precipitate was collected by centrifugation through a 0.45 μm nylon membrane filter at 14,000 rpm to obtain crystalline Form 5.

Crystalline Form 5 was characterized by XRPD, DSC, and TGA. By XRPD, Form 5 had high crystallinity (FIG. 18). In an essentially pure material of crystalline Form 5, peaks can be observed at angles of refraction 2θ as set forth in Table 15. By DSC, Form 5 exhibited a dehydration peak at T_{onset} of 32.1°C with an enthalpy of 48 J/g, a recrystallization peak at T_{onset} of 81.2°C, and a melting peak at T_{onset} of 172.5°C combined with recrystallization (FIG. 19). TGA shows 4.8% weight loss at 150°C (FIG. 20).

Table 15. X-ray powder diffraction peaks of crystalline Form 5 of Compound A.

Position [2θ°]	Relative Intensity
4.2	100.00
6.5	65.96
6.8	37.63
10.1	55.81
10.7	24.95
11.3	35.73
11.7	83.09
12.2	24.10
13.4	35.73
14.6	38.69
14.9	86.26
15.5	28.75
15.7	36.58
17.1	39.75
17.5	42.92
17.9	39.32
18.2	70.82
19.0	100.00
19.7	68.50
20.7	67.86
21.4	42.71
22.4	31.92
23.6	41.23
24.3	29.39

Example 14

This example demonstrates exemplary methods of preparing and characterizing crystalline Form 6 of Compound A.

About 100 mg of crystalline Form 1 of Compound A was equilibrated in 0.5 mL of 1:1 (v:v) ethyl acetate/heptane at 25°C. About 2 mg of Form 4 seeds were added to the mixture. After stirring the mixture at 25°C for two days, the resulting solids were collected by vacuum filtration to obtain crystalline Form 5.

Crystalline Form 6 was characterized by XRPD, DSC, and TGA. By XRPD, Form 6 had low crystallinity (FIG. 21). In an essentially pure material of crystalline Form 6, peaks can be observed at angles of refraction 2θ as set forth in Table 16. By DSC, Form 6 exhibited a desolvation peak at T_{onset} of 53.3°C with an enthalpy of 44 J/g and an endothermic peak at T_{onset} of 166.4°C with an enthalpy of 4 J/g (FIG. 22). By TGA, Form 6 exhibited 6.7% weight loss at 150°C (FIG. 23).

Table 16. X-ray powder diffraction peaks of crystalline Form 6 of Compound A.

Position [2θ°]	Relative Intensity
4.7	8.30
7.5	68.40
7.9	100.00
10.3	38.90
11.2	11.50
13.1	13.70
17.4	29.90
17.7	49.00
19.1	16.60
19.8	27.80
20.6	10.70
21.7	24.30
22.4	55.90
23.1	24.80
24.1	44.10
25.2	13.30
26.2	41.50

Example 15