

# Supplementary Materials: Glasdegib Dimaleate Form: Synthesis, Characterization and Comparison of its Properties with Monomaleate Analogue

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## Table of Contents

1.	DSC & TGA thermograms .....	S2
2.	Infrared spectra .....	S4
3.	Raman spectra .....	S8
4.	PXRD diffractograms .....	S10
5.	LC Chromatograms.....	S14
6.	ssNMR spectra .....	S15
7.	Solution <sup>1</sup> H- and <sup>13</sup> C-NMR spectra of glasdegib derivatives.....	S16
7.1.	Glasdegib monomaleate .....	S16
7.2.	Glasdegib dimaleate .....	S18
8.	Calculated pK <sub>a</sub> data of glasdegib and maleic acid.....	S20

## 1. DSC & TGA thermograms

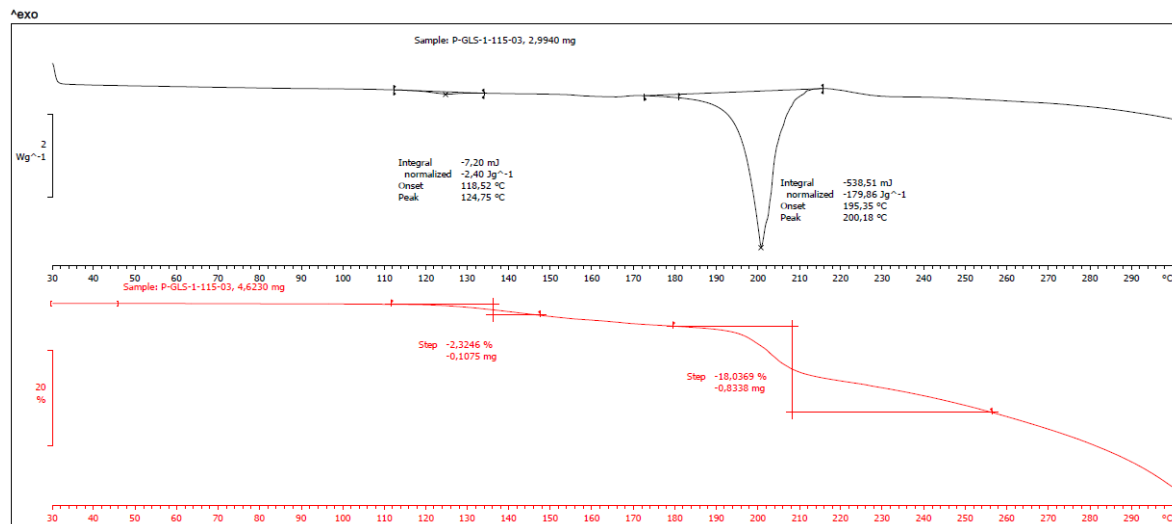


Figure S1: DSC & TGA thermogram of glasdegib monomaleate.

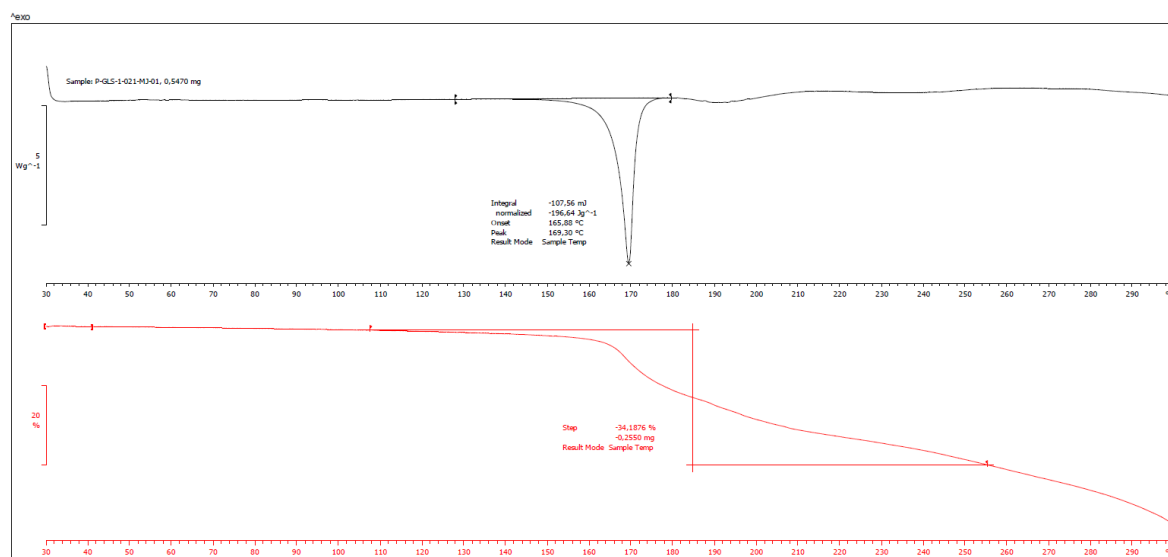


Figure S2: DSC & TGA thermogram of glasdegib dimaleate.

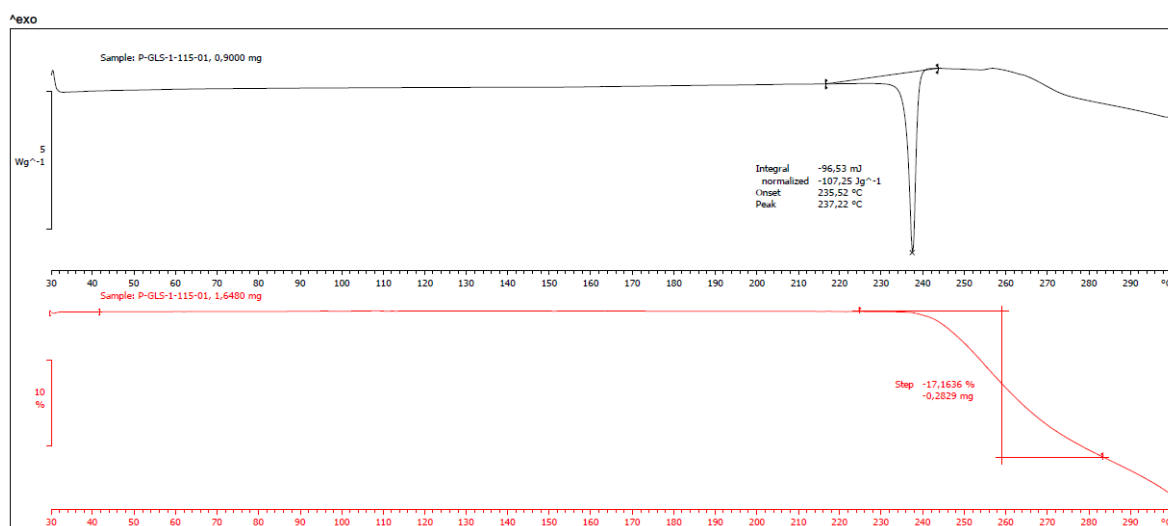


Figure S3: DSC &amp; TGA thermogram of glasdegib base.

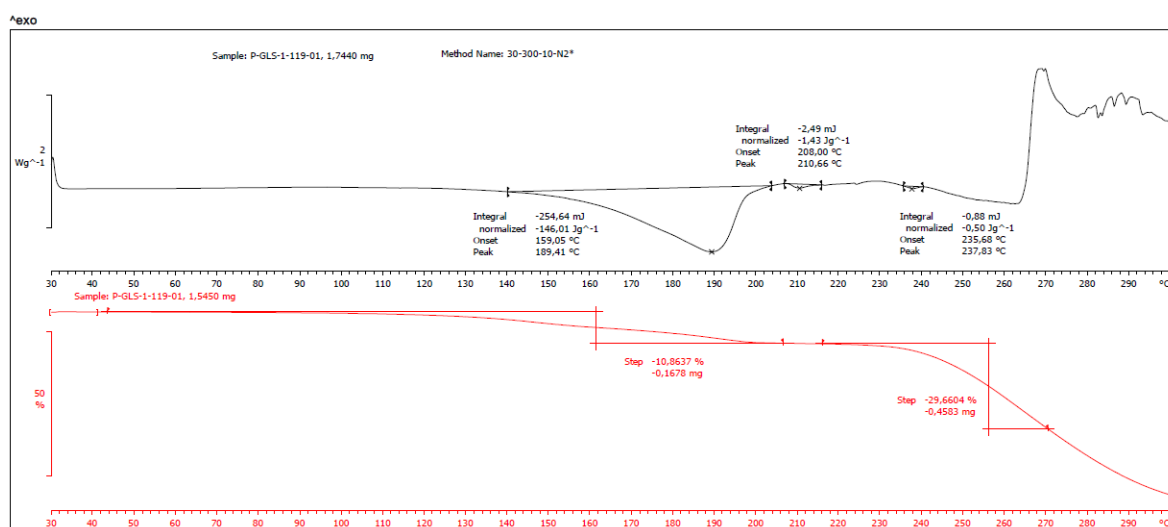


Figure S4: DSC &amp; TGA thermogram of glasdegib dihydrochloride hydrate.

## 2. Infrared spectra

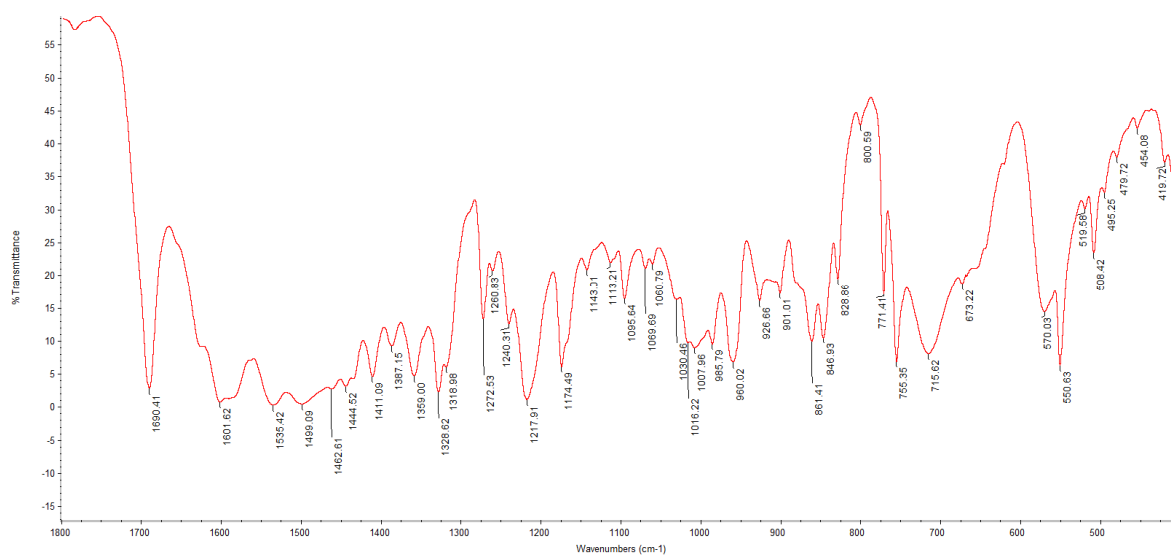


Figure S5: IR spectrum of glasdegib monomaleate 1800-400 cm<sup>-1</sup>.

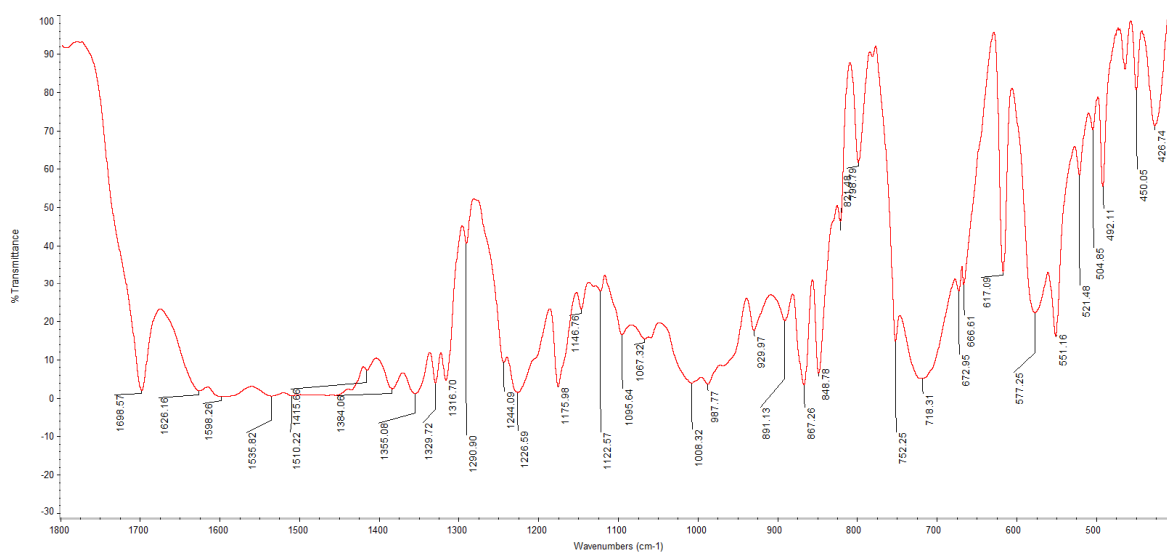


Figure S6: IR spectrum of glasdegib dimaleate 1800-400 cm<sup>-1</sup>.

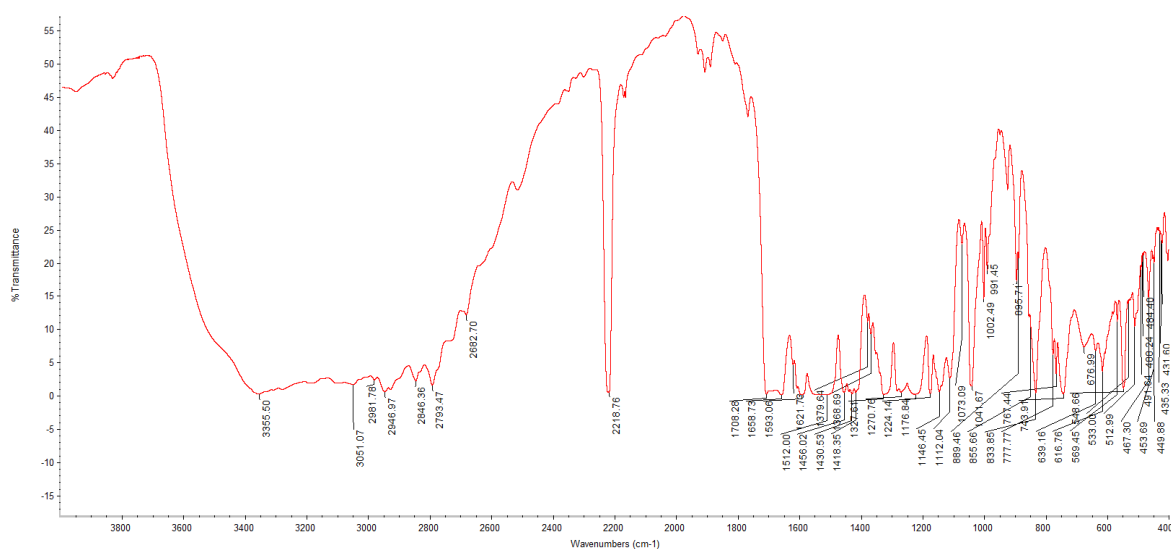


Figure S7: IR spectrum of glasdegib base 4000-400 cm<sup>-1</sup>.

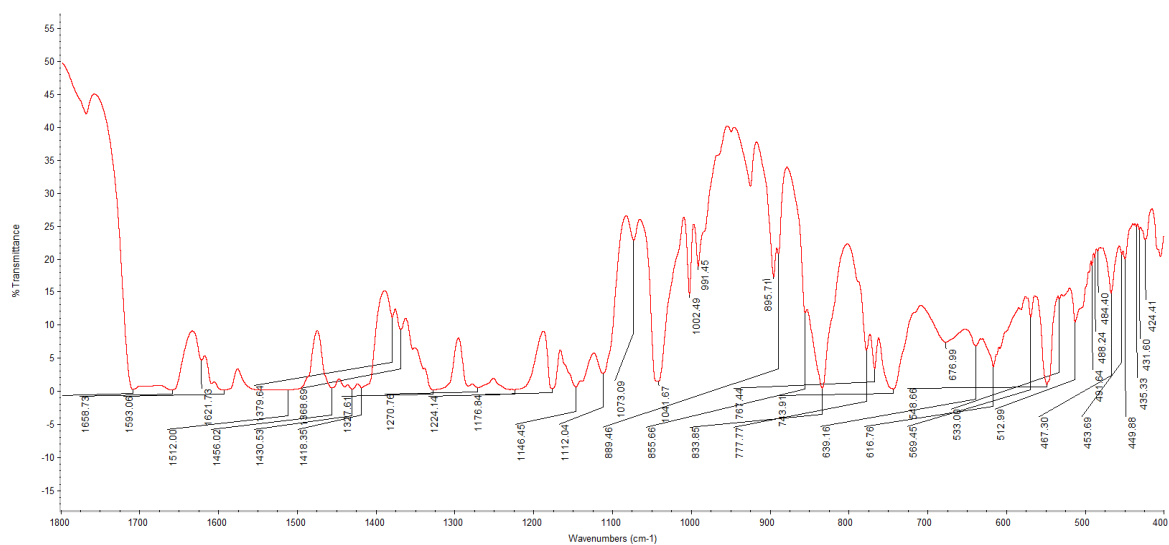


Figure S8: IR spectrum of glasdegib base 1800-400 cm<sup>-1</sup>.

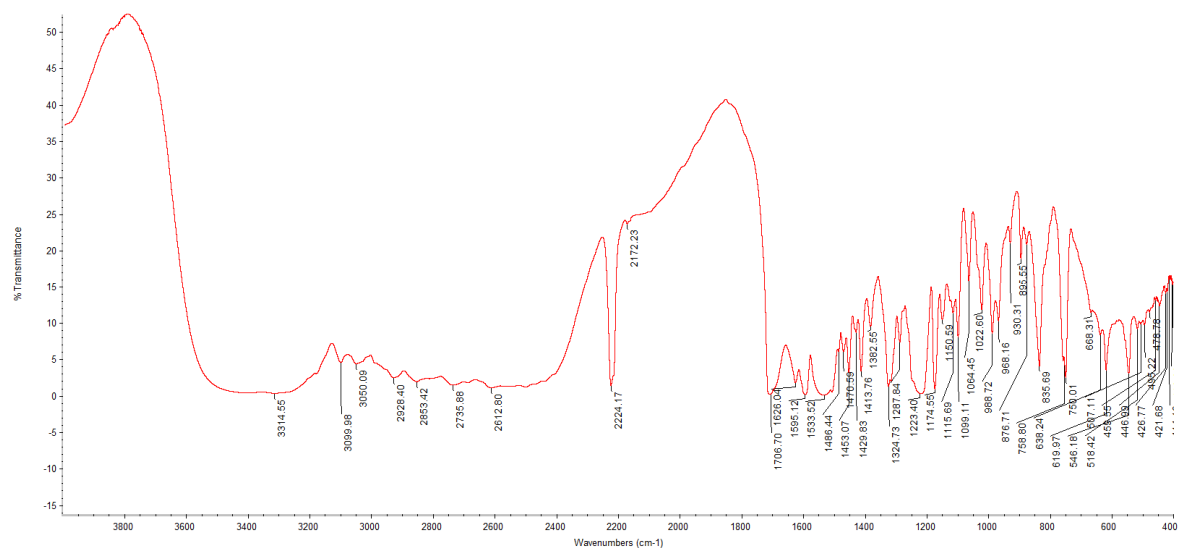


Figure S9. IR spectrum of glasdegib dihydrochloride hydrate 4000-400 cm<sup>-1</sup>.

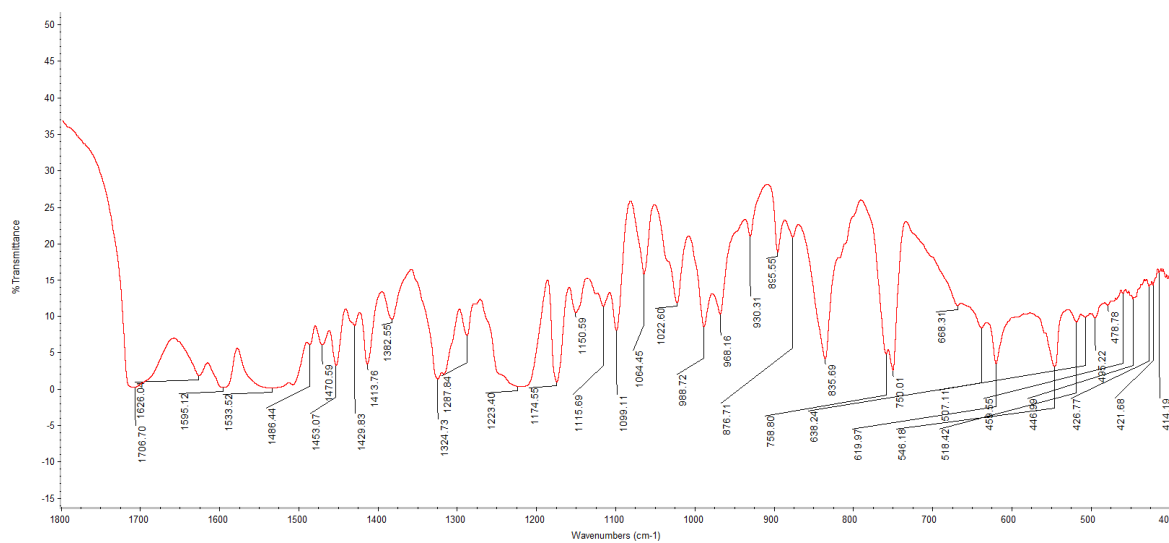


Figure S10. IR spectrum of glasdegib dihydrochloride hydrate 1800-400 cm<sup>-1</sup>.

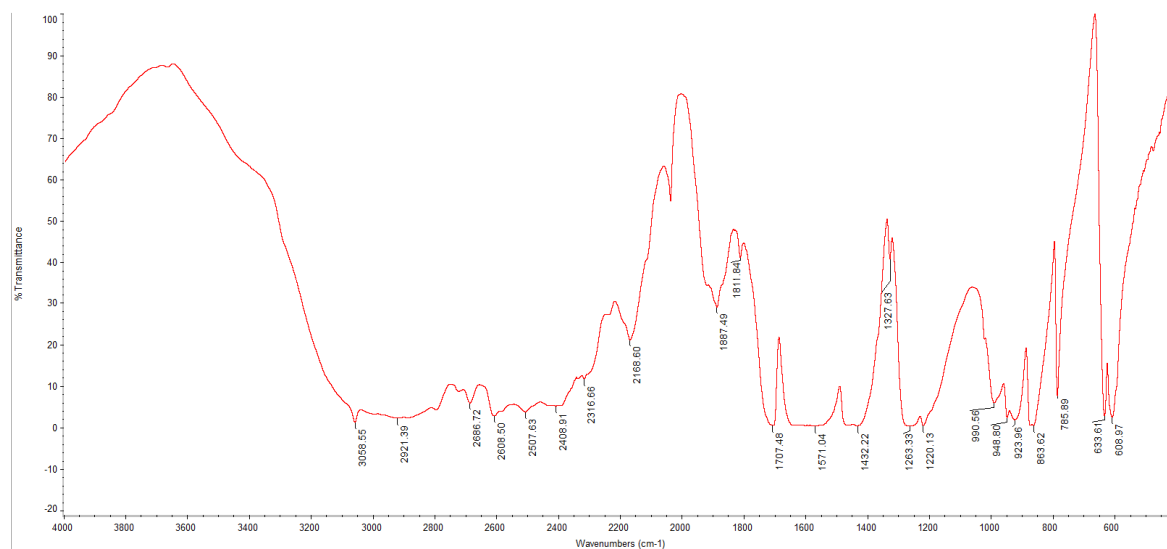


Figure S11. IR spectrum of maleic acid 4000-400 cm<sup>-1</sup>.

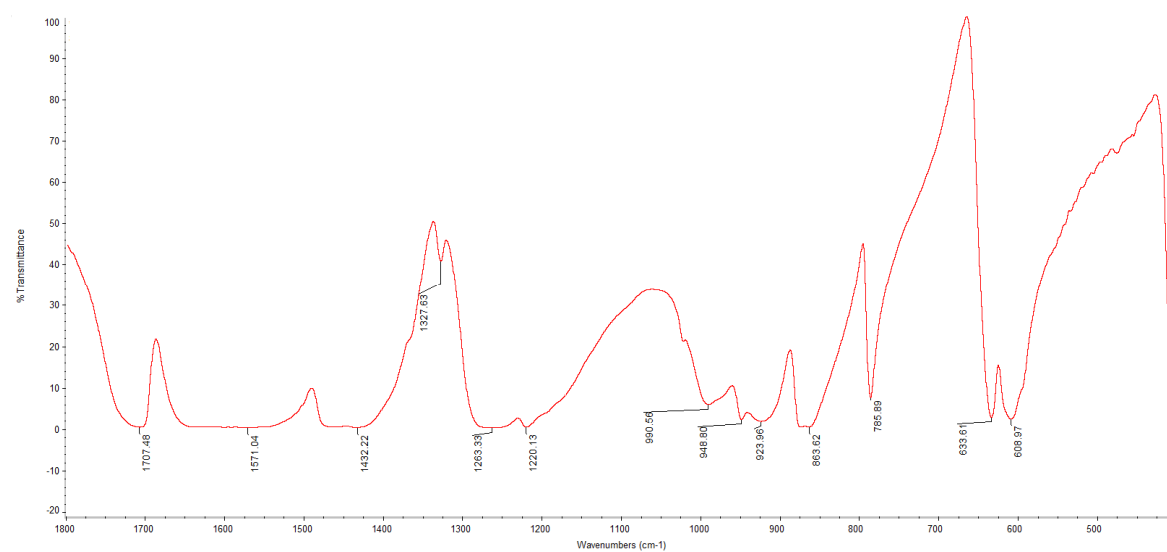


Figure S12. IR spectrum of maleic acid 1800-400 cm<sup>-1</sup>.

### 3. Raman spectra

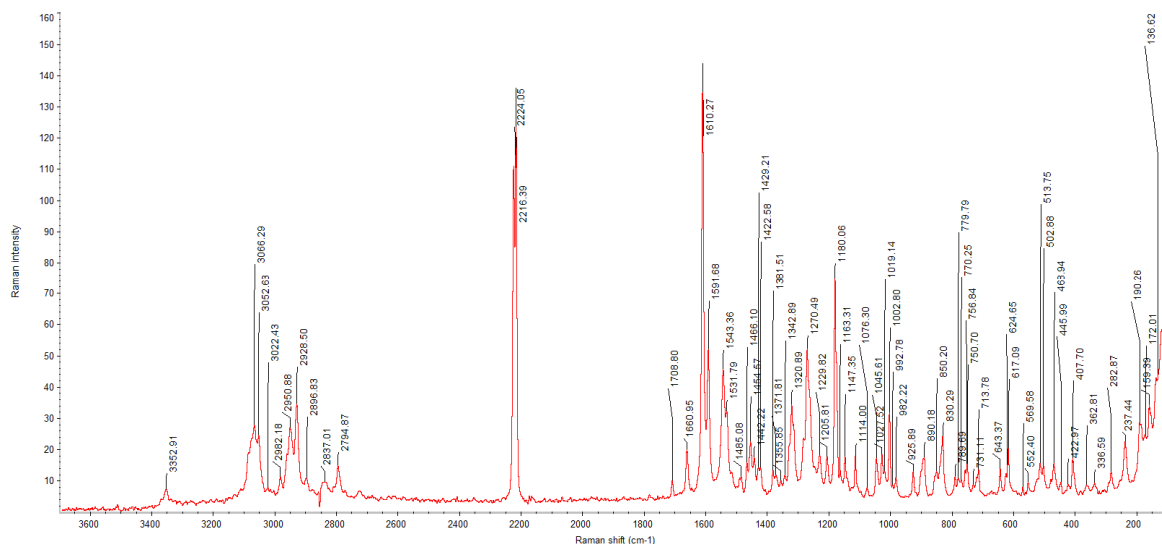


Figure S13. Raman spectrum of glasdegib base.

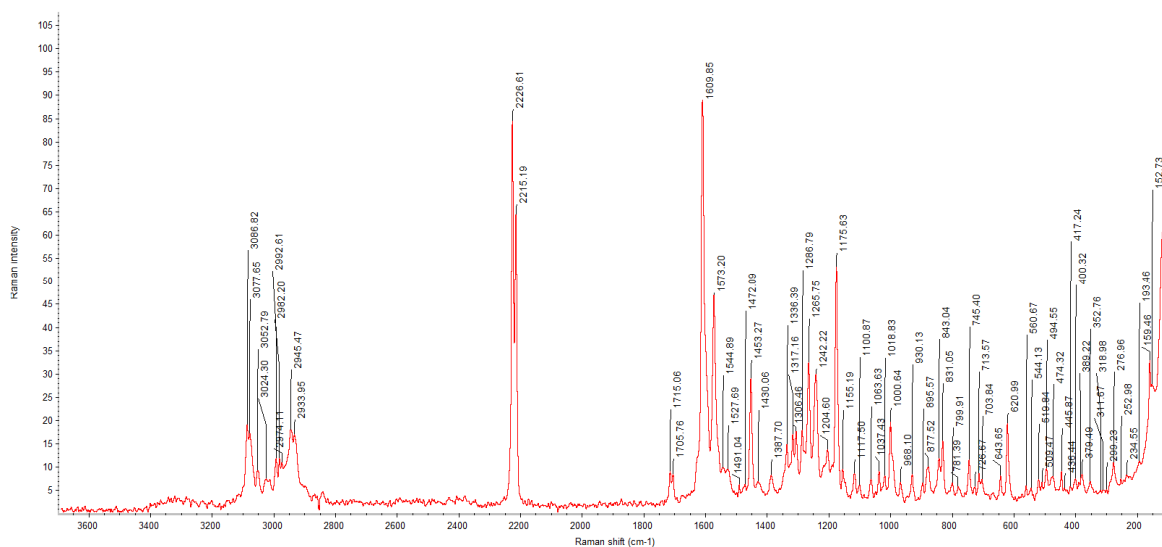


Figure S14. Raman spectrum of glasdegib dihydrochloride.



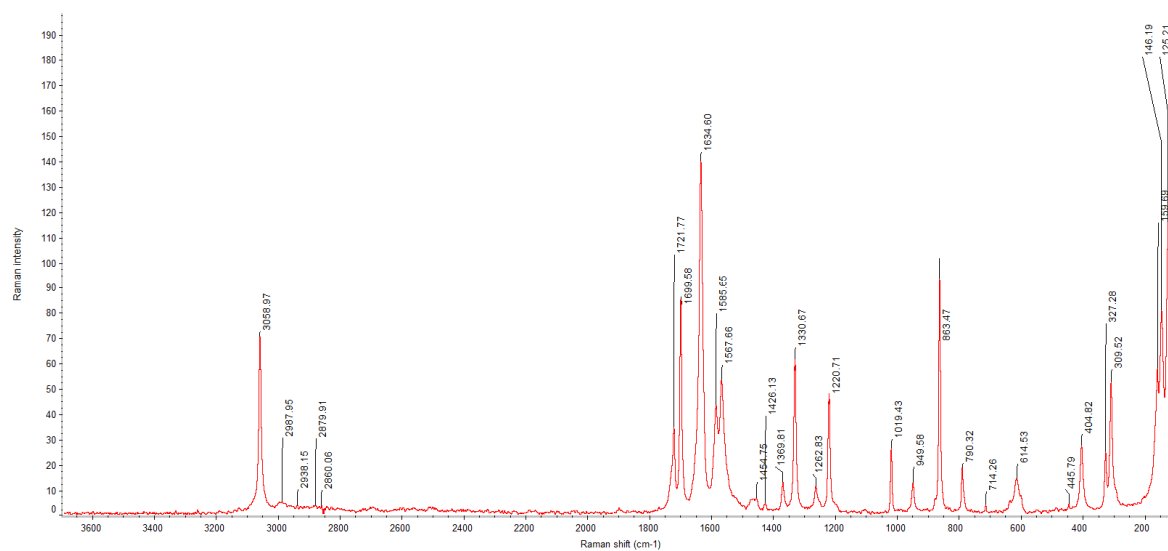
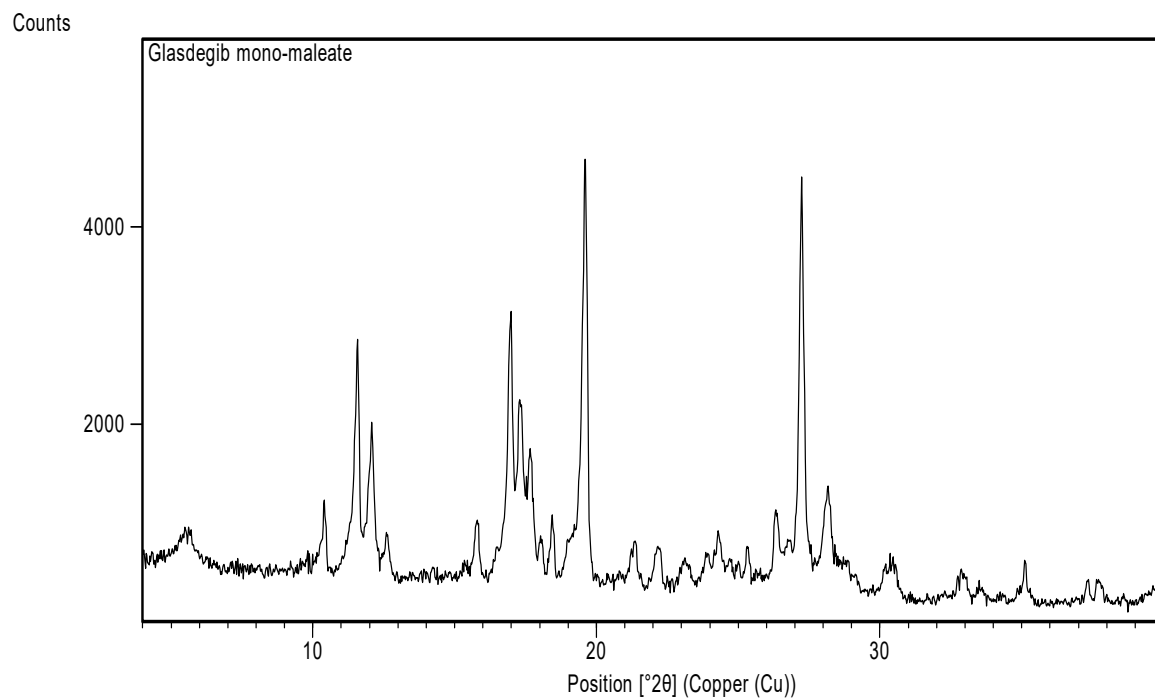
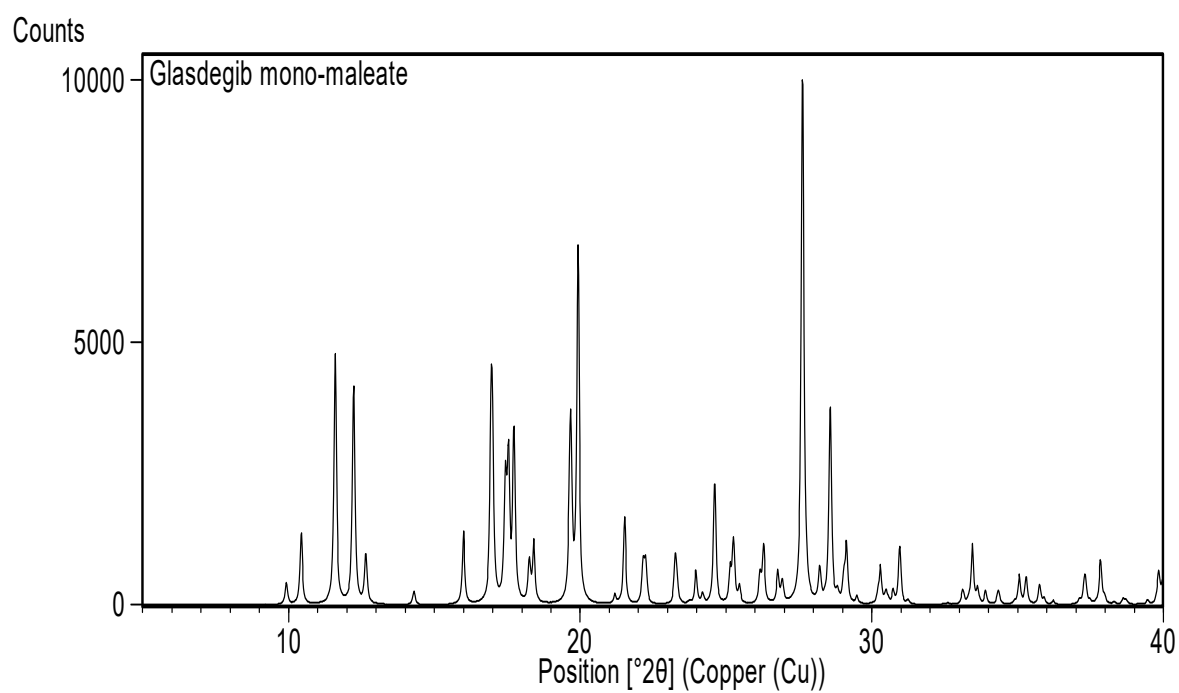


Figure S15. Raman spectrum of maleic acid.

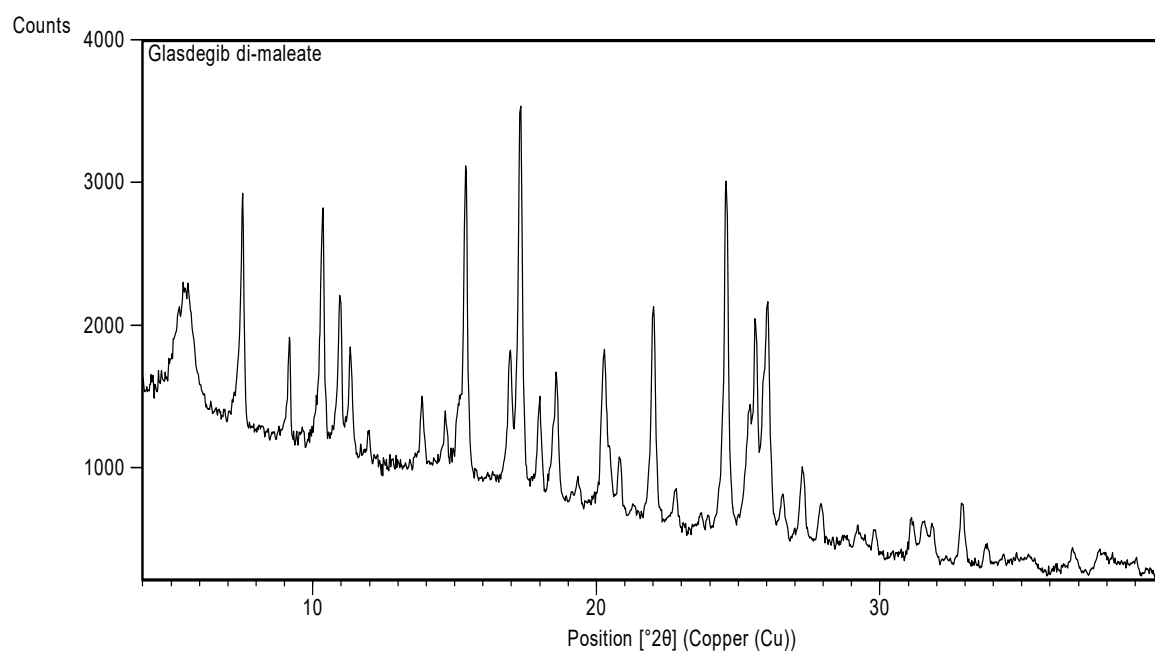
#### 4. PXRD diffractograms



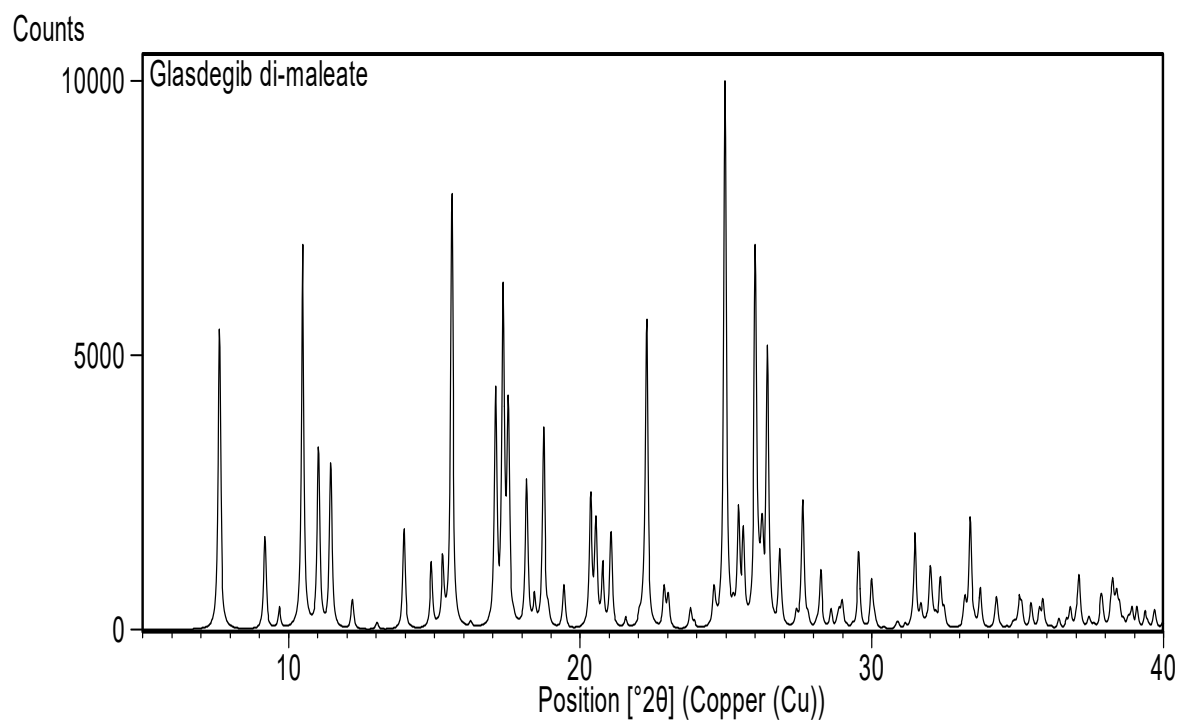
**Figure S16.** Measured PXRD pattern of glasdegib monomaleate powder.



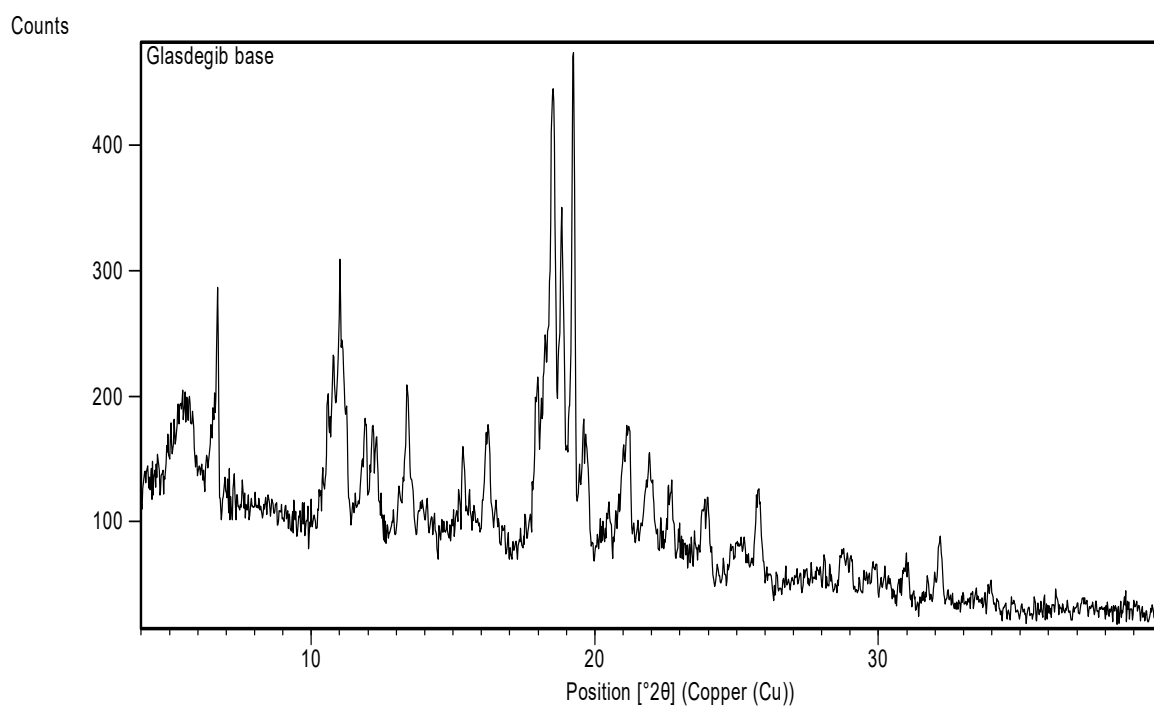
**Figure S17.** Calculated PXRD pattern from the single-crystal structure of glasdegib monomaleate.



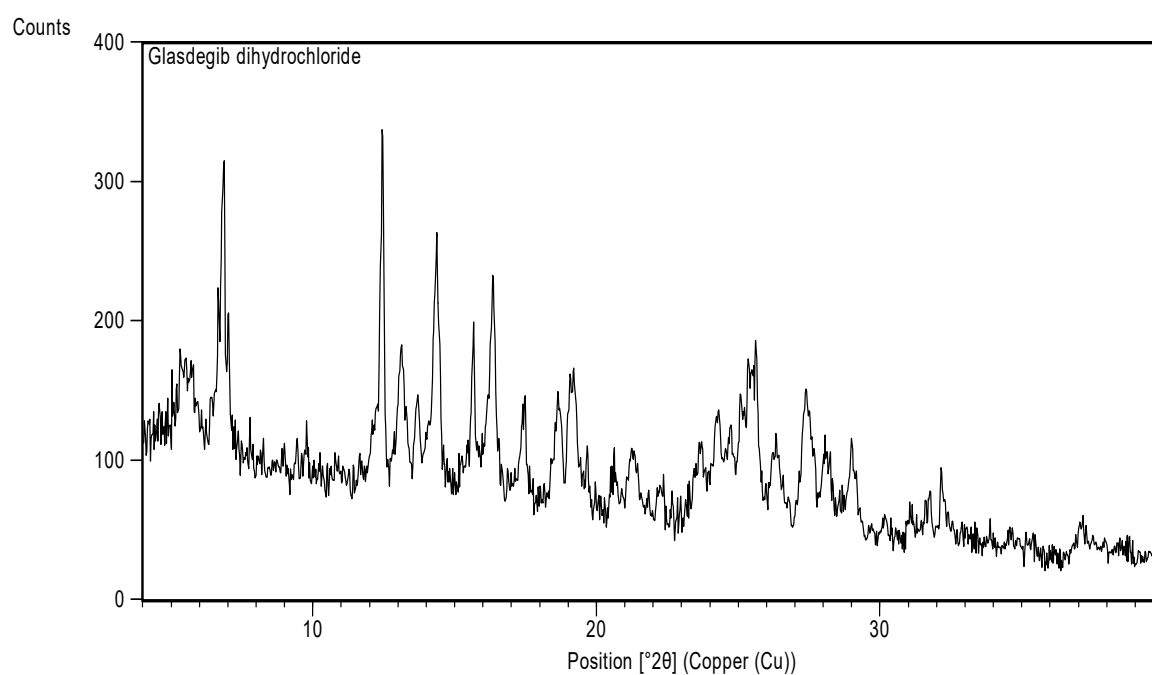
**Figure S18.** Measured PXRD pattern of glasdegib dimaleate powder.



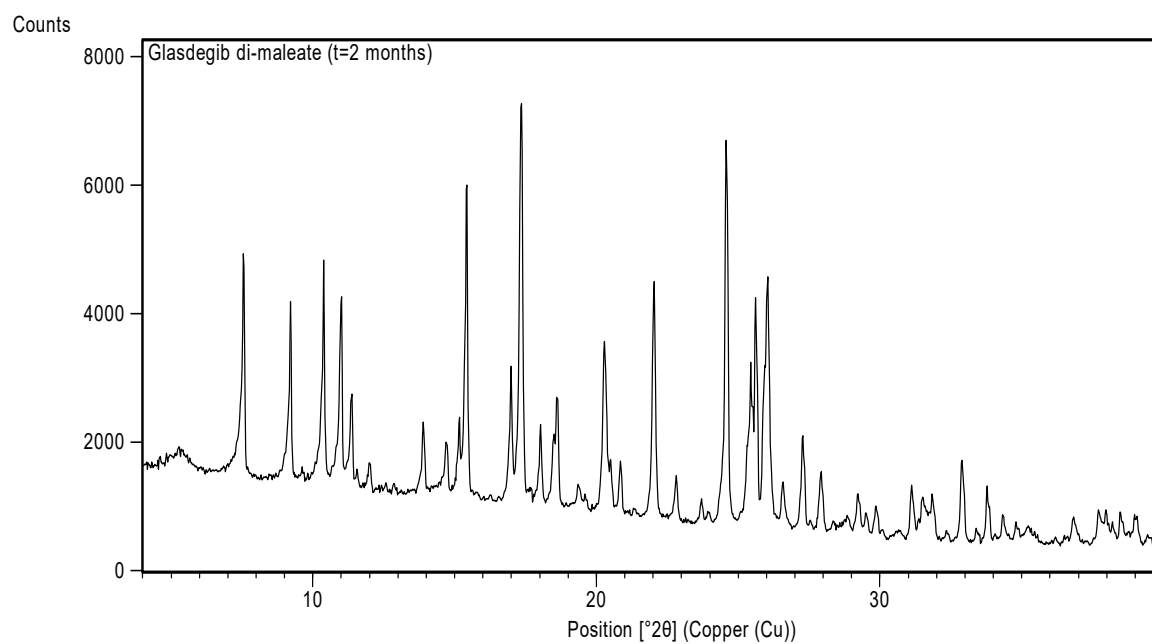
**Figure S19.** Calculated PXRD pattern from the single-crystal structure of glasdegib dimaleate.



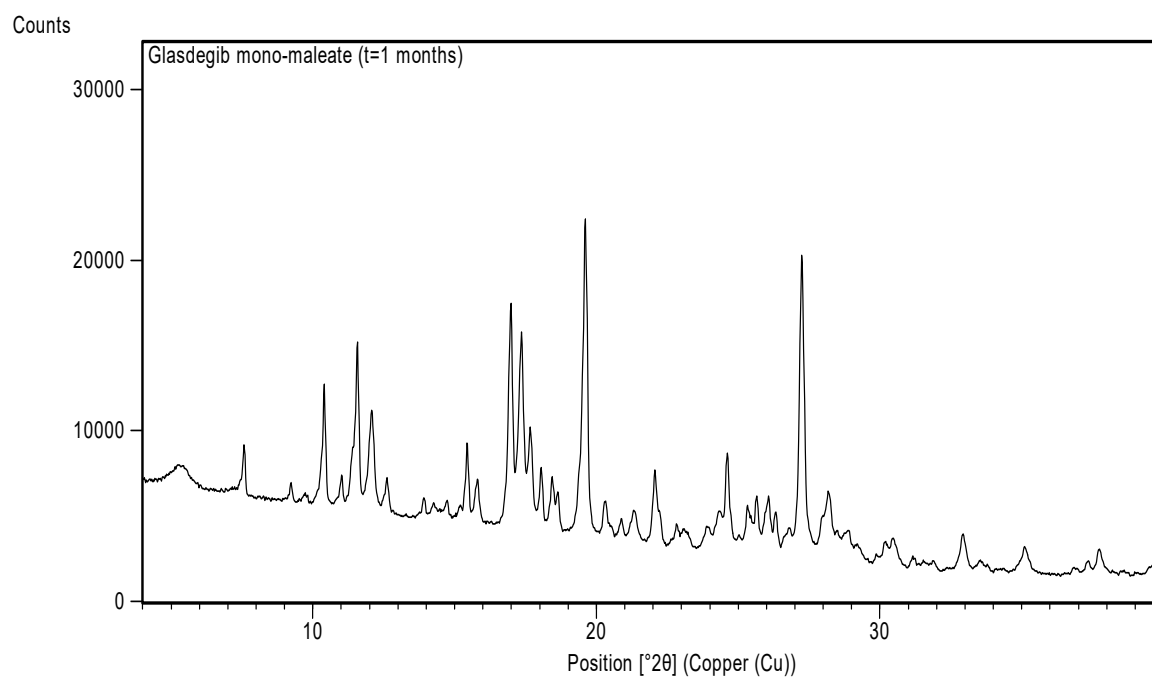
**Figure S20.** Measured PXRD pattern of glasdegib base powder.



**Figure S21.** Measured PXRD pattern of glasdegib dihydrochloride powder.

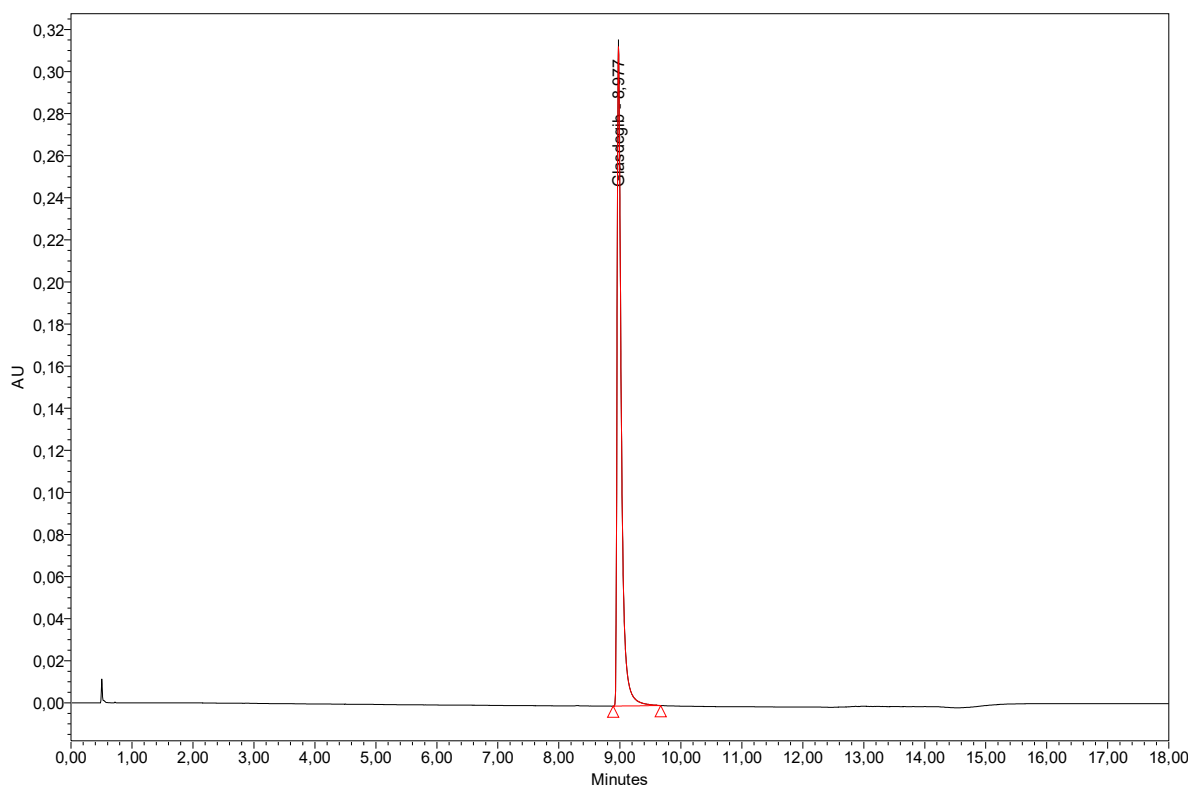


**Figure S22.** Measured PXRD pattern of glasdegib dimaleate powder exposed to stress conditions for 2 months.

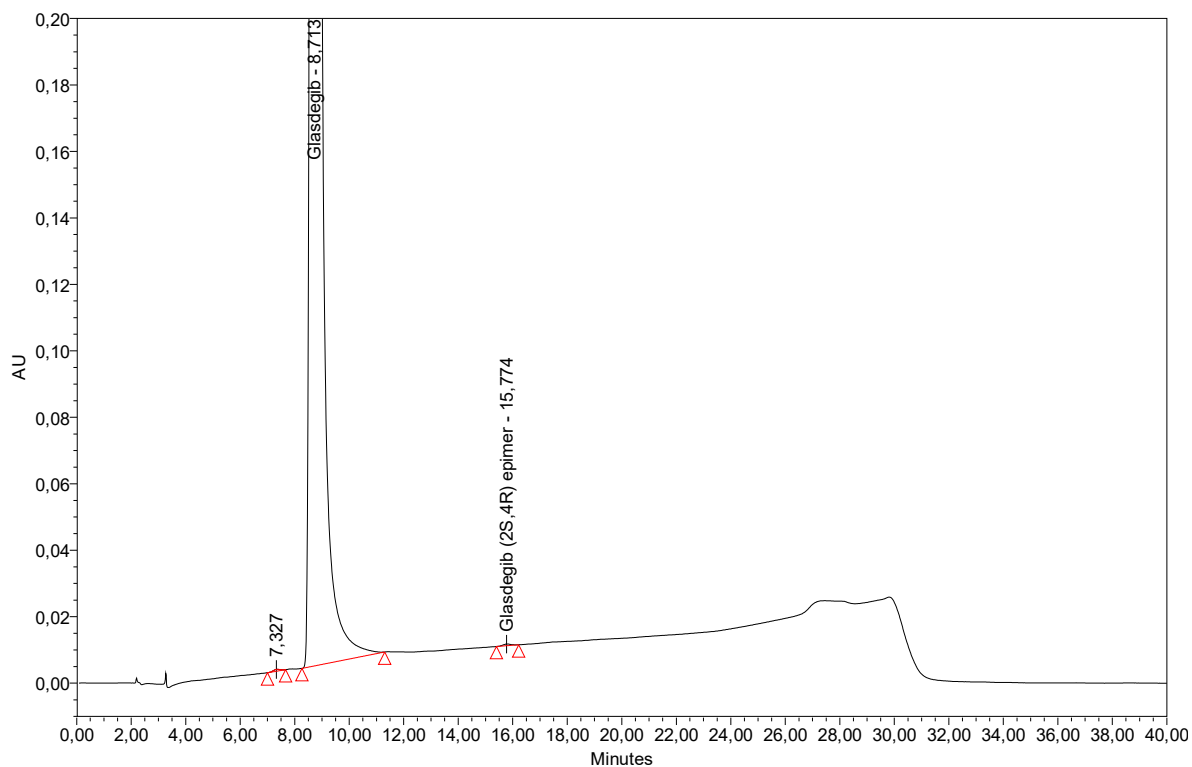


**Figure S23.** Measured PXRD pattern of glasdegib monomaleate powder exposed to stress conditions for 1 months.

## 5. LC Chromatograms



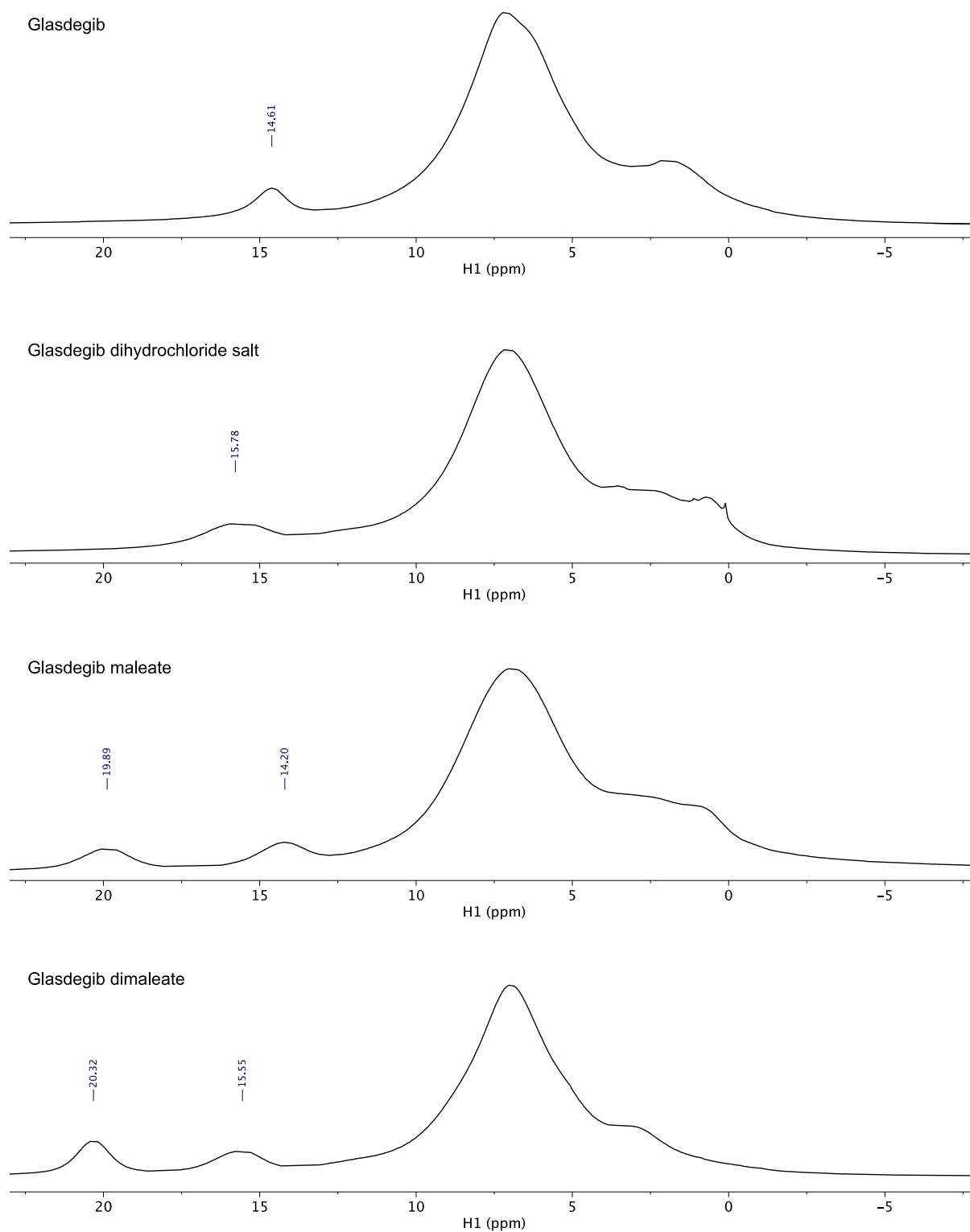
**Figure S24.** Ultra high performance liquid chromatogram of glasdegib for determination of solubility.



**Figure S25.** Chiral high performance liquid chromatogram for determination of chiral purity.

## 6. ssNMR spectra

### *<sup>1</sup>H echo MAS NMR spectra of glasdegib derivatives*



**Figure S26.** <sup>1</sup>H echo MAS NMR spectra of glasdegib base, glasdegib dihydrochloride hydrate, glasdegib monomaleate and glasdegib dimaleate.

## 7. Solution $^1\text{H}$ - and $^{13}\text{C}$ -NMR spectra of glasdegib derivatives

### 7.1. Glasdegib monomaleate

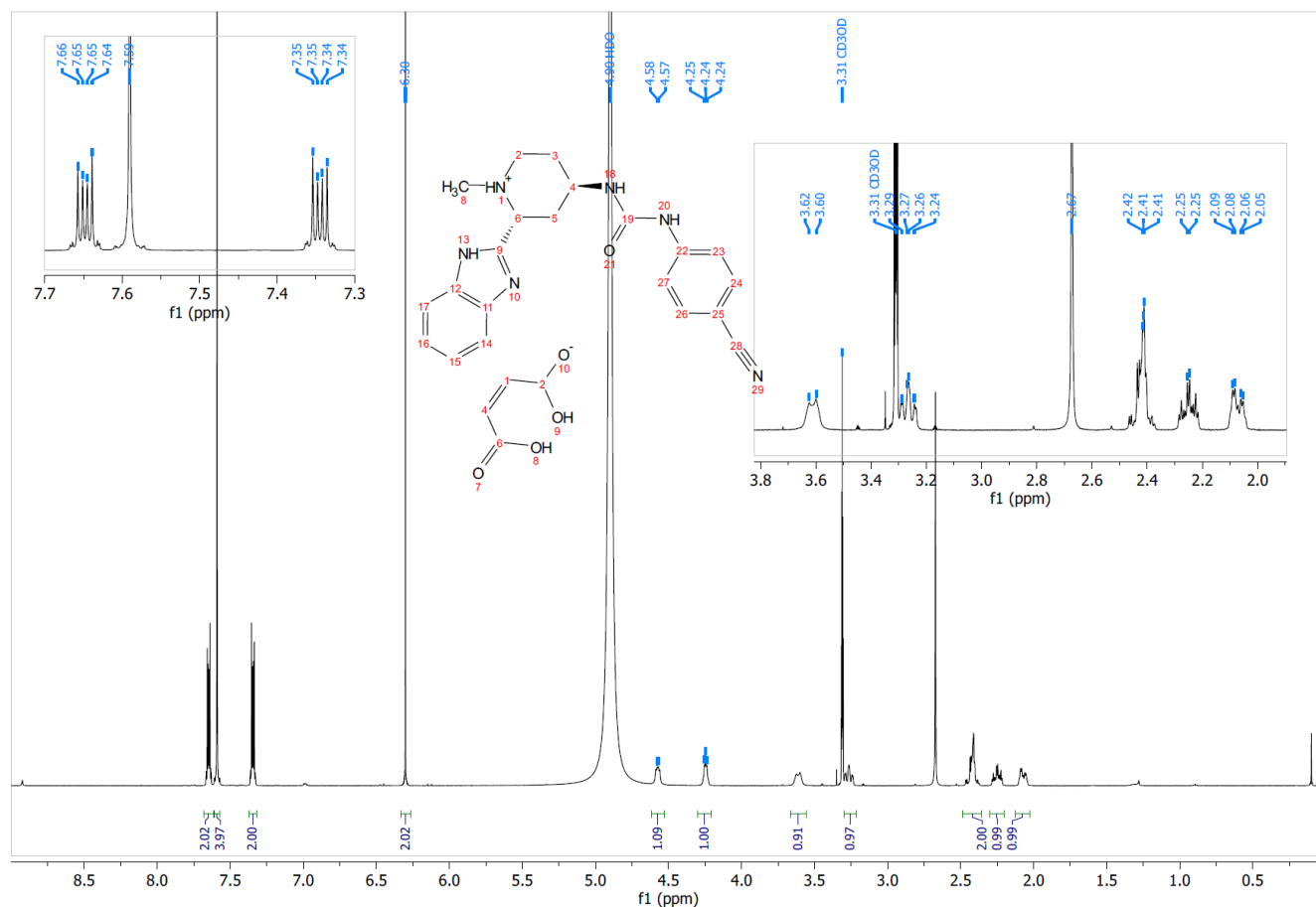


Figure S27.  $^1\text{H}$ -NMR spectrum of glasdegib monomaleate in  $\text{MeOD}$  at 500 MHz.



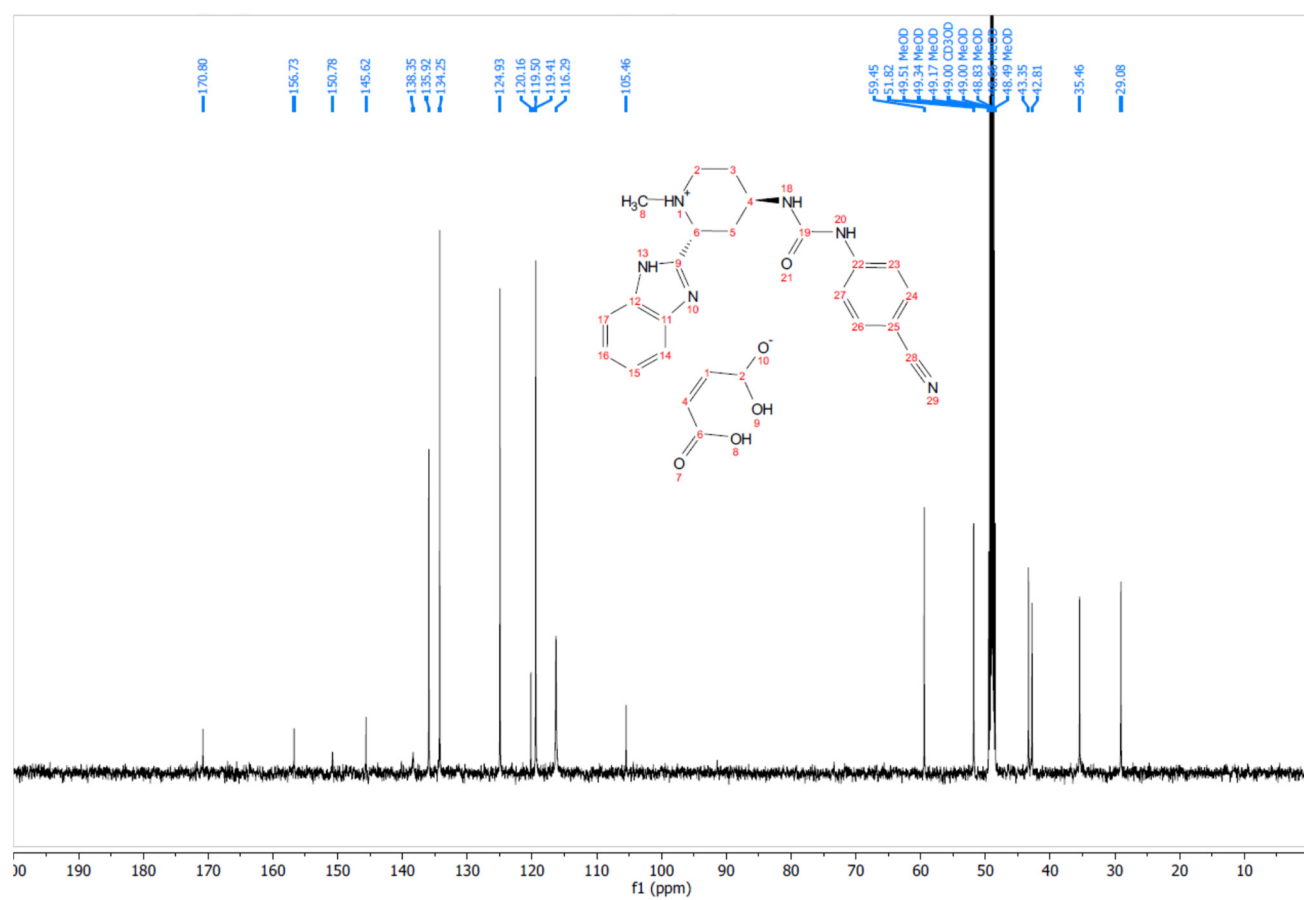


Figure S28. <sup>13</sup>C-NMR spectrum of glasdegib monomaleate in MeOD at 125 MHz.

## 7.2. Glasdegib dimaleate

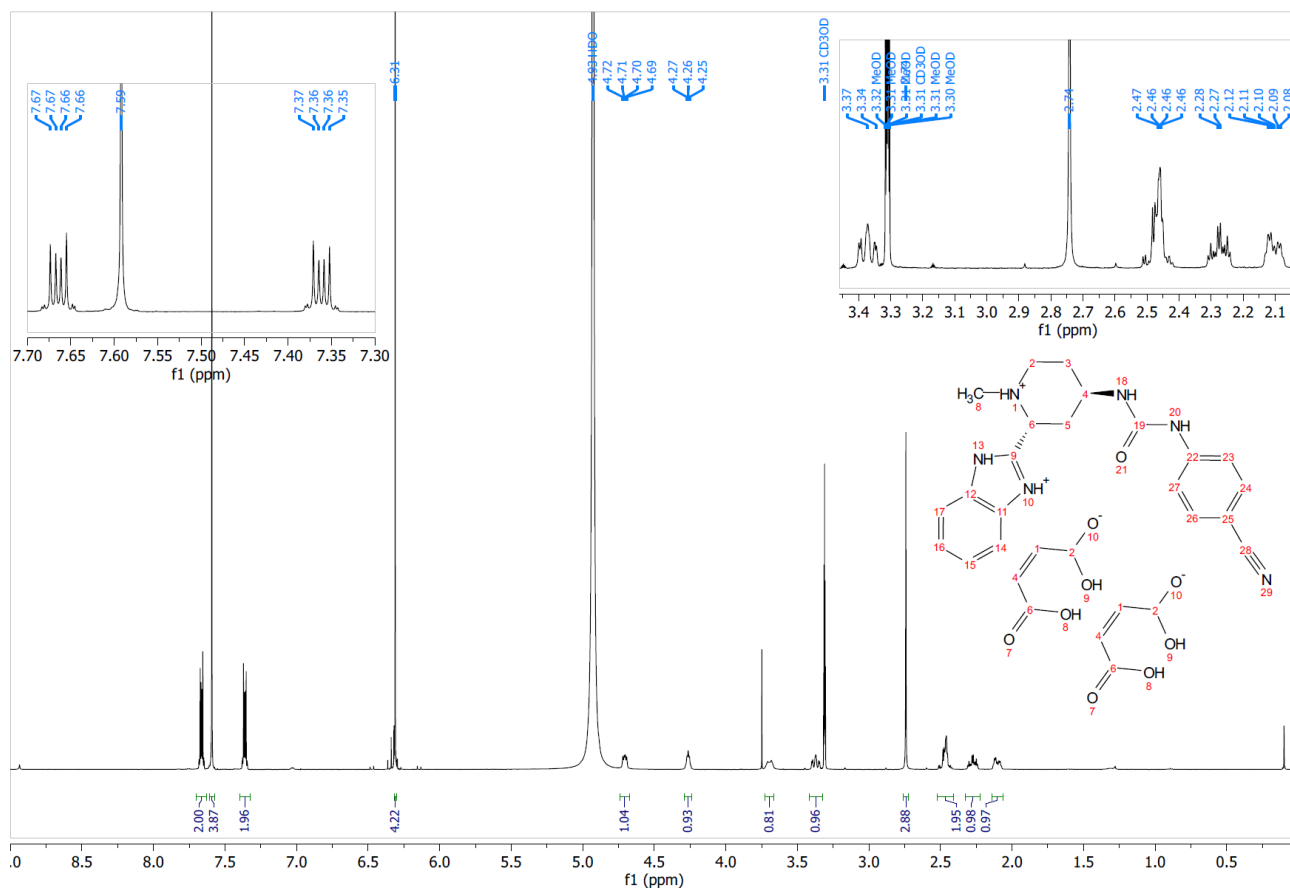


Figure S29. <sup>1</sup>H-NMR spectrum of glasdegib dimaleate in MeOD at 500 MHz.

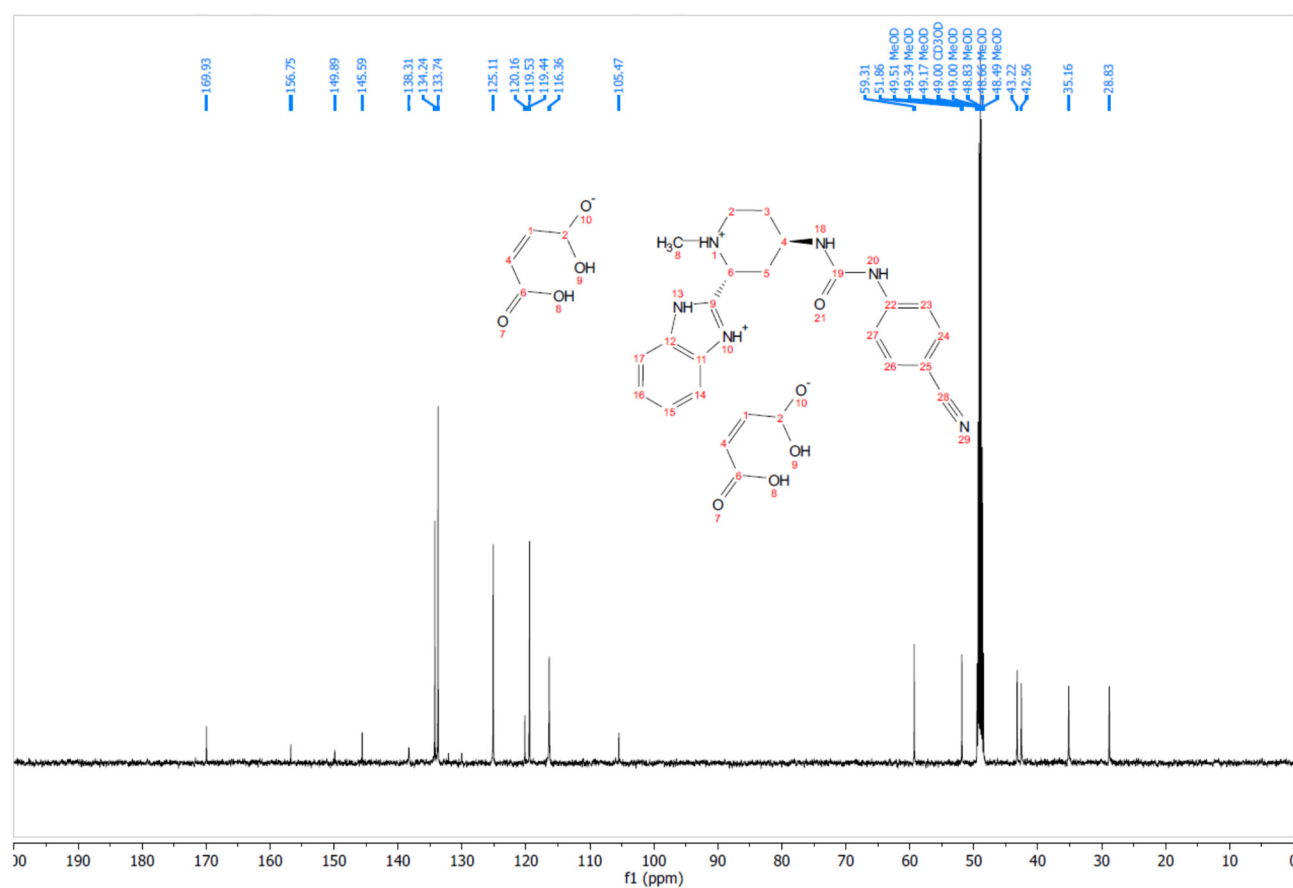


Figure S30.  $^{13}\text{C}$ -NMR spectrum of glasdegib dimaleate in MeOD at 125 MHz.

## 8. Calculated $\text{pK}_a$ data

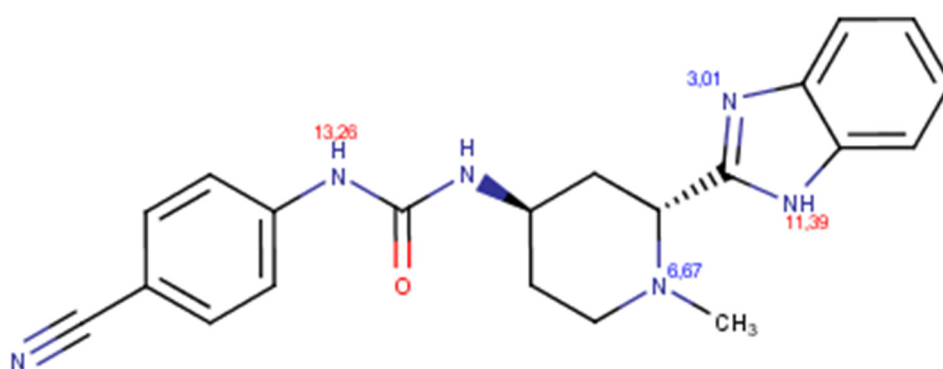
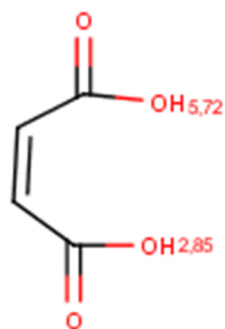


Figure S31. MarvinSketch 17.28 calculated  $\text{pK}_a$  values for glasdegib.



**Figure S32.** MarvinSketch 17.28 calculated  $pK_a$  values for maleic acid.



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