CLAIMS

WHAT IS CLAIMED IS:

1. A crystalline free acid form of (*2S,4R*)-5-(5'-chloro-2'-fluoro-[1,1'-biphenyl]-4-yl)-2-(ethoxymethyl)-4-(3-hydroxyisoxazole-5-carboxamido)-2-methylpentanoic acid (**I′**), wherein the crystalline form is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.51±0.20, 11.62±0.20, 13.05±0.20, 15.07±0.20, and 23.28±0.20.

2. The crystalline form of Claim 1, wherein the crystalline form is characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values of 6.51±0.20, 11.62±0.20, 13.05±0.20, 15.07±0.20, 17.12±0.20, 23.28±0.20, and 26.19±0.20.

3. The crystalline form of Claim 1, wherein the powder x-ray diffraction pattern further comprises one or more additional diffraction peaks at 2θ values selected from 15.72±0.20, 17.12±0.20, 20.79±0.20, 21.10±0.20, 24.48±0.20, 25.81±0.20, and 26.19±0.20.

4. The crystalline form of Claim 1, wherein the crystalline form is characterized by a differential scanning calorimetry trace recorded at a heating rate of 10 ^{o}C per minute which shows a maximum in endothermic heat flow at a temperature between 214 ^{o}C and 218 ^{o}C.

5. A pharmaceutical composition comprising the crystalline form of any one of Claims 1 to 4 and a pharmaceutically acceptable carrier.

6. The pharmaceutical composition of Claim 5, wherein the pharmaceutically acceptable carrier is magnesium stearate.

7. A pharmaceutical composition comprising the crystalline form of any one of Claims 1 to 4 and an AT_1 receptor antagonist, a phosphodiesterase inhibitor, a renin inhibitor, a soluble guanylate cyclase inhibitor, a mineralocorticoid-receptor antagonist, a diuretic, or combinations

thereof.

8. The pharmaceutical composition of Claim 7, further comprising a pharmaceutically acceptable carrier.

9. An oral dosage form comprising the crystalline form of any one of Claims 1 to 4 in a capsule, tablet, or suspension.

10. The oral dosage form of Claim 9, wherein the dosage form is formulated for an immediate, controlled or delayed release.

11. The oral dosage form of Claim 9, wherein the capsule comprises gelatin, polysaccharide, chitosan or synthetic polymers.

12. The oral dosage form of Claim 9, wherein the capsule is a hard capsule that comprises gelatin, polysaccharides, or synthetic polymers.

13. The oral dosage form of Claim 9, wherein the capsule comprises hydroxypropyl methylcellulose.

14. A crystalline form according to any one of Claims 1 to 4 for use in therapy.

15. The use of a crystalline form according to any one of Claims 1 to 4, for the manufacture of a medicament for treating hypertension, heart failure, or renal disease.

16. The use according to Claim 15, wherein the hypertension is primary hypertension, pulmonary arterial hypertension, chronic thromboembolic pulmonary hypertension, or hypertension with renal artery stenosis.

17. The use according to Claim 15, wherein the renal disease is diabetic nephropathy, chronic kidney disease, proteinuria, acute kidney injury, nephrotic syndrome, focal segmental glomerulosclerosis or polycystic kidney disease.

18. The use of the compound of any one of claims 1 to 4 for the manufacture of a medicament for treating a renally-impaired patient once-daily.

19. The use according to claim 18, wherein the renally-impaired patient has chronic kidney disease with an estimated glomerular filtration rate (eGFR) between 60 mL/min/1.73 m^2 and 15 mL/min/1.73 m^2.

20. The use according to claim 15, where atrial natriuretic peptide (ANP) or cyclic guanosine monophosphate (cGMP) levels in a human increase for at least 24 hours.

21. The use according to Claim 20, wherein levels of ANP and cGMP are measured in either urine or plasma or both in the human.

22. A process for preparing the crystalline form of Claim 1, the process comprising

(a) forming a solution comprising (*2S,4R*)-5-(5'-chloro-2'-fluoro-[1,1'-biphenyl]-4-yl)-2-(ethoxymethyl)-4-(3-hydroxyisoxazole-5-carboxamido)-2-methylpentanoic acid and a solvent, optionally with a metal scavenger, at an elevated temperature;

(b) cooling the solution to a temperature between about -20 ^{o}C and 5 ^{o}C; and

(c) isolating the resulting solids to yield the crystalline form.

23. The process according to Claim 22, where the solvent in step (a) is a polar solvent.

24. The process according to Claim 23, where the solvent is a mixture of ethyl acetate and water, ethyl acetate, or ethanol.

25. The process according to Claim 22, where the temperature in step (a) is between about 60 ^{o}C and 95 ^{o}C.

26. The process according to Claim 25, where the temperature is between about 70 ^{o}C and 85 ^{o}C.

27. The process according to Claim 22, where isolating the solids in step (c) involves filtering, washing with one or more solvents, drying in air or under vacuum, or a combination of these steps.

28. The process according to Claim 27, where the solvent is ethyl acetate and water, ethyl acetate or ethanol.

29. The process according to Claim 27, where drying under vacuum is performed at a temperature between 25 ^{o}C and 70 ^{o}C.