

Figure 1 – Form I XRPD Diffractogram

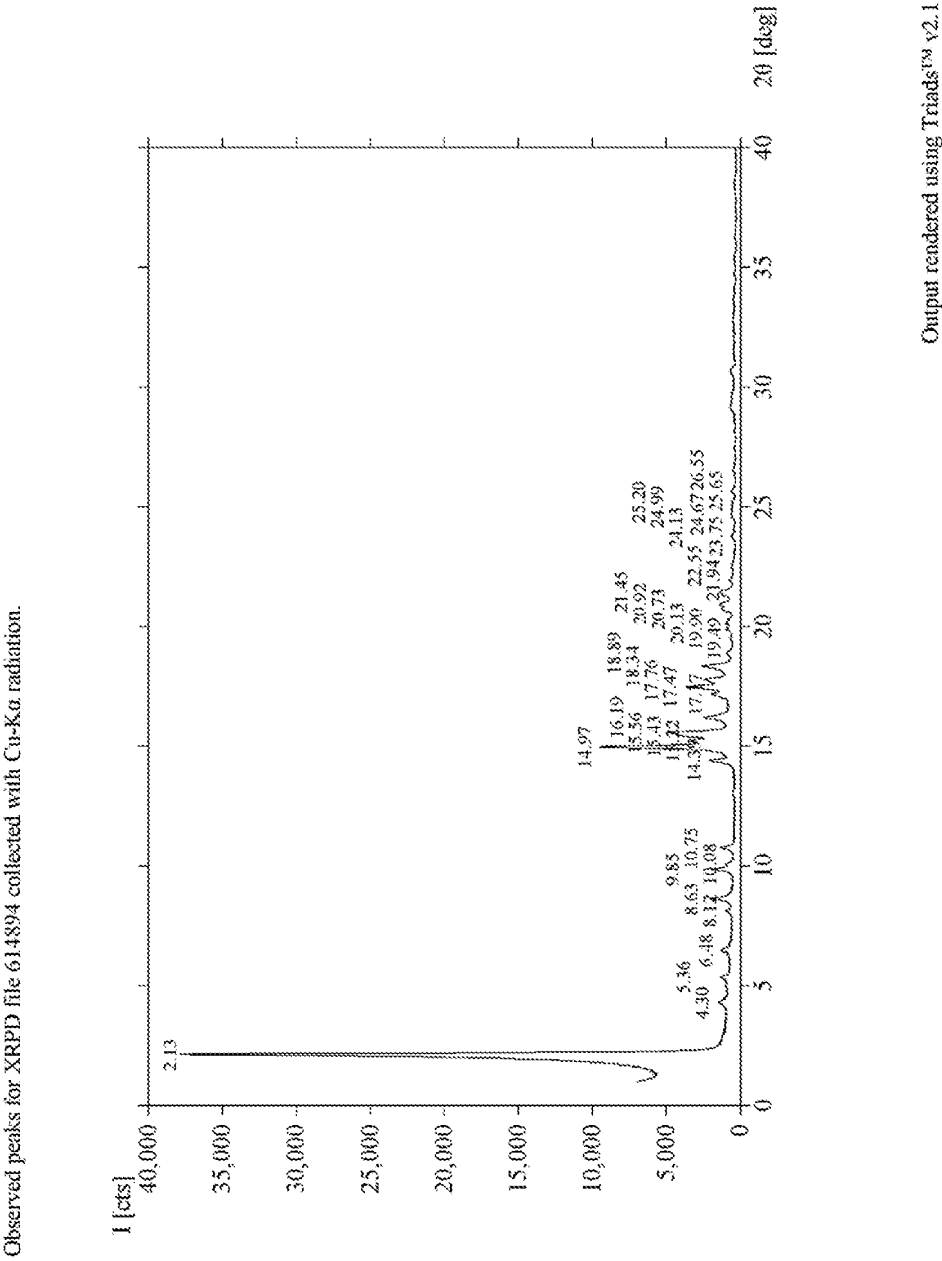


Figure 2 – Form II XRPD Diffractogram

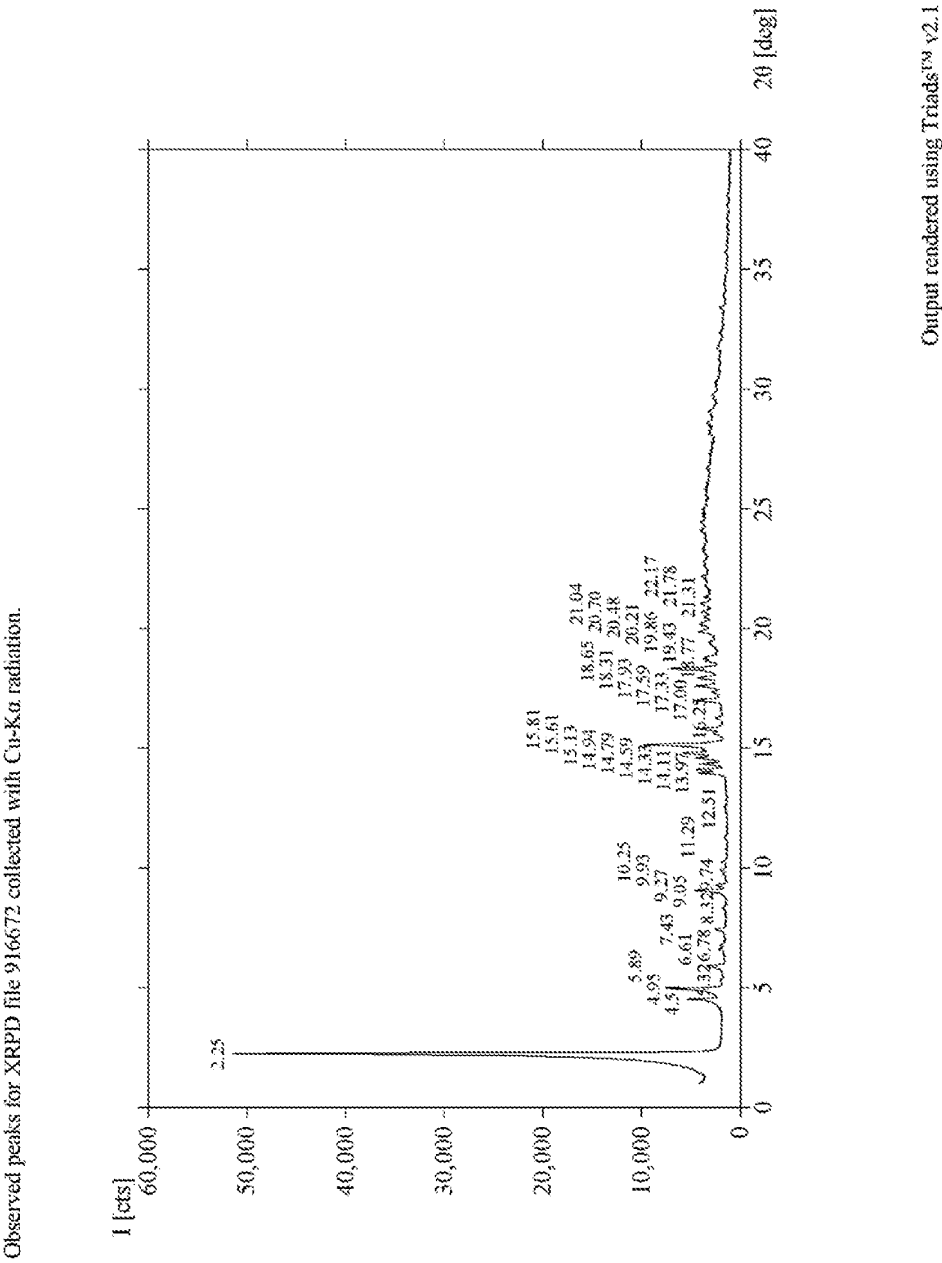


Figure 3 – Form V XRPD Diffractogram

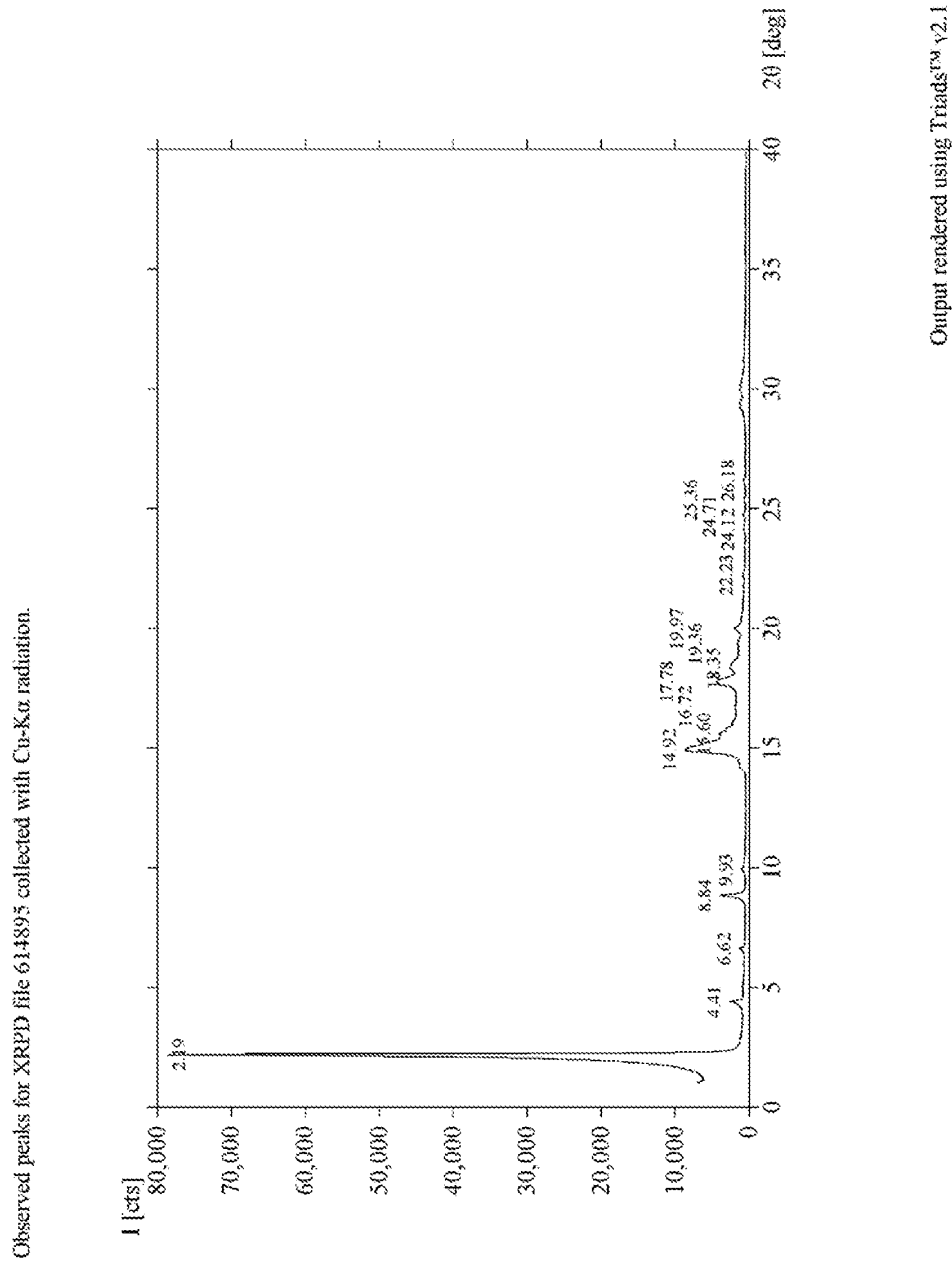
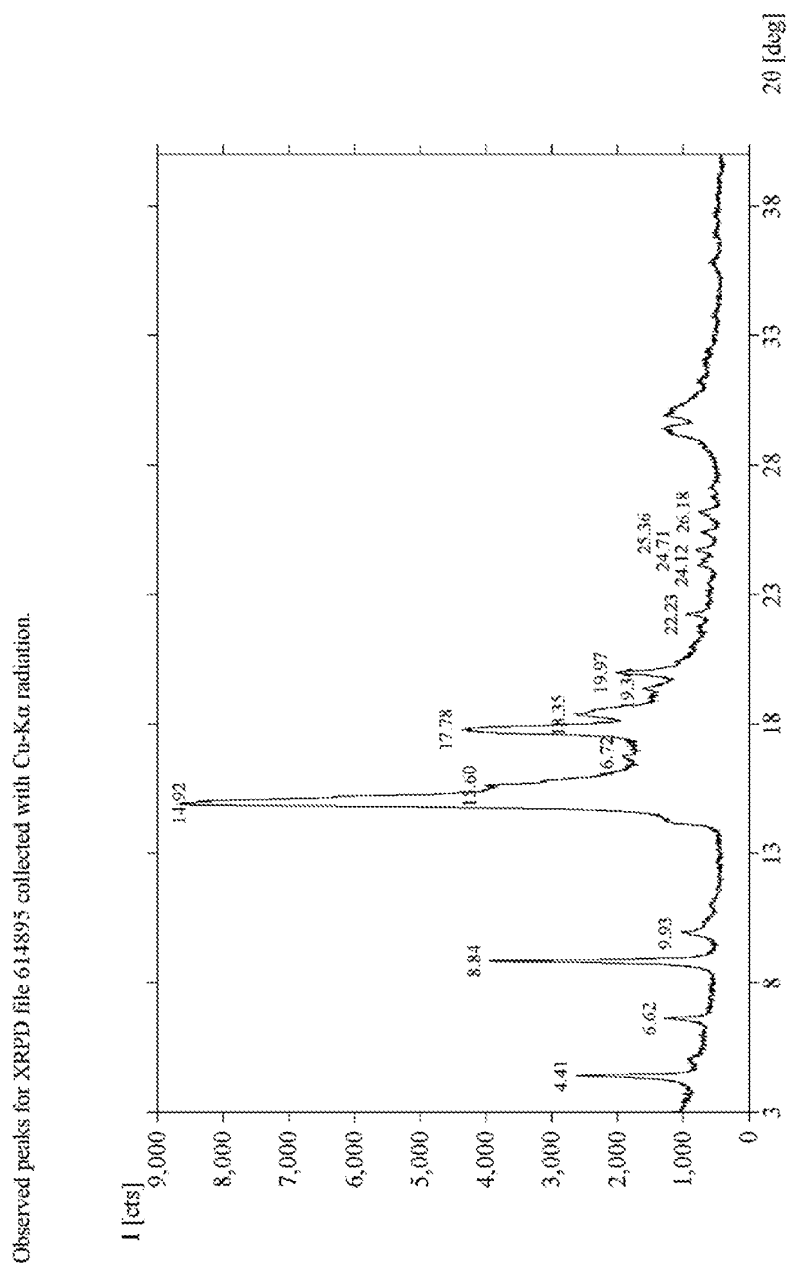


Figure 4 – Form V XRPD Diffractogram



Output rendered using Triads™ v2.1

Figure 5 – Form IX XRPD Diffractogram

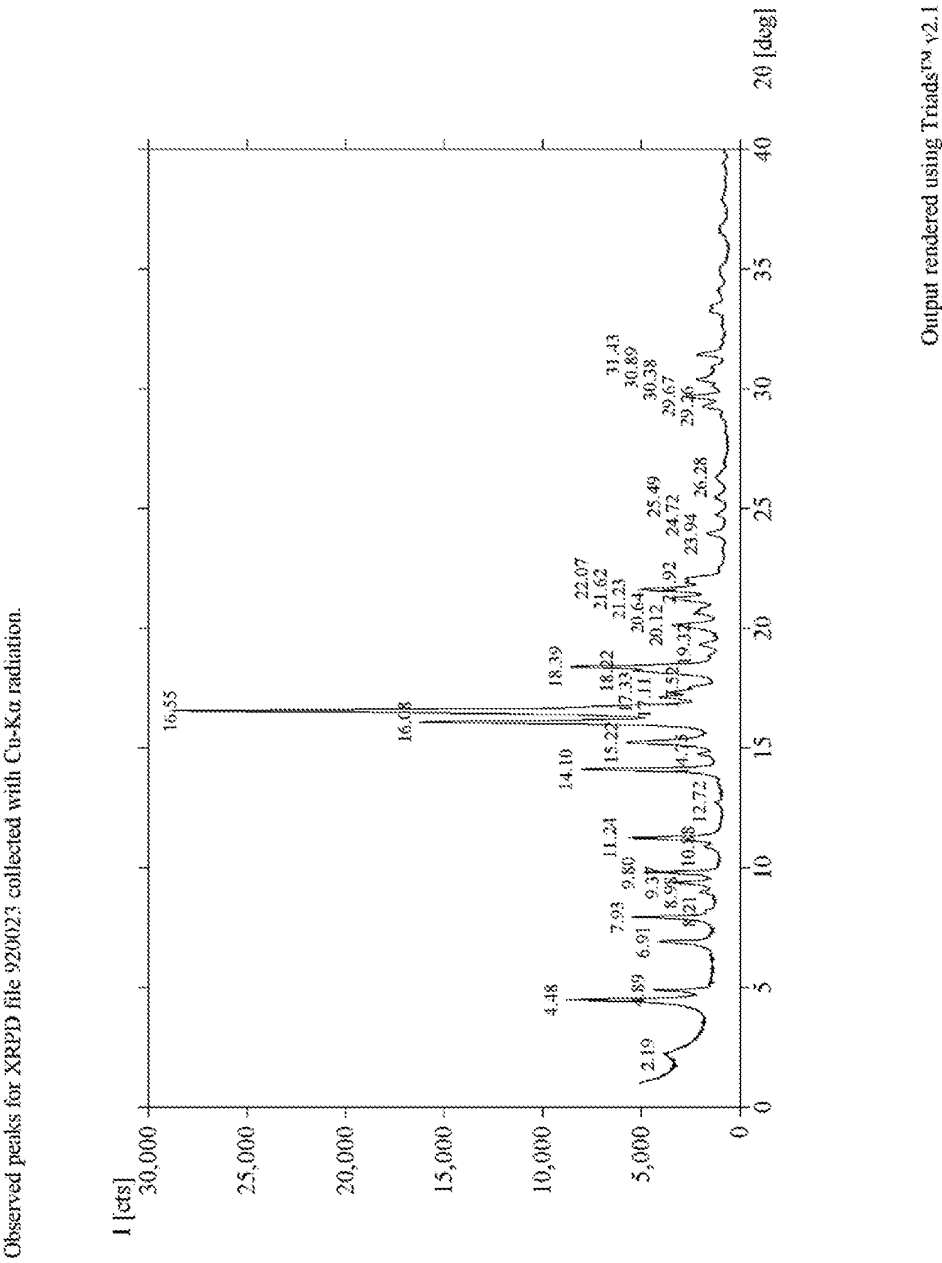


Figure 6 – Form XI XRPD Diffractogram

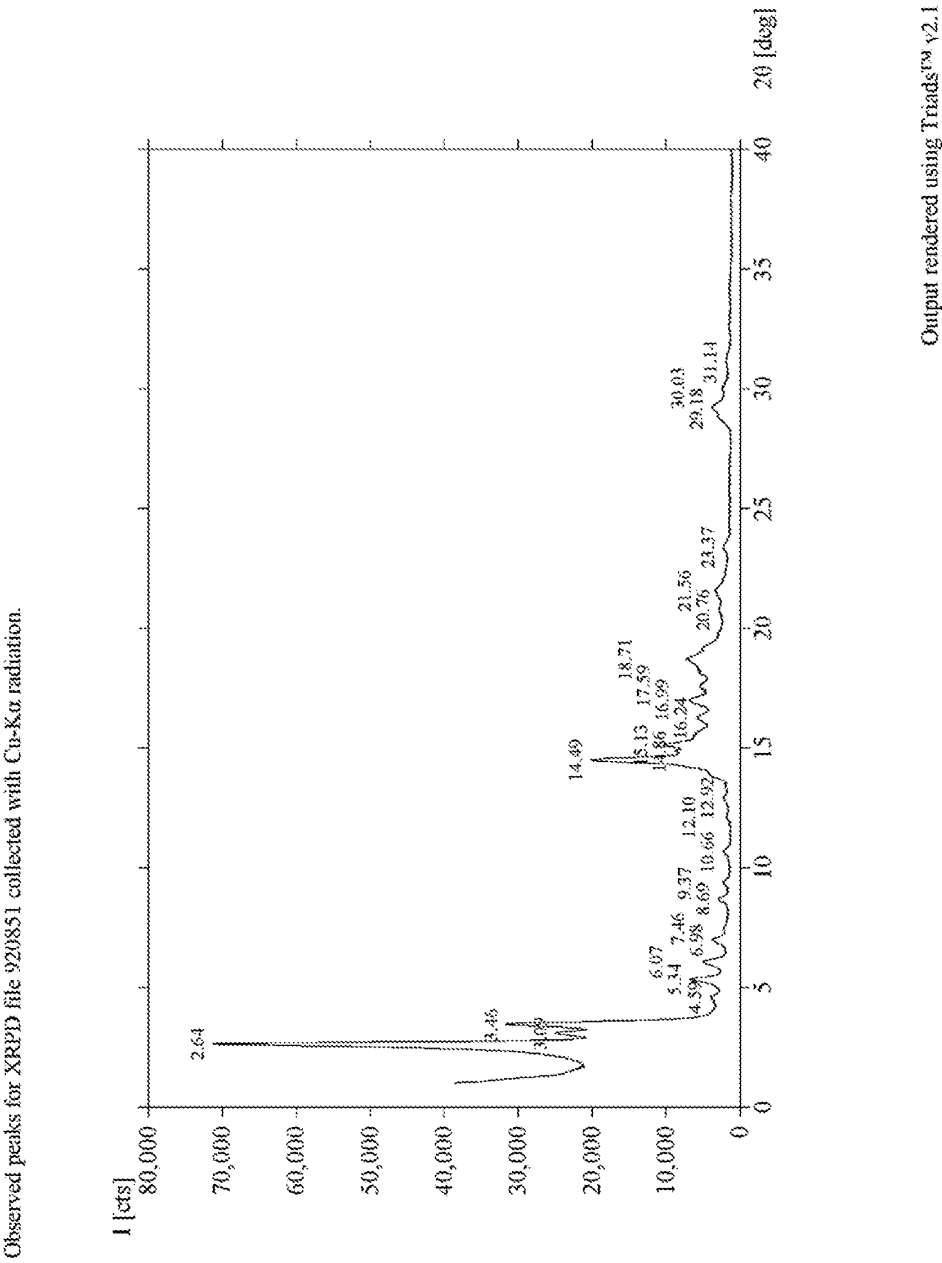


Figure 7 – Form XI XRPD Diffractogram

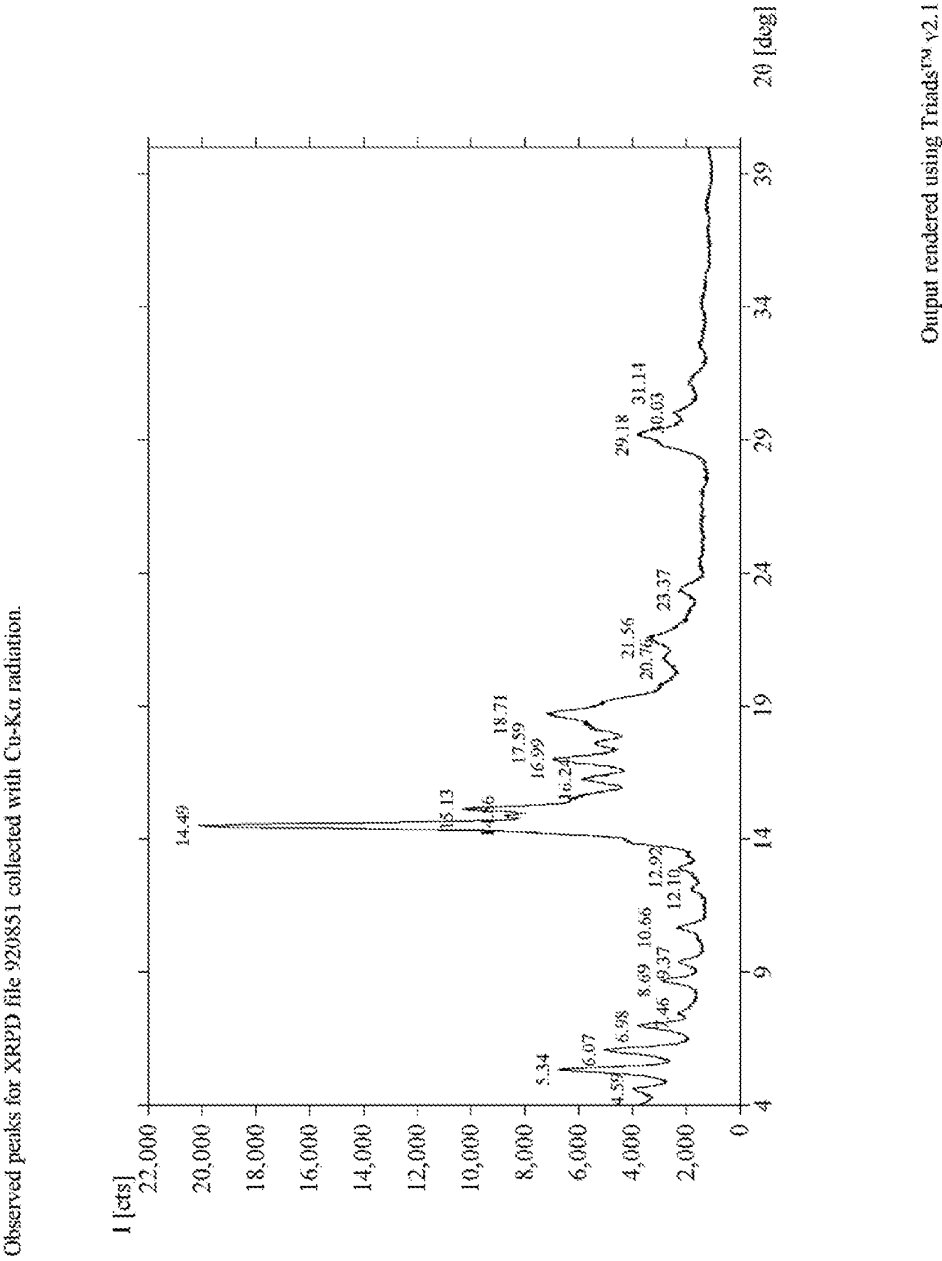
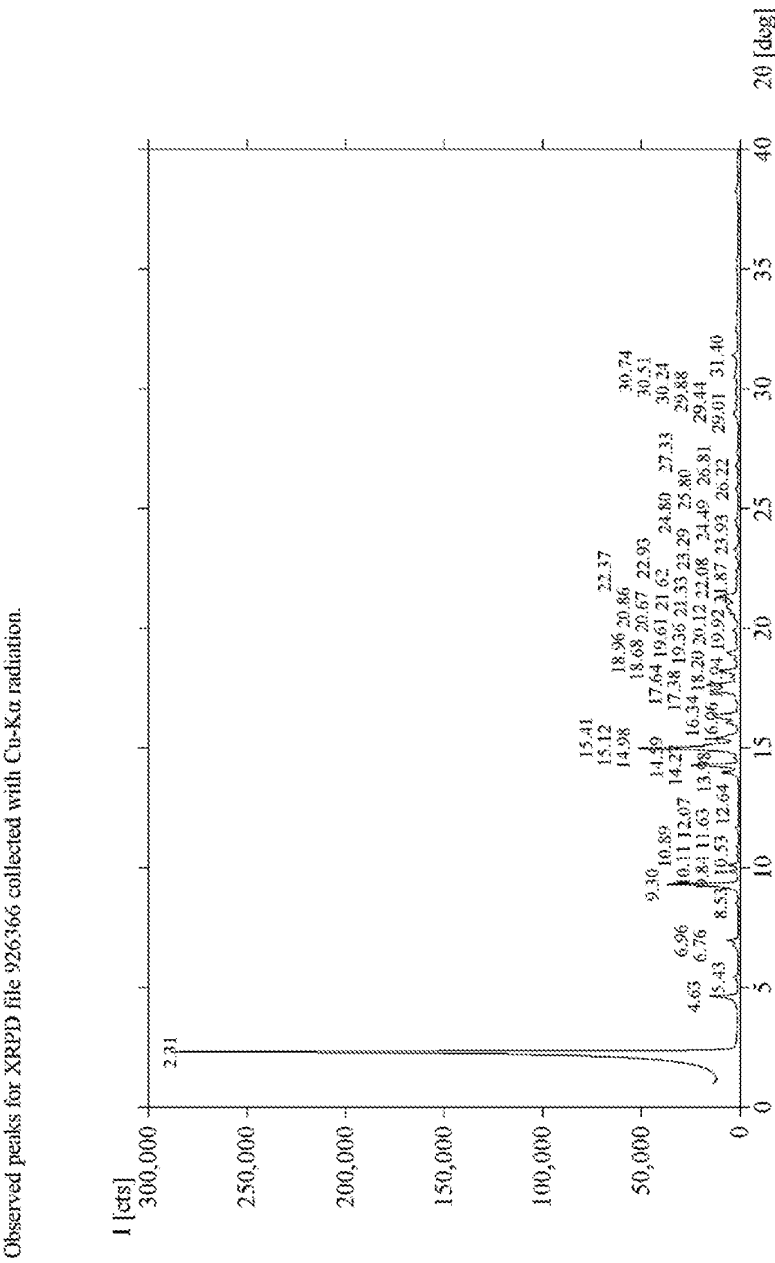
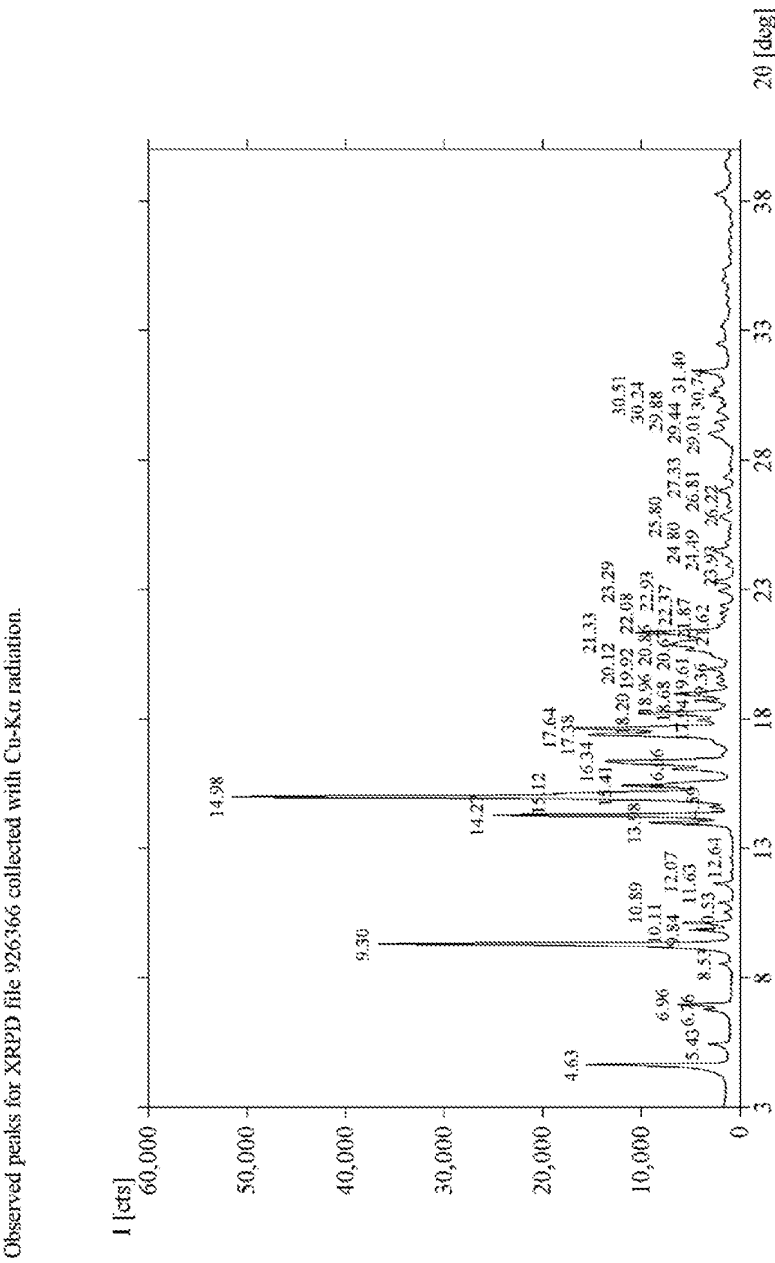


Figure 8 – Form XIII XRPD Diffractogram



Output rendered using Triads™ v2.1

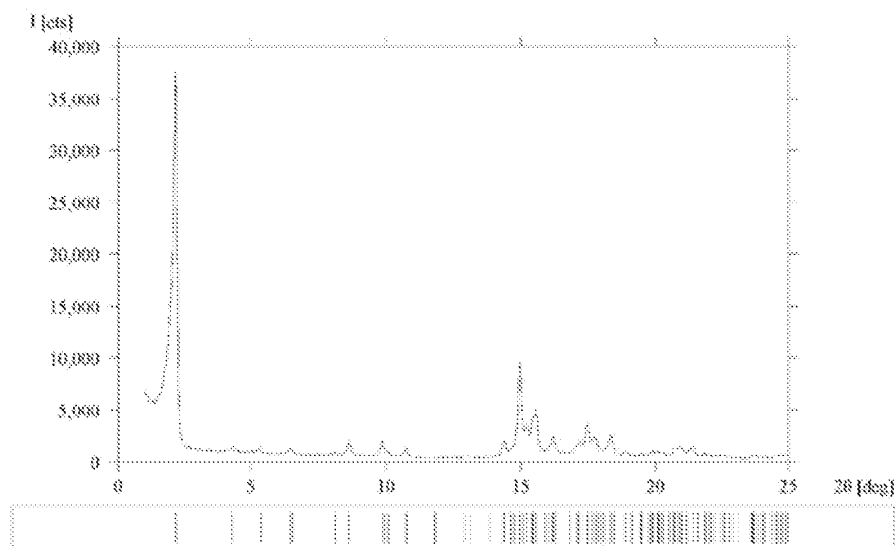
Figure 9 – Form XIII XRPD Diffractogram



Output rendered using Triads v2.1

Figure 10 – Indexing results for Form I

Indexing results for XRPD file 614894 collected with Cu-K α radiation.



Bravais Type	Primitive Monoclinic
a [Å]	6.175
b [Å]	17.937
c [Å]	41.093
α [deg]	90
β [deg]	93.14
γ [deg]	90
Volume [Å ³ /cell]	4,544.7
Chiral Contents?	Chiral
Extinction Symbol	P 1 2, 1
Space Group(s)	P2 ₁ , (4)
Source	Manual Input

Indexing is conducted under the "Procedures for SSC/Non-GRIP Activities."

Output rendered using Triads v1.1f

Figure 11 – Thermograms for Form I

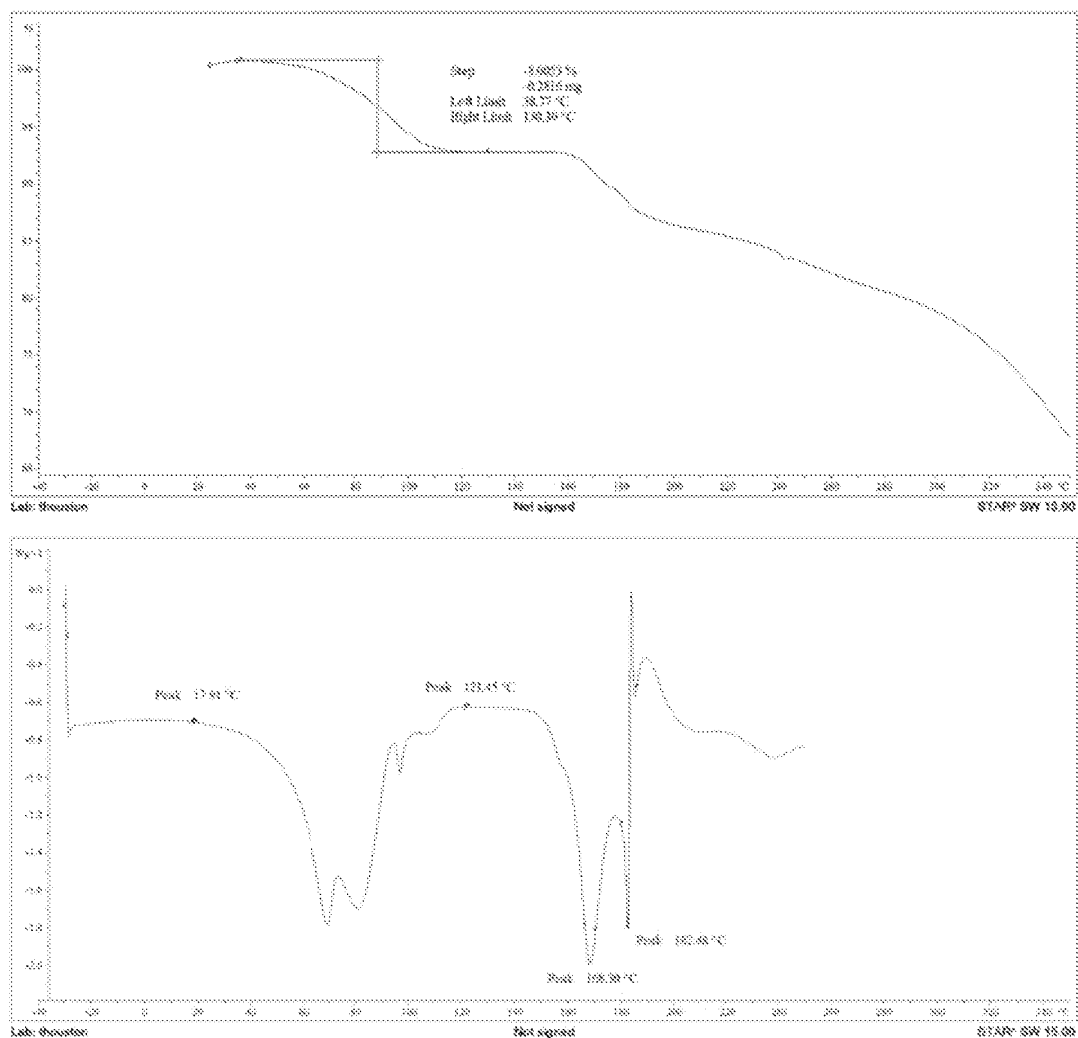
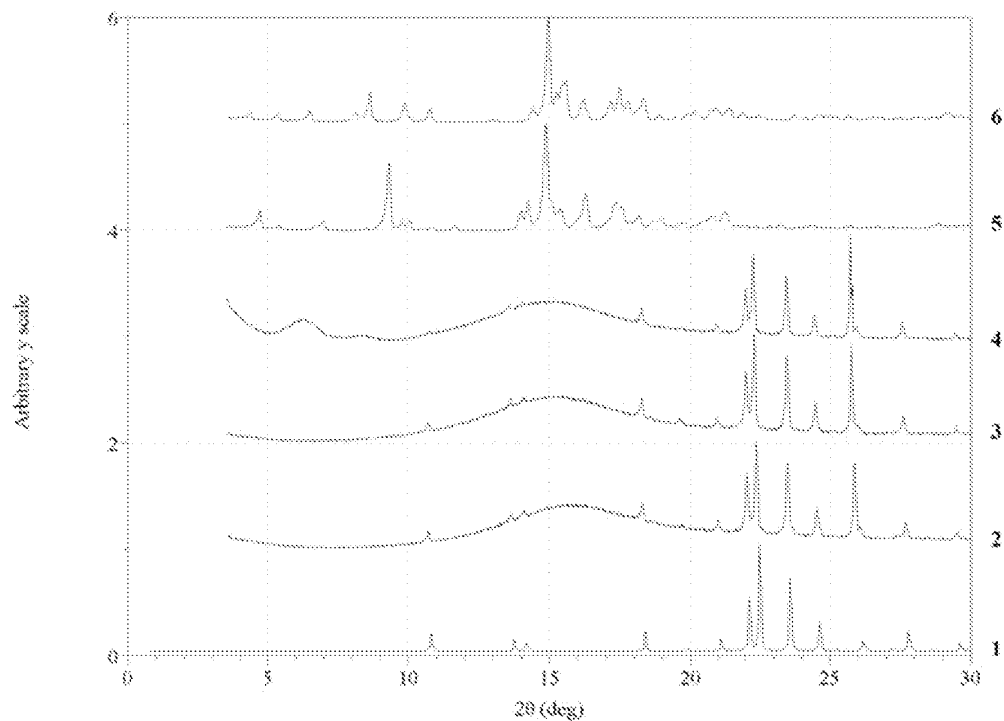


Figure 12 – VT-XRPD experiment for Form I



1 = Reference pattern of $\text{HNa}_3(\text{SO}_4)_2$ calculated from Cambridge Structural Database single crystal data

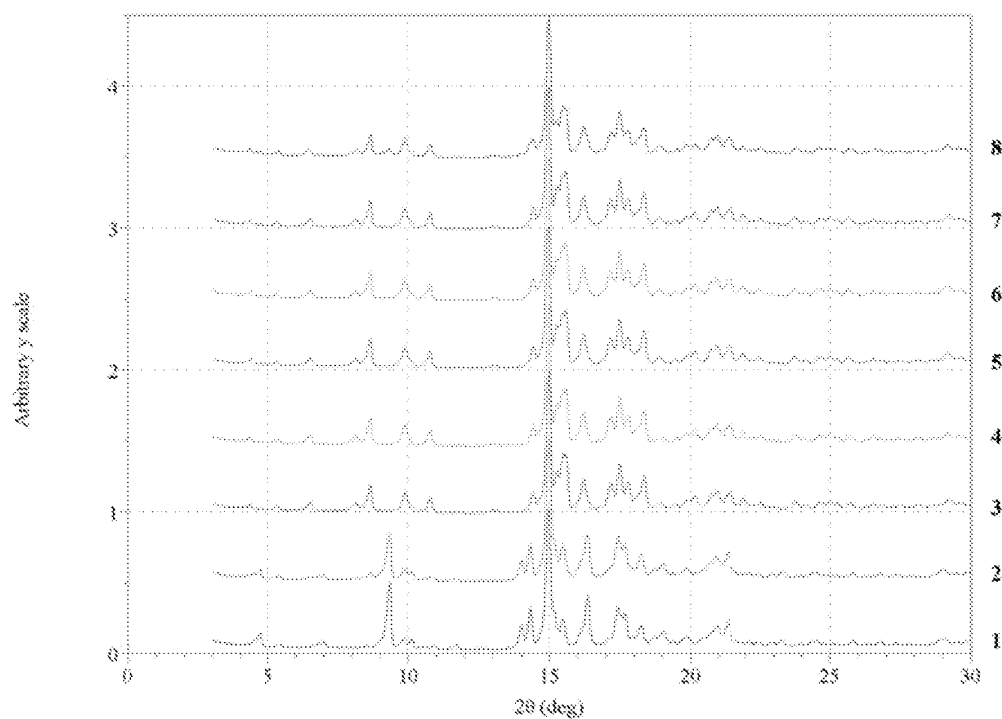
2 = $\text{HNa}_3(\text{SO}_4)_2$ (30°C)

3 = $\text{HNa}_3(\text{SO}_4)_2$ (135°C)

4 = Broad reflections + $\text{HNa}_3(\text{SO}_4)_2$ (170°C)

5 = Form XIII (135°C)

6 = Form I + peak at 9.0° (30°C)

Figure 13 – VRH-XRPD experiment for Form I and Form XIII

1 = Form XIII, 0% RH

2 = Forms XIII + I, 0% RH

3 = Form I, 25% RH

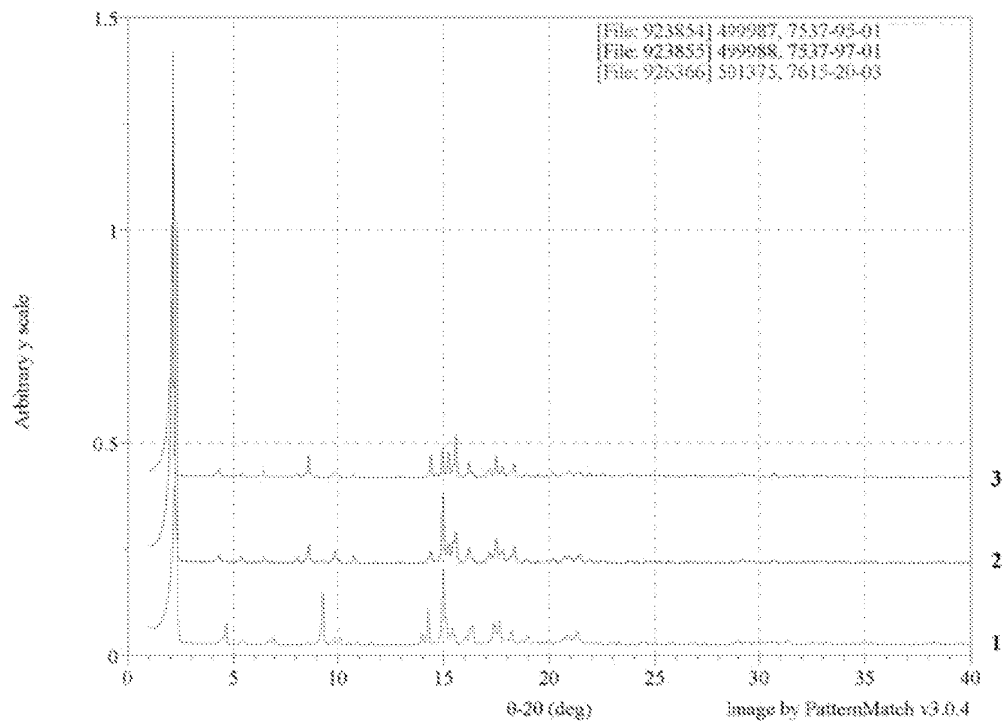
4 = Form I, 75% RH

5 = Form I, 85% RH

6 = Form I, 75% RH

7 = Form I, 55% RH

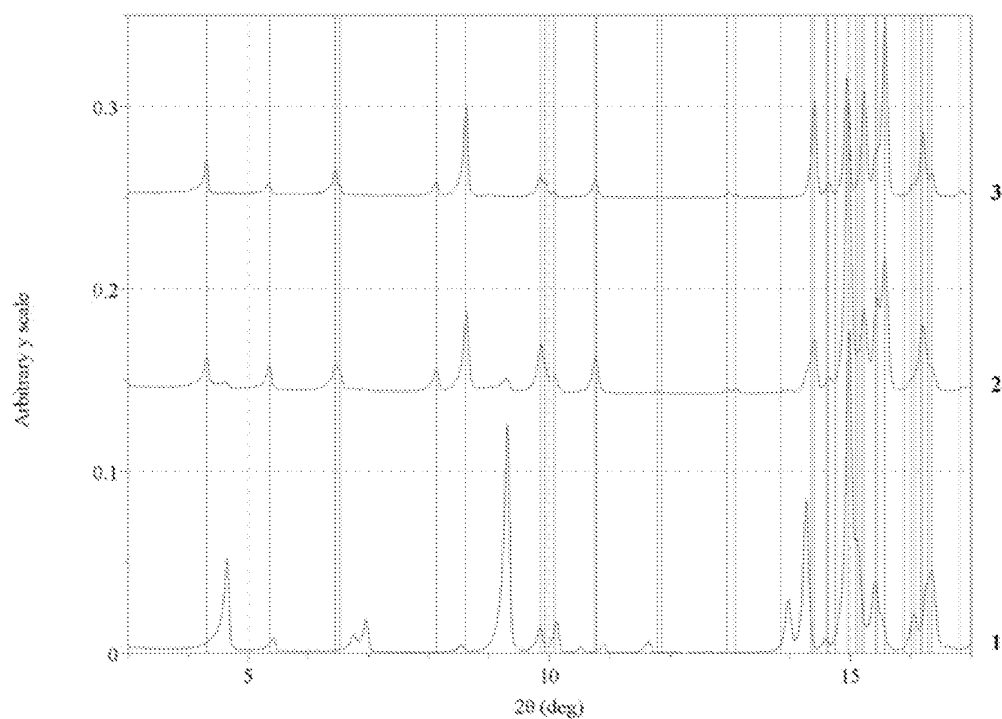
8 = Forms I + XIII, lab humidity 14.7% RH, initial XRPD

Figure 14 – XRPD Diffractograms from dehydration of Form I

1 = Dried @ 70 °C vacuum for 2 days; Form XIII

2 = Dried under N₂ for 30 min, Forms I + XIII

3 = 0.50 a_w ACN/H₂O slurry 4 days, Form I + peak @ 9.0°

Figure 15 – XRPD Diffractograms from dehydration of Form I (expanded)

1 = Dried @ 70 °C vacuum for 2 days; Form XIII

2 = Dried under N₂ for 30 min, Forms I + XIII

3 = 0.50 a_w ACN/H₂O slurry 4 days, Form I + peak @ 9.0°

Figure 16 – DVS Isotherm for Form II

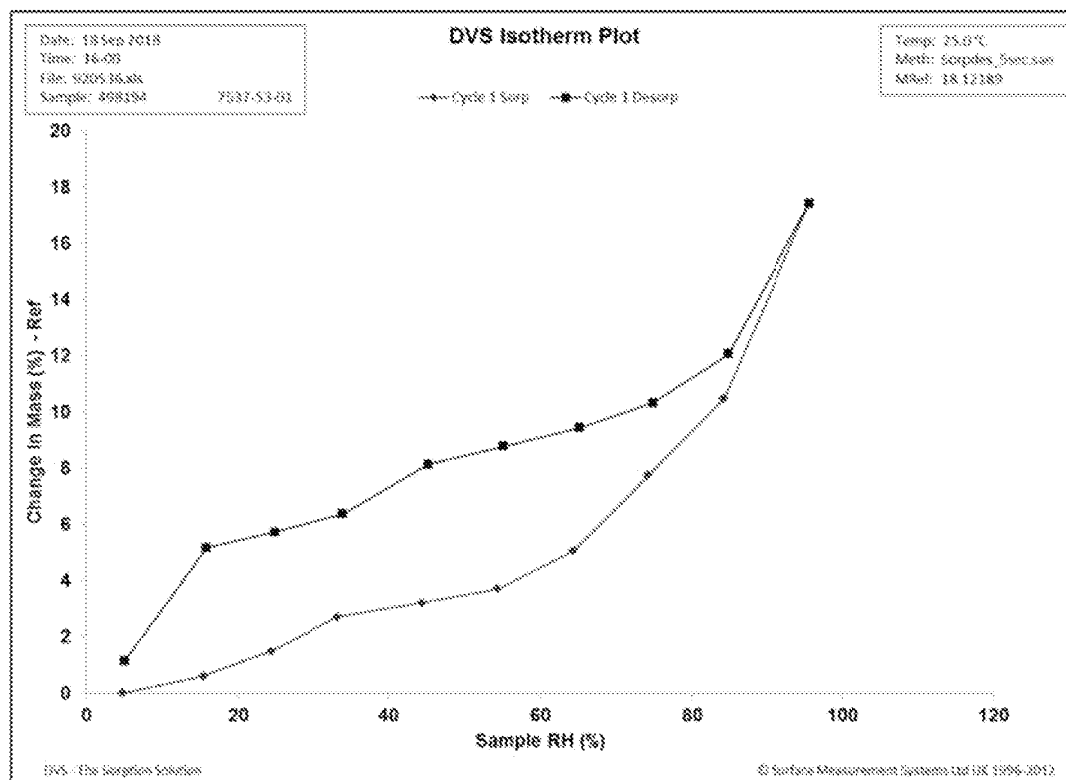
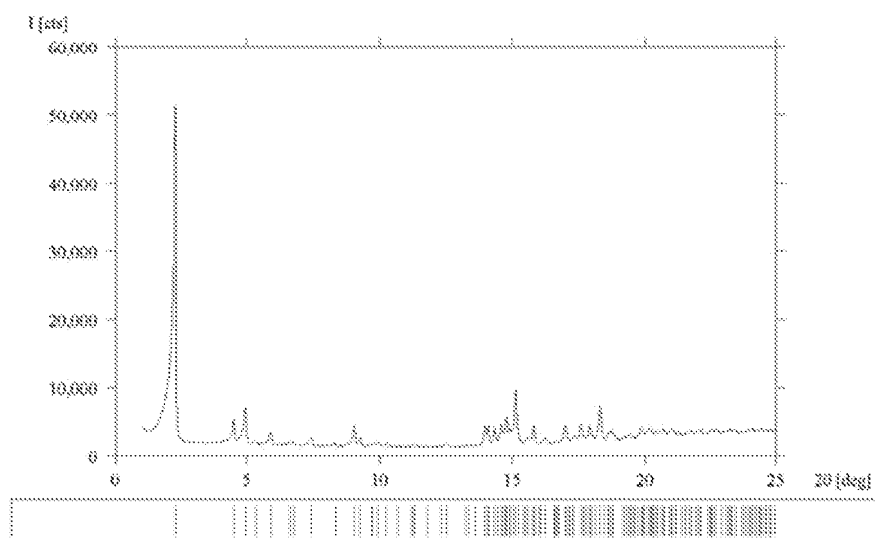


Figure 17 – Indexing results for Form II (99:01 ACN/H₂O slurry, 0.21 wt, 3 days)

Indexing results for XRPD file 916672 collected with Cu-K α radiation.



Bravais Type	Primitive
a [Å]	6.553
b [Å]	18.259
c [Å]	78.133
α [deg]	90
β [deg]	90
γ [deg]	90
Volume [Å ³ /cell]	9,066.5
Chiral Contents?	Chiral
Extinction Symbol	P 2 ₁ 2 ₁ 2 ₁
Space Group(s)	P2 ₁ 2 ₁ 2 ₁ (19)

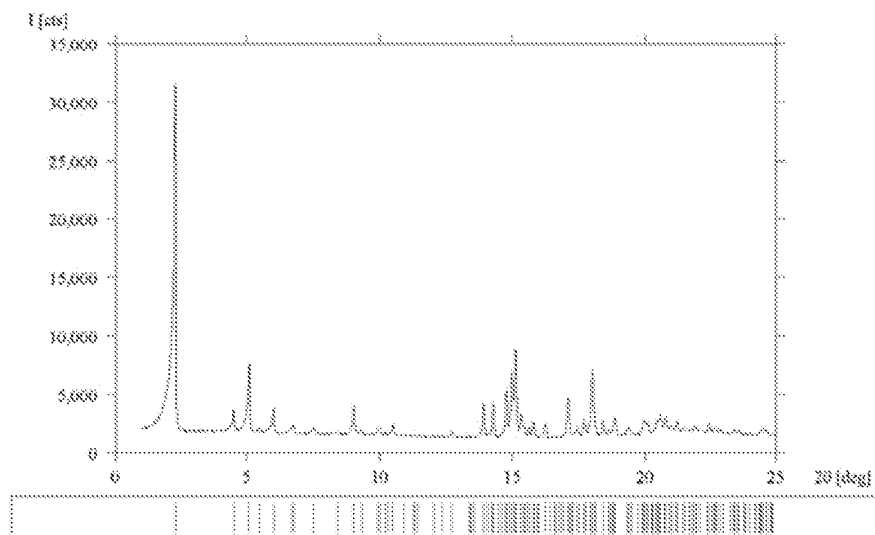
Source	Manual Input
--------	--------------

Indexing is conducted under the "Procedures for SSC/T Non-cGMP Activities."

Output rendered using Trade™ v2.0

Figure 18 – Indexing results for Form II (95:05 EtOH/H₂O slurry, 0.30a_w, 10 days)

Indexing results for XRPD file 916677 collected with Cu-K α radiation.



Bravais Type	Primitive
a [Å]	6.573
b [Å]	17.766
c [Å]	78.213
α [deg]	90
β [deg]	90
γ [deg]	90
Volume [Å ³ /cell]	8,858.5
Chiral Contents?	Chiral
Extinction Symbol	P 2 ₁ 2 ₁ 2 ₁
Space Group(s)	P2 ₁ 2 ₁ 2 ₁ (19)

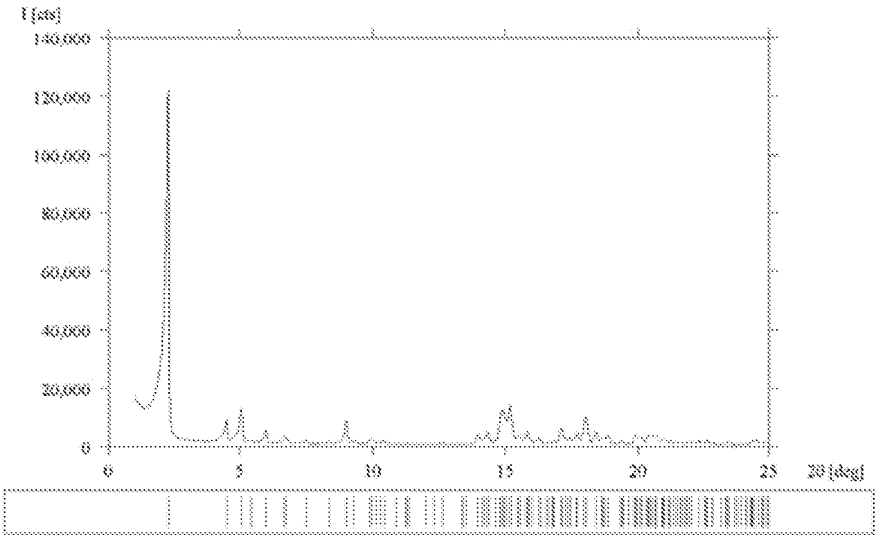
Source	Manual Input
--------	--------------

Indexing is conducted under the "Procedures for SSC/T Non-cGMP Activities."

Output rendered using Trade™ v2.0

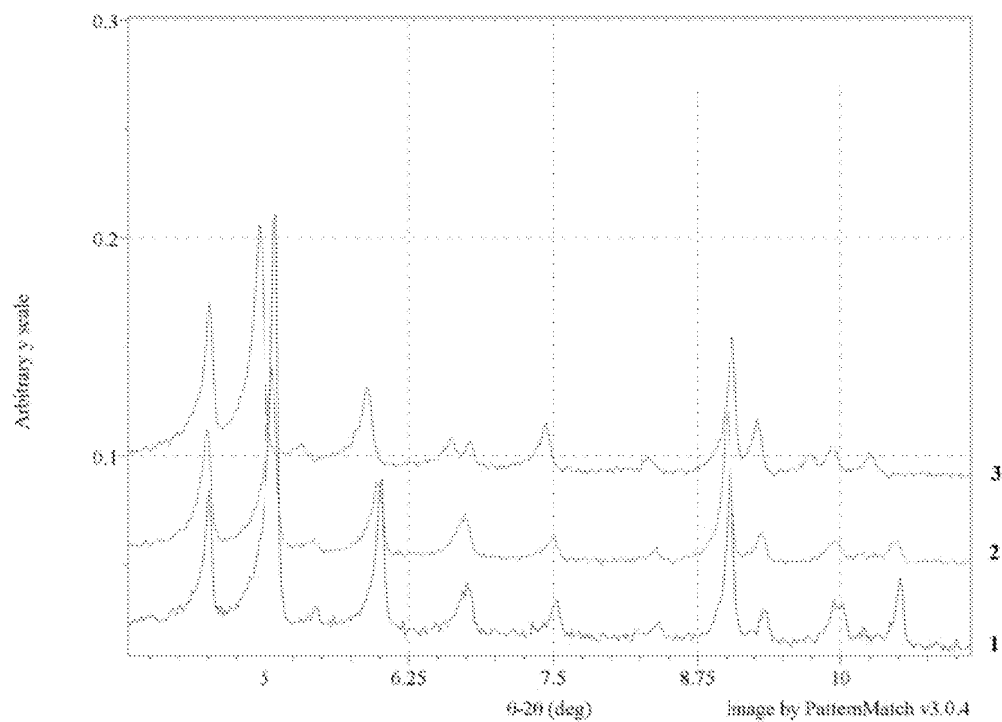
Figure 19 – Indexing results for Form II (Acetone slurry, 55°C, 1 day)

Indexing results for XRPD file 916689 collected with Cu-K α radiation.



Bravais Type	Primitive
a [Å]	6.553
b [Å]	17.863
c [Å]	78.433
α [deg]	90
β [deg]	90
γ [deg]	90
Volume [Å ³ /cell]	8,900.9
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 20 – XRPD peak position variability for Form II for slurries indexed in Figures 17 - 19, shown from approximately 4°2 θ to approximately 11°2 θ

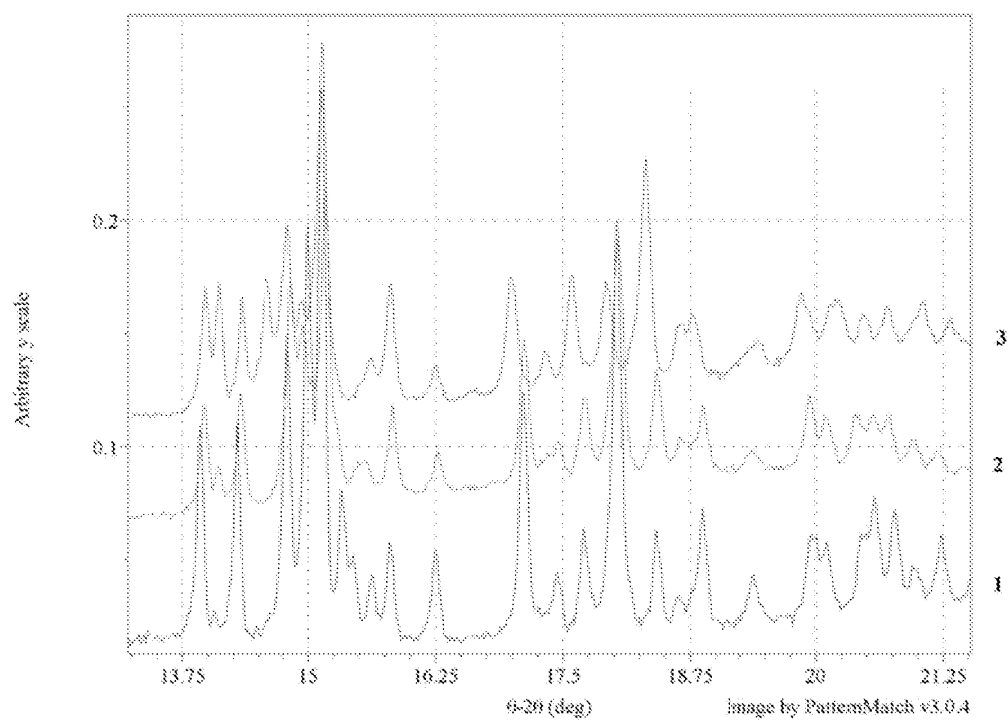


1 = 95:05 (EtOH/H₂O slurry, 0.30aw)

2 = Acetone slurry

3 = 99:01 (ACN/H₂O slurry, 0.21aw)

Figure 21 – XRPD peak position variability for Form II for slurries indexed in Figures 17 - 19, shown from approximately 13°2 θ to approximately 21.5°2 θ

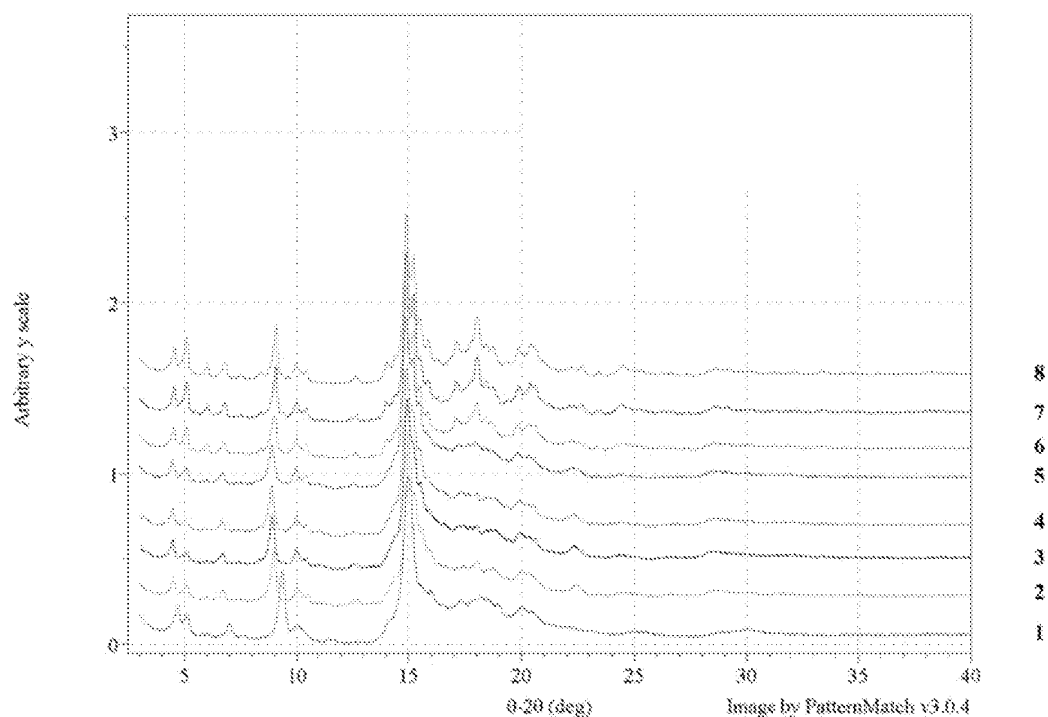


1 = 95:05 (EtOH/H₂O slurry, 0.30aw)

2 = Acetone slurry

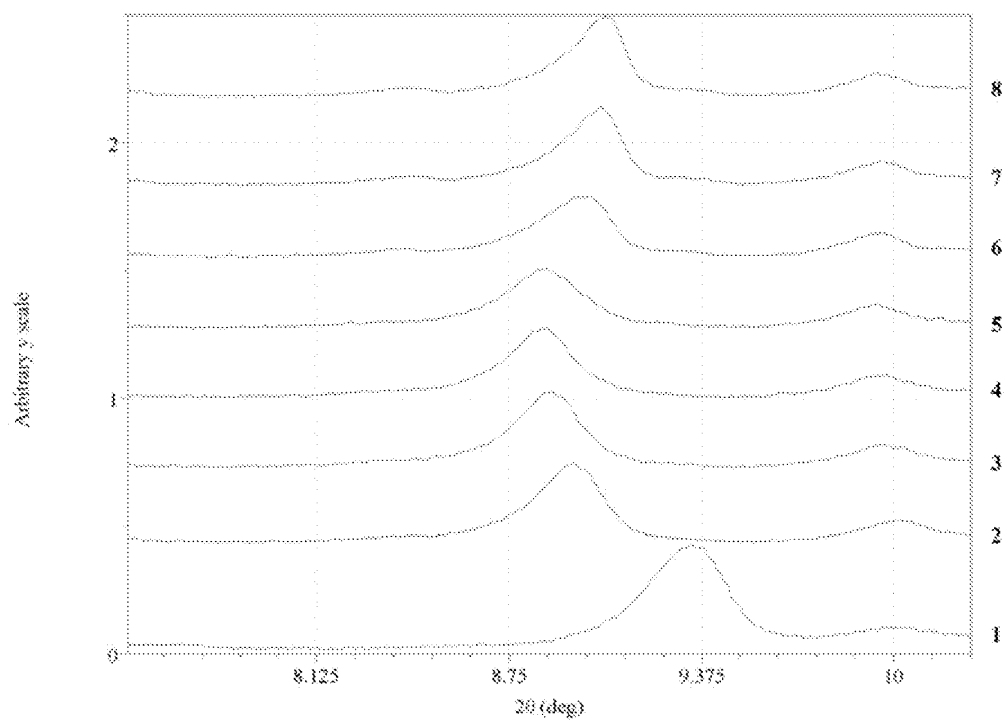
3 = 99:01 (ACN/H₂O slurry, 0.21aw)

Figure 22 – VRH-XRPD experiment for Form II



- 1 = Form II, 0% RH
- 2 = Form II, 25% RH
- 3 = Form II, 55% RH
- 4 = Form II, 75% RH
- 5 = Form II, 85% RH
- 6 = Form II, 75% RH
- 7 = Form II, 55% RH
- 8 = Form II, initial, 40% RH

Figure 23 – XRPD peak position variability for Form II observed by VH-XRPD, shown from approximately 7.5°2 θ to approximately 10.2°2 θ



1 = Form II, 0% RH

2 = Form II, 25% RH

3 = Form II, 55% RH

4 = Form II, 75% RH

5 = Form II, 85% RH

6 = Form II, 75% RH

7 = Form II, 55% RH

8 = Form II, initial, 40% RH

Figure 24 – TGA thermograms for Form II

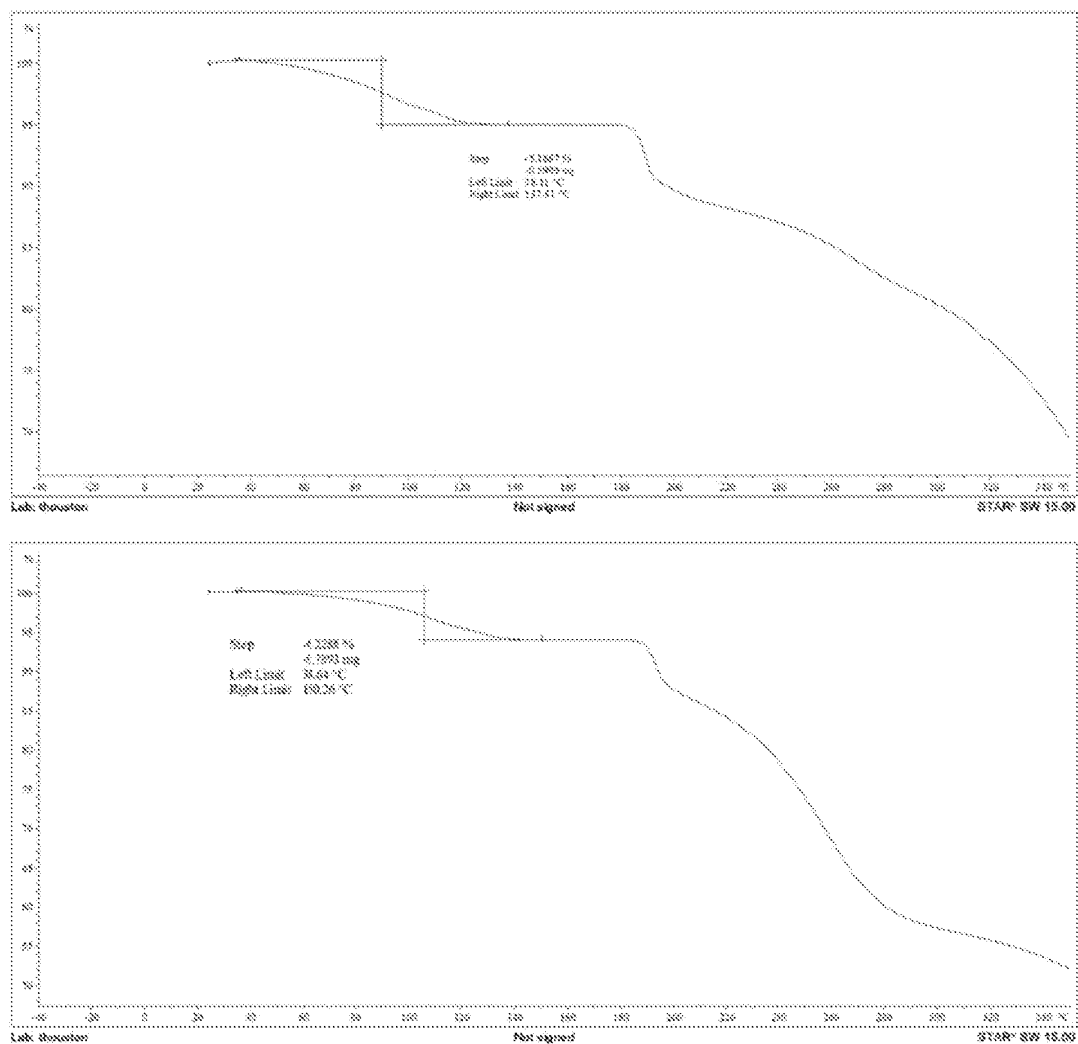


Figure 25 – DSC thermograms for Form II

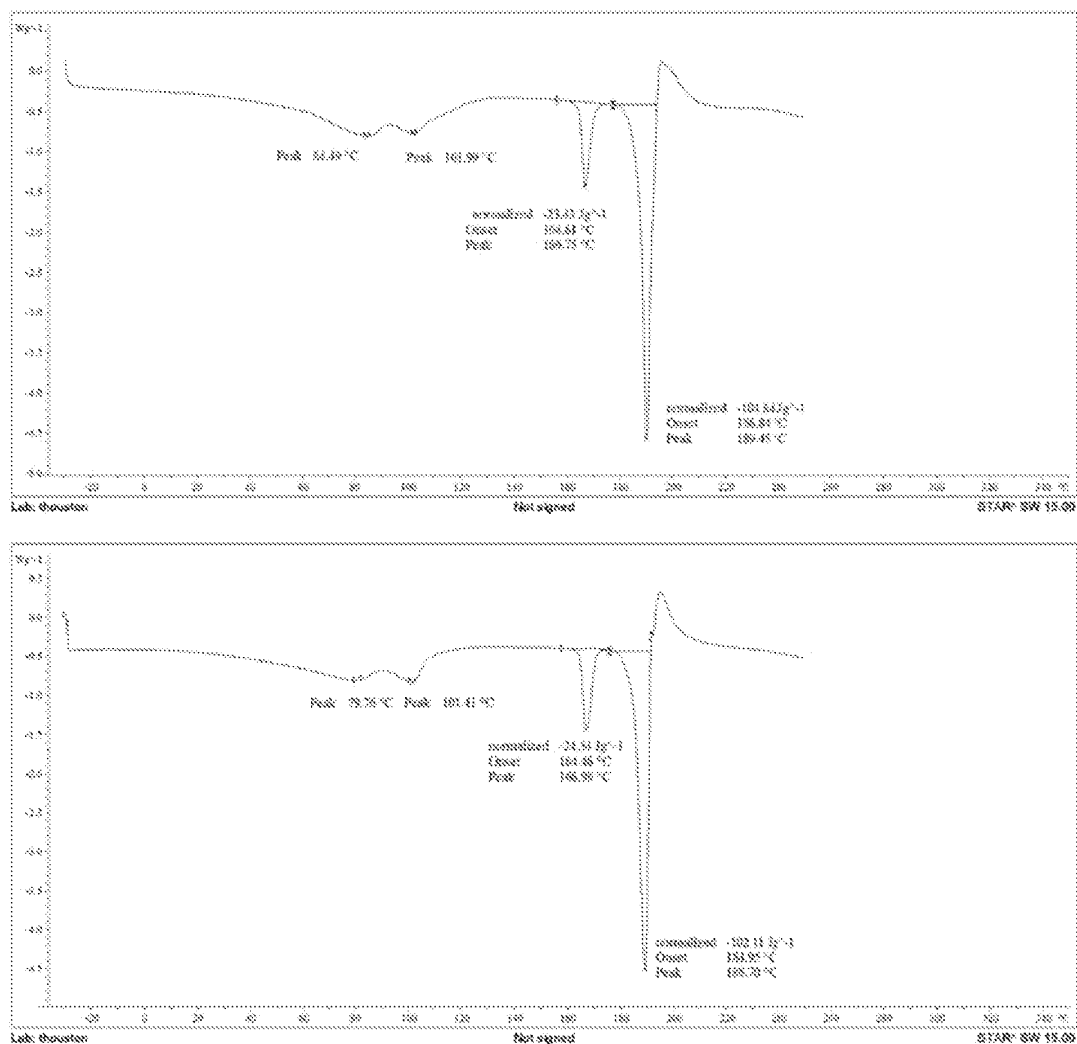
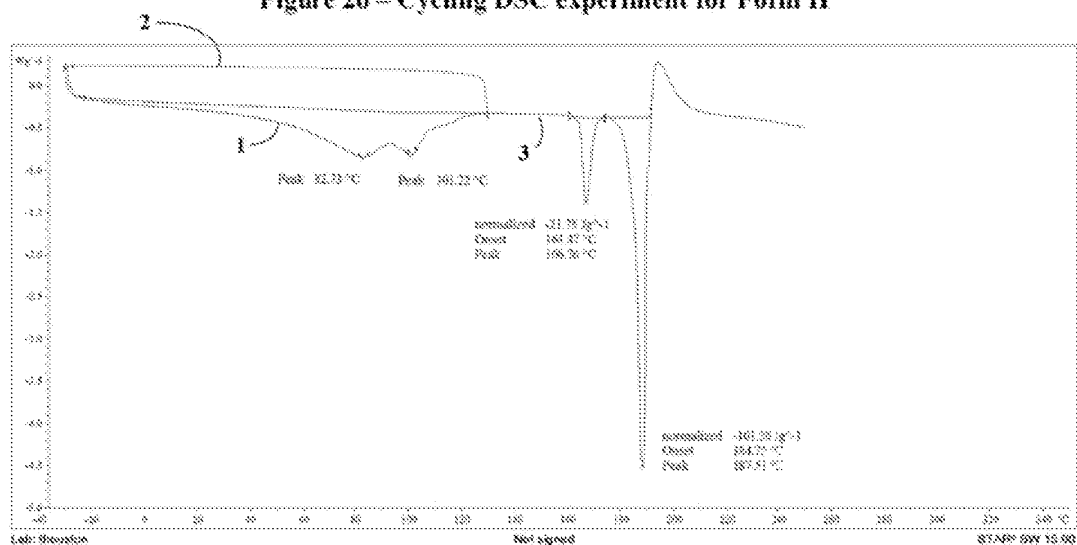


Figure 26 – Cycling DSC experiment for Form II

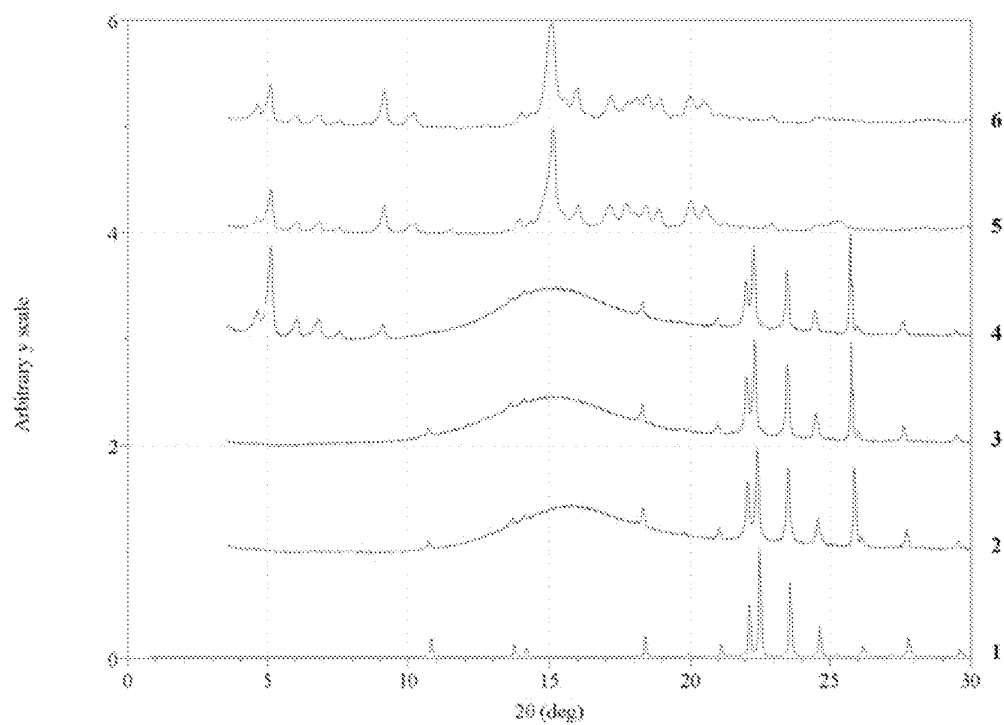


1 = Trace 1 (-30°C to 130°C)

2 = Trace 2 (130°C to -30°C)

3 = Trace 3 (-30°C to 250°C)

Figure 27 – VT-XRPD experiment for Form II



1 = Reference pattern of $\text{HNa}_3(\text{SO}_4)_2$ calculated from Cambridge Structural Database single crystal data

2 = $\text{HNa}_3(\text{SO}_4)_2$ (25.0°C)

3 = $\text{HNa}_3(\text{SO}_4)_2$ (135.0°C)

4 = Form II + $\text{HNa}_3(\text{SO}_4)_2$ (170.0°C)

5 = Form II (135.0°C)

6 = Form II (25.0°C)

Figure 28 – TGA thermogram for Form V

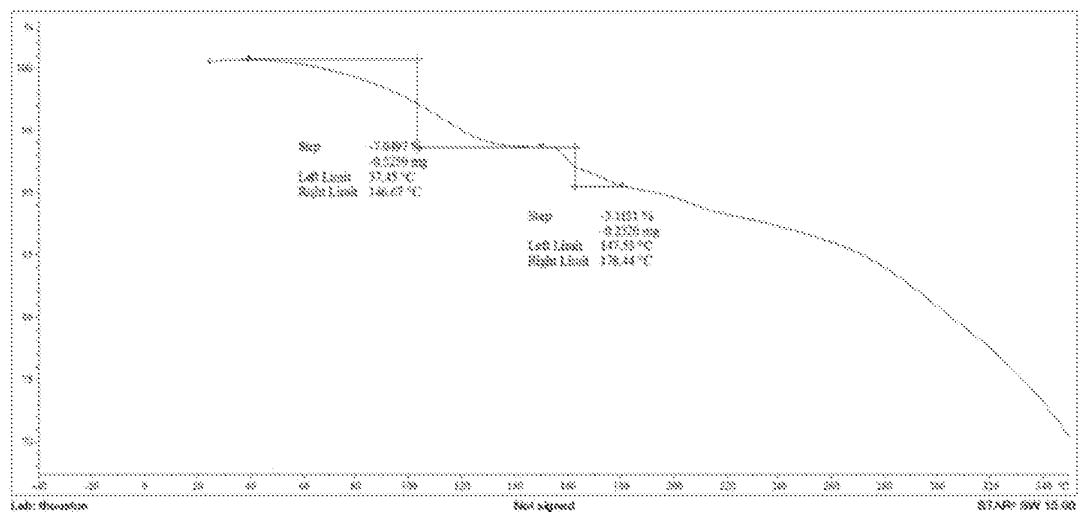


Figure 29 – DSC thermogram for Form V

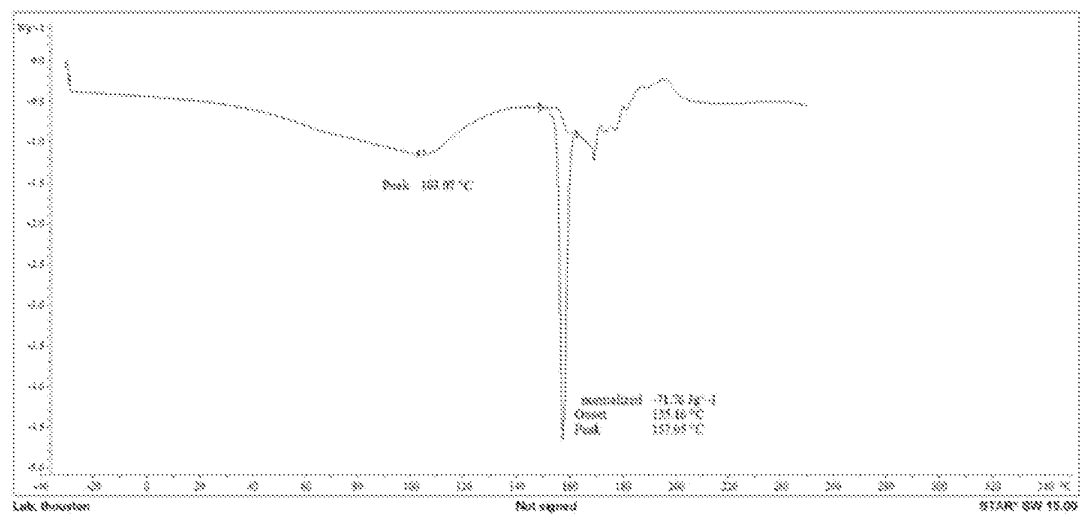
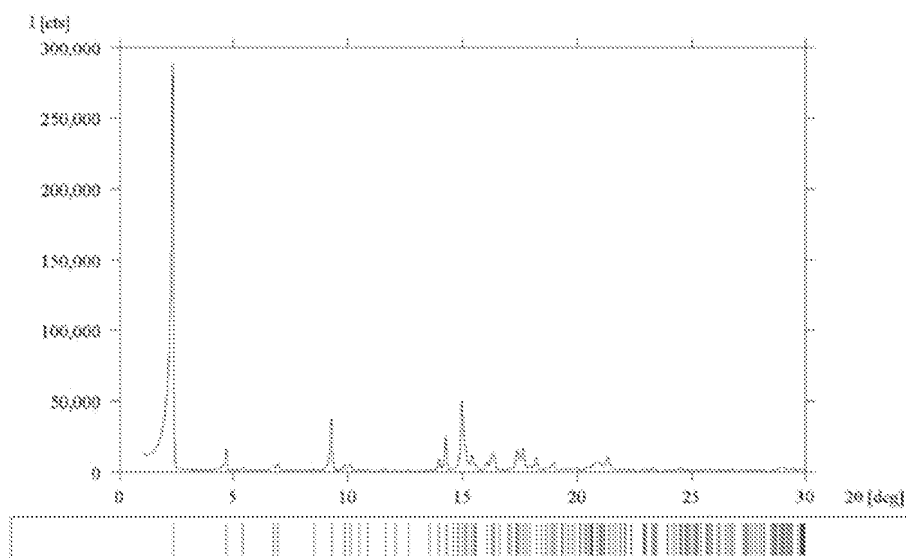
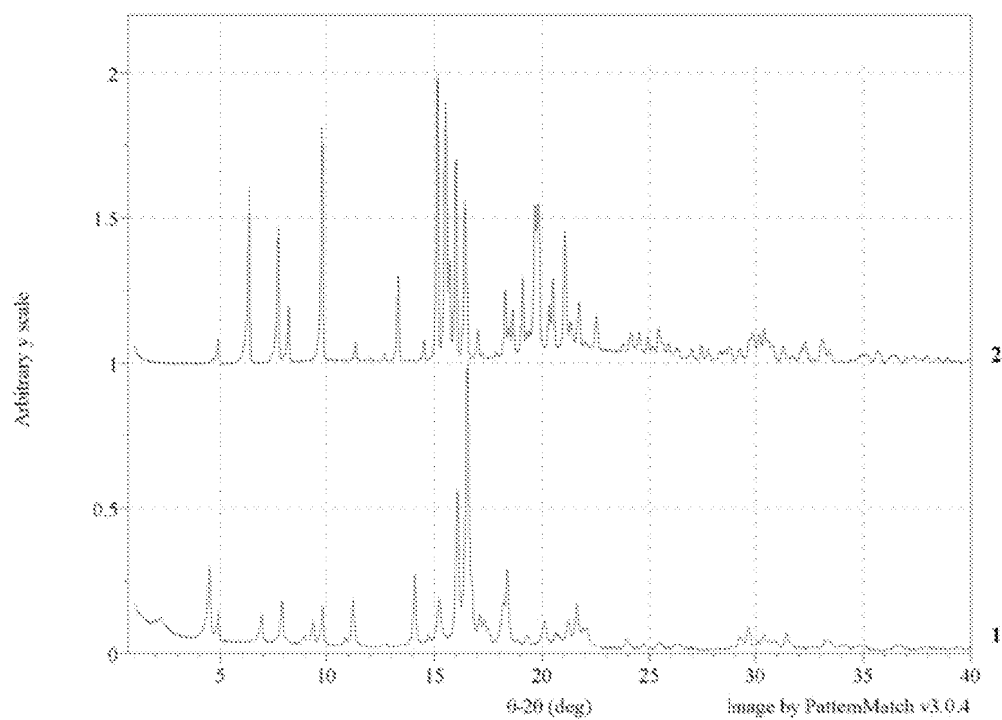


Figure 30 – Indexing results for Form XIII
(generated from a mixture of Form I and Form XIII exposed to 70°C vacuum for 2 days)

Indexing results for XRPD file 926366 collected with Cu-K α radiation.



Parameter Type	Primitive
Monoclinic	
a [Å]	6.208
b [Å]	17.944
c [Å]	38.040
α [deg]	90
β [deg]	93.22
γ [deg]	90
Volume [Å ³ /cell]	4,230.8
Chiral Contents?	Chiral
Extinction Symbol	P 1 2 ₁ 1
Space Group(s)	P2 ₁ (4)
Source	Manual Input

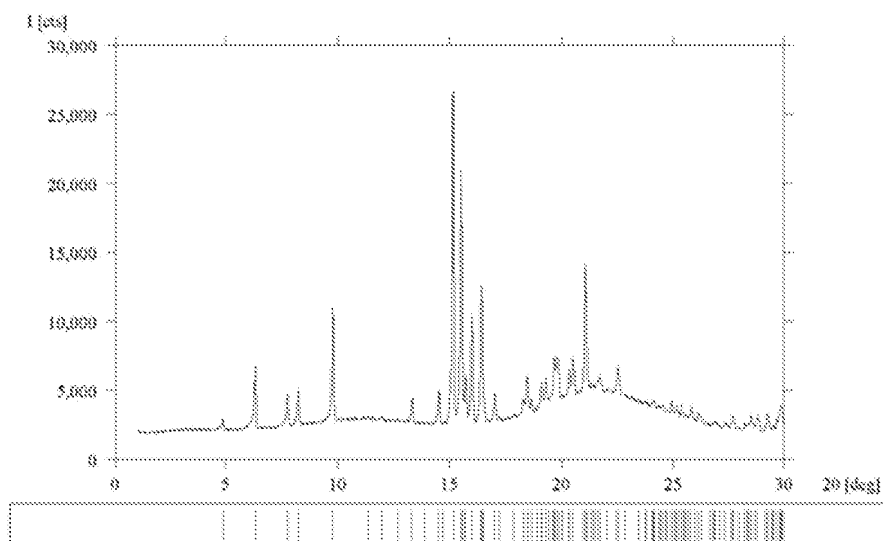
Figure 31 – XRPD Diffractograms of Form III and Form IX

1 = Form IX

2 = Form III

Figure 32 – Indexing results for Form III

Indexing results for XRPD file 914954 collected with Cu-K α radiation.



Bravais Type	Triclinic
a [Å]	6.099
b [Å]	13.962
c [Å]	18.090
α [deg]	93.42
β [deg]	92.09
γ [deg]	91.25
Volume [Å ³ /cell]	1,536.2
Chiral Contents?	Chiral
Extinction Symbol	P -
Space Group(s)	P1 (1)
Source	Triads™ Algorithm

Indexing is conducted under the "Procedures for SSC/NON-GLBP Activities."

Output modeled using Triads™ v2.0

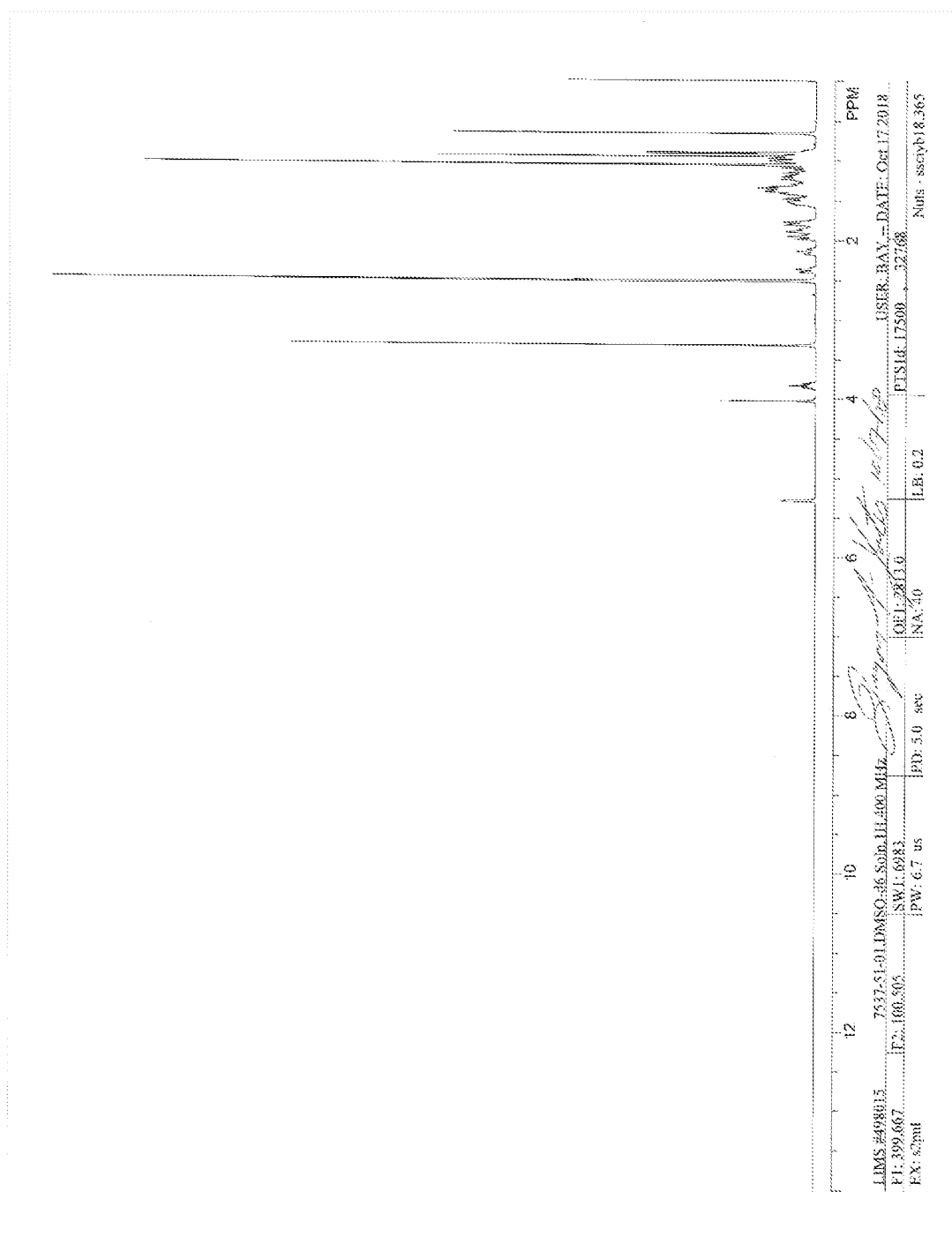
Figure 33 – ^1H -NMR spectrum of Form IX

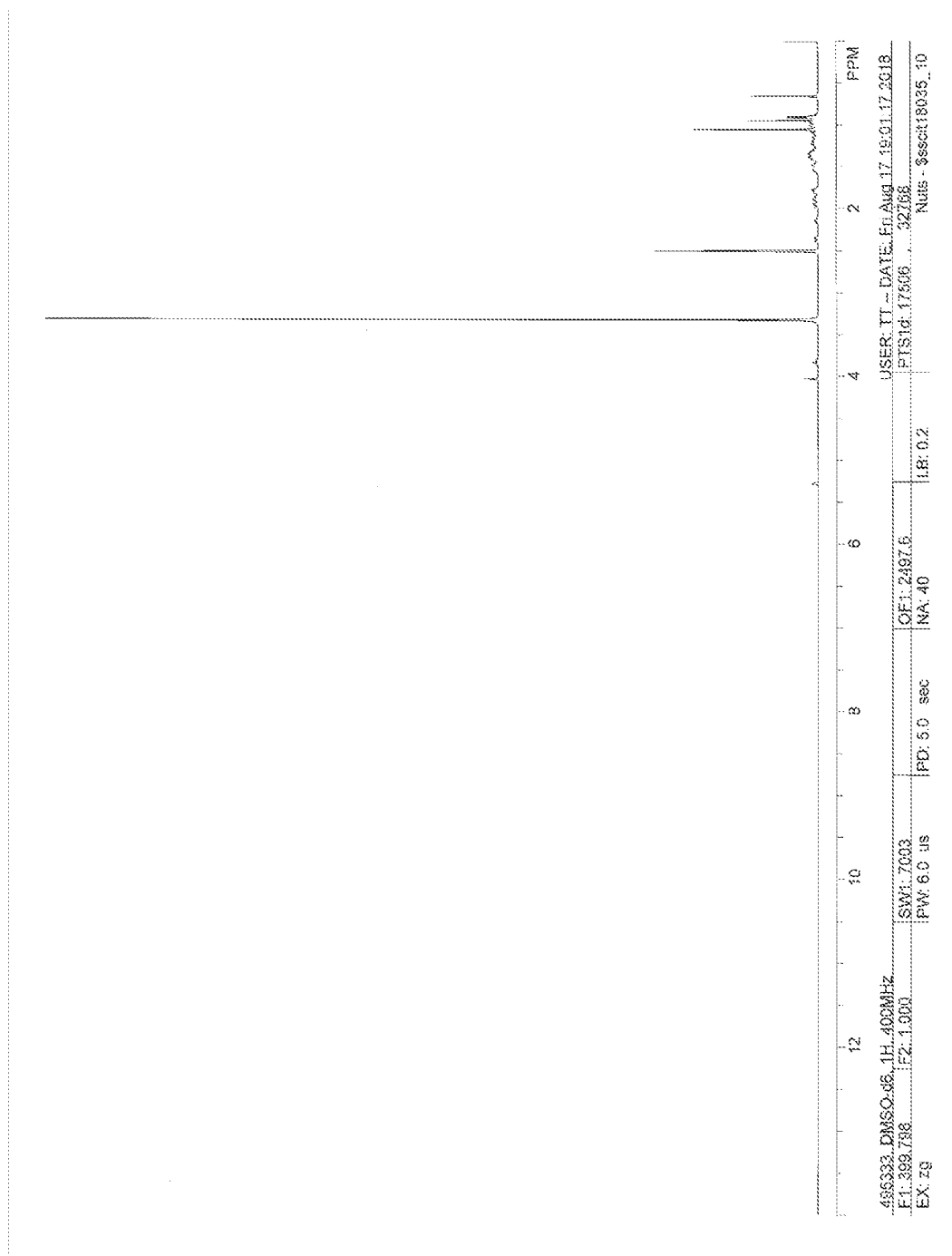
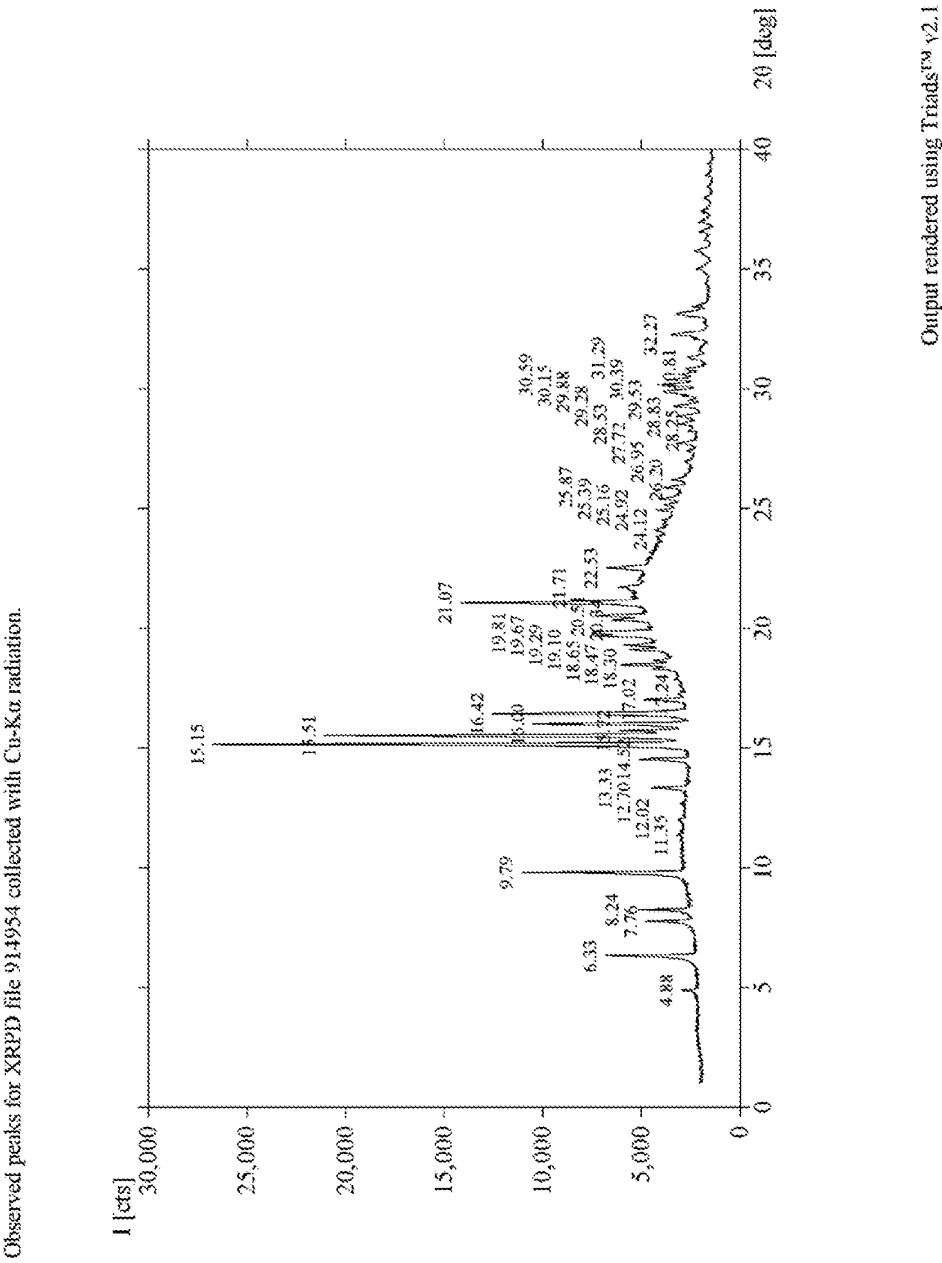
Figure 34 – ^1H -NMR spectrum of 25HC3S sodium

Figure 35 – Form III XRPD Diffractogram



Output rendered using Triads™ v2.1

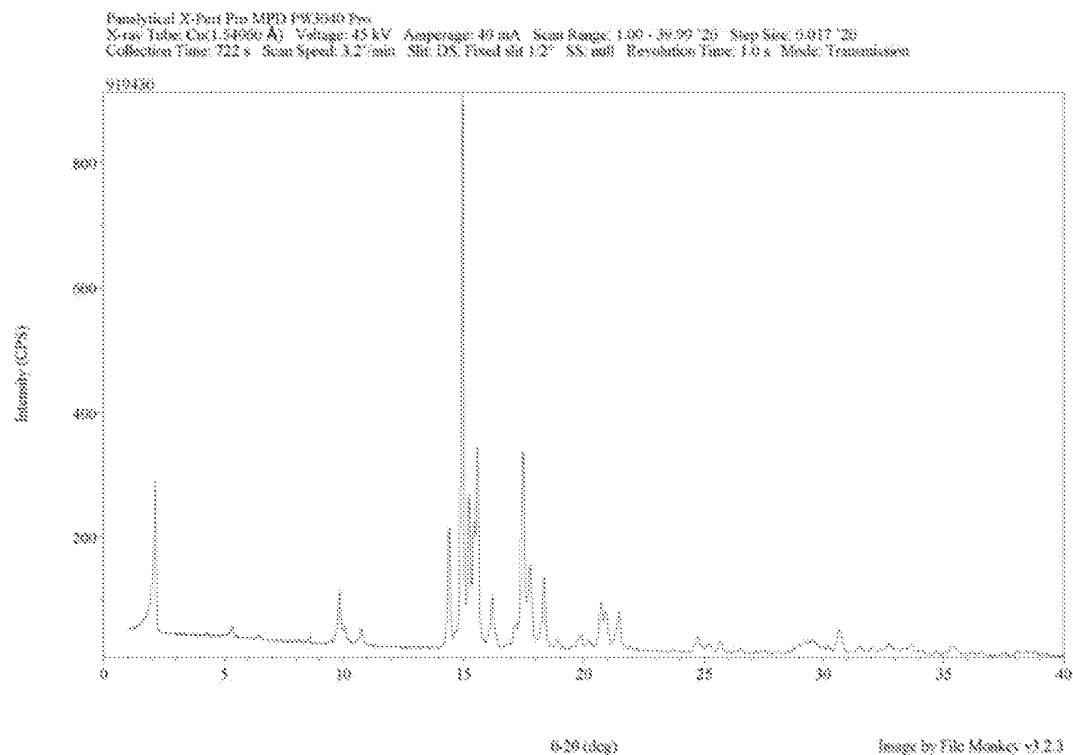
Figure 36 – Form I XRPD Diffractogram

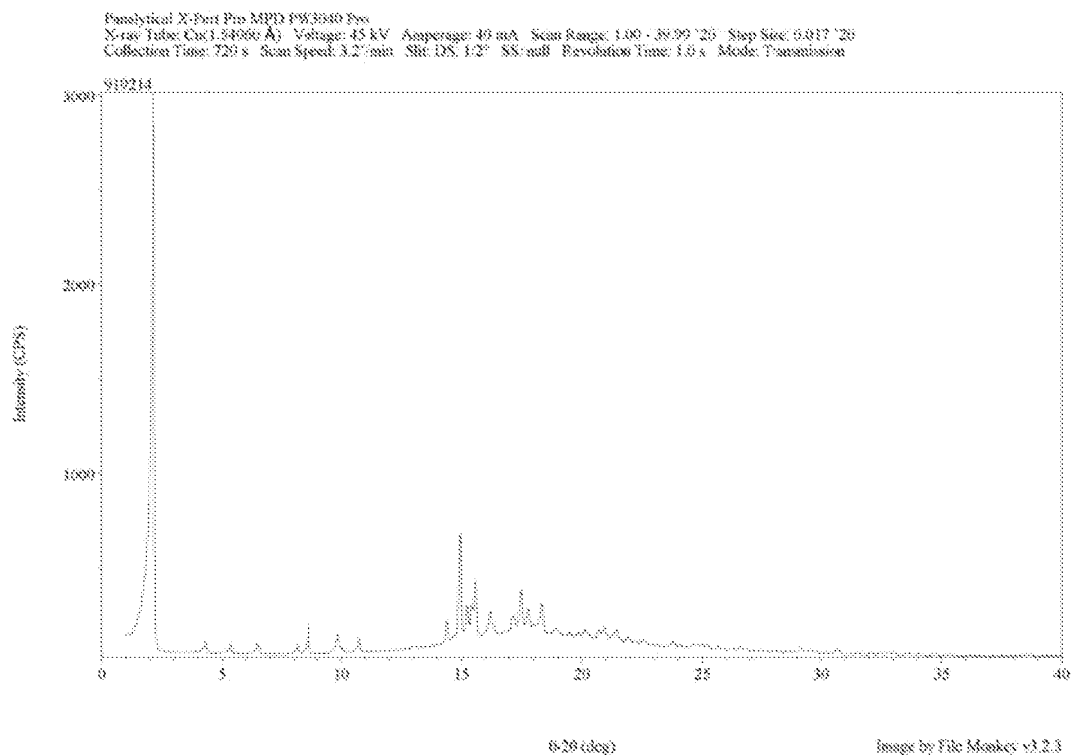
Figure 37 – Form I XRPD Diffractogram

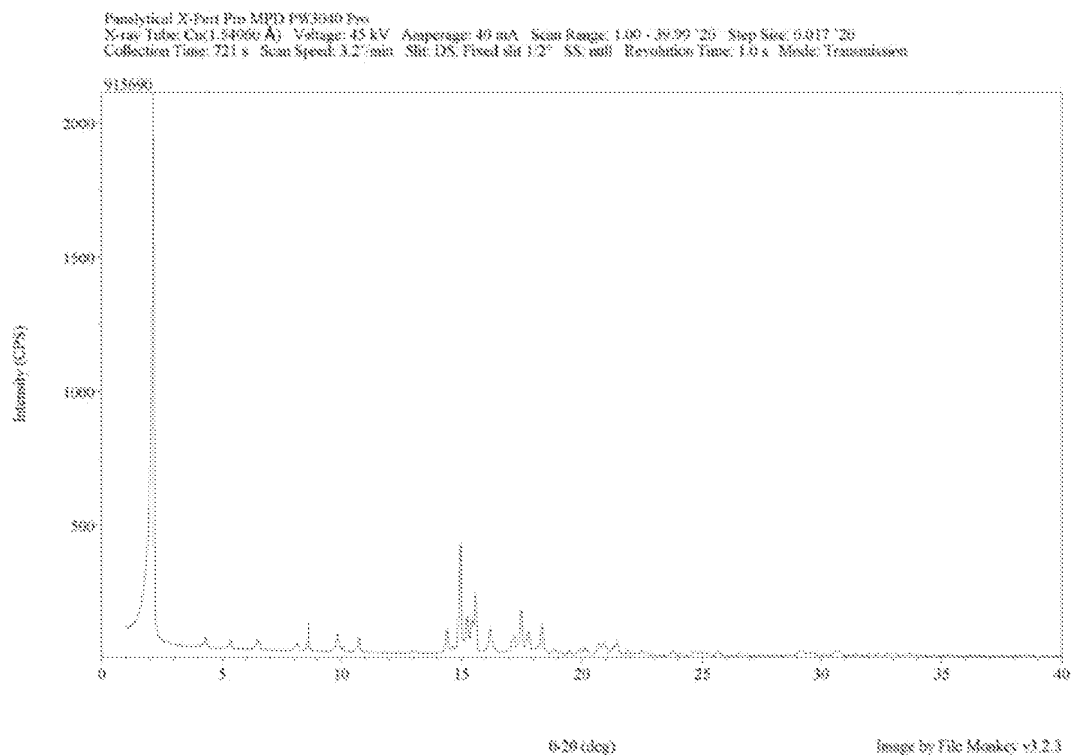
Figure 38 – Form I XRPD Diffractogram

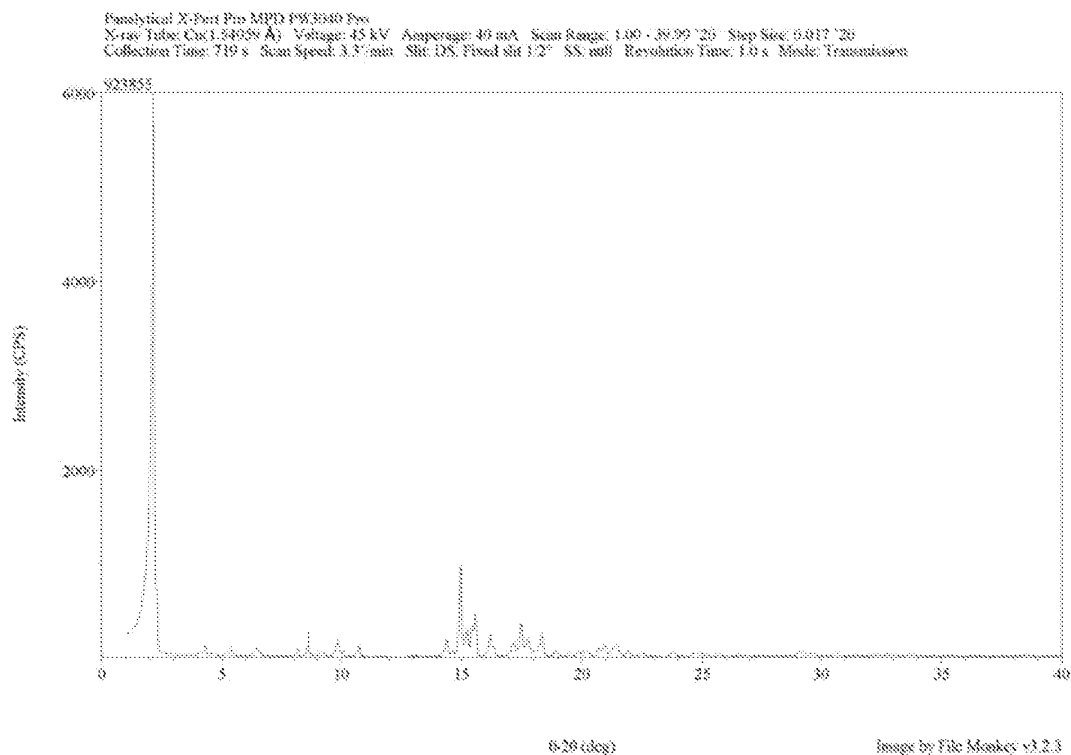
Figure 39 – XRPD Diffractograms of Form I and Form XIII

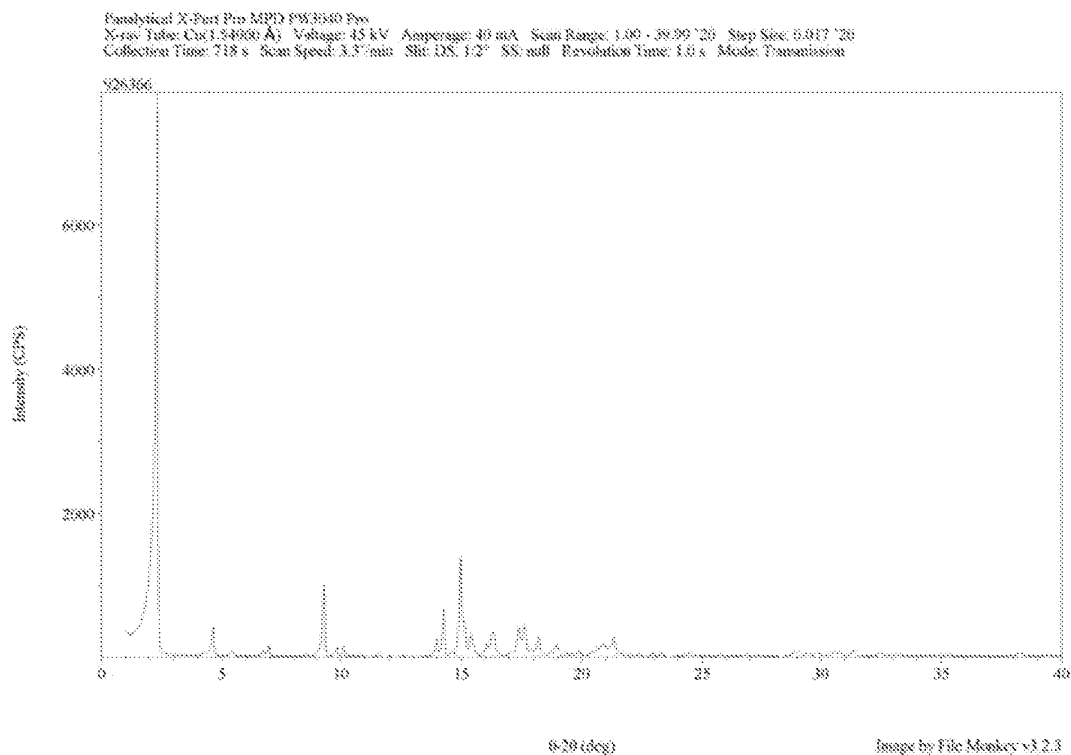
Figure 40 – Form XIII XRPD Diffractogram

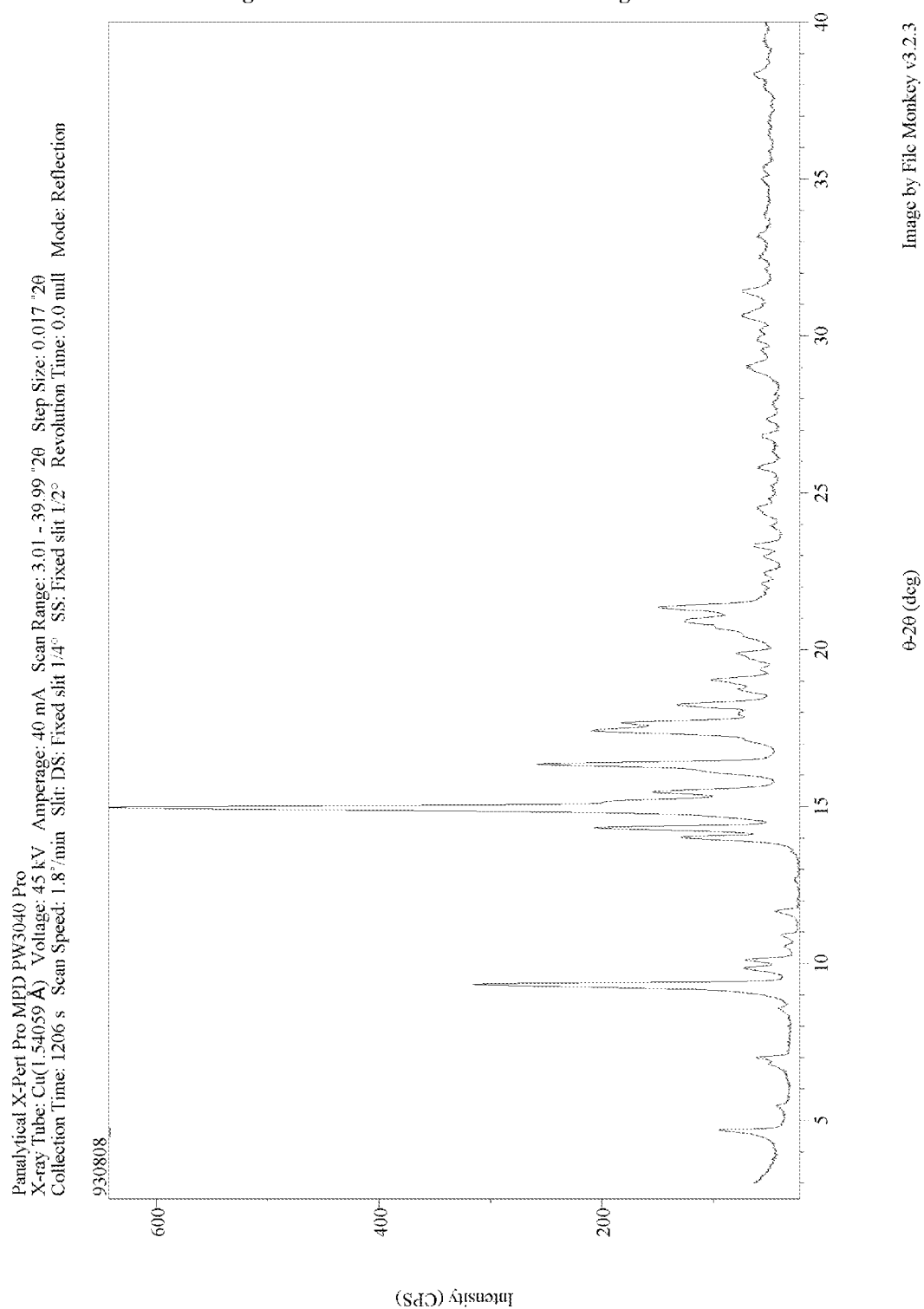
Figure 41 – Form XIII XRPD Diffractogram

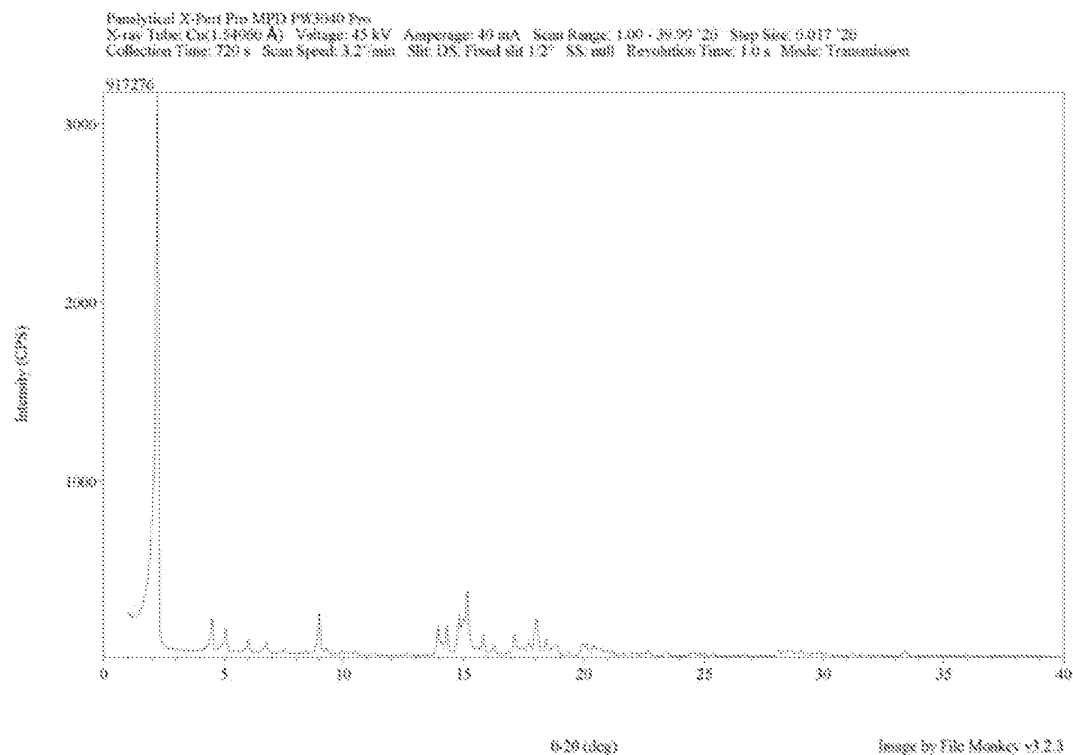
Figure 42 – Form II XRPD Diffractogram

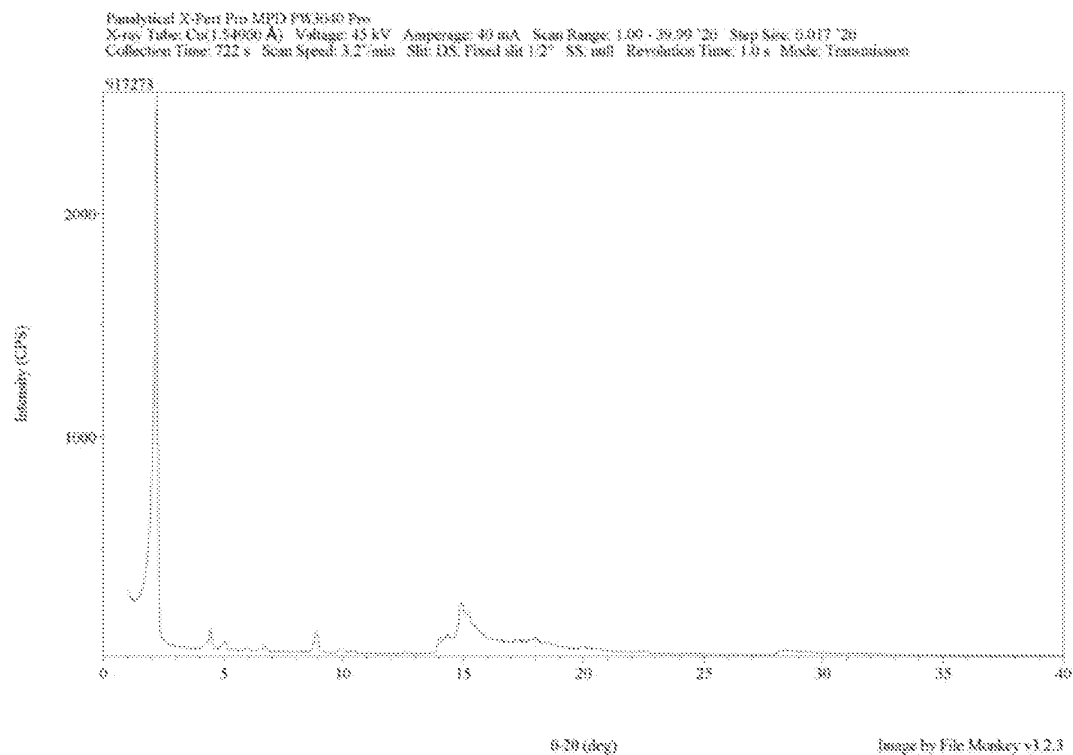
Figure 43 – Form II XRPD Diffractogram

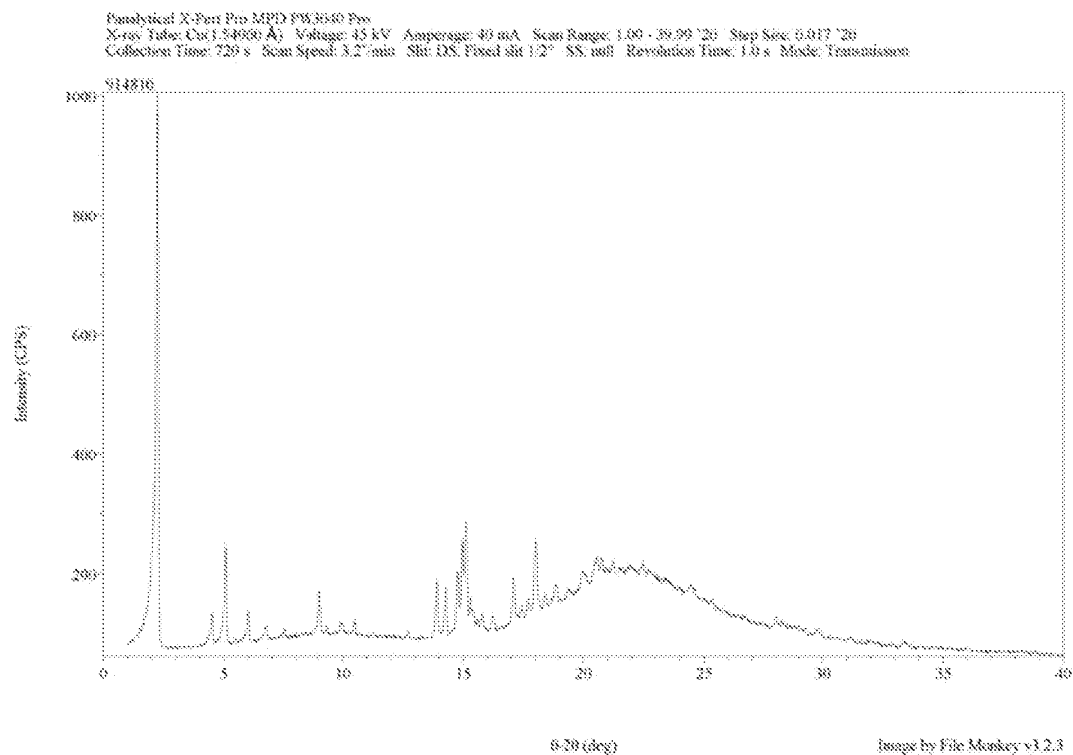
Figure 44 – Form II XRPD Diffractogram

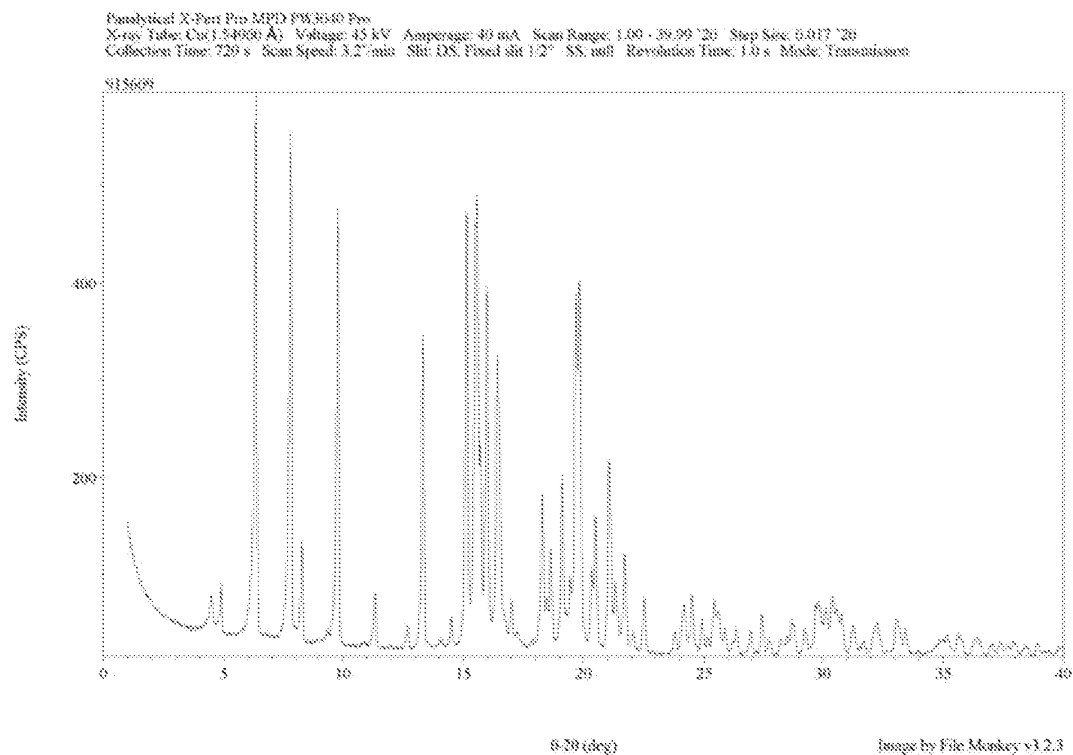
Figure 45 – XRPD Diffractogram of Form III and Form IX Mixture

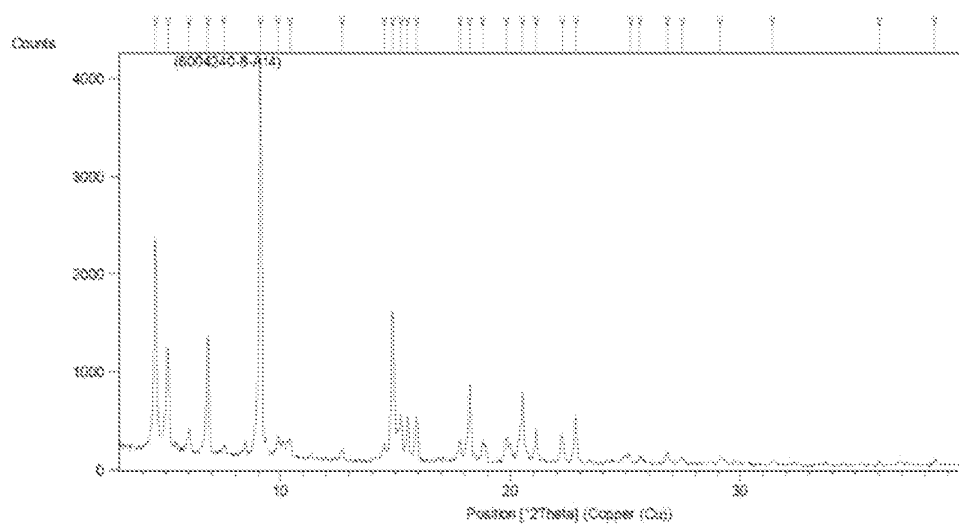
Figure 46 – Form II XRPD Diffractogram

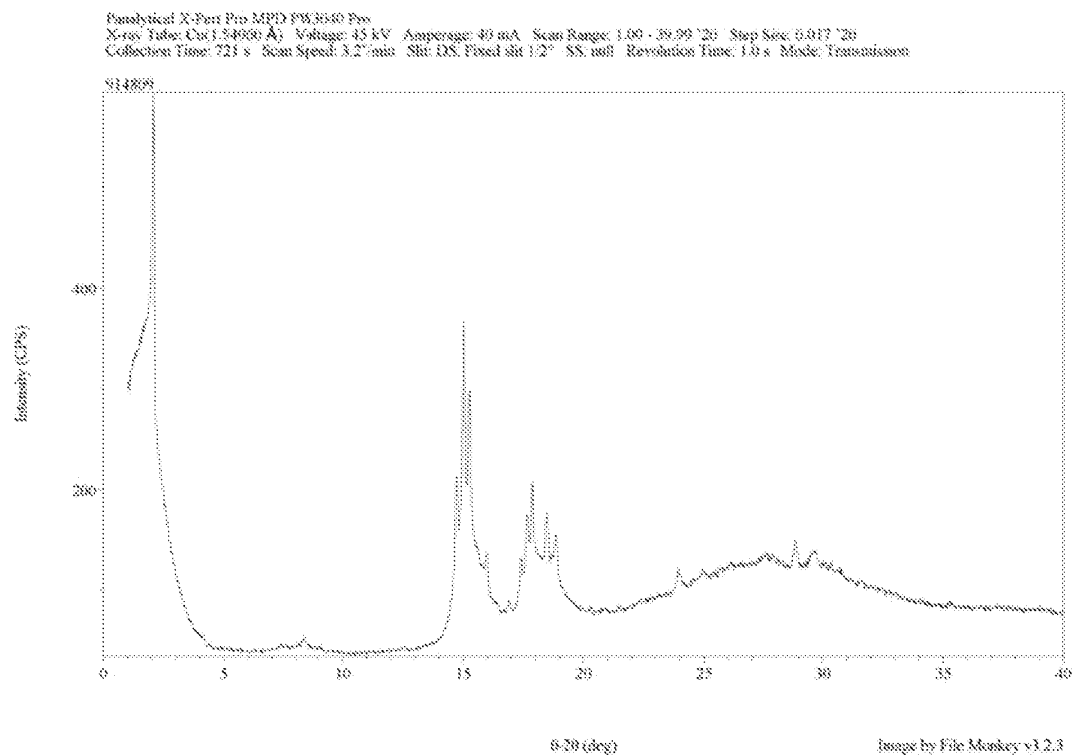
Figure 47 – Form V XRPD Diffractogram

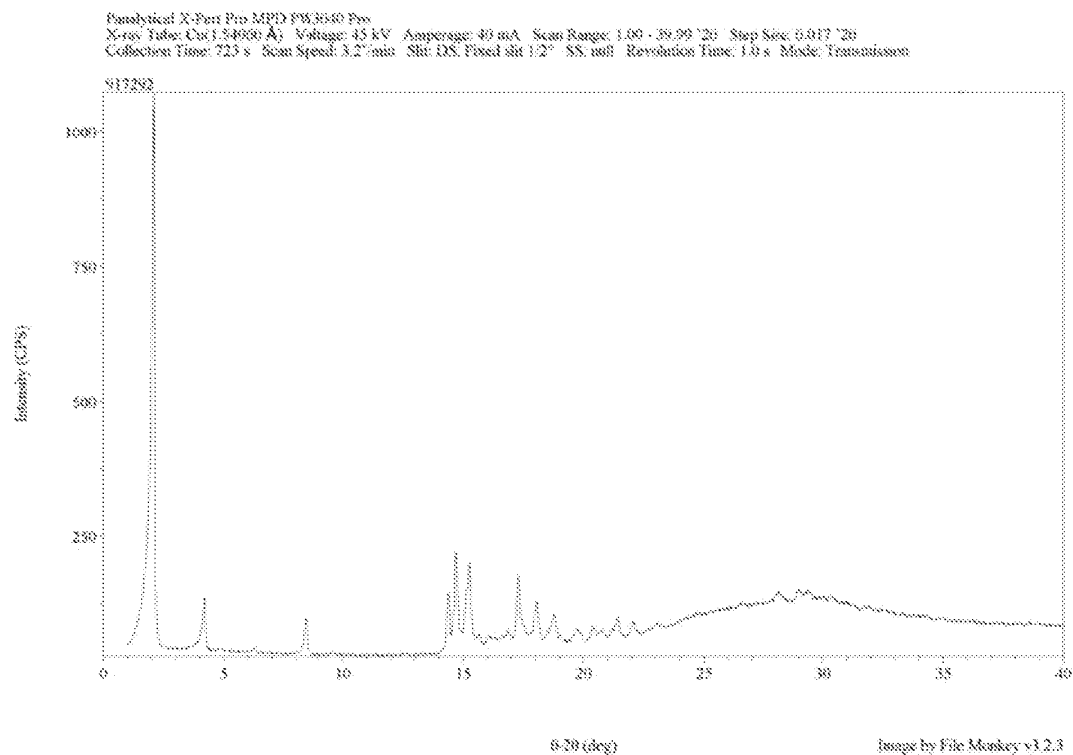
Figure 48 – Form V XRPD Diffractogram

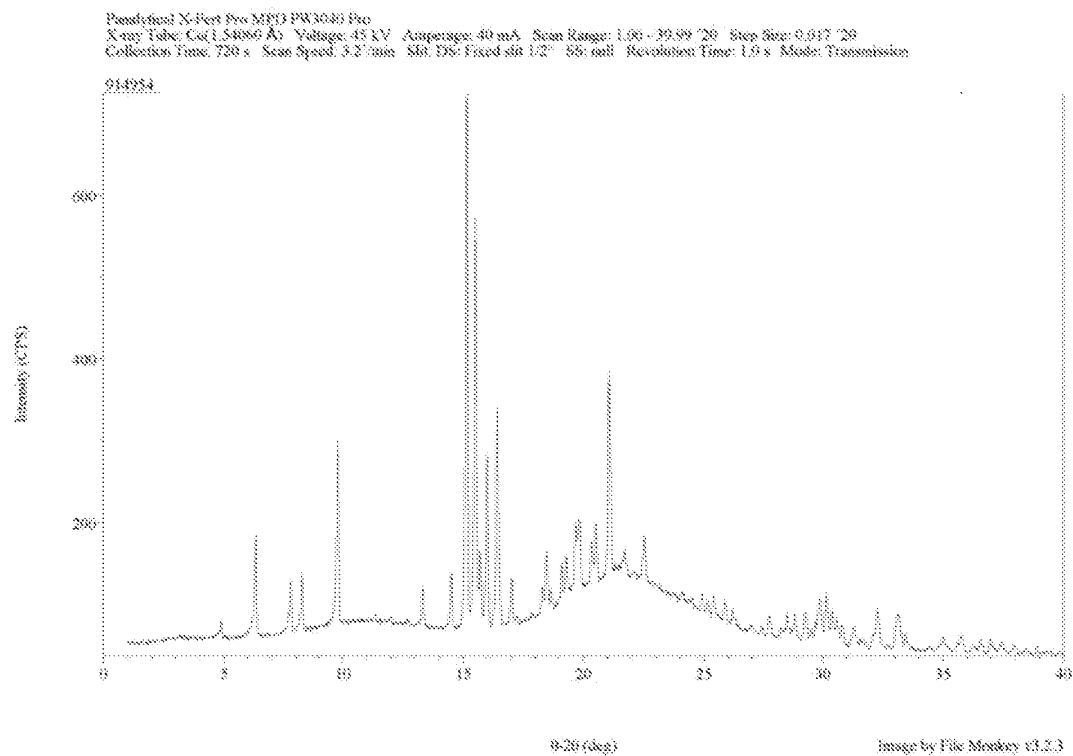
Figure 49 – Form III XRPD Diffractogram

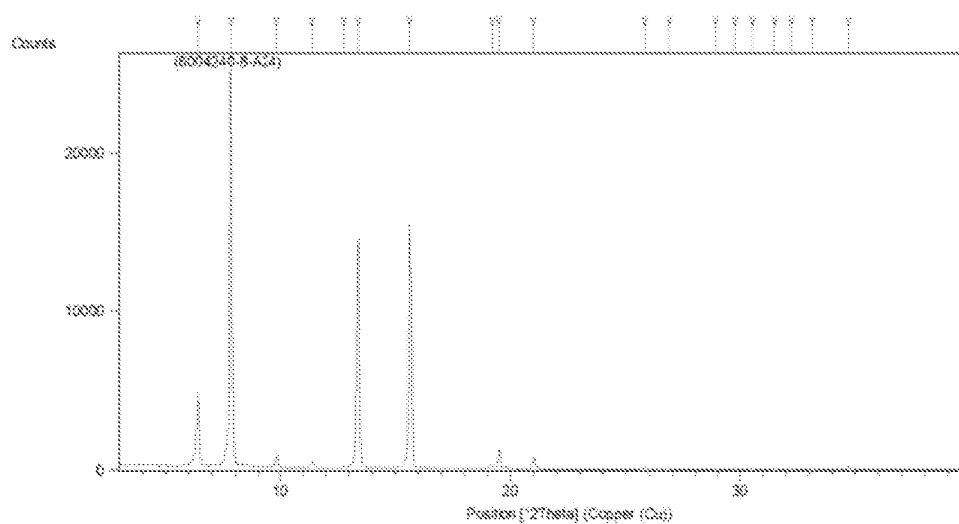
Figure 50 – Form III XRPD Diffractogram

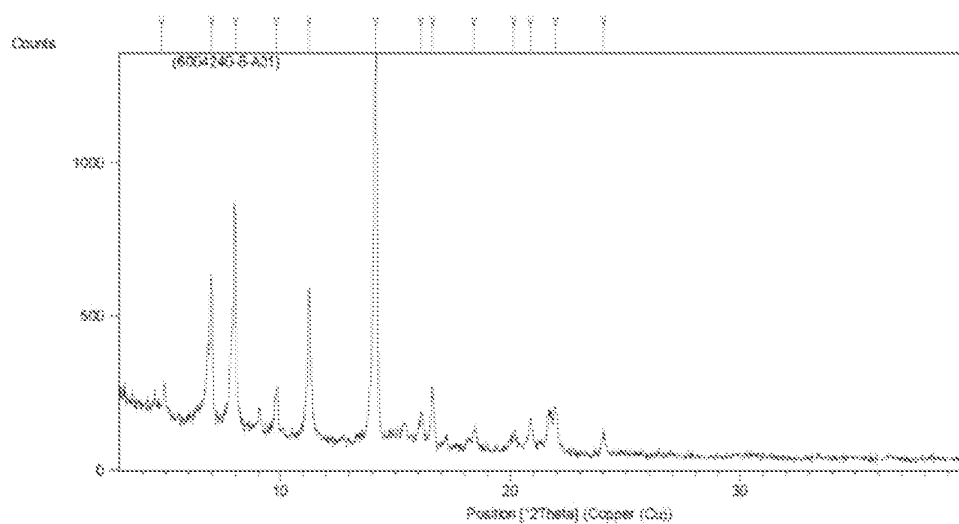
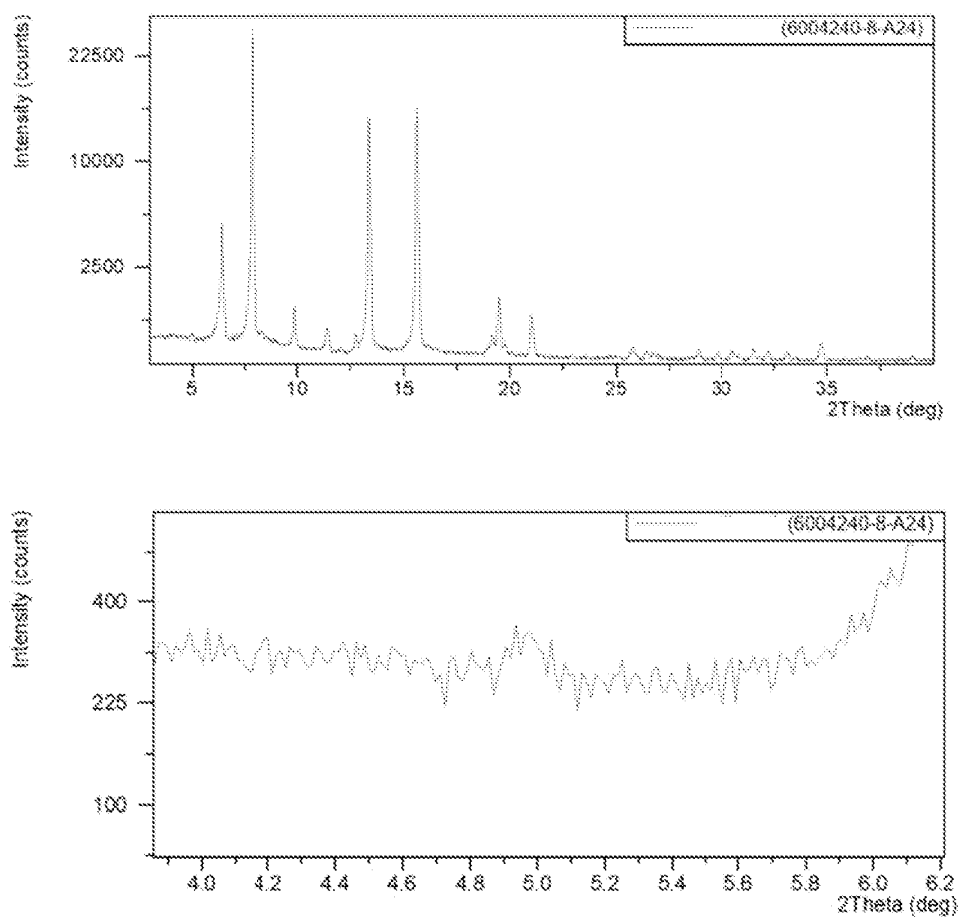
Figure 51 – Form IX XRPD Diffractogram

Figure 52 – Form III XRPD Diffractogram and Expanded

XRPD Results indicate that there is a small peak at $2\theta \sim 4.9$

Figure 53 – Form I XRPD Diffractogram

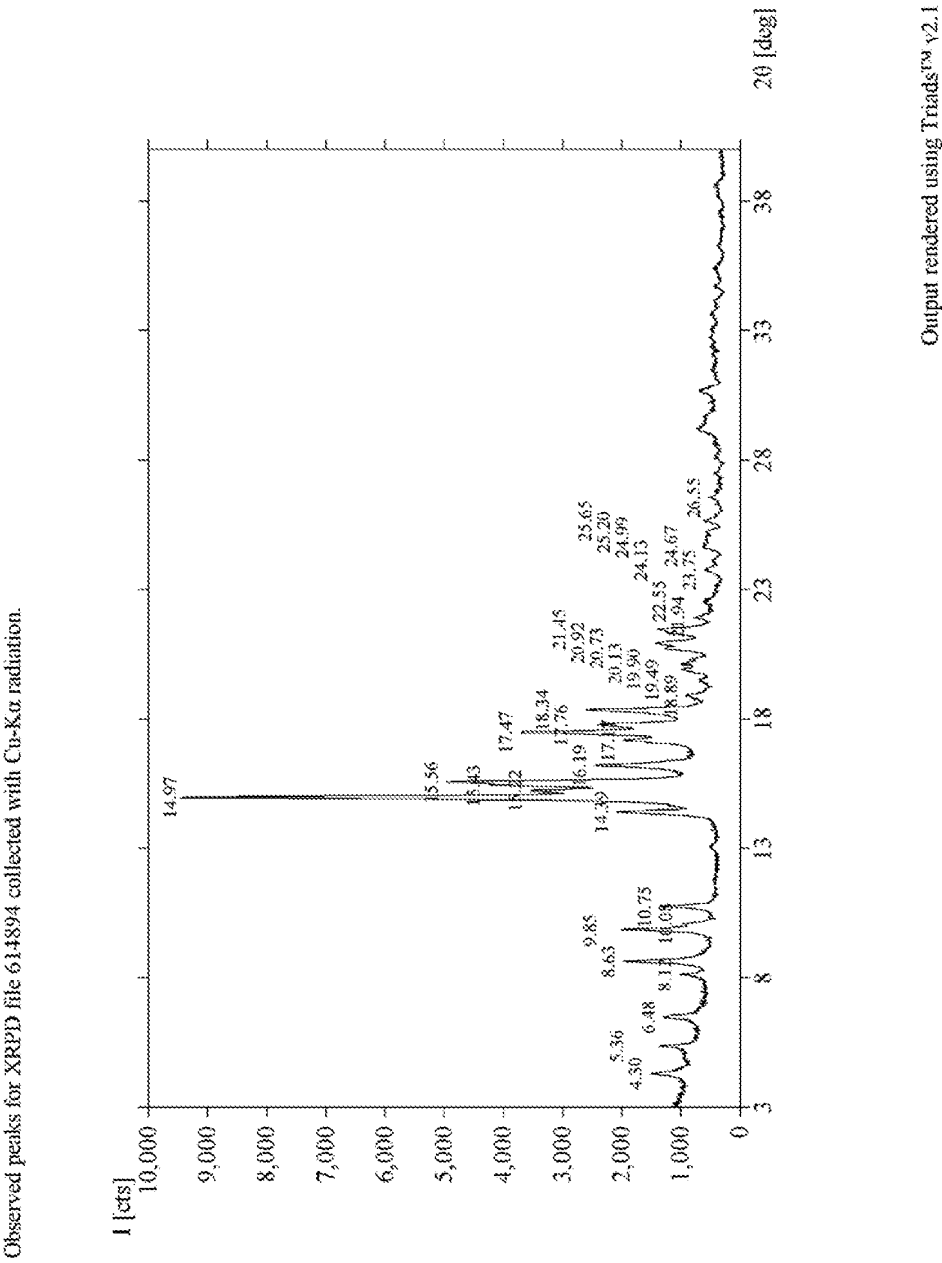
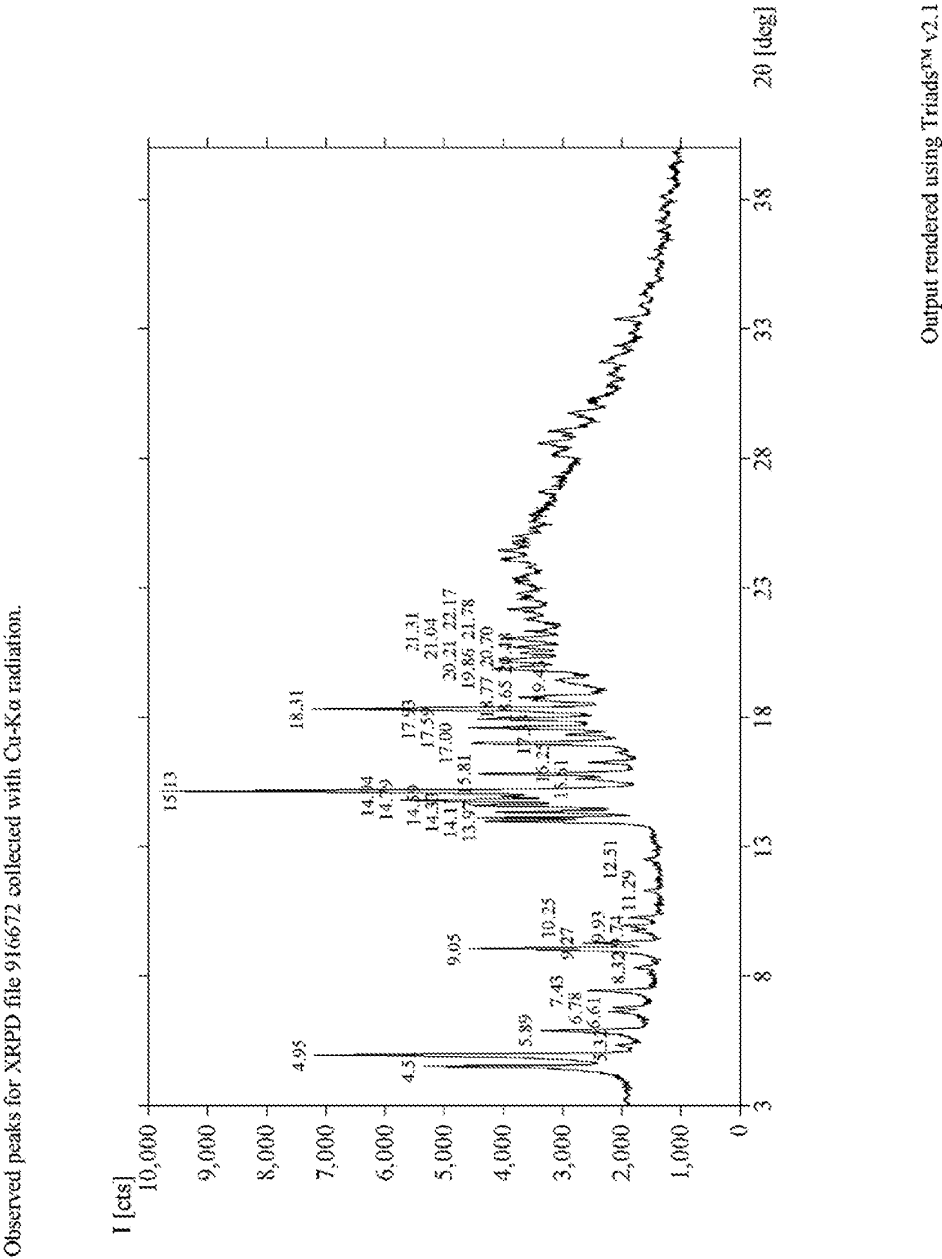


Figure 54 – Form II XRPD Diffractogram



Output rendered using Triads™ v2.1

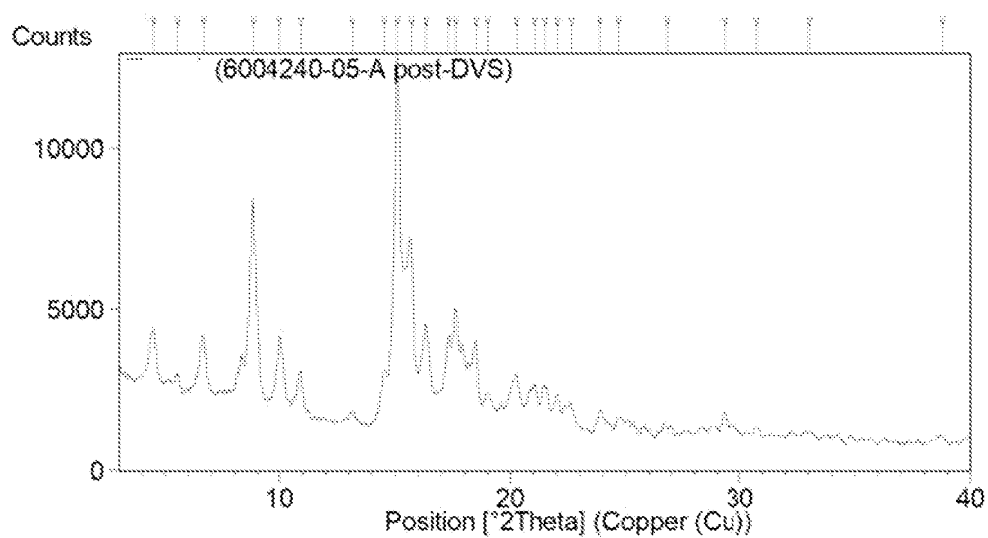
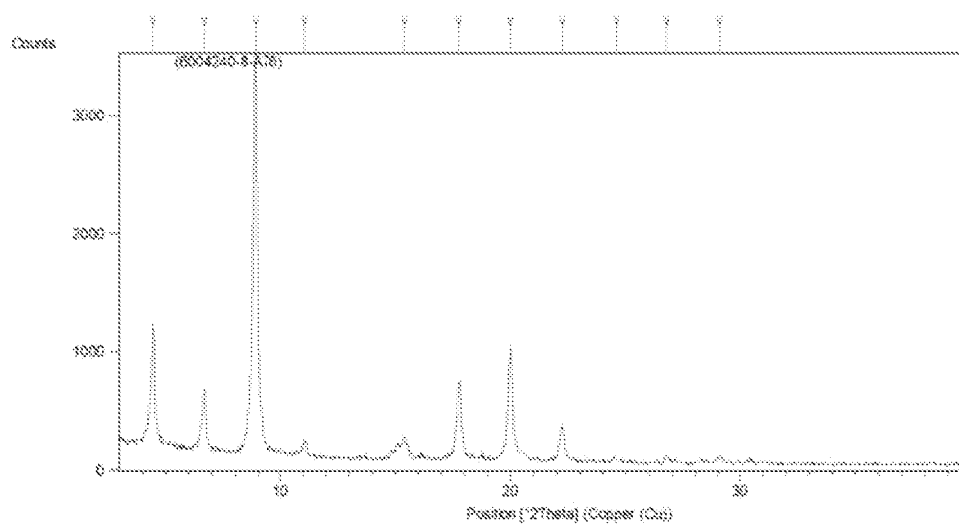
Figure 55 – Form I XRPD Diffractogram

Figure 56 – Form V XRPD Diffractogram



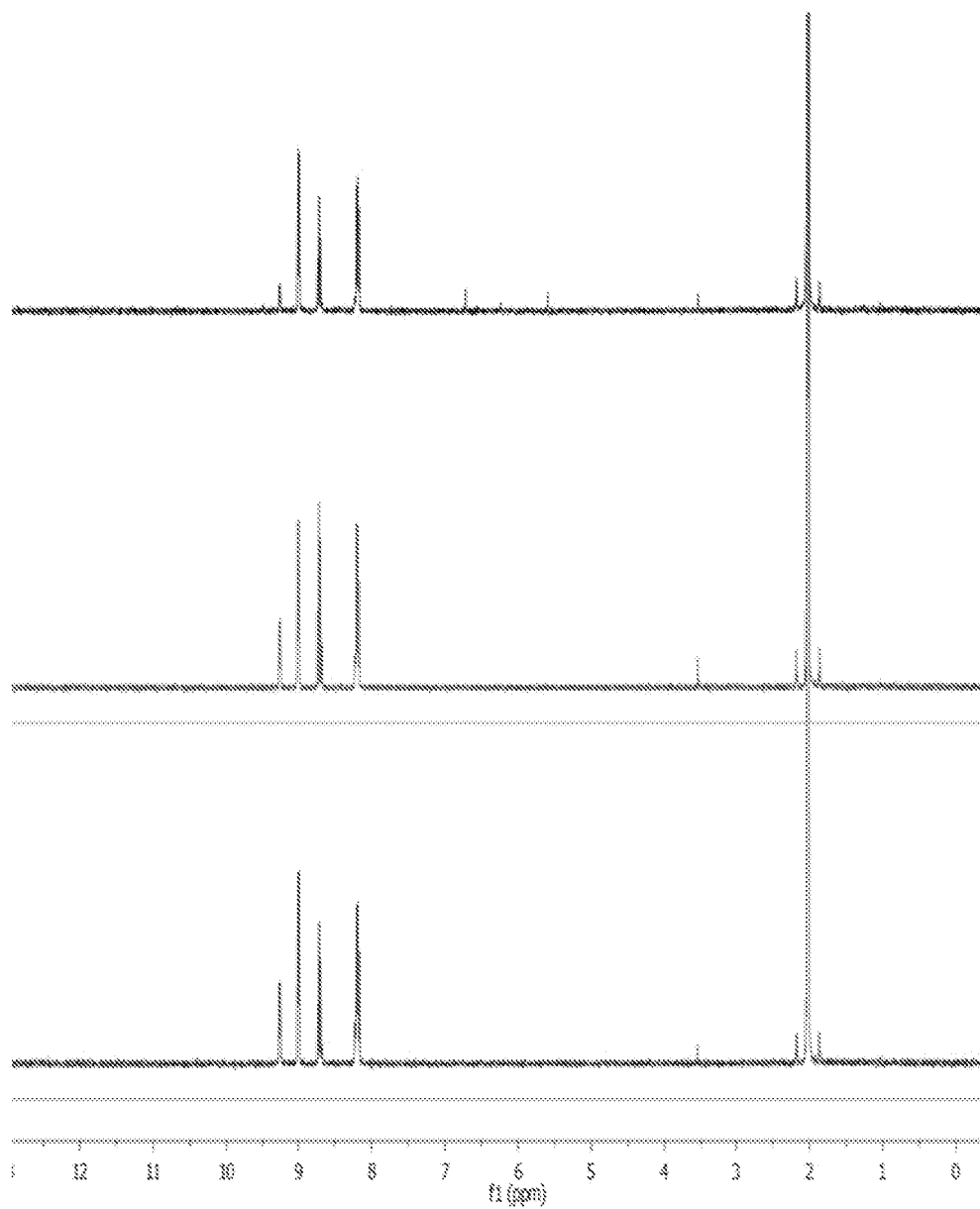


Figure S7

Figure 58A

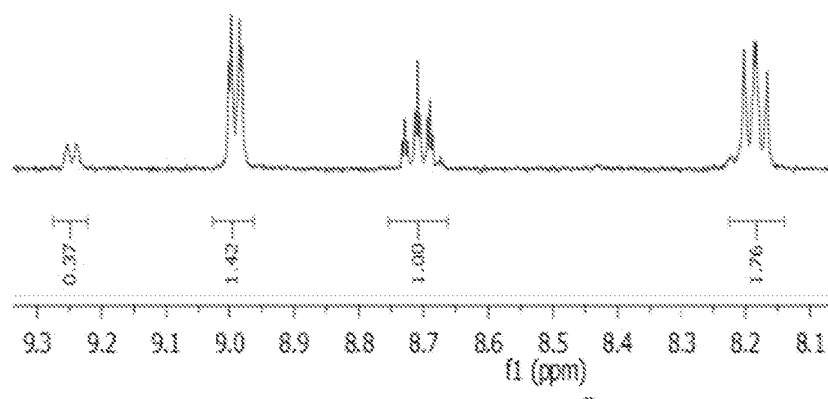


Figure 58B

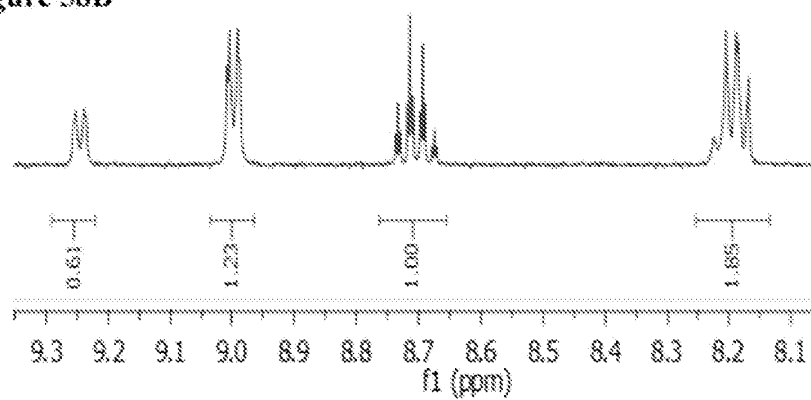


Figure 58C

