

FUSED RING NITROGEN-CONTAINING COMPOUND, CRYSTAL FORM
THEREOF, PREPARATION METHOD THEREFOR, AND USE THEREOF

This application claims priority to Chinese Patent Application No. 202410500685X, filed on April 24, 2024. This application incorporates the entire content of the aforementioned Chinese patent application by reference.

TECHNICAL FIELD

The present invention relates to a nitrogen-containing fused-ring compound, crystalline forms thereof, and preparation method therefor, and application thereof.

BACKGROUND

DNA damage is an important mechanism by which many chemotherapeutic drugs exert their antitumor effects. DNA double-strand breaks (DSBs) are one of the most common types of damage in cells, which can be directly caused by ionizing radiation, or induced by ultraviolet light, reactive oxygen species (ROS), or other mutagens, such as common chemotherapeutic drugs like cisplatin, 5-FU, etoposide, etc. Unrepaired DNA damage can lead to functional blocks in cell transcription and replication, induce apoptosis or necrosis, and result in clearance by immune cells. Tumor cells possess efficient damage repair or alternative repair pathways. Targeting key proteins in DNA damage repair pathways, leading to the accumulation of unrepaired DNA damage and ultimately cell death, is one of the important strategies in cancer treatment.

DNA double-strand breaks are typically repaired by three pathways: non-homologous end joining (NHEJ), homologous recombination (HR), and alternative non-homologous end joining (Alt-NHEJ, also known as microhomology-mediated end joining, MMEJ, or TMEJ). Among these, the HR repair pathway only occurs during the G2 and S phases, achieving error-free repair under conditions where sister chromatids are present. In mammals, over 90% of DSB damage is repaired via the NHEJ pathway. It is an error-prone repair, resulting in deletion mutations of <30 bp, insertion mutations of <5 bp, or microhomologous sequences of <2 bp. Recent studies indicate that when HR and/or NHEJ are deficient, cells become highly dependent on the third pathway, MMEJ, to repair broken DNA. Inhibiting MMEJ leads to cell apoptosis. MMEJ is also an error-prone repair pathway; it relies on DNA polymerase theta to join microhomologous end sequences (theta-mediated end joining, TMEJ) to complete the

repair of DNA double-strand breaks.

DNA polymerase theta (Pol theta or Pol θ) is a key protein in the MMEJ repair pathway. It is a unique multifunctional polymerase composed of an N-terminal helicase domain, a central domain, and a C-terminal polymerase domain. Basic research has found that the polymerase domain is essential for DNA extension at DSB damage repair sites, while the helicase domain and the central domain play key roles in the recognition and binding of Pol θ to substrates. Through Pol θ , the interaction between DNA and damage repair complexes (such as competitive binding to single-stranded DNA with RAD51) can be disrupted, thereby inhibiting the HR repair pathway. Furthermore, the helicase domain of Pol θ is also involved in DNA replication arrest; its functional loss will lead to increased replication stress in tumor cells, resulting in apoptosis.

Pol θ is not expressed or is expressed at low levels in normal tissue cells, but it is highly expressed in various tumors such as lung cancer, breast cancer, HR-deficient ovarian cancer, gastric cancer, and colon cancer, and is associated with poorer prognosis. Among these, Pol θ is overexpressed in over 70% of breast cancers. These phenomena indicate that Pol θ may play an important role in these cancers and is a potential tumor-specific target.

Studies involving knockdown or knockout of Pol θ in tumor cells have found that Pol θ deficiency can sensitize these cells to radiation, induce DSB generation, enhance replication fork instability, make tumor cells sensitive to genotoxic agents, potentially enhancing the effects of radiotherapy and chemotherapy, and is a potential drug target. Studies have also found that Pol θ exhibits synthetic lethality with HR deficiency, and its small molecule inhibitors can kill HR-deficient tumor cells both in vitro and in vivo. Particularly in PARP inhibitor (Olaparib, etc.) resistant tumors with HR deficiency reversion mutations, Pol θ inhibitors can resensitize cells to PARPi, providing valuable therapeutic opportunities. Additionally, given that Pol θ is a key protein in the MMEJ pathway, its functional impairment can also lead to increased genomic instability in cancer cells, increase somatic mutations, and facilitate the generation of tumor neoantigens. Furthermore, it has been reported that Pol θ is involved in cGAS-STING-mediated immune activation; therefore, targeting Pol θ also has the potential to enhance immunotherapy.

In summary, Pol θ is a highly promising target for cancer therapy. Designing ATPase activity inhibitors targeting the Pol θ protein to inhibit intracellular MMEJ, used alone or in combination with other chemotherapy, radiotherapy, antibody therapy, or immunotherapy, aims

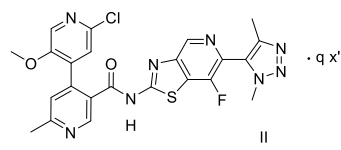
to kill tumor cells and holds great potential in the treatment of tumors such as lung cancer, breast cancer, HR-deficient ovarian cancer, gastric cancer, colon cancer, prostate cancer, and pancreatic cancer.

SUMMARY OF THE INVENTION

The technical problem to be solved by the present invention is to overcome the single solid form of polymerase theta inhibitor compounds in the prior art. For this purpose, the present invention provides a nitrogen-containing fused-ring compound, crystalline forms thereof, and preparation method therefor, and application thereof. The nitrogen-containing fused-ring compound and crystalline forms thereof provided by the present invention show significant improvement in ATPase activity and protein inhibitory effects compared to the prior art.

The present invention solves the above technical problem through the following technical solutions.

The present invention provides a compound of formula II,



wherein the structure of the compound of formula II is:

x' (solvent) is water or acetone;

q (molar equivalent of solvent) is 0.01-2.5.

Preferably, in the compound of formula II, q (molar equivalent of solvent) is 0.1, 0.5, 0.56, 0.6, 1, 1.5, 2, or 2.5.

In one embodiment, the compound of formula II is crystalline form A of the compound of formula II, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $9.00\pm 0.2^\circ$, $10.84\pm 0.2^\circ$, $17.91\pm 0.2^\circ$, and $22.05\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula II has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following positions: $5.98\pm 0.2^\circ$, $11.94\pm 0.2^\circ$, $18.36\pm 0.2^\circ$, $21.30\pm 0.2^\circ$, $23.29\pm 0.2^\circ$, and $27.72\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula II has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks at $9.00\pm 0.2^\circ$, $10.84\pm 0.2^\circ$, $17.91\pm 0.2^\circ$, $22.05\pm 0.2^\circ$, $5.98\pm 0.2^\circ$, $11.94\pm 0.2^\circ$, $18.36\pm 0.2^\circ$,

In one embodiment, crystalline form A of the compound of formula II has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following positions: 13.46 $\pm$ 0.2 $^\circ$, 14.16 $\pm$ 0.2 $^\circ$, 14.72 $\pm$ 0.2 $^\circ$, 19.99 $\pm$ 0.2 $^\circ$, 20.65 $\pm$ 0.2 $^\circ$, 24.6 $\pm$ 0.2 $^\circ$, 25.62 $\pm$ 0.2 $^\circ$, 28.96 $\pm$ 0.2 $^\circ$, 29.68 $\pm$ 0.2 $^\circ$, 30.29 $\pm$ 0.2 $^\circ$, and 31.08 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula II has its X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 1.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form A of the compound of formula II is substantially as shown in FIG. 2.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form A of the compound of formula II is substantially as shown in FIG. 3.

In one embodiment, the compound of formula II is crystalline form B of the compound of formula II, which has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, having diffraction peaks at 8.10 $\pm$ 0.2 $^\circ$, 8.37 $\pm$ 0.2 $^\circ$, 13.94 $\pm$ 0.2 $^\circ$, 15.67 $\pm$ 0.2 $^\circ$, 20.72 $\pm$ 0.2 $^\circ$, 24.4 $\pm$ 0.2 $^\circ$, and 24.83 $\pm$ 0.2 $^\circ$.

4

and $21.15 \pm 0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula II has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks at $8.10 \pm 0.2^\circ$, $8.37 \pm 0.2^\circ$, $13.94 \pm 0.2^\circ$, $15.67 \pm 0.2^\circ$, $20.72 \pm 0.2^\circ$, $14.26 \pm 0.2^\circ$, $16.61 \pm 0.2^\circ$, $17.91 \pm 0.2^\circ$, and $18.70 \pm 0.2^\circ$.

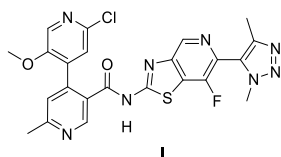
In one embodiment, crystalline form B of the compound of formula II has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following positions: $14.26 \pm 0.2^\circ$, $16.61 \pm 0.2^\circ$, $17.91 \pm 0.2^\circ$, $18.70 \pm 0.2^\circ$, $19.13 \pm 0.2^\circ$, $19.62 \pm 0.2^\circ$, $20.03 \pm 0.2^\circ$, $23.92 \pm 0.2^\circ$, $24.20 \pm 0.2^\circ$, and $25.42 \pm 0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula II has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks as shown in Table 2.

In one embodiment, crystalline form B of the compound of formula II has its X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 4.

In one embodiment, in crystalline form B of the compound of formula II, x' is acetone, and q is 0.56, 0.5, or 0.6, for example, q is 0.56.

The present invention provides crystalline form C of a compound of formula I, which has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks at $11.51 \pm 0.2^\circ$, $18.01 \pm 0.2^\circ$, $22.56 \pm 0.2^\circ$, and $25.68 \pm 0.2^\circ$;



In one embodiment, crystalline form C of the compound of formula I has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following positions: $12.88 \pm 0.2^\circ$, $15.05 \pm 0.2^\circ$, $20.85 \pm 0.2^\circ$, $21.68 \pm 0.2^\circ$, $23.11 \pm 0.2^\circ$, and $24.79 \pm 0.2^\circ$.

Preferably, in crystalline form C of the compound of formula I, the diffraction peak at $11.51 \pm 0.2^\circ$ in its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, is the strongest peak.

In one embodiment, crystalline form C of the compound of formula I has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction

peaks at $11.51\pm0.2^\circ$, $18.01\pm0.2^\circ$, $22.56\pm0.2^\circ$, $25.68\pm0.2^\circ$, $12.88\pm0.2^\circ$, $15.05\pm0.2^\circ$, $20.85\pm0.2^\circ$, $21.68\pm0.2^\circ$, and $23.11\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula I has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following positions: $9.022\pm0.2^\circ$, $10.212\pm0.2^\circ$, $16.163\pm0.2^\circ$, $16.96\pm0.2^\circ$, $17.22\pm0.2^\circ$, $26.937\pm0.2^\circ$, $29.445\pm0.2^\circ$, $30.137\pm0.2^\circ$, and $30.354\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula I has its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks as shown in Table 3.

In one embodiment, crystalline form C of the compound of formula I has its X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 5.

In one embodiment, crystalline form C of the compound of formula I has its differential scanning calorimetry (DSC) curve exhibiting an endothermic peak at a peak temperature of $238.92\pm3^\circ\text{C}$.

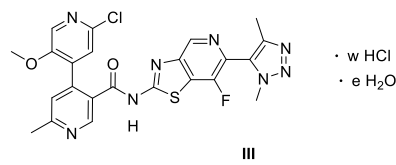
In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form C of the compound of formula I is substantially as shown in FIG. 6.

In one embodiment, crystalline form C of the compound of formula I has its thermogravimetric analysis (TGA) curve showing a weight loss of about 0.81% over the temperature range from $35.56\pm3^\circ\text{C}$ to $230.00\pm3^\circ\text{C}$.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form C of the compound of formula I is substantially as shown in FIG. 7.

In one embodiment, crystalline form C of the compound of formula I is solvent-free.

The present invention provides a compound of formula III,



wherein the structure of the compound of formula III is:

wherein w is 0.5-2.5; e is 0-2.

In one embodiment, in the compound of formula III, w (molar equivalents of hydrochloric acid) is 0.5, 1, 1.1, 1.6, 1.7, 1.8, 1.76, 1.06, 1.5, or 2.

In one embodiment, in the compound of formula III, e is 0 (solvent-free).

In one embodiment, in the compound of formula III, e (molar equivalents of water) is 1-2, for example, e is 1.5.

In one embodiment, the compound of formula III is crystalline form A of the compound of formula III, and its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, has diffraction peaks at $4.61\pm0.2^\circ$, $5.02\pm0.2^\circ$, $9.19\pm0.2^\circ$, and $13.76\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, further having diffraction peaks at one or more of the following positions: $6.57\pm0.2^\circ$, $6.89\pm0.2^\circ$, $9.96\pm0.2^\circ$, $10.48\pm0.2^\circ$, $10.64\pm0.2^\circ$, $14.69\pm0.2^\circ$, and $14.90\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, having diffraction peaks at $4.61\pm0.2^\circ$, $5.02\pm0.2^\circ$, $9.19\pm0.2^\circ$, $13.76\pm0.2^\circ$, $6.57\pm0.2^\circ$, $6.89\pm0.2^\circ$, $9.96\pm0.2^\circ$, $10.48\pm0.2^\circ$, and $10.64\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, further having diffraction peaks at one or more of the following positions: $18.34\pm0.2^\circ$, $19.13\pm0.2^\circ$, $19.30\pm0.2^\circ$, $20.81\pm0.2^\circ$, $21.68\pm0.2^\circ$, $23.09\pm0.2^\circ$, $24.03\pm0.2^\circ$, $26.13\pm0.2^\circ$, and $28.17\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, having diffraction peaks as shown in Table 4.

In one embodiment, the X-ray powder diffraction pattern of crystalline form A of the compound of formula III, obtained using Cu-K α radiation, is substantially as shown in FIG. 8.

In one embodiment, in crystalline form A of the compound of formula III, w (molar equivalents of hydrochloric acid) is 1, 1.1, or 1.06, for example 1.06.

In one embodiment, in crystalline form A of the compound of formula III, e is 0 or 1.5. Preferably, e is 0. In one embodiment, the compound of formula III is crystalline form B of the compound of formula III, and its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, has diffraction peaks at $6.47\pm0.2^\circ$, $7.19\pm0.2^\circ$, $9.11\pm0.2^\circ$, $11.63\pm0.2^\circ$, and $14.45\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, further having diffraction peaks at one or more of the following positions: $17.37\pm0.2^\circ$, $18.21\pm0.2^\circ$,

19.44±0.2°, 20.17±0.2°, 20.82±0.2°, 22.41±0.2°, and 24.58±0.2°.

In one embodiment, crystalline form B of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, having diffraction peaks at 6.47±0.2°, 7.19±0.2°, 9.11±0.2°, 11.63±0.2°, 14.45±0.2°, 17.37±0.2°, 18.21±0.2°, 19.44±0.2°, 20.17±0.2°, and 20.82±0.2°.

In one embodiment, crystalline form B of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, further having diffraction peaks at one or more of the following positions: 12.94±0.2°, 13.41±0.2°, 15.41±0.2°, 15.75±0.2°, 16.23±0.2°, 16.72±0.2°, 26.01±0.2°, 26.17±0.2°, 26.47±0.2°, 27.96±0.2°, 28.62±0.2°, 30.04±0.2°, and 30.37±0.2°.

In one embodiment, crystalline form B of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, having diffraction peaks as shown in Table 5.

In one embodiment, the X-ray powder diffraction pattern of crystalline form B of the compound of formula III, obtained using Cu-K α radiation, is substantially as shown in FIG. 9.

In one embodiment, crystalline form B of the compound of formula III has its differential scanning calorimetry (DSC) curve showing an endothermic peak at a peak temperature of 186.88±3°C.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form B of the compound of formula III is substantially as shown in FIG. 10.

In one embodiment, crystalline form B of the compound of formula III has its thermogravimetric analysis (TGA) curve showing a weight loss of about 1.42% in the temperature range from 24.07±3°C to 120.0±3°C, and a weight loss of about 9.84% in the temperature range from 120.00±3°C to 205.00±3°C.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form B of the compound of formula III is substantially as shown in FIG. 11.

In one embodiment, in crystalline form B of the compound of formula III, w (molar equivalent of hydrochloric acid) is 1.76, 1.7, or 1.8, for example, 1.76.

In one embodiment, in crystalline form B of the compound of formula III, e is 0.

In one embodiment, the compound of formula III is crystalline form C of the compound of formula III, and its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angles, has diffraction peaks at 9.84±0.2°, 10.51±0.2°, 12.19±0.2°, 13.39±0.2°, 19.44±0.2°, 20.17±0.2°, 20.82±0.2°, 22.41±0.2°, and 24.58±0.2°.

and $14.40 \pm 0.2^\circ$.

In one embodiment, the X-ray powder diffraction pattern of crystalline form C of the compound of formula III, obtained using Cu-K α radiation and expressed in terms of 2θ angles, further has diffraction peaks at one or more of the following: $18.16 \pm 0.2^\circ$, $18.29 \pm 0.2^\circ$, $19.39 \pm 0.2^\circ$, $20.81 \pm 0.2^\circ$, $21.75 \pm 0.2^\circ$, $26.41 \pm 0.2^\circ$, and $27.48 \pm 0.2^\circ$.

In one embodiment, the X-ray powder diffraction pattern of crystalline form C of the compound of formula III, obtained using Cu-K α radiation and expressed in terms of 2θ angles, has diffraction peaks at $9.84 \pm 0.2^\circ$, $10.51 \pm 0.2^\circ$, $12.19 \pm 0.2^\circ$, $13.39 \pm 0.2^\circ$, $14.40 \pm 0.2^\circ$, $18.16 \pm 0.2^\circ$, $18.29 \pm 0.2^\circ$, $19.39 \pm 0.2^\circ$, $20.81 \pm 0.2^\circ$, $21.75 \pm 0.2^\circ$, and $26.41 \pm 0.2^\circ$.

In one embodiment, the X-ray powder diffraction pattern of crystalline form C of the compound of formula III, obtained using Cu-K α radiation and expressed in terms of 2θ angles, further additionally has diffraction peaks at one or more of the following: $11.61 \pm 0.2^\circ$, $20.02 \pm 0.2^\circ$, $22.51 \pm 0.2^\circ$, $22.90 \pm 0.2^\circ$, $23.25 \pm 0.2^\circ$, $23.49 \pm 0.2^\circ$, $24.46 \pm 0.2^\circ$, $25.11 \pm 0.2^\circ$, $26.94 \pm 0.2^\circ$, $28.37 \pm 0.2^\circ$, $28.97 \pm 0.2^\circ$, and $29.32 \pm 0.2^\circ$.

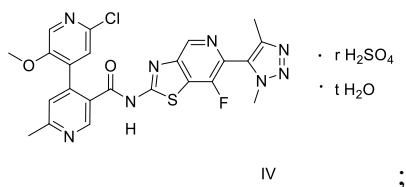
In one embodiment, crystalline form C of the compound of formula III has its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks as shown in Table 6.

In one embodiment, the X-ray powder diffraction pattern of crystalline form C of the compound of formula III, obtained using Cu-K α radiation, is substantially as shown in FIG. 12.

In one embodiment, in crystalline form C of the compound of formula III, w is 1.7, 1.8, or 1.6, for example, 1.7.

In one embodiment, in crystalline form C of the compound of formula III, e is 0.

The present invention provides a compound of formula IV,



wherein r is 0.5-2; t is 0-2.

In one embodiment, in the compound of formula IV, r is 0.5, 0.9, 1, 0.97, 0.96, or 1.5.

In one embodiment, in the compound of formula IV, t is 0.

In one embodiment, in the compound of formula IV, t is 1-2, for example, t is 1.7.

In one embodiment, the compound of formula IV is crystalline form A of the compound of

formula IV, and its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ , has diffraction peaks at $5.14\pm0.2^\circ$, $9.01\pm0.2^\circ$, and $15.03\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ , which further has diffraction peaks at one or more of the following positions: $11.45\pm0.2^\circ$, $17.44\pm0.2^\circ$, $21.45\pm0.2^\circ$, $21.78\pm0.2^\circ$, $24.82\pm0.2^\circ$, $25.95\pm0.2^\circ$, and $26.68\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ , which has diffraction peaks at $5.14\pm0.2^\circ$, $9.01\pm0.2^\circ$, $15.03\pm0.2^\circ$, $11.45\pm0.2^\circ$, $17.44\pm0.2^\circ$, $21.45\pm0.2^\circ$, $21.78\pm0.2^\circ$, $24.82\pm0.2^\circ$, and $25.95\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ , which further has diffraction peaks at one or more of the following positions: $6.11\pm0.2^\circ$, $6.60\pm0.2^\circ$, $7.35\pm0.2^\circ$, $8.63\pm0.2^\circ$, $14.62\pm0.2^\circ$, $16.45\pm0.2^\circ$, $20.40\pm0.2^\circ$, $23.81\pm0.2^\circ$, $25.47\pm0.2^\circ$, $27.96\pm0.2^\circ$, and $28.43\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, which has diffraction peaks as shown in Table 7.

In one embodiment, crystalline form A of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, which is substantially as shown in FIG. 13.

In one embodiment, in crystalline form A of the compound of formula IV, r is 1, 0.97, or 0.9, for example, 0.97.

In one embodiment, in crystalline form A of the compound of formula IV, t is 0.

In one embodiment, the compound of formula IV is crystalline form B of the compound of formula IV, and its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ , has diffraction peaks at $7.11\pm0.2^\circ$, $7.57\pm0.2^\circ$, $7.72\pm0.2^\circ$, $8.10\pm0.2^\circ$, and $10.27\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ , which further has diffraction peaks at one or more of the following positions: $13.86\pm0.2^\circ$, $14.11\pm0.2^\circ$, $14.71\pm0.2^\circ$, $15.44\pm0.2^\circ$, $16.07\pm0.2^\circ$, $23.18\pm0.2^\circ$, $24.18\pm0.2^\circ$, and $25.17\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula IV has an X-ray powder

diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ , which has diffraction peaks at $7.11\pm0.2^\circ$, $7.57\pm0.2^\circ$, $7.72\pm0.2^\circ$, $8.10\pm0.2^\circ$, $13.86\pm0.2^\circ$, $14.11\pm0.2^\circ$, $14.71\pm0.2^\circ$, $23.18\pm0.2^\circ$, $24.18\pm0.2^\circ$, and $25.17\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ , which further has diffraction peaks at one or more of the following positions: $10.27\pm0.2^\circ$, $12.55\pm0.2^\circ$, $12.67\pm0.2^\circ$, $13.19\pm0.2^\circ$, $16.73\pm0.2^\circ$, $17.92\pm0.2^\circ$, $18.38\pm0.2^\circ$, $18.58\pm0.2^\circ$, $20.18\pm0.2^\circ$, $21.2\pm0.2^\circ$, $22.2\pm0.2^\circ$, $25.58\pm0.2^\circ$, and $27.82\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, which has diffraction peaks as shown in Table 8.

In one embodiment, crystalline form B of the compound of formula IV has an X-ray powder diffraction pattern obtained using Cu-K α radiation, which is substantially as shown in FIG. 14.

In one embodiment, crystalline form B of the compound of formula IV has a differential scanning calorimetry (DSC) curve with endothermic peaks at peak temperatures of $34.85\pm3^\circ\text{C}$, $90.79\pm3^\circ\text{C}$, and $204.38\pm3^\circ\text{C}$.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form B of the compound of formula IV is substantially as shown in FIG. 15.

In one embodiment, crystalline form B of the compound of formula IV has a thermogravimetric analysis (TGA) curve showing a weight loss of about 2.06% in the temperature range from $33.44\pm3^\circ\text{C}$ to $50.0\pm3^\circ\text{C}$, a weight loss of about 0.56% in the temperature range from $50.0\pm3^\circ\text{C}$ to $100.0\pm3^\circ\text{C}$, a weight loss of about 0.39% in the temperature range from $100.00\pm3^\circ\text{C}$ to $180.00\pm3^\circ\text{C}$, and a weight loss of about 1.0% in the temperature range from $180.00\pm3^\circ\text{C}$ to $230.00\pm3^\circ\text{C}$.

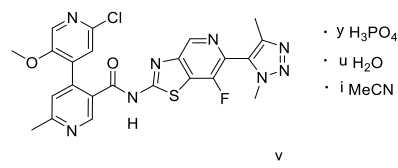
In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form B of the compound of formula IV is substantially as shown in FIG. 16.

In one embodiment, in crystalline form B of the compound of formula IV, r is 1, 0.9, or 0.96, for example, 0.96.

In one embodiment, in crystalline form B of the compound of formula IV, t is 0 or 1.7. Preferably, t is 0.

In one embodiment, in crystalline form B of the compound of formula IV, r is 1 and t is 0; or, r is 0.96 and t is 0.

The present invention provides a compound of formula V,



wherein the structure of the compound of formula V is:

y is 0.5-1.5; u is 0-2; i is 0-2. Preferably, i and u are 0.

In one embodiment, in the compound of formula V, y is 1.1, 1.2, 1.14, 1, 1.09, or 1.05.

In one embodiment, in the compound of formula V, y and u are 0.

In one embodiment, in the compound of formula V, i is 0, and u is 0.1, 0.91, 0.9, or 1, for example, 0.91.

In one embodiment, in the compound of formula V, u is 0, and i is 0.1, 0.18, 0.2, or 0.5, for example, 0.18 or 0.1.

In one embodiment, the compound of formula V is crystalline form A of the compound of formula V, and its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.15\pm 0.2^\circ$, $8.34\pm 0.2^\circ$, $15.51\pm 0.2^\circ$, and $15.88\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which further has diffraction peaks at one or more of the following positions: $7.97\pm 0.2^\circ$, $8.96\pm 0.2^\circ$, $17.93\pm 0.2^\circ$, $18.39\pm 0.2^\circ$, $21.99\pm 0.2^\circ$, and $27.73\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which has diffraction peaks at $5.15\pm 0.2^\circ$, $8.34\pm 0.2^\circ$, $15.51\pm 0.2^\circ$, $15.88\pm 0.2^\circ$, $8.96\pm 0.2^\circ$, $17.93\pm 0.2^\circ$, $18.39\pm 0.2^\circ$, $21.99\pm 0.2^\circ$, and $27.73\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which further has diffraction peaks at one or more of the following positions: $5.98\pm 0.2^\circ$, $8.96\pm 0.2^\circ$, $10.49\pm 0.2^\circ$, $10.80\pm 0.2^\circ$, $11.34\pm 0.2^\circ$, $11.66\pm 0.2^\circ$, $11.99\pm 0.2^\circ$, $12.73\pm 0.2^\circ$, $13.15\pm 0.2^\circ$, $18.68\pm 0.2^\circ$, $19.73\pm 0.2^\circ$, $20.14\pm 0.2^\circ$, $22.76\pm 0.2^\circ$, and $23.24\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which has diffraction peaks as shown in Table 9.

In one embodiment, crystalline form A of the compound of formula V has an X-ray powder

diffraction pattern obtained using Cu-K α radiation which is substantially as shown in FIG. 17.

In one embodiment, crystalline form A of the compound of formula V has a differential scanning calorimetry (DSC) curve with endothermic peaks at peak temperatures of $88.96\pm 3^{\circ}\text{C}$ and $192.67\pm 3^{\circ}\text{C}$.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form A of the compound of formula V is substantially as shown in FIG. 18.

In one embodiment, crystalline form A of the compound of formula V has a thermogravimetric analysis (TGA) curve showing a weight loss of about 0.92% in the temperature range from $32.34\pm 3^{\circ}\text{C}$ to $70.0\pm 3^{\circ}\text{C}$, and a weight loss of about 2.1% in the temperature range from $70.0\pm 3^{\circ}\text{C}$ to $180.0\pm 3^{\circ}\text{C}$.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form A of the compound of formula V is substantially as shown in FIG. 19.

In one embodiment, in crystalline form A of the compound of formula V, y is 1.1, 1.2, or 1.14, for example, 1.14.

In one embodiment, in crystalline form A of the compound of formula V, i is 0, y is 1.1, and u is 0.9; or, i is 0, y is 1.14, and u is 0.91.

In one embodiment, the compound of formula V is crystalline form B of the compound of formula V, and its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.83\pm 0.2^{\circ}$, $12.89\pm 0.2^{\circ}$, $14.44\pm 0.2^{\circ}$, $15.91\pm 0.2^{\circ}$, $25.08\pm 0.2^{\circ}$, and $25.50\pm 0.2^{\circ}$.

In one embodiment, crystalline form B of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which further has diffraction peaks at one or more of the following positions: $8.32\pm 0.2^{\circ}$, $10.26\pm 0.2^{\circ}$, $13.93\pm 0.2^{\circ}$, $14.67\pm 0.2^{\circ}$, $20.35\pm 0.2^{\circ}$, $20.49\pm 0.2^{\circ}$, $21.15\pm 0.2^{\circ}$, and $21.65\pm 0.2^{\circ}$.

In one embodiment, crystalline form B of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which has diffraction peaks at $6.83\pm 0.2^{\circ}$, $12.89\pm 0.2^{\circ}$, $14.44\pm 0.2^{\circ}$, $15.91\pm 0.2^{\circ}$, $25.08\pm 0.2^{\circ}$, $25.50\pm 0.2^{\circ}$, $8.32\pm 0.2^{\circ}$, $20.35\pm 0.2^{\circ}$, and $20.49\pm 0.2^{\circ}$.

In one embodiment, crystalline form B of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which further has diffraction peaks at one or more of the following positions: $8.52\pm 0.2^{\circ}$, $9.17\pm 0.2^{\circ}$, $13.46\pm 0.2^{\circ}$, $16.25\pm 0.2^{\circ}$, and $17.94\pm 0.2^{\circ}$.

In one embodiment, crystalline form B of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which has diffraction peaks as shown in Table 10.

In one embodiment, crystalline form B of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation which is substantially as shown in FIG. 20.

In one embodiment, in crystalline form B of the compound of formula V, y is 1.1, 1, or 1.09, for example, 1.09.

In one embodiment, in crystalline form B of the compound of formula V, u and i are 0.

In one embodiment, the compound of formula V is crystalline form C of the compound of formula V, and its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.19\pm0.2^\circ$, $8.39\pm0.2^\circ$, $10.47\pm0.2^\circ$, and $11.51\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which further has diffraction peaks at one or more of the following positions: $15.68\pm0.2^\circ$, $15.87\pm0.2^\circ$, $18.01\pm0.2^\circ$, $22.55\pm0.2^\circ$, and $25.67\pm0.2^\circ$.

Preferably, in crystalline form C of the compound of formula V, the relative intensity of the diffraction peak at $11.51\pm0.2^\circ$ in its X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, is 80%-100%, and preferably the diffraction peak at $11.51\pm0.2^\circ$ is the strongest peak.

In one embodiment, crystalline form C of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which further has diffraction peaks at one or more of the following positions: $11.67\pm0.2^\circ$, $12.73\pm0.2^\circ$, $12.89\pm0.2^\circ$, $14.59\pm0.2^\circ$, $15.05\pm0.2^\circ$, $18.76\pm0.2^\circ$, $19.81\pm0.2^\circ$, $20.48\pm0.2^\circ$, $20.84\pm0.2^\circ$, $21.68\pm0.2^\circ$, $22.10\pm0.2^\circ$, $24.80\pm0.2^\circ$, and $25.27\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which has diffraction peaks at $5.19\pm0.2^\circ$, $8.39\pm0.2^\circ$, $10.47\pm0.2^\circ$, $11.51\pm0.2^\circ$, $11.67\pm0.2^\circ$, $15.68\pm0.2^\circ$, $15.87\pm0.2^\circ$, $18.01\pm0.2^\circ$, and $22.55\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula V has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which has diffraction peaks as shown in Table 11.

In one embodiment, crystalline form C of the compound of formula V has an X-ray powder

diffraction pattern obtained using Cu-K α radiation which is substantially as shown in FIG. 21.

In one embodiment, crystalline form C of the compound of formula V has a differential scanning calorimetry (DSC) curve with endothermic peaks at peak temperatures of $188.32\pm 3^{\circ}\text{C}$ and $221.66\pm 3^{\circ}\text{C}$.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form C of the compound of formula V is substantially as shown in FIG. 22.

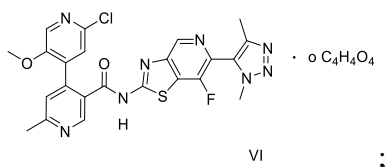
In one embodiment, crystalline form C of the compound of formula V has a thermogravimetric analysis (TGA) curve showing a weight loss of about 0.71% in the temperature range from $29.55\pm 3^{\circ}\text{C}$ to $180.0\pm 3^{\circ}\text{C}$ and a weight loss of about 0.85% in the temperature range from $180.0\pm 3^{\circ}\text{C}$ to $220.0\pm 3^{\circ}\text{C}$.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form C of the compound of formula V is substantially as shown in FIG. 23.

In one embodiment, in crystalline form C of the compound of formula V, y is 1.1, 1, or 1.05, for example, 1.05.

In one embodiment, in crystalline form C of the compound of formula V, u and i are 0, and y is 1; or, u is 0, i is 0.1, and y is 1.05.

The present invention provides a compound of formula VI,



wherein, C₄H₄O₄ is fumaric acid;

o (molar equivalent of fumaric acid) is 1-1.1.

In one embodiment, in the compound of formula VI, o is 1, 1.01, 1.05, or 1.1.

In one embodiment, the compound of formula VI is crystalline form A of the compound of formula VI, which has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, with diffraction peaks at $10.58\pm 0.2^{\circ}$, $12.16\pm 0.2^{\circ}$, and $13.98\pm 0.2^{\circ}$.

In one embodiment, crystalline form A of the compound of formula VI has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2θ angle, which further has diffraction peaks at one or more of the following positions: $6.96\pm 0.2^{\circ}$, $8.60\pm 0.2^{\circ}$, $14.84\pm 0.2^{\circ}$, $15.39\pm 0.2^{\circ}$, $16.63\pm 0.2^{\circ}$, and $17.08\pm 0.2^{\circ}$.

In one embodiment, crystalline form A of the compound of formula VI has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angle, with diffraction peaks at 10.58 $\pm$ 0.2 $^\circ$, 12.16 $\pm$ 0.2 $^\circ$, 13.98 $\pm$ 0.2 $^\circ$, 6.96 $\pm$ 0.2 $^\circ$, 8.60 $\pm$ 0.2 $^\circ$, 14.84 $\pm$ 0.2 $^\circ$, 15.39 $\pm$ 0.2 $^\circ$, 16.63 $\pm$ 0.2 $^\circ$, and 17.08 $\pm$ 0.2 $^\circ$.

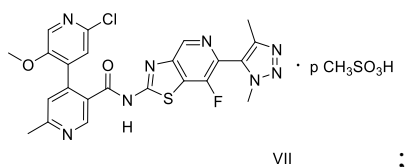
In one embodiment, crystalline form A of the compound of formula VI has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angle, which further has diffraction peaks at one or more of the following positions: 6.73 $\pm$ 0.2 $^\circ$, 9.38 $\pm$ 0.2 $^\circ$, 10.28 $\pm$ 0.2 $^\circ$, 13.45 $\pm$ 0.2 $^\circ$, 19.28 $\pm$ 0.2 $^\circ$, and 18.67 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula VI has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angle, which has diffraction peaks as shown in Table 12.

In one embodiment, crystalline form A of the compound of formula VI has an X-ray powder diffraction pattern obtained using Cu-K α radiation which is substantially as shown in FIG. 24.

In one embodiment, in crystalline form A of the compound of formula VI, o is 1, 1.1, or 1.01, for example, 1.01.

The present invention provides a compound of formula VII,



wherein CH₃SO₃H is methanesulfonic acid;

p (the molar equivalent of methanesulfonic acid) is 1-1.1.

In one embodiment, in the compound of formula VII, p is 1:1, 1:1.03, 1:1.05, or 1:1.1.

In one embodiment, in the compound of formula VII, p is 1, 1.03, 1.05, or 1.1.

In one embodiment, the compound of formula VII is crystalline form A of the compound of formula VII, which has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angle, with diffraction peaks at 9.00 $\pm$ 0.2 $^\circ$, 17.44 $\pm$ 0.2 $^\circ$, and 21.74 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula VII has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angle, which further has diffraction peaks at one or more of the following positions: 5.13 $\pm$ 0.2 $^\circ$, 6.73 $\pm$ 0.2 $^\circ$, 8.61 $\pm$ 0.2 $^\circ$, 17.74 $\pm$ 0.2 $^\circ$, 24.43 $\pm$ 0.2 $^\circ$, and 25.32 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula VII has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angle, with diffraction peaks at 9.00 $\pm$ 0.2 $^\circ$, 17.44 $\pm$ 0.2 $^\circ$, 21.74 $\pm$ 0.2 $^\circ$, 5.13 $\pm$ 0.2 $^\circ$, 6.73 $\pm$ 0.2 $^\circ$, 8.61 $\pm$ 0.2 $^\circ$, 17.74 $\pm$ 0.2 $^\circ$, 24.43 $\pm$ 0.2 $^\circ$, and 25.32 $\pm$ 0.2 $^\circ$.

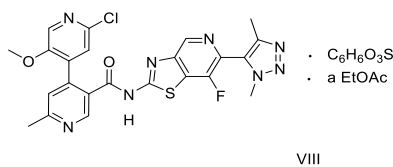
In one embodiment, crystalline form A of the compound of formula VII has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angle, which further has diffraction peaks at one or more of the following positions: 15.55 $\pm$ 0.2 $^\circ$, 10.05 $\pm$ 0.2 $^\circ$, 11.57 $\pm$ 0.2 $^\circ$, 22.37 $\pm$ 0.2 $^\circ$, and 26.36 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula VII has an X-ray powder diffraction pattern obtained using Cu-K α radiation, expressed in terms of 2 θ angle, which has diffraction peaks as shown in Table 13.

In one embodiment, crystalline form A of the compound of formula VII has an X-ray powder diffraction pattern obtained using Cu-K α radiation which is substantially as shown in FIG. 25.

In one embodiment, in crystalline form A of the compound of formula VII, p is 1, 1.1, or 1.03, for example, 1.03.

The present invention provides a compound of formula VIII,



wherein C₆H₅O₃S is benzenesulfonic acid;

a (the molar equivalent of ethyl acetate) is 0-1.

In one embodiment, in the compound of formula VIII, a is 0 (solvent-free).

In one embodiment, in the compound of formula VIII, a is 0-0.1, for example, 0.07, 0.1, or 0.

In one embodiment, the compound of formula VIII is crystalline form A of the compound of formula VIII, which has X-ray powder diffraction peaks expressed in 2 θ angles using Cu-K α radiation at 7.66 $\pm$ 0.2 $^\circ$, 10.09 $\pm$ 0.2 $^\circ$, 11.92 $\pm$ 0.2 $^\circ$, and 13.91 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula VIII has X-ray powder diffraction peaks expressed in 2 θ angles using Cu-K α radiation further at one or more of the following: 9.06 $\pm$ 0.2 $^\circ$, 9.72 $\pm$ 0.2 $^\circ$, 11.10 $\pm$ 0.2 $^\circ$, 15.91 $\pm$ 0.2 $^\circ$, 16.55 $\pm$ 0.2 $^\circ$, 18.00 $\pm$ 0.2 $^\circ$, 18.27 $\pm$ 0.2 $^\circ$, and 19.16 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula VIII has X-ray powder

diffraction peaks expressed in 2θ angles using Cu-K α radiation at $7.66\pm 0.2^\circ$, $10.09\pm 0.2^\circ$, $11.92\pm 0.2^\circ$, $13.91\pm 0.2^\circ$, $9.06\pm 0.2^\circ$, $9.72\pm 0.2^\circ$, $11.10\pm 0.2^\circ$, $15.91\pm 0.2^\circ$, and $16.55\pm 0.2^\circ$.

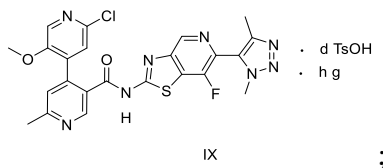
In one embodiment, crystalline form A of the compound of formula VIII has X-ray powder diffraction peaks expressed in 2θ angles using Cu-K α radiation further at one or more of the following: $20.35\pm 0.2^\circ$, $20.89\pm 0.2^\circ$, $21.10\pm 0.2^\circ$, $21.68\pm 0.2^\circ$, $22.27\pm 0.2^\circ$, $22.49\pm 0.2^\circ$, $22.97\pm 0.2^\circ$, $23.50\pm 0.2^\circ$, $24.84\pm 0.2^\circ$, $25.52\pm 0.2^\circ$, and $29.36\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula VIII has X-ray powder diffraction peaks using Cu-K α radiation as shown in Table 14.

In one embodiment, crystalline form A of the compound of formula VIII has an X-ray powder diffraction pattern obtained using Cu-K α radiation which is substantially as shown in FIG. 26.

In one embodiment, in crystalline form A of the compound of formula VIII, a is 0, 0.1, or 0.07, for example, 0.07.

The present invention provides a compound of formula IX,



wherein d (the molar equivalent of p-toluenesulfonic acid) is 1-2;

g (solvent) is water, tetrahydrofuran, isopropanol, 1,4-dioxane, or methyl tert-butyl ether;

h (the molar equivalent of solvent) is 0-3.

In one embodiment, in the compound of formula IX, d is 1, 1.1, 1.2, 1.02, or 1.5.

In one embodiment, in the compound of formula IX, h is 0 (solvent-free).

In one embodiment, in the compound of formula IX, h is 0.1-3. For example, g is water and h is 3; or g is tetrahydrofuran and h is 0.2, 0.1, or 0.16; or g is isopropanol and h is 0.9, 0.98, or 1; or g is 1,4-dioxane and h is 3, 2.96, or 2.9; or g is methyl tert-butyl ether and h is 0.4, 0.45, or 0.5.

In one embodiment, the compound of formula IX is crystalline form A of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, has diffraction peaks at $5.96\pm 0.2^\circ$, $16.71\pm 0.2^\circ$, $17.79\pm 0.2^\circ$, $20.33\pm 0.2^\circ$, and $24.66\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having

diffraction peaks at one or more of the following positions: $10.57\pm0.2^\circ$, $12.34\pm0.2^\circ$, $12.77\pm0.2^\circ$, $14.06\pm0.2^\circ$, $14.40\pm0.2^\circ$, $14.97\pm0.2^\circ$, $19.88\pm0.2^\circ$, $22.64\pm0.2^\circ$, $23.18\pm0.2^\circ$, and $24.22\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks at $5.96\pm0.2^\circ$, $16.71\pm0.2^\circ$, $17.79\pm0.2^\circ$, $20.33\pm0.2^\circ$, $24.66\pm0.2^\circ$, $10.57\pm0.2^\circ$, $12.34\pm0.2^\circ$, $12.77\pm0.2^\circ$, $14.06\pm0.2^\circ$, $14.40\pm0.2^\circ$, and $14.97\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having diffraction peaks at one or more of the following positions: $9.93\pm0.2^\circ$, $10.13\pm0.2^\circ$, $11.03\pm0.2^\circ$, $11.88\pm0.2^\circ$, $17.56\pm0.2^\circ$, $18.41\pm0.2^\circ$, $18.70\pm0.2^\circ$, $20.68\pm0.2^\circ$, $22.11\pm0.2^\circ$, $25.43\pm0.2^\circ$, $25.98\pm0.2^\circ$, $27.35\pm0.2^\circ$, $27.52\pm0.2^\circ$, $28.08\pm0.2^\circ$, and $29.17\pm0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation with diffraction peaks as shown in Table 15.

In one embodiment, crystalline form A of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 27.

In one embodiment, crystalline form A of the compound of formula IX has a differential scanning calorimetry (DSC) curve with endothermic peaks at peak temperatures of $95.83\pm3^\circ\text{C}$ and $194.7\pm3^\circ\text{C}$.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form A of the compound of formula IX is substantially as shown in FIG. 28.

In one embodiment, crystalline form A of the compound of formula IX has a thermogravimetric analysis (TGA) curve showing a weight loss of about 2.03% in the temperature range from $30.52\pm3^\circ\text{C}$ to $60.0\pm3^\circ\text{C}$, a weight loss of about 5.13% in the temperature range from $60.0\pm3^\circ\text{C}$ to $100.0\pm3^\circ\text{C}$, and a weight loss of about 0.5% in the temperature range from $100.0\pm3^\circ\text{C}$ to $190.0\pm3^\circ\text{C}$.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form A of the compound of formula IX is substantially as shown in FIG. 29.

In one embodiment, in crystalline form A of the compound of formula IX, g is water, d is 1, and h is 3.

In one embodiment, the compound of formula IX is a single crystal of the compound of formula IX, wherein

g is water, h is 3, and d is 1;

it has the following unit cell parameters: triclinic crystal system (Triclinic), space group $P\bar{1}$; $a = 10.6990(12) \text{ \AA}$, $\alpha = 89.299(7)^\circ$, $b = 10.7640(13) \text{ \AA}$, $\beta = 86.519(6)^\circ$, $c = 14.9358(17) \text{ \AA}$, $\gamma = 84.364(6)^\circ$, unit cell volume = $1708.6(3) \text{ \AA}^3$, number of asymmetric units per unit cell $Z = 2$, crystal density is 1.46 mg/m^3 .

In one embodiment, the compound of formula IX is crystalline form B of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, has diffraction peaks at $5.34 \pm 0.2^\circ$, $9.34 \pm 0.2^\circ$, $9.92 \pm 0.2^\circ$, $12.84 \pm 0.2^\circ$, and $16.61 \pm 0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having diffraction peaks at one or more of the following positions: $8.20 \pm 0.2^\circ$, $10.13 \pm 0.2^\circ$, $11.12 \pm 0.2^\circ$, $13.18 \pm 0.2^\circ$, $16.13 \pm 0.2^\circ$, $18.67 \pm 0.2^\circ$, $19.47 \pm 0.2^\circ$, and $19.85 \pm 0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks at $5.34 \pm 0.2^\circ$, $9.34 \pm 0.2^\circ$, $9.92 \pm 0.2^\circ$, $12.84 \pm 0.2^\circ$, $16.61 \pm 0.2^\circ$, $8.20 \pm 0.2^\circ$, $10.13 \pm 0.2^\circ$, $13.18 \pm 0.2^\circ$, and $16.13 \pm 0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having diffraction peaks at one or more of the following positions: $11.47 \pm 0.2^\circ$, $16.36 \pm 0.2^\circ$, $18.60 \pm 0.2^\circ$, and $21.53 \pm 0.2^\circ$.

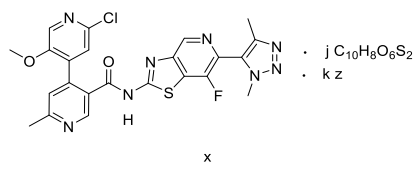
In one embodiment, crystalline form B of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks as shown in Table 16.

In one embodiment, crystalline form B of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 30.

In one embodiment, in crystalline form B of the compound of formula IX, d is 1.

In one embodiment, in crystalline form B of the compound of formula IX, h is 0.

The present invention provides a compound of formula X,



wherein $C_{10}H_8O_6S_2$ is 1,5-naphthalenedisulfonic acid;

j (molar equivalent of 1,5-naphthalenedisulfonic acid) is 0.5-1.5;

z (solvent) is water or acetonitrile;

k is 0-5.

In one embodiment, in the compound of formula X, j is 1, 0.9, 1.1, 1.02, or 0.93.

In one embodiment, in the compound of formula X, k is 0 (solvent-free).

In one embodiment, in the compound of formula X, k is 0.01-5, for example, z is water and k is 5 or 4.8; or, for example, z is acetonitrile and k is 0.07.

In one embodiment, the compound of formula X is crystalline form A of the compound of formula X, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, has diffraction peaks at $7.46\pm 0.2^\circ$, $12.01\pm 0.2^\circ$, and $21.70\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having diffraction peaks at one or more of the following positions: $11.43\pm 0.2^\circ$, $13.53\pm 0.2^\circ$, $17.24\pm 0.2^\circ$, $18.49\pm 0.2^\circ$, $18.77\pm 0.2^\circ$, $21.08\pm 0.2^\circ$, $24.57\pm 0.2^\circ$, and $28.44\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks at $7.46\pm 0.2^\circ$, $12.01\pm 0.2^\circ$, $21.70\pm 0.2^\circ$, $11.43\pm 0.2^\circ$, $13.53\pm 0.2^\circ$, $17.24\pm 0.2^\circ$, $18.49\pm 0.2^\circ$, $18.77\pm 0.2^\circ$, and $21.08\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having diffraction peaks at one or more of the following positions: $5.71\pm 0.2^\circ$, $13.93\pm 0.2^\circ$, $14.91\pm 0.2^\circ$, $15.34\pm 0.2^\circ$, $22.12\pm 0.2^\circ$, $22.80\pm 0.2^\circ$, $22.95\pm 0.2^\circ$, $24.99\pm 0.2^\circ$, and $26.08\pm 0.2^\circ$.

In one embodiment, crystalline form A of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks as shown in Table 17.

In one embodiment, crystalline form A of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 31.

In one embodiment, in crystalline form A of the compound of formula X, k is 0, and j is 1, 1.1, or 1.02, for example, 1.02.

In one embodiment, the compound of formula X is crystalline form B of the compound of formula X, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angles, has diffraction peaks at $8.96\pm0.2^\circ$, $10.81\pm0.2^\circ$, $17.92\pm0.2^\circ$, and $22.07\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angles, further having diffraction peaks at one or more of the following positions: $11.98\pm0.2^\circ$, $18.4\pm0.2^\circ$, $21.27\pm0.2^\circ$, $23.27\pm0.2^\circ$, $27.26\pm0.2^\circ$, and $27.77\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angles, further having diffraction peaks at one or more of the following positions: $5.95\pm0.2^\circ$, $14.09\pm0.2^\circ$, $15.80\pm0.2^\circ$, $16.97\pm0.2^\circ$, $20.03\pm0.2^\circ$, $22.42\pm0.2^\circ$, and $23.79\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angles, having diffraction peaks at $8.96\pm0.2^\circ$, $10.81\pm0.2^\circ$, $17.92\pm0.2^\circ$, $22.07\pm0.2^\circ$, $11.98\pm0.2^\circ$, $18.4\pm0.2^\circ$, $21.27\pm0.2^\circ$, and $23.27\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angles, having diffraction peaks as shown in Table 18.

In one embodiment, crystalline form B of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 32.

In one embodiment, in crystalline form B of the compound of formula X, k is 0, and j is 0.9, 1, or 0.93, for example, 0.93.

In one embodiment, the compound of formula X is crystalline form C of the compound of formula X, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angles, has diffraction peaks at $11.30\pm0.2^\circ$, $22.61\pm0.2^\circ$, $24.69\pm0.2^\circ$, $26.16\pm0.2^\circ$, and $28.57\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angles, further having diffraction peaks at one or more of the following positions: $6.36\pm0.2^\circ$, $12.82\pm0.2^\circ$, $17.07\pm0.2^\circ$, $17.48\pm0.2^\circ$, $18.61\pm0.2^\circ$, $20.24\pm0.2^\circ$, and $20.94\pm0.2^\circ$.

Preferably, crystalline form C of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, wherein the relative intensity of the diffraction peak at $22.61\pm0.2^\circ$ is 50%-100%, and preferably the diffraction peak at $22.61\pm0.2^\circ$ is the strongest peak.

In one embodiment, crystalline form C of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks at $11.30\pm0.2^\circ$, $22.61\pm0.2^\circ$, $24.69\pm0.2^\circ$, $26.16\pm0.2^\circ$, $28.57\pm0.2^\circ$, $6.36\pm0.2^\circ$, $18.61\pm0.2^\circ$, $12.82\pm0.2^\circ$, and $17.07\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having diffraction peaks at one or more of the following positions: $12.11\pm0.2^\circ$ and $15.12\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks as shown in Table 19.

In one embodiment, crystalline form C of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 33.

In one embodiment, in crystalline form C of the compound of formula X, j is 1.

In one embodiment, in crystalline form C of the compound of formula X, k is 0 and j is 1, or, z is acetonitrile and k is 0.07.

In one embodiment, the compound of formula X is crystalline form D of the compound of formula X, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, has diffraction peaks at $10.68\pm0.2^\circ$ and $21.44\pm0.2^\circ$; wherein z is water.

In one embodiment, crystalline form D of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having diffraction peaks at one or more of the following positions: $15.77\pm0.2^\circ$, $16.88\pm0.2^\circ$, $19.82\pm0.2^\circ$, $21.76\pm0.2^\circ$, $23.62\pm0.2^\circ$, $24.95\pm0.2^\circ$, and $27.32\pm0.2^\circ$.

In one embodiment, crystalline form D of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks at $10.68\pm0.2^\circ$, $21.44\pm0.2^\circ$, $15.77\pm0.2^\circ$, $16.88\pm0.2^\circ$, $19.82\pm0.2^\circ$, $21.76\pm0.2^\circ$, $23.62\pm0.2^\circ$, and $24.95\pm0.2^\circ$.

In one embodiment, crystalline form D of the compound of formula X has an X-ray powder

diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, further having diffraction peaks at one or more of the following positions: $7.27\pm 0.2^\circ$, $7.87\pm 0.2^\circ$, $13.59\pm 0.2^\circ$, $14.54\pm 0.2^\circ$, $16.25\pm 0.2^\circ$, $17.29\pm 0.2^\circ$, $17.66\pm 0.2^\circ$, $19.63\pm 0.2^\circ$, $20.27\pm 0.2^\circ$, $22.62\pm 0.2^\circ$, $23.09\pm 0.2^\circ$, $24.33\pm 0.2^\circ$, $24.64\pm 0.2^\circ$, $26.37\pm 0.2^\circ$, and $28.88\pm 0.2^\circ$.

In one embodiment, crystalline form D of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angles, having diffraction peaks as shown in Table 20.

In one embodiment, crystalline form D of the compound of formula X has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 34.

In one embodiment, crystalline form D of the compound of formula X has a differential scanning calorimetry (DSC) curve with endothermic peaks at peak temperatures of $62.55\pm 3^\circ\text{C}$, $86.97\pm 3^\circ\text{C}$, and $267.15\pm 3^\circ\text{C}$.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form D of the compound of formula X is substantially as shown in FIG. 35.

In one embodiment, crystalline form D of the compound of formula X has a thermogravimetric analysis (TGA) curve showing a weight loss of about 4.49% in the temperature range from $32.27\pm 3^\circ\text{C}$ to $60.0\pm 3^\circ\text{C}$, a weight loss of about 2.41% in the temperature range from $60.0\pm 3^\circ\text{C}$ to $120.0\pm 3^\circ\text{C}$, and a weight loss of about 0.6% in the temperature range from $120.0\pm 3^\circ\text{C}$ to $240.0\pm 3^\circ\text{C}$.

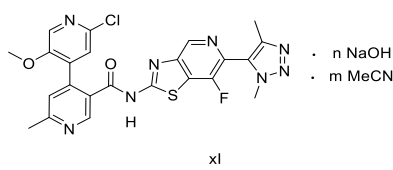
In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form D of the compound of formula X is substantially as shown in FIG. 36.

In one embodiment, in crystalline form D of the compound of formula X, j is 1 or 1.02, for example 1.02.

In one embodiment, in crystalline form D of the compound of formula X, k is 5, 4.8, or 4, for example 4.8.

In one embodiment, in crystalline form D of the compound of formula X, z is water, j is 1, and k is 5; or, j is 1.02, and k is 4.8.

The present invention provides a compound of formula XI,



wherein n (the molar equivalent of sodium ion) is 0-1.5.

In one embodiment, in the compound of formula XI, n is 0.6, 1.1, 1.13, or 1.2.

In one embodiment, in the compound of formula XI, m is 0 (solvent-free).

In one embodiment, in the compound of formula XI, m is 0.5-1.5, for example 1, 1.07, or 1.1.

In one embodiment, the compound of formula XI is crystalline form A of the compound of formula XI, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 12.78 $\pm$ 0.2 $^\circ$, 14.09 $\pm$ 0.2 $^\circ$, and 14.98 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 10.67 $\pm$ 0.2 $^\circ$, 18.82 $\pm$ 0.2 $^\circ$, 19.49 $\pm$ 0.2 $^\circ$, and 23.58 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 6.9 $\pm$ 0.2 $^\circ$, 9.39 $\pm$ 0.2 $^\circ$, 17.22 $\pm$ 0.2 $^\circ$, 19.35 $\pm$ 0.2 $^\circ$, 21.80 $\pm$ 0.2 $^\circ$, 22.45 $\pm$ 0.2 $^\circ$, 24.15 $\pm$ 0.2 $^\circ$, and 27.926 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, having diffraction peaks at 12.78 $\pm$ 0.2 $^\circ$, 14.09 $\pm$ 0.2 $^\circ$, 14.98 $\pm$ 0.2 $^\circ$, 10.67 $\pm$ 0.2 $^\circ$, 18.82 $\pm$ 0.2 $^\circ$, 19.49 $\pm$ 0.2 $^\circ$, 23.58 $\pm$ 0.2 $^\circ$, and 21.80 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form A of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, having diffraction peaks as shown in Table 21.

In one embodiment, crystalline form A of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 37.

In one embodiment, in crystalline form A of the compound of formula XI, m is 0, and n is 0.6, 0.7, or 0.62, for example 0.62.

In one embodiment, the compound of formula XI is crystalline form B of the compound of formula XI, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 8.22 $\pm$ 0.2 $^\circ$, 19.38 $\pm$ 0.2 $^\circ$, 20.38 $\pm$ 0.2 $^\circ$, 23.43 $\pm$ 0.2 $^\circ$, and 24.74 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form B of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having

diffraction peaks at one or more of the following: $9.31\pm0.2^\circ$, $12.92\pm0.2^\circ$, $13.62\pm0.2^\circ$, $24.17\pm0.2^\circ$, and $27.37\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks at $8.22\pm0.2^\circ$, $19.38\pm0.2^\circ$, $20.38\pm0.2^\circ$, $23.43\pm0.2^\circ$, $24.74\pm0.2^\circ$, $9.31\pm0.2^\circ$, $12.92\pm0.2^\circ$, and $24.17\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $15.13\pm0.2^\circ$, $16.29\pm0.2^\circ$, $17.63\pm0.2^\circ$, $17.77\pm0.2^\circ$, $18.12\pm0.2^\circ$, $18.63\pm0.2^\circ$, $21.04\pm0.2^\circ$, $21.28\pm0.2^\circ$, and $26.46\pm0.2^\circ$.

In one embodiment, crystalline form B of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks as shown in Table 22.

In one embodiment, crystalline form B of the compound of formula XI has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 38.

In one embodiment, in crystalline form B of the compound of formula XI, n is 1.1, 1.2, or 1.13, for example 1.13.

In one embodiment, in crystalline form B of the compound of formula XI, n is 1.1 and m is 1.1; or, n is 1.13 and m is 1.07.

In one embodiment, the compound of formula IX is crystalline form C of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.23\pm0.2^\circ$ and $19.82\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $9.21\pm0.2^\circ$, $16.11\pm0.2^\circ$, $16.57\pm0.2^\circ$, $19.39\pm0.2^\circ$, and $20.01\pm0.2^\circ$.

Preferably, crystalline form C of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, wherein the relative intensity of the diffraction peak at $19.82\pm0.2^\circ$ is 60%-100%, preferably the relative intensity is 70%-80%, for example 71.2%.

In one embodiment, crystalline form C of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction

peaks at $5.23\pm0.2^\circ$, $19.82\pm0.2^\circ$, $9.21\pm0.2^\circ$, $16.11\pm0.2^\circ$, $16.57\pm0.2^\circ$, $19.39\pm0.2^\circ$, $9.80\pm0.2^\circ$, $11.12\pm0.2^\circ$, and $20.01\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $8.07\pm0.2^\circ$, $8.26\pm0.2^\circ$, $9.80\pm0.2^\circ$, $11.12\pm0.2^\circ$, $12.72\pm0.2^\circ$, $15.24\pm0.2^\circ$, $15.48\pm0.2^\circ$, $18.20\pm0.2^\circ$, $18.71\pm0.2^\circ$, $21.52\pm0.2^\circ$, $23.51\pm0.2^\circ$, and $23.83\pm0.2^\circ$.

In one embodiment, crystalline form C of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks as shown in Table 23.

In one embodiment, crystalline form C of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 39.

In one embodiment, in crystalline form C of the compound of formula IX, d is 1, 1.1, or 1.02, for example 1.02.

In one embodiment, in crystalline form C of the compound of formula IX, g is tetrahydrofuran, d is 1, and h is 0.2; or, g is tetrahydrofuran, d is 1.02, and h is 0.16; or, d is 1 and h is 0.

In one embodiment, the compound of formula IX is crystalline form D of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.13\pm0.2^\circ$, $10.87\pm0.2^\circ$, and $18.38\pm0.2^\circ$.

In one embodiment, crystalline form D of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $8.52\pm0.2^\circ$, $12.18\pm0.2^\circ$, $13.12\pm0.2^\circ$, $16.60\pm0.2^\circ$, $20.07\pm0.2^\circ$, and $24.00\pm0.2^\circ$.

Preferably, crystalline form D of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, wherein the relative intensity of the diffraction peak at $6.13\pm0.2^\circ$ is 80%-100%, and preferably the diffraction peak at $6.13\pm0.2^\circ$ is the strongest peak.

In one embodiment, the compound of formula IX is crystalline form D of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.13\pm0.2^\circ$, $10.87\pm0.2^\circ$, $18.38\pm0.2^\circ$, $8.52\pm0.2^\circ$, $12.18\pm0.2^\circ$, $13.12\pm0.2^\circ$, $16.60\pm0.2^\circ$, $20.07\pm0.2^\circ$, and $24.00\pm0.2^\circ$.

In one embodiment, crystalline form D of the compound of formula IX has an X-ray powder

diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 14.25 $\pm$ 0.2 $^\circ$, 14.42 $\pm$ 0.2 $^\circ$, 17.00 $\pm$ 0.2 $^\circ$, 19.09 $\pm$ 0.2 $^\circ$, 19.34 $\pm$ 0.2 $^\circ$, 21.12 $\pm$ 0.2 $^\circ$, 21.28 $\pm$ 0.2 $^\circ$, 21.59 $\pm$ 0.2 $^\circ$, 21.72 $\pm$ 0.2 $^\circ$, 23.14 $\pm$ 0.2 $^\circ$, and 24.27 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form D of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, having diffraction peaks as shown in Table 24.

In one embodiment, crystalline form D of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 40.

In one embodiment, in crystalline form D of the compound of formula IX, d is 1/3.

In one embodiment, in crystalline form D of the compound of formula IX, g is water, d is 1, and h is 0, 1, or 3.

In one embodiment, the compound of formula IX is crystalline form E of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 5.66 $\pm$ 0.2 $^\circ$, 11.50 $\pm$ 0.2 $^\circ$, 17.97 $\pm$ 0.2 $^\circ$, 19.83 $\pm$ 0.2 $^\circ$, 23.95 $\pm$ 0.2 $^\circ$, and 26.58 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form E of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 13.85 $\pm$ 0.2 $^\circ$, 14.30 $\pm$ 0.2 $^\circ$, 17.74 $\pm$ 0.2 $^\circ$, 19.04 $\pm$ 0.2 $^\circ$, 21.93 $\pm$ 0.2 $^\circ$, 22.25 $\pm$ 0.2 $^\circ$, 23.08 $\pm$ 0.2 $^\circ$, and 23.43 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form E of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, having diffraction peaks at 5.66 $\pm$ 0.2 $^\circ$, 11.50 $\pm$ 0.2 $^\circ$, 17.97 $\pm$ 0.2 $^\circ$, 19.83 $\pm$ 0.2 $^\circ$, 23.95 $\pm$ 0.2 $^\circ$, 13.85 $\pm$ 0.2 $^\circ$, 14.30 $\pm$ 0.2 $^\circ$, 17.74 $\pm$ 0.2 $^\circ$, 19.04 $\pm$ 0.2 $^\circ$, and 21.93 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form E of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 8.45 $\pm$ 0.2 $^\circ$, 9.02 $\pm$ 0.2 $^\circ$, 9.84 $\pm$ 0.2 $^\circ$, 11.19 $\pm$ 0.2 $^\circ$, 12.70 $\pm$ 0.2 $^\circ$, 15.59 $\pm$ 0.2 $^\circ$, 16.56 $\pm$ 0.2 $^\circ$, 16.93 $\pm$ 0.2 $^\circ$, 18.75 $\pm$ 0.2 $^\circ$, 20.55 $\pm$ 0.2 $^\circ$, 25.05 $\pm$ 0.2 $^\circ$, and 26.98 $\pm$ 0.2 $^\circ$.

In one embodiment, crystalline form E of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, having diffraction peaks as shown in Table 25.

In one embodiment, crystalline form E of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 41.

In one embodiment, crystalline form E of the compound of formula IX has a differential scanning calorimetry (DSC) curve with endothermic peaks at peak temperatures of $102.94\pm 3^{\circ}\text{C}$ and $183.71\pm 3^{\circ}\text{C}$.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form E of the compound of formula IX is substantially as shown in FIG. 42.

In one embodiment, crystalline form E of the compound of formula IX has a thermogravimetric analysis (TGA) curve showing a weight loss of about 3.11% in the temperature range from $33.06\pm 3^{\circ}\text{C}$ to $55.0\pm 3^{\circ}\text{C}$, a weight loss of about 3.82% in the temperature range from $55.0\pm 3^{\circ}\text{C}$ to $120.0\pm 3^{\circ}\text{C}$, and a weight loss of about 1.21% in the temperature range from $120.0\pm 3^{\circ}\text{C}$ to $190.0\pm 3^{\circ}\text{C}$.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form E of the compound of formula IX is substantially as shown in FIG. 43.

In one embodiment, in crystalline form E of the compound of formula IX, d is 1, 1.1, or 1.02, for example, 1.02.

In one embodiment, in crystalline form E of the compound of formula IX, d is 1, g is isopropanol, and h is 1; or, d is 1.02, g is isopropanol, and h is 0.98.

In one embodiment, the compound of formula IX is crystalline form F of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.15\pm 0.2^{\circ}$, $16.55\pm 0.2^{\circ}$, $17.30\pm 0.2^{\circ}$, and $21.48\pm 0.2^{\circ}$.

In one embodiment, crystalline form F of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $10.34\pm 0.2^{\circ}$, $15.73\pm 0.2^{\circ}$, $16.34\pm 0.2^{\circ}$, $20.42\pm 0.2^{\circ}$, $20.72\pm 0.2^{\circ}$, $24.34\pm 0.2^{\circ}$, and $25.74\pm 0.2^{\circ}$.

In one embodiment, crystalline form F of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, with diffraction peaks at $6.15\pm 0.2^{\circ}$, $16.55\pm 0.2^{\circ}$, $17.30\pm 0.2^{\circ}$, $21.48\pm 0.2^{\circ}$, $10.34\pm 0.2^{\circ}$, $15.73\pm 0.2^{\circ}$, $16.34\pm 0.2^{\circ}$, $20.42\pm 0.2^{\circ}$, and $20.72\pm 0.2^{\circ}$.

In one embodiment, crystalline form F of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $6.99\pm 0.2^{\circ}$, $11.85\pm 0.2^{\circ}$, $12.27\pm 0.2^{\circ}$,

12.57±0.2°, 13.97±0.2°, 18.42±0.2°, 20.98±0.2°, 21.22±0.2°, 21.83±0.2°, 21.97±0.2°, 26.02±0.2°, and 26.83±0.2°.

In one embodiment, crystalline form F of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, having diffraction peaks as shown in Table 26.

In one embodiment, the X-ray powder diffraction pattern of crystalline form F of the compound of formula IX, using Cu-K α radiation, is substantially as shown in FIG. 44.

In one embodiment, crystalline form F of the compound of formula IX has a differential scanning calorimetry (DSC) curve with an endothermic peak at a peak temperature of 266.84±3°C.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form F of the compound of formula IX is substantially as shown in FIG. 45.

In one embodiment, crystalline form F of the compound of formula IX has a thermogravimetric analysis (TGA) curve showing a weight loss of about 2.2% in the temperature range from 34.19±3°C to 260.0±3°C.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form F of the compound of formula IX is substantially as shown in FIG. 46.

In one embodiment, in crystalline form F of the compound of formula IX, d is 1, 1.1, or 1.02, for example, 1.02.

In one embodiment, in crystalline form F of the compound of formula IX, d is 1, and h is 0.

In one embodiment, the compound of formula IX is crystalline form G of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 7.65±0.2°, 13.76±0.2°, 20.35±0.2°, 21.01±0.2°, and 21.57±0.2°;

g is 1,4-dioxane.

In one embodiment, crystalline form G of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 23.83±0.2°, 25.37±0.2°, 25.63±0.2°, and 27.60±0.2°.

In one embodiment, crystalline form G of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, with diffraction peaks at 7.65±0.2°, 13.76±0.2°, 20.35±0.2°, 21.01±0.2°, 21.57±0.2°, 23.83±0.2°, 25.37±0.2°, 25.63±0.2°, and 27.60±0.2°.

25.63±0.2°, and 27.60±0.2°.

In one embodiment, crystalline form G of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 11.87±0.2°, 12.60±0.2°, 12.76±0.2°, 16.07±0.2°, 20.05±0.2°, 22.75±0.2°, and 23.83±0.2°.

In one embodiment, crystalline form G of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, with diffraction peaks as shown in Table 27.

In one embodiment, the X-ray powder diffraction pattern of crystalline form G of the compound of formula IX using Cu-K α radiation is substantially as shown in FIG. 47.

In one embodiment, in crystalline form G of the compound of formula IX, d is 1, 1.1, or 1.2, for example, 1.1.

In one embodiment, in crystalline form G of the compound of formula IX, d is 1, g is 1,4-dioxane, and h is 3; or, d is 1.1, g is 1,4-dioxane, and h is 2.96.

In one embodiment, the compound of formula IX is crystalline form H of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 8.27±0.2°, 9.70±0.2°, 16.55±0.2°, and 19.78±0.2°; g is methyl tert-butyl ether.

In one embodiment, crystalline form H of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 10.39±0.2°, 11.07±0.2°, 11.60±0.2°, 12.56±0.2°, 17.07±0.2°, 19.09±0.2°, 21.12±0.2°, 21.46±0.2°, 25.23±0.2°, and 27.27±0.2°.

In one embodiment, crystalline form H of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, with diffraction peaks at 8.27±0.2°, 9.70±0.2°, 16.55±0.2°, 19.78±0.2°, 11.07±0.2°, 12.56±0.2°, 17.07±0.2°, 19.09±0.2°, and 21.46±0.2°.

In one embodiment, crystalline form H of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, further having diffraction peaks at one or more of the following: 5.21±0.2°, 11.60±0.2°, 15.29±0.2°, 18.79±0.2°, and 23.36±0.2°.

In one embodiment, crystalline form H of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, with diffraction

peaks as shown in Table 28.

In one embodiment, crystalline form H of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 48.

In one embodiment, crystalline form H of the compound of formula IX has a differential scanning calorimetry (DSC) curve with endothermic peaks at peak temperatures of $45.53\pm 3^\circ\text{C}$, $155.23\pm 3^\circ\text{C}$, and $191.45\pm 3^\circ\text{C}$.

In one embodiment, the differential scanning calorimetry (DSC) curve of crystalline form H of the compound of formula IX is substantially as shown in FIG. 49.

In one embodiment, crystalline form H of the compound of formula IX has a thermogravimetric analysis (TGA) curve showing a weight loss of about 1.74% in the temperature range from $32.99\pm 3^\circ\text{C}$ to $100.0\pm 3^\circ\text{C}$, and a weight loss of about 5.38% in the temperature range from $100.0\pm 3^\circ\text{C}$ to $180.0\pm 3^\circ\text{C}$.

In one embodiment, the thermogravimetric analysis (TGA) curve of crystalline form H of the compound of formula IX is substantially as shown in FIG. 50.

In one embodiment, in crystalline form H of the compound of formula IX, g is methyl tert-butyl ether, and h is 0.5, 0.4, or 0.45, for example, 0.45.

In one embodiment, in crystalline form H of the compound of formula IX, d is 1:1 (i.e., 1).

In one embodiment, in crystalline form H of the compound of formula IX, g is methyl tert-butyl ether, h is 0.5, and d is 1.

In one embodiment, the compound of formula IX is crystalline form I of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.01\pm 0.2^\circ$, $10.63\pm 0.2^\circ$, and $12.79\pm 0.2^\circ$.

In one embodiment, crystalline form I of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $14.20\pm 0.2^\circ$, $16.67\pm 0.2^\circ$, $18.00\pm 0.2^\circ$, $18.74\pm 0.2^\circ$, $20.07\pm 0.2^\circ$, and $24.06\pm 0.2^\circ$.

Preferably, crystalline form I of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, wherein the relative intensity of the diffraction peak at $6.01\pm 0.2^\circ$ is 80%-100%, and preferably the diffraction peak at $6.01\pm 0.2^\circ$ is the strongest peak.

In one embodiment, crystalline form I of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction

peaks at $6.01\pm0.2^\circ$, $10.63\pm0.2^\circ$, $12.79\pm0.2^\circ$, $14.20\pm0.2^\circ$, $16.67\pm0.2^\circ$, $18.00\pm0.2^\circ$, $20.07\pm0.2^\circ$, and $24.06\pm0.2^\circ$.

In one embodiment, crystalline form I of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $11.99\pm0.2^\circ$, $17.84\pm0.2^\circ$, and $23.22\pm0.2^\circ$.

In one embodiment, crystalline form I of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks as shown in Table 29.

In one embodiment, crystalline form I of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 51.

In one embodiment, in crystalline form I of the compound of formula IX, d is 1.

In one embodiment, in crystalline form I of the compound of formula IX, h is 0.

In one embodiment, in crystalline form I of the compound of formula IX, d is 1 and h is 0.

In one embodiment, the compound of formula IX is crystalline form J of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.27\pm0.2^\circ$, $8.18\pm0.2^\circ$, $9.72\pm0.2^\circ$, $10.54\pm0.2^\circ$, $11.06\pm0.2^\circ$, $12.44\pm0.2^\circ$, $18.79\pm0.2^\circ$, and $20.21\pm0.2^\circ$.

In one embodiment, crystalline form J of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further having diffraction peaks at one or more of the following: $15.30\pm0.2^\circ$, $16.41\pm0.2^\circ$, $18.79\pm0.2^\circ$, $19.07\pm0.2^\circ$, $19.66\pm0.2^\circ$, and $21.25\pm0.2^\circ$.

In one embodiment, crystalline form J of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, having diffraction peaks as shown in Table 30.

In one embodiment, crystalline form J of the compound of formula IX has an X-ray powder diffraction pattern using Cu-K α radiation substantially as shown in FIG. 52.

In one embodiment, in crystalline form J of the compound of formula IX, d is 1.

In one embodiment, in crystalline form J of the compound of formula IX, d is 1 and h is 0.

The present invention provides a method for preparing the compound of formula II described above, comprising the step of: reacting the compound of formula II (e.g., crystalline form A of the compound of formula II) with a mixed solvent of a ketone solvent (e.g., acetone) and water

to obtain the compound of formula II; preferably, the mixed solvent of the ketone solvent (e.g., acetone) and water is a mixed solution of acetone and water in a volume ratio of 19:1 (acetone/water).

The present invention provides a method for preparing the compound of formula III described above, comprising the step of: performing a salt formation reaction of the compound of formula I with hydrochloric acid in an ester solvent (e.g., ethyl acetate) or a nitrile solvent (e.g., acetonitrile) to obtain the compound of formula III.

The present invention provides a method for preparing the compound of formula IV described above, comprising the step of: performing a salt formation reaction of the compound of formula I with sulfuric acid in an ester solvent (e.g., ethyl acetate) or a nitrile solvent (e.g., acetonitrile) to obtain the compound of formula IV.

The present invention provides a method for preparing the compound of formula V described above, comprising the step of: performing a salt formation reaction of the compound of formula I with phosphoric acid in an ester solvent (e.g., ethyl acetate) or a nitrile solvent (e.g., acetonitrile) to obtain the compound of formula V.

The present invention provides a method for preparing the compound of formula VI or formula VII described above, comprising the step of: performing a salt formation reaction of the compound of formula I with methanesulfonic acid or fumaric acid in a nitrile solvent (e.g., acetonitrile) to obtain the compound of formula VI or formula VII.

The present invention provides a method for preparing the compound of formula VIII described above, comprising the step of: performing a salt formation reaction of the compound of formula I with benzenesulfonic acid in an ester solvent (e.g., ethyl acetate) to obtain the compound of formula VIII.

The present invention provides a method for preparing the compound of formula IX described above, comprising the step of: performing a salt formation reaction of the compound of formula I with p-toluenesulfonic acid in a mixed solvent of a ketone solvent (e.g., acetone) and water, or in a nitrile solvent (e.g., acetonitrile), to obtain the compound of formula IX; preferably, the mixed solvent of the ketone solvent (e.g., acetone) and water is a mixed solution of acetone and water in a volume ratio of 19:1 (acetone/water);

or, mixing the compound of formula IX with a solvent and drying to obtain the compound of formula IX; wherein the solvent is one or more selected from an ether solvent (e.g., tetrahydrofuran, 1,4-dioxane, or methyl tert-butyl ether), an alcohol solvent (e.g., isopropanol),

and a nitrile solvent (e.g., acetonitrile).

The present invention provides a method for preparing the compound of formula X described above, comprising the step of: performing a salt formation reaction of the compound of formula I with 1,5-naphthalenedisulfonic acid in a mixed solvent of a ketone solvent (e.g., acetone) and water, an ester solvent (e.g., ethyl acetate), or a nitrile solvent (e.g., acetonitrile), to obtain the compound of formula X; preferably, the mixed solvent of the ketone solvent and water is a mixed solution of acetone and water in a volume ratio of 19:1 (acetone/water).

The present invention provides a method for preparing the compound of formula XI described above, comprising the step of: performing a salt formation reaction of the compound of formula I with NaOH in an ester solvent (e.g., ethyl acetate) to obtain the compound of formula XI.

The present invention provides a method for preparing crystalline form B of the compound of formula II, crystalline form C of the compound of formula I, crystalline form A of the compound of formula III described above, crystalline form B of the compound of formula III, crystalline form C of the compound of formula III, crystalline form A of the compound of formula IV, crystalline form B of the compound of formula IV, crystalline form A of the compound of formula V, crystalline form B of the compound of formula V, crystalline form C of the compound of formula V, crystalline form A of the compound of formula VI, crystalline form A of the compound of formula VII, crystalline form A of the compound of formula VIII, crystalline form A of the compound of formula IX, crystalline form B of the compound of formula IX, crystalline form A of the compound of formula X, crystalline form B of the compound of formula X, crystalline form C of the compound of formula X, crystalline form D of the compound of formula X, crystalline form A of the compound of formula XI, or crystalline form B of the compound of formula XI, comprising the steps of: cooling a mixture of the compound and a solvent to induce crystallization, and drying to obtain the crystalline form;

wherein, when the compound is the compound of formula II and the solvent is a mixed solvent of acetone and water, crystalline form B of the compound of formula II is obtained;

when the compound is the compound of formula II and the solvent is ethyl acetate or acetonitrile, crystalline form C of the compound of formula I is obtained;

when the compound is the compound of formula III and the solvent is ethyl acetate, crystalline

form A or crystalline form B of the compound of formula III is obtained;

when the compound is the compound of formula III and the solvent is acetonitrile, crystalline form C of the compound of formula III is obtained;

when the compound is the compound of formula IV and the solvent is ethyl acetate, crystalline form A of the compound of formula IV is obtained;

when the compound is the compound of formula IV and the solvent is acetonitrile, crystalline form B of the compound of formula IV is obtained;

when the compound is the compound of formula V and the solvent is ethyl acetate, crystalline form A of the compound of formula V is obtained;

when the compound is the compound of formula V and the solvent is acetonitrile, crystalline form B of the compound of formula V is obtained;

when the compound is the compound of formula V and the solvent is acetonitrile, and crystalline form B of the compound of formula V is added to the mixture, crystalline form C of the compound of formula V is obtained;

when the compound is the compound of formula VI and the solvent is acetonitrile, crystalline form A of the compound of formula VI is obtained;

when the compound is the compound of formula VII and the solvent is acetonitrile, crystalline form A of the compound of formula VII is obtained;

when the compound is the compound of formula VIII and the solvent is ethyl acetate, crystalline form A of the compound of formula VIII is obtained;

when the compound is the compound of formula IX and the solvent is a mixed solution of acetone and water, crystalline form A of the compound of formula IX is obtained;

when the compound is the compound of formula IX and the solvent is acetonitrile, crystalline form B of the compound of formula IX is obtained;

when the compound is the compound of formula X and the solvent is a mixed solution of acetone and water, crystalline form A of the compound of formula X is obtained;

when the compound is the compound of formula X and the solvent is ethyl acetate, crystalline form B of the compound of formula X is obtained;

when the compound is the compound of formula X and the solvent is acetonitrile, crystalline form C of the compound of formula X is obtained;

when the compound is the compound of formula X and the solvent is a mixed solution of acetone and water, crystalline form D of the compound of formula X is obtained;

when the compound is the compound of formula XI and the solvent is ethyl acetate, crystalline form A of the compound of formula XI is obtained;

when the compound is the compound of formula XI and the solvent is acetonitrile, crystalline form B of the compound of formula XI is obtained.

The present invention provides a method for preparing crystalline form C, crystalline form E, crystalline form F, crystalline form G, and crystalline form H of the compound of formula IX, comprising the following steps: subjecting a mixture of the compound and a solvent to drying and crystallization to obtain the crystalline form;

when the compound is the compound of formula IX (preferably crystalline form A of the compound of formula IX) and the solvent is tetrahydrofuran, crystalline form C of the compound of formula IX is obtained;

when the compound is the compound of formula IX (preferably crystalline form A of the compound of formula IX) and the solvent is isopropanol, crystalline form E of the compound of formula IX is obtained;

when the compound is the compound of formula IX (preferably crystalline form A of the compound of formula IX) and the solvent is acetonitrile and n-heptane, crystalline form F of the compound of formula IX is obtained;

when the compound is the compound of formula IX (preferably crystalline form A of the compound of formula IX) and the solvent is 1,4-dioxane, crystalline form G of the compound of formula IX is obtained;

when the compound is the compound of formula IX (preferably crystalline form A of the compound of formula IX) and the solvent is a mixed solution of acetonitrile and methyl tert-butyl ether, crystalline form H of the compound of formula IX is obtained.

In the preparation method, the cooling crystallization is, for example, cooling from 50°C to 25°C.

In the preparation method, the mixed solution of acetone and water may be a mixed solution of acetone and water with a volume ratio of 19:1 (acetone/water).

In the preparation method, the drying is, for example, centrifugation and/or vacuum drying.

Preferably, the method for preparing crystalline form B of the compound of formula II comprises the following steps: subjecting crystalline form A of the compound of formula II to crystallization with a mixture of acetone and water (e.g., a suspension) to obtain crystalline form B of the compound of formula II.

In the method for preparing crystalline form B of the compound of formula II, the crystallization is, for example, cooling crystallization (e.g., cooling from 50°C to 25°C) and drying (e.g., centrifugation and/or vacuum drying) to obtain crystalline form B of the compound of formula II.

In the method for preparing crystalline form B of the compound of formula II, the mixed solution of acetone and water may be a mixed solution of acetone and water with a volume ratio of 19:1 (acetone/water).

Preferably, the method for preparing crystalline form C of the compound of formula I comprises the following steps: subjecting crystalline form A of the compound of formula II to crystallization with a mixture of ethyl acetate or acetonitrile (e.g., a suspension) to obtain crystalline form C of the compound of formula I.

In one embodiment, in the method for preparing crystalline form C of the compound of formula I, the crystallization is cooling crystallization (e.g., cooling from 50°C to 25°C) and drying (e.g., centrifugation and/or vacuum drying).

Preferably, the method for preparing crystalline form A of the compound of formula III, crystalline form B of the compound of formula III, crystalline form C of the compound of formula III, crystalline form A of the compound of formula IV, crystalline form B of the compound of formula IV, crystalline form A of the compound of formula V, crystalline form B of the compound of formula V, crystalline form A of the compound of formula VI, crystalline form A of the compound of formula VII, crystalline form A of the compound of formula VIII, crystalline form A of the compound of formula IX, crystalline form B of the compound of formula IX, crystalline form A of the compound of formula X, crystalline form B of the compound of formula X, or crystalline form C of the compound of formula X comprises the following steps: subjecting a mixture of the compound of formula I, a solvent, and an acid to crystallization to obtain a crystalline form;

wherein the solvent is ethyl acetate and the acid is hydrochloric acid to obtain crystalline form A of the compound of formula III;

the solvent is ethyl acetate and the acid is hydrochloric acid to obtain crystalline form B of the compound of formula III;

the solvent is acetonitrile and the acid is hydrochloric acid to obtain crystalline form C of the compound of formula III;

the solvent is ethyl acetate and the acid is sulfuric acid to obtain crystalline form A of the

compound of formula IV;

the solvent is acetonitrile and the acid is sulfuric acid to obtain crystalline form B of the compound of formula IV;

the solvent is ethyl acetate and the acid is phosphoric acid to obtain crystalline form A of the compound of formula V;

the solvent is acetonitrile and the acid is phosphoric acid to obtain crystalline form B of the compound of formula V;

the solvent is acetonitrile and the acid is fumaric acid to obtain crystalline form A of the compound of formula VI;

the solvent is acetonitrile and the acid is methanesulfonic acid to obtain crystalline form A of the compound of formula VII;

the solvent is ethyl acetate and the acid is benzenesulfonic acid to obtain crystalline form A of the compound of formula VIII;

the solvent is a mixed solution of acetone and water and the acid is p-toluenesulfonic acid to obtain crystalline form A of the compound of formula IX;

the solvent is acetonitrile and the acid is p-toluenesulfonic acid to obtain crystalline form B of the compound of formula IX;

the solvent is a mixed solution of acetone and water and the acid is 1,5-naphthalenedisulfonic acid to obtain crystalline form A of the compound of formula X;

the solvent is ethyl acetate and the acid is 1,5-naphthalenedisulfonic acid to obtain crystalline form B of the compound of formula X;

the solvent is acetonitrile and the acid is 1,5-naphthalenedisulfonic acid to obtain crystalline form C of the compound of formula X.

Preferably, in the preparation method, the crystallization is cooling crystallization (e.g., cooling from 50°C to 25°C) and drying (e.g., vacuum drying).

Preferably, in the preparation method, the molar ratio of the hydrochloric acid to the compound of formula I may be (0.8-1.2):1 (preferably 1:1) to obtain crystalline form A of the compound of formula III.

Preferably, in the preparation method, the molar ratio of the hydrochloric acid to the compound of formula I may be (1.6-2.2):1 (preferably 2:1) to obtain crystalline form B of the compound of formula III.

Preferably, in the preparation method, the mixed solution of acetone and water is a mixed

solution of acetone and water with a volume ratio of 19:1 (acetone/water).

Preferably, in the preparation method, the compound of formula I is in a conventional form in the art (e.g., amorphous form of the compound of formula I or crystalline form A of the compound of formula II). Preferably, when obtaining crystalline form A of the compound of formula III, crystalline form B of the compound of formula III, crystalline form C of the compound of formula III, or crystalline form A of the compound of formula IV, the compound of formula I is crystalline form A of the compound of formula II.

Preferably, in the preparation method, when the acid is hydrochloric acid, sulfuric acid, or phosphoric acid, the acid is an ethyl acetate solution of the acid, for example, a mixed solution obtained by mixing a concentrated aqueous acid solution with ethyl acetate in a volume ratio of 1:9 (acid/ethyl acetate).

Preferably, the preparation method for crystalline form C of the compound of formula V comprises the following steps: mixing a mixture of the compound of formula I, acetonitrile, and phosphoric acid with crystalline form B of the compound of formula V, followed by crystallization to obtain crystalline form C of the compound of formula V;

wherein the compound of formula I and the acid are as described in any embodiment of the present invention.

Preferably, in the preparation method for crystalline form C of the compound of formula V, the crystallization is cooling crystallization (e.g., cooling from 50°C to 25°C) and drying (e.g., vacuum drying).

Preferably, the preparation methods for crystalline form C, crystalline form E, crystalline form F, crystalline form G, and crystalline form H of the compound of formula IX comprise the following step: crystallizing a mixture of crystalline form A of the compound of formula IX and a solvent;

wherein the solvent is tetrahydrofuran to obtain crystalline form C of the compound of formula IX;

the solvent is isopropanol to obtain crystalline form E of the compound of formula IX;

the solvent is acetonitrile and n-heptane to obtain crystalline form F of the compound of formula IX;

the solvent is 1,4-dioxane to obtain crystalline form G of the compound of formula IX;

the solvent is a mixed solution of acetonitrile and methyl tert-butyl ether (e.g., a mixed solution with a volume ratio of 1:4 (acetonitrile/methyl tert-butyl ether)) to obtain crystalline form H of

the compound of formula IX.

In one embodiment, in the preparation method, the crystallization is drying crystallization (e.g., vacuum drying at 50°C).

In one embodiment, in the preparation method, when obtaining crystalline form G of the compound of formula IX, the crystallization is filtration.

In one embodiment, in the preparation method, when obtaining crystalline form F of the compound of formula IX, the preparation method comprises the following step: adding n-heptane to a mixed solvent of crystalline form A of the compound of formula IX and acetonitrile, followed by crystallization.

In one embodiment, in the preparation method, when obtaining crystalline form H of the compound of formula IX, the preparation method comprises the following step: adding methyl tert-butyl ether to a mixed solvent of crystalline form A of the compound of formula IX and acetonitrile, followed by crystallization.

The present invention provides a preparation method for crystalline form I of the compound of formula IX, comprising the following step: heating crystalline form A of the compound of formula IX at 25°C (preferably under nitrogen protection) to obtain crystalline form I of the compound of formula IX.

Preferably, the preparation method for crystalline form D of the compound of formula IX comprises the following step: heating crystalline form A of the compound of formula IX at 55°C (preferably under nitrogen protection) to obtain crystalline form D of the compound of formula IX.

Preferably, the preparation method for crystalline form J of the compound of formula IX comprises the following step: heating crystalline form H of the compound of formula IX at 170°C (preferably under nitrogen protection) to obtain crystalline form J of the compound of formula IX.

The present invention provides a preparation method for a single crystal of the compound of formula IX, comprising the following steps:

Step (1): mixing a mixture of the compound of formula I, acetone, and water with crystalline form A of the compound of formula IX;

Step (2): adding a mixture of toluene-4-sulfonic acid, acetone and water, and crystallizing to obtain a single crystal of the compound of formula IX.

In the preparation method for the single crystal of the compound of formula IX, in step (1), the mass ratio of the compound of formula I to acetone may be (0.5-2):(8-12), for example, 1.63:10.3.

In the preparation method for the single crystal of the compound of formula IX, in step (1), the mass ratio of acetone to water may be (9-11):(0.5-1.5), for example, 10.3:1.3.

In the preparation method for the single crystal of the compound of formula IX, in step (1), the mixing may be performed at 45-55°C.

In the preparation method for the single crystal of the compound of formula IX, in step (1), the mass ratio of crystalline form A of the compound of formula IX to the compound of formula I may be 1:100 to 1:10, for example, 8:163.

In the preparation method for the single crystal of the compound of formula IX, in step (2), the mass ratio of toluene-4-sulfonic acid, acetone, and water may be (1-2.5):(5-10):(0.5-1.5), for example, 0.58:2.57:0.32.

In the preparation method for the single crystal of the compound of formula IX, in step (2), the toluene-4-sulfonic acid may be toluene-4-sulfonic acid monohydrate.

In the preparation method for the single crystal of the compound of formula IX, in step (2), the mixture may be added in two portions, preferably the first addition is performed at 45-55°C, with a mass ratio of toluene-4-sulfonic acid to the compound of formula I of 1:(2-4), for example, 0.58:1.63, and the second addition is performed at 20-30°C, with a mass ratio of toluene-4-sulfonic acid to the compound of formula I of 1:(4-8), for example, 0.29:1.63.

In one embodiment, in the preparation method for the single crystal of the compound of formula IX, after the crystallization, filtration is performed to obtain the single crystal of the compound of formula IX.

Preferably, the preparation method for crystalline form D of the compound of formula X comprises the following step: crystallizing a mixture of crystalline form A of the compound of formula X, a mixed solution of acetone and water, and 1,5-naphthalenedisulfonic acid to obtain crystalline form D of the compound of formula X.

In one embodiment, in the preparation method for crystalline form D of the compound of formula X, the crystallization is cooling crystallization (e.g., suspension cooling from 50°C to 25°C) and drying (e.g., vacuum drying).

In one embodiment, in the preparation method for crystalline form D of the compound of formula X, the mixed solution of acetone and water is a mixed solution of acetone and water

with a volume ratio of 19:1 (acetone/water).

Preferably, the preparation method for crystalline form A or crystalline form B of the compound of formula XI comprises the following step: crystallizing a mixture of the compound of formula I, a solvent, and a sodium salt (e.g., NaOH) to obtain a crystalline form of the sodium salt of the compound of formula I or a solvate thereof;

wherein, when the solvent is ethyl acetate, crystalline form A of the compound of formula XI is obtained;

when the solvent is acetonitrile, crystalline form B of the compound of formula XI is obtained.

In one embodiment, in the preparation method for crystalline form A or crystalline form B of the compound of formula XI, the crystallization is cooling crystallization (e.g., suspension cooling from 50°C to 25°C) and drying (e.g., vacuum drying).

The present invention provides a pharmaceutical composition comprising substance X and a pharmaceutically acceptable excipient, wherein said substance X is the compound of formula II, III, IV, V, VI, VII, VIII, IX, X, or XI as described above, or crystalline form C of the compound of formula I as described above.

The present invention provides a use of substance X or the pharmaceutical composition as described above in the manufacture of a medicament, wherein in said use, said medicament is for treating lung cancer, breast cancer, HR-deficient ovarian cancer, gastric cancer, prostate cancer, pancreatic cancer, or colon cancer; said substance X is the compound of formula II, III, IV, V, VI, VII, VIII, IX, X, or XI as described above, or crystalline form C of the compound of formula I as described above.

Preferably, said substance X is crystalline form A of the compound of formula II, crystalline form B of the compound of formula II, crystalline form A of the compound of formula III, crystalline form B of the compound of formula III, crystalline form C of the compound of formula III, crystalline form A of the compound of formula IV, crystalline form B of the compound of formula IV, crystalline form A of the compound of formula V, crystalline form B of the compound of formula V, crystalline form C of the compound of formula V, crystalline form A of the compound of formula VI, crystalline form A of the compound of formula VII, crystalline form A of the compound of formula VIII, crystalline form A of the compound of formula X, crystalline form B of the compound of formula X, crystalline form C of the compound of formula X, crystalline form D of the compound of formula X, crystalline form A

of the compound of formula XI, crystalline form B of the compound of formula XI, crystalline form A of the compound of formula IX, crystalline form B of the compound of formula IX, crystalline form C of the compound of formula IX, crystalline form D of the compound of formula IX, crystalline form E of the compound of formula IX, crystalline form F of the compound of formula IX, crystalline form G of the compound of formula IX, crystalline form H of the compound of formula IX, crystalline form I of the compound of formula IX, or crystalline form J of the compound of formula IX.

Definition of Terms:

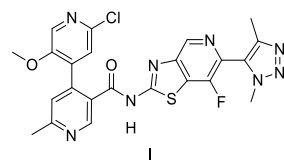
As used herein and unless otherwise specified, the term "solvate" refers to a crystalline form of a substance containing solvent in its crystal structure. The term "hydrate" refers to a solvate in which the solvent in the crystal structure is water.

As used herein and unless otherwise specified, the terms "about" and "approximately," when used in connection with providing a numerical value or range of values to characterize a specific solid form, indicate that the value or range of values may deviate to an extent considered reasonable by a person skilled in the art (e.g., taking error into account) while still describing said specific solid form: for example, a specific temperature or temperature range describing a melting, dehydration, desolvation, or glass transition temperature; a mass change, such as a mass change with temperature or humidity; a solvent content or water content in units such as mass or percentage; or a peak position in an analysis such as by IR or Raman spectroscopy or XRPD, or a peak position (temperature of thermal event) in an analysis such as DSC or TGA. For example, in specific embodiments, the terms "about" and "approximately," when used in this context, indicate that the numerical value or range of values may vary within $\pm 5\%$, $\pm 4\%$, $\pm 3\%$, $\pm 2\%$, $\pm 1.5\%$, $\pm 1\%$, $\pm 0.5\%$, or $\pm 0.25\%$ of said value or range of values. The tilde (i.e., "~") preceding a numerical value or range of values as used herein indicates "about" or "approximately."

As used herein and unless otherwise specified, in an X-ray powder diffraction pattern, the position of a diffraction peak or the relative intensity of diffraction peaks may vary due to factors such as the measurement instrument, measurement method/conditions, etc. For any specific crystalline form, there may be an error in the peak positions, for example, the measurement error for the 2θ value is $\pm 0.2^\circ$. Therefore, when identifying each crystalline form, this error should be taken into account, and variations within this error also fall within the

scope of the present application.

As used herein, when referring to a specific salt, composition, excipient, etc., as "pharmaceutically acceptable," it means that the salt, composition, excipient, etc., is generally non-toxic, safe, and suitable for use in a subject, preferably a mammalian subject, more preferably a human subject.



As used herein, the structure of the compound of formula I is:

As used herein and unless otherwise specified, the term "excipient" refers to those excipients widely adopted in the field of pharmaceutical production. Excipients are primarily used to provide a safe, stable, and functional pharmaceutical composition, and may also provide means to achieve a desired dissolution rate of the active ingredient after administration to a subject, or to facilitate effective absorption of the active ingredient after administration of the composition to a subject. An excipient may be an inert filler, or it may provide a certain function, such as stabilizing the overall pH of the composition or preventing degradation of the active ingredient in the composition.

Without departing from common knowledge in the art, the aforementioned preferred conditions can be combined in any manner to obtain various preferred examples of the present invention.

The reagents and raw materials used in the present invention are all commercially available.

The positive and progressive effects of the present invention are as follows: the salt forms and crystalline forms of the nitrogen-containing fused-ring compound provided by the present invention exhibit good drug activity, possess favorable stability and solubility, and have promising pharmaceutical prospects.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula II.

FIG. 2 is a differential scanning calorimetry thermogram of crystalline form A of the compound of formula II.

FIG. 3 is a thermogravimetric analysis thermogram of crystalline form A of the compound of

formula II.

FIG. 4 is an X-ray powder diffraction pattern of crystalline form B of the compound of formula II.

FIG. 5 is an X-ray powder diffraction pattern of crystalline form C of the compound of formula I.

FIG. 6 is a differential scanning calorimetry thermogram of crystalline form C of the compound of formula I.

FIG. 7 is a thermogravimetric analysis thermogram of crystalline form C of the compound of formula I.

FIG. 8 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula III.

FIG. 9 is an X-ray powder diffraction pattern of crystalline form B of the compound of formula III.

FIG. 10 is a differential scanning calorimetry thermogram of crystalline form B of the compound of formula III.

FIG. 11 is a thermogravimetric analysis thermogram of crystalline form B of the compound of formula III.

FIG. 12 is an X-ray powder diffraction pattern of crystalline form C of the compound of formula III.

FIG. 13 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula IV.

FIG. 14 is an X-ray powder diffraction pattern of crystalline form B of the compound of formula IV.

FIG. 15 is a differential scanning calorimetry thermogram of crystalline form B of the compound of formula IV.

FIG. 16 is a thermogravimetric analysis thermogram of crystalline form B of the compound of formula IV.

FIG. 17 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula V.

FIG. 18 is a differential scanning calorimetry thermogram of crystalline form A of the compound of formula V.

FIG. 19 is a thermogravimetric analysis thermogram of crystalline form A of the compound of

formula V.

FIG. 20 is an X-ray powder diffraction pattern of crystalline form B of the compound of formula V.

FIG. 21 is an X-ray powder diffraction pattern of crystalline form C of the compound of formula V.

FIG. 22 is a differential scanning calorimetry thermogram of crystalline form C of the compound of formula V.

FIG. 23 is a thermogravimetric analysis thermogram of crystalline form C of the compound of formula V.

FIG. 24 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula VI.

FIG. 25 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula VII.

FIG. 26 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula VIII.

FIG. 27 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula IX.

FIG. 28 is a differential scanning calorimetry thermogram of crystalline form A of the compound of formula IX.

FIG. 29 is a thermogravimetric analysis thermogram of crystalline form A of the compound of formula IX.

FIG. 30 is an X-ray powder diffraction pattern of crystalline form B of the compound of formula IX.

FIG. 31 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula X.

FIG. 32 is an X-ray powder diffraction pattern of crystalline form B of the compound of formula X.

FIG. 33 is an X-ray powder diffraction pattern of crystalline form C of the compound of formula X.

FIG. 34 is an X-ray powder diffraction pattern of crystalline form D of the compound of formula X.

FIG. 35 is a differential scanning calorimetry thermogram of crystalline form D of the

compound of formula X.

FIG. 36 is a thermogravimetric analysis thermogram of crystalline form D of the compound of formula X.

FIG. 37 is an X-ray powder diffraction pattern of crystalline form A of the compound of formula XI.

FIG. 38 is an X-ray powder diffraction pattern of crystalline form B of the compound of formula XI.

FIG. 39 is an X-ray powder diffraction pattern of crystalline form C of the compound of formula IX.

FIG. 40 is an X-ray powder diffraction pattern of crystalline form D of the compound of formula IX.

FIG. 41 is an X-ray powder diffraction pattern of crystalline form E of the compound of formula IX.

FIG. 42 is a differential scanning calorimetry thermogram of crystalline form E of the compound of formula IX.

FIG. 43 is a thermogravimetric analysis thermogram of crystalline form E of the compound of formula IX.

FIG. 44 is an X-ray powder diffraction pattern of crystalline form F of the compound of formula IX.

FIG. 45 is a differential scanning calorimetry thermogram of crystalline form F of the compound of formula IX.

FIG. 46 is a thermogravimetric analysis thermogram of crystalline form F of the compound of formula IX.

FIG. 47 is an X-ray powder diffraction pattern of crystalline form G of the compound of formula IX.

FIG. 48 is an X-ray powder diffraction pattern of crystalline form H of the compound of formula IX.

FIG. 49 is a differential scanning calorimetry thermogram of crystalline form H of the compound of formula IX.

FIG. 50 is a thermogravimetric analysis thermogram of crystalline form H of the compound of formula IX.

FIG. 51 is an X-ray powder diffraction pattern of crystalline form I of the compound of

formula IX.

FIG. 52 is an X-ray powder diffraction pattern of crystalline form J of the compound of formula IX.

FIG. 53 shows XRPD patterns of crystalline form C of the compound of formula I after storage under open conditions at 25°C/60% RH and under sealed conditions at 60°C.

FIG. 54 shows XRPD patterns of crystalline form C of the compound of formula V after storage under open conditions at 25°C/60% RH and under sealed conditions at 60°C.

FIG. 55 shows XRPD patterns of crystalline form A of the compound of formula IX after storage under open conditions at 25°C/60% RH and under sealed conditions at 60°C.

FIG. 56 shows XRPD patterns of crystalline form F of the compound of formula IX after storage under open conditions at 25°C/60% RH and under sealed conditions at 60°C.

FIG. 57 is a molecular structure diagram of a single crystal of the compound of formula IX.

DETAILED DESCRIPTION

The present invention is further illustrated by the following examples, which are not intended to limit the scope of the present invention. For experimental methods without specified conditions in the following examples, conventional methods and conditions were followed, or instructions of commercial products were selected.

The test method for X-ray powder diffraction (XRPD) involved in the present invention is shown in the following table:

Parameter	
Model	Bruker A8 advance X-ray diffractometer
X-ray tube setting	Cu/K-Alpha1 ($\lambda = 1.5406\text{\AA}$)
Tube voltage	40 kV
Tube current	40 mA
Scan range	2°~40°
Step size	0.02°
Step time	0.4 or 0.3 or 0.12 or 0.06 seconds
Sample stage rotation speed	15 rpm

The single crystal test method involved in the present invention is as follows:

Model: Bruker A8 advance X-ray diffractometer

X-ray source: High-intensity micro-focus Cu/Mo automatic switching dual-source system, using diamond heat conduction technology, with Mo source power of 70 W and Cu source power of 60 W.

Micro-focus source, spot size not larger than 100 μm .

X-ray optics system, multilayer film micro-focus optics system.

Goniometer: Kappa (Kappa, ω , 2θ , ϕ) four-circle goniometer, equipped with an automatic goniometer head.

Detector: New semiconductor two-dimensional imaging technology detector, with both photon counting and integration functions; detection area $208 \times 128 \text{ mm}^2$; pixel size: $\leq 135 \mu\text{m} \times 135 \mu\text{m}$, 1:1 coupling with the chip, no beam cone ratio.

Low-temperature cooling system: Supports testing in the temperature range of 80 K to 500 K.

The test methods for thermogravimetric analysis (TGA) curves and differential scanning calorimetry (DSC) curves involved in the present invention are shown in the following table:

Parameter	DSC	TGA
Model	TA Instruments Discovery 2500	TA Instruments Discovery 5500
Sample pan	Tzero pan and sealed lid with a manually punched pinhole of about 0.7 mm in diameter	Aluminum pan
Temperature range	0~250°C	Room temperature to 300°C
Heating rate (°C/min)	10°C/min	10°C/min
Purge gas	N ₂ , 50 mL/min	N ₂ , balance 10 mL/min, Sample chamber 25 mL/min

The hygroscopicity characterization and the definition of hygroscopic weight gain involved in the present invention (refer to the Hygroscopicity Test Guideline for Drugs, Part IV, Chinese Pharmacopoeia 2020 Edition) are as follows:

Deliquescent: Absorbs sufficient moisture to form a liquid;

Extremely hygroscopic: Hygroscopic weight gain not less than 15%;

Hygroscopic: Hygroscopic weight gain less than 15% but not less than 2%;

Slightly hygroscopic: Hygroscopic weight gain less than 2% but not less than 0.2%;

Non-hygroscopic or almost non-hygroscopic: Hygroscopic weight gain less than 0.2%.

The water content test method is as follows: Karl Fischer titration (KF)

Equipment model	Metrohm 851/885 system
Method	Coulometric titration
Oven temperature (Oven temperature)	150°C
Extraction time	120 s
Nitrogen flow rate	50 mL/min
Sample amount	~5-30 mg

The ion chromatography test conditions involved in this application are as follows:

Instrument model	Metrohm 940 professional IC
Liquid collector	889 IC
Detector	Conductivity detector
Eluent (anion)	3.2 mmol/L aqueous sodium carbonate solution + 1.0 mmol/L aqueous sodium bicarbonate solution
Eluent (cation)	1.7 mmol/L HNO ₃ + 0.7 mmol/L pyridinecarboxylic acid
Suppressor solutions	2% H ₃ PO ₄
Column	Anion A SUPP 5-150 or Cation Column C4-150
Column temperature	30°C
Flow rate	0.7 mL/min (anion) or 0.9 mL/min (cation)
Diluent	ACN/water (1 : 1; v : v)
Injection volume	20 µL

The nuclear magnetic resonance (NMR) test conditions involved in this application are as follows:

Equipment model	Bruker Avance-AV 400M (for ¹ H NMR)
Frequency	400 MHz
Probe	5 mm PABBO BB/19F-1H/D Z-GRD Z108618/0406 (for ¹ H NMR)
Number of scans	8
Temperature	297.6K
Relaxation delay	1 second

The polarized light microscopy (PLM) test conditions involved in this application are as follows:

Equipme	Leica DM4 P
---------	-------------

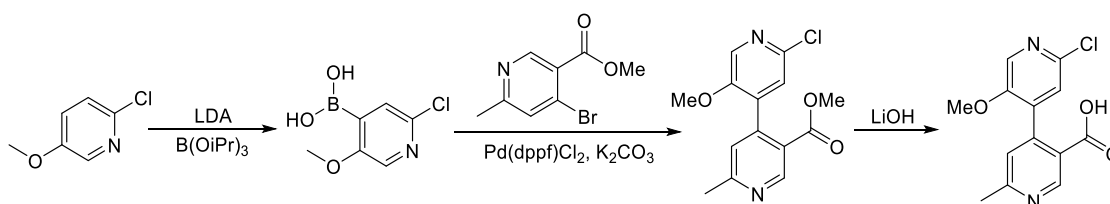
nt model	
Method	Crossed polarizers, with silicone oil (Crossed polarizer, silicone oil added)

The high-performance liquid chromatography (HPLC) test conditions involved in this application are as follows:

Equipment model	SHIMADZU LC-40		
UPLC method	Column: XBridge-C18 4.6 mm*150 mm 3.5 μm		
	Detector: DAD		
	Wavelength: 220 nm		
	Column temperature: 40°C		
	Flow rate: 1.2 mL/min		
	Mobile phase A: 0.1% TFA aqueous solution		
	Mobile phase B: Acetonitrile		
	Diluent: ACN/water (1 : 1; v : v)		
	Injection volume: 5 μL		
Gradient setting:			
	Time (min)	Mobile phase A (%)	Mobile phase B (%)
	0.0	95	5
	9.0	5	95
	13.0	5	95
	13.1	95	5
	17.0	95	5

Example 1. Preparation method of the compound of Formula II

Intermediate 1: 2'-chloro-5'-methoxy-6-methyl-[4,4'-bipyridine]-3-carboxylic acid



Step 1: (2-chloro-5-methoxypyridin-4-yl)boronic acid

2-Chloro-5-methoxypyridine (10.0 g, 69.5 mmol) was dissolved in 250 mL of tetrahydrofuran,

and under nitrogen protection, the solution was cooled to -65°C. A 2 mol/L solution of lithium diisopropylamide (70 mL) was added dropwise slowly while maintaining the temperature below -60°C. After the addition was complete, the reaction was maintained at the low temperature for 2 hours. Then, triisopropyl borate (26.2 g, 139 mmol) was added dropwise slowly at -65°C. The mixture was stirred at -65°C for an additional hour and then allowed to warm to room temperature overnight. The reaction mixture was quenched by adding 100 mL of water under an ice-water bath. The resulting aqueous solution was extracted twice with ethyl acetate, and the organic phases were discarded. The aqueous phase was adjusted to pH 5-6 with a 2M hydrochloric acid aqueous solution, resulting in the precipitation of a large amount of solid. The solid was collected by filtration under reduced pressure and dried to afford the title compound (11.4 g, white solid, yield 88.1%). LC/MS (ESI) m/z: 188.0 [M+H]⁺.

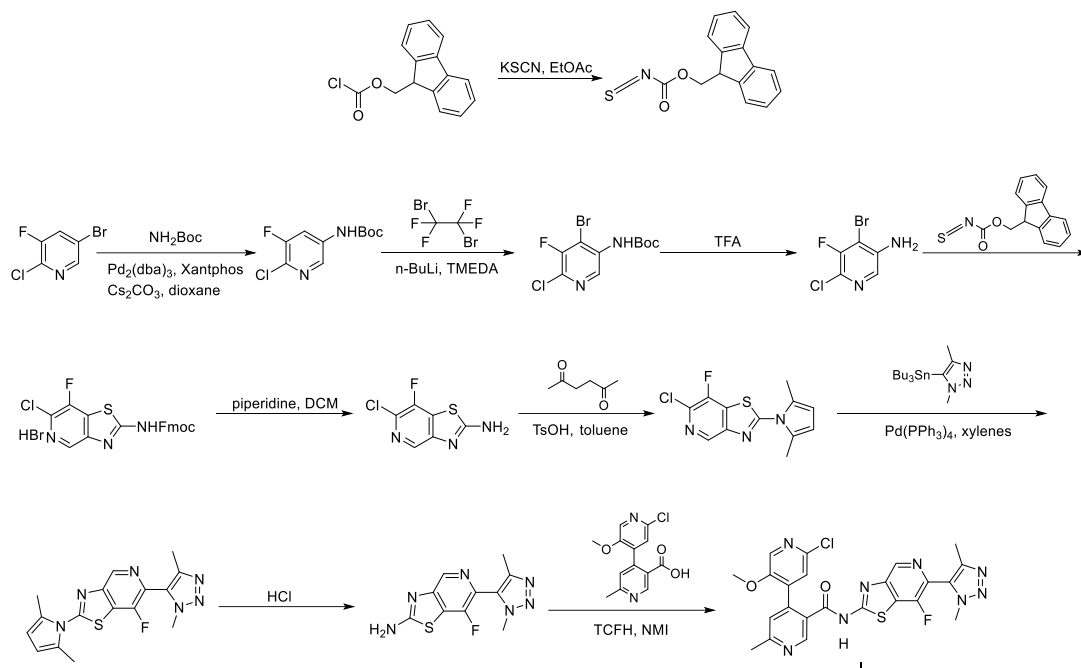
Step 2: Methyl 2'-chloro-5'-methoxy-6-methyl-[4,4'-bipyridine]-3-carboxylate

Methyl 4-bromo-6-methylnicotinate (5 g, 21.7 mmol), (2-chloro-5-methoxypyridin-4-yl)boronic acid (4.07 g, 21.7 mmol), and potassium carbonate (9 g, 65.2 mmol) were dissolved in a mixture of 180 mL of 1,4-dioxane and 36 mL of water. Under nitrogen protection, bis(triphenylphosphine)palladium(II) chloride (1.59 g, 2.17 mmol) was added, and the mixture was heated to 80°C and reacted for 2 hours. The reaction mixture was cooled to room temperature, filtered, and then partitioned with water and ethyl acetate for extraction. The organic phase was concentrated and purified by silica gel column chromatography (petroleum ether/ethyl acetate system: 0-50% ethyl acetate) to afford the title compound (5.20 g, white solid, yield 81.7%). LC/MS (ESI) m/z: 293.0 [M+H]⁺.

Step 3: 2'-chloro-5'-methoxy-6-methyl-[4,4'-bipyridine]-3-carboxylic acid

Methyl 2'-chloro-5'-methoxy-6-methyl-[4,4'-bipyridine]-3-carboxylate (5.2 g, 17.7 mmol) was dissolved in a mixture of 50 mL of tetrahydrofuran and 50 mL of water. Lithium hydroxide (0.64 g, 26.6 mmol) was added, and the mixture was stirred at room temperature overnight. After concentrating under reduced pressure to remove tetrahydrofuran, the pH was adjusted to 5-6 with hydrochloric acid, resulting in the precipitation of a large amount of white solid. The solid was collected by filtration under reduced pressure, and the filter cake was dried to afford the title compound (4.82 g, white solid, yield 96.7%). LC/MS (ESI) m/z: 279.1 [M+H]⁺.

2'-chloro-N-(6-(1,4-dimethyl-1H-1,2,3-triazol-5-yl)-7-fluorothiazolo[4,5-c]pyridin-2-yl)-5'-methoxy-6-methyl-[4,4'-bipyridine]-3-carboxamide



Step 1: O-((9H-fluoren-9-yl)methyl)carbonisothiocyanatide

To a solution of potassium thiocyanate (20.7 g, 213 mmol) in ethyl acetate (160 mL) at 0°C was added dropwise a solution of 9-fluorenylmethyl chloroformate (50 g, 193 mmol) in ethyl acetate (160 mL). The mixture was stirred at room temperature for 16 hours. The mixture was filtered, and the filtrate was concentrated under reduced pressure. The residue was purified by silica gel column chromatography (eluent: petroleum ether/ethyl acetate = 20, *V/V*) to afford the title compound (yellow oil, 16.0 g, yield 29.4%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.89 (d, *J* = 7.6 Hz, 2H), 7.69 (d, *J* = 7.6 Hz, 2H), 7.45-7.39 (m, 2H), 7.36-7.31 (m, 2H), 4.30-4.23 (m, 2H), 4.23-4.18 (m, 1H).

Step 2: Tert-butyl (6-chloro-5-fluoropyridin-3-yl)carbamate

To a solution of 2-chloro-3-fluoro-5-bromopyridine (8.00 g, 38.0 mmol), tert-butyl carbamate (4.90 g, 41.8 mmol), cesium carbonate (24.8 g, 76.0 mmol), and 4,5-bis(diphenylphosphino)-9,9-dimethylxanthene (880 mg, 1.52 mmol) in 1,4-dioxane (160 mL) was added bis(dibenzylideneacetone)palladium(0) (1.04 g, 1.14 mmol). The mixture was stirred at 85°C under nitrogen atmosphere for 20 hours. The mixture was concentrated under reduced pressure. The residue was purified by silica gel column chromatography (eluent: petroleum ether/ethyl acetate = 4, *V/V*) to afford the title compound (yellow solid, 6.10 g, yield 65.0%). LC/MS (ESI) *m/z*: 246.9 [M+H]⁺.

Step 3: Tert-butyl (4-bromo-6-chloro-5-fluoropyridin-3-yl)carbamate

Tert-butyl (6-chloro-5-fluoropyridin-3-yl)carbamate (6.10 g, 24.7 mmol) and

N,N,N',N'-tetramethylethylenediamine (8.62 g, 74.2 mmol) were dissolved in diethyl ether (130 mL). Under nitrogen atmosphere, the reaction mixture was cooled to -60°C, and 1.6 M n-butyllithium (46.4 mL, 74.2 mmol) was added dropwise. After the addition was complete, the mixture was warmed to -20°C and stirred for 1.5 hours. The reaction mixture was cooled to -60°C, and 1,2-dibromotetrafluoroethane (19.9 g, 76.7 mmol) was added dropwise. After the addition was complete, the mixture was slowly warmed to room temperature, quenched with 1 N HCl (92 mL), and extracted with ethyl acetate. The organic phase was washed with water and brine, dried over anhydrous sodium sulfate, and filtered. The mixture was concentrated under reduced pressure to afford the title compound (yellow solid, 8.0 g, yield 99.4%). LC/MS (ESI) m/z: 326.9 [M+H]⁺.

Step 4: 4-bromo-6-chloro-5-fluoropyridin-3-amine

Tert-butyl (4-bromo-6-chloro-5-fluoropyridin-3-yl)carbamate (8.0 g, 24.6 mmol) was dissolved in dichloromethane (100 mL) and trifluoroacetic acid (50 mL). The reaction mixture was stirred at room temperature for one hour. The mixture was concentrated under reduced pressure to give a residue, which was redissolved in ethyl acetate and neutralized with saturated aqueous sodium bicarbonate solution. The mixture was extracted with ethyl acetate. The organic phase was washed with water and brine, dried over anhydrous sodium sulfate, and filtered. The mixture was concentrated under reduced pressure to afford the title compound (yellow solid, 5.7 g, yield 103%). LC/MS (ESI) m/z: 224.8 [M+H]⁺.

Step 5: (9H-fluoren-9-yl)methyl (6-chloro-7-fluorothiazolo[4,5-c]pyridin-2-yl)carbamate hydrobromide

4-bromo-6-chloro-5-fluoropyridin-3-amine (5.7 g, 25.3 mmol) was dissolved in acetone (100 mL), and O-((9H-fluoren-9-yl)methyl)carbonisothiocyanatide (8.54 g, 30.3 mmol) was added. The mixture was stirred at 50°C overnight. The reaction mixture was cooled to room temperature, filtered, and the filter cake was rinsed with acetone. The solid was dried under reduced pressure to afford the title compound (7.7 g, white solid, yield 60.1%). LC/MS (ESI) m/z: 426.0 [M+H]⁺.

Step 6: 6-chloro-7-fluorothiazolo[4,5-c]pyridin-2-amine

To a solution of (9H-fluoren-9-yl)methyl (6-chloro-7-fluorothiazolo[4,5-c]pyridin-2-yl)carbamate hydrobromide (7.7 g, 18.1 mmol) in dichloromethane (80 mL) was added piperidine (17.9 mL, 181 mmol), and the mixture was stirred at room temperature for one hour. Water was added to the reaction mixture, and the mixture was back-extracted with ethyl acetate.

The aqueous phase was concentrated under reduced pressure. The residue was first purified by silica gel column chromatography (eluent: dichloromethane/methanol = 10, *V/V*), and then further purified by slurrying in dichloromethane to afford the title compound (white solid, 1.45 g, yield 39.4%). LC/MS (ESI) *m/z*: 204.1 [M+H]⁺.

Step 7: 6-chloro-2-(2,5-dimethyl-1H-pyrrol-1-yl)-7-fluorothiazolo[4,5-c]pyridine

To a solution of 6-chloro-7-fluorothiazolo[4,5-c]pyridin-2-amine (1.45 g, 7.12 mmol) and acetylacetone (1.65 g, 14.5 mmol) in toluene (30 mL) was added *p*-toluenesulfonic acid monohydrate (0.25 g, 1.42 mmol), and the mixture was refluxed at 140°C with water separation for two hours. The reaction mixture was cooled to room temperature, ethyl acetate was added, and the mixture was washed with an aqueous sodium bicarbonate solution and then with saturated brine. The mixture was concentrated under reduced pressure, and the residue was purified by silica gel column chromatography (eluent: petroleum ether/ethyl acetate = 10, *V/V*) to afford the title compound (white solid, 1.5 g, yield 74.8%). LC/MS (ESI) *m/z*: 282.1 [M+H]⁺.

Step 8: 6-(1,4-dimethyl-1H-1,2,3-triazol-5-yl)-2-(2,5-dimethyl-1H-pyrrol-1-yl)-7-fluorothiazolo[4,5-c]pyridine

To a solution of 6-chloro-2-(2,5-dimethyl-1H-pyrrol-1-yl)-7-fluorothiazolo[4,5-c]pyridine (1.5 g, 5.32 mmol) and 1,4-dimethyl-5-(tributylstannyl)-1H-1,2,3-triazole (3.08 g, 7.99 mmol) in xylene (30 mL) was added tetrakis(triphenylphosphine)palladium (1.85 g, 1.60 mmol). The mixture was stirred at 150°C under nitrogen protection for 5 hours. The mixture was concentrated under reduced pressure, and the residue was purified by silica gel column chromatography (eluent: petroleum ether/ethyl acetate = 3, *V/V*) to afford the title compound (pale yellow solid, 1.25 g, yield 68.7%). LC/MS (ESI) *m/z*: 343.1 [M+H]⁺.

Step 9: 6-(1,4-dimethyl-1H-1,2,3-triazol-5-yl)-7-fluorothiazolo[4,5-c]pyridin-2-amine hydrochloride

6-(1,4-dimethyl-1H-1,2,3-triazol-5-yl)-2-(2,5-dimethyl-1H-pyrrol-1-yl)-7-fluorothiazolo[4,5-c]pyridine (1.25 g, 3.65 mmol) was dissolved in 2 N HCl (15 mL). The reaction mixture was stirred at 80°C for two hours. The mixture was concentrated under reduced pressure, and the residue was slurried with acetonitrile to afford the title compound (pale yellow solid, 1.0 g, yield 91.1%). LC/MS (ESI) *m/z*: 265.1 [M+H]⁺.

Step 10: 2'-chloro-N-(6-(1,4-dimethyl-1H-1,2,3-triazol-5-yl)-7-fluorothiazolo[4,5-c]pyridin-2-yl)-5'-methoxy-6-methyl-[4,4'-bipyridine]-3-carboxamide

6-(1,4-dimethyl-1H-1,2,3-triazol-5-yl)-7-fluorothiazolo[4,5-c]pyridin-2-amine hydrochloride (1.0 g, 3.78 mmol), 2'-chloro-5'-methoxy-6-methyl-[4,4'-bipyridine]-3-carboxylic acid (1.16 g, 4.16 mmol), and N-methylimidazole (2.17 g, 26.5 mmol) were dissolved in acetonitrile (20 mL). The mixture was stirred at 70°C for 5 minutes, then N,N,N',N'-tetramethylchloroformamidinium hexafluorophosphate (5.31 g, 18.9 mmol) was added to the reaction mixture, and stirring was continued at 70°C for 2 hours. As the starting material remained unreacted, additional N-methylimidazole (0.62 g, 7.56 mmol) and N,N,N',N'-tetramethylchloroformamidinium hexafluorophosphate (1.06 g, 3.78 mmol) were added to the reaction mixture. Water was added to the reaction mixture, and the mixture was extracted with ethyl acetate. The organic phase was washed with water and brine, then concentrated under reduced pressure. The residue was first purified by silica gel column chromatography (eluent: dichloromethane/methanol = 20, *V/V*), then slurried with ethyl acetate to afford the title compound (834.7 mg, white solid, yield 42.0%). ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.63 (s, 1H), 9.14 (d, *J* = 2.0 Hz, 1H), 8.90 (s, 1H), 8.18 (s, 1H), 7.62 (s, 1H), 7.51 (s, 1H), 4.03 (s, 3H), 3.61 (s, 3H), 2.62 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H). LC/MS (ESI) *m/z*: 525.2 [M+H]⁺.

The obtained white solid is crystalline form A of the compound of formula I, its XRPD pattern is shown in FIG. 1, and it has the diffraction peaks shown in Table 1 below.

Table 1

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.984	14.75874	18.40%	27	24.6	3.6159	13.90%
2	9.003	9.81492	68.00%	28	25.347	3.51101	3.60%
3	10.5	8.41809	6.10%	29	25.62	3.47424	8.20%
4	10.837	8.15715	62.30%	30	26.413	3.3717	1.60%
5	11.941	7.40557	28.40%	31	27.165	3.28003	6.30%
6	13.462	6.57211	4.10%	32	27.717	3.216	63.30%
7	14.156	6.25128	8.50%	33	28.498	3.12958	7.20%
8	14.72	6.013	8.70%	34	28.956	3.08106	12.50%
9	15.31	5.78276	1.40%	35	29.184	3.0575	6.80%
10	15.455	5.72891	1.60%	36	29.679	3.00767	15.80%
11	15.79	5.6079	1.20%	37	30.294	2.94797	9.40%
12	17.153	5.16531	2.20%	38	31.083	2.87498	5.90%

13	17.91	4.94869	100.00%	39	31.992	2.7953	7.20%
14	18.357	4.82916	40.90%	40	32.218	2.77621	7.00%
15	19.02	4.66238	2.50%	41	32.567	2.74722	6.60%
16	19.994	4.43722	12.90%	42	32.731	2.73381	5.30%
17	20.649	4.29795	8.90%	43	33.609	2.66443	1.40%
18	21.304	4.16734	40.50%	44	34.17	2.62191	3.90%
19	21.71	4.0903	18.00%	45	34.631	2.58808	0.70%
20	22.052	4.02768	77.00%	46	35.461	2.52937	0.50%
21	22.448	3.9574	23.80%	47	35.815	2.50521	1.70%
22	22.631	3.92595	14.80%	48	36.41	2.46559	3.40%
23	23.291	3.81612	23.90%	49	37.525	2.39489	4.00%
24	23.679	3.75448	4.60%	50	37.961	2.36834	3.90%
25	23.96	3.71101	8.40%	51	38.629	2.32896	7.00%
26	24.139	3.68389	7.70%	52	39.669	2.27022	1.50%

The DSC pattern of crystalline form A of the compound of formula II is shown in FIG. 2. The DSC curve shows two endothermic peaks at peak temperatures of 98.33°C (enthalpy value 121.36 J/g) and 206.94°C (enthalpy value 66.798 J/g), with onset points (Onset x) at 72.55°C and 200.15°C, respectively. The TGA pattern is shown in FIG. 3. The sample lost 0.48% of its weight when heated from 34.69°C to 70.0°C, 2.11% from 70.0°C to 95.0°C, 1.38% from 95.0°C to 140.0°C, and approximately 0.34% in the temperature range from 140.0°C to 220.0°C. Based on the DSC and TGA analysis, crystalline form A is a monohydrate.

Example 1-2 Preparation of Crystalline Form C of the Compound of Formula I

Approximately 30 mg of crystalline form A of the compound of formula II was weighed and placed in a 2 mL glass vial, and 0.5 mL of ethyl acetate or acetonitrile was added to form a suspension. The resulting sample was suspended at 50°C for 2 hours, then allowed to cool naturally to 25°C and suspended at 25°C for 3 days. The resulting suspension was centrifuged through a 0.45 µm nylon filter membrane at 14,000 rpm. The obtained solid was vacuum dried at 50°C for 2 hours to yield crystalline form C.

The obtained white solid is crystalline form C of the compound of formula I, its XRPD pattern is shown in FIG. 5, and it has the diffraction peaks shown in Table 3 below. The DSC pattern of crystalline form C of the compound of formula I is shown in FIG. 6. The DSC curve shows an endothermic peak at a peak temperature of 238.92°C (enthalpy value 106.67 J/g). The TGA pattern is shown in FIG. 7. The sample lost 0.81% of its weight when heated from 35.56°C to 230.0°C. Based on the DSC and TGA analysis, crystalline form C of the free base of the

compound of formula I is an anhydrate.

Table 3

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	9.022	9.79429	16.10%	26	28.103	3.17262	2.40%
2	10.212	8.65524	11.40%	27	28.604	3.11826	2.30%
3	11.25	7.85911	3.90%	28	29.038	3.07259	4.90%
4	11.506	7.68478	100.00%	29	29.445	3.03098	24.40%
5	12.878	6.86853	28.70%	30	29.491	3.02643	14.70%
6	13.61	6.50087	6.60%	31	30.137	2.963	13.50%
7	14.575	6.07273	14.20%	32	30.354	2.94229	12.90%
8	15.052	5.88114	22.80%	33	30.683	2.91145	2.00%
9	16.163	5.47954	5.70%	34	31.261	2.85896	1.80%
10	16.96	5.22352	7.10%	35	31.719	2.81869	1.70%
11	17.22	5.14526	5.40%	36	32.564	2.74745	1.90%
12	18.011	4.92107	59.10%	37	33.128	2.70203	1.90%
13	18.91	4.68917	12.10%	38	34.117	2.62587	4.40%
14	19.944	4.44835	1.60%	39	34.866	2.57115	2.40%
15	20.479	4.33321	26.60%	40	34.973	2.56353	8.80%
16	20.853	4.25635	42.00%	41	35.477	2.5283	5.60%
17	21.676	4.09669	32.30%	42	36.053	2.48919	1.90%
18	22.56	3.93806	98.50%	43	36.415	2.46528	2.30%
19	23.106	3.84624	29.20%	44	36.885	2.43497	1.80%
20	24.789	3.58872	31.90%	45	37.062	2.42369	1.90%
21	25.679	3.46636	84.80%	46	38.06	2.36241	2.70%
22	26.207	3.39769	5.00%	47	38.395	2.34256	3.10%
23	26.937	3.30724	15.00%	48	39.217	2.29537	2.80%
24	27.259	3.26894	11.20%	49	39.732	2.26679	2.00%
25	27.439	3.24784	35.40%				

Example 2 Crystalline Form A of the Compound of Formula III

30 mg of crystalline form A of the compound of formula II was added to 0.5 mL of ethyl acetate, and the temperature was raised to 50°C. 0.05 mL of a dilute hydrochloric acid solution was added to the system (preparation method for dilute hydrochloric acid: 0.1 mL of concentrated hydrochloric acid (37% wt) dissolved in 0.9 mL of ethyl acetate). The entire system was stirred at 50°C for 2 hours, cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to yield crystalline form A of the compound of formula III. Ion chromatography showed that the molar ratio of the compound of formula I

to hydrochloric acid was 1:1.06. The XRPD pattern of crystalline form A of the compound of formula III is shown in FIG. 8, and it has the diffraction peaks shown in Table 4 below.

Table 4

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	4.61	19.15403	100.00%	18	19.13	4.63574	6.10%
2	5.023	17.58006	34.20%	19	19.304	4.59425	8.20%
3	6.572	13.43895	21.10%	20	19.964	4.44391	2.40%
4	6.892	12.81446	14.10%	21	20.808	4.26544	6.40%
5	9.191	9.61374	70.70%	22	21.684	4.09519	8.00%
6	9.966	8.86786	16.20%	23	23.087	3.84938	5.90%
7	10.48	8.43464	8.90%	24	24.031	3.70025	11.90%
8	10.641	8.30701	8.30%	25	24.497	3.6309	5.70%
9	11.12	7.95062	2.40%	26	25.141	3.53926	4.20%
10	12.193	7.25335	2.00%	27	25.56	3.48225	7.00%
11	13.76	6.4306	62.30%	28	26.127	3.4079	14.40%
12	14.686	6.02699	9.60%	29	27.539	3.23628	1.40%
13	14.902	5.94022	14.80%	30	28.173	3.16496	7.20%
14	15.952	5.55141	1.80%	31	31.795	2.81213	3.10%
15	17.087	5.18506	1.20%	32	34.164	2.62238	1.90%
16	18.101	4.89688	4.20%	33	35.404	2.53331	1.80%
17	18.344	4.83252	9.10%	34	36.041	2.48998	2.20%

Example 3 Crystalline Form B of the Compound of Formula III

30 mg of crystalline form A of the compound of formula II was added to 0.5 mL of ethyl acetate, and the temperature was raised to 50°C. 0.096 mL of a dilute hydrochloric acid solution was added to the system (preparation method for dilute hydrochloric acid: 0.1 mL of concentrated hydrochloric acid dissolved in 0.9 mL of ethyl acetate). The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to yield crystalline form B of the compound of formula III. Ion chromatography showed that the molar ratio of the compound of formula I to hydrochloric acid was 1:1.76. The XRPD pattern of crystalline form B of the compound of formula III is shown in FIG. 9, and it has the diffraction peaks shown in Table 5 below.

Table 5

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	6.469	13.65234	32.50%	34	26.962	3.30424	13.30%
2	7.191	12.28265	18.80%	35	27.149	3.28196	14.10%
3	9.111	9.69816	13.90%	36	27.471	3.24414	4.90%
4	11.634	7.60053	19.40%	37	27.956	3.18897	40.00%
5	12.94	6.83578	7.20%	38	28.616	3.11698	44.60%
6	13.406	6.59963	8.00%	39	29.004	3.07608	7.10%
7	14.45	6.125	71.00%	40	29.161	3.0599	8.10%
8	15.407	5.74645	9.70%	41	29.389	3.03669	4.40%
9	15.75	5.62221	10.40%	42	30.04	2.97233	15.60%
10	16.234	5.45541	15.50%	43	30.365	2.94122	29.10%
11	16.429	5.3914	10.70%	44	30.626	2.9168	6.10%
12	16.722	5.2973	13.20%	45	30.999	2.88256	16.20%
13	17.367	5.10202	49.40%	46	31.421	2.84476	5.90%
14	18.209	4.86807	51.20%	47	31.869	2.80577	4.20%
15	18.817	4.71221	3.70%	48	32.198	2.77791	25.10%
16	19.443	4.56187	47.20%	49	32.644	2.74097	11.10%
17	20.166	4.39987	51.30%	50	32.917	2.71881	10.10%
18	20.496	4.32982	23.70%	51	33.638	2.66217	1.30%
19	20.628	4.30239	32.20%	52	34.045	2.63129	13.20%
20	20.823	4.26252	100.00%	53	34.445	2.60161	2.00%
21	21.657	4.10023	4.60%	54	35.025	2.55988	2.90%
22	22.215	3.99843	2.30%	55	35.664	2.51546	5.10%
23	22.406	3.96476	31.50%	56	36.067	2.48825	4.60%
24	23.012	3.86166	12.10%	57	36.293	2.47331	6.50%
25	23.445	3.79133	2.80%	58	36.489	2.46042	5.50%
26	23.628	3.76249	6.90%	59	37.026	2.42601	6.20%
27	23.81	3.73402	2.50%	60	37.339	2.40634	2.70%
28	24.578	3.61916	83.80%	61	38.15	2.35709	3.90%
29	25.33	3.5134	9.00%	62	38.451	2.33931	5.20%
30	25.839	3.44522	20.50%	63	39.006	2.30729	2.50%
31	26.006	3.42347	39.10%	64	39.263	2.29276	5.20%
32	26.166	3.40301	27.40%	65	39.814	2.26231	5.90%

33	26.468	3.36485	47.20%				
----	--------	---------	--------	--	--	--	--

The DSC pattern of crystalline form B of the compound of formula III is shown in FIG. 10. The DSC curve shows an endothermic peak at a peak temperature of 186.88°C (enthalpy value 164.82 J/g), with an onset point (Onset x) of 172.09°C. The TGA pattern is shown in FIG. 11. The sample lost 1.42% of its weight when heated from 24.07°C to 120.0°C, and 9.84% from 120.0°C to 205.0°C. Based on the DSC and TGA analysis, crystalline form B of the compound of formula III is an anhydrate.

Example 4 Crystalline Form C of the Compound of Formula III

30 mg of crystalline form A of the compound of formula II was added to 0.5 mL of acetonitrile, and the temperature was raised to 50°C. 0.096 mL of a dilute hydrochloric acid solution was added to the system (preparation method for dilute hydrochloric acid: 0.1 mL of concentrated hydrochloric acid dissolved in 0.9 mL of ethyl acetate). The entire system was stirred at 50°C for 2 hours, cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to obtain crystalline form C of the compound of formula III. The molar ratio of the compound of formula I to hydrochloric acid was 1:1.7. The XRPD pattern of crystalline form C of the compound of formula III is shown in FIG. 12, and it has the diffraction peaks shown in Table 6 below.

Table 6

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	9.84	8.9813	22.30%	21	26.935	3.30754	17.80%
2	10.511	8.4094	14.40%	22	27.482	3.24292	51.00%
3	11.606	7.61836	12.00%	23	28.37	3.1434	25.40%
4	12.187	7.25646	61.90%	24	28.965	3.08014	25.90%
5	13.393	6.60555	33.30%	25	29.322	3.04348	26.90%
6	14.401	6.14577	24.90%	26	29.949	2.98119	6.50%
7	17.352	5.10637	10.30%	27	30.421	2.93597	15.70%
8	18.158	4.8815	86.70%	28	31.245	2.86039	11.50%
9	18.293	4.84598	78.90%	29	31.602	2.82887	5.70%
10	19.386	4.57505	53.40%	30	32.238	2.7745	11.00%
11	20.016	4.43254	15.90%	31	32.62	2.74286	9.60%
12	20.805	4.2661	37.40%	32	33.448	2.67686	3.30%
13	21.747	4.08336	64.40%	33	34.041	2.6316	6.10%
14	22.514	3.94607	33.80%	34	34.684	2.58422	7.70%
15	22.896	3.88101	13.30%	35	35.296	2.54081	10.10%

16	23.245	3.82352	34.10%	36	36.157	2.48228	13.90%
17	23.488	3.78457	29.40%	37	36.574	2.45491	4.00%
18	24.462	3.63601	12.20%	38	37.903	2.37183	6.30%
19	25.113	3.54325	16.90%	39	38.471	2.33815	7.40%
20	26.409	3.37219	100.00%				

Example 5 Crystalline Form A of the Compound of Formula IV

30 mg of crystalline form A of the compound of formula II was added to 0.5 mL of ethyl acetate, and the temperature was raised to 50°C. 0.033 mL of a dilute sulfuric acid solution was added to the system (preparation method for dilute sulfuric acid: 0.1 mL of sulfuric acid dissolved in 0.9 mL of ethyl acetate). The entire system was stirred at 50°C for 2 hours, cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to obtain crystalline form A of the compound of formula IV. The molar ratio of the compound of formula I to sulfuric acid was 1:0.97. The XRPD pattern of crystalline form A of the compound of formula IV is shown in FIG. 13, and it has the diffraction peaks shown in Table 7 below.

Table 7

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.143	17.16934	60.20%	12	20.403	4.34933	33.50%
2	6.108	14.45907	16.10%	13	21.449	4.13949	33.20%
3	6.598	13.38491	14.80%	14	21.781	4.07705	58.50%
4	7.345	12.02656	20.40%	15	23.807	3.73458	40.20%
5	8.628	10.24052	23.70%	16	24.819	3.58454	67.70%
6	9.008	9.80933	100.00%	17	25.473	3.49394	51.50%
7	11.45	7.72193	39.40%	18	25.946	3.4313	49.20%
8	14.624	6.05231	18.30%	19	26.679	3.33862	54.90%
9	15.033	5.8885	35.20%	20	27.959	3.18865	6.00%
10	16.445	5.38619	17.10%	21	28.427	3.13718	29.90%
11	17.437	5.08168	33.50%				

Example 6 Crystalline Form B of the Compound of Formula IV

30 mg of the compound of formula I was added to 0.5 mL of acetonitrile, and the temperature was raised to 50°C. 0.033 mL of a dilute sulfuric acid solution was added to the system (preparation method for dilute sulfuric acid: 0.1 mL of sulfuric acid dissolved in 0.9 mL of ethyl acetate). The entire system was stirred at 50°C for 2 hours, cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to obtain

crystalline form B of the compound of formula IV. The molar ratio of the compound of formula I to sulfuric acid was 1:0.96. The XRPD pattern of crystalline form B of the compound of formula IV is shown in FIG. 14, and it has the diffraction peaks shown in Table 8 below.

Table 8

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	6.868	12.86005	9.90%	35	21.518	4.12631	8.90%
2	7.106	12.42925	22.80%	36	21.975	4.04158	11.20%
3	7.566	11.67521	24.00%	37	22.201	4.00088	20.90%
4	7.716	11.44879	24.70%	38	22.509	3.94688	14.50%
5	8.1	10.90706	26.40%	39	22.856	3.88771	8.00%
6	9.963	8.87076	5.10%	40	23.179	3.83422	67.20%
7	10.269	8.60717	16.20%	41	23.617	3.76419	11.60%
8	11.2	7.89415	2.00%	42	24.18	3.67773	94.90%
9	11.554	7.65303	2.30%	43	24.741	3.59564	12.60%
10	11.859	7.45666	2.80%	44	25.171	3.53519	100.00%
11	12.546	7.04959	13.30%	45	25.575	3.48024	31.60%
12	12.674	6.97885	14.40%	46	25.996	3.42481	14.30%
13	13.192	6.70605	12.00%	47	26.391	3.37441	4.40%
14	13.486	6.56063	8.90%	48	26.802	3.32368	13.60%
15	13.859	6.38461	27.20%	49	27.162	3.28036	18.40%
16	14.114	6.26974	55.00%	50	27.403	3.25211	14.20%
17	14.71	6.01728	63.20%	51	27.814	3.20496	41.70%
18	15.439	5.73483	21.70%	52	28.093	3.1738	14.80%
19	16.074	5.50951	43.00%	53	28.575	3.1213	5.40%
20	16.445	5.38608	5.20%	54	29.494	3.02609	14.30%
21	16.729	5.2952	18.90%	55	29.893	2.98659	16.50%
22	17.005	5.20983	4.60%	56	30.118	2.9648	13.30%
23	17.552	5.04867	4.20%	57	31.454	2.84184	8.00%
24	17.918	4.94655	19.00%	58	32.036	2.79159	14.30%
25	18.378	4.82369	46.30%	59	32.834	2.72552	4.20%
26	18.576	4.77263	44.20%	60	33.662	2.66033	2.50%
27	19.097	4.64357	8.20%	61	34.515	2.59649	4.60%
28	19.463	4.55724	15.30%	62	35.429	2.53161	2.70%
29	19.796	4.4813	12.50%	63	35.851	2.50278	2.10%
30	20.182	4.39642	28.90%	64	36.348	2.46966	3.90%
31	20.394	4.35117	11.90%	65	36.7	2.4468	7.70%
32	20.588	4.31055	15.90%	66	38.306	2.34784	6.40%
33	20.818	4.26344	11.70%	67	38.66	2.32714	3.80%

34	21.206	4.18636	27.10%	68	39.469	2.28128	4.30%
----	--------	---------	--------	----	--------	---------	-------

The DSC pattern of crystalline form B of the compound of formula IV is shown in FIG. 15. The DSC curve shows endothermic peaks at peak temperatures of 34.85°C (enthalpy value 56.75 J/g), 90.79°C (enthalpy value 28.413 J/g), and 204.38°C (enthalpy value 70.95 J/g), with onset points (Onset x) of 11.47°C, 67.49°C, and 196.17°C, respectively. The TGA pattern is shown in FIG. 16. The sample lost 2.06% of its weight when heated from 33.44°C to 50.0°C, 0.56% from 50.0°C to 100.0°C, 0.39% from 100.0°C to 180.0°C, and 1% from 180.0°C to 230.0°C.

Example 7 Crystalline Form A of the Compound of Formula V

30 mg of the compound of formula I was added to 0.5 mL of ethyl acetate, and the temperature was raised to 50°C. 0.041 mL of a dilute phosphoric acid solution was added to the system (preparation method for dilute phosphoric acid: 0.1 mL of phosphoric acid dissolved in 0.9 mL of ethyl acetate). The entire system was stirred at 50°C for 2 hours, cooled to 25°C with stirring, filtered, and the resulting wet product was vacuum dried at 50°C for 2 hours to obtain crystalline form A of the compound of formula V. The molar ratio of the compound of formula I to phosphoric acid was 1:1.14, and crystalline form A of the compound of formula V is a hydrate. The XRPD pattern of crystalline form A of the phosphate salt of the compound of formula I is shown in FIG. 17, and it has the diffraction peaks shown in Table 9 below.

Table 9

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.153	17.13442	24.70%	35	23.241	3.82427	33.70%
2	5.982	14.76142	8.10%	36	23.632	3.76186	23.60%
3	7.969	11.08548	16.00%	37	23.98	3.70798	58.60%
4	8.343	10.58926	44.50%	38	24.1	3.68976	43.00%
5	8.96	9.86201	21.40%	39	24.465	3.6355	7.40%
6	10.489	8.42719	33.70%	40	24.875	3.57651	14.30%
7	10.799	8.18598	26.70%	41	25.132	3.5405	39.10%
8	11.337	7.79853	32.40%	42	25.542	3.48466	5.60%
9	11.657	7.58527	24.10%	43	25.831	3.44632	6.90%
10	11.989	7.37587	21.90%	44	26.177	3.40159	13.30%
11	12.726	6.95054	15.30%	45	26.438	3.3685	10.80%
12	13.146	6.72941	7.00%	46	26.83	3.3202	11.90%
13	13.457	6.57471	-0.10%	47	27.281	3.26636	20.30%
14	14.069	6.28966	2.80%	48	27.734	3.21404	100.00%

15	14.607	6.05938	1.40%	49	28.441	3.13574	9.60%
16	14.842	5.96398	5.00%	50	28.63	3.11548	11.10%
17	15.505	5.71038	38.40%	51	28.936	3.08321	22.30%
18	15.884	5.57482	44.10%	52	29.165	3.0595	18.40%
19	16.687	5.3085	6.50%	53	29.592	3.0163	5.80%
20	17.26	5.13366	4.00%	54	30.38	2.93983	7.70%
21	17.929	4.94353	75.20%	55	31.268	2.85833	5.70%
22	18.387	4.82144	84.80%	56	31.763	2.81491	8.30%
23	18.679	4.7465	37.50%	57	32.113	2.78507	11.50%
24	19.062	4.65205	11.40%	58	32.719	2.73479	5.20%
25	19.731	4.49575	38.50%	59	32.966	2.71493	4.70%
26	20.14	4.4055	31.90%	60	33.517	2.67154	8.90%
27	20.516	4.32562	12.40%	61	33.934	2.63964	6.50%
28	20.76	4.27521	14.20%	62	34.599	2.5904	3.10%
29	21.054	4.2162	29.80%	63	35.435	2.53122	7.30%
30	21.278	4.17241	26.00%	64	36.132	2.48393	2.10%
31	21.643	4.10273	22.90%	65	37.141	2.41876	4.10%
32	21.992	4.03848	88.50%	66	37.699	2.38421	6.90%
33	22.507	3.94723	13.30%	67	38.63	2.32888	6.10%
34	22.762	3.90364	42.90%	68	39.602	2.2739	2.50%

The DSC pattern of crystalline form A of the compound of formula V is shown in FIG. 18. The DSC curve shows two endothermic peaks at peak temperatures of 88.96°C (enthalpy value 42.827 J/g) and 192.67°C (enthalpy value 52.713 J/g), with onset points (Onset x) of 66.74°C and 183.41°C, respectively. The TGA pattern is shown in FIG. 19. The sample lost 0.92% of its weight when heated from 32.34°C to 70.0°C, and 2.10% from 70.0°C to 180.0°C. Based on the DSC and TGA analyses, crystalline form A of the phosphate salt of the compound of formula I (crystalline form A of the compound of formula V) is a hydrate, and the water content calculated from TGA is 0.91 equivalents.

Example 8 Crystalline Form B of the Compound of Formula V

30 mg of the compound of formula I was added to 0.5 mL of acetonitrile, and the temperature was raised to 50°C. 0.041 mL of a dilute phosphoric acid solution was added to the system (preparation method for dilute phosphoric acid: 0.1 mL of phosphoric acid dissolved in 0.9 mL of ethyl acetate). The entire system was stirred at 50°C for 2 hours, cooled to 25°C with stirring, filtered, and the resulting wet product was vacuum dried at 50°C for 2 hours to obtain crystalline form B of the phosphate salt of the compound of formula I (crystalline form B of the compound of formula V). The molar ratio of the compound of formula I to phosphoric acid

was 1:1.09. The XRPD pattern of crystalline form B of the phosphate salt of the compound of formula I (crystalline form B of the compound of formula V) is shown in FIG. 20, and it has the diffraction peaks shown in Table 10 below.

Table 10

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	3.412	25.87783	5.60%	25	21.646	4.10215	22.50%
2	6.833	12.92626	38.40%	26	22.054	4.02735	20.10%
3	7.273	12.14545	6.10%	27	22.311	3.98146	15.20%
4	8.32	10.61883	22.40%	28	22.826	3.89276	13.30%
5	8.523	10.36627	17.60%	29	23.005	3.86291	16.40%
6	9.17	9.6366	12.30%	30	23.559	3.77327	24.00%
7	10.256	8.61794	21.70%	31	23.983	3.70754	25.70%
8	11.718	7.5458	8.60%	32	24.675	3.60507	19.70%
9	12.885	6.86488	100.00%	33	25.081	3.54768	49.20%
10	13.462	6.57226	16.40%	34	25.496	3.49088	59.20%
11	13.761	6.43017	7.80%	35	25.917	3.43502	9.70%
12	13.928	6.35326	20.10%	36	26.578	3.35112	2.90%
13	14.438	6.1301	51.20%	37	27.083	3.28974	9.50%
14	14.668	6.03436	21.10%	38	27.63	3.22589	8.30%
15	15.905	5.56779	47.80%	39	28.154	3.16703	24.60%
16	16.247	5.45135	23.60%	40	29.058	3.07055	17.10%
17	16.609	5.33328	10.10%	41	31.515	2.83649	8.10%
18	17.103	5.18037	7.60%	42	31.958	2.79817	-3.30%
19	17.943	4.93961	14.80%	43	32.727	2.7342	11.80%
20	19.204	4.61801	3.90%	44	34.248	2.61615	9.60%
21	20	4.436	8.00%	45	35.914	2.4985	7.00%
22	20.352	4.36009	50.60%	46	37.149	2.41827	2.40%
23	20.488	4.33131	48.30%	47	38.731	2.32303	12.90%
24	21.145	4.1982	19.40%				

Example 9 Crystalline Form C of the Compound of Formula V

800.6 mg of the compound of formula I was added to 12 mL of acetonitrile, and the temperature was raised to 50°C. 1.09 mL of a dilute phosphoric acid solution was added to the system (preparation method for dilute phosphoric acid: 0.1 mL of phosphoric acid dissolved in 0.9 mL of ethyl acetate). 3 mg of seeds of crystalline form B of the compound of formula V were added. The entire system was stirred at 50°C for 2 hours, cooled to 25°C with stirring, filtered, and the resulting wet product was vacuum dried at 50°C for 2 hours to obtain

crystalline form C of the compound of formula V. The molar ratio of the compound of formula I to phosphoric acid was 1:1.05. Crystalline form C of the compound of formula V is an anhydrate, and NMR shows that crystalline form C of the phosphate salt of the compound of formula I (crystalline form C of the compound of formula V) contains 0.1 equivalent of acetonitrile. The XRPD pattern of crystalline form C of the compound of formula I is shown in FIG. 21, and it has the diffraction peaks shown in Table 11 below.

Table 11

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	5.194	17.00128	49.10%	41	24.561	3.62162	2.70%
2	7.95	11.11257	4.30%	42	24.804	3.58669	30.20%
3	8.388	10.53338	51.00%	43	25.27	3.52153	28.10%
4	9.027	9.78834	9.70%	44	25.665	3.46825	60.90%
5	9.443	9.35808	0.90%	45	26.002	3.42398	4.60%
6	10.215	8.65246	8.60%	46	26.193	3.39955	18.70%
7	10.468	8.44417	48.10%	47	26.423	3.37046	11.90%
8	11.192	7.89926	16.20%	48	26.94	3.30687	12.40%
9	11.506	7.68472	100.00%	49	27.185	3.27765	13.70%
10	11.672	7.57563	23.90%	50	27.467	3.24469	39.90%
11	12.726	6.95069	21.10%	51	28.108	3.17204	7.00%
12	12.885	6.86505	17.90%	52	28.431	3.13677	2.50%
13	13.137	6.73409	5.10%	53	28.725	3.10532	8.90%
14	13.623	6.49459	3.90%	54	29.083	3.06794	13.50%
15	14.586	6.06817	12.80%	55	29.412	3.03439	33.20%
16	15.052	5.88112	13.40%	56	30.123	2.96435	10.90%
17	15.682	5.64618	33.50%	57	30.326	2.94497	9.90%
18	15.865	5.58146	26.00%	58	30.528	2.92594	3.90%
19	16.158	5.48123	5.20%	59	31.259	2.85912	2.00%
20	16.787	5.27694	9.60%	60	31.674	2.82261	9.10%
21	16.935	5.23145	7.20%	61	32.155	2.78146	2.40%
22	17.236	5.14075	3.60%	62	32.544	2.74915	3.70%
23	17.57	5.04367	3.80%	63	32.701	2.73627	2.80%
24	17.898	4.95191	25.20%	64	33.023	2.71038	3.50%
25	18.012	4.92095	56.50%	65	33.438	2.67767	7.20%
26	18.393	4.81986	10.90%	66	33.929	2.64	6.00%
27	18.756	4.72738	34.70%	67	34.274	2.61419	3.70%
28	19.092	4.64491	7.00%	68	34.754	2.57922	3.40%
29	19.809	4.47827	32.70%	69	34.975	2.56342	9.70%
30	20.481	4.33291	32.90%	70	35.465	2.52912	8.40%
31	20.843	4.25843	28.10%	71	35.731	2.51089	3.10%
32	21.676	4.09663	29.00%	72	36.389	2.46699	4.20%
33	22.103	4.01842	32.30%	73	36.885	2.43497	1.20%
34	22.551	3.93962	79.40%	74	37.388	2.40336	6.80%
35	23.098	3.84751	34.60%	75	37.963	2.36822	4.20%

36	23.226	3.82662	25.20%	76	38.424	2.34089	3.70%
37	23.486	3.78492	9.60%	77	38.711	2.32418	4.50%
38	23.704	3.75051	12.70%	78	39.164	2.29832	6.40%
39	23.943	3.71361	27.10%	79	39.661	2.27068	3.30%
40	24.147	3.68273	30.10%				

The DSC curve of the crystalline form C of the compound of formula V is shown in FIG. 22. The DSC curve shows endothermic peaks at peak temperatures of 188.32°C (enthalpy value 41.702 J/g) and 221.66°C (enthalpy value 32.668 J/g), with onset points (Onset x) at 175.87°C and 32.668°C, respectively. The TGA curve is shown in FIG. 23. The sample shows a weight loss of 0.71% when heated from 29.55°C to 180.0°C, and a weight loss of 0.85% when heated from 180.0°C to 220.0°C.

Example 10 Crystalline Form A of the Compound of Formula VI

30 mg of the compound of formula I and 7.04 mg of fumaric acid were added to 0.5 mL of acetonitrile, and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the resulting wet product was vacuum dried at 50°C for 2 hours to obtain crystalline form A of the compound of formula VI. The molar ratio of the compound of formula I to fumaric acid was 1:1.01. The XRPD pattern of crystalline form A of the fumarate salt of the compound of formula I (crystalline form A of the compound of formula VI) is shown in FIG. 24, and it has diffraction peaks as shown in Table 12 below.

NMR: ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.65 (s, 1H), 13.11 (s, 2H), 9.16 (d, *J* = 2.0 Hz, 1H), 8.92 (s, 1H), 8.19 (s, 1H), 7.63 (s, 1H), 7.53 (s, 1H), 6.63 (s, 2H), 4.03 (s, 3H), 3.61 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 12

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.527	15.97563	16.80%	24	19.864	4.466	7.80%
2	6.733	13.11834	20.90%	25	20.599	4.30833	15.60%
3	6.959	12.69269	47.50%	26	21.267	4.1745	14.80%
4	7.737	11.41816	9.70%	27	21.759	4.08112	11.20%
5	8.599	10.2745	42.10%	28	22.345	3.97554	13.80%
6	9.379	9.42209	14.10%	29	22.552	3.93942	32.30%
7	9.91	8.91803	9.40%	30	22.758	3.90426	21.80%
8	10.278	8.59978	21.00%	31	23.378	3.80204	39.60%
9	10.577	8.35741	90.90%	32	23.946	3.71313	11.20%
10	12.156	7.2748	53.50%	33	24.521	3.6274	9.20%

11	13.45	6.57797	21.50%	34	25.146	3.53865	25.40%
12	13.735	6.44195	12.60%	35	25.709	3.4624	40.00%
13	13.982	6.3287	100.00%	36	27.37	3.2559	13.20%
14	14.844	5.96313	42.10%	37	27.682	3.21995	12.40%
15	15.391	5.75233	43.10%	38	28.842	3.09299	16.60%
16	15.733	5.62829	7.20%	39	29.908	2.98517	23.30%
17	16.631	5.32636	35.60%	40	31.774	2.81395	11.50%
18	17.077	5.18824	52.80%	41	32.663	2.73942	10.20%
19	17.37	5.10132	5.40%	42	34.767	2.57831	16.80%
20	18.669	4.74913	21.60%	43	35.226	2.54572	6.90%
21	18.97	4.67444	20.90%	44	37.118	2.42017	3.80%
22	19.16	4.62861	26.60%	45	38.115	2.35915	8.40%
23	19.276	4.60083	34.30%				

Example 11 Crystalline Form A of the Compound of Formula VII

30 mg of the compound of formula I was added to 0.5 mL of acetonitrile, and the temperature was raised to 50°C. 0.039 mL of a methanesulfonic acid solution (0.1 mL of methanesulfonic acid dissolved in 0.9 mL of ethyl acetate) was added. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the resulting wet product was vacuum dried at 50°C for 2 hours to obtain crystalline form A of the compound of formula VII. The molar ratio of the compound of formula I to methanesulfonic acid was 1:1.03. The XRPD pattern of crystalline form A of the compound of formula VII is shown in FIG. 25, and it has diffraction peaks as shown in Table 13 below.

NMR:¹H NMR (400 MHz, DMSO-*d*₆) δ 13.64 (s, 1H), 9.15 (d, *J* = 2.0 Hz, 1H), 8.92 (s, 1H), 8.19 (s, 1H), 7.63 (s, 1H), 7.54 (s, 1H), 4.03 (s, 3H), 3.62 (s, 3H), 2.63 (s, 3H), 2.29 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 13

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	5.132	17.20602	16.30%	16	21.155	4.19632	9.00%
2	6.727	13.12872	13.80%	17	21.744	4.08391	49.70%
3	8.614	10.2569	32.10%	18	22.365	3.97189	12.10%
4	8.997	9.82161	100.00%	19	23.183	3.8336	6.10%
5	10.049	8.79483	9.80%	20	23.707	3.75013	7.80%
6	10.341	8.5475	6.50%	21	24.426	3.64125	18.70%
7	11.167	7.91683	4.40%	22	25.322	3.51441	31.50%
8	11.566	7.64514	9.50%	23	26.356	3.37883	12.50%

9	13.927	6.35347	5.60%	24	27.293	3.26497	7.90%
10	15.062	5.87733	7.50%	25	27.926	3.19232	11.90%
11	15.551	5.69372	13.90%	26	28.326	3.14817	13.10%
12	17.441	5.08075	58.10%	27	29.137	3.0624	10.00%
13	17.739	4.99592	18.10%	28	29.856	2.99021	8.70%
14	18.62	4.76144	8.90%	29	31.067	2.87641	1.70%
15	20.076	4.41946	3.30%				

Example 12 Crystalline Form A of the Compound of Formula VIII

30 mg of the compound of formula I and 9.68 mg of benzenesulfonic acid were added to 0.5 mL of ethyl acetate, and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the resulting wet product was vacuum dried at 50°C for 2 hours to obtain crystalline form A of the compound of formula VIII. The molar ratio of the compound of formula I to benzenesulfonic acid was 1:1.00, and NMR showed the molar ratio of the compound of formula I to ethyl acetate was 1:0.07. The XRPD pattern of crystalline form A of the compound of formula VIII is shown in FIG. 26, and it has diffraction peaks as shown in Table 14 below.

NMR: ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.64 (s, 1H), 9.15 (d, *J* = 2.0 Hz, 1H), 8.92 (s, 1H), 8.19 (s, 1H), 7.63 (s, 1H), 7.60-7.58 (m, 2H), 7.54 (s, 1H), 7.34-7.29 (m, 3H), 4.03 (s, 3H), 3.61 (s, 3H), 2.63 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 14

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	6.475	13.6404	14.30%	22	20.349	4.3607	19.20%
2	7.661	11.53042	100.00%	23	20.89	4.24904	20.10%
3	9.059	9.75377	21.40%	24	21.099	4.20735	9.60%
4	9.719	9.09297	20.80%	25	21.675	4.09672	31.10%
5	10.087	8.76259	45.10%	26	22.266	3.98938	24.30%
6	11.104	7.96192	18.60%	27	22.494	3.94942	19.60%
7	11.921	7.41792	44.30%	28	22.968	3.86906	28.30%
8	12.359	7.15589	10.80%	29	23.497	3.78306	22.00%
9	12.984	6.81282	9.10%	30	24.844	3.58097	32.30%
10	13.906	6.36334	79.00%	31	25.515	3.48834	42.40%
11	14.416	6.13904	10.20%	32	25.974	3.42768	18.20%
12	15.142	5.84662	6.30%	33	26.695	3.33665	10.20%
13	15.912	5.56516	19.50%	34	27.335	3.25998	8.50%
14	16.551	5.35171	20.20%	35	28.189	3.16317	8.50%

15	17.038	5.19977	9.40%	36	29.356	3.04006	20.20%
16	17.601	5.03481	12.70%	37	30.275	2.94982	10.90%
17	17.995	4.92535	18.50%	38	30.805	2.90025	10.10%
18	18.269	4.85222	41.70%	39	31.765	2.81476	5.80%
19	18.507	4.79024	58.50%	40	32.965	2.715	9.50%
20	19.159	4.62871	50.10%	41	34.371	2.6071	11.60%
21	19.716	4.49913	11.20%				

Example 13 Crystalline Form A of the Compound of Formula IX

30 mg of the compound of Formula I and 10.44 mg of p-toluenesulfonic acid were added to 0.5 mL of acetone/water (95 : 5, v : v), and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, cooled to 25°C with stirring, filtered, and the resulting wet product was vacuum dried at 50°C for 2 hours to obtain Crystalline Form A of the compound of Formula IX. The XRPD pattern of Crystalline Form A of the compound of Formula IX is shown in FIG. 27, and it has diffraction peaks as shown in Table 15 below.

NMR: ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.64 (s, 1H), 9.15 (d, *J* = 2.0 Hz, 1H), 8.91 (s, 1H), 8.18 (s, 1H), 7.63 (s, 1H), 7.53 (s, 1H), 7.47 (d, *J* = 8.4 Hz, 2H), 7.11 (d, *J* = 7.6 Hz, 2H), 4.03 (s, 3H), 3.61 (s, 3H), 2.63 (s, 3H), 2.29 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 15

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.962	14.81202	57.90%	39	25.978	3.42709	27.10%
2	8.204	10.76821	4.10%	40	26.258	3.39119	6.70%
3	8.371	10.55406	5.70%	41	26.662	3.3408	6.70%
4	9.927	8.90302	10.20%	42	27.349	3.25839	21.60%
5	10.133	8.72234	8.40%	43	27.516	3.23902	30.80%
6	10.573	8.36071	36.50%	44	27.793	3.20733	7.70%
7	11.032	8.01334	14.20%	45	28.075	3.1757	31.60%
8	11.882	7.44222	14.90%	46	28.299	3.15109	10.80%
9	12.339	7.16786	33.30%	47	28.965	3.08021	15.00%
10	12.767	6.9283	24.90%	48	29.166	3.05937	39.40%
11	13.419	6.59304	5.20%	49	29.35	3.04064	12.90%
12	14.06	6.29386	32.00%	50	29.915	2.9845	7.40%
13	14.396	6.14788	28.30%	51	30.364	2.94138	40.50%
14	14.974	5.91163	29.20%	52	31.21	2.86355	6.60%
15	15.682	5.64651	2.80%	53	31.645	2.82513	5.50%
16	15.845	5.58857	1.80%	54	31.834	2.80877	16.00%
17	16.714	5.30007	100.00%	55	31.958	2.7982	37.80%

18	17.555	5.04799	29.70%	56	32.619	2.74298	2.80%
19	17.785	4.98319	73.50%	57	32.954	2.71585	11.80%
20	18.413	4.81446	31.00%	58	33.386	2.68169	6.40%
21	18.696	4.74244	15.60%	59	33.718	2.65606	3.40%
22	19.188	4.62193	5.90%	60	34.173	2.62168	10.50%
23	19.668	4.51003	13.00%	61	34.59	2.59107	8.40%
24	19.877	4.4632	43.60%	62	35.191	2.54821	3.00%
25	20.33	4.36466	57.50%	63	35.191	2.54821	3.00%
26	20.684	4.29085	15.20%	64	35.477	2.52831	3.80%
27	21.198	4.18782	29.10%	65	35.913	2.49857	5.40%
28	21.486	4.13245	2.70%	66	36.124	2.48447	4.50%
29	22.112	4.01691	32.50%	67	36.345	2.4699	9.70%
30	22.637	3.92478	45.20%	68	36.981	2.42881	2.20%
31	23.176	3.83469	32.40%	69	37.406	2.40222	6.20%
32	23.856	3.72701	2.20%	70	37.623	2.38887	10.10%
33	24.224	3.67124	46.00%	71	37.83	2.37624	4.30%
34	24.66	3.6073	72.50%	72	38.317	2.34719	3.70%
35	25.148	3.53832	6.60%	73	38.881	2.31439	7.90%
36	25.427	3.5001	32.70%	74	39.015	2.30674	5.20%
37	25.651	3.47007	8.80%	75	39.544	2.27711	5.00%
38	25.836	3.4456	13.40%				

The DSC pattern of Crystalline Form A of the compound of Formula IX is shown in FIG. 28. The DSC curve shows two endothermic peaks at peak temperatures of 95.83°C (enthalpy value 244.34 J/g) and 194.7°C (enthalpy value 34.862 J/g), with onset points (Onset x) at 57.37°C and 181.84°C, respectively. The TGA pattern is shown in FIG. 29. The sample showed a weight loss of 2.03% when heated from 30.52°C to 60.0°C, a weight loss of 5.13% when heated from 60.0°C to 100.0°C, and a weight loss of 0.50% when heated from 100.0°C to 190.0°C. NMR data indicate that in Crystalline Form A of the compound of Formula IX, the molar ratio of water to the compound of Formula I to p-toluenesulfonic acid is 3:1:1.

Example 13-1 Single Crystal of the Compound of Formula IX

The compound of Formula I (1.63 kg, 3.11 mol) was added to a mixed solution of 10.3 kg of acetone and 1.30 kg of water, the temperature was controlled at 45-55°C, and 80 g of Crystalline Form A of the compound of Formula IX was added to obtain a mixture. 0.58 kg of p-toluenesulfonic acid monohydrate was dissolved in 2.57 kg of acetone and 0.32 kg of water, and the resulting solution was added dropwise to the above mixture. The mixture was stirred at a controlled temperature of 45-55°C for 2 hours. The temperature was lowered to 20-30°C, and

the mixture was stirred for 2 hours. 0.29 kg of p-toluenesulfonic acid monohydrate was dissolved in 1.28 kg of acetone and 0.16 kg of water, and the resulting solution was added dropwise to the above mixture. The mixture was stirred at a controlled temperature of 20-30°C for 2 hours. The mixture was filtered to obtain a single crystal of the compound of Formula IX, wherein the molar ratio of water to the compound of Formula I to p-toluenesulfonic acid is 3:1:1.

The molecular structure of the single crystal of the compound of Formula IX is shown in FIG.

57, and the unit cell parameters are shown in the table below:

Unit cell dimensions/mm ³	0.200 × 0.180 × 0.150 mm ³
Wavelength	1.54178 Å
Crystal system	Triclinic
Space group	P-1
a/Å	10.6990(12) Å
b/Å	10.7640(13) Å
c/Å	14.9358(17) Å
α/°	89.299(7)°
β/°	86.519(6)°
γ/°	84.364(6)°
Unit cell volume/Å ³	1708.6(3) Å ³
Z	2
Unit cell density mg/m ³	1.460 mg/m ³
F (000)	780
Absorption coefficient mm ⁻¹	2.717 mm ⁻¹
Diffraction index range (h/k/l)	-12≤h≤12, -12≤k≤12, -17≤l≤17
Temperature/K	173(2) K
Theta range for data collection/°	2.964 to 66.596°
Data/restraints/parameters	5746 / 1 / 477
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7531 and 0.6736
Refinement method	Full-matrix least-squares on F ²
Goodness-of-fit on F ²	0.894
Final R indices [I>=2σ(I)]	R1 = 0.0359, wR2 = 0.1027
R factor (Final R indices) [all data]	R1 = 0.0435, wR2 = 0.1109
Largest diff. peak and hole / e.Å ⁻³	0.300 and -0.432 e.Å ⁻³
Reflections collected	21870
Independent reflections	5746 [R(int) = 0.0379]
Absolute structure parameter	N/A

Example 14 Crystalline Form B of the Compound of Formula IX

30 mg of the compound of Formula I and 10.44 mg of p-toluenesulfonic acid were added to 0.5 mL of acetonitrile, and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to obtain Crystalline Form B of the compound of Formula IX. The molar ratio of the compound of Formula I to p-toluenesulfonic acid is 1:1.00. The XRPD pattern of Crystalline Form B of the p-toluenesulfonate salt of the compound of Formula I (Crystalline Form B of the compound of Formula IX) is shown in FIG. 30, and it has diffraction peaks as shown in Table 16 below.

NMR: ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.65 (s, 1H), 9.15 (d, *J* = 2.0 Hz, 1H), 8.92 (s, 1H), 8.19 (s, 1H), 7.63 (s, 1H), 7.54 (s, 1H), 7.47 (d, *J* = 8.4 Hz, 2H), 7.11 (d, *J* = 7.6 Hz, 2H), 4.03 (s, 3H), 3.61 (s, 3H), 2.63 (s, 3H), 2.29 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 16

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.339	16.53947	100.00%	21	19.465	4.55658	31.00%
2	8.195	10.78059	23.30%	22	19.846	4.46999	52.90%
3	9.083	9.72794	13.30%	23	21.533	4.12355	18.40%
4	9.335	9.46653	63.50%	24	22.137	4.0123	7.00%
5	9.921	8.90842	50.50%	25	22.875	3.88447	22.70%
6	10.125	8.72971	32.20%	26	24.412	3.6434	31.40%
7	11.117	7.95254	16.10%	27	25.425	3.50047	23.50%
8	11.469	7.70919	17.90%	28	25.749	3.45708	23.10%
9	12.189	7.25545	13.10%	29	26.479	3.36337	23.50%
10	12.839	6.88971	54.40%	30	26.833	3.31984	12.40%
11	13.182	6.71091	34.90%	31	27.344	3.25898	8.80%
12	14.888	5.94554	8.90%	32	27.945	3.19024	19.80%
13	15.262	5.80086	13.80%	33	28.263	3.15503	9.20%
14	16.132	5.48968	63.90%	34	28.665	3.11168	10.00%
15	16.362	5.41311	46.10%	35	29.119	3.06425	9.00%
16	16.606	5.3341	73.50%	36	29.963	2.97978	22.10%
17	16.891	5.24471	13.20%	37	30.835	2.89751	8.80%
18	17.307	5.11958	9.50%	38	32.466	2.75553	14.10%
19	18.597	4.76732	37.50%	39	35.254	2.54373	5.30%
20	18.67	4.74878	60.70%				

Example 15 Crystalline Form A of the compound of Formula X

30 mg of the compound of Formula I and 22.06 mg of 1,5-naphthalenedisulfonic acid were added to 0.5 mL of acetone/water (95:5, v:v), and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to obtain Crystalline Form A of the compound of Formula X. The molar ratio of the compound of Formula I to 1,5-naphthalenedisulfonic acid is 1:1.02. The XRPD pattern of Crystalline Form A of the compound of Formula X is shown in FIG. 31, and it has diffraction peaks as shown in Table 17 below.

NMR: ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.64 (s, 1H), 9.15 (d, *J* = 2.0 Hz, 1H), 8.91 (s, 1H), 8.85 (d, *J* = 8.4 Hz, 2H), 8.18 (s, 1H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.63 (s, 1H), 7.53 (s, 1H), 7.40 (dd, *J* = 8.4, 7.6 Hz, 2H), 4.03 (s, 3H), 3.61 (s, 3H), 2.63 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 17

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.713	15.4567	7.20%	25	23.845	3.7287	3.90%
2	7.456	11.84734	81.90%	26	24.23	3.67024	3.00%
3	8.673	10.18701	2.90%	27	24.571	3.62016	20.40%
4	11.43	7.73558	25.50%	28	24.994	3.55975	9.80%
5	12.012	7.36179	100.00%	29	25.441	3.49833	3.30%
6	12.345	7.16417	2.10%	30	26.083	3.41361	8.90%
7	13.532	6.53813	27.00%	31	26.593	3.34923	10.50%
8	13.703	6.45705	9.00%	32	26.898	3.31198	6.70%
9	13.928	6.35325	7.70%	33	28.049	3.17861	5.80%
10	14.751	6.00068	5.80%	34	28.435	3.13633	20.50%
11	14.914	5.93552	13.20%	35	28.687	3.10937	8.10%
12	15.324	5.77744	9.10%	36	28.976	3.07906	6.30%
13	17.235	5.14087	28.30%	37	29.329	3.04281	4.20%
14	17.708	5.00472	1.60%	38	30.168	2.95998	3.30%
15	18.485	4.79589	25.50%	39	30.869	2.89436	6.10%
16	18.767	4.72462	21.20%	40	31.214	2.86319	5.40%
17	19.02	4.66231	4.70%	41	32.378	2.76287	2.70%
18	21.077	4.21171	24.30%	42	33.52	2.6713	2.80%
19	21.695	4.093	49.30%	43	34.636	2.58771	1.80%
20	22.12	4.01545	11.90%	44	36.84	2.43779	2.90%
21	22.48	3.95186	2.60%	45	37.805	2.37776	2.60%
22	22.796	3.89782	10.50%	46	38.41	2.34167	1.90%
23	22.948	3.87227	17.80%	47	38.842	2.31667	5.70%

24	23.354	3.80594	2.00%	48	39.543	2.27716	2.50%
----	--------	---------	-------	----	--------	---------	-------

Example 16 Crystalline Form B of the compound of Formula X

30 mg of the compound of Formula I and 22.06 mg of 1,5-naphthalenedisulfonic acid were added to 0.5 mL of ethyl acetate, and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to obtain Crystalline Form B of the compound of Formula X. The molar ratio of the compound of Formula I to 1,5-naphthalenedisulfonic acid is 1:0.93. The XRPD pattern of Crystalline Form B of the compound of Formula X is shown in FIG. 32, and it has diffraction peaks as shown in Table 18 below.

NMR: ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.64 (s, 1H), 9.15 (d, *J* = 2.0 Hz, 1H), 8.91 (s, 1H), 8.86 (d, *J* = 8.4 Hz, 2H), 8.18 (s, 1H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.63 (s, 1H), 7.53 (s, 1H), 7.40 (dd, *J* = 8.4, 7.6 Hz, 2H), 4.03 (s, 3H), 3.61 (s, 3H), 2.63 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 18

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.946	14.85159	20.80%	18	23.793	3.73676	58.00%
2	8.964	9.85725	81.20%	19	24.141	3.6836	23.80%
3	10.807	8.18027	91.00%	20	24.553	3.62279	42.60%
4	11.977	7.38357	46.80%	21	25.565	3.48153	28.00%
5	14.091	6.28001	23.00%	22	26.293	3.38677	12.50%
6	14.696	6.02294	14.40%	23	26.989	3.30096	3.00%
7	15.797	5.6055	48.20%	24	27.256	3.26925	74.60%
8	16.97	5.22056	29.20%	25	27.772	3.2097	70.10%
9	17.916	4.94708	100.00%	26	29.656	3.00995	34.10%
10	18.4	4.81795	64.10%	27	30.281	2.94924	15.00%
11	20.028	4.42978	30.00%	28	31.009	2.88165	15.20%
12	20.734	4.28054	23.10%	29	32.208	2.77701	15.40%
13	21.272	4.17353	45.00%	30	32.528	2.75045	26.80%
14	22.072	4.02397	86.10%	31	36.294	2.47322	25.50%
15	22.422	3.96191	36.20%	32	37.499	2.39649	16.40%
16	22.76	3.90387	32.30%	33	38.636	2.32852	20.10%
17	23.273	3.81897	51.70%	34	39.292	2.29116	20.30%

Example 17 Crystalline Form C of the compound of Formula X

30 mg of the compound of Formula X and 22.06 mg of 1,5-naphthalenedisulfonic acid were added to 0.5 mL of acetonitrile, and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the obtained wet

product was vacuum dried at 50°C for 2 hours to obtain Crystalline Form C of the compound of Formula X. The molar ratio of the compound of Formula I to 1,5-naphthalenedisulfonic acid is 1:1.00. The XRPD pattern of Crystalline Form C of the compound of Formula X is shown in FIG. 33, and it has diffraction peaks as shown in Table 19 below.

NMR: ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.65 (s, 1H), 9.16 (d, *J* = 2.0 Hz, 1H), 8.92 (s, 1H), 8.86 (d, *J* = 8.4 Hz, 2H), 8.19 (s, 1H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.63 (s, 1H), 7.53 (s, 1H), 7.40 (dd, *J* = 8.4, 7.6 Hz, 2H), 4.03 (s, 3H), 3.61 (s, 3H), 2.63 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 19

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	4.648	18.99788	-0.90%	11	18.606	4.76513	39.10%
2	6.359	13.88777	51.00%	12	20.236	4.38474	26.60%
3	9.423	9.37769	16.00%	13	20.944	4.23819	35.50%
4	11.302	7.82283	98.80%	14	22.606	3.93014	100.00%
5	12.105	7.30558	26.70%	15	24.693	3.60257	71.00%
6	12.82	6.89996	55.10%	16	26.159	3.40389	52.30%
7	13.646	6.48392	15.50%	17	28.569	3.12198	38.80%
8	15.115	5.85673	30.30%	18	31.642	2.82537	9.30%
9	17.068	5.1909	40.10%	19	32.751	2.73219	4.40%
10	17.476	5.07047	37.10%				

Example 18 Crystalline Form D of the compound of Formula X

400.23 mg of the compound of Formula I, 294.79 mg of 1,5-naphthalenedisulfonic acid, and 3 mg of Crystalline Form A of the 1,5-naphthalenedisulfonate salt of the compound of Formula I were added to 6.6 mL of acetone/water (95:5, v:v), and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to obtain Crystalline Form D of the compound of Formula X. The molar ratio of the compound of Formula I to 1,5-naphthalenedisulfonic acid is 1:1.02. The XRPD pattern of Crystalline Form D of the compound of Formula X is shown in FIG. 34, and it has diffraction peaks as shown in Table 20 below.

NMR: ¹H NMR (400 MHz, DMSO-*d*₆) δ 13.65 (s, 1H), 9.16 (d, *J* = 2.0 Hz, 1H), 8.92 (s, 1H), 8.86 (d, *J* = 8.4 Hz, 2H), 8.19 (s, 1H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.63 (s, 1H), 7.53 (s, 1H), 7.40 (dd, *J* = 8.4, 7.6 Hz, 2H), 4.03 (s, 3H), 3.61 (s, 3H), 2.63 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 20

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	7.269	12.15102	11.50%	32	24.953	3.56557	30.20%
2	7.872	11.22197	7.90%	33	25.376	3.50703	1.20%
3	9.251	9.55183	4.50%	34	25.665	3.46825	1.60%
4	9.566	9.23802	2.80%	35	26.37	3.37708	2.40%
5	10.138	8.71834	1.70%	36	26.691	3.33723	9.50%
6	10.682	8.27576	100.00%	37	27.319	3.26192	24.90%
7	11.491	7.69425	3.20%	38	28.123	3.17044	6.50%
8	12.029	7.35148	3.10%	39	28.418	3.13815	2.00%
9	13.333	6.63559	1.60%	40	28.881	3.08892	20.30%
10	13.593	6.50901	8.50%	41	29.287	3.047	1.90%
11	14.537	6.08826	6.30%	42	29.544	3.02108	2.90%
12	15.551	5.69372	8.20%	43	29.954	2.98063	7.20%
13	15.77	5.61503	24.60%	44	30.343	2.94335	3.30%
14	16.245	5.45179	1.30%	45	30.626	2.9168	7.00%
15	16.5	5.36836	7.40%	46	31.161	2.86788	5.70%
16	16.875	5.24981	20.10%	47	31.818	2.81021	8.00%
17	17.287	5.12542	4.30%	48	31.993	2.79519	9.20%
18	17.66	5.01827	10.70%	49	32.37	2.76349	12.40%
19	18.586	4.77007	1.60%	50	32.703	2.73616	5.40%
20	19.627	4.51945	7.10%	51	33.037	2.70921	3.30%
21	19.815	4.47691	26.00%	52	33.33	2.68606	2.40%
22	20.266	4.37825	11.50%	53	34.29	2.61305	4.70%
23	21.438	4.1416	73.20%	54	34.759	2.57886	2.60%
24	21.762	4.08063	21.90%	55	35.072	2.55657	1.60%
25	22.623	3.92722	10.80%	56	36.503	2.45954	2.00%
26	23.09	3.84893	9.80%	57	37.339	2.40638	2.30%
27	23.366	3.80395	8.90%	58	37.585	2.39118	2.80%
28	23.618	3.76395	32.00%	59	37.892	2.37252	8.20%
29	24.004	3.70429	6.80%	60	38.562	2.33283	1.20%
30	24.333	3.65496	11.80%	61	38.815	2.3182	4.10%
31	24.642	3.60992	11.70%				

The DSC profile of Crystalline Form D of the compound of Formula X is shown in FIG. 35. The DSC curve shows three endothermic peaks at peak temperatures of 62.55°C (enthalpy value 111.00 J/g), 86.97°C (enthalpy value 39.972 J/g), and 267.15°C (enthalpy value 55.716 J/g), with onset points (Onset x) at 39.64°C, 70.37°C, and 256.54°C, respectively. The TGA profile is shown in FIG. 36. The sample lost 4.49% of its weight when heated from 32.27°C to

60.0°C, lost 2.41% when heated from 60.0°C to 120.0°C, and lost 0.60% when heated from 120.0°C to 240.0°C. NMR data show that Crystalline Form D of the compound of Formula X contains only 0.1% acetone. The KF moisture test result shows a water content of 9.1%, indicating that Crystalline Form D contains water equivalent to 4.8 equivalents.

Example 19 Crystalline Form A of the compound of Formula XI

30 mg of the compound of Formula I and 2.50 mg of sodium hydroxide were added to 0.5 mL of ethyl acetate, and the temperature was raised to 50°C. The entire system was stirred at 50°C for 2 hours, then cooled to 25°C with stirring, filtered, and the obtained wet product was vacuum dried at 50°C for 2 hours to obtain Crystalline Form A of the compound of Formula XI. The molar ratio of the compound of Formula I to sodium is 1:0.62. The XRPD pattern of Crystalline Form A of the compound of Formula XI is shown in FIG. 37, and it has diffraction peaks as shown in Table 21 below.

Table 21

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	4.687	18.83695	1.80%	19	23.031	3.85854	1.40%
2	5.583	15.81629	2.20%	20	23.577	3.77046	45.80%
3	6.9	12.80028	4.30%	21	24.148	3.68254	6.00%
4	9.385	9.41639	5.20%	22	26.476	3.36378	1.90%
5	10.665	8.28879	37.80%	23	27.172	3.27925	1.00%
6	11.146	7.93194	3.10%	24	27.926	3.19239	13.80%
7	12.782	6.91996	95.20%	25	28.848	3.09237	2.40%
8	14.093	6.27919	100.00%	26	30.214	2.95564	5.60%
9	14.975	5.9112	76.20%	27	31.248	2.86014	2.40%
10	15.521	5.70471	2.40%	28	32.47	2.75519	3.00%
11	15.881	5.57597	0.90%	29	33.25	2.69236	4.70%
12	17.222	5.14467	9.30%	30	35.119	2.55326	4.00%
13	18.821	4.71122	27.70%	31	36.156	2.48235	2.70%
14	19.351	4.58332	9.80%	32	36.698	2.44694	5.60%
15	19.489	4.55115	16.80%	33	37.176	2.41653	2.20%
16	20.821	4.26281	1.70%	34	38.159	2.35655	4.50%
17	21.804	4.07295	15.90%	35	39.026	2.30615	3.10%
18	22.448	3.9575	7.40%				

Example 20 Crystalline Form B of the compound of Formula XI

30 mg of the compound of Formula I and 2.50 mg of sodium hydroxide were added to 0.5 mL of acetonitrile, and the temperature was raised to 50°C. The entire system was stirred at 50°C

for 2 hours, cooled to 25°C and stirred, filtered, and the resulting wet product was vacuum dried at 50°C for 2 hours to obtain Crystalline Form B of the compound of Formula XI. The molar ratio of the compound of Formula I to sodium was 1:1.13, and the molar ratio of the compound of Formula I to the solvent acetonitrile was 1:1.07. The XRPD pattern of Crystalline Form B of the compound of Formula XI is shown in FIG. 38, and it has the diffraction peaks shown in Table 22 below.

Table 22

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.541	15.93667	3.50%	23	22.743	3.90677	6.00%
2	6.923	12.7585	5.20%	24	23.048	3.85572	11.60%
3	8.22	10.74816	77.00%	25	23.433	3.79331	82.60%
4	9.311	9.49097	44.20%	26	24.167	3.67965	43.60%
5	9.596	9.20947	13.00%	27	24.736	3.59641	68.40%
6	10.632	8.31437	8.80%	28	25.913	3.43554	2.60%
7	11.563	7.64664	11.30%	29	26.456	3.36626	14.90%
8	12.922	6.84547	46.00%	30	26.863	3.31623	11.40%
9	13.323	6.64052	31.20%	31	27.373	3.25556	27.40%
10	13.622	6.49544	7.90%	32	29.056	3.07075	11.70%
11	13.827	6.39957	15.60%	33	30.037	2.97261	13.70%
12	15.128	5.85168	18.50%	34	30.266	2.95061	20.30%
13	16.288	5.43764	18.70%	35	30.853	2.89585	20.40%
14	17.021	5.20501	8.70%	36	31.404	2.84628	18.40%
15	17.629	5.027	16.60%	37	32.211	2.77675	12.00%
16	17.773	4.98662	23.40%	38	33.098	2.7044	10.30%
17	18.124	4.89075	33.20%	39	34.323	2.61062	15.80%
18	18.632	4.7585	31.00%	40	34.876	2.57048	11.10%
19	19.378	4.57704	90.40%	41	36.294	2.47322	2.20%
20	20.377	4.35482	100.00%	42	37.623	2.38884	5.40%
21	21.035	4.22005	32.30%	43	38.731	2.32303	9.20%
22	21.278	4.17238	30.90%	44	39.873	2.25907	5.30%

Example 21 Crystalline Form C of the compound of Formula IX

199.4 mg of Crystalline Form A of the p-toluenesulfonate salt of the compound of Formula I (Crystalline Form A of the compound of Formula IX) was added to 5.2 mL of tetrahydrofuran, stirred at 25°C, filtered, and vacuum dried at 50°C for 2 hours to obtain Crystalline Form C of the compound of Formula IX, which is a solvate of tetrahydrofuran. The molar ratio of the

compound of Formula I to p-toluenesulfonic acid and tetrahydrofuran was 1:1.02:0.16. The XRPD pattern of Crystalline Form C of the compound of Formula IX is shown in FIG. 39, and it has the diffraction peaks shown in Table 23 below.

Table 23

Peak	Diffraction Angle (°, 2θ)	d-Spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	5.23	16.88487	100.00%	24	19.385	4.57526	32.40%
2	5.781	15.27577	5.00%	25	19.82	4.47596	71.20%
3	8.067	10.9514	10.80%	26	20.007	4.43447	44.60%
4	8.259	10.69689	15.40%	27	20.87	4.25293	9.40%
5	9.205	9.59966	38.30%	28	21.518	4.12633	12.70%
6	9.797	9.02074	15.20%	29	21.885	4.05803	7.90%
7	10.137	8.71938	8.20%	30	22.265	3.98958	15.40%
8	10.442	8.46535	6.60%	31	22.76	3.90396	6.20%
9	10.917	8.09813	8.20%	32	23.512	3.78074	21.30%
10	11.117	7.95271	14.80%	33	23.829	3.73113	12.00%
11	11.559	7.64976	4.40%	34	24.282	3.66256	6.90%
12	12.719	6.95419	24.10%	35	24.532	3.6258	8.90%
13	13.084	6.76127	6.90%	36	25.261	3.52275	12.70%
14	14.601	6.06167	4.30%	37	25.498	3.49051	12.60%
15	15.243	5.8078	17.70%	38	26.44	3.36825	22.20%
16	15.482	5.71887	14.20%	39	26.718	3.33385	8.70%
17	16.113	5.49622	35.50%	40	27.335	3.26006	10.20%
18	16.569	5.34603	40.00%	41	28.317	3.14917	11.00%
19	16.976	5.21884	17.70%	42	29.731	3.00254	9.60%
20	17.355	5.10576	6.40%	43	30.06	2.97044	5.40%
21	17.894	4.95308	11.00%	44	31.485	2.8391	5.00%
22	18.198	4.87091	19.60%	45	32.604	2.74421	5.40%
23	18.712	4.73836	15.10%				

Example 22 Crystalline Form D of the compound of Formula IX

Crystalline Form A of the compound of Formula IX was heated under a nitrogen atmosphere at 55°C to obtain Crystalline Form D of the compound of Formula IX. The molar ratio of the compound of Formula I to p-toluenesulfonic acid was 1:1. The XRPD pattern of Crystalline Form D of the p-toluenesulfonate salt of the compound of Formula I (Crystalline Form D of the compound of Formula IX) is shown in FIG. 40, and it has the diffraction peaks shown in Table 24 below.

Table 24

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	6.127	14.41406	100.00%	27	23.136	3.84136	7.90%
2	8.518	10.37289	16.50%	28	23.997	3.70544	19.80%
3	10.193	8.67124	4.60%	29	24.266	3.66497	8.20%
4	10.87	8.13289	27.30%	30	25.276	3.52065	0.90%
5	12.179	7.26166	22.40%	31	25.551	3.4835	1.10%
6	12.59	7.02527	3.10%	32	25.788	3.45202	2.10%
7	13.119	6.74327	19.70%	33	26.012	3.42274	1.20%
8	13.401	6.60209	1.40%	34	26.47	3.36459	3.20%
9	14.247	6.21148	14.00%	35	26.93	3.30808	3.20%
10	14.418	6.13826	10.90%	36	27.442	3.24752	2.30%
11	15.416	5.74307	10.40%	37	27.867	3.19895	2.30%
12	16.596	5.33741	24.70%	38	28.331	3.14765	12.30%
13	17.002	5.21068	5.80%	39	28.816	3.09575	5.10%
14	17.574	5.04238	1.60%	40	29.353	3.04029	3.00%
15	17.839	4.96809	3.60%	41	29.783	2.99737	2.70%
16	18.376	4.82432	33.50%	42	30.728	2.90733	1.80%
17	19.089	4.64558	11.90%	43	31.199	2.86452	2.10%
18	19.342	4.58548	13.10%	44	31.956	2.79836	1.30%
19	19.638	4.51695	3.30%	45	32.518	2.75125	2.10%
20	20.069	4.42091	20.90%	46	34.492	2.59823	0.80%
21	20.391	4.35189	3.10%	47	35.57	2.52192	0.20%
22	21.123	4.20265	8.40%	48	36	2.49272	1.70%
23	21.28	4.17204	8.20%	49	37.011	2.42697	0.90%
24	21.589	4.11296	10.60%	50	37.645	2.38754	1.40%
25	21.72	4.08848	8.50%	51	38.269	2.35	2.00%
26	22.095	4.01988	2.30%	52	39.074	2.30341	1.30%

Example 23 Crystalline Form E of the compound of Formula IX

40 mg of Crystalline Form A of the compound of Formula IX was added to 0.2 mL of isopropanol, heated to 50°C with shaking, filtered, and vacuum dried at 50°C for 2 hours to obtain Crystalline Form E of the isopropanol solvate of the p-toluenesulfonate salt of the compound of Formula I (Crystalline Form E of the compound of Formula IX). Its ¹H NMR spectrum showed that this crystalline form contained 0.98 equivalents of isopropanol. In Crystalline Form E of the compound of Formula IX, the molar ratio of the compound of Formula I, p-toluenesulfonic acid, and isopropanol was 1:1.02:0.98. The XRPD pattern of Crystalline Form E of the compound of Formula IX is shown in FIG. 41, and it has the

diffraction peaks shown in Table 25 below.

NMR: ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 13.65 (s, 1H), 9.15 (d, $J = 2.0$ Hz, 1H), 8.92 (s, 1H), 8.19 (s, 1H), 7.63 (s, 1H), 7.54 (s, 1H), 7.47 (d, $J = 8.4$ Hz, 2H), 7.11 (d, $J = 7.6$ Hz, 2H), 4.03 (s, 3H), 3.78 (sept, $J = 7.0$ Hz, 1H), 3.61 (s, 3H), 2.63 (s, 3H), 2.29 (s, 3H), 2.26 (d, $J = 1.6$ Hz, 3H), 1.09 (d, $J = 7.0$ Hz, 6H).

Table 25

Peak	Diffraction Angle ($^\circ$, 2 θ)	d-spacing ($\text{\AA}$)	Relative Intensity	Peak	Diffraction Angle ($^\circ$, 2 θ)	d-spacing ($\text{\AA}$)	Relative Intensity
1	5.105	17.29659	1.20%	34	23.946	3.7132	89.20%
2	5.656	15.61287	78.90%	35	24.461	3.63618	8.70%
3	8.105	10.89934	4.90%	36	25.046	3.55248	20.10%
4	8.451	10.45407	14.30%	37	25.494	3.4911	9.20%
5	9.022	9.79358	22.30%	38	25.85	3.44377	14.10%
6	9.839	8.98291	12.30%	39	26.135	3.40691	11.30%
7	11.192	7.89963	25.00%	40	26.582	3.35059	76.50%
8	11.501	7.68798	65.80%	41	26.981	3.30196	25.20%
9	12.286	7.19858	7.80%	42	27.858	3.19999	35.60%
10	12.704	6.96221	13.30%	43	28.054	3.17809	27.00%
11	13.853	6.38731	36.70%	44	28.344	3.14618	4.30%
12	14.296	6.1907	41.20%	45	28.859	3.09124	29.50%
13	15.359	5.76451	5.00%	46	29.053	3.07103	5.60%
14	15.586	5.68079	14.80%	47	29.717	3.00391	13.70%
15	16.204	5.46575	8.60%	48	30.36	2.94177	17.10%
16	16.56	5.349	23.50%	49	30.578	2.92124	32.90%
17	16.925	5.23427	6.90%	50	30.951	2.88692	6.40%
18	17.18	5.15736	18.90%	51	31.536	2.83466	6.50%
19	17.741	4.99534	68.20%	52	31.738	2.81713	5.10%
20	17.974	4.93107	92.90%	53	32.454	2.75658	6.10%
21	18.754	4.72791	22.60%	54	32.801	2.72817	2.10%
22	19.043	4.65667	64.50%	55	33.226	2.69424	5.10%
23	19.341	4.58571	17.70%	56	33.693	2.65797	4.10%
24	19.516	4.54484	18.90%	57	34.651	2.58661	1.80%
25	19.834	4.47269	100.00%	58	35.358	2.53655	3.40%
26	20.549	4.31873	12.80%	59	36.121	2.48464	3.30%
27	21.069	4.21326	3.60%	60	36.545	2.45681	8.60%
28	21.373	4.1541	5.60%	61	37.162	2.41742	3.80%
29	21.934	4.049	47.00%	62	37.521	2.39514	7.80%
30	22.246	3.99292	58.60%	63	38.009	2.36545	11.40%

31	22.467	3.95407	15.70%	64	38.724	2.32343	6.20%
32	23.082	3.8502	40.90%	65	39.556	2.27643	2.40%
33	23.431	3.79359	53.10%				

The DSC profile of Crystalline Form E of the compound of Formula IX is shown in FIG. 42. The DSC curve shows two endothermic peaks at peak temperatures of 102.94°C (enthalpy value 207.35 J/g) and 183.71°C (enthalpy value 34.855 J/g), with onset points (Onset x) at 65.61°C and 176.33°C, respectively. The TGA profile is shown in FIG. 43. The sample showed a weight loss of 3.11% upon heating from 33.06°C to 55.0°C, a weight loss of 3.82% upon heating from 55.0°C to 120.00°C, and a weight loss of 1.21% upon heating from 120.0°C to 190.0°C.

Example 24 Crystalline Form F of the compound of Formula IX

40 mg of Crystalline Form A of the compound of Formula IX was added to 1.6 mL of acetonitrile and dissolved with filtration. The filtrate was maintained at 25°C, and 3.2 mL of n-heptane was added to the filtrate. Solids precipitated from the system, which were filtered and vacuum dried at 50°C for 2 hours to obtain Crystalline Form F of the compound of Formula IX. The molar ratio of the compound of Formula I to p-toluenesulfonic acid was 1:1.02. The XRPD pattern of Crystalline Form F of the compound of Formula IX is shown in FIG. 44, and it has the diffraction peaks shown in Table 26 below.

¹H NMR (400 MHz, DMSO-*d*₆) δ 13.64 (s, 1H), 9.15 (d, *J* = 2.0 Hz, 1H), 8.91 (s, 1H), 8.19 (s, 1H), 7.63 (s, 1H), 7.53 (s, 1H), 7.47 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 7.6 Hz, 2H), 4.02 (s, 3H), 3.61 (s, 3H), 2.63 (s, 3H), 2.29 (s, 3H), 2.26 (d, *J* = 1.6 Hz, 3H).

Table 26

Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2θ)	d-spacing (Å)	Relative Intensity
1	6.146	14.36817	55.40%	39	23.461	3.7888	3.70%
2	6.985	12.6444	9.10%	40	24.029	3.70048	2.60%
3	9.906	8.92193	0.40%	41	24.342	3.65369	60.90%
4	10.338	8.55009	26.50%	42	24.635	3.6108	3.60%
5	10.747	8.22588	0.80%	43	25.251	3.52415	10.30%
6	11.517	7.67738	4.60%	44	25.738	3.45859	45.90%
7	11.849	7.46291	12.00%	45	26.024	3.42116	20.90%
8	12.265	7.21058	8.20%	46	26.651	3.34211	6.20%
9	12.57	7.03612	8.70%	47	26.827	3.32061	26.20%

10	12.727	6.94986	1.30%	48	27.268	3.26786	6.30%
11	13.351	6.62658	5.50%	49	27.871	3.19849	7.20%
12	13.965	6.33642	15.30%	50	28.126	3.17008	9.90%
13	14.137	6.25979	2.80%	51	28.463	3.13334	13.30%
14	15.519	5.70521	4.30%	52	28.743	3.10342	1.30%
15	15.732	5.62843	24.80%	53	29.705	3.00513	7.40%
16	16.335	5.42215	26.30%	54	29.989	2.97725	2.10%
17	16.549	5.35248	67.90%	55	30.185	2.95844	1.50%
18	16.727	5.29583	5.50%	56	30.524	2.9263	1.60%
19	17.083	5.1862	5.00%	57	30.716	2.90842	4.30%
20	17.303	5.12084	86.30%	58	31.277	2.85758	18.20%
21	17.726	4.99973	0.90%	59	31.732	2.81761	4.60%
22	18.067	4.90588	1.30%	60	32.331	2.76673	1.90%
23	18.423	4.81198	17.40%	61	32.696	2.73667	4.00%
24	18.917	4.68734	7.80%	62	32.983	2.71352	4.30%
25	19.365	4.58003	0.70%	63	33.237	2.69335	8.80%
26	19.824	4.47498	0.50%	64	33.376	2.6825	9.50%
27	20.419	4.34589	41.00%	65	33.68	2.65897	7.50%
28	20.72	4.28351	39.90%	66	34.419	2.60351	8.60%
29	20.98	4.23092	23.30%	67	35.046	2.55837	7.20%
30	21.217	4.18425	26.20%	68	35.674	2.51474	6.40%
31	21.479	4.13372	100.00%	69	36.241	2.47671	1.10%
32	21.825	4.06909	19.90%	70	36.574	2.45491	1.80%
33	21.972	4.04201	18.40%	71	36.884	2.43503	1.10%
34	22.242	3.99369	8.20%	72	37.361	2.40501	6.30%
35	22.433	3.96011	6.30%	73	38.144	2.3574	3.10%
36	22.689	3.91601	1.80%	74	38.48	2.33761	6.40%
37	22.973	3.86827	8.10%	75	39.118	2.30093	1.70%
38	23.269	3.81961	4.20%				

The DSC profile of Crystalline Form F of the compound of Formula IX is shown in FIG. 45. The DSC curve shows an endothermic peak at a peak temperature of 266.84°C (enthalpy value 70.167 J/g), with an onset point (Onset x) of 263.55°C. The TGA profile is shown in FIG. 46. The sample showed a weight loss of 2.2% upon heating from 34.19°C to 260.0°C.

Example 25 Crystalline Form G of the compound of Formula IX

40 mg of Crystalline Form A of the compound of Formula IX was added to 0.2 mL of 1,4-dioxane, heated to 50°C with shaking, filtered, and vacuum dried at 50°C for 2 hours to obtain Crystalline Form G of the compound of Formula IX. NMR analysis indicated that this crystalline form contained 3 equivalents of dioxane. The molar ratio of the compound of

Formula I, p-toluenesulfonic acid, and 1,4-dioxane was 1:1.10:2.96. The XRPD pattern of Crystalline Form G of the compound of Formula IX is shown in FIG. 47, and it has the diffraction peaks shown in Table 27 below.

NMR: ^1H NMR (400 MHz, DMSO- d_6) δ 13.65 (s, 1H), 9.15 (d, J = 2.0 Hz, 1H), 8.92 (s, 1H), 8.19 (s, 1H), 7.63 (s, 1H), 7.54 (s, 1H), 7.47 (d, J = 8.4 Hz, 2H), 7.11 (d, J = 7.6 Hz, 2H), 4.03 (s, 3H), 3.57 (s, 24H), 3.61 (s, 3H), 2.63 (s, 3H), 2.29 (s, 3H), 2.26 (d, J = 1.6 Hz, 3H).

Table 27

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	7.646	11.55367	25.60%	26	23.096	3.8478	7.00%
2	9.655	9.15311	3.90%	27	23.513	3.78055	6.80%
3	10.734	8.23557	1.20%	28	23.827	3.73146	23.40%
4	11.096	7.96733	1.80%	29	24.468	3.63515	3.10%
5	11.868	7.45081	7.90%	30	25.372	3.50758	100.00%
6	12.603	7.01819	8.50%	31	25.632	3.47261	35.80%
7	12.759	6.93236	8.00%	32	26.652	3.34203	12.20%
8	13.756	6.43239	28.70%	33	27.17	3.2794	8.70%
9	14.113	6.27053	5.20%	34	27.595	3.22992	51.60%
10	16.072	5.5101	13.10%	35	28.363	3.14416	8.50%
11	17.036	5.2006	3.70%	36	29.174	3.05861	15.80%
12	17.432	5.08315	5.10%	37	29.887	2.98725	2.90%
13	17.641	5.02357	11.10%	38	31.461	2.84122	4.60%
14	17.82	4.97352	8.60%	39	31.788	2.8128	9.50%
15	18.409	4.8157	8.90%	40	32.382	2.76247	2.80%
16	18.753	4.72799	6.10%	41	33.264	2.69128	6.70%
17	18.969	4.67472	9.60%	42	33.57	2.6674	4.70%
18	19.41	4.56954	2.20%	43	33.857	2.64545	3.80%
19	20.052	4.42453	12.10%	44	34.249	2.61606	6.50%
20	20.348	4.36084	40.00%	45	34.781	2.57725	2.50%
21	21.009	4.22519	49.00%	46	36.21	2.47876	2.60%
22	21.306	4.16701	10.50%	47	37.185	2.41595	1.70%
23	21.568	4.11689	63.70%	48	38.068	2.36195	4.70%
24	22.232	3.99544	2.10%	49	38.407	2.34188	3.40%
25	22.748	3.90587	15.20%	50	38.739	2.32259	1.90%

Example 26 Crystalline Form H of the compound of Formula IX

40 mg of Crystalline Form A of the compound of Formula IX was added to 3.0 mL of acetonitrile and dissolved with filtration. 12 mL of methyl tert-butyl ether was added to the filtrate, resulting in precipitation of a solid, which was filtered to obtain Crystalline Form H of the compound of Formula IX. NMR analysis indicated it contained 0.45 equivalents of methyl tert-butyl ether. The molar ratio of the compound of Formula I, p-toluenesulfonic acid, and the

solvent methyl tert-butyl ether was 1:1.00:0.5. The XRPD pattern of Crystalline Form H of the compound of Formula IX is shown in FIG. 48, and it has the diffraction peaks shown in Table 28 below.

NMR: ^1H NMR (400 MHz, DMSO- d_6) δ 13.65 (s, 1H), 9.15 (d, $J = 2.0$ Hz, 1H), 8.92 (s, 1H), 8.19 (s, 1H), 7.63 (s, 1H), 7.54 (s, 1H), 7.47 (d, $J = 8.4$ Hz, 2H), 7.11 (d, $J = 7.6$ Hz, 2H), 4.03 (s, 3H), 3.61 (s, 3H), 3.08 (s, 1.5H) 2.63 (s, 3H), 2.29 (s, 3H), 2.26 (d, $J = 1.6$ Hz, 3H), 1.11 (s, 4.5H).

Table 28

Peak	Diffraction Angle ($^\circ$, 2θ)	d-spacing ($\text{\AA}$)	Relative Intensity	Peak	Diffraction Angle ($^\circ$, 2θ)	d-spacing ($\text{\AA}$)	Relative Intensity
1	5.207	16.95771	14.10%	24	22.437	3.95941	11.80%
2	8.269	10.6847	27.00%	25	23.355	3.80574	16.20%
3	9.701	9.11034	29.10%	26	24.458	3.63664	4.60%
4	10.392	8.5059	9.80%	27	24.922	3.56991	4.80%
5	11.07	7.98592	17.20%	28	25.232	3.52673	30.70%
6	11.597	7.62467	11.30%	29	26.139	3.40641	26.00%
7	12.557	7.04336	18.00%	30	26.334	3.38168	19.10%
8	13.05	6.77887	4.30%	31	27.268	3.26793	16.00%
9	14.964	5.91575	4.00%	32	27.951	3.18958	16.80%
10	15.286	5.79185	10.80%	33	28.311	3.14978	7.50%
11	16.554	5.35087	43.40%	34	29.557	3.01977	23.20%
12	17.072	5.18967	23.20%	35	30.05	2.97142	10.70%
13	17.463	5.07429	2.80%	36	30.383	2.93961	10.10%
14	18.079	4.90283	2.20%	37	30.874	2.89397	9.50%
15	18.485	4.79597	11.50%	38	31.272	2.85799	4.00%
16	18.793	4.71809	19.70%	39	32.212	2.77672	12.70%
17	19.087	4.64603	29.50%	40	33.403	2.68034	1.80%
18	19.784	4.48383	100.00%	41	35.024	2.55994	2.10%
19	20.812	4.26473	9.10%	42	36.282	2.474	3.00%
20	21.118	4.20364	15.80%	43	37.312	2.40804	2.10%
21	21.463	4.13686	23.00%	44	38.273	2.3498	3.70%
22	21.926	4.05052	3.50%	45	38.916	2.31242	2.80%
23	22.237	3.99455	7.90%				

The DSC pattern of Crystalline Form H of the compound of Formula IX is shown in FIG. 49. The DSC curve shows endothermic peaks at peak temperatures of 45.53°C (enthalpy value 10.478 J/g), 155.23°C (enthalpy value 29.331 J/g), and 191.45°C (enthalpy value 23.665 J/g), with onset points (Onset x) at 29.17°C, 134.74°C, and 184.26°C, respectively. The TGA pattern

is shown in FIG. 50. The sample showed a weight loss of 1.74% when heated from 32.99°C to 100.0°C, and a weight loss of 5.38% when heated from 100.0°C to 180.0°C.

Example 27 Crystalline Form I of the compound of Formula IX

Crystalline Form A of the compound of Formula IX was heated under a nitrogen atmosphere at 25°C to obtain Crystalline Form I of the compound of Formula IX. The molar ratio of the compound of Formula I to p-toluenesulfonic acid was 1:1. The XRPD pattern of Crystalline Form I of the compound of Formula I is shown in FIG. 51, and it has the diffraction peaks shown in Table 29 below.

Table 29

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	6.01	14.69474	100.00%	22	22.213	3.99885	2.80%
2	8.343	10.5898	2.70%	23	22.458	3.95579	7.20%
3	10.023	8.81831	4.60%	24	22.676	3.91814	3.60%
4	10.63	8.31585	16.60%	25	23.219	3.82777	14.60%
5	11.082	7.97786	3.70%	26	24.06	3.69589	10.00%
6	11.991	7.37471	7.00%	27	24.725	3.59791	2.20%
7	12.4	7.13247	5.80%	28	25.351	3.51041	5.30%
8	12.792	6.91459	18.40%	29	25.658	3.46913	3.90%
9	14.204	6.23046	14.50%	30	26.084	3.41342	4.80%
10	15.035	5.88764	6.30%	31	26.374	3.37659	3.40%
11	16.674	5.31265	26.20%	32	27.684	3.2197	5.90%
12	17.842	4.96727	17.20%	33	28.076	3.17566	6.60%
13	17.999	4.92439	19.30%	34	28.81	3.09642	2.00%
14	18.468	4.80044	3.40%	35	29.18	3.05794	4.90%
15	18.743	4.73046	8.60%	36	30.919	2.88983	2.90%
16	19.542	4.53886	1.30%	37	31.837	2.80853	1.40%
17	20.069	4.42085	11.90%	38	32.985	2.71338	1.00%
18	20.069	4.42085	11.90%	39	33.718	2.65607	1.50%
19	20.418	4.34616	1.30%	40	37.941	2.36955	2.80%
20	20.9	4.2469	3.50%	41	39.251	2.29347	1.80%
21	21.201	4.18728	4.40%				

Example 28 Crystalline Form J of the compound of Formula IX

Crystalline Form H of the compound of Formula IX was heated under a nitrogen atmosphere at 170°C. After solvent removal, Crystalline Form J of the compound of Formula IX was

obtained. The molar ratio of the compound of Formula I to p-toluenesulfonic acid was 1:1.01. The XRPD pattern of Crystalline Form J of the compound of Formula IX is shown in FIG. 52, and it has the diffraction peaks shown in Table 30 below.

Table 30

Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity	Peak	Diffraction Angle (°, 2 θ)	d-spacing (Å)	Relative Intensity
1	5.272	16.74922	57.30%	13	19.073	4.64948	35.50%
2	8.181	10.79936	50.00%	14	19.655	4.51309	35.70%
3	9.719	9.09269	40.00%	15	20.205	4.3915	62.00%
4	10.542	8.38506	39.80%	16	21.25	4.17776	27.60%
5	11.063	7.99099	100.00%	17	22.327	3.97857	2.00%
6	11.681	7.56951	12.90%	18	23.074	3.85153	20.40%
7	12.442	7.10847	84.30%	19	25.084	3.54725	0.50%
8	12.876	6.8697	36.10%	20	26.478	3.36356	35.00%
9	15.295	5.7885	32.70%	21	27.524	3.23811	7.20%
10	16.408	5.39808	32.30%	22	28.157	3.16666	20.90%
11	17.765	4.98876	8.00%	23	29.266	3.04914	20.40%
12	18.786	4.71982	95.30%				

Test Example 1

The crystalline form C of the compound of formula I obtained in Example 1-2, the crystalline form C of the compound of formula V obtained in Example 9, the crystalline form A of the compound of formula IX obtained in Example 13, and the crystalline form F of the compound of formula IX obtained in Example 24 were placed under open conditions at 25°C/60% RH and sealed conditions at 60°C for two weeks. As shown in FIG. 53, FIG. 54, FIG. 55, and FIG. 56, the crystalline forms exhibited good stability.

Test Example 2

Test method: A gradient of 40-0-95-0-40% RH was set, with dm/dt 0.002/s. The system was equilibrated for 60~360 min for every 10% RH change. The test temperature was 25°C.

Test results showed that the crystalline form C of the compound of formula I was non-hygroscopic, and the crystalline form remained unchanged before and after the test. The crystalline form A of the compound of formula III exhibited moderate hygroscopicity, absorbing 4.8% water. The crystalline form A of the compound of formula IX absorbed 0.2% water, and the crystalline form C of the compound of formula V absorbed 1.7% water.

Test Example 3

The crystalline form C of the compound of formula I, the crystalline form A of the compound of formula III, the crystalline form C of the compound of formula V, and the crystalline form A of the compound of formula IX obtained in the above examples were subjected to solubility testing. The test method was as follows:

The crystalline form C of the compound of formula I (8 mg), the crystalline form A of the compound of formula III (9 mg), the crystalline form A of the compound of formula IX (11.7 mg), and the crystalline form C of the compound of formula V (10 mg) were weighed into 20 mL glass vials, respectively. 4 mL of medium was added to each. The resulting suspensions or clear solutions were stirred at 37°C at 400 rpm. Samples (0.5 mL each) were taken at 0.5 hours and 2 hours, and centrifuged at 37°C at 14,000 rpm for 5 min. The resulting filtrates were finally subjected to content testing.

The results showed that the crystalline forms obtained in the present application had good solubility, as shown in the table below:

A indicates solubility of 0.01-0.1 mg/mL, B indicates solubility of 0.1-1 mg/mL.

	Physical Form							
	Crystalline form C of the compound of formula I		Crystalline form A of the compound of formula III		Crystalline form C of the compound of formula V		Crystalline form A of the compound of formula IX	
	Solubility (mg/mL)							
Dissolution Time	0.5h	2h	0.5h	2h	0.5h	2h	0.5h	2h
Water	A	A	A	A	A	A	A	A
Fasted State Simulated Gastric Fluid	A	A	B	B	B	B	B	B
Fasted State Simulated Intestinal Fluid	A	A	B	A	B	B	B	B
Fed State Simulated Intestinal Fluid	A	A	B	B	B	B	B	B

Rat PK Assay

The purpose of this experiment was to study the pharmacokinetics of the test compounds administered orally to SD rats (SPF grade, Beijing Vital River Laboratory Animal Technology Co., Ltd.). The experimental procedure was as follows:

- 1) The above compound crystalline forms were uniformly dispersed in a mixture of 99.9% (0.5% MC (450 cp)/water) + 0.1% Tween 80 at a concentration of 5.0 mg/mL, and then administered orally (10 mL/kg);
- 2) Blood samples were collected at 0.083, 0.25, 0.5, 1, 2, 4, 6, 8, and 24 hours, with 0.25 mL collected each time, and EDTA-K2 was added. After collection, the blood samples were placed on ice and centrifuged within 1 hour to separate plasma (centrifugation conditions: 6000 g, 3 minutes, 2-8°C). Plasma samples were stored in a -80°C freezer prior to analysis;
- 3) For sample analysis, the concentration of the compound in the plasma samples was analyzed using an LC-MS/MS method. For the plasma assay results, pharmacokinetic parameters were calculated using WinNonlin (Phoenix, version 8.2.0) or other similar software.

The results showed that the crystalline forms of the present application exhibited high exposure and favorable metabolic performance, as shown in the table below:

Physical Form	Dose	T _{max} (h)	C _{max} (ng/mL)	AUC _(0-t) (h*ng/mL)
Crystalline Form A of the compound of Formula II	50mg/kg	10.7	543	5997
Crystalline Form A of the compound of Formula IX	50mg/kg	0.833	97800	743182

Test Example 4

1. Biochemical Activity Assay for Pol θ Inhibition (ADP Glo Assay)

The N-terminus of the Pol θ protein contains a helicase domain with ATPase activity, capable of hydrolyzing ATP to ADP. The ATPase activity of the Pol θ protein and the inhibitory effect of small molecule compounds on the protein can be analyzed using the ADP-Glo detection kit (ADP-Glo™ Kinase Assay, Promega, V9102). The specific assay method is as follows:

- 1) The helicase domain of the Pol θ protein was purified using an insect system to obtain protein with purity >90%. ssDNA (SEQ ID NO.1: 5'-CCAGTGAATTGTTGCTCGGTACCTGCTAAC-3', Hangzhou Yocon Biotechnology Co., Ltd.) was ordered as the substrate for the Pol θ protein; a reaction buffer containing 10 mM DTT (Dithiothreitol), 20 mM MgCl₂, and Tris-HCl pH 7.5 was prepared;
- 2) A 2x ssDNA-Pol θ premix was prepared, and the reaction was carried out in a 384-well plate.

A control group containing only buffer was set up, and the premix was added to all other groups. Compounds were added using an automated pipettor (Thermo, Multidrop 8), starting at a concentration of 10 μ M and diluted at a 1/3 ratio, with a total of 9 detection points set, each with 2 replicates;

3) A 2x ATP solution was prepared, and an equal volume of the 2x ATP solution was added to all wells, followed by incubation at room temperature for 60 min;

4) According to the Promega reagent instructions, the ADP-Glo Detection Reagent was added to the reaction system, followed by incubation at room temperature for 60 min;

5) According to the Promega reagent instructions, the Kinase Detection Reagent was added to the reaction system, followed by incubation at room temperature for 60 min. The chemiluminescence signal was detected using a microplate reader (Thermo, VarioskanLUX), with the reading interval per well set to 1000 ms;

Data analysis was performed on the measurement results: the CV% of the control group detection results should be less than 10%, and the z' value should be greater than 0.5. For data meeting the above quality control results, the compound inhibition rate was calculated (Inhibition rate (%) = 100 * (control group average - experimental group) / (control group average - buffer control group average)). GraphPad Prism8 was used to perform nonlinear regression fitting of the compound inhibition rate curve and derive the IC50 value.

2. Pol θ Inhibitor Cell Viability Assay

The in vitro efficacy of the inhibitors was evaluated using a cell viability assay with CellTiter-Glo reagent (Promega, G7573), i.e., the commonly used CTG assay. The specific assay method is as follows:

1) DLD1 and DLD1-/- cells were cultured. One day before the assay, cells were digested with trypsin (0.025% Trypsin-EDTA, Hyclone), centrifuged at 1000 rpm for 3 min, and collected. Cells were counted using a cell counter (Shanghai Monwei Biomedical Technology Co.,Ltd., SmartCell600A.SC1006) and seeded into a 96-well white culture plate at an appropriate seeding ratio.

2) Drug treatment was performed 24 hours after cell plating, recorded as Day 0. Compound addition was performed using an automated pipettor (Thermo, Multidrop 8), with the plate set as a 96-well plate, starting concentration set at 30 μ M, diluted at a 1/3 ratio, with a total of 9 detection points set, each with 2 replicates. Cells were cultured in a 37°C, 5% CO₂ incubator.

3) Medium changes were performed on Day 3 and Day 7, and compounds were added according to step 2).

4) On Day 10, the cell culture plate was removed, and an equal volume of CTG reagent (which needed to be equilibrated to temperature and premixed according to the reagent instructions beforehand) was added. The plate was gently mixed for 10 min (speed 300) on a constant temperature mixer (Hangzhou Allsheng Instruments Co.,Ltd., MSC-100), and chemiluminescence detection was performed using a microplate reader (Thermo, VarioskanLUX).

5) Data analysis was performed on the measurement results: the CV% of the control group detection results should be less than 20%, and the z' value should be greater than 0.5. For data meeting the above quality control results, cell viability was calculated (Inhibition rate (%) = $100 * (\text{control group average} - \text{experimental group}) / (\text{control group average} - \text{medium control group average})$). GraphPad Prism8 was used to perform nonlinear regression fitting of the compound inhibition rate curve and derive the IC₅₀ value.

Data List:

Specific data for the biochemical assay and cell viability assay are shown in the table below:

Example	Biochemical Assay IC ₅₀ (nM)	Cell Viability Assay IC ₅₀ (nM)
1	3.31	<1.52

3. Solubility Assay

1) Preparation of 0.1 M Na₂PO₄ buffer (pH 7.4):

Add 11 g of Na₂HPO₄ (FW: 141.96) and 3.5 g of NaH₂PO₄·2H₂O (FW: 156.03) to 1 L of Milli-Q water, and adjust the pH to 7.4 using phosphoric acid or sodium hydroxide;

2) Add 10 µL of the test compound (concentration: 10 mM in DMSO) to 990 µL of the Na₂PO₄ buffer prepared in step 1 (final DMSO concentration: 1%);

3) Shake the sample tube at room temperature for 2 hours (1000 rpm/min);

4) Calibration curve preparation:

a) Prepare a 300 µM spiking solution (SS):

Add 6 µL (10 mM in DMSO) of the test compound stock solution to 194 µL of MeOH/ACN (4:1);

b) Draw the standard curve:

	MeOH:ACN (4:1)	STD ID	STD Conc. (µM)
--	----------------	--------	----------------

	mixture (μL)		
100 μL SS (300 μM)	400	STD 8	60
100 μL STD 8	200	STD 7	20
100 μL STD 7	100	STD 6	10
100 μL STD 6	150	STD 5	4
100 μL STD 5	400	STD 4	0.8
100 μL STD 4	300	STD 3	0.2
100 μL STD 3	100	STD 2	0.1
100 μL STD 2	400	STD 1	0.02

5) Centrifuge the sample (10 minutes, 12000 rpm) to precipitate insoluble particles. Filter the supernatant through a 0.22 μm filter, then transfer the supernatant to a new centrifuge tube.

6) Dilute the supernatant 10-fold with 100 mM buffer

Add 10 μL of the supernatant to 90 μL of buffer (100 mM) to achieve a 10-fold dilution;

7) LC-MS/MS (API 4000) sample preparation

Add 10 μL of the sample (10-fold dilution) and standard curve samples to 400 μL of solution (MeOH : ACN = 1 : 1), then perform the detection. Calculate the solubility value based on the standard curve.

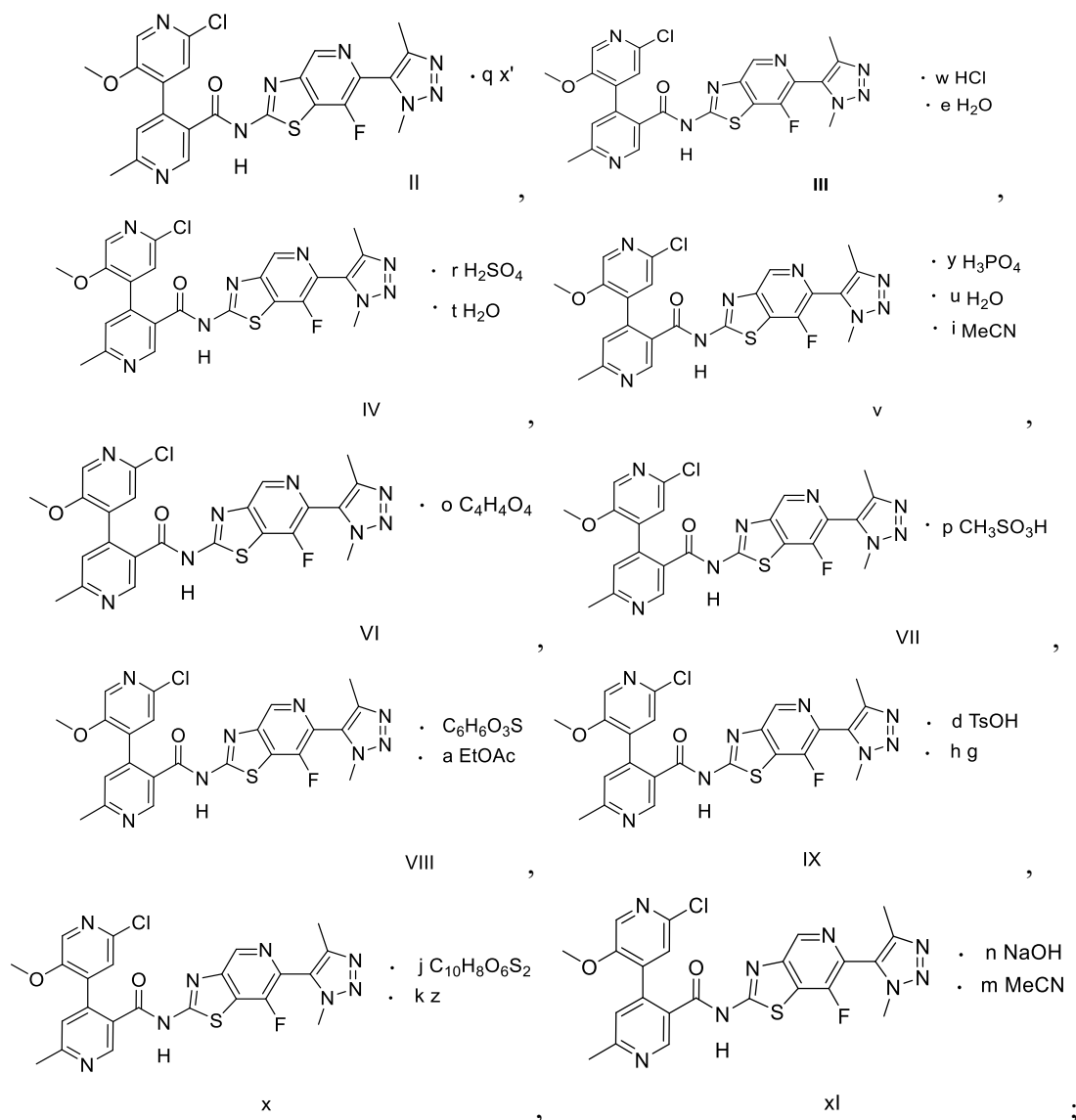
The experimental results are shown in the table below:

Example	Solubility (μM)
1	61.8

Although specific embodiments of the present invention have been described above, those skilled in the art should understand that these are merely illustrative examples. Various changes or modifications can be made to these embodiments without departing from the principle and essence of the present invention. Therefore, the scope of protection of the present invention is defined by the appended claims.

Claims

1. A nitrogen-containing fused-ring compound, characterized in that it is a compound of formula II, a compound of formula III, a compound of formula IV, a compound of formula V, a compound of formula VI, a compound of formula VII, a compound of formula VIII, a compound of formula IX, a compound of formula X, or a compound of formula XI;



wherein,

in the compound of formula II, x' is water or acetone, and q is 0.01-2.5;

in the compound of formula III, w is 0.5-2.5, and e is 0-2;

in the compound of formula IV, r is 0.5-2, and t is 0-2;

in the compound of formula V, y is 0.5-1.5, u is 0-2, and i is 0-2;

in the compound of formula VI, C₄H₄O₄ is fumaric acid, and o is 1-1.1;

in the compound of formula VII, $\text{CH}_3\text{SO}_3\text{H}$ is methanesulfonic acid, and p is 1-1.1;
 in the compound of formula VIII, $\text{C}_6\text{H}_5\text{O}_3\text{S}$ is benzenesulfonic acid, and a is 0-1;
 in the compound of formula IX, d is 1-2, g is water, tetrahydrofuran, isopropanol, 1,4-dioxane, or methyl tert-butyl ether, and h is 0-3;
 in the compound of formula X, $\text{C}_{10}\text{H}_8\text{O}_6\text{S}_2$ is 1,5-naphthalenedisulfonic acid, j is 0.5-1.5, z is water or acetonitrile, and k is 0-5;
 in the compound of formula XI, n is 0-1.5, and m is 0 or 0.1-1.5.

2. The nitrogen-containing fused-ring compound according to claim 1, characterized in that it satisfies one or more of the following conditions:

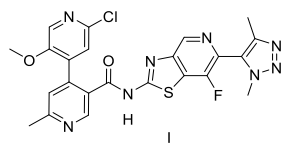
- (1) in said compound of formula II, q is 0.1, 0.5, 0.56, 0.6, 1, 1.5, 2, or 2.5;
- (2) in said compound of formula III, w is 0.5, 1, 1.1, 1.6, 1.7, 1.8, 1.76, 1.06, 1.5, or 2;
- (3) in said compound of formula III, e is 0 or 1-2, for example, e is 1.5;
- (4) in said compound of formula IV, r is 0.5, 0.9, 1, 0.97, 0.96, or 1.5;
- (5) in said compound of formula IV, t is 0 or 1-2, for example, t is 1.7;
- (6) in said compound of formula V, y is 1.1, 1.2, 1.14, 1, 1.09, or 1.05;
 preferably, y and u are 0; or, i is 0, and u is 0.1, 0.91, 0.9, or 1, for example, i is 0 and u is 0.91;
 or, u is 0, and i is 0.1, 0.18, 0.2, or 0.5, for example, u is 0 and i is 0.18 or 0.1;
- (7) in said compound of formula VI, o is 1, 1.01, 1.05, or 1.1;
- (8) in said compound of formula VII, p is 1, 1.03, 1.05, or 1.1;
- (9) in said compound of formula VIII, a is 0, or, a is 0-0.1, for example, 0.07, 0.1, or 0;
- (10) in said compound of formula IX, d is 1, 1.1, 1.2, 1.02, or 1.5;
- (11) in said compound of formula IX, h is 0; or, h is 0.1-3, for example, g is water and h is 3; or, g is tetrahydrofuran and h is 0.2, 0.1, or 0.16; or, g is isopropanol and h is 0.9, 0.98, or 1; or, g is 1,4-dioxane and h is 3, 2.96, or 2.9; or, g is methyl tert-butyl ether and h is 0.4, 0.45, or 0.5;
- (12) in said compound of formula X, j is 1, 0.9, 1.1, 1.02, or 0.93;
- (13) in said compound of formula X, k is 0, or, k is 0.01-5, for example, z is water and k is 5 or 4.8; or, z is acetonitrile and k is 0.07;
- (14) in said compound of formula XI, n is 0.6, 1.1, 1.13, or 1.2;
- (15) in said compound of formula XI, m is 0, or, m is 0.5-1.5, for example, 1, 1.07, or 1.1.

3. A crystalline form C of a compound of formula I or the nitrogen-containing fused-ring compound according to claim 1 or 2, characterized in that it satisfies one or more of the following conditions:

(1) said compound of formula II is crystalline form A of the compound of formula II, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 9.00 $\pm$ 0.2 $^\circ$, 10.84 $\pm$ 0.2 $^\circ$, 17.91 $\pm$ 0.2 $^\circ$, and 22.05 $\pm$ 0.2 $^\circ$;

(2) said compound of formula II is crystalline form B of the compound of formula II, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 8.10 $\pm$ 0.2 $^\circ$, 8.37 $\pm$ 0.2 $^\circ$, 13.94 $\pm$ 0.2 $^\circ$, 15.67 $\pm$ 0.2 $^\circ$, 20.72 $\pm$ 0.2 $^\circ$, 24.4 $\pm$ 0.2 $^\circ$, and 24.83 $\pm$ 0.2 $^\circ$;

(3) said crystalline form C of the compound of formula I has an X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, with diffraction peaks at 11.51 $\pm$ 0.2 $^\circ$, 18.01 $\pm$ 0.2 $^\circ$, 22.56 $\pm$ 0.2 $^\circ$, and 25.68 $\pm$ 0.2 $^\circ$;



wherein the structure of the compound of formula I is:

(4) said compound of formula III is crystalline form A of the compound of formula III, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 4.61 $\pm$ 0.2 $^\circ$, 5.02 $\pm$ 0.2 $^\circ$, 9.19 $\pm$ 0.2 $^\circ$, and 13.76 $\pm$ 0.2 $^\circ$;

(5) said compound of formula III is crystalline form B of the compound of formula III, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 6.47 $\pm$ 0.2 $^\circ$, 7.19 $\pm$ 0.2 $^\circ$, 9.11 $\pm$ 0.2 $^\circ$, 11.63 $\pm$ 0.2 $^\circ$, and 14.45 $\pm$ 0.2 $^\circ$;

(6) said compound of formula III is crystalline form C of the compound of formula III, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 9.84 $\pm$ 0.2 $^\circ$, 10.51 $\pm$ 0.2 $^\circ$, 12.19 $\pm$ 0.2 $^\circ$, 13.39 $\pm$ 0.2 $^\circ$, and 14.40 $\pm$ 0.2 $^\circ$;

(7) said compound of formula IV is crystalline form A of the compound of formula IV, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 5.14 $\pm$ 0.2 $^\circ$, 9.01 $\pm$ 0.2 $^\circ$, and 15.03 $\pm$ 0.2 $^\circ$;

(8) said compound of formula IV is crystalline form B of the compound of formula IV, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 7.11 $\pm$ 0.2 $^\circ$, 7.57 $\pm$ 0.2 $^\circ$, 7.72 $\pm$ 0.2 $^\circ$, 8.10 $\pm$ 0.2 $^\circ$, and 10.27 $\pm$ 0.2 $^\circ$;

(9) said compound of formula V is crystalline form A of the compound of formula V, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks at 5.15 $\pm$ 0.2 $^\circ$, 8.34 $\pm$ 0.2 $^\circ$, 15.51 $\pm$ 0.2 $^\circ$, and 15.88 $\pm$ 0.2 $^\circ$;

- (10) said compound of formula V is crystalline form B of the compound of formula V, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.83\pm0.2^\circ$, $12.89\pm0.2^\circ$, $14.44\pm0.2^\circ$, $15.91\pm0.2^\circ$, $25.08\pm0.2^\circ$, and $25.50\pm0.2^\circ$;
- (11) said compound of formula V is crystalline form C of the compound of formula V, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.19\pm0.2^\circ$, $8.39\pm0.2^\circ$, $10.47\pm0.2^\circ$, and $11.51\pm0.2^\circ$;
- (12) said compound of formula VI is crystalline form A of the compound of formula VI, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $10.58\pm0.2^\circ$, $12.16\pm0.2^\circ$, and $13.98\pm0.2^\circ$;
- (13) said compound of formula VII is crystalline form A of the compound of formula VII, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $9.00\pm0.2^\circ$, $17.44\pm0.2^\circ$, and $21.74\pm0.2^\circ$;
- (14) said compound of formula VIII is crystalline form A of the compound of formula VIII, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $7.66\pm0.2^\circ$, $10.09\pm0.2^\circ$, $11.92\pm0.2^\circ$, and $13.91\pm0.2^\circ$;
- (15) said compound of formula IX is crystalline form A of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.96\pm0.2^\circ$, $16.71\pm0.2^\circ$, $17.79\pm0.2^\circ$, $20.33\pm0.2^\circ$, and $24.66\pm0.2^\circ$;
- (16) said compound of formula IX is crystalline form B of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.34\pm0.2^\circ$, $9.34\pm0.2^\circ$, $9.92\pm0.2^\circ$, $12.84\pm0.2^\circ$, and $16.61\pm0.2^\circ$;
- (17) said compound of formula X is crystalline form A of the compound of formula X, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $7.46\pm0.2^\circ$, $12.01\pm0.2^\circ$, and $21.70\pm0.2^\circ$;
- (18) said compound of formula X is crystalline form B of the compound of formula X, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $8.96\pm0.2^\circ$, $10.81\pm0.2^\circ$, $17.92\pm0.2^\circ$, and $22.07\pm0.2^\circ$;
- (19) said compound of formula X is crystalline form C of the compound of formula X, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $11.30\pm0.2^\circ$, $22.61\pm0.2^\circ$, $24.69\pm0.2^\circ$, $26.16\pm0.2^\circ$, and $28.57\pm0.2^\circ$;
- (20) said compound of formula X is crystalline form D of the compound of formula X, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has

diffraction peaks at $10.68 \pm 0.2^\circ$ and $21.44 \pm 0.2^\circ$, wherein z is water;

(21) said compound of formula XI is crystalline form A of the compound of formula XI, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $12.78 \pm 0.2^\circ$, $14.09 \pm 0.2^\circ$, and $14.98 \pm 0.2^\circ$;

(22) said compound of formula XI is crystalline form B of the compound of formula XI, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $8.22 \pm 0.2^\circ$, $19.38 \pm 0.2^\circ$, $20.38 \pm 0.2^\circ$, $23.43 \pm 0.2^\circ$, and $24.74 \pm 0.2^\circ$;

(23) said compound of formula IX is crystalline form C of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.23 \pm 0.2^\circ$ and $19.82 \pm 0.2^\circ$;

(24) said compound of formula IX is crystalline form D of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.13 \pm 0.2^\circ$, $10.87 \pm 0.2^\circ$, and $18.38 \pm 0.2^\circ$;

(25) said compound of formula IX is crystalline form E of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.66 \pm 0.2^\circ$, $11.50 \pm 0.2^\circ$, $17.97 \pm 0.2^\circ$, $19.83 \pm 0.2^\circ$, $23.95 \pm 0.2^\circ$, and $26.58 \pm 0.2^\circ$;

(26) said compound of formula IX is crystalline form F of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.15 \pm 0.2^\circ$, $16.55 \pm 0.2^\circ$, $17.30 \pm 0.2^\circ$, and $21.48 \pm 0.2^\circ$;

(27) said compound of formula IX is crystalline form G of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $7.65 \pm 0.2^\circ$, $13.76 \pm 0.2^\circ$, $20.35 \pm 0.2^\circ$, $21.01 \pm 0.2^\circ$, and $21.57 \pm 0.2^\circ$, wherein g is 1,4-dioxane;

(28) said compound of formula IX is crystalline form H of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $8.27 \pm 0.2^\circ$, $9.70 \pm 0.2^\circ$, $16.55 \pm 0.2^\circ$, and $19.78 \pm 0.2^\circ$, wherein g is methyl tert-butyl ether;

(29) said compound of formula IX is crystalline form I of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.01 \pm 0.2^\circ$, $10.63 \pm 0.2^\circ$, and $12.79 \pm 0.2^\circ$;

(30) said compound of formula IX is crystalline form J of the compound of formula IX, and its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has

diffraction peaks at $5.27\pm0.2^\circ$, $8.18\pm0.2^\circ$, $9.72\pm0.2^\circ$, $10.54\pm0.2^\circ$, $11.06\pm0.2^\circ$, $12.44\pm0.2^\circ$, $18.79\pm0.2^\circ$, and $20.21\pm0.2^\circ$.

4. The crystalline form C of the compound of formula I or said nitrogen-containing fused-ring compound according to claim 3, characterized in that it satisfies one or more of the following conditions:

(1) said crystalline form A of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $5.98\pm0.2^\circ$, $11.94\pm0.2^\circ$, $18.36\pm0.2^\circ$, $21.30\pm0.2^\circ$, $23.29\pm0.2^\circ$, and $27.72\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $9.00\pm0.2^\circ$, $10.84\pm0.2^\circ$, $17.91\pm0.2^\circ$, $22.05\pm0.2^\circ$, $5.98\pm0.2^\circ$, $11.94\pm0.2^\circ$, $18.36\pm0.2^\circ$, $21.30\pm0.2^\circ$, and $23.29\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $13.46\pm0.2^\circ$, $14.16\pm0.2^\circ$, $14.72\pm0.2^\circ$, $19.99\pm0.2^\circ$, $20.65\pm0.2^\circ$, $24.6\pm0.2^\circ$, $25.62\pm0.2^\circ$, $28.96\pm0.2^\circ$, $29.68\pm0.2^\circ$, $30.29\pm0.2^\circ$, and $31.08\pm0.2^\circ$;

(2) said crystalline form B of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $13.33\pm0.2^\circ$, $14.79\pm0.2^\circ$, $17.63\pm0.2^\circ$, and $21.15\pm0.2^\circ$;

for example, said crystalline form B of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $8.10\pm0.2^\circ$, $8.37\pm0.2^\circ$, $13.94\pm0.2^\circ$, $15.67\pm0.2^\circ$, $20.72\pm0.2^\circ$, $14.26\pm0.2^\circ$, $16.61\pm0.2^\circ$, $17.91\pm0.2^\circ$, and $18.70\pm0.2^\circ$;

preferably, said crystalline form B of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $14.26\pm0.2^\circ$, $16.61\pm0.2^\circ$, $17.91\pm0.2^\circ$, $18.70\pm0.2^\circ$, $19.13\pm0.2^\circ$, $19.62\pm0.2^\circ$, $20.03\pm0.2^\circ$, $23.92\pm0.2^\circ$, $24.20\pm0.2^\circ$, and $25.42\pm0.2^\circ$;

(3) said crystalline form C of the compound of formula I, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $12.88\pm0.2^\circ$, $15.05\pm0.2^\circ$, $20.85\pm0.2^\circ$, $21.68\pm0.2^\circ$, $23.11\pm0.2^\circ$, and $24.79\pm0.2^\circ$;

for example, in said crystalline form C of the compound of formula I, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has the diffraction peak at $11.51\pm0.2^\circ$ as the strongest peak;

for example, said crystalline form C of the compound of formula I, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $11.51\pm0.2^\circ$, $18.01\pm0.2^\circ$, $22.56\pm0.2^\circ$, $25.68\pm0.2^\circ$, $12.88\pm0.2^\circ$, $15.05\pm0.2^\circ$, $20.85\pm0.2^\circ$, $21.68\pm0.2^\circ$, and $23.11\pm0.2^\circ$;

preferably, said crystalline form C of the compound of formula I, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $9.022\pm0.2^\circ$, $10.212\pm0.2^\circ$, $16.163\pm0.2^\circ$, $16.96\pm0.2^\circ$, $17.22\pm0.2^\circ$, $26.937\pm0.2^\circ$, $29.445\pm0.2^\circ$, $30.137\pm0.2^\circ$, and $30.354\pm0.2^\circ$;

(4) said crystalline form A of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $6.57\pm0.2^\circ$, $6.89\pm0.2^\circ$, $9.96\pm0.2^\circ$, $10.48\pm0.2^\circ$, $10.64\pm0.2^\circ$, $14.69\pm0.2^\circ$, and $14.90\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $4.61\pm0.2^\circ$, $5.02\pm0.2^\circ$, $9.19\pm0.2^\circ$, $13.76\pm0.2^\circ$, $6.57\pm0.2^\circ$, $6.89\pm0.2^\circ$, $9.96\pm0.2^\circ$, $10.48\pm0.2^\circ$, and $10.64\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $18.34\pm0.2^\circ$, $19.13\pm0.2^\circ$, $19.30\pm0.2^\circ$, $20.81\pm0.2^\circ$, $21.68\pm0.2^\circ$, $23.09\pm0.2^\circ$, $24.03\pm0.2^\circ$, $26.13\pm0.2^\circ$, and $28.17\pm0.2^\circ$;

(5) said crystalline form B of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $17.37\pm0.2^\circ$, $18.21\pm0.2^\circ$, $19.44\pm0.2^\circ$, $20.17\pm0.2^\circ$, $20.82\pm0.2^\circ$, $22.41\pm0.2^\circ$, and $24.58\pm0.2^\circ$;

for example, said crystalline form B of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.47\pm0.2^\circ$, $7.19\pm0.2^\circ$, $9.11\pm0.2^\circ$, $11.63\pm0.2^\circ$, $14.45\pm0.2^\circ$, $17.37\pm0.2^\circ$, $18.21\pm0.2^\circ$, $19.44\pm0.2^\circ$, $20.17\pm0.2^\circ$, and $20.82\pm0.2^\circ$;

preferably, said crystalline form B of the compound of formula III, its X-ray powder diffraction

pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $12.94\pm0.2^\circ$, $13.41\pm0.2^\circ$, $15.41\pm0.2^\circ$, $15.75\pm0.2^\circ$, $16.23\pm0.2^\circ$, $16.72\pm0.2^\circ$, $26.01\pm0.2^\circ$, $26.17\pm0.2^\circ$, $26.47\pm0.2^\circ$, $27.96\pm0.2^\circ$, $28.62\pm0.2^\circ$, $30.04\pm0.2^\circ$, and $30.37\pm0.2^\circ$;

(6) said crystalline form C of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $18.16\pm0.2^\circ$, $18.29\pm0.2^\circ$, $19.39\pm0.2^\circ$, $20.81\pm0.2^\circ$, $21.75\pm0.2^\circ$, $26.41\pm0.2^\circ$, and $27.48\pm0.2^\circ$;

for example, said crystalline form C of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $9.84\pm0.2^\circ$, $10.51\pm0.2^\circ$, $12.19\pm0.2^\circ$, $13.39\pm0.2^\circ$, $14.40\pm0.2^\circ$, $18.16\pm0.2^\circ$, $18.29\pm0.2^\circ$, $19.39\pm0.2^\circ$, $20.81\pm0.2^\circ$, $21.75\pm0.2^\circ$, and $26.41\pm0.2^\circ$;

preferably, said crystalline form C of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $11.61\pm0.2^\circ$, $20.02\pm0.2^\circ$, $22.51\pm0.2^\circ$, $22.90\pm0.2^\circ$, $23.25\pm0.2^\circ$, $23.49\pm0.2^\circ$, $24.46\pm0.2^\circ$, $25.11\pm0.2^\circ$, $26.94\pm0.2^\circ$, $28.37\pm0.2^\circ$, $28.97\pm0.2^\circ$, and $29.32\pm0.2^\circ$;

(7) said crystalline form A of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $11.45\pm0.2^\circ$, $17.44\pm0.2^\circ$, $21.45\pm0.2^\circ$, $21.78\pm0.2^\circ$, $24.82\pm0.2^\circ$, $25.95\pm0.2^\circ$, and $26.68\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.14\pm0.2^\circ$, $9.01\pm0.2^\circ$, $15.03\pm0.2^\circ$, $11.45\pm0.2^\circ$, $17.44\pm0.2^\circ$, $21.45\pm0.2^\circ$, $21.78\pm0.2^\circ$, $24.82\pm0.2^\circ$, and $25.95\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $6.11\pm0.2^\circ$, $6.60\pm0.2^\circ$, $7.35\pm0.2^\circ$, $8.63\pm0.2^\circ$, $14.62\pm0.2^\circ$, $16.45\pm0.2^\circ$, $20.40\pm0.2^\circ$, $23.81\pm0.2^\circ$, $25.47\pm0.2^\circ$, $27.96\pm0.2^\circ$, and $28.43\pm0.2^\circ$;

(8) said crystalline form B of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $13.86\pm0.2^\circ$, $14.11\pm0.2^\circ$, $14.71\pm0.2^\circ$, $15.44\pm0.2^\circ$, $16.07\pm0.2^\circ$, $23.18\pm0.2^\circ$, $24.18\pm0.2^\circ$, and $25.17\pm0.2^\circ$;

for example, said crystalline form B of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $7.11\pm0.2^\circ$, $7.57\pm0.2^\circ$, $7.72\pm0.2^\circ$, $8.10\pm0.2^\circ$, $13.86\pm0.2^\circ$, $14.11\pm0.2^\circ$, $14.71\pm0.2^\circ$, $23.18\pm0.2^\circ$, $24.18\pm0.2^\circ$, and $25.17\pm0.2^\circ$;

preferably, said crystalline form B of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $10.27\pm0.2^\circ$, $12.55\pm0.2^\circ$, $12.67\pm0.2^\circ$, $13.19\pm0.2^\circ$, $16.73\pm0.2^\circ$, $17.92\pm0.2^\circ$, $18.38\pm0.2^\circ$, $18.58\pm0.2^\circ$, $20.18\pm0.2^\circ$, $21.2\pm0.2^\circ$, $22.2\pm0.2^\circ$, $25.58\pm0.2^\circ$, and $27.82\pm0.2^\circ$;

(9) said crystalline form A of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $7.97\pm0.2^\circ$, $8.96\pm0.2^\circ$, $17.93\pm0.2^\circ$, $18.39\pm0.2^\circ$, $21.99\pm0.2^\circ$, and $27.73\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.15\pm0.2^\circ$, $8.34\pm0.2^\circ$, $15.51\pm0.2^\circ$, $15.88\pm0.2^\circ$, $8.96\pm0.2^\circ$, $17.93\pm0.2^\circ$, $18.39\pm0.2^\circ$, $21.99\pm0.2^\circ$, and $27.73\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $5.98\pm0.2^\circ$, $8.96\pm0.2^\circ$, $10.49\pm0.2^\circ$, $10.80\pm0.2^\circ$, $11.34\pm0.2^\circ$, $11.66\pm0.2^\circ$, $11.99\pm0.2^\circ$, $12.73\pm0.2^\circ$, $13.15\pm0.2^\circ$, $18.68\pm0.2^\circ$, $19.73\pm0.2^\circ$, $20.14\pm0.2^\circ$, $22.76\pm0.2^\circ$, and $23.24\pm0.2^\circ$;

(10) said crystalline form B of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $8.32\pm0.2^\circ$, $10.26\pm0.2^\circ$, $13.93\pm0.2^\circ$, $14.67\pm0.2^\circ$, $20.35\pm0.2^\circ$, $20.49\pm0.2^\circ$, $21.15\pm0.2^\circ$, and $21.65\pm0.2^\circ$;

for example, said crystalline form B of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.83\pm0.2^\circ$, $12.89\pm0.2^\circ$, $14.44\pm0.2^\circ$, $15.91\pm0.2^\circ$, $25.08\pm0.2^\circ$, $25.50\pm0.2^\circ$, $8.32\pm0.2^\circ$, $20.35\pm0.2^\circ$, and $20.49\pm0.2^\circ$;

preferably, said crystalline form B of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at

one or more of the following positions: $8.52\pm0.2^\circ$, $9.17\pm0.2^\circ$, $13.46\pm0.2^\circ$, $16.25\pm0.2^\circ$, and $17.94\pm0.2^\circ$;

(11) said crystalline form C of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $15.68\pm0.2^\circ$, $15.87\pm0.2^\circ$, $18.01\pm0.2^\circ$, $22.55\pm0.2^\circ$, and $25.67\pm0.2^\circ$;

for example, in said crystalline form C of the compound of formula V, the relative intensity of the diffraction peak at $11.51\pm0.2^\circ$ in its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, is 80%-100%, preferably the diffraction peak at $11.51\pm0.2^\circ$ is the strongest peak;

for example, said crystalline form C of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.19\pm0.2^\circ$, $8.39\pm0.2^\circ$, $10.47\pm0.2^\circ$, $11.51\pm0.2^\circ$, $11.67\pm0.2^\circ$, $15.68\pm0.2^\circ$, $15.87\pm0.2^\circ$, $18.01\pm0.2^\circ$, and $22.55\pm0.2^\circ$;

preferably, said crystalline form C of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $11.67\pm0.2^\circ$, $12.73\pm0.2^\circ$, $12.89\pm0.2^\circ$, $14.59\pm0.2^\circ$, $15.05\pm0.2^\circ$, $18.76\pm0.2^\circ$, $19.81\pm0.2^\circ$, $20.48\pm0.2^\circ$, $20.84\pm0.2^\circ$, $21.68\pm0.2^\circ$, $22.10\pm0.2^\circ$, $24.80\pm0.2^\circ$, and $25.27\pm0.2^\circ$;

(12) said crystalline form A of the compound of formula VI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $6.96\pm0.2^\circ$, $8.60\pm0.2^\circ$, $14.84\pm0.2^\circ$, $15.39\pm0.2^\circ$, $16.63\pm0.2^\circ$, and $17.08\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula VI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $10.58\pm0.2^\circ$, $12.16\pm0.2^\circ$, $13.98\pm0.2^\circ$, $6.96\pm0.2^\circ$, $8.60\pm0.2^\circ$, $14.84\pm0.2^\circ$, $15.39\pm0.2^\circ$, $16.63\pm0.2^\circ$, and $17.08\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula VI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $6.73\pm0.2^\circ$, $9.38\pm0.2^\circ$, $10.28\pm0.2^\circ$, $13.45\pm0.2^\circ$, $19.28\pm0.2^\circ$, and $18.67\pm0.2^\circ$;

(13) said crystalline form A of the compound of formula VII, its X-ray powder diffraction

pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $5.13\pm0.2^\circ$, $6.73\pm0.2^\circ$, $8.61\pm0.2^\circ$, $17.74\pm0.2^\circ$, $24.43\pm0.2^\circ$, and $25.32\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula VII, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $9.00\pm0.2^\circ$, $17.44\pm0.2^\circ$, $21.74\pm0.2^\circ$, $5.13\pm0.2^\circ$, $6.73\pm0.2^\circ$, $8.61\pm0.2^\circ$, $17.74\pm0.2^\circ$, $24.43\pm0.2^\circ$, and $25.32\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula VII, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $15.55\pm0.2^\circ$, $10.05\pm0.2^\circ$, $11.57\pm0.2^\circ$, $22.37\pm0.2^\circ$, and $26.36\pm0.2^\circ$;

(14) said crystalline form A of the compound of formula VIII, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $9.06\pm0.2^\circ$, $9.72\pm0.2^\circ$, $11.10\pm0.2^\circ$, $15.91\pm0.2^\circ$, $16.55\pm0.2^\circ$, $18.00\pm0.2^\circ$, $18.27\pm0.2^\circ$, and $19.16\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula VIII, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $7.66\pm0.2^\circ$, $10.09\pm0.2^\circ$, $11.92\pm0.2^\circ$, $13.91\pm0.2^\circ$, $9.06\pm0.2^\circ$, $9.72\pm0.2^\circ$, $11.10\pm0.2^\circ$, $15.91\pm0.2^\circ$, and $16.55\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula VIII, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $20.35\pm0.2^\circ$, $20.89\pm0.2^\circ$, $21.10\pm0.2^\circ$, $21.68\pm0.2^\circ$, $22.27\pm0.2^\circ$, $22.49\pm0.2^\circ$, $22.97\pm0.2^\circ$, $23.50\pm0.2^\circ$, $24.84\pm0.2^\circ$, $25.52\pm0.2^\circ$, and $29.36\pm0.2^\circ$;

(15) said crystalline form A of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $10.57\pm0.2^\circ$, $12.34\pm0.2^\circ$, $12.77\pm0.2^\circ$, $14.06\pm0.2^\circ$, $14.40\pm0.2^\circ$, $14.97\pm0.2^\circ$, $19.88\pm0.2^\circ$, $22.64\pm0.2^\circ$, $23.18\pm0.2^\circ$, and $24.22\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.96\pm0.2^\circ$, $16.71\pm0.2^\circ$, $17.79\pm0.2^\circ$, $20.33\pm0.2^\circ$, $24.66\pm0.2^\circ$, $10.57\pm0.2^\circ$, $12.34\pm0.2^\circ$, $12.77\pm0.2^\circ$, $14.06\pm0.2^\circ$, $14.40\pm0.2^\circ$, and $14.97\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $9.93\pm0.2^\circ$, $10.13\pm0.2^\circ$, $11.03\pm0.2^\circ$, $11.88\pm0.2^\circ$, $17.56\pm0.2^\circ$, $18.41\pm0.2^\circ$, $18.70\pm0.2^\circ$, $20.68\pm0.2^\circ$, $22.11\pm0.2^\circ$, $25.43\pm0.2^\circ$, $25.98\pm0.2^\circ$, $27.35\pm0.2^\circ$, $27.52\pm0.2^\circ$, $28.08\pm0.2^\circ$, and $29.17\pm0.2^\circ$;

(16) said crystalline form B of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $8.20\pm0.2^\circ$, $10.13\pm0.2^\circ$, $11.12\pm0.2^\circ$, $13.18\pm0.2^\circ$, $16.13\pm0.2^\circ$, $18.67\pm0.2^\circ$, $19.47\pm0.2^\circ$, and $19.85\pm0.2^\circ$;

for example, said crystalline form B of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.34\pm0.2^\circ$, $9.34\pm0.2^\circ$, $9.92\pm0.2^\circ$, $12.84\pm0.2^\circ$, $16.61\pm0.2^\circ$, $8.20\pm0.2^\circ$, $10.13\pm0.2^\circ$, $13.18\pm0.2^\circ$, and $16.13\pm0.2^\circ$;

preferably, said crystalline form B of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $11.47\pm0.2^\circ$, $16.36\pm0.2^\circ$, $18.60\pm0.2^\circ$, and $21.53\pm0.2^\circ$;

(17) said crystalline form A of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $11.43\pm0.2^\circ$, $13.53\pm0.2^\circ$, $17.24\pm0.2^\circ$, $18.49\pm0.2^\circ$, $18.77\pm0.2^\circ$, $21.08\pm0.2^\circ$, $24.57\pm0.2^\circ$, and $28.44\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $7.46\pm0.2^\circ$, $12.01\pm0.2^\circ$, $21.70\pm0.2^\circ$, $11.43\pm0.2^\circ$, $13.53\pm0.2^\circ$, $17.24\pm0.2^\circ$, $18.49\pm0.2^\circ$, $18.77\pm0.2^\circ$, and $21.08\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $5.71\pm0.2^\circ$, $13.93\pm0.2^\circ$, $14.91\pm0.2^\circ$, $15.34\pm0.2^\circ$, $22.12\pm0.2^\circ$, $22.80\pm0.2^\circ$, $22.95\pm0.2^\circ$, $24.99\pm0.2^\circ$, and $26.08\pm0.2^\circ$;

(18) said crystalline form B of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $11.98\pm0.2^\circ$, $18.4\pm0.2^\circ$, $21.27\pm0.2^\circ$, $23.27\pm0.2^\circ$, $27.26\pm0.2^\circ$, and $27.77\pm0.2^\circ$;

for example, said crystalline form B of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $8.96\pm0.2^\circ$, $10.81\pm0.2^\circ$, $17.92\pm0.2^\circ$, $22.07\pm0.2^\circ$, $11.98\pm0.2^\circ$, $18.4\pm0.2^\circ$, $21.27\pm0.2^\circ$, and $23.27\pm0.2^\circ$;

preferably, said crystalline form B of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $5.95\pm0.2^\circ$, $14.09\pm0.2^\circ$, $15.80\pm0.2^\circ$, $16.97\pm0.2^\circ$, $20.03\pm0.2^\circ$, $22.42\pm0.2^\circ$, and $23.79\pm0.2^\circ$;

(19) said crystalline form C of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $6.36\pm0.2^\circ$, $12.82\pm0.2^\circ$, $17.07\pm0.2^\circ$, $17.48\pm0.2^\circ$, $18.61\pm0.2^\circ$, $20.24\pm0.2^\circ$, and $20.94\pm0.2^\circ$;

for example, said crystalline form C of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has a relative intensity of 50%-100% for the diffraction peak at $22.61\pm0.2^\circ$, preferably the diffraction peak at $22.61\pm0.2^\circ$ is the strongest peak;

for example, said crystalline form C of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $11.30\pm0.2^\circ$, $22.61\pm0.2^\circ$, $24.69\pm0.2^\circ$, $26.16\pm0.2^\circ$, $28.57\pm0.2^\circ$, $6.36\pm0.2^\circ$, $18.61\pm0.2^\circ$, $12.82\pm0.2^\circ$, and $17.07\pm0.2^\circ$;

preferably, said crystalline form C of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $12.11\pm0.2^\circ$ and $15.12\pm0.2^\circ$;

(20) said crystalline form D of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $15.77\pm0.2^\circ$, $16.88\pm0.2^\circ$, $19.82\pm0.2^\circ$, $21.76\pm0.2^\circ$, $23.62\pm0.2^\circ$, $24.95\pm0.2^\circ$, and $27.32\pm0.2^\circ$;

for example, said crystalline form D of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $10.68\pm0.2^\circ$, $21.44\pm0.2^\circ$, $15.77\pm0.2^\circ$, $16.88\pm0.2^\circ$, $19.82\pm0.2^\circ$, $21.76\pm0.2^\circ$, $23.62\pm0.2^\circ$, and $24.95\pm0.2^\circ$;

preferably, said crystalline form D of the compound of formula X, its X-ray powder diffraction

pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $7.27\pm0.2^\circ$, $7.87\pm0.2^\circ$, $13.59\pm0.2^\circ$, $14.54\pm0.2^\circ$, $16.25\pm0.2^\circ$, $17.29\pm0.2^\circ$, $17.66\pm0.2^\circ$, $19.63\pm0.2^\circ$, $20.27\pm0.2^\circ$, $22.62\pm0.2^\circ$, $23.09\pm0.2^\circ$, $24.33\pm0.2^\circ$, $24.64\pm0.2^\circ$, $26.37\pm0.2^\circ$, and $28.88\pm0.2^\circ$;

(21) said crystalline form A of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $10.67\pm0.2^\circ$, $18.82\pm0.2^\circ$, $19.49\pm0.2^\circ$, and $23.58\pm0.2^\circ$;

for example, said crystalline form A of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $12.78\pm0.2^\circ$, $14.09\pm0.2^\circ$, $14.98\pm0.2^\circ$, $10.67\pm0.2^\circ$, $18.82\pm0.2^\circ$, $19.49\pm0.2^\circ$, $23.58\pm0.2^\circ$, and $21.80\pm0.2^\circ$;

preferably, said crystalline form A of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following positions: $6.9\pm0.2^\circ$, $9.39\pm0.2^\circ$, $17.22\pm0.2^\circ$, $19.35\pm0.2^\circ$, $21.80\pm0.2^\circ$, $22.45\pm0.2^\circ$, $24.15\pm0.2^\circ$, and $27.926\pm0.2^\circ$;

(22) said crystalline form B of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $9.31\pm0.2^\circ$, $12.92\pm0.2^\circ$, $13.62\pm0.2^\circ$, $24.17\pm0.2^\circ$, and $27.37\pm0.2^\circ$;

for example, said crystalline form B of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $8.22\pm0.2^\circ$, $19.38\pm0.2^\circ$, $20.38\pm0.2^\circ$, $23.43\pm0.2^\circ$, $24.74\pm0.2^\circ$, $9.31\pm0.2^\circ$, $12.92\pm0.2^\circ$, and $24.17\pm0.2^\circ$;

preferably, said crystalline form B of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $15.13\pm0.2^\circ$, $16.29\pm0.2^\circ$, $17.63\pm0.2^\circ$, $17.77\pm0.2^\circ$, $18.12\pm0.2^\circ$, $18.63\pm0.2^\circ$, $21.04\pm0.2^\circ$, $21.28\pm0.2^\circ$, and $26.46\pm0.2^\circ$;

(23) said crystalline form C of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $9.21\pm0.2^\circ$, $16.11\pm0.2^\circ$, $16.57\pm0.2^\circ$, $19.39\pm0.2^\circ$, and $20.01\pm0.2^\circ$;

for example, said crystalline form C of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has a diffraction peak at $19.82\pm0.2^\circ$ with a relative intensity of 60%-100%, preferably a relative intensity of

70%-80%, for example 71.2%;

for example, said crystalline form C of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $5.23\pm0.2^\circ$, $19.82\pm0.2^\circ$, $9.21\pm0.2^\circ$, $16.11\pm0.2^\circ$, $16.57\pm0.2^\circ$, $19.39\pm0.2^\circ$, $9.80\pm0.2^\circ$, $11.12\pm0.2^\circ$, and $20.01\pm0.2^\circ$;

preferably, said crystalline form C of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $8.07\pm0.2^\circ$, $8.26\pm0.2^\circ$, $9.80\pm0.2^\circ$, $11.12\pm0.2^\circ$, $12.72\pm0.2^\circ$, $15.24\pm0.2^\circ$, $15.48\pm0.2^\circ$, $18.20\pm0.2^\circ$, $18.71\pm0.2^\circ$, $21.52\pm0.2^\circ$, $23.51\pm0.2^\circ$, and $23.83\pm0.2^\circ$;

(24) said crystalline form D of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $8.52\pm0.2^\circ$, $12.18\pm0.2^\circ$, $13.12\pm0.2^\circ$, $16.60\pm0.2^\circ$, $20.07\pm0.2^\circ$, and $24.00\pm0.2^\circ$;

for example, said crystalline form D of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has a diffraction peak at $6.13\pm0.2^\circ$ with a relative intensity of 80%-100%, preferably the diffraction peak at $6.13\pm0.2^\circ$ is the strongest peak;

for example, said crystalline form D of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.13\pm0.2^\circ$, $10.87\pm0.2^\circ$, $18.38\pm0.2^\circ$, $8.52\pm0.2^\circ$, $12.18\pm0.2^\circ$, $13.12\pm0.2^\circ$, $16.60\pm0.2^\circ$, $20.07\pm0.2^\circ$, and $24.00\pm0.2^\circ$;

preferably, said crystalline form D of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $14.25\pm0.2^\circ$, $14.42\pm0.2^\circ$, $17.00\pm0.2^\circ$, $19.09\pm0.2^\circ$, $19.34\pm0.2^\circ$, $21.12\pm0.2^\circ$, $21.28\pm0.2^\circ$, $21.59\pm0.2^\circ$, $21.72\pm0.2^\circ$, $23.14\pm0.2^\circ$, and $24.27\pm0.2^\circ$;

(25) said crystalline form E of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $13.85\pm0.2^\circ$, $14.30\pm0.2^\circ$, $17.74\pm0.2^\circ$, $19.04\pm0.2^\circ$, $21.93\pm0.2^\circ$, $22.25\pm0.2^\circ$, $23.08\pm0.2^\circ$, and $23.43\pm0.2^\circ$;

for example, said crystalline form E of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks

at $5.66\pm0.2^\circ$, $11.50\pm0.2^\circ$, $17.97\pm0.2^\circ$, $19.83\pm0.2^\circ$, $23.95\pm0.2^\circ$, $13.85\pm0.2^\circ$, $14.30\pm0.2^\circ$, $17.74\pm0.2^\circ$, $19.04\pm0.2^\circ$, and $21.93\pm0.2^\circ$;

preferably, said crystalline form E of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $8.45\pm0.2^\circ$, $9.02\pm0.2^\circ$, $9.84\pm0.2^\circ$, $11.19\pm0.2^\circ$, $12.70\pm0.2^\circ$, $15.59\pm0.2^\circ$, $16.56\pm0.2^\circ$, $16.93\pm0.2^\circ$, $18.75\pm0.2^\circ$, $20.55\pm0.2^\circ$, $25.05\pm0.2^\circ$, and $26.98\pm0.2^\circ$;

(26) said crystalline form F of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $10.34\pm0.2^\circ$, $15.73\pm0.2^\circ$, $16.34\pm0.2^\circ$, $20.42\pm0.2^\circ$, $20.72\pm0.2^\circ$, $24.34\pm0.2^\circ$, and $25.74\pm0.2^\circ$;

for example, said crystalline form F of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $6.15\pm0.2^\circ$, $16.55\pm0.2^\circ$, $17.30\pm0.2^\circ$, $21.48\pm0.2^\circ$, $10.34\pm0.2^\circ$, $15.73\pm0.2^\circ$, $16.34\pm0.2^\circ$, $20.42\pm0.2^\circ$, and $20.72\pm0.2^\circ$;

preferably, said crystalline form F of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $6.99\pm0.2^\circ$, $11.85\pm0.2^\circ$, $12.27\pm0.2^\circ$, $12.57\pm0.2^\circ$, $13.97\pm0.2^\circ$, $18.42\pm0.2^\circ$, $20.98\pm0.2^\circ$, $21.22\pm0.2^\circ$, $21.83\pm0.2^\circ$, $21.97\pm0.2^\circ$, $26.02\pm0.2^\circ$, and $26.83\pm0.2^\circ$;

(27) crystalline form G of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $23.83\pm0.2^\circ$, $25.37\pm0.2^\circ$, $25.63\pm0.2^\circ$, and $27.60\pm0.2^\circ$;

for example, crystalline form G of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks at $7.65\pm0.2^\circ$, $13.76\pm0.2^\circ$, $20.35\pm0.2^\circ$, $21.01\pm0.2^\circ$, $21.57\pm0.2^\circ$, $23.83\pm0.2^\circ$, $25.37\pm0.2^\circ$, $25.63\pm0.2^\circ$, and $27.60\pm0.2^\circ$;

preferably, crystalline form G of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $11.87\pm0.2^\circ$, $12.60\pm0.2^\circ$, $12.76\pm0.2^\circ$, $16.07\pm0.2^\circ$, $20.05\pm0.2^\circ$, $22.75\pm0.2^\circ$, and $23.83\pm0.2^\circ$;

(28) said crystalline form H of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: $10.39\pm0.2^\circ$, $11.07\pm0.2^\circ$, $11.60\pm0.2^\circ$, $12.56\pm0.2^\circ$, $17.07\pm0.2^\circ$,

19.09±0.2°, 21.12±0.2°, 21.46±0.2°, 25.23±0.2°, and 27.27±0.2°;

for example, said crystalline form H of the compound of formula IX, its X-ray powder diffraction pattern using Cu-Kα radiation, expressed in terms of 2θ angle, has diffraction peaks at 8.27±0.2°, 9.70±0.2°, 16.55±0.2°, 19.78±0.2°, 11.07±0.2°, 12.56±0.2°, 17.07±0.2°, 19.09±0.2°, and 21.46±0.2°;

preferably, said crystalline form H of the compound of formula IX, its X-ray powder diffraction pattern using Cu-Kα radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: 5.21±0.2°, 11.60±0.2°, 15.29±0.2°, 18.79±0.2°, and 23.36±0.2°;

(29) said crystalline form I of the compound of formula IX, its X-ray powder diffraction pattern using Cu-Kα radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: 14.20±0.2°, 16.67±0.2°, 18.00±0.2°, 18.74±0.2°, 20.07±0.2°, and 24.06±0.2°;

for example, said crystalline form I of the compound of formula IX, its X-ray powder diffraction pattern using Cu-Kα radiation, expressed in terms of 2θ angle, has a relative intensity of 80%-100% for the diffraction peak at 6.01±0.2°, preferably the diffraction peak at 6.01±0.2° is the strongest peak;

for example, said crystalline form I of the compound of formula IX, its X-ray powder diffraction pattern using Cu-Kα radiation, expressed in terms of 2θ angle, has diffraction peaks at 6.01±0.2°, 10.63±0.2°, 12.79±0.2°, 14.20±0.2°, 16.67±0.2°, 18.00±0.2°, 20.07±0.2°, and 24.06±0.2°;

preferably, said crystalline form I of the compound of formula IX, its X-ray powder diffraction pattern using Cu-Kα radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: 11.99±0.2°, 17.84±0.2°, and 23.22±0.2°;

(30) said crystalline form J of the compound of formula IX, its X-ray powder diffraction pattern using Cu-Kα radiation, expressed in terms of 2θ angle, further has diffraction peaks at one or more of the following: 15.30±0.2°, 16.41±0.2°, 18.79±0.2°, 19.07±0.2°, 19.66±0.2°, and 21.25±0.2°.

5. The crystalline form C of the compound of formula I or said nitrogen-containing fused-ring compound according to claim 3, characterized in that it satisfies one or more of the following conditions:

(1) said crystalline form A of the compound of formula II, its X-ray powder diffraction pattern

using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 1;

table 1

peak	diffraction Angle (°, 2 θ)	relative Intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.98	18.40%	27	24.6	13.90%
2	9.00	68.00%	28	25.35	3.60%
3	10.5	6.10%	29	25.62	8.20%
4	10.84	62.30%	30	26.41	1.60%
5	11.94	28.40%	31	27.17	6.30%
6	13.46	4.10%	32	27.72	63.30%
7	14.16	8.50%	33	28.50	7.20%
8	14.72	8.70%	34	28.96	12.50%
9	15.31	1.40%	35	29.18	6.80%
10	15.46	1.60%	36	29.68	15.80%
11	15.79	1.20%	37	30.29	9.40%
12	17.15	2.20%	38	31.08	5.90%
13	17.91	100.00%	39	31.99	7.20%
14	18.36	40.90%	40	32.22	7.00%
15	19.02	2.50%	41	32.57	6.60%
16	19.99	12.90%	42	32.73	5.30%
17	20.65	8.90%	43	33.61	1.40%
18	21.30	40.50%	44	34.17	3.90%
19	21.71	18.00%	45	34.63	0.70%
20	22.05	77.00%	46	35.46	0.50%
21	22.45	23.80%	47	35.82	1.70%
22	22.63	14.80%	48	36.41	3.40%
23	23.29	23.90%	49	37.53	4.00%
24	23.68	4.60%	50	37.96	3.90%
25	23.96	8.40%	51	38.63	7.00%
26	24.14	7.70%	52	39.67	1.50%

;

preferably, said crystalline form A of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 1;

(2) said crystalline form A of the compound of formula II, its differential scanning calorimetry curve has endothermic peaks at peak temperatures of 98.33 $\pm$ 3°C and 206.94 $\pm$ 3°C;

preferably, the differential scanning calorimetry curve of said crystalline form A of the compound of formula II is substantially as shown in figure 2;

(3) said crystalline form A of the compound of formula II, its thermogravimetric analysis curve shows a weight loss of about 0.48% in the temperature range from $34.7\pm 3^{\circ}\text{C}$ to $70.0\pm 3^{\circ}\text{C}$, a weight loss of about 2.11% in the temperature range from $70.0\pm 3^{\circ}\text{C}$ to $95.0\pm 3^{\circ}\text{C}$, a weight loss of about 1.38% in the temperature range from $95.0\pm 3^{\circ}\text{C}$ to $140.0\pm 3^{\circ}\text{C}$, and a weight loss of about 0.34% in the temperature range from $140.0\pm 3^{\circ}\text{C}$ to $220.0\pm 3^{\circ}\text{C}$;

preferably, the thermogravimetric analysis curve of said crystalline form A of the compound of formula II is substantially as shown in figure 3;

(4) in said crystalline form A of the compound of formula II, x' is water, and q is 1;

(5) said crystalline form B of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks as shown in table 2;

table 2

peak	diffraction angle ($^{\circ}$, 2θ)	relative intensity	peak	diffraction angle ($^{\circ}$, 2θ)	relative intensity
1	8.10	23.20%	25	23.32	4.00%
2	8.37	37.80%	26	23.92	18.60%
3	10.22	9.10%	27	24.20	13.60%
4	11.46	3.80%	28	24.4	65.40%
5	11.83	2.40%	29	24.83	100.00%
6	13.33	26.90%	30	25.42	16.90%
7	13.94	41.50%	31	25.70	2.90%
8	14.26	10.40%	32	26.24	2.80%
9	14.79	17.30%	33	26.82	4.20%
10	15.67	42.90%	34	27.43	7.20%
11	16.61	5.90%	35	27.60	9.30%
12	17.63	17.40%	36	28.04	10.20%
13	17.91	6.40%	37	28.67	14.60%
14	18.70	5.20%	38	29.01	7.00%
15	19.13	9.10%	39	30.1	11.20%
16	19.62	5.40%	40	31.02	10.90%
17	20.03	9.80%	41	32.10	6.70%
18	20.72	53.70%	42	32.72	5.60%
19	21.15	30.20%	43	33.49	2.70%
20	21.65	2.20%	44	33.70	2.60%

21	21.84	6.00%	45	34.38	7.30%
22	22.14	54.20%	46	34.82	10.80%
23	22.36	4.40%	47	36.33	1.60%
24	23.02	3.30%	48	38.57	6.30%

;

preferably, said crystalline form B of the compound of formula II, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 4;

(6) in said crystalline form B of the compound of formula II, x' is acetone, and q is 0.56, 0.5, or 0.6, for example, q is 0.56;

(7) said crystalline form C of the compound of formula I, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 3;

table 3

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	9.02	16.10%	26	28.10	2.40%
2	10.21	11.40%	27	28.60	2.30%
3	11.25	3.90%	28	29.04	4.90%
4	11.51	100.00%	29	29.45	24.40%
5	12.88	28.70%	30	29.49	14.70%
6	13.61	6.60%	31	30.14	13.50%
7	14.58	14.20%	32	30.35	12.90%
8	15.05	22.80%	33	30.68	2.00%
9	16.16	5.70%	34	31.26	1.80%
10	16.96	7.10%	35	31.72	1.70%
11	17.22	5.40%	36	32.56	1.90%
12	18.01	59.10%	37	33.13	1.90%
13	18.91	12.10%	38	34.12	4.40%
14	19.94	1.60%	39	34.87	2.40%
15	20.48	26.60%	40	34.97	8.80%
16	20.85	42.00%	41	35.48	5.60%
17	21.68	32.30%	42	36.05	1.90%
18	22.56	98.50%	43	36.42	2.30%
19	23.11	29.20%	44	36.89	1.80%
20	24.79	31.90%	45	37.06	1.90%
21	25.68	84.80%	46	38.06	2.70%
22	26.21	5.00%	47	38.40	3.10%
23	26.94	15.00%	48	39.22	2.80%

24	27.26	11.20%	49	39.73	2.00%
25	27.44	35.40%			

;

preferably, said crystalline form C of the compound of formula I, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 5;

(8) said crystalline form C of the compound of formula I, its differential scanning calorimetry curve has an endothermic peak at a peak temperature of 238.92 $\pm$ 3 $^{\circ}$ C;

preferably, the differential scanning calorimetry curve of said crystalline form C of the compound of formula I is substantially as shown in figure 6;

(9) said crystalline form C of the compound of formula I, its thermogravimetric analysis curve shows a weight loss of about 0.81% in the temperature range from 35.56 $\pm$ 3 $^{\circ}$ C to 230.00 $\pm$ 3 $^{\circ}$ C;

preferably, the thermogravimetric analysis curve of said crystalline form C of the compound of formula I is substantially as shown in figure 7;

(10) said crystalline form C of the compound of formula I is solvent-free;

(11) said crystalline form A of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 4;

table 4

peak	diffraction angle ($^{\circ}$, 2 θ)	relative intensity	peak	diffraction angle ($^{\circ}$, 2 θ)	relative intensity
1	4.61	100.00%	18	19.13	6.10%
2	5.02	34.20%	19	19.30	8.20%
3	6.57	21.10%	20	19.96	2.40%
4	6.89	14.10%	21	20.81	6.40%
5	9.19	70.70%	22	21.68	8.00%
6	9.97	16.20%	23	23.09	5.90%
7	10.48	8.90%	24	24.03	11.90%
8	10.64	8.30%	25	24.50	5.70%
9	11.12	2.40%	26	25.14	4.20%
10	12.19	2.00%	27	25.56	7.00%
11	13.76	62.30%	28	26.13	14.40%
12	14.69	9.60%	29	27.54	1.40%
13	14.90	14.80%	30	28.17	7.20%
14	15.95	1.80%	31	31.80	3.10%
15	17.09	1.20%	32	34.16	1.90%
16	18.10	4.20%	33	35.40	1.80%

17	18.34	9.10%	34	36.04	2.20%
----	-------	-------	----	-------	-------

;

preferably, said crystalline form A of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 8;

(12) in said crystalline form A of the compound of formula III, w is 1, 1.1, or 1.06, for example 1.06;

(13) in said crystalline form A of the compound of formula III, e is 0 or 1.5;

(14) said crystalline form B of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 5;

table 5

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	6.47	32.50%	34	26.96	13.30%
2	7.19	18.80%	35	27.15	14.10%
3	9.11	13.90%	36	27.47	4.90%
4	11.63	19.40%	37	27.96	40.00%
5	12.94	7.20%	38	28.62	44.60%
6	13.41	8.00%	39	29.00	7.10%
7	14.45	71.00%	40	29.16	8.10%
8	15.41	9.70%	41	29.39	4.40%
9	15.75	10.40%	42	30.04	15.60%
10	16.23	15.50%	43	30.37	29.10%
11	16.43	10.70%	44	30.63	6.10%
12	16.72	13.20%	45	31.00	16.20%
13	17.37	49.40%	46	31.42	5.90%
14	18.21	51.20%	47	31.87	4.20%
15	18.82	3.70%	48	32.20	25.10%
16	19.44	47.20%	49	32.64	11.10%
17	20.17	51.30%	50	32.92	10.10%
18	20.50	23.70%	51	33.64	1.30%
19	20.63	32.20%	52	34.05	13.20%
20	20.82	100.00%	53	34.44	2.00%
21	21.66	4.60%	54	35.03	2.90%
22	22.22	2.30%	55	35.66	5.10%
23	22.41	31.50%	56	36.07	4.60%
24	23.01	12.10%	57	36.29	6.50%

25	23.45	2.80%	58	36.49	5.50%
26	23.63	6.90%	59	37.03	6.20%
27	23.81	2.50%	60	37.34	2.70%
28	24.58	83.80%	61	38.15	3.90%
29	25.33	9.00%	62	38.45	5.20%
30	25.84	20.50%	63	39.01	2.50%
31	26.01	39.10%	64	39.26	5.20%
32	26.17	27.40%	65	39.81	5.90%
33	26.47	47.20%			

;

preferably, said crystalline form B of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 9;

(15) said crystalline form B of the compound of formula III, its differential scanning calorimetry curve has an endothermic peak at a peak temperature of 186.88 $\pm$ 3 $^{\circ}$ C;

preferably, the differential scanning calorimetry curve of said crystalline form B of the compound of formula III is substantially as shown in figure 10;

(16) said crystalline form B of the compound of formula III, its thermogravimetric analysis curve shows a weight loss of about 1.42% in the temperature range from 24.07 $\pm$ 3 $^{\circ}$ C to 120.0 $\pm$ 3 $^{\circ}$ C, and a weight loss of about 9.84% in the temperature range from 120.00 $\pm$ 3 $^{\circ}$ C to 205.00 $\pm$ 3 $^{\circ}$ C;

preferably, the thermogravimetric analysis curve of said crystalline form B of the compound of formula III is substantially as shown in figure 11;

(17) in said crystalline form B of the compound of formula III, w is 1.76, 1.7, or 1.8, for example 1.76;

(18) in said crystalline form B of the compound of formula III, e is 0;

(19) said crystalline form C of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 6;

table 6

peak	diffraction angle ($^{\circ}$, 2 θ)	relative intensity	peak	diffraction angle ($^{\circ}$, 2 θ)	relative intensity
1	9.84	22.30%	21	26.94	17.80%
2	10.51	14.40%	22	27.48	51.00%
3	11.61	12.00%	23	28.37	25.40%
4	12.19	61.90%	24	28.97	25.90%

5	13.39	33.30%	25	29.32	26.90%
6	14.40	24.90%	26	29.95	6.50%
7	17.35	10.30%	27	30.42	15.70%
8	18.16	86.70%	28	31.25	11.50%
9	18.29	78.90%	29	31.60	5.70%
10	19.39	53.40%	30	32.24	11.00%
11	20.02	15.90%	31	32.62	9.60%
12	20.81	37.40%	32	33.45	3.30%
13	21.75	64.40%	33	34.04	6.10%
14	22.51	33.80%	34	34.68	7.70%
15	22.90	13.30%	35	35.30	10.10%
16	23.25	34.10%	36	36.16	13.90%
17	23.49	29.40%	37	36.57	4.00%
18	24.46	12.20%	38	37.90	6.30%
19	25.11	16.90%	39	38.47	7.40%
20	26.41	100.00%			

;

preferably, said crystalline form C of the compound of formula III, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 12;

(20) in said crystalline form C of the compound of formula III, w is 1.7, 1.8, or 1.6, for example 1.7;

(21) said crystalline form A of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 7;

table 7

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.14	60.20%	12	20.40	33.50%
2	6.11	16.10%	13	21.45	33.20%
3	6.60	14.80%	14	21.78	58.50%
4	7.35	20.40%	15	23.81	40.20%
5	8.63	23.70%	16	24.82	67.70%
6	9.01	100.00%	17	25.47	51.50%
7	11.45	39.40%	18	25.95	49.20%
8	14.62	18.30%	19	26.68	54.90%
9	15.03	35.20%	20	27.96	6.00%
10	16.45	17.10%	21	28.43	29.90%
11	17.44	33.50%			

;

preferably, said crystalline form A of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 13;

(22) in said crystalline form A of the compound of formula IV, r is 1, 0.97, or 0.9, for example 0.97;

(23) in said crystalline form A of the compound of formula IV, t is 0;

(24) said crystalline form B of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 8;

table 8

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	6.87	9.90%	35	21.52	8.90%
2	7.11	22.80%	36	21.98	11.20%
3	7.57	24.00%	37	22.20	20.90%
4	7.72	24.70%	38	22.51	14.50%
5	8.10	26.40%	39	22.86	8.00%
6	9.96	5.10%	40	23.18	67.20%
7	10.27	16.20%	41	23.62	11.60%
8	11.20	2.00%	42	24.18	94.90%
9	11.55	2.30%	43	24.74	12.60%
10	11.86	2.80%	44	25.17	100.00%
11	12.55	13.30%	45	25.58	31.60%
12	12.67	14.40%	46	26.00	14.30%
13	13.19	12.00%	47	26.39	4.40%
14	13.49	8.90%	48	26.80	13.60%
15	13.86	27.20%	49	27.16	18.40%
16	14.11	55.00%	50	27.40	14.20%
17	14.71	63.20%	51	27.81	41.70%
18	15.44	21.70%	52	28.09	14.80%
19	16.07	43.00%	53	28.58	5.40%
20	16.45	5.20%	54	29.49	14.30%
21	16.73	18.90%	55	29.89	16.50%
22	17.01	4.60%	56	30.12	13.30%
23	17.55	4.20%	57	31.45	8.00%
24	17.92	19.00%	58	32.04	14.30%
25	18.38	46.30%	59	32.83	4.20%
26	18.58	44.20%	60	33.66	2.50%

27	19.10	8.20%	61	34.52	4.60%
28	19.46	15.30%	62	35.43	2.70%
29	19.80	12.50%	63	35.85	2.10%
30	20.18	28.90%	64	36.35	3.90%
31	20.39	11.90%	65	36.70	7.70%
32	20.59	15.90%	66	38.31	6.40%
33	20.82	11.70%	67	38.66	3.80%
34	21.21	27.10%	68	39.47	4.30%

;

preferably, said crystalline form B of the compound of formula IV, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 14;

(25) said crystalline form B of the compound of formula IV, its differential scanning calorimetry curve has endothermic peaks at peak temperatures of 34.85 $\pm$ 3°C, 90.79 $\pm$ 3°C, and 204.38 $\pm$ 3°C;

preferably, the differential scanning calorimetry curve of said crystalline form B of the compound of formula IV is substantially as shown in figure 15;

(26) said crystalline form B of the compound of formula IV, its thermogravimetric analysis curve shows a weight loss of about 2.06% in the temperature range from 33.44 $\pm$ 3°C to 50.0 $\pm$ 3°C, a weight loss of about 0.56% in the temperature range from 50.0 $\pm$ 3°C to 100.0 $\pm$ 3°C, a weight loss of about 0.39% in the temperature range from 100.00 $\pm$ 3°C to 180.00 $\pm$ 3°C, and a weight loss of about 1% in the temperature range from 180.00 $\pm$ 3°C to 230.00 $\pm$ 3°C;

Preferably, the thermogravimetric analysis curve of said crystalline form B of the compound of formula IV is substantially as shown in figure 16;

(27) in said crystalline form B of the compound of formula IV, r is 1, 0.9, or 0.96, for example 0.96;

(28) in said crystalline form B of the compound of formula IV, t is 0 or 1.7;

preferably, in said crystalline form B of the compound of formula IV, r is 1 and t is 0; or, r is 0.96 and t is 0;

(29) said crystalline form A of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 9;

table 9

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
------	------------------------------------	--------------------	------	------------------------------------	--------------------

1	5.15	24.70%	35	23.24	33.70%
2	5.98	8.10%	36	23.63	23.60%
3	7.97	16.00%	37	23.98	58.60%
4	8.34	44.50%	38	24.1	43.00%
5	8.96	21.40%	39	24.47	7.40%
6	10.49	33.70%	40	24.88	14.30%
7	10.80	26.70%	41	25.13	39.10%
8	11.34	32.40%	42	25.54	5.60%
9	11.66	24.10%	43	25.83	6.90%
10	11.99	21.90%	44	26.18	13.30%
11	12.73	15.30%	45	26.44	10.80%
12	13.15	7.00%	46	26.83	11.90%
13	13.46	-0.10%	47	27.28	20.30%
14	14.07	2.80%	48	27.73	100.00%
15	14.61	1.40%	49	28.44	9.60%
16	14.84	5.00%	50	28.63	11.10%
17	15.51	38.40%	51	28.94	22.30%
18	15.88	44.10%	52	29.17	18.40%
19	16.68	6.50%	53	29.59	5.80%
20	17.26	4.00%	54	30.38	7.70%
21	17.93	75.20%	55	31.27	5.70%
22	18.39	84.80%	56	31.76	8.30%
23	18.68	37.50%	57	32.11	11.50%
24	19.06	11.40%	58	32.72	5.20%
25	19.73	38.50%	59	32.97	4.70%
26	20.14	31.90%	60	33.52	8.90%
27	20.52	12.40%	61	33.93	6.50%
28	20.76	14.20%	62	34.60	3.10%
29	21.05	29.80%	63	35.44	7.30%
30	21.28	26.00%	64	36.13	2.10%
31	21.64	22.90%	65	37.14	4.10%
32	21.99	88.50%	66	37.70	6.90%
33	22.51	13.30%	67	38.63	6.10%
34	22.76	42.90%	68	39.60	2.50%

;

preferably, said crystalline form A of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 17;

(30) said crystalline form A of the compound of formula V, its differential scanning calorimetry curve has endothermic peaks at peak temperatures of 88.96 $\pm$ 3°C and 192.67 $\pm$ 3°C;

preferably, the differential scanning calorimetry curve of said crystalline form A of the compound of formula V is substantially as shown in figure 18;

(31) said crystalline form A of the compound of formula V, its thermogravimetric analysis curve shows a weight loss of about 0.92% in the temperature range from $32.34\pm 3^{\circ}\text{C}$ to $70.0\pm 3^{\circ}\text{C}$, and a weight loss of about 2.1% in the temperature range from $70.0\pm 3^{\circ}\text{C}$ to $180.0\pm 3^{\circ}\text{C}$;

preferably, the thermogravimetric analysis curve of said crystalline form A of the compound of formula V is substantially as shown in figure 19;

(32) in said crystalline form A of the compound of formula V, y is 1.1, 1.2, or 1.14, for example 1.14;

preferably, in said crystalline form A of the compound of formula V, i is 0, y is 1.1, u is 0.9; or, i is 0, y is 1.14, u is 0.91;

(33) said crystalline form B of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks as shown in table 10;

table 10

peak	diffraction angle ($^{\circ}$, 2θ)	relative intensity	peak	diffraction angle ($^{\circ}$, 2θ)	relative intensity
1	3.41	5.60%	25	21.65	22.50%
2	6.83	38.40%	26	22.05	20.10%
3	7.27	6.10%	27	22.31	15.20%
4	8.32	22.40%	28	22.83	13.30%
5	8.52	17.60%	29	23.01	16.40%
6	9.17	12.30%	30	23.56	24.00%
7	10.26	21.70%	31	23.98	25.70%
8	11.72	8.60%	32	24.68	19.70%
9	12.89	100.00%	33	25.08	49.20%
10	13.46	16.40%	34	25.50	59.20%
11	13.76	7.80%	35	25.92	9.70%
12	13.93	20.10%	36	26.58	2.90%
13	14.44	51.20%	37	27.08	9.50%
14	14.67	21.10%	38	27.63	8.30%
15	15.91	47.80%	39	28.15	24.60%
16	16.25	23.60%	40	29.06	17.10%
17	16.61	10.10%	41	31.52	8.10%
18	17.10	7.60%	42	31.96	-3.30%

19	17.94	14.80%	43	32.73	11.80%
20	19.20	3.90%	44	34.25	9.60%
21	20.00	8.00%	45	35.91	7.00%
22	20.35	50.60%	46	37.15	2.40%
23	20.49	48.30%	47	38.73	12.90%
24	21.15	19.40%			

;

preferably, said crystalline form B of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 20;

(34) in said crystalline form B of the compound of formula V, y is 1.1, 1, or 1.09, for example 1.09;

(35) in said crystalline form B of the compound of formula V, u and i are 0;

(36) said crystalline form C of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 11;

table 11

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.19	49.10%	41	24.56	2.70%
2	7.95	4.30%	42	24.80	30.20%
3	8.39	51.00%	43	25.27	28.10%
4	9.03	9.70%	44	25.67	60.90%
5	9.44	0.90%	45	26.00	4.60%
6	10.22	8.60%	46	26.19	18.70%
7	10.47	48.10%	47	26.42	11.90%
8	11.19	16.20%	48	26.94	12.40%
9	11.51	100.00%	49	27.19	13.70%
10	11.67	23.90%	50	27.47	39.90%
11	12.73	21.10%	51	28.11	7.00%
12	12.89	17.90%	52	28.43	2.50%
13	13.14	5.10%	53	28.73	8.90%
14	13.62	3.90%	54	29.08	13.50%
15	14.59	12.80%	55	29.41	33.20%
16	15.05	13.40%	56	30.12	10.90%
17	15.68	33.50%	57	30.33	9.90%
18	15.87	26.00%	58	30.53	3.90%
19	16.16	5.20%	59	31.260	2.00%
20	16.79	9.60%	60	31.67	9.10%

21	16.94	7.20%	61	32.16	2.40%
22	17.24	3.60%	62	32.54	3.70%
23	17.57	3.80%	63	32.70	2.80%
24	17.90	25.20%	64	33.02	3.50%
25	18.01	56.50%	65	33.44	7.20%
26	18.39	10.90%	66	33.93	6.00%
27	18.76	34.70%	67	34.27	3.70%
28	19.09	7.00%	68	34.75	3.40%
29	19.81	32.70%	69	34.98	9.70%
30	20.48	32.90%	70	35.47	8.40%
31	20.84	28.10%	71	35.73	3.10%
32	21.68	29.00%	72	36.39	4.20%
33	22.10	32.30%	73	36.89	1.20%
34	22.55	79.40%	74	37.39	6.80%
35	23.10	34.60%	75	37.96	4.20%
36	23.23	25.20%	76	38.42	3.70%
37	23.49	9.60%	77	38.71	4.50%
38	23.70	12.70%	78	39.16	6.40%
39	23.94	27.10%	79	39.66	3.30%
40	24.15	30.10%			

;

preferably, said crystalline form C of the compound of formula V, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 21;

(37) said crystalline form C of the compound of formula V, its differential scanning calorimetry curve has endothermic peaks at peak temperatures of 188.32 $\pm$ 3°C and 221.66 $\pm$ 3°C;

preferably, the differential scanning calorimetry curve of said crystalline form C of the compound of formula V is substantially as shown in figure 22;

(38) said crystalline form C of the compound of formula V, its thermogravimetric analysis curve shows a weight loss of about 0.71% in the temperature range from 29.55 $\pm$ 3°C to 180.0 $\pm$ 3°C, and a weight loss of about 0.85% in the temperature range from 180.0 $\pm$ 3°C to 220.0 $\pm$ 3°C;

preferably, the thermogravimetric analysis curve of said crystalline form C of the compound of formula V is substantially as shown in figure 23;

(39) in said crystalline form C of the compound of formula V, y is 1.1, 1, or 1.05, for example 1.05;

preferably, in said crystalline form C of the compound of formula V, u and i are 0, y is 1; or, u is

0, i is 0.1, y is 1.05;

(40) said crystalline form A of the compound of formula VI, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 12;

table 12

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.53	16.80%	24	19.86	7.80%
2	6.73	20.90%	25	20.60	15.60%
3	6.96	47.50%	26	21.27	14.80%
4	7.74	9.70%	27	21.76	11.20%
5	8.60	42.10%	28	22.35	13.80%
6	9.38	14.10%	29	22.55	32.30%
7	9.91	9.40%	30	22.76	21.80%
8	10.28	21.00%	31	23.38	39.60%
9	10.58	90.90%	32	23.95	11.20%
10	12.16	53.50%	33	24.52	9.20%
11	13.45	21.50%	34	25.15	25.40%
12	13.74	12.60%	35	25.71	40.00%
13	13.98	100.00%	36	27.37	13.20%
14	14.84	42.10%	37	27.68	12.40%
15	15.39	43.10%	38	28.84	16.60%
16	15.73	7.20%	39	29.91	23.30%
17	16.63	35.60%	40	31.77	11.50%
18	17.08	52.80%	41	32.66	10.20%
19	17.37	5.40%	42	34.77	16.80%
20	18.67	21.60%	43	35.23	6.90%
21	18.97	20.90%	44	37.12	3.80%
22	19.16	26.60%	45	38.12	8.40%
23	19.28	34.30%			

;

preferably, said crystalline form A of the compound of formula VI, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 24;

(41) in said crystalline form A of the compound of formula VI, o is 1, 1.1, or 1.01, for example 1.01;

(42) said crystalline form A of the compound of formula VII, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown

in table 13;

table 13

peak	diffraction angle (°, 2θ)	relative intensity	peak	diffraction angle (°, 2θ)	relative intensity
1	5.13	16.30%	16	21.16	9.00%
2	6.73	13.80%	17	21.74	49.70%
3	8.61	32.10%	18	22.37	12.10%
4	9.00	100.00%	19	23.18	6.10%
5	10.05	9.80%	20	23.71	7.80%
6	10.34	6.50%	21	24.43	18.70%
7	11.17	4.40%	22	25.32	31.50%
8	11.57	9.50%	23	26.36	12.50%
9	13.93	5.60%	24	27.29	7.90%
10	15.06	7.50%	25	27.93	11.90%
11	15.55	13.90%	26	28.33	13.10%
12	17.44	58.10%	27	29.14	10.00%
13	17.74	18.10%	28	29.86	8.70%
14	18.62	8.90%	29	31.07	1.70%
15	20.08	3.30%			

;

preferably, said crystalline form A of the compound of formula VII, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 25;

(43) in said crystalline form A of the compound of formula VII, p is 1:1, 1:1.1, or 1:1.03, for example 1:1.03;

(44) said crystalline form A of the compound of formula VIII, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks as shown in table 14;

table 14

peak	diffraction angle (°, 2θ)	relative intensity	peak	diffraction angle (°, 2θ)	relative intensity
1	6.48	14.30%	22	20.35	19.20%
2	7.66	100.00%	23	20.89	20.10%
3	9.06	21.40%	24	21.10	9.60%
4	9.72	20.80%	25	21.68	31.10%
5	10.09	45.10%	26	22.27	24.30%
6	11.10	18.60%	27	22.49	19.60%
7	11.92	44.30%	28	22.97	28.30%

8	12.36	10.80%	29	23.50	22.00%
9	12.98	9.10%	30	24.84	32.30%
10	13.91	79.00%	31	25.52	42.40%
11	14.42	10.20%	32	25.97	18.20%
12	15.14	6.30%	33	26.70	10.20%
13	15.91	19.50%	34	27.34	8.50%
14	16.55	20.20%	35	28.19	8.50%
15	17.04	9.40%	36	29.36	20.20%
16	17.60	12.70%	37	30.28	10.90%
17	18.00	18.50%	38	30.81	10.10%
18	18.27	41.70%	39	31.77	5.80%
19	18.51	58.50%	40	32.97	9.50%
20	19.16	50.10%	41	34.37	11.60%
21	19.72	11.20%			

;

preferably, said crystalline form A of the compound of formula VIII, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 26;

(45) in said crystalline form A of the compound of formula VIII, a is 0, 0.1, or 0.07, for example 0.07;

(46) said crystalline form A of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 15;

table 15

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.96	57.90%	39	25.98	27.10%
2	8.20	4.10%	40	26.26	6.70%
3	8.37	5.70%	41	26.66	6.70%
4	9.93	10.20%	42	27.35	21.60%
5	10.13	8.40%	43	27.52	30.80%
6	10.57	36.50%	44	27.79	7.70%
7	11.03	14.20%	45	28.08	31.60%
8	11.88	14.90%	46	28.30	10.80%
9	12.34	33.30%	47	28.97	15.00%
10	12.77	24.90%	48	29.17	39.40%
11	13.42	5.20%	49	29.35	12.90%
12	14.06	32.00%	50	29.92	7.40%
13	14.40	28.30%	51	30.36	40.50%

14	14.97	29.20%	52	31.21	6.60%
15	15.68	2.80%	53	31.65	5.50%
16	15.85	1.80%	54	31.83	16.00%
17	16.71	100.00%	55	31.96	37.80%
18	17.56	29.70%	56	32.62	2.80%
19	17.79	73.50%	57	32.95	11.80%
20	18.41	31.00%	58	33.39	6.40%
21	18.70	15.60%	59	33.72	3.40%
22	19.19	5.90%	60	34.17	10.50%
23	19.67	13.00%	61	34.59	8.40%
24	19.88	43.60%	62	35.19	3.00%
25	20.33	57.50%	63	35.19	3.00%
26	20.68	15.20%	64	35.48	3.80%
27	21.20	29.10%	65	35.91	5.40%
28	21.49	2.70%	66	36.12	4.50%
29	22.11	32.50%	67	36.35	9.70%
30	22.64	45.20%	68	36.98	2.20%
31	23.18	32.40%	69	37.41	6.20%
32	23.86	2.20%	70	37.62	10.10%
33	24.22	46.00%	71	37.83	4.30%
34	24.66	72.50%	72	38.32	3.70%
35	25.15	6.60%	73	38.88	7.90%
36	25.43	32.70%	74	39.02	5.20%
37	25.65	8.80%	75	39.54	5.00%
38	25.84	13.40%			

;

preferably, said crystalline form A of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 27;

(47) said crystalline form A of the compound of formula IX, its differential scanning calorimetry curve has endothermic peaks at peak temperatures of 95.83 $\pm$ 3°C and 194.7 $\pm$ 3°C;

preferably, the differential scanning calorimetry curve of said crystalline form A of the compound of formula IX is substantially as shown in figure 28;

(48) said crystalline form A of the compound of formula IX, its thermogravimetric analysis curve shows a weight loss of about 2.03% in the temperature range from 30.52 $\pm$ 3°C to 60.0 $\pm$ 3°C, a weight loss of about 5.13% in the temperature range from 60.0 $\pm$ 3°C to 100.0 $\pm$ 3°C, and a weight loss of about 0.5% in the temperature range from 100.0 $\pm$ 3°C to 190.0 $\pm$ 3°C;

preferably, the thermogravimetric analysis curve of said crystalline form A of the compound of

formula IX is substantially as shown in figure 29;

(49) in said crystalline form A of the compound of formula IX, g is water, d is 1, and h is 3;

(50) said crystalline form B of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 16;

table 16

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.34	100.00%	21	19.47	31.00%
2	8.20	23.30%	22	19.85	52.90%
3	9.08	13.30%	23	21.53	18.40%
4	9.34	63.50%	24	22.14	7.00%
5	9.92	50.50%	25	22.88	22.70%
6	10.13	32.20%	26	24.41	31.40%
7	11.12	16.10%	27	25.43	23.50%
8	11.47	17.90%	28	25.75	23.10%
9	12.19	13.10%	29	26.48	23.50%
10	12.84	54.40%	30	26.83	12.40%
11	13.18	34.90%	31	27.34	8.80%
12	14.89	8.90%	32	27.95	19.80%
13	15.26	13.80%	33	28.26	9.20%
14	16.13	63.90%	34	28.67	10.00%
15	16.36	46.10%	35	29.12	9.00%
16	16.61	73.50%	36	29.96	22.10%
17	16.89	13.20%	37	30.84	8.80%
18	17.31	9.50%	38	32.47	14.10%
19	18.60	37.50%	39	35.25	5.30%
20	18.67	60.70%			

;

preferably, said crystalline form B of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 30;

(51) in said crystalline form B of the compound of formula IX, d is 1;

(52) in said crystalline form B of the compound of formula IX, h is 0;

(53) said crystalline form A of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 17;

table 17

peak	diffraction angle (°, 2θ)	relative intensity	peak	diffraction angle (°, 2θ)	relative intensity
1	5.71	7.20%	25	23.85	3.90%
2	7.46	81.90%	26	24.23	3.00%
3	8.67	2.90%	27	24.57	20.40%
4	11.43	25.50%	28	24.99	9.80%
5	12.01	100.00%	29	25.44	3.30%
6	12.35	2.10%	30	26.08	8.90%
7	13.53	27.00%	31	26.59	10.50%
8	13.70	9.00%	32	26.90	6.70%
9	13.93	7.70%	33	28.05	5.80%
10	14.75	5.80%	34	28.44	20.50%
11	14.91	13.20%	35	28.69	8.10%
12	15.32	9.10%	36	28.98	6.30%
13	17.24	28.30%	37	29.33	4.20%
14	17.71	1.60%	38	30.17	3.30%
15	18.49	25.50%	39	30.87	6.10%
16	18.77	21.20%	40	31.21	5.40%
17	19.02	4.70%	41	32.38	2.70%
18	21.08	24.30%	42	33.52	2.80%
19	21.70	49.30%	43	34.64	1.80%
20	22.12	11.90%	44	36.84	2.90%
21	22.48	2.60%	45	37.81	2.60%
22	22.80	10.50%	46	38.41	1.90%
23	22.95	17.80%	47	38.84	5.70%
24	23.35	2.00%	48	39.54	2.50%

;

preferably, the X-ray powder diffraction pattern of said crystalline form A of the compound of formula X, using Cu-K α radiation, is substantially as shown in figure 31;

(54) in said crystalline form A of the compound of formula X, k is 0, and j is 1, 1.1, or 1.02, for example 1.02;

(55) said crystalline form B of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in Table 18;

table 18

peak	diffraction angle (°, 2θ)	relative intensity	peak	diffraction angle (°, 2θ)	relative intensity
------	---------------------------	--------------------	------	---------------------------	--------------------

1	5.95	20.80%	18	23.79	58.00%
2	8.96	81.20%	19	24.14	23.80%
3	10.81	91.00%	20	24.55	42.60%
4	11.98	46.80%	21	25.57	28.00%
5	14.09	23.00%	22	26.29	12.50%
6	14.70	14.40%	23	26.99	3.00%
7	15.80	48.20%	24	27.26	74.60%
8	16.97	29.20%	25	27.77	70.10%
9	17.92	100.00%	26	29.66	34.10%
10	18.4	64.10%	27	30.28	15.00%
11	20.03	30.00%	28	31.01	15.20%
12	20.73	23.10%	29	32.21	15.40%
13	21.27	45.00%	30	32.53	26.80%
14	22.07	86.10%	31	36.29	25.50%
15	22.42	36.20%	32	37.50	16.40%
16	22.76	32.30%	33	38.64	20.10%
17	23.27	51.70%	34	39.29	20.30%

;

preferably, the X-ray powder diffraction pattern of said crystalline form B of the compound of formula X, using Cu-K α radiation, is substantially as shown in figure 32;

(56) in said crystalline form B of the compound of formula X, k is 0, and j is 0.9, 1, or 0.93, for example 0.93;

(57) said crystalline form C of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 19;

table 19

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	4.65	-0.90%	11	18.61	39.10%
2	6.36	51.00%	12	20.24	26.60%
3	9.42	16.00%	13	20.94	35.50%
4	11.30	98.80%	14	22.61	100.00%
5	12.11	26.70%	15	24.69	71.00%
6	12.82	55.10%	16	26.16	52.30%
7	13.65	15.50%	17	28.57	38.80%
8	15.12	30.30%	18	31.64	9.30%
9	17.07	40.10%	19	32.75	4.40%
10	17.48	37.10%			

;

preferably, the X-ray powder diffraction pattern of said crystalline form C of the compound of formula X, using Cu-K α radiation, is substantially as shown in figure 33;

(58) in said crystalline form C of the compound of formula X, j is 1;

Preferably, in said crystalline form C of the compound of formula X, k is 0 and j is 1; or, z is acetonitrile and k is 0.07;

(59) said crystalline form D of the compound of formula X, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 20;

table 20

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	7.27	11.50%	32	24.95	30.20%
2	7.87	7.90%	33	25.38	1.20%
3	9.25	4.50%	34	25.67	1.60%
4	9.57	2.80%	35	26.37	2.40%
5	10.14	1.70%	36	26.69	9.50%
6	10.68	100.00%	37	27.32	24.90%
7	11.49	3.20%	38	28.12	6.50%
8	12.03	3.10%	39	28.42	2.00%
9	13.33	1.60%	40	28.88	20.30%
10	13.59	8.50%	41	29.29	1.90%
11	14.54	6.30%	42	29.54	2.90%
12	15.55	8.20%	43	29.95	7.20%
13	15.77	24.60%	44	30.34	3.30%
14	16.25	1.30%	45	30.63	7.00%
15	16.5	7.40%	46	31.16	5.70%
16	16.88	20.10%	47	31.82	8.00%
17	17.29	4.30%	48	31.99	9.20%
18	17.66	10.70%	49	32.37	12.40%
19	18.59	1.60%	50	32.70	5.40%
20	19.63	7.10%	51	33.04	3.30%
21	19.82	26.00%	52	33.33	2.40%
22	20.27	11.50%	53	34.29	4.70%
23	21.44	73.20%	54	34.76	2.60%
24	21.76	21.90%	55	35.07	1.60%
25	22.62	10.80%	56	36.50	2.00%
26	23.09	9.80%	57	37.34	2.30%

27	23.37	8.90%	58	37.59	2.80%
28	23.62	32.00%	59	37.90	8.20%
29	24.00	6.80%	60	38.56	1.20%
30	24.33	11.80%	61	38.82	4.10%
31	24.64	11.70%			

;

preferably, the X-ray powder diffraction pattern of said crystalline form D of the compound of formula X, using Cu-K α radiation, is substantially as shown in figure 34;

(60) said crystalline form D of the compound of formula X, its differential scanning calorimetry curve has endothermic peaks at peak temperatures of 62.55 $\pm$ 3°C, 86.97 $\pm$ 3°C, and 267.15 $\pm$ 3°C;

preferably, the differential scanning calorimetry curve of said crystalline form D of the compound of formula X is substantially as shown in figure 35;

(61) said crystalline form D of the compound of formula X, its thermogravimetric analysis curve shows a weight loss of about 4.49% in the temperature range from 32.27 $\pm$ 3°C to 60.0 $\pm$ 3°C, a weight loss of about 2.41% in the temperature range from 60.0 $\pm$ 3°C to 120.0 $\pm$ 3°C, and a weight loss of about 0.6% in the temperature range from 120.0 $\pm$ 3°C to 240.0 $\pm$ 3°C;

preferably, the thermogravimetric analysis curve of said crystalline form D of the compound of formula X is substantially as shown in figure 36;

(62) in said crystalline form D of the compound of formula X, j is 1 or 1.02, for example 1.02;

(63) in said crystalline form D of the compound of formula X, k is 5, 4.8, or 4, for example 4.8;

preferably, in said crystalline form D of the compound of formula X, z is water, j is 1, and k is 5; or, j is 1.02 and k is 4.8;

(64) said crystalline form A of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation and expressed in terms of 2 θ angle has diffraction peaks as shown in table 21;

table 21

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	4.69	1.80%	19	23.03	1.40%
2	5.58	2.20%	20	23.58	45.80%
3	6.9	4.30%	21	24.15	6.00%
4	9.39	5.20%	22	26.48	1.90%
5	10.67	37.80%	23	27.17	1.00%

6	11.15	3.10%	24	27.93	13.80%
7	12.78	95.20%	25	28.85	2.40%
8	14.09	100.00%	26	30.21	5.60%
9	14.98	76.20%	27	31.25	2.40%
10	15.52	2.40%	28	32.47	3.00%
11	15.88	0.90%	29	33.25	4.70%
12	17.22	9.30%	30	35.12	4.00%
13	18.82	27.70%	31	36.16	2.70%
14	19.35	9.80%	32	36.70	5.60%
15	19.49	16.80%	33	37.18	2.20%
16	20.82	1.70%	34	38.16	4.50%
17	21.80	15.90%	35	39.03	3.10%
18	22.45	7.40%			

;

preferably, said crystalline form A of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 37;

(65) in said crystalline form A of the compound of formula XI, m is 0, and n is 0.6, 0.7, or 0.62, for example 0.62;

(66) said crystalline form B of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation and expressed in terms of 2 θ angle has diffraction peaks as shown in table 22;

table 22

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.54	3.50%	23	22.74	6.00%
2	6.92	5.20%	24	23.05	11.60%
3	8.22	77.00%	25	23.43	82.60%
4	9.31	44.20%	26	24.17	43.60%
5	9.60	13.00%	27	24.77	68.40%
6	10.63	8.80%	28	25.91	2.60%
7	11.56	11.30%	29	26.46	14.90%
8	12.92	46.00%	30	26.86	11.40%
9	13.32	31.20%	31	27.37	27.40%
10	13.62	7.90%	32	29.06	11.70%
11	13.83	15.60%	33	30.04	13.70%
12	15.13	18.50%	34	30.27	20.30%
13	16.29	18.70%	35	30.85	20.40%
14	17.02	8.70%	36	31.40	18.40%

15	17.63	16.60%	37	32.21	12.00%
16	17.77	23.40%	38	33.10	10.30%
17	18.12	33.20%	39	34.32	15.80%
18	18.63	31.00%	40	34.88	11.10%
19	19.38	90.40%	41	36.29	2.20%
20	20.38	100.00%	42	37.62	5.40%
21	21.04	32.30%	43	38.73	9.20%
22	21.28	30.90%	44	39.87	5.30%

;

preferably, said crystalline form B of the compound of formula XI, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 38;

(67) in said crystalline form B of the compound of formula XI, n is 1.1, 1.2, or 1.13, for example 1.13;

preferably, in said crystalline form B of the compound of formula XI, n is 1.1 and m is 1.1; or, n is 1.13 and m is 1.07;

(68) said crystalline form C of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation and expressed in terms of 2 θ angle has diffraction peaks as shown in table 23;

table 23

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.23	100.00%	24	19.39	32.40%
2	5.78	5.00%	25	19.82	71.20%
3	8.07	10.80%	26	20.01	44.60%
4	8.26	15.40%	27	20.87	9.40%
5	9.21	38.30%	28	21.52	12.70%
6	9.80	15.20%	29	21.89	7.90%
7	10.14	8.20%	30	22.27	15.40%
8	10.44	6.60%	31	22.76	6.20%
9	10.92	8.20%	32	23.51	21.30%
10	11.12	14.80%	33	23.83	12.00%
11	11.56	4.40%	34	24.28	6.90%
12	12.72	24.10%	35	24.53	8.90%
13	13.08	6.90%	36	25.26	12.70%
14	14.60	4.30%	37	25.50	12.60%
15	15.24	17.70%	38	26.44	22.20%
16	15.48	14.20%	39	26.72	8.70%

17	16.11	35.50%	40	27.34	10.20%
18	16.57	40.00%	41	28.32	11.00%
19	16.98	17.70%	42	29.73	9.60%
20	17.36	6.40%	43	30.06	5.40%
21	17.89	11.00%	44	31.49	5.00%
22	18.20	19.60%	45	32.60	5.40%
23	18.71	15.10%			

;

preferably, said crystalline form C of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 39;

(69) in said crystalline form C of the compound of formula IX, d is 1, 1.1, or 1.02, for example 1.02;

preferably, in said crystalline form C of the compound of formula IX, g is tetrahydrofuran, d is 1, and h is 0.2; or, g is tetrahydrofuran, d is 1.02, and h is 0.16; or, d is 1 and h is 0;

(70) said crystalline form D of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation and expressed in terms of 2 θ angle has diffraction peaks as shown in table 24;

table 24

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	6.13	100.00%	27	23.14	7.90%
2	8.52	16.50%	28	24.00	19.80%
3	10.19	4.60%	29	24.27	8.20%
4	10.87	27.30%	30	25.28	0.90%
5	12.18	22.40%	31	25.55	1.10%
6	12.59	3.10%	32	25.79	2.10%
7	13.12	19.70%	33	26.01	1.20%
8	13.40	1.40%	34	26.47	3.20%
9	14.25	14.00%	35	26.93	3.20%
10	14.42	10.90%	36	27.44	2.30%
11	15.42	10.40%	37	27.87	2.30%
12	16.60	24.70%	38	28.33	12.30%
13	17.00	5.80%	39	28.82	5.10%
14	17.57	1.60%	40	29.35	3.00%
15	17.84	3.60%	41	29.78	2.70%
16	18.38	33.50%	42	30.73	1.80%
17	19.09	11.90%	43	31.20	2.10%

18	19.34	13.10%	44	31.96	1.30%
19	19.64	3.30%	45	32.52	2.10%
20	20.07	20.90%	46	34.49	0.80%
21	20.39	3.10%	47	35.57	0.20%
22	21.12	8.40%	48	36.00	1.70%
23	21.28	8.20%	49	37.01	0.90%
24	21.59	10.60%	50	37.65	1.40%
25	21.72	8.50%	51	38.27	2.00%
26	22.10	2.30%	52	39.07	1.30%

;

preferably, said crystalline form D of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 40;

(71) in said crystalline form D of the compound of formula IX, d is 1;

Preferably, in said crystalline form D of the compound of formula IX, g is water, d is 1, and h is 0, 1, or 3;

(72) said crystalline form E of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation and expressed in terms of 2 θ angle has diffraction peaks as shown in table 25;

table 25

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.11	1.20%	34	23.95	89.20%
2	5.66	78.90%	35	24.46	8.70%
3	8.11	4.90%	36	25.05	20.10%
4	8.45	14.30%	37	25.49	9.20%
5	9.02	22.30%	38	25.85	14.10%
6	9.84	12.30%	39	26.14	11.30%
7	11.19	25.00%	40	26.58	76.50%
8	11.50	65.80%	41	26.98	25.20%
9	12.29	7.80%	42	27.86	35.60%
10	12.70	13.30%	43	28.05	27.00%
11	13.85	36.70%	44	28.34	4.30%
12	14.30	41.20%	45	28.86	29.50%
13	15.36	5.00%	46	29.05	5.60%
14	15.59	14.80%	47	29.72	13.70%
15	16.20	8.60%	48	30.36	17.10%
16	16.56	23.50%	49	30.58	32.90%

17	16.93	6.90%	50	30.95	6.40%
18	17.18	18.90%	51	31.54	6.50%
19	17.74	68.20%	52	31.74	5.10%
20	17.97	92.90%	53	32.45	6.10%
21	18.75	22.60%	54	32.80	2.10%
22	19.04	64.50%	55	33.23	5.10%
23	19.34	17.70%	56	33.69	4.10%
24	19.52	18.90%	57	34.65	1.80%
25	19.83	100.00%	58	35.36	3.40%
26	20.55	12.80%	59	36.12	3.30%
27	21.07	3.60%	60	36.55	8.60%
28	21.37	5.60%	61	37.16	3.80%
29	21.93	47.00%	62	37.52	7.80%
30	22.25	58.60%	63	38.01	11.40%
31	22.47	15.70%	64	38.72	6.20%
32	23.08	40.90%	65	39.56	2.40%
33	23.43	53.10%			

;

preferably, said crystalline form E of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 41;

(73) said crystalline form E of the compound of formula IX, its differential scanning calorimetry curve has endothermic peaks at peak temperatures of 102.94 $\pm$ 3°C and 183.71 $\pm$ 3°C; preferably, the differential scanning calorimetry curve of said crystalline form E of the compound of formula IX is substantially as shown in figure 42;

(74) said crystalline form E of the compound of formula IX, its thermogravimetric analysis curve shows a weight loss of about 3.11% in the temperature range from 33.06 $\pm$ 3°C to 55.0 $\pm$ 3°C, a weight loss of about 3.82% in the temperature range from 55 $\pm$ 3°C to 120.0 $\pm$ 3°C, and a weight loss of about 1.21% in the temperature range from 120.0 $\pm$ 3°C to 190.0 $\pm$ 3°C; preferably, the thermogravimetric analysis curve of said crystalline form E of the compound of formula IX is substantially as shown in figure 43;

(75) in said crystalline form E of the compound of formula IX, d is 1, 1.1, or 1.02, for example 1.02;

preferably, in said crystalline form E of the compound of formula IX, d is 1, g is isopropanol, and h is 1; or, d is 1.02, g is isopropanol, and h is 0.98;

(76) said crystalline form F of the compound of formula IX, its X-ray powder diffraction

pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks as shown in table 26;

table 26

peak	diffraction angle ($^{\circ}$, 2θ)	relative intensity	peak	diffraction angle ($^{\circ}$, 2θ)	relative intensity
1	6.15	55.40%	39	23.46	3.70%
2	6.99	9.10%	40	24.03	2.60%
3	9.91	0.40%	41	24.34	60.90%
4	10.34	26.50%	42	24.64	3.60%
5	10.75	0.80%	43	25.25	10.30%
6	11.52	4.60%	44	25.74	45.90%
7	11.85	12.00%	45	26.02	20.90%
8	12.27	8.20%	46	26.65	6.20%
9	12.57	8.70%	47	26.83	26.20%
10	12.73	1.30%	48	27.27	6.30%
11	13.35	5.50%	49	27.87	7.20%
12	13.97	15.30%	50	28.13	9.90%
13	14.14	2.80%	51	28.46	13.30%
14	15.52	4.30%	52	28.74	1.30%
15	15.73	24.80%	53	29.71	7.40%
16	16.34	26.30%	54	29.99	2.10%
17	16.55	67.90%	55	30.19	1.50%
18	16.73	5.50%	56	30.52	1.60%
19	17.08	5.00%	57	30.72	4.30%
20	17.30	86.30%	58	31.28	18.20%
21	17.73	0.90%	59	31.73	4.60%
22	18.07	1.30%	60	32.33	1.90%
23	18.42	17.40%	61	32.70	4.00%
24	18.92	7.80%	62	32.98	4.30%
25	19.37	0.70%	63	33.24	8.80%
26	19.82	0.50%	64	33.38	9.50%
27	20.42	41.00%	65	33.68	7.50%
28	20.72	39.90%	66	34.42	8.60%
29	20.98	23.30%	67	35.05	7.20%
30	21.22	26.20%	68	35.67	6.40%
31	21.48	100.00%	69	36.24	1.10%
32	21.83	19.90%	70	36.57	1.80%
33	21.97	18.40%	71	36.88	1.10%
34	22.24	8.20%	72	37.36	6.30%
35	22.43	6.30%	73	38.14	3.10%

36	22.69	1.80%	74	38.48	6.40%
37	22.97	8.10%	75	39.12	1.70%
38	23.27	4.20%			

;

preferably, said crystalline form F of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 44;

(77) said crystalline form F of the compound of formula IX, its differential scanning calorimetry curve has an endothermic peak at a peak temperature of 266.84 $\pm$ 3°C;

preferably, the differential scanning calorimetry curve of said crystalline form F of the compound of formula IX is substantially as shown in figure 45;

(78) said crystalline form F of the compound of formula IX, its thermogravimetric analysis curve shows a weight loss of about 2.2% in the temperature range from 34.19 $\pm$ 3°C to 260.0 $\pm$ 3°C;

preferably, the thermogravimetric analysis curve of said crystalline form F of the compound of formula IX is substantially as shown in figure 46;

(79) in said crystalline form F of the compound of formula IX, d is 1, 1.1, or 1.02, for example 1.02;

preferably, in said crystalline form F of the compound of formula IX, d is 1, and h is 0;

(80) said crystalline form G of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 27;

table 27

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	7.65	25.60%	26	23.10	7.00%
2	9.66	3.90%	27	23.51	6.80%
3	10.73	1.20%	28	23.83	23.40%
4	11.10	1.80%	29	24.47	3.10%
5	11.87	7.90%	30	25.37	100.00%
6	12.60	8.50%	31	25.63	35.80%
7	12.76	8.00%	32	26.65	12.20%
8	13.76	28.70%	33	27.17	8.70%
9	14.11	5.20%	34	27.60	51.60%
10	16.07	13.10%	35	28.36	8.50%
11	17.04	3.70%	36	29.17	15.80%

12	17.43	5.10%	37	29.89	2.90%
13	17.64	11.10%	38	31.46	4.60%
14	17.82	8.60%	39	31.79	9.50%
15	18.41	8.90%	40	32.38	2.80%
16	18.75	6.10%	41	33.26	6.70%
17	18.97	9.60%	42	33.57	4.70%
18	19.41	2.20%	43	33.86	3.80%
19	20.05	12.10%	44	34.25	6.50%
20	20.35	40.00%	45	34.78	2.50%
21	21.01	49.00%	46	36.21	2.60%
22	21.31	10.50%	47	37.19	1.70%
23	21.57	63.70%	48	38.07	4.70%
24	22.23	2.10%	49	38.41	3.40%
25	22.75	15.20%	50	38.74	1.90%

;

preferably, said crystalline form G of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 47;

(81) in said crystalline form G of the compound of formula IX, d is 1, 1.1, or 1.2, for example 1.1;

preferably, in said crystalline form G of the compound of formula IX, d is 1, g is 1,4-dioxane, and h is 3, or, d is 1.1, g is 1,4-dioxane, and h is 2.96;

(82) said crystalline form H of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 28;

table 28

peak	diffraction angle (°, 2 θ)	relative intensity	peak	diffraction angle (°, 2 θ)	relative intensity
1	5.21	14.10%	24	22.44	11.80%
2	8.27	27.00%	25	23.36	16.20%
3	9.70	29.10%	26	24.46	4.60%
4	10.39	9.80%	27	24.92	4.80%
5	11.07	17.20%	28	25.23	30.70%
6	11.60	11.30%	29	26.14	26.00%
7	12.56	18.00%	30	26.33	19.10%
8	13.05	4.30%	31	27.27	16.00%
9	14.96	4.00%	32	27.95	16.80%
10	15.29	10.80%	33	28.31	7.50%

11	16.55	43.40%	34	29.56	23.20%
12	17.07	23.20%	35	30.05	10.70%
13	17.46	2.80%	36	30.38	10.10%
14	18.08	2.20%	37	30.87	9.50%
15	18.49	11.50%	38	31.27	4.00%
16	18.79	19.70%	39	32.21	12.70%
17	19.09	29.50%	40	33.40	1.80%
18	19.78	100.00%	41	35.02	2.10%
19	20.81	9.10%	42	36.28	3.00%
20	21.12	15.80%	43	37.31	2.10%
21	21.46	23.00%	44	38.27	3.70%
22	21.93	3.50%	45	38.92	2.80%
23	22.24	7.90%			

;

preferably, said crystalline form H of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 48;

(83) said crystalline form H of the compound of formula IX, its differential scanning calorimetry curve has endothermic peaks at peak temperatures of 45.53 $\pm$ 3°C, 155.23 $\pm$ 3°C, and 191.45 $\pm$ 3°C;

preferably, the differential scanning calorimetry curve of said crystalline form H of the compound of formula IX is substantially as shown in figure 49;

(84) said crystalline form H of the compound of formula IX, its thermogravimetric analysis curve shows a weight loss of about 1.74% in the temperature range from 32.99 $\pm$ 3°C to 100.0 $\pm$ 3°C, and a weight loss of about 5.38% in the temperature range from 100 $\pm$ 3°C to 180.0 $\pm$ 3°C;

preferably, the thermogravimetric analysis curve of said crystalline form H of the compound of formula IX is substantially as shown in figure 50;

(85) said crystalline form H of the compound of formula IX, wherein g is methyl tert-butyl ether, and h is 0.5, 0.4, or 0.45, for example 0.45;

(86) said crystalline form H of the compound of formula IX, wherein d is 1;

preferably, in said crystalline form H of the compound of formula IX, g is methyl tert-butyl ether, h is 0.5, and d is 1;

(87) said crystalline form I of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2 θ angle, has diffraction peaks as shown in table 29;

table 29

peak	diffraction angle (°, 2θ)	relative intensity	peak	diffraction angle (°, 2θ)	relative intensity
1	6.01	100.00%	21	22.21	2.80%
2	8.34	2.70%	22	22.46	7.20%
3	10.02	4.60%	23	22.68	3.60%
4	10.63	16.60%	24	23.22	14.60%
5	11.08	3.70%	25	24.06	10.00%
6	11.99	7.00%	26	24.73	2.20%
7	12.4	5.80%	27	25.35	5.30%
8	12.79	18.40%	28	25.66	3.90%
9	14.20	14.50%	29	26.08	4.80%
10	15.04	6.30%	30	26.37	3.40%
11	16.67	26.20%	31	27.68	5.90%
12	17.84	17.20%	32	28.08	6.60%
13	18.00	19.30%	33	28.81	2.00%
14	18.47	3.40%	34	29.18	4.90%
15	18.74	8.60%	35	30.92	2.90%
16	19.54	1.30%	36	31.84	1.40%
17	20.07	11.90%	37	32.99	1.00%
18	20.42	1.30%	38	33.72	1.50%
19	20.90	3.50%	39	37.94	2.80%
20	21.20	4.40%	40	39.25	1.80%

;

preferably, said crystalline form I of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 51;

(88) said crystalline form I of the compound of formula IX, wherein d is 1;

(89) in said crystalline form I of the compound of formula IX, h is 0;

preferably, in said crystalline form I of the compound of formula IX, d is 1 and h is 0;

(90) said crystalline form J of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation, expressed in terms of 2θ angle, has diffraction peaks as shown in table 30;

table 30

peak	diffraction angle (°, 2θ)	relative intensity	peak	diffraction angle (°, 2θ)	relative intensity
1	5.27	57.30%	13	19.07	35.50%
2	8.18	50.00%	14	19.66	35.70%

3	9.72	40.00%	15	20.21	62.00%
4	10.54	39.80%	16	21.25	27.60%
5	11.06	100.00%	17	22.33	2.00%
6	11.68	12.90%	18	23.07	20.40%
7	12.44	84.30%	19	25.08	0.50%
8	12.88	36.10%	20	26.48	35.00%
9	15.30	32.70%	21	27.52	7.20%
10	16.41	32.30%	22	28.16	20.90%
11	17.77	8.00%	23	29.27	20.40%
12	18.79	95.30%			

;

preferably, said crystalline form J of the compound of formula IX, its X-ray powder diffraction pattern using Cu-K α radiation is substantially as shown in figure 52;

(91) said crystalline form J of the compound of formula IX, wherein d is 1;

Preferably, in said crystalline form J of the compound of formula IX, d is 1 and h is 0.

6. The nitrogen-containing fused-ring compound according to claim 1, characterized in that said compound of formula IX is a single crystal of the compound of formula IX, wherein g is water, h is 3, and d is 1;

it has the following unit cell parameters: triclinic crystal system, space group P-1; a = 10.6990(12) Å, α = 89.299(7)°, b = 10.7640(13) Å, β = 86.519(6)°, c = 14.9358(17) Å, γ = 84.364(6)°, unit cell volume = 1708.6(3) Å³, number of asymmetric units per unit cell Z = 2, crystal density is 1.46 mg/m³.

7. A method for preparing the crystalline form C of the compound of formula I according to any one of claims 3-6 or said nitrogen-containing fused-ring compound, characterized in that it is any one of the following schemes:

scheme 1:

scheme 1 comprises the following steps: cooling a mixture of a compound and a solvent to induce crystallization, and drying to obtain a crystalline form;

wherein said compound is said compound of formula II, the solvent is a mixed solvent of acetone and water, to obtain crystalline form B of the compound of formula II;

wherein said compound is said compound of formula II, the solvent is ethyl acetate or acetonitrile, to obtain crystalline form C of the compound of formula I;

wherein said compound is said compound of formula III, the solvent is ethyl acetate, to obtain crystalline form A or crystalline form B of the compound of formula III;

wherein said compound is said compound of formula III, the solvent is acetonitrile, to obtain crystalline form C of the compound of formula III;

wherein said compound is said compound of formula IV, the solvent is ethyl acetate, to obtain crystalline form A of the compound of formula IV;

wherein said compound is said compound of formula IV, the solvent is acetonitrile, to obtain crystalline form B of the compound of formula IV;

wherein said compound is said compound of formula V, the solvent is ethyl acetate, to obtain crystalline form A of the compound of formula V;

wherein said compound is said compound of formula V, the solvent is acetonitrile, to obtain crystalline form B of the compound of formula V;

wherein said compound is said compound of formula V, the solvent is acetonitrile, and crystalline form B of the compound of formula V is added to said mixture, to obtain crystalline form C of the compound of formula V;

wherein said compound is said compound of formula VI, the solvent is acetonitrile, to obtain crystalline form A of the compound of formula VI;

wherein said compound is said compound of formula VII, the solvent is acetonitrile, to obtain crystalline form A of the compound of formula VII;

wherein said compound is said compound of formula VIII, the solution is ethyl acetate, to obtain crystalline form A of the compound of formula VIII;

wherein said compound is said compound of formula IX, the solution is a mixed solution of acetone and water, to obtain crystalline form A of the compound of formula IX;

wherein said compound is said compound of formula IX, the solution is acetonitrile, to obtain crystalline form B of the compound of formula IX;

wherein said compound is said compound of formula X, the solvent is a mixed solution of acetone and water, to obtain crystalline form A of the compound of formula X;

wherein said compound is said compound of formula X, the solvent is ethyl acetate, to obtain crystalline form B of the compound of formula X;

wherein said compound is said compound of formula X, the solvent is acetonitrile, to obtain crystalline form C of the compound of formula X;

wherein said compound is said compound of formula X, the solvent is a mixed solution of acetone and water, to obtain crystalline form D of the compound of formula X;

wherein said compound is said compound of formula XI, the solvent is ethyl acetate, to obtain

crystalline form A of the compound of formula XI;

wherein said compound is said compound of formula XI, the solvent is acetonitrile, to obtain crystalline form B of the compound of formula XI;

scheme 2,

scheme 2 comprises the following steps: subjecting a mixture of a compound and a solvent to crystallization and drying to obtain a crystalline form;

wherein said compound is crystalline form A of said compound of formula IX, the solution is tetrahydrofuran, to obtain crystalline form C of the compound of formula IX;

wherein said compound is crystalline form A of said compound of formula IX, the solution is isopropanol, to obtain crystalline form E of the compound of formula IX;

wherein said compound is crystalline form A of said compound of formula IX, the solution is acetonitrile and n-heptane, to obtain crystalline form F of the compound of formula IX;

wherein said compound is crystalline form A of said compound of formula IX, the solution is 1,4-dioxane, to obtain crystalline form G of the compound of formula IX;

wherein said compound is crystalline form A of said compound of formula IX, the solution is a mixed solution of acetonitrile and methyl tert-butyl ether, to obtain crystalline form H of the compound of formula IX;

scheme 3:

scheme 3 comprises the following steps: under nitrogen protection, heating said crystalline form A of the compound of formula IX at 55°C to obtain crystalline form D of the compound of formula IX;

or, under nitrogen protection, heating said crystalline form A of the compound of formula IX at 25°C to obtain crystalline form I of the compound of formula IX;

or, under nitrogen protection, heating said crystalline form H of the compound of formula IX at 170°C to obtain crystalline form J of the compound of formula IX;

scheme 4:

scheme 4 comprises the following steps:

step (1): mixing a mixture of the compound of formula I, acetone, and water with crystalline form A of the compound of formula IX;

step (2): adding a mixture of toluene-4-sulfonic acid, acetone and water, and crystallizing to obtain a single crystal of the compound of formula IX.

8. The preparation method according to claim 7, characterized in that it satisfies one or more of

the following conditions:

- (1) said cooling crystallization is cooling from 50°C to 25°C;
 - (2) said mixed solution of acetone and water is a mixed solution of acetone and water in a volume ratio of 19:1;
 - (3) said drying is centrifugation and/or vacuum drying;
 - (4) in step (1), the mass ratio of said compound of formula I to acetone is (0.5-2):(8-12), for example 1.63:10.3;
 - (5) in step (1), the mass ratio of said acetone to water is (9-11):(0.5-1.5), for example 10.3:1.3;
 - (6) in step (1), the mass ratio of said crystalline form A of the compound of formula IX to said compound of formula I is 1:100 to 1:10, for example 8:163;
 - (7) in step (2), the mass ratio of said toluene-4-sulfonic acid, said acetone and said water is (1-2.5):(5-10):(0.5-1.5), for example 0.58:2.57:0.32;
 - (8) in step (2), said toluene-4-sulfonic acid is toluene-4-sulfonic acid monohydrate;
 - (9) in step (2), said mixture is added in two portions, preferably the first addition is performed at 45-55°C, and the mass ratio of said toluene-4-sulfonic acid to said compound of formula I is 1:(2-4), for example 0.58:1.63; the second addition is performed at 20-30°C, and the mass ratio of said toluene-4-sulfonic acid to said compound of formula I is 1:(4-8), for example 0.29:1.63.
9. A pharmaceutical composition comprising the compound of formula II, the compound of formula III, the compound of formula IV, the compound of formula V, the compound of formula VI, the compound of formula VII, the compound of formula VIII, the compound of formula IX, the compound of formula X, or the compound of formula XI according to any one of claims 1-6, or crystalline form C of the compound of formula I according to any one of claims 3-5, and a pharmaceutically acceptable excipient.
10. Use of the pharmaceutical composition according to claim 9, or the compound of formula II, the compound of formula III, the compound of formula IV, the compound of formula V, the compound of formula VI, the compound of formula VII, the compound of formula VIII, the compound of formula IX, the compound of formula X, or the compound of formula XI according to any one of claims 1-6, or crystalline form C of the compound of formula I according to any one of claims 3-5, in the manufacture of a medicament, wherein said medicament is for treating lung cancer, breast cancer, HR-deficient ovarian cancer, gastric cancer, prostate cancer, pancreatic cancer, or colon cancer.