Claims:

1. A diketopiperazine composition comprising a plurality of substantially uniformly formed, microcrystalline particles, wherein the particles have a substantially hollow spherical structure and comprise a shell comprising crystallites of a diketopiperazine that do not self-assemble, and the particles have a volumetric mean geometric diameter less than or equal to 5 μm.

2. The composition of claim 1, wherein said composition comprises an active agent adsorbed to said microcrystalline particles.

3. The composition of claim 1, wherein said composition comprises an active agent entrapped within said microcrystalline particles.

4. The composition of claim 3, wherein said composition comprises an active agent entrapped within the interior void volume of said microcrystalline particles.

5. The composition of claim 1, wherein said microcrystalline particles comprise a trans isomer content of about 45% to about 65%.

6. The composition of claim 1, wherein said diketopiperazine comprises 3,6-bis(N-X-4-aminobutyl)-2,5-diketopiperazine, wherein X is selected from the group consisting of fumaryl, succinyl, maleyl, malonyl, and glutaryl, or a salt thereof.

7. The composition of claim 6, wherein X is fumaryl.

8. The composition of claim 7, wherein said diketopiperazine is a diketopiperazine salt.

9. The composition of claim 2 or 3, wherein said active agent comprises at least one of vasoactive agents, neuroactive agents, hormones, anticoagulants, immunomodulating agents, cytotoxic agents, antibiotics, antiviral agents, antigens, infectious agents, inflammatory mediators, hormones, cell surface receptor agonist and antagonists, cell surface antigens, cytokines, lipokines, enkephalins, alkynes, cyclosporins, anti-IL-8 antibodies, IL-8 antagonists; prostaglandins including PG-12, LTB receptor blockers; triptans, insulin and analogs thereof, growth hormone and analogs thereof, parathyroid hormone (PTH) and analogs thereof, parathyroid hormone related peptide (PTHrP),

ghrelin, obestatin, enterostatin, granulocyte macrophage colony stimulating factor (GM-CSF), amylin, amylin analogs, glucagon-like peptide 1 (GLP-1), clopidogrel, PPACK (D-phenylalanyl-L-prolyl-L-arginine chloromethyl ketone), oxyntomodulin (OXM), peptide YY(3-36) (PYY), adiponectin, cholecystokinin (CCK), secretin, gastrin, glucagon, motilin, somatostatin, brain natriuretic peptide (BNP), atrial natriuretic peptide (ANP), IGF-1, growth hormone releasing factor (GHRF), integrin beta-4 precursor (ITB4) receptor antagonist, analgesics, nociceptin, nocistatin, orphanin FQ2, calcitonin, CGRP, angiotensin, substance P, neurokinin A, pancreatic polypeptide, neuropeptide Y, delta-sleep-inducing peptide and vasoactive intestinal peptide; and analogs of the active agents.

10. The composition of claim 9, wherein said active agent comprises a triptan.

11. The composition of claim 9, wherein said active agent comprises PYY.

12. The composition of claim 9, wherein said active agent comprises a prostaglandin.

13. The composition of claim 9, wherein said active agent comprises PPACK.

14. The composition of claim 9, wherein said active agent comprises a cytotoxic agent.

15. The composition of claim 9, wherein said active agent comprises an antiviral agent.

16. The composition of claim 9, wherein said active agent comprises clopidogrel.

17. The composition of claim 9, wherein said active agent comprises calcitonin.

18. The composition of claim 9, wherein said active agent comprises substance P.

19. The composition of claim 9, wherein said active agent comprises nociceptin.

20. The composition of claim 9, wherein said active agent comprises nocistatin.

21. A diketopiperazine composition comprising a plurality of substantially uniformly formed, microcrystalline particles, wherein the particles are formed by a method comprising:

combining diketopiperazine in a solution and a solution of acetic acid without the presence of a surfactant;

homogenizing in a high shear mixer at high pressures of up to 2,000 psi through an orifice to form a precipitate;

washing the precipitate in suspension with deionized water; and

concentrating the suspension and drying the suspension in a spray drying apparatus.

22. The composition of claim 21, wherein said orifice is about 0.001 in^2 in diameter.

23. The composition of claim 21, wherein said particles comprise an active agent.

24. The composition of claim 23, wherein said active agent comprises at least one of vasoactive agents, neuroactive agents, hormones, anticoagulants, immunomodulating agents, cytotoxic agents, antibiotics, antiviral agents, antigens, infectious agents, inflammatory mediators, hormones, cell surface receptor agonist and antagonists, cell surface antigens, cytokines, lipokines, enkephalins, alkynes, cyclosporins, anti-IL-8 antibodies, IL-8 antagonists; prostaglandins including PG-12, LTB receptor blockers; triptans, insulin and analogs thereof, growth hormone and analogs thereof, parathyroid hormone (PTH) and analogs thereof, parathyroid hormone related peptide (PTHrP), ghrelin, obestatin, enterostatin, granulocyte macrophage colony stimulating factor (GM-CSF), amylin, amylin analogs, glucagon-like peptide 1 (GLP-1), clopidogrel, PPACK (D-phenylalanyl-L-prolyl-L-arginine chloromethyl ketone), oxyntomodulin (OXM), peptide YY(3-36) (PYY), adiponectin, cholecystokinin (CCK), secretin, gastrin, glucagon, motilin, somatostatin, brain natriuretic peptide (BNP), atrial natriuretic peptide (ANP), IGF-1, growth hormone releasing factor (GHRF), integrin beta-4 precursor (ITB4) receptor antagonist, analgesics, nociceptin, nocistatin, orphanin FQ2, calcitonin, CGRP, angiotensin, substance P, neurokinin A, pancreatic polypeptide, neuropeptide Y, delta-sleep-inducing peptide and vasoactive intestinal peptide; and analogs of the active agents.

25. The composition of claim 24, wherein said active agent comprises a triptan.

26. The composition of claim 24, wherein said active agent comprises PYY.

27. The composition of claim 24, wherein said active agent comprises a prostaglandin.

28. The composition of claim 24, wherein said active agent comprises PPACK.29. The composition of claim 24, wherein said active agent comprises a cytotoxic agent.

30. The composition of claim 24, wherein said active agent comprises an antiviral agent.

31. The composition of claim 24, wherein said active agent comprises clopidogrel.

32. The composition of claim 24, wherein said active agent comprises calcitonin.

33. The composition of claim 24, wherein said active agent comprises substance P.

34. The composition of claim 24, wherein said active agent comprises nociceptin.

35. The composition of claim 24, wherein said active agent comprises nocistatin.