CLAIMS

WHAT IS CLAIMED IS:

1. ~~A crystalline~~ Crystalline freebase Form III of biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester characterized by a powder x-ray diffraction comprising diffraction peaks at 2θ values of 6.6±0.1, 13.1±0.1, 18.6±0.1, 19.7±0.1, and 20.2±0.1~~.~~; and

~~2. The crystalline freebase of Claim 1, further characterized by~~ having five or more additional diffraction peaks at 2θ values selected from 8.8±0.1, 10.1±0.1, 11.4±0.1, 11.6±0.1, 14.8±0.1, 15.2±0.1, 16.1±0.1, 16.4±0.1, 16.9±0.1, 17.5±0.1, 18.2±0.1, 19.3±0.1, 19.9±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.

~~3~~2. The crystalline compound of Claim ~~2~~1, characterized by a powder x-ray diffraction pattern comprising diffraction peaks at 2θ values ~~selected from~~of 6.6±0.1, 11.4±0.1, 13.1±0.1, 16.1±0.1, 17.5±0.1, 18.2±0.1, 18.6±0.1, 19.3±0.1, 19.7±0.1, 19.9±0.1, 20.2±0.1, 20.8±0.1, 21.1±0.1, 21.7±0.1, and 22.3±0.1.

~~4. The crystalline compound of Claim 2, further characterized by a powder x-ray diffraction pattern in which the peak positions are substantially in accordance with the peak positions of the pattern shown in FIG. 1.~~

~~5~~3. The crystalline compound of Claim ~~2~~1, further characterized by a differential scanning calorimetry thermogram which shows a melting point of about 125 °C.

~~6. The compound of Claim 2, further characterized by a differential scanning calorimetry thermogram substantially in accordance with that shown in FIG. 4.~~

~~7. The crystalline freebase of Claim 1, further characterized by having five or more additional diffraction peaks at 2θ values selected from 10.6±0.1, 15.0±0.1, 16.0±0.1, 17.3±0.1, 17.7±0.1, 20.9±0.1, 21.4±0.1, 22.6±0.1, 24.6±0.1, and 27.8±0.1.~~

~~8. The crystalline compound of Claim 7, characterized by a powder x-ray diffraction~~

~~pattern comprising diffraction peaks at 2θ values selected from 6.6±0.1, 13.1±0.1, 15.0±0.1, 17.3±0.1, 17.7±0.1, 18.6±0.1, 19.7±0.1, 20.2±0.1, 20.9±0.1, 21.4±0.1, and 22.6±0.1.~~

~~9. The crystalline compound of Claim 7, further characterized by a powder x-ray diffraction pattern in which the peak positions are substantially in accordance with the peak positions of the pattern shown in FIG. 2.~~

~~10. The compound of Claim 7, further characterized by a differential scanning calorimetry thermogram which shows a melting point of about 119 °C.~~

~~11. The compound of Claim 7, further characterized by a differential scanning calorimetry thermogram substantially in accordance with that shown in FIG. 5.~~

~~12~~4. A pharmaceutical composition comprising a pharmaceutically acceptable carrier and the compound of any one of Claims 1 to ~~11~~3.

~~13~~5. The composition of Claim ~~12~~4, which further comprises an agent selected from β_2 adrenergic receptor agonists, steroidal anti-inflammatory agents, phosphodiesterase-4 inhibitors, and combinations thereof; wherein the crystalline form and the agent are formulated together or separately.

~~14~~6. The composition of Claim ~~13~~5, which comprises a β_2 adrenergic receptor agonist and a steroidal anti-inflammatory agent.

~~15~~7. The compound of any one of Claims 1-~~11~~3 in micronized form.

~~16~~8. A process for preparing the crystalline freebase Form III of Claim ~~2~~1, comprising contacting biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester with acetonitrile.

~~17. A process for preparing the crystalline freebase Form IV of Claim 7, comprising a)~~

~~forming a seed crystal of the crystalline freebase Form III; b) dissolving the crystalline freebase Form III in acetonitrile to form a solution; c) and adding the seed crystal to the solution.~~

~~18~~9. A process for purifying biphenyl-2-ylcarbamic acid 1-(2-{[4-(4-carbamoylpiperidin-1-ylmethyl)benzoyl]methylamino}ethyl)piperidin-4-yl ester, comprising forming the compound of any one of Claims 1-~~11~~3.

~~19~~10. A compound as claimed in any one of Claims 1 to ~~11~~3, for use in therapy.

~~20~~11. A compound as claimed in Claim ~~19~~10, for use in the treatment of chronic obstructive pulmonary disease or asthma, or for producing bronchodilation.

~~21~~12. The use of a compound as claimed in any one of Claims 1 to ~~11~~3, for the manufacture of a medicament for the treatment of chronic obstructive pulmonary disease or asthma, or for producing bronchodilation.