WHAT IS CLAIMED IS:

1. A method of making microparticles suitable for pulmonary administration as a dry powder comprising: a) dissolving a fumaryl diketopiperazine (FDKP) composition having an FDKP trans isomer content of about 45% to about 65% in a solution having a basic pH to form an FDKP solution; b) providing a solution of a volatile acid, and c) mixing the solutions together in a high-shear mixer to produce said microparticles in a suspension.

2. The method of claim 1, further comprising determining the trans isomer content of the FDKP.

3. The method of claim 2, wherein the step of determining the trans isomer content of the FDKP is performed using high pressure liquid chromatography.

4. The method of claim 1, further comprising mixing said microparticles with a solution comprising a drug.

5. The method of claim 1, wherein the drug is an endocrine hormone.

6. The method of claim 5, wherein the drug is insulin, parathyroid hormone, calcitonin, glucagon, glucagon-like peptide 1, oxyntomodulin, or an analog of said drug.

7. A method for synthesizing a fumaryl diketopiperazine having a trans isomer content of about 45% to about 65% comprising: a) saponifying an ester of ~~a~~ the fumaryl diketopiperazine in a mixture of water and a water-miscible solvent at a basic pH; and b) recrystallizing the fumaryl diketopiperazine in a reaction mixture comprising trifluoroacetic acid and glacial acid at a resultant ratio ranging from about 0.4 to about 0.7.

8. The method of claim 7, wherein the water-miscible solvent is an alcohol, tetrahydrofuran, dioxane, and acetonitrile.

9. The method of claim 8, wherein the alcohol is methanol, ethanol, or isopropanol.

10. The method of claim 9, wherein the methanol is in a water methanol ratio ranging from 1:1 to about 3:1.

11. The method of claim 7, further comprising the step of determining the trans isomer content of the fumaryl diketopiperazine.

~~12. The method of claim 7, wherein the diketopiperazine is of the formula 2,5-diketo-3,6-bis(N-X-4-aminobutyl)piperazine, wherein X is selected from the group consisting of fumaryl, succinyl, maleyl, and glutaryl.~~

12~~13~~. The method of claim 7, wherein the fumaryl diketopiperazine is ~~2,5-diketo-~~3,6-bis(N-fumaryl-4-aminobutyl)-2,5-diketopiperazine.

13~~14~~. The method of claim 7, wherein the reaction mixture in the recrystallizing step is held for a period of about 4 hours or greater than 4 hours.