2)	118.1(2)
C(33)-C(34)	1.523(4)	N(2)-C(8)-O(2)	112.30(19)
C(33)-H(33A)	0.9900	N(3)-C(9)-C(10)	109.7(2)
C(33)-H(33B)	0.9900	N(3)-C(9)-C(13)	110.3(2)
C(34)-C(35)	1.504(4)	C(10)-C(9)-C(13)	112.3(2)
C(34)-H(34A)	0.9900	N(3)-C(9)-H(9)	108.1
C(34)-H(34B)	0.9900	C(10)-C(9)-H(9)	108.1
C(35)-O(5)	1.219(3)	C(13)-C(9)-H(9)	108.1
C(35)-O(6)	1.312(3)	N(4)-C(10)-C(9)	110.5(2)
N(4)-C(10)-H(10A)	109.5	C(17)-C(18)-C(22)	123.0(2)
C(9)-C(10)-H(10A)	109.5	C(19)-C(18)-C(22)	118.8(2)
N(4)-C(10)-H(10B)	109.5	C(20)-C(19)-C(18)	117.8(2)
C(9)-C(10)-H(10B)	109.5	C(20)-C(19)-C(23)	119.3(2)

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H(10A)-C(10)-H(10B)	108.1	C(18)-C(19)-C(23)	122.9(2)
N(4)-C(11)-C(12)	111.2(2)	C(19)-C(20)-C(21)	120.9(2)
N(4)-C(11)-H(11A)	109.4	C(19)-C(20)-H(20)	119.5
C(12)-C(11)-H(11A)	109.4	C(21)-C(20)-H(20)	119.5
N(4)-C(11)-H(11B)	109.4	N(6)-C(21)-N(5)	117.5(2)
C(12)-C(11)-H(11B)	109.4	N(6)-C(21)-C(20)	121.9(2)
H(11A)-C(11)-H(11B)	108.0	N(5)-C(21)-C(20)	120.6(2)
N(3)-C(12)-C(11)	111.5(2)	F(3)-C(22)-F(2)	105.9(2)
N(3)-C(12)-H(12A)	109.3	F(3)-C(22)-F(4)	105.6(2)
C(11)-C(12)-H(12A)	109.3	F(2)-C(22)-F(4)	105.7(2)
N(3)-C(12)-H(12B)	109.3	F(3)-C(22)-C(18)	114.3(2)
C(11)-C(12)-H(12B)	109.3	F(2)-C(22)-C(18)	112.0(2)
H(12A)-C(12)-H(12B)	108.0	F(4)-C(22)-C(18)	112.8(2)
C(9)-C(13)-H(13A)	109.5	C(19)-C(23)-H(23A)	109.5
C(9)-C(13)-H(13B)	109.5	C(19)-C(23)-H(23B)	109.5
H(13A)-C(13)-H(13B)	109.5	H(23A)-C(23)-H(23B)	109.5
C(9)-C(13)-H(13C)	109.5	C(19)-C(23)-H(23C)	109.5
H(13A)-C(13)-H(13C)	109.5	H(23A)-C(23)-H(23C)	109.5
H(13B)-C(13)-H(13C)	109.5	H(23B)-C(23)-H(23C)	109.5
O(1)-C(14)-N(4)	121.9(2)	O(2)-C(24)-C(25)	107.61(19)
O(1)-C(14)-C(15)	120.2(3)	O(2)-C(24)-H(24A)	110.2
N(4)-C(14)-C(15)	117.9(3)	C(25)-C(24)-H(24A)	110.2
C(16)-C(15)-C(14)	120.9(3)	O(2)-C(24)-H(24B)	110.2
C(16)-C(15)-H(15)	119.5	C(25)-C(24)-H(24B)	110.2
C(14)-C(15)-H(15)	119.5	H(24A)-C(24)-H(24B)	108.5
C(15)-C(16)-H(16A)	120.0	C(24)-C(25)-N(7)	110.4(2)
C(15)-C(16)-H(16B)	120.0	C(24)-C(25)-C(26)	111.8(2)

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H(16A)-C(16)-H(16B)	120.0	N(7)-C(25)-C(26)	105.2(2)
N(5)-C(17)-C(18)	123.3(2)	C(24)-C(25)-C(26A)	111.8(2)
N(5)-C(17)-C(5)	110.8(2)	N(7)-C(25)-C(26A)	105.2(2)
C(18)-C(17)-C(5)	125.7(2)	C(24)-C(25)-H(25)	109.8
C(17)-C(18)-C(19)	118.3(2)	N(7)-C(25)-H(25)	109.8
C(26)-C(25)-H(25)	109.8	H(28C)-C(28A)-H(28D)	109.3
C(25)-C(26)-C(27)	105.6(3)	N(7)-C(29)-H(29A)	109.5
C(25)-C(26)-H(26A)	110.6	N(7)-C(29)-H(29B)	109.5
C(27)-C(26)-H(26A)	110.6	H(29A)-C(29)-H(29B)	109.5
C(25)-C(26)-H(26B)	110.6	N(7)-C(29)-H(29C)	109.5
C(27)-C(26)-H(26B)	110.6	H(29A)-C(29)-H(29C)	109.5
H(26A)-C(26)-H(26B)	108.8	H(29B)-C(29)-H(29C)	109.5
C(28)-C(27)-C(26)	100.6(3)	O(4)-C(30)-O(3)	123.5(2)
C(28)-C(27)-H(27A)	111.6	O(4)-C(30)-C(31)	119.3(2)
C(26)-C(27)-H(27A)	111.6	O(3)-C(30)-C(31)	117.3(2)
C(28)-C(27)-H(27B)	111.6	C(30)-C(31)-C(32)	118.1(2)
C(26)-C(27)-H(27B)	111.6	C(30)-C(31)-H(31A)	107.8
H(27A)-C(27)-H(27B)	109.4	C(32)-C(31)-H(31A)	107.8
N(7)-C(28)-C(27)	105.4(2)	C(30)-C(31)-H(31B)	107.8
N(7)-C(28)-H(28A)	110.7	C(32)-C(31)-H(31B)	107.8
C(27)-C(28)-H(28A)	110.7	H(31A)-C(31)-H(31B)	107.1
N(7)-C(28)-H(28B)	110.7	C(31)-C(32)-C(33)	110.2(2)
C(27)-C(28)-H(28B)	110.7	C(31)-C(32)-H(32A)	109.6
H(28A)-C(28)-H(28B)	108.8	C(33)-C(32)-H(32A)	109.6
C(27A)-C(26A)-C(25)	102.4(4)	C(31)-C(32)-H(32B)	109.6
C(27A)-C(26A)-H(26C)	111.3	C(33)-C(32)-H(32B)	109.6
C(25)-C(26A)-H(26C)	111.3	H(32A)-C(32)-H(32B)	108.1

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C(27A)-C(26A)-H(26D)	111.3	C(34)-C(33)-C(32)	115.7(3)
C(25)-C(26A)-H(26D)	111.3	C(34)-C(33)-H(33A)	108.4
H(26C)-C(26A)-H(26D)	109.2	C(32)-C(33)-H(33A)	108.4
C(26A)-C(27A)-C(28A)	106.2(5)	C(34)-C(33)-H(33B)	108.4
C(26A)-C(27A)-H(27C)	110.5	C(32)-C(33)-H(33B)	108.4
C(28A)-C(27A)-H(27C)	110.5	H(33A)-C(33)-H(33B)	107.4
C(26A)-C(27A)-H(27D)	110.5	C(35)-C(34)-C(33)	118.6(3)
C(28A)-C(27A)-H(27D)	110.5	C(35)-C(34)-H(34A)	107.7
H(27C)-C(27A)-H(27D)	108.7	C(33)-C(34)-H(34A)	107.7
N(7)-C(28A)-C(27A)	101.2(3)	C(35)-C(34)-H(34B)	107.7
N(7)-C(28A)-H(28C)	111.5	C(33)-C(34)-H(34B)	107.7
C(27A)-C(28A)-H(28C)	111.5	H(34A)-C(34)-H(34B)	107.1
N(7)-C(28A)-H(28D)	111.5	O(5)-C(35)-O(6)	123.2(2)
C(27A)-C(28A)-H(28D)	111.5	O(5)-C(35)-C(34)	121.0(2)
O(6)-C(35)-C(34)	115.8(2)	H(6A)-N(6)-H(6B)	120.0
C(8)-N(1)-C(7)	113.4(2)	C(29)-N(7)-C(28A)	111.8(2)
C(1)-N(2)-C(8)	117.93(19)	C(29)-N(7)-C(28)	111.8(2)
C(1)-N(3)-C(12)	124.79(19)	C(29)-N(7)-C(25)	112.2(2)
C(1)-N(3)-C(9)	119.8(2)	C(28A)-N(7)-C(25)	106.6(2)
C(12)-N(3)-C(9)	114.6(2)	C(28)-N(7)-C(25)	106.6(2)
C(14)-N(4)-C(11)	119.8(2)	C(29)-N(7)-H(7)	108.7
C(14)-N(4)-C(10)	128.3(2)	C(28)-N(7)-H(7)	108.7
C(11)-N(4)-C(10)	111.7(2)	C(25)-N(7)-H(7)	108.7
C(17)-N(5)-C(21)	119.0(2)	C(8)-O(2)-C(24)	115.77(17)
C(21)-N(6)-H(6A)	120.0	C(35)-O(6)-H(6)	109.5
C(21)-N(6)-H(6B)	120.0	H(6A)-N(6)-H(6B)	120.0

[0304] Table 22: Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound A

Form D. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^2U^{11} + \dots + 2hkab^*U^{12}]$.

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
C(1)	25(1)	26(1)	18(1)	0(1)	7(1)	1(1)
C(2)	23(1)	26(1)	19(1)	2(1)	7(1)	3(1)
C(3)	25(1)	28(1)	23(1)	2(1)	11(1)	-1(1)
C(4)	21(1)	31(1)	23(1)	-1(1)	9(1)	1(1)
C(5)	23(1)	26(1)	18(1)	0(1)	6(1)	3(1)
C(6)	23(1)	24(1)	21(1)	4(1)	7(1)	-1(1)
C(7)	21(1)	24(1)	19(1)	0(1)	6(1)	1(1)
C(8)	21(1)	25(1)	20(1)	1(1)	5(1)	0(1)
C(9)	32(1)	33(1)	22(1)	8(1)	4(1)	-3(1)
C(10)	27(1)	39(2)	27(1)	10(1)	7(1)	2(1)
C(11)	33(1)	31(1)	25(1)	4(1)	9(1)	0(1)
C(12)	26(1)	28(1)	26(1)	4(1)	7(1)	-3(1)
C(13)	47(2)	39(2)	25(1)	5(1)	12(1)	2(1)
C(14)	35(1)	28(1)	33(1)	6(1)	19(1)	9(1)
C(15)	43(2)	42(2)	37(1)	10(1)	18(1)	8(1)
C(16)	43(2)	51(2)	50(2)	23(1)	24(1)	15(1)
C(17)	19(1)	28(1)	20(1)	2(1)	6(1)	1(1)
C(18)	22(1)	31(1)	21(1)	2(1)	5(1)	4(1)
C(19)	21(1)	34(1)	24(1)	3(1)	5(1)	4(1)
C(20)	23(1)	35(1)	20(1)	4(1)	3(1)	3(1)
C(21)	23(1)	31(1)	21(1)	1(1)	7(1)	-1(1)
C(22)	27(1)	37(2)	24(1)	1(1)	6(1)	9(1)
C(23)	42(2)	37(2)	27(1)	4(1)	5(1)	14(1)
C(24)	22(1)	27(1)	29(1)	5(1)	5(1)	-2(1)
C(25)	25(1)	29(1)	29(1)	-1(1)	4(1)	-2(1)
C(26)	29(1)	30(2)	48(2)	2(1)	13(1)	-3(1)
C(27)	30(2)	35(2)	37(2)	-2(2)	13(2)	-2(2)
C(28)	41(1)	34(2)	34(1)	-1(1)	16(1)	-3(1)
C(26A)	29(1)	30(2)	48(2)	2(1)	13(1)	-3(1)
C(27A)	42(5)	33(5)	41(5)	1(3)	24(4)	-5(3)
C(28A)	41(1)	34(2)	34(1)	-1(1)	16(1)	-3(1)
C(29)	33(1)	34(2)	32(1)	3(1)	5(1)	4(1)
C(30)	35(1)	27(1)	23(1)	2(1)	11(1)	2(1)
C(31)	35(1)	26(1)	31(1)	3(1)	8(1)	-1(1)

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C(32)	31(1)	25(1)	31(1)	1(1)	8(1)	-1(1)
C(33)	35(1)	27(1)	46(2)	2(1)	1(1)	-1(1)
C(34)	38(2)	36(2)	48(2)	-17(1)	-6(1)	6(1)
C(35)	27(1)	31(1)	32(1)	-6(1)	4(1)	0(1)
N(1)	20(1)	28(1)	21(1)	4(1)	4(1)	0(1)
N(2)	23(1)	27(1)	19(1)	3(1)	5(1)	1(1)
N(3)	29(1)	28(1)	20(1)	6(1)	3(1)	-3(1)
N(4)	33(1)	31(1)	28(1)	7(1)	9(1)	1(1)
N(5)	22(1)	30(1)	20(1)	1(1)	4(1)	2(1)
N(6)	35(1)	36(1)	20(1)	-1(1)	4(1)	7(1)
N(7)	26(1)	28(1)	28(1)	2(1)	5(1)	-1(1)
O(1)	45(1)	32(1)	44(1)	6(1)	20(1)	-2(1)
O(2)	22(1)	29(1)	21(1)	4(1)	3(1)	-2(1)
O(3)	36(1)	27(1)	28(1)	2(1)	4(1)	-5(1)
O(4)	41(1)	37(1)	28(1)	3(1)	1(1)	4(1)
O(5)	37(1)	41(1)	29(1)	-8(1)	5(1)	6(1)
O(6)	40(1)	35(1)	27(1)	-2(1)	7(1)	10(1)
O(7)	160(5)	47(2)	28(2)	0	17(2)	0
F(1)	25(1)	34(1)	30(1)	14(1)	3(1)	-4(1)
F(2)	32(1)	63(1)	34(1)	-11(1)	11(1)	16(1)
F(3)	58(1)	45(1)	19(1)	1(1)	8(1)	22(1)
F(4)	53(1)	36(1)	42(1)	-11(1)	15(1)	-6(1)
Cl(1)	19(1)	48(1)	33(1)	10(1)	6(1)	-2(1)

[0305] Table 23: Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound A Form D.

	x	y	z	U(eq)
H(3)	2096	4916	6185	30
H(9)	4338	4290	7859	37
H(10A)	4606	2079	7417	38
H(10B)	4618	1649	8120	38
H(11A)	2929	394	6556	36
H(11B)	3578	1242	6450	36
H(12A)	2677	3049	6257	33
H(12B)	2583	2634	6920	33
H(13A)	3335	4448	8166	56
H(13B)	3912	3264	8617	56

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H(13C)	3208	2647	8050	56
H(15)	4366	39	8616	47
H(16A)	3751	-2873	8400	55
H(16B)	4322	-2215	9093	55
H(20)	661	9674	2852	33
H(23A)	1089	12646	3764	56
H(23B)	385	12144	3879	56
H(23C)	411	12017	3172	56
H(24A)	5119	9692	6754	33
H(24B)	5065	8475	6197	33
H(25)	6289	8764	7388	35
H(26A)	6572	10697	6866	43
H(26B)	5996	10186	6173	43
H(27A)	7049	9467	6048	41
H(27B)	7430	8971	6799	41
H(28A)	6296	7382	5806	43
H(28B)	7034	6648	6291	43
H(26C)	6066	10732	6456	43
H(26D)	6864	10125	6856	43
H(27C)	6677	9566	5835	43
H(27D)	5879	8983	5694	43
H(28C)	6502	6667	5891	43
H(28D)	7185	7400	6460	43
H(29A)	6941	5275	7187	53
H(29B)	6590	6129	7629	53
H(29C)	7252	6885	7513	53
H(31A)	4265	3079	6166	38
H(31B)	4696	2038	5853	38
H(32A)	4335	3644	4929	36
H(32B)	3899	4688	5244	36
H(33A)	3598	1500	5032	48
H(33B)	3105	2753	5178	48
H(34A)	3335	2762	3997	56
H(34B)	2709	1781	4074	56
H(6A)	1406	6037	3009	39
H(6B)	974	7223	2516	39
H(7)	5892	6419	6525	34

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H(6)	2113	5599	4268	53
H(7X)	4400	5674	5035	101
H(7X)	4950(40)	6470(80)	5350(30)	110(20)

[0306] Table 24: Torsion angles [°] for Compound A Form D.

N(2)-C(1)-C(2)-C(7)	7.6(3)
N(3)-C(1)-C(2)-C(7)	-173.2(2)
N(2)-C(1)-C(2)-C(3)	-168.9(2)
N(3)-C(1)-C(2)-C(3)	10.2(4)
C(7)-C(2)-C(3)-C(4)	6.1(3)
C(1)-C(2)-C(3)-C(4)	-177.5(2)
C(2)-C(3)-C(4)-C(5)	-1.1(4)
C(2)-C(3)-C(4)-Cl(1)	178.61(18)
C(3)-C(4)-C(5)-C(6)	-3.2(3)
Cl(1)-C(4)-C(5)-C(6)	177.16(18)
C(3)-C(4)-C(5)-C(17)	169.9(2)
Cl(1)-C(4)-C(5)-C(17)	-9.8(3)
C(4)-C(5)-C(6)-F(1)	-177.9(2)
C(17)-C(5)-C(6)-F(1)	8.7(3)
C(4)-C(5)-C(6)-C(7)	2.4(3)
C(17)-C(5)-C(6)-C(7)	-171.0(2)
F(1)-C(6)-C(7)-N(1)	4.2(3)
C(5)-C(6)-C(7)-N(1)	-176.1(2)
F(1)-C(6)-C(7)-C(2)	-177.2(2)
C(5)-C(6)-C(7)-C(2)	2.5(3)
C(3)-C(2)-C(7)-N(1)	171.8(2)
C(1)-C(2)-C(7)-N(1)	-5.0(3)
C(3)-C(2)-C(7)-C(6)	-6.7(3)
C(1)-C(2)-C(7)-C(6)	176.5(2)
N(3)-C(9)-C(10)-N(4)	-55.9(3)
C(13)-C(9)-C(10)-N(4)	67.1(3)
N(4)-C(11)-C(12)-N(3)	50.9(3)
O(1)-C(14)-C(15)-C(16)	10.9(4)
N(4)-C(14)-C(15)-C(16)	-171.1(3)
C(6)-C(5)-C(17)-N(5)	93.6(3)
C(4)-C(5)-C(17)-N(5)	-79.2(3)
C(6)-C(5)-C(17)-C(18)	-81.4(3)
C(4)-C(5)-C(17)-C(18)	105.7(3)

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N(5)-C(17)-C(18)-C(19)	-0.9(3)
C(5)-C(17)-C(18)-C(19)	173.5(2)
N(5)-C(17)-C(18)-C(22)	178.1(2)
C(5)-C(17)-C(18)-C(22)	-7.4(4)
C(17)-C(18)-C(19)-C(20)	3.4(3)
C(22)-C(18)-C(19)-C(20)	-175.7(2)
C(17)-C(18)-C(19)-C(23)	-174.6(2)
C(22)-C(18)-C(19)-C(23)	6.3(4)
C(18)-C(19)-C(20)-C(21)	-3.6(4)
C(23)-C(19)-C(20)-C(21)	174.4(2)
C(19)-C(20)-C(21)-N(6)	-177.9(2)
C(19)-C(20)-C(21)-N(5)	1.3(4)
C(17)-C(18)-C(22)-F(3)	-1.7(3)
C(19)-C(18)-C(22)-F(3)	177.4(2)
C(17)-C(18)-C(22)-F(2)	-122.1(2)
C(19)-C(18)-C(22)-F(2)	56.9(3)
C(17)-C(18)-C(22)-F(4)	118.9(2)
C(19)-C(18)-C(22)-F(4)	-62.0(3)
O(2)-C(24)-C(25)-N(7)	63.7(2)
O(2)-C(24)-C(25)-C(26)	-179.6(2)
O(2)-C(24)-C(25)-C(26A)	-179.6(2)
C(24)-C(25)-C(26)-C(27)	-137.9(3)
N(7)-C(25)-C(26)-C(27)	-18.1(3)
C(25)-C(26)-C(27)-C(28)	35.9(3)
C(26)-C(27)-C(28)-N(7)	-40.4(3)
C(24)-C(25)-C(26A)-C(27A)	-88.5(4)
N(7)-C(25)-C(26A)-C(27A)	31.3(4)
C(25)-C(26A)-C(27A)-C(28A)	-43.3(5)
C(26A)-C(27A)-C(28A)-N(7)	38.8(5)
O(4)-C(30)-C(31)-C(32)	-162.5(2)
O(3)-C(30)-C(31)-C(32)	18.5(3)
C(30)-C(31)-C(32)-C(33)	179.9(2)
C(31)-C(32)-C(33)-C(34)	-165.8(2)
C(32)-C(33)-C(34)-C(35)	-76.3(4)
C(33)-C(34)-C(35)-O(5)	173.2(3)
C(33)-C(34)-C(35)-O(6)	-7.6(4)
N(2)-C(8)-N(1)-C(7)	3.4(4)

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O(2)-C(8)-N(1)-C(7)	-178.41(19)
C(6)-C(7)-N(1)-C(8)	178.4(2)
C(2)-C(7)-N(1)-C(8)	-0.1(3)
N(3)-C(1)-N(2)-C(8)	175.6(2)
C(2)-C(1)-N(2)-C(8)	-5.2(3)
N(1)-C(8)-N(2)-C(1)	-0.7(4)
O(2)-C(8)-N(2)-C(1)	-178.91(19)
N(2)-C(1)-N(3)-C(12)	-150.4(2)
C(2)-C(1)-N(3)-C(12)	30.4(3)
N(2)-C(1)-N(3)-C(9)	18.5(3)
C(2)-C(1)-N(3)-C(9)	-160.7(2)
C(11)-C(12)-N(3)-C(1)	119.6(2)
C(11)-C(12)-N(3)-C(9)	-49.9(3)
C(10)-C(9)-N(3)-C(1)	-117.9(2)
C(13)-C(9)-N(3)-C(1)	117.9(3)
C(10)-C(9)-N(3)-C(12)	52.1(3)
C(13)-C(9)-N(3)-C(12)	-72.1(3)
O(1)-C(14)-N(4)-C(11)	6.3(4)
C(15)-C(14)-N(4)-C(11)	-171.7(2)
O(1)-C(14)-N(4)-C(10)	-177.8(2)
C(15)-C(14)-N(4)-C(10)	4.2(4)
C(12)-C(11)-N(4)-C(14)	119.4(2)
C(12)-C(11)-N(4)-C(10)	-57.1(3)
C(9)-C(10)-N(4)-C(14)	-116.2(3)
C(9)-C(10)-N(4)-C(11)	60.0(3)
C(18)-C(17)-N(5)-C(21)	-1.4(3)
C(5)-C(17)-N(5)-C(21)	-176.6(2)
N(6)-C(21)-N(5)-C(17)	-179.4(2)
C(20)-C(21)-N(5)-C(17)	1.3(3)
C(27A)-C(28A)-N(7)-C(29)	-140.1(4)
C(27A)-C(28A)-N(7)-C(25)	-17.2(4)
C(27)-C(28)-N(7)-C(29)	-92.4(3)
C(27)-C(28)-N(7)-C(25)	30.6(3)
C(24)-C(25)-N(7)-C(29)	-123.6(2)
C(26)-C(25)-N(7)-C(29)	115.7(2)
C(26A)-C(25)-N(7)-C(29)	115.7(2)
C(24)-C(25)-N(7)-C(28A)	113.7(2)

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C(26A)-C(25)-N(7)-C(28A)	-7.0(2)
C(24)-C(25)-N(7)-C(28)	113.7(2)
C(26)-C(25)-N(7)-C(28)	-7.0(2)
N(1)-C(8)-O(2)-C(24)	-7.4(3)
N(2)-C(8)-O(2)-C(24)	171.03(19)
C(25)-C(24)-O(2)-C(8)	-158.6(2)

[0307] The single crystal structure of Compound A Form D is shown in FIG. 20. C6 and C25 of Compound A Form D are in the *S* configuration. The Flack [2] parameter for this structure is 0.007(8). For the inverted structure, with C6 and C25 in the *R* configuration the Flack parameter is 0.996(8). Determination of the absolute structure using Bayesian statistics on Bijvoet differences [3] reveals that the probability of the absolute structure as presented being correct is 1.000, while the probabilities of the absolute structure being a racemic twin or false are both 0.000. The Flack equivalent and its uncertainty are calculated through this program to be 0.011(6). The calculation was based on 3344 Bijvoet pairs with a coverage of 92 %.

[0308] There are five intermolecular hydrogen bonds in the crystal structure of Form D. A water molecule (O7) sits on a 2-fold axis and is hydrogen bonded to two symmetry related carbonyl oxygen atoms (O4). The carboxylic group of the adipate forms a heterodimer through the O-H...N and O...H-N intramolecular interactions with nitrogen N5 and N6 of the API molecule. Anime nitrogen atom N6 is also involved in a hydrogen bond interaction to oxygen O3 of the adipate. Additionally, nitrogen N7 of disordered 1-methylpyrrolidine is involved in one N-H...O intermolecular interaction to oxygen O4 of the -COO- group of adipate. Views of the crystal packing within the unit cell can be seen in FIG. 21 and FIG. 22. For clarity all hydrogen atoms have been removed from packing diagrams.

[0309] Overall Summary. Compound A was the most stable form for further development of Compound 1. The amorphous form is considered for development purposes to be unstable for further development. Not only is the adipate salt of Compound A far more stable than the other identified salt forms, but it is also useful in the crystallization and purification of the compound during large scale synthesis.

Example 6 – Compound A Form C

[0310] X-ray quality crystals were grown from a saturated methylethylketone solution that was allowed to slowly evaporate under ambient conditions. A colorless prism 0.170 x 0.080 x 0.060 mm in size was mounted on a Cryoloop with Paratone oil. Data

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were collected in a nitrogen gas stream at 90(2) K using phi and omega scans. Crystal-to-detector distance was 40 mm and exposure time was 0.05 seconds per frame using a scan width of 0.5°. Data collection was 100.0% complete to 67.000° in θ . A total of 43859 reflections were collected covering the indices, $-25 \leq h \leq 25$, $-10 \leq k \leq 10$, $-27 \leq l \leq 27$. 7083 reflections were found to be symmetry independent, with an R_{int} of 0.0360. Indexing and unit cell refinement indicated an I-centered, monoclinic lattice. The space group was found to be I 2 (No. 5). The data were integrated and scaled using CrysAlisPro 1.171.41.76a. Solution by iterative methods (SHELXT-2014) produced a complete heavy-atom phasing model. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2018). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-2018.

[0311] Table 25: Hydrogen bond information for Form C

D-H...A	d(D-H)	d(H...A)	d(D...A)	$\angle(\text{DHA})$
N6-H6B...O5	0.85(7)	2.13(7)	2.976(8)	175(5)
N6-H6A...O3#1	0.90(7)	1.96(7)	2.799(8)	156(6)
N6-H6A...O3A#1	0.90(7)	1.95(7)	2.800(10)	158(6)
O6-H6C...N5	0.84	1.85	2.662(6)	163.2
N7A-H7A...O4A#2	1.00	1.58	2.559(13)	163.5
N7-H7...O4	1.00	1.64	2.62(2)	166.2

#1 $x+1/2, y+1/2, z+1/2$ #2 $x, y+1, z$

[0312] Table 26: Crystal data and structure refinement for Compound A Form C

Empirical formula	C35 H42 Cl F4 N7 O6
Formula weight	768.20
Temperature	90(2) K
Wavelength	1.54184 Å
Crystal system	Monoclinic
Space group	I 2
Unit cell dimensions	$a = 20.4022(2)$ Å $\alpha = 90^\circ$. $b = 8.63050(10)$ Å $\beta = 113.7020(10)^\circ$. $c = 22.3422(3)$ Å $\gamma = 90^\circ$.
Volume	$3602.20(8)$ Å ³
Z	4

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Density (calculated)	1.417 Mg/m ³
Absorption coefficient	1.604 mm ⁻¹
F(000)	1608
Crystal size	0.170 x 0.080 x 0.060 mm ³
Theta range for data collection	2.480 to 75.125°.
Index ranges	-25<=h<=25, -10<=k<=10, -27<=l<=27
Reflections collected	43859
Independent reflections	7083 [R(int) = 0.0360]
Completeness to theta = 67.000°	100.0 %
Absorption correction	Gaussian
Max. and min. transmission	1.000 and 0.678
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	7083 / 1 / 574
Goodness-of-fit on F ²	1.082
Final R indices [I>2sigma(I)]	R1 = 0.0333, wR2 = 0.0862
R indices (all data)	R1 = 0.0337, wR2 = 0.0864
Absolute structure parameter	0.009(5)
Extinction coefficient	0.00053(7)
Largest diff. peak and hole	0.211 and -0.190 e.Å ⁻³

[0313] Table 27: Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å²x 10³) for Compound A Form C. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C(1)	3569(1)	5187(3)	6766(1)	28(1)
C(2)	3022(1)	5959(3)	6211(1)	27(1)
C(3)	2277(1)	5656(3)	5958(1)	29(1)
C(4)	1824(1)	6453(3)	5424(1)	30(1)
C(5)	2064(1)	7612(3)	5116(1)	30(1)
C(6)	2781(1)	7942(3)	5392(1)	32(1)
C(7)	3277(1)	7176(3)	5942(1)	29(1)
C(8)	4381(1)	6963(3)	6725(1)	29(1)
C(9)	2944(1)	2666(3)	6687(1)	35(1)
C(10)	3349(2)	1165(4)	6713(1)	40(1)
C(11)	4321(2)	1985(4)	7702(1)	38(1)
C(12)	3936(2)	3479(4)	7725(1)	37(1)
C(13)	3534(2)	3353(5)	8168(2)	51(1)

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C(14)	3665(2)	-542(4)	7659(2)	39(1)
C(15)	4067(2)	-775(4)	8372(2)	48(1)
C(16)	4021(2)	-2091(5)	8648(2)	56(1)
C(17)	1612(1)	8368(3)	4481(1)	29(1)
C(18)	1315(1)	9823(3)	4409(1)	31(1)
C(19)	966(1)	10437(4)	3766(1)	33(1)
C(20)	914(1)	9507(3)	3251(1)	33(1)
C(21)	1218(1)	8023(3)	3356(1)	30(1)
C(22)	1342(2)	10781(4)	4979(1)	38(1)
C(23)	685(2)	12064(4)	3633(2)	46(1)
C(24)	5314(1)	8584(4)	6710(1)	37(1)
C(25)	6106(3)	8603(6)	6915(2)	28(1)
C(26)	6685(3)	7468(7)	6261(3)	38(1)
C(27)	7039(8)	9033(17)	6491(8)	48(3)
C(28)	6404(3)	9875(7)	6613(3)	34(1)
C(29)	6845(3)	6234(7)	7308(3)	36(1)
C(30)	5223(3)	4190(6)	6172(2)	29(1)
C(31)	4575(2)	3362(5)	5781(2)	61(1)
C(24A)	5314(1)	8584(4)	6710(1)	37(1)
C(25A)	5580(3)	7619(6)	6293(3)	32(1)
C(26A)	6541(4)	9165(9)	6316(5)	44(2)
C(27A)	6865(3)	7775(8)	6773(3)	43(1)
C(28A)	6236(10)	6689(19)	6628(9)	38(3)
C(29A)	5735(4)	8081(8)	5271(3)	44(1)
C(24X)	5314(1)	8584(4)	6710(1)	37(1)
C(25X)	6106(3)	8603(6)	6915(2)	28(1)
C(26X)	6685(3)	7468(7)	6261(3)	38(1)
C(27X)	6359(8)	9077(16)	5978(7)	35(3)
C(28X)	6404(3)	9875(7)	6613(3)	34(1)
C(29X)	6845(3)	6234(7)	7308(3)	36(1)
C(30A)	5080(4)	2199(7)	5921(3)	43(1)
C(31A)	4575(2)	3362(5)	5781(2)	61(1)
C(32)	4027(2)	3718(4)	5108(2)	49(1)
C(33)	3397(2)	2605(4)	4900(2)	43(1)
C(34)	2910(2)	2681(5)	4181(2)	63(1)
C(35)	2454(2)	4099(4)	3935(2)	48(1)
N(1)	3972(1)	7681(3)	6192(1)	32(1)

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N(2)	4233(1)	5756(3)	7026(1)	29(1)
N(3)	3436(1)	3900(3)	7054(1)	34(1)
N(4)	3806(1)	759(3)	7383(1)	38(1)
N(5)	1573(1)	7475(3)	3971(1)	29(1)
N(6)	1184(1)	7084(3)	2865(1)	38(1)
N(7)	6365(8)	7098(15)	6742(7)	35(2)
N(7A)	5795(3)	8759(6)	5901(2)	34(1)
N(7X)	6365(8)	7098(15)	6742(7)	35(2)
O(1)	3208(1)	-1477(3)	7332(1)	48(1)
O(2)	5072(1)	7420(2)	7032(1)	34(1)
O(3)	5233(2)	5533(4)	5952(2)	35(1)
O(4)	5711(2)	3642(5)	6664(2)	39(1)
O(3A)	4993(2)	1157(5)	5494(2)	43(1)
O(4A)	5551(3)	2146(6)	6493(2)	62(1)
O(5)	2368(2)	4928(3)	4384(1)	54(1)
O(6)	2169(1)	4400(3)	3354(1)	60(1)
F(1)	3028(1)	9071(2)	5121(1)	45(1)
F(2)	1703(1)	12108(2)	5040(1)	54(1)
F(3)	682(1)	11204(3)	4915(1)	56(1)
F(4)	1643(1)	10074(2)	5554(1)	53(1)
Cl(1)	906(1)	6064(1)	5100(1)	38(1)

[0314] Table 28: Bond lengths [Å] and angles [°] for Compound A Form C.

C(1)-N(2)	1.334(3)	C(14)-C(15)	1.482(4)
C(1)-N(3)	1.364(4)	C(15)-C(16)	1.315(5)
C(1)-C(2)	1.455(3)	C(15)-H(15)	0.9500
C(2)-C(7)	1.410(4)	C(16)-H(16A)	0.9500
C(2)-C(3)	1.416(3)	C(16)-H(16B)	0.9500
C(3)-C(4)	1.365(4)	C(17)-N(5)	1.352(3)
C(3)-H(3)	0.9500	C(17)-C(18)	1.375(4)
C(4)-C(5)	1.409(4)	C(18)-C(19)	1.425(4)
C(4)-Cl(1)	1.747(3)	C(18)-C(22)	1.501(4)
C(5)-C(6)	1.369(4)	C(19)-C(20)	1.370(4)
C(5)-C(17)	1.496(3)	C(19)-C(23)	1.500(4)
C(6)-F(1)	1.348(3)	C(20)-C(21)	1.401(4)
C(6)-C(7)	1.404(4)	C(20)-H(20)	0.9500
C(7)-N(1)	1.368(3)	C(21)-N(6)	1.344(4)

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C(8)-N(1)	1.303(3)	C(21)-N(5)	1.355(3)
C(8)-N(2)	1.340(4)	C(22)-F(4)	1.329(3)
C(8)-O(2)	1.354(3)	C(22)-F(2)	1.339(4)
C(9)-N(3)	1.467(4)	C(22)-F(3)	1.347(3)
C(9)-C(10)	1.525(4)	C(23)-H(23A)	0.9800
C(9)-H(9A)	0.9900	C(23)-H(23B)	0.9800
C(9)-H(9B)	0.9900	C(23)-H(23C)	0.9800
C(10)-N(4)	1.452(3)	C(24)-O(2)	1.434(4)
C(10)-H(10A)	0.9900	C(24)-C(25)	1.491(5)
C(10)-H(10B)	0.9900	C(24)-H(24A)	0.9900
C(11)-N(4)	1.460(4)	C(24)-H(24B)	0.9900
C(11)-C(12)	1.521(4)	C(25)-N(7)	1.510(15)
C(11)-H(11A)	0.9900	C(25)-C(28)	1.537(8)
C(11)-H(11B)	0.9900	C(25)-H(25)	1.0000
C(12)-N(3)	1.480(3)	C(26)-N(7)	1.499(17)
C(12)-C(13)	1.522(4)	C(26)-C(27)	1.521(18)
C(12)-H(12)	1.0000	C(26)-H(26A)	0.9900
C(13)-H(13A)	0.9800	C(26)-H(26B)	0.9900
C(13)-H(13B)	0.9800	C(27)-C(28)	1.603(14)
C(13)-H(13C)	0.9800	C(27)-H(27A)	0.9900
C(14)-O(1)	1.228(4)	C(27)-H(27B)	0.9900
C(14)-N(4)	1.366(4)	C(28)-H(28A)	0.9900
C(28)-H(28B)	0.9900	C(25X)-C(28X)	1.537(8)
C(29)-N(7)	1.456(17)	C(25X)-H(25X)	1.0000
C(29)-H(29A)	0.9800	C(26X)-N(7X)	1.499(17)
C(29)-H(29B)	0.9800	C(26X)-C(27X)	1.560(16)
C(29)-H(29C)	0.9800	C(26X)-H(26E)	0.9900
C(30)-O(4)	1.242(6)	C(26X)-H(26F)	0.9900
C(30)-O(3)	1.263(7)	C(27X)-C(28X)	1.546(14)
C(30)-C(31)	1.448(7)	C(27X)-H(27E)	0.9900
C(31)-C(32)	1.501(5)	C(27X)-H(27F)	0.9900
C(31)-H(31A)	0.9900	C(28X)-H(28E)	0.9900
C(31)-H(31B)	0.9900	C(28X)-H(28F)	0.9900
C(24A)-O(2)	1.434(4)	C(29X)-N(7X)	1.456(17)

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C(24A)-C(25A)	1.504(6)	C(29X)-H(29G)	0.9800
C(24A)-H(24C)	0.9900	C(29X)-H(29H)	0.9800
C(24A)-H(24D)	0.9900	C(29X)-H(29I)	0.9800
C(25A)-C(28A)	1.48(2)	C(30A)-O(4A)	1.253(8)
C(25A)-N(7A)	1.495(7)	C(30A)-O(3A)	1.271(8)
C(25A)-H(25A)	1.0000	C(30A)-C(31A)	1.383(8)
C(26A)-N(7A)	1.470(9)	C(31A)-C(32)	1.501(5)
C(26A)-C(27A)	1.542(10)	C(31A)-H(31C)	0.9900
C(26A)-H(26C)	0.9900	C(31A)-H(31D)	0.9900
C(26A)-H(26D)	0.9900	C(32)-C(33)	1.520(5)
C(27A)-C(28A)	1.516(15)	C(32)-H(32A)	0.9900
C(27A)-H(27C)	0.9900	C(32)-H(32B)	0.9900
C(27A)-H(27D)	0.9900	C(33)-C(34)	1.514(5)
C(28A)-H(28C)	0.9900	C(33)-H(33A)	0.9900
C(28A)-H(28D)	0.9900	C(33)-H(33B)	0.9900
C(29A)-N(7A)	1.482(8)	C(34)-C(35)	1.501(5)
C(29A)-H(29D)	0.9800	C(34)-H(34A)	0.9900
C(29A)-H(29E)	0.9800	C(34)-H(34B)	0.9900
C(29A)-H(29F)	0.9800	C(35)-O(6)	1.219(4)
C(24X)-O(2)	1.434(4)	C(35)-O(5)	1.300(4)
C(24X)-C(25X)	1.491(5)	N(6)-H(6A)	0.8800
C(24X)-H(24E)	0.9900	N(6)-H(6B)	0.8800
C(24X)-H(24F)	0.9900	N(7)-H(7)	1.0000
C(25X)-N(7X)	1.510(15)	N(7A)-H(7A)	1.0000
N(7X)-H(7X)	1.0000	C(9)-C(10)-H(10B)	109.4
O(5)-H(5)	0.8400	H(10A)-C(10)-H(10B)	108.0
N(2)-C(1)-N(3)	117.1(2)	N(4)-C(11)-C(12)	110.5(2)
N(2)-C(1)-C(2)	119.9(2)	N(4)-C(11)-H(11A)	109.5
N(3)-C(1)-C(2)	123.0(2)	C(12)-C(11)-H(11A)	109.5
C(7)-C(2)-C(3)	119.2(2)	N(4)-C(11)-H(11B)	109.5
C(7)-C(2)-C(1)	114.6(2)	C(12)-C(11)-H(11B)	109.5
C(3)-C(2)-C(1)	126.0(2)	H(11A)-C(11)-H(11B)	108.1
C(4)-C(3)-C(2)	119.9(2)	N(3)-C(12)-C(11)	109.2(2)
C(4)-C(3)-H(3)	120.1	N(3)-C(12)-C(13)	110.2(2)

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C(2)-C(3)-H(3)	120.1	C(11)-C(12)-C(13)	112.4(3)
C(3)-C(4)-C(5)	122.5(2)	N(3)-C(12)-H(12)	108.3
C(3)-C(4)-Cl(1)	120.2(2)	C(11)-C(12)-H(12)	108.3
C(5)-C(4)-Cl(1)	117.35(19)	C(13)-C(12)-H(12)	108.3
C(6)-C(5)-C(4)	116.5(2)	C(12)-C(13)-H(13A)	109.5
C(6)-C(5)-C(17)	118.4(2)	C(12)-C(13)-H(13B)	109.5
C(4)-C(5)-C(17)	124.7(2)	H(13A)-C(13)-H(13B)	109.5
F(1)-C(6)-C(5)	118.2(2)	C(12)-C(13)-H(13C)	109.5
F(1)-C(6)-C(7)	117.7(2)	H(13A)-C(13)-H(13C)	109.5
C(5)-C(6)-C(7)	124.1(2)	H(13B)-C(13)-H(13C)	109.5
N(1)-C(7)-C(6)	118.2(2)	O(1)-C(14)-N(4)	121.2(3)
N(1)-C(7)-C(2)	124.2(2)	O(1)-C(14)-C(15)	120.7(3)
C(6)-C(7)-C(2)	117.6(2)	N(4)-C(14)-C(15)	118.1(3)
N(1)-C(8)-N(2)	129.5(2)	C(16)-C(15)-C(14)	120.6(4)
N(1)-C(8)-O(2)	118.6(2)	C(16)-C(15)-H(15)	119.7
N(2)-C(8)-O(2)	111.8(2)	C(14)-C(15)-H(15)	119.7
N(3)-C(9)-C(10)	110.9(2)	C(15)-C(16)-H(16A)	120.0
N(3)-C(9)-H(9A)	109.5	C(15)-C(16)-H(16B)	120.0
C(10)-C(9)-H(9A)	109.5	H(16A)-C(16)-H(16B)	120.0
N(3)-C(9)-H(9B)	109.5	N(5)-C(17)-C(18)	123.2(2)
C(10)-C(9)-H(9B)	109.5	N(5)-C(17)-C(5)	110.9(2)
H(9A)-C(9)-H(9B)	108.1	C(18)-C(17)-C(5)	125.8(2)
N(4)-C(10)-C(9)	110.9(2)	C(17)-C(18)-C(19)	118.4(2)
N(4)-C(10)-H(10A)	109.4	C(17)-C(18)-C(22)	122.6(2)
C(9)-C(10)-H(10A)	109.4	C(19)-C(18)-C(22)	119.0(3)
N(4)-C(10)-H(10B)	109.4	C(20)-C(19)-C(18)	117.9(3)
C(20)-C(19)-C(23)	119.2(2)	C(27)-C(26)-H(26B)	111.1
C(18)-C(19)-C(23)	122.9(3)	H(26A)-C(26)-H(26B)	109.0
C(19)-C(20)-C(21)	121.0(2)	C(26)-C(27)-C(28)	98.8(9)
C(19)-C(20)-H(20)	119.5	C(26)-C(27)-H(27A)	112.0
C(21)-C(20)-H(20)	119.5	C(28)-C(27)-H(27A)	112.0
N(6)-C(21)-N(5)	116.7(3)	C(26)-C(27)-H(27B)	112.0
N(6)-C(21)-C(20)	122.7(2)	C(28)-C(27)-H(27B)	112.0
N(5)-C(21)-C(20)	120.6(2)	H(27A)-C(27)-H(27B)	109.7

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F(4)-C(22)-F(2)	105.8(2)	C(25)-C(28)-C(27)	103.9(7)
F(4)-C(22)-F(3)	106.3(2)	C(25)-C(28)-H(28A)	111.0
F(2)-C(22)-F(3)	105.4(3)	C(27)-C(28)-H(28A)	111.0
F(4)-C(22)-C(18)	114.4(3)	C(25)-C(28)-H(28B)	111.0
F(2)-C(22)-C(18)	112.9(2)	C(27)-C(28)-H(28B)	111.0
F(3)-C(22)-C(18)	111.4(2)	H(28A)-C(28)-H(28B)	109.0
C(19)-C(23)-H(23A)	109.5	N(7)-C(29)-H(29A)	109.5
C(19)-C(23)-H(23B)	109.5	N(7)-C(29)-H(29B)	109.5
H(23A)-C(23)-H(23B)	109.5	H(29A)-C(29)-H(29B)	109.5
C(19)-C(23)-H(23C)	109.5	N(7)-C(29)-H(29C)	109.5
H(23A)-C(23)-H(23C)	109.5	H(29A)-C(29)-H(29C)	109.5
H(23B)-C(23)-H(23C)	109.5	H(29B)-C(29)-H(29C)	109.5
O(2)-C(24)-C(25)	113.8(3)	O(4)-C(30)-O(3)	124.2(5)
O(2)-C(24)-H(24A)	108.8	O(4)-C(30)-C(31)	124.0(5)
C(25)-C(24)-H(24A)	108.8	O(3)-C(30)-C(31)	111.8(4)
O(2)-C(24)-H(24B)	108.8	C(30)-C(31)-C(32)	129.4(4)
C(25)-C(24)-H(24B)	108.8	C(30)-C(31)-H(31A)	104.9
H(24A)-C(24)-H(24B)	107.7	C(32)-C(31)-H(31A)	104.9
C(24)-C(25)-N(7)	111.0(6)	C(30)-C(31)-H(31B)	104.9
C(24)-C(25)-C(28)	116.3(4)	C(32)-C(31)-H(31B)	104.9
N(7)-C(25)-C(28)	105.1(7)	H(31A)-C(31)-H(31B)	105.8
C(24)-C(25)-H(25)	108.0	O(2)-C(24A)-C(25A)	101.9(3)
N(7)-C(25)-H(25)	108.0	O(2)-C(24A)-H(24C)	111.4
C(28)-C(25)-H(25)	108.0	C(25A)-C(24A)-H(24C)	111.4
N(7)-C(26)-C(27)	103.4(7)	O(2)-C(24A)-H(24D)	111.4
N(7)-C(26)-H(26A)	111.1	C(25A)-C(24A)-H(24D)	111.4
C(27)-C(26)-H(26A)	111.1	H(24C)-C(24A)-H(24D)	109.2
N(7)-C(26)-H(26B)	111.1	C(28A)-C(25A)-N(7A)	103.7(8)
C(28A)-C(25A)-C(24A)	117.6(8)	C(24X)-C(25X)-C(28X)	116.3(4)
N(7A)-C(25A)-C(24A)	105.2(4)	N(7X)-C(25X)-C(28X)	105.1(7)
C(28A)-C(25A)-H(25A)	110.0	C(24X)-C(25X)-H(25X)	108.0
N(7A)-C(25A)-H(25A)	110.0	N(7X)-C(25X)-H(25X)	108.0
C(24A)-C(25A)-H(25A)	110.0	C(28X)-C(25X)-H(25X)	108.0
N(7A)-C(26A)-C(27A)	107.0(5)	N(7X)-C(26X)-C(27X)	104.0(9)

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N(7A)-C(26A)-H(26C)	110.3	N(7X)-C(26X)-H(26E)	110.9
C(27A)-C(26A)-H(26C)	110.3	C(27X)-C(26X)-H(26E)	110.9
N(7A)-C(26A)-H(26D)	110.3	N(7X)-C(26X)-H(26F)	110.9
C(27A)-C(26A)-H(26D)	110.3	C(27X)-C(26X)-H(26F)	110.9
H(26C)-C(26A)-H(26D)	108.6	H(26E)-C(26X)-H(26F)	109.0
C(28A)-C(27A)-C(26A)	103.8(9)	C(28X)-C(27X)-C(26X)	99.6(9)
C(28A)-C(27A)-H(27C)	111.0	C(28X)-C(27X)-H(27E)	111.9
C(26A)-C(27A)-H(27C)	111.0	C(26X)-C(27X)-H(27E)	111.9
C(28A)-C(27A)-H(27D)	111.0	C(28X)-C(27X)-H(27F)	111.9
C(26A)-C(27A)-H(27D)	111.0	C(26X)-C(27X)-H(27F)	111.9
H(27C)-C(27A)-H(27D)	109.0	H(27E)-C(27X)-H(27F)	109.6
C(25A)-C(28A)-C(27A)	106.7(11)	C(25X)-C(28X)-C(27X)	101.4(6)
C(25A)-C(28A)-H(28C)	110.4	C(25X)-C(28X)-H(28E)	111.5
C(27A)-C(28A)-H(28C)	110.4	C(27X)-C(28X)-H(28E)	111.5
C(25A)-C(28A)-H(28D)	110.4	C(25X)-C(28X)-H(28F)	111.5
C(27A)-C(28A)-H(28D)	110.4	C(27X)-C(28X)-H(28F)	111.5
H(28C)-C(28A)-H(28D)	108.6	H(28E)-C(28X)-H(28F)	109.3
N(7A)-C(29A)-H(29D)	109.5	N(7X)-C(29X)-H(29G)	109.5
N(7A)-C(29A)-H(29E)	109.5	N(7X)-C(29X)-H(29H)	109.5
H(29D)-C(29A)-H(29E)	109.5	H(29G)-C(29X)-H(29H)	109.5
N(7A)-C(29A)-H(29F)	109.5	N(7X)-C(29X)-H(29I)	109.5
H(29D)-C(29A)-H(29F)	109.5	H(29G)-C(29X)-H(29I)	109.5
H(29E)-C(29A)-H(29F)	109.5	H(29H)-C(29X)-H(29I)	109.5
O(2)-C(24X)-C(25X)	113.8(3)	O(4A)-C(30A)-O(3A)	124.0(6)
O(2)-C(24X)-H(24E)	108.8	O(4A)-C(30A)-C(31A)	116.9(5)
C(25X)-C(24X)-H(24E)	108.8	O(3A)-C(30A)-C(31A)	118.5(6)
O(2)-C(24X)-H(24F)	108.8	C(30A)-C(31A)-C(32)	124.2(4)
C(25X)-C(24X)-H(24F)	108.8	C(30A)-C(31A)-H(31C)	106.3
H(24E)-C(24X)-H(24F)	107.7	C(32)-C(31A)-H(31C)	106.3
C(24X)-C(25X)-N(7X)	111.0(6)	C(30A)-C(31A)-H(31D)	106.3
C(32)-C(31A)-H(31D)	106.3	C(14)-N(4)-C(10)	120.0(3)
H(31C)-C(31A)-H(31D)	106.4	C(14)-N(4)-C(11)	128.0(2)
C(31A)-C(32)-C(33)	112.0(3)	C(10)-N(4)-C(11)	111.5(2)
C(31)-C(32)-C(33)	112.0(3)	C(17)-N(5)-C(21)	118.8(2)

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C(31)-C(32)-H(32A)	109.2	C(21)-N(6)-H(6A)	120.0
C(33)-C(32)-H(32A)	109.2	C(21)-N(6)-H(6B)	120.0
C(31)-C(32)-H(32B)	109.2	H(6A)-N(6)-H(6B)	120.0
C(33)-C(32)-H(32B)	109.2	C(29)-N(7)-C(26)	113.4(11)
H(32A)-C(32)-H(32B)	107.9	C(29)-N(7)-C(25)	113.6(9)
C(34)-C(33)-C(32)	114.3(3)	C(26)-N(7)-C(25)	107.4(9)
C(34)-C(33)-H(33A)	108.7	C(29)-N(7)-H(7)	107.4
C(32)-C(33)-H(33A)	108.7	C(26)-N(7)-H(7)	107.4
C(34)-C(33)-H(33B)	108.7	C(25)-N(7)-H(7)	107.4
C(32)-C(33)-H(33B)	108.7	C(26A)-N(7A)-C(29A)	111.2(6)
H(33A)-C(33)-H(33B)	107.6	C(26A)-N(7A)-C(25A)	105.0(5)
C(35)-C(34)-C(33)	118.0(3)	C(29A)-N(7A)-C(25A)	111.4(5)
C(35)-C(34)-H(34A)	107.8	C(26A)-N(7A)-H(7A)	109.7
C(33)-C(34)-H(34A)	107.8	C(29A)-N(7A)-H(7A)	109.7
C(35)-C(34)-H(34B)	107.8	C(25A)-N(7A)-H(7A)	109.7
C(33)-C(34)-H(34B)	107.8	C(29X)-N(7X)-C(26X)	113.4(11)
H(34A)-C(34)-H(34B)	107.1	C(29X)-N(7X)-C(25X)	113.6(9)
O(6)-C(35)-O(5)	123.4(3)	C(26X)-N(7X)-C(25X)	107.4(9)
O(6)-C(35)-C(34)	121.6(3)	C(29X)-N(7X)-H(7X)	107.4
O(5)-C(35)-C(34)	115.0(3)	C(26X)-N(7X)-H(7X)	107.4
C(8)-N(1)-C(7)	113.6(2)	C(25X)-N(7X)-H(7X)	107.4
C(1)-N(2)-C(8)	117.6(2)	C(8)-O(2)-C(24X)	116.7(2)
C(1)-N(3)-C(9)	123.3(2)	C(8)-O(2)-C(24)	116.7(2)
C(1)-N(3)-C(12)	119.4(2)	C(8)-O(2)-C(24A)	116.7(2)
C(9)-N(3)-C(12)	115.2(2)	C(35)-O(5)-H(5)	109.5

[0315] Table 29: Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound A Form C. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
C(1)	26(1)	36(1)	22(1)	-2(1)	9(1)	0(1)
C(2)	25(1)	36(1)	20(1)	-1(1)	9(1)	1(1)
C(3)	24(1)	39(1)	25(1)	-2(1)	11(1)	-3(1)
C(4)	22(1)	43(2)	25(1)	-2(1)	8(1)	-2(1)
C(5)	26(1)	41(1)	20(1)	1(1)	7(1)	2(1)

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C(6)	27(1)	46(2)	22(1)	4(1)	8(1)	-6(1)
C(7)	23(1)	43(1)	21(1)	1(1)	8(1)	-2(1)
C(8)	21(1)	39(1)	23(1)	-6(1)	6(1)	-1(1)
C(9)	28(1)	41(2)	28(1)	3(1)	4(1)	-4(1)
C(10)	41(1)	41(2)	30(1)	3(1)	7(1)	-2(1)
C(11)	29(1)	53(2)	28(1)	8(1)	9(1)	-2(1)
C(12)	35(1)	45(2)	24(1)	4(1)	4(1)	-2(1)
C(13)	48(2)	73(2)	29(2)	3(2)	13(1)	10(2)
C(14)	39(2)	39(2)	43(2)	6(1)	20(1)	6(1)
C(15)	43(2)	59(2)	44(2)	18(2)	18(1)	8(2)
C(16)	46(2)	71(2)	61(2)	28(2)	32(2)	17(2)
C(17)	22(1)	43(2)	20(1)	-1(1)	6(1)	-1(1)
C(18)	25(1)	43(2)	21(1)	-3(1)	5(1)	2(1)
C(19)	26(1)	47(2)	24(1)	1(1)	7(1)	5(1)
C(20)	29(1)	44(2)	21(1)	0(1)	5(1)	1(1)
C(21)	26(1)	41(1)	22(1)	-1(1)	6(1)	-2(1)
C(22)	33(1)	52(2)	24(1)	-2(1)	7(1)	9(1)
C(23)	46(2)	53(2)	31(1)	-1(1)	8(1)	17(2)
C(24)	27(1)	42(2)	40(2)	-8(1)	11(1)	-8(1)
C(25)	22(2)	31(2)	27(2)	-1(2)	6(2)	-2(2)
C(26)	43(3)	44(3)	33(3)	-3(2)	22(2)	-2(3)
C(27)	35(7)	59(9)	63(8)	18(7)	32(7)	12(6)
C(28)	32(3)	35(3)	37(3)	-4(3)	16(2)	-5(2)
C(29)	30(2)	39(3)	35(3)	4(2)	10(2)	3(2)
C(30)	30(2)	33(3)	23(2)	-1(2)	9(2)	4(2)
C(31)	45(2)	75(3)	57(2)	-23(2)	16(2)	3(2)
C(24A)	27(1)	42(2)	40(2)	-8(1)	11(1)	-8(1)
C(25A)	33(3)	29(3)	34(3)	-4(2)	14(2)	-6(2)
C(26A)	36(5)	45(4)	47(5)	5(4)	13(4)	-15(3)
C(27A)	29(3)	44(3)	49(4)	4(3)	7(3)	-1(3)
C(28A)	25(6)	45(8)	42(7)	5(5)	11(5)	-3(5)
C(29A)	51(3)	42(3)	37(3)	-8(3)	16(3)	-8(3)
C(24X)	27(1)	42(2)	40(2)	-8(1)	11(1)	-8(1)
C(25X)	22(2)	31(2)	27(2)	-1(2)	6(2)	-2(2)
C(26X)	43(3)	44(3)	33(3)	-3(2)	22(2)	-2(3)
C(27X)	44(9)	37(6)	23(6)	-4(6)	13(6)	-5(6)
C(28X)	32(3)	35(3)	37(3)	-4(3)	16(2)	-5(2)

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C(29X)	30(2)	39(3)	35(3)	4(2)	10(2)	3(2)
C(30A)	64(4)	37(3)	38(3)	-3(3)	31(3)	-5(3)
C(31A)	45(2)	75(3)	57(2)	-23(2)	16(2)	3(2)
C(32)	46(2)	35(2)	68(2)	13(2)	26(2)	6(1)
C(33)	43(2)	35(2)	49(2)	3(1)	16(1)	6(1)
C(34)	58(2)	62(2)	53(2)	-19(2)	5(2)	20(2)
C(35)	47(2)	55(2)	38(2)	-8(1)	12(1)	12(2)
N(1)	22(1)	51(1)	21(1)	-1(1)	6(1)	-4(1)
N(2)	24(1)	35(1)	26(1)	-1(1)	7(1)	2(1)
N(3)	31(1)	40(1)	24(1)	2(1)	4(1)	-3(1)
N(4)	38(1)	41(1)	30(1)	6(1)	7(1)	-1(1)
N(5)	25(1)	39(1)	22(1)	0(1)	7(1)	0(1)
N(6)	42(1)	44(1)	22(1)	-3(1)	6(1)	3(1)
N(7)	21(5)	43(6)	36(5)	-1(4)	8(4)	-4(4)
N(7A)	34(3)	36(2)	32(2)	-3(2)	12(2)	-6(2)
N(7X)	21(5)	43(6)	36(5)	-1(4)	8(4)	-4(4)
O(1)	50(1)	41(1)	56(1)	4(1)	23(1)	-3(1)
O(2)	23(1)	41(1)	33(1)	-2(1)	4(1)	-2(1)
O(3)	34(2)	33(2)	30(2)	0(2)	5(2)	-1(2)
O(4)	43(2)	41(2)	24(2)	5(2)	4(2)	7(2)
O(3A)	53(2)	39(2)	35(2)	-4(2)	17(2)	-2(2)
O(4A)	97(4)	44(3)	37(2)	-1(2)	18(3)	1(3)
O(5)	71(2)	54(1)	35(1)	2(1)	18(1)	29(1)
O(6)	58(1)	81(2)	35(1)	-7(1)	12(1)	26(1)
F(1)	31(1)	68(1)	29(1)	15(1)	3(1)	-14(1)
F(2)	62(1)	53(1)	43(1)	-18(1)	16(1)	-7(1)
F(3)	38(1)	89(2)	40(1)	-15(1)	13(1)	18(1)
F(4)	67(1)	65(1)	20(1)	0(1)	12(1)	20(1)
Cl(1)	20(1)	58(1)	33(1)	5(1)	7(1)	-1(1)

[0316] Table 30: Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for Compound A Form C.

	x	y	z	U(eq)
H(3)	2094	4900	6159	35
H(9A)	2686	2991	6226	41
H(9B)	2585	2488	6875	41
H(10A)	3003	318	6513	47

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H(10B)	3645	1290	6458	47
H(11A)	4627	2163	7459	45
H(11B)	4633	1662	8152	45
H(12)	4301	4321	7896	45
H(13A)	3300	4343	8170	76
H(13B)	3872	3101	8613	76
H(13C)	3172	2535	8006	76
H(15)	4360	34	8631	58
H(16A)	3729	-2904	8391	67
H(16B)	4281	-2230	9105	67
H(20)	667	9873	2817	40
H(23A)	371	12255	3860	69
H(23B)	415	12205	3162	69
H(23C)	1087	12794	3790	69
H(24A)	5159	9612	6800	44
H(24B)	5083	8408	6232	44
H(25)	6334	8724	7401	33
H(26A)	6310	7535	5811	45
H(26B)	7041	6676	6273	45
H(27A)	7151	9561	6150	58
H(27B)	7479	8946	6897	58
H(28A)	6587	10761	6916	41
H(28B)	6031	10253	6197	41
H(29A)	7274	6855	7546	54
H(29B)	6984	5264	7163	54
H(29C)	6601	6002	7597	54
H(31A)	4300	3303	6059	73
H(31B)	4732	2290	5753	73
H(24C)	5704	9215	7028	44
H(24D)	4918	9273	6440	44
H(25A)	5186	6942	5995	38
H(26C)	6557	10107	6574	53
H(26D)	6812	9367	6043	53
H(27C)	7247	7275	6675	52
H(27D)	7066	8103	7237	52
H(28C)	6258	5826	6345	45
H(28D)	6240	6248	7039	45

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H(29D)	6112	7305	5353	66
H(29E)	5788	8902	4991	66
H(29F)	5266	7589	5053	66
H(24E)	5159	9612	6800	44
H(24F)	5083	8408	6232	44
H(25X)	6334	8724	7401	33
H(26E)	6551	6676	5911	45
H(26F)	7213	7526	6480	45
H(27E)	6648	9625	5780	42
H(27F)	5858	8986	5653	42
H(28E)	6903	10143	6901	41
H(28F)	6105	10822	6518	41
H(29G)	6606	6030	7603	54
H(29H)	6972	5249	7164	54
H(29I)	7280	6841	7539	54
H(31C)	4843	4335	5948	73
H(31D)	4304	3155	6055	73
H(32A)	4255	3655	4793	58
H(32B)	3851	4790	5100	58
H(33A)	3583	1536	5007	52
H(33B)	3112	2832	5159	52
H(34A)	3211	2570	3929	76
H(34B)	2587	1771	4077	76
H(6A)	1386	6164	2951	45
H(6B)	959	7389	2457	45
H(7)	5935	6435	6512	42
H(7A)	5487	9704	5816	41
H(7X)	5935	6435	6512	42
H(5)	2083	5659	4208	82

[0317] Similarly to Compound A Form D, the carboxylic group of the adipate forms a heterodimer with the API molecule through the O-H $\cdots$ N and O $\cdots$ H-N intramolecular interactions. The hydrogen H6A of amine N6 forms one intermolecular N-H $\cdots$ O bond to either O3 or O3A oxygen atoms of positionally disordered -COO- group of the adipate anion (50/50). Additionally, the nitrogen atom N7 of one component of positionally disordered 1-methylpyrrolidine is involved in one N-H $\cdots$ O intermolecular interaction to oxygen O4 of also disordered -COO- group of adipate. Nitrogen N7A of

the second component is hydrogen bonded to the analogous oxygen O4A. There appear to be no further hydrogen bonding interactions and as such the crystal structure packing is further stabilised by Van der Waals interactions only.

[0318] Views of the crystal packing within the unit cell can be seen in FIG. 23 and FIG. 24. A comparison of the crystal packing of Compound A Form C and Compound A Form D is shown in FIG. 25. For clarity all hydrogen atoms have been removed from packing diagrams.

Example 6 – Compound B Form A.

[0319] Compound B Form A was obtained from an acetone system and the sample was air dried at RT. No form change was observed before and after drying (FIG. 11). A weight loss of 0.7% up to 100 °C was observed in TGA and DSC result showed one endotherm at 53.8 °C (peak temperature) before the possible overlapped peak with peak temperature at 156.3 °C (FIG. 12). Based on the integrals, the stoichiometric ratio of fumaric acid:freebase was determined to be 1.03. A peak of acetone was observed and approximately 0.49 molar acetone (4.4 wt%) was detected. The HPLC purity of Compound B Form A was determined to be 93.56 area%. After heating Compound B Form A to 100 °C under N₂ protection, cooled down and exposed to ambient conditions, no form change was observed before and after heating. Peak of acetone was observed in the heated sample. Around 0.39 molar acetone (3.5 wt%) was still detected. One endotherm at 58.7 °C and an overlapped peak with peak temperature at 157.1 °C were still observed on the sample obtained after heating. The first endotherm might be caused by re-adsorption of moisture. Since ~3.5 wt% acetone was still detected from NMR, Compound B Form A is expected to be an acetone/H₂O co-solvate.

Example 7 – Compound C Form A

[0320] Compound C Form A was obtained from EtOAc system and the sample was air dried at RT. No form change was observed before and after drying (FIG. 13). A weight loss of 8.6% up to 100 °C was observed in TGA and two endotherms at 81.0 °C and 149.2 °C (peak temperature) were observed in DSC curve (FIG. 14). HPLC purity of Compound C Form A was determined to be 95.18 area%.

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Example 8 – Compound D Form A:

[0321] Compound D Form A was obtained from evaporation of acetone/n-heptane (1:1, v/v) solution. The XRPD result is displayed in FIG. 15. A weight loss of 7.7% was observed up to 170 °C in TGA, and DSC result (FIG. 16) showed one endotherm at 75.8 °C (peak temperature). Based on the integrals by NMR analysis, the stoichiometric ratio of benzenesulfonic acid:freebase was determined to be 1.03. Peak of acetone was observed. Around 0.30 molar acetone (2.7 wt%) was detected. HPLC purity of Compound D Form A was determined to be 96.23 area%.

Example 9: Compound E Form A:

[0322] Compound E Form A was obtained from acetone system. The XRPD spectrum overlay is shown in FIG. 17. Limited solids were observed after stirring at 5 °C. During XRPD test, it was observed that the sample tended to absorb moisture and turn to liquid. The suspension was transferred to -20 °C, but limited solids were still observed. Anti-solvent (n-heptane) was added and the sample was transferred to 5 °C. However, gel was obtained.

Example 10: Compound F Form A:

[0323] Compound F Form A (810935-03-A2) was obtained from acetone/n-heptane (1:1, v/v) system by evaporation at RT (FIG. 26).

[0324] A weight loss of 9.0% up to 100 °C was observed in TGA and DSC result showed two endotherms at 65.5 °C and 125.4 °C (peak temperature) (FIG. 27).

[0325] Based on the integrals, the stoichiometric ratio of acetic acid:freebase was determined to be 0.69. Peak of acetone was observed. Around 0.16 molar acetone (1.5 wt%) was detected. The purity of Compound F Form A was 88.42 area%.

[0326] After heating Compound F Form A to 100 °C under N₂ protection, cooled down and exposed to ambient conditions, no form change was observed. Two broad DSC endotherms at 64.2 °C and 124.2 °C (peak temperature) were still observed on the sample obtained after heating, suggesting the re-adsorption of moisture. Since no form change was observed after heating, Compound F Form A is likely a hydrate.

[0327] Table 31: Representative XRPD Peaks for Compound F Form A.

Pos. [°2θ]	d-spacing [Å]	Rel. Int. [%]
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6.6132	13.35494	55.67
7.7347	11.42087	100
8.9607	9.86079	6.47
10.3787	8.51654	4.48
12.9417	6.83509	21.07
13.7826	6.41992	16.86
14.5599	6.07887	4.79
16.0886	5.50455	12.29
17.4322	5.08318	4.97
18.0779	4.90307	9.75
20.3112	4.36871	20.73
21.2392	4.17987	43.46
24.2764	3.66337	20.25
25.6495	3.47029	8.21
29.6647	3.00907	5.28

Example 10 - Freebase screening procedure

[0328] The solubility of starting material was estimated at RT in 20 solvents. For each experiment, approximately 2 mg of sample was added into a 3-mL glass vial. Solvents in Table 32 were then added stepwise (50, 50, 200, 700 μ L) into the vials until the solids were dissolved visually or a total volume of 1 mL was reached. Solubility results are summarized in Table 5-11 and were used to guide the solvent selection in salt screening and crystal form screening.

[0329] Table 32: Approximate solubility of starting material at RT

Solvent	Solubility (mg/mL)	Solvent	Solubility (mg/mL)
MeOH	S > 42.0	1,4-Dioxane	S > 38.0
EtOH	7.0 < S < 21.0	ACN	6.7 < S < 20.0
IPA	2.1 < S < 7.0	MTBE	2.2 < S < 7.3
Acetone	S > 40.0	n-Heptane	S < 2.0
MEK	S > 46.0	Toluene	S > 40.0
EtOAc	S > 40.0	DMSO	S > 38.0
IPAc	S > 40.0	H ₂ O	S < 2.1
MIBK	S > 42.0	DCM	S > 38.0
THF	S > 44.0	CPME	S > 40.0

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Solvent	Solubility (mg/mL)	Solvent	Solubility (mg/mL)
2-MeTHF	S > 40.0	Anisole	S > 38.0

[0330] A total of 64 polymorph screening experiments were performed for freebase Compound 1 using the sample received as starting material through different crystallization methods. The methods utilized and crystal forms identified are summarized in Table 33. As a result, Form A was obtained from polymorph screening.

[0331] Table 33: Summary of polymorph screening experiments of freebase

Method	Result
Slurry at RT/5 °C/50 °C	Form A, amorphous, gel,
Liquid Vapor Diffusion	Form A, amorphous, gel, clear
Slow Evaporation	Form A, amorphous, gel
Anti-solvent Addition	Form A, amorphous, gel,
Total	Form A, amorphous, gel, clear

[0332] Slurry at RT

[0333] Slurry conversion experiments were conducted at RT in different solvent systems. About 15~20 mg of freebase starting material (Compound 1) was suspended in 0.5 mL of solvent in an HPLC vial. Solids were isolated for XRPD analysis after magnetically stirring at RT. Results summarized in Table 34 indicated that Form A and amorphous were obtained.

[0334] Table 34: Summary of slurry conversion experiments at RT

Solvent, v:v	Result
IPA	Form A (Low crystallinity)
MTBE	Gel
ACN	Form A
EtOH	Form A
EtOH/H ₂ O, 1:1	Form A
ACN/H ₂ O, 1:1	Form A (Low crystallinity)
Acetone/H ₂ O, 1:4	Form A
1,4-Dioxane/H ₂ O, 1:4	Amorphous
Benzyl alcohol/n-Heptane, 1:4	Gel
THF/n-Heptane, 1:4	Form A

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Solvent, v:v	Result
DCM/n-Heptane, 1:4	Form A
Anisole/n-Heptane, 1:4	Gel
Toluene/Cyclohexane, 1:4	Amorphous
CPME/Cyclohexane, 1:4	Amorphous
MEK/Cyclohexane, 1:4	Amorphous
EtOAc/Cyclohexane, 1:4	Amorphous

[0335] Slurry at 5 °. Slurry conversion experiments were conducted at 5 °C in different solvent systems. About 15~20 mg of freebase starting material (Compound 1) was suspended in 0.5 mL of solvent in an HPLC vial. Solids were isolated for XRPD analysis after magnetically stirring at 5 °C. Results summarized in Table 35 indicated that Form A and amorphous were obtained.

[0336] Table 35: Summary of slurry conversion experiments at 5 °C

Solvent, v:v	Result
ACN	Form A
MIBK/n-Heptane, 1:3	Gel
IPAc/n-Heptane, 1:3	Amorphous
2-MeTHF/n-Heptane, 1:3	Gel
Xylene/n-Heptane, 1:3	Amorphous
DMAc/n-Heptane, 1:3	Gel
NMP/H ₂ O, 1:4	Amorphous
Ethyl lactate/H ₂ O, 1:3	Gel
DMF/H ₂ O, 1:3	Amorphous

[0337] Slurry at 50 °. Slurry conversion experiments were conducted at 50 °C in different solvent systems. About 20 mg of freebase starting material (Compound 1) was suspended in 0.5 mL of solvent in an HPLC vial. Solids were isolated for XRPD analysis after magnetical stirring at 50 °C. Results summarized in Table 36 indicated that amorphous was obtained.

[0338] Table 36:

Solvent	Result
H ₂ O	Amorphous

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n-Heptane

Amorphous

[0339] Liquid vapor diffusion. Liquid vapor diffusion experiments were conducted in different solvent systems. Approximate 20 mg of freebase starting material (Compound 1) was dissolved in 0.3~1.5 mL corresponding solvent to obtain a clear solution in a 3-mL vial (if a suspension was observed, the sample was filtered through a PTFE filter with 0.45 μ m pore size). This solution was then placed into a 20-mL vial with 3 mL of corresponding anti-solvent. The 20-mL vial was sealed with a cap and kept at RT allowing sufficient time for organic vapor to interact with the solution. The precipitates were isolated for XRPD analysis. The results summarized in Table 37 showed that Form A and amorphous were observed.

[0340] Table 37:

Solvent	Anti-solvent	Result
IPAc		Amorphous
Toluene	n-Heptane	Gel
1,4-Dioxane		Gel
2-Butanol		Gel
DMF		Clear solution
ACN	H ₂ O	Form A
DMSO		Amorphous
THF		Form A (Low crystallinity)
CPME		Gel
Anisole	n-Pentane	Gel
Acetone		Form A
EtOAc		Gel
Xylene		Gel

[0341] Slow evaporation. Slow evaporation experiments were performed under 10 conditions. Briefly, about 15~20 mg of freebase starting material (810935-01-A) was dissolved in 0.5~1.0 mL of solvent in a 3-mL glass vial. The visually clear solutions were subjected to evaporation at RT with vials sealed by Parafilm®. The solids were isolated for XRPD analysis, and the results summarized in Table 38 indicated that Form A and amorphous were obtained.

[0342] Table 38:

Solvent	Result
ACN	Form A
DCM	Form A (Low crystallinity)
MEK	Amorphous
EtOAc	Amorphous
IPAc	Gel
MeOH	Amorphous
EtOH	Form A
Acetone	Form A
THF	Amorphous
2-MeTHF	Gel

[0343] Anti-solvent addition. A total of 12 anti-solvent addition experiments were carried out. About 20 mg of freebase starting material (Compound 1) was dissolved in 0.5~1.5 mL solvent to obtain a clear solution (if a suspension was observed, the sample was filtered through a PTFE filter with 0.45 μm pore size), and the solution was magnetically stirred followed by addition of anti-solvent stepwise till precipitate appeared or the total amount of anti-solvent reached 10.0 mL. The obtained precipitate was isolated for XRPD analysis. Results summarized in Table 39 showed that Compound A Form A with low crystallinity and amorphous were generated.

[0344] Table 39:

Solvent	Anti-solvent	Result
Toluene		Amorphous
EtOAc		Amorphous
CPME	n-Heptane	Amorphous
Acetone		Form A (Low crystallinity)
ACN		Form A (Low crystallinity)
1,4-Dioxane	H ₂ O	Gel
MeOH		Amorphous
DMSO		Low crystallinity

Solvent	Anti-solvent	Result
THF		Form A (Low crystallinity)
Anisole	Cyclohexane	Gel
MEK		Gel
DCM		Form A (Low crystallinity)

Example 11 - Polymorphic screening of Compound A.

[0345] The solubility of Compound A Form A (810935-33-A) was estimated at RT, following similar procedure as described herein. Results summarized in Table 40 were used to guide the solvent selection in polymorph screening of adipate. The methods utilized and crystal forms identified are summarized in Table 41.

[0346] Table 40: Approximate solubility of Compound A Form A at RT

Solvent	Solubility (mg/mL)	Solvent	Solubility (mg/mL)
MeOH	S > 48.0	1,4-Dioxane	7.0 < S < 21.0
EtOH	S > 42.0	ACN	2.4 < S < 8.0
IPA	2.2 < S < 7.3	MTBE	S < 1.9
Acetone	6.0 < S < 18.0	n-Heptane	S < 2.0
MEK	6.7 < S < 20.0	Toluene	S < 1.9
EtOAc	S < 2.0	DMSO	S > 44.0
IPAc	S < 1.9	H ₂ O	22.0 < S < 44.0
MIBK	S < 2.0	DCM	19.0 < S < 38.0
THF	S > 48.0	CPME	S < 2.0
2-MeTHF	20.0 < S < 40.0	Anisole	S < 1.9

[0347] Table 41: Summary of polymorph screening experiments of adipate

Method	Result
Slurry at RT/50 °C	Compound A Form A, gel, clear
Liquid Vapor Diffusion	Amorphous, clear, gel
Slow Evaporation	Gel, clear
Solid Vapor Diffusion	Compound A Form A
Anti-solvent Addition	Compound A Form A, B, clear, gel
Slow Cooling	Compound A Form A

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Method	Result
Total	Compound A Form A, Compound A Form B, clear, gel

[0348] Slurry at RT for Compound A. Slurry conversion experiments were conducted at RT in different solvent systems. About 15 mg of Compound A Form A was suspended in 0.5 mL of solvent in an HPLC vial. Solids were isolated for XRPD analysis after magnetic stirring at RT. Results summarized in Table 42 indicated that adipate Type A was obtained.

[0349] Table 42:

Solvent, v:v	Result
IPA	Compound A Form A
ACN	Compound A Form A
Acetone	Compound A Form A
MEK	Compound A Form A
1,4-Dioxane	Compound A Form A
H ₂ O	Clear solution
H ₂ O/ACN, 1:4	Gel
H ₂ O/IPA, 1:4	Gel
DCM/IPA, 1:4	Compound A Form A
2-MeTHF/Toluene, 1:4	Compound A Form A
Acetone/n-Heptane, 1:3	Compound A Form A
Acetone/Anisole, 1:3	Compound A Form A
MEK/n-Heptane, 1:3	Compound A Form A
MEK/MTBE, 1:3	Compound A Form A
1,4-Dioxane/n-Heptane, 1:3	Compound A Form A
1,4-Dioxane/IPAc, 1:3	Compound A Form A
DMSO/H ₂ O, 1:6	Clear solution
THF/H ₂ O, 1:6	Clear solution
EtOH/n-Heptane, 1:6	Compound A Form A
MeOH/CPME, 1:6	Compound A Form A

*: Clear solution was transferred to 5 °C, followed by transferring to -20 °C. **: Clear solution was transferred to 5 °C, followed by transferring to -20 °C. Clear solution was

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Solvent, v:v	Result
still observed, so anti-solvent was added. #: Clear solution was transferred to 5 °C, followed by transferring to -20 °C. Clear solution was still observed, so anti-solvent was added. Since clear solution was still observed, the sample was transferred to evaporation at RT. &: The clear solution was transferred to evaporation at RT	

[0350] Slurry at 50 ° for Compound A. Slurry conversion experiments were conducted at 50 °C in different solvent systems. About 15 mg of Compound A Form A was suspended in 0.5 mL of solvent in an HPLC vial. Solids were isolated for XRPD analysis after magnetic stirring at 50 °C. Results summarized in Table 43 indicated that Compound A Form A was obtained.

[0351] Table 43:

Solvent	Result
MIBK	Compound A Form A
EtOAc	Compound A Form A
IPAc	Compound A Form A
Anisole	Compound A Form A
n-Heptane	Compound A Form A
CPME	Compound A Form A
MTBE	Compound A Form A
Toluene	Compound A Form A

[0352] Liquid vapor diffusion Compound A. Liquid vapor diffusion experiments were conducted in different solvent systems. Approximate 15 mg of Compound A Form A was dissolved in 0.5 mL corresponding solvent to obtain a clear solution in a 3-mL vial. This solution was then placed into a 20-mL vial with 3 mL of corresponding anti-solvent. The 20-mL vial was sealed with a cap and kept at RT allowing sufficient time for organic vapor to interact with the solution. The precipitates were isolated for XRPD analysis. The results summarized in Table 44 showed that amorphous was obtained.

[0353] Table 44:

Solvent	Anti-solvent	Result
DMSO	EtOAc	Clear solution
THF	n-Heptane	Amorphous
	MTBE	Gel

EtOH	n-Pentane	Gel
	MTBE	Gel
H ₂ O	IPA	Gel
	ACN	Gel

*: clear solutions obtained from liquid vapor diffusion were transferred to evaporation at RT.

[0354] Slow evaporation Compound A. Slow evaporation experiments were performed under seven conditions. Briefly, about 15 mg of Compound A Form A was dissolved in 0.5~1.0 mL of solvent in a 3-mL glass vial. The visually clear solutions were subjected to evaporation at RT with vials sealed by Parafilm® with a few pinholes. The results summarized in Table 45 indicated that only gel or clear solution was obtained.

[0355] Table 45:

Solvent	Result
MeOH	Gel
EtOH	Gel
THF	Gel
DCM	Gel
2-MeTHF	Gel
H ₂ O	Clear solution
Acetone	Gel

[0356] Solid vapor diffusion Compound A. Solid vapor diffusion experiments were conducted using 12 different solvents. Approximate 15 mg of Compound A Form A (810935-40-A) was weighed into a 3-mL vial, which was placed into a 20-mL vial with 2 mL of corresponding solvent. The 20-mL vial was sealed with a cap and kept at RT allowing solvent vapor to interact with the sample. The solids were tested by XRPD and the results summarized in Table 46 showed that Compound A Form A was obtained.

[0357] Table 46:

Solvent	Result
H ₂ O	Compound A Form A
DCM	Compound A Form A

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Solvent	Result
EtOH	Compound A Form A
MeOH	Compound A Form A
ACN	Compound A Form A
THF	Compound A Form A
Acetone	Compound A Form A
DMF	Compound A Form A
EtOAc	Compound A Form A
1,4-Dioxane	Compound A Form A
IPA	Compound A Form A
DMSO	Compound A Form A

[0358] Anti-solvent addition Compound A. A total of 14 anti-solvent addition experiments were carried out. About 15 mg of Compound A Form A was dissolved in 0.5~1.5 mL solvent to obtain a clear solution (if a suspension was observed, the sample was filtered through a PTFE filter with 0.45 μ m pore size), and the solution was magnetically stirred followed by addition of anti-solvent stepwise until precipitate appeared or the total amount of anti-solvent reached 10.0 mL. The obtained precipitate was isolated for XRPD analysis. Results summarized in Table 47 showed that Compound A Form A and Compound A Form B were generated.

[0359] Table 47:

Solvent	Anti-solvent	Result
THF	n-Heptane	Compound A Form A
EtOH		Compound A Form A
DCM		Compound A Form A
2-MeTHF	Anisole	Gel
EtOH		Clear solution
DMSO	EtOAc	Clear
THF		Compound A Form A
MeOH	Toluene	Compound A Form A
THF		Compound A Form A
DCM	MTBE	Compound A Form B
MeOH		Gel

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Solvent	Anti-solvent	Result
EtOH		Gel
DCM		Compound A Form A
THF		Compound A Form A

[0360] Slow cooling Compound A. Slow cooling experiments were conducted in four solvent systems. About 15 mg of Compound A Form A was suspended in 1.0 mL of solvent in a 3-mL glass vial at RT. The suspension was then heated to 50 °C, equilibrated for about 2 hr and filtered to a new vial using a seal membrane (PTFE, pore size of 0.45 µm). Filtrates were slowly cooled down to 5 °C at a rate of 0.1 °C/min. The obtained solids were kept isothermal at 5 °C before isolation for XRPD analysis. Results summarized in Table 48 indicated Compound A Form A was obtained.

[0361] Table 48:

Solvent	Result
Acetone	Compound A Form A
MEK	Compound A Form A
IPA	Compound A Form A
ACN	Compound A Form A

[0362] All technical and scientific terms used herein have the same meaning. Efforts have been made to ensure accuracy with respect to numbers used (e.g. amounts, temperature, etc.) but some experimental errors and deviations should be accounted for.

[0363] Throughout this specification and the claims, the words “comprise,” “comprises,” and “comprising” are used in a non-exclusive sense, except where the context requires otherwise. It is understood that embodiments described herein include “consisting of” and/or “consisting essentially of” embodiments.

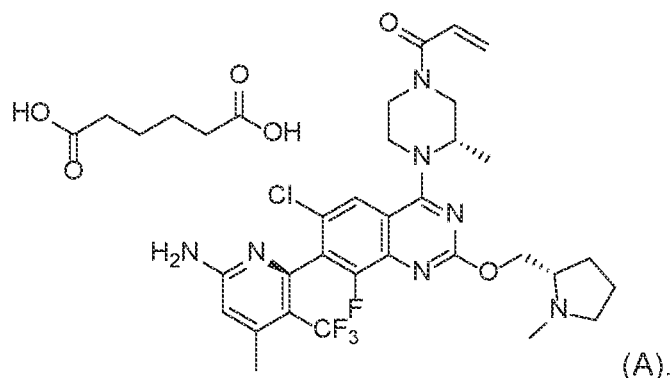
[0364] Where a range of values is provided, it is understood that each intervening value, to the tenth of the unit of the lower limit, unless the context clearly dictates otherwise, between the upper and lower limit of the range and any other stated or intervening value in that stated range, is encompassed herein. The upper and lower limits of these small ranges which can independently be included in the smaller ranges is also encompassed herein, subject to any specifically excluded limit in the

stated range. Where the stated range includes one or both of the limits, ranges excluding either or both of those included limits are also included herein.

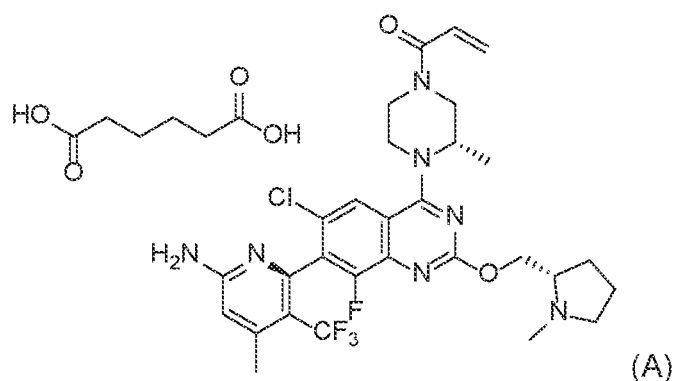
[0365] Many modifications and other embodiments of the inventions set forth herein will come to mind to one skilled in the art to which these inventions pertain having the benefit of the teachings presented in the foregoing descriptions and the associated drawings. Therefore, it is to be understood that the inventions are not to be limited to the specific embodiments disclosed and that modifications and other embodiments are intended to be included within the scope of the appended claims. Although specific terms are employed herein, they are used in a generic and descriptive sense only and not for purposes of limitation.

What is claimed is:

1. A compound having the structure:



2. A crystal form comprising Compound A:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 9.6474, 11.0365, 15.4059, 16.4193, 18.7038 ($\pm 0.1^\circ 2\theta$).

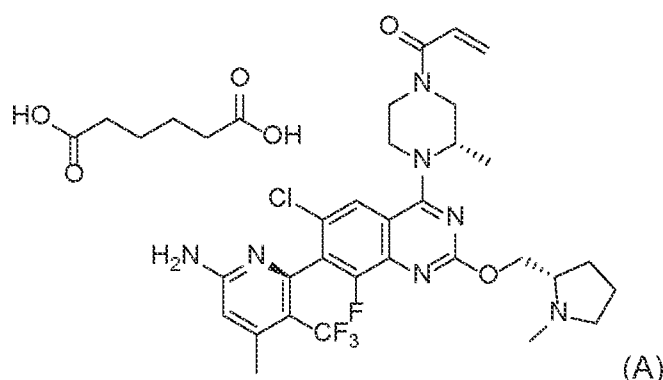
3. The crystal form of claim 2, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 1.
4. The crystal form of claim 2, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 1.
5. The crystal form of claim 2, wherein the crystal form of Compound A has an X-

ray powder diffraction pattern substantially as shown in FIG 1.

6. The crystal form of claim 2, wherein the crystal form of Compound A has a TGA thermograph corresponding substantially to the representative TGA thermogram as depicted in FIG 2.

7. The crystal form of claim 2, wherein the crystal form of Compound A has a DSC thermograph corresponding substantially as depicted in FIG 2.

8. A crystal form comprising Compound A:



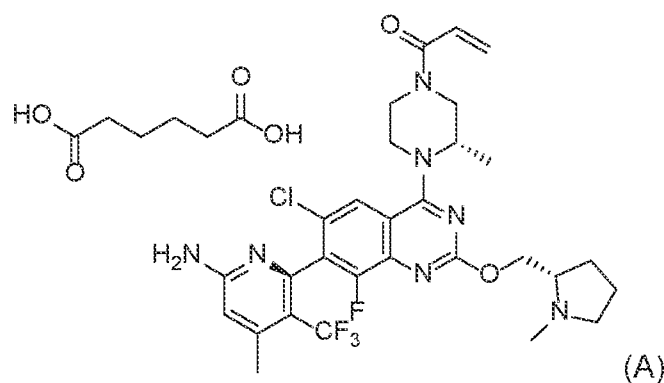
having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 5.2063, 14.63, 17.7161, 21.9253, 25.1335 ($\pm 0.1^\circ 2\theta$).

9. The crystal form of claim 8, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 2.

10. The crystal form of claim 8, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 2.

11. The crystal form of claim 8, wherein the crystal form of Compound A has an X-ray powder diffraction pattern substantially as shown in FIG 5.

12. A crystal form comprising Compound A:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 9.7745, 11.0487, 15.2452, 18.6477, 18.7908 ($\pm 0.1^\circ 2\theta$).

13. The crystal form of claim 12, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 3.

14. The crystal form of claim 12, wherein the crystal form of Compound A has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 3.

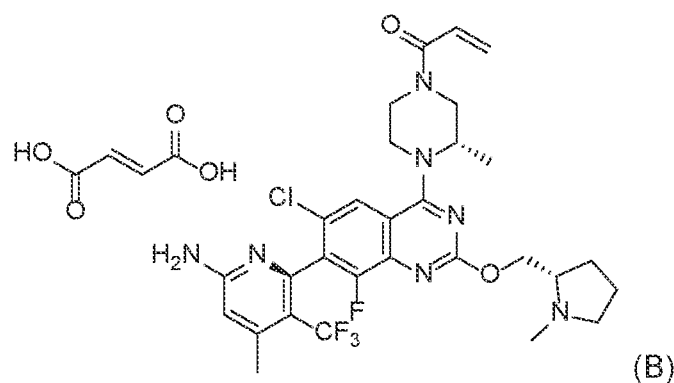
15. The crystal form of claim 12, wherein the crystal form of Compound A has an X-ray powder diffraction pattern substantially as shown in FIG 8.

16. The crystal form of claim 12, wherein the crystal form of Compound A has a TGA thermograph corresponding substantially to the representative TGA thermogram as depicted in FIG 10.

17. The crystal form of claim 12, wherein the crystal form of Compound A has a DSC thermograph corresponding substantially as depicted in FIG 9.

18. A crystal form comprising Compound B:

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having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 6.6796, 14.9097, 15.2963, 19.4416, 23.7124 ($\pm 0.1^\circ 2\theta$).

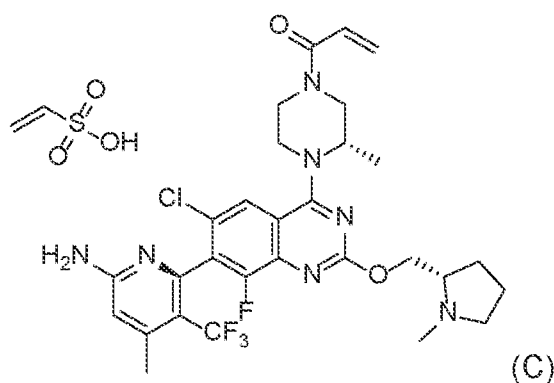
19. The crystal form of claim 18, wherein the crystal form of Compound B has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 4.

20. The crystal form of claim 18, wherein the crystal form of Compound B has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 4.

21. The crystal form of claim 18, wherein the crystal form of Compound B has an X-ray powder diffraction pattern substantially as shown in FIG 11.

22. The crystal form of claim 18, wherein the crystal form of Compound B has a DSC thermograph corresponding substantially as depicted in FIG 12.

23. A crystal form comprising Compound C:



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having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 12.5401, 17.5707, 16.834, 21.3525, 26.5569 ($\pm 0.1^\circ 2\theta$).

24. The crystal form of claim 23, wherein the crystal form of Compound C has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 5.

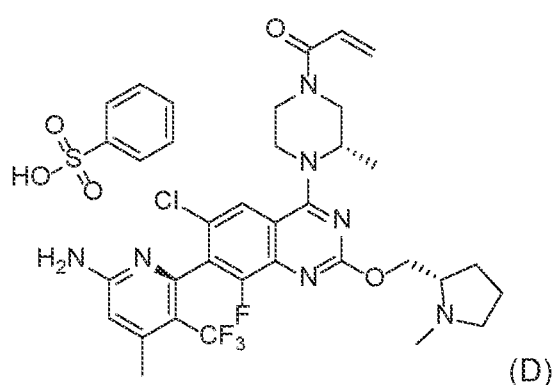
25. The crystal form of claim 23, wherein the crystal form of Compound C has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 5.

26. The crystal form of claim 23, wherein the crystal form of Compound C has an X-ray powder diffraction pattern substantially as shown in FIG 13.

27. The crystal form of claim 23, wherein the crystal form of Compound C has a TGA thermograph corresponding substantially to the representative TGA thermogram as depicted in FIG 14.

28. The crystal form of claim 23, wherein the crystal form of Compound C has a DSC thermograph corresponding substantially as depicted in FIG 14.

29. A crystal form comprising Compound D:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 7.3809, 10.7407, 14.654, 18.5979, 25.2558 ($\pm 0.1^\circ 2\theta$).

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30. The crystal form of claim 29, wherein the crystal form of Compound D has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 6.

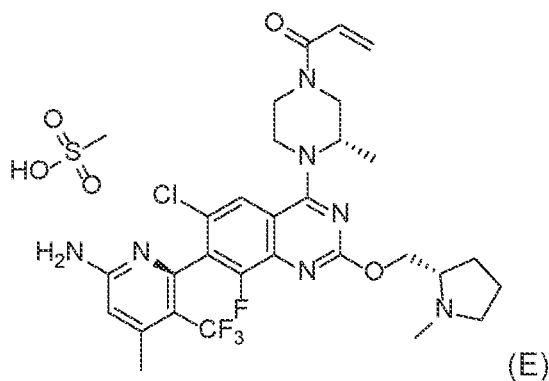
31. The crystal form of claim 29, wherein the crystal form of Compound D has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 6.

32. The crystal form of claim 29, wherein the crystal form of Compound D has an X-ray powder diffraction pattern substantially as shown in FIG 15.

33. The crystal form of claim 29, wherein the crystal form of Compound D has a TGA thermograph corresponding substantially to the representative TGA thermogram as depicted in FIG 16.

34. The crystal form of claim 29, wherein the crystal form of Compound D has a DSC thermograph corresponding substantially as depicted in FIG 16.

35. A crystal form comprising Compound E:



having an X-ray powder diffraction pattern comprising characteristic X-ray powder diffraction peaks using CuK α radiation at 5.9702, 6.2798, 12.4314, 18.9264, 20.6365 ($\pm 0.1^\circ 2\theta$).

36. The crystal form of claim 35, wherein the crystal form of Compound E has an X-ray powder diffraction pattern comprising at least 5 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 7.

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37. The crystal form of claim 35, wherein the crystal form of Compound E has an X-ray powder diffraction pattern comprising at least 10 characteristic X-ray powder diffraction peaks using CuK α radiation as set forth in Table 7.
38. The crystal form of claim 35, wherein the crystal form of Compound E has an X-ray powder diffraction pattern substantially as shown in FIG 17.
39. A pharmaceutical composition comprising the solid form of any one of claims 2-38 and at least one pharmaceutically acceptable excipient.
40. A pharmaceutical composition of claim 39, wherein said composition is formulated for oral administration.
41. A method of treating a cancer comprising a KRasG12C mutation in a patient having said cancer, the method comprising administering an effective amount of a compound of claim 1 or a crystal form of any one of claims 2-38.
42. A method for treating a cancer comprising a KRasG12C mutation in a patient having said cancer, the method comprising:
determining if the patient has the mutation; and

if the patient is determined to have the mutation, then administering to the patient an effective amount of a compound of claim 1 or a crystal form of any one of claims 2-38.
43. The method of claim 41 or 42, wherein the cancer comprises lung cancer, colorectal cancer, or pancreatic cancer.
44. The method of claim 41 or 42, wherein the cancer is lung cancer.
45. The method of claim 44, wherein the lung cancer is non-small cell lung cancer (NSCLC).
46. The method of claim 41 or 42, wherein the cancer is colorectal cancer.
47. The method of claim 41 or 42, wherein the cancer is pancreatic cancer.
48. A method for inhibiting proliferation of a cell population wherein the cell

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population comprises a KRasG12C mutation, the method comprising contacting the cell population with a compound of claim 1 or a crystal form of any one of claims 2-38.

49. A method for inhibiting tumor metastasis comprising administering to an individual in need thereof a therapeutically effective amount of the compound of claim 1 or a crystal form of any one of claims 2-38.

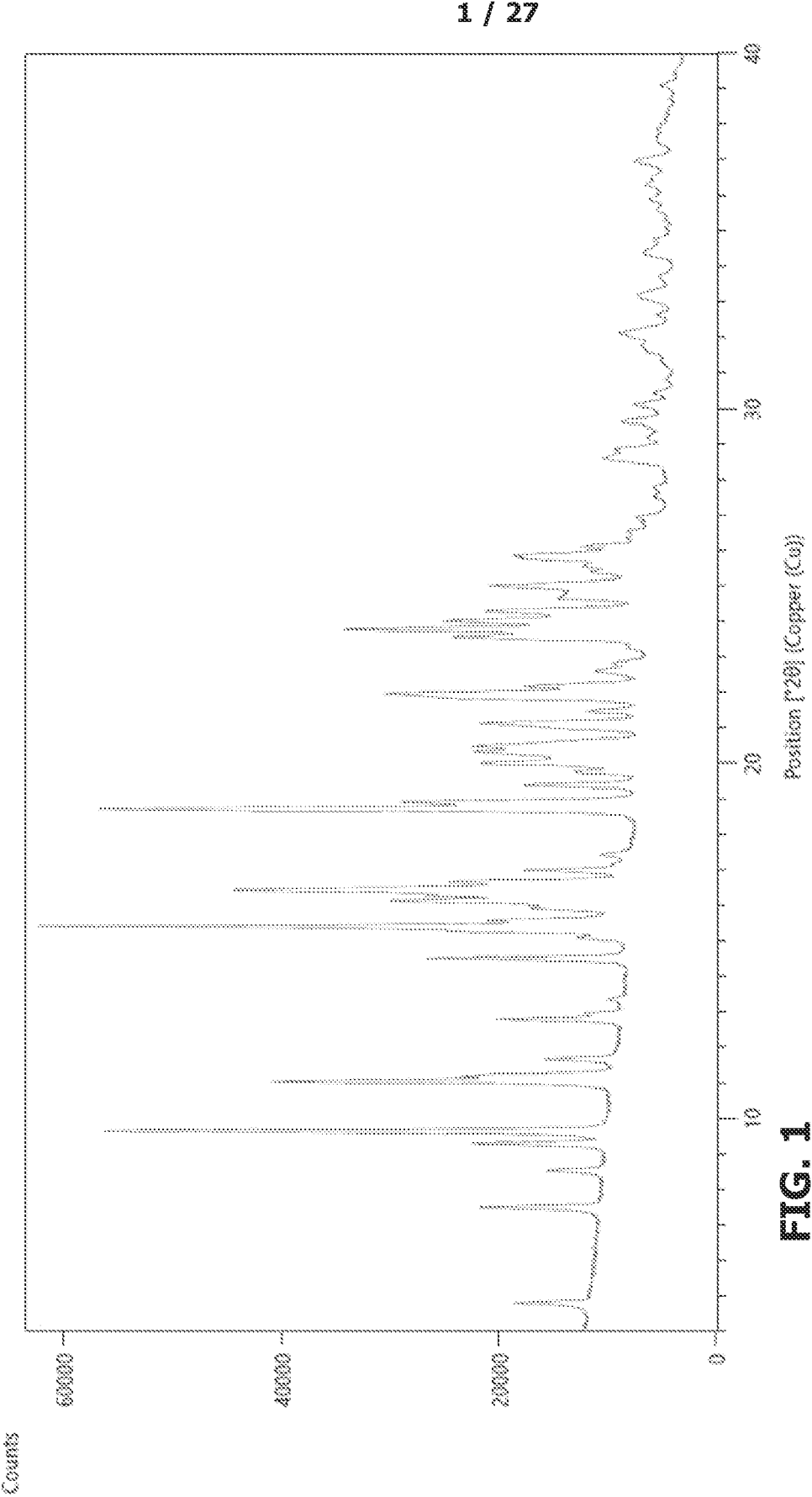
50. Use of a compound of claim 1 or a crystal form of any one of claims 2-38 in the manufacture of a medicament for treating a cancer.

51. The use of claim 50, wherein the cancer comprises lung cancer, colorectal cancer, or pancreatic cancer.

52. The compound of claim 1 or a crystal form of any one of claims 2-38 for use in a method of treating cancer.

53. The compound of compound of claim 52, wherein the cancer comprises lung cancer, colorectal cancer, or pancreatic cancer.

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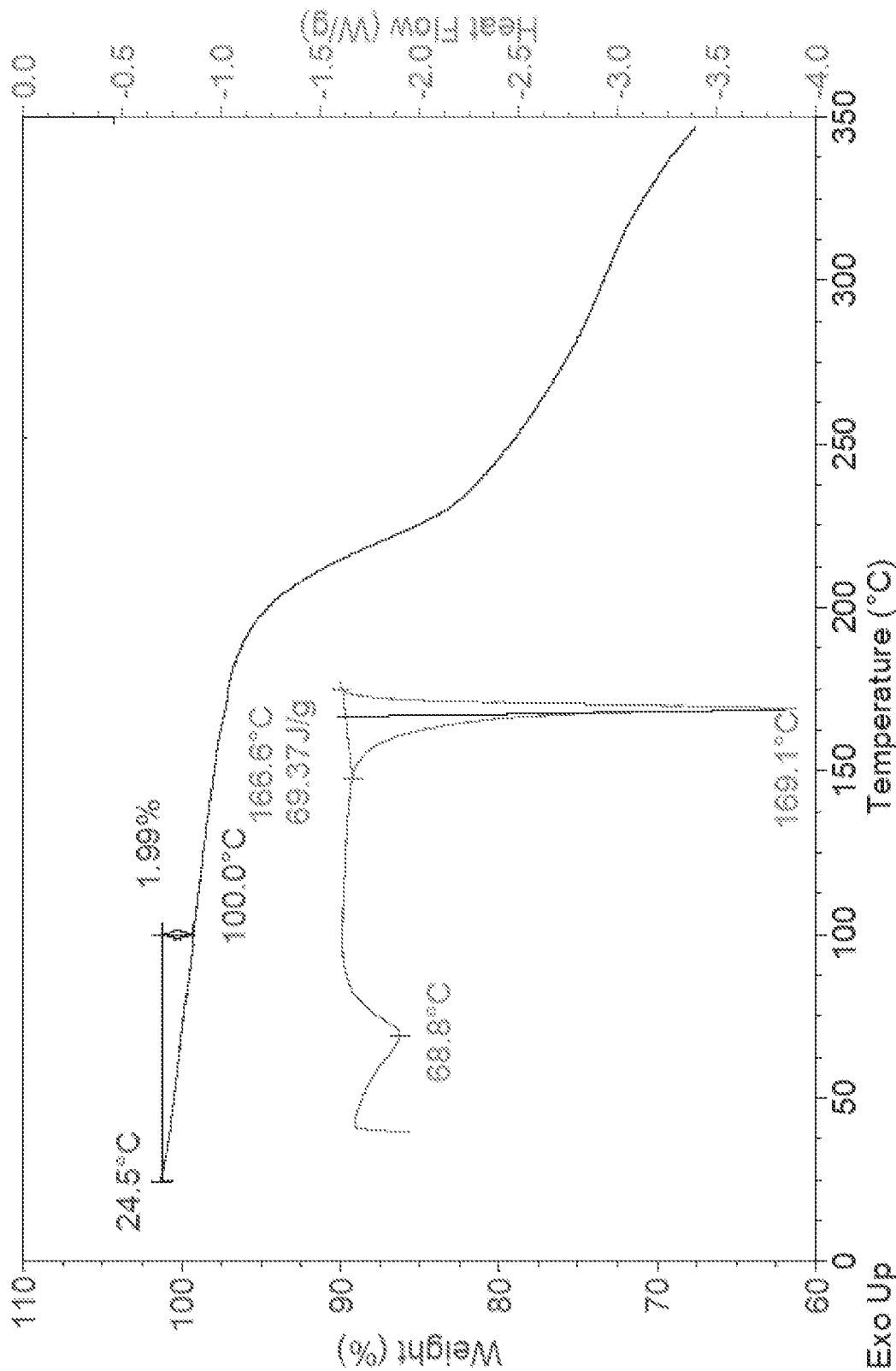


FIG. 2

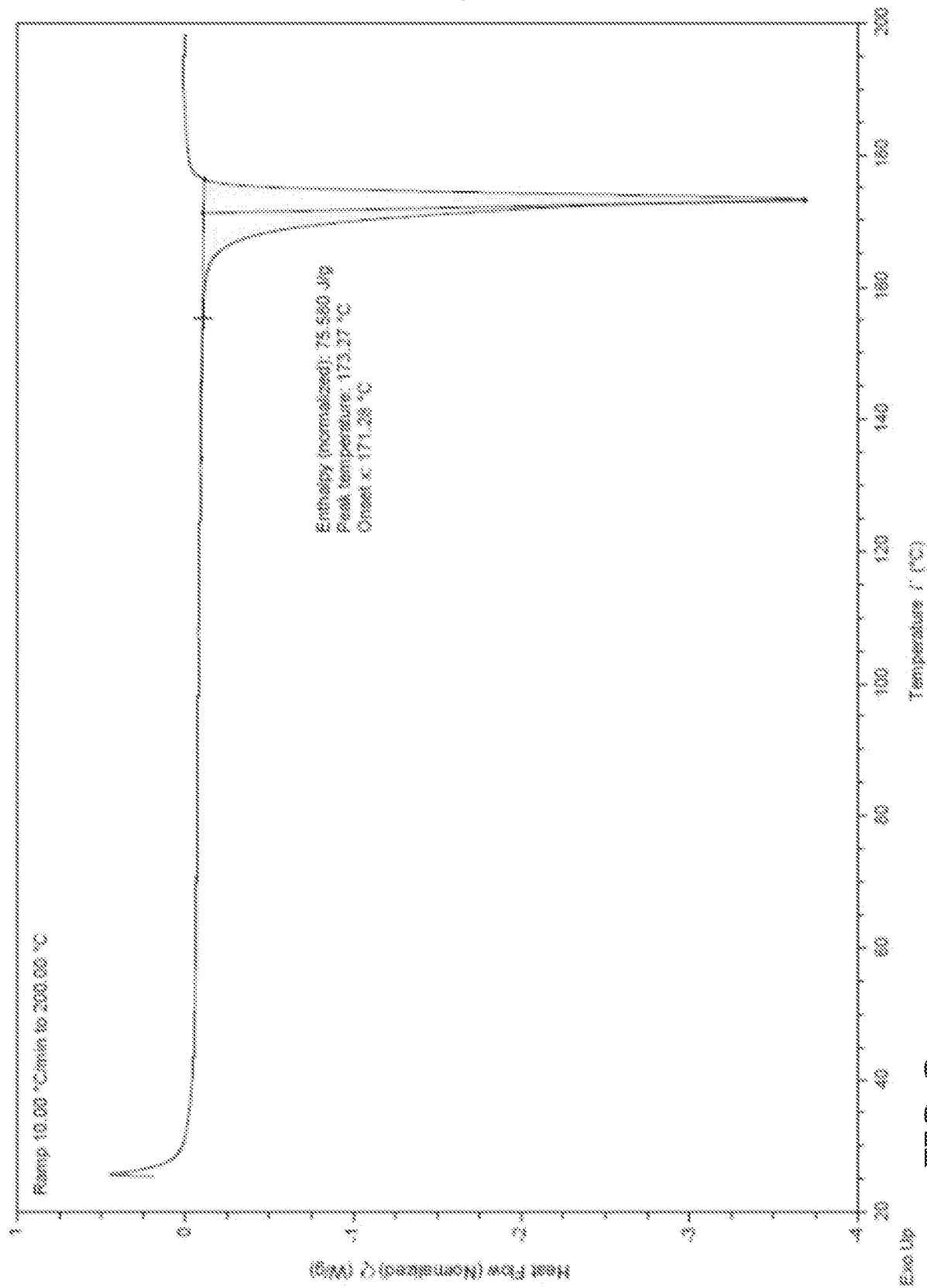


FIG. 3

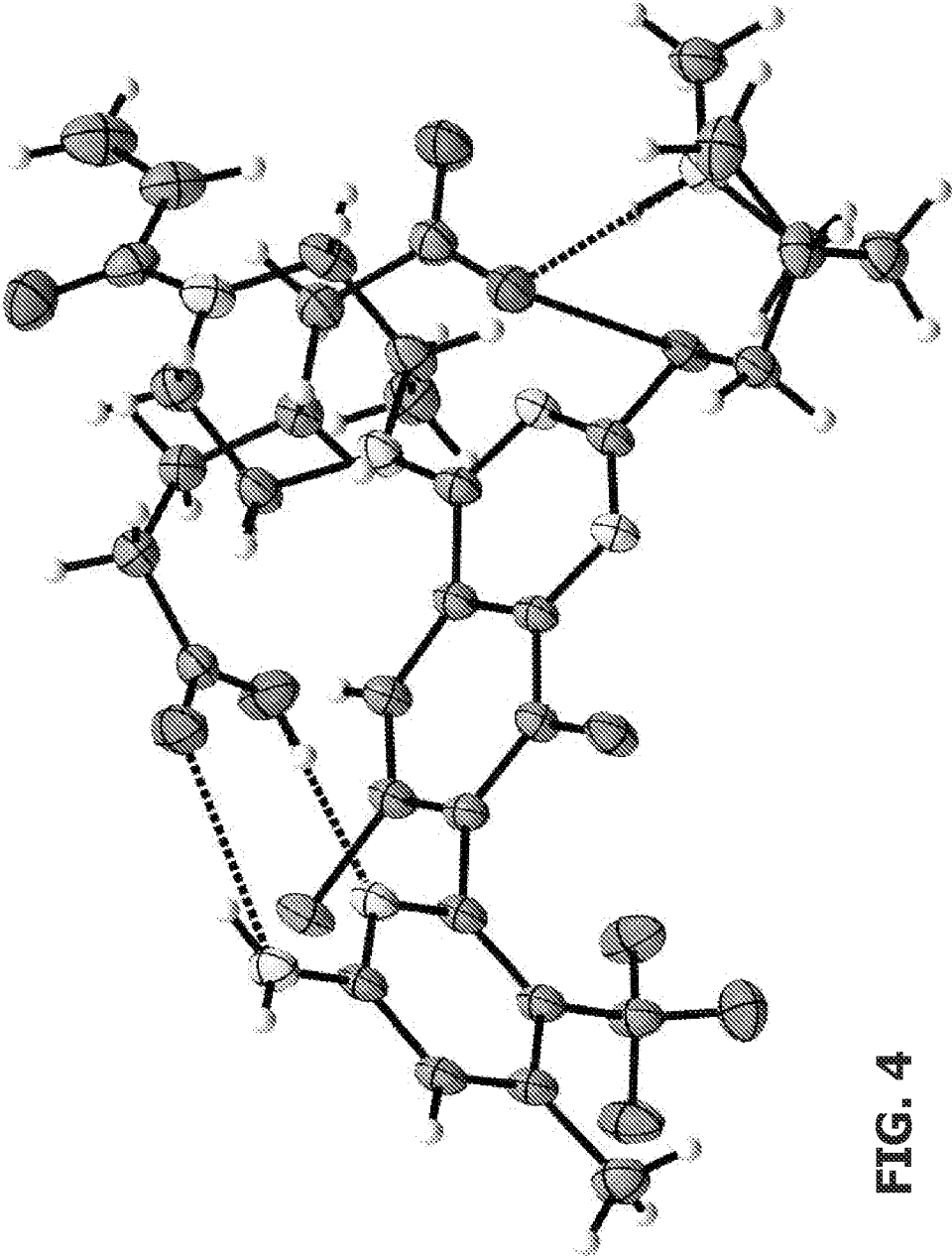
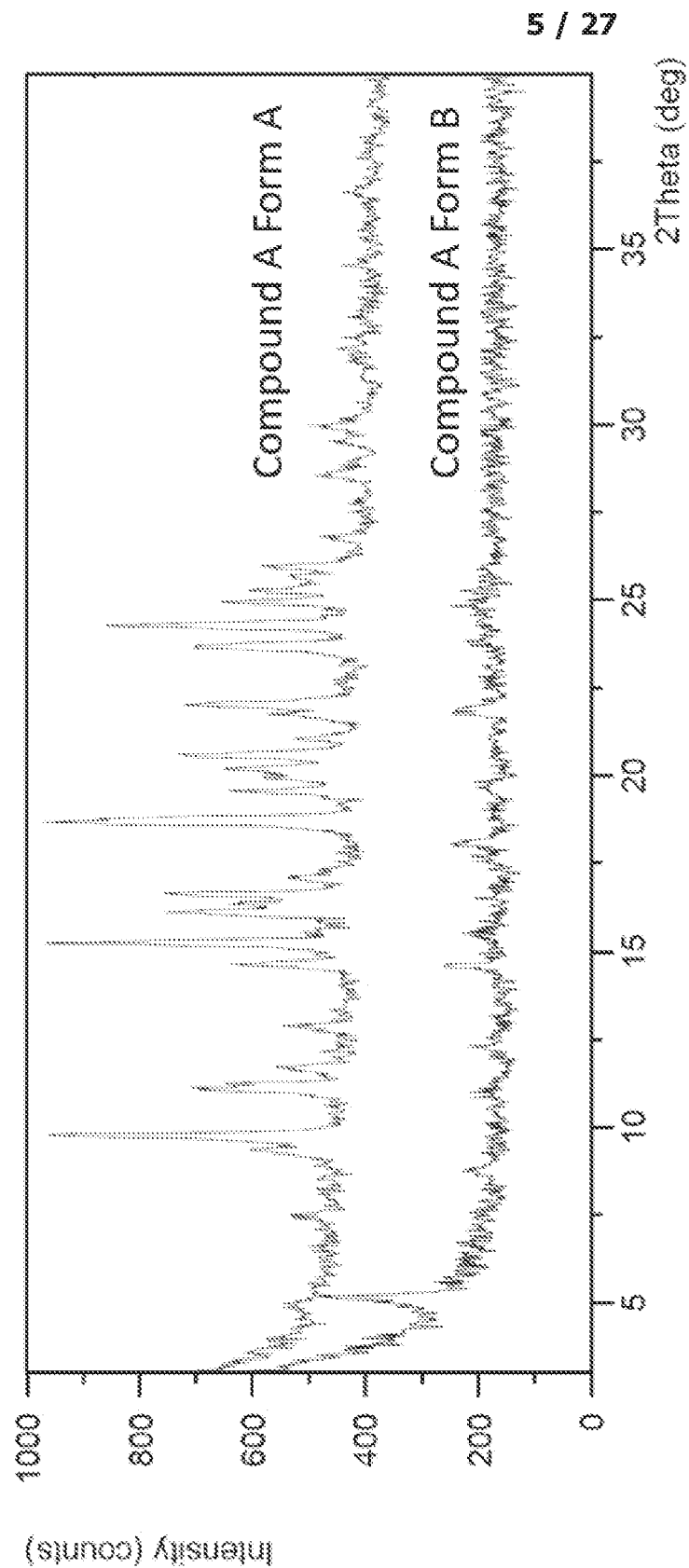


FIG. 4

**FIG. 5**

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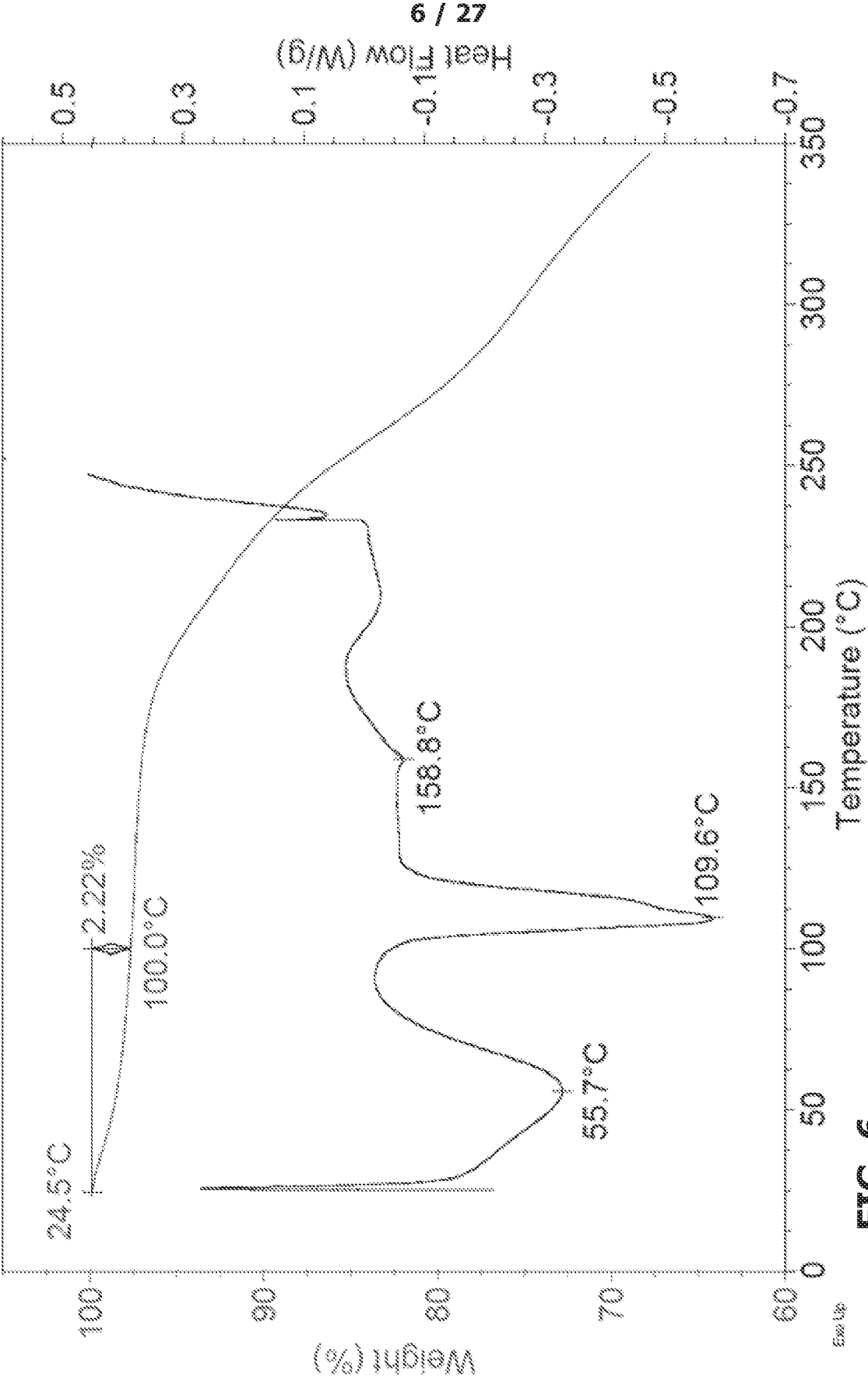
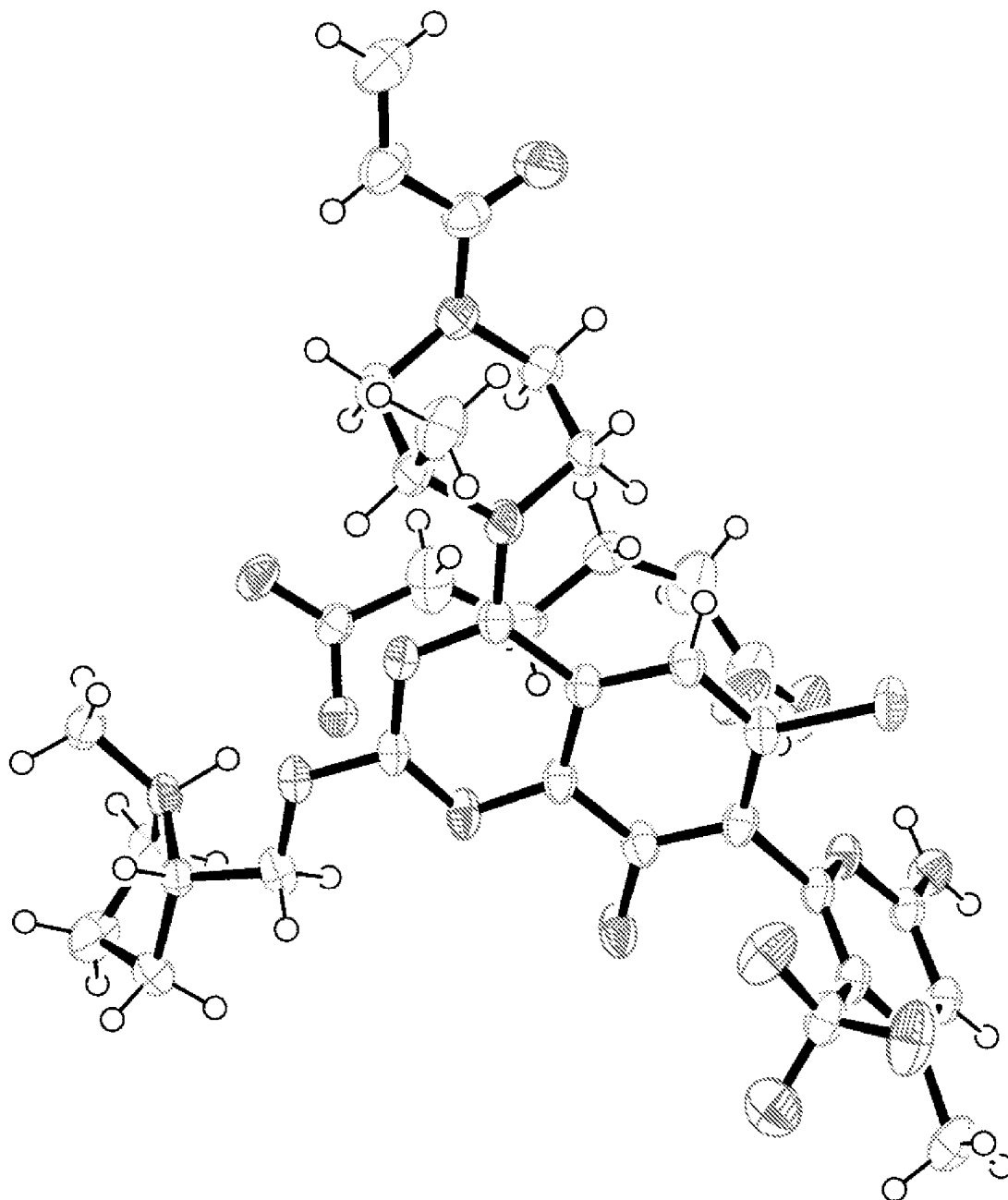


FIG. 6

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**FIG. 7**

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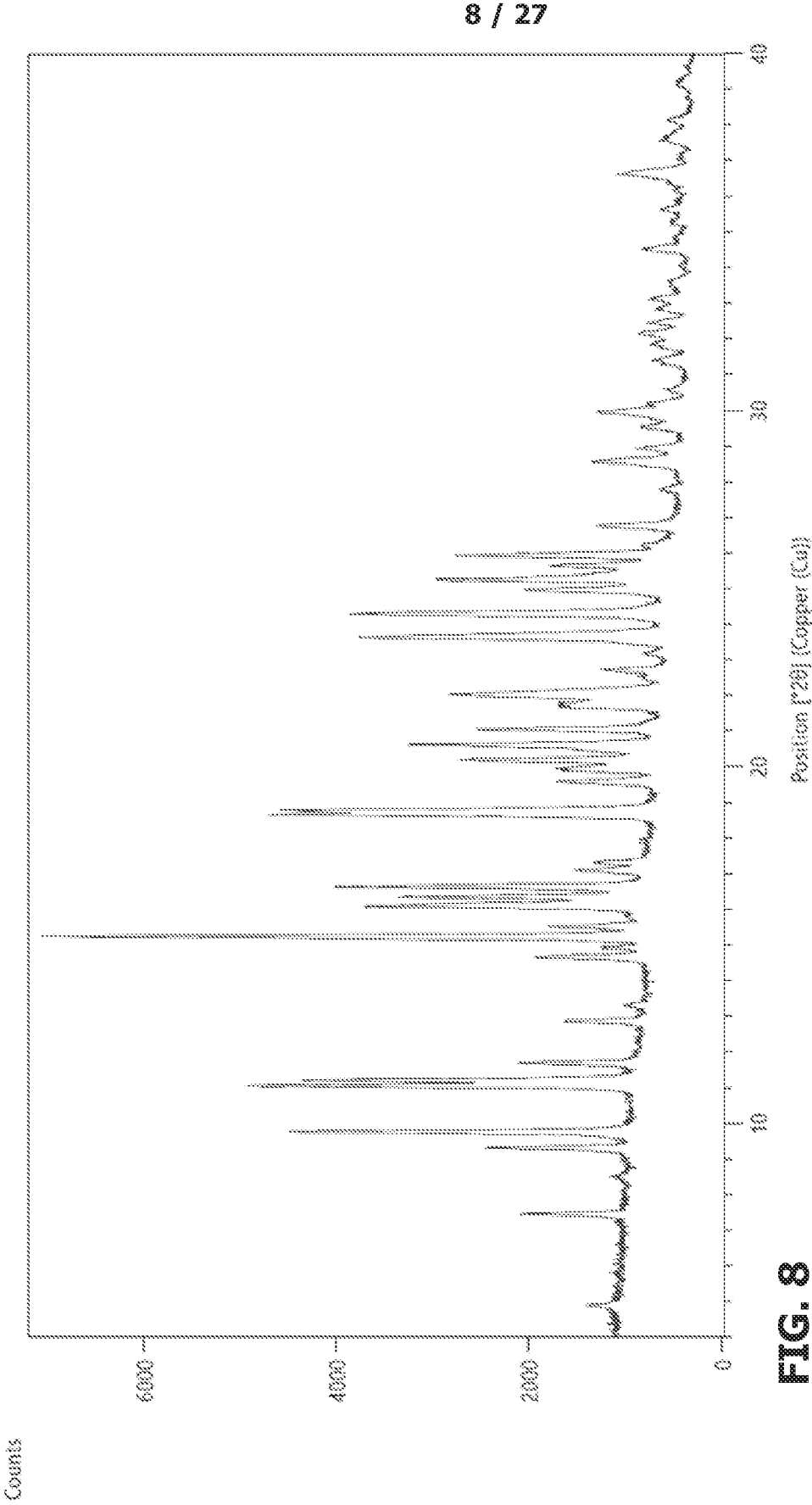


FIG. 8

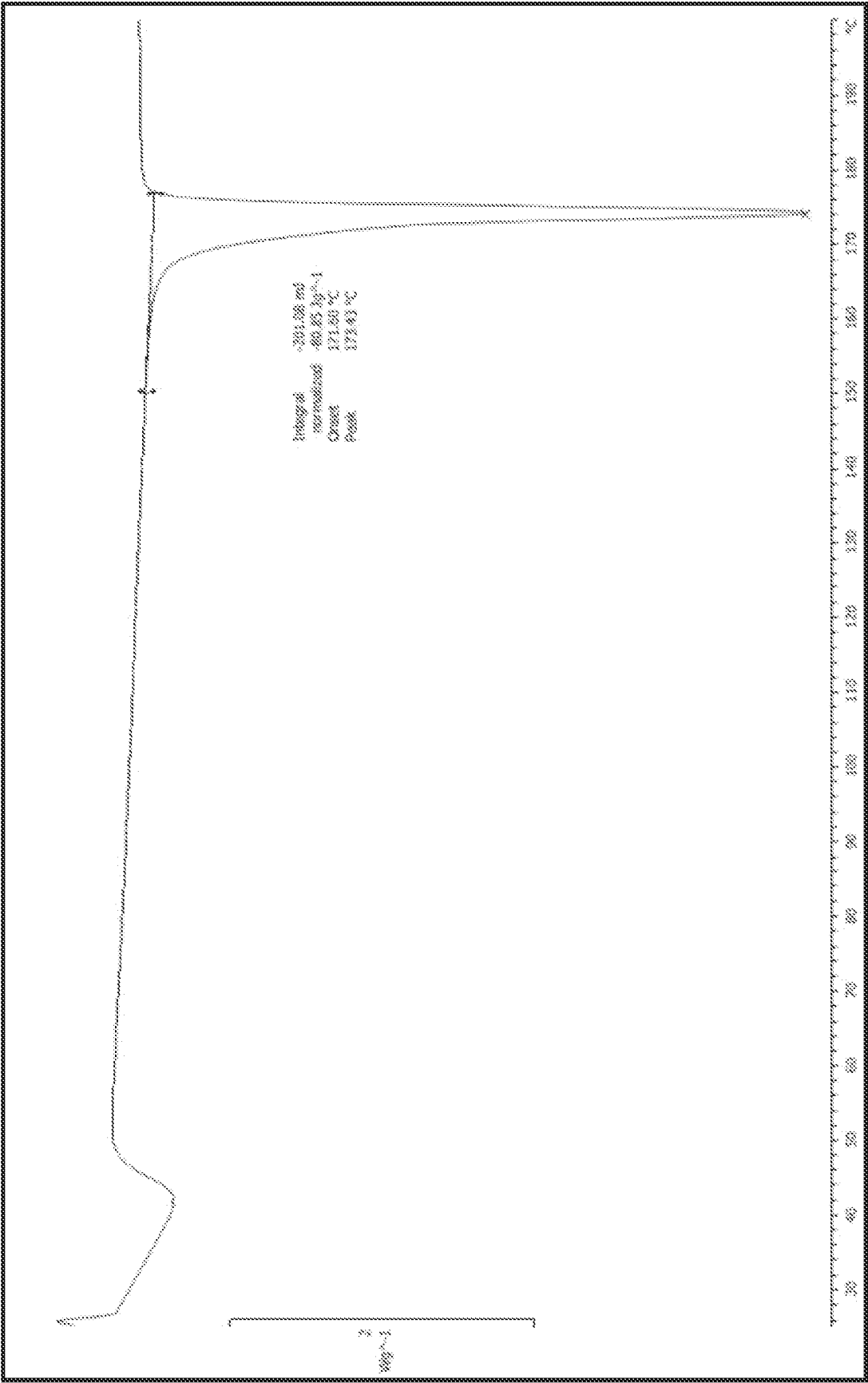


FIG. 9

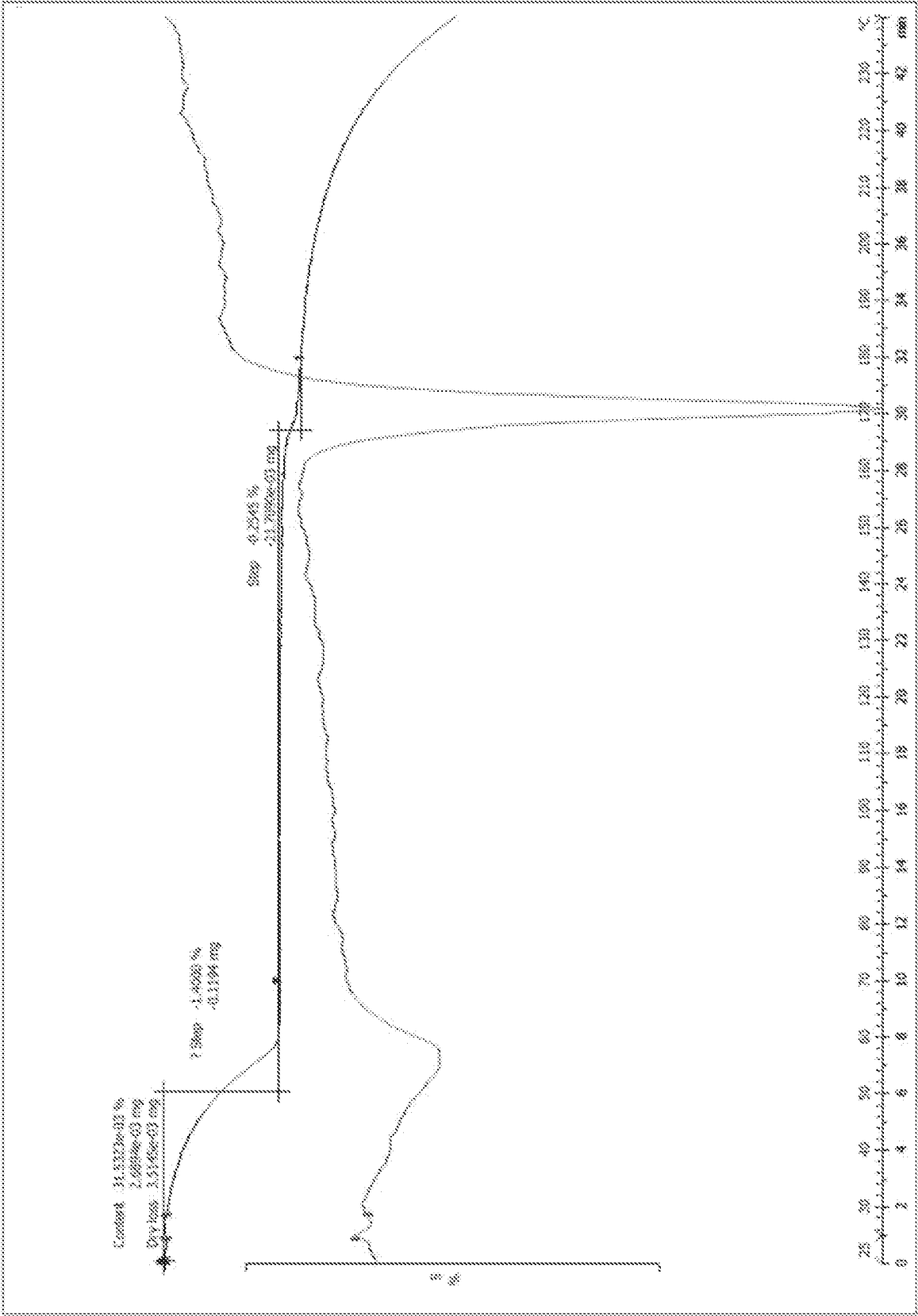
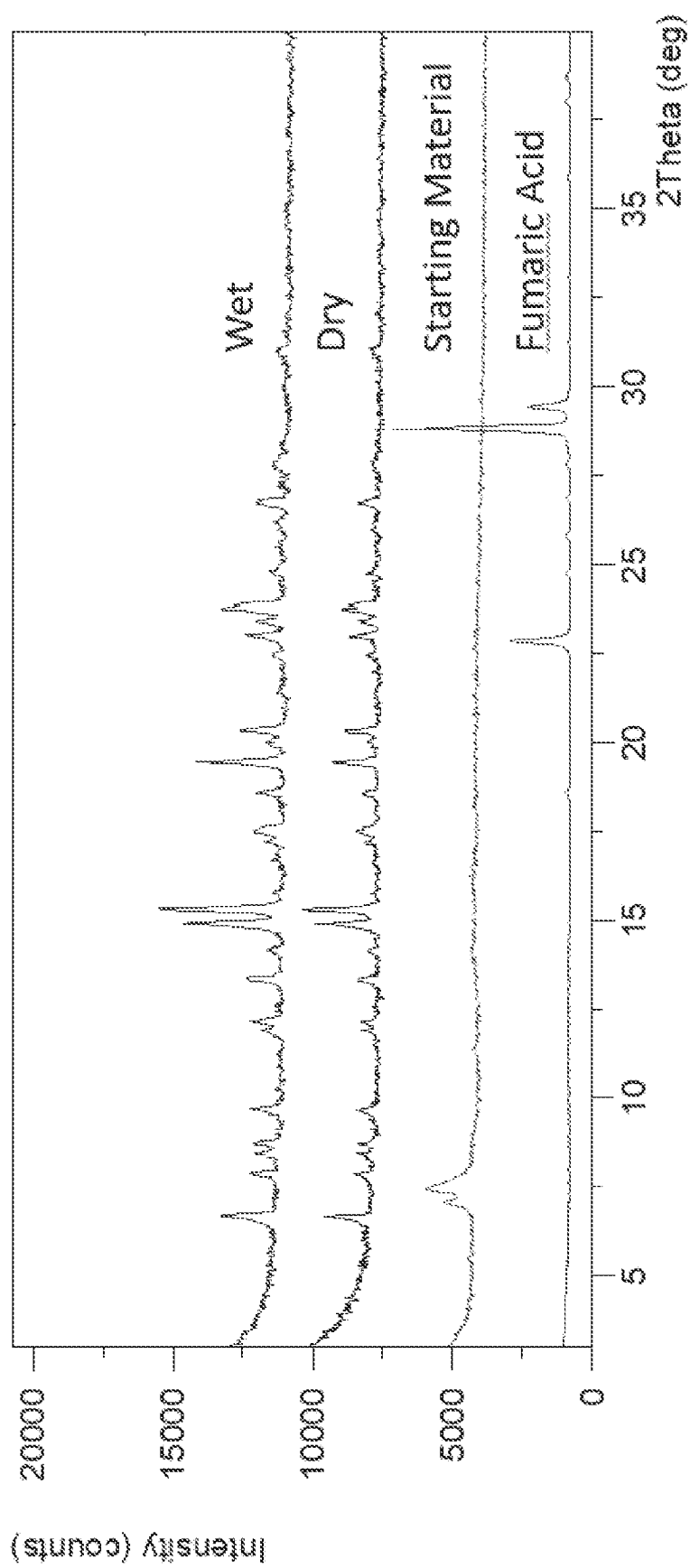


FIG. 10

**FIG. 11**

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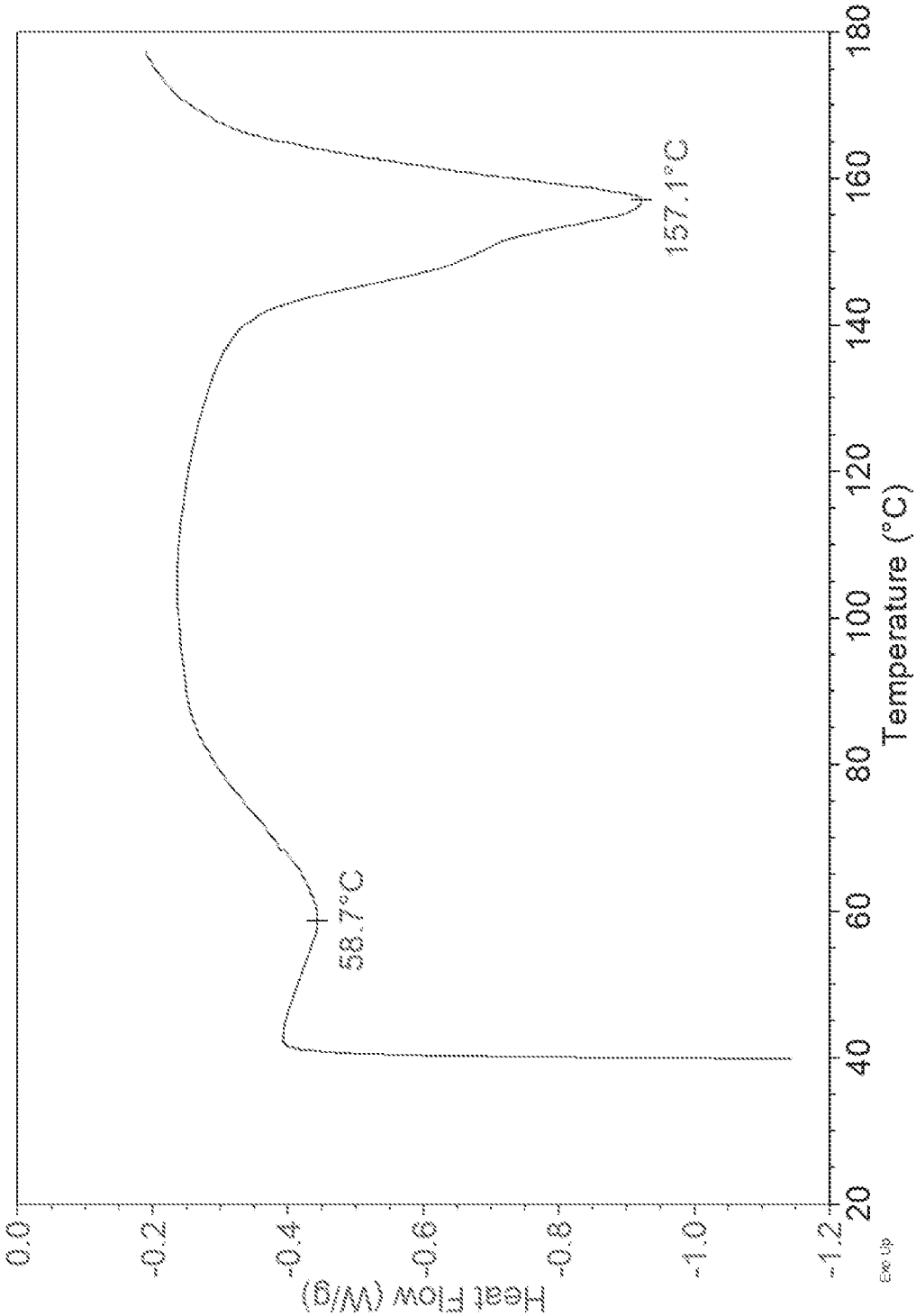


FIG. 12

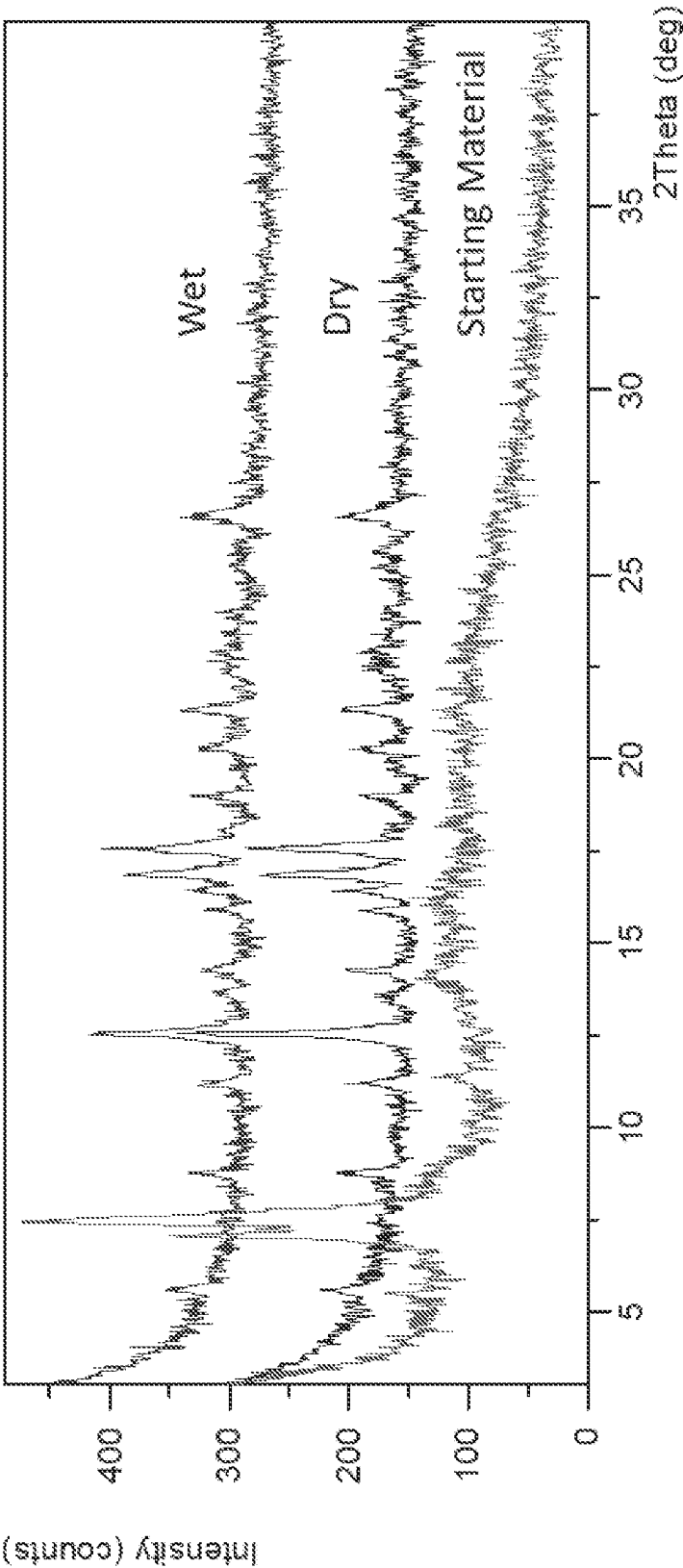
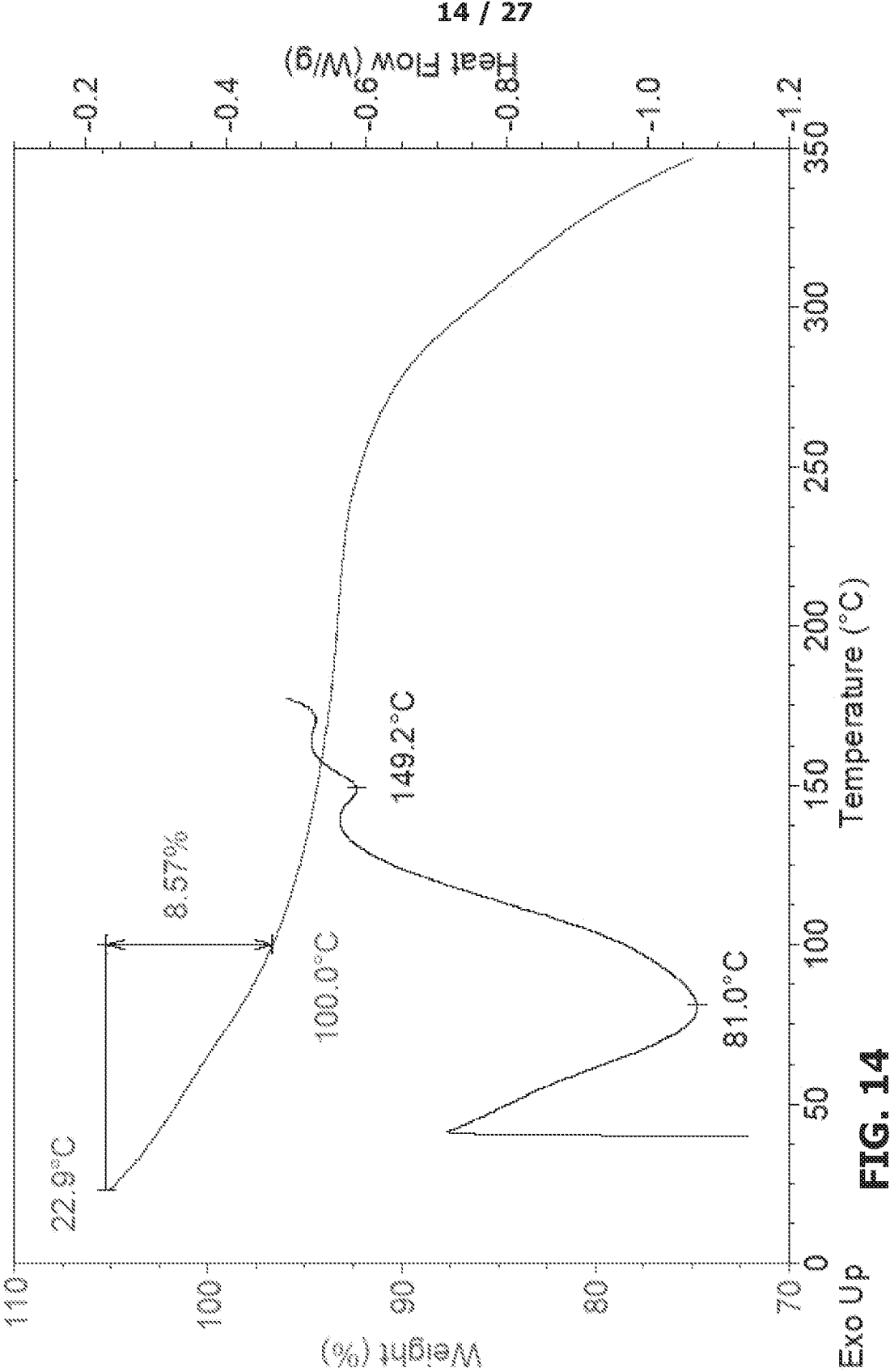
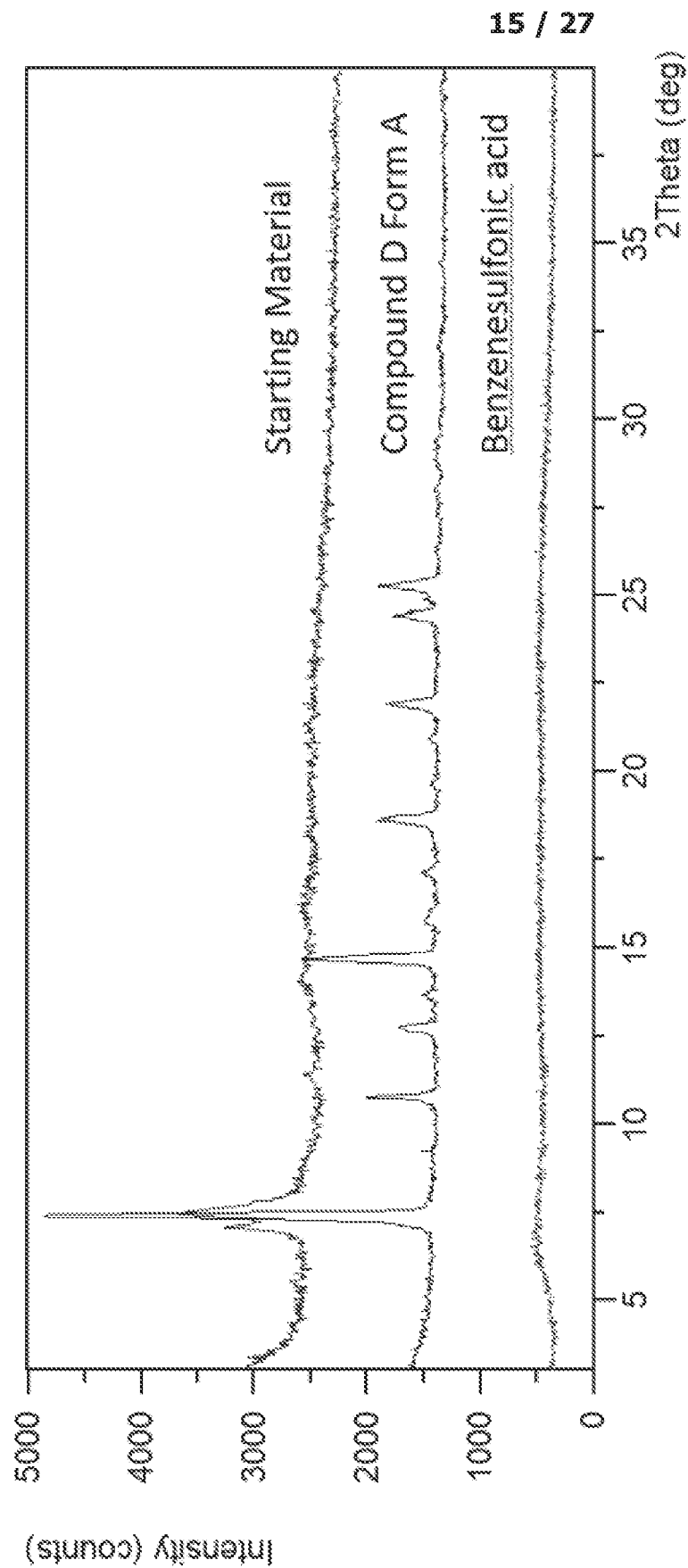


FIG. 13

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Exo Up
FIG. 14

**FIG. 15**

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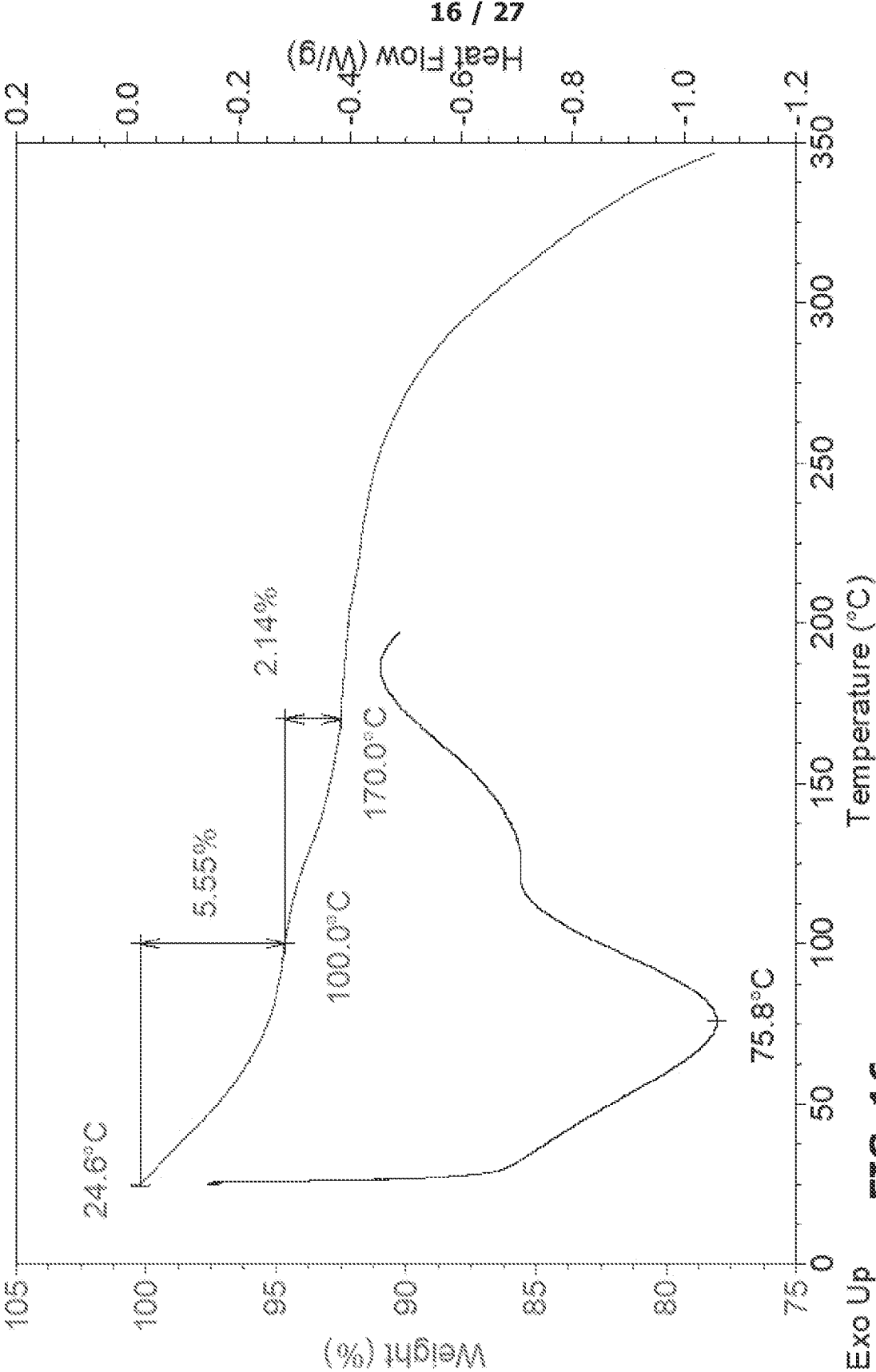


FIG. 16

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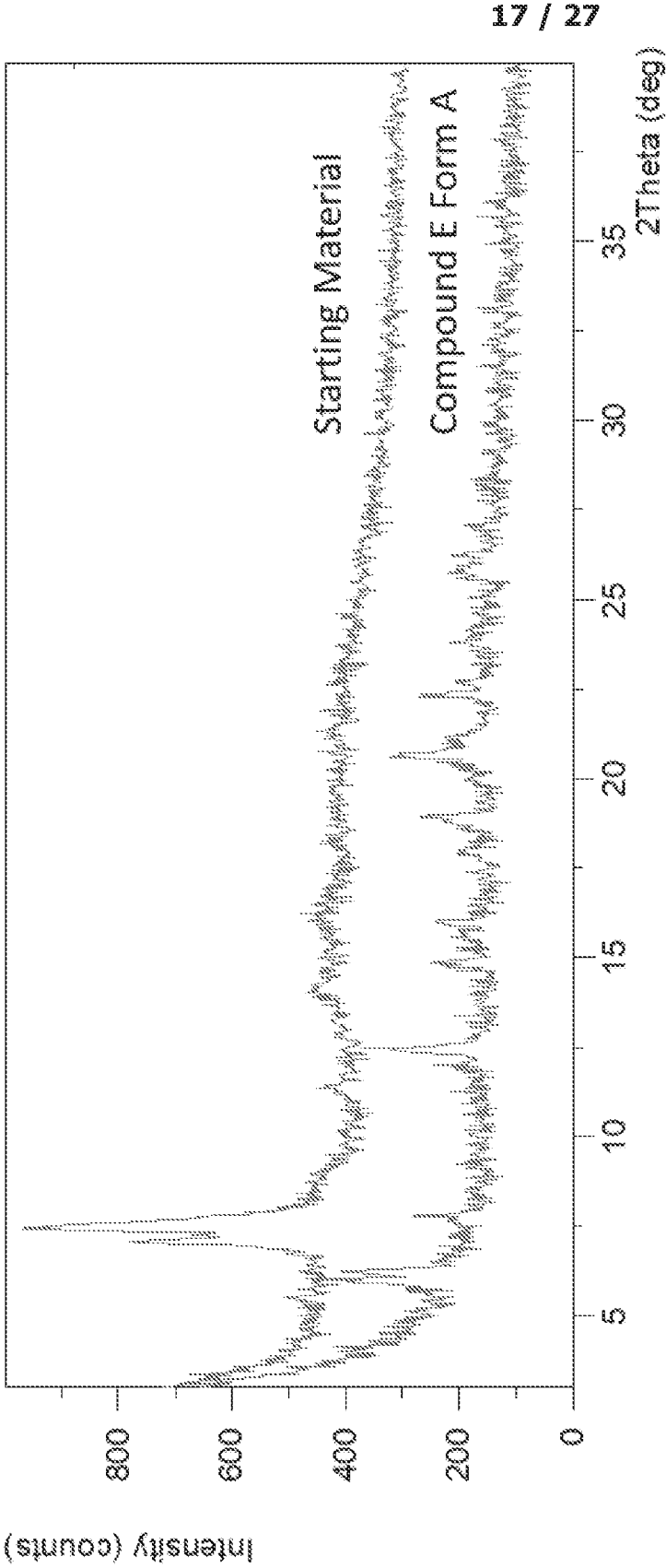
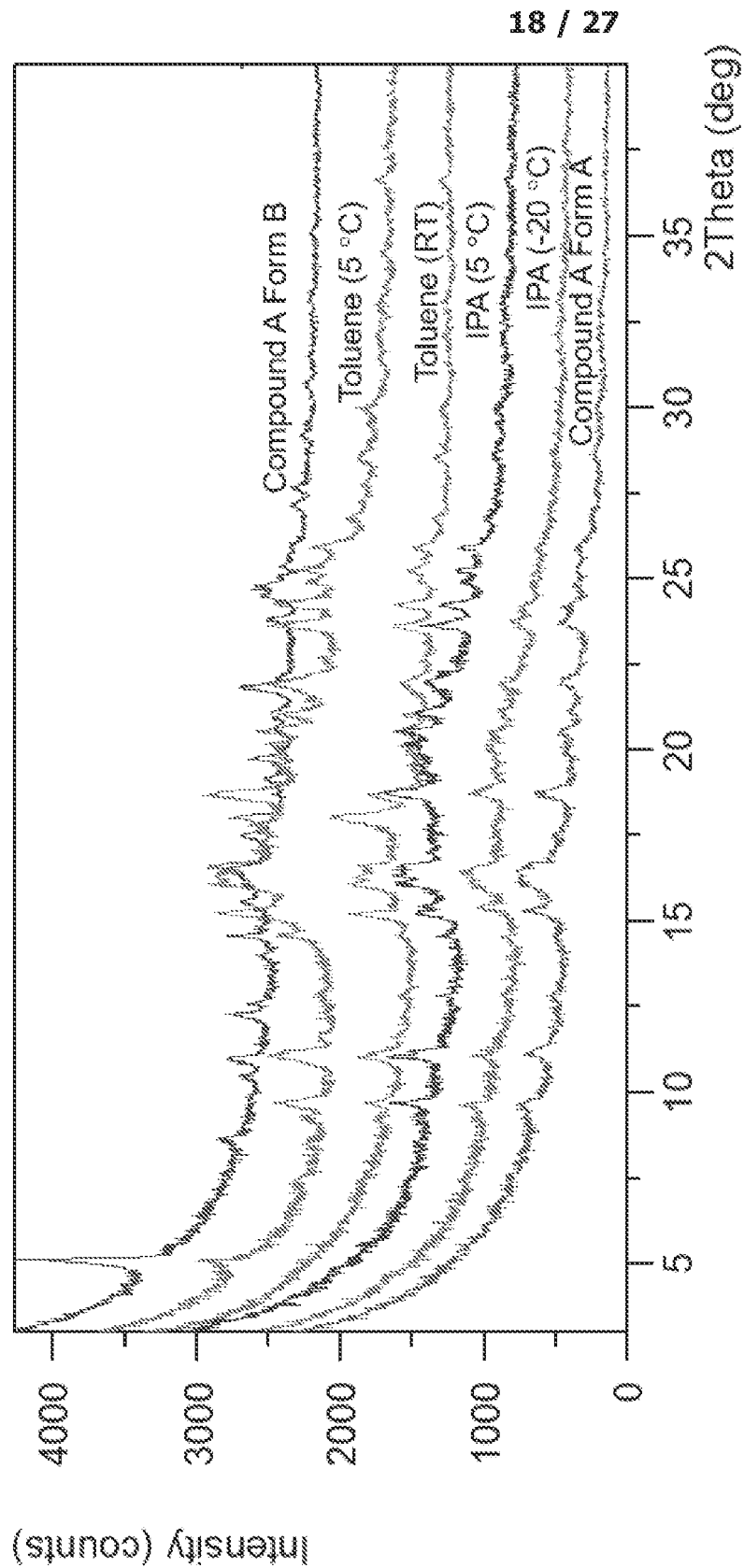
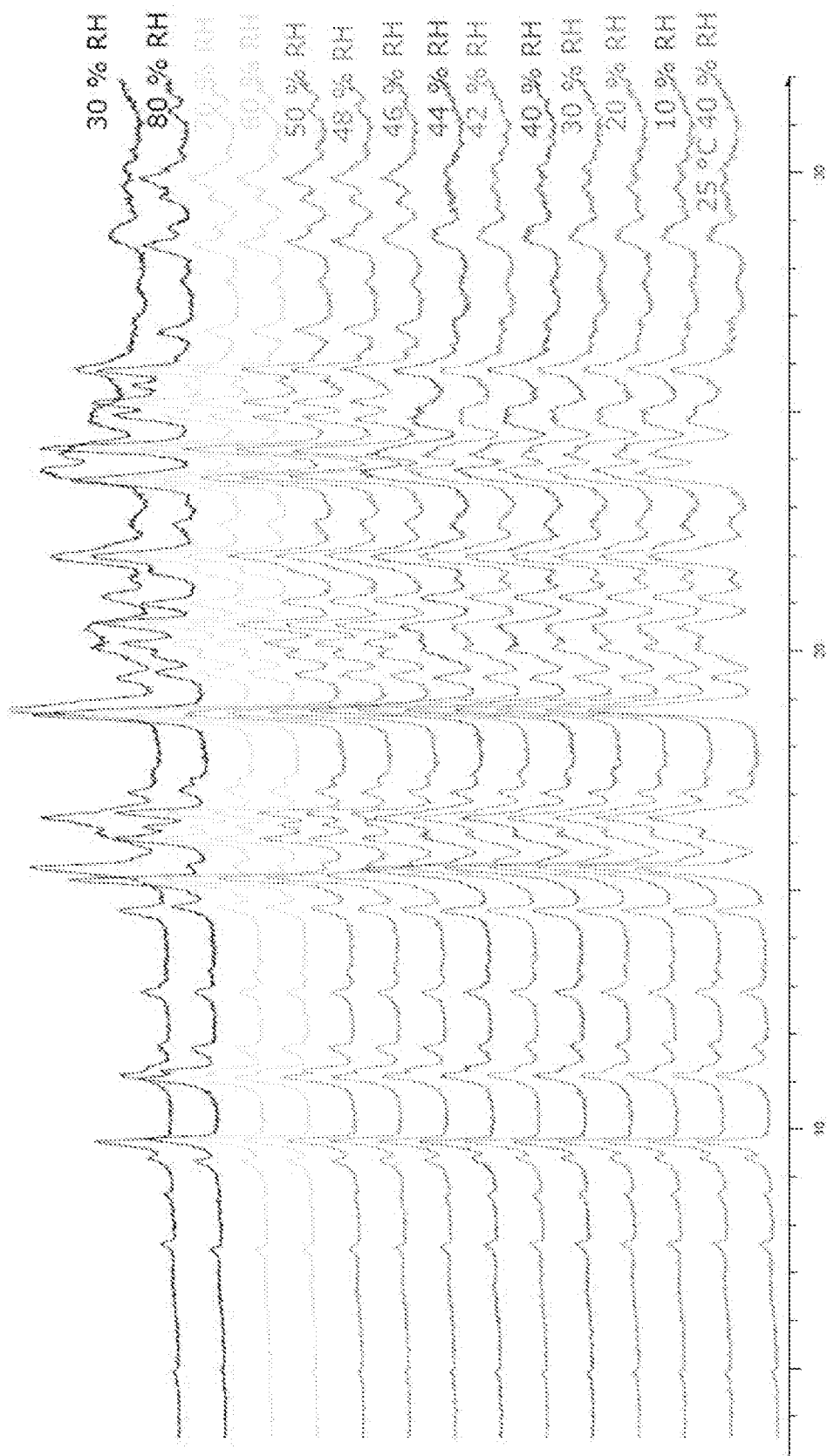


FIG. 17

**FIG. 18**

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2-Theta - Scale

FIG. 19

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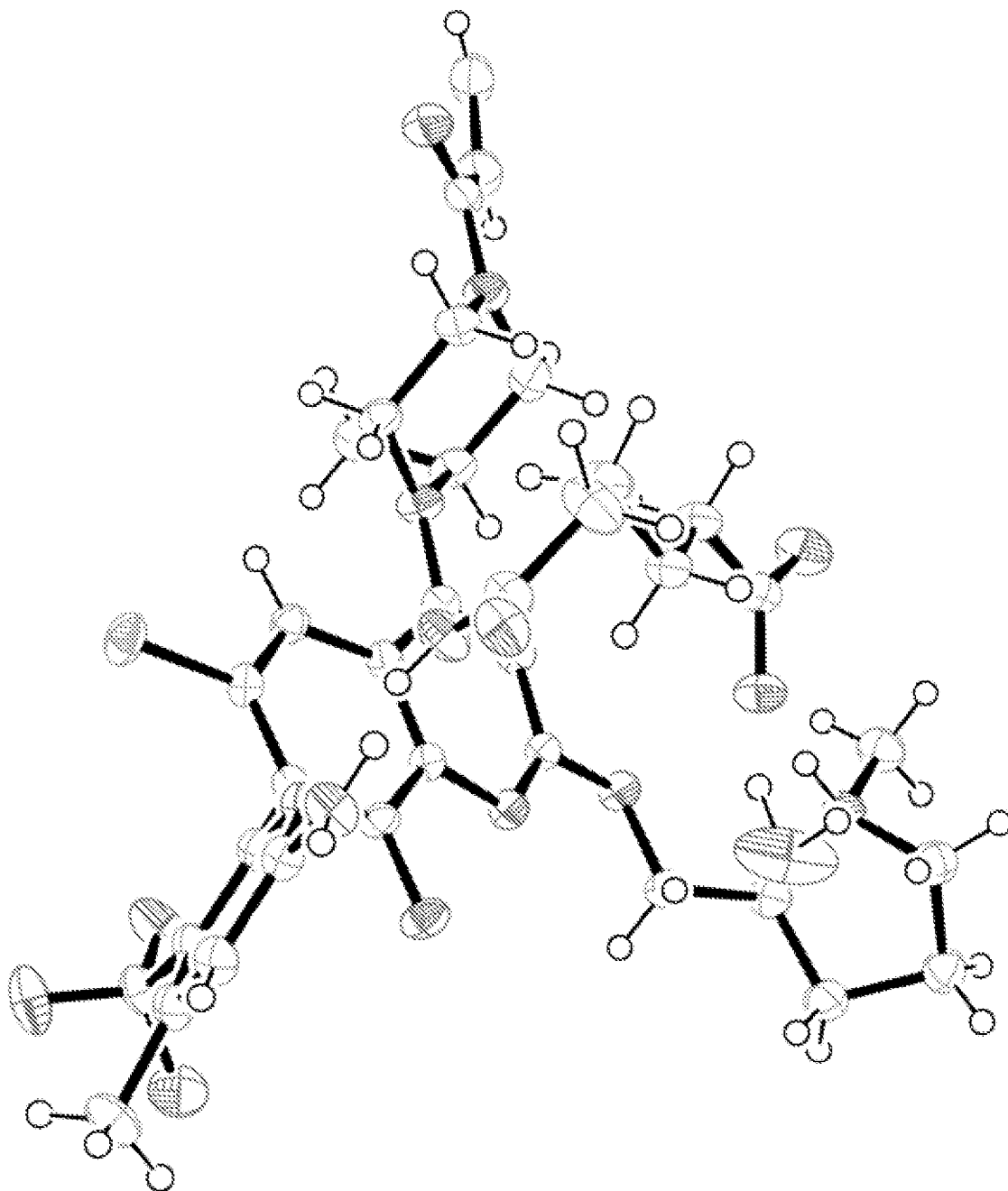


FIG. 20

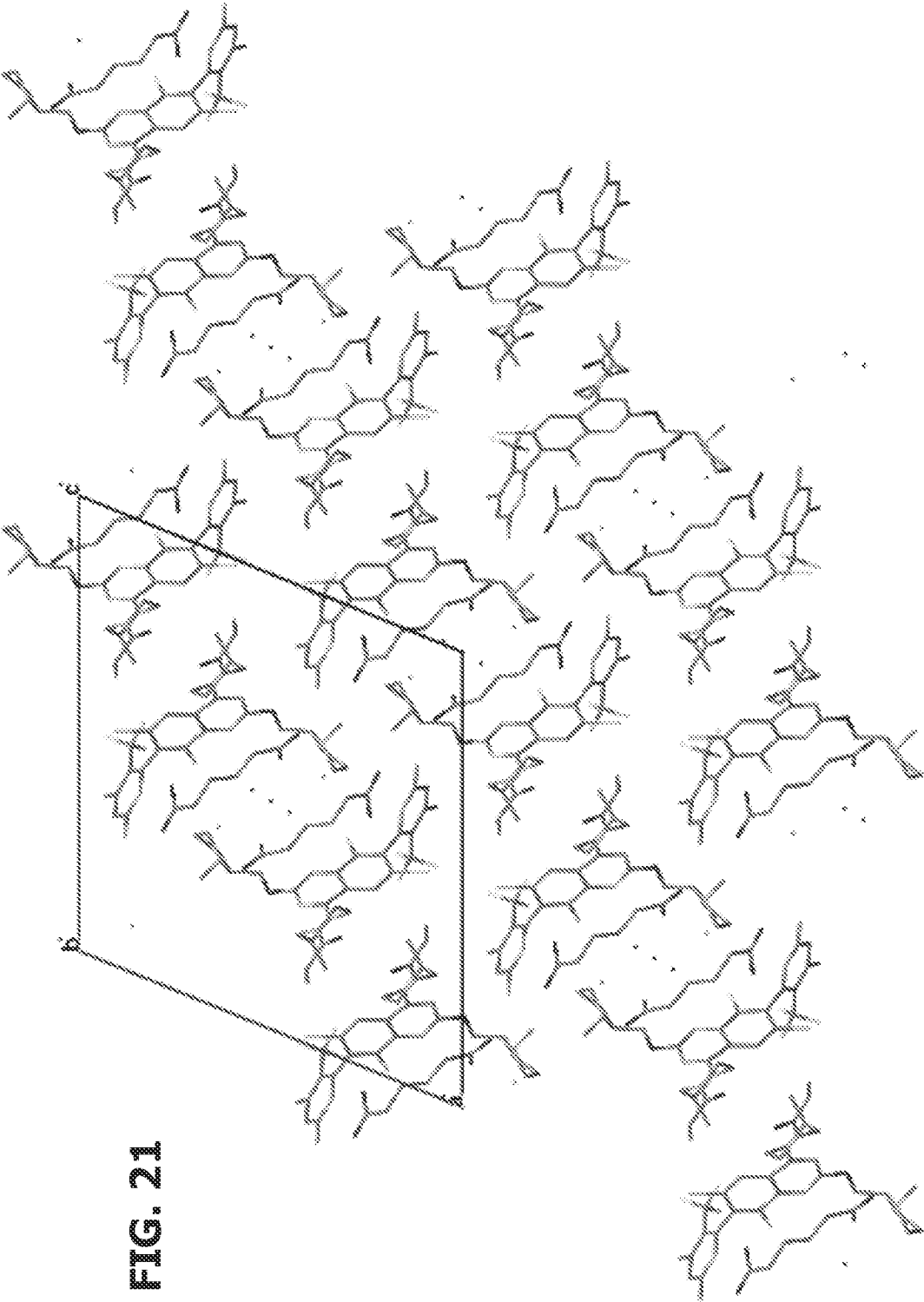


FIG. 21

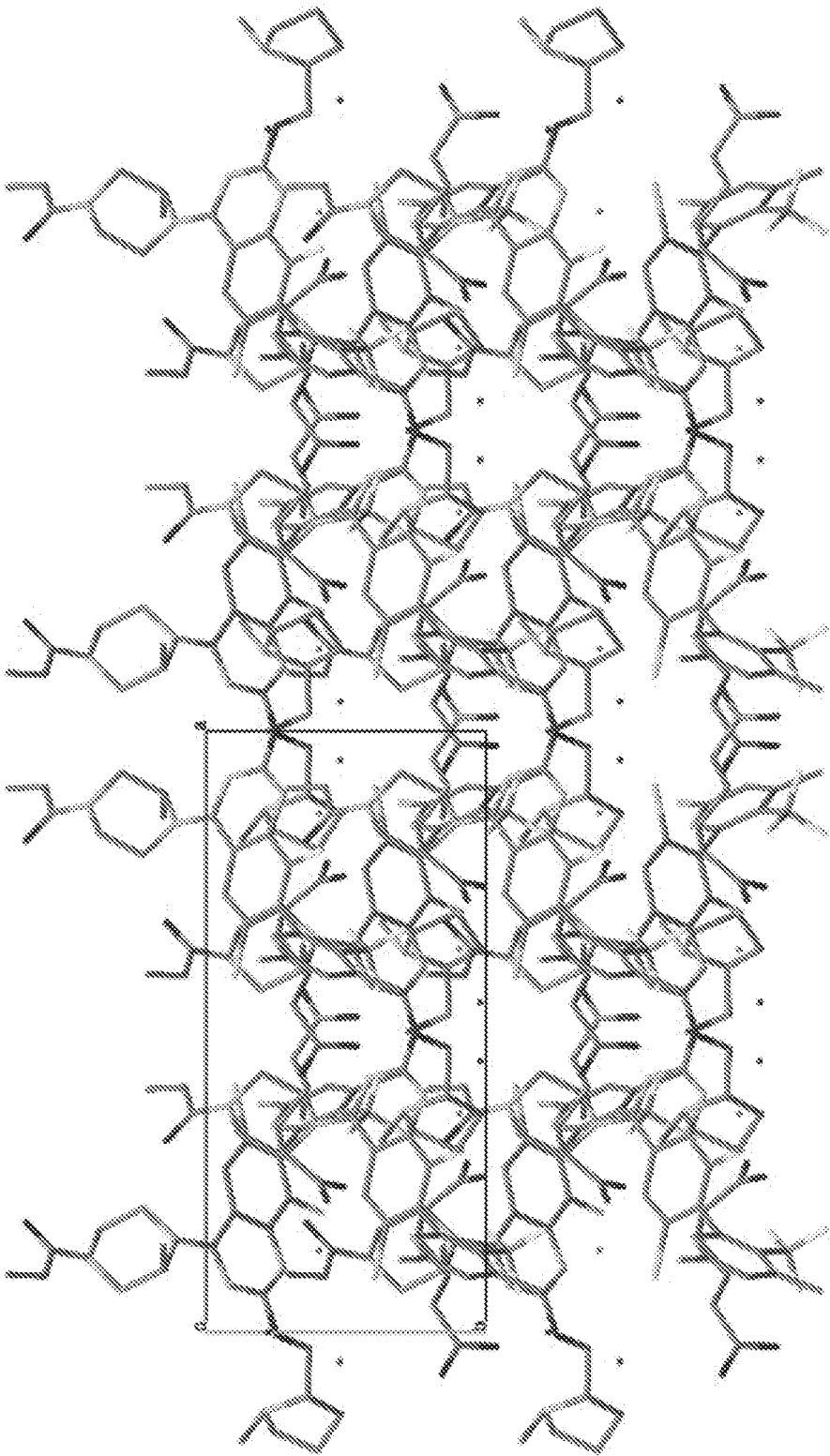
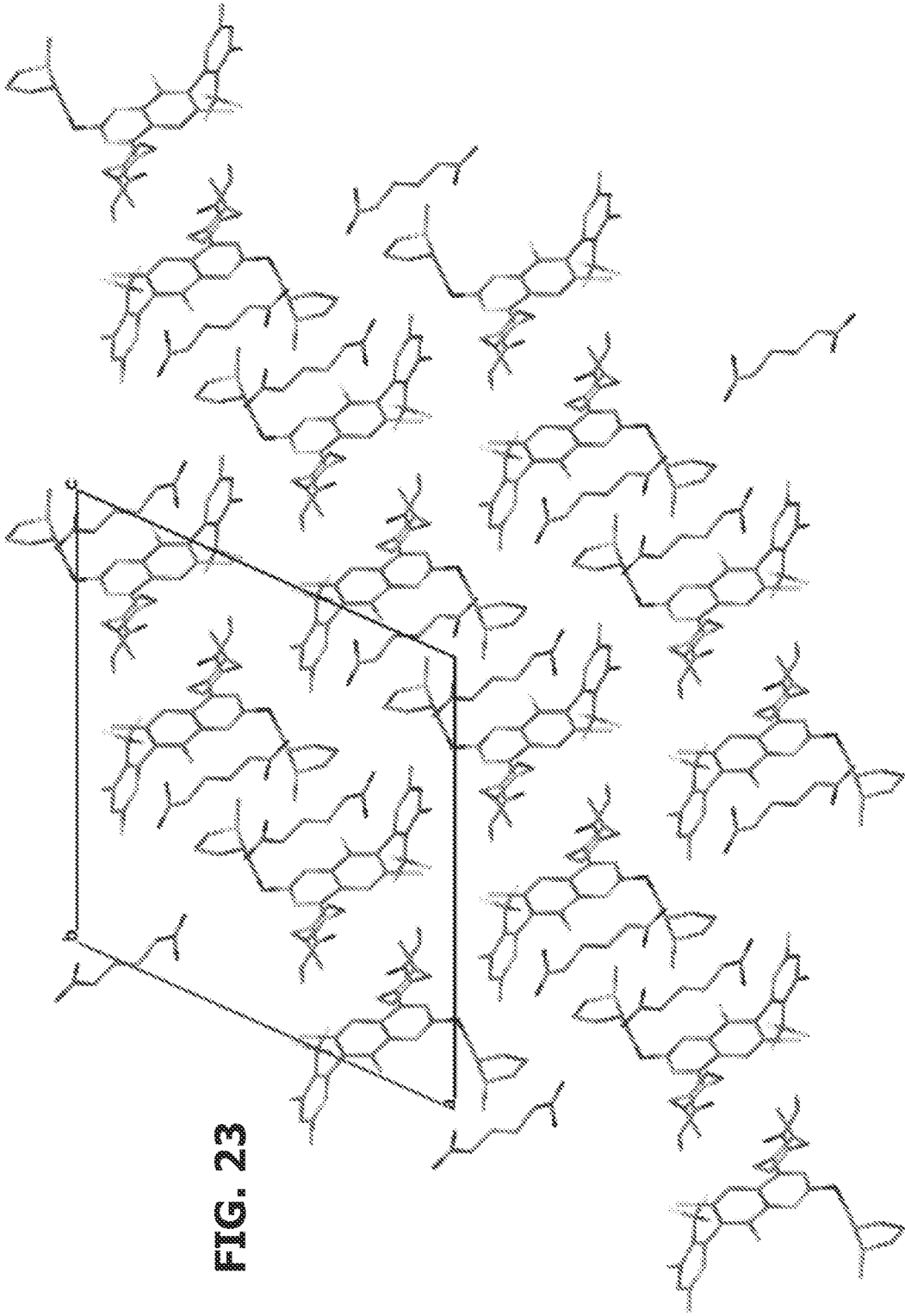


FIG. 22



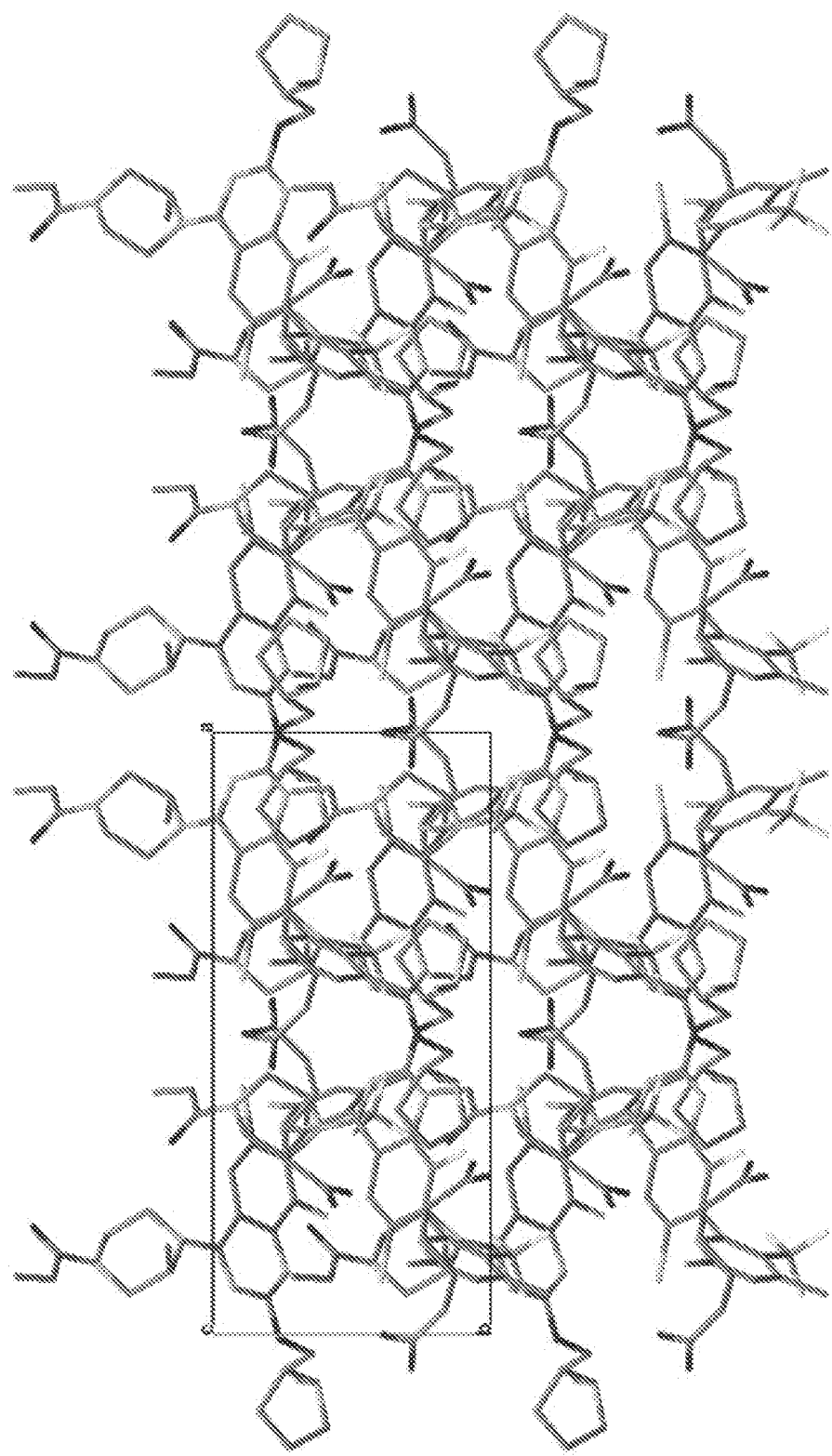


FIG. 24

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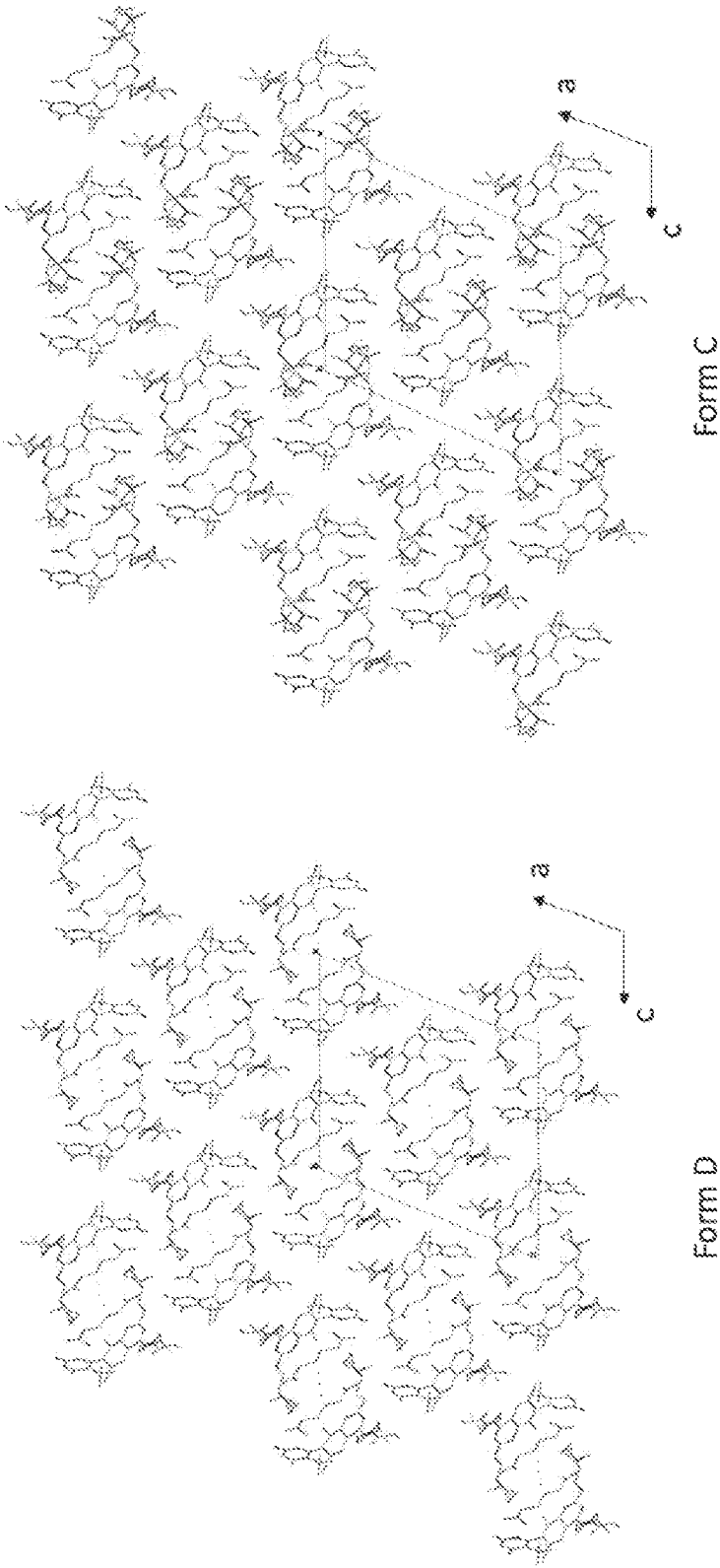


FIG. 25

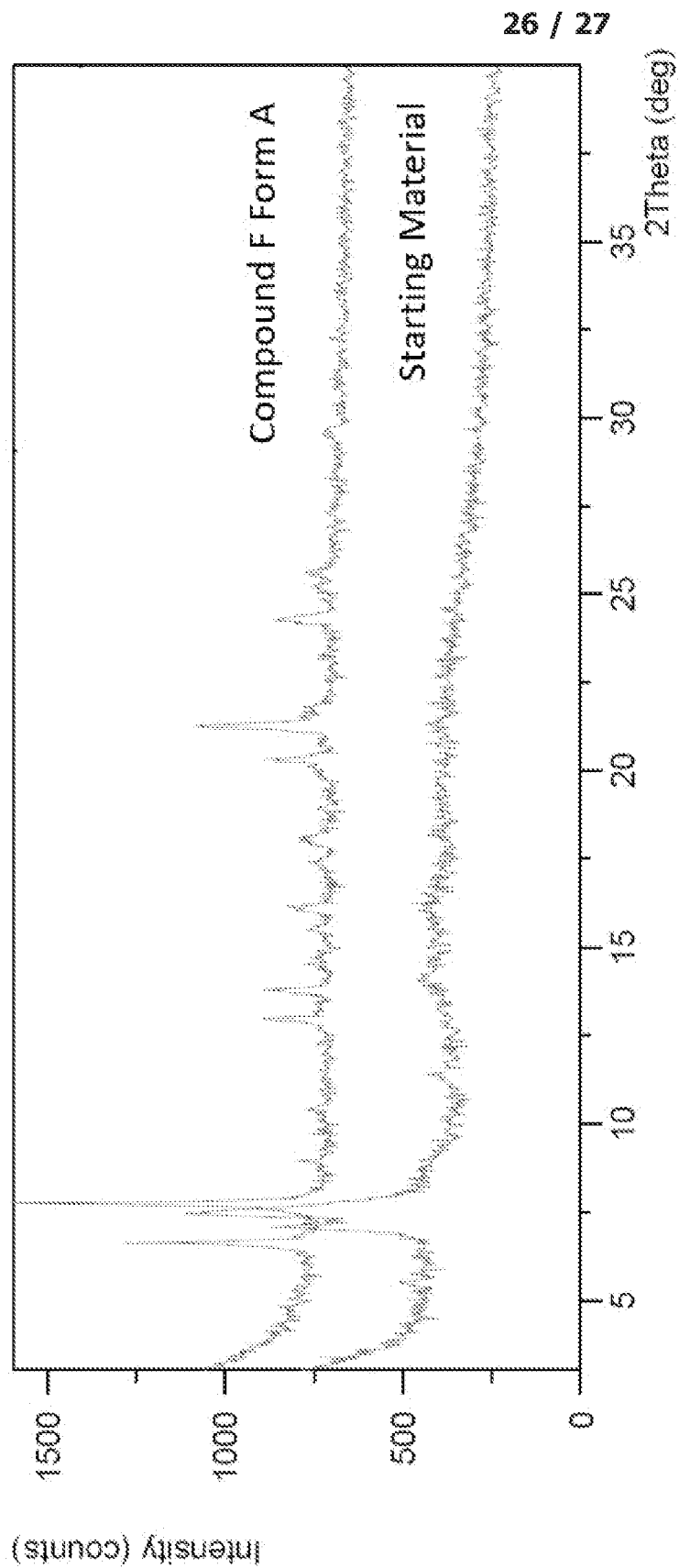


FIG. 26

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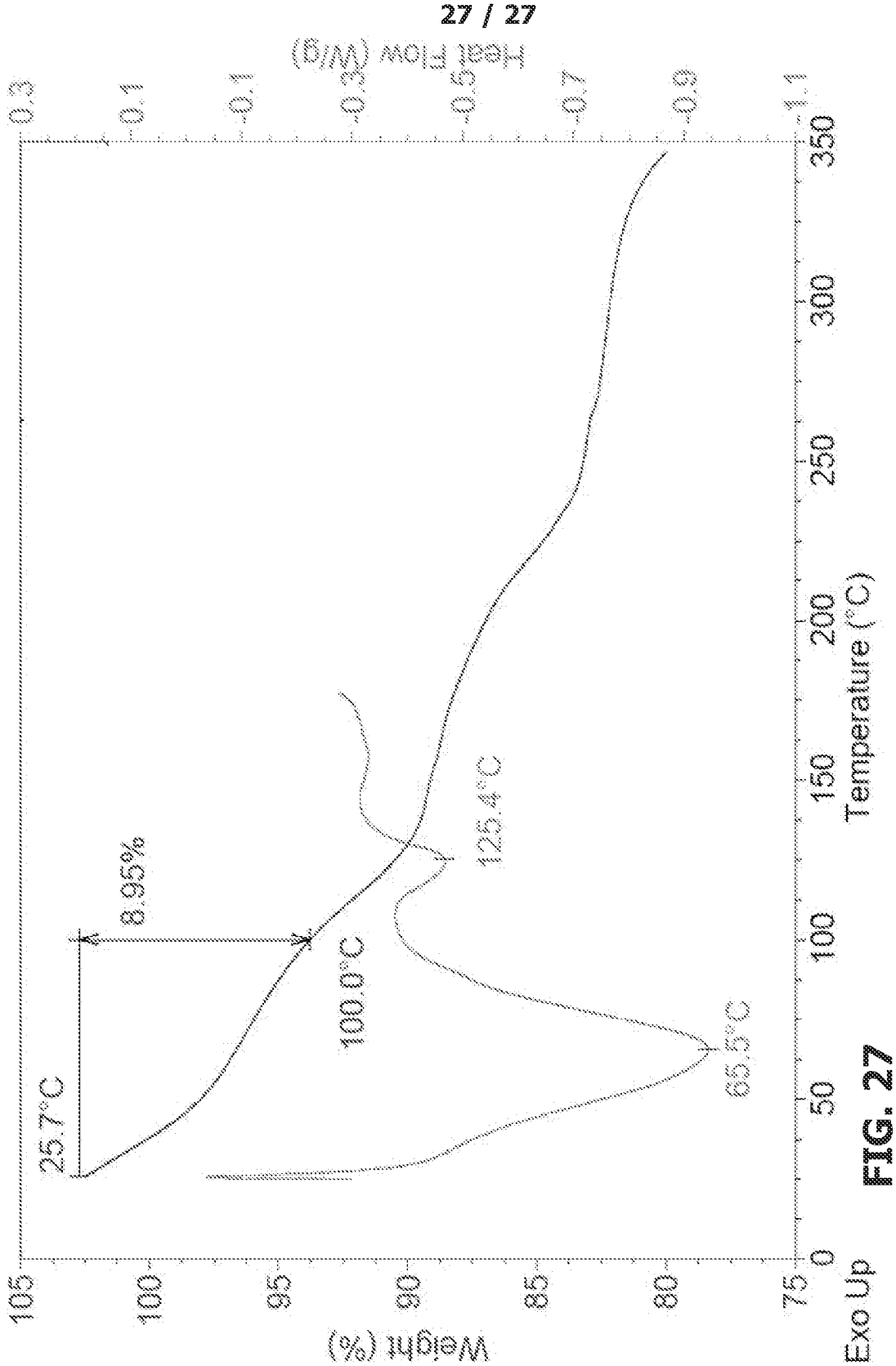


FIG. 27