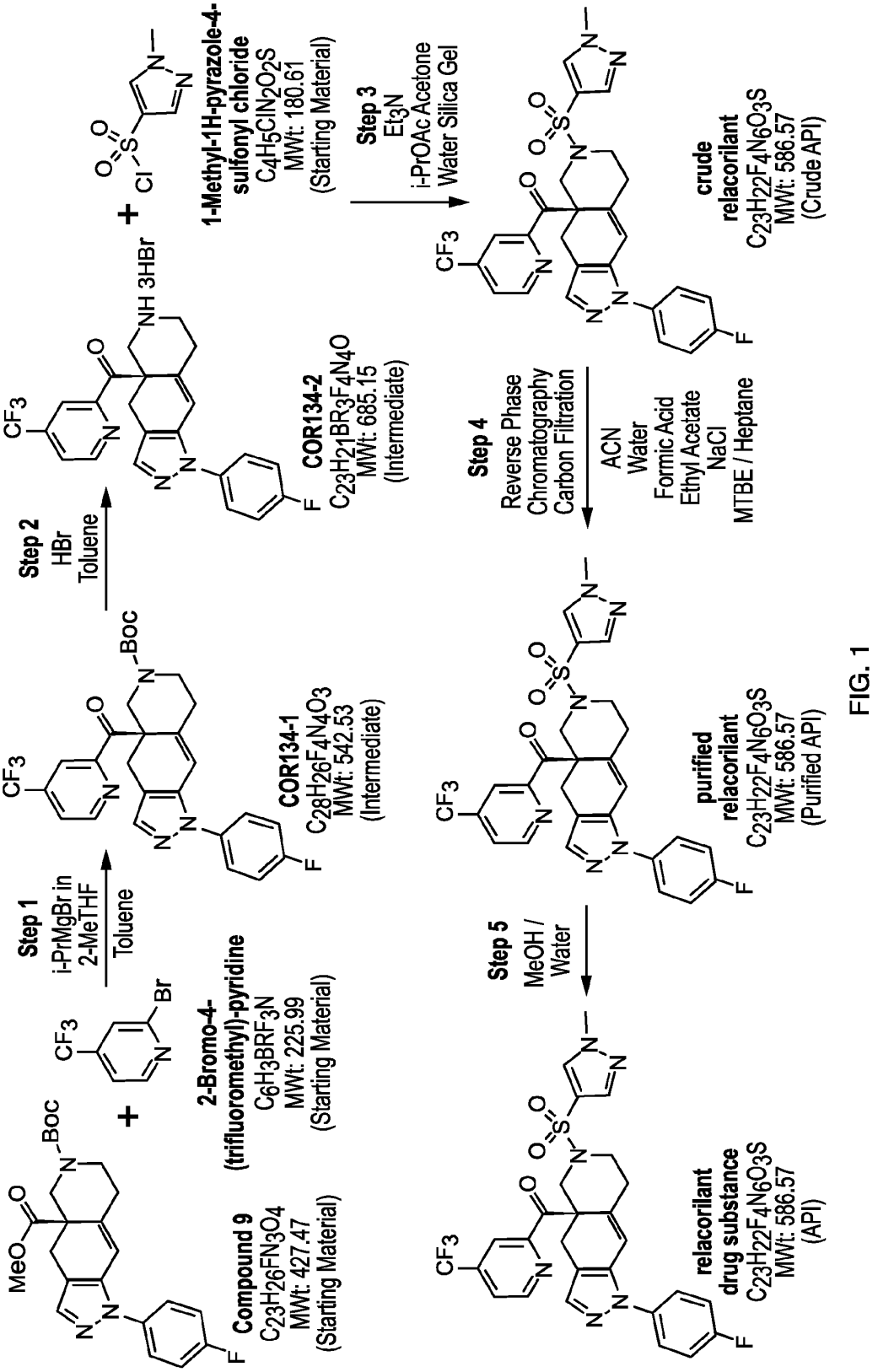
Compound 9
C23H26FN3O4
MWt: 427.47
(Starting Material)
2-Bromo-4-(trifluoromethyl)-pyridine
C6H3BRF3N
MWt: 225.99
(Starting Material)
Step 1
i-PrMgBr in 2-MeTHF
Toluene
COR134-1
C28H26F4N4O3
MWt: 542.53
(Intermediate)
Step 2
HBr
Toluene
COR134-2
C23H21BR3F4N4O
MWt: 685.15
(Intermediate)
1-Methyl-1H-pyrazole-4-sulfonyl chloride
C4H5ClN2O2S
MWt: 180.61
(Starting Material)
Step 3
Et3N
i-PrOAc Acetone
Water Silica Gel
crude relacorilant
C23H22F4N6O3S
MWt: 586.57
(Crude API)
Step 4
Reverse Phase Chromatography
Carbon Filtration
ACN
Water
Formic Acid
Ethyl Acetate
NaCl
MTBE / Heptane
purified relacorilant
C23H22F4N6O3S
MWt: 586.57
(Purified API)
Step 5
MeOH / Water
relacorilant drug substance
C23H22F4N6O3S
MWt: 586.57
(API)

FIG. 1

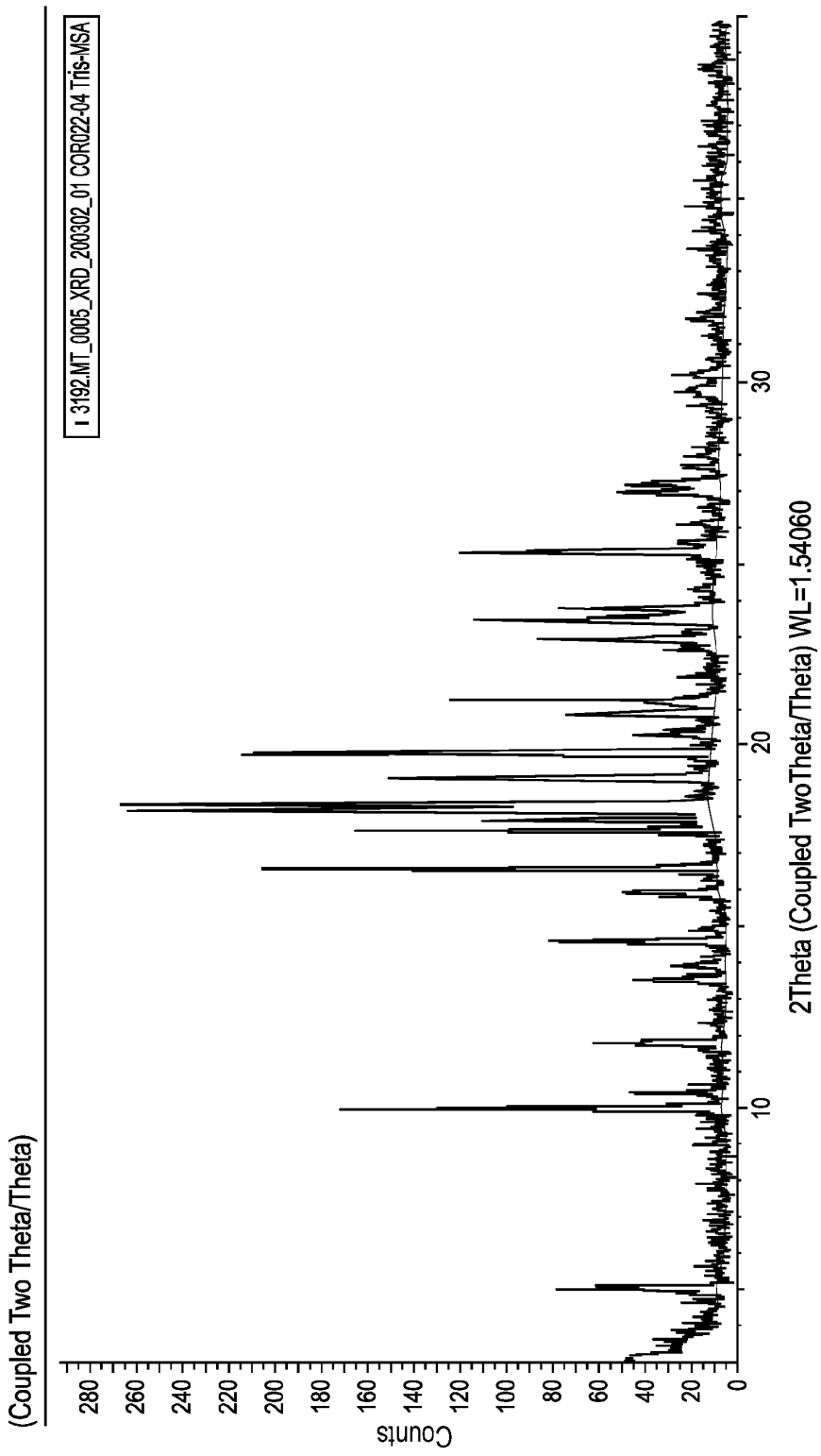
(Coupled Two Theta/Theta)
3192.MT_0005_XRD_200302_01 COR022-04 Tris-MSA
Counts
0
20
40
60
80
100
120
140
160
180
200
220
240
260
280
10
20
30
2Theta (Coupled TwoTheta/Theta) WL=1.54060

FIG. 2

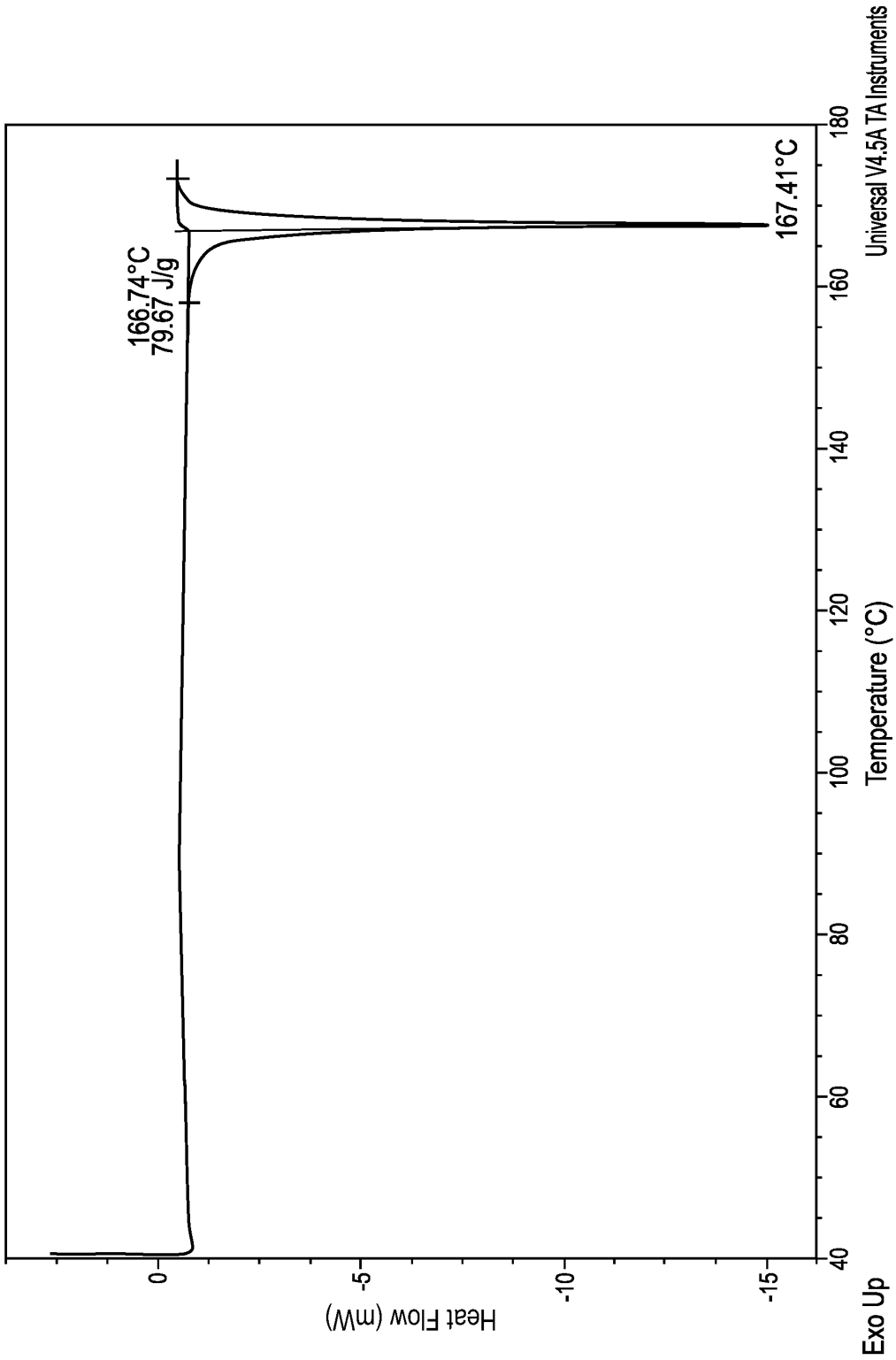
166.74°C
79.67 J/g
167.41°C
Universal V4.5A TA Instruments
Heat Flow (mW)
Temperature (°C)
Exo Up
0
-5
-10
-15
40
60
80
100
120
140
160
180

FIG. 3

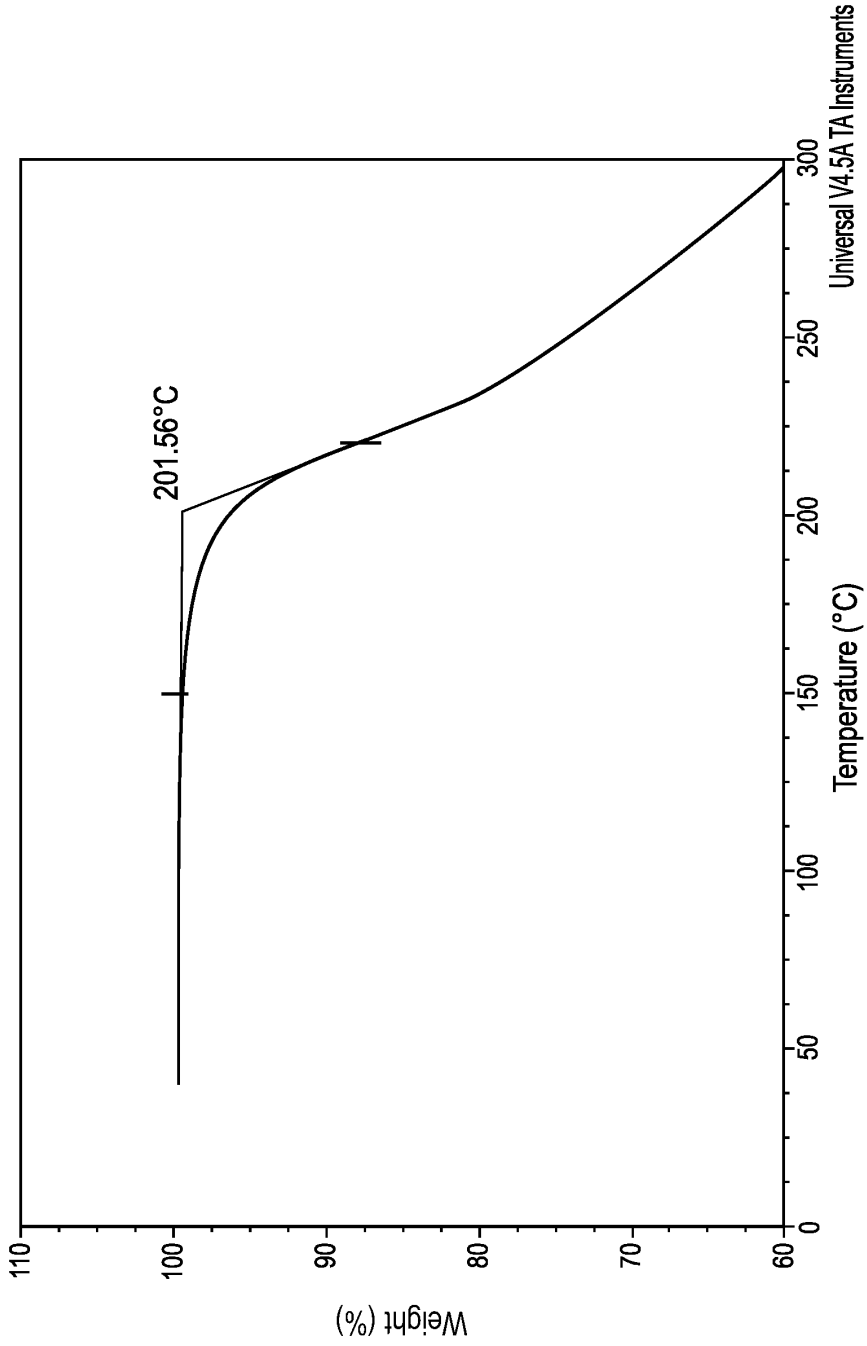
Universal V4.5A TA Instruments
201.56°C
Temperature (°C)
Weight (%)
110
100
90
80
70
60
0
50
100
150
200
250
300

FIG. 4

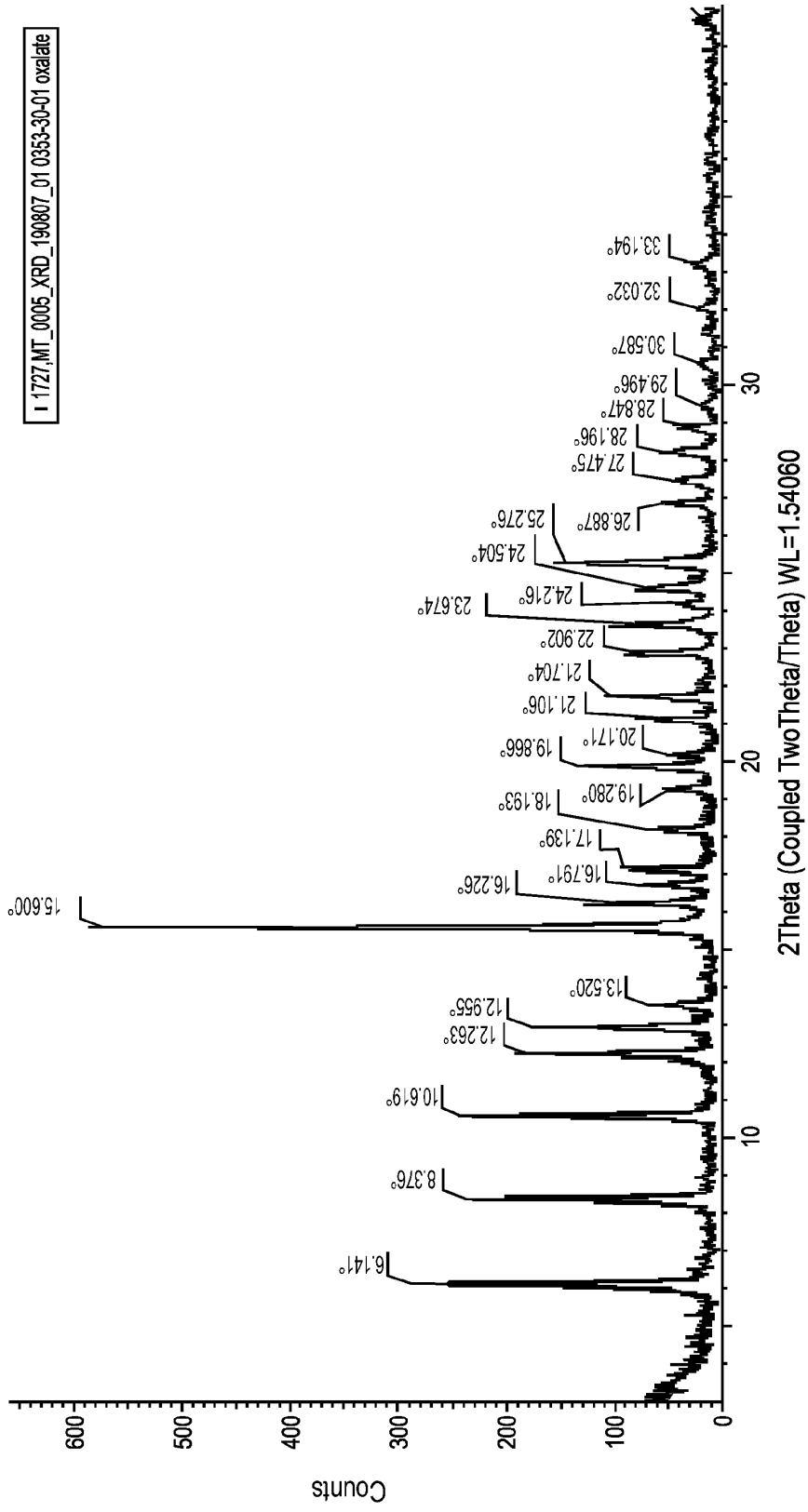
1727,MT_0005_XRD_190807_01 0353-30-01 oxalate
Counts
2Theta (Coupled TwoTheta/Theta) WL=1.54060
0
100
200
300
400
500
600
10
20
30
6.141°
8.376°
10.619°
12.263°
12.955°
13.520°
15.600°
16.226°
16.791°
17.139°
18.193°
19.280°
19.866°
20.171°
21.106°
21.704°
22.902°
23.674°
24.216°
24.504°
25.276°
26.887°
27.475°
28.196°
28.847°
29.496°
30.587°
32.032°
33.194°

FIG. 5

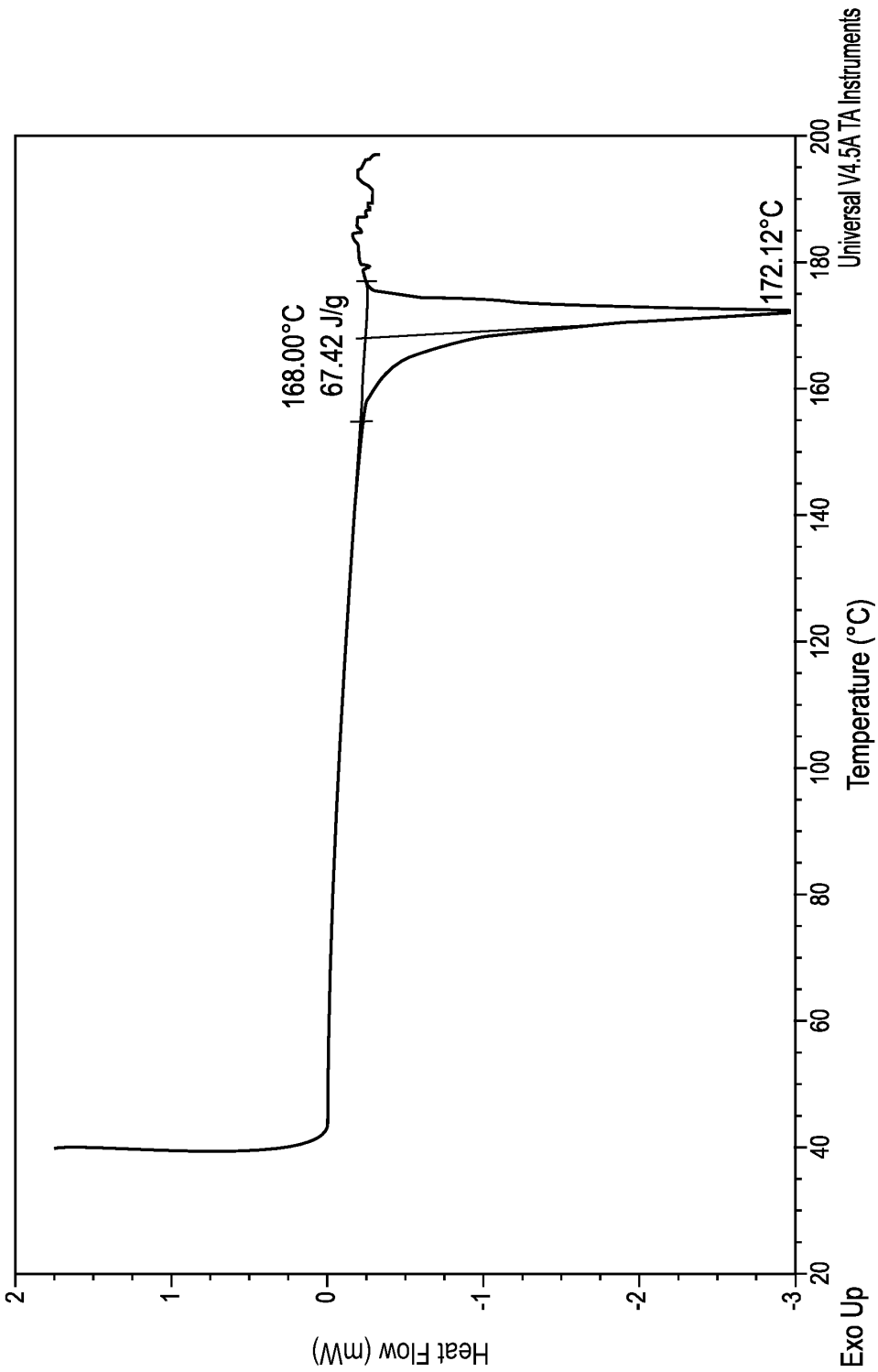
168.00°C
67.42 J/g
172.12°C
Universal V4.5A TA Instruments
Exo Up
Heat Flow (mW)
Temperature (°C)
2
1
0
-1
-2
-3
20
40
60
80
100
120
140
160
180
200

FIG. 6

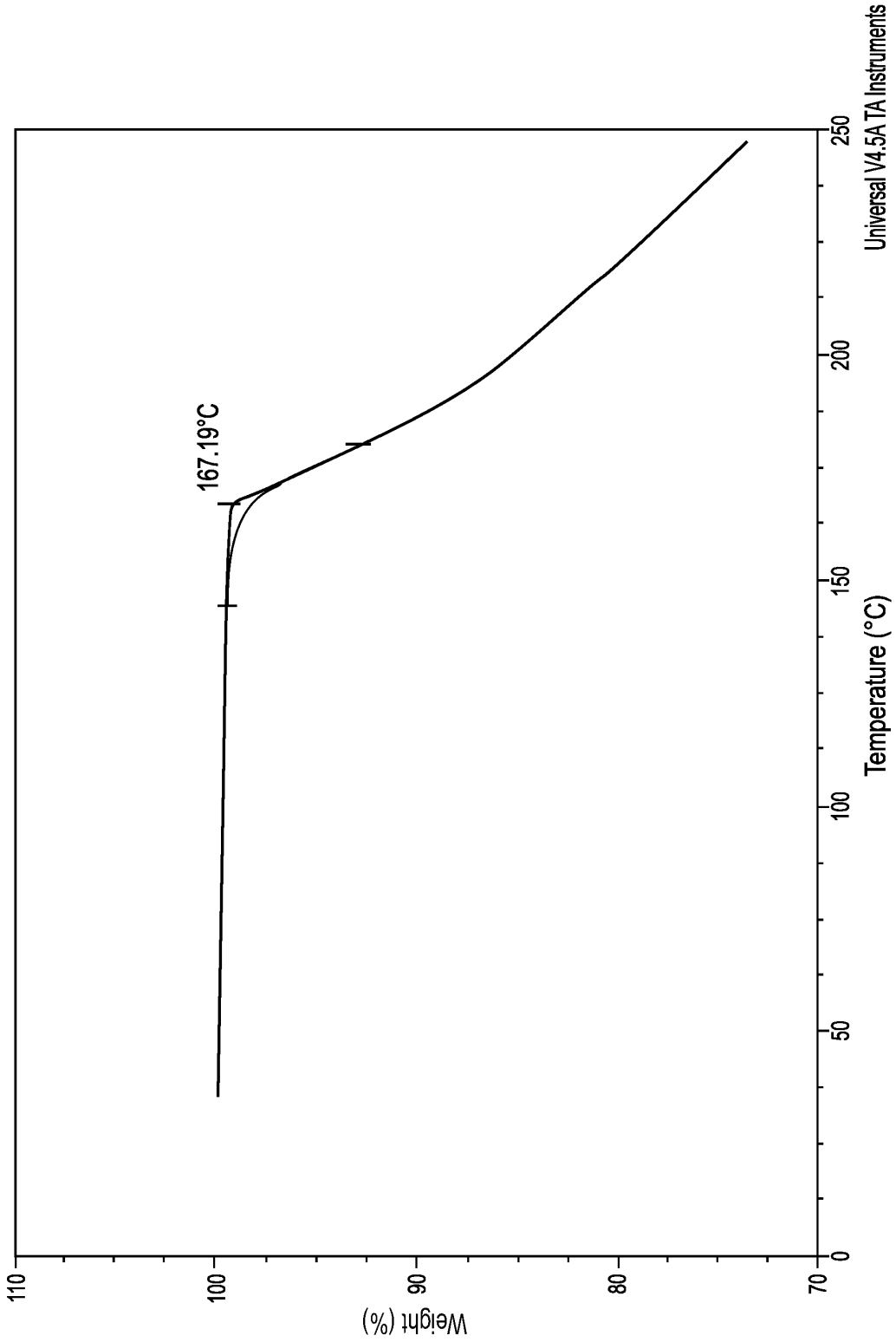
Universal V4.5A TA Instruments
167.19°C
Weight (%)
110
100
90
80
70
0
50
100
150
200
250
Temperature (°C)

FIG. 7

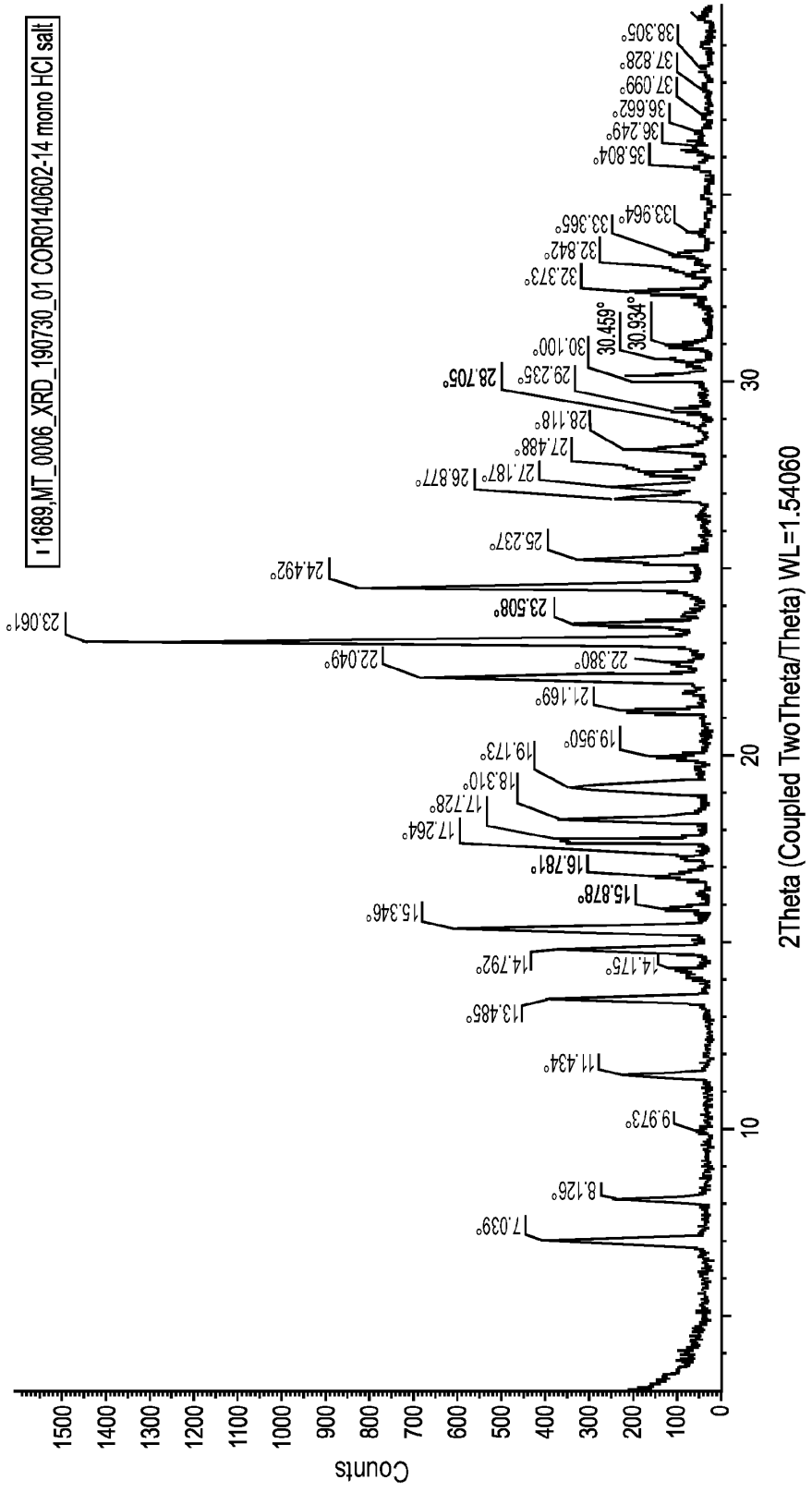
1689,MT_0006_XRD_190730_01 COR0140602-14 mono HCl salt
Counts
2Theta (Coupled TwoTheta/Theta) WL=1.54060
7.039°
8.126°
9.973°
11.434°
13.485°
14.175°
14.792°
15.346°
15.878°
16.781°
17.264°
17.728°
18.310°
19.173°
19.950°
21.169°
22.049°
22.380°
23.061°
23.508°
24.492°
25.237°
26.877°
27.187°
27.488°
28.118°
28.705°
29.235°
30.100°
30.459°
30.934°
32.373°
32.842°
33.365°
33.964°
35.804°
36.249°
36.662°
37.099°
37.828°
38.305°

FIG. 8

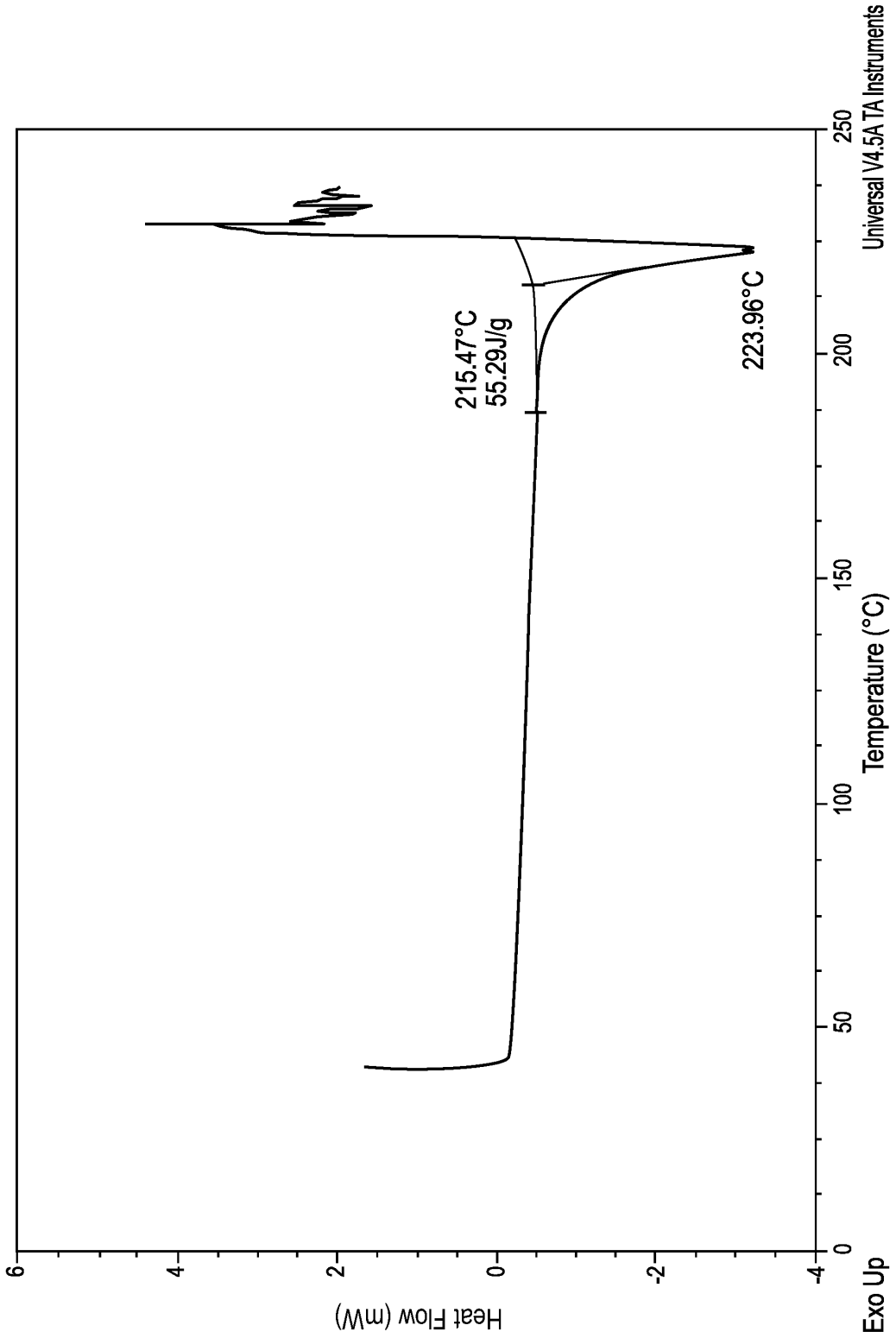
Universal V4.5A TA Instruments
Exo Up
Heat Flow (mW)
6
4
2
0
-2
-4
0
50
100
150
200
250
Temperature (°C)
215.47°C
55.29J/g
223.96°C

FIG. 9

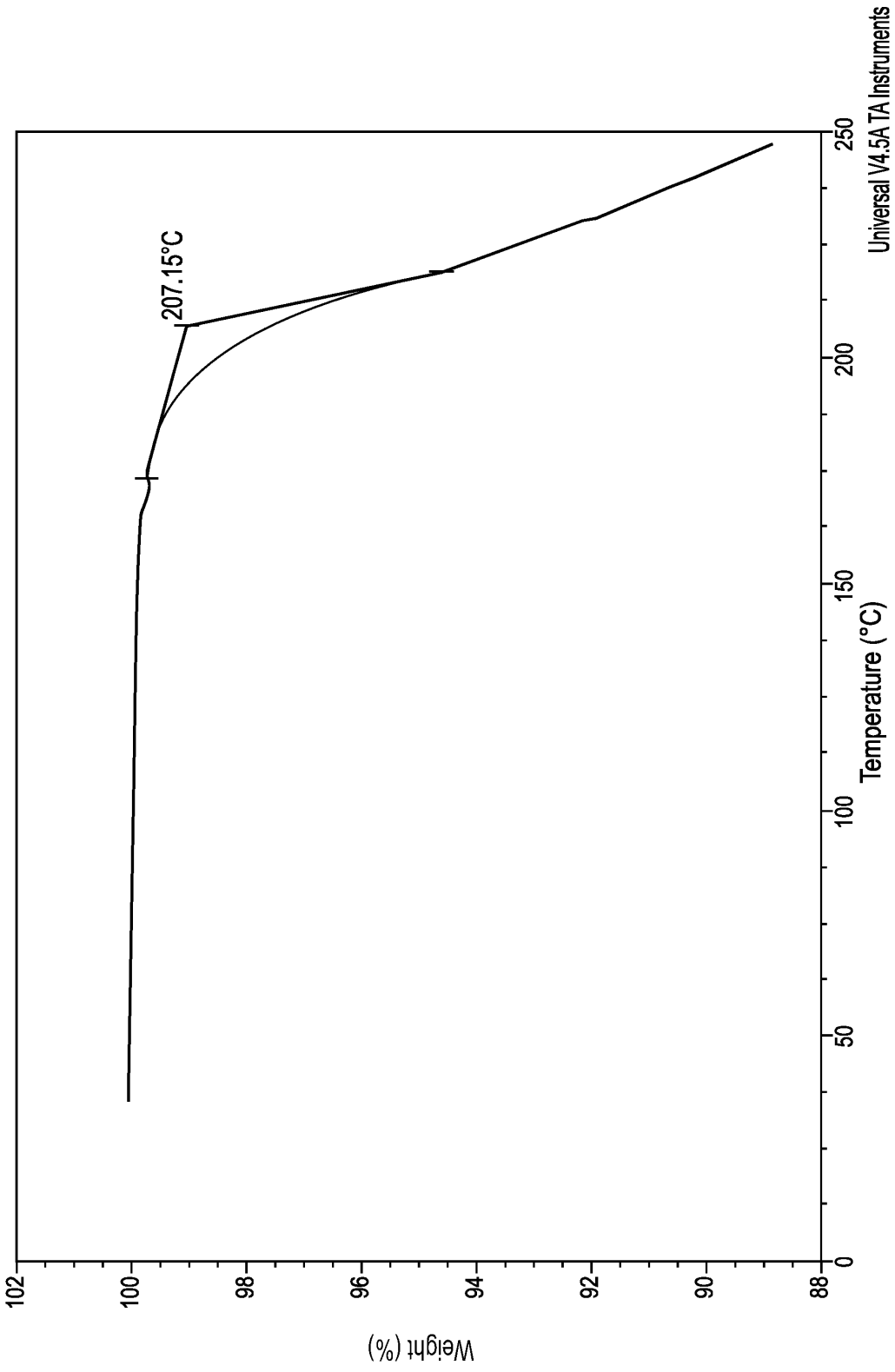
207.15°C
Weight (%)
102
100
98
96
94
92
90
88
0
50
100
150
200
250
Temperature (°C)
Universal V4.5A TA Instruments

FIG. 10