

CLAIMS

1. A crystal of
7-{(3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquin
5 oline-3-carboxylic acid hydrochloride salt, having powder X-ray
diffraction peaks at diffraction angles 2θ of $10.8^\circ \pm 0.2^\circ$, $12.9^\circ \pm 0.2^\circ$, and $24.7^\circ \pm 0.2^\circ$.

2. The crystal according to claim 1, having powder X-ray
10 diffraction peaks at diffraction angles 2θ of $4.9^\circ \pm 0.2^\circ$, $10.8^\circ \pm 0.2^\circ$, $12.9^\circ \pm 0.2^\circ$, $18.2^\circ \pm 0.2^\circ$, $21.7^\circ \pm 0.2^\circ$, $24.7^\circ \pm 0.2^\circ$, and $26.4^\circ \pm 0.2^\circ$.

3. The crystal according to claim 1 or 2, having a powder X-ray
15 diffraction pattern identical to a powder X-ray diffraction pattern
shown in FIG. 1.

4. The crystal according to any one of claims 1 to 3, wherein,
in differential thermal analysis (DTA), an exothermic peak appears
20 only in the vicinity of 210°C , and in thermogravimetric (TG) analysis,
no weight loss occurs below 100°C .

5. A crystal of
7-{(3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquin
25 yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquin

oline-3-carboxylic acid hydrochloride salt hydrate, having powder X-ray diffraction peaks at diffraction angles 2θ of $9.4^\circ \pm 0.2^\circ$ and $17.7^\circ \pm 0.2^\circ$.

5 6. The crystal according to claim 5, having powder X-ray diffraction peaks at diffraction angles 2θ of $4.8^\circ \pm 0.2^\circ$, $9.4^\circ \pm 0.2^\circ$, $17.7^\circ \pm 0.2^\circ$, $22.8^\circ \pm 0.2^\circ$, $25.8^\circ \pm 0.2^\circ$, and $27.0^\circ \pm 0.2^\circ$.

7. The crystal according to claim 5 or 6, having a powder X-ray
10 diffraction pattern identical to a powder X-ray diffraction pattern shown in FIG. 2.

8. The crystal according to any one of claims 5 to 7, wherein, in differential thermal analysis (DTA), an endothermic peak appears
15 between room temperature and 100°C , and an exothermic peak appears in the vicinity of 140°C , and in thermogravimetric (TG) analysis, weight loss until 100°C is about 7%.

9. The crystal according to any one of claims 5 to 8, wherein
20 a water content determined by a Karl Fischer water content determination is about 7%.

10. A crystal of
7-{(3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquin
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oline-3-carboxylic acid methanesulfonate salt, having powder X-ray diffraction peaks at diffraction angles 2θ of $9.9^\circ \pm 0.2^\circ$, $14.1^\circ \pm 0.2^\circ$, and $28.0^\circ \pm 0.2^\circ$.

5 11. The crystal according to claim 10, having powder X-ray diffraction peaks at diffraction angles 2θ of $9.9^\circ \pm 0.2^\circ$, $14.1^\circ \pm 0.2^\circ$, $16.6^\circ \pm 0.2^\circ$, $19.8^\circ \pm 0.2^\circ$, $22.3^\circ \pm 0.2^\circ$, and $28.0^\circ \pm 0.2^\circ$.

12. The crystal according to claim 10 or 11, having a powder
10 X-ray diffraction pattern identical to a powder X-ray diffraction pattern shown in FIG. 3.

13. The crystal according to any one of claims 10 to 12, wherein,
in differential thermal analysis (DTA), an endothermic peak appears
15 in the vicinity of 213°C , and an exothermic peak appears only in the vicinity of 220°C , and in thermogravimetric (TG) analysis, no weight loss occurs below 100°C .

14. A pharmaceutical composition comprising the crystal
20 according to any one of claims 1 to 13, and a pharmaceutically acceptable carrier, solvent or diluent.

15. An antibacterial agent comprising the crystal according to any one of claims 1 to 13 as an active pharmaceutical ingredient
25 or prepared by incorporating the crystal according to any one of

claims 1 to 13 as an active pharmaceutical ingredient.

16. The antibacterial agent according to claim 15, wherein the antibacterial agent is an oral dosage formulation.

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17. The antibacterial agent according to claim 15, wherein the antibacterial agent is an injection.

18. A method for producing the crystal according to any one
10 of claims 1 to 4, comprising:

adding hydrochloric acid to a 2-propanol solution of
7-{(3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-
yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquin
oline-3-carboxylic acid to obtain a crude hydrochloride salt; and
15 crystallizing the obtained crude hydrochloride salt with
hydrous ethanol having a water activity of less than 0.5 or hydrous
2-propanol having a water activity of less than 0.5.

19. A crystal obtained by: adding hydrochloric acid to a
20 2-propanol solution of
7-{(3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-
yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquin
oline-3-carboxylic acid to obtain a crude hydrochloride salt; and
crystallizing the obtained crude hydrochloride salt with hydrous
25 ethanol having a water activity of less than 0.5 or hydrous 2-propanol

having a water activity of less than 0.5.

20. A method for producing the crystal according to any one of claims 5 to 9, comprising:

5 adding hydrochloric acid to a 2-propanol solution of
7-{(3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquinoline-3-carboxylic acid to obtain a crude hydrochloride salt; and
 crystallizing the obtained crude hydrochloride salt with
10 hydrous ethanol having a water activity of 0.5 or higher or hydrous 2-propanol having a water activity of 0.5 or higher.

21. A crystal obtained by: adding hydrochloric acid to a 2-propanol solution of
15 7-{(3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquinoline-3-carboxylic acid to obtain a crude hydrochloride salt; and
 crystallizing the obtained crude hydrochloride salt with hydrous ethanol having a water activity of 0.5 or higher or hydrous 2-propanol
20 having a water activity of 0.5 or higher.

22. A method of producing the crystal according to any one of claims 10 to 13, comprising:

 adding methanesulfonic acid to an acetone solution of
25 7-{(3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-

yl}-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquinoline-3-carboxylic acid to obtain a crude methanesulfonate salt; and

crystallizing the obtained crude methanesulfonate salt with 5 hydrous acetone.

23. A crystal obtained by: adding methanesulfonic acid to an acetone solution of

7-((3S,4S)-3-[(cyclopropylamino)methyl]-4-fluoropyrrolidine-1-yl)-6-fluoro-1-(2-fluoroethyl)-8-methoxy-4-oxo-1,4-dihydroquinoline-3-carboxylic acid to obtain a crude methanesulfonate salt; and crystallizing the obtained crude methanesulfonate salt with 10 hydrous acetone.