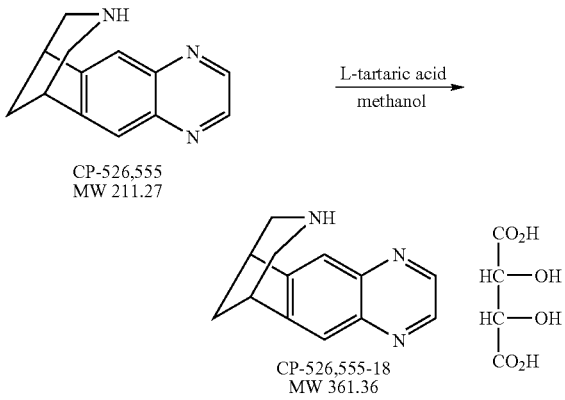
Example 1

L-Tartrate Salt of 5,8,14-Triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene (Anhydrous Polymorph, Form B)



A speck-free vessel was charged with L-tartaric acid (780 grams, 1.1 equiv.) and methanol (7.5 L). The contents of the vessel were stirred until solution and speck free filtered into the crystallization vessel. 5,8,14-triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene free base (992 grams) and methanol (7.5 L) were dissolved in the vessel; the mixture was maintained at between 20 to 25° C. The solution of 5,8,14-triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene free base was added over about 45 minutes to the L-tartaric acid solution through a filter to render the solution speck and fiber free. The product was allowed to stir at 20 to 25° C. overnight and isolated by filtration. The product was dried under vacuum at 35 to 45° C. to give 1618.4 grams (95.4%) of 5,8,14-triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene L-tartrate salt Form B (MW 361.36). M.p. 210.5° C.; verified as Form B by powder x-ray diffraction.

Example 2

L-Tartrate Salt of 5,8,14-Triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene (Anhydrous Polymorph, Form A)

A reactor was charged with 5,8,14-triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene free base (2 g; 0.0095 mole, 1.0 equiv.) and methanol (60 mL, 30 mL/g). The mixture was stirred at 20 to 25° C. until completely dissolved. A second reactor containing a solution of L-tartaric acid (1.55 g, 0.0103 mole, 1.1 equiv.) dissolved in methanol (60 mL, 30 mL/g) was heated to reflux in methanol (i.e., 60 to 66° C.). The free base solution was added to the L-tartaric acid solution at methanolic reflux temperature over 20 minutes. The resulting slurry was cooled to 20 to 25° C. over a 1 hour period. The reaction mixture was allowed to stir for approximately 2 hours followed by isolation of the product by filtration. The solid product was washed with methanol (10 mL), then dried under vacuum at 30 to 35° C. to give 3.3 grams (97%) of 5,8,14-triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene L-tartrate Form A. The identity as Form A was determined by PXRD as compared with standard samples.

Example 3

L-Tartrate Saft Form C of 5,8,14-Triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene (Form C)

Preparation of CP-526,555-18 Form C from Form A or Form B: L-tartrate salt Form B (~5 g) was dissolved in water (10 to 15 ml). Acetonitrile (200 to 300 ml) was added and Form C formed as a white precipitate. The resulting slurry was allowed to stir for 10 minutes and then filtered. The wet cake was then allowed to air dry. Product was determined to be Form C by NIR spectroscopy, DSC and PXRD analysis. This procedure may be run with Form A to yield Form C.

Example 4

L-Tartrate Salt Form A of 5,8,14-Triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene (Form A)

Preparation of Form A from Form C: L-tartrate salt Form C (~2 g) was added to 200 to 300 mL hot ethanol (~75° C.) and allowed to stir for 30 minutes. The sample was filtered hot and then dried in a 45° C. vacuum oven (house vacuum). The material was determined to be Form A by NIR spectroscopy, DSC, and PXRD analysis.

What is claimed is:

1. The anhydrous L-tartrate salt of 5,8,14-triazatetracyclo[10.3.1.$0^{2,11}$.$0^{4,9}$]-hexadeca-2(11),3,5,7,9-pentaene, characterized substantially by at least one of the following powder x-ray diffraction pattern peaks expressed in term of 2θ as measured with copper radiation chosen from: 6.1, 16.8 and 21.9.

2. A compound according to claim **1** characterized substantially by the following principal powder x-ray diffraction pattern peaks expressed in terms of 2θ and d-spacings as measured with copper radiation:

Angle 2θ	d-value (Å)
6.1	14.5
12.2	7.2
13.0	6.8
14.7	6.0
16.8	5.3
19.4	4.6
21.9	4.1
24.6	3.6.

3. A compound according to claim **1** characterized substantially by solid state ^{13}C NMR resonance peaks at 178.4, 145.1, and 122.9 ppm.

4. The anhydrous L-tartrate salt of 5,8,14-triazatetracyclo[10.3.1.$0^{2,11}$0.^{4,9}]-hexadeca-2(11),3,5,7,9-pentaene, characterized substantially by at least one of the following powder x-ray diffraction pattern peaks expressed in terms of 2θ as measured with copper radiation chosen from: 5.9 and 21.8.

5. A compound according to claim **4** characterized substantially by the principal powder x-ray diffraction pattern peaks in terms of 2θ and d-spacings measured with copper radiation: