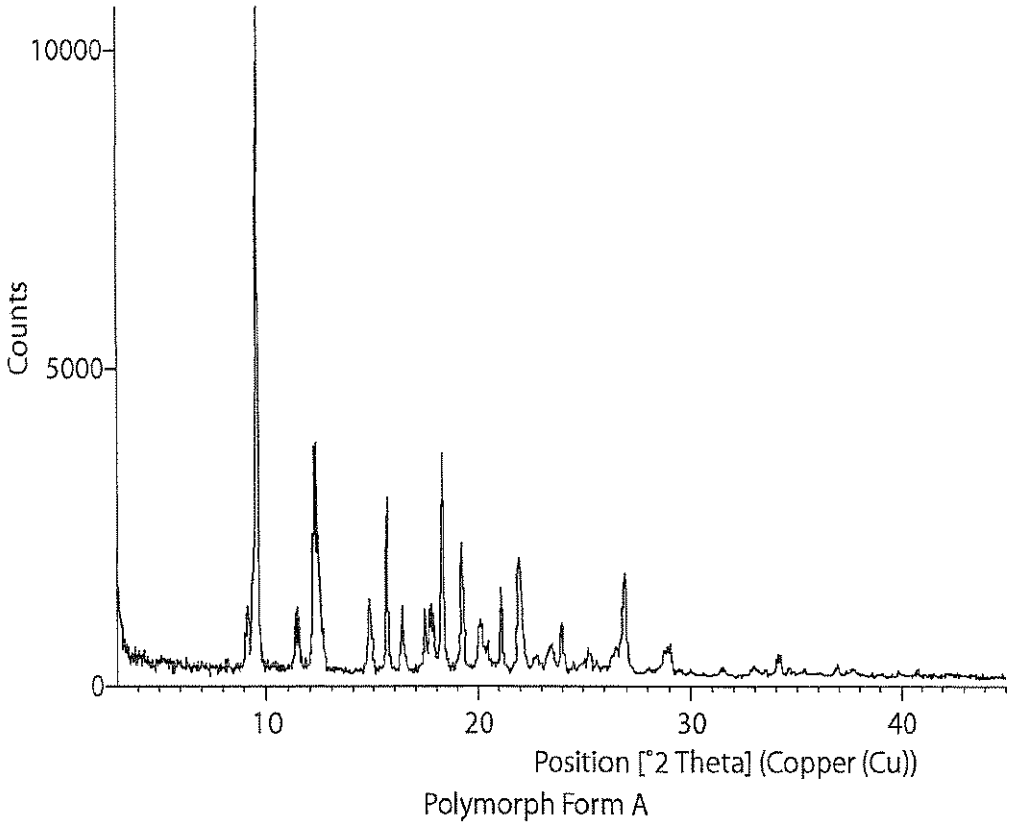

10000
5000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form A

Fig. 1

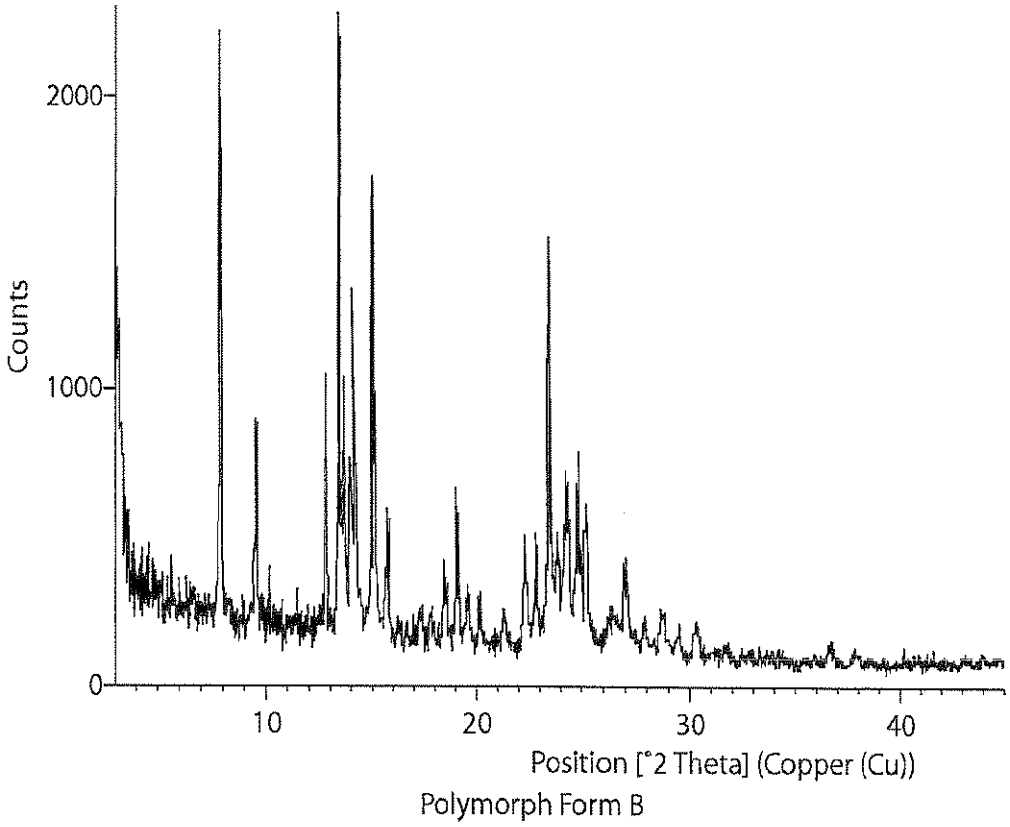

2000
1000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form B

Fig. 2

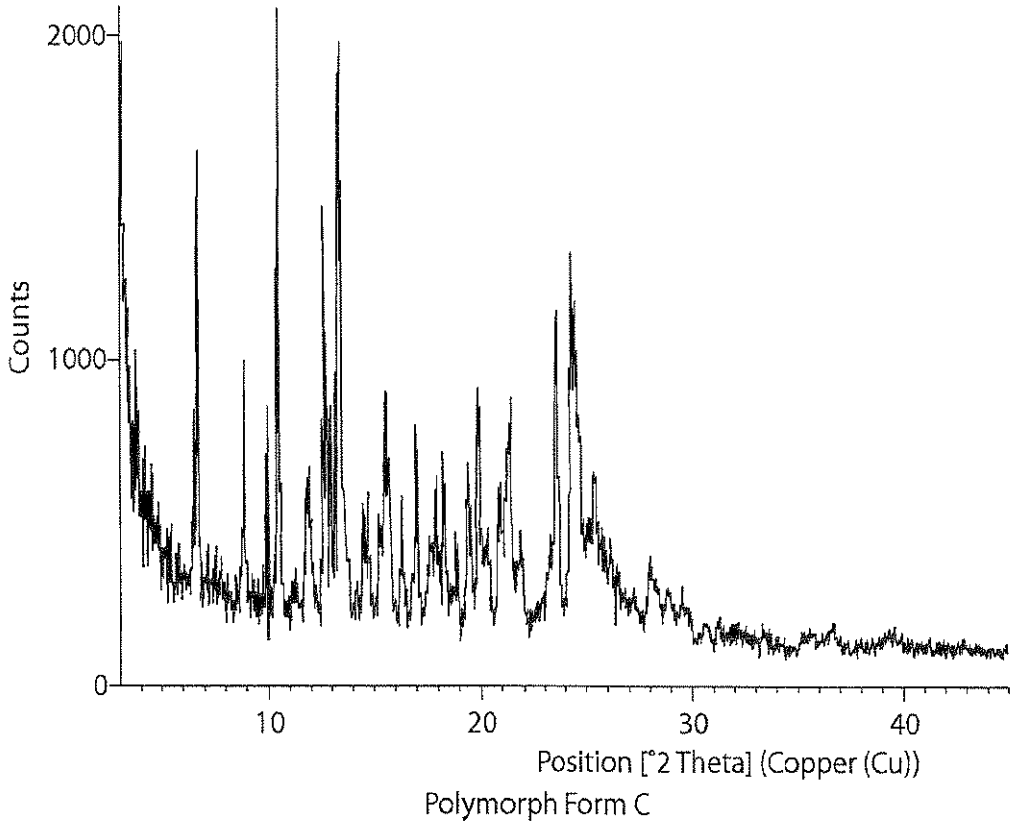

2000
1000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form C

Fig. 3

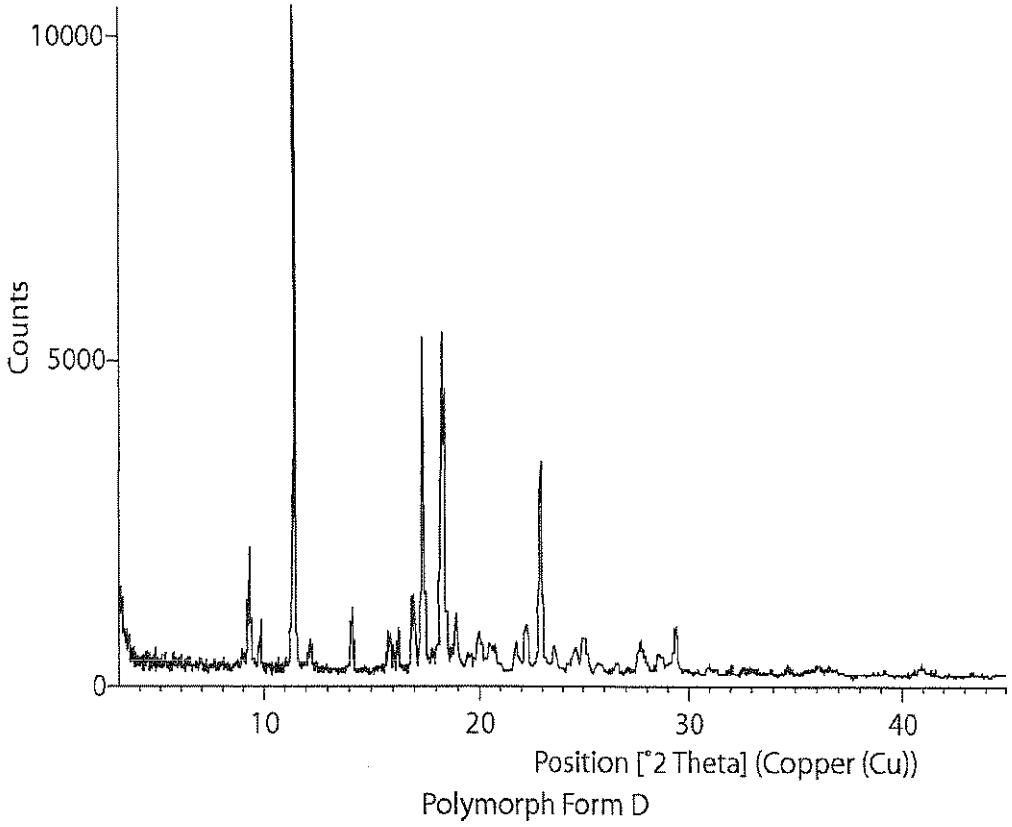

10000
5000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form D

Fig. 4

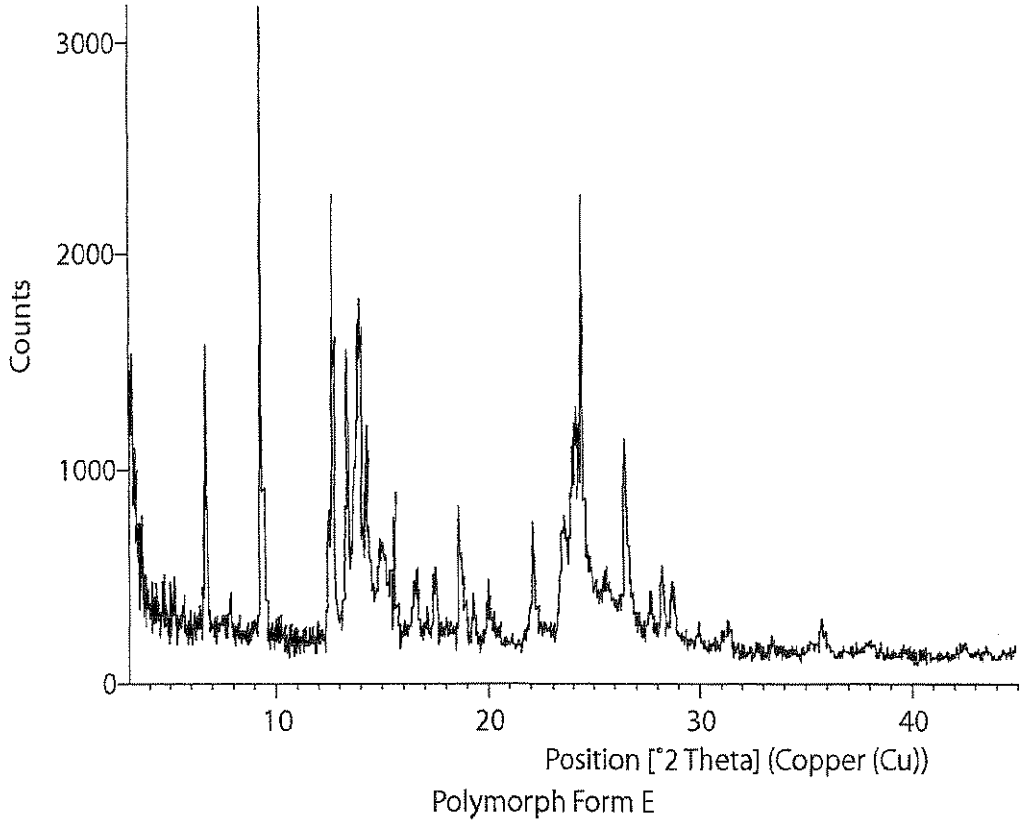

3000
2000
1000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form E

Fig. 5

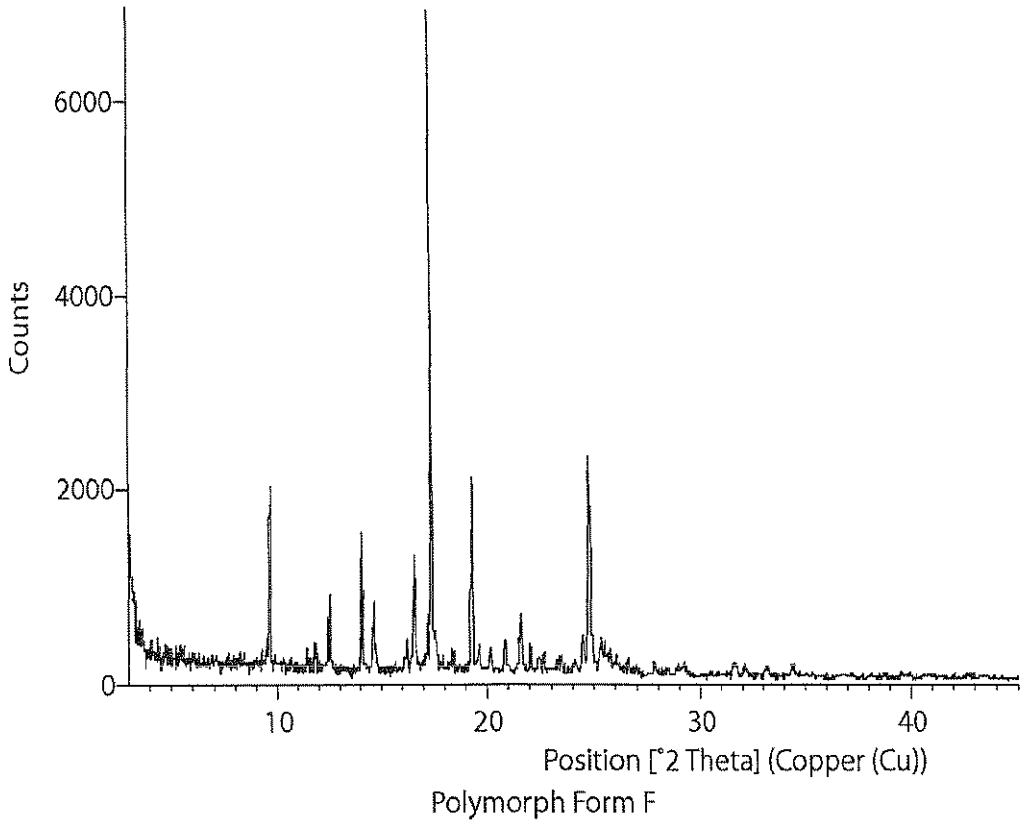

6000
4000
2000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form F

Fig. 6

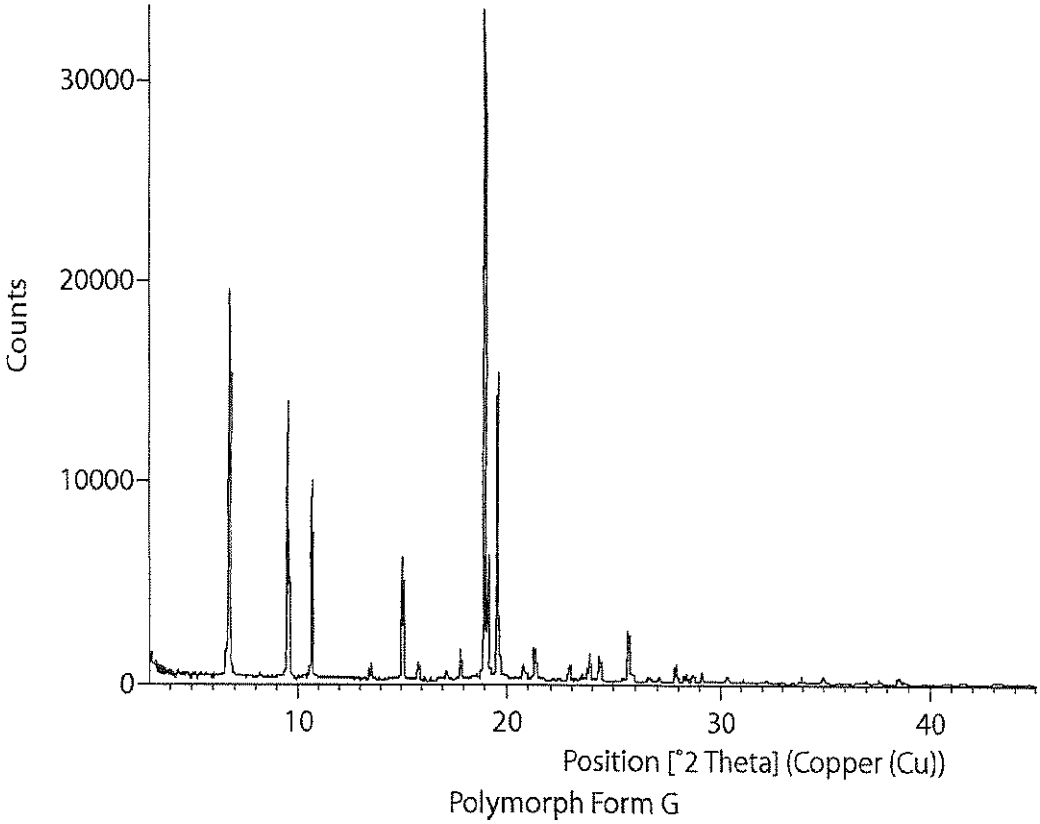

30000
20000
10000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form G

Fig. 7

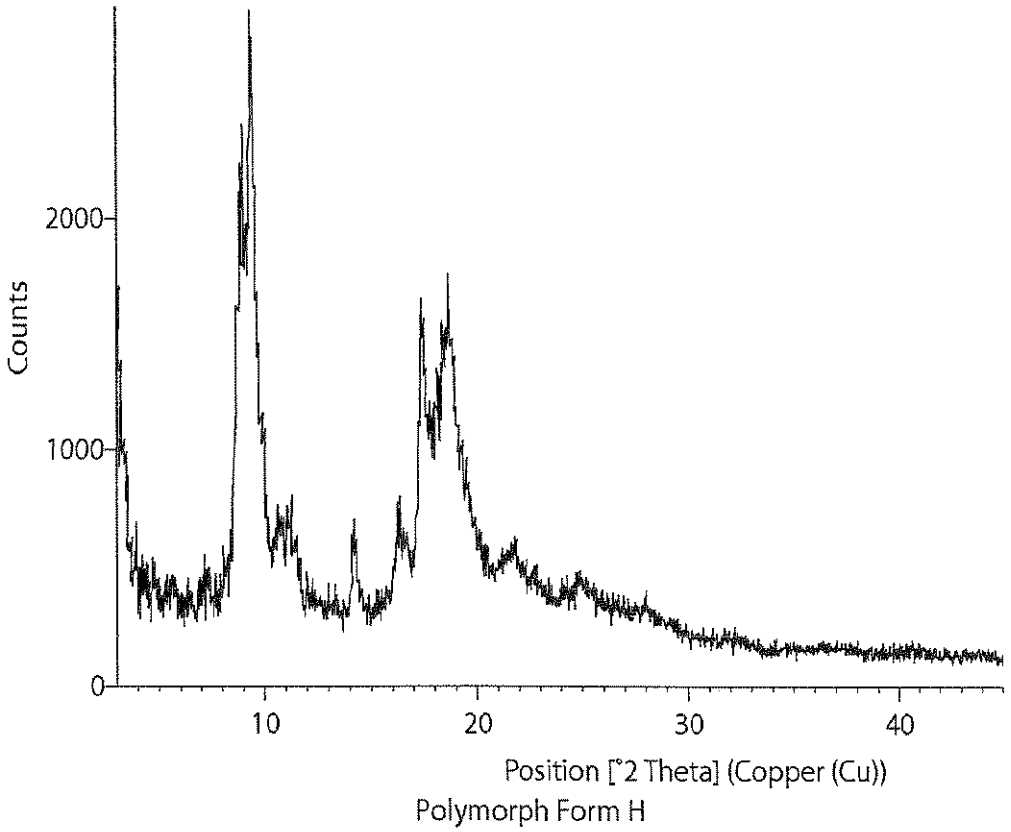

Counts
2000
1000
0
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form H

Fig. 8

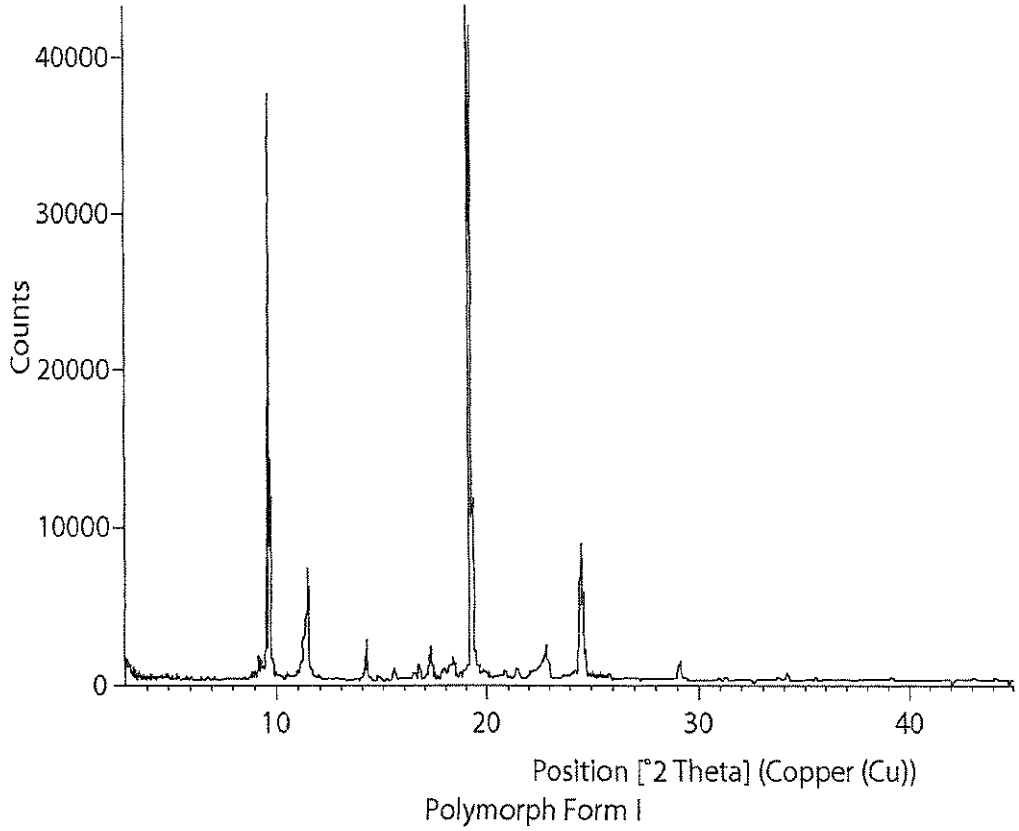

40000
30000
20000
10000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form I

Fig. 9

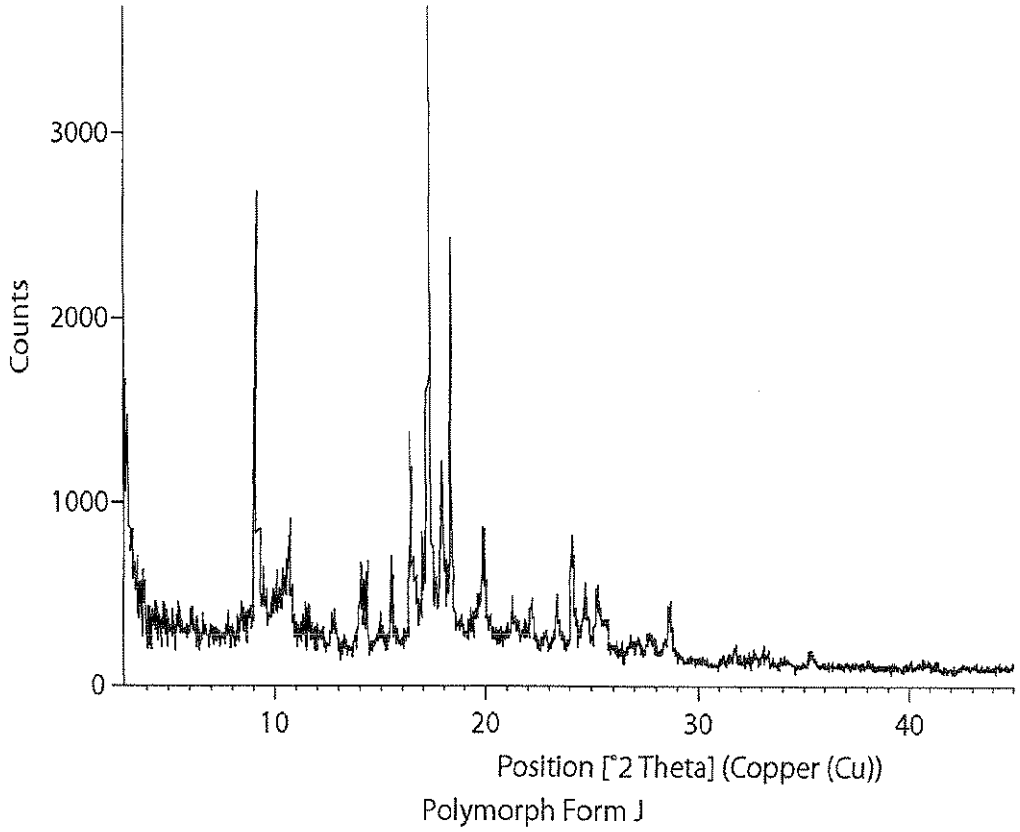

3000
2000
1000
0
Counts
10
20
30
40
Position [°2 Theta] (Copper (Cu))
Polymorph Form J

Fig. 10

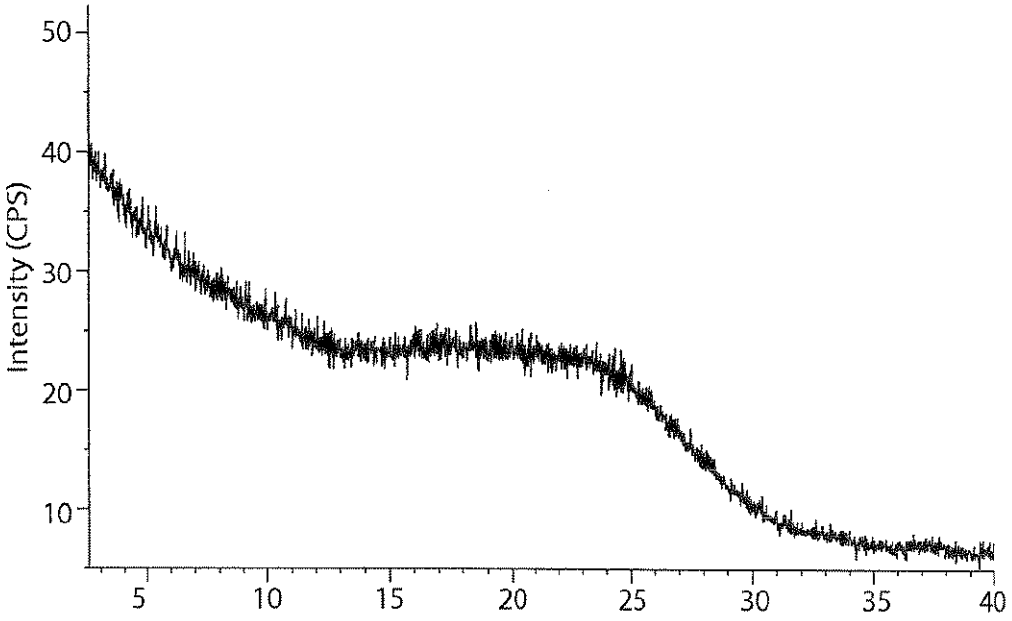

50
40
30
20
10
Intensity (CPS)
5
10
15
20
25
30
35
40
θ-2θ (deg)
Amorphous Compound of Formula (I)

Fig. 11

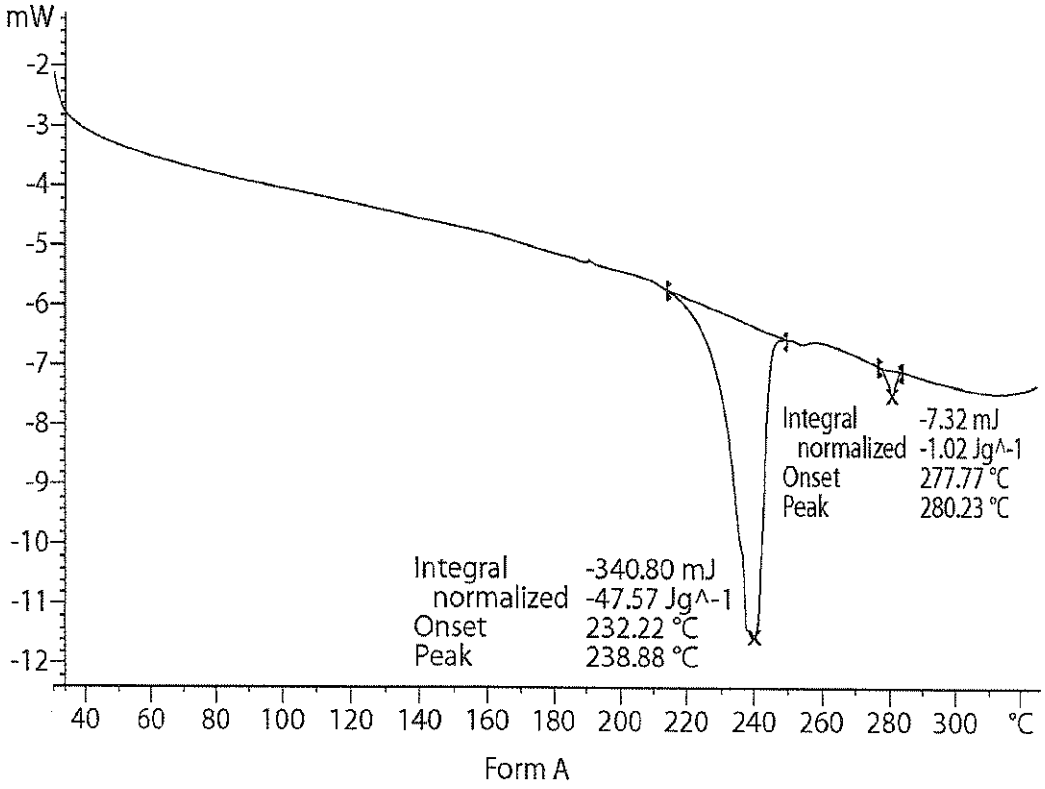

mW
-2
-3
-4
-5
-6
-7
-8
-9
-10
-11
-12
40
60
80
100
120
140
160
180
200
220
240
260
280
300
°C
Integral -340.80 mJ
normalized -47.57 Jg^-1
Onset 232.22 °C
Peak 238.88 °C
Integral -7.32 mJ
normalized -1.02 Jg^-1
Onset 277.77 °C
Peak 280.23 °C
Form A

Fig. 12

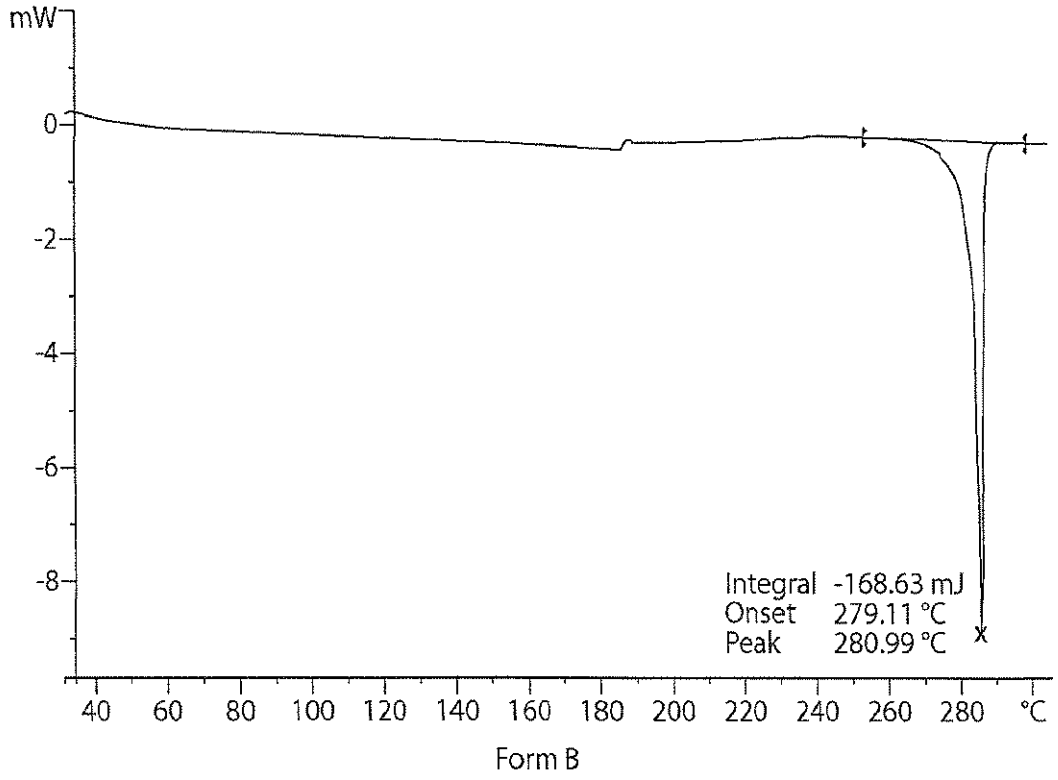

mW
0
-2
-4
-6
-8
Integral -168.63 mJ
Onset 279.11 °C
Peak 280.99 °C
40
60
80
100
120
140
160
180
200
220
240
260
280
°C
Form B

Fig. 13

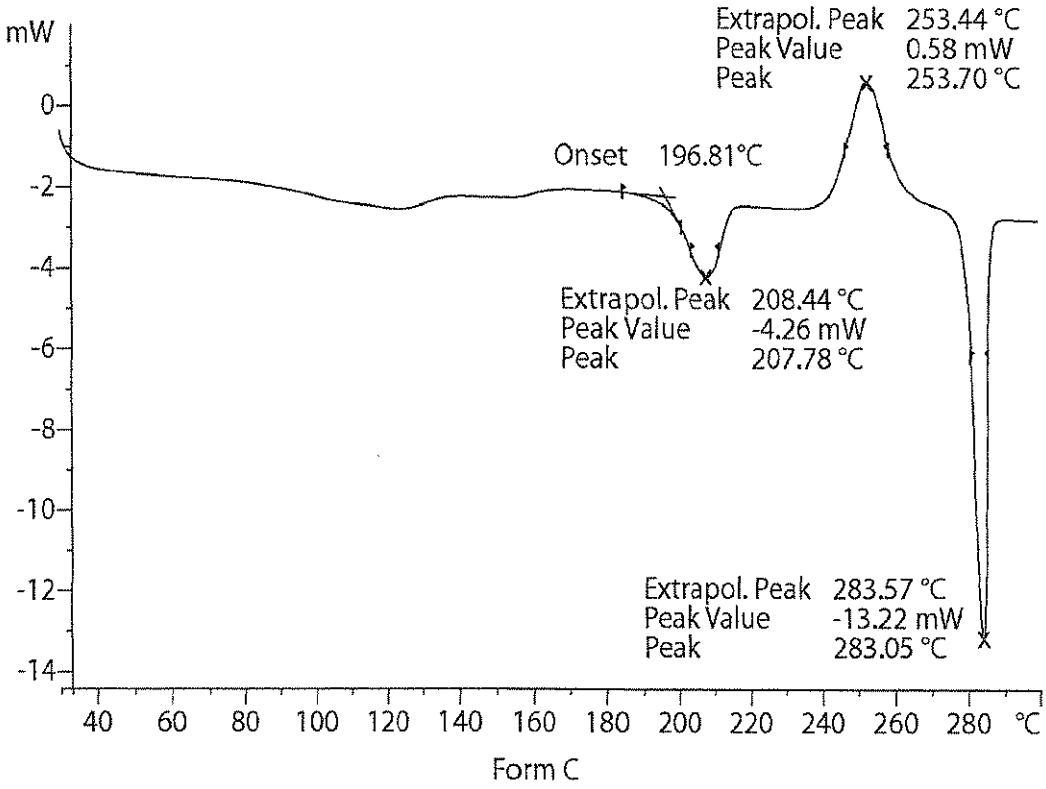

mW
0
-2
-4
-6
-8
-10
-12
-14
Extrapol. Peak 253.44 °C
Peak Value 0.58 mW
Peak 253.70 °C
Onset 196.81°C
Extrapol. Peak 208.44 °C
Peak Value -4.26 mW
Peak 207.78 °C
Extrapol. Peak 283.57 °C
Peak Value -13.22 mW
Peak 283.05 °C
40 60 80 100 120 140 160 180 200 220 240 260 280 °C
Form C

Fig. 14

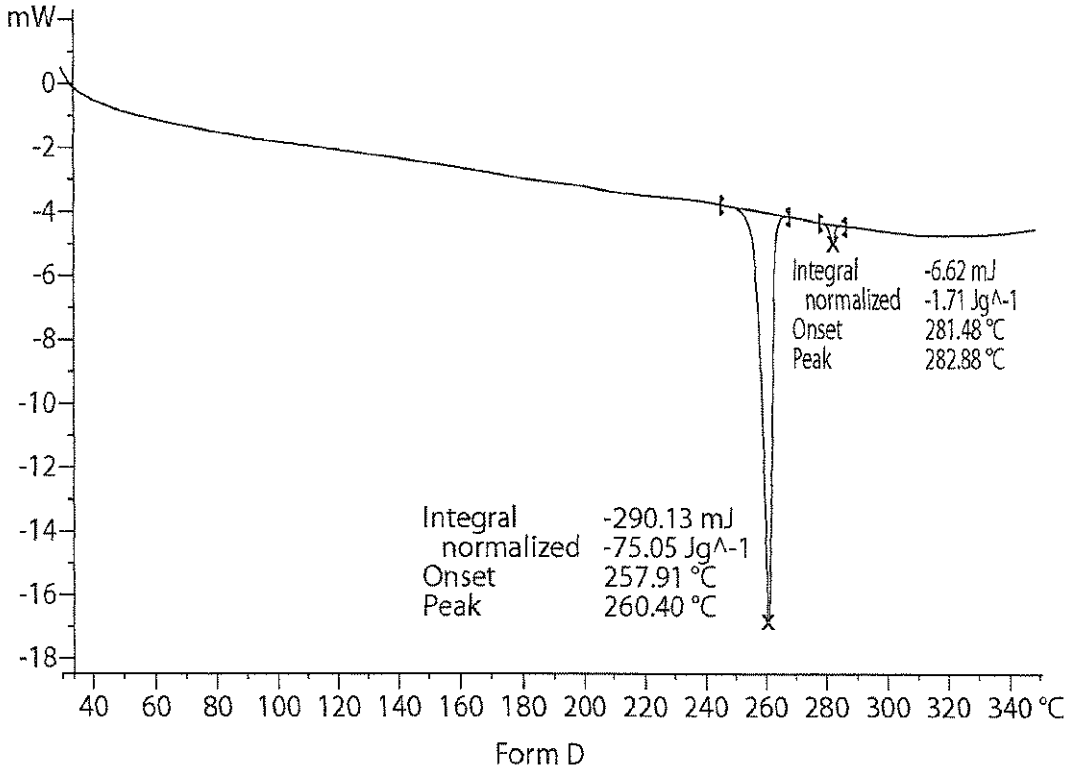

mW
0
-2
-4
-6
-8
-10
-12
-14
-16
-18
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 °C
Integral -6.62 mJ
normalized -1.71 Jg^-1
Onset 281.48 °C
Peak 282.88 °C
Integral -290.13 mJ
normalized -75.05 Jg^-1
Onset 257.91 °C
Peak 260.40 °C
Form D

Fig. 15

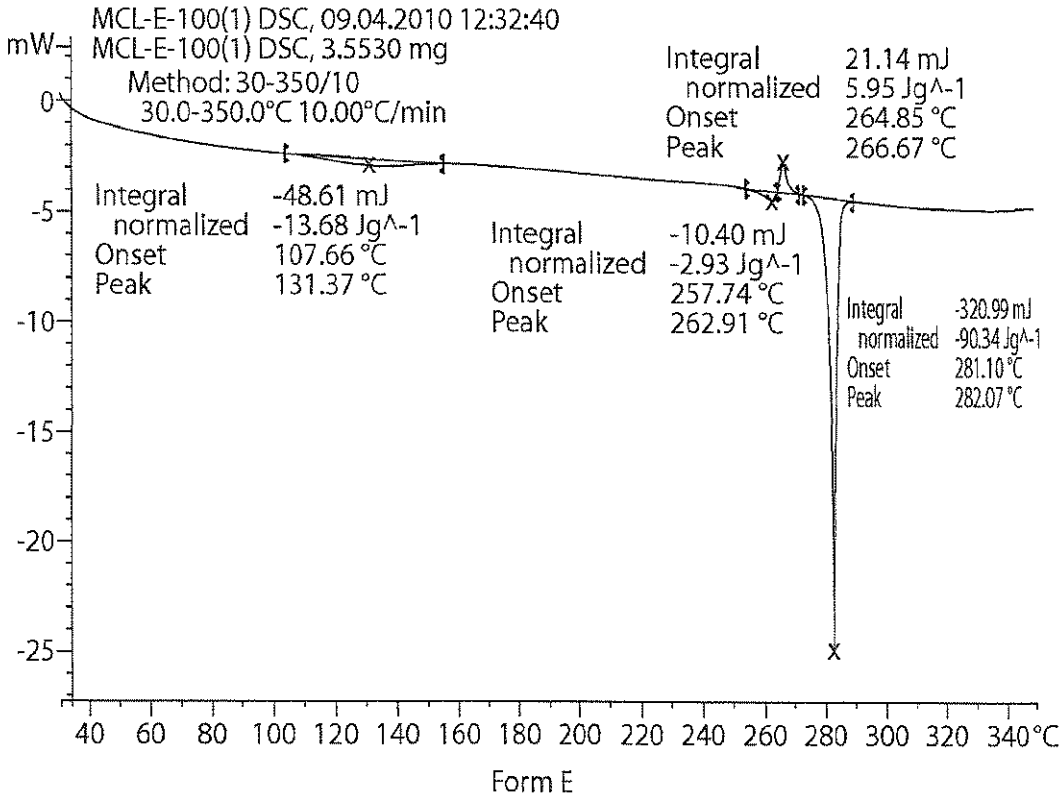

MCL-E-100(1) DSC, 09.04.2010 12:32:40
MCL-E-100(1) DSC, 3.5530 mg
Method: 30-350/10
30.0-350.0°C 10.00°C/min
mW
0
-5
-10
-15
-20
-25
Integral -48.61 mJ
normalized -13.68 Jg^-1
Onset 107.66 °C
Peak 131.37 °C
Integral -10.40 mJ
normalized -2.93 Jg^-1
Onset 257.74 °C
Peak 262.91 °C
Integral 21.14 mJ
normalized 5.95 Jg^-1
Onset 264.85 °C
Peak 266.67 °C
Integral -320.99 mJ
normalized -90.34 Jg^-1
Onset 281.10 °C
Peak 282.07 °C
40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 °C
Form E

Fig. 16

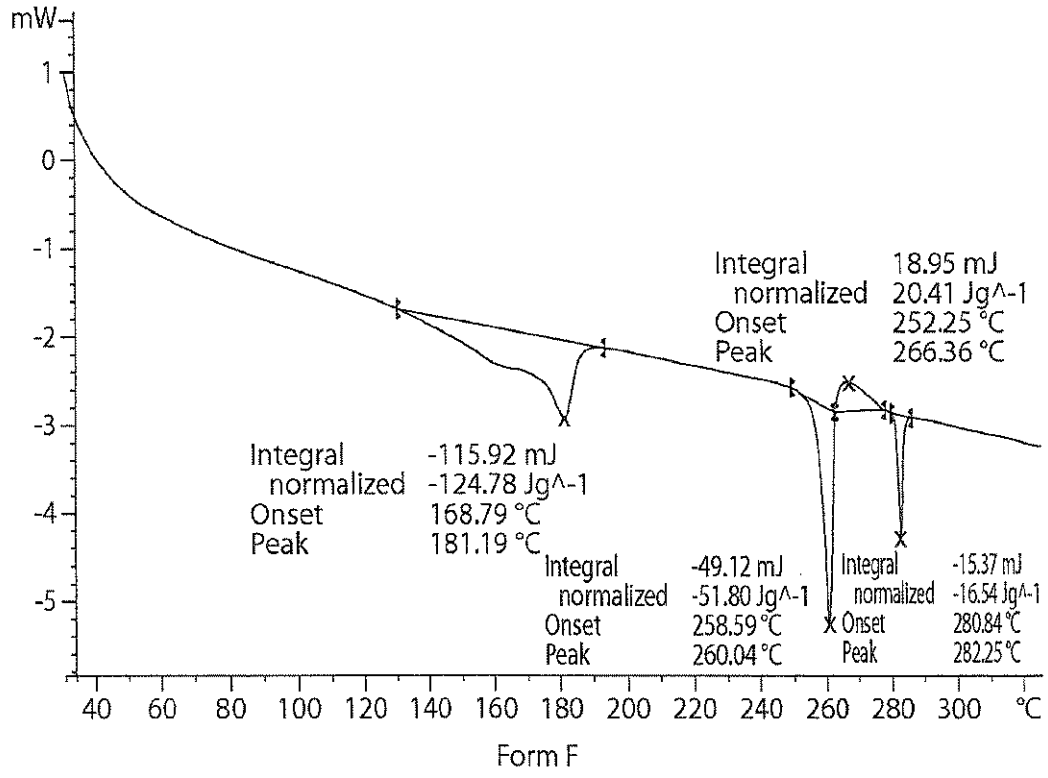

mW
1
0
-1
-2
-3
-4
-5
40
60
80
100
120
140
160
180
200
220
240
260
280
300
°C
Integral 18.95 mJ
normalized 20.41 Jg^-1
Onset 252.25 °C
Peak 266.36 °C
Integral -115.92 mJ
normalized -124.78 Jg^-1
Onset 168.79 °C
Peak 181.19 °C
Integral -49.12 mJ
normalized -51.80 Jg^-1
Onset 258.59 °C
Peak 260.04 °C
Integral -15.37 mJ
normalized -16.54 Jg^-1
Onset 280.84 °C
Peak 282.25 °C
Form F

Fig. 17

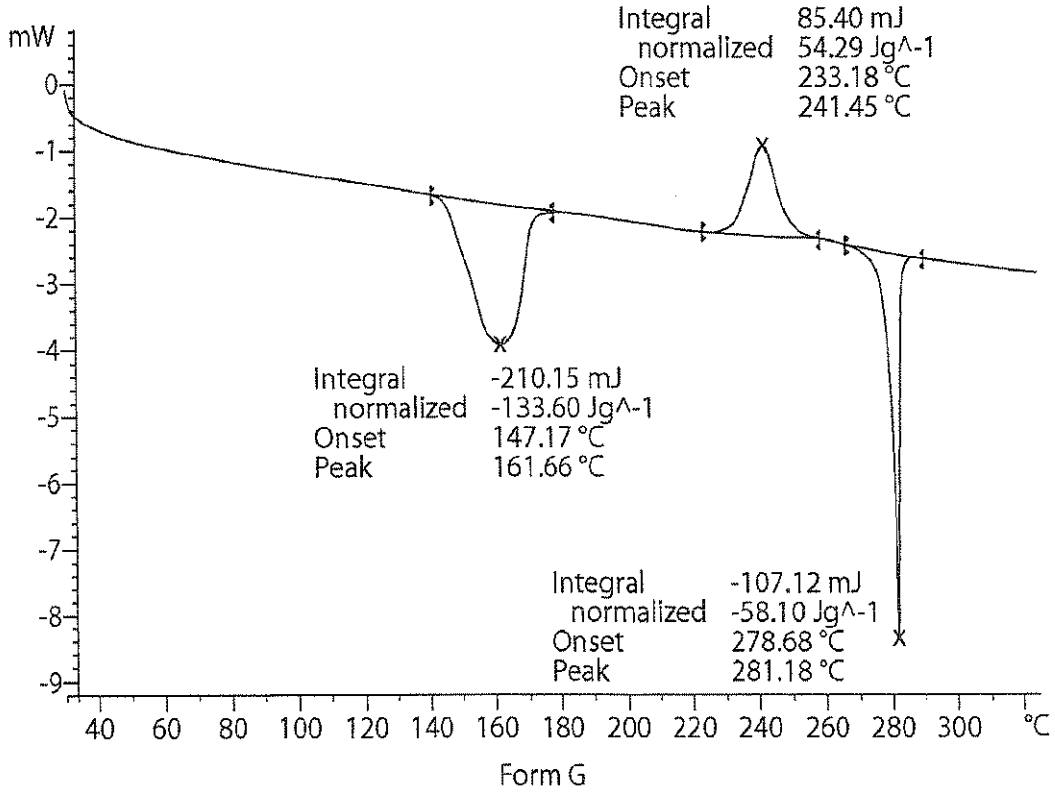

Integral 85.40 mJ
normalized 54.29 Jg^-1
Onset 233.18 °C
Peak 241.45 °C
Integral -210.15 mJ
normalized -133.60 Jg^-1
Onset 147.17 °C
Peak 161.66 °C
Integral -107.12 mJ
normalized -58.10 Jg^-1
Onset 278.68 °C
Peak 281.18 °C
mW
0
-1
-2
-3
-4
-5
-6
-7
-8
-9
40 60 80 100 120 140 160 180 200 220 240 260 280 300 °C
Form G

Fig. 18

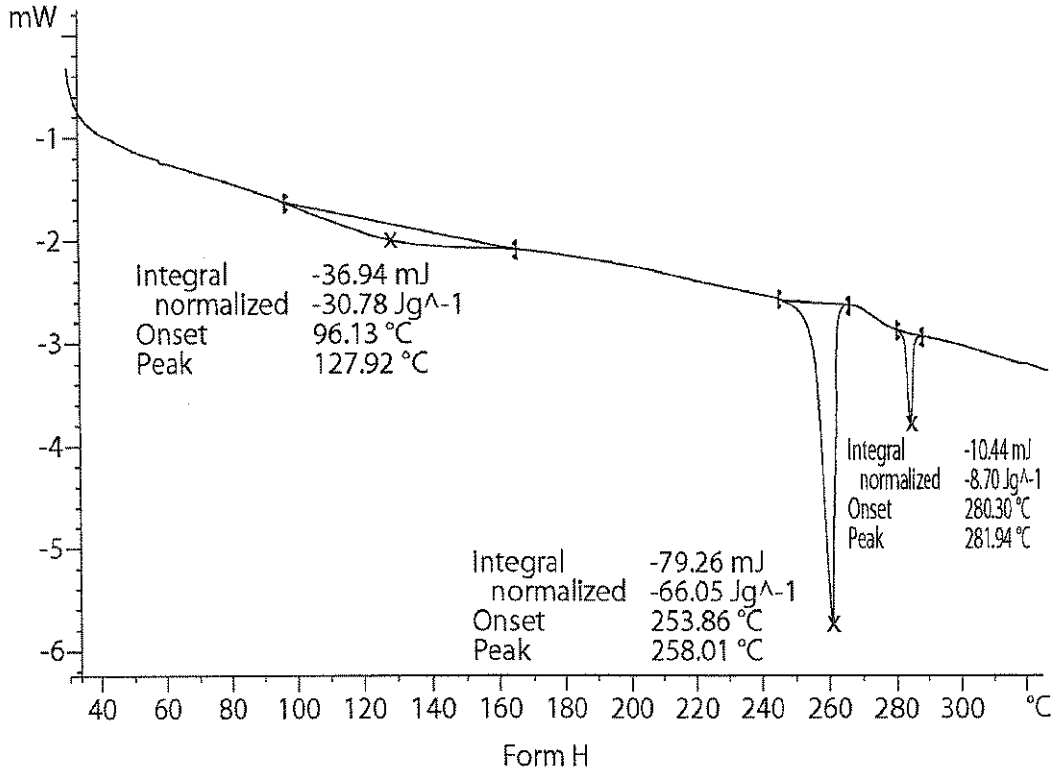

mW
-1
-2
-3
-4
-5
-6
Integral -36.94 mJ
normalized -30.78 Jg^-1
Onset 96.13 °C
Peak 127.92 °C
Integral -10.44 mJ
normalized -8.70 Jg^-1
Onset 280.30 °C
Peak 281.94 °C
Integral -79.26 mJ
normalized -66.05 Jg^-1
Onset 253.86 °C
Peak 258.01 °C
40 60 80 100 120 140 160 180 200 220 240 260 280 300 °C
Form H

Fig. 19

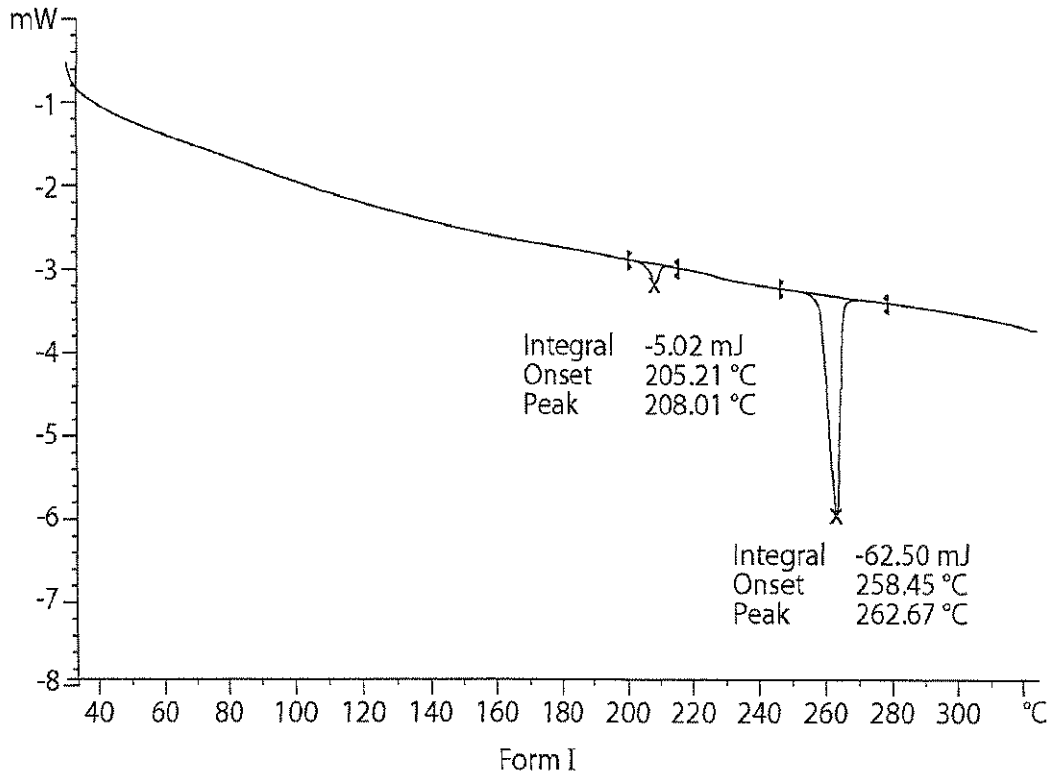

mW
-1
-2
-3
-4
-5
-6
-7
-8
Integral -5.02 mJ
Onset 205.21 °C
Peak 208.01 °C
Integral -62.50 mJ
Onset 258.45 °C
Peak 262.67 °C
40 60 80 100 120 140 160 180 200 220 240 260 280 300 °C
Form I

Fig. 20

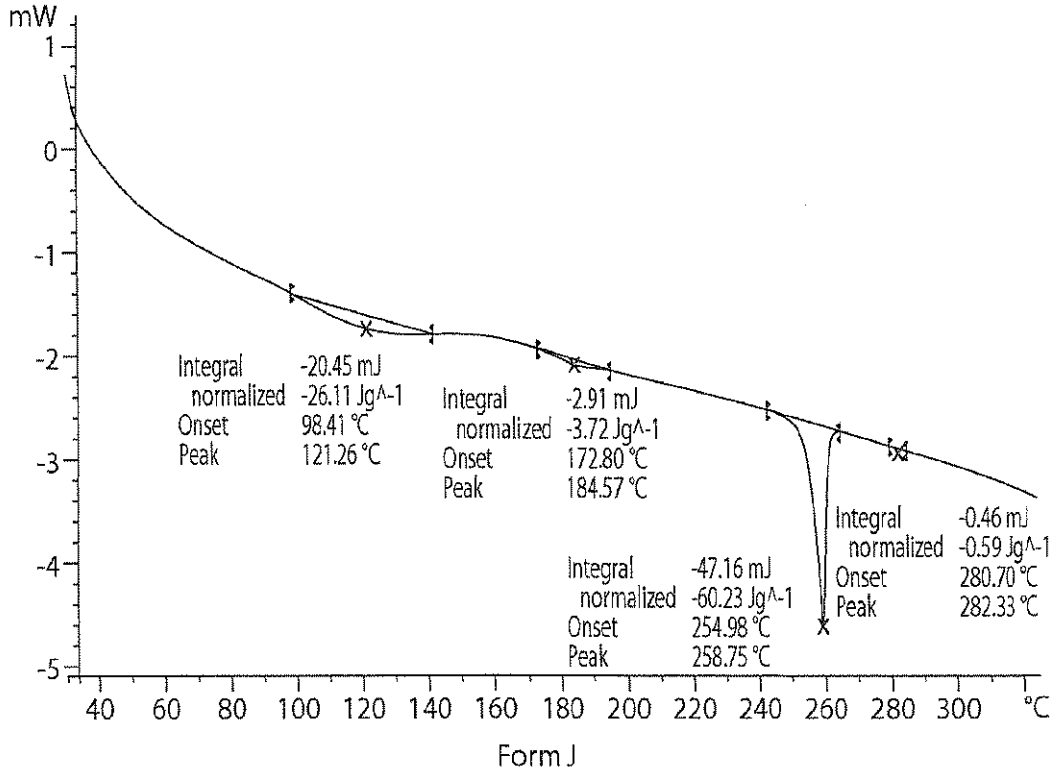
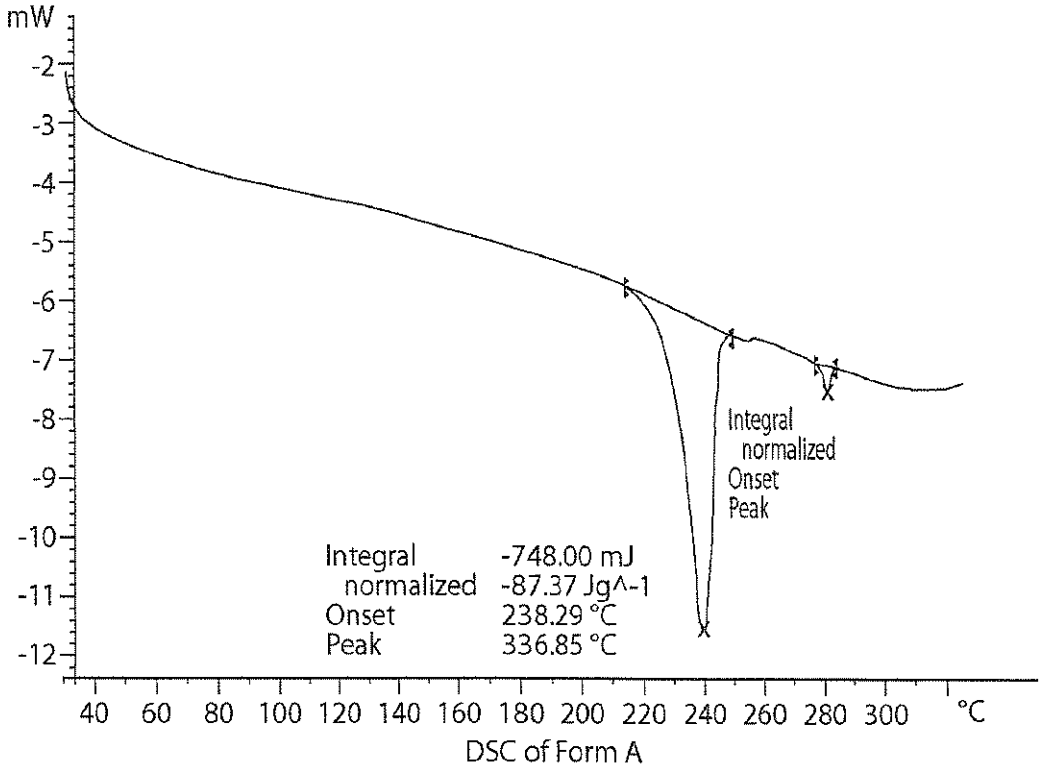
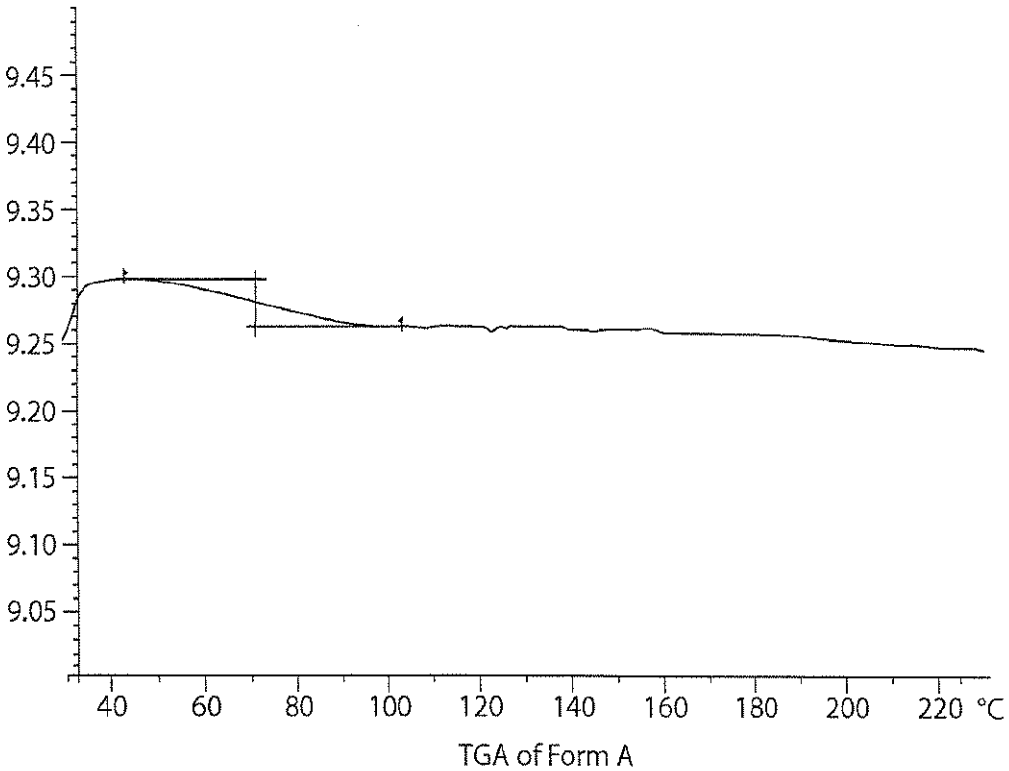

mW
1
0
-1
-2
-3
-4
-5
40
60
80
100
120
140
160
180
200
220
240
260
280
300
°C
Integral -20.45 mJ
normalized -26.11 Jg^-1
Onset 98.41 °C
Peak 121.26 °C
Integral -2.91 mJ
normalized -3.72 Jg^-1
Onset 172.80 °C
Peak 184.57 °C
Integral -47.16 mJ
normalized -60.23 Jg^-1
Onset 254.98 °C
Peak 258.75 °C
Integral -0.46 mJ
normalized -0.59 Jg^-1
Onset 280.70 °C
Peak 282.33 °C
Form J

Fig. 21

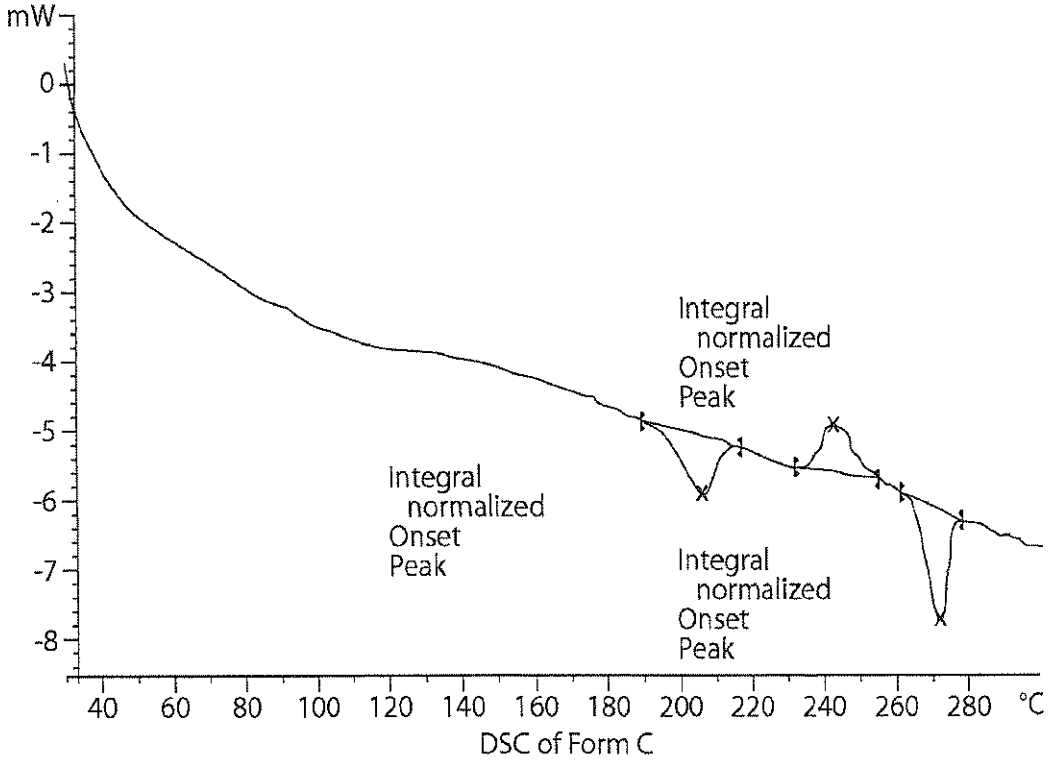


DSC of Form A



TGA of Form A

Fig. 22


mW
0
-1
-2
-3
-4
-5
-6
-7
-8
Integral
normalized
Onset
Peak
Integral
normalized
Onset
Peak
Integral
normalized
Onset
Peak
40 60 80 100 120 140 160 180 200 220 240 260 280 °C
DSC of Form C

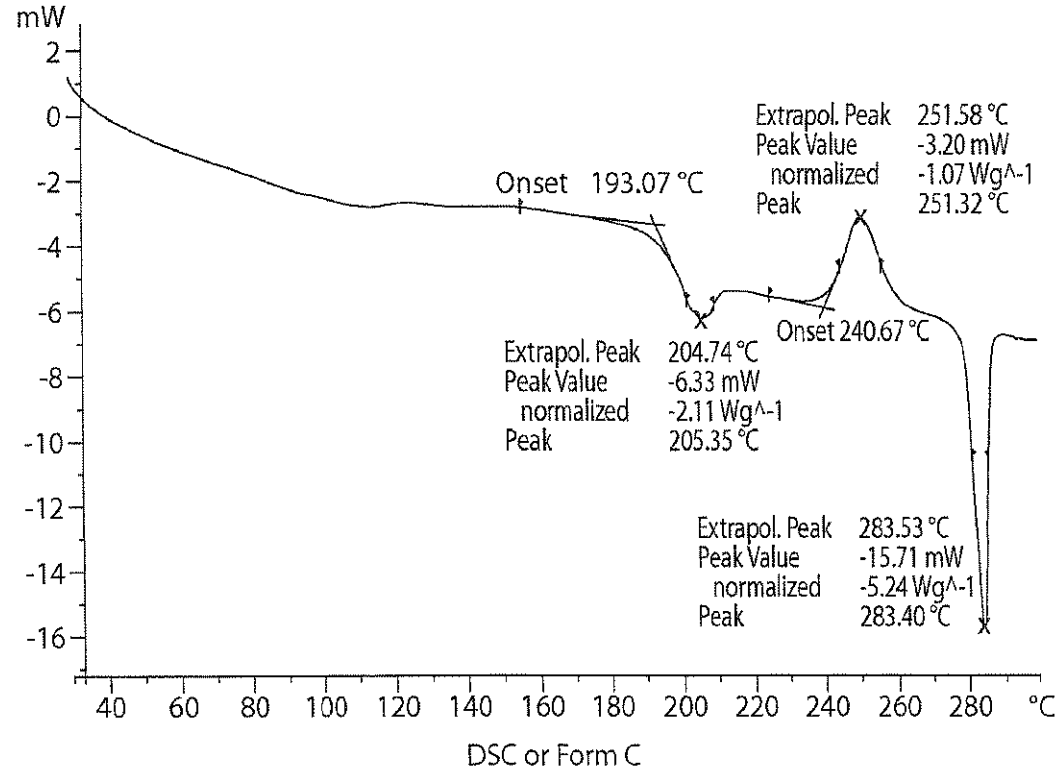
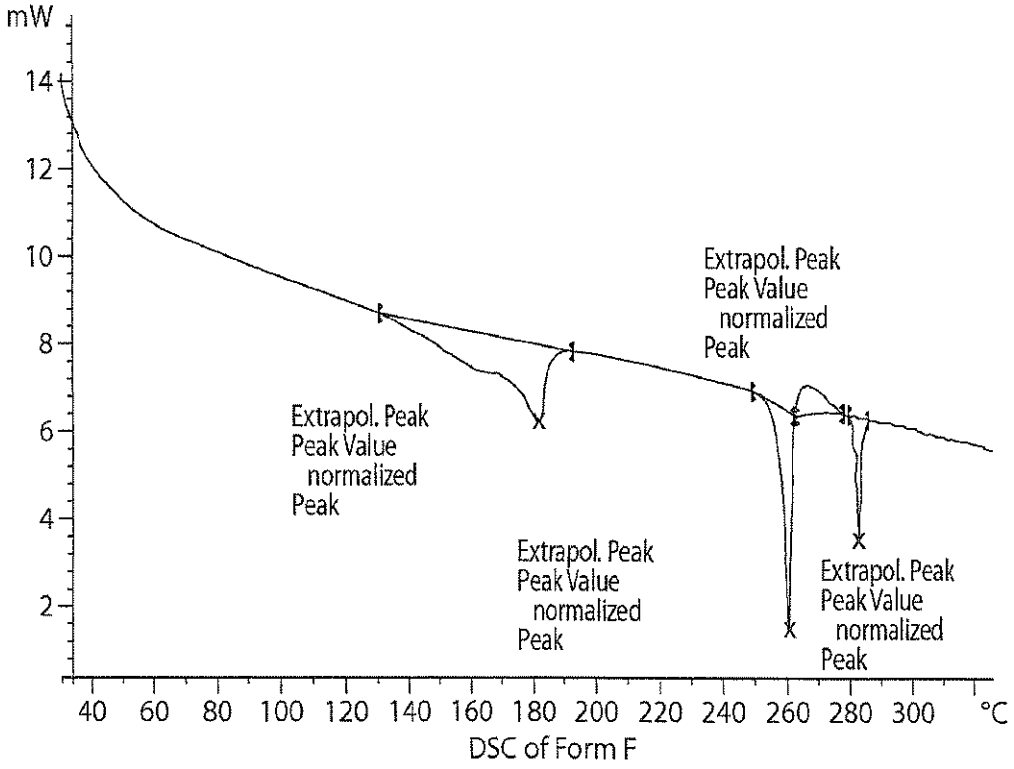

mW
2
0
-2
-4
-6
-8
-10
-12
-14
-16
Onset 193.07 °C
Extrapol. Peak 251.58 °C
Peak Value -3.20 mW
normalized -1.07 Wg^-1
Peak 251.32 °C
Onset 240.67 °C
Extrapol. Peak 204.74 °C
Peak Value -6.33 mW
normalized -2.11 Wg^-1
Peak 205.35 °C
Extrapol. Peak 283.53 °C
Peak Value -15.71 mW
normalized -5.24 Wg^-1
Peak 283.40 °C
40 60 80 100 120 140 160 180 200 220 240 260 280 °C
DSC or Form C

Fig. 23


mW
14
12
10
8
6
4
2
Extrapol. Peak
Peak Value
normalized
Peak
Extrapol. Peak
Peak Value
normalized
Peak
Extrapol. Peak
Peak Value
normalized
Peak
Extrapol. Peak
Peak Value
normalized
Peak
40 60 80 100 120 140 160 180 200 220 240 260 280 300 °C
DSC of Form F

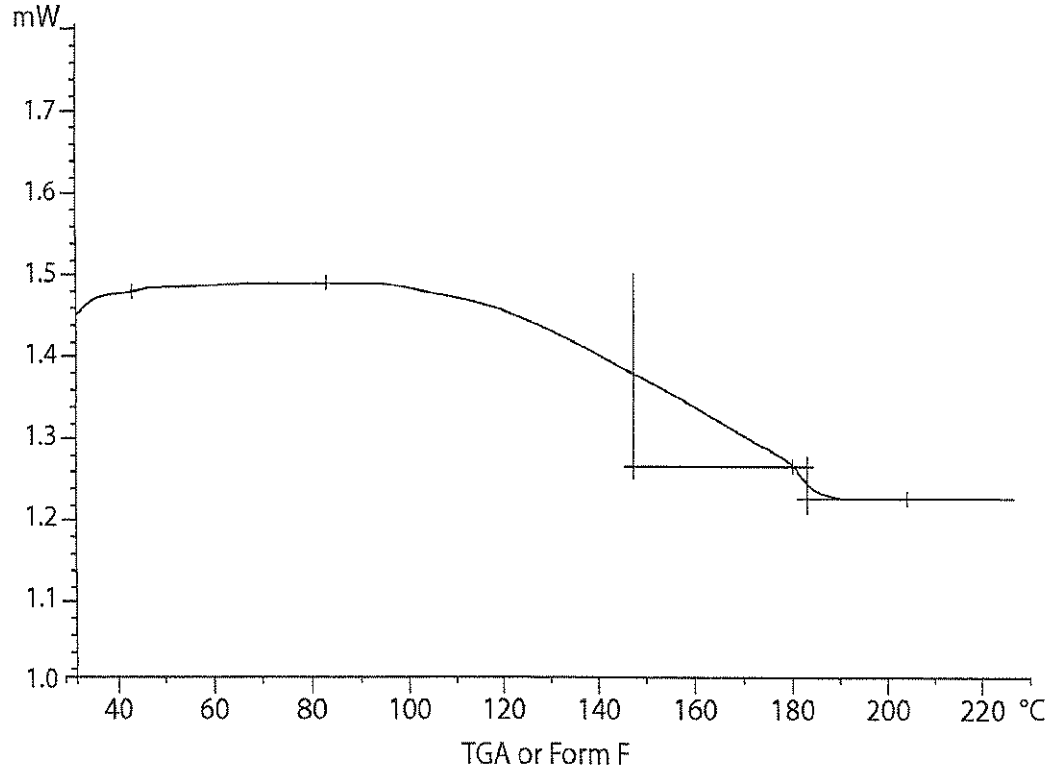

mW
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
40 60 80 100 120 140 160 180 200 220 °C
TGA or Form F

Fig. 24

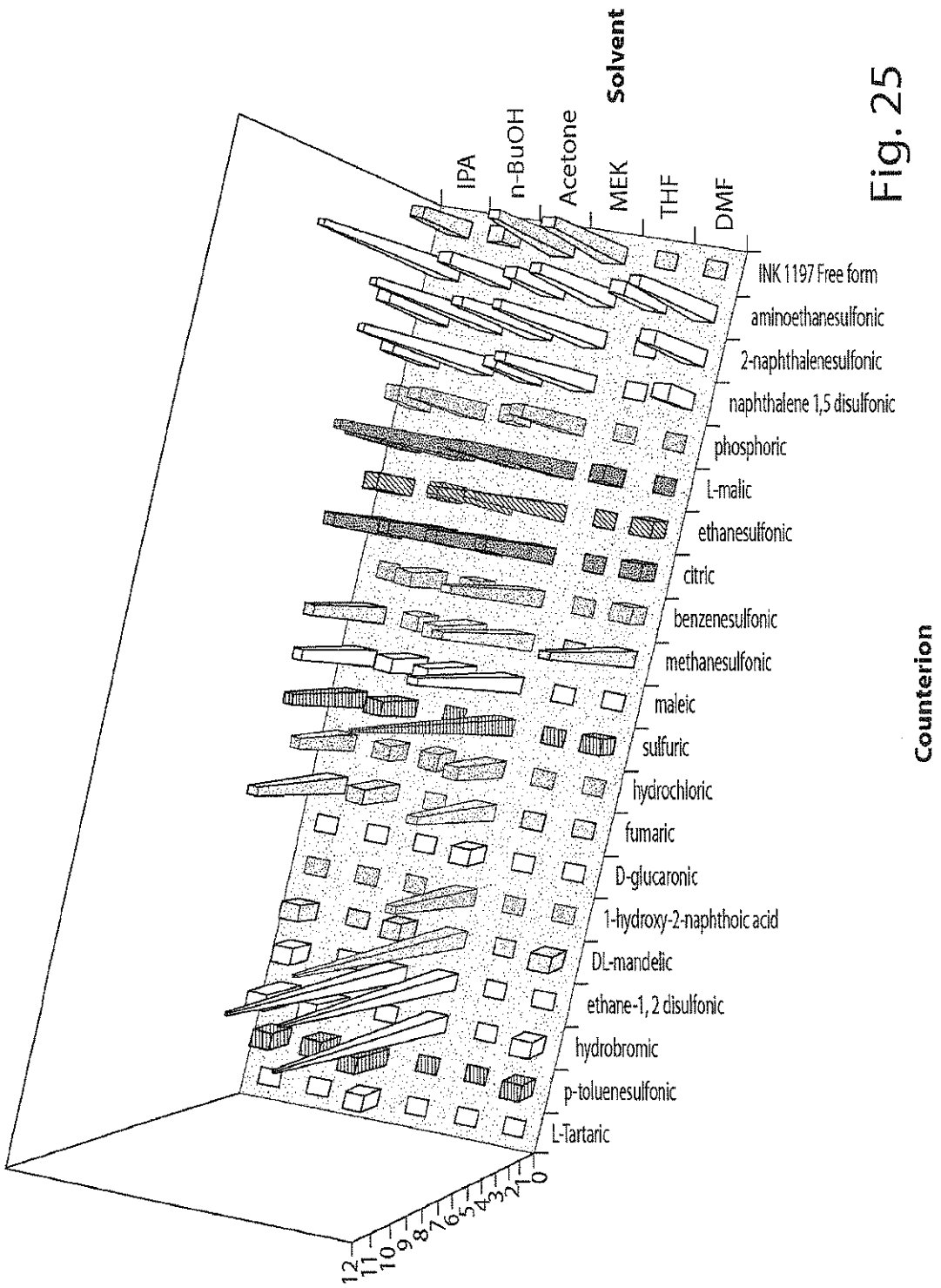

Solvent
IPA
n-BuOH
Acetone
MEK
THF
DMF
Counterion
INK 1197 Free form
aminoethanesulfonic
2-naphthalenesulfonic
naphthalene 1,5 disulfonic
phosphoric
L-malic
ethanesulfonic
citric
benzenesulfonic
methanesulfonic
maleic
sulfuric
hydrochloric
fumaric
D-glucaronic
1-hydroxy-2-naphthoic acid
DL-mandelic
ethane-1, 2 disulfonic
hydrobromic
p-toluenesulfonic
L-Tartaric
0
1
2
3
4
5
6
8
9
10
11
12

Fig. 25

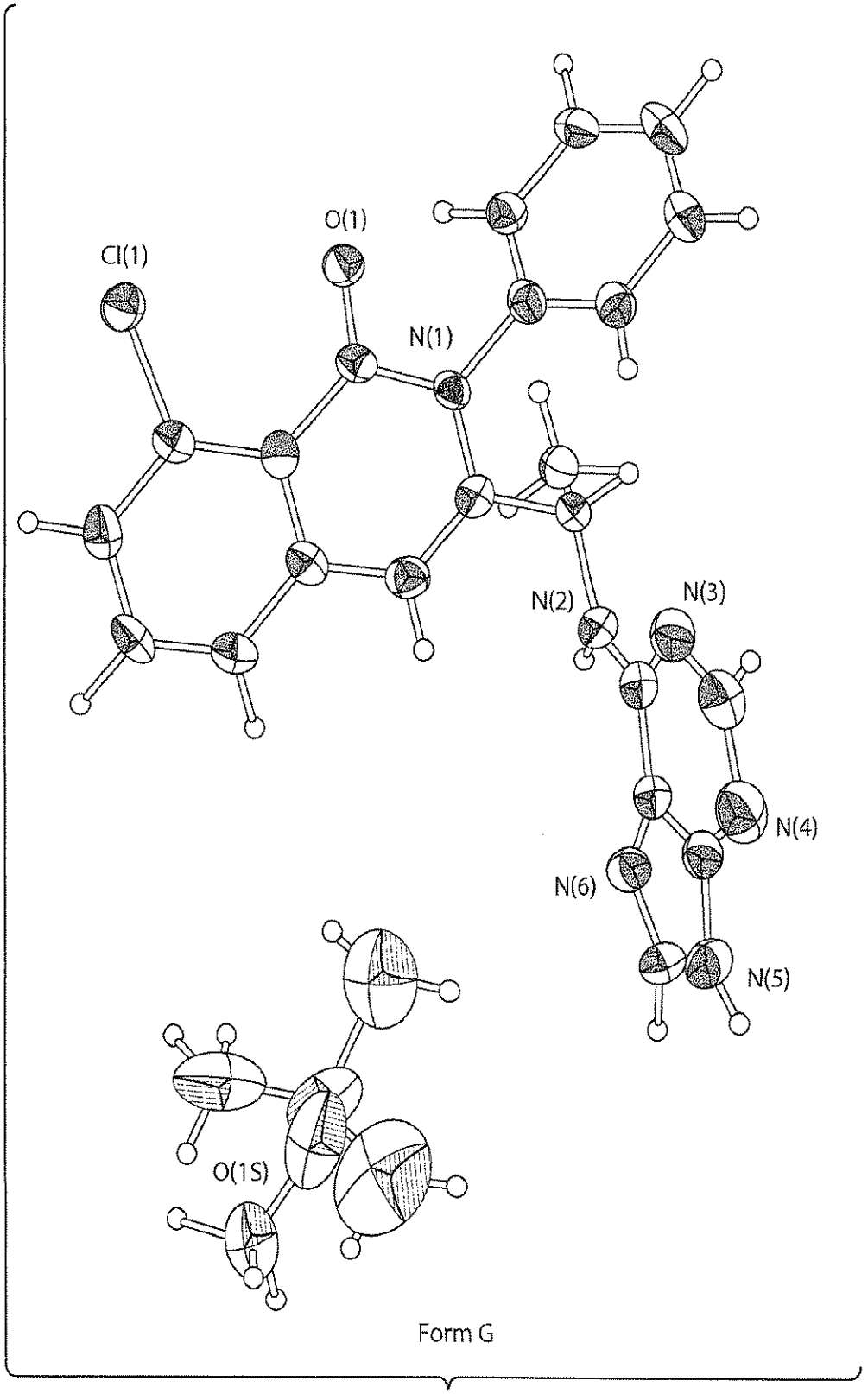


Fig. 26

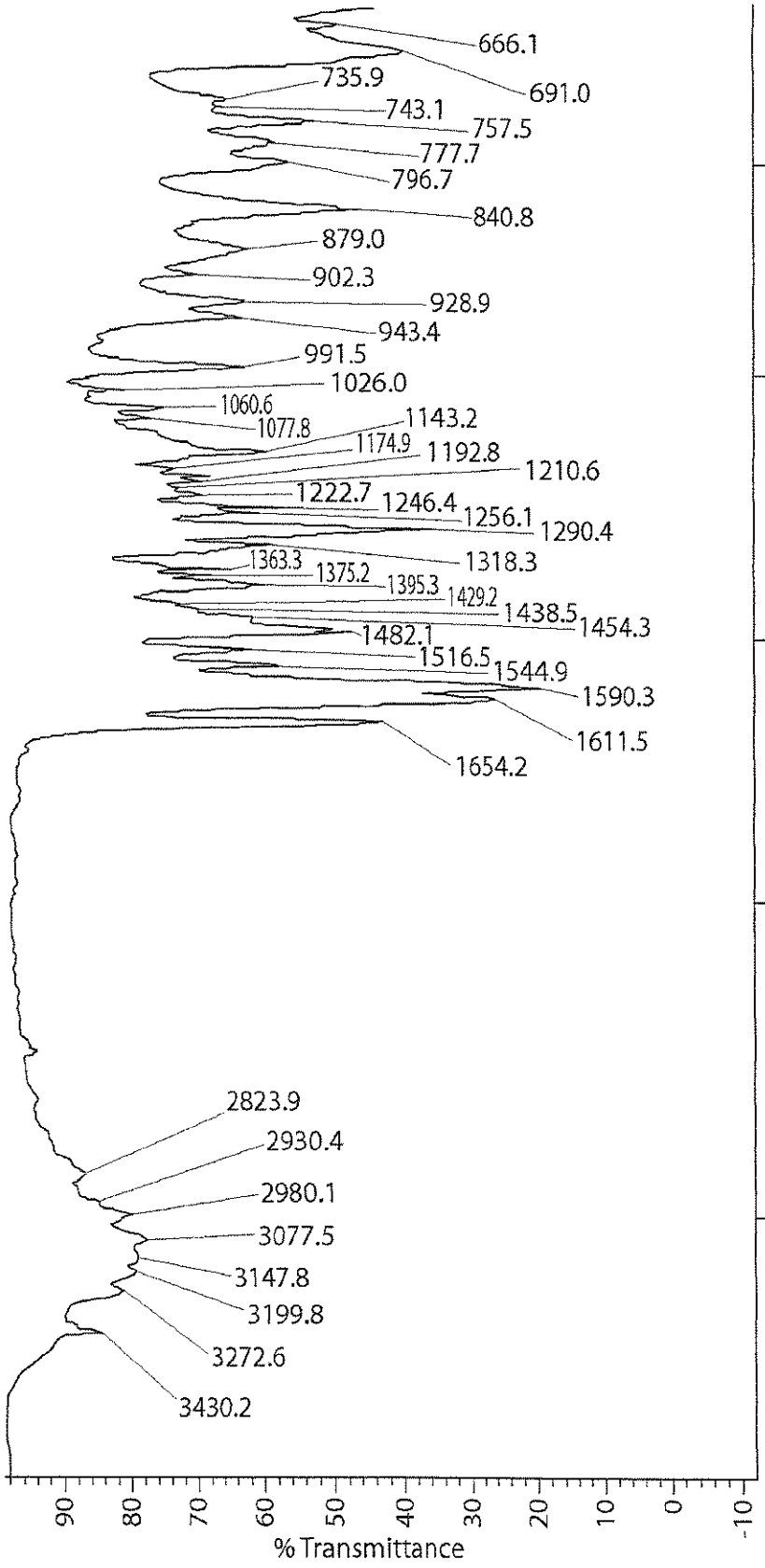

666.1
691.0
735.9
743.1
757.5
777.7
796.7
840.8
879.0
902.3
928.9
943.4
991.5
1026.0
1060.6
1077.8
1143.2
1174.9
1192.8
1210.6
1222.7
1246.4
1256.1
1290.4
1318.3
1363.3
1375.2
1395.3
1429.2
1438.5
1454.3
1482.1
1516.5
1544.9
1590.3
1611.5
1654.2
2823.9
2930.4
2980.1
3077.5
3147.8
3199.8
3272.6
3430.2
800
1000
1500
2000
3000
Wavenumbers (cm-1)
90
80
70
60
50
40
30
20
10
0
-10
% Transmittance

Fig. 27

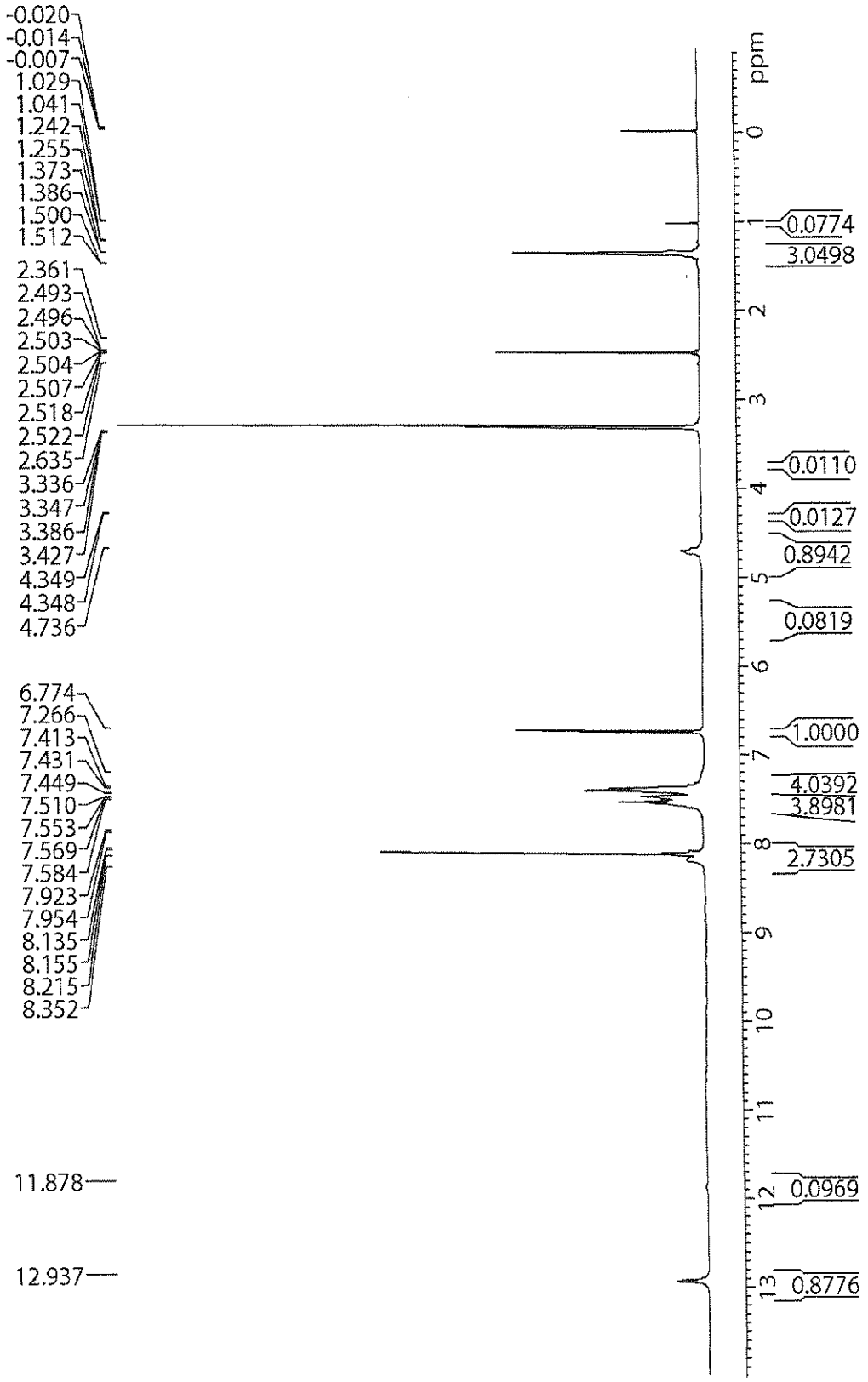

-0.020
-0.014
-0.007
1.029
1.041
1.242
1.255
1.373
1.386
1.500
1.512
2.361
2.493
2.496
2.503
2.504
2.507
2.518
2.522
2.635
3.336
3.347
3.386
3.427
4.349
4.348
4.736
6.774
7.266
7.413
7.431
7.449
7.510
7.553
7.569
7.584
7.923
7.954
8.135
8.155
8.215
8.352
11.878
12.937
ppm
0.0774
3.0498
0.0110
0.0127
0.8942
0.0819
1.0000
4.0392
3.8981
2.7305
0.0969
0.8776

Fig. 28

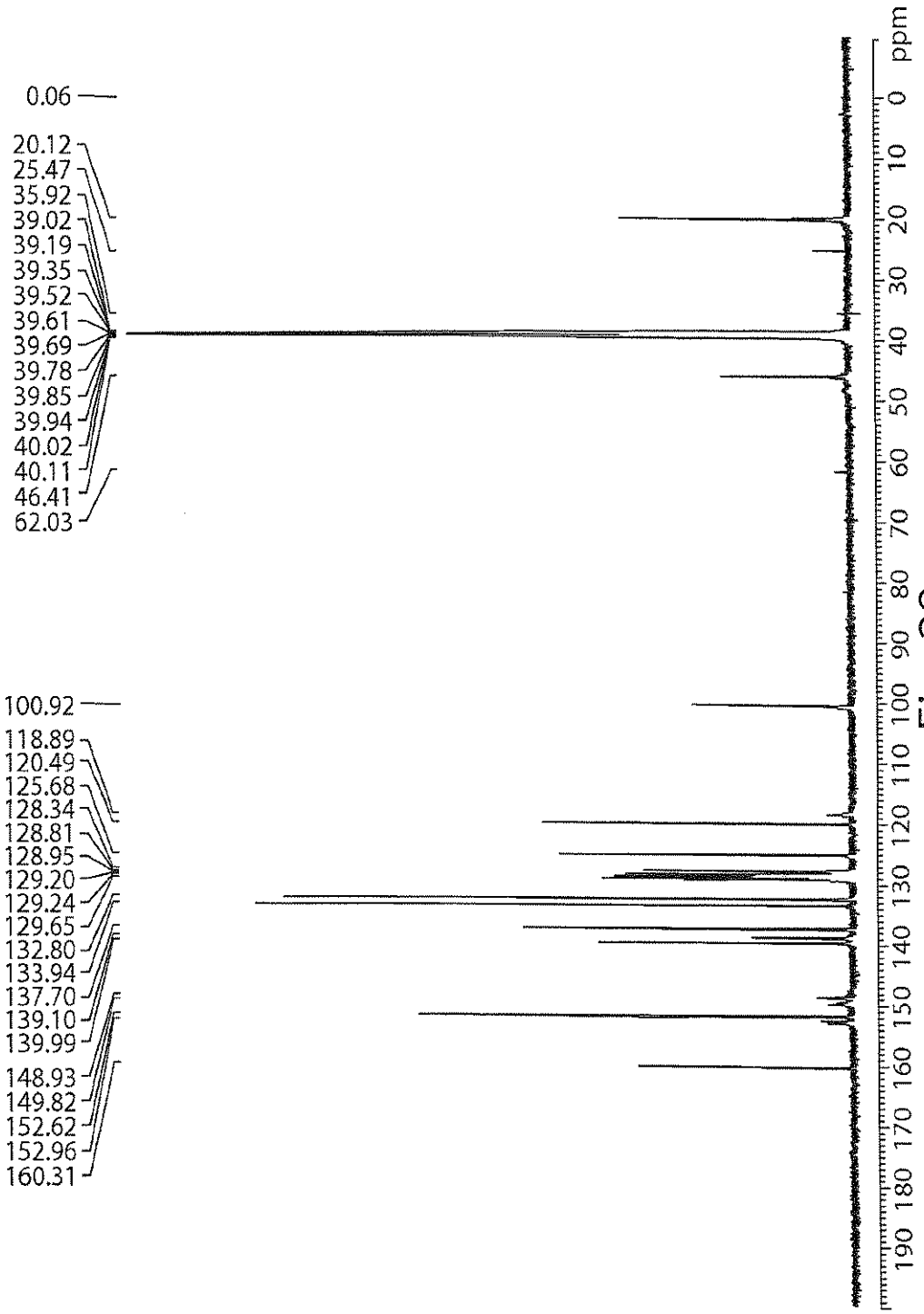

0.06
20.12
25.47
35.92
39.02
39.19
39.35
39.52
39.61
39.69
39.78
39.85
39.94
40.02
40.11
46.41
62.03
100.92
118.89
120.49
125.68
128.34
128.81
128.95
129.20
129.24
129.65
132.80
133.94
137.70
139.10
139.99
148.93
149.82
152.62
152.96
160.31
ppm
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190

Fig. 29

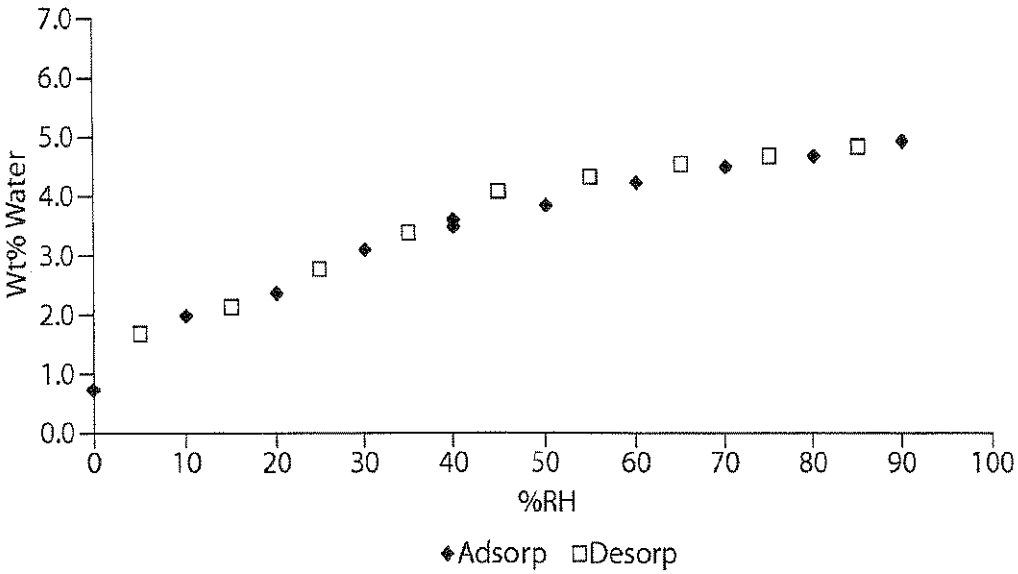

7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
Wt% Water
0
10
20
30
40
50
60
70
80
90
100
%RH
♦Adsorp □Desorp

Fig. 30

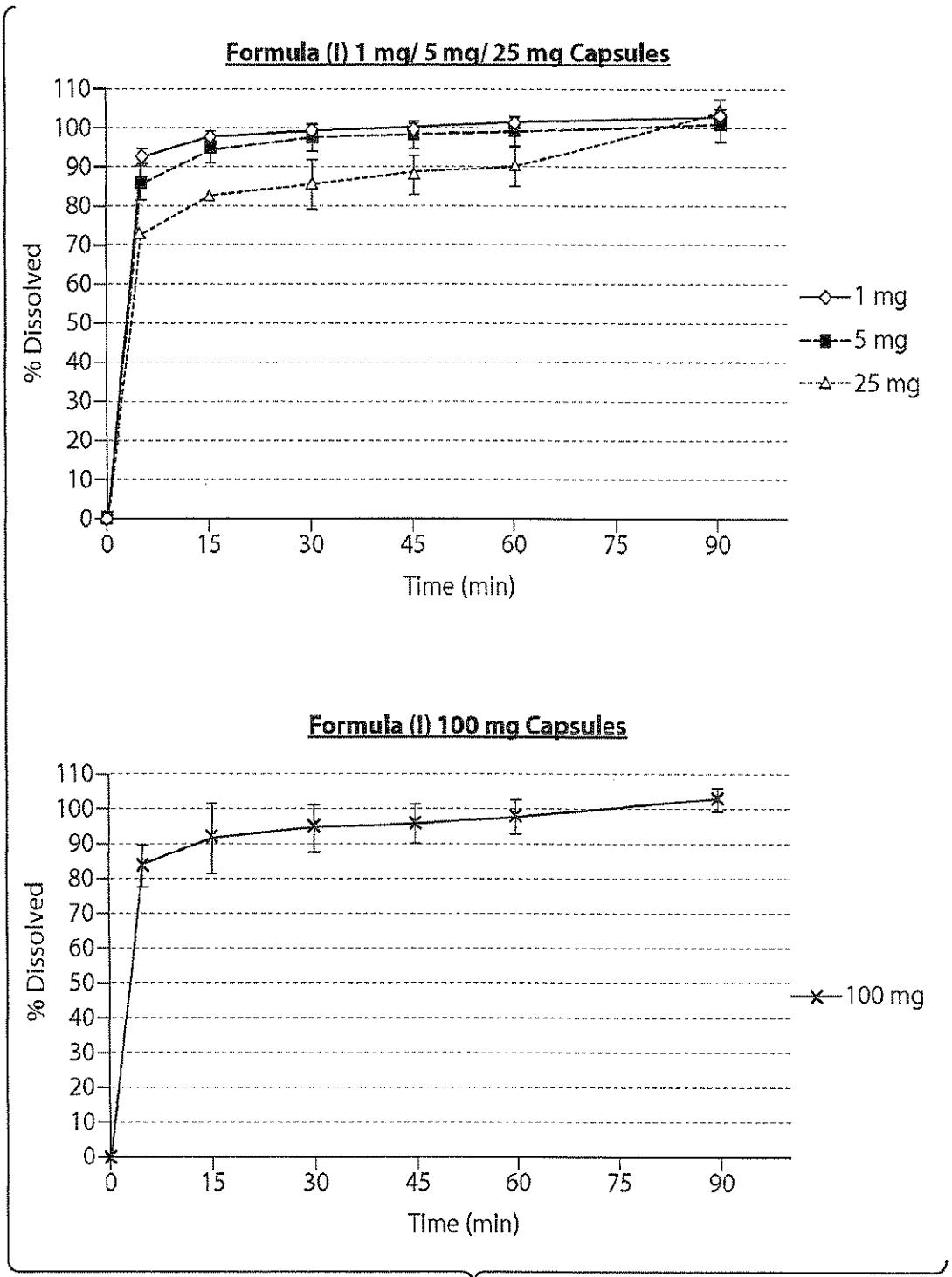

Formula (I) 1 mg/ 5 mg/ 25 mg Capsules
% Dissolved
Time (min)
1 mg
5 mg
25 mg
Formula (I) 100 mg Capsules
% Dissolved
Time (min)
100 mg

Fig. 31