CLAIMS

1. Anhydrous crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide, characterized by ^{13}C solid state NMR peaks at 50.8 ppm, 58.3 ppm and 43.5 ppm, wherein each peak is ± 0.2 ppm.

2. The compound of claim 1 which is further characterized by a ^{19}F solid state NMR peak at -70.7 ppm ± 0.2 ppm.

3. The compound of claim 1 which is further characterized by one to four powder X-ray diffraction peaks (Cu Kα radiation) selected from the group consisting of peaks at 9.1, 9.6, 10.3 and 16.2 degrees 2θ wherein each peak is ± 0.2 degrees 2θ.

4. The compound of claim 1 which is further characterized by a ^{19}F solid state NMR peak at -70.7 ppm ± 0.2 ppm and one to four powder X-ray diffraction peaks (Cu Kα radiation) selected from the group consisting of peaks at 9.1, 9.6, 10.3 and 16.2 degrees 2θ wherein each peak is ± 0.2 degrees 2θ.

5. The compound according to any one of claims 1 to 4 comprising less than 5% by weight of any other solid form or solid forms of the compound *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide.

6. A pharmaceutical composition comprising a therapeutically effective amount of the compound according to any one of claims 1 to 5 and a pharmaceutically acceptable carrier.

7. Anhydrous crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide characterized by ^{19}F solid state NMR peaks at -72.6 ppm and -73.8 ppm wherein each peak is ± 0.2 ppm and ^{13}C solid state NMR peaks at 182.6 ppm, 156.1 ppm and 52.6 ppm wherein each peak is ± 0.2 ppm.

8. The compound of claim 7 which is further characterized by one to four powder X-ray diffraction peaks (Cu Kα radiation) selected from the group consisting of peaks at 3.6, 7.1, 10.7 and 17.1 degrees 2θ wherein each peak is ± 0.2 degrees 2θ.

9. Anhydrous crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide characterized by ^{19}F solid state NMR peaks at -72.6 ppm ± 0.2 ppm and -73.8 ppm ± 0.2 ppm, one to three ^{13}C solid state NMR peaks selected from the group consisting of peaks at 182.6 ppm, 156.1 ppm and 52.6 ppm wherein each peak is ± 0.2 ppm and one to four powder X-ray diffraction peaks (Cu Kα radiation) selected from the group consisting of peaks at 3.6, 7.1, 10.7 and 17.1 degrees 2θ wherein each peak is ± 0.2 degrees 2θ.

10. The compound according to any one of claims 7 to 9 which comprises less than 5% by weight of any other solid forms of the compound *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide.

11. A pharmaceutical composition comprising a therapeutically effective amount of anhydrous crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide according to any one of claims 7 to 10 and a pharmaceutically acceptable carrier.

12. Crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide, cyclopentyl methyl ether solvate characterized by ^{19}F solid state NMR peaks at -70.2 ppm and -70.5 ppm wherein each peak is ± 0.2 ppm and one to three ^{13}C solid state NMR peaks selected from the group of peaks at 32.7 ppm, 24.2 ppm and 56.0 ppm wherein each peak is ± 0.2 ppm.

13. The compound of claim 12 which is crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide, cyclopentyl methyl ether solvate, further characterized by one to three powder X-ray diffraction peaks (Cu Kα radiation) selected from peaks at 7.1, 7.9 and 19.8 degrees 2θ wherein each peak is ± 0.2 degrees 2θ.

14. Crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide, isopropyl acetate solvate characterized by ^{19}F solid state NMR peaks at -69.8 ppm, -71.9 ppm and -72.4 ppm wherein each peak is ± 0.2 ppm, and one to three ^{13}C solid state NMR peaks selected from the group of peaks at 20.9 ppm ± 0.2 ppm, 38.7 ppm ± 0.2 ppm and 52.0 ppm ± 0.2 ppm.

15. The compound according to claim 14 further characterized by one to four powder X-ray diffraction peaks (Cu Kα radiation) selected from the group of peaks at 8.5, 6.3, 10.7, and 19.1 degrees 2θ wherein each peak is ± 0.2 degrees 2θ.

16. Crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide, ethyl acetate solvate, characterized by a single crystal X-ray diffraction pattern (SXRD) wherein the crystal system is orthorhombic, the space group is $P2_12_12$, the unit cell has dimensions a = 24.7926(16) Å, α = 90°, b = 25.0341(15) Å, β = 90° and c = 9.6240(6) Å, γ = 90°, volume is 5973.2(6) $Å^3$, Z is 8, calculated density is 1.187 g/cm^3, Goodness-of-fit on F^2 is 1.077, Final R indexes [I>=2σ (I)] are R_1 = 0.0880, wR_2 = 0.2433, Final R indexes [all data] are R_1 = 0.1056, wR_2 = 0.2666.

17. Anhydrous crystalline *N*-(Methoxycarbonyl)-3-methyl-L-valyl-(4*R*)-*N*-{(1*S*)-1-cyano-2-[(3*S*)-2-oxopyrrolidin-3-yl]ethyl}-4-(trifluoromethyl)-L-prolinamide, characterized by ^{19}F solid state NMR peaks at -71.0 and -71.5 ppm, each ± 0.2 ppm.

18. The compound according to claim 17 further characterized by one to three ^{13}C solid state NMR peaks selected from the group of peaks at 53.3 ppm, 39.8 ppm and 169.1 ppm wherein each peak is ± 0.2 ppm.

19. The compound according to claim 17 further characterized by one to four ^{13}C solid state NMR peaks selected from the group of peaks at 53.3 ppm, 39.8 ppm, 169.1 ppm and 40.8 ppm, wherein each peak is ± 0.2 ppm.

20. The compound according to claim 17 further characterized by one to two powder X-ray diffraction peaks (Cu Kα radiation) selected from peaks at 11.6 and 14.6 degrees 2θ wherein each peak is ± 0.2 degrees 2θ.