WHAT IS CLAIMED:

1. A composition comprising a plurality of diketopiperazine microparticles and a drug or an active agent; wherein each of the diketopiperazine microparticles comprises a plurality of structural domains; said diketopiperazine microparticles have a mass-weighted average particle size of ~~about~~ 33,000 to ~~about~~ 216,000 voxels measured by X-ray tomography for particle size measuring ~~about~~ 0.5 μm to ~~about~~ 4 μm in geometric diameter, wherein said diketopiperazine is 3,6-bis(N-fumaryl-4-aminobutyl)-2,5-diketo-diketopiperazine; or a salt thereof.

2. The composition of claim 1, wherein the ~~diketopiperazine~~ microparticles comprise ~~about~~ 140 to ~~about~~ 170 structural domains.

3. The composition of claim 1 or 2, wherein the ~~diketopiperazine~~ microparticles comprise structural domains ranging in number-weighted size from ~~about~~ 250 voxels to ~~about~~ 1,400 voxels.

4. The composition of any one of the preceding claims, wherein the ~~diketopiperazine~~ microparticles comprise structural domains ranging in voxel-weighted size from ~~about~~ 500 voxels to ~~about~~ 2,000 voxels.

5. The composition of any one of the preceding claims, wherein each of the ~~diketopiperazine~~ microparticles have a specific surface area greater than 35 m^2/g.

6. The composition of any one of the preceding claims, wherein each of the ~~diketopiperazine~~ microparticles have a specific surface area greater than 70 m^2/g.

7. The composition of any one of the preceding claims, wherein the plurality of structural domains have an average size of ~~about~~ 300 nm to ~~about~~ 450 nm as measured by radius of gyration.

8. The composition of any one of the preceding claims, wherein said drug or active agent is a small ~~organic~~ molecule, peptide or protein, a nucleic acid molecule, or combinations thereof.

9. The composition of claim 8, wherein said peptide is an endocrine hormone.

10. The composition of claim 8, wherein said peptide or protein is insulin, parathyroid hormone, calcitonin, glucagon, glucagon-like peptide 1, oxyntomodulin, oxytocin, CCK-8, PYY3-36, ghrelin, vasoactive intestinal peptide, leuprolide, growth hormone, RGD (Arg-Gly-As[[s]]p) peptide, growth hormone releasing peptide, DDAVP (desamino-Cys-

1, D-arg8)vasopressin peptide, cyclosporine, detirelex, somatostatin, interferon-α, granulocyte colony stimulating factor, IgG, or an analog or active fragment thereof.

11. The composition of claim 8, wherein the small ~~organic~~ molecule is a neurotransmitter agonist, a neurotransmitter antagonist, a pain inhibitory agent, a vaccine, an anti-inflammatory agent, an anti-cancer agent, a cell receptor agonist molecule, a cell receptor antagonist molecule, an immunosuppressant, a statin, or an anti-infective agent.

12. The composition of any one of the preceding claims, wherein said diketopiperazine is a fumaryl diketopiperazine ~~(bis—~~3,6-bis(N-fumaryl-4-aminobutyl)-2,5-diketo-diketopiperazine~~; or a~~ salt ~~thereof~~.

13. The composition of any one of the preceding claims in the form of a dry powder.

14. The composition of claim 13, further comprising insulin in an amount greater than 4 units per milligram of the dry powder.

15. The composition of claim 14, wherein the amount of insulin is 6 units per milligram of dry powder.

16. The composition of claim 15, wherein the dry powder comprises an insulin dose comprising 60 units or more of insulin for delivering to a patient in a single inhalation using a dry powder inhaler.

17. A method of synthesizing fumaryl diketopiperazine microparticles, the method comprising:

feeding equal masses of a first solution comprising ~~about~~ 11 wt% to ~~about~~ 12 wt% acetic acid and a second solution comprising ~~about~~ 2.75 wt% fumaryl diketopiperazine solutions and containing a surfactant at a concentration of 0.05 wt% at a temperature of ~~about~~ 17°C to ~~about~~ 22°C through a high shear mixer, and

collecting the fumaryl diketopiperazine microparticles.

18. A method of synthesizing fumaryl diketopiperazine microparticles comprising: collecting fumaryl diketopiperazine microparticles that are a product of feeding a precursor solution through a high shear mixer; wherein the precursor solution comprises a first solution comprising ~~about~~ 11 wt% to ~~about~~ 12 wt% acetic acid, a second solution comprising ~~about~~ 2.75 wt% fumaryl diketopiperazine, and the precursor solution comprises a surfactant at a concentration of ~~about~~ 0.05 wt%.

19. The method according to claim 17 or 18, wherein said surfactant is polysorbate 80.

20. The method according to any one of claims 17-19, further comprising the step of washing the suspension with deionized water to remove excess acid.

21. The method according to any one of claims 17-19, further comprising the step of adding a solution comprising an active agent to said suspension and adjusting the pH of the solution to pH 4.5 with an aqueous ammonia solution.

22. A diketopiperazine microparticle comprising a plurality of structural domains, and having a mass-weighted average particle size of ~~about~~ 33,000 to ~~about~~ 216,000 voxels measured by X-ray tomography for a particle size measuring ~~about~~ 0.5 μm to ~~about~~ 4 μm in geometric size.