

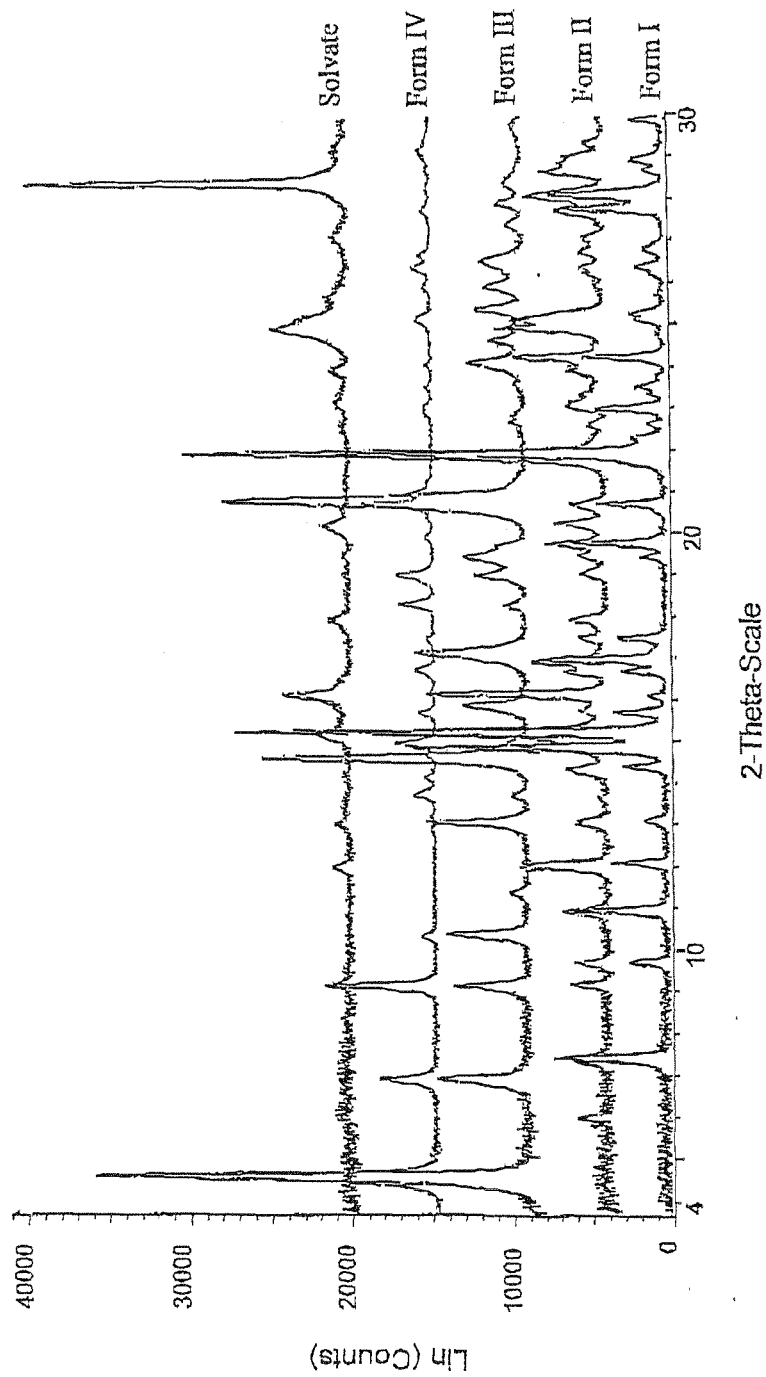


Figure 1. X-ray patterns of various crystal forms of hydrochloric salt

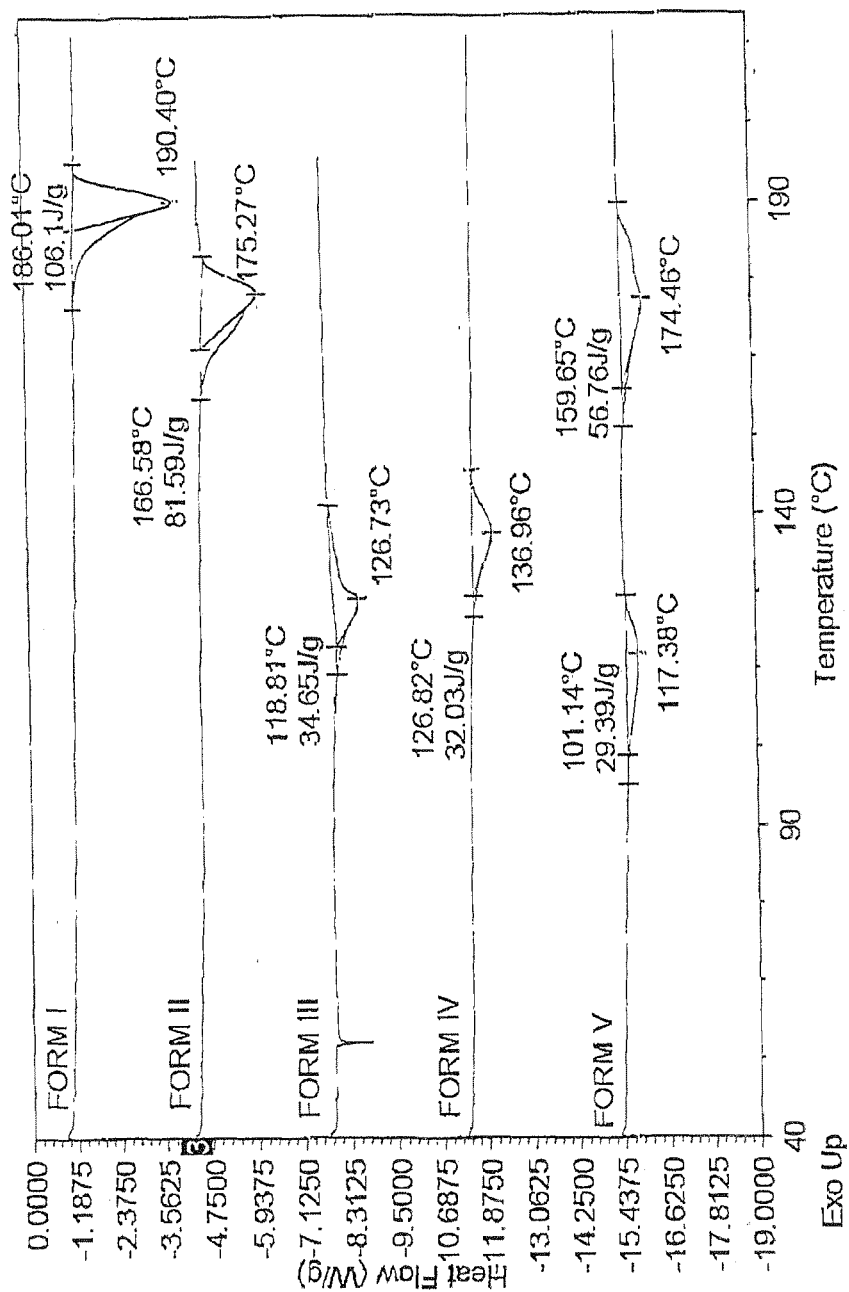


Figure 2- DSC scan of various crystal forms of hydrochloric salt

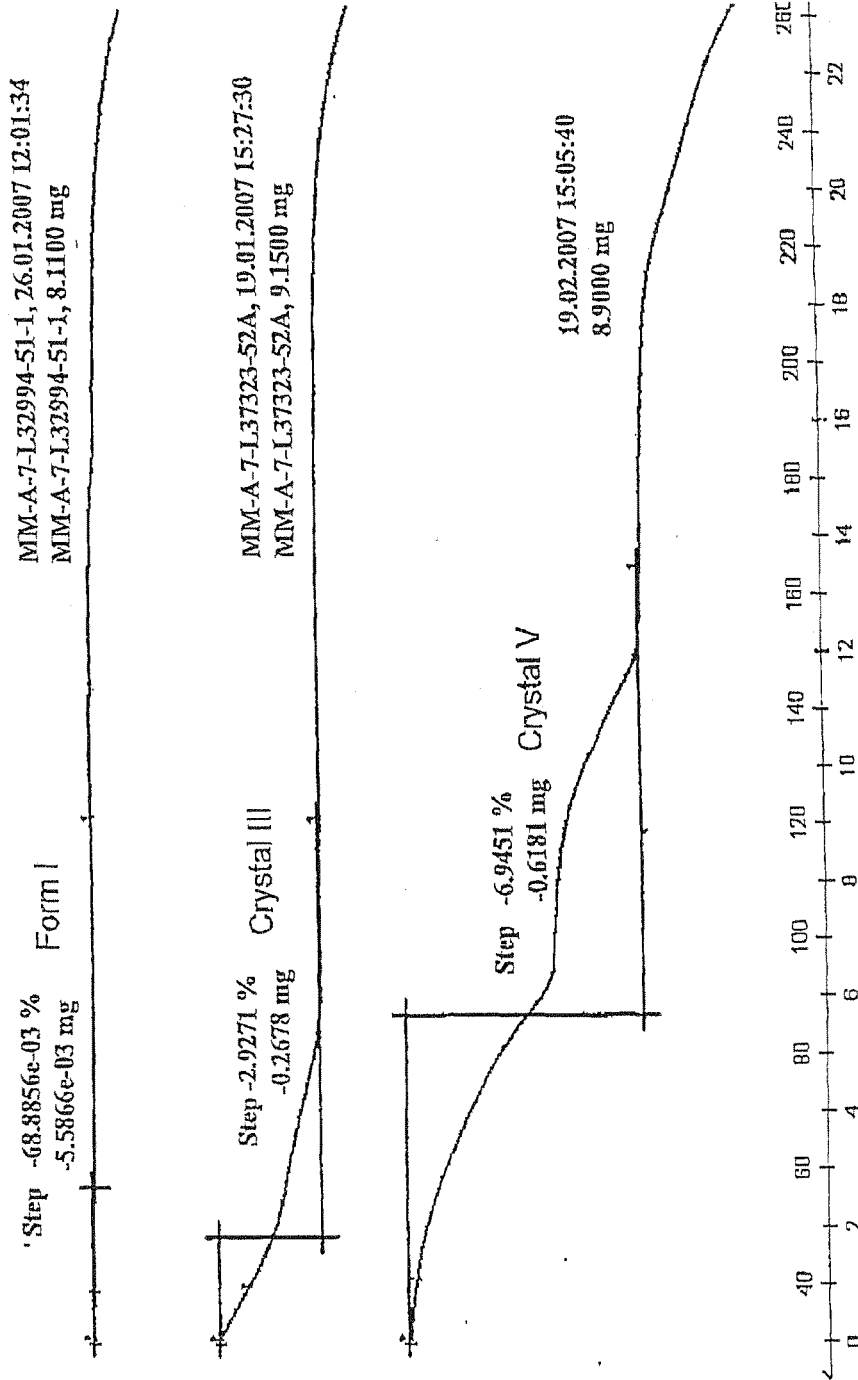


Figure 3- TGA of various crystal forms of hydrochloric salt

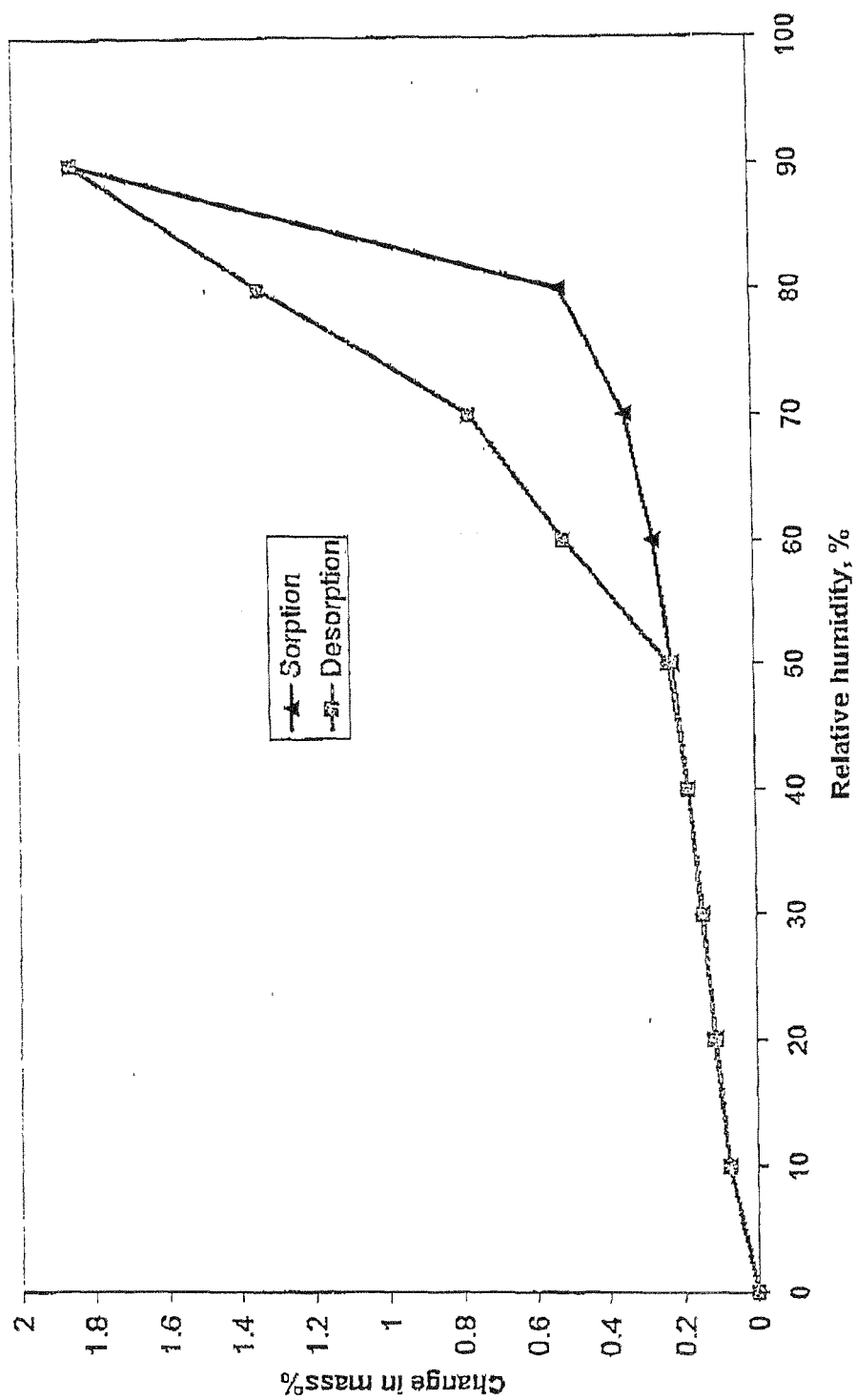


Figure 4- DVS of mono-HCl salt (NO form change after DVS test)

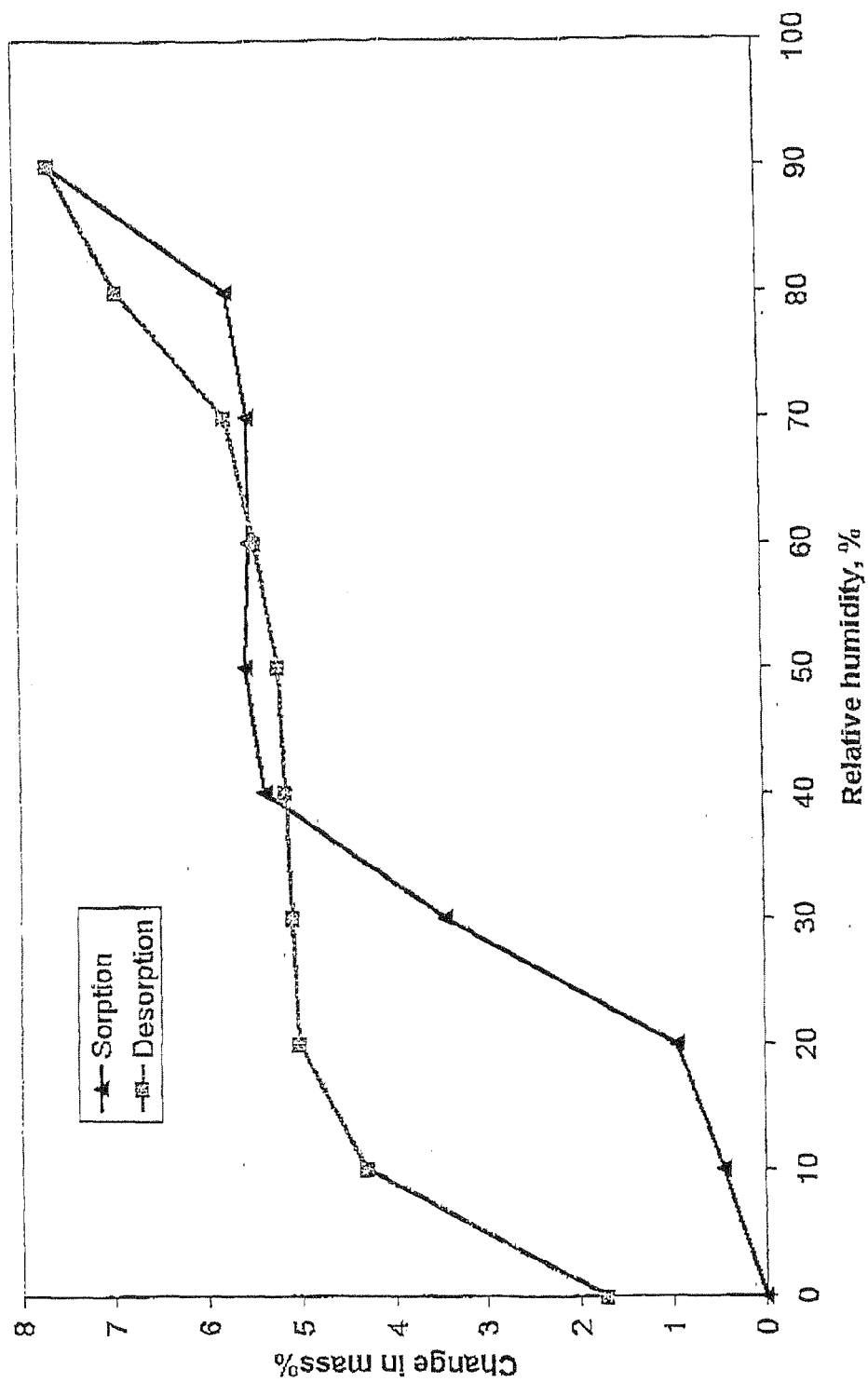


Figure 5- DVS of hydrochloric salt (crystal II) (NO form change after DVS)

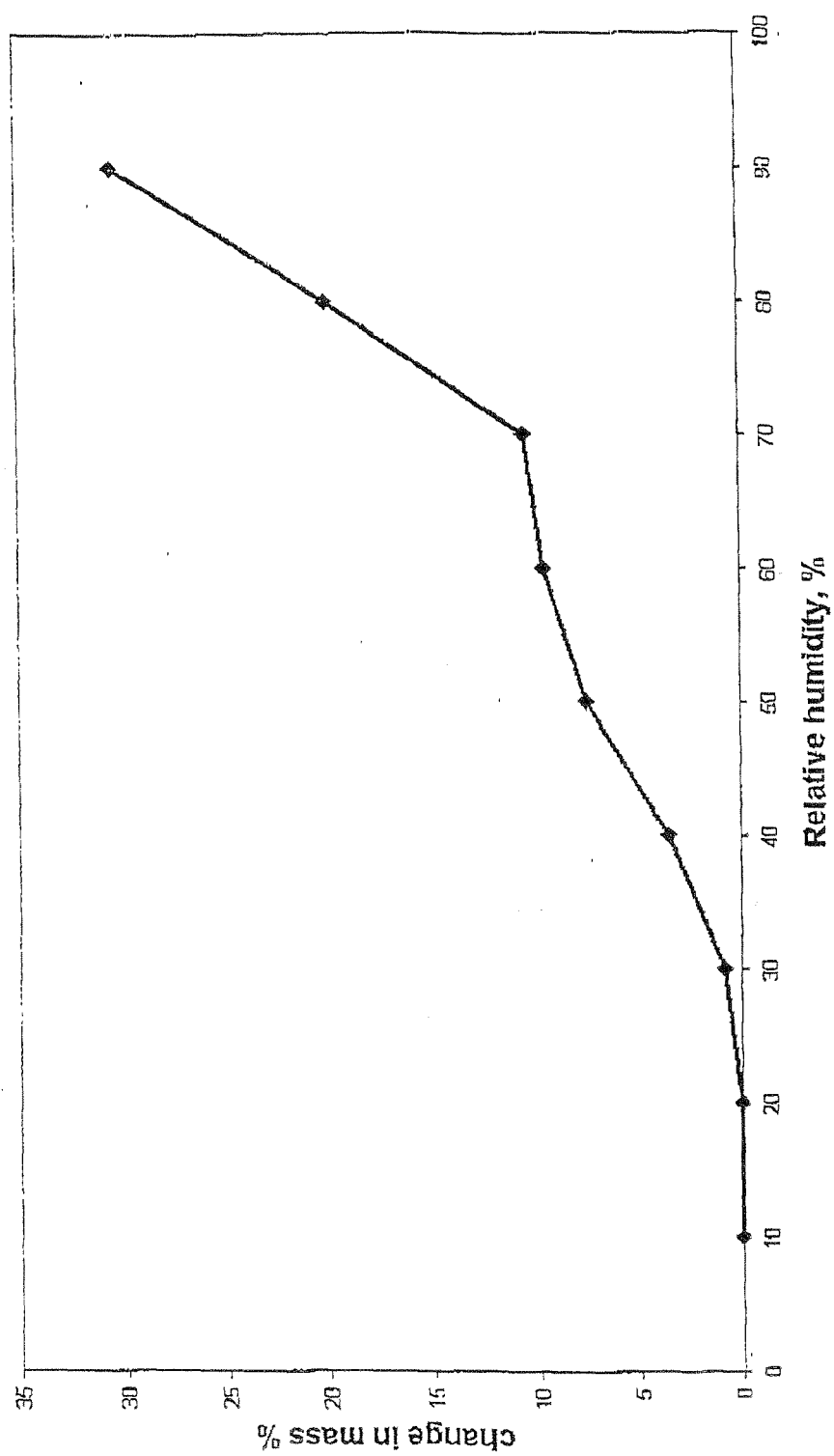


Figure 6- DVS of hydrochloric salt (crystal III) (data from pre-selection minute)

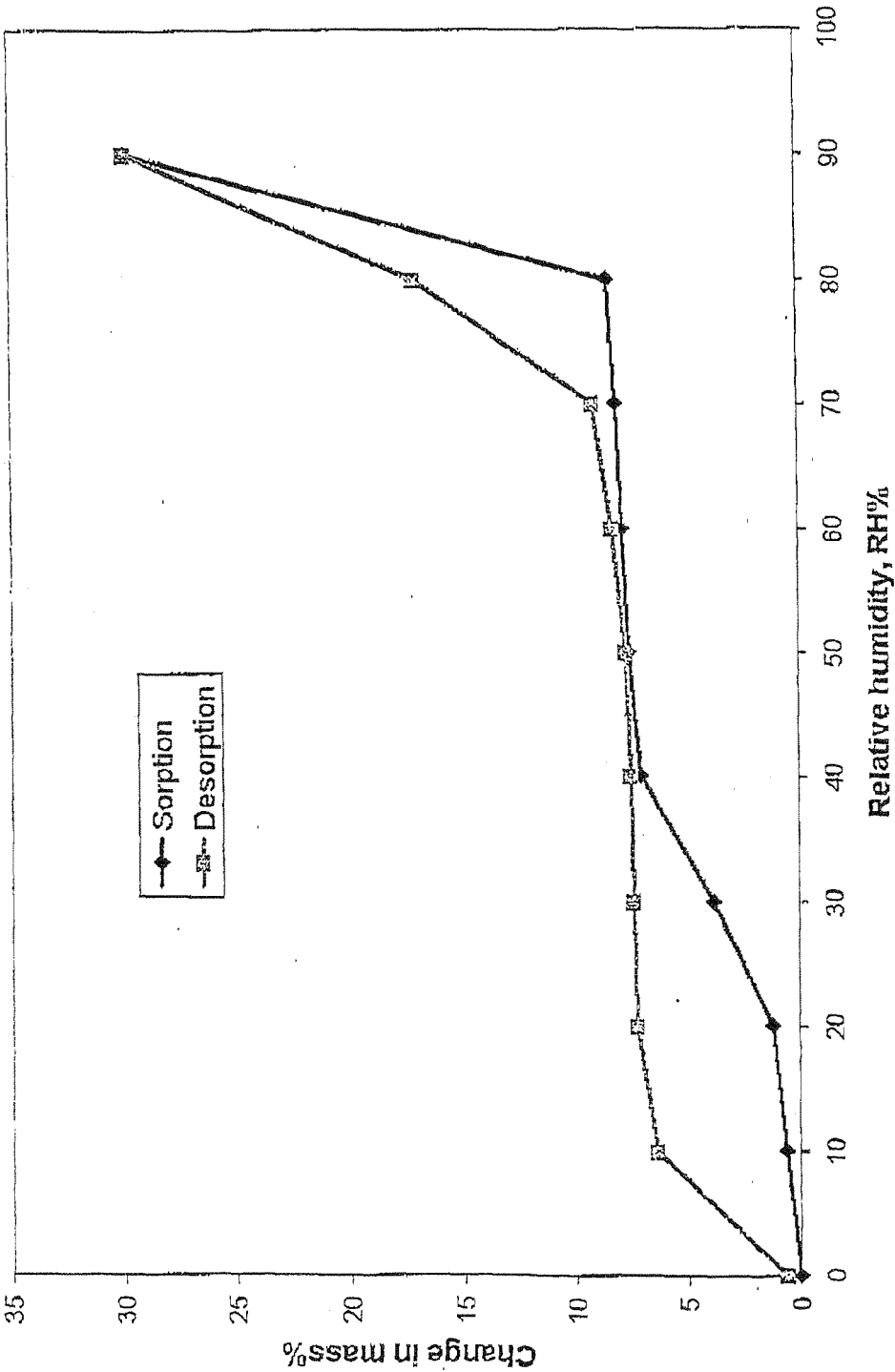


Figure 7- DVS of hydrochloric salt (crystal V)

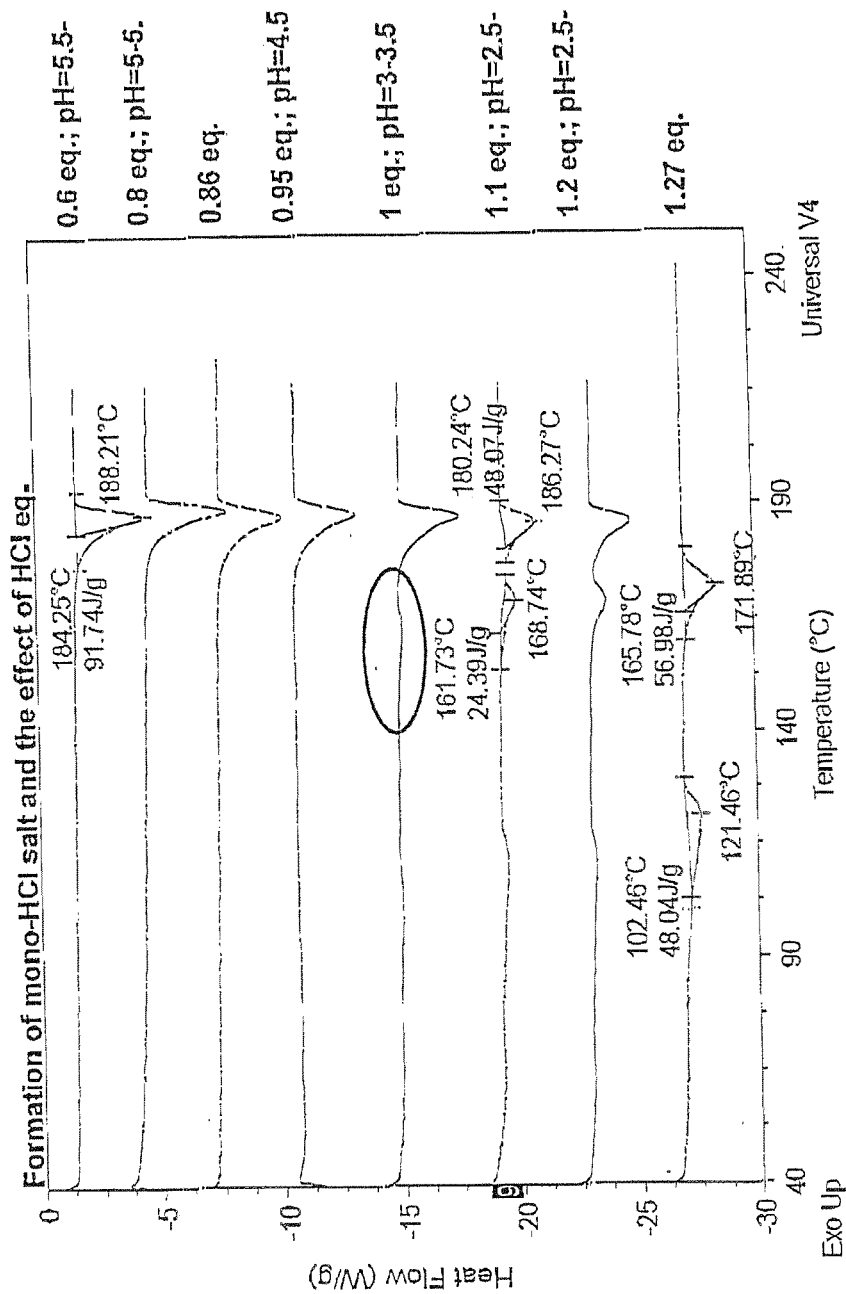


Figure 8- Effect of pH and HCl equivalence on HCl salt formation

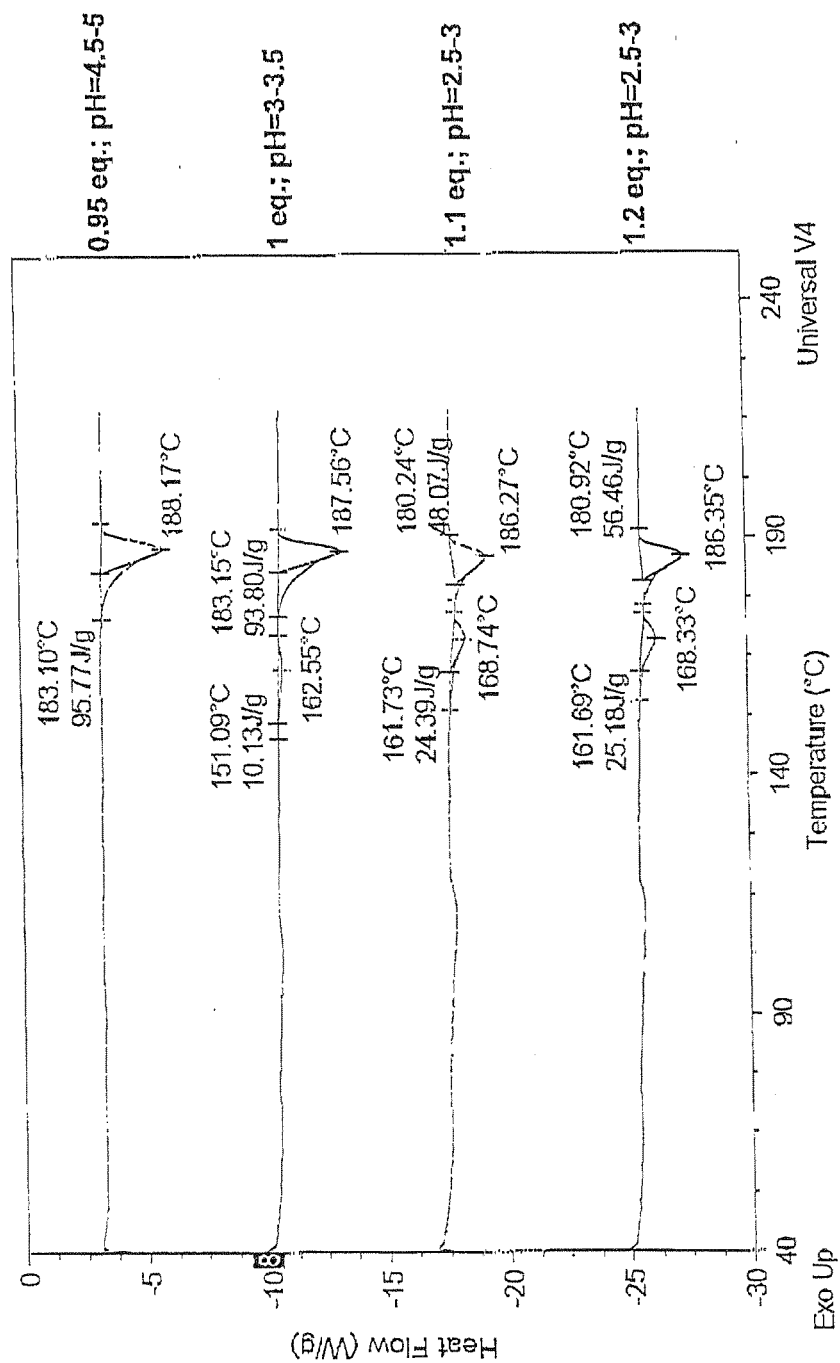


Figure 9- Effect of pH and HCl equivalence on HCl salt formation

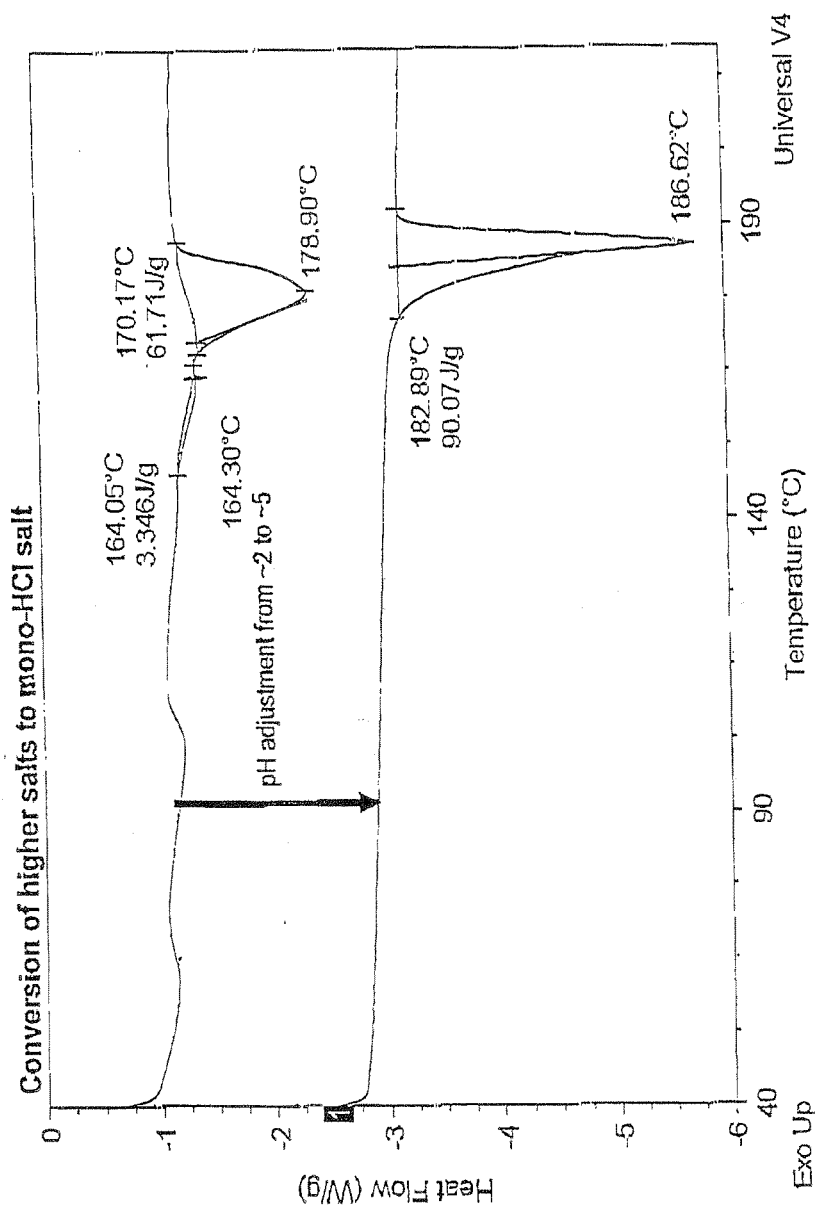


Figure 10- Conversion of higher salts to mono-HCl crystal I 259 mg di-HCl salt was slurried in 4 volumes acetone + 0.5 volume ethanol ASDQ at room temperature. The resulting slurry gave a pH of ~2. To increase the pH, 0.02 mL NaOH 30% was added which increased the pH to 5-5.5. The slurry was stirred overnight and converted to mono-HCl. 173 mg mono-HCl was obtained.

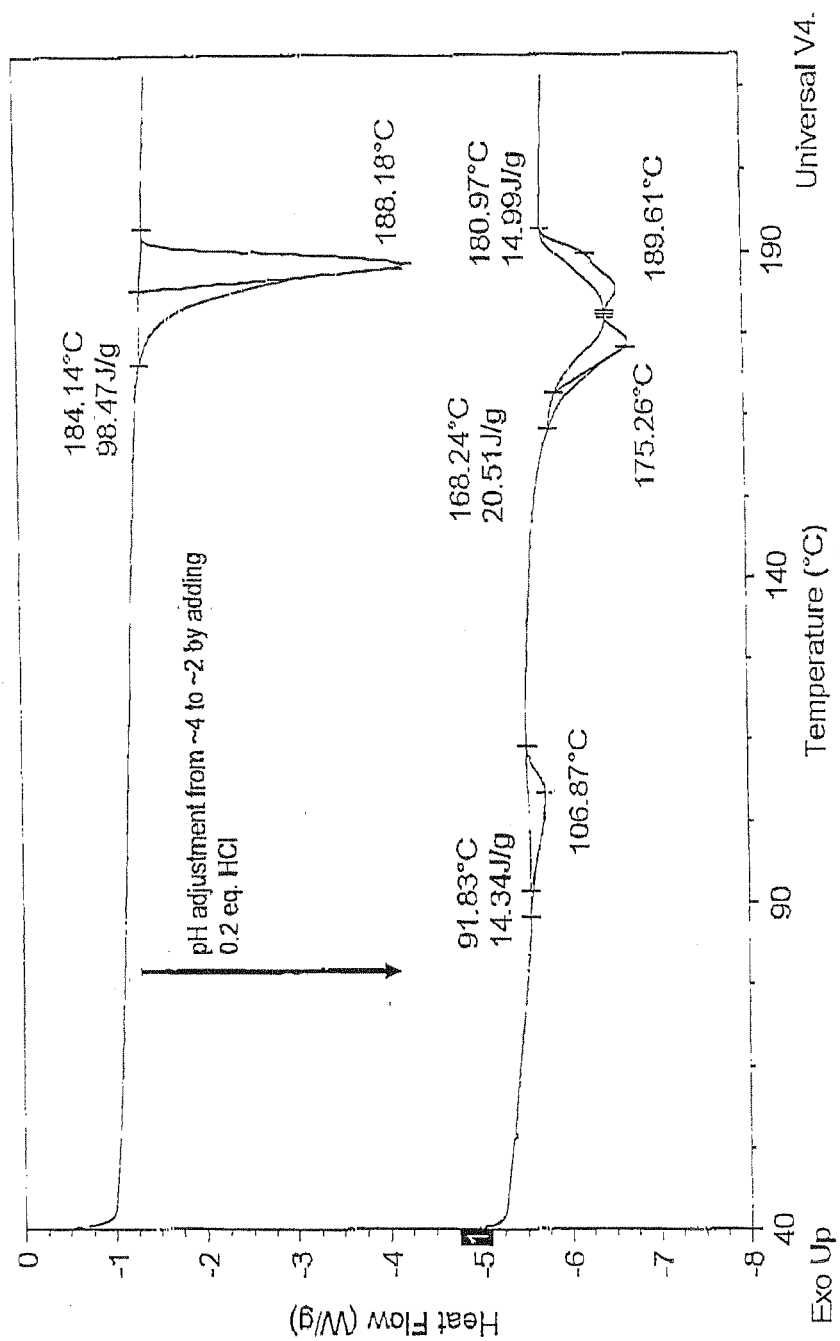


Figure 11- Conversion of mono-HCl to Form II by decreasing the pH (slurried overnight)

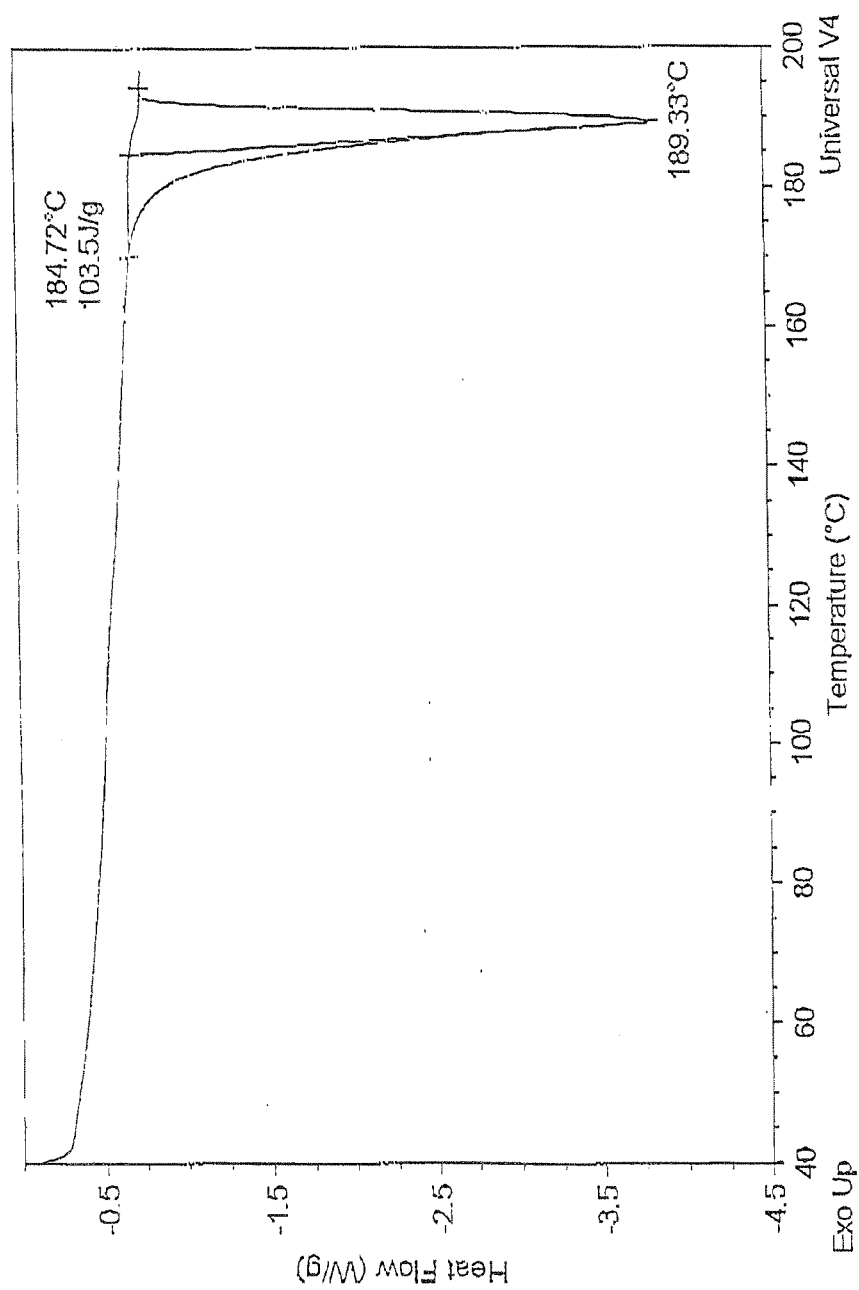


Figure 12- DSC scan of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl))-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form I

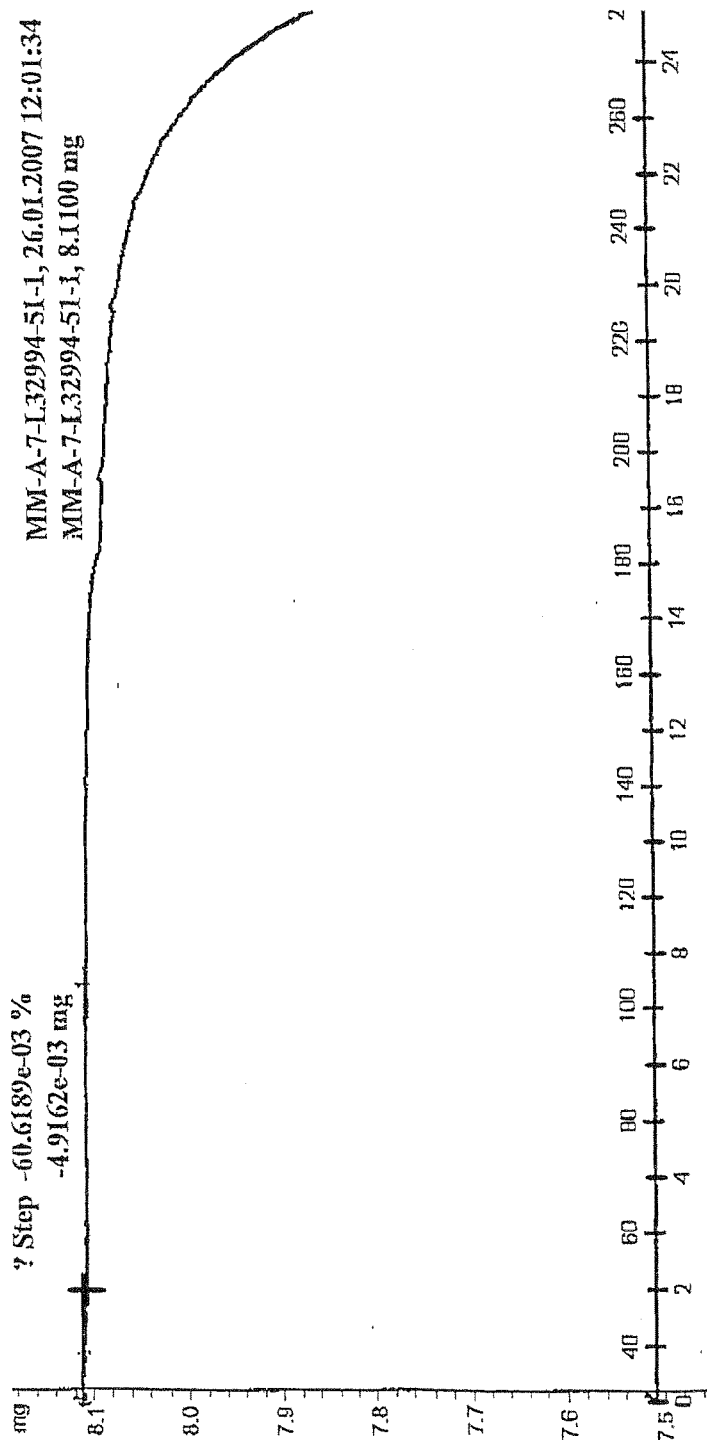


Figure 13- TGA thermogram of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl))-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form I

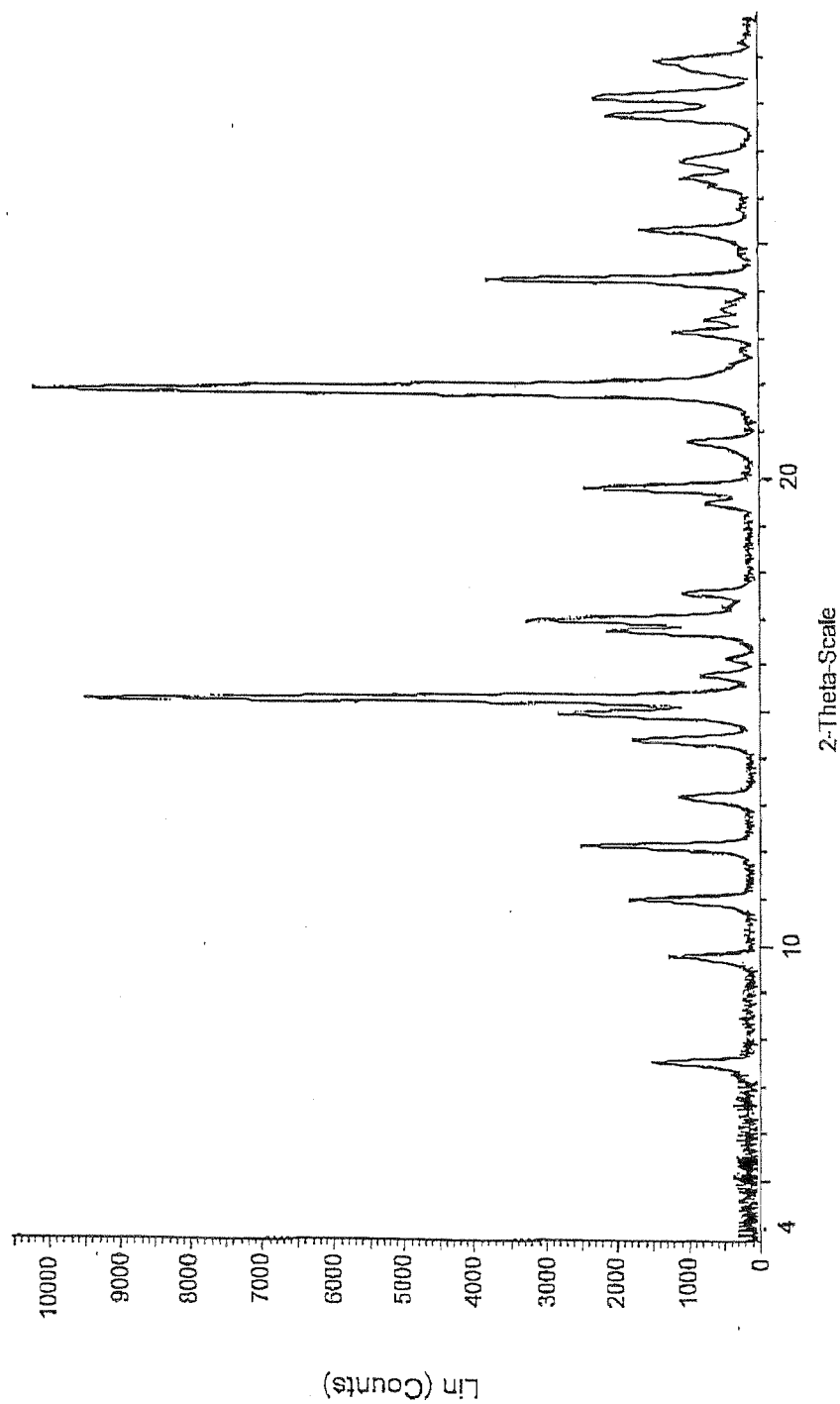


Figure 14- X-ray diffraction pattern of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl)-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form I

Figure 14 continued

Table 1 - X-ray diffraction data of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl)-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form I

Angle, 2-Theta °	d value, Angstrom	Intensity, Count	Intensity, %
7.5	11.8	1484	14.5
9.7	9.1	1252	12.2
11.0	8.1	1795	17.5
12.1	7.3	2502	24.5
13.1	6.7	1122	11.0
14.4	6.2	1750	17.1
14.9	5.9	2814	27.5
15.3	5.8	9510	93.0
15.8	5.6	789	7.7
16.1	5.5	437	4.3
16.7	5.3	2108	20.6
17.0	5.2	3257	31.6
17.5	5.1	1075	10.5
19.5	4.6	740	7.2
19.8	4.5	2439	23.8
20.8	4.3	987	9.5
21.9	4.1	10228	100.0
23.1	3.8	1184	11.6
23.4	3.8	712	7.0
24.3	3.7	3787	37.0
25.9	3.5	1634	16.0
26.4	3.4	1000	9.8
26.5	3.3	1058	10.3
27.8	3.2	2091	20.4
28.1	3.2	2278	22.3
28.7	3.1	934	9.1
28.9	3.1	1433	14.0

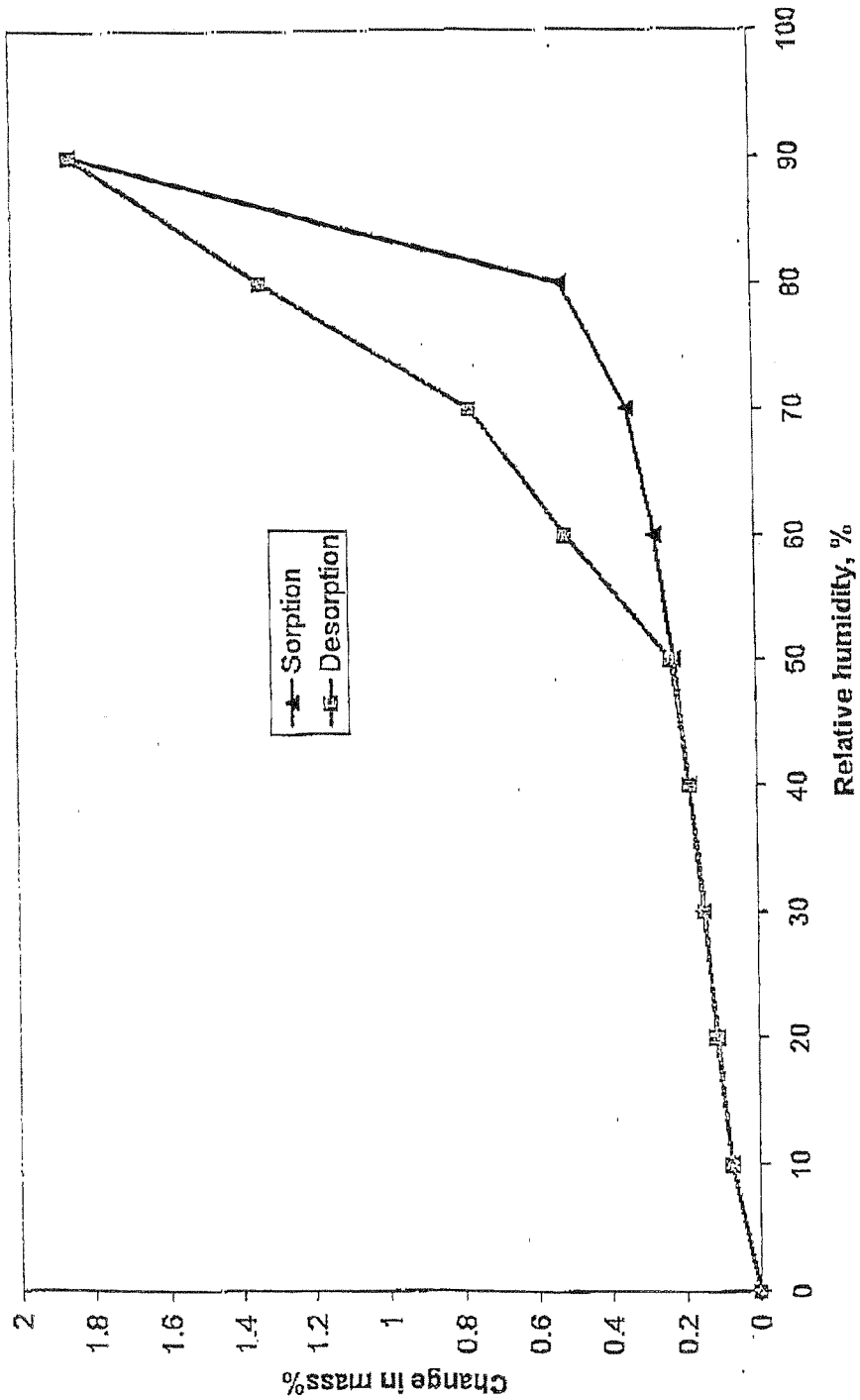


Figure 15- DVS isothermal analysis of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl)-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form I

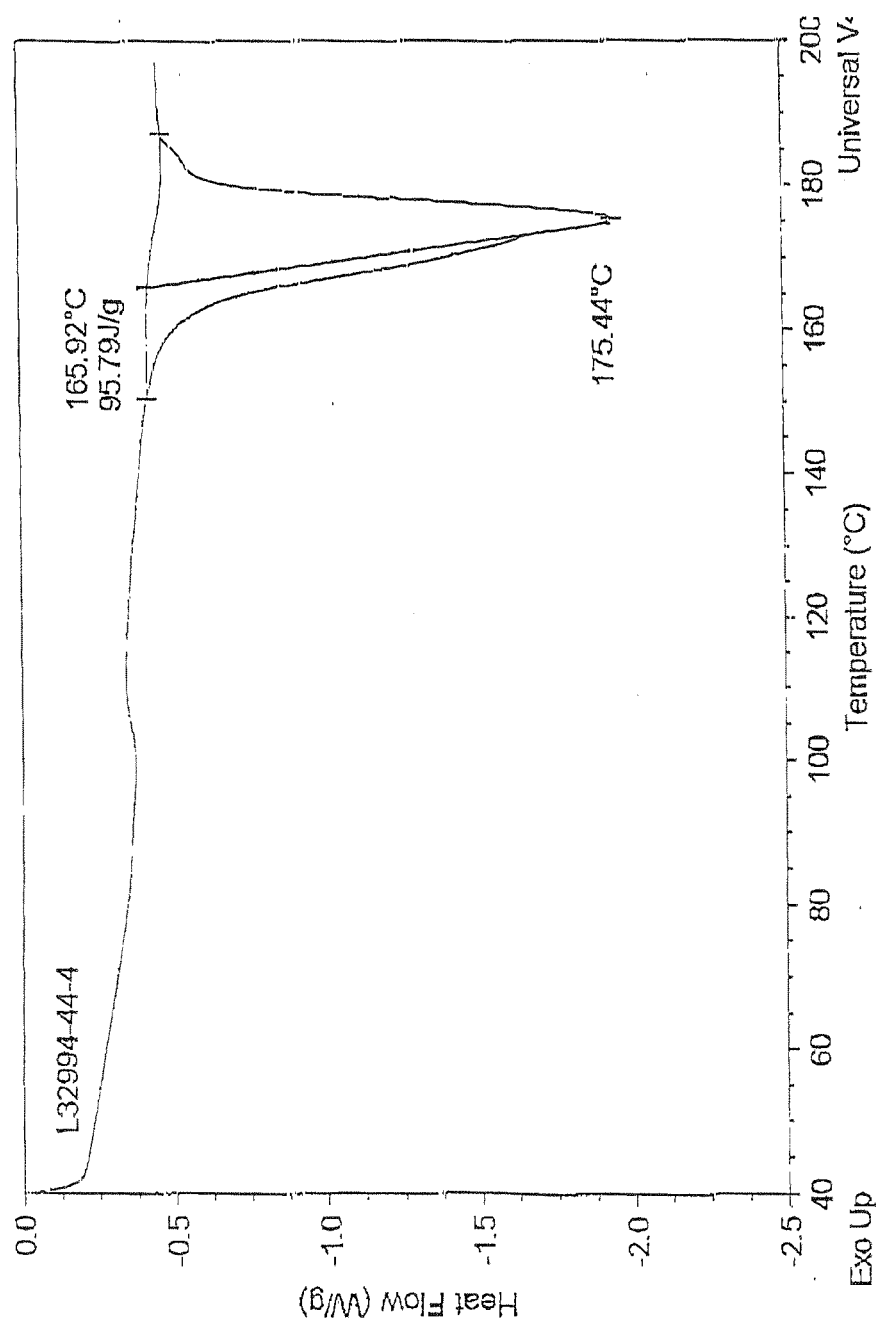


Figure 16- DSC scan of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl)-1H-pyrazol-3-yl) pentanamide hydrochloric salt Form II

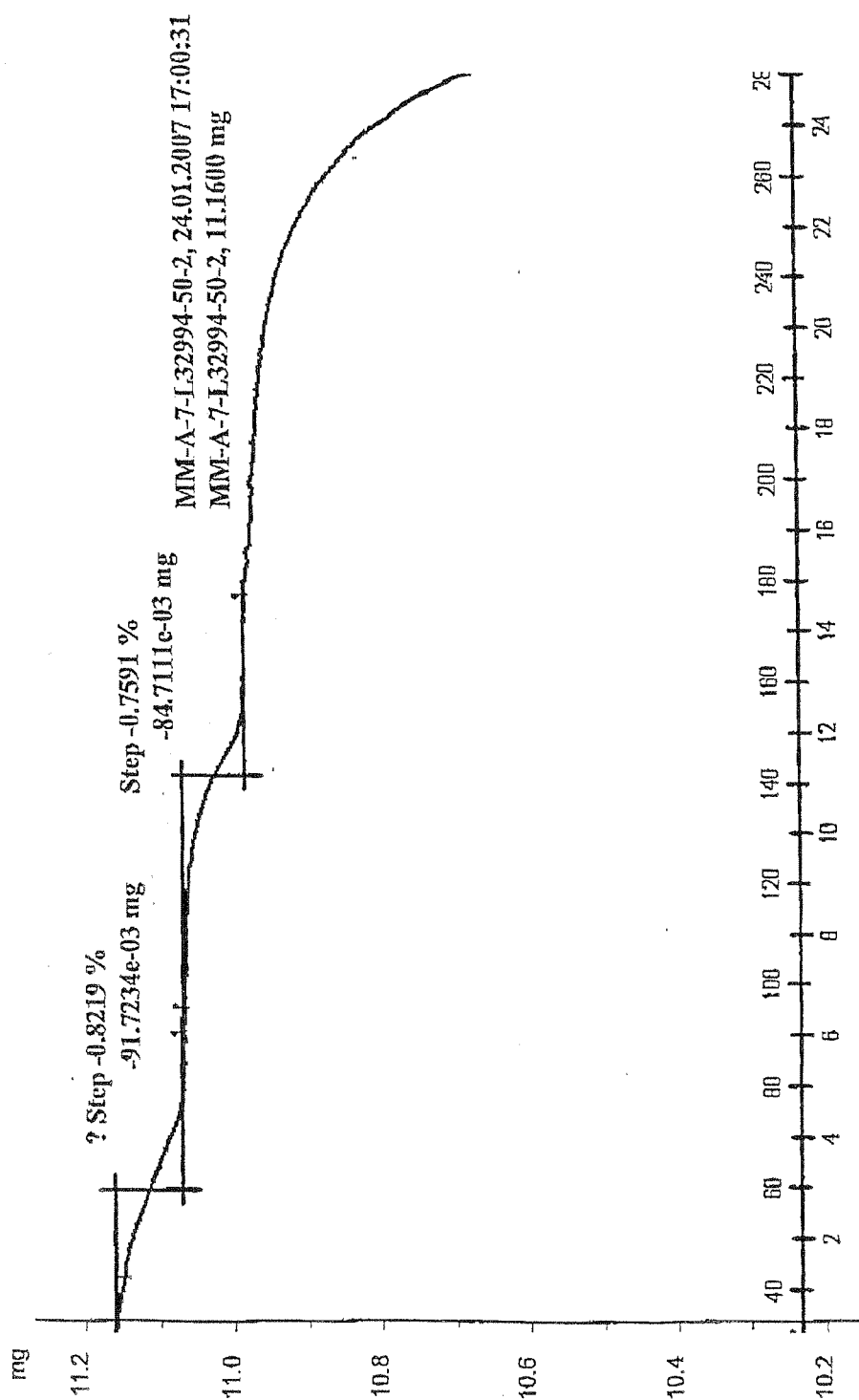


Figure 17- TGA thermogram of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl))-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form II

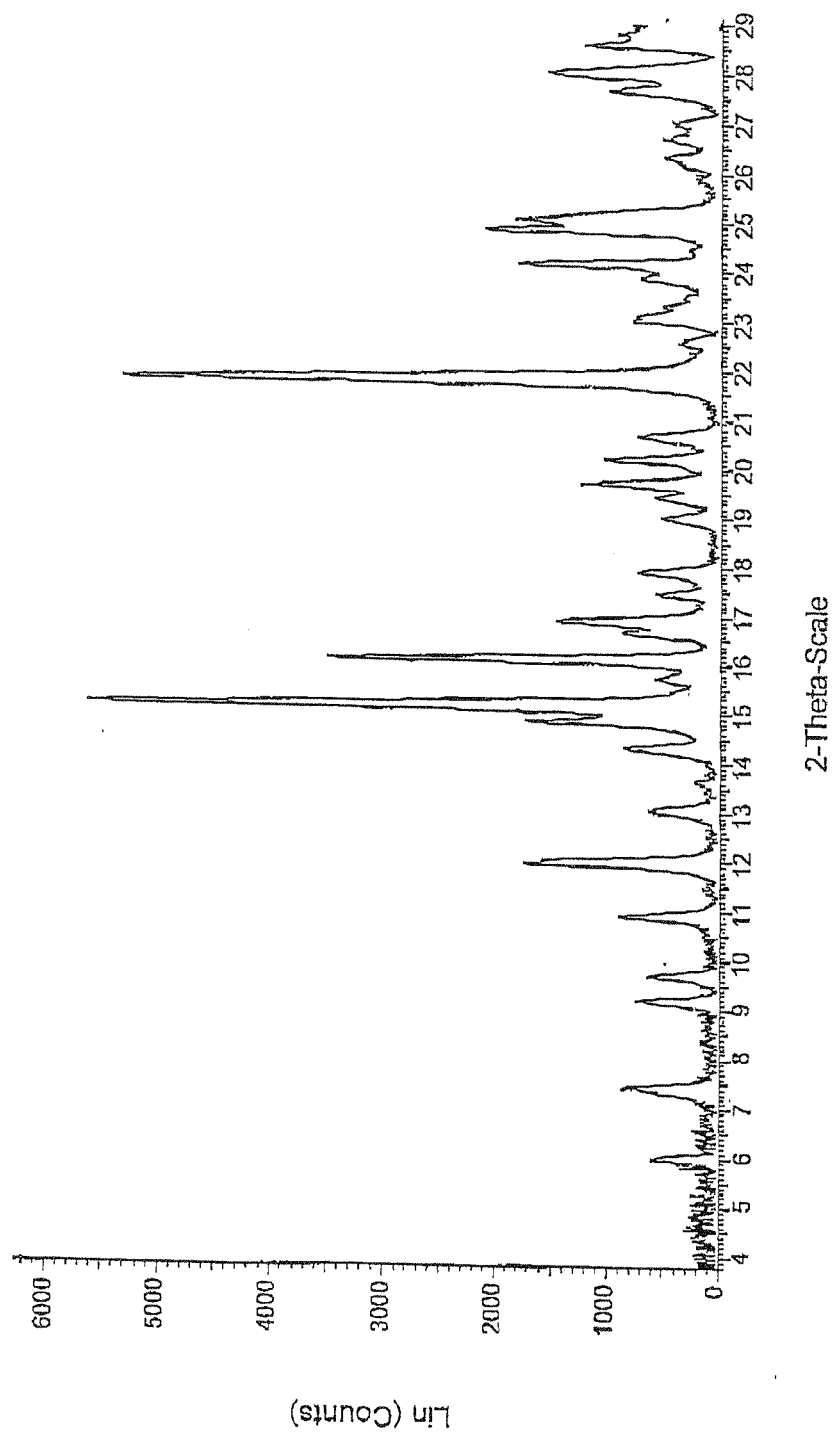


Figure 18- X-ray diffraction pattern of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl)-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form II

Figure 18 continued

Table 2 - X-ray diffraction data of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl)-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form II

Angle, 2-Theta °	d value, Angstrom	Intensity, Count	Intensity %
6.0	14.8	588	10.4
7.4	11.9	827	14.6
9.2	9.6	735	13.0
9.7	9.1	686	11.3
10.9	8.1	888	15.7
12.0	7.4	1741	30.8
13.0	6.8	625	11.1
14.3	6.2	849	15.0
14.9	6.0	1723	30.6
15.2	5.8	5647	100.0
16.1	5.5	3506	62.1
16.7	5.3	855	15.1
16.9	5.2	1454	25.7
17.5	5.1	542	9.6
17.9	4.9	730	12.8
19.0	4.7	491	8.7
19.5	4.6	576	10.2
19.7	4.5	1239	21.9
20.2	4.4	1032	18.3
20.7	4.3	727	12.9
21.9	4.1	5332	94.4
22.6	3.9	358	6.3
23.1	3.8	747	13.2
23.9	3.7	706	12.5
24.2	3.7	1791	31.7
24.9	3.6	2089	37.0
25.1	3.5	1828	32.4
26.4	3.4	495	8.8
26.7	3.3	500	8.9
27.7	3.2	989	17.5
28.1	3.2	1527	27.0
28.7	3.1	1204	21.3
29.0	3.1	762	13.5
29.5	3.0	390	6.9

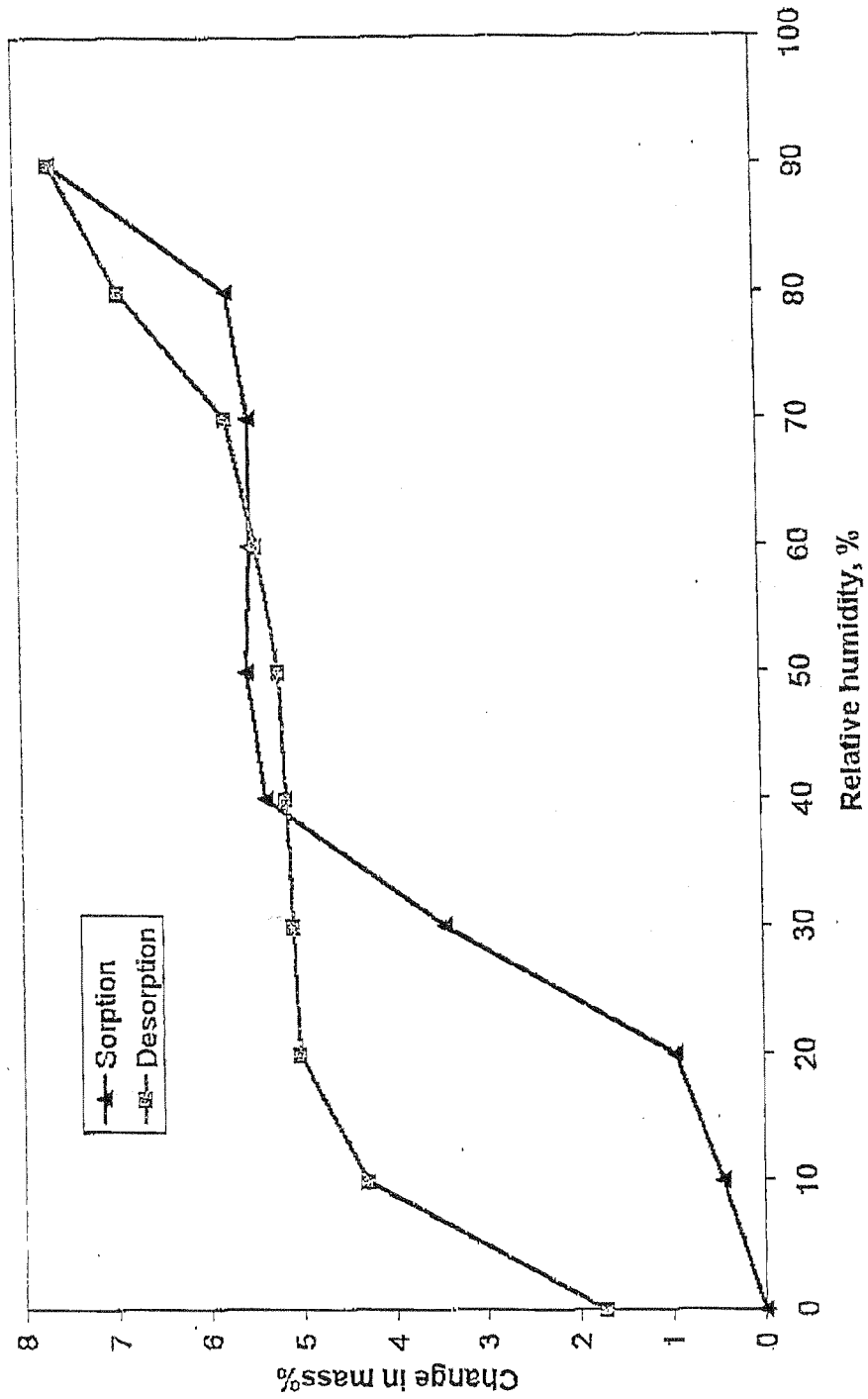


Figure 19. DVS isothermal analysis of 5-(4-acetyl-1,4-diazepan-1-yl)-N-(5-(4-methoxyphenyl))-1H-pyrazol-3-yl)pentanamide hydrochloric salt Form II