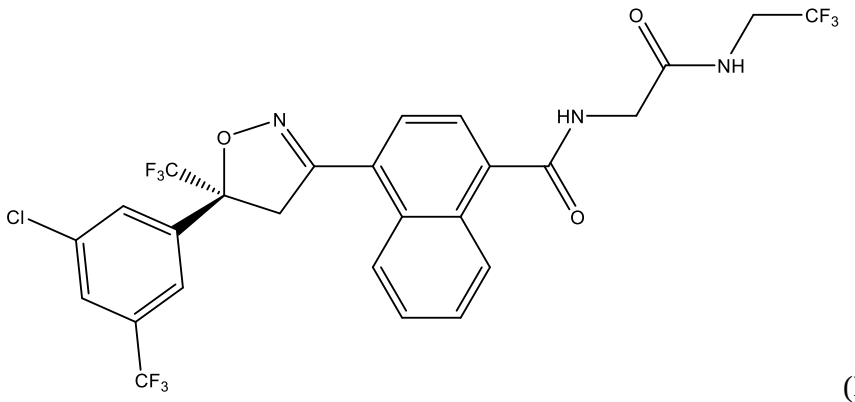
CLAIMS:

1. A crystalline compound of formula (Ia), designated as Form I**,**



(Ia)

wherein said crystals are characterized by having an x-ray powder diffraction pattern comprising three, four, five, six, seven or more peaks selected from the group consisting of: 10.03°, 10.48°, 13.16°, 15.42°, 15.80°, 16.07°, 17.65°, 20.16°, 22.15°, 23.68°, 26.52°, and 28.13° 2θ ±0.2 as determined on a diffractometer using Cu-Kα radiation.

2. The crystalline compound of formula (Ia) according to claim 1, characterized by having an x-ray powder diffraction pattern comprising three or more peaks selected from the group consisting of: 10.03°, 10.48°, 13.16°, 20.16°, and 22.15° 2θ ±0.2 as determined on a diffractometer using Cu-Kα radiation.

3. The crystalline compound of formula (Ia) according to claim 1, characterized by having a differential scanning calorimetry (DSC) thermogram having an peak at a temperature of about 146°C, and an onset at about 143°C, measured with the heating rate of 5°C /min.

4. The crystalline compound of formula (Ia) according to claim 1, characterized by having a differential scanning calorimetry (DSC) thermogram having a heat of fusion of about 61.7 J/g.

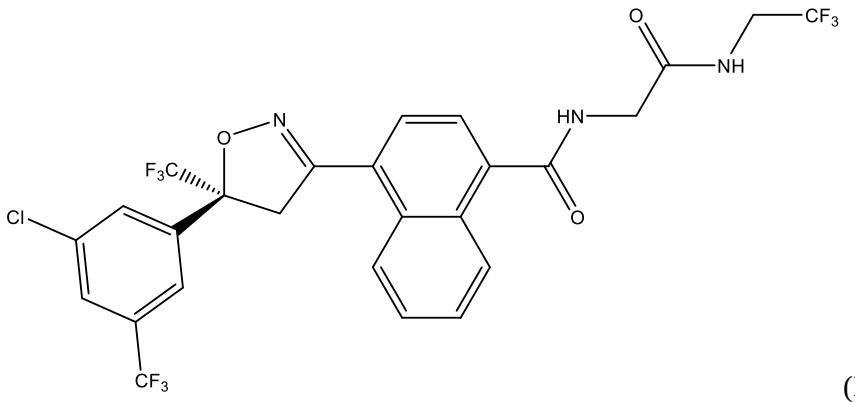
5. The crystalline compound of formula (Ia) according to claim 1, which is enantiomerically pure.

6. The crystalline compound of formula (Ia) according to claim 1, having a degree of chemical purity of at least 97% (w/w).

7. The crystalline compound of formula (Ia) according to claim 1, having an enantiomeric purity of at least 98%.

8. The crystalline compound of formula (Ia) according to claim 1, in substantially pure crystal form.

9. A crystalline compound of formula (Ia), designated as Form II**,**



(Ia),

wherein said crystals are characterized by having an x-ray powder diffraction pattern comprising three, four, five, six, seven or more peaks selected from the group consisting of: 5.99°, 12.99°, 15.80°, 18.71°, 19.33°, 20.24°, 21.65°, 22.17°, 26.11° and 29.00° 2θ ±0.2 as determined on a diffractometer using Cu-Kα radiation.

10. The crystalline compound of formula (Ia) according to claim 9, wherein said crystals are characterized by having an x-ray powder diffraction pattern comprising three or more peaks selected from the group consisting of: 5.99°, 12.99°, 15.80°, 22.17°, 26.11° 2θ ±0.2 as determined on a diffractometer using Cu-Kα radiation.

11. The crystalline compound of formula (Ia) according to claim 9, characterized having a differential scanning calorimetry (DSC) thermogram having an peak at a temperature of about 149°C, and an onset at about 146°C, measured with the heating rate of 5°C /min.

12. The crystalline compound of formula (Ia) according to claim 9, characterized by having a differential scanning calorimetry (DSC) thermogram having a heat of fusion about 65.7 J/g.

13. The crystalline compound of formula (Ia) according to claim 9, which is enantiomerically pure.

14. The crystalline compound of formula (Ia) according to claim 9, having a degree of chemical purity of at least 97%.

15. The crystalline compound of formula (Ia) according to claim 1, wherein the compound of formula (Ia) further comprises a crystalline Form II and/or an amorphous form of the compound of formula (Ia); wherein the crystalline compound of formula (Ia) designated Form II is characterized by having

an X-ray powder diffraction pattern comprising three, four, five, six, seven or more peaks selected from the group consisting of: 5.99°, 12.99°, 15.80°, 18.71°, 19.33°, 20.24°, 21.65°, 22.17°, 26.11° and 29.00° 2θ ±0.2 as determined on a diffractometer using Cu-Kα radiation.

16. A pharmaceutical composition comprising the crystalline compound of formula (Ia) Form I according to claim 1, and at least one pharmaceutically acceptable excipient.

17. The pharmaceutical composition according to claim 16, wherein the composition further comprises a crystalline compound of formula (Ia) designated Form II and/or an amorphous form of the compound of formula (Ia); wherein the crystalline compound of formula (Ia) designated Form II is characterized by having an X-ray powder diffraction pattern comprising three, four, five, six, seven or more peaks selected from the group consisting of: 5.99°, 12.99°, 15.80°, 18.71°, 19.33°, 20.24°, 21.65°, 22.17°, 26.11° and 29.00° 2θ ±0.2 as determined on a diffractometer using Cu-Kα radiation.

18. The pharmaceutical composition of claim 17, wherein the composition comprises at least 90% by weight of the crystalline compound of formula (Ia) Form I according to claim 1, based on the total weight of compound of formula (Ia) in the composition.

19. A process for preparing a crystalline compound of formula (Ia) according to claim 1, which comprises:—

(a) heating a mixture of the toluene solvate of (*S*)-afoxolaner in a solvent, wherein the solvent is acetonitrile, ethyl acetate, a linear, branched or cyclic aliphatic solvent or an alcohol, or a mixture thereof, until dissolution has occurred;

(b) reducing the temperature of the solvent system to induce nucleation;

(c) maintaining the mixture at a temperature below that at which nucleation has commenced; and

(d) isolating the crystalline compound of formula (Ia) so deposited.

20. The process according to claim 19 wherein the alcohol is a lower alkyl alcohol.

21. The process according to claim 20 wherein the lower alkyl alcohol is ethanol.

22. The process according to any one of claims 19 to 21, wherein the aliphatic solvent is a linear, branched or cyclic alkane solvent.

23. The process of claim 19 wherein the solvent is a mixture comprising ethanol and cyclohexane.

24. The process of claim 23 wherein the mixture of ethanol and cyclohexane is about 3:97 to about 10:90 (v/v) ethanol to cyclohexane.

25. The process of claim 23 wherein the mixture of ethanol and cyclohexane is about 8:92 (v/v) ethanol to cyclohexane.

26. The process of claim 19 comprising seeding with enantiomerically pure (*S*)-afoxolaner Form I.

27. The process of claim 19, wherein the heating is to about 50 to about 80 degrees Celsius.

28. The process of claim 19, wherein reducing the temperature is to a temperature of about 5 degree Celsius.

29. Use of the crystalline compound of Formula (Ia) Form I according to claim 1, or a pharmaceutical composition according to any one of claims 16-18 in the manufacture of a medicament for the treatment or prevention of a parasitic infestation in an animal; wherein an effective amount of the medicament is to be administered to the animal.

30. The crystalline compound of formula (Ia) according to claim 1, wherein at least about 90% (w/w) of the crystalline compound is a crystalline Form I form.

31. The crystalline compound of formula (Ia) according to claim 1, wherein the crystalline compound has a chemical purity of about at least 95% (w/w).

32. The crystalline compound of formula (Ia) according to claim 1, wherein the crystalline compound has a chemical purity if about at least 99% (w/w).

33. The crystalline compound of formula (Ia) according to claim 1, wherein the crystalline compound has a chemical purity of about 99.00%(w/w) to about 99.95% (w/w) and an enantiomeric purity of about 99.0 to about 100%.

34. A crystalline compound of Formula (Ia) Form I according to claim 1, or a pharmaceutical composition according to any one of claims 16-18 for use as a medicament.