

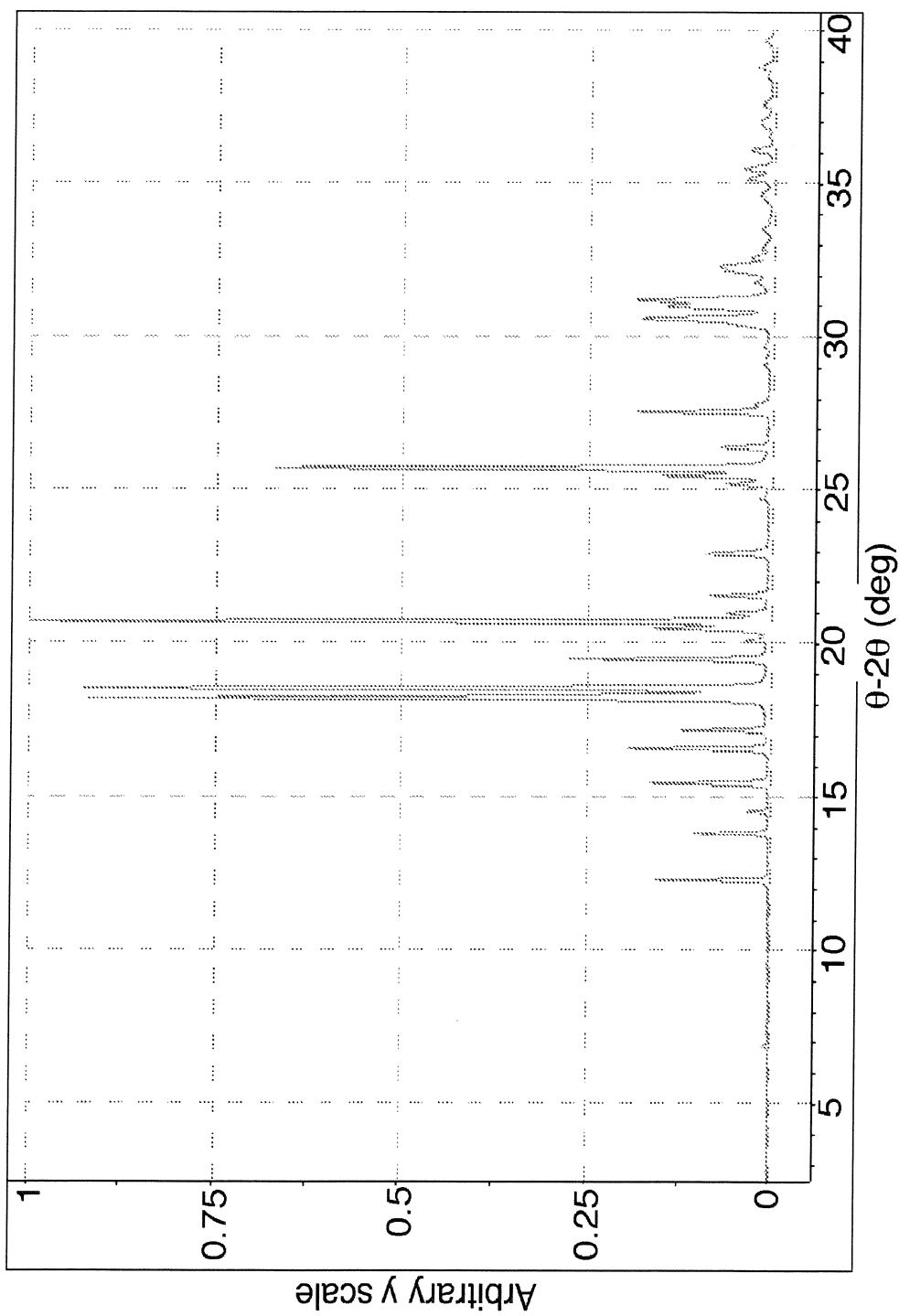


Figure 1 - High-resolution XRPD pattern of Crystalline Form A

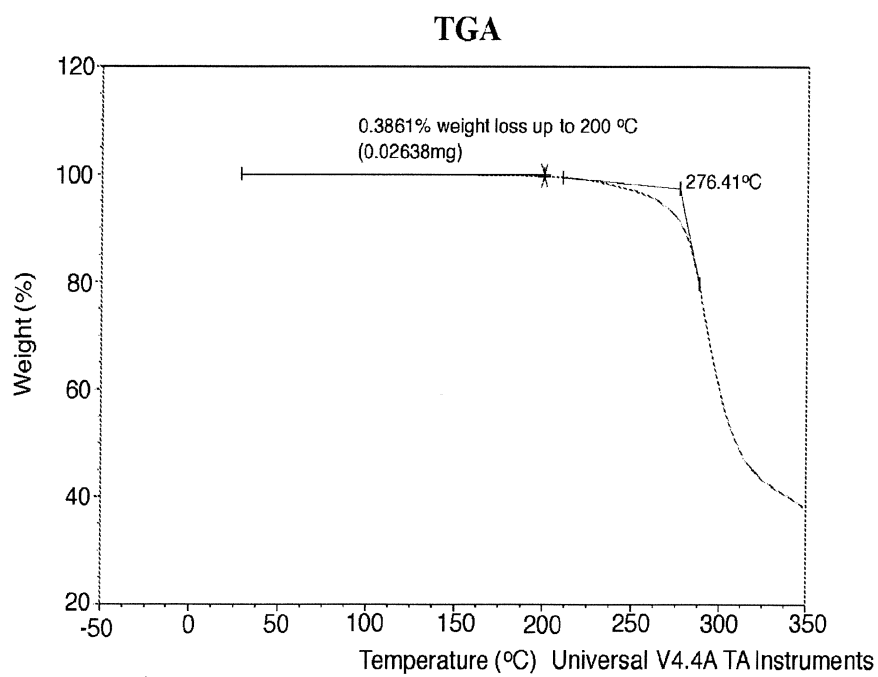
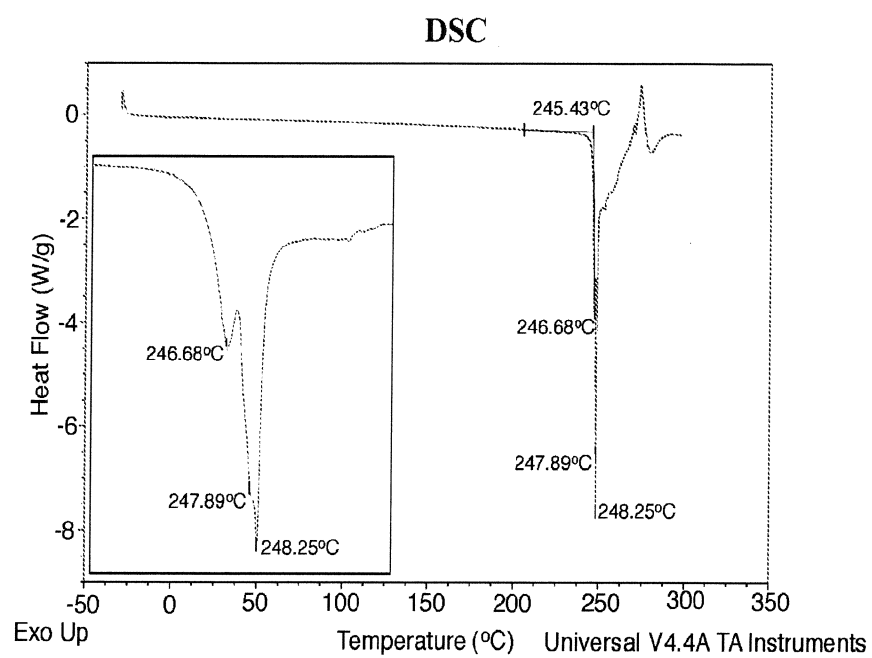


Figure 2 - DSC and TGA thermograms of Crystalline Form A

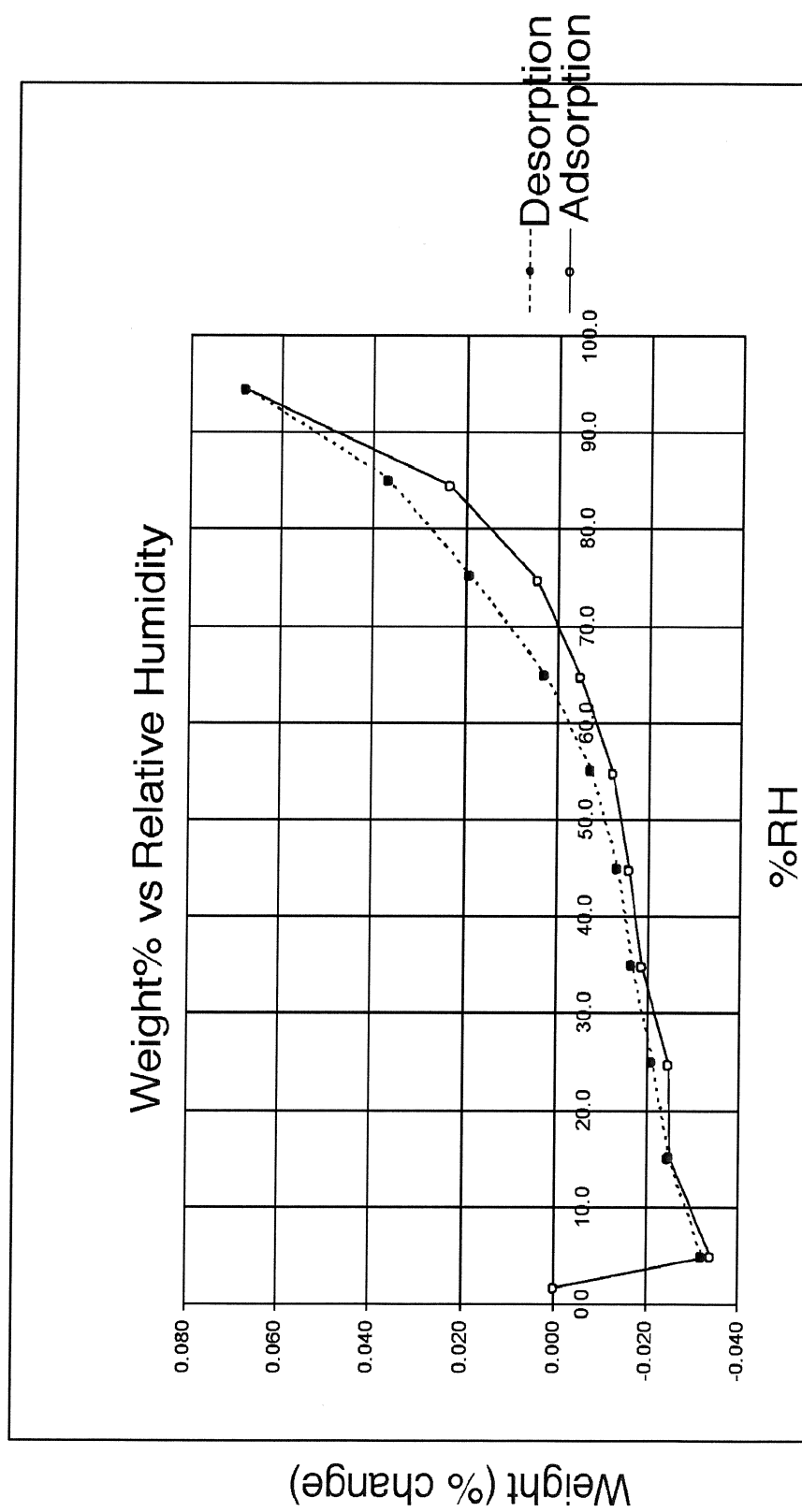


Figure 3 - Dynamic vapor sorption/desorption isotherm of Crystalline Form A
Weight% vs Relative Humidity

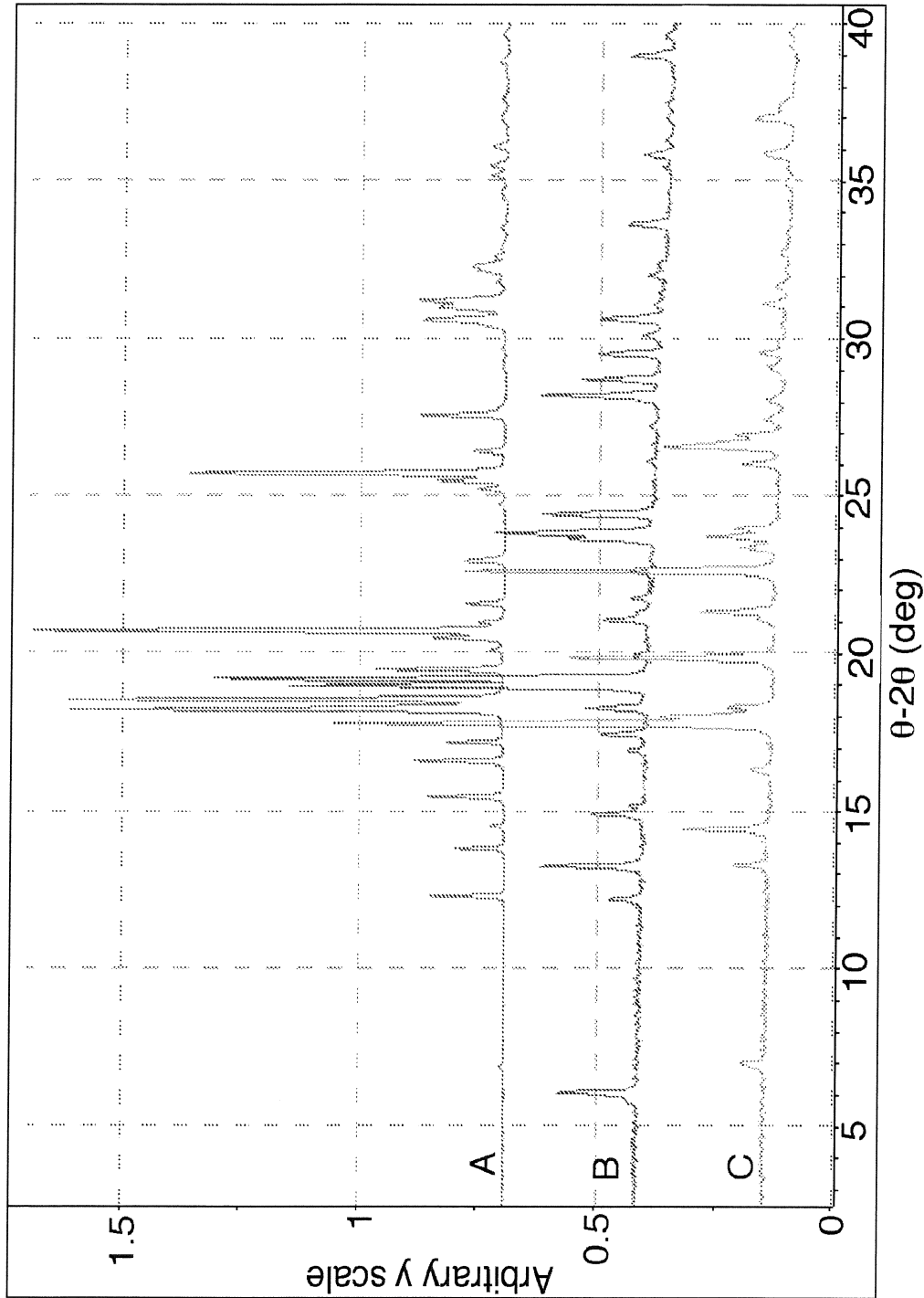


Figure 4 - Overlay of XRPD pattern of Crystalline Form A, Form B, and Form C (top to bottom):
Figure 4A - Form A; Figure 4B - Form B; Figure 4C - Form C

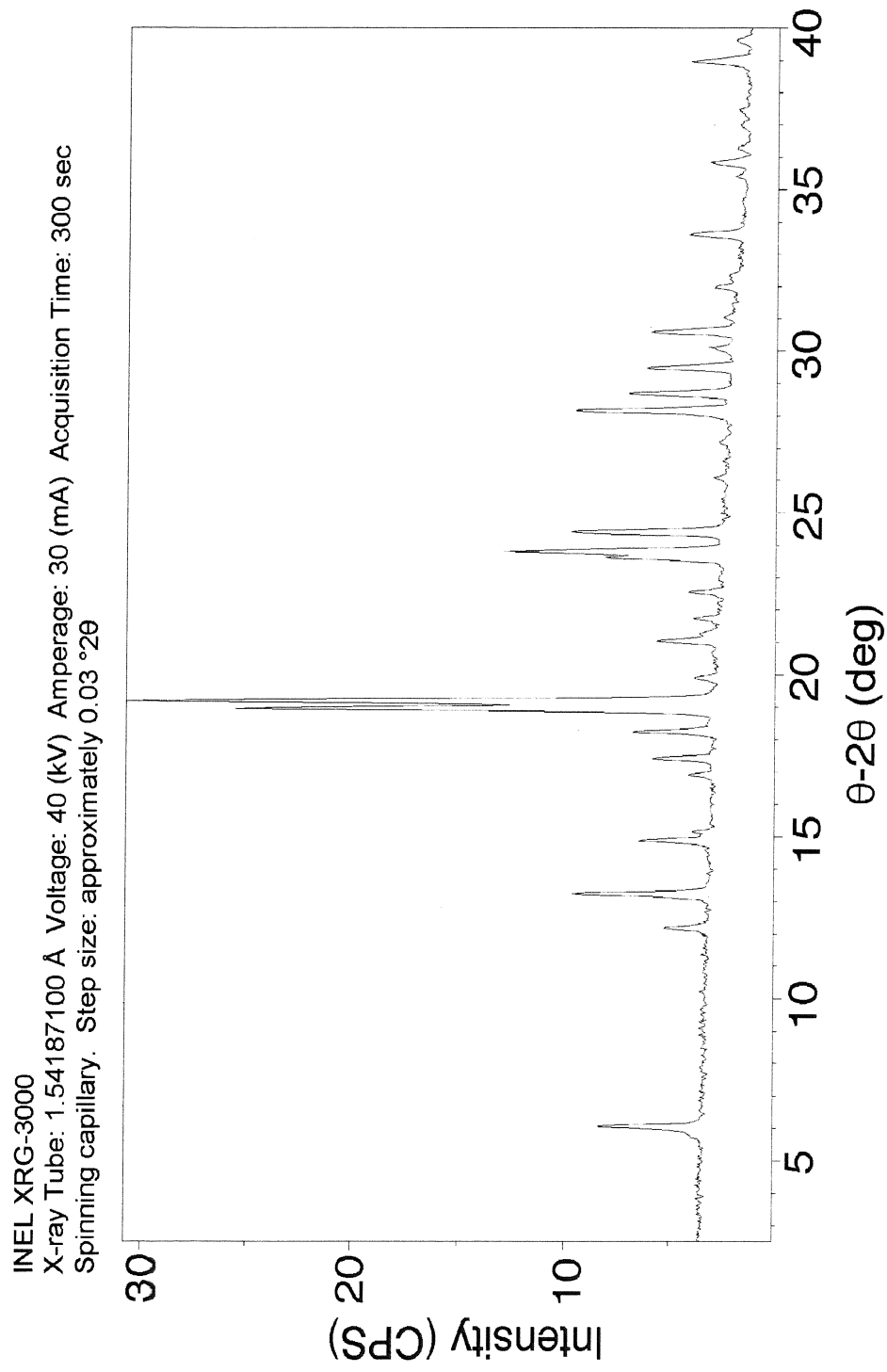
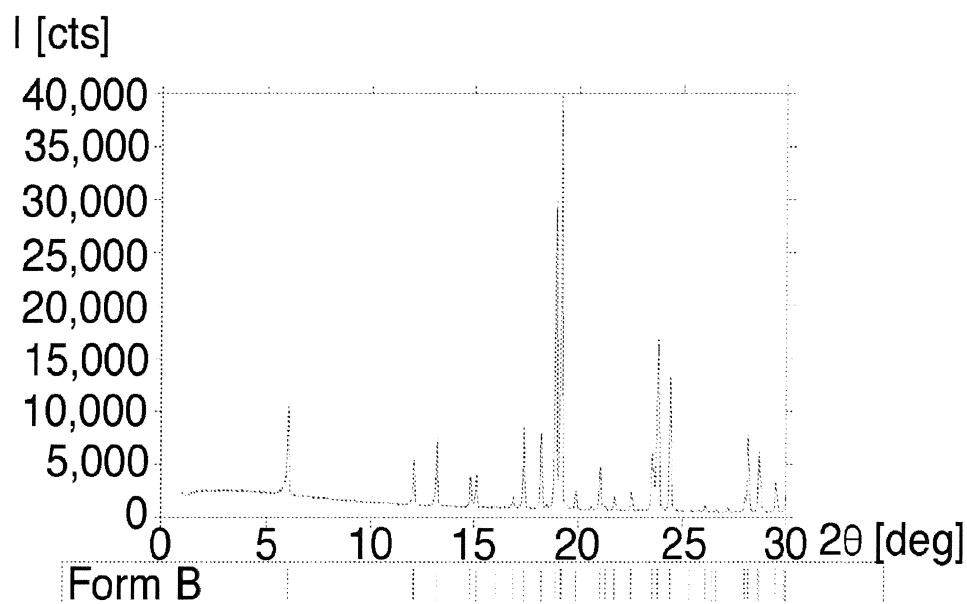


Figure 5 - XRPD pattern of Crystalline Form B

Indexing results for XRPD collected with Cu-K α radiation.



Form B

Bravais Type	Primitive
	Orthorhombic
a [Å]	5.971
b [Å]	7.540
c [Å]	29.155
α [deg]	90
β [deg]	90
γ [deg]	90
Volume [Å ³ /cell]	1,312.6
Chiral Contents?	Chiral
Extinction Symbol	P 21 21 21
Space Group(s)	P 21 21 21 (19)

Source

Manual Input

Figure 6 - Indexing Solution for Crystalline Form B

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 1.00 – 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1939 s Scan Speed: 1.2°/min Slit: DS: 1/2° SS: null Revolution Time: 1.0 s Mode: Transmission

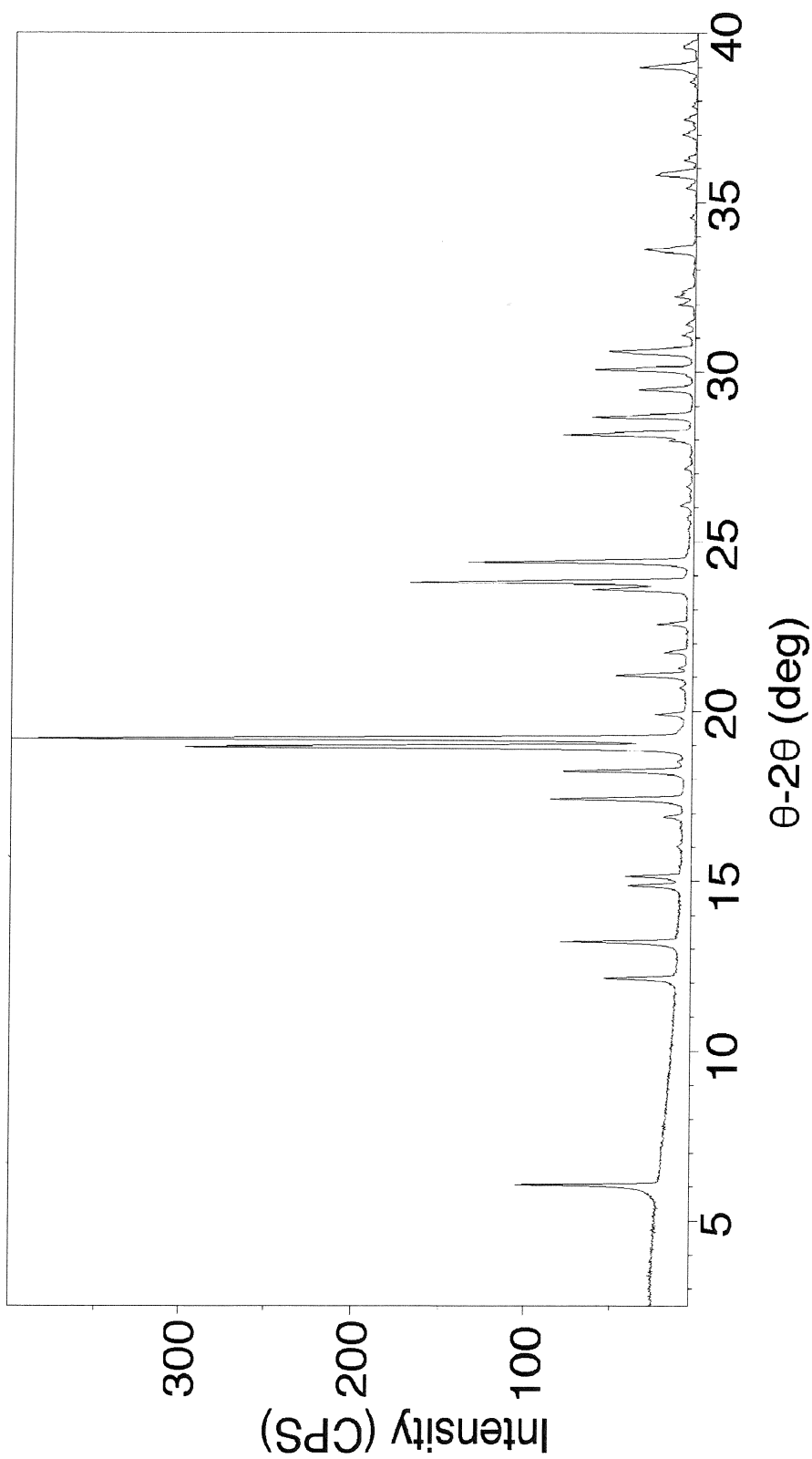


Figure 7 - High-resolution XRPD pattern of Crystalline Form B

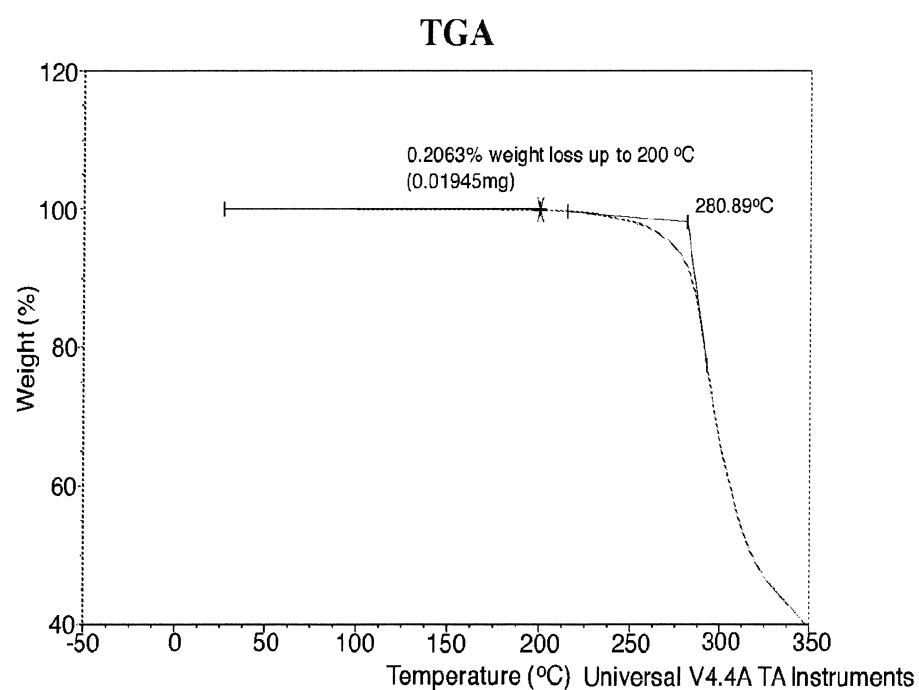
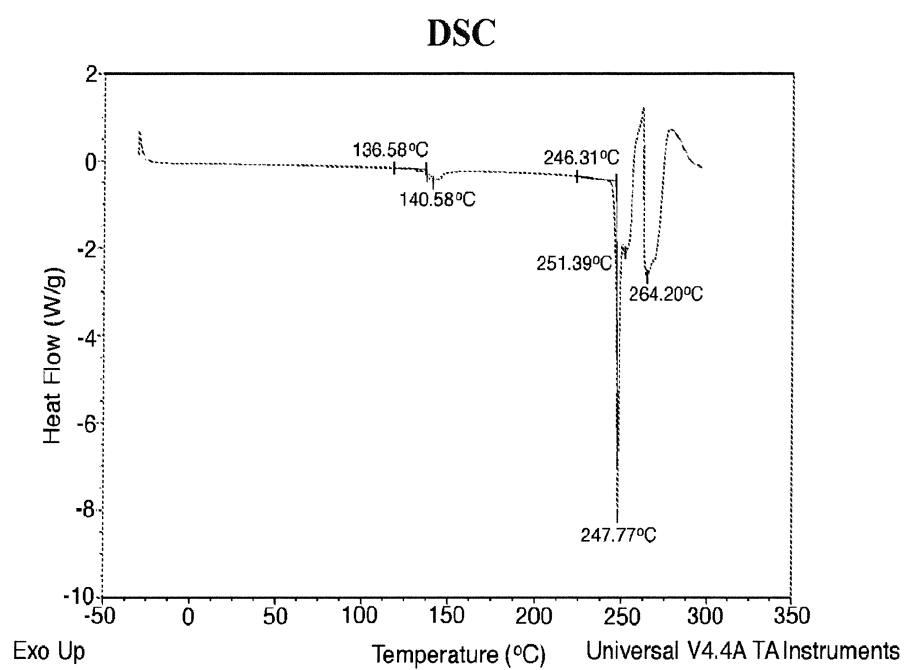


Figure 8 - DSC and TGA thermograms of Crystalline Form B

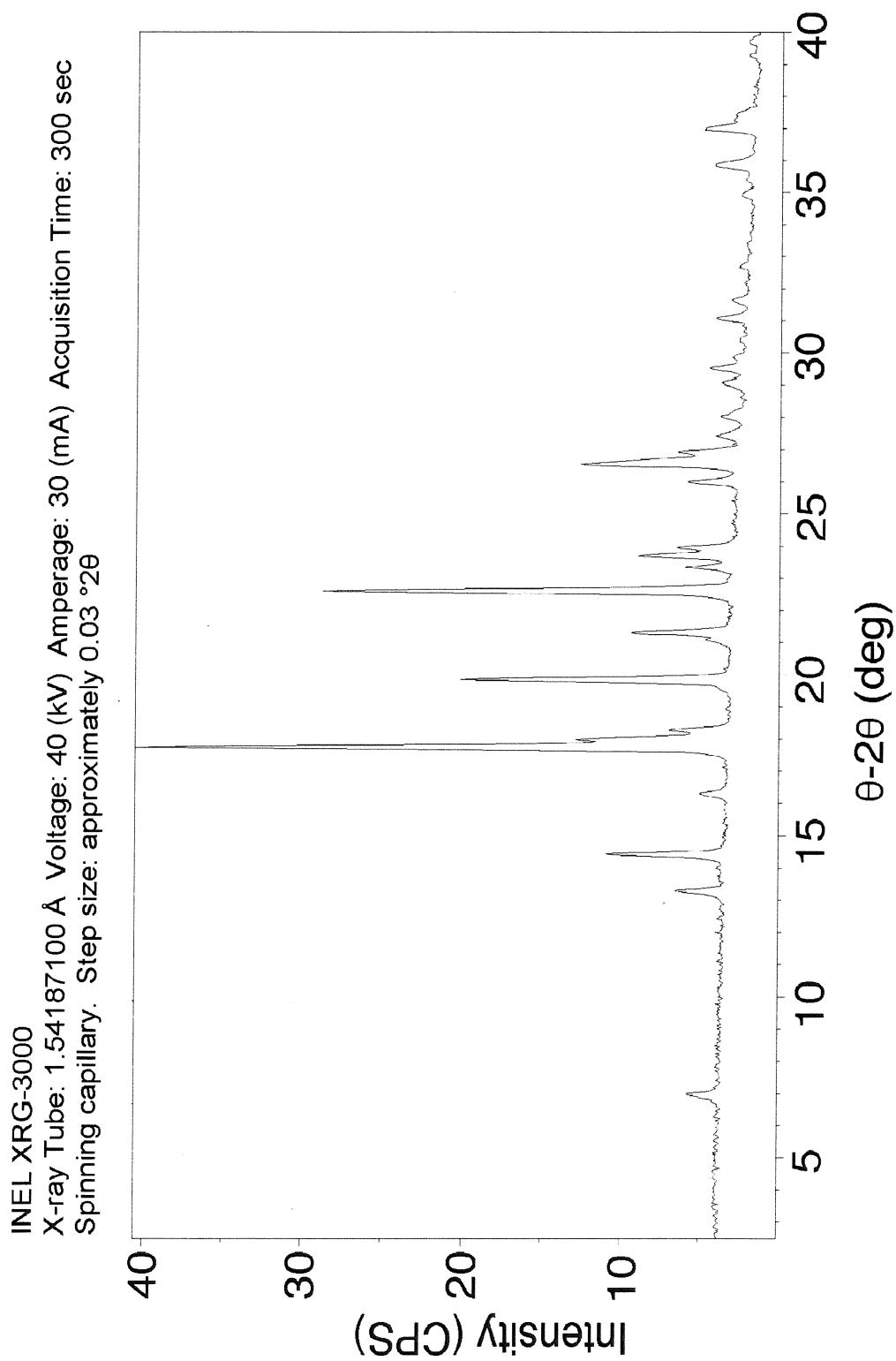
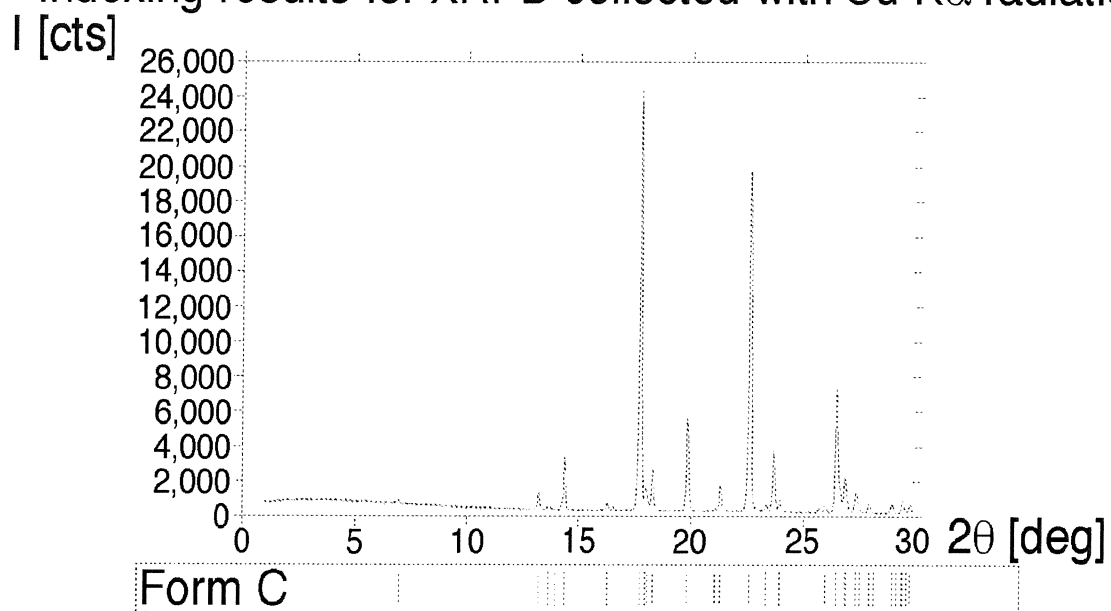


Figure 9 - XRPD pattern of Crystalline Form C

Indexing results for XRPD collected with Cu-K α radiation.



Form C

Bravais Type	Primitive Monoclinic
a [Å]	6.539
b [Å]	7.861
c [Å]	12.791
α [deg]	90
β [deg]	98.76
γ [deg]	90
Volume [Å ³ /cell]	649.9
Chiral Contents?	Chiral
Extinction Symbol	P 1 21 1
Space Group(s)	P 21 (4)

Source

Triads Algorithm

Figure 10 - Indexing Solution for Crystalline Form C

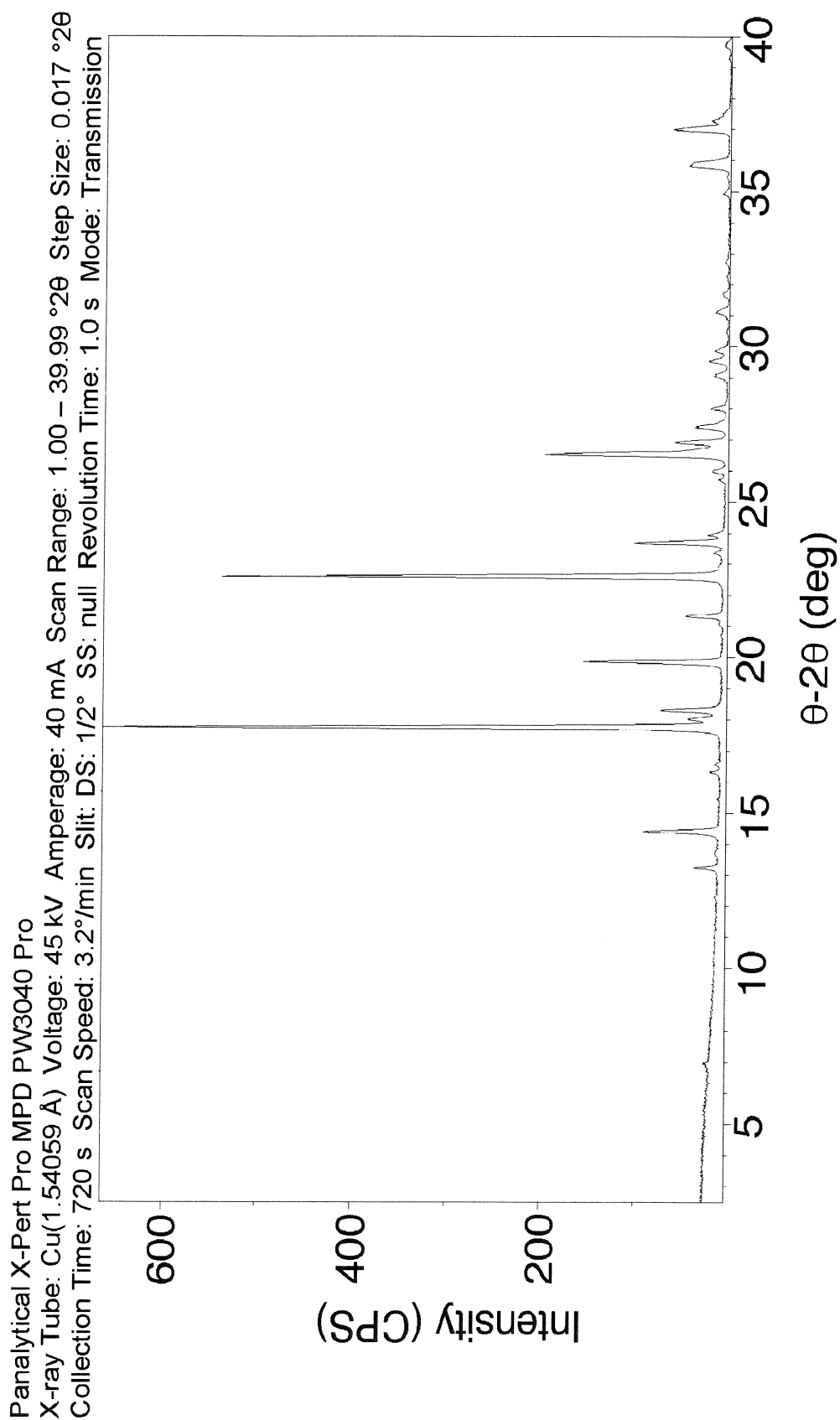


Figure 11 - High-resolution XRPD pattern of Crystalline Form C

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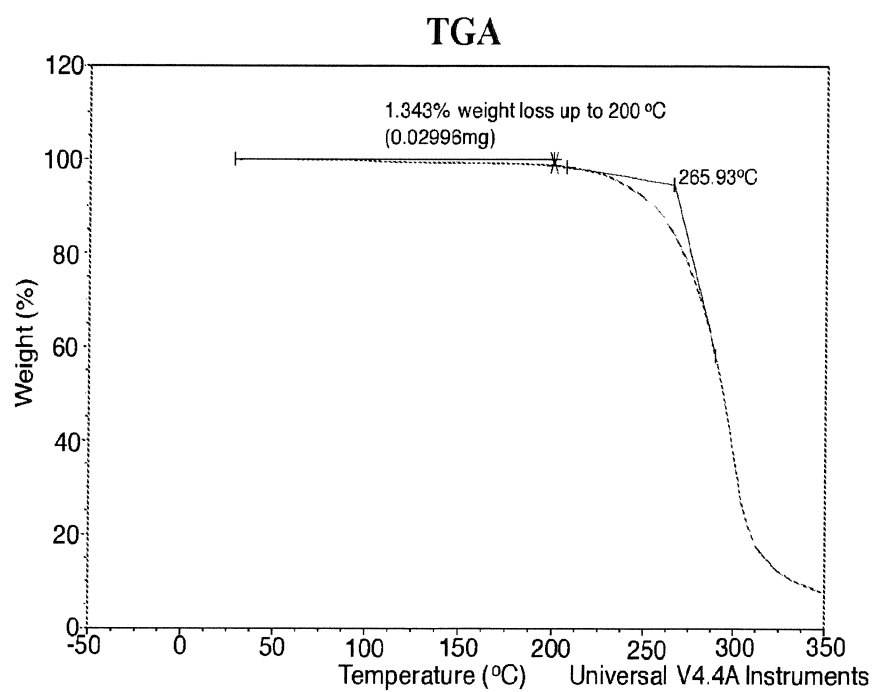
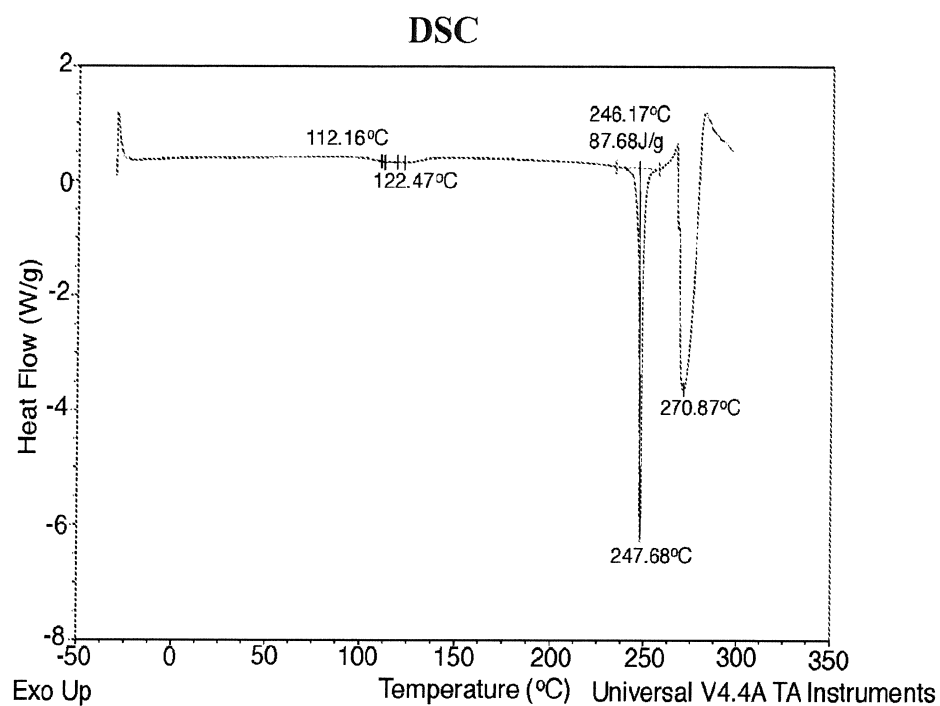


Figure 12 - DSC and TGA thermograms of Crystalline Form C

Figure 13 – Overlay of the X-ray powder diffraction patterns of Crystalline Form B, Form C, and combinations of Crystalline Form C + Crystalline Form B, Crystalline Form C + Crystalline Form A, Crystalline Form B + Crystalline Form A, and Crystalline Form A + Crystalline Form C (top to bottom):

Figure 13A – Crystalline Form B, slow cooling in IPA, solids precipitate in refrigerator

Figure 13B – Crystalline Form C + Crystalline Form B, slow crystalline cooling in IPA, with seeds, solids precipitate in freezer

Figure 13C – Crystalline Form C + Crystalline Form A, slow cooling in IPA, solids precipitate in freezer

Figure 13D – Crystalline Form B, slow cooling in IPA, solids precipitate in freezer

Figure 13E – Crystalline Form B + Crystalline Form A, crash cooling in IPA, solids precipitate in dry ice/IPA

Figure 13F – Crystalline Form A + Crystalline Form C, slow cooling in IPA, solids precipitate in freezer

Figure 13G – Form C, slow cooling in IPA

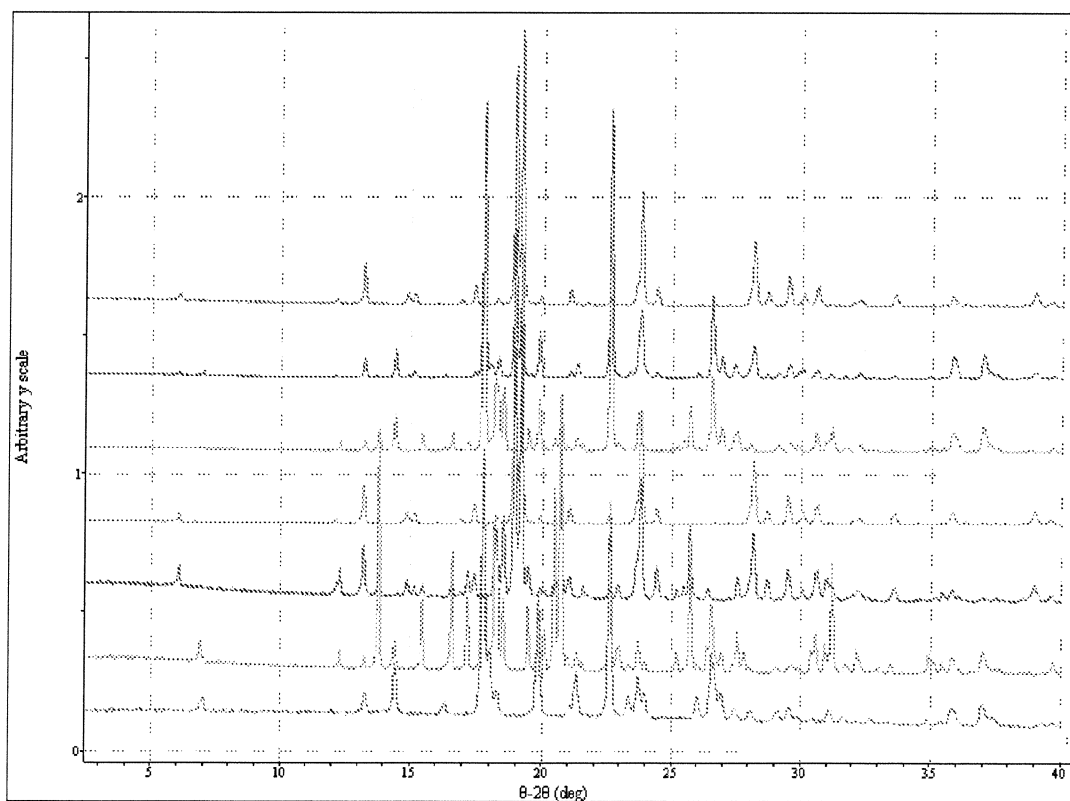
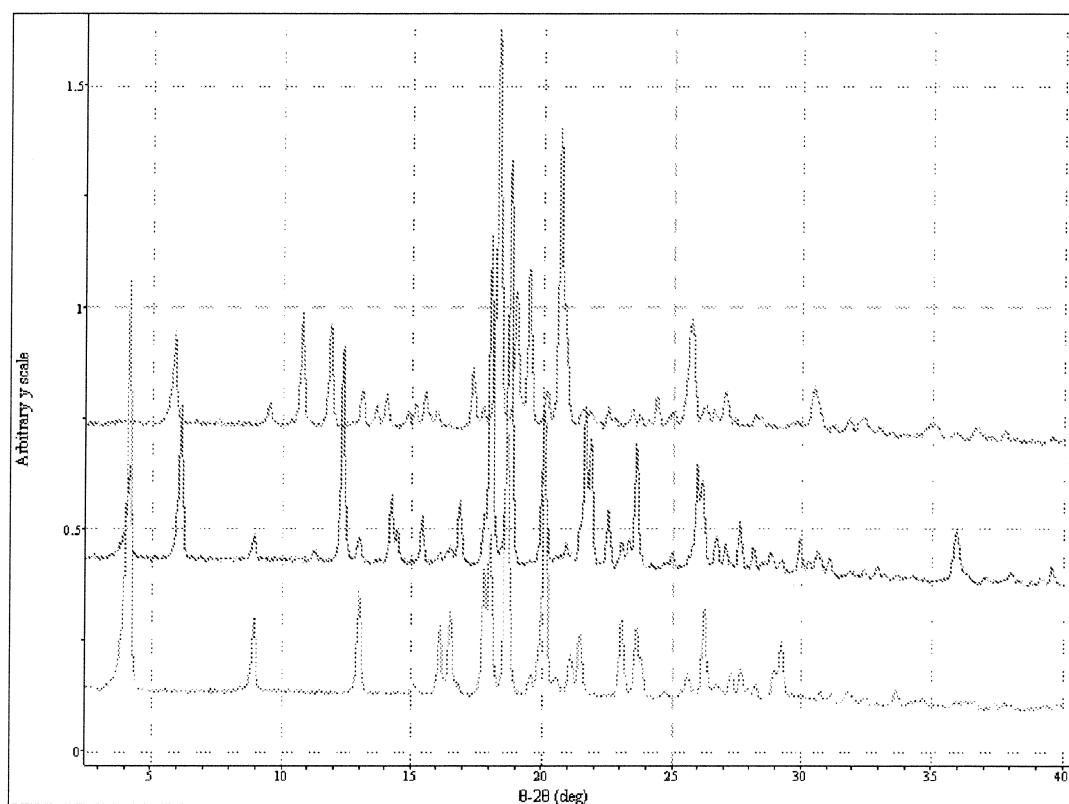


Figure 14 – Overlay of X-ray powder diffraction patterns of Crystalline Form D, Form E, and Form F (top to bottom):

Figure 14D – Crystalline Form D, 30-min stir at 70 °C in pH 4.4 buffer

Figure 14E – Crystalline Form E (contains peaks of Crystalline Form F), slurry at 50 °C in pH 6.0 buffer

Figure 14F – Crystalline Form F, 30-min stir at 70 °C in pH 8.1 buffer



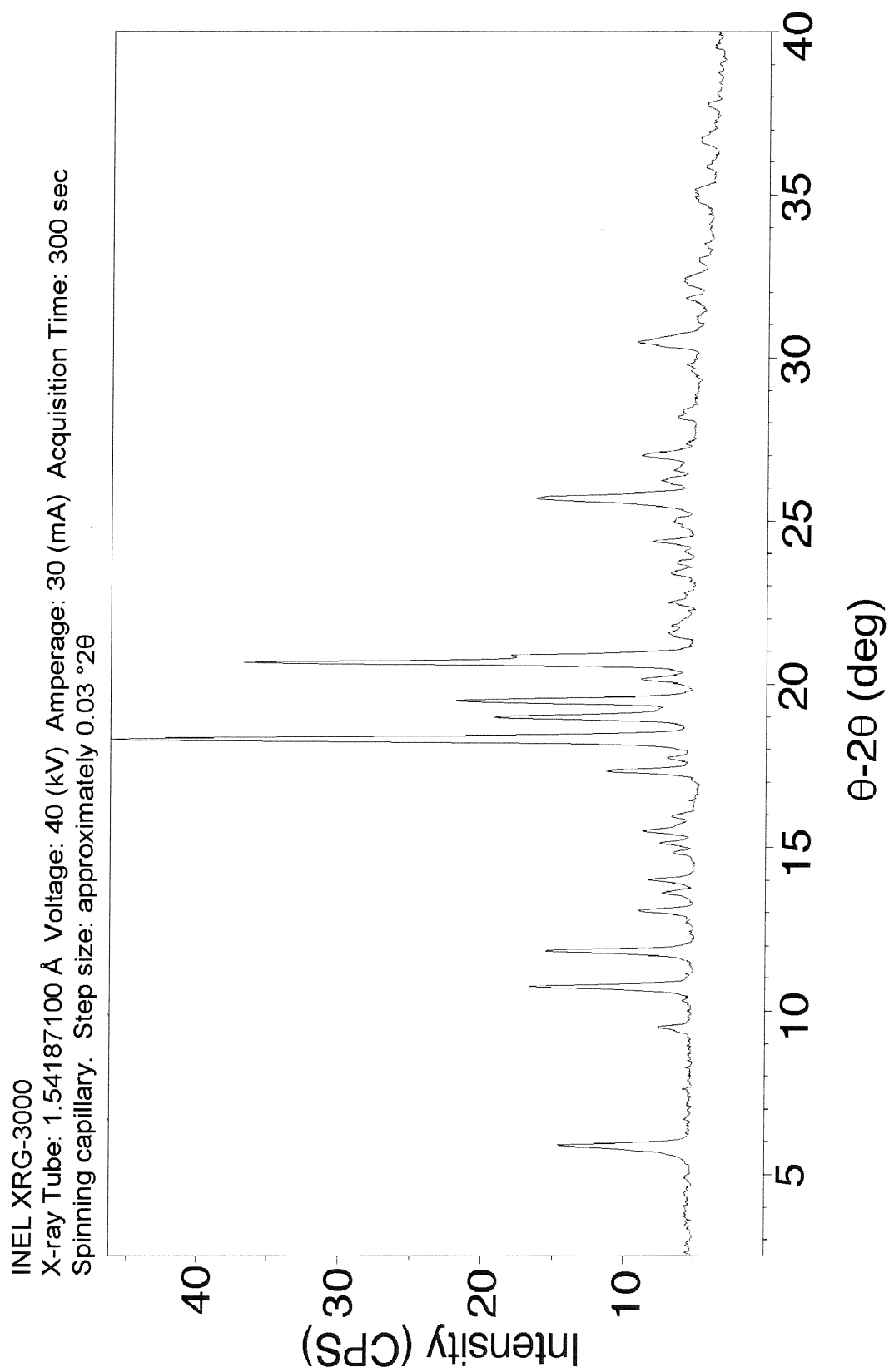


Figure 15 - XRPD pattern of Crystalline Form D

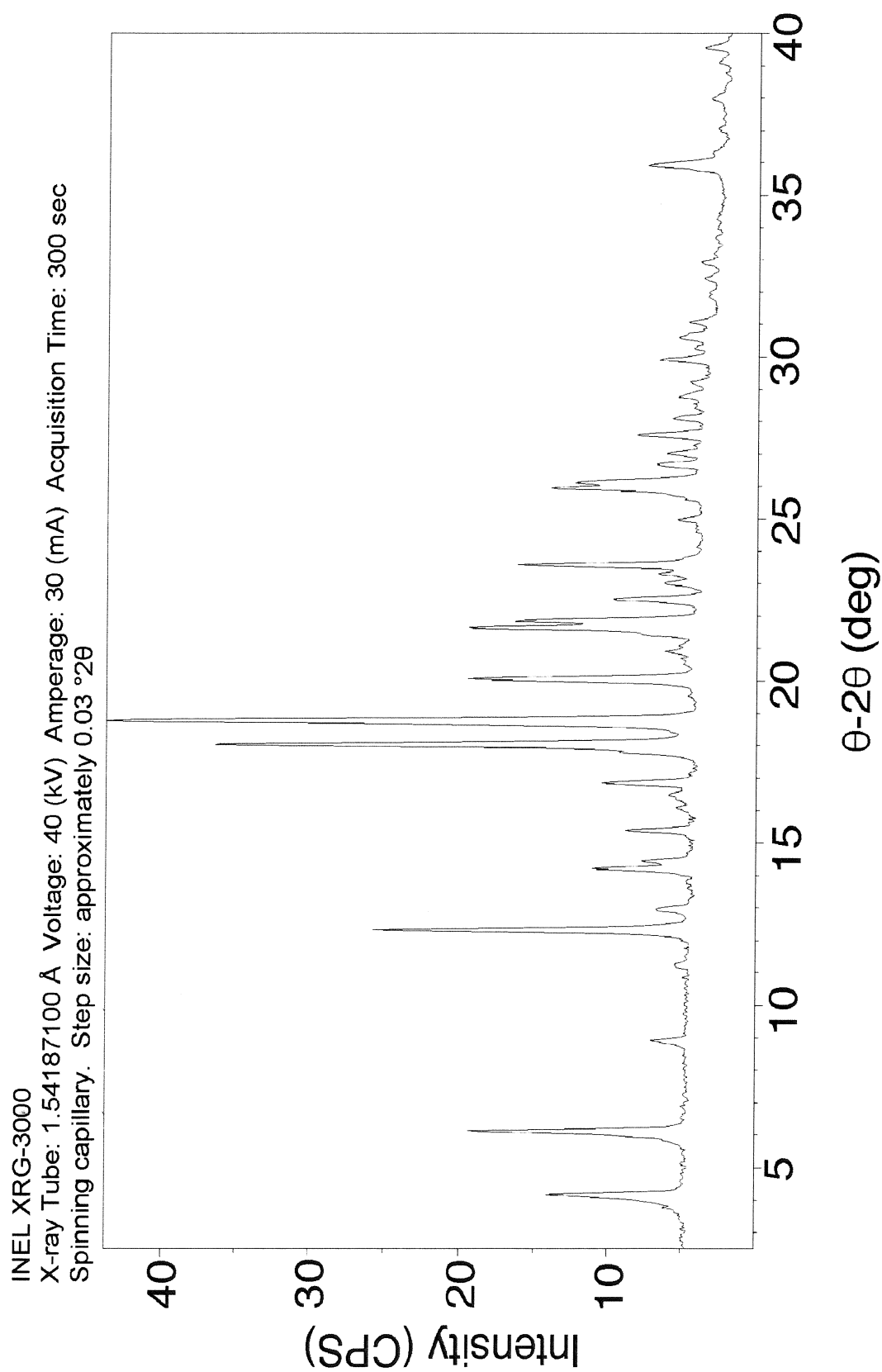


Figure 16 - XRPD pattern of Crystalline Form E (contains peaks of Crystalline Form F)

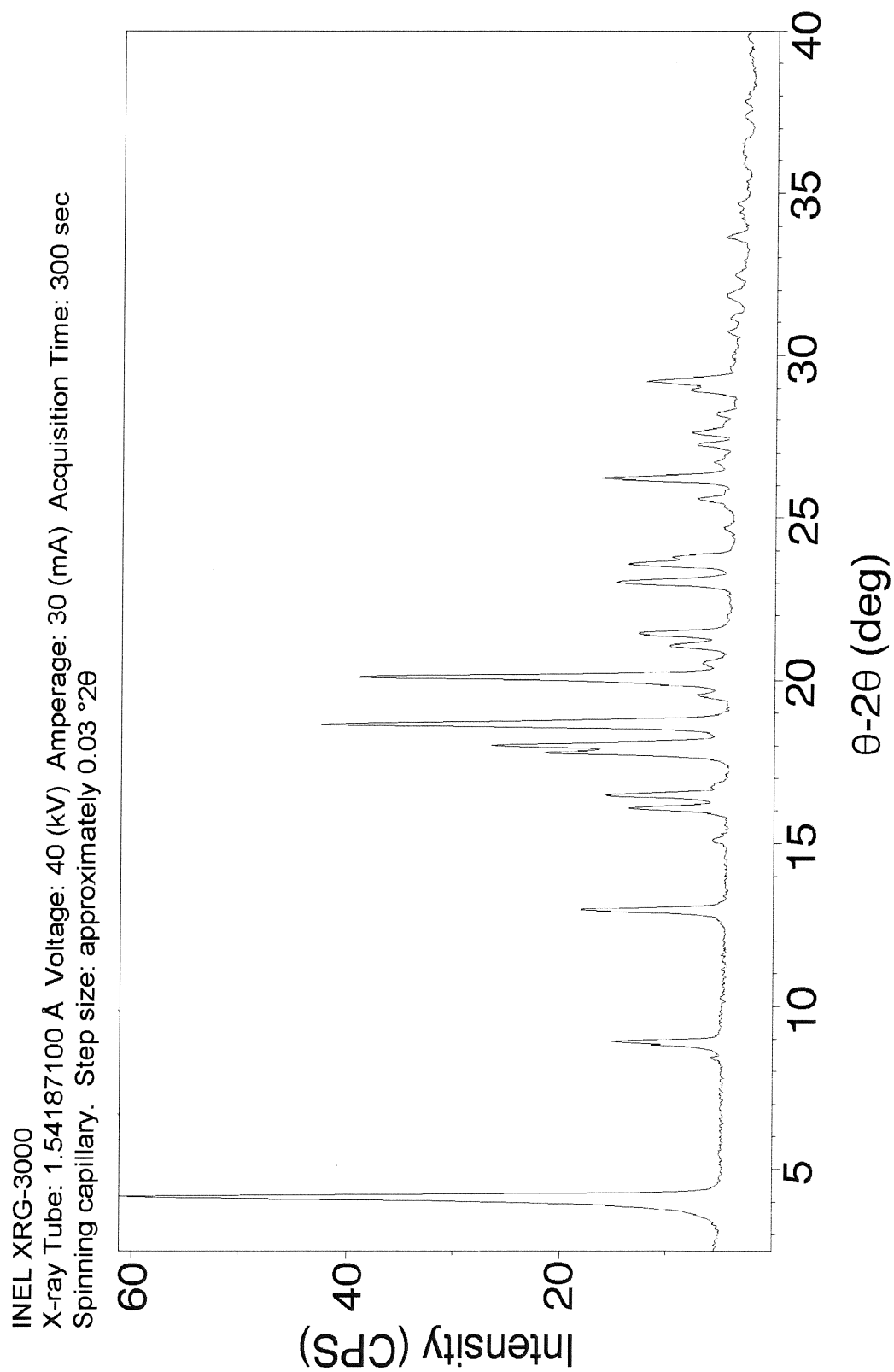


Figure 17 - XRPD pattern of Crystalline Form F

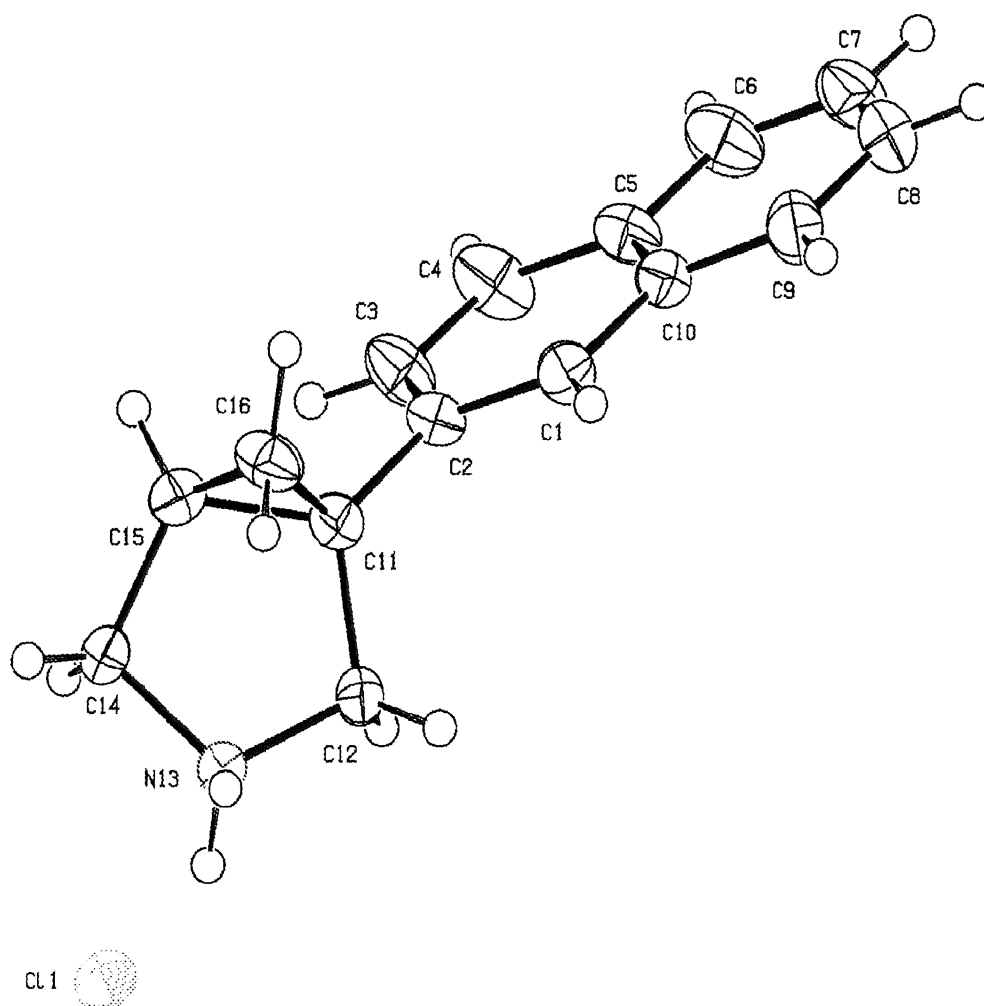


Figure 18 - ORTEP drawing of Crystalline Form A (atoms are represented by 50% probability anisotropic thermal ellipsoids)

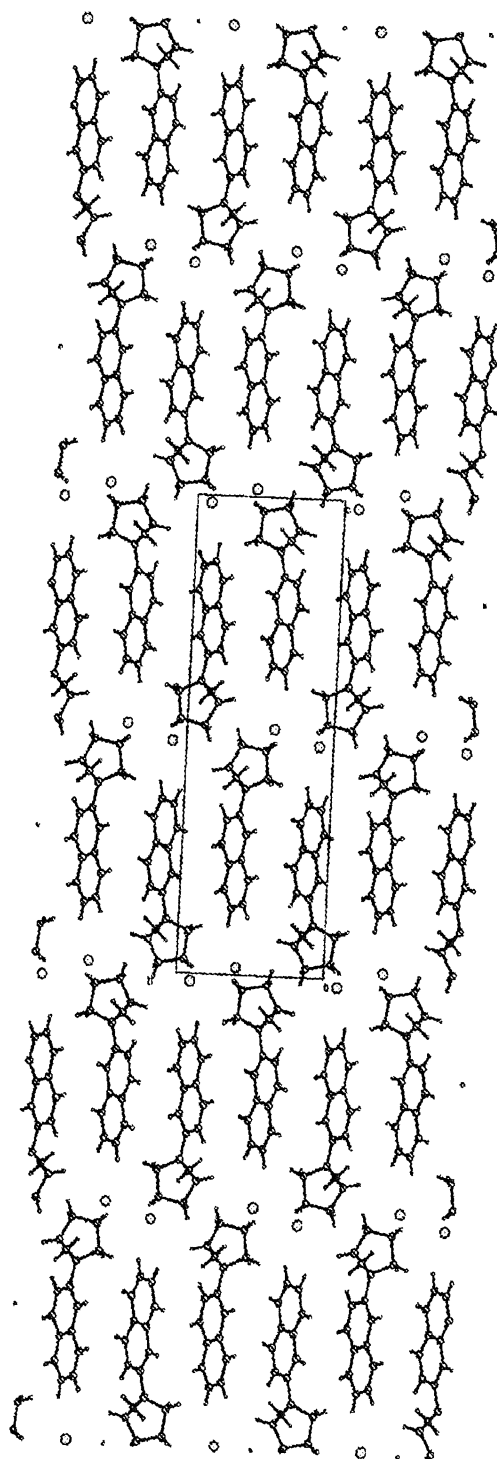


Figure 19 - Packing diagram of Crystalline Form A viewed down the crystallographic *a* axis

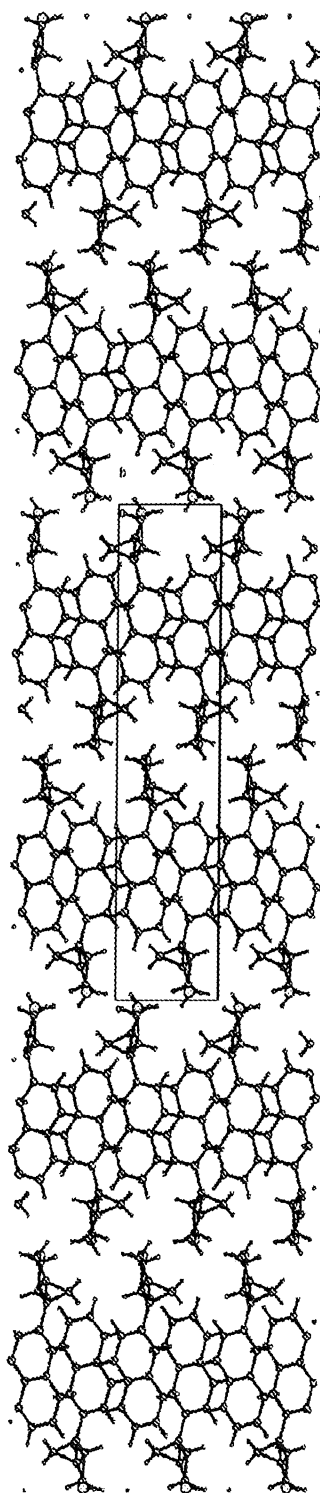


Figure 20 - Packing diagram of Crystalline Form A viewed down the crystallographic *b* axis

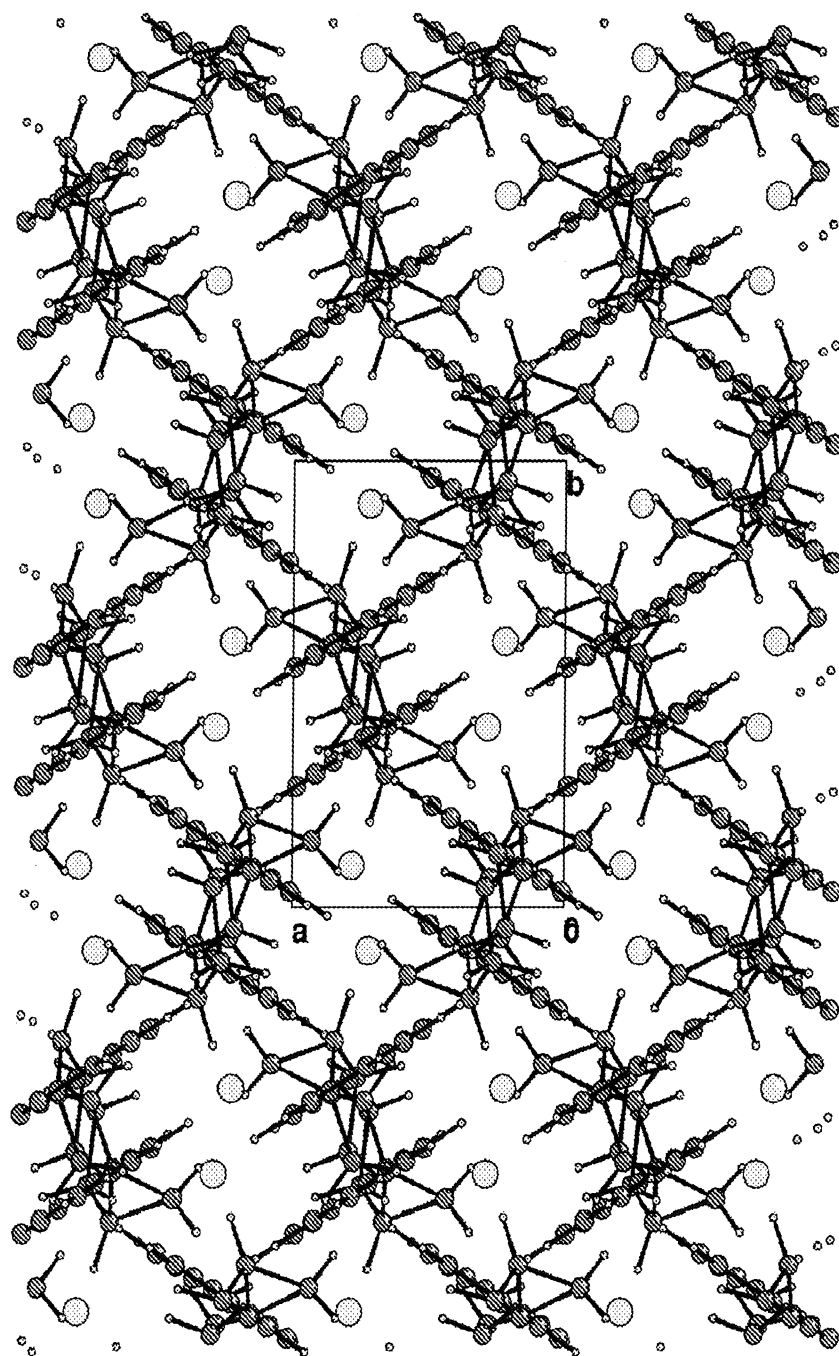


Figure 21 - Packing diagram of Crystalline Form A viewed down the crystallographic *c* axis

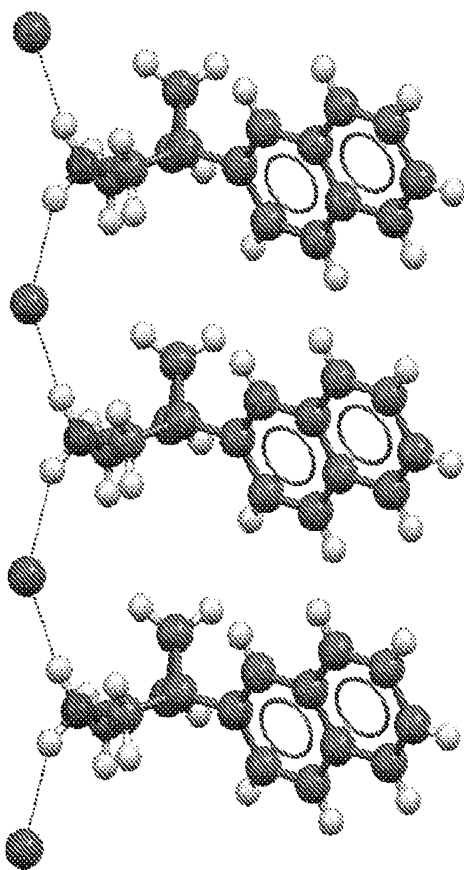


Figure 22 - Hydrogen bonding in Crystalline Form A

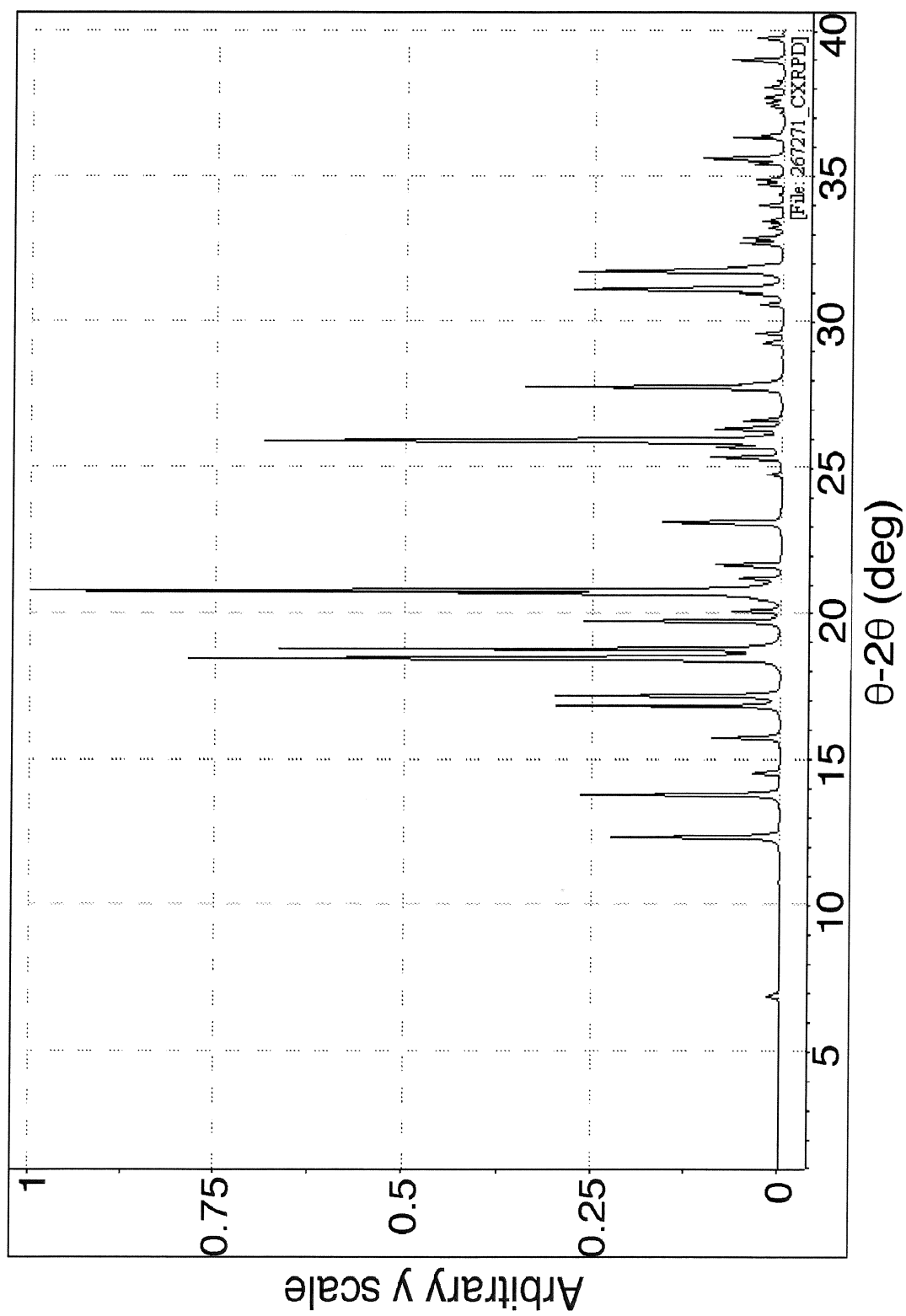


Figure 23 - Calculated XRPD pattern of Crystalline Form A

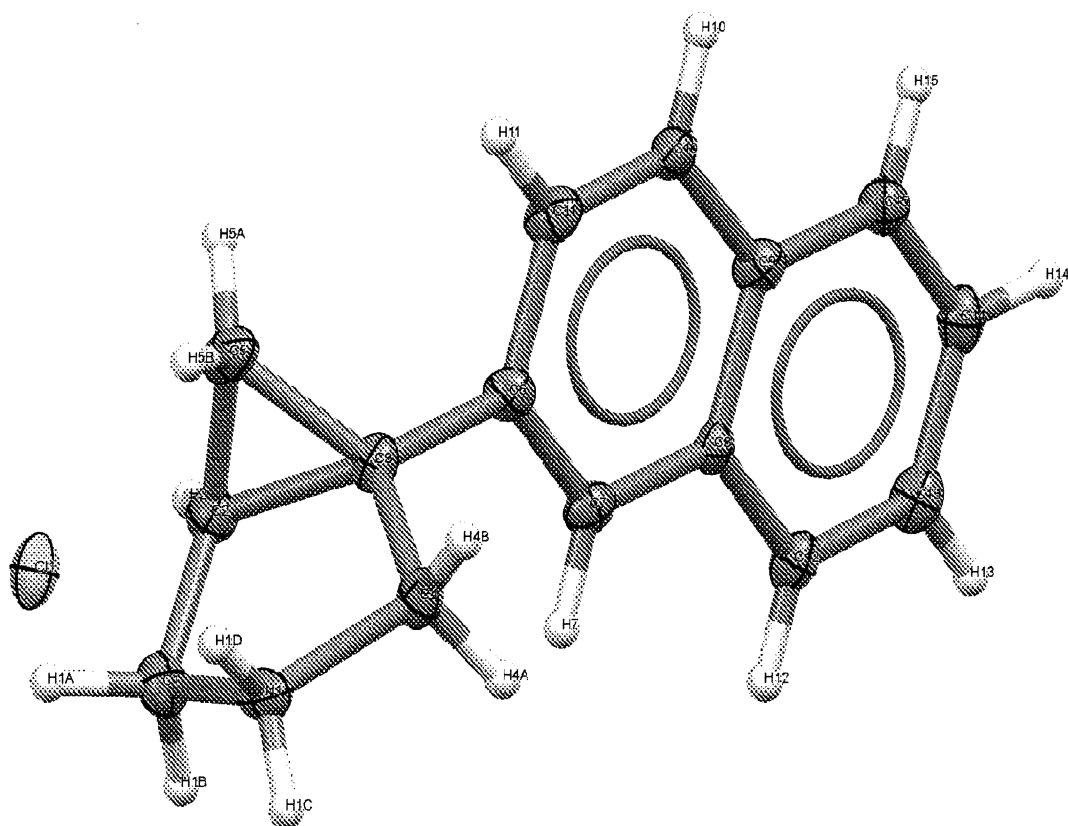


Figure 24 - Atomic displacement ellipsoid drawing for Crystalline Form B (atoms are represented by 50% probability anisotropic thermal ellipsoids)

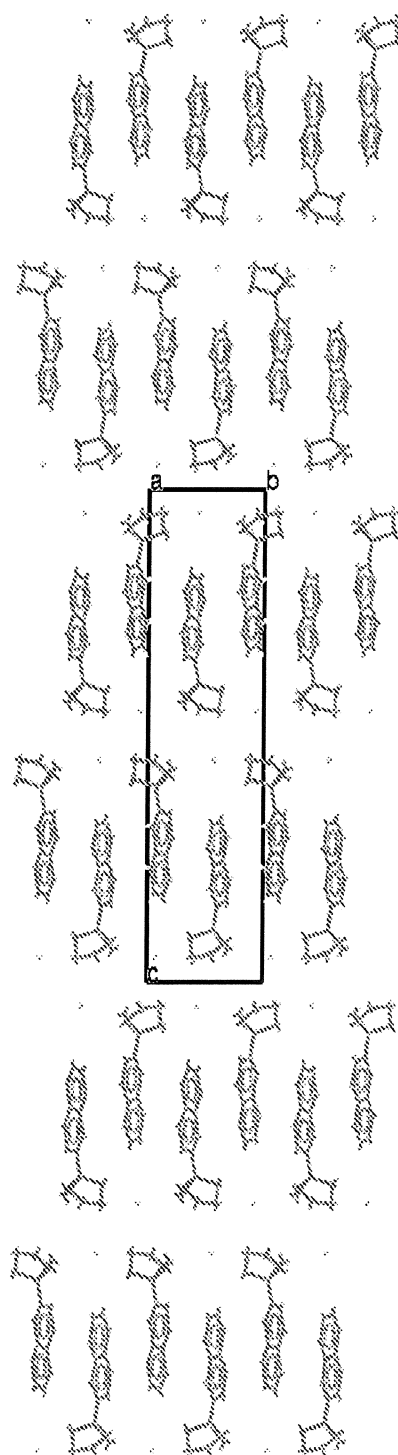


Figure 25 - Packing diagram of Crystalline Form B viewed along the crystallographic *a* axis

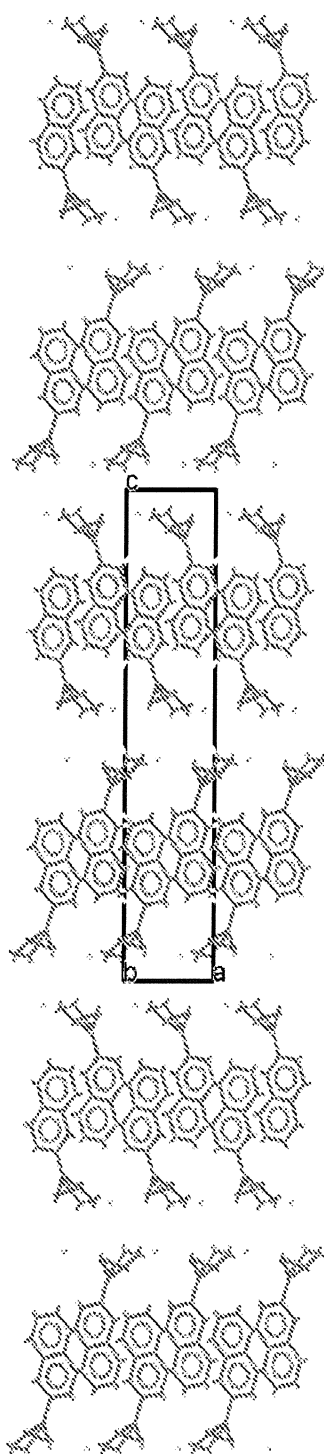


Figure 26 - Packing diagram of Crystalline Form B viewed along the crystallographic *b* axis

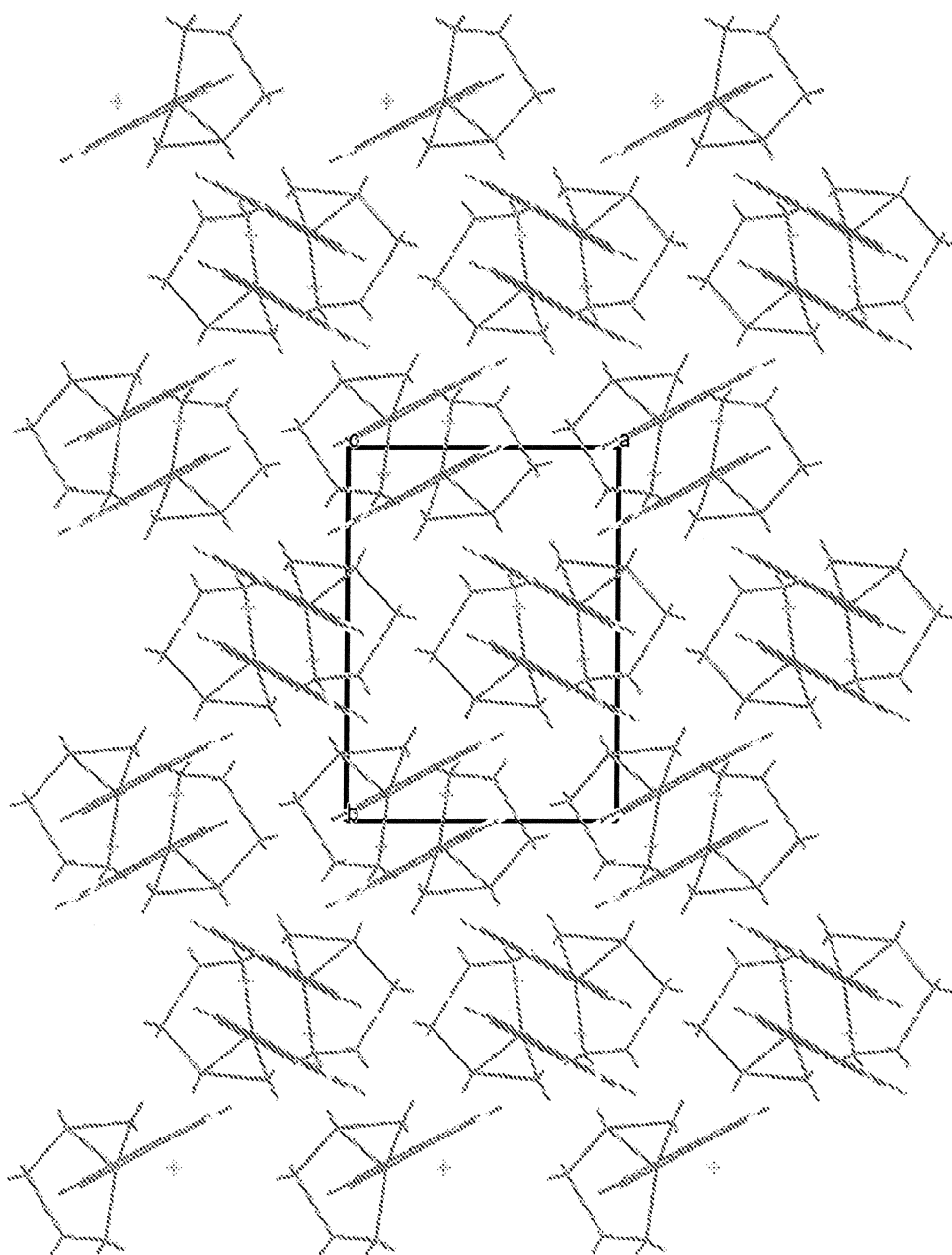


Figure 27 - Packing diagram of Crystalline Form B viewed along the crystallographic *c* axis

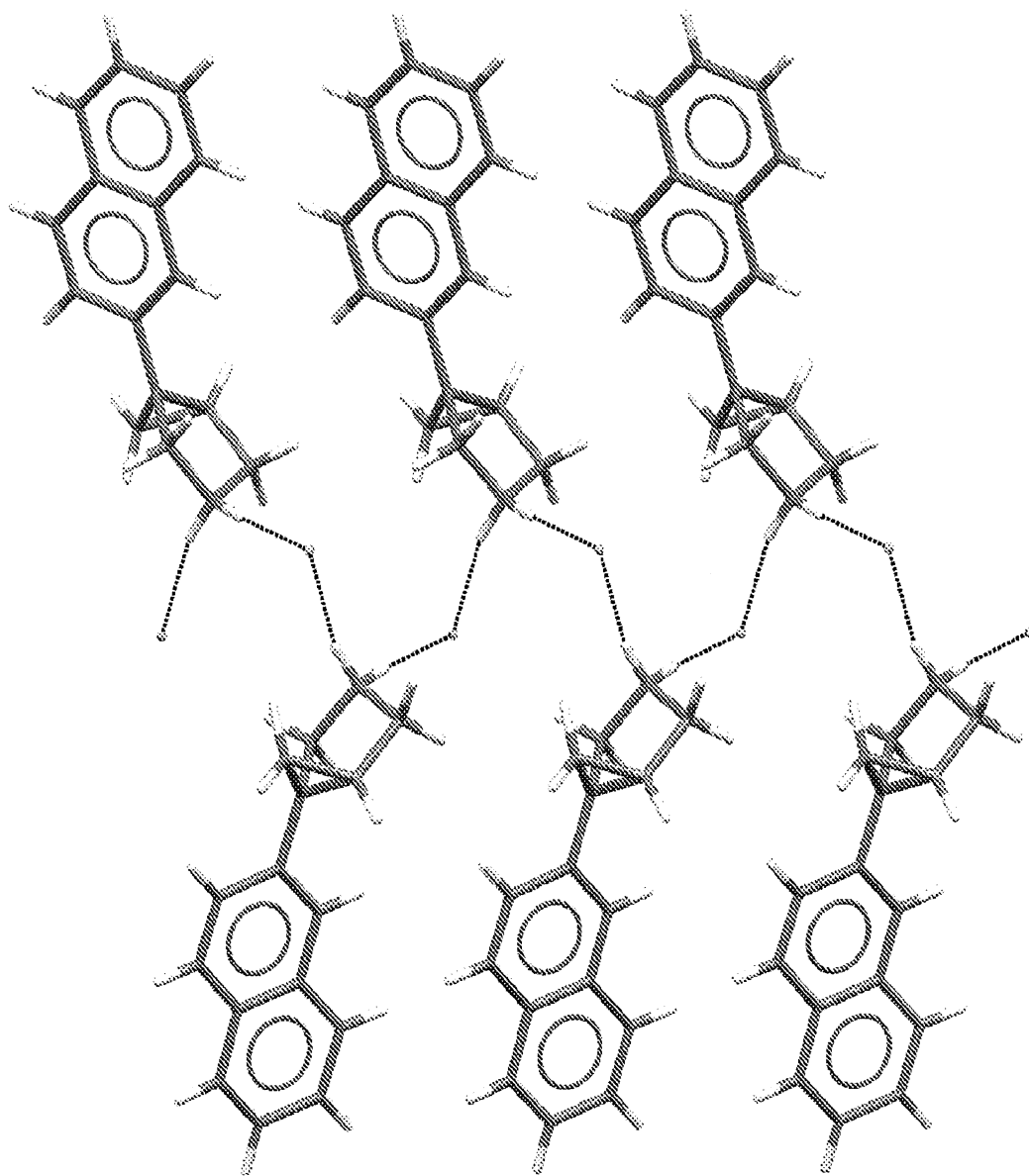


Figure 28 - Hydrogen bonding in the structure of Crystalline Form B

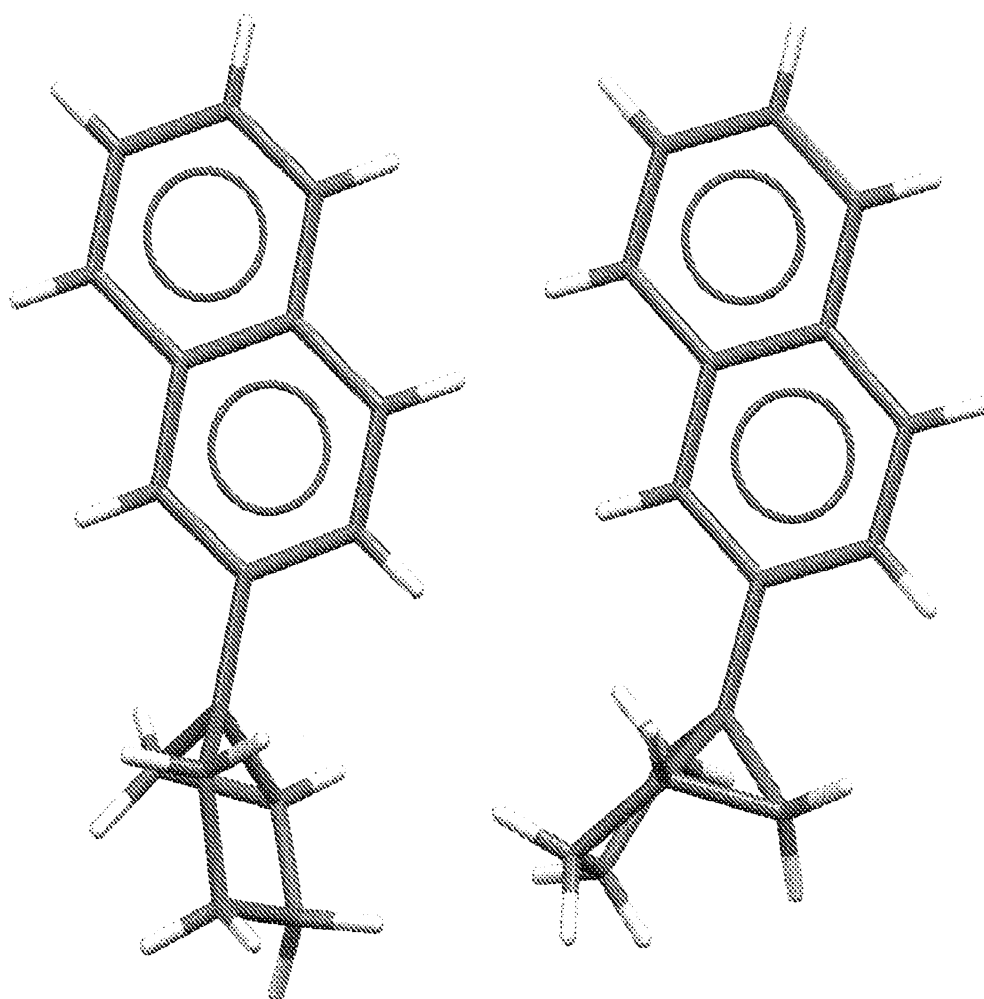


Figure 29 - The molecular conformations of (1R,5S)-1-(naphthalen-2-yl)-3-azabicyclo[3.1.0]hexane in the structures of Crystalline Forms A and B (left: (1R,5S)-1-(naphthalen-2-yl)-3-azabicyclo[3.1.0]hexane in the structure of Crystalline Form A; right: (1R,5S)-1-(naphthalen-2-yl)-3-azabicyclo[3.1.0]hexane in the structure of Crystalline Form B)

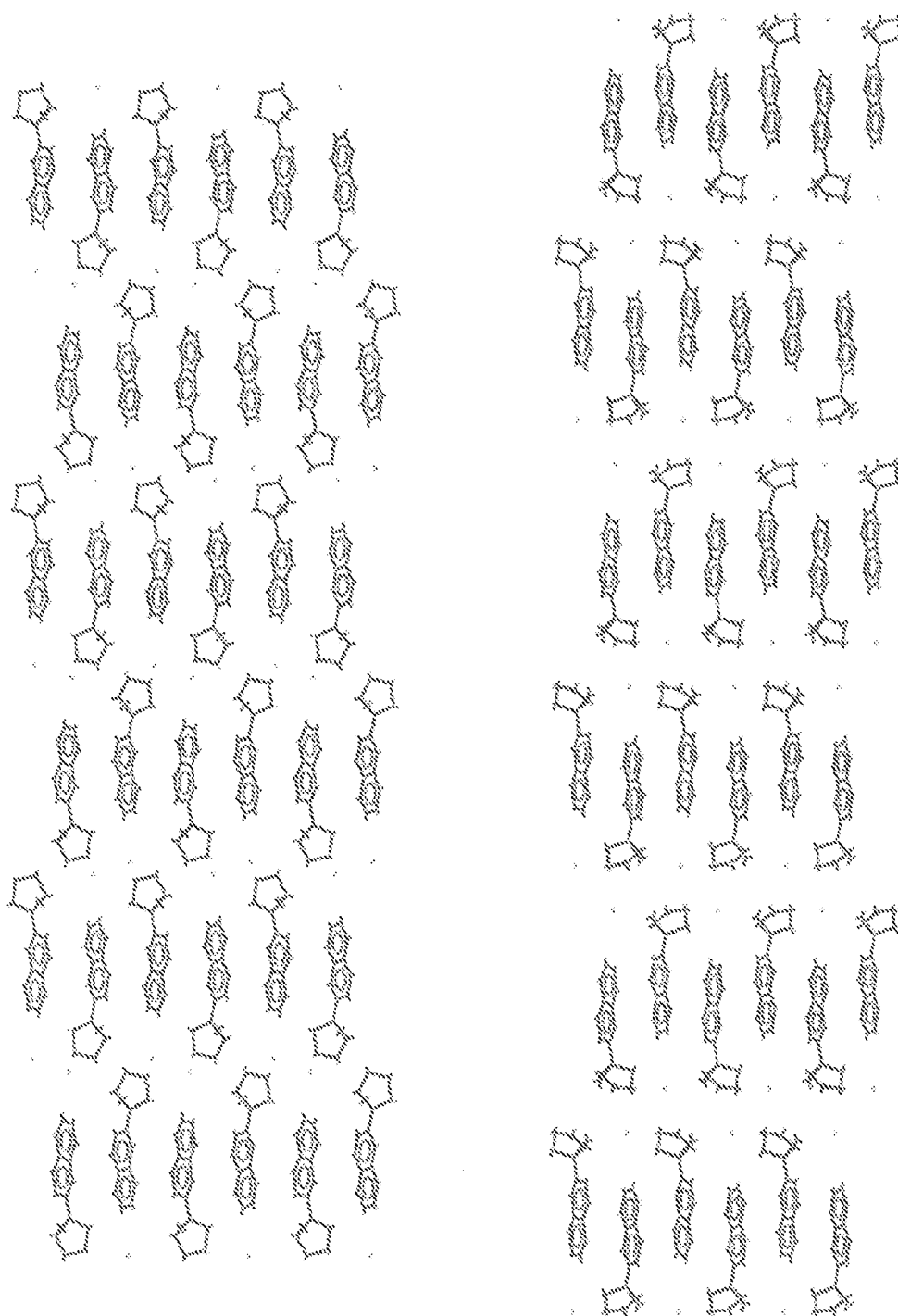


Figure 30 - Packing diagram of Crystalline Forms A and B viewed along the crystallographic a axis (left: packing of Crystalline Form A; right: packing of Crystalline Form B)

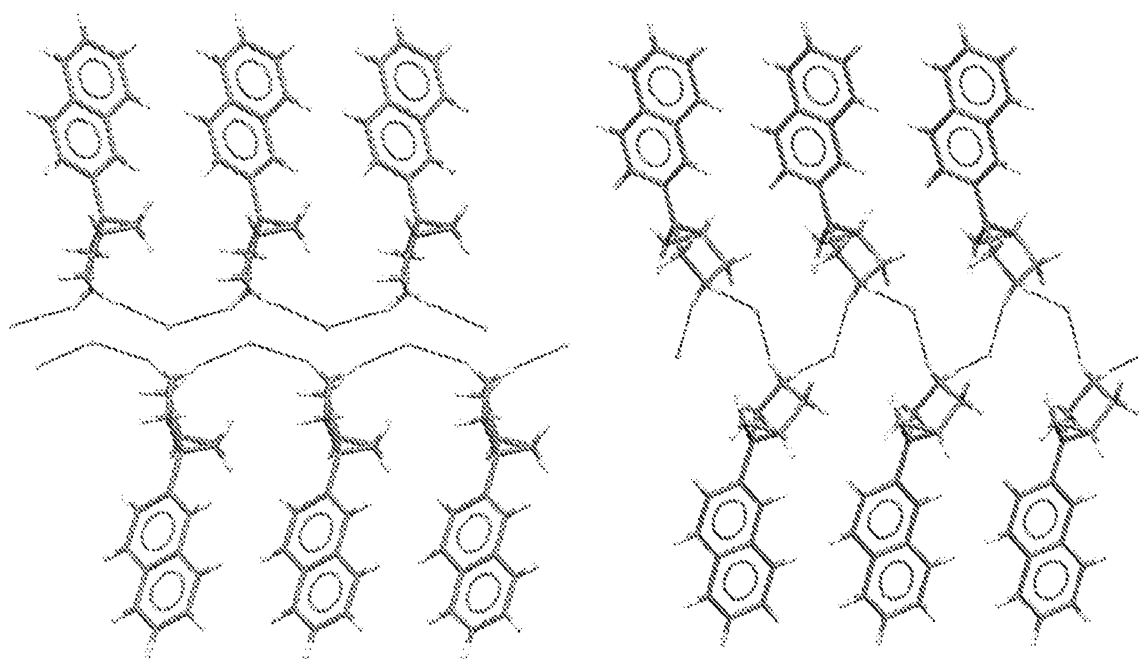


Figure 31 - Hydrogen bonding in the structures of Crystalline Forms A and B (left: hydrogen bonding in the structure of Crystalline Form A; right: hydrogen bonding in the structure of Crystalline Form B)

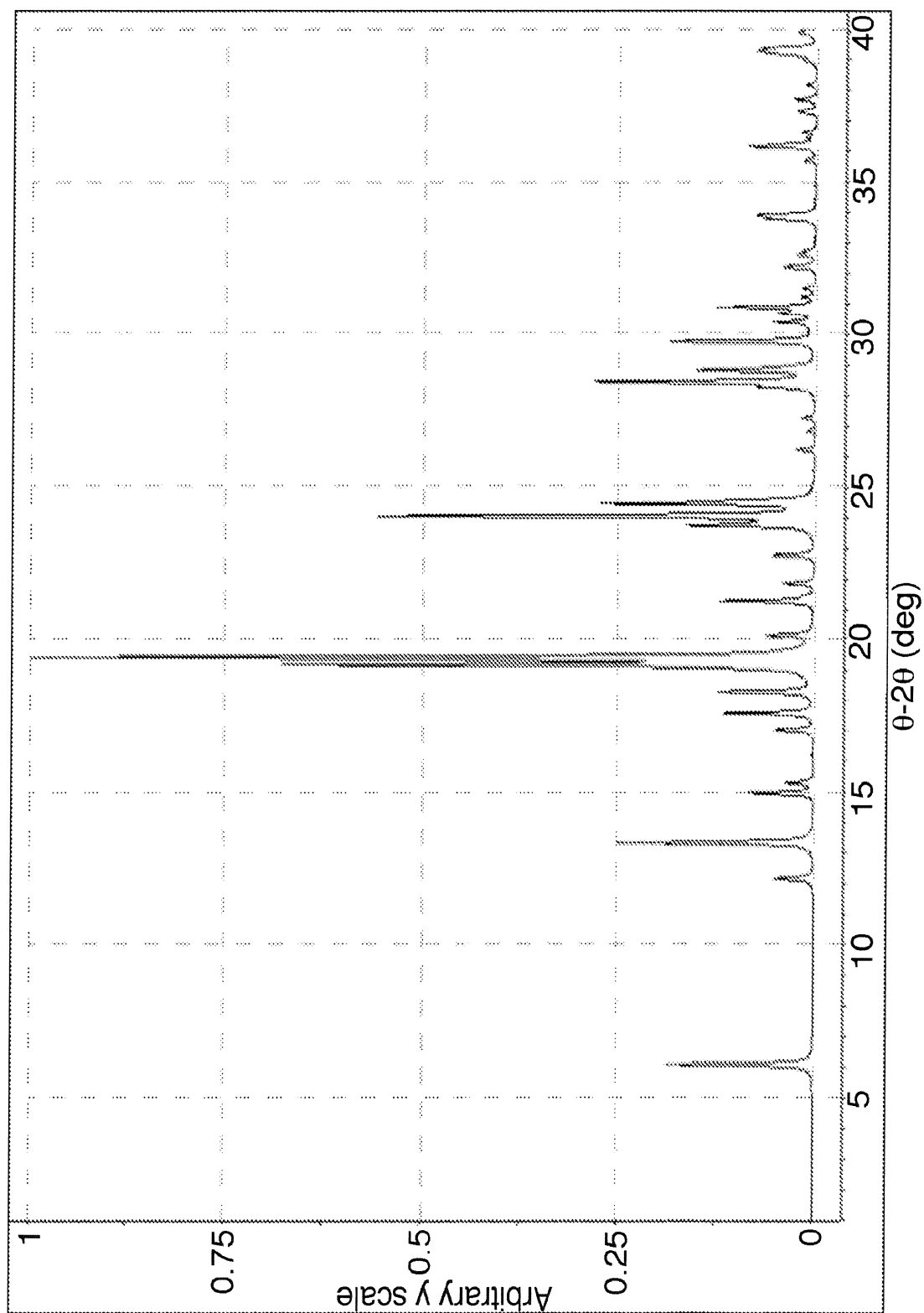


Figure 32 - Calculated X-ray powder pattern of Crystalline Form B

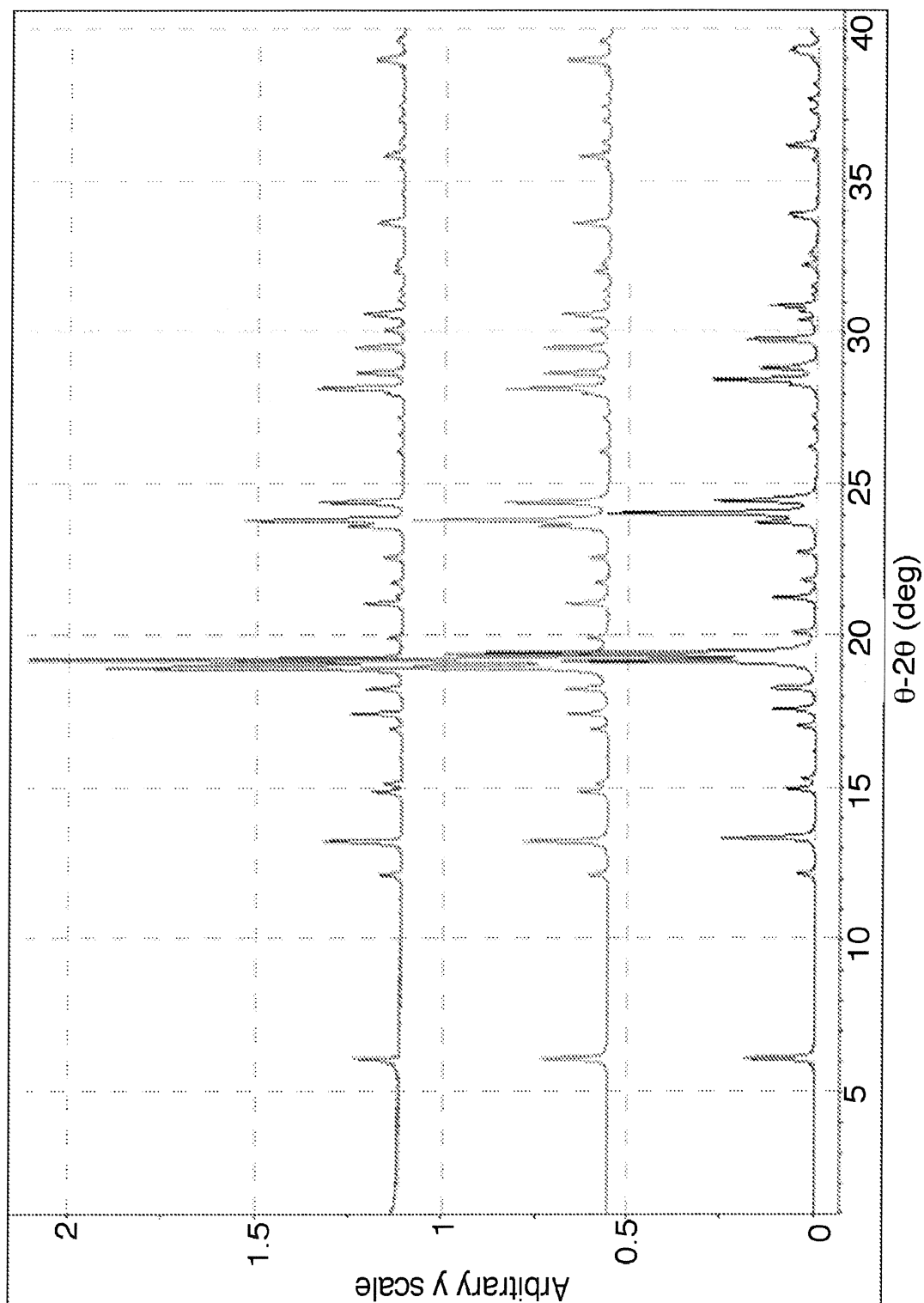


Figure 33 - Experimental and calculated XRPD patterns of Crystalline Form B (top: experimental XRPD pattern at room temperature; middle: calculated XRPD pattern adjusted to room temperature; bottom: calculated XRPD pattern at 100 K)

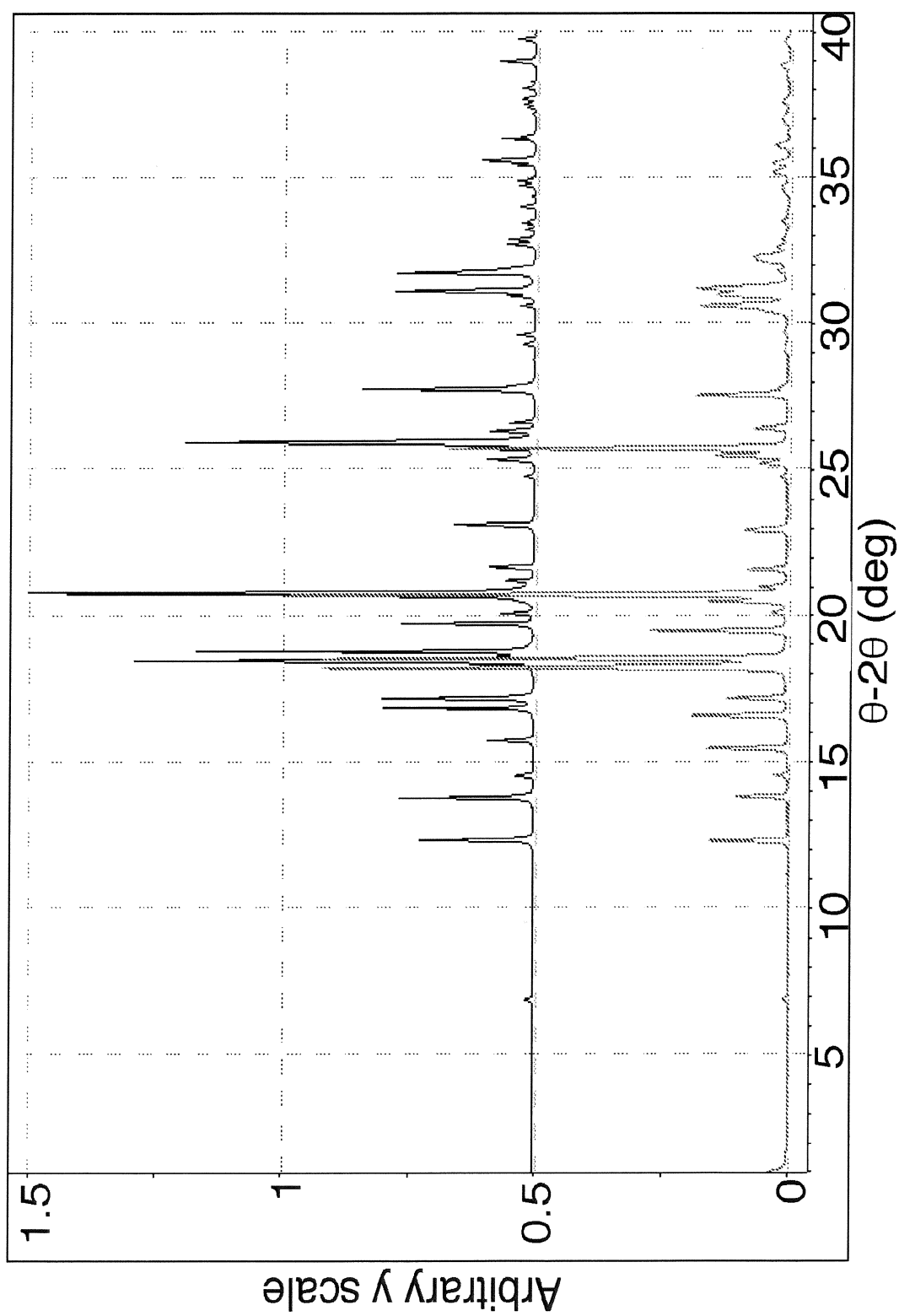


Figure 34 - Experimental and calculated XRPD patterns of Crystalline Form A (top: calculated XRPD pattern; bottom: experimental XRPD pattern at room temperature)

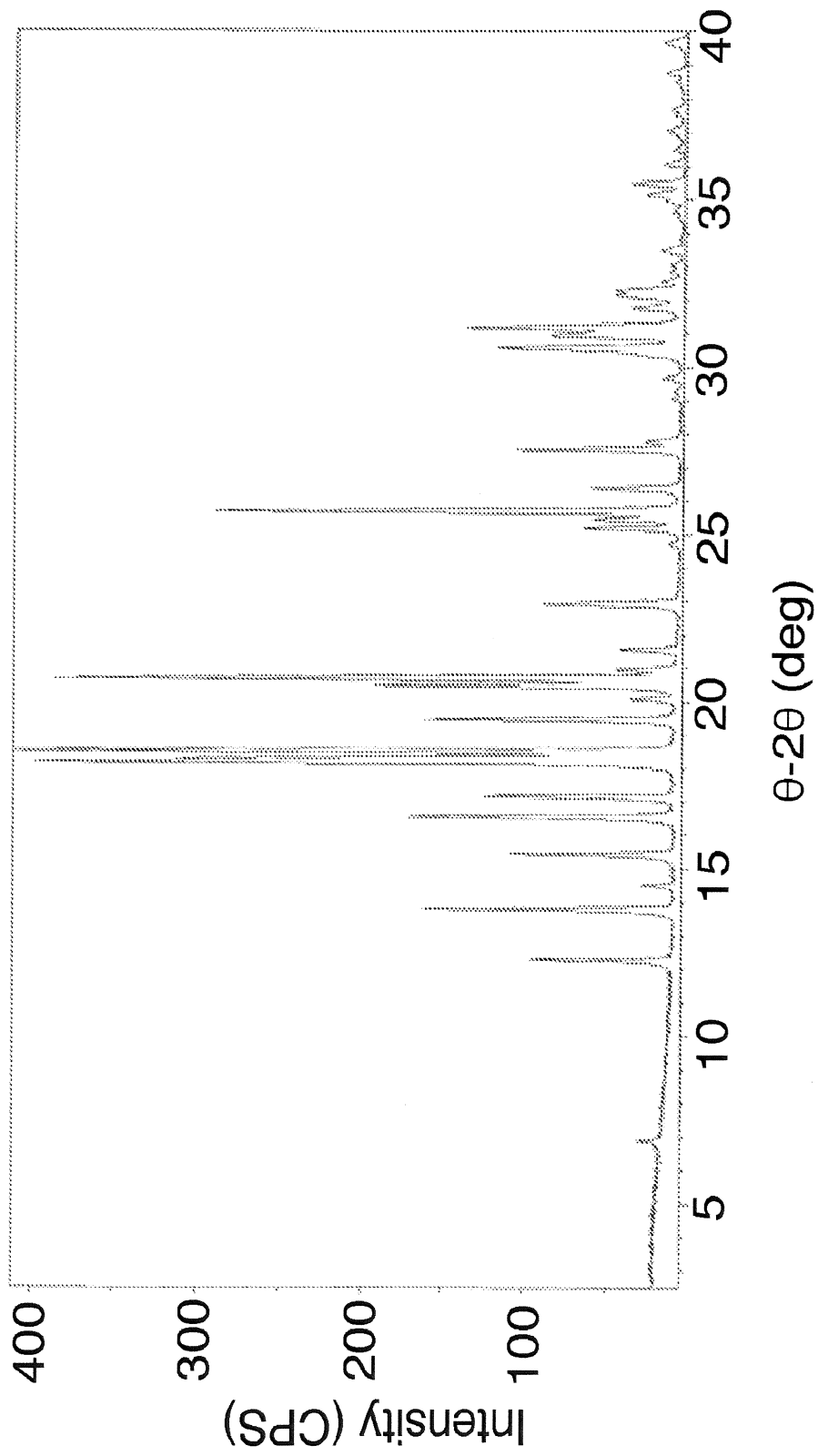


Figure 35 - XRPD pattern of Crystalline Form A

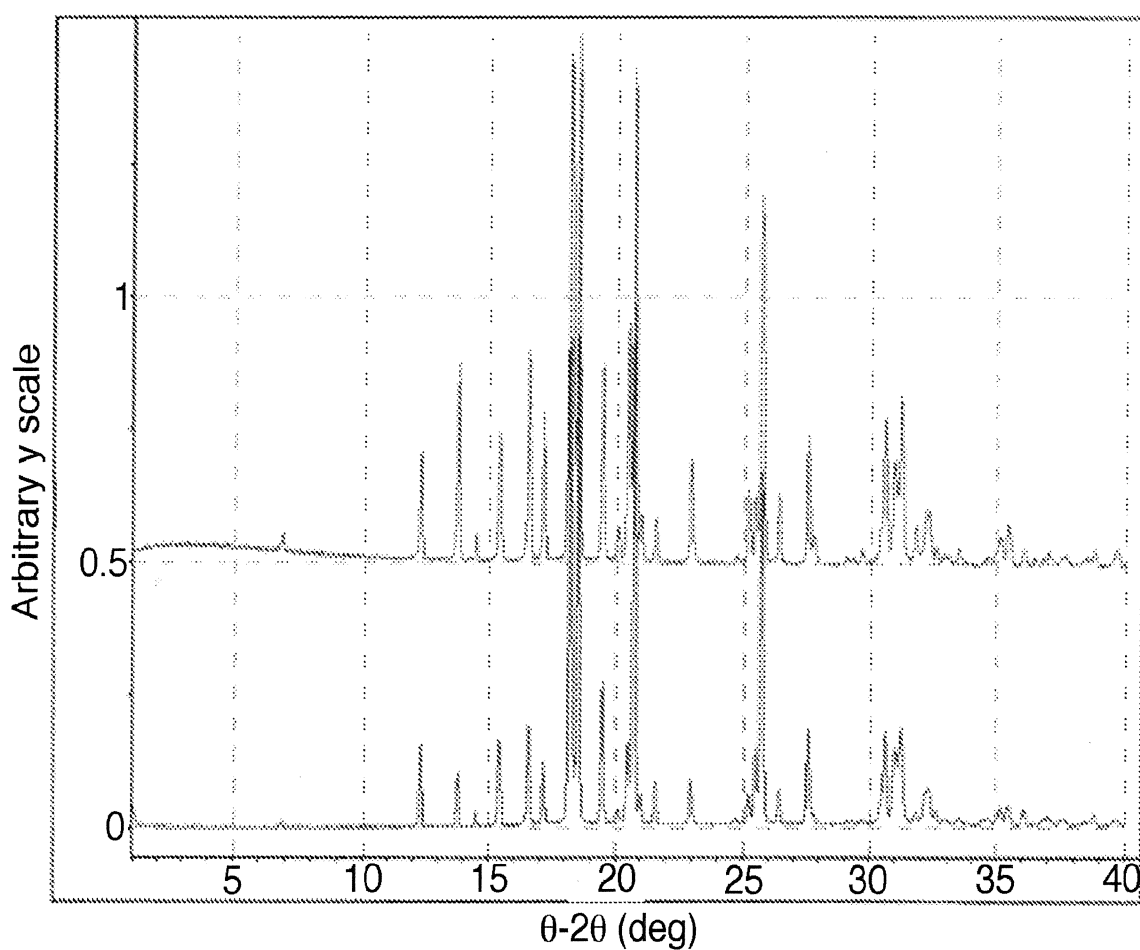


Figure 36 - XRPD pattern comparison of (1R,5S)-1-(naphthalen-2-yl)-3-azabicyclo[3.1.0]hexane hydrochloride from Examples 1 and 5 (top: Example 5; bottom: Example 1) (patterns are offset along the y-axis for comparison)

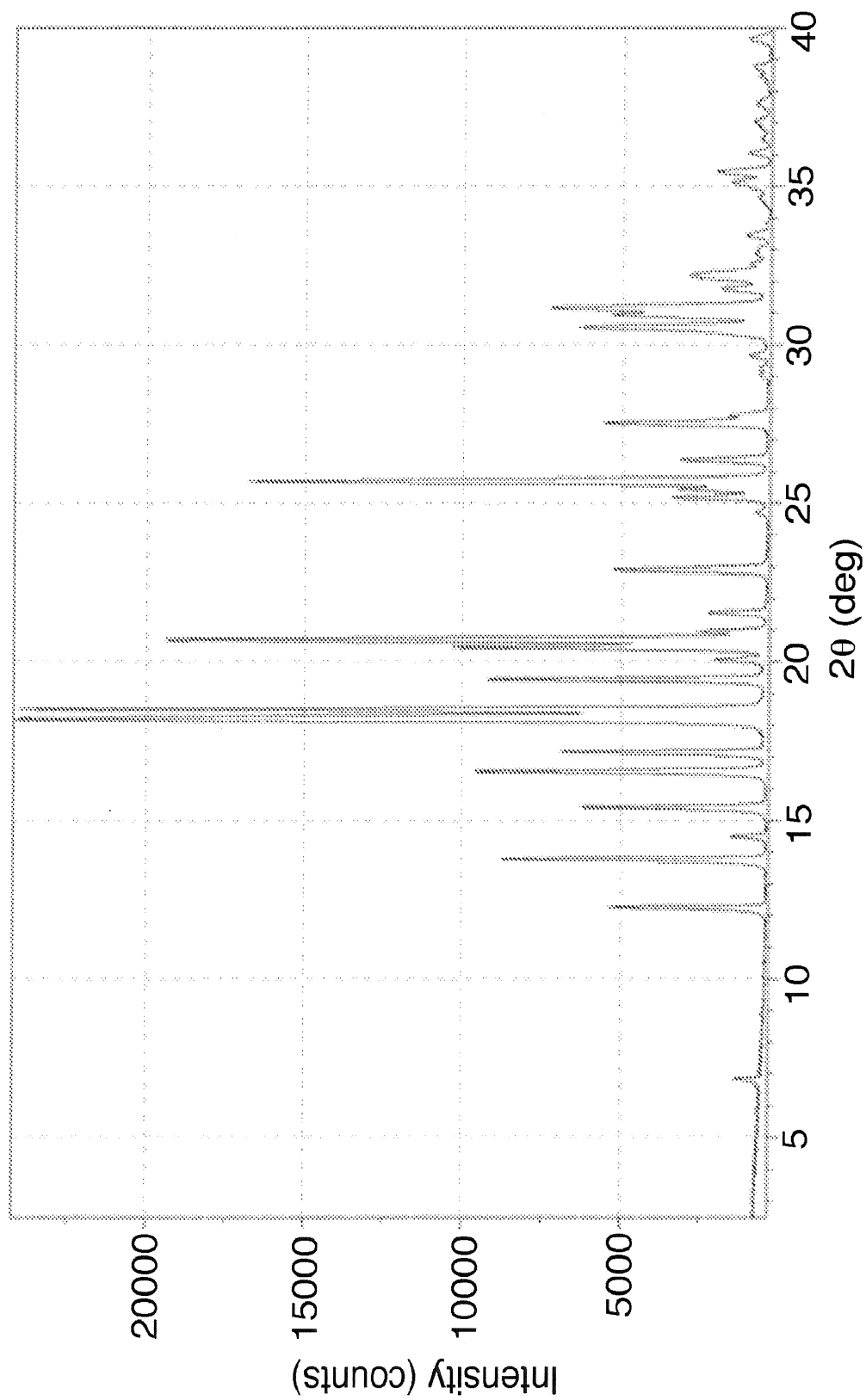
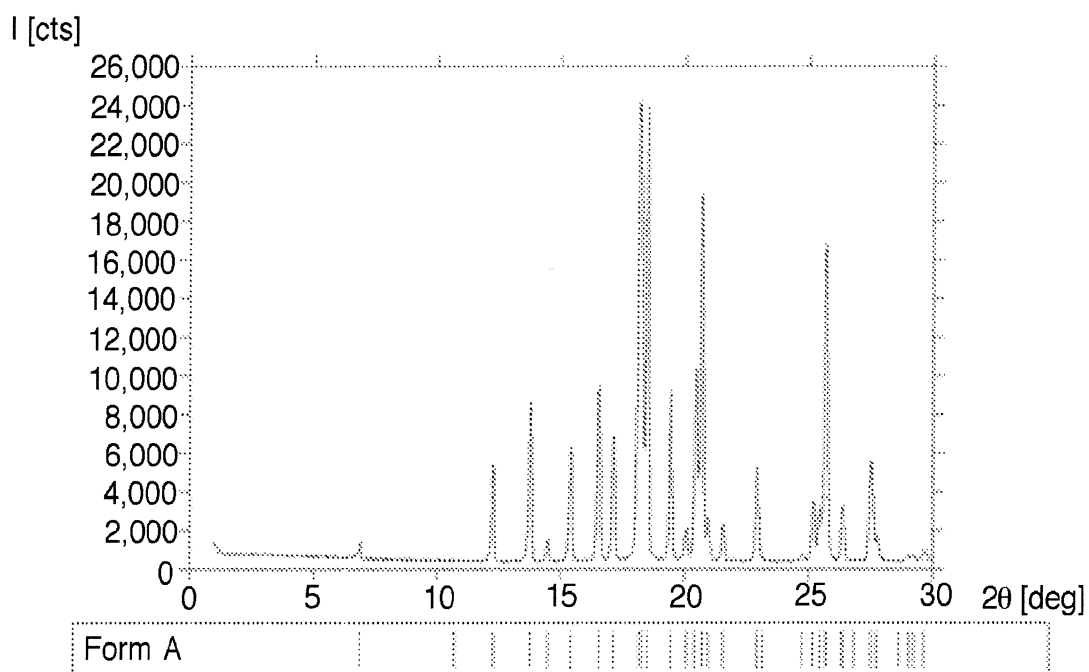


Figure 37 - XRPD pattern of Crystalline Form A collected with Cu K α radiation



Form A	
Bravais Type	Primitive Orthorhombic
a [Å]	5.886
b [Å]	8.704
c [Å]	25.684
α [deg]	90
β [deg]	90
γ [deg]	90
Volume [Å ³ /cell]	1,315.8
Chiral Contents?	Chiral
Extinction Symbol	P 2 ₁ 2 ₁ 2 ₁
Space Group(s)	P2 ₁ 2 ₁ 2 ₁ (19)

Source

Manual Input

Figure 38 - Indexing result for the XRPD depicted in Figure 37 collected with Cu K α radiation

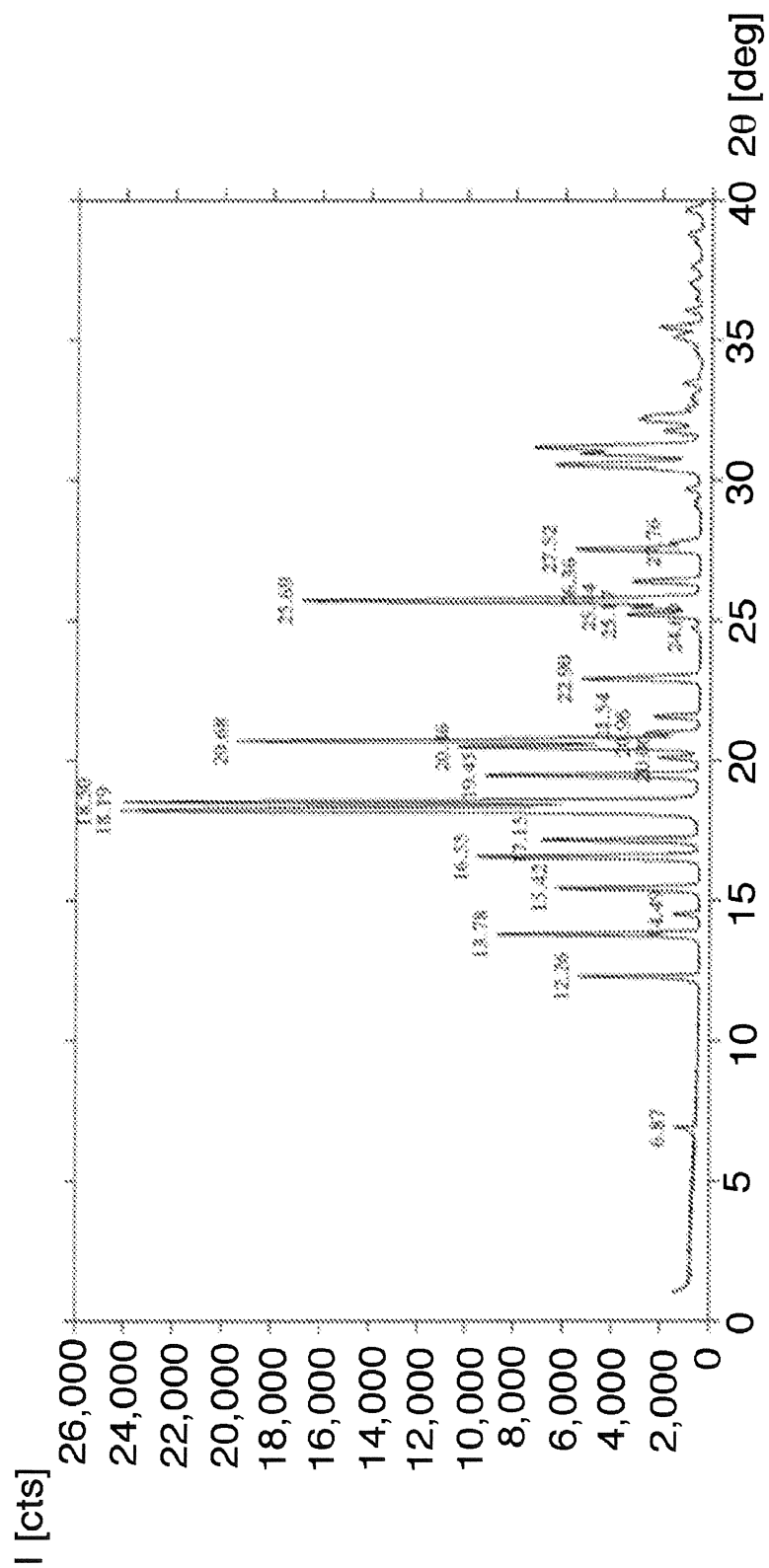


Figure 39 - Observed peaks for the XRPD pattern depicted in
Figure 37 collected with Cu K α radiation

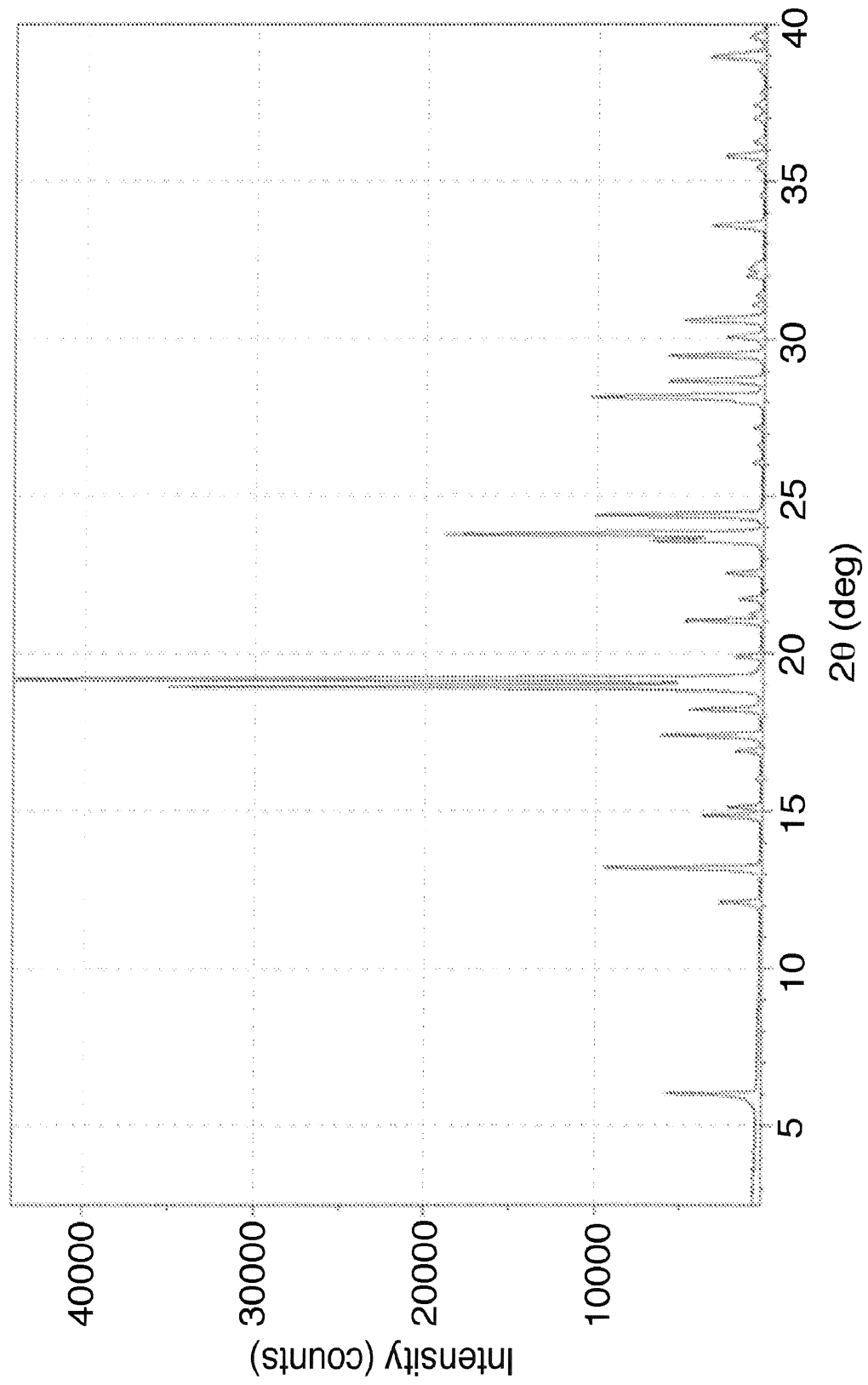
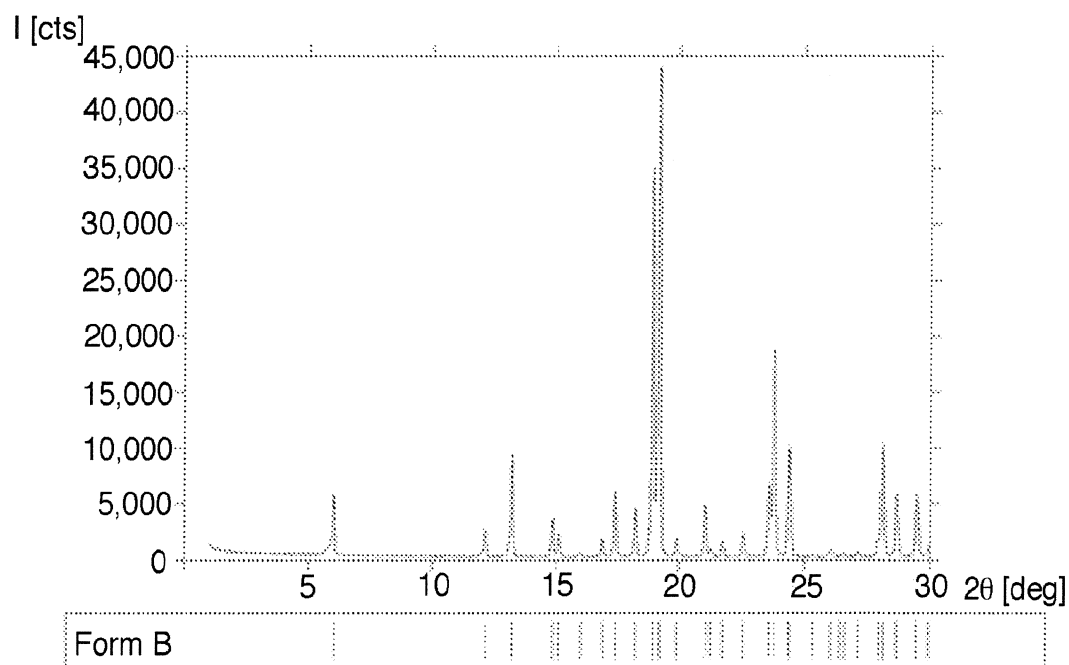


Figure 40 - XRPD pattern of Crystalline Form B



Form B	
Bravais Type	Primitive Orthorhombic
a [Å]	5.973
b [Å]	7.537
c [Å]	29.168
α [deg]	90
β [deg]	90
γ [deg]	90
Volume [Å ³ /cell]	1,313.1
Chiral Contents?	Chiral
Extinction Symbol	P 2 ₁ 2 ₁ 2 ₁
Space Group(s)	P2 ₁ 2 ₁ 2 ₁ (19)

Source

Manual Input

Figure 41 - Indexing result for the XRPD pattern depicted in Figure 40 collected with Cu K α radiation

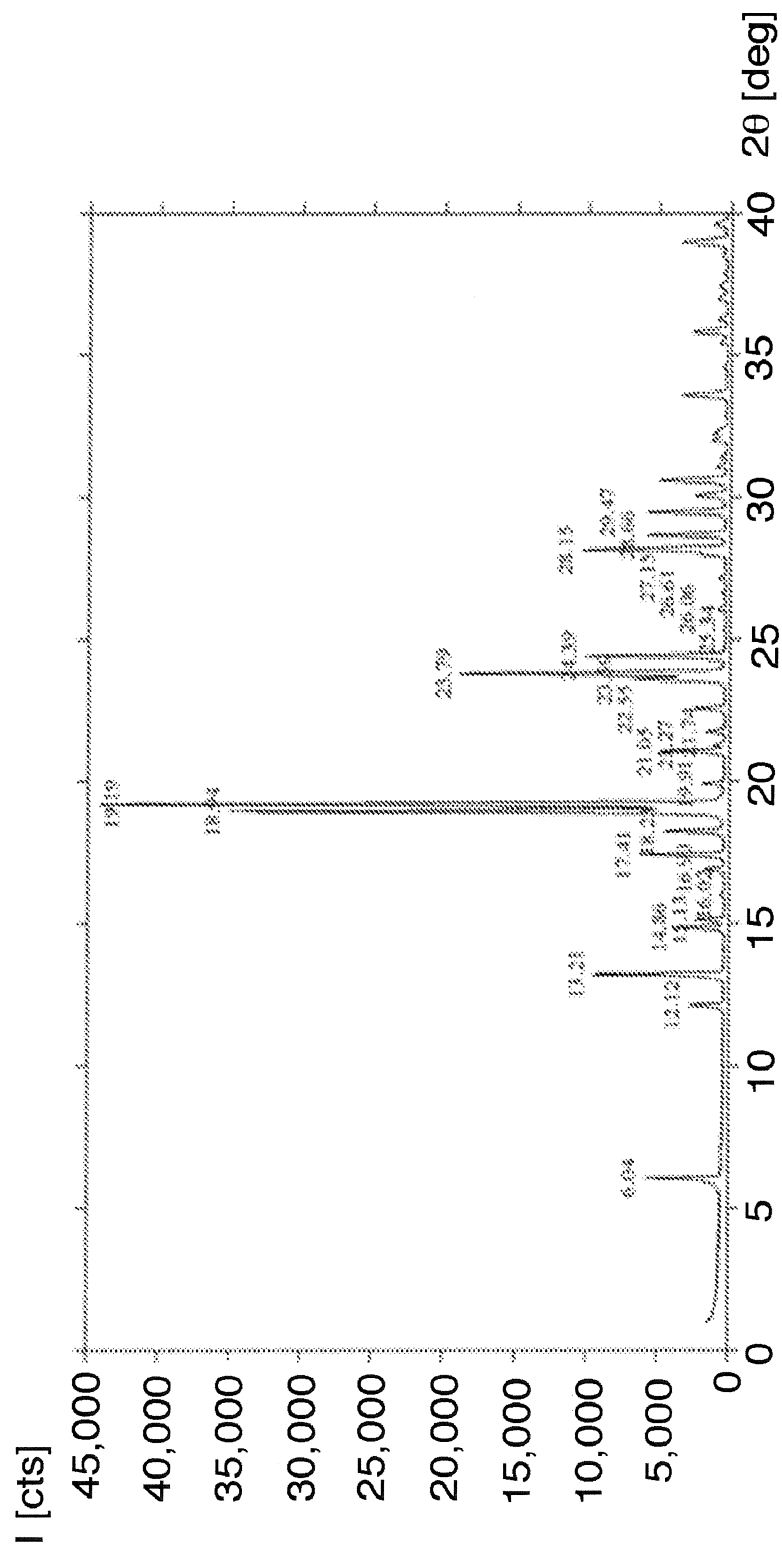


Figure 42 - Observed peaks for the XRPD pattern depicted in Figure 40 collected with Cu $K\alpha$ radiation

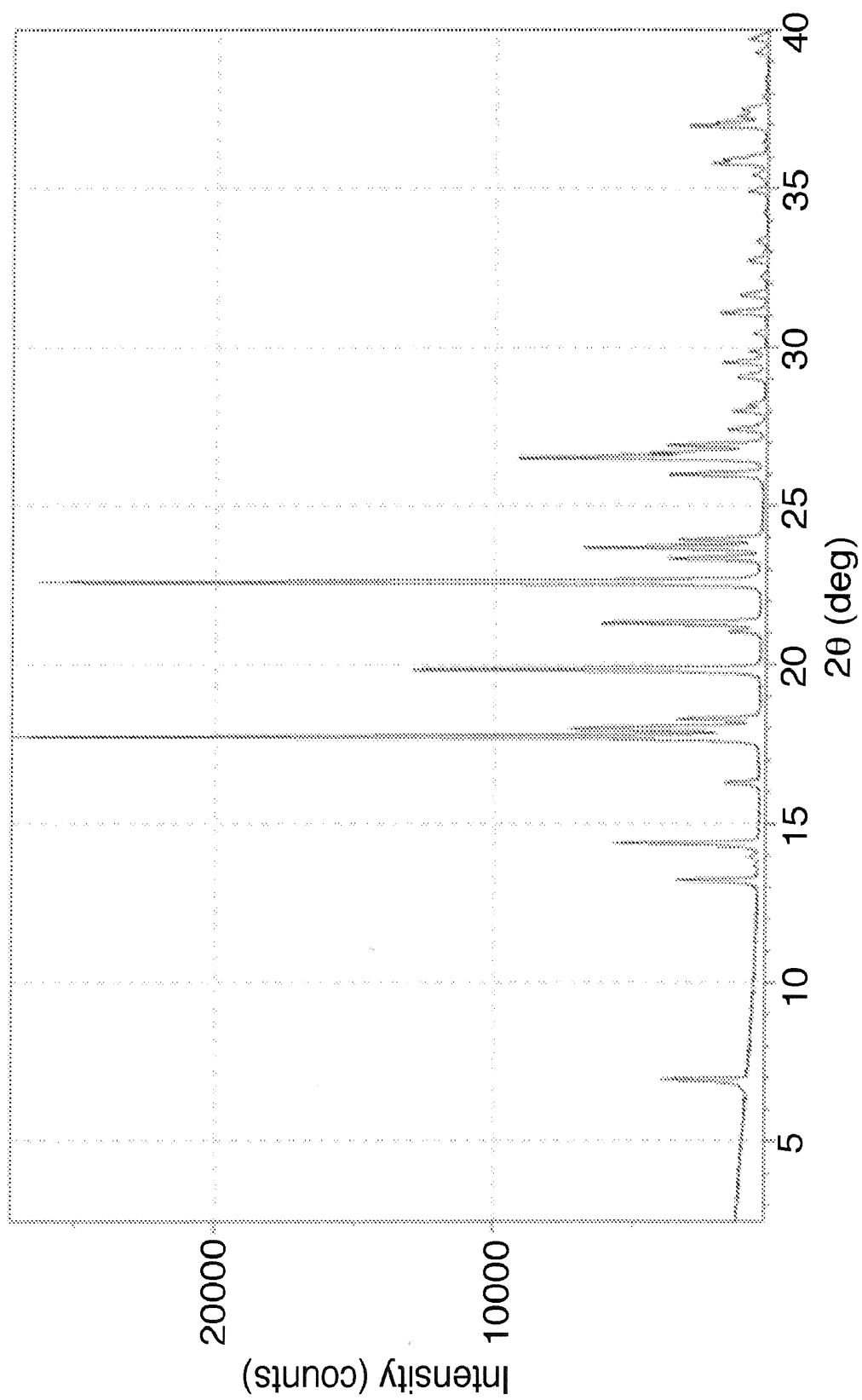
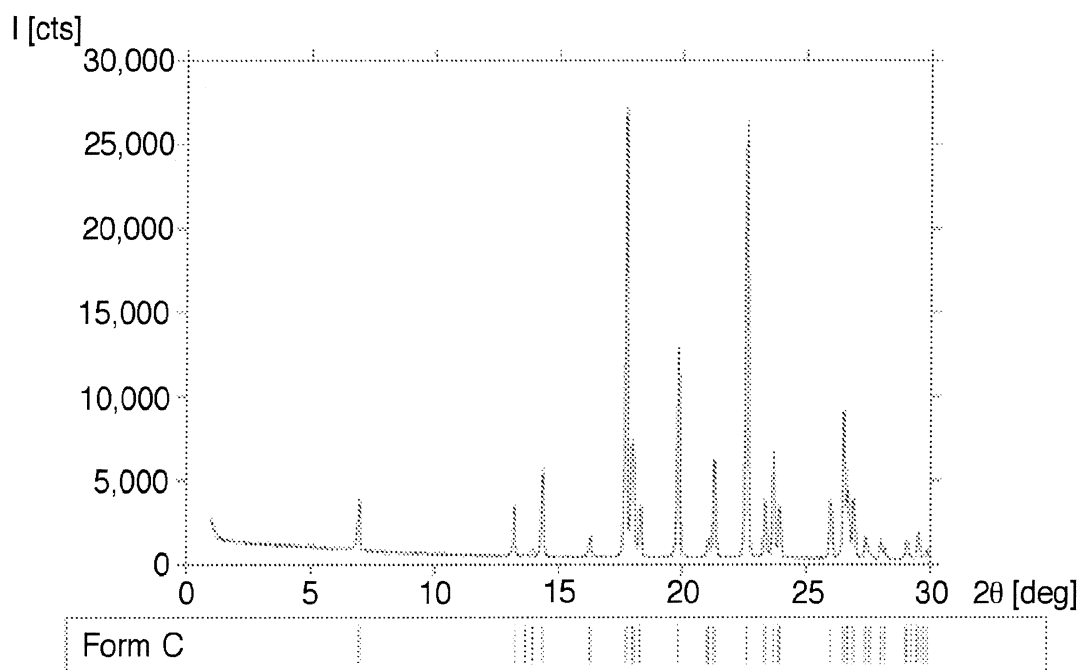


Figure 43 - XRPD pattern of Crystalline Form C



Form C

Bravais Type	Primitive Monoclinic
a [Å]	6.541
b [Å]	7.863
c [Å]	12.809
α [deg]	90
β [deg]	98.70
γ [deg]	90
Volume [Å ³ /cell]	651.2
Chiral Contents?	Chiral
Extinction Symbol	P 1 2 ₁ 1
Space Group(s)	P2 ₁ (4)

Source

Triads™ Algorithm

Figure 44 - Indexing result for the XRPD pattern depicted in Figure 43 collected with Cu K α radiation

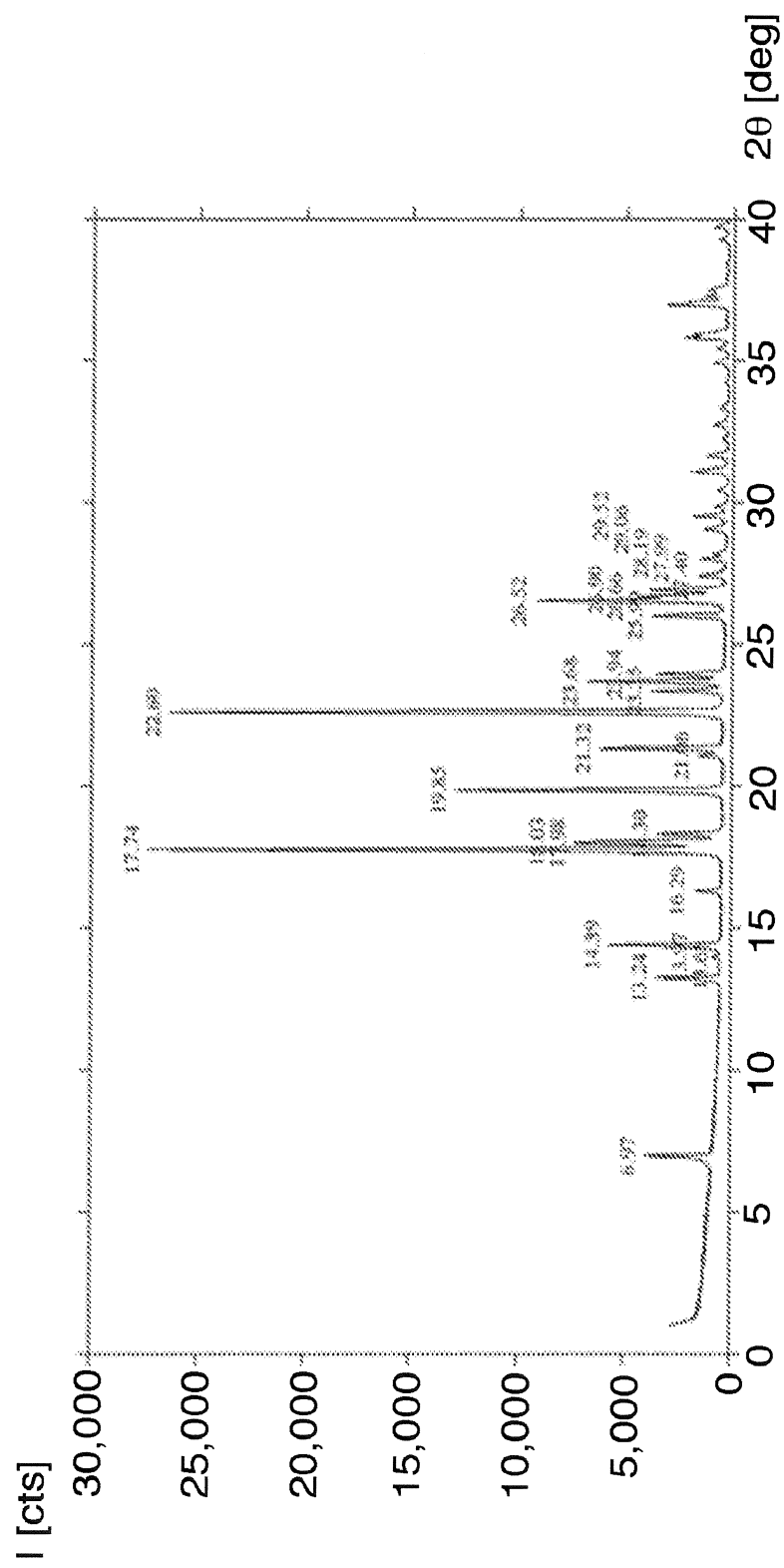
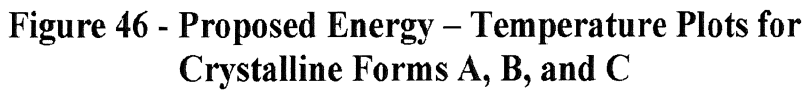


Figure 45 - Observed peaks for the XPRD pattern depicted in Figure 43 collected with Cu K α radiation



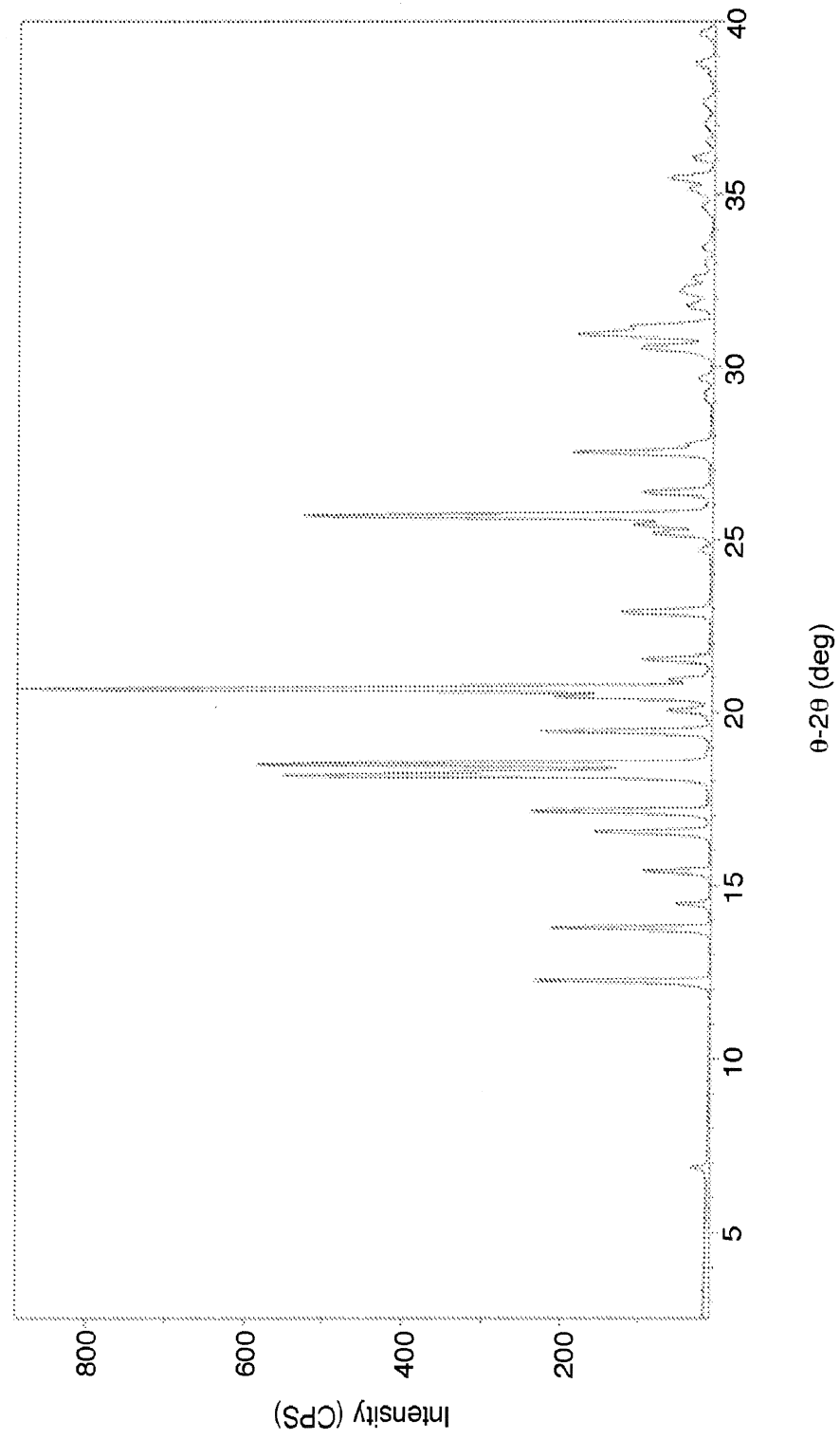


Figure 47 - XRPD pattern of Crystalline Form A

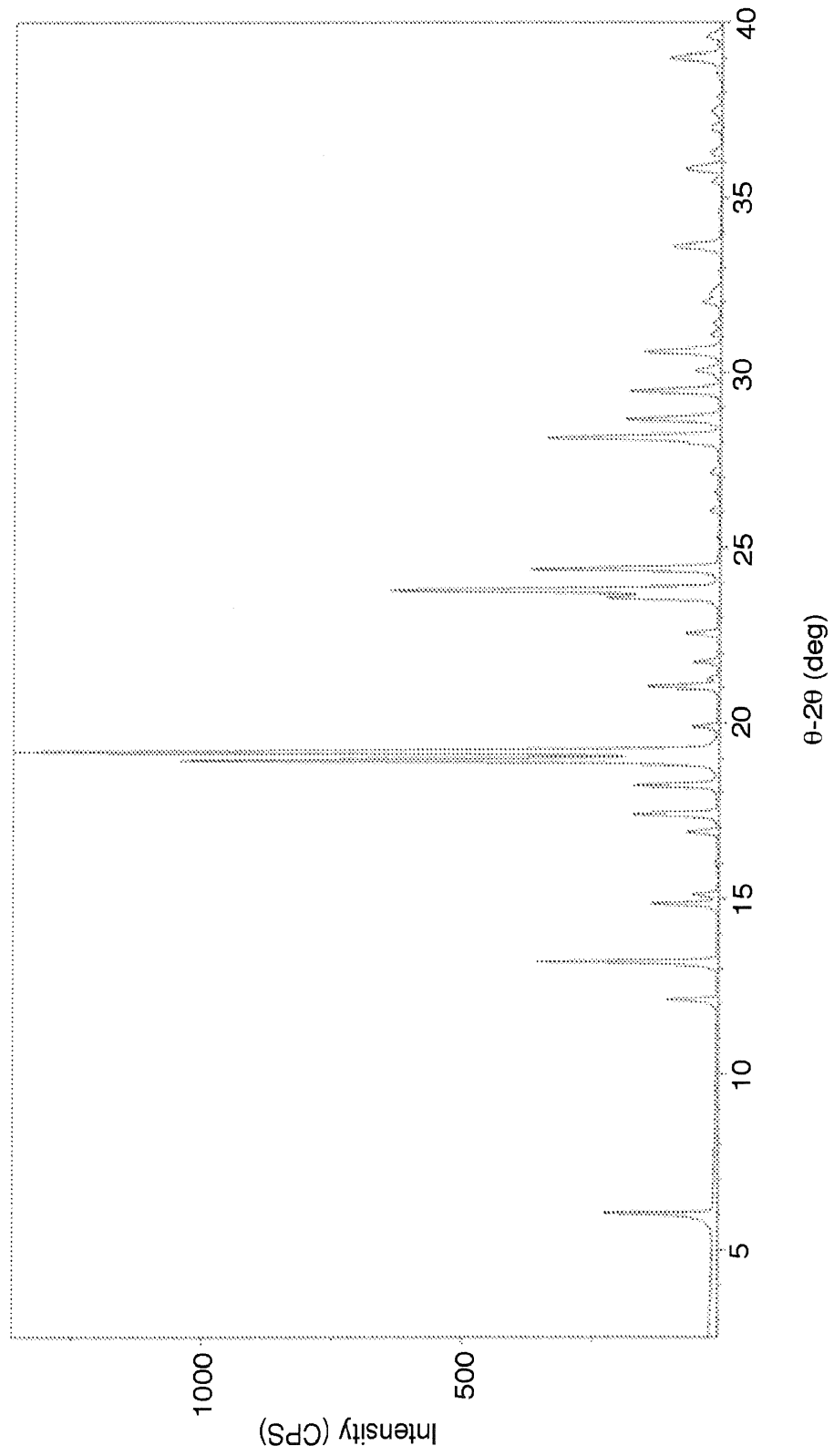


Figure 48 - XRPD pattern of Crystalline Form B

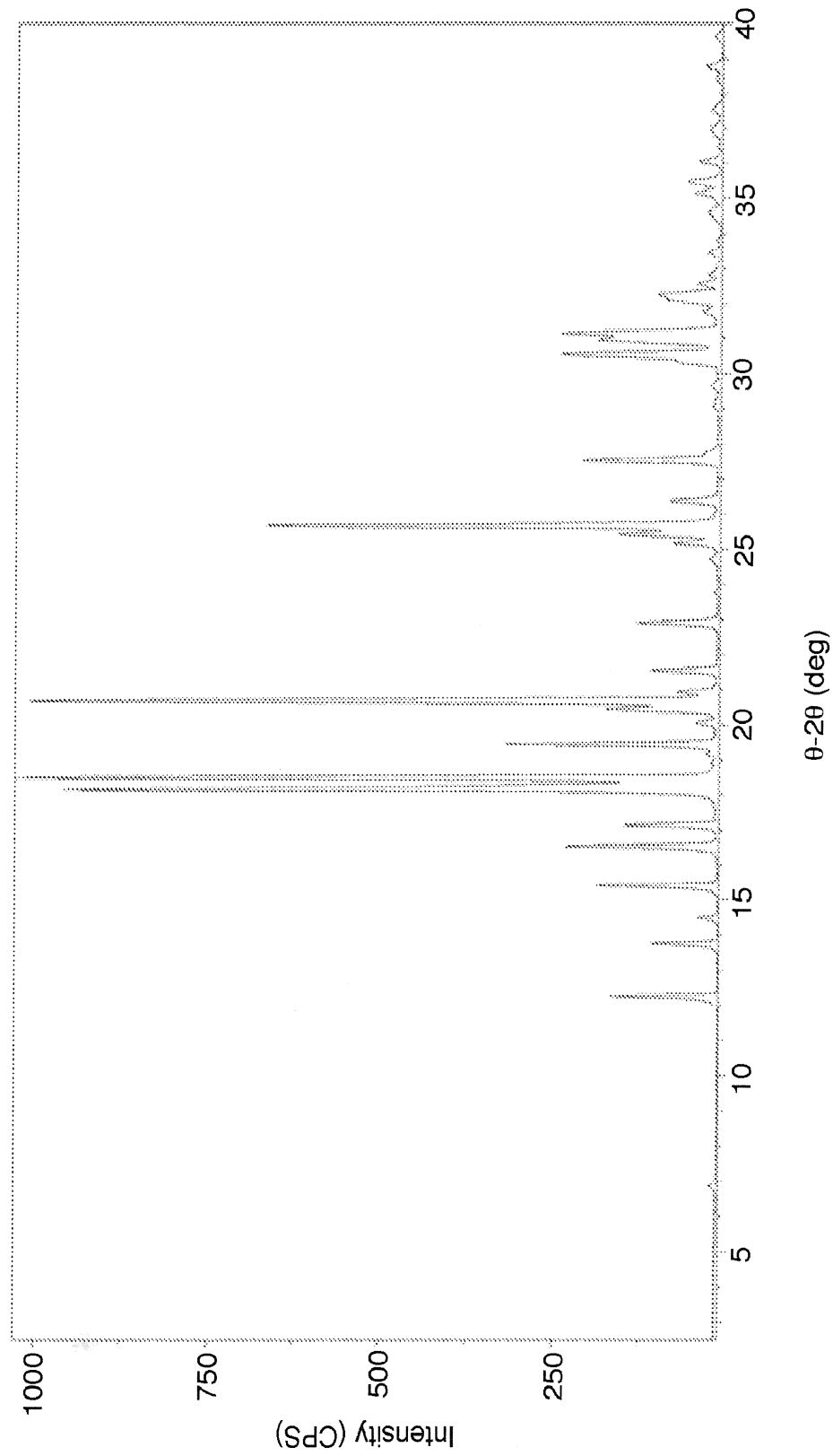


Figure 49 - XRPD pattern of mixture of Crystalline Form A and a Minor Quantity of Crystalline Form B

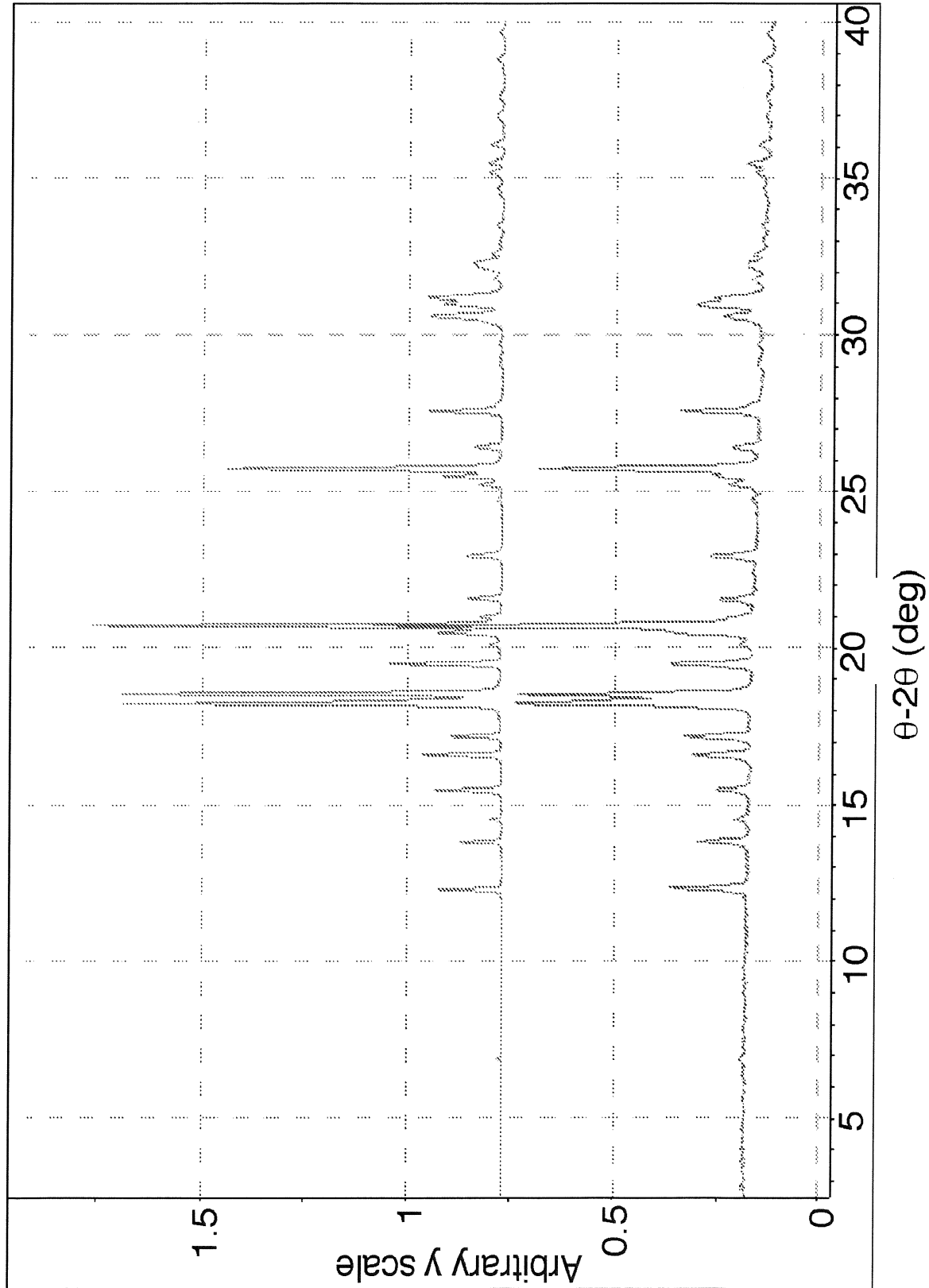


Figure 50 - XRPD patterns of Crystalline Form A Before and After DVS Analysis (top: before, bottom: after)

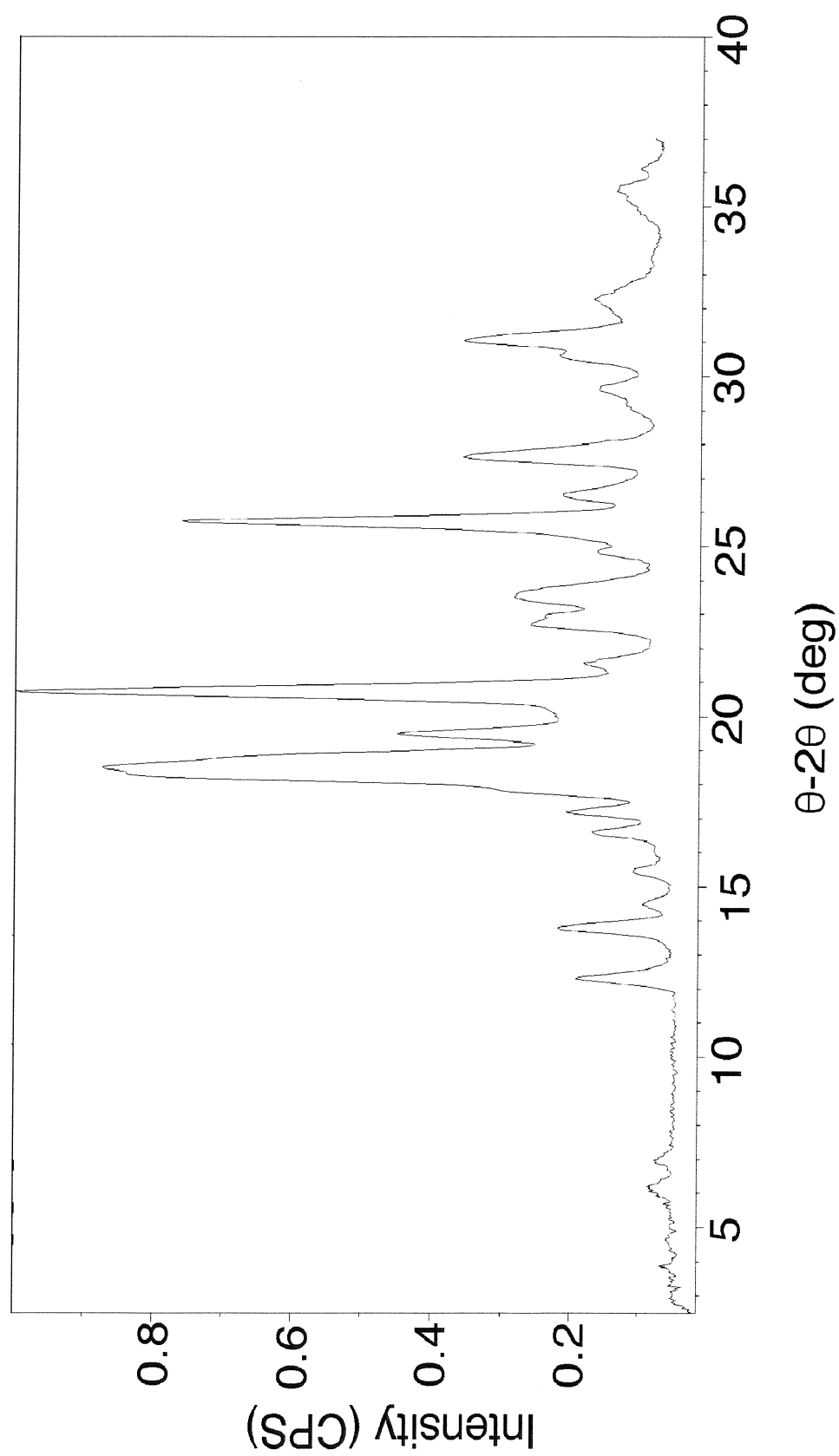


Figure 51 - XRPD Pattern of Disordered Crystalline Form A

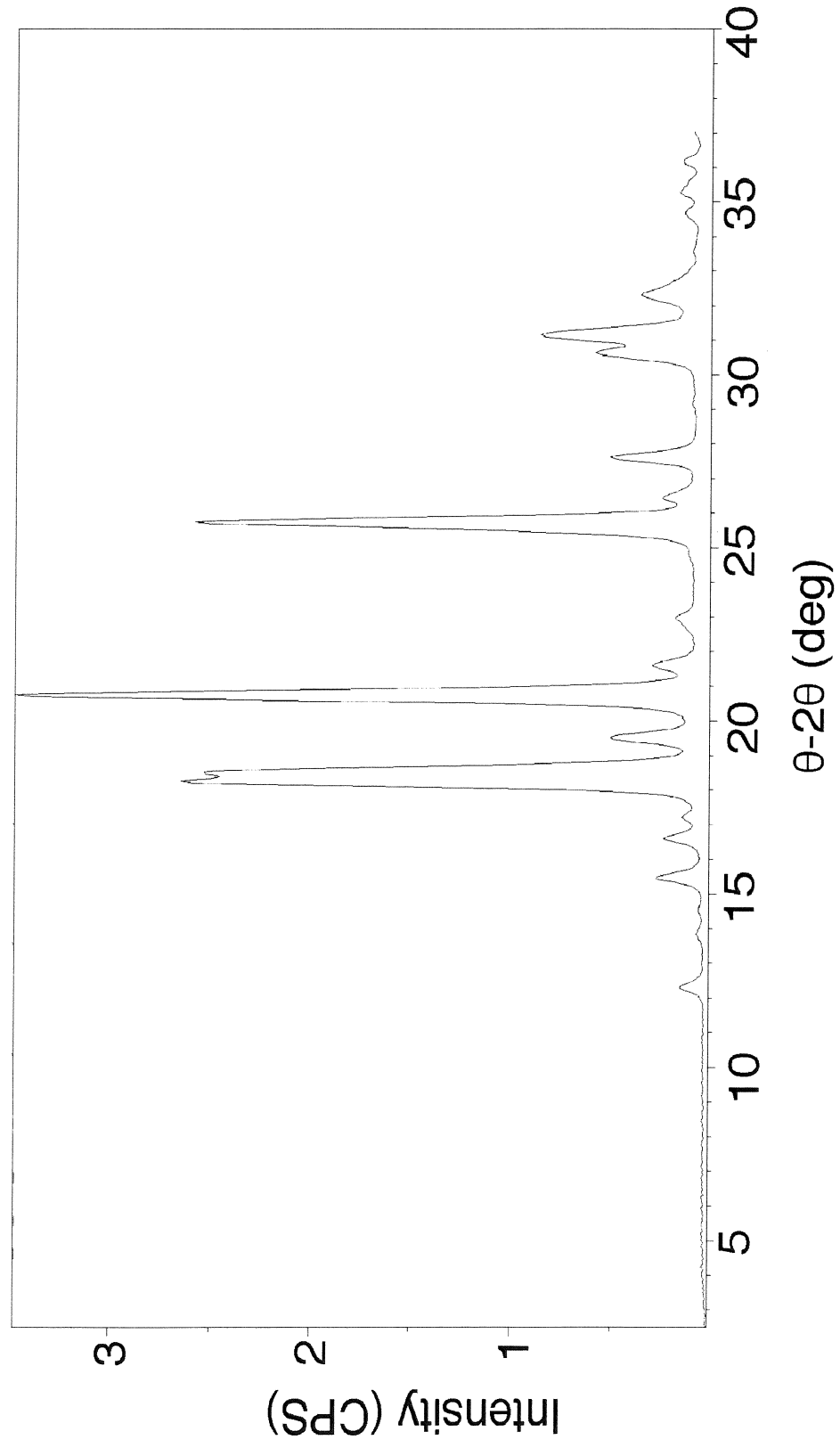


Figure 52 - XRPD Pattern of Disordered Crystalline Form A

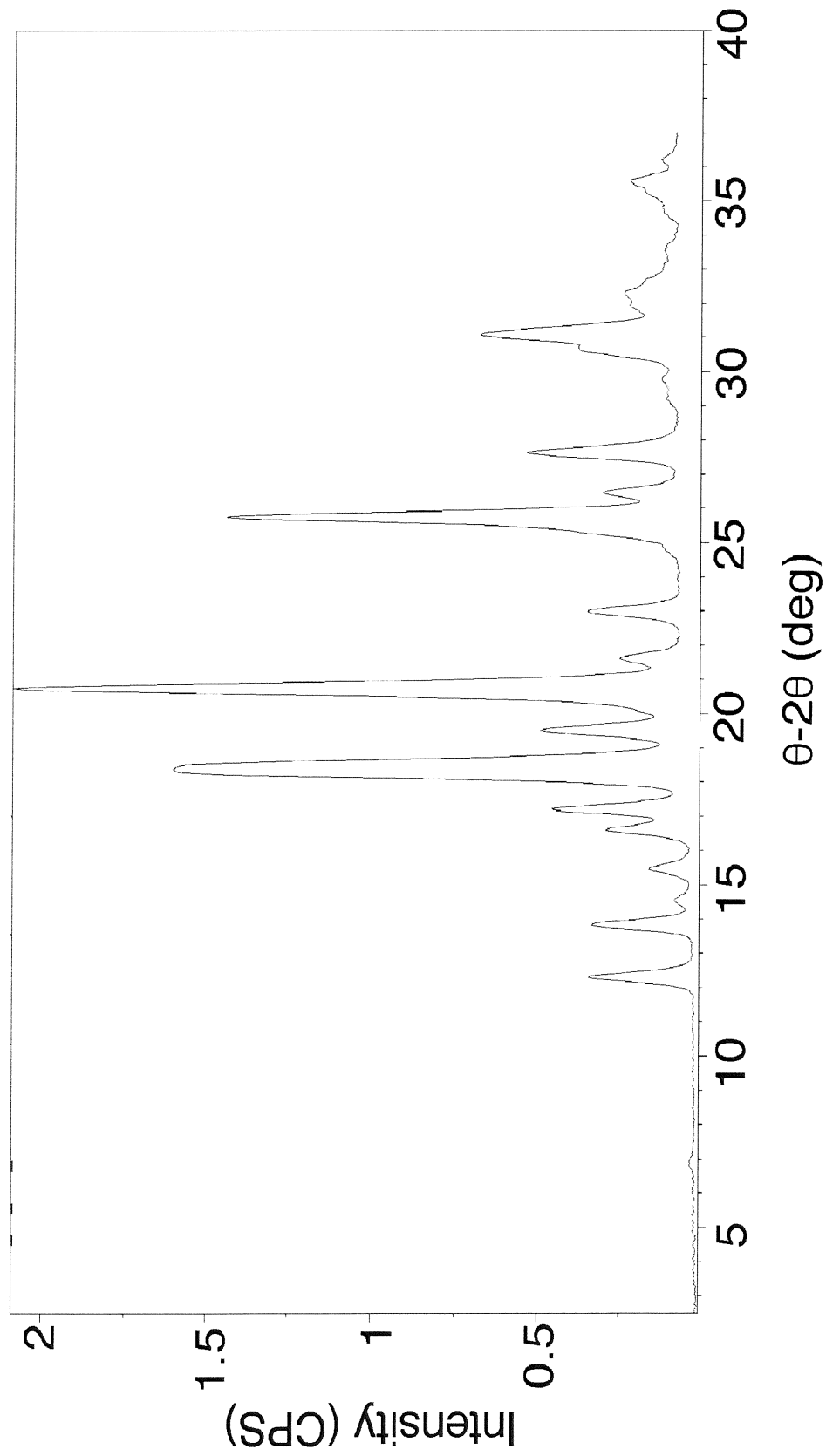


Figure 53 - XRPD Pattern of Disordered Crystalline Form A

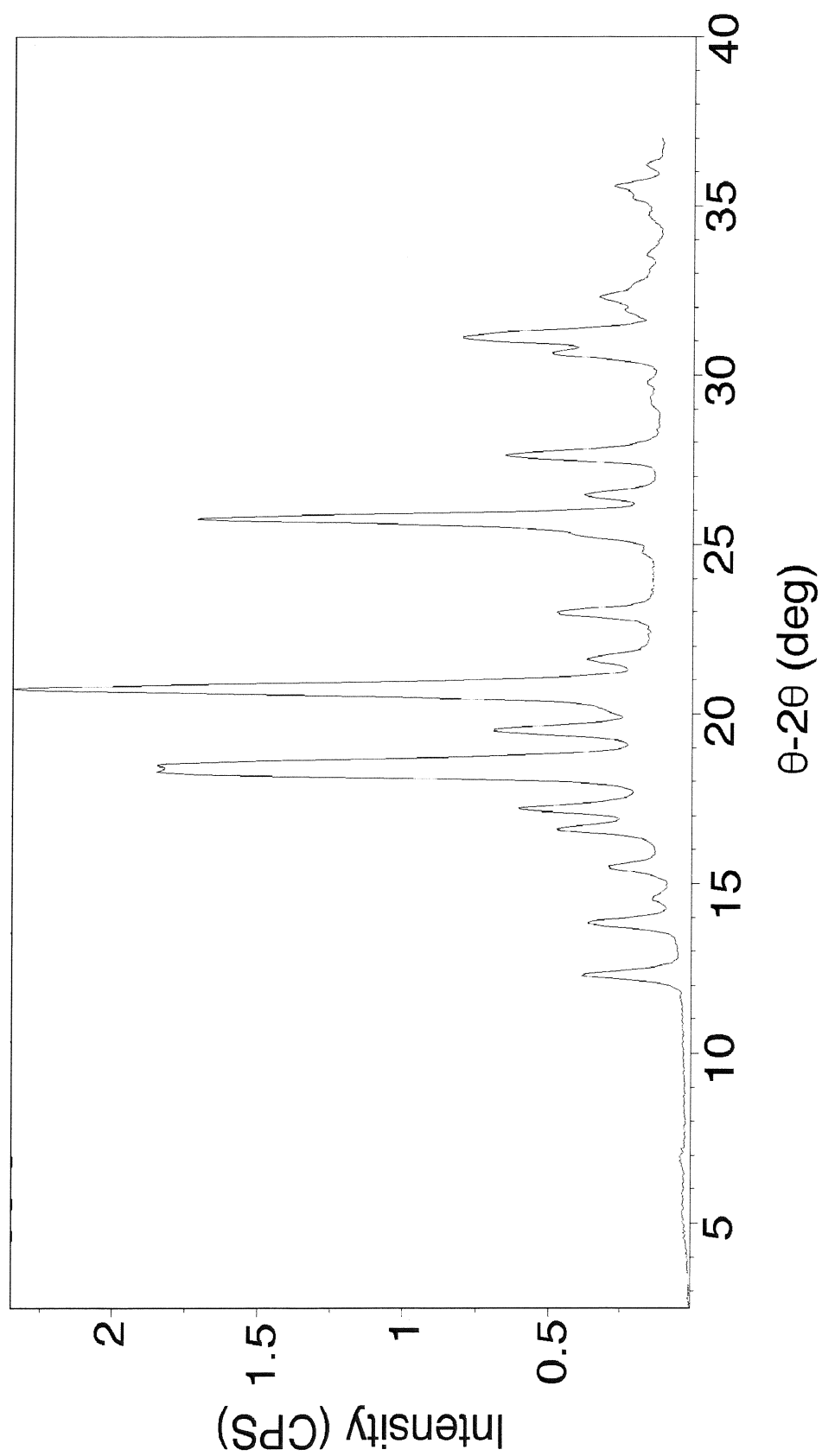
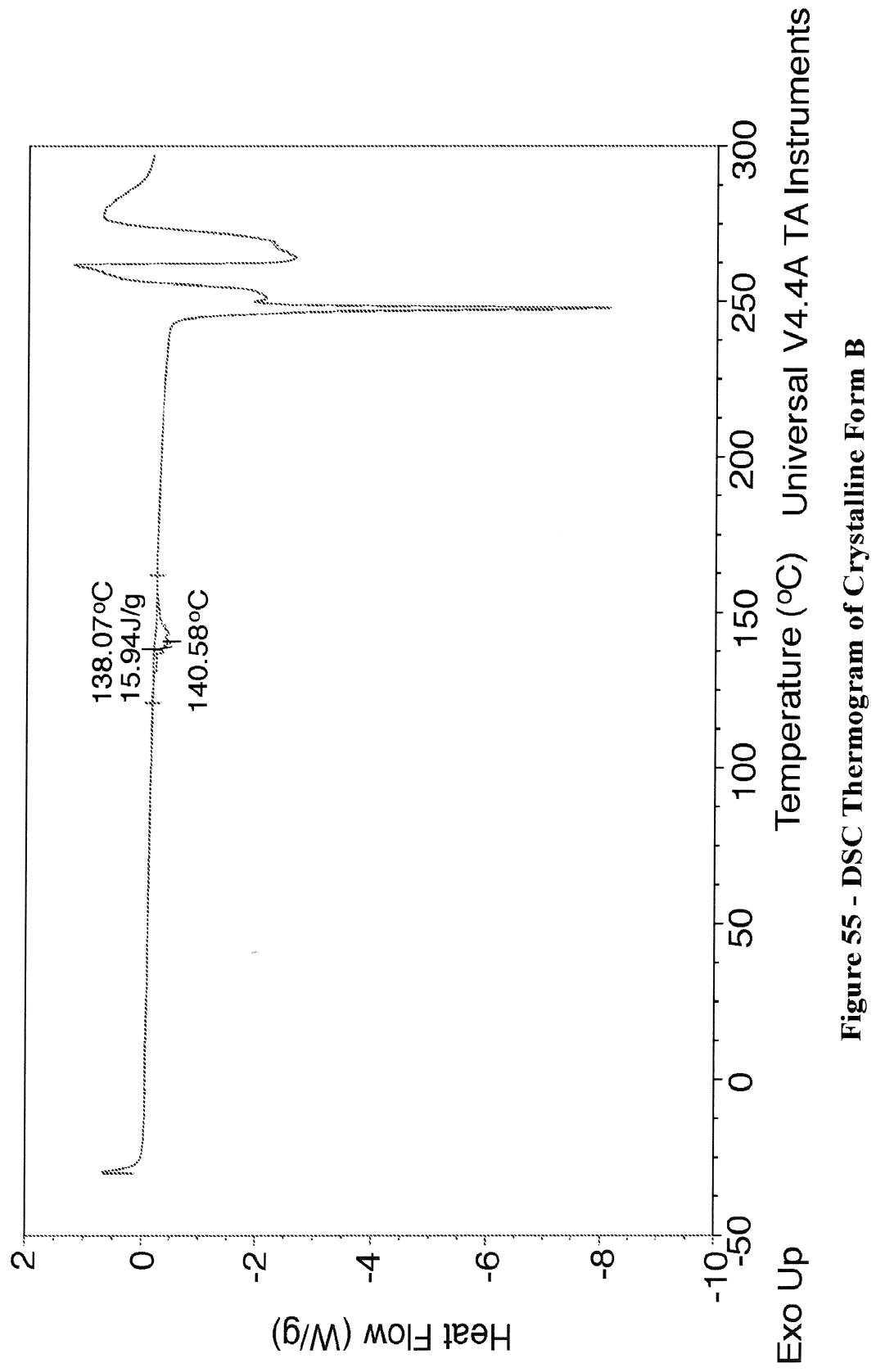


Figure 54 - XRPD Pattern of Disordered Crystalline Form A



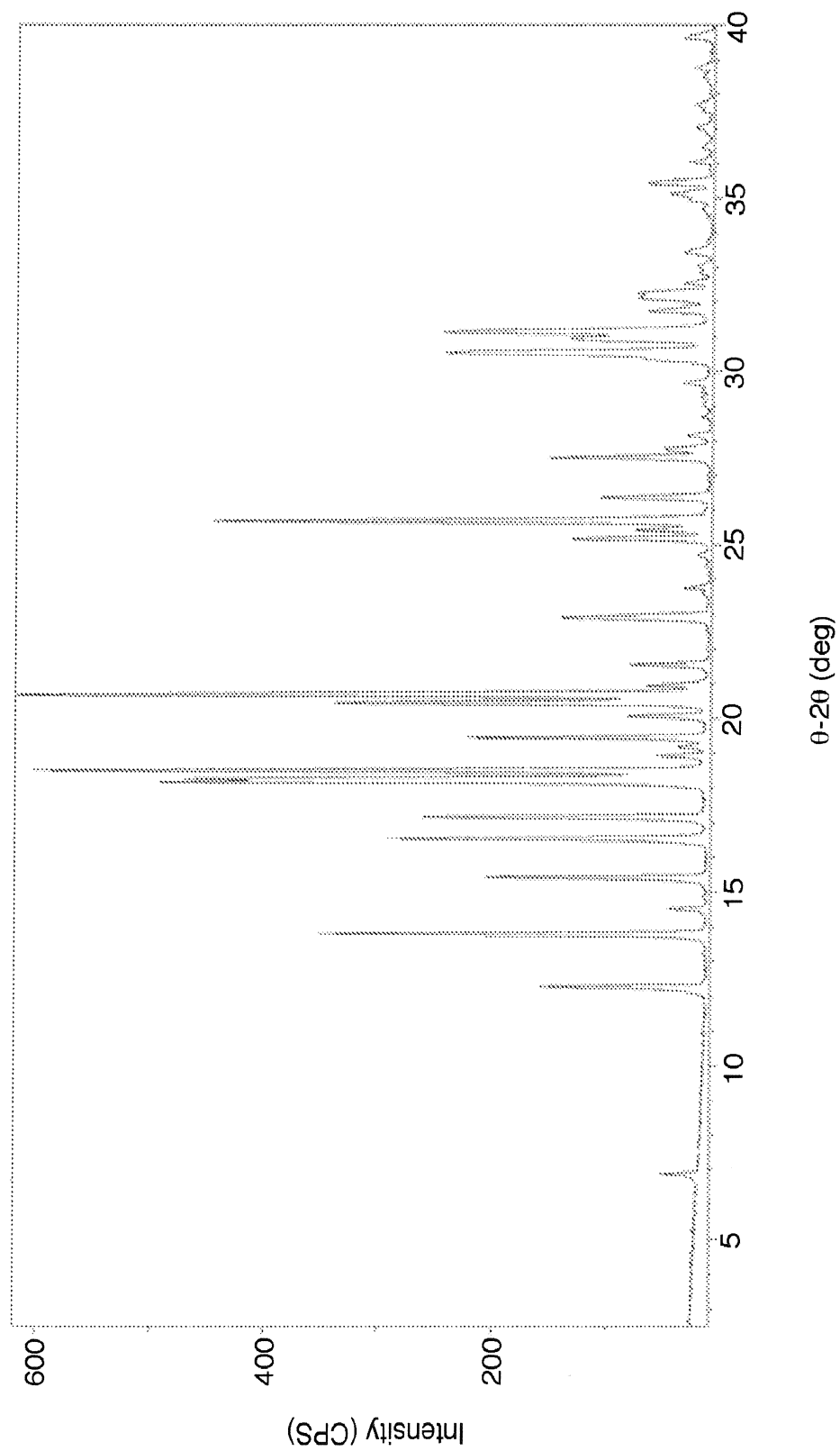


Figure 56 - XRPD Pattern of Mixture of Crystalline Forms A and B