

CLAIMS

1. A solid crystalline form of (R)-oxybutynin HCl, designated as Form C.
2. The solid crystalline form of claim 1, having an X-ray powder diffraction pattern comprising at least three peaks, in terms of 2-theta, selected from the group consisting of 6.9 degrees $2\theta \pm 0.2$ degree 2θ , 18.3 degrees $2\theta \pm 0.2$ degree 2θ , 11.7 degrees $2\theta \pm 0.2$ degree 2θ , 16.8 degrees $2\theta \pm 0.2$ degree 2θ and 14.2 degrees $2\theta \pm 0.2$ degree 2θ , at about ambient relative humidity.
3. The solid crystalline form of claim 1, having an X-ray powder diffraction pattern comprising at least four peaks, in terms of 2-theta, selected from the group consisting of 6.9 degrees $2\theta \pm 0.2$ degree 2θ , 18.3 degrees $2\theta \pm 0.2$ degree 2θ , 11.7 degrees $2\theta \pm 0.2$ degree 2θ , 16.8 degrees $2\theta \pm 0.2$ degree 2θ , 14.2 degrees $2\theta \pm 0.2$ degree 2θ , 7.6 degrees $2\theta \pm 0.2$ degree 2θ , and 14.8 degrees $2\theta \pm 0.2$ degree 2θ , at about ambient relative humidity.
4. The solid crystalline form of claim 1, having an X-ray powder diffraction pattern comprising at least five peaks, in terms of 2-theta, selected from the group consisting of 6.9 degrees $2\theta \pm 0.2$ degree 2θ , 18.3 degrees $2\theta \pm 0.2$ degree 2θ , 11.7 degrees $2\theta \pm 0.2$ degree 2θ , 16.8 degrees $2\theta \pm 0.2$ degree 2θ , 14.2 degrees $2\theta \pm 0.2$ degree 2θ , 7.6 degrees $2\theta \pm 0.2$ degree 2θ , and 14.8 degrees $2\theta \pm 0.2$ degree 2θ , 24.2 degrees $2\theta \pm 0.2$ degree 2θ , 13.9 degrees $2\theta \pm 0.2$ degree 2θ , and 8.7 degrees $2\theta \pm 0.2$ degree 2θ , at about ambient relative humidity.
5. The solid crystalline form of claim 1, having an X-ray powder diffraction pattern comprising at least seven peaks, in terms of 2-theta, selected from the group consisting of 6.9 degrees $2\theta \pm 0.2$ degree 2θ , 18.3 degrees $2\theta \pm 0.2$ degree 2θ , 11.7 degrees $2\theta \pm 0.2$ degree 2θ , 16.8 degrees $2\theta \pm 0.2$ degree 2θ , 14.2 degrees $2\theta \pm 0.2$ degree 2θ , 7.6 degrees $2\theta \pm 0.2$ degree 2θ , and 14.8 degrees $2\theta \pm 0.2$ degree 2θ , 24.2 degrees $2\theta \pm 0.2$ degree 2θ , 13.9 degrees $2\theta \pm 0.2$ degree 2θ , and 8.7 degrees $2\theta \pm 0.2$ degree 2θ , at about ambient relative humidity.
6. The solid crystalline form of claim 1, having an X-ray powder diffraction pattern comprising the peaks, in terms of 2-theta, of 6.9 degrees $2\theta \pm 0.2$ degree 2θ , 18.3 degrees $2\theta \pm 0.2$ degree 2θ , 11.7 degrees $2\theta \pm 0.2$ degree 2θ , 16.8 degrees $2\theta \pm 0.2$ degree 2θ , 14.2

degrees $2\theta \pm 0.2$ degree 2θ , 7.6 degrees $2\theta \pm 0.2$ degree 2θ , and 14.8 degrees $2\theta \pm 0.2$ degree 2θ , 24.2 degrees $2\theta \pm 0.2$ degree 2θ , 13.9 degrees $2\theta \pm 0.2$ degree 2θ , and 8.7 degrees $2\theta \pm 0.2$ degree 2θ , at about ambient relative humidity.

7. The solid crystalline form of claim 1, having an X-ray powder diffraction pattern substantially as shown in Figure 4 at about ambient relative humidity.
8. The solid crystalline form of any one of claims 1-7, having a differential scanning calorimetry (DSC) thermogram comprising a melting onset at 109.6 °C and an endothermic peak at 119.1 °C.
9. The solid crystalline form of claim 8, having a differential scanning calorimetry (DSC) thermogram substantially as shown in Figure 7.
10. A solid crystalline form of (R)-oxybutynin HCl, having a combination of the Form B and Form C polymorphs as shown in Figure 9.
11. A pharmaceutical composition comprising the solid crystalline form of any one of claims 1-9 and one or more pharmaceutically acceptable excipients.
12. A process for preparing the solid crystalline form of any one of claims 1-9, comprising precipitating the solid crystalline form from a solution comprising (R)-oxybutynin HCl and a solvent, or slurrying (R)-oxybutynin HCl in a solvent, wherein the solvent comprises an organic solvent excluding methanol, and the content of water is at or below 5% v/v.
13. The process according to claim 12, wherein the organic solvent is selected from the group consisting of n-heptane, propyl acetate, ethyl acetate, isopropyl acetate, methyl isobutyl ketone (MIBK), methyl ethyl ketone (MEK), 1-propanol, ethanol, methyl t-butyl ether (MTBE), 1,4-dioxane, toluene, 1,2-dimethoxyethane, tetrahydrofuran, dichloromethane, acetonitrile, nitromethane, and mixtures thereof.
14. The process of claim 13, wherein the organic solvent is MTBE.
15. A method of treating a condition associated with pharyngeal airway collapse comprising administering to a subject in need thereof the solid crystalline form of any one of claims 1-9.

16. The method of claim 15, wherein the condition associated with pharyngeal airway collapse is sleep apnea or snoring.
17. The method of claim 15, wherein the condition associated with pharyngeal airway collapse is Obstructive Sleep Apnea (OSA).
18. A solid crystalline form of (R)-oxybutynin HCl.
19. The solid crystalline form of claim 18, which is Form A.
20. The solid crystalline form of claim 19, having an X-ray powder diffraction pattern substantially as shown in Figure 2 at about ambient relative humidity.
21. The solid crystalline form of claim 18, which is Form B.
22. The solid crystalline form of claim 21, having an X-ray powder diffraction pattern substantially as shown in Figure 3 at about ambient relative humidity.
23. A pharmaceutical composition comprising a solid crystalline form of (R)-oxybutynin HCl according to any one of claims 18-22 and optionally one or more pharmaceutically acceptable excipients.
24. A solid crystalline form of (R)-oxybutynin citrate.
25. A pharmaceutical composition comprising a solid crystalline form of (R)-oxybutynin citrate according to claim 24 and optionally one or more pharmaceutically acceptable excipients.
26. A process for producing crystalline R-oxybutynin HCl of Form C, the process comprising:
 - isolating (R)-oxybutynin from racemic oxybutynin via chiral resolution with D-malic acid; and
 - adding HCl to the isolated (R)-oxybutynin to produce crystalline (R)-oxybutynin HCl of Form C.
27. The process of claim 26, wherein isolating (R)-oxybutynin from racemic oxybutynin comprises adding D-malic acid to racemic oxybutynin free base.

28. The process of claim 27, wherein D-malic acid is added to racemic oxybutynin free base in the presence of 2-propanol.
29. The process of claim 26, wherein the HCl is added in the presence of ethyl acetate.
30. The process of claim 26, further comprising adding MTBE to the isolated (R)-oxybutynin after addition of HCl.