

Figure 1. Representative XRPD patterns of rifaximin Form μ .

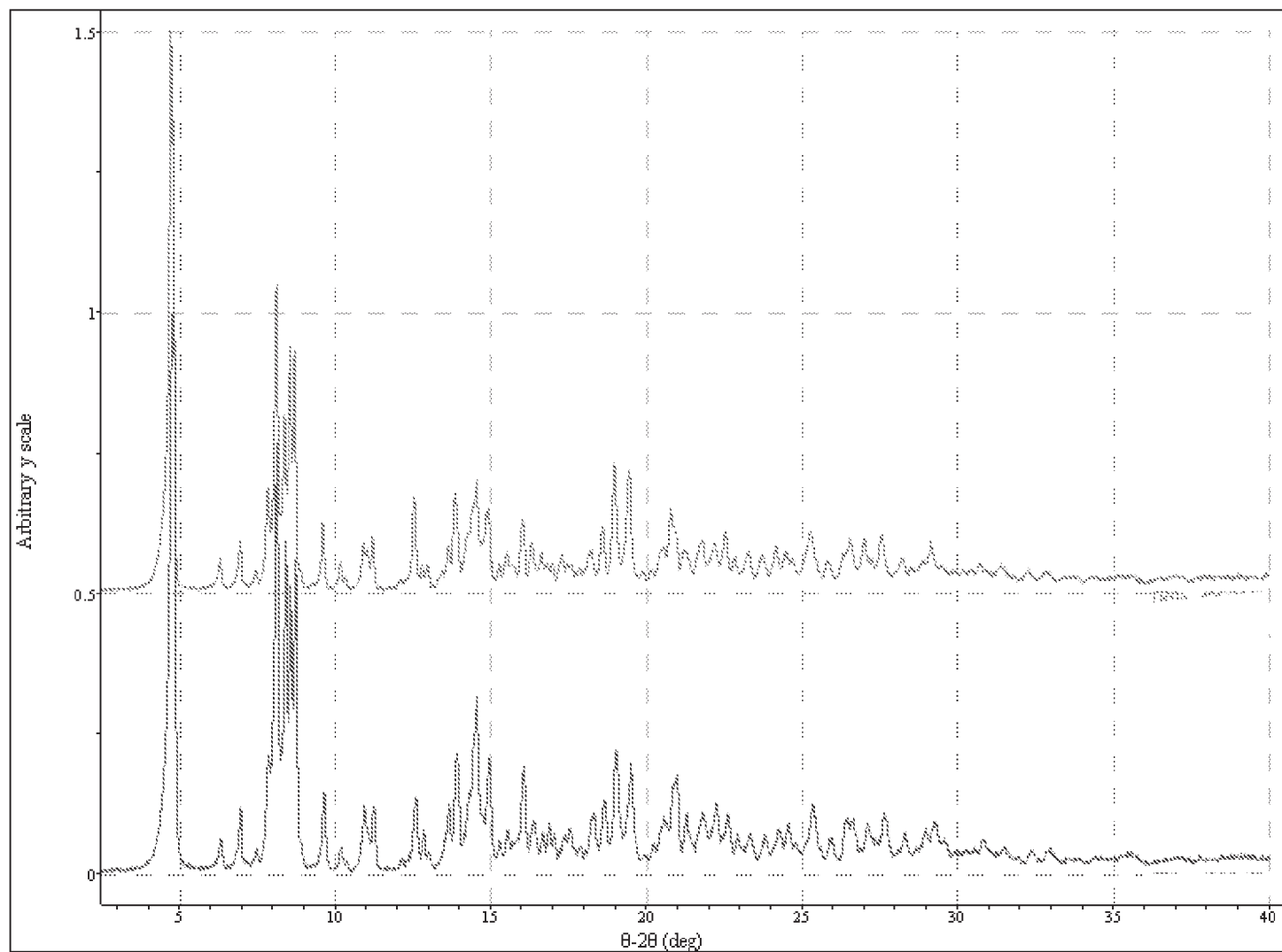


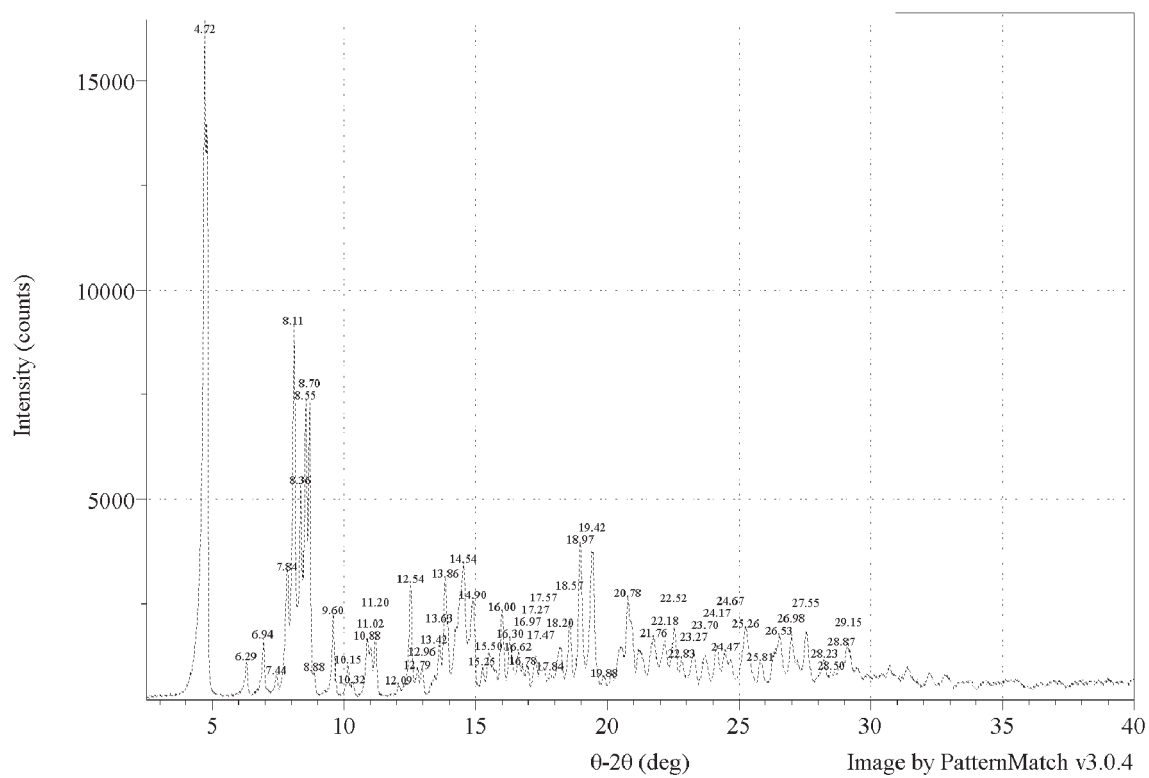
Figure 2. List of observed peaks for rifaximin Form μ .

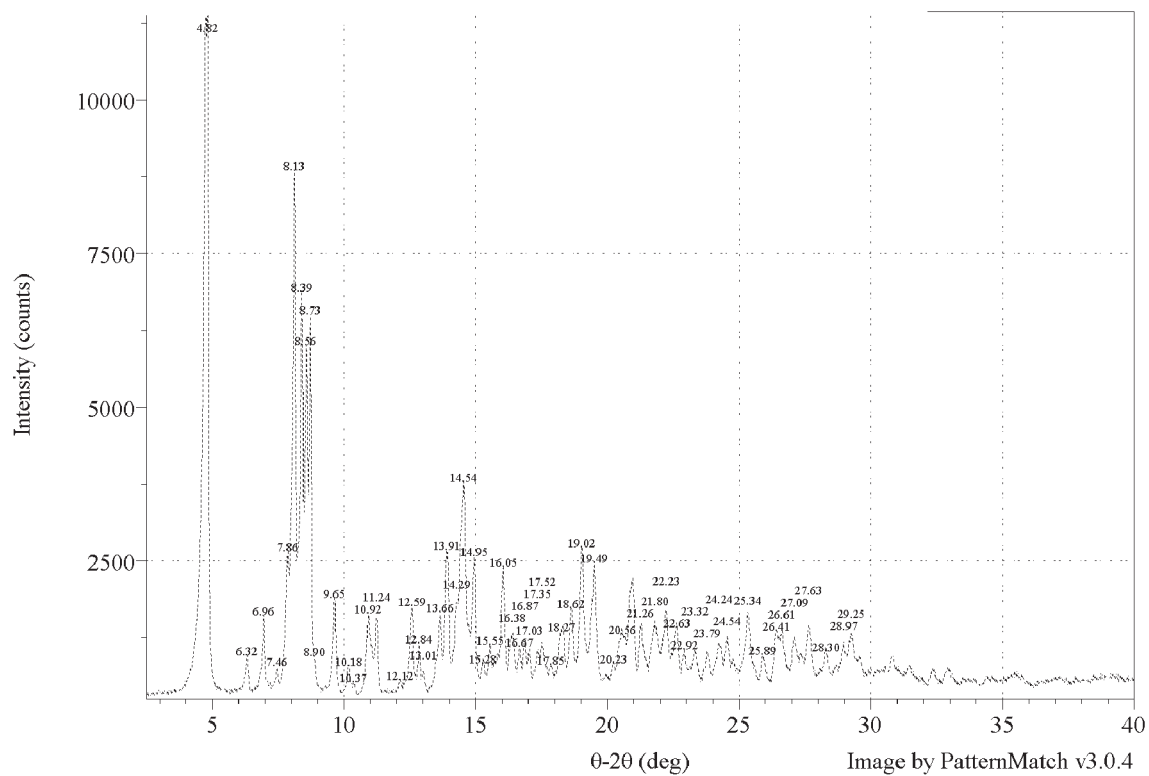
Figure 3. List of observed peaks for rifaximin Form μ .

Figure 4. Tentative indexing solution for rifaximin Form μ . bars indicate allowed reflections based on the unit cell dimensions and the assigned space group.

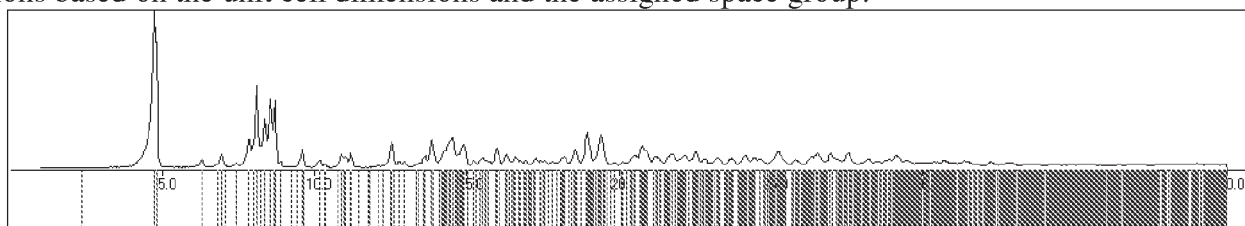


Figure 5. Tentative indexing solution for rifaximin Form μ . bars indicate allowed reflections based on the unit cell dimensions and the assigned space group ($P2_1$, #4).

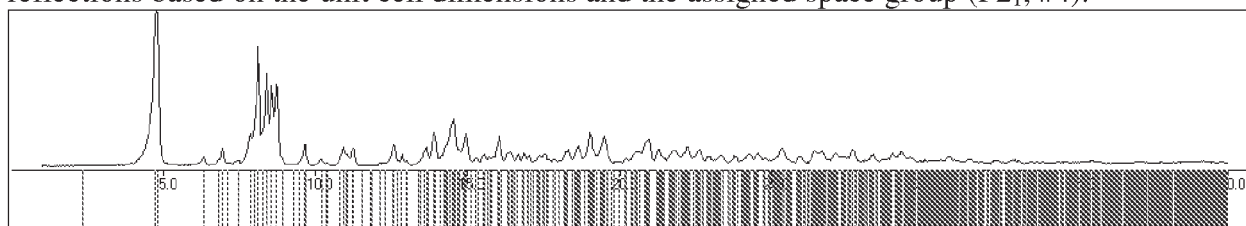


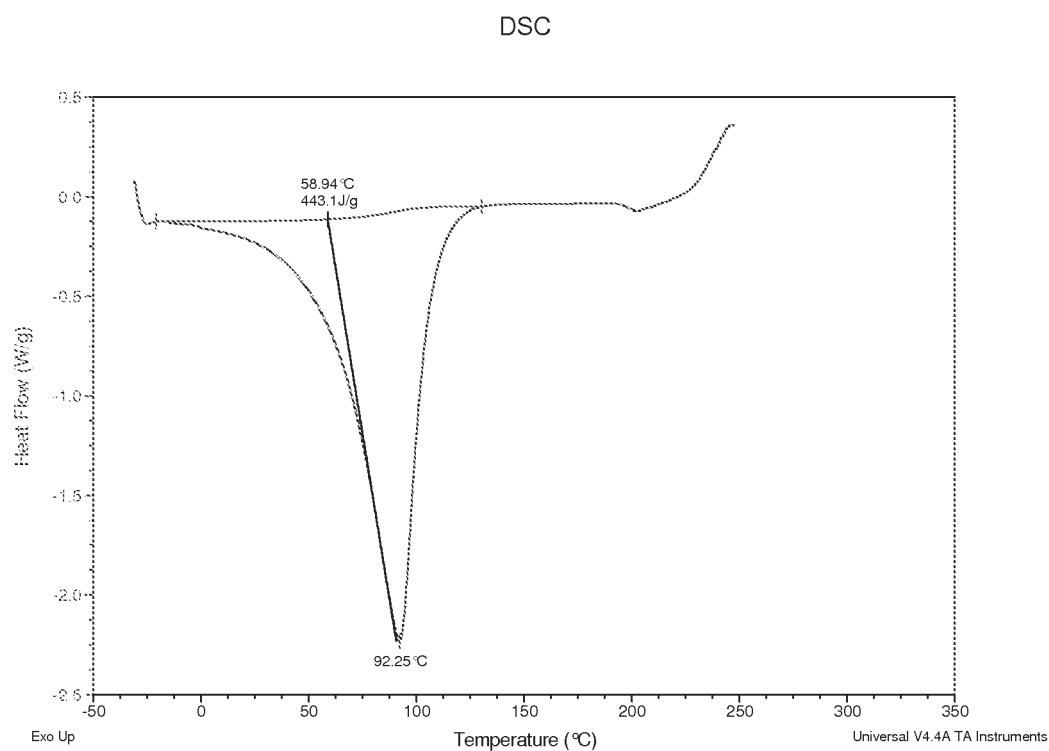
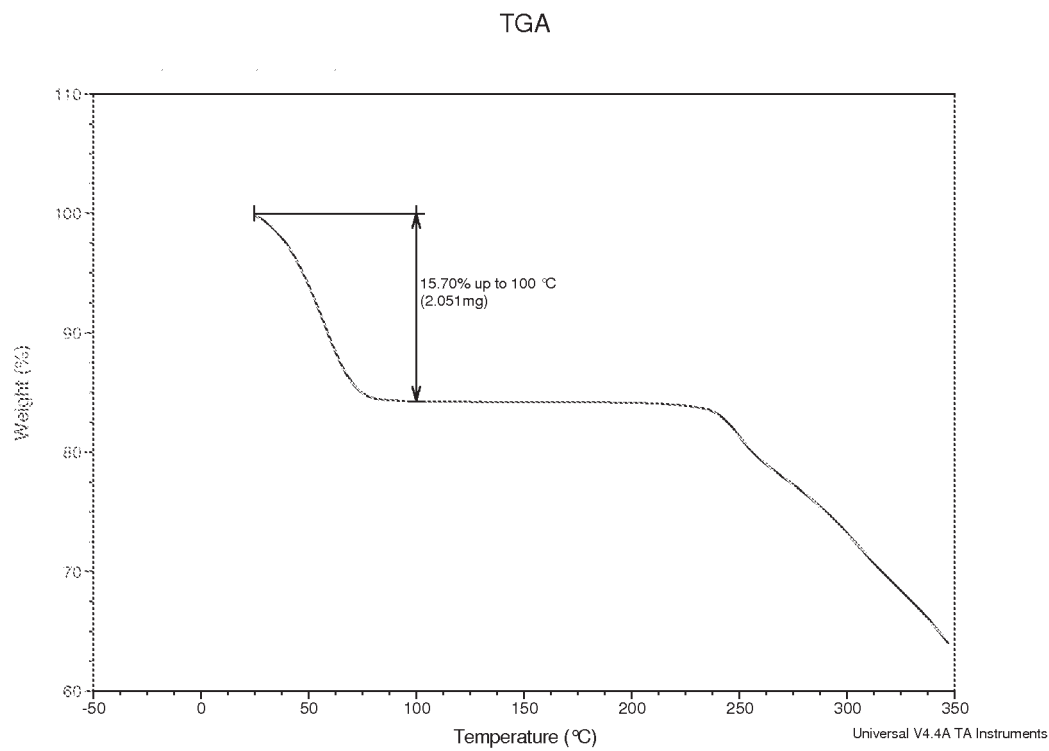
Figure 6. DSC and TGA thermograms of rifaximin Form μ .

Figure 7. Moisture sorption of rifaximin Form μ .

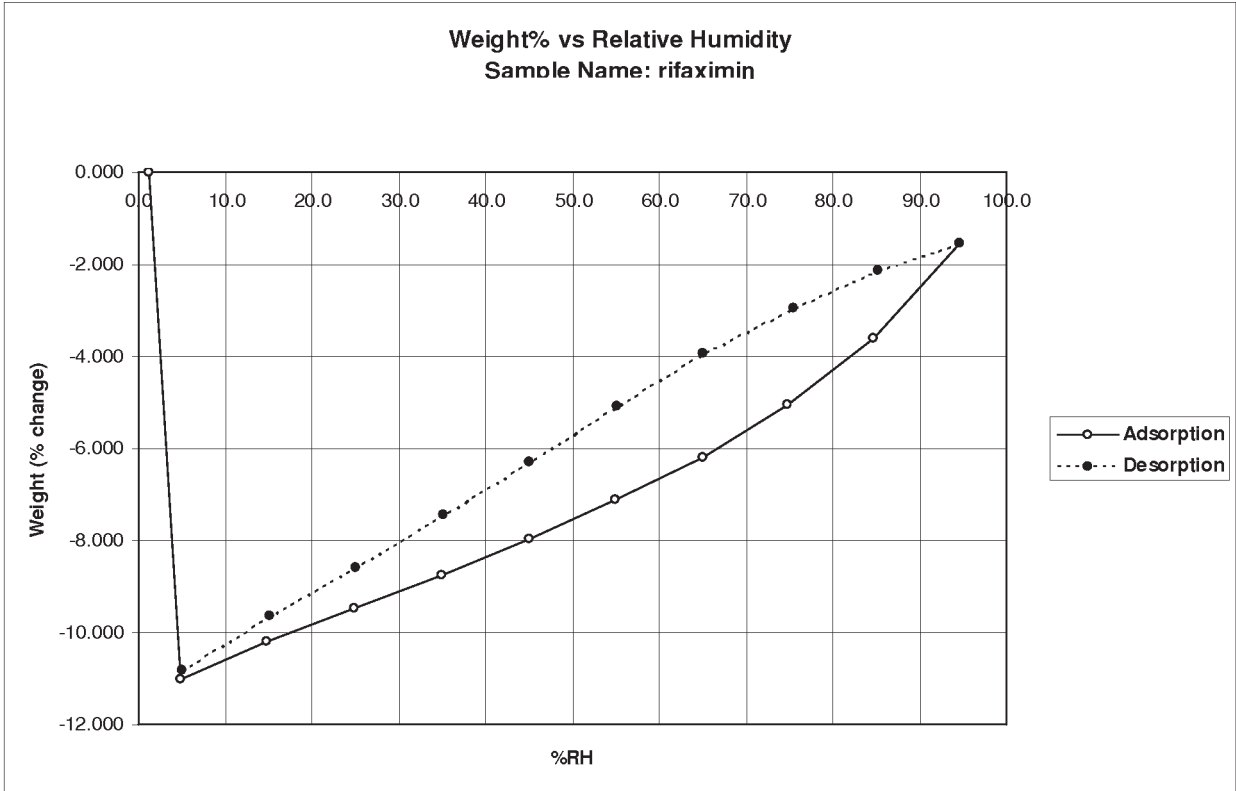
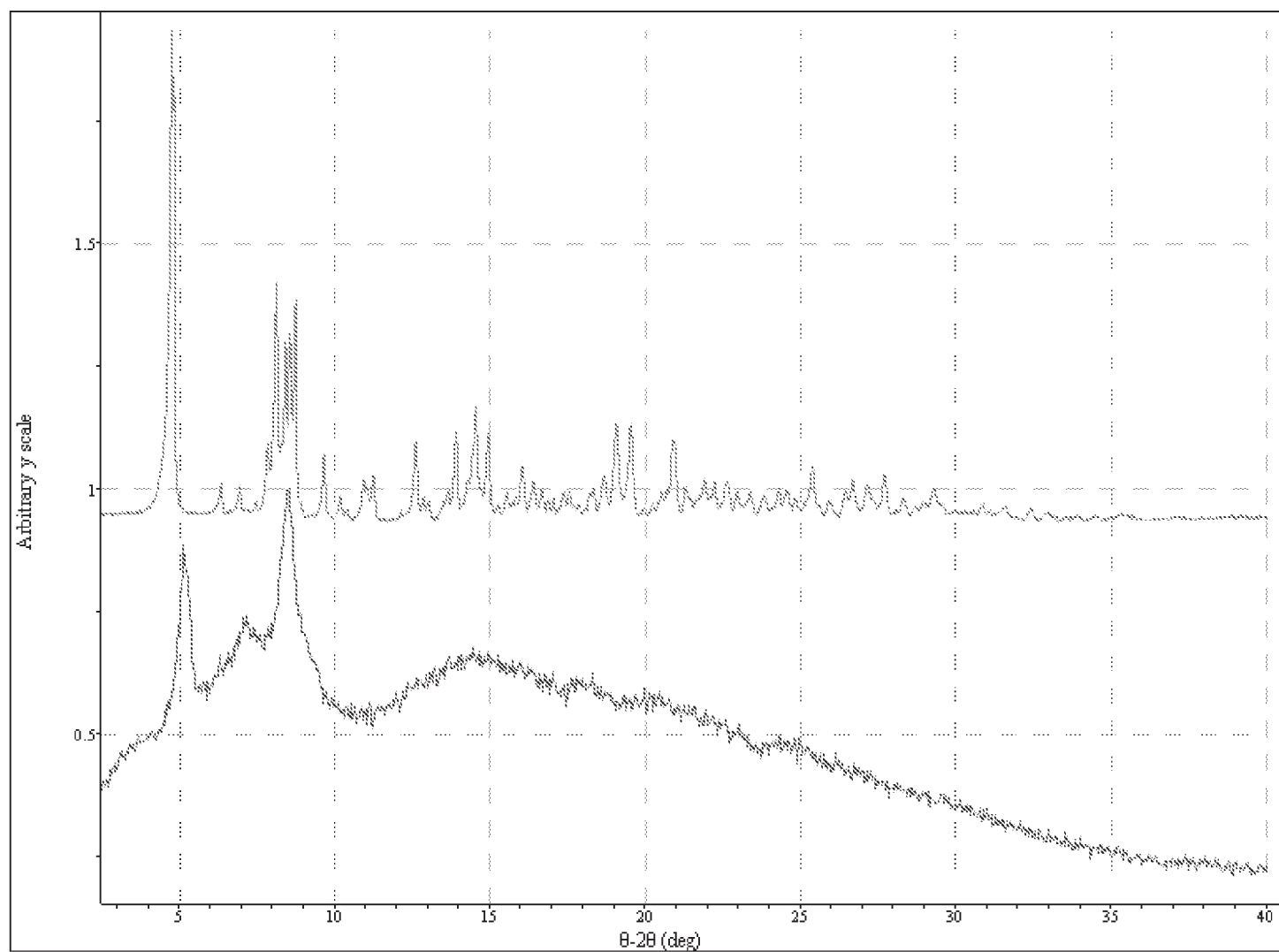


Figure 8. Post-Moisture sorption XRPD of rifaximin Form μ .
Top: before moisture sorption;
Bottom: after moisture sorption (Form γ).



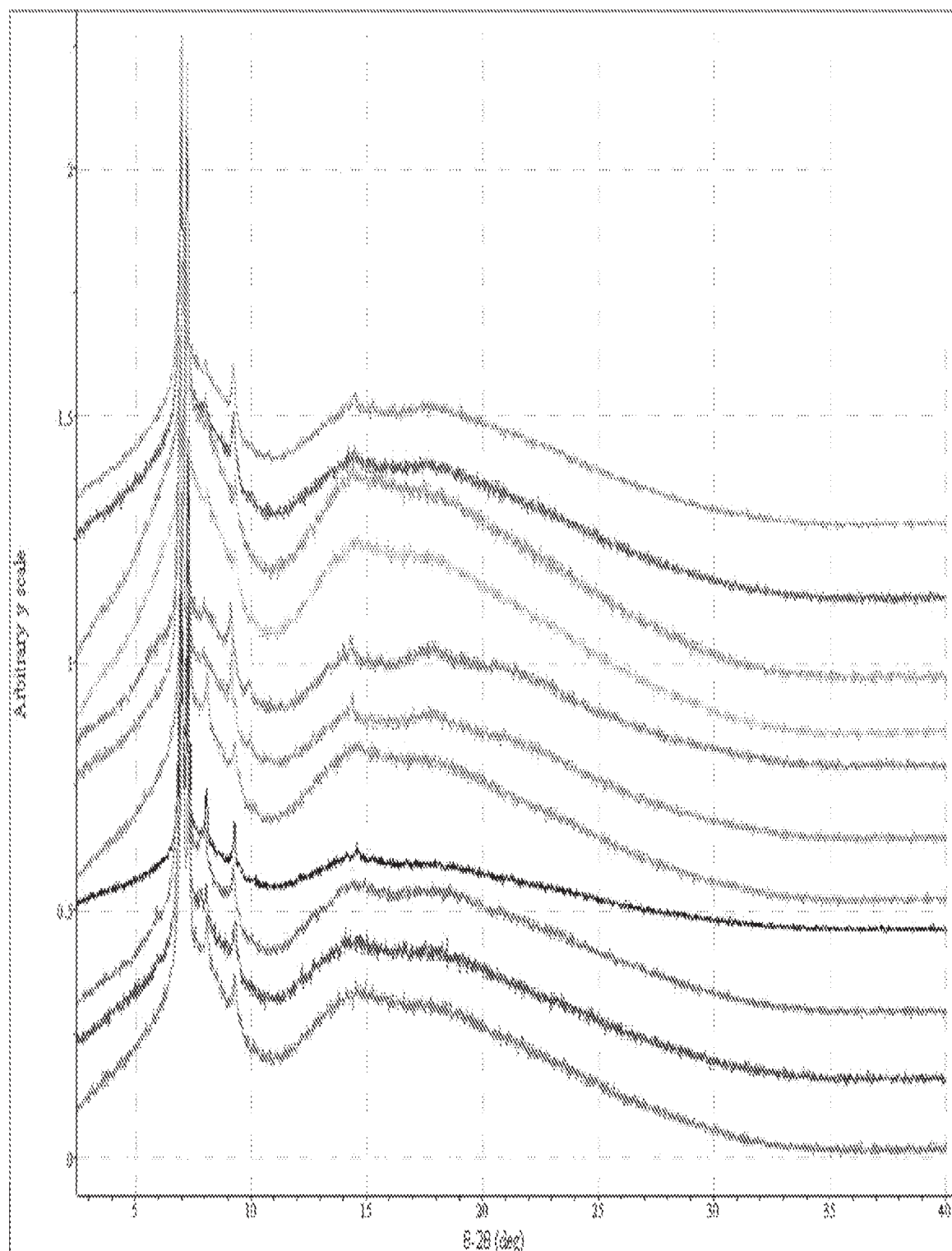


Figure 9

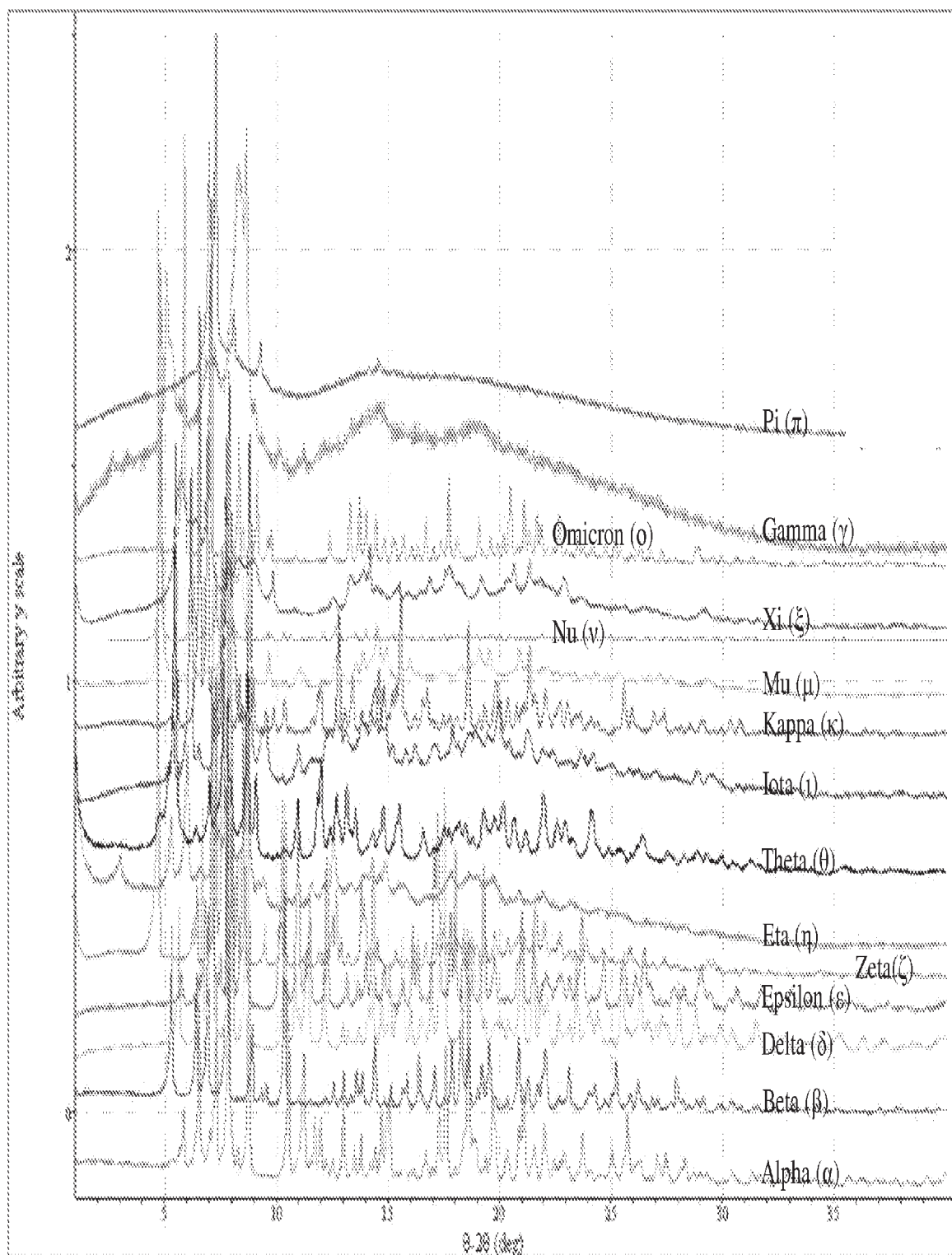


Figure 10

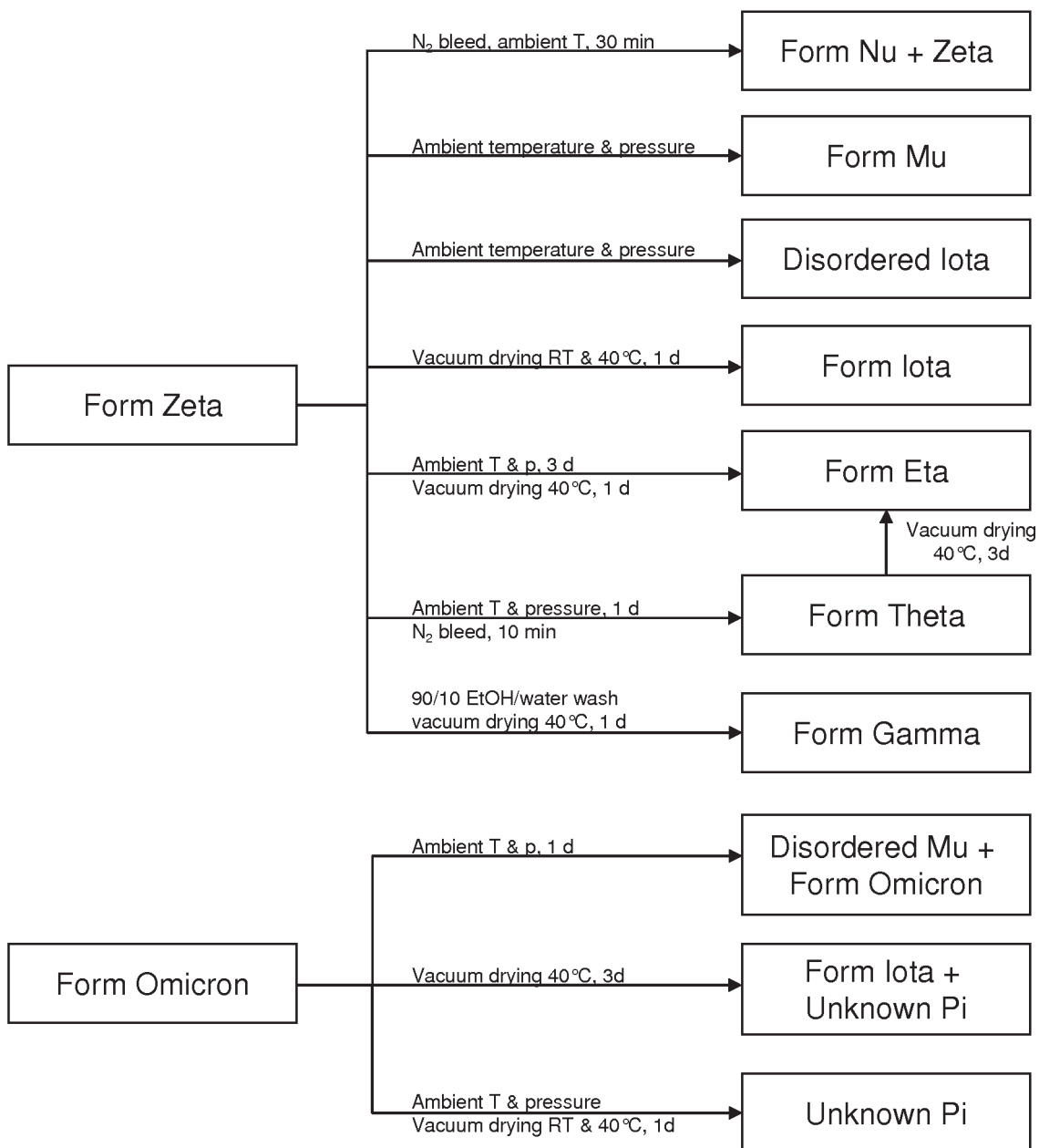
Figure 11 Summary of Results of Drying Experiments.

Figure 12: XRPD patterns of samples of Rifaximin Unknown Pi.

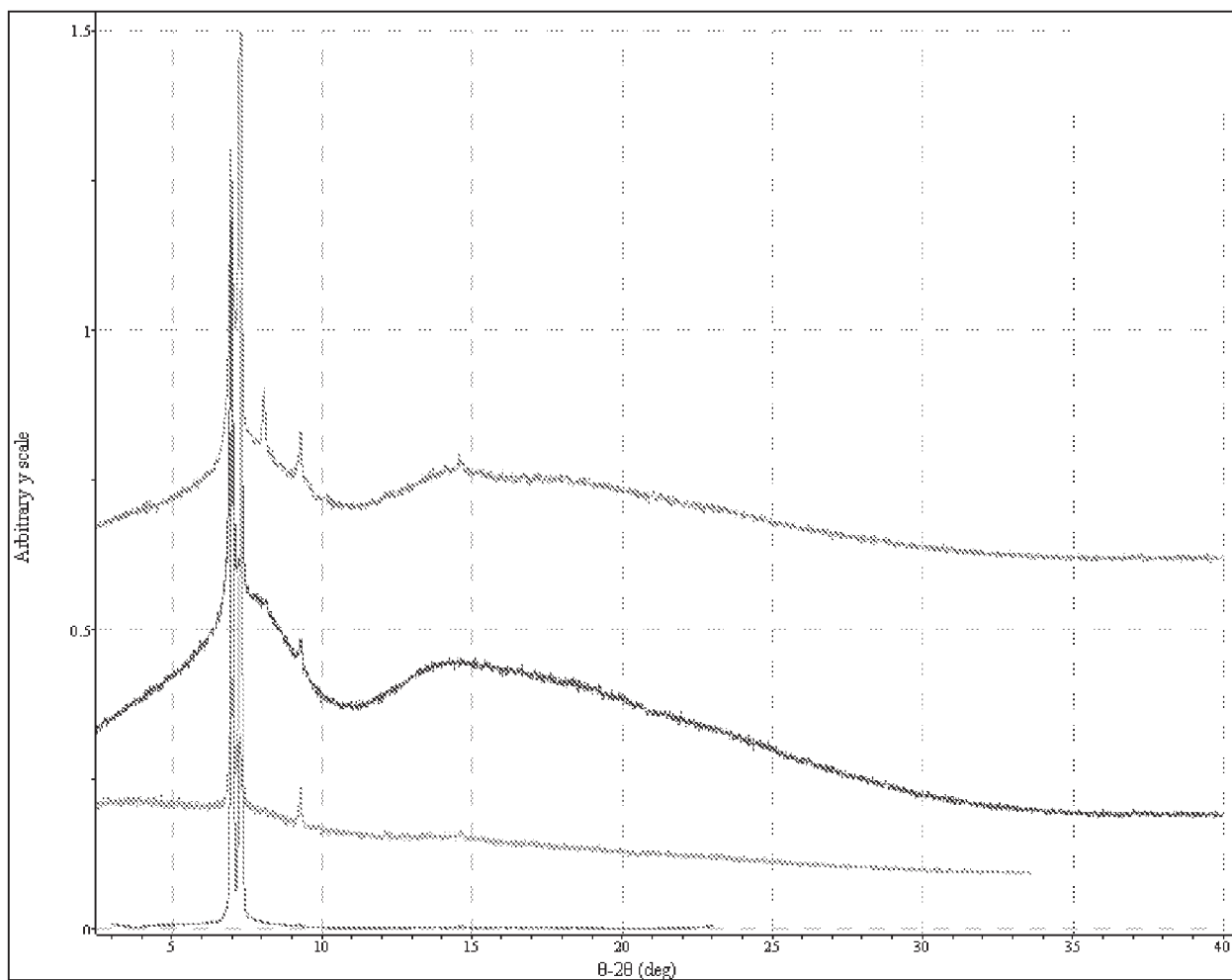


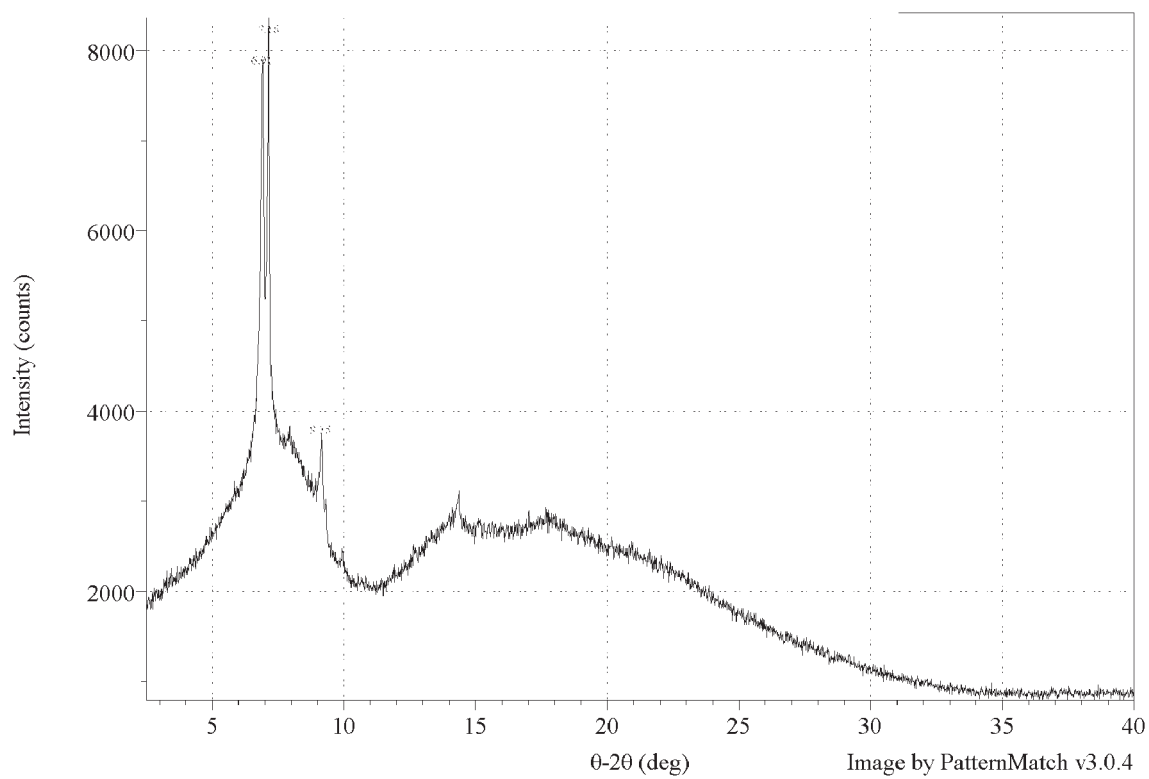
Figure 13. List of observed peaks for Rifaximin Pi,

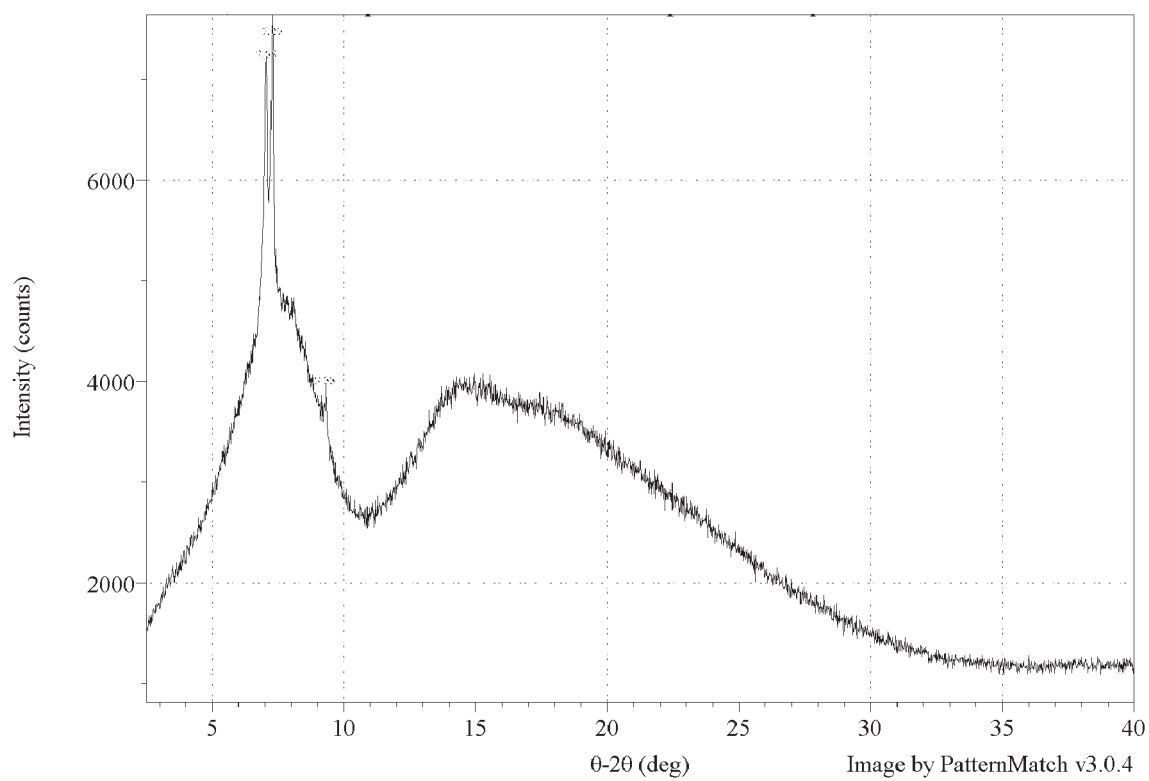
Figure 14. List of observed peaks for Rifaximin Pi,

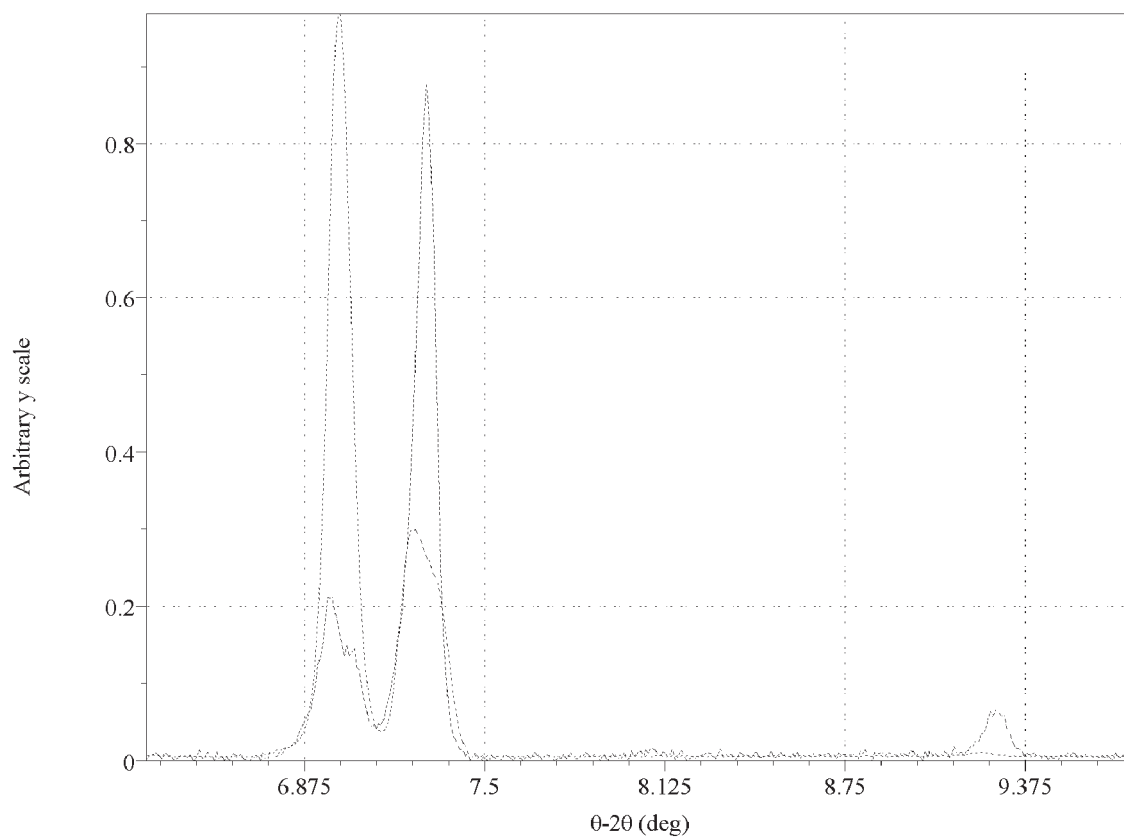
Figure 15. Comparison of XRPD patterns of Rifaximin

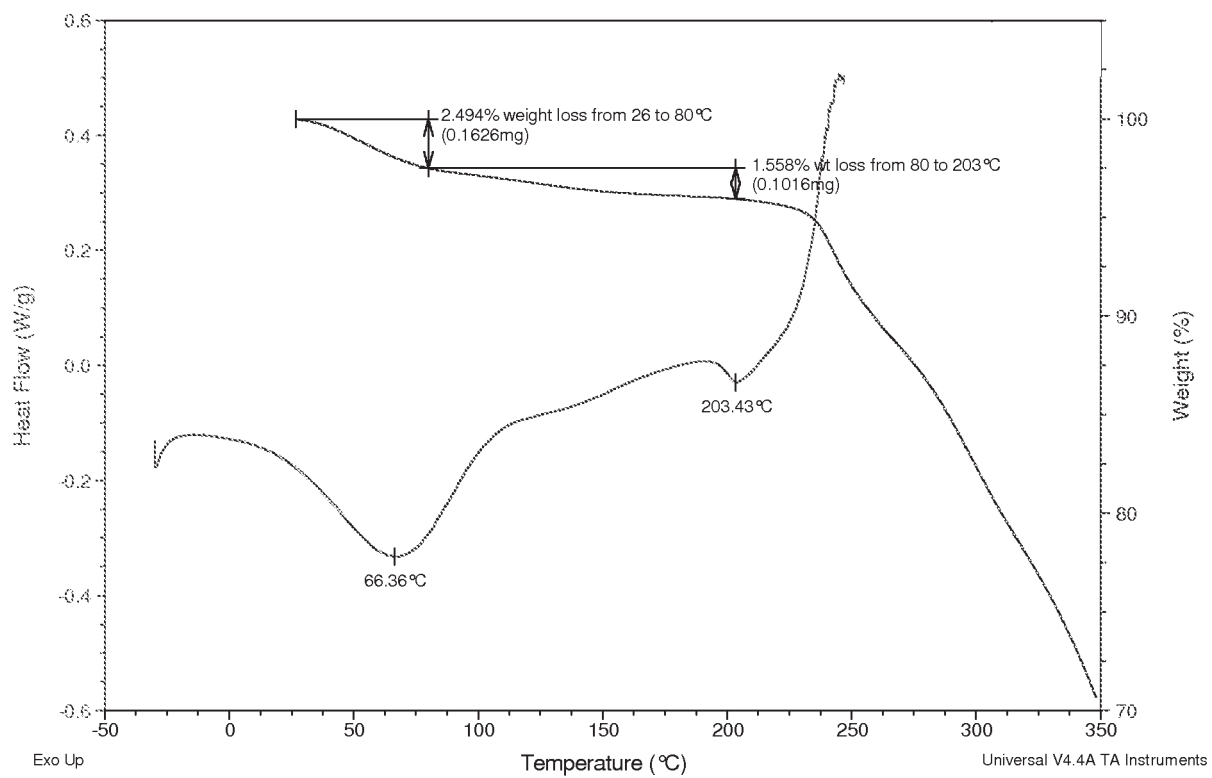
Figure 16. Thermal analysis of Rifaximin Pi

Figure 17. Dynamic vapor sorption isotherm of a sample of Rifaximin Pi

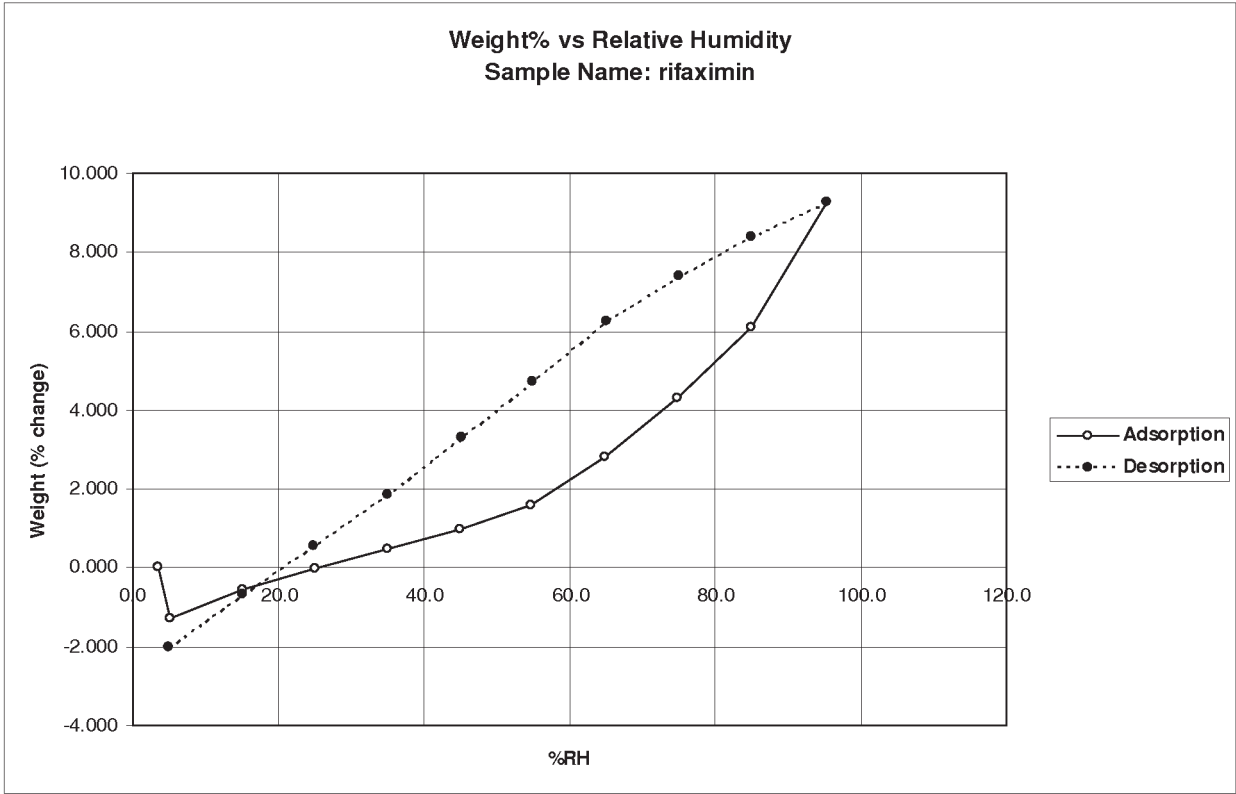


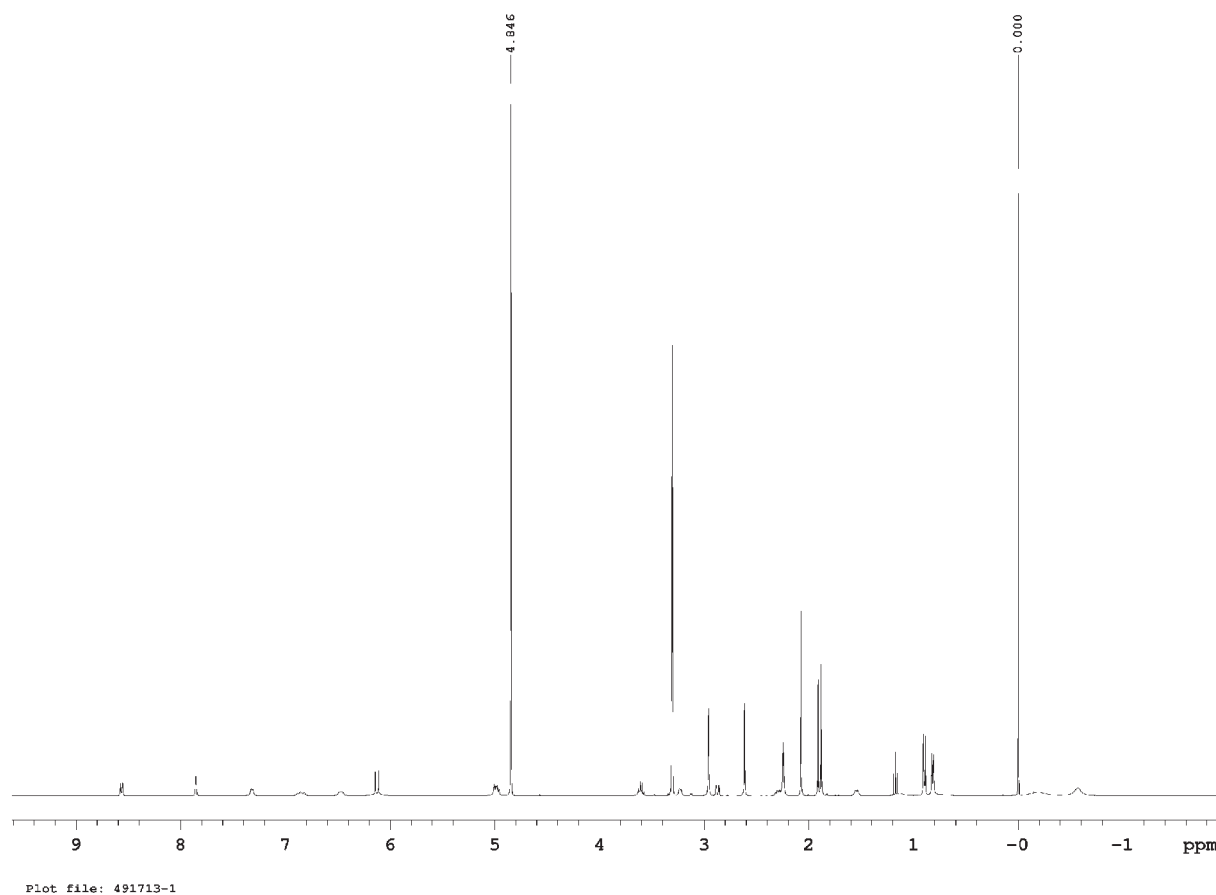
Figure 18. Proton NMR spectrum of Rifaximin Pi

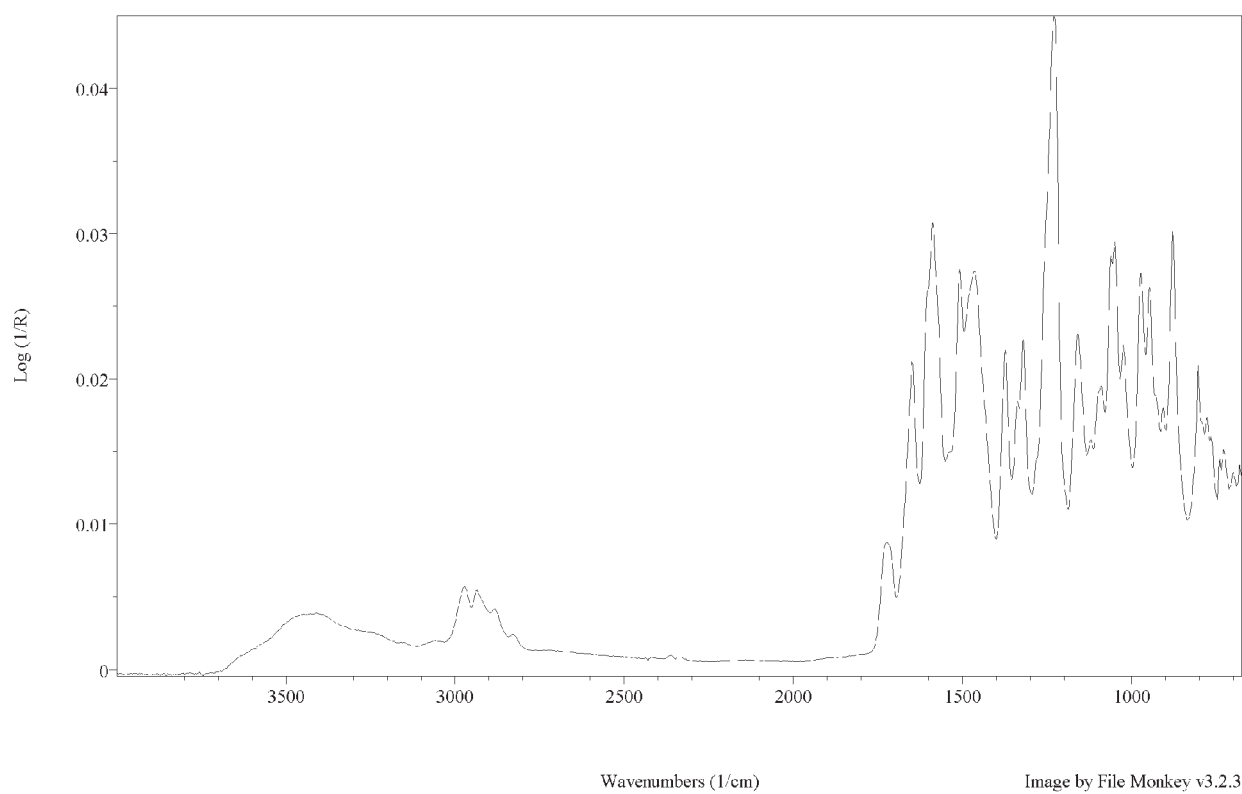
Figure 19. ATR-IR spectrum of Rifaximin Pi

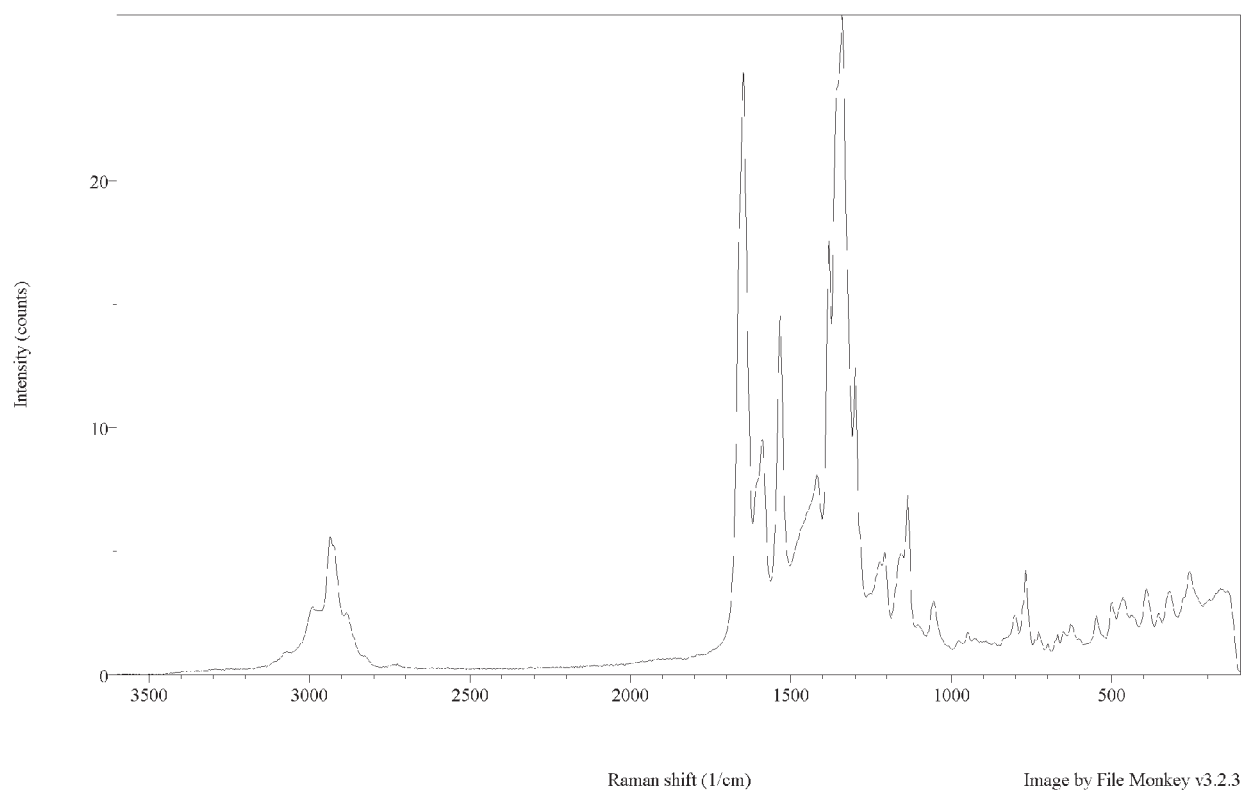
Figure 20. Raman spectrum of Rifaximin Pi

Figure 21. Solid state carbon NMR spectrum of Rifaximin D_2O

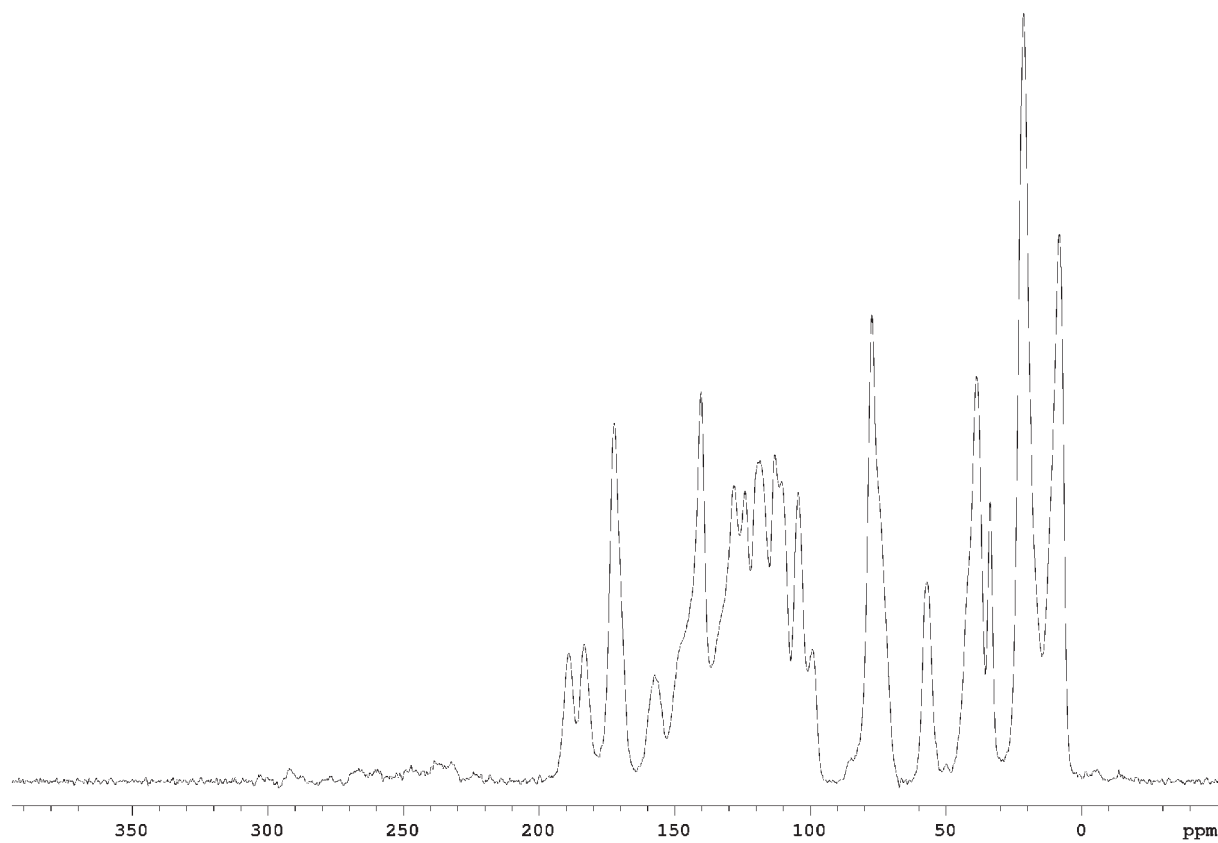


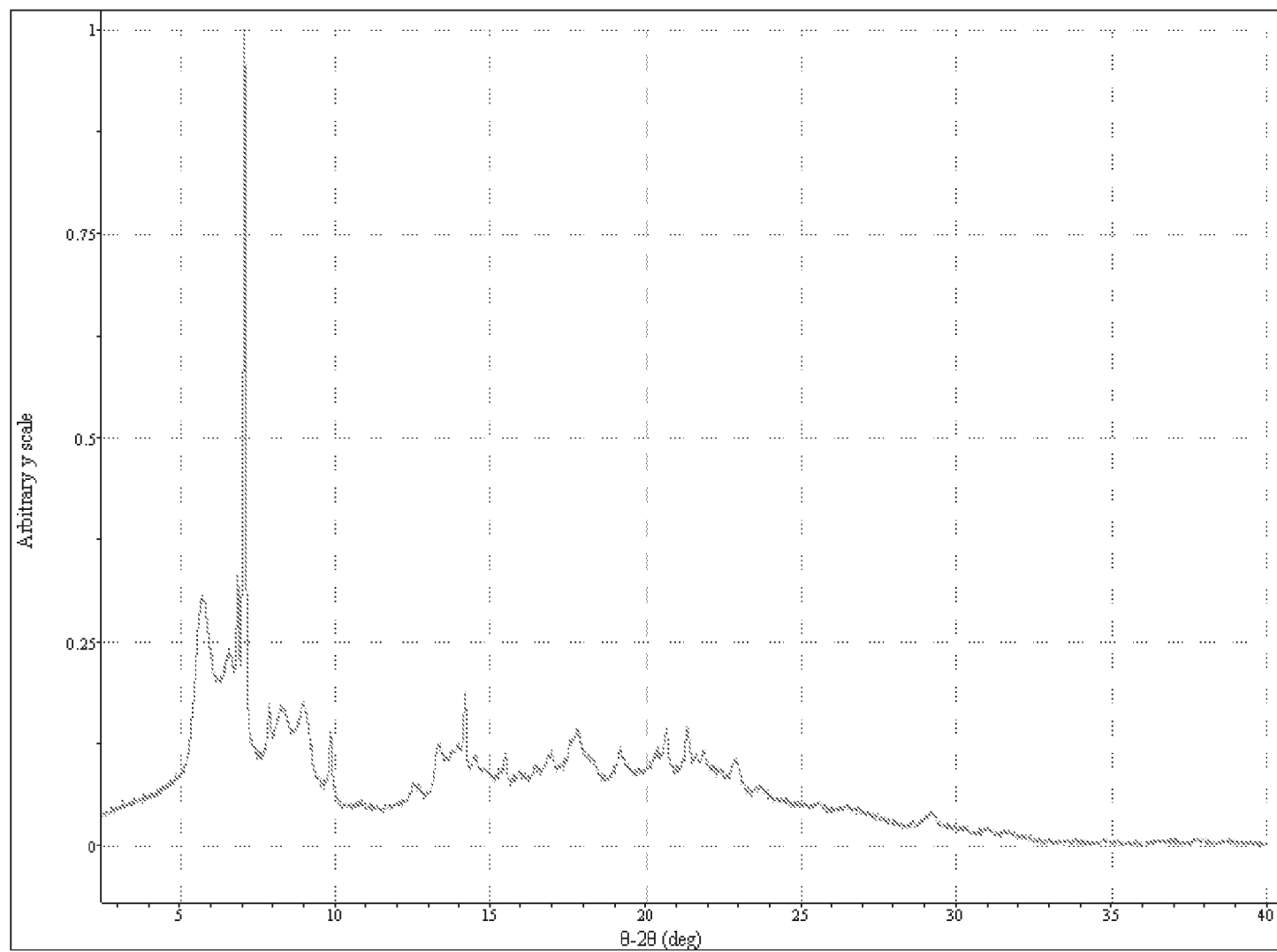
Figure 22 XRPD Pattern of Rifaximin Form Xi (ξ).

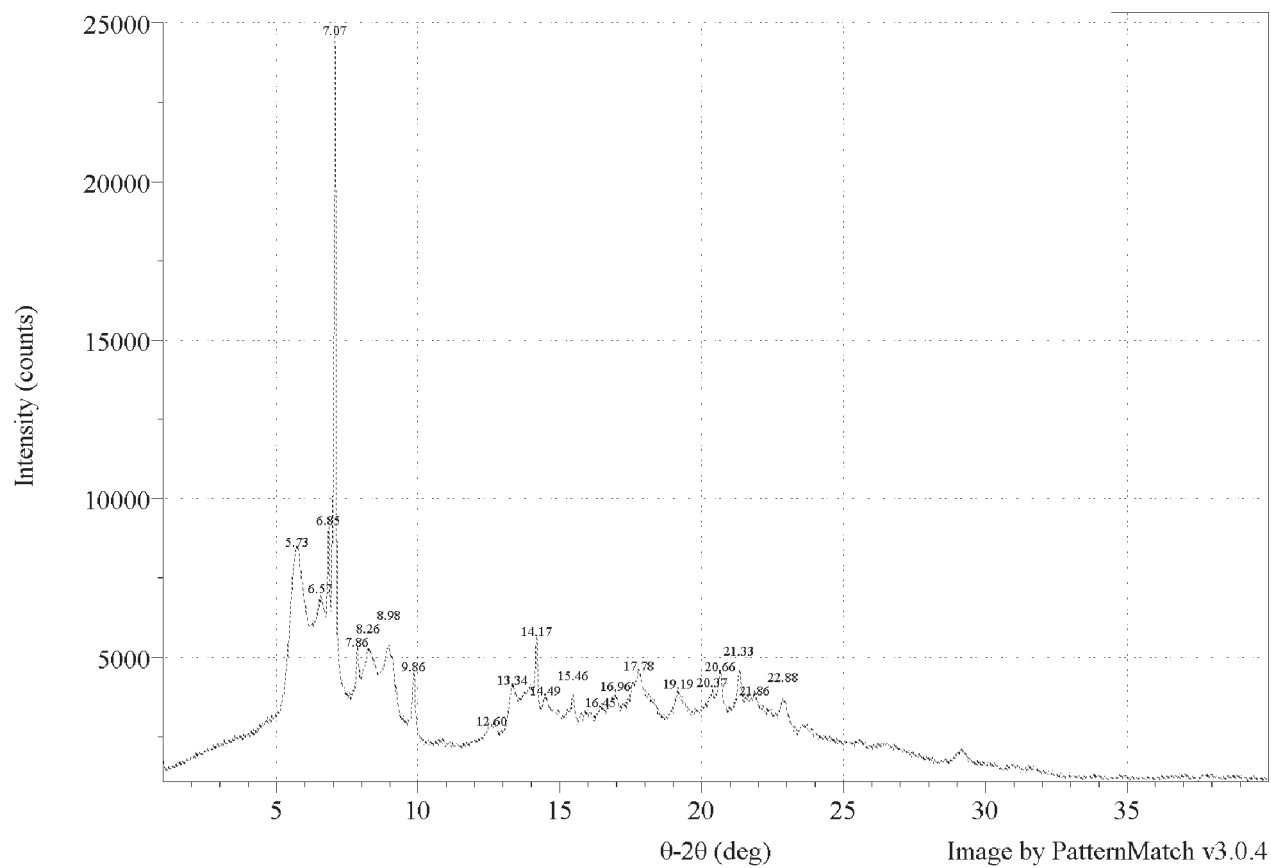
Figure 23 List of Observed Peaks for Rifaximin Form Xi (ξ)

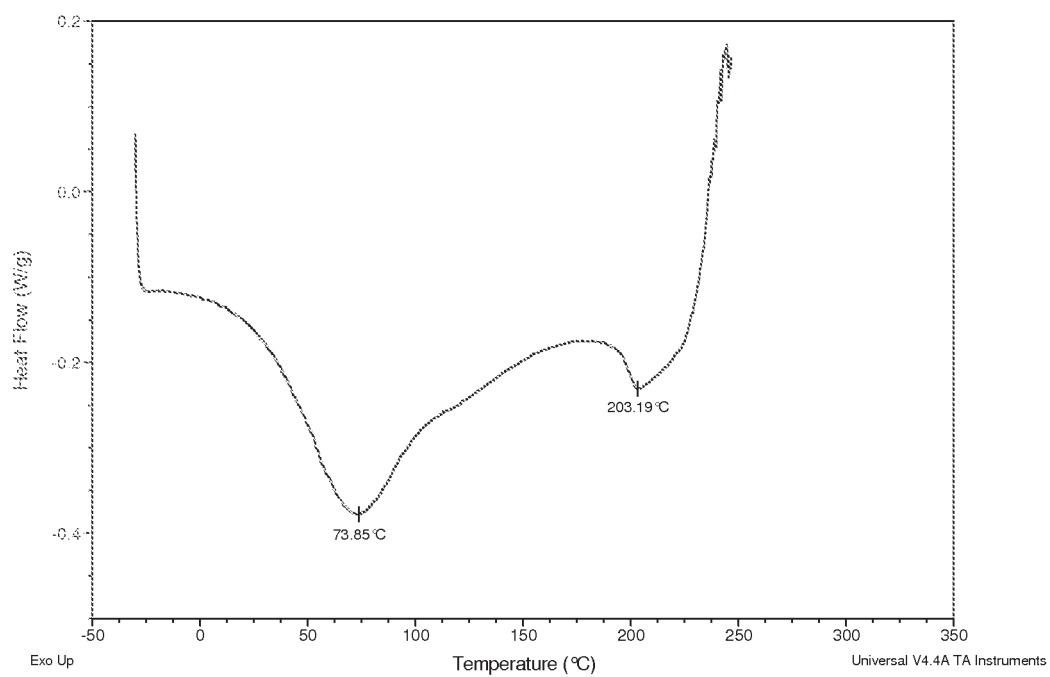
Figure 24 DSC Thermogram of Rifaximin Form Xi (ξ).

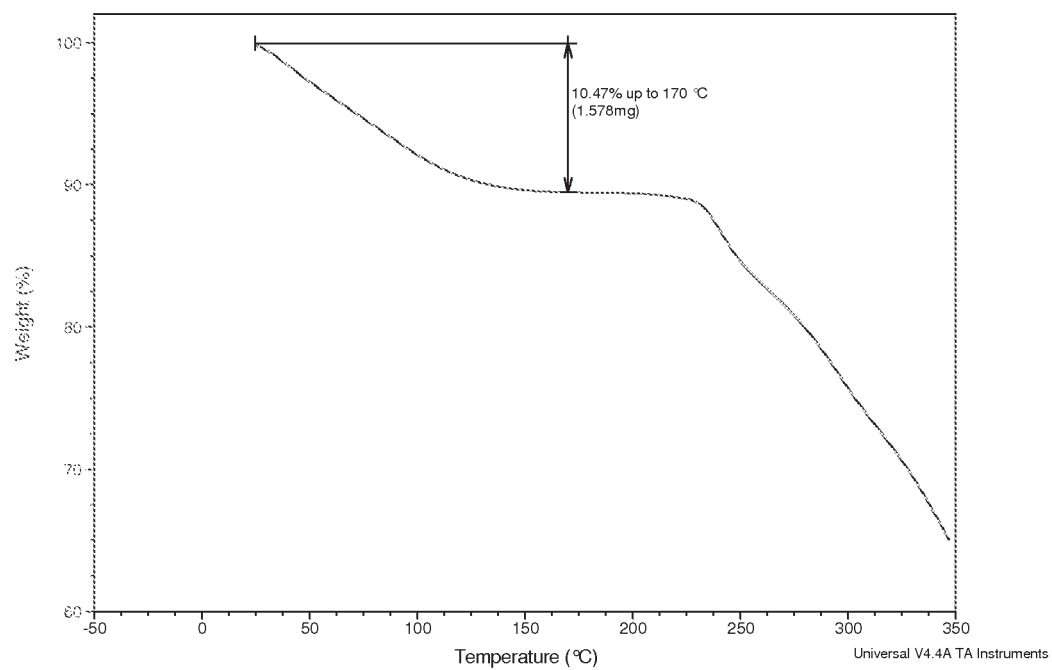
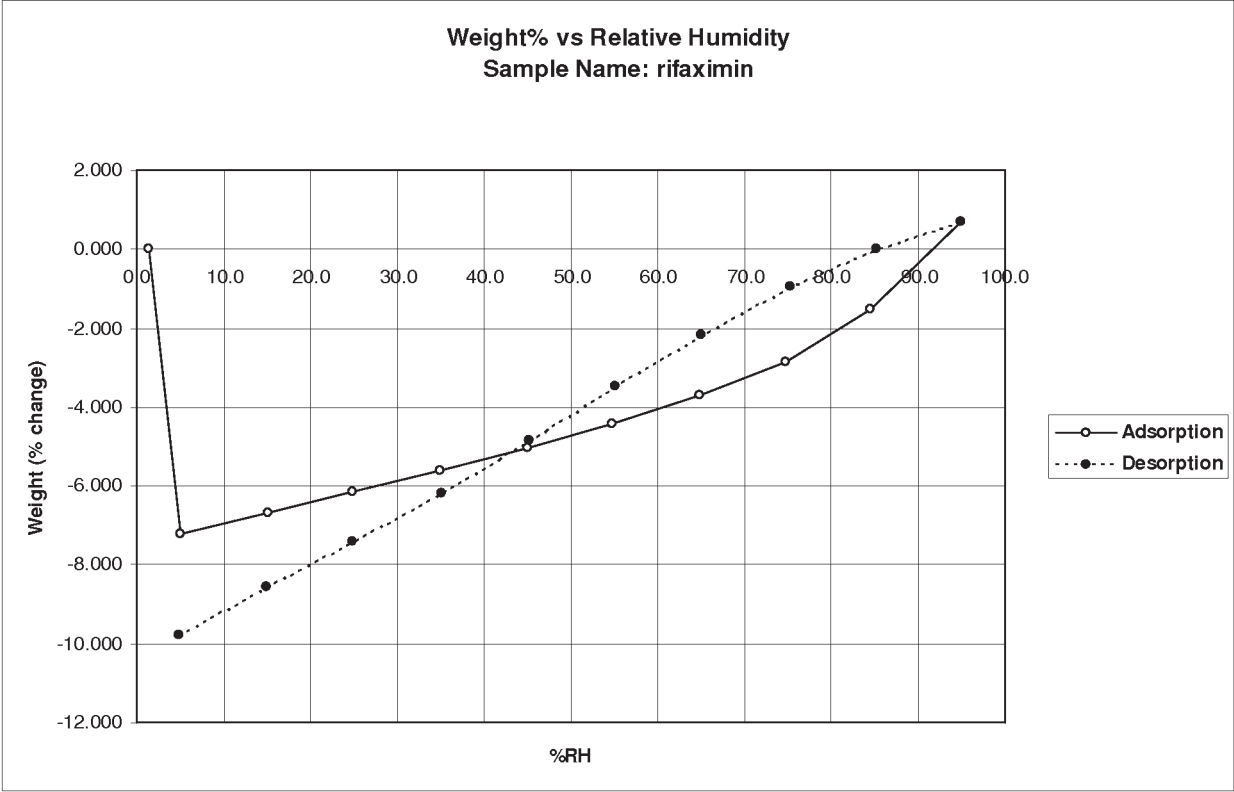
Figure 25 TGA Thermogram of Rifaximin Form Xi (ξ).

Figure 26 Moisture Sorption Isotherm of Rifaximin Form Xi (ξ).



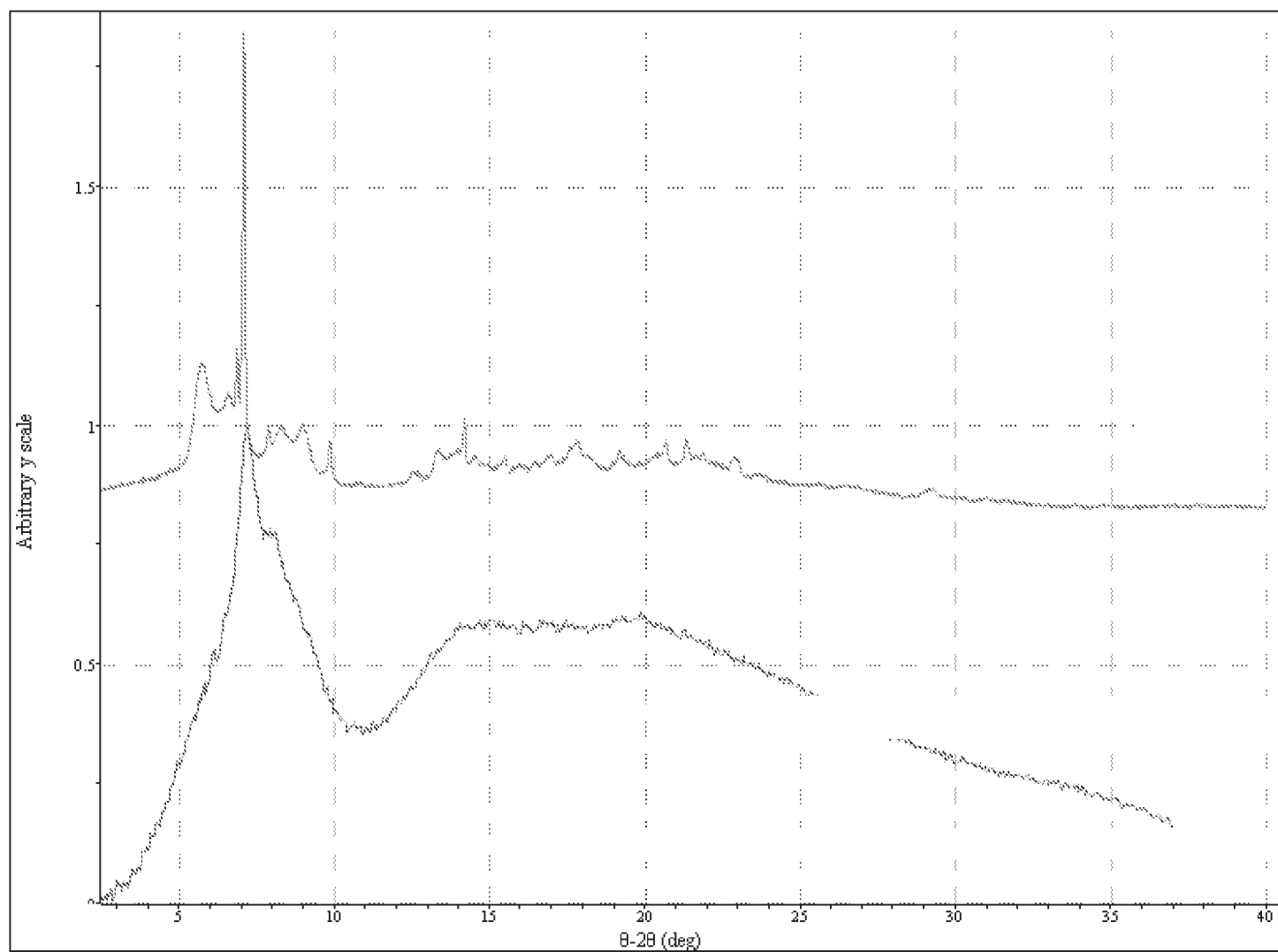


Figure 27 XRPD pattern of Rifaximin Form Xi (ξ) Before and After the Moisture Sorption Experiment

Figure 28

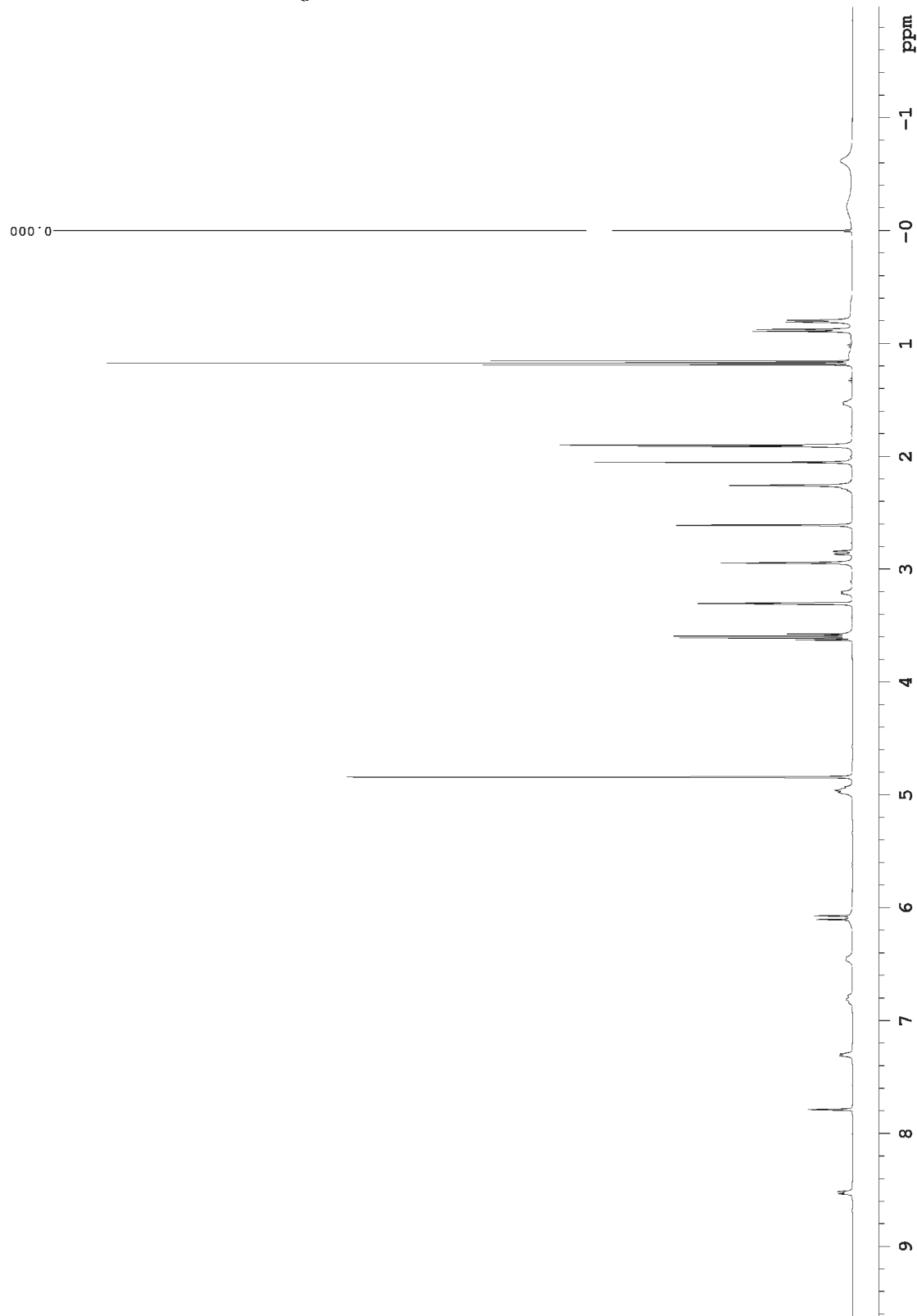


Figure 29 Solid State NMR Spectrum of Rifaximin Form Xi(ξ).

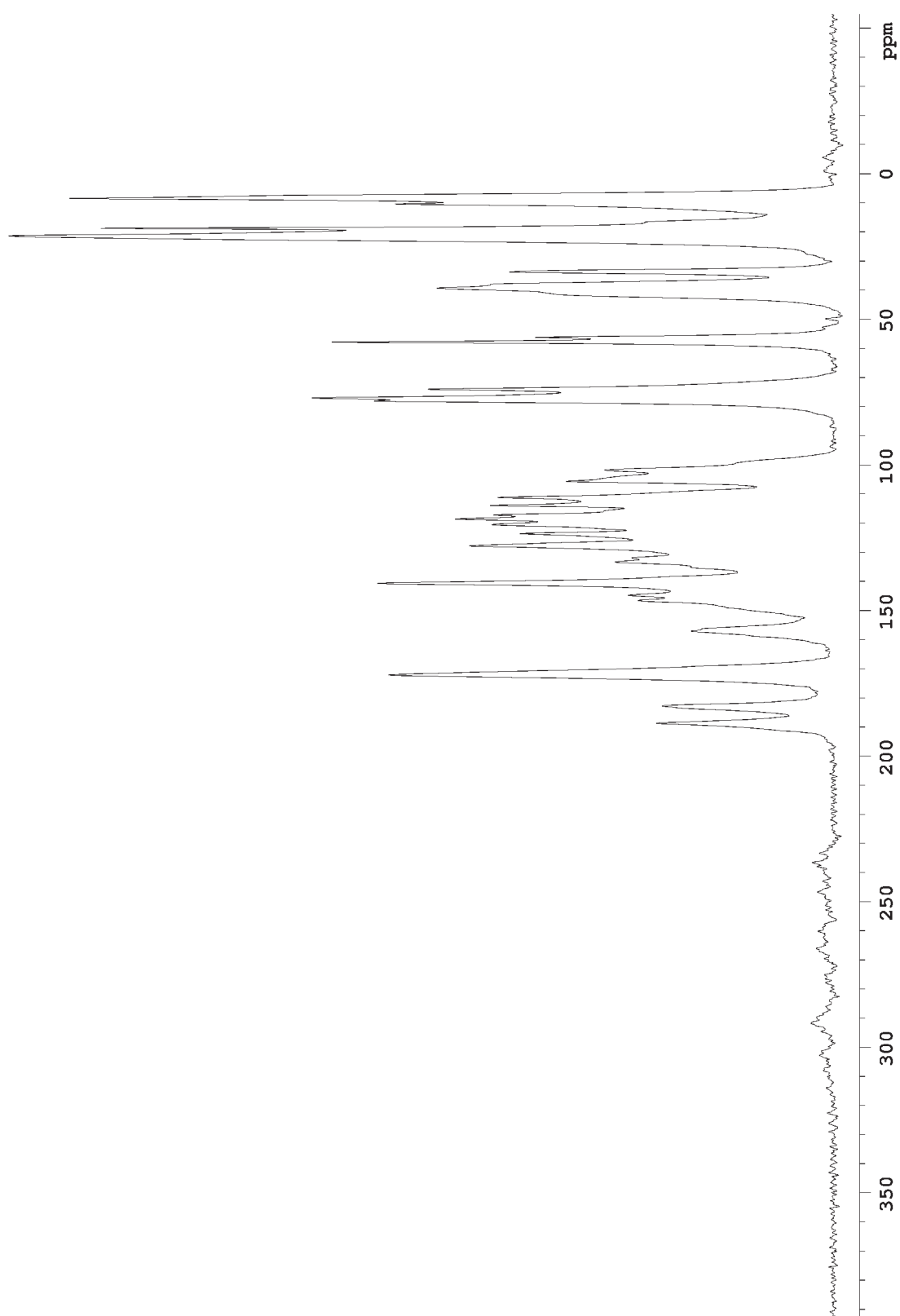


Figure 30 Infrared (IR) Spectrum of Rifaximin Form Xi(ξ).

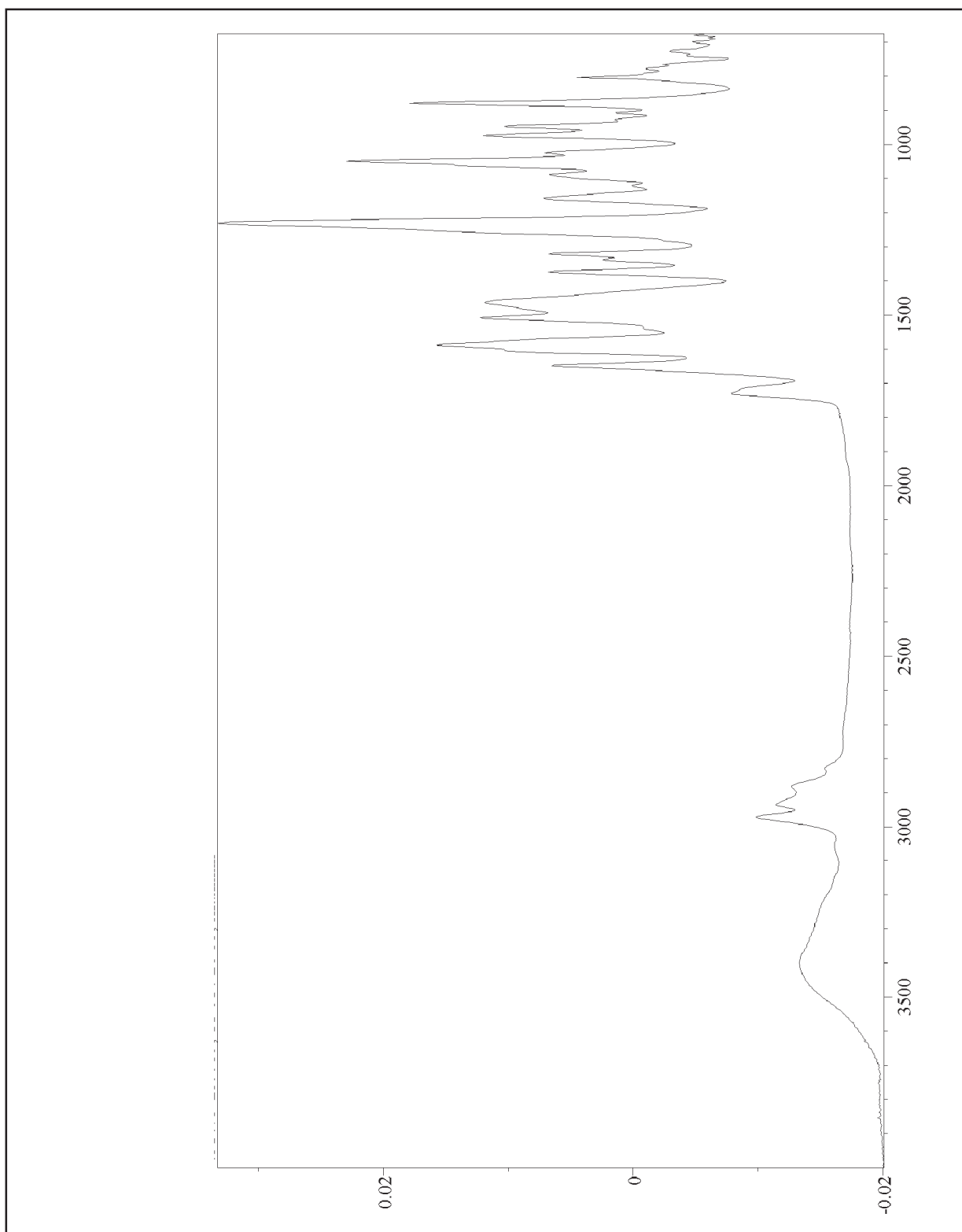


Figure 31 Raman Spectrum of Rifaximin Form Xi(ξ).

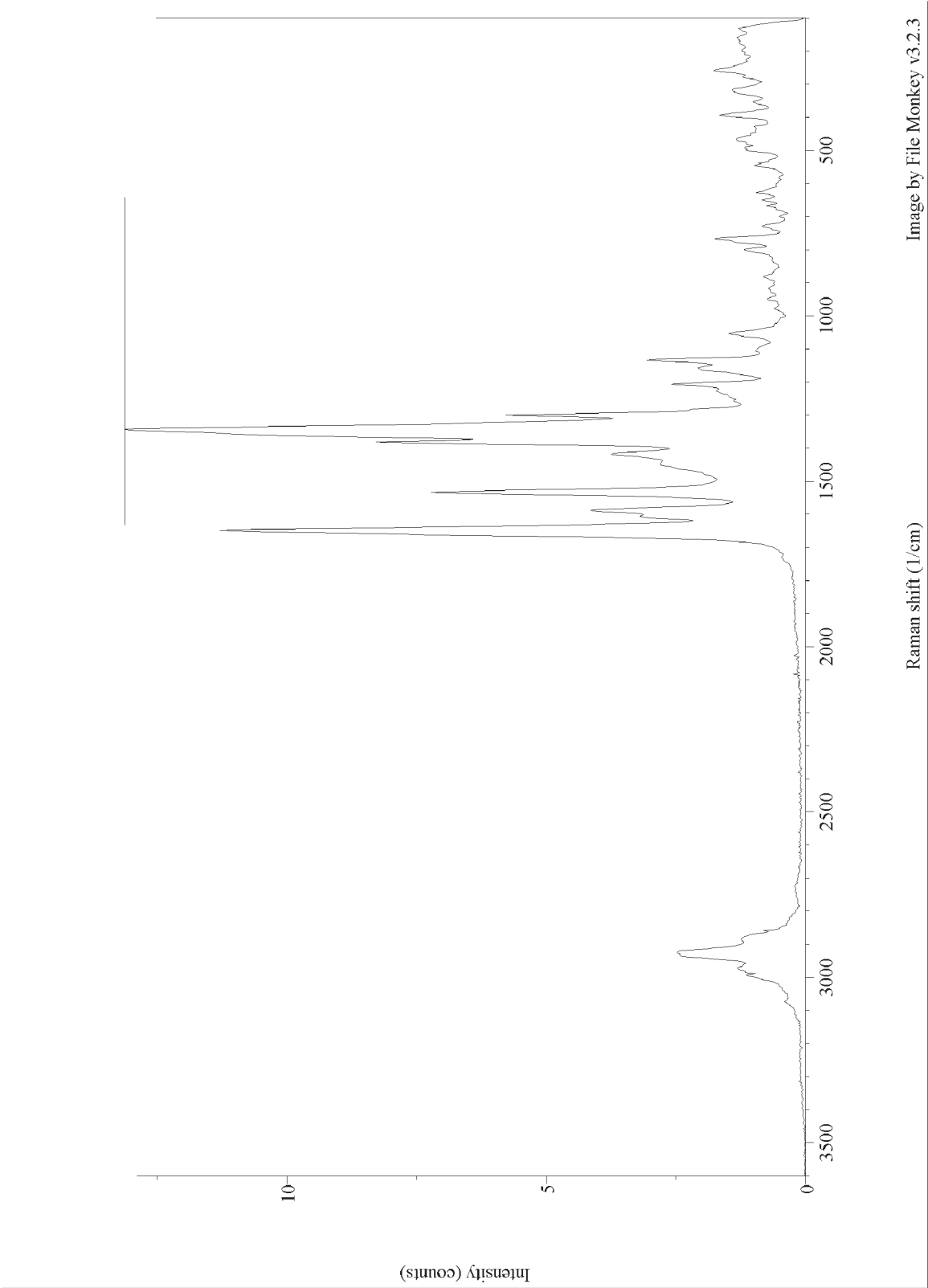


Figure 32

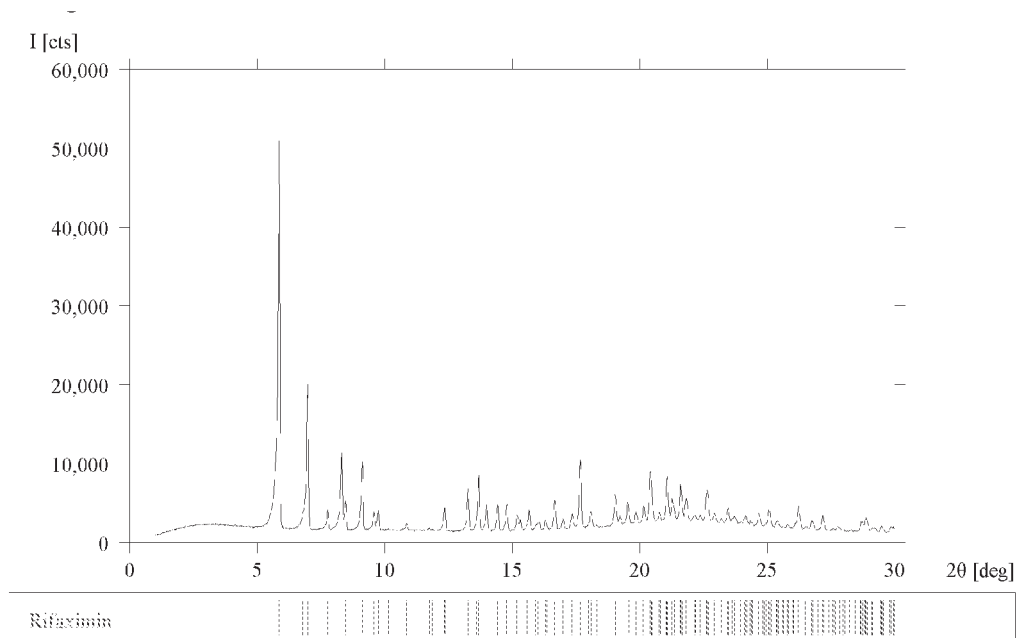


Figure 33. Indexed unit cell parameters of Rifaximin

	Rifaximin
Bravais Type	Primitive Orthorhombic
a [Å]	12.636
b [Å]	18.461
c [Å]	26.003
α [deg]	90
β [deg]	90
γ [deg]	90
Volume [Å ³ /cell]	6,065.7
Chiral Contents?	Chiral
Extinction Symbol	P - 21 21
Space Group(s)	P22 ₁ 2 ₁ (18)*
Source	Triads Algorithm

* = Unconventional space group setting.

[illegible]

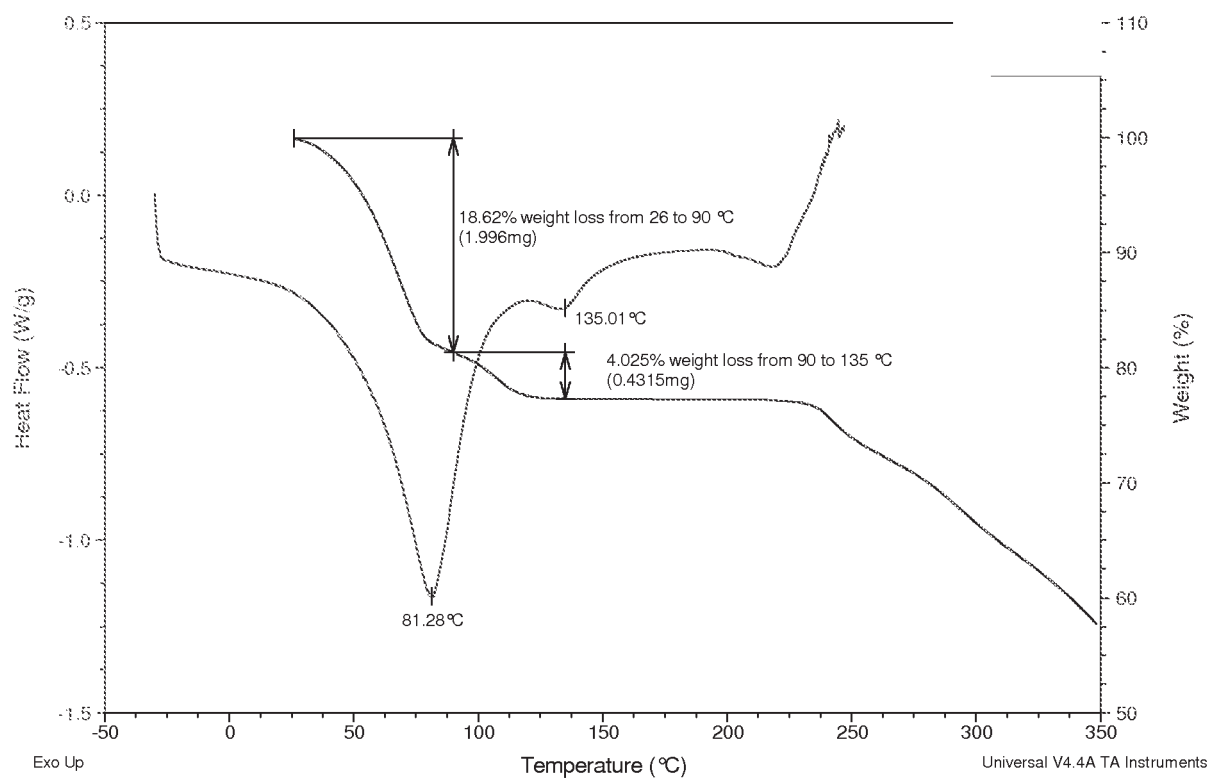
Figure 35. Thermal analysis of Rifaximin Form Omicron,

Figure 36. Dynamic vapor sorption isotherm of a sample of Rifaximin Form Omicron

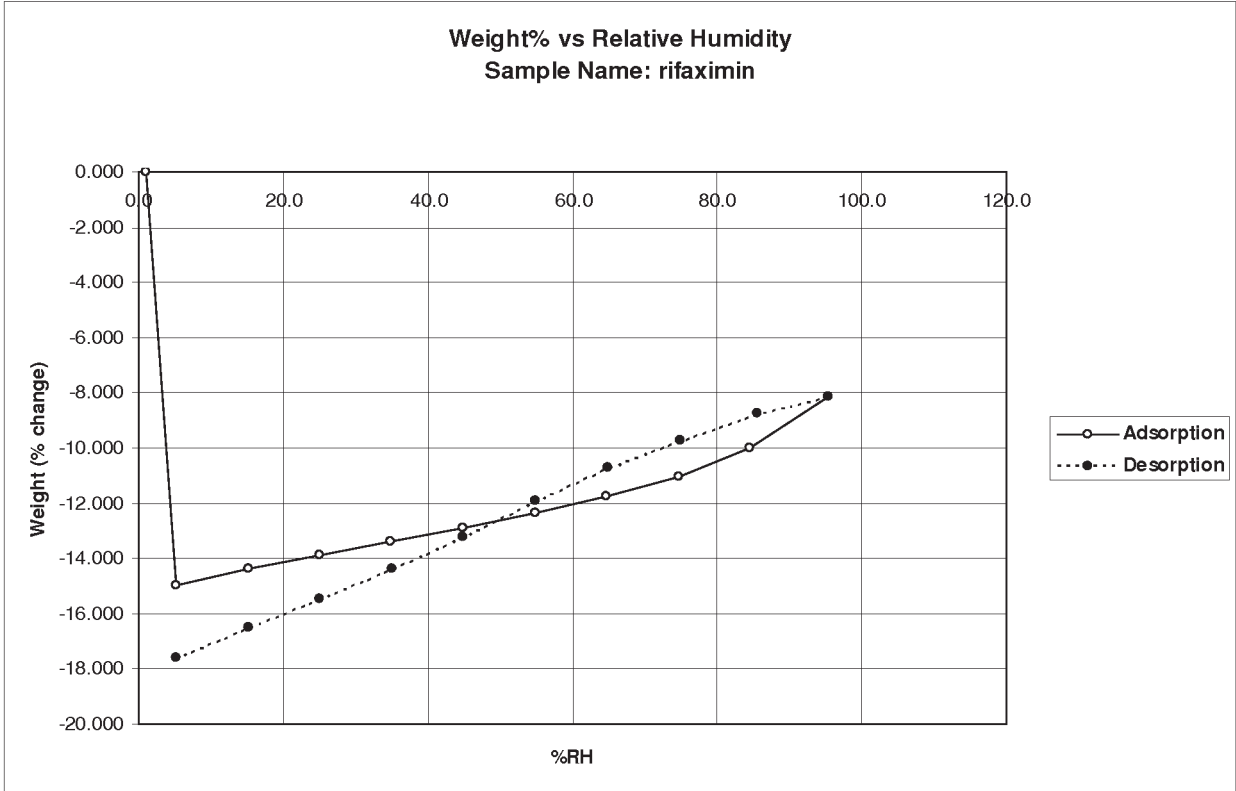


Figure 37. Comparison of XRPD patterns of Rifaximin Form Omicron and the post-DVS sample, Form Iota.

Top: Form Iota

Bottom: Form Omicron

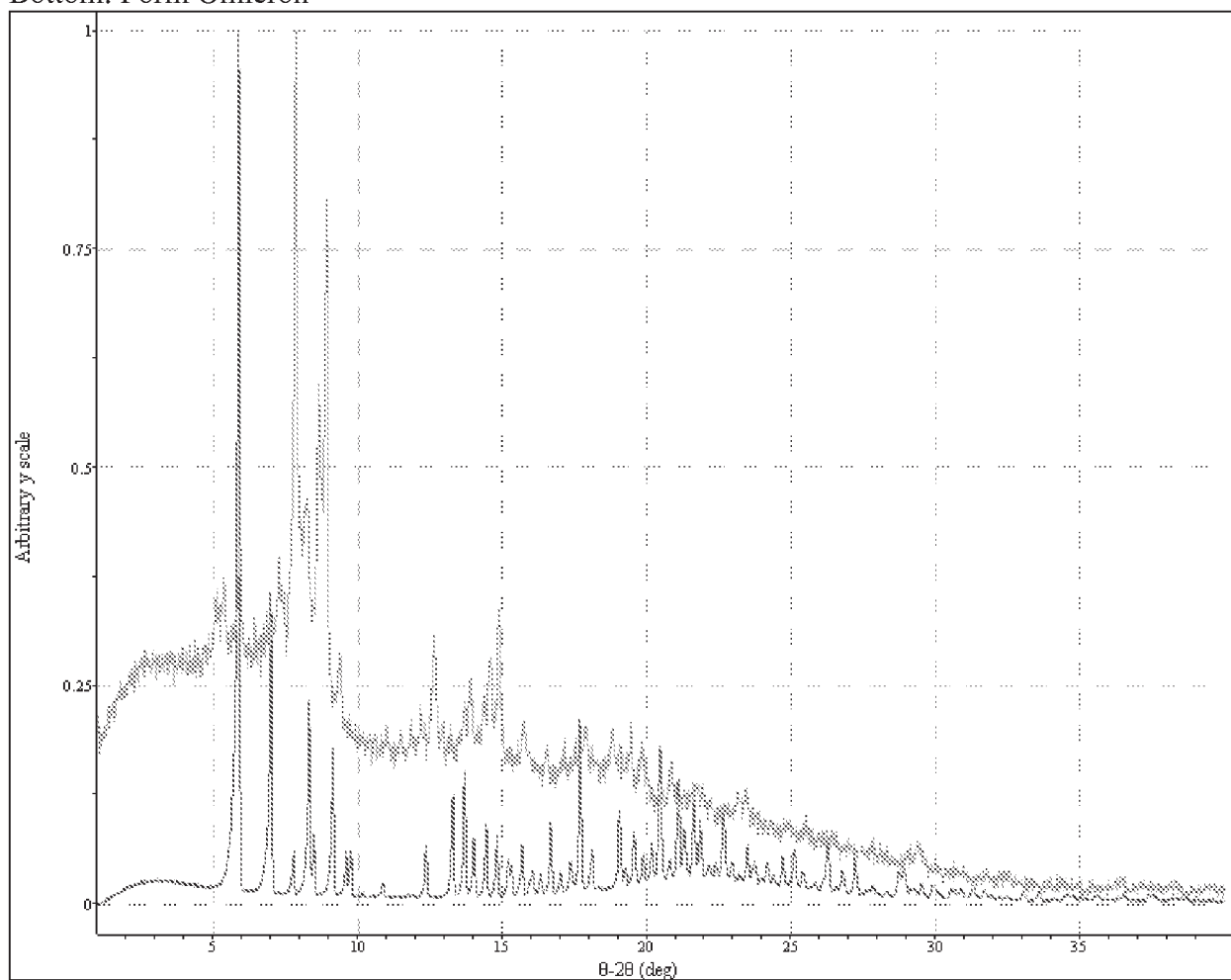


Figure 38. ATR-IR spectrum of Rifaximin Form Omicron,

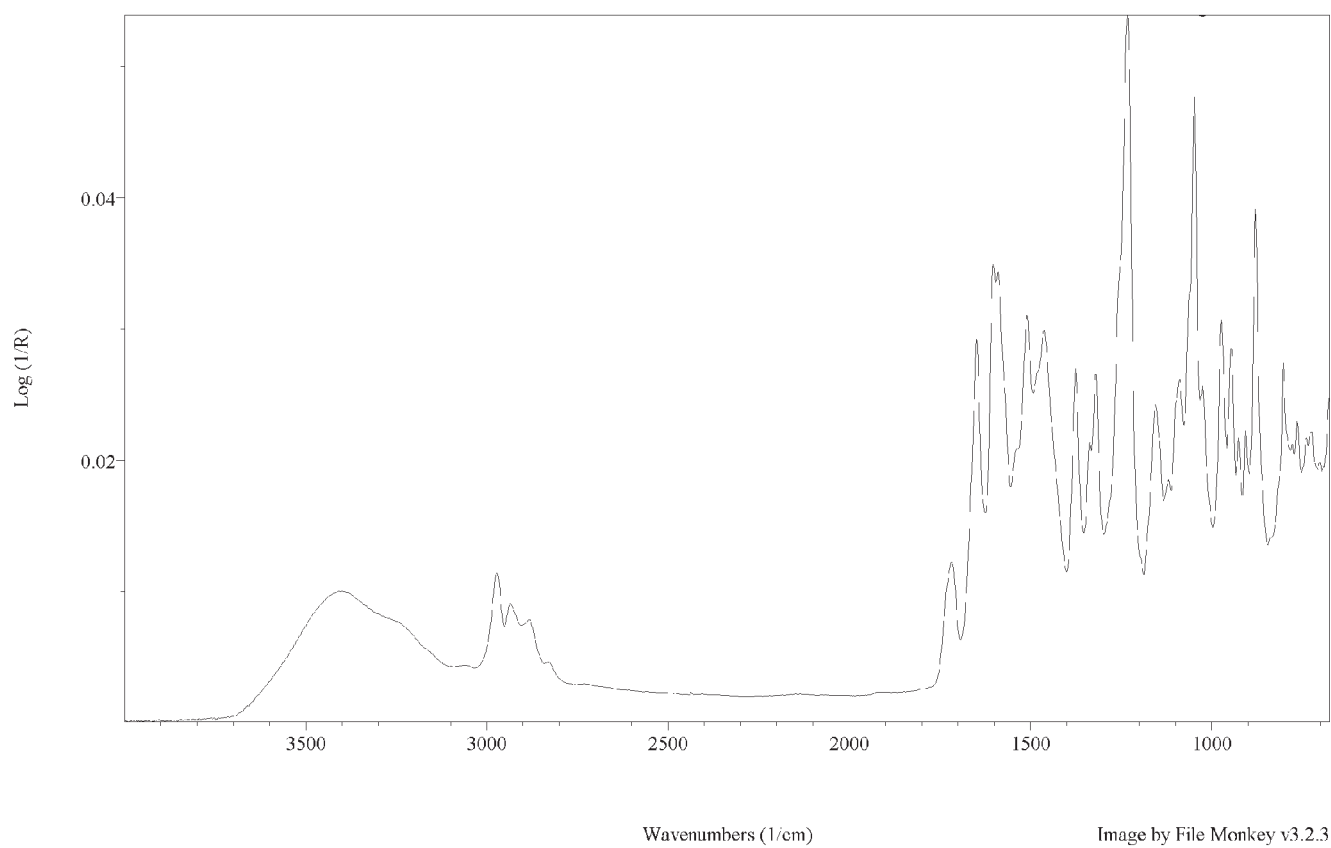


Figure 39. Raman spectrum of Rifaximin Form Omicron,

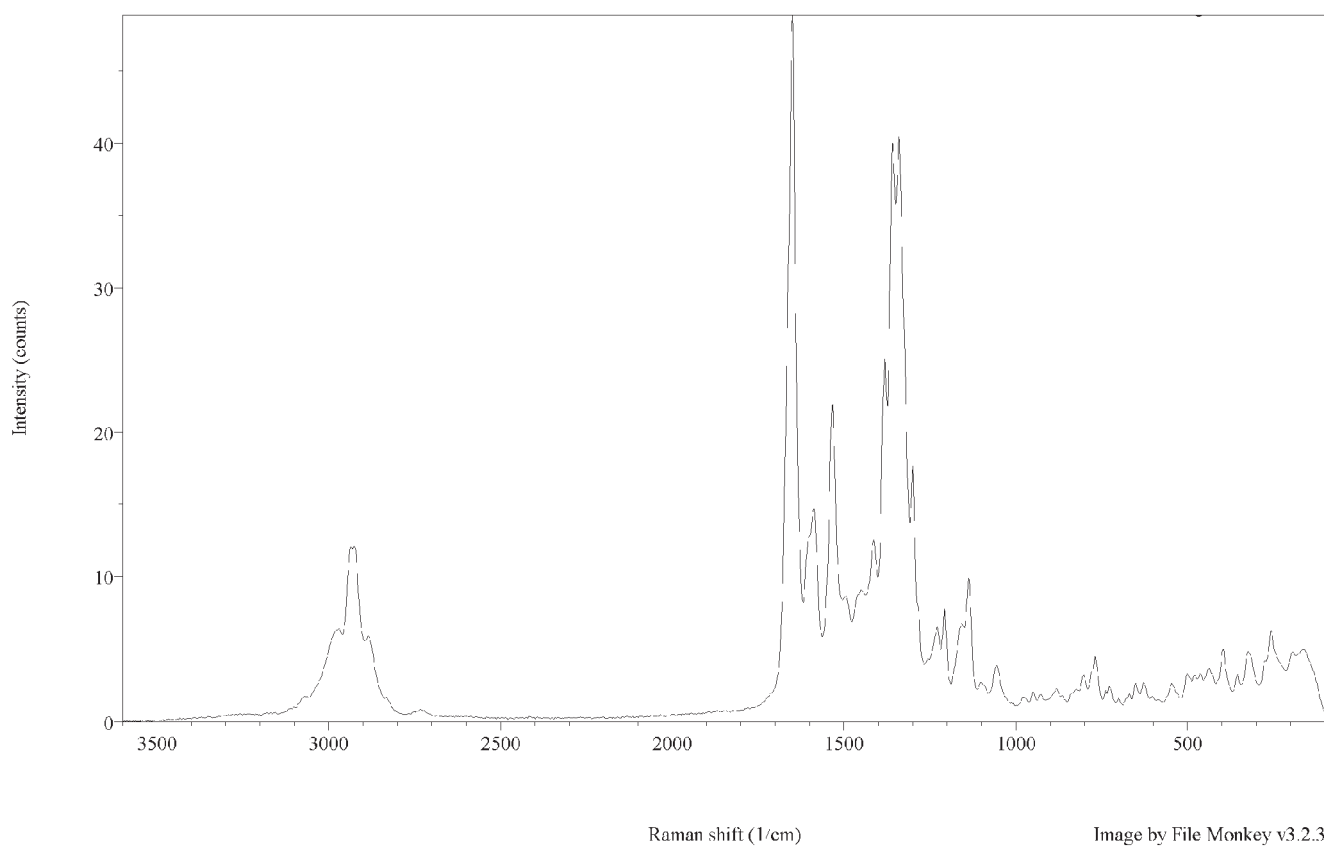


Figure 40. Solution ^1H NMR spectrum of Rifaximin Form Omicron

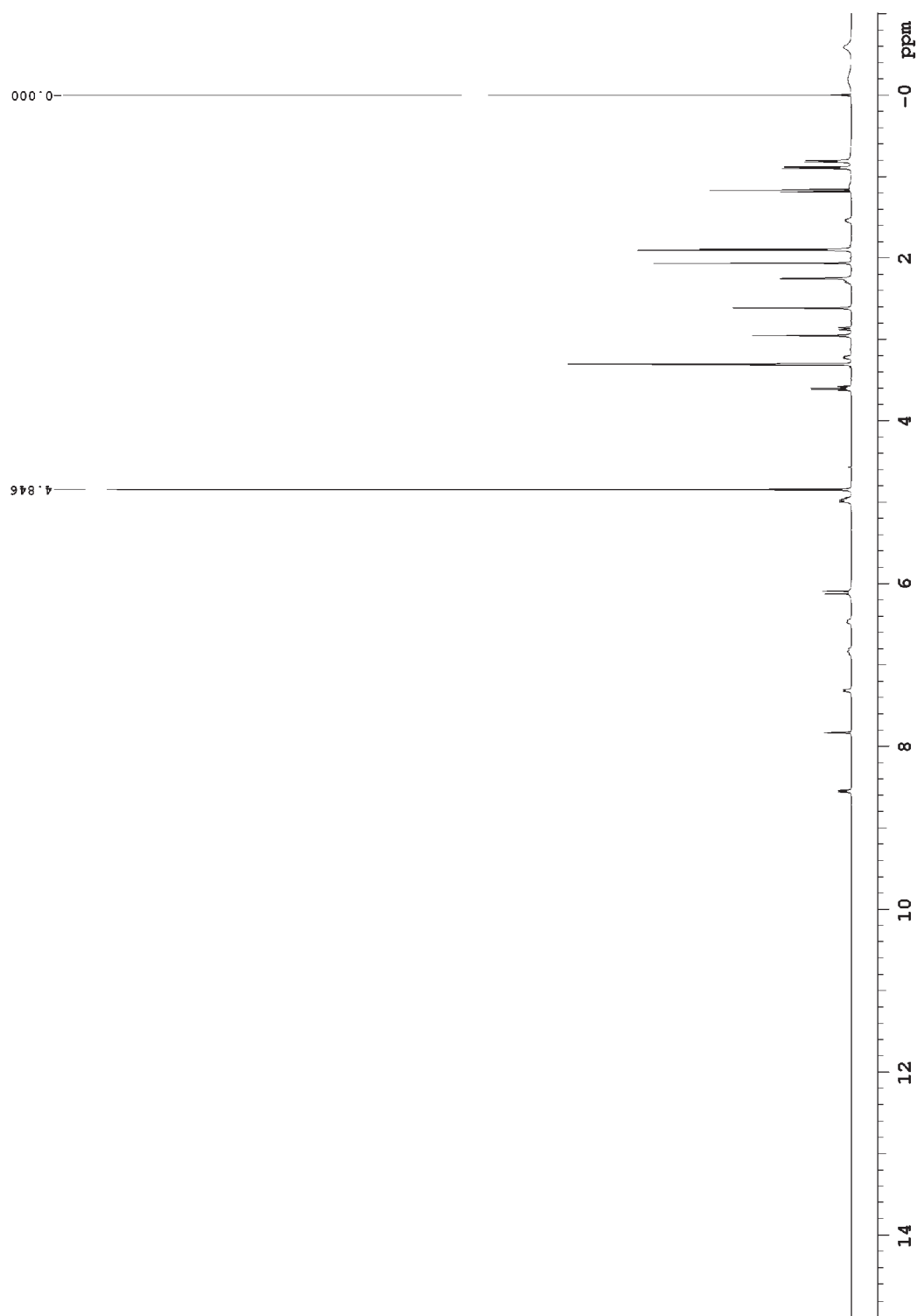


Figure 41. Solid state ^{13}C NMR spectrum of Rifaximin Form Omicron

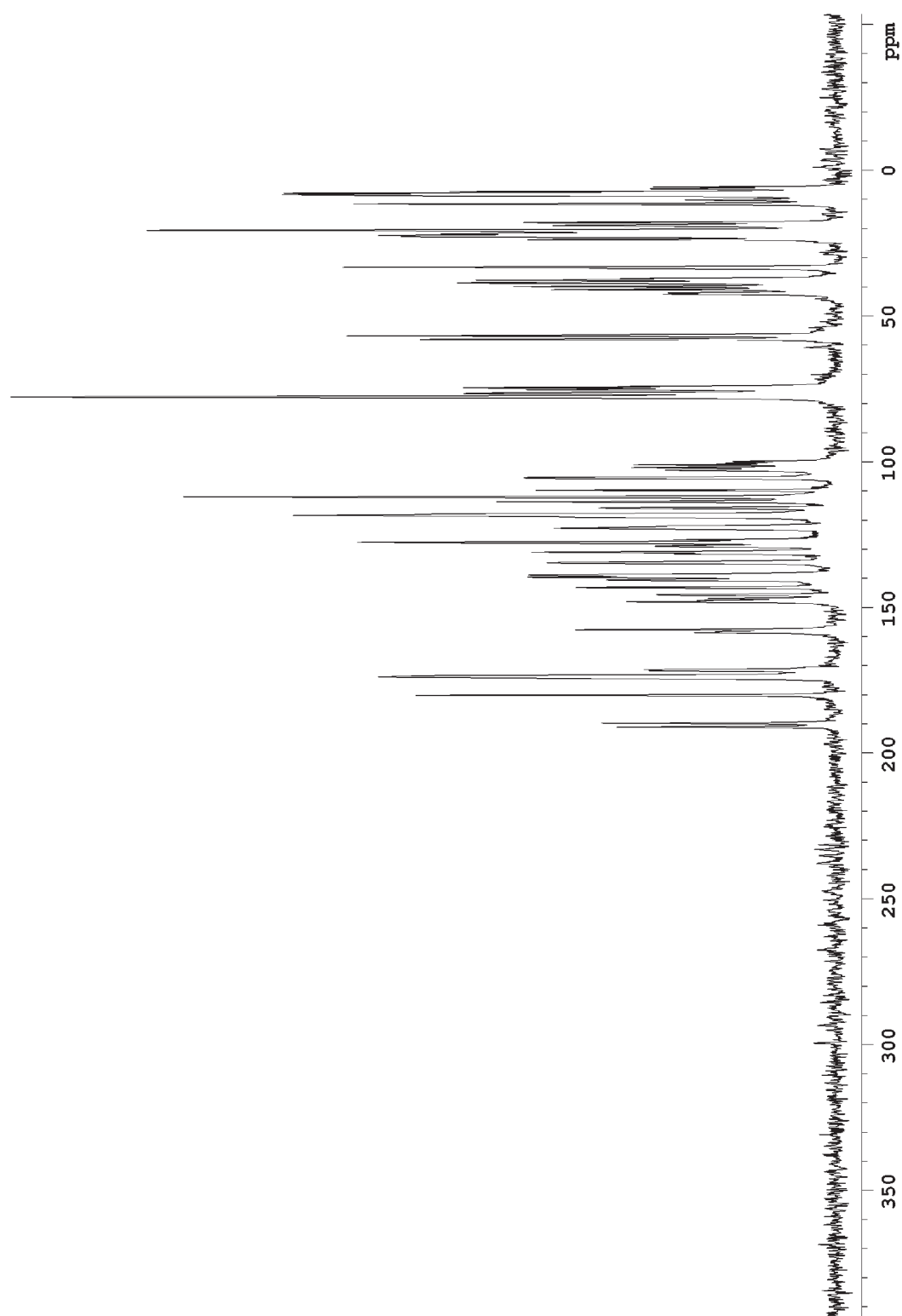


Figure 42

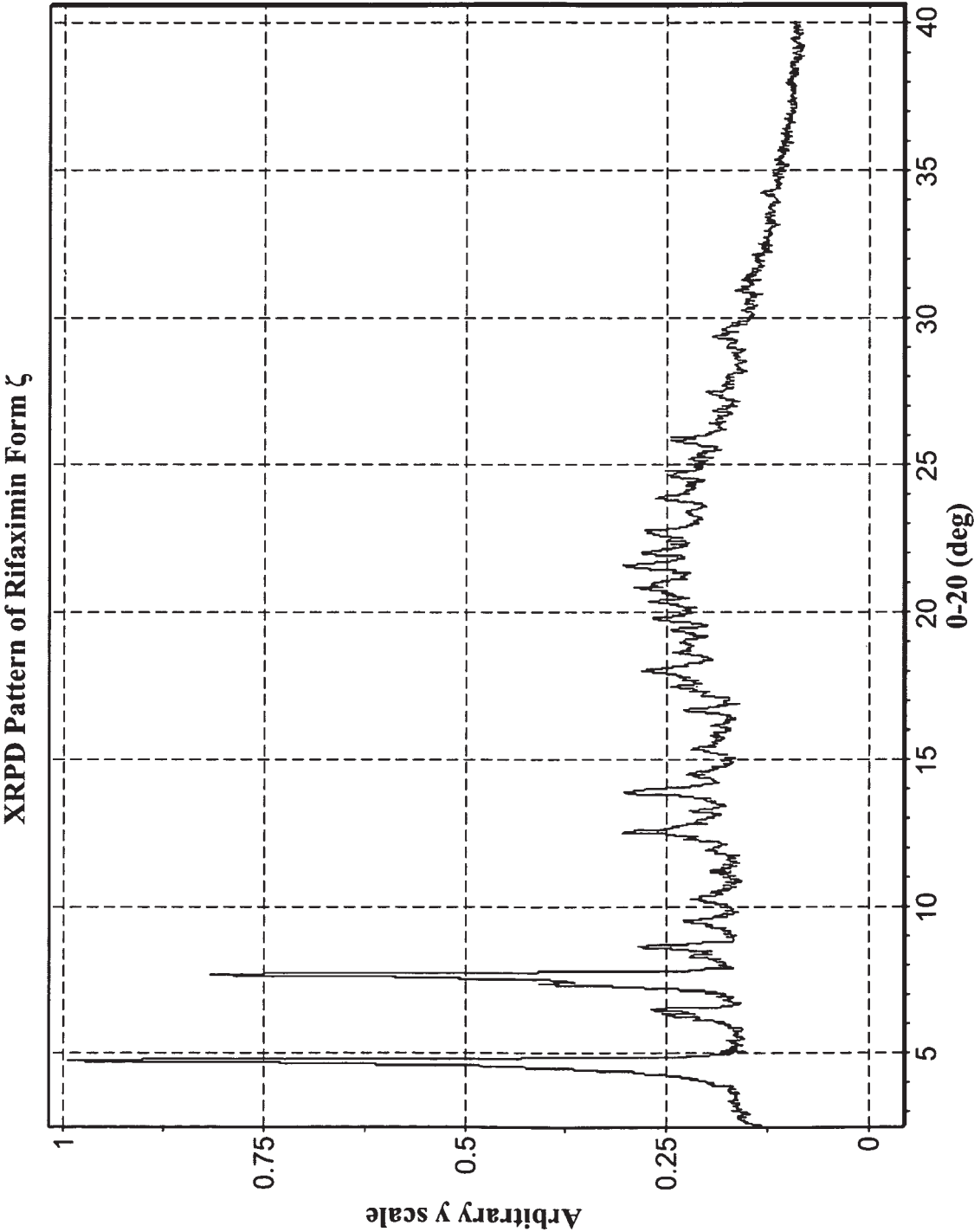


Figure 43

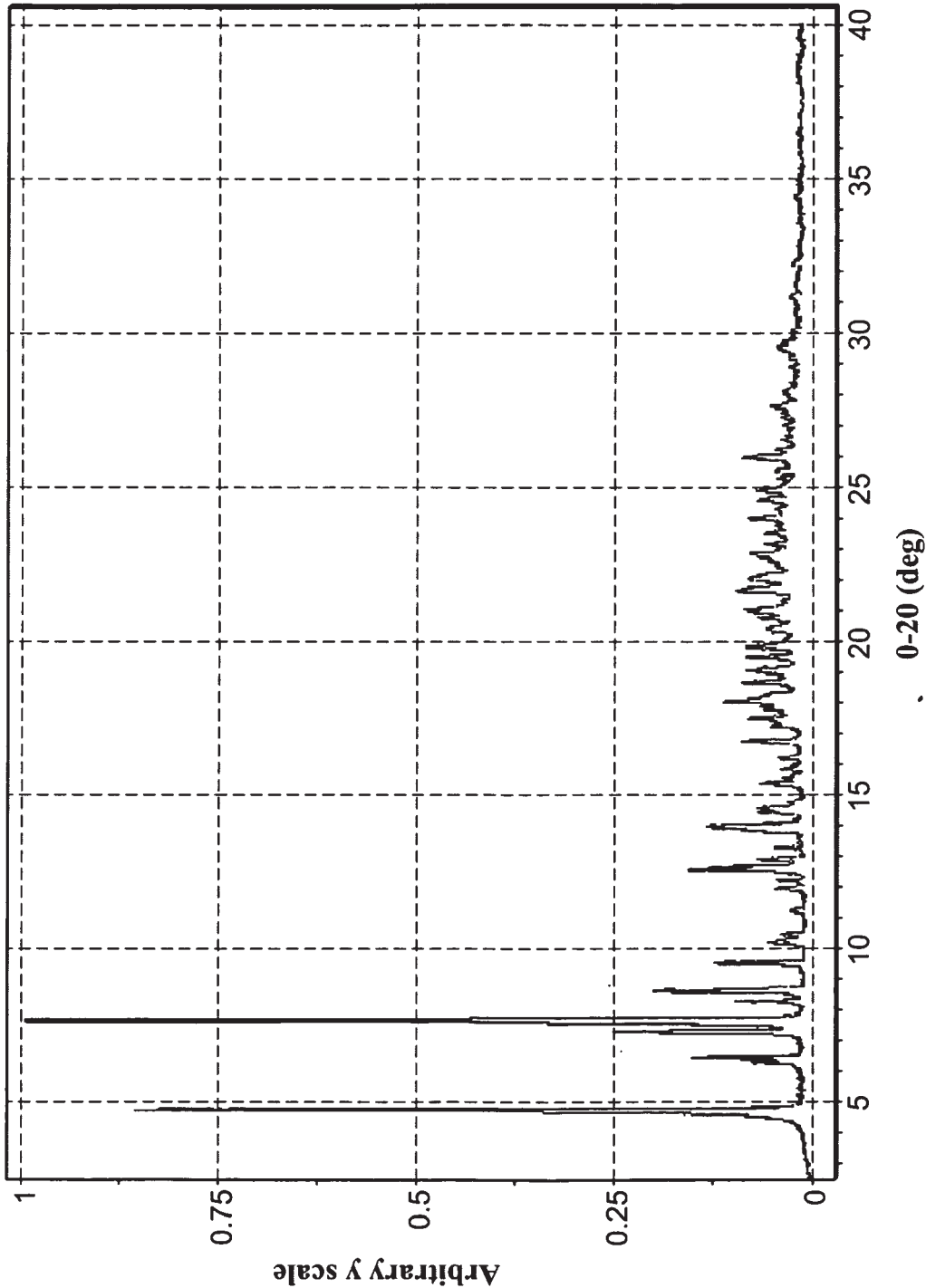


Figure 44

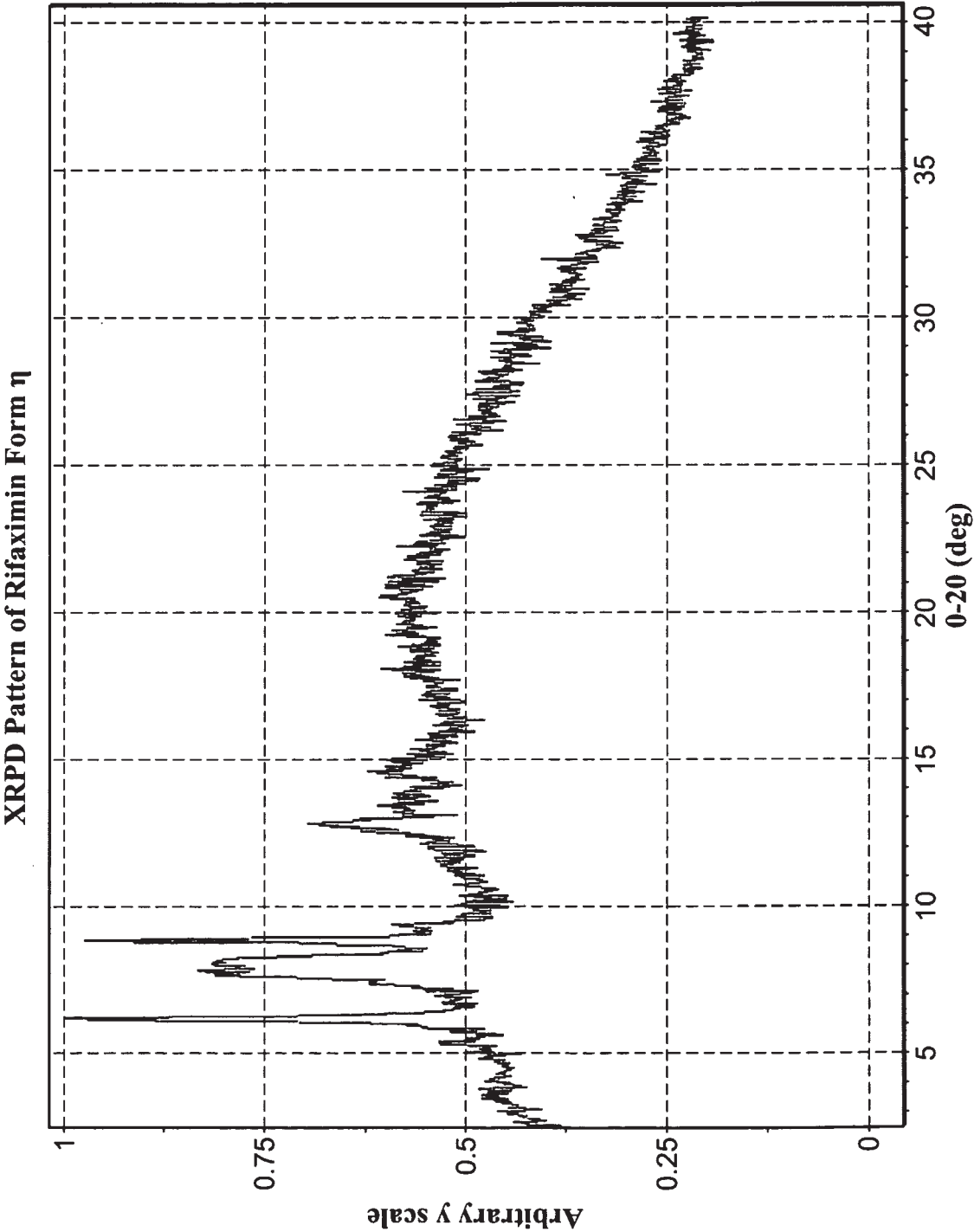


Figure 45

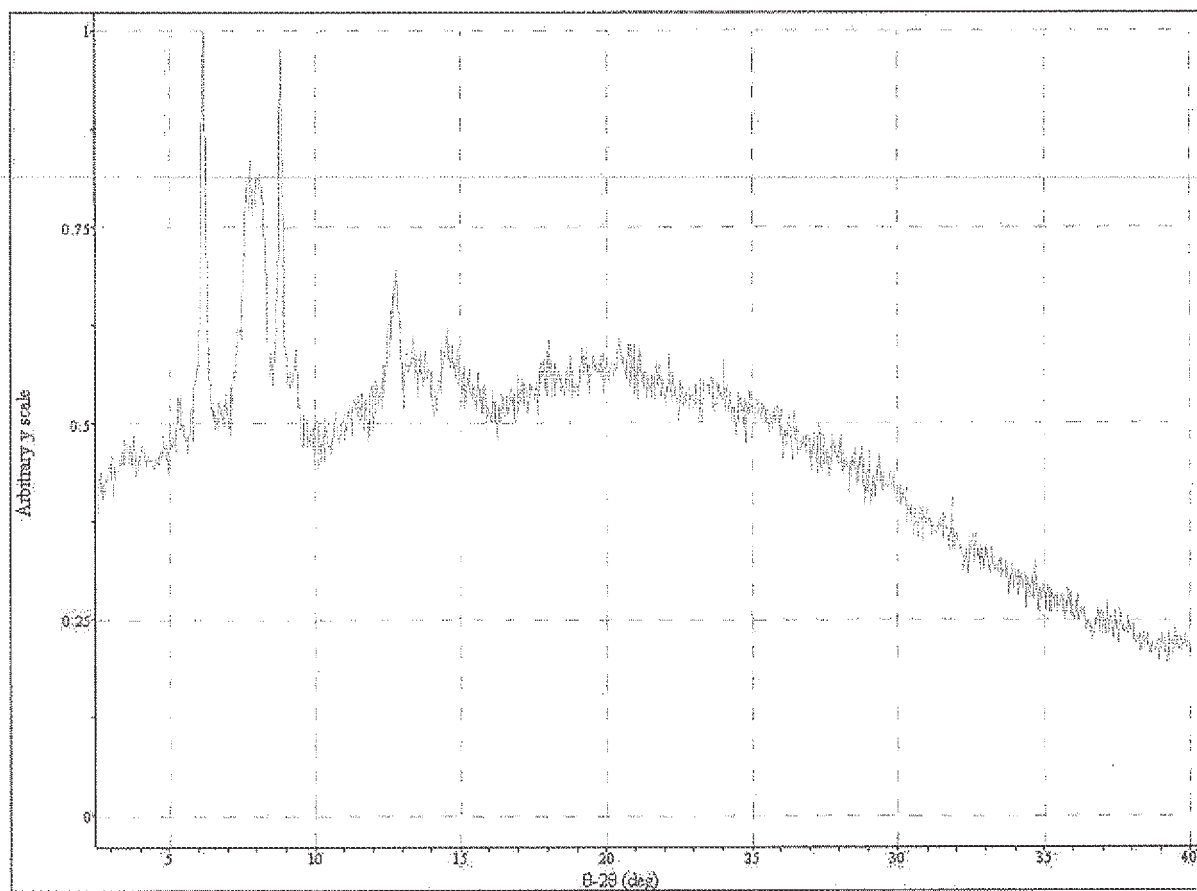
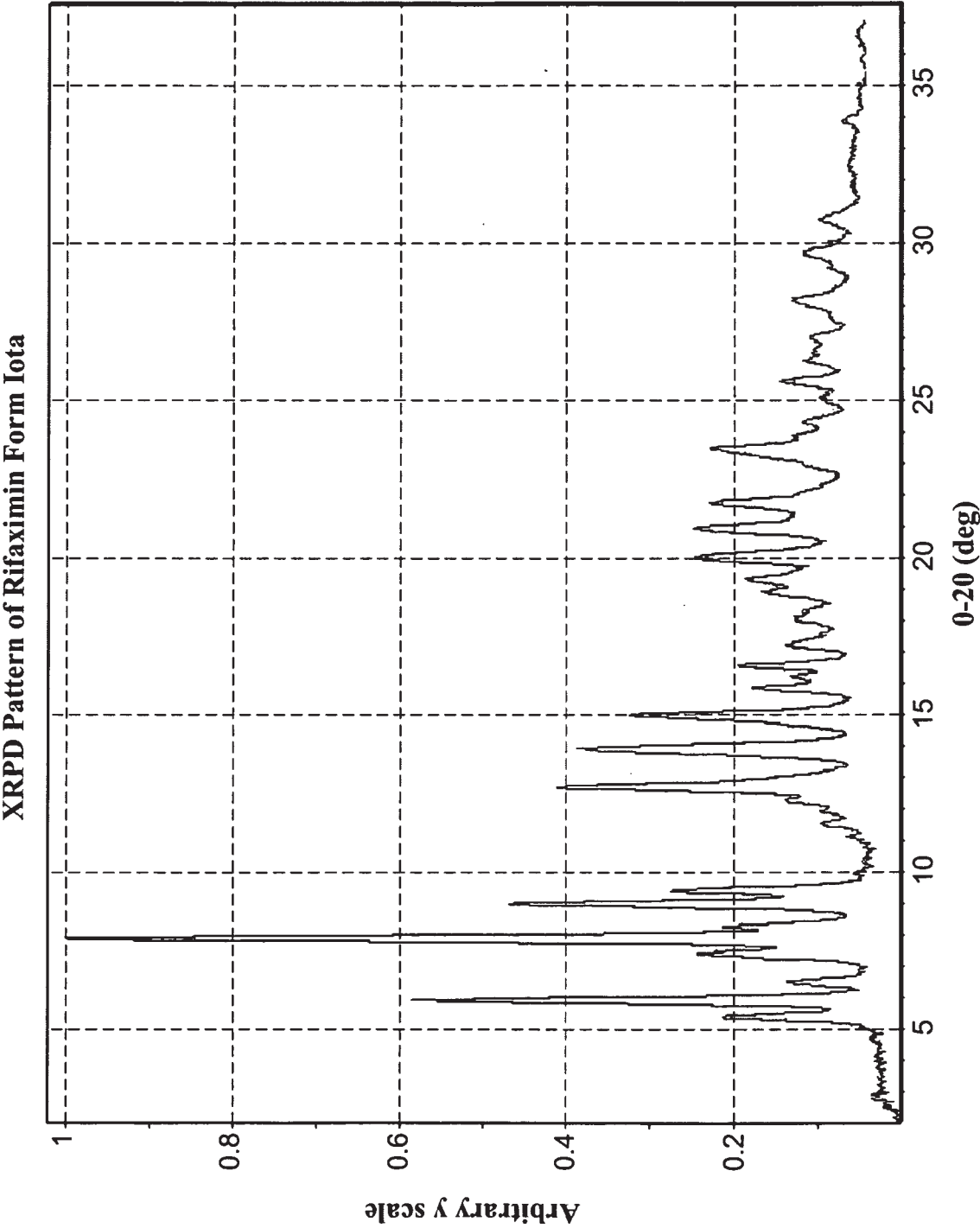
XRPD Pattern of Rifaximin Form η 

Figure 46



Background Subtracted XRPD Pattern of Rifaximin, Form Iota

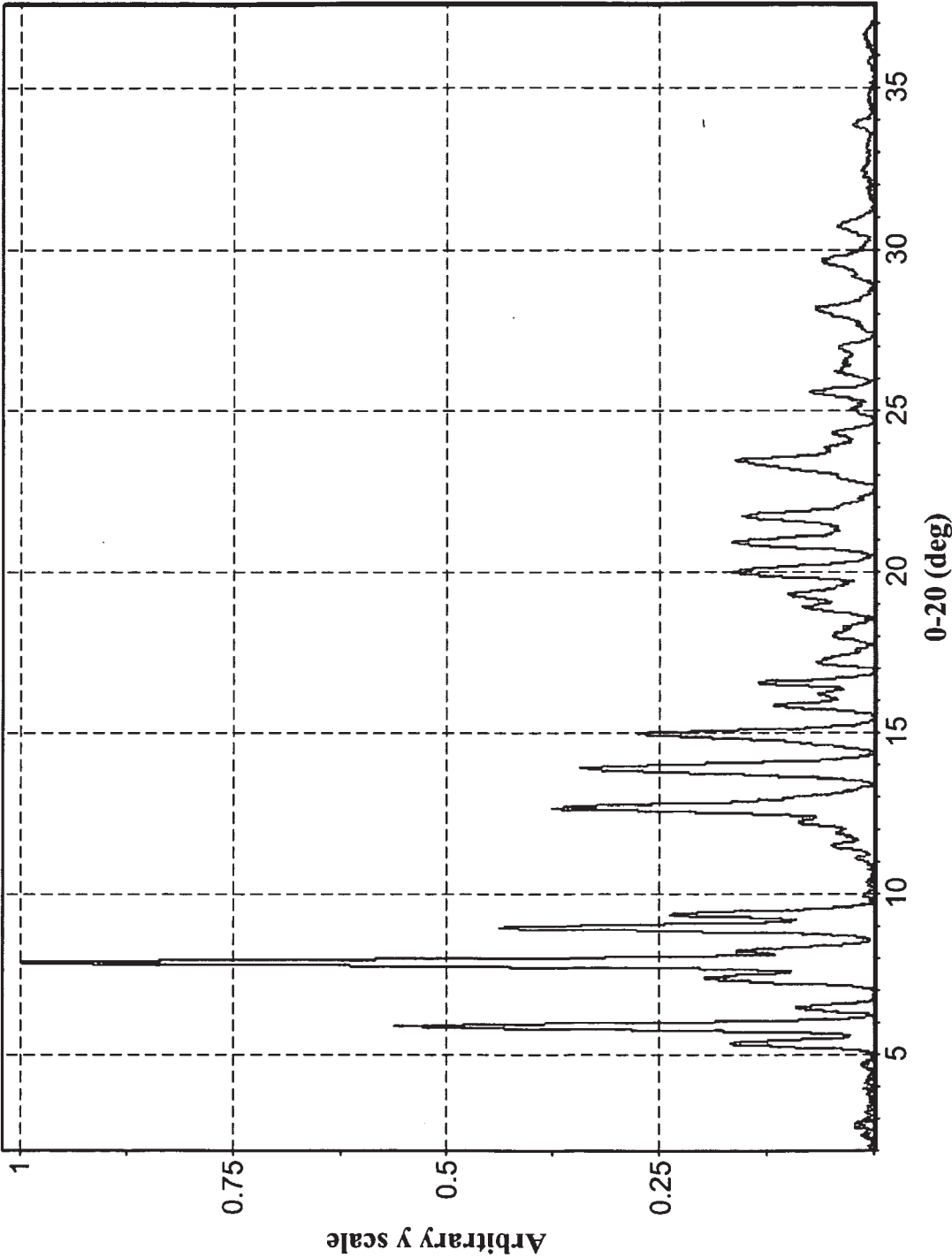


Figure 47

List of observed peaks for Rifaximin, Form Iota. Note that the peak labels in this image are meant as a visual aid.

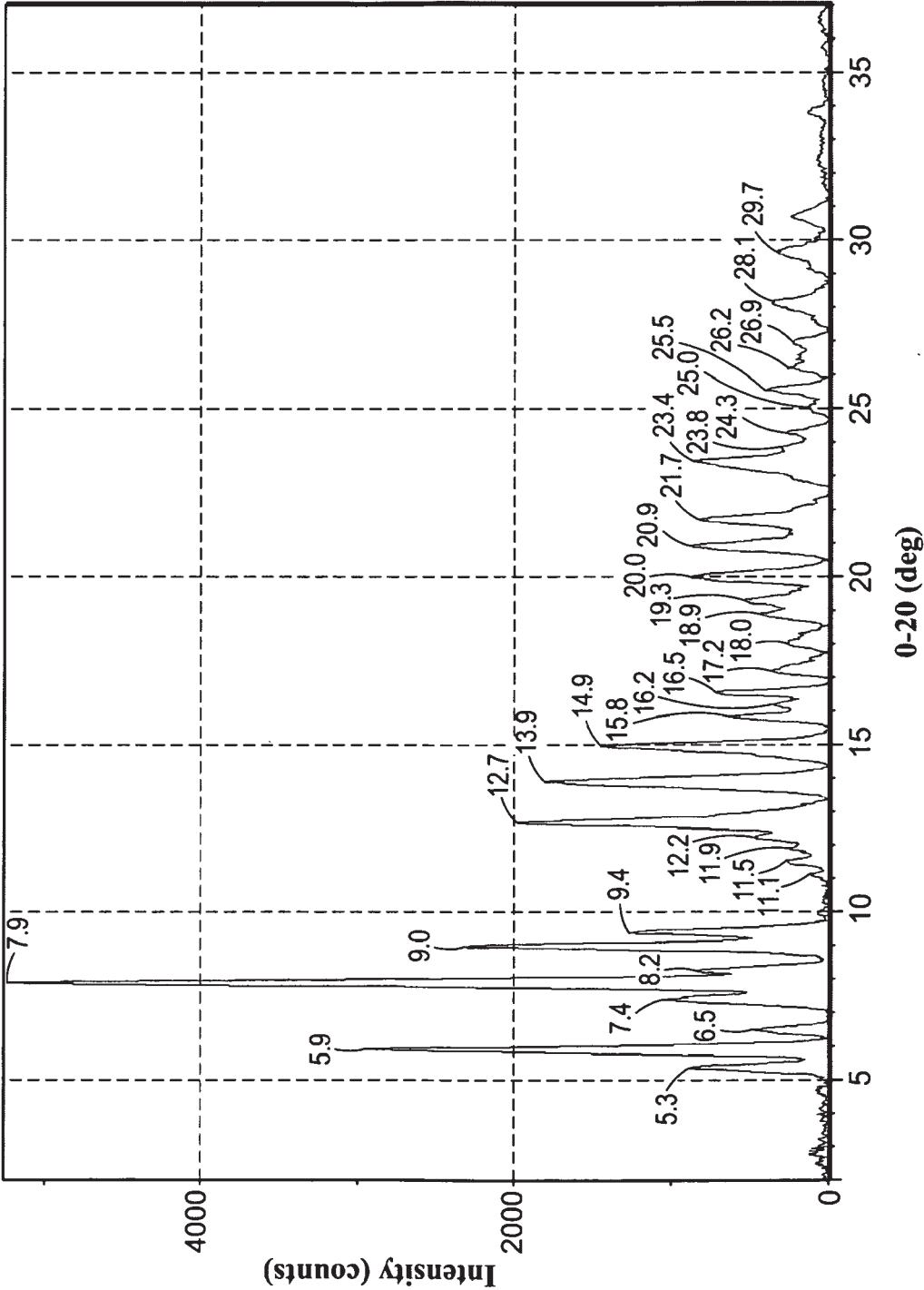


Figure 48

Figure 49A

Observed peaks for Rifaximin, Form Iota

2θ	d space (Å)	Intensity (%)
5.3 ± 0.1	16.550 ± 0.316	17
5.9 ± 0.1	15.031 ± 0.260	54
6.5 ± 0.1	13.683 ± 0.215	9
7.4 ± 0.1	12.011 ± 0.165	20
7.9 ± 0.1	11.220 ± 0.144	100
8.2 ± 0.1	10.757 ± 0.132	16
9.0 ± 0.1	9.870 ± 0.111	44
9.4 ± 0.1	9.449 ± 0.102	24
11.1 ± 0.1	7.957 ± 0.072	2
11.5 ± 0.1	7.695 ± 0.067	5
11.9 ± 0.1	7.450 ± 0.063	5
12.2 ± 0.1	7.231 ± 0.059	9
12.7 ± 0.1	6.992 ± 0.055	37
13.9 ± 0.1	6.371 ± 0.046	34
14.9 ± 0.1	5.930 ± 0.040	27
15.8 ± 0.1	5.602 ± 0.035	12
16.2 ± 0.1	5.478 ± 0.034	7
16.5 ± 0.1	5.360 ± 0.032	14
17.2 ± 0.1	5.162 ± 0.030	7
18.0 ± 0.1	4.928 ± 0.027	5
18.9 ± 0.1	4.700 ± 0.025	8
19.3 ± 0.1	4.604 ± 0.024	10
20.0 ± 0.1	4.448 ± 0.022	17
20.9 ± 0.1	4.250 ± 0.020	17
21.7 ± 0.1	4.099 ± 0.019	15
23.4 ± 0.1	3.795 ± 0.016	16
23.8 ± 0.1	3.733 ± 0.015	6
24.3 ± 0.1	3.663 ± 0.015	5
25.0 ± 0.1	3.559 ± 0.014	3
25.5 ± 0.1	3.488 ± 0.013	8
26.2 ± 0.1	3.401 ± 0.013	5
26.9 ± 0.1	3.310 ± 0.012	4
28.1 ± 0.1	3.176 ± 0.011	7
29.7 ± 0.1	3.010 ± 0.010	6

Figure 49B

Prominent peaks for Rifaximin, Form Iota

2θ	d space (Å)	Intensity (%)
5.9 ± 0.1	15.031 ± 0.260	54
7.9 ± 0.1	11.220 ± 0.144	100
9.0 ± 0.1	9.870 ± 0.111	44
12.7 ± 0.1	6.992 ± 0.055	37
13.9 ± 0.1	6.371 ± 0.046	34
14.9 ± 0.1	5.930 ± 0.040	27

iota

Thermal data for Rifaximin, Form

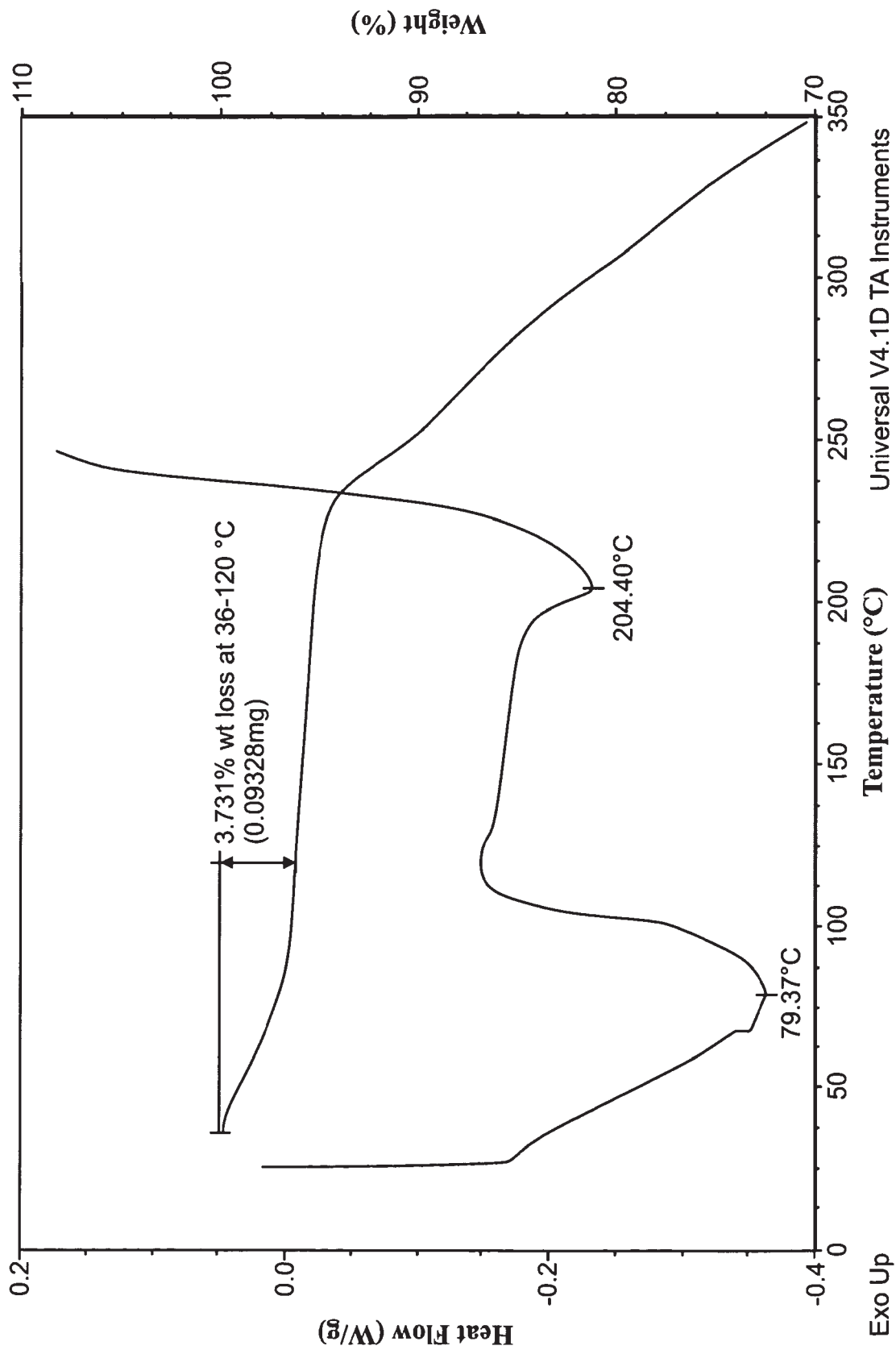
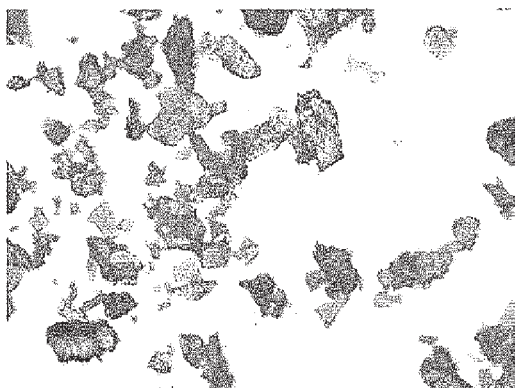


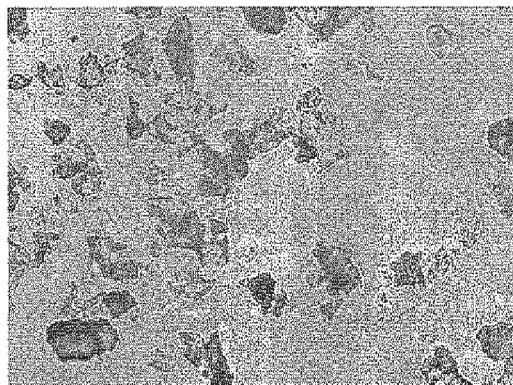
Figure 50

Figure 51A

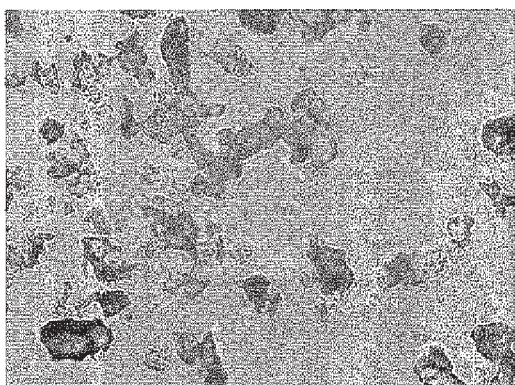
Hot Stage microscopy of Rifaximin, Form i.



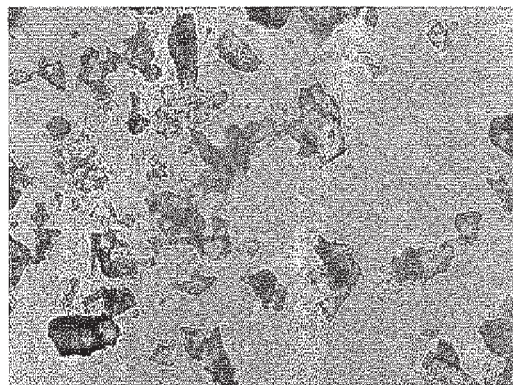
T= 30°C



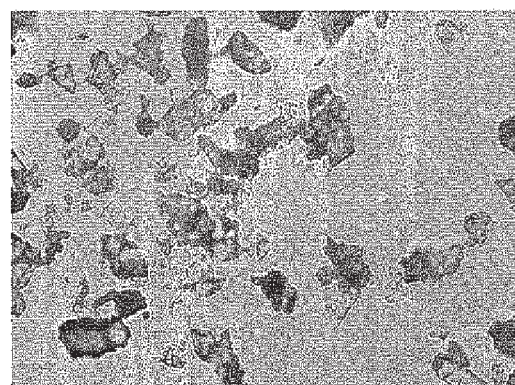
T= 30°C



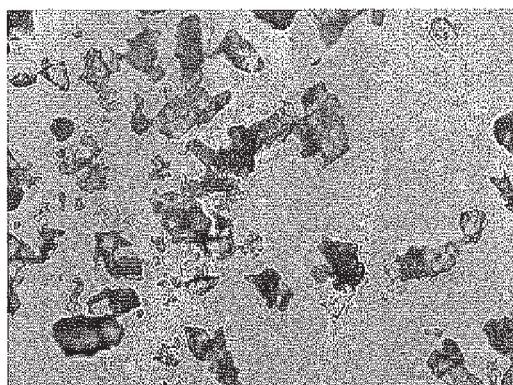
T= 80°C



T= 120°C



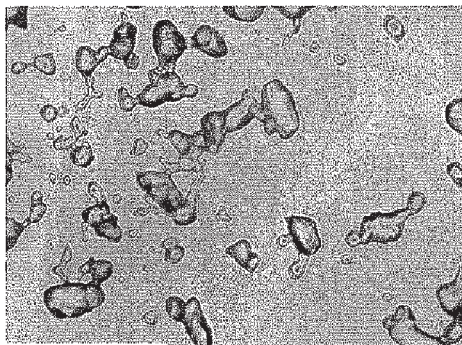
T= 190°C



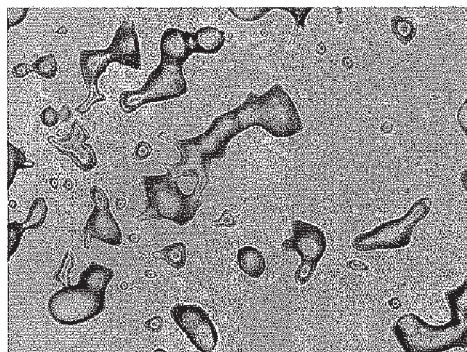
T= 204°C

Figure 51B

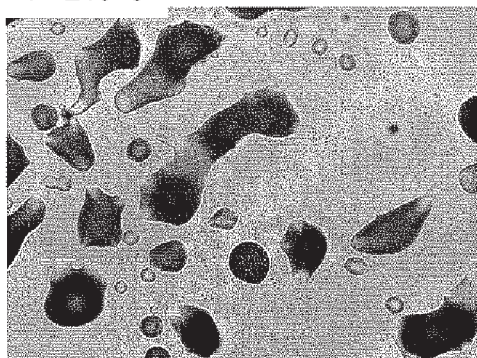
Hot Stage microscopy of Rifaximin, Form i.



T= 218°C



T= 222°C



T= 250°C

Figure 52

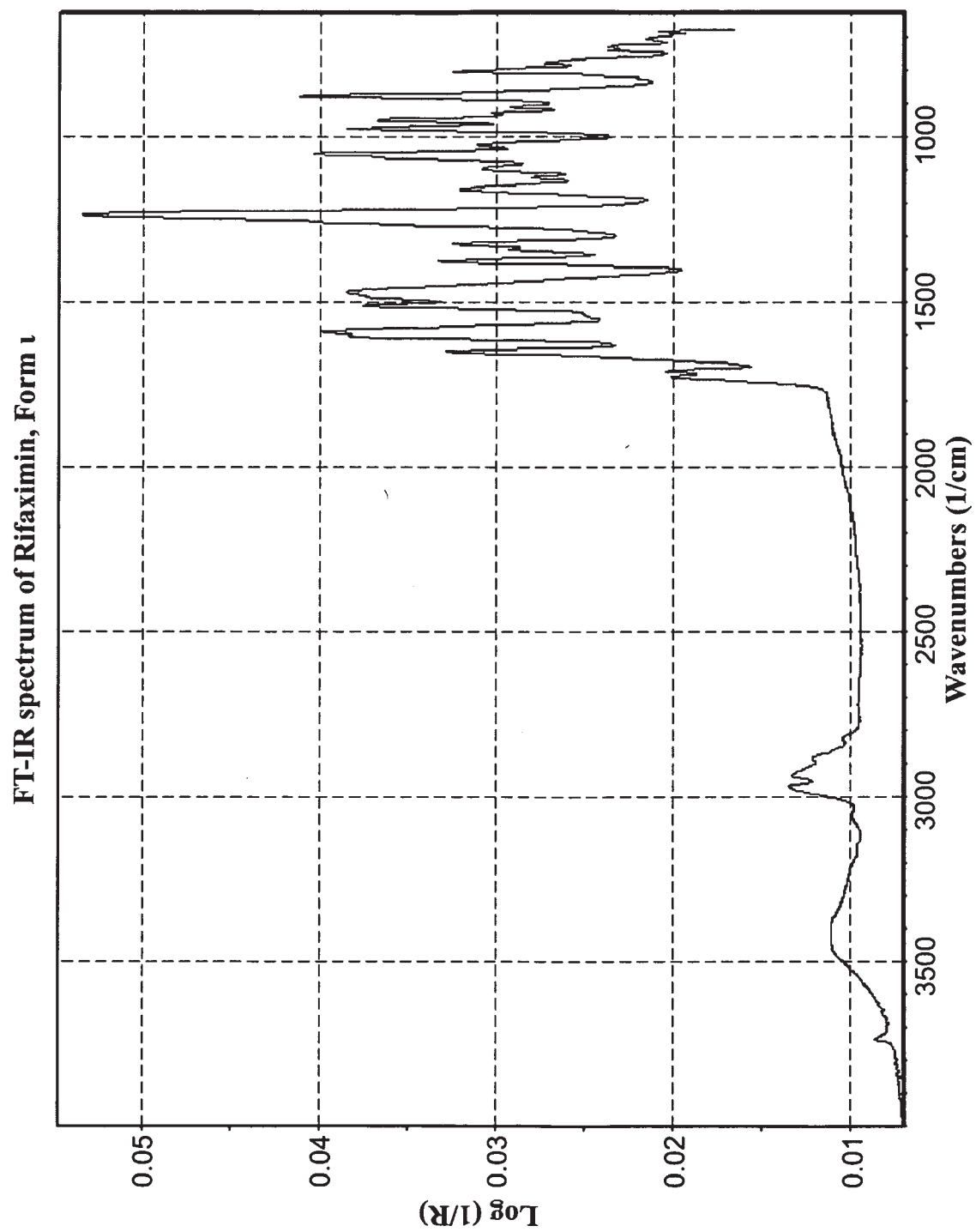


Figure 53

