We claim:

1. Form I of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid.

2. A crystalline monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern having at least one peak in degrees 2Θ± 0.2° 2Θselected from 7.5, 10.4, 14.2, 15.0, 15.5, 16.1, 18.4, 20.8, 23.6, 24.1, 24.5, 24.8, 25.4, 26.6, 27.2, 27.5, 29.6, and 31.5.

3. The crystalline monohydrate of claim 2, wherein the monohydrate exhibits an X-ray powder diffraction pattern having at least two peaks in degrees 2Θ± 0.2° 2Θselected from 7.5, 10.4, 14.2, 15.0, 15.5, 16.1, 18.4, 20.8, 23.6, 24.1, 24.5, 24.8, 25.4, 26.6, 27.2, 27.5, 29.6, and 31.5.

4. The crystalline monohydrate of claim 2, wherein the monohydrate exhibits an X-ray powder diffraction pattern having at least one peak in degrees 2Θ± 0.2° 2Θselected from 15.5, 24.5, and 24.8.

5. Form II of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid.

6. A crystalline ethanol solvate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern

having at least one peak in degrees 2Θ± 0.2° 2Θselected from 7.5, 14.1, 16.5, 18.5, 25, and 26.

7. The crystalline ethanol solvate of claim 6, wherein the crystalline ethanol solvate exhibits an X-ray powder diffraction pattern having at least one peak in degrees 2Θ± 0.2° 2Θselected from 16.5, 25, and 26.

8. Form III of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid.

9. An octahydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid.

10. An octahydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern having at least one peak in degrees 2Θ± 0.2° 2Θselected from 4.8, 10.4, 11.7, and 14.6.

11. Form IV of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid.

12. A crystalline monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern having at least one peak in degrees 2Θ± 0.2° 2Θselected from 7.2, 18.2, and 24.7.

13. A pharmaceutical composition comprising the crystalline polymorph of any one of claims 1-12 and an active agent.

14. The pharmaceutical composition of claim 13, wherein the active agent is calcitonin.

15. The pharmaceutical composition of claim 14, further comprising parathyroid hormone.

16. The pharmaceutical composition of claim 13, wherein the active agent is insulin.

17. The pharmaceutical composition of claim 13, wherein the active agent is a growth hormone.

18. A process for preparing Form I of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid comprising the steps of:

(a) dissolving a monoethanol solvate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid in a mixture of acetonitrile and water; and

(b) cooling the solution prepared in step (a) to an effective temperature to yield Form I of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid.

19. A process for preparing a monoethanol solvate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid comprising the steps of:

(a) adding sodium hydroxide to a solution of N-(5-chlorosalicyloyl)-8-aminocaprylic acid and ethanol; and

(b) precipitating Form II of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid from the solution.

20. A process for preparing an octahydrate of 5-CNAC by maintaining (i) a monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid, (ii) a monoethanol solvate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid, or (iii) a mixture thereof at a relative humidity of at least 75% for a sufficient time to form the octahydrate.

21. A process for preparing Form IV of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid comprising the steps of:

(a) dissolving the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid in methyl ethyl ketone at an elevated temperature; and

(b) cooling the methyl ethyl ketone solution of step (a) to yield Form IV of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid.

22. A process for preparing a mixture of Forms I and IV of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid comprising the step of cooling a solution of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid and acetone for a sufficient time to produce the mixture of Forms I and IV.

The claims defining the invention are as follows:

1. A crystalline form of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid which is a crystalline ethanol solvate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern having a characteristic peak at 26 degrees 2Θ ± 0.2° 2Θ.

2. The crystalline form of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid of claim 1, wherein the crystalline ethanol solvate exhibits an X-ray powder diffraction pattern having additional characteristic peaks at 16.5 and 25 degrees 2Θ ± 0.2° 2Θ.

3. A crystalline form of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid which is an octahydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern having a characteristic peak at 4.8 degrees 2Θ ± 0.2° 2Θ.

4. A crystalline form of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid which is a crystalline monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern having its most intense peak at 18.2 degrees 2Θ ± 0.2° 2Θ.

5. A pharmaceutical composition comprising a crystalline form of any one of claims 1 to 4 and a biologically active agent.

6. The pharmaceutical composition of claim 5, wherein the biologically active agent is calcitonin.

7. The pharmaceutical composition of claim 6, further comprising parathyroid hormone.

8. The pharmaceutical composition of claim 5, wherein the biologically active agent is insulin.

9. The pharmaceutical composition of claim 5, wherein the biologically active agent is a growth hormone.

10. A process for preparing a monoethanol solvate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid of claim 1 comprising the steps of:

(a) adding sodium hydroxide to a solution of N-(5-chlorosalicyloyl)-8-aminocaprylic acid and ethanol; and

(b) precipitating the monoethanol solvate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid from the solution.

11. A process for preparing an octahydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid of claim 3 comprising maintaining a monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid, (ii) a monoethanol solvate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid, or (iii) a

mixture thereof at a relative humidity of at least 75% for a sufficient time to form the octahydrate.

12. A process for preparing a monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid of claim 4 comprising the steps of:

(a) dissolving a monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid in methyl ethyl ketone at an elevated temperature; and

(b) cooling the methyl ethyl ketone solution of step (a) to yield the monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid.

13. A process for preparing a mixture of Forms I and IV of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid comprising the step of cooling a solution of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid and acetone for a sufficient time to produce the mixture of Forms I and IV.

14. The crystalline form of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid of claim 1, wherein the crystalline form exhibits an X-ray powder diffraction pattern having additional characteristic peaks at 7.5, 14.1, 16.5, 18.5 and 25 degrees $2\Theta \pm 0.2° 2\Theta$.

15. The crystalline form of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid of claim 3, wherein the crystalline form exhibits an X-ray powder diffraction pattern having additional characteristic peaks at 10.4, 11.7 and 14.6 degrees 2Θ

± 0.2° 2Θ.

16. The crystalline form of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid of claim 4, wherein the crystalline form exhibits an X-ray powder diffraction pattern having additional characteristic peaks at 7.2 and 24.7 degrees 2Θ ± 0.2° 2Θ.

17. A pharmaceutical composition comprising:

(a) a crystalline monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern having characteristic peaks in degrees 2Θ ± 0.2° 2Θ at 7.5, 10.4, 14.2, 15.0, 15.5, 16.1, 18.4, 20.8, 23.6, 24.1, 24.5, 24.8, 25.4, 26.6, 27.2, 27.5, 29.6, and 31.5;

(b) a crystalline monohydrate of the disodium salt of N-(5-chlorosalicyloyl)-8-aminocaprylic acid exhibiting an X-ray powder diffraction pattern having characteristic peaks in degrees 2Θ ± 0.2° 2Θ at 7.2, 18.2, and 24.7; and

(c) a biologically active agent.

18. The pharmaceutical composition of claim 17, wherein the biologically active agent is calcitonin.

19. The pharmaceutical composition of claim 17, wherein the biologically active agent is human growth hormone.

20. The pharmaceutical composition of claim 17, wherein the biologically

active agent is parathyroid hormone.