

Supporting Information for

Transition-Metal-Free Synthesis of C-Glycosylated Phenanthridines *via* K₂S₂O₈-Mediated Oxidative Radical Decarboxylation of Uronic Acids

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X-ray crystal structure of 4c

The single crystal of **4c** was prepared by slow evaporation of its solution in EtOH/dichloromethane. The structure of **4c** was established by X-ray analysis of its crystal. Thermal ellipsoids are set at 50% probability.

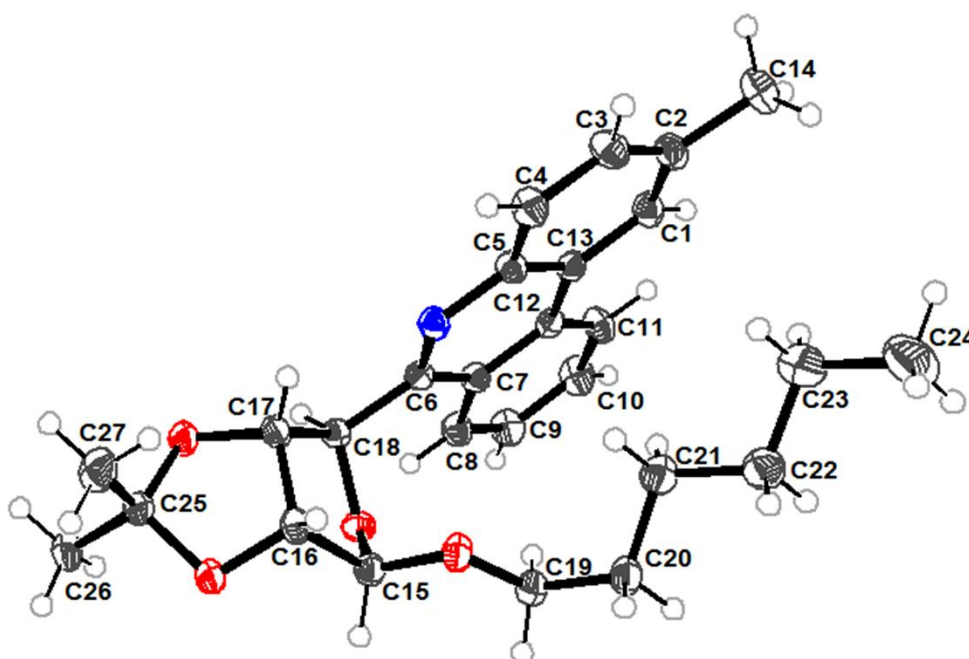


Figure S1. ORTEP diagrams of **4c**.

X-ray crystal structure of 7b'

The single crystal of **7b'** was prepared by slow evaporation of its solution in EtOH/dichloromethane. The structure of **7b'** was established by X-ray analysis of its crystal. Thermal ellipsoids are set at 50% probability.

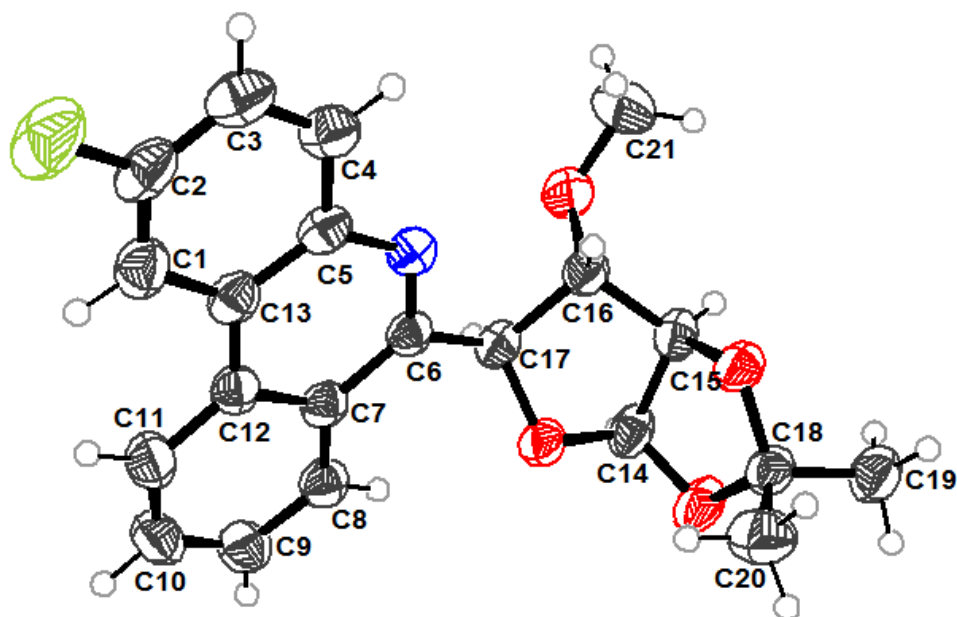


Figure S2. ORTEP diagrams of 7b'.

X-ray crystal structure of 7d'

The single crystal of **7d'** was prepared by slow evaporation of its solution in EtOH/dichloromethane. The structure of **7d'** was established by X-ray analysis of its crystal. Thermal ellipsoids are set at 50% probability.

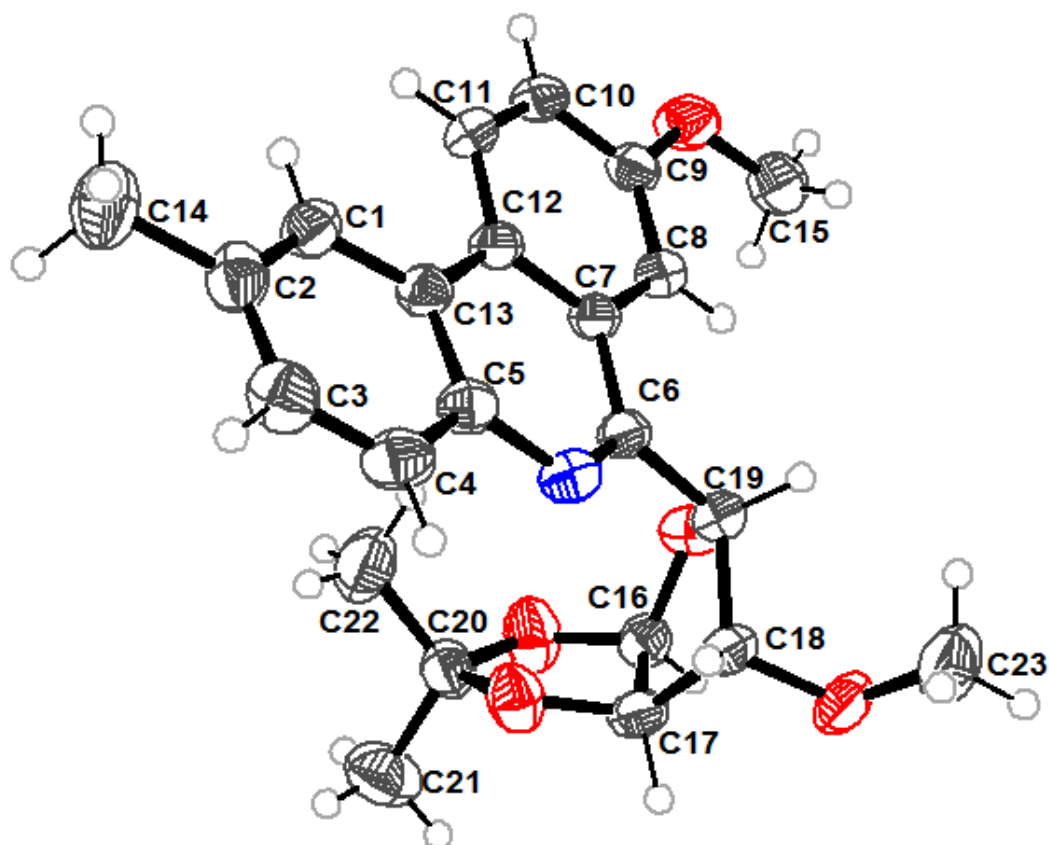
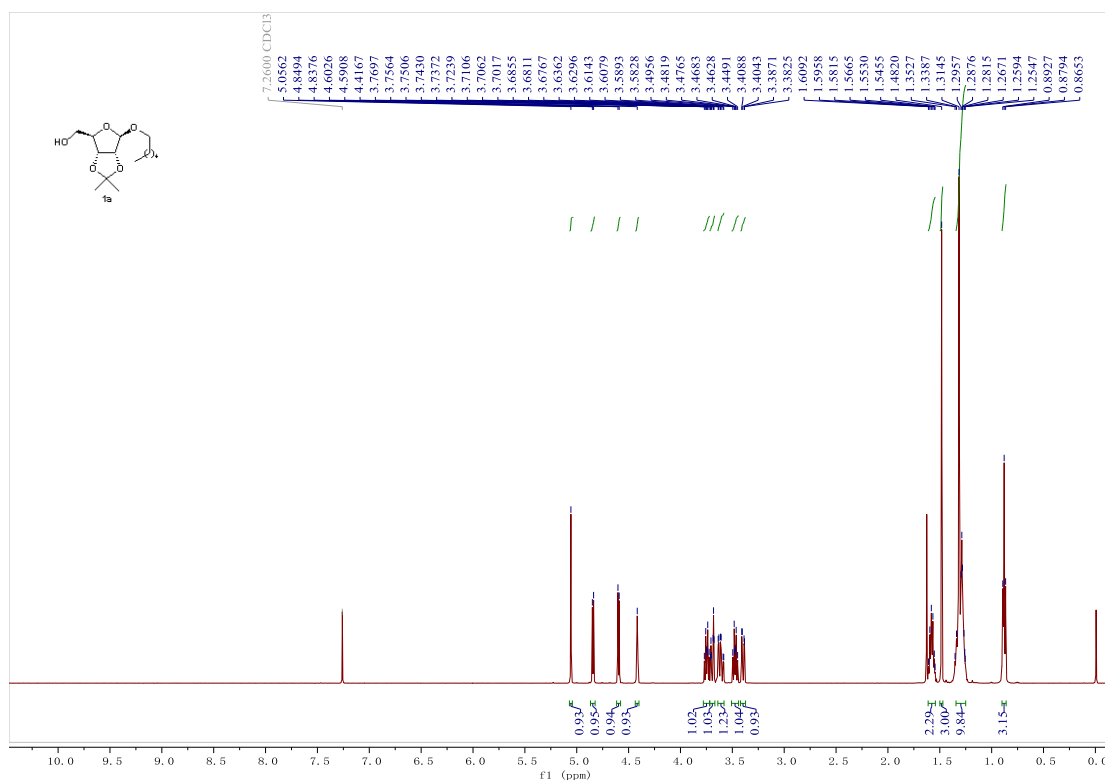
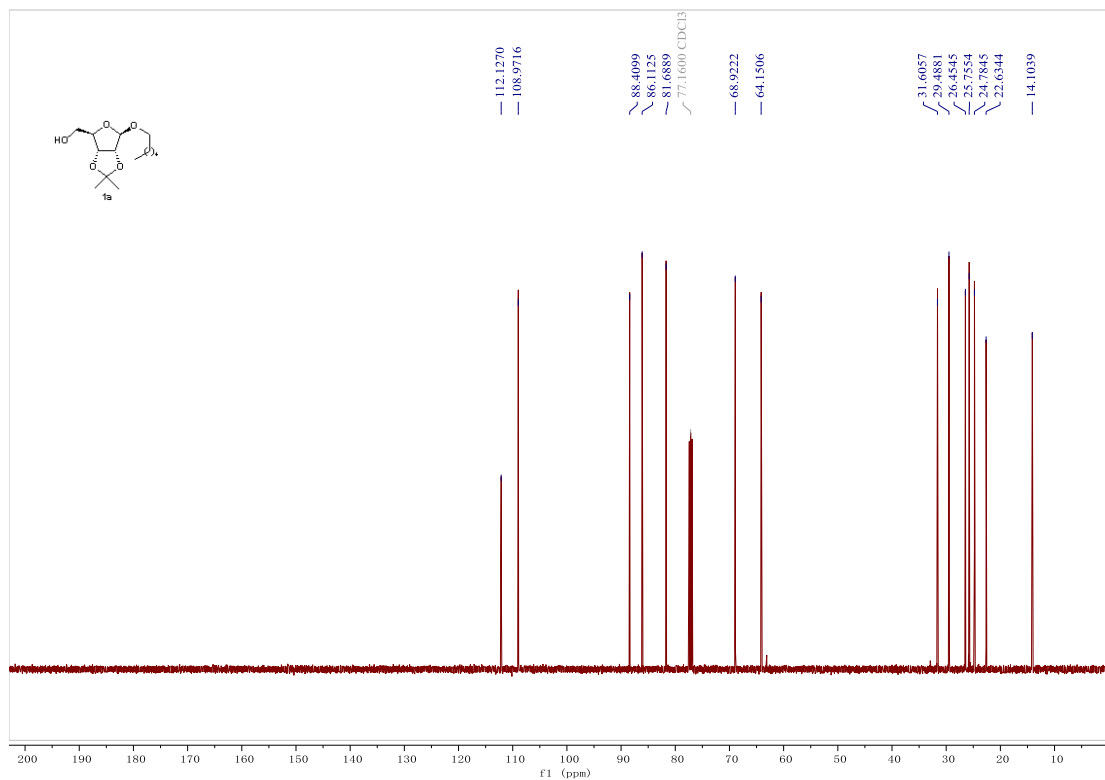


Figure S3. ORTEP diagrams of 7d'.

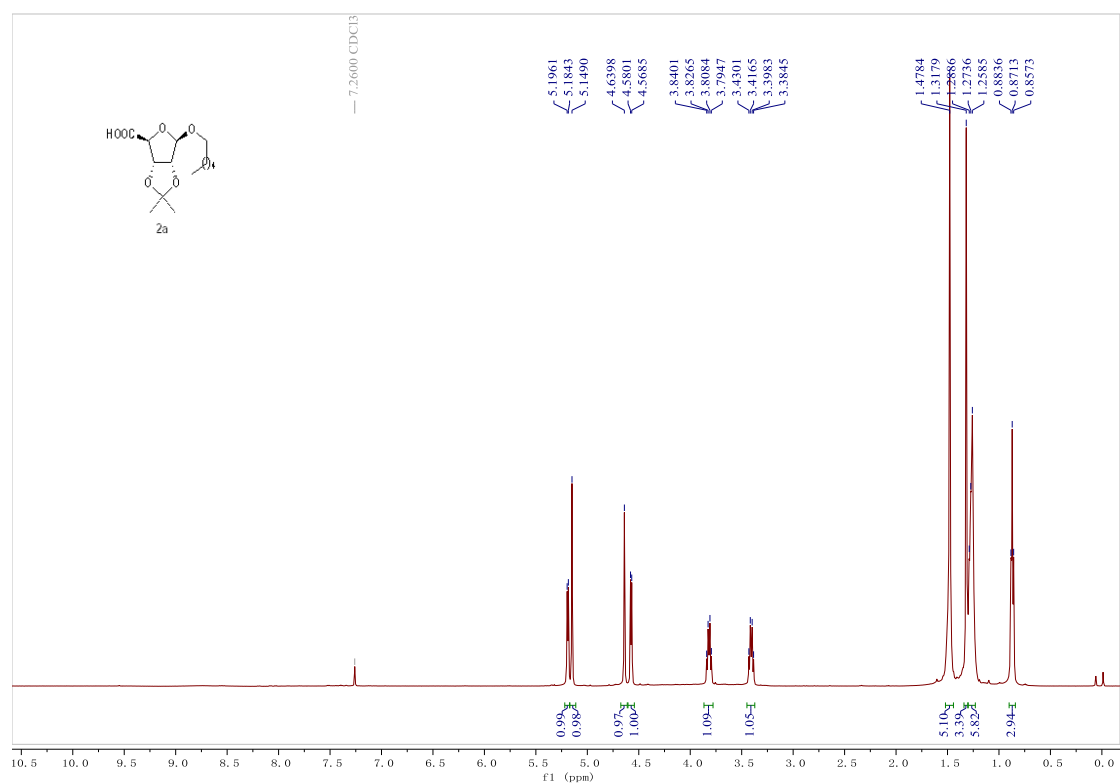
NMR spectra



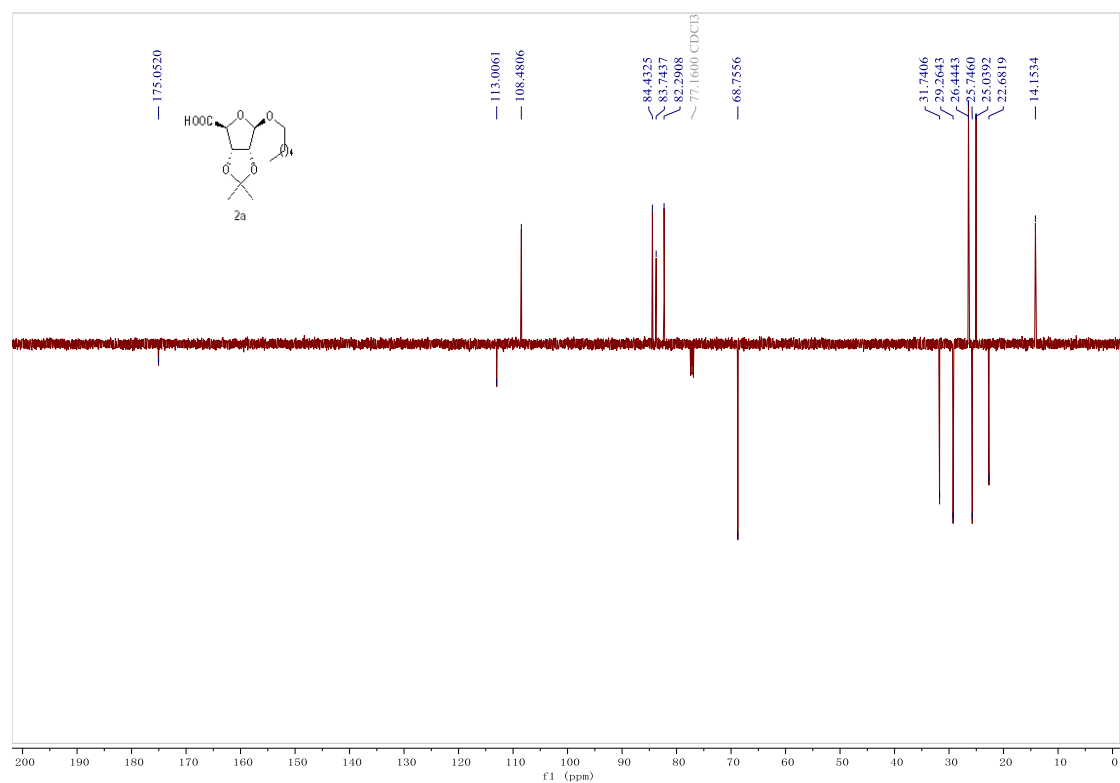
¹H NMR spectrum for 1a (500 MHz, CDCl₃)



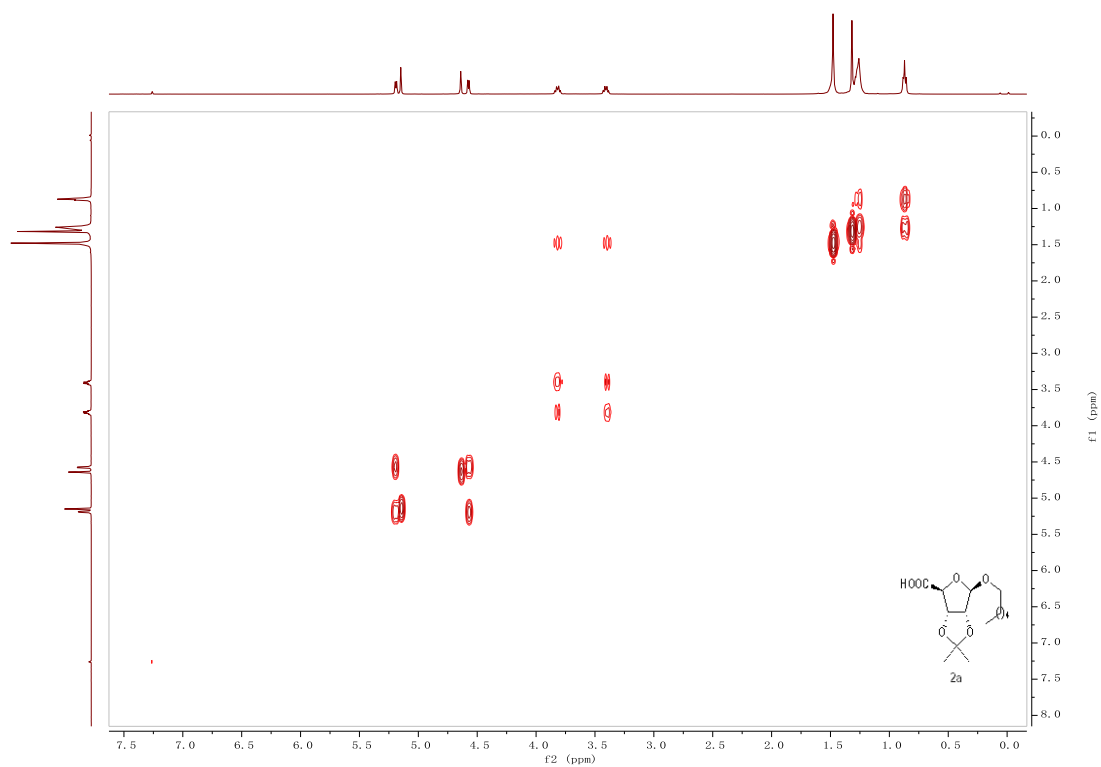
¹³C NMR spectrum for 1a (126 MHz, CDCl₃)



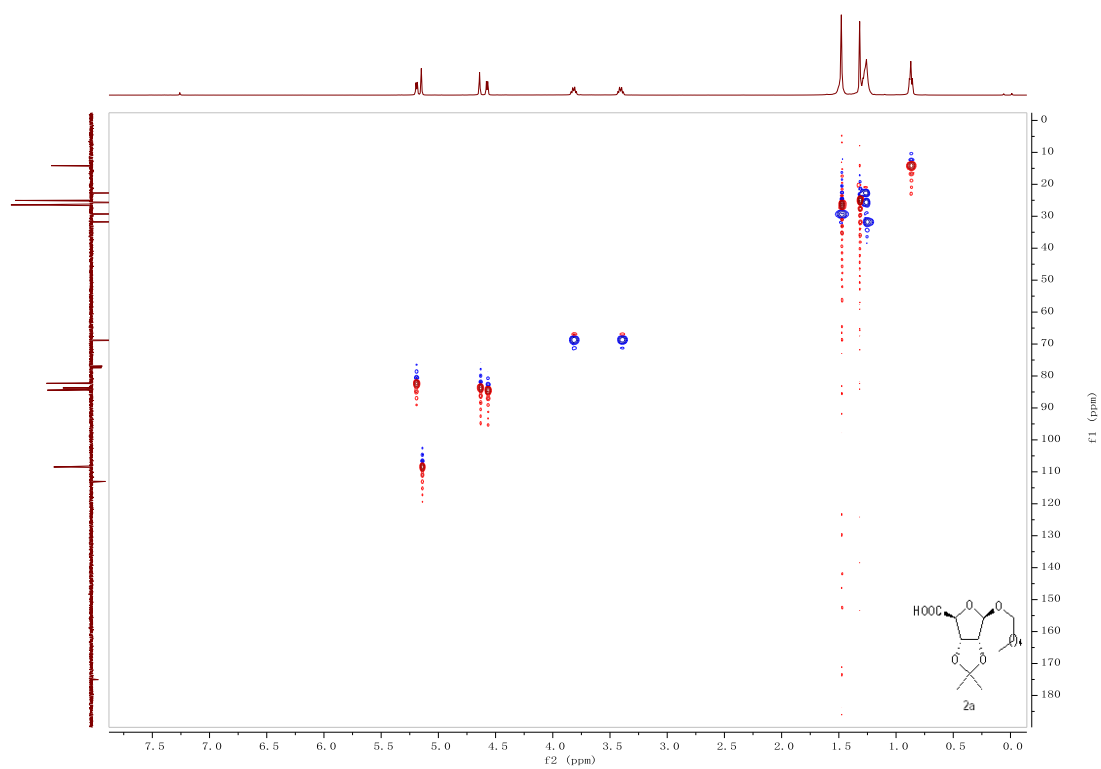
¹H NMR spectrum for **2a** (500 MHz, CDCl₃)



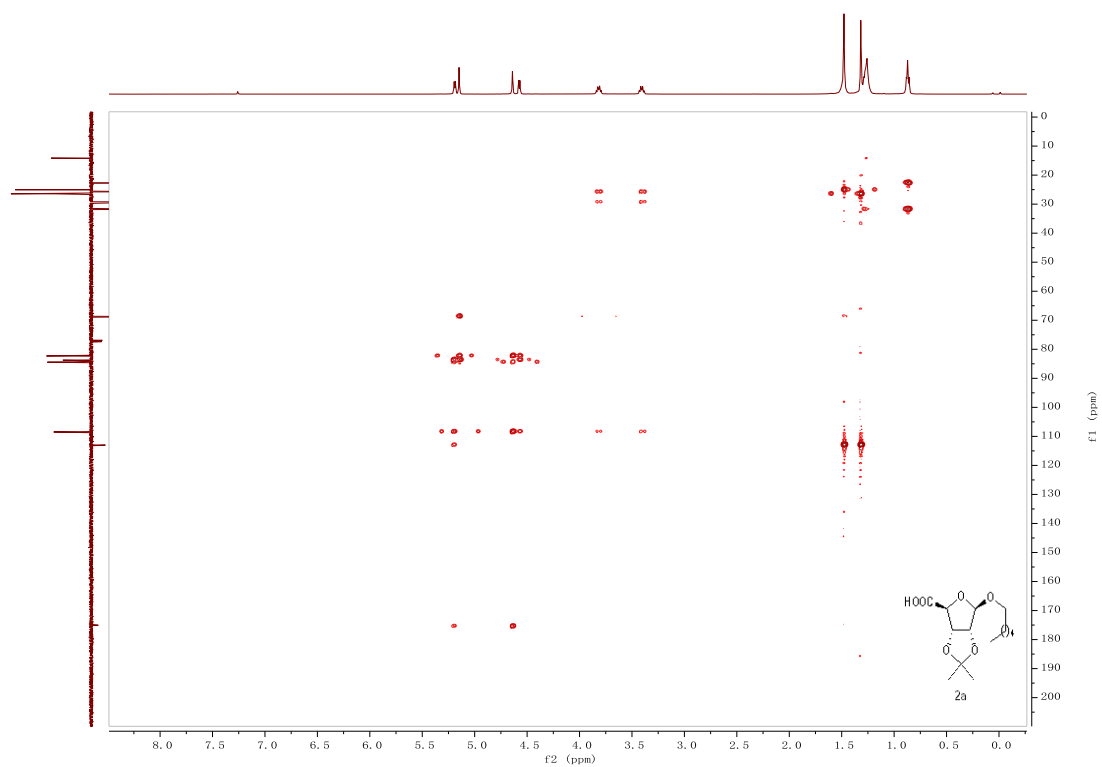
DEPT-Q spectrum for **2a** (126 MHz, CDCl₃)



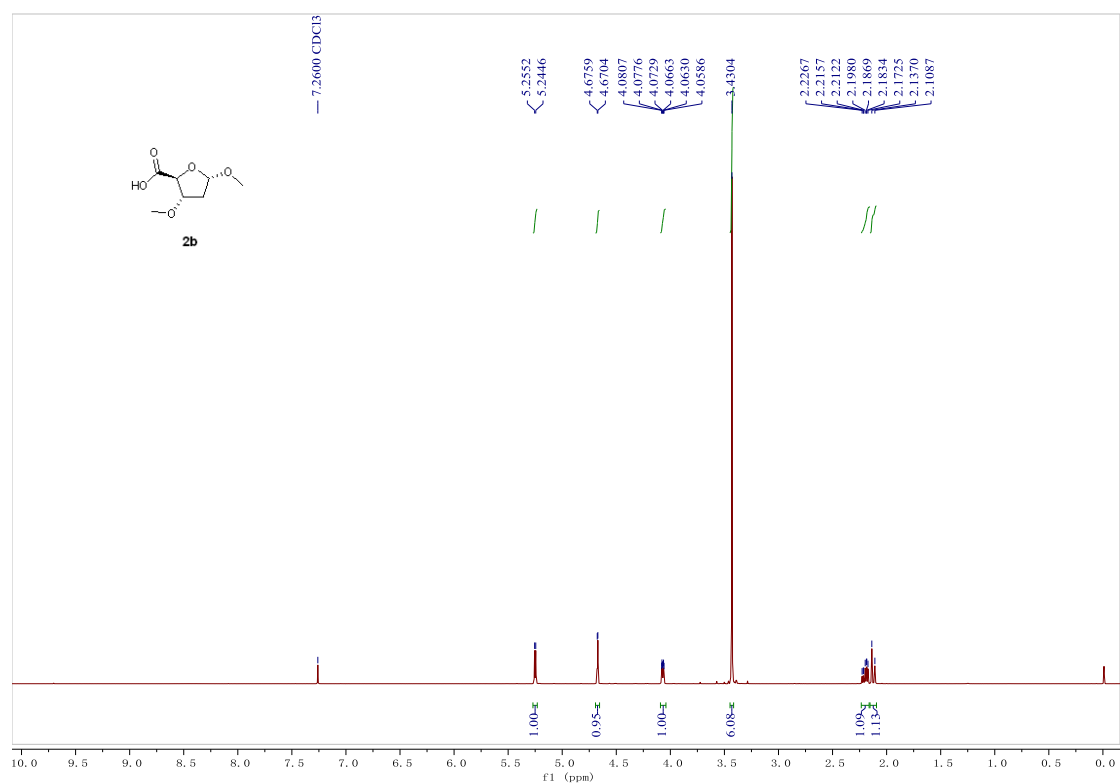
COSY spectrum for 2a



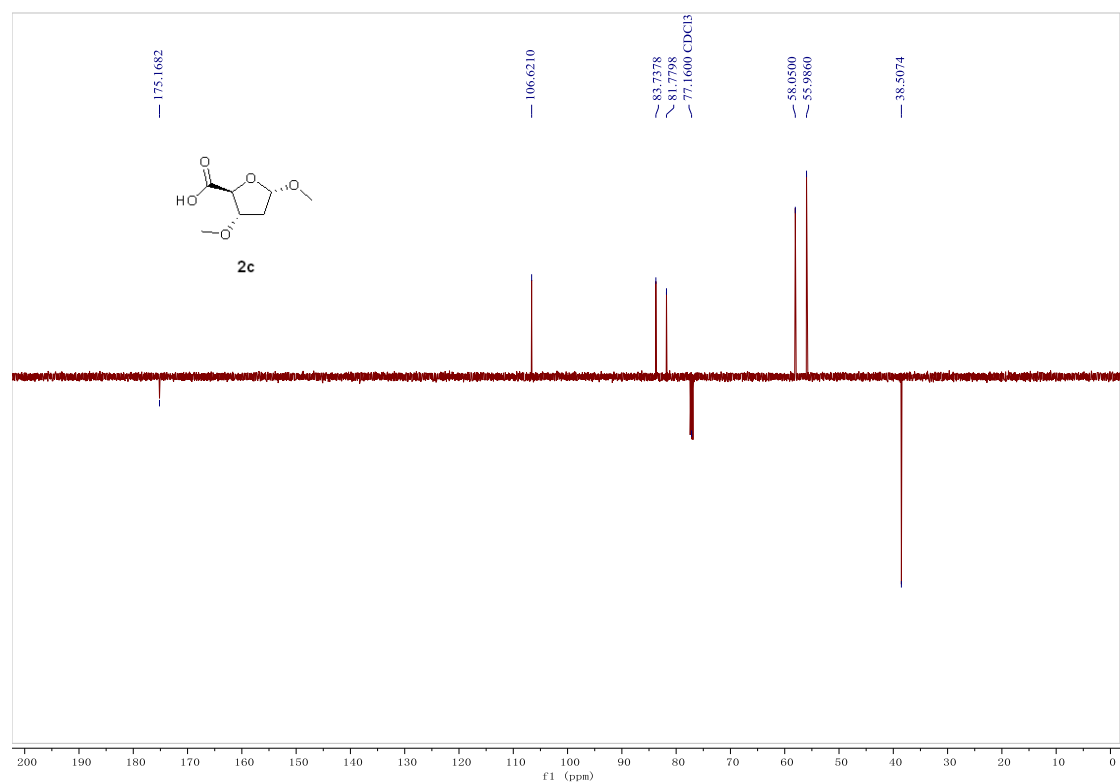
HSQC spectrum for 2a



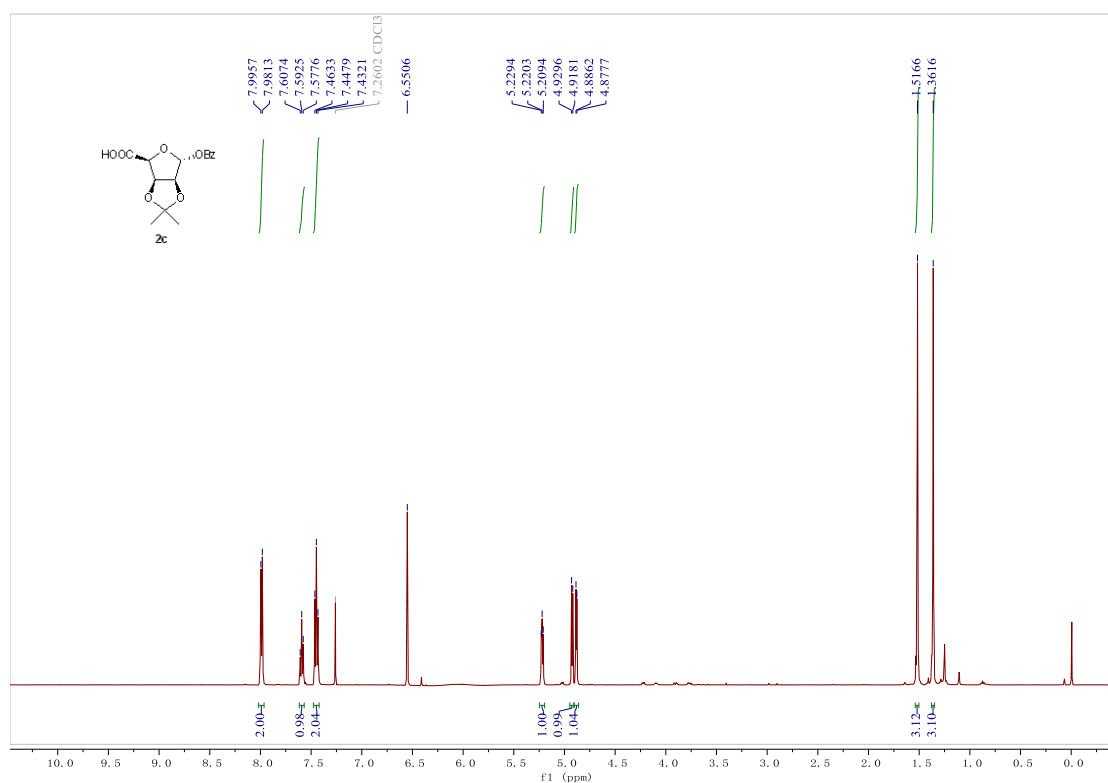
HMBC spectrum for **2a**



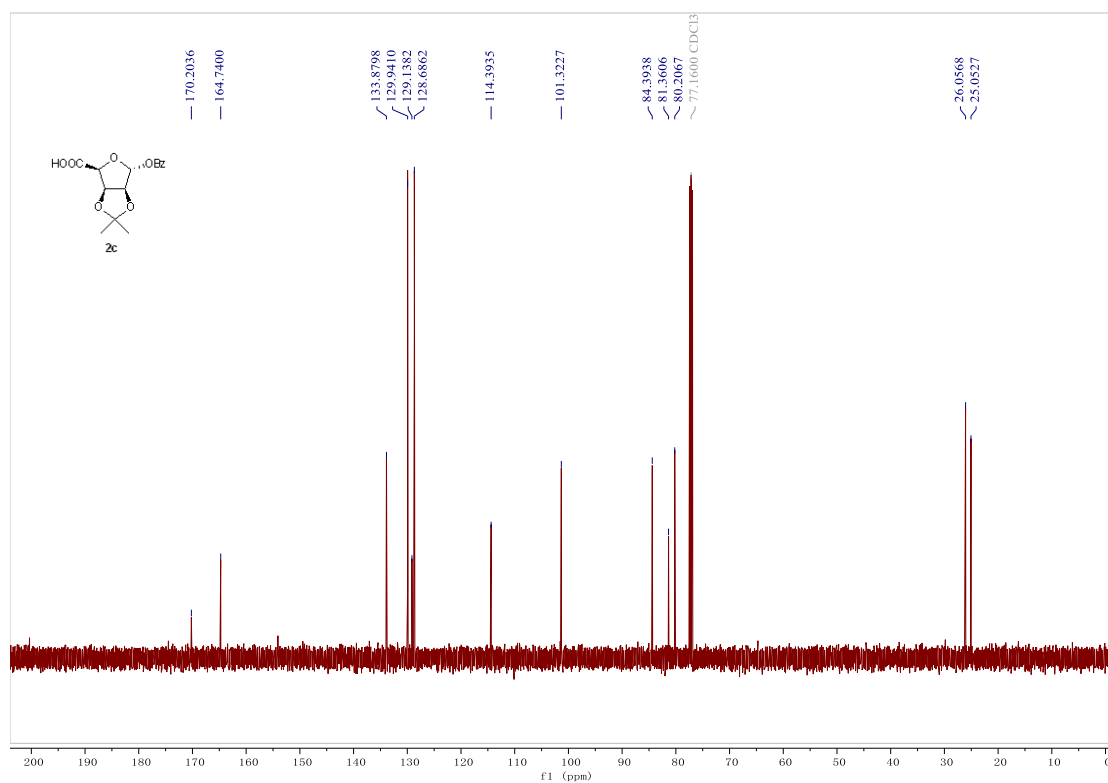
¹H NMR spectrum for **2b** (500 MHz, CDCl₃)



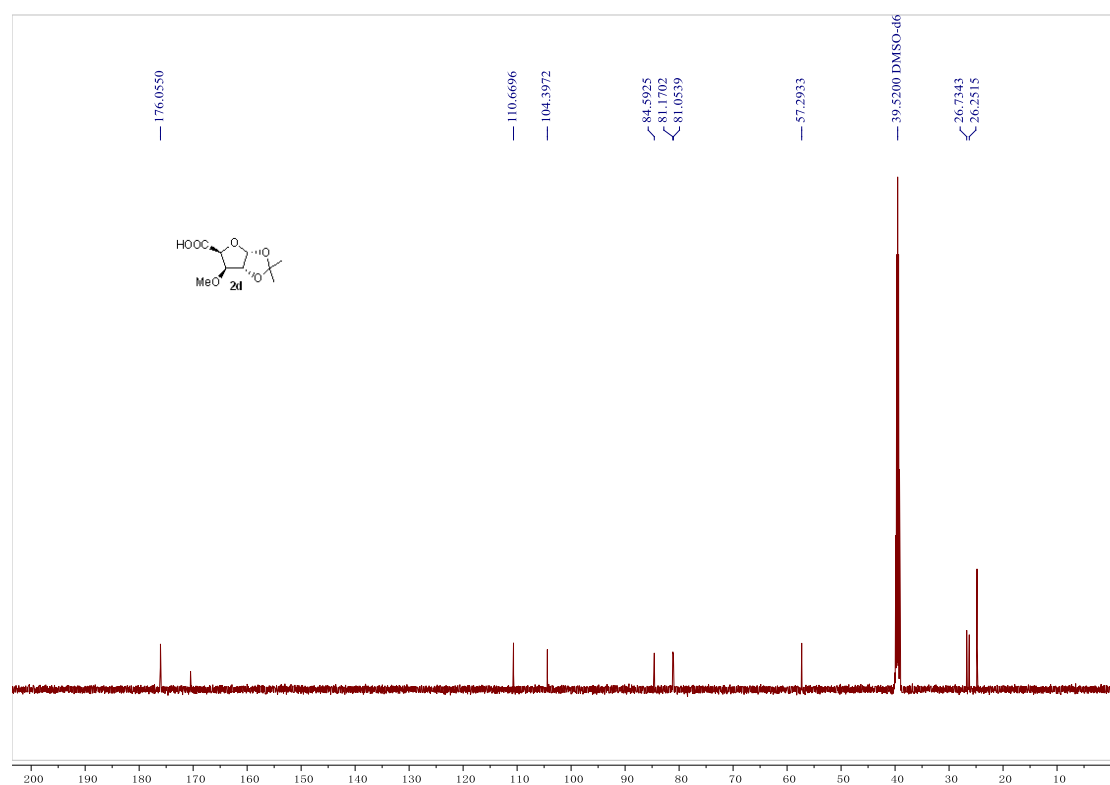
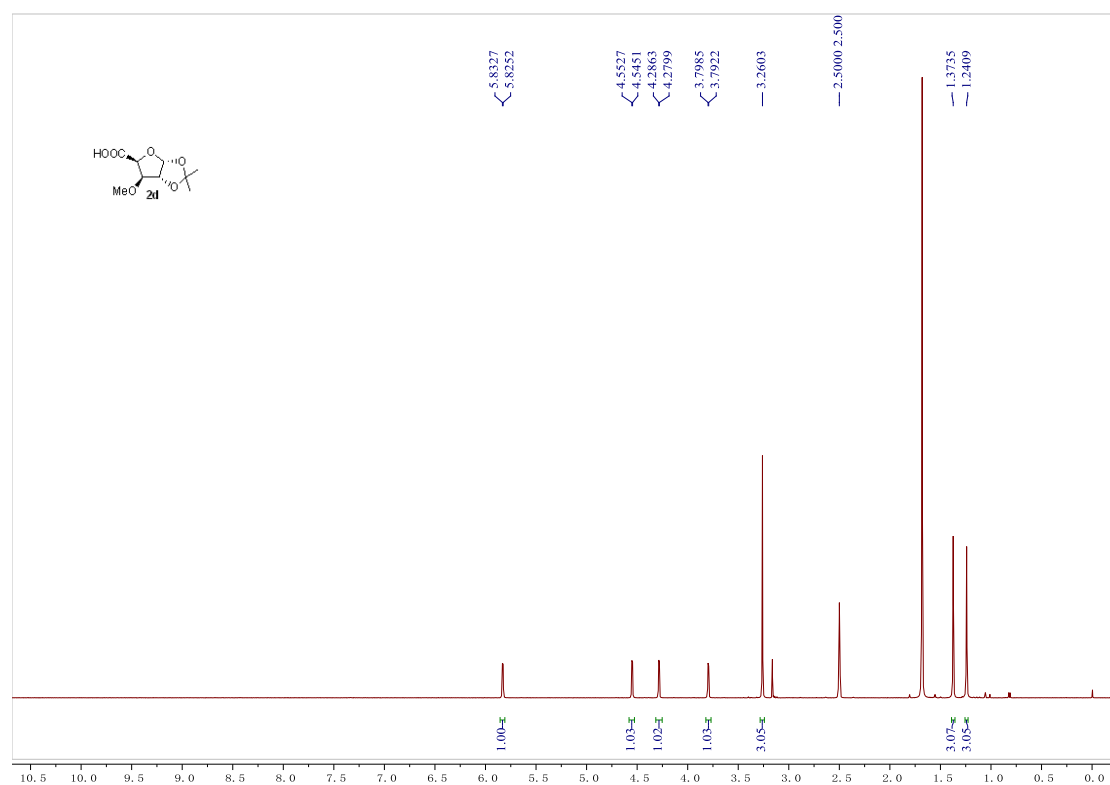
DEPT-Q spectrum for **2b** (126 MHz, CDCl₃)



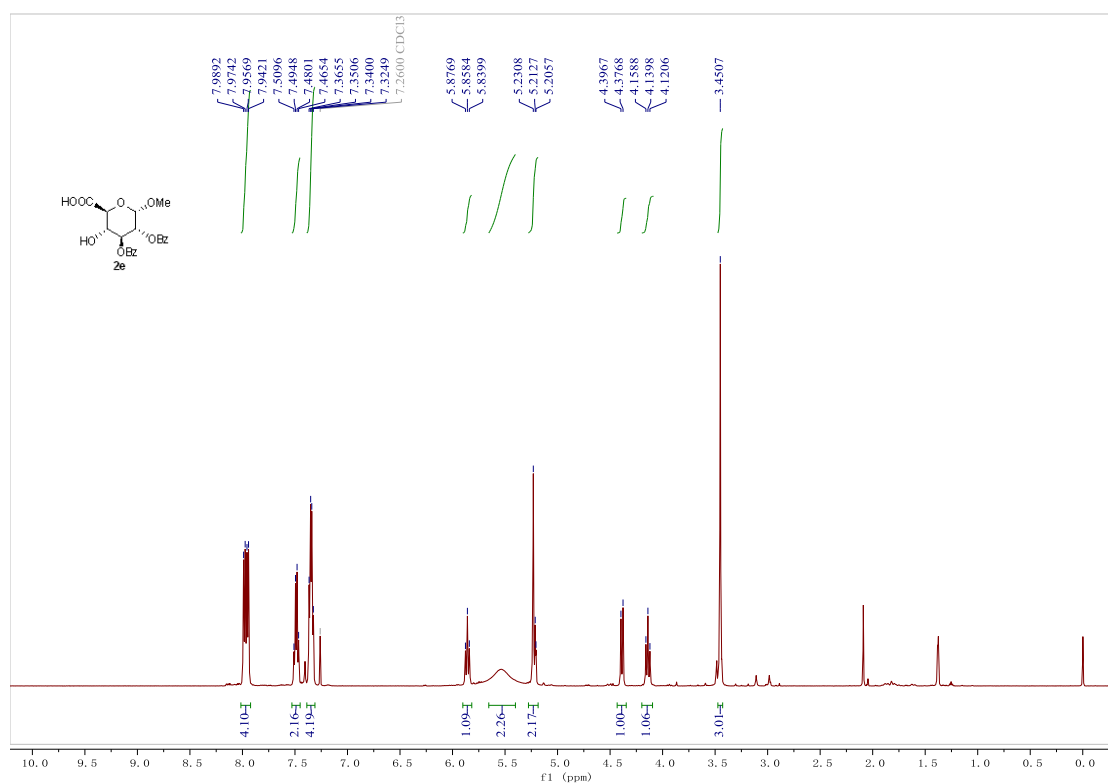
¹H NMR spectrum for 2c (500 MHz, CDCl₃)



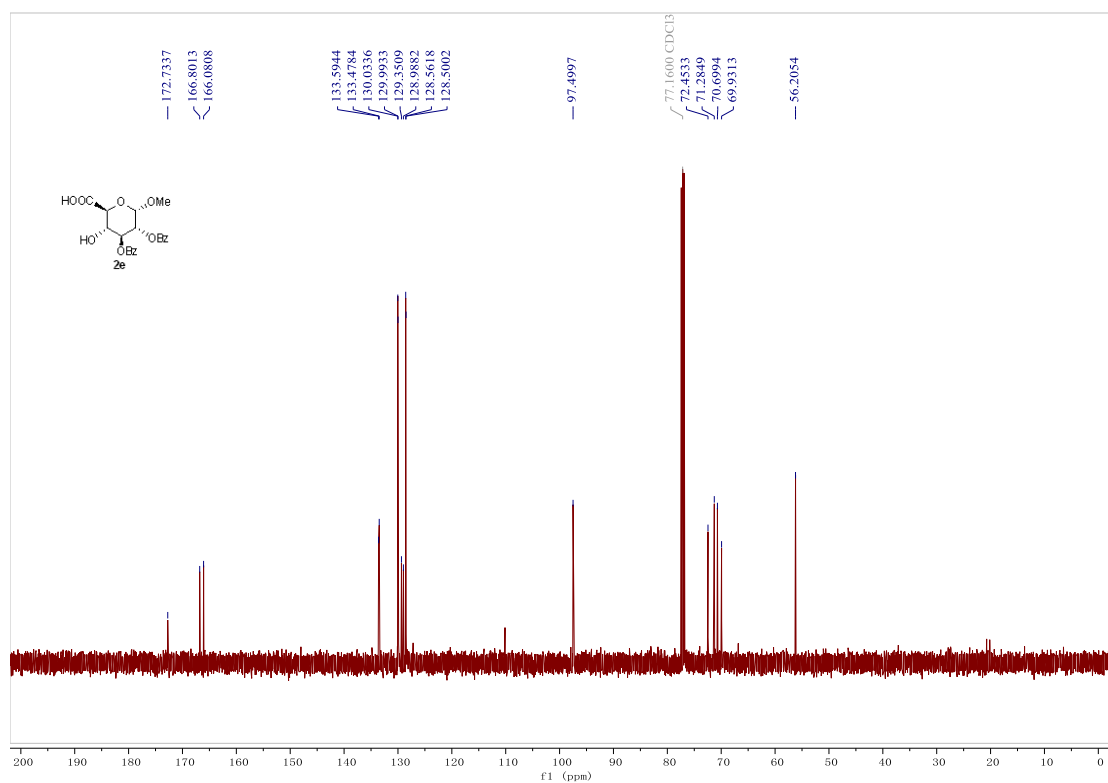
¹³C NMR spectrum for 2c (126 MHz, CDCl₃)



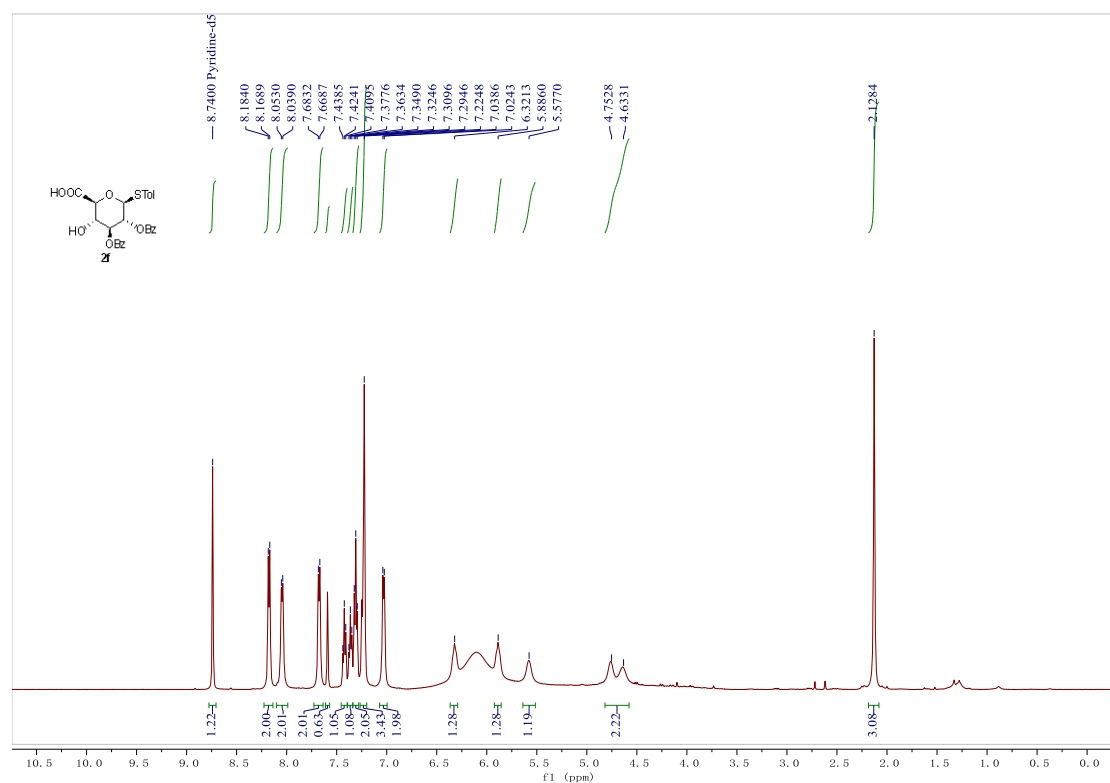
¹³C NMR spectrum for **2d** (126 MHz, DMSO-*d*₆)



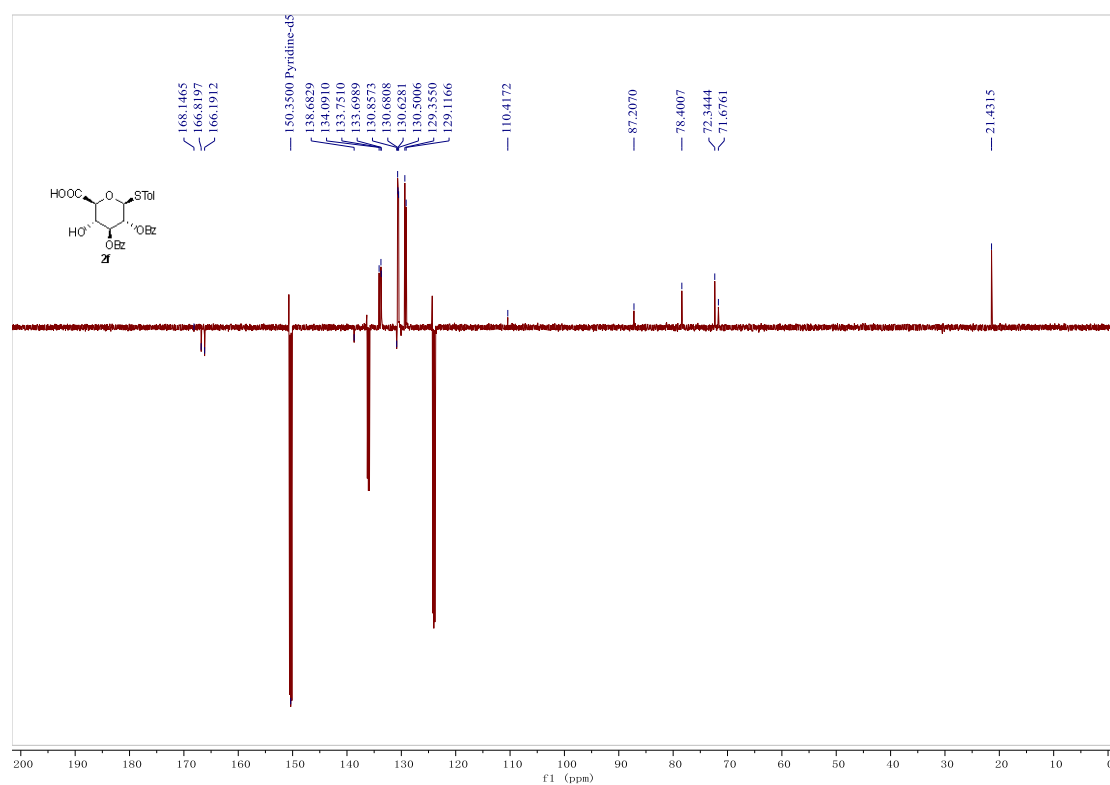
¹H NMR spectrum for 2e (500 MHz, CDCl₃)



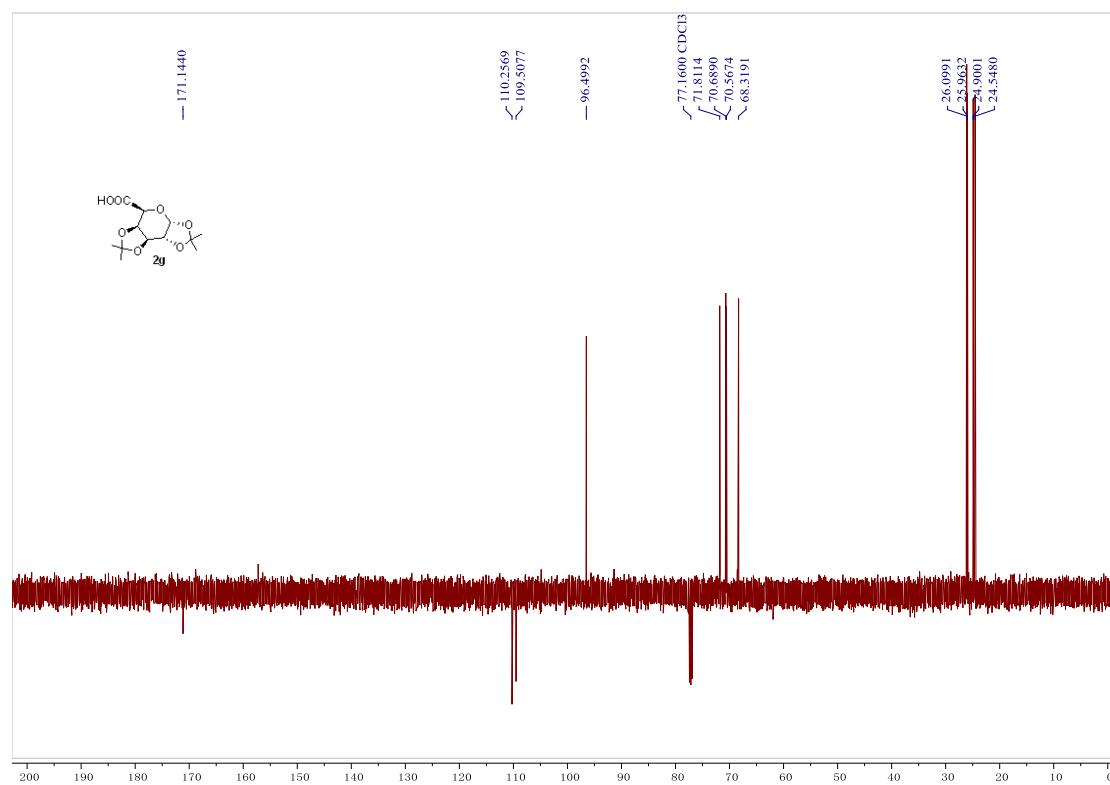
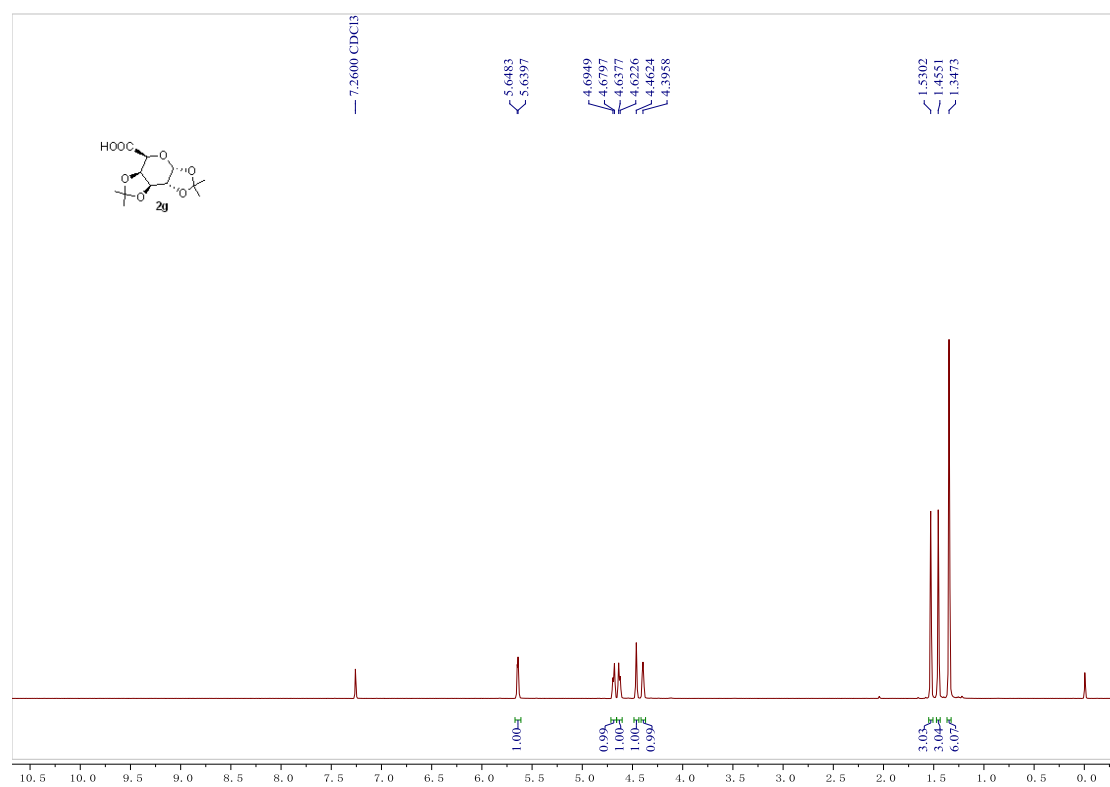
¹³C NMR spectrum for 2e (126 MHz, CDCl₃)

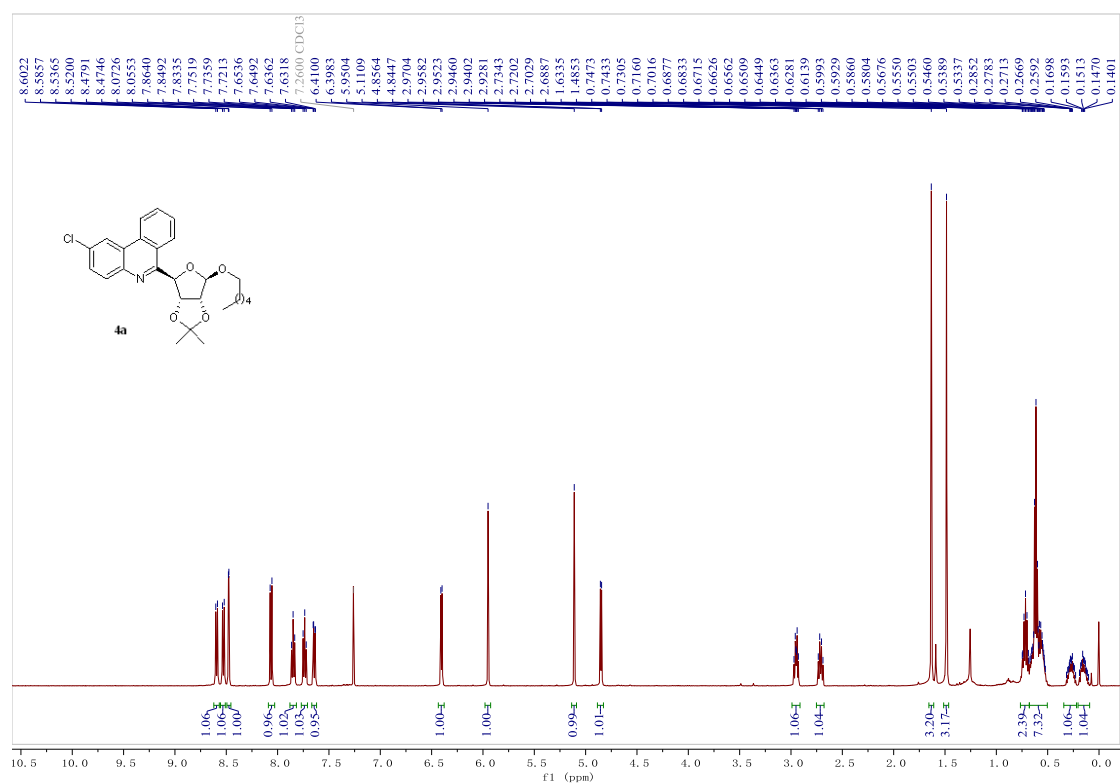


¹H NMR spectrum for 2f (500 MHz, pyridine-d₅)

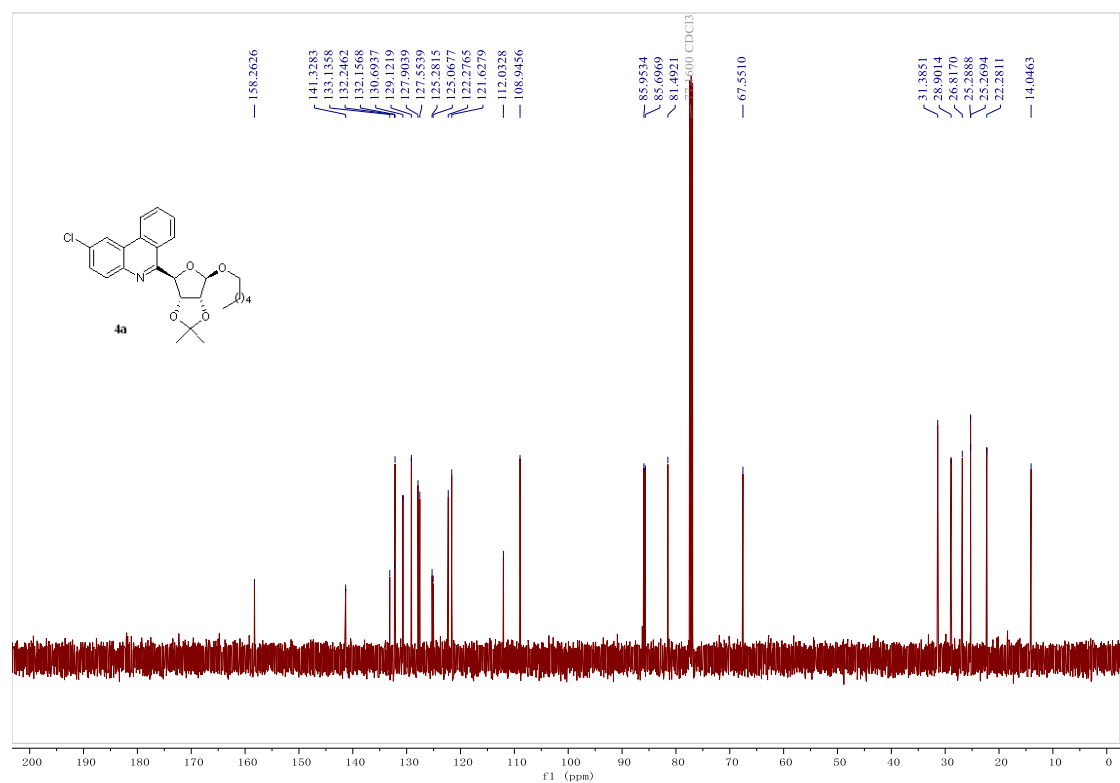


DEPT-Q spectrum for 2f (126 MHz, pyridine-d₅)

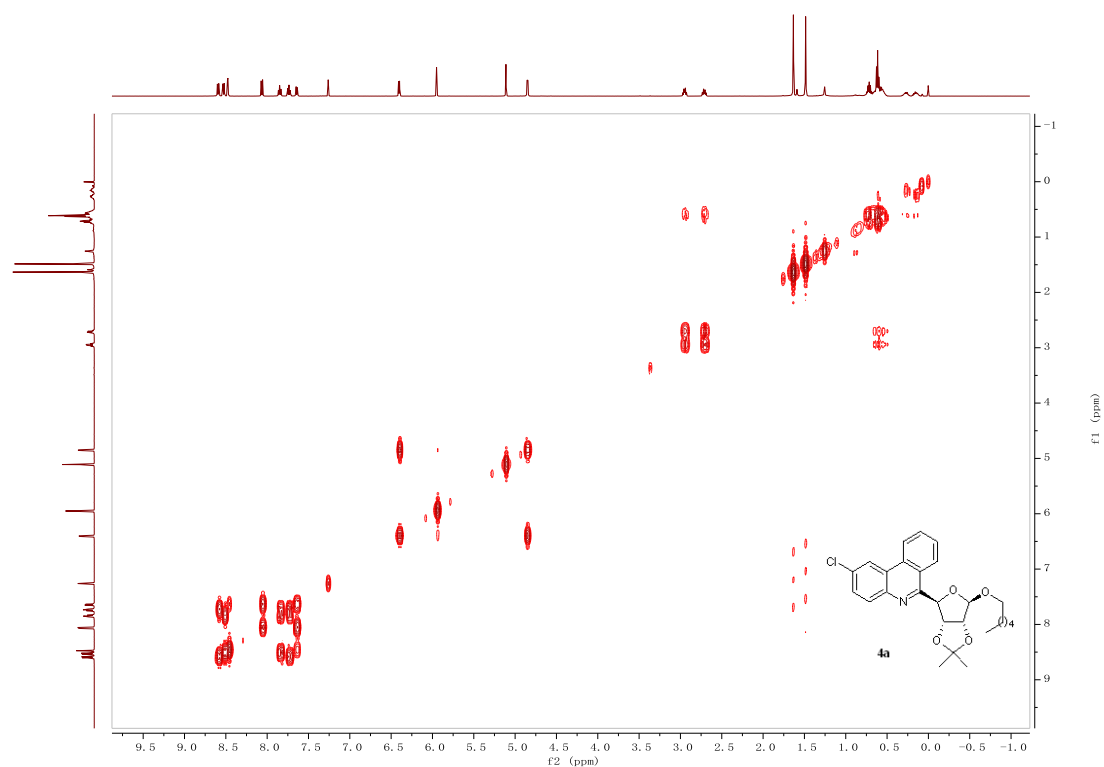




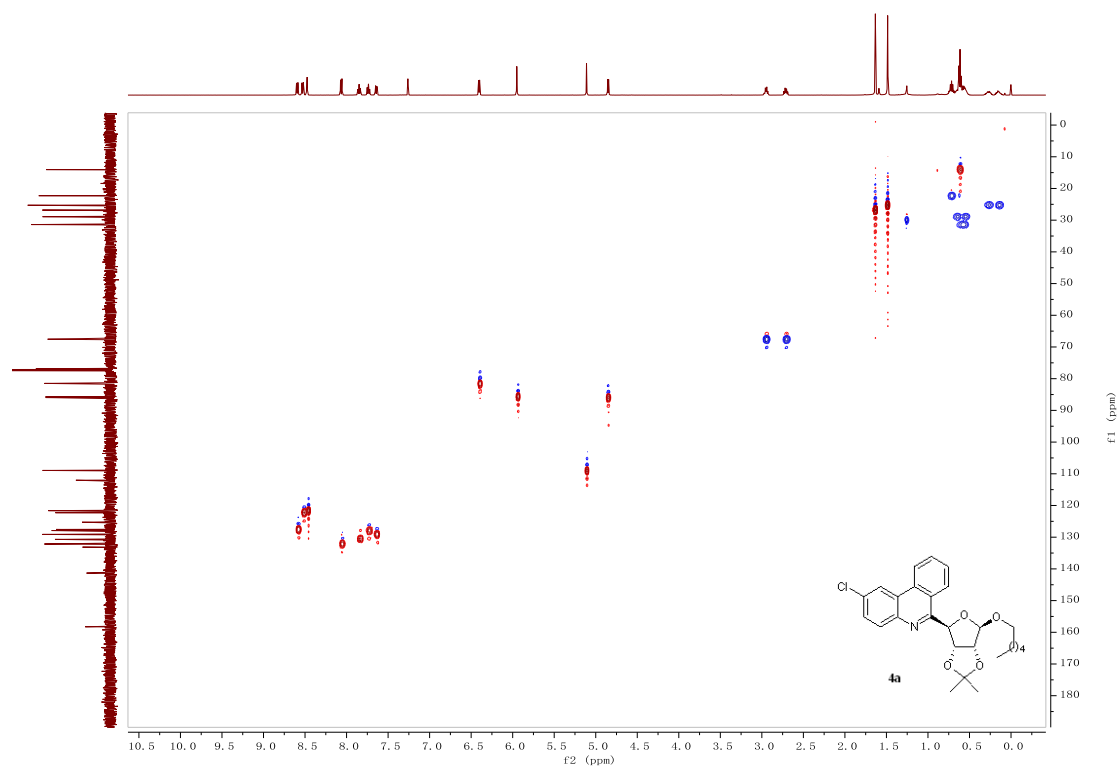
¹H NMR spectrum for 4a (500 MHz, CDCl₃)



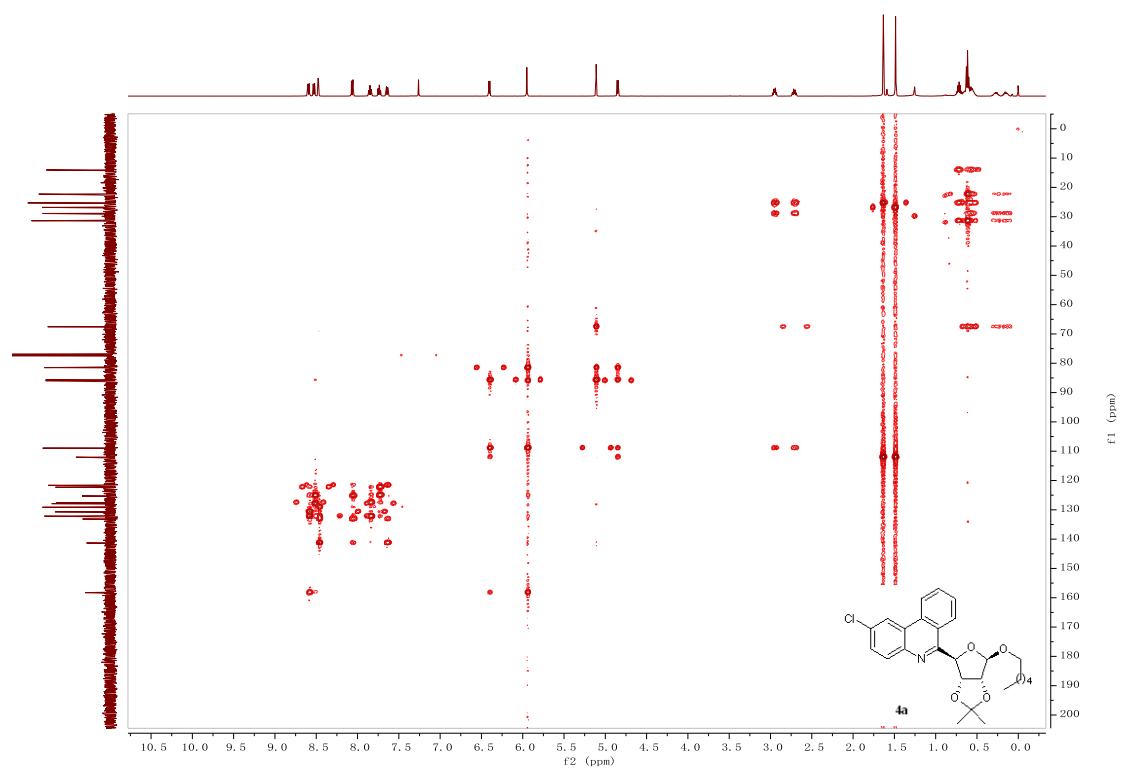
¹³C NMR spectrum for 4a (126 MHz, CDCl₃)



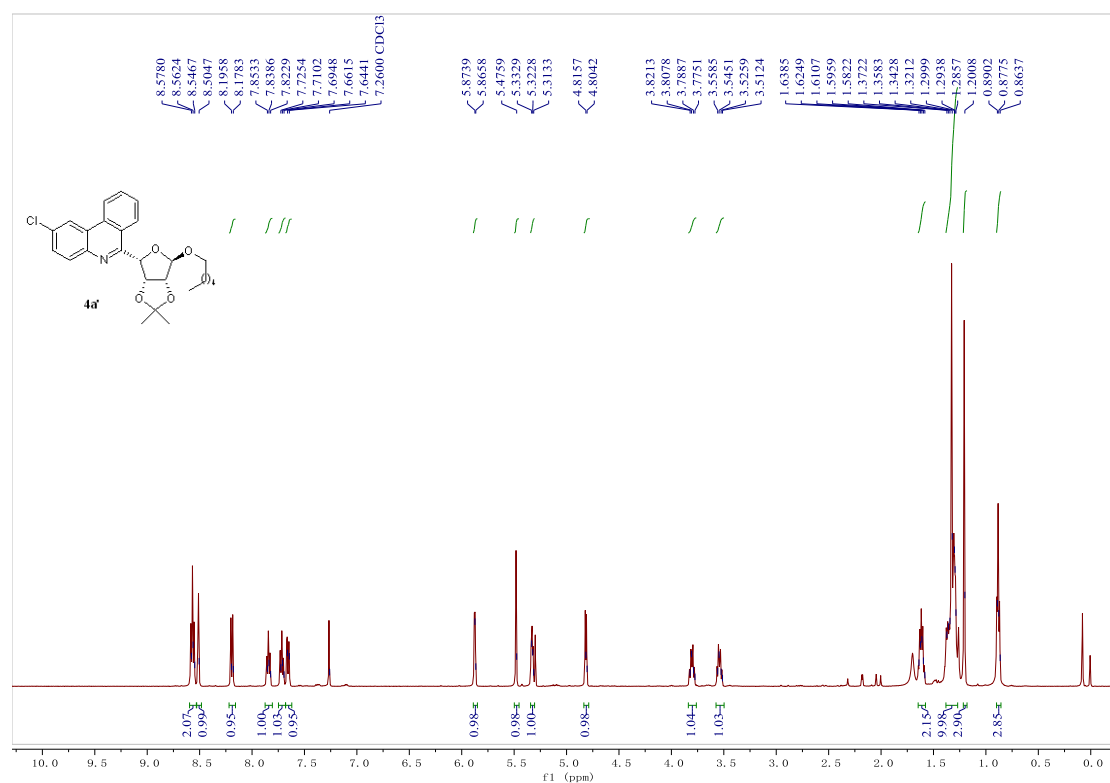
COSY spectrum for **4a**



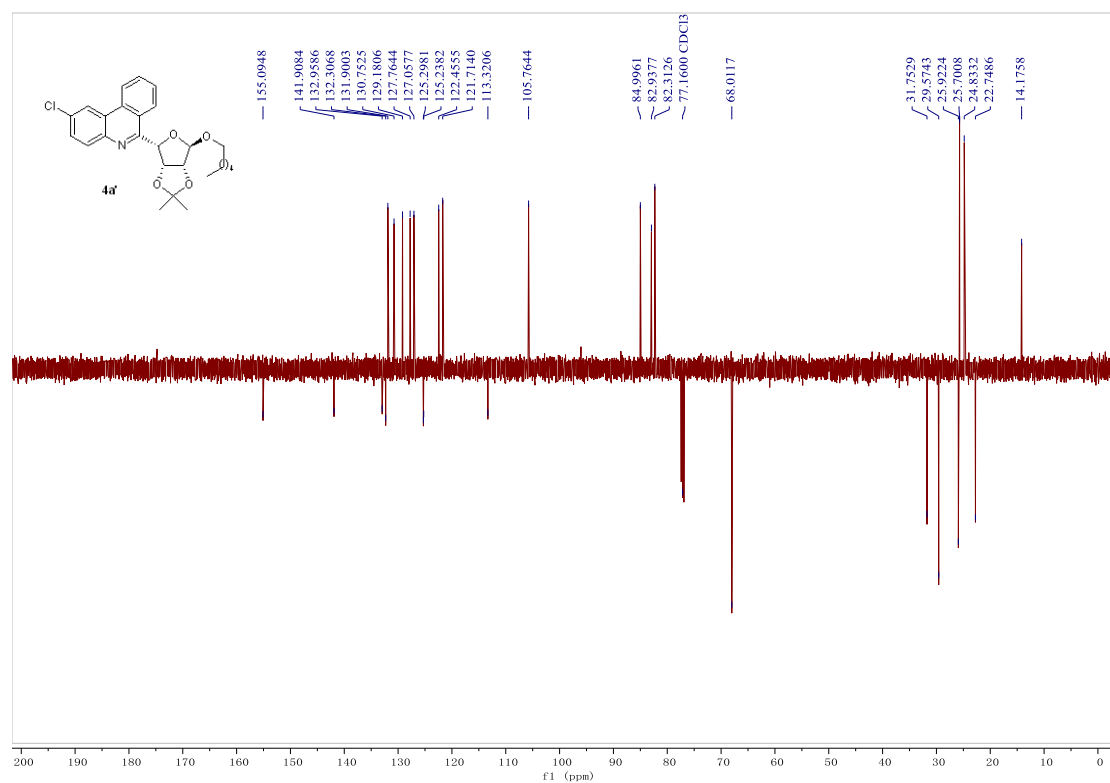
HSQC spectrum for **4a**



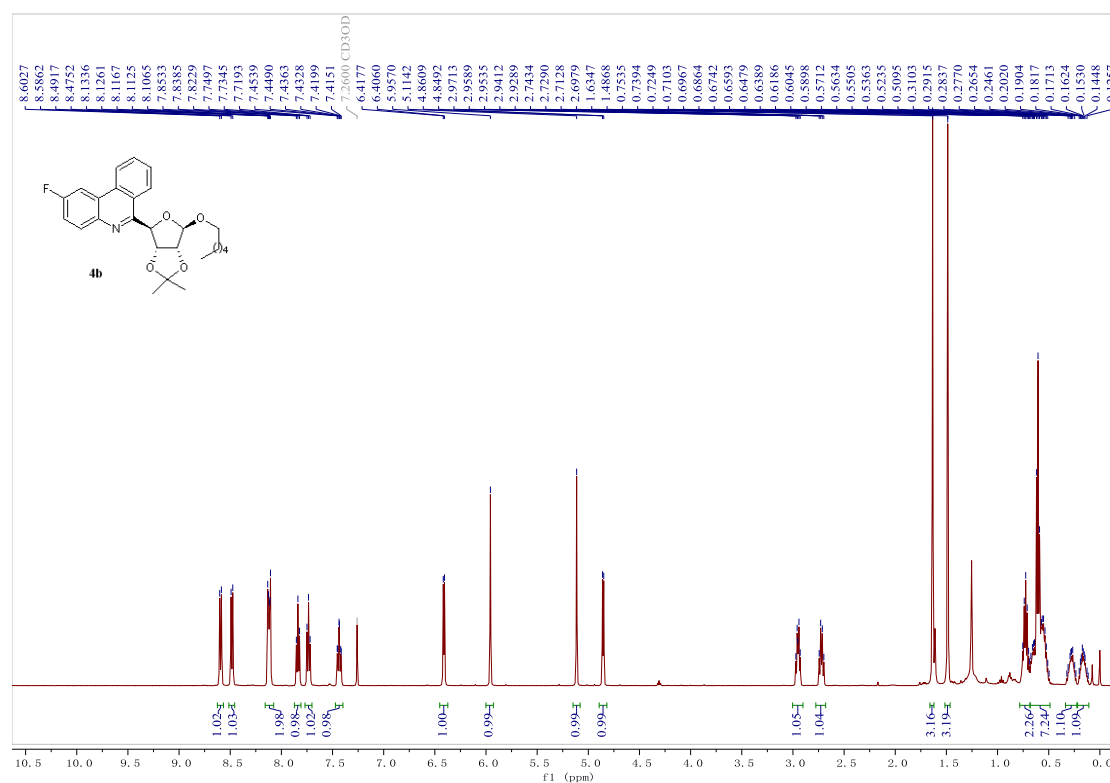
HMBC spectrum for **4a**



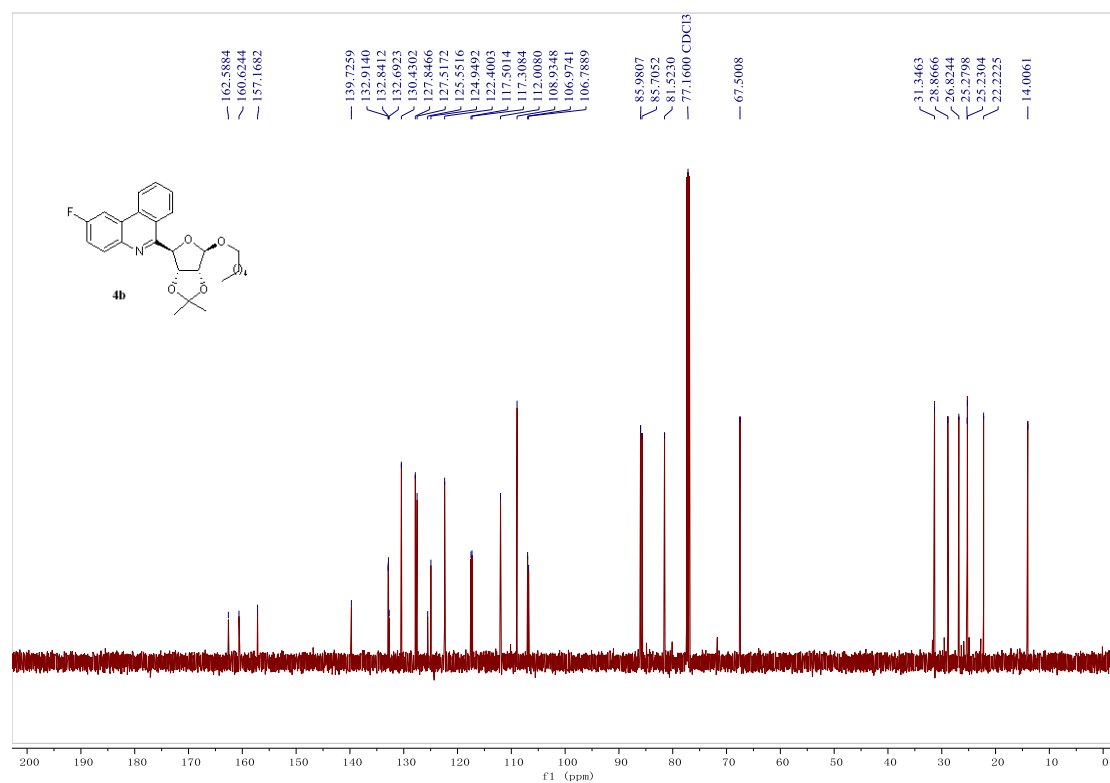
¹H NMR spectrum for 4a' (500 MHz, CDCl₃)



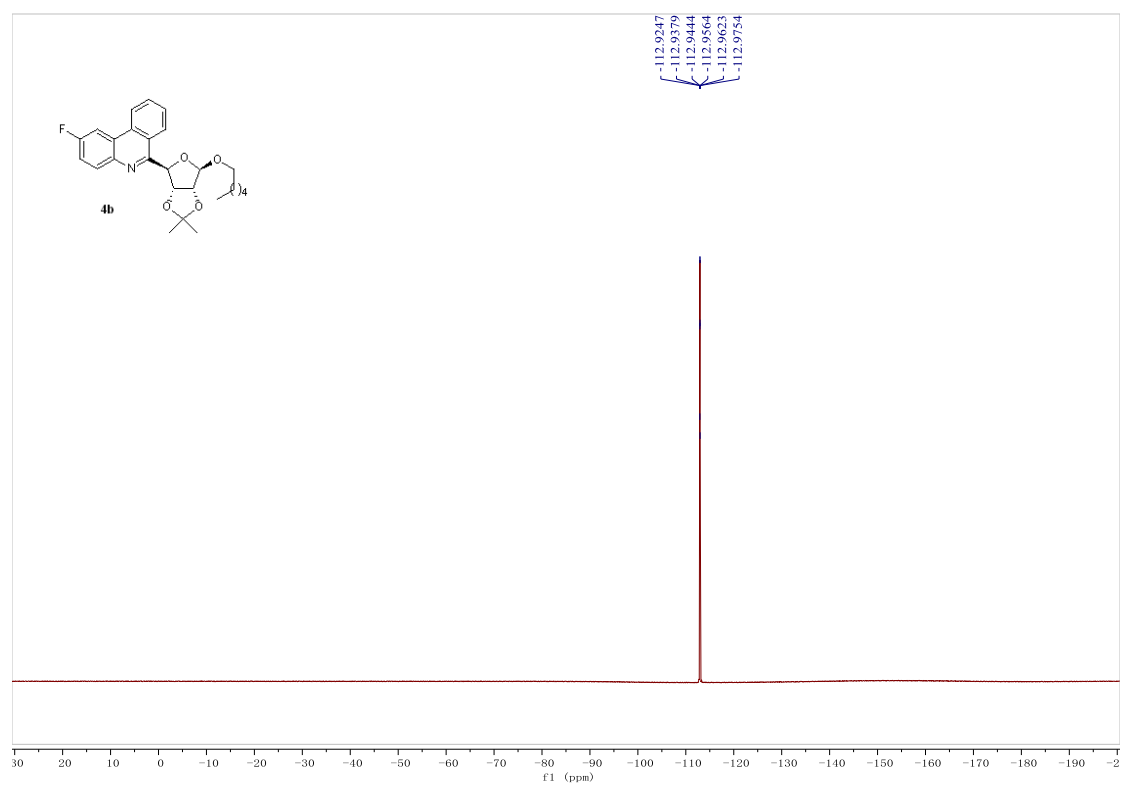
DEPT-Q spectrum for 4a' (126 MHz, CDCl₃)



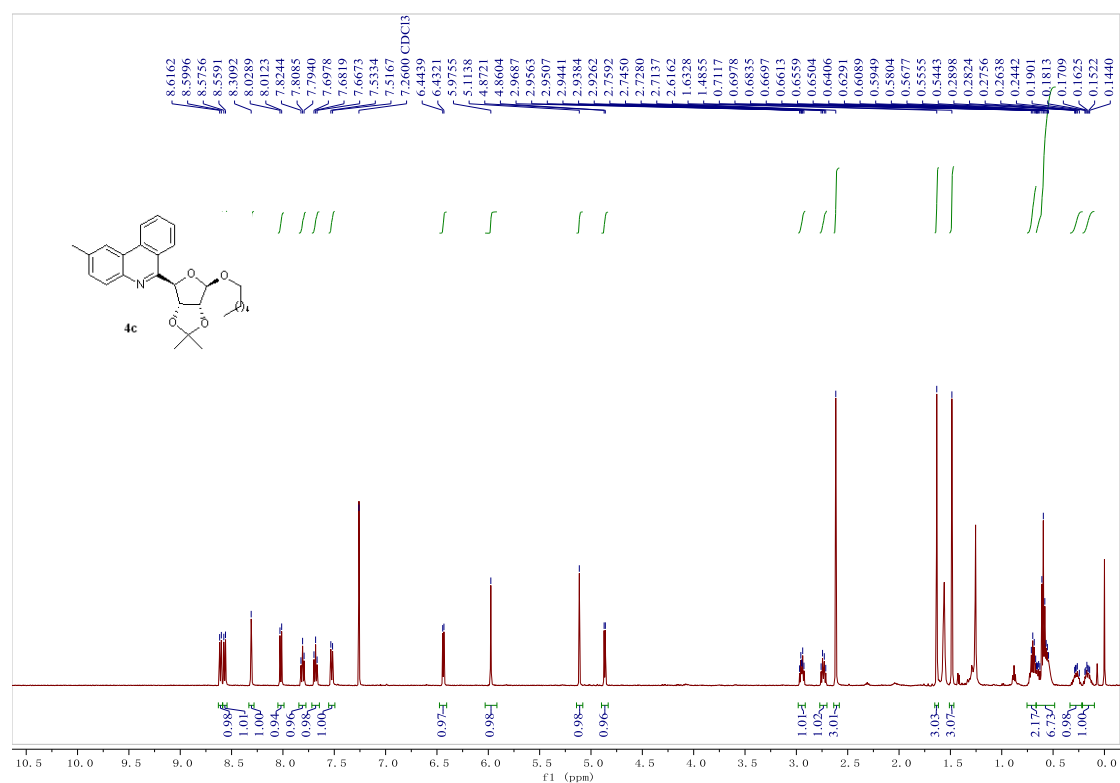
¹H NMR spectrum for 4b (500 MHz, CDCl₃)



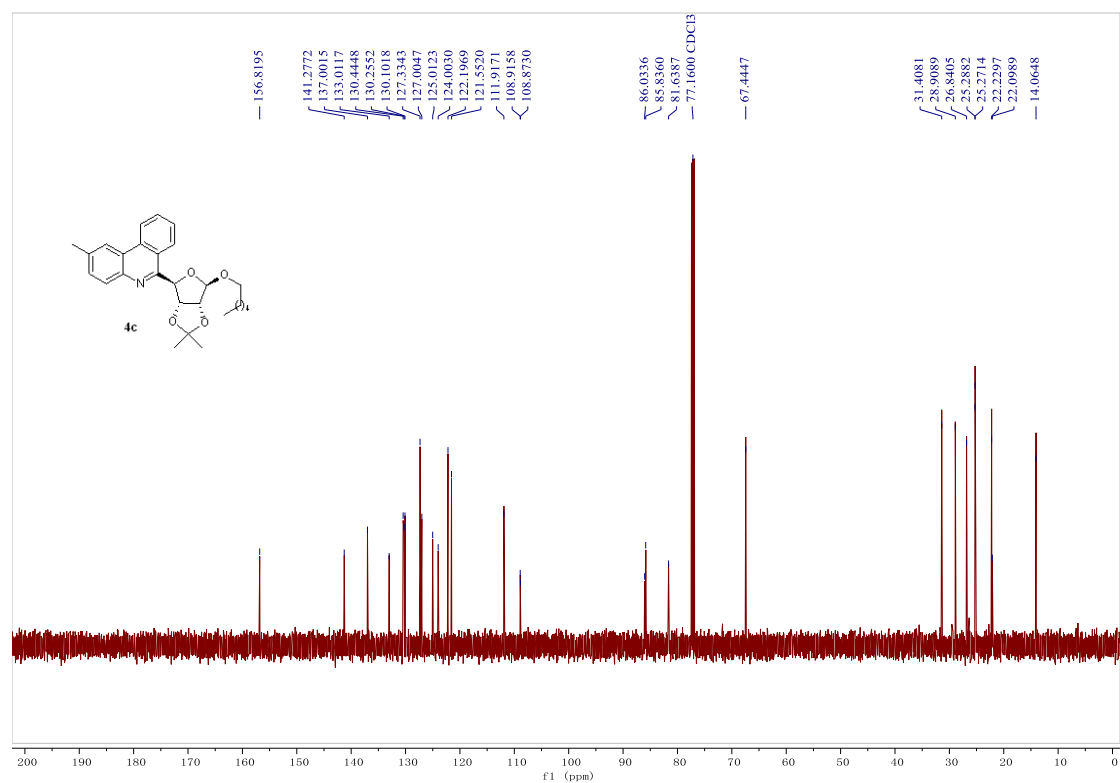
¹³C NMR spectrum for 4b (126 MHz, CDCl₃)



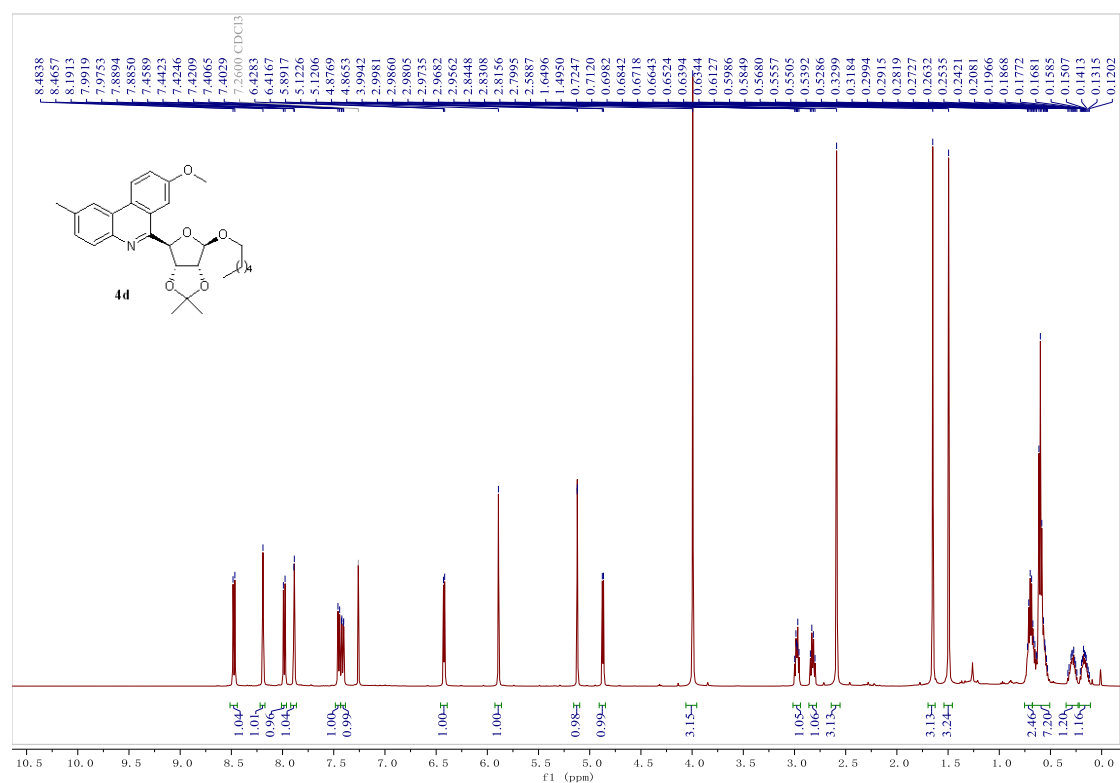
^{19}F NMR spectrum for **4b**



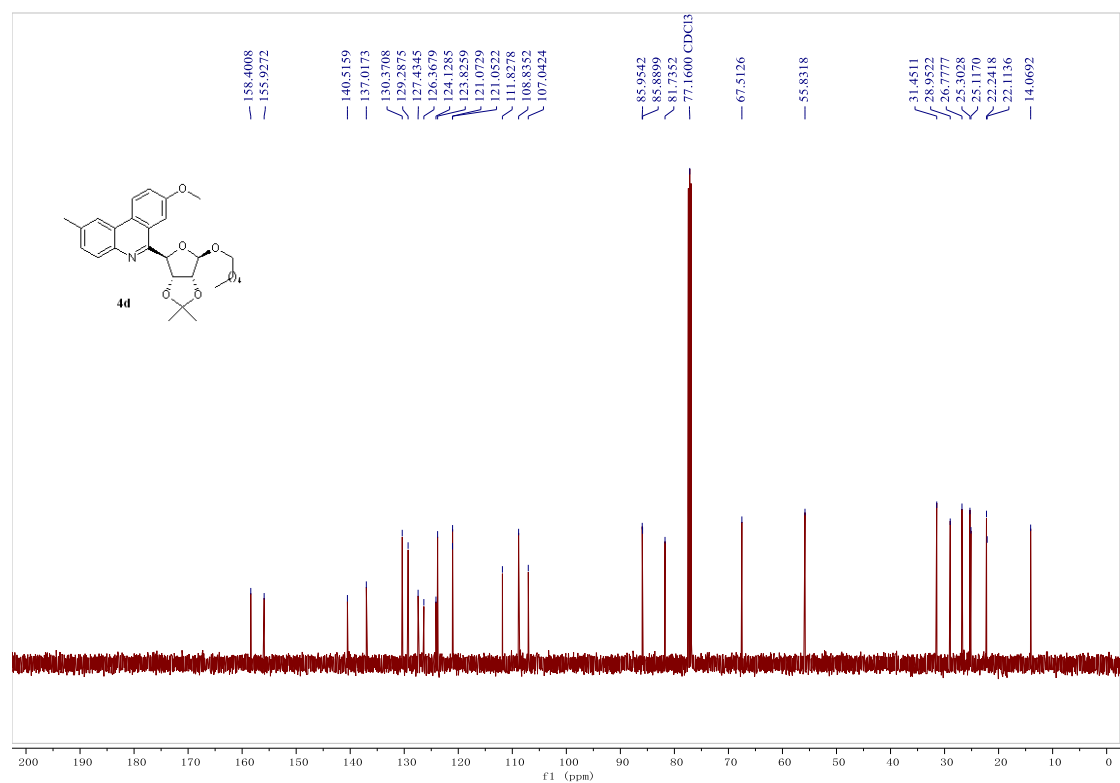
¹H NMR spectrum for 4c (500 MHz, CDCl₃)



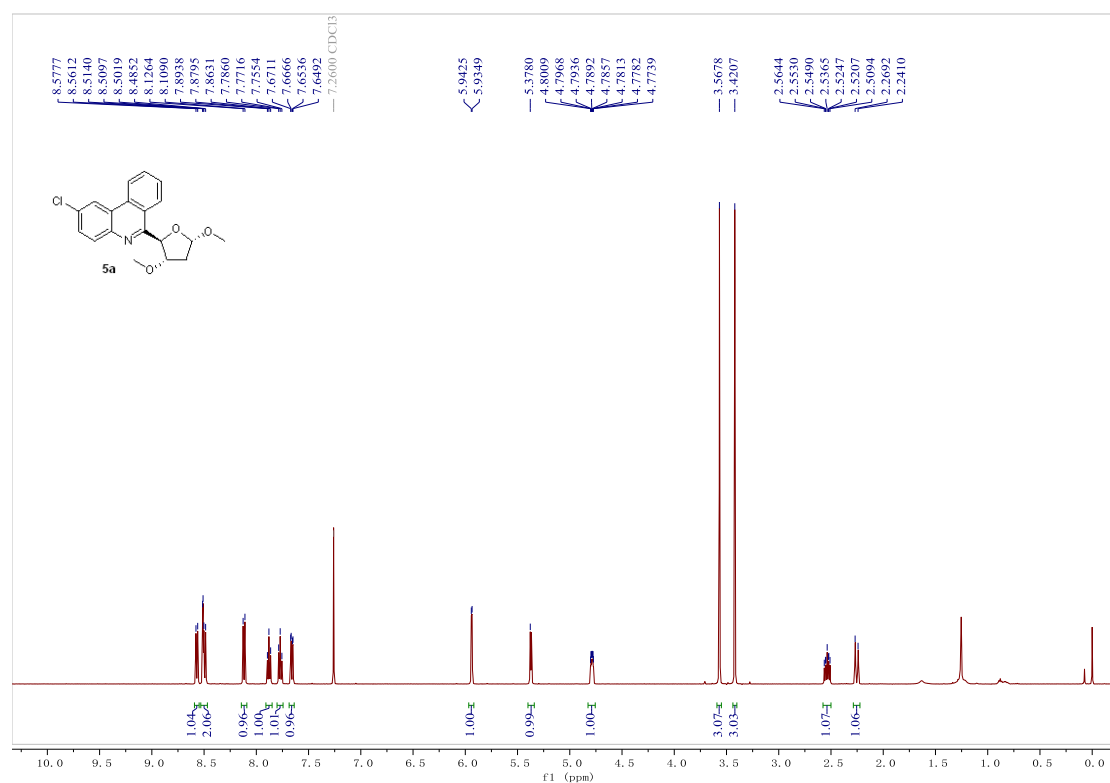
¹³C NMR spectrum for 4c (126 MHz, CDCl₃)



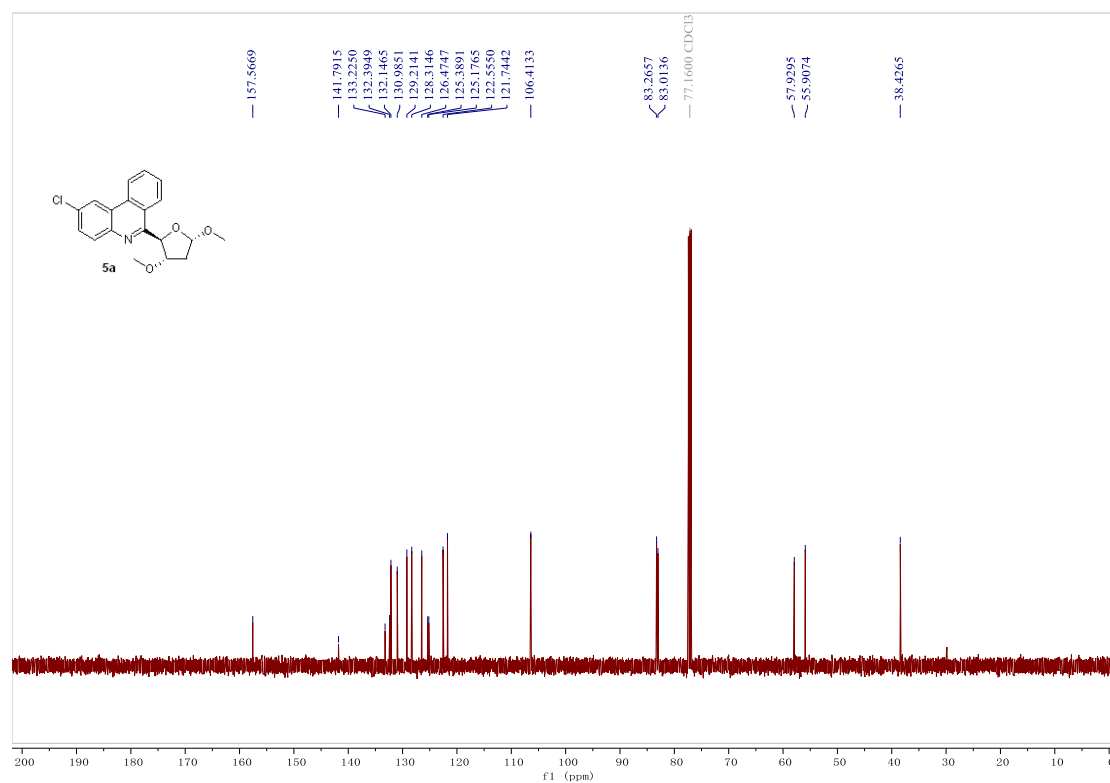
¹H NMR spectrum for 4d (500 MHz, CDCl₃)



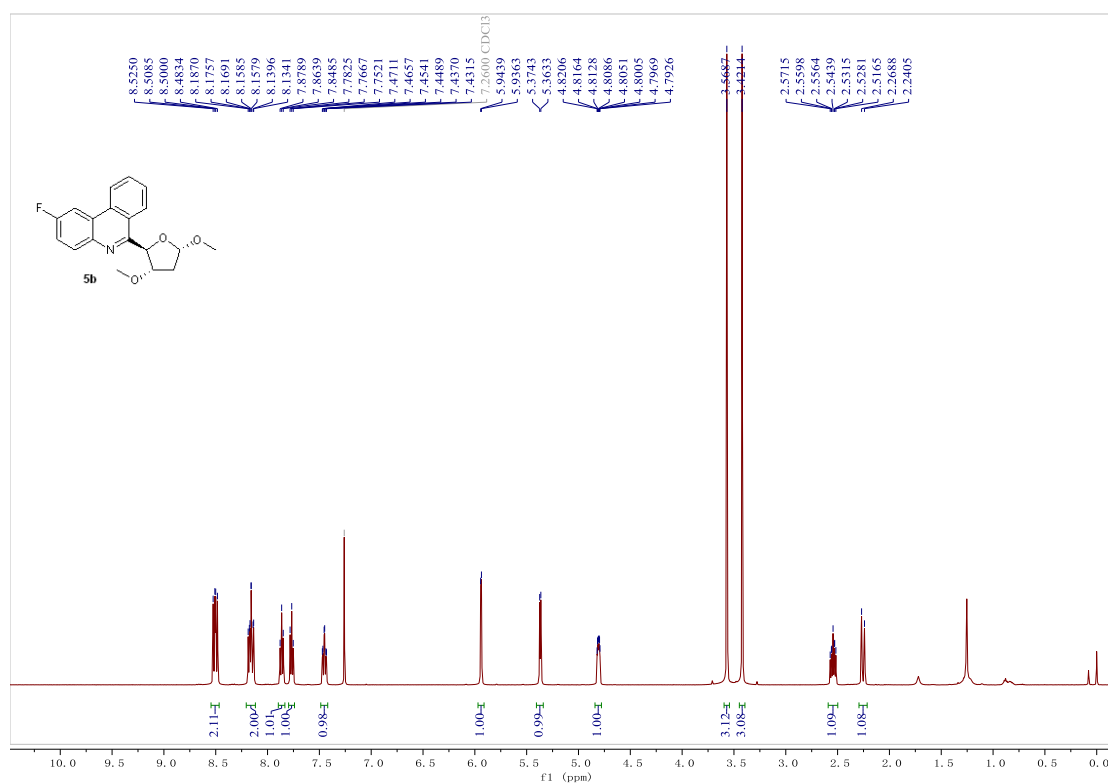
¹³C NMR spectrum for 4d (126 MHz, CDCl₃)



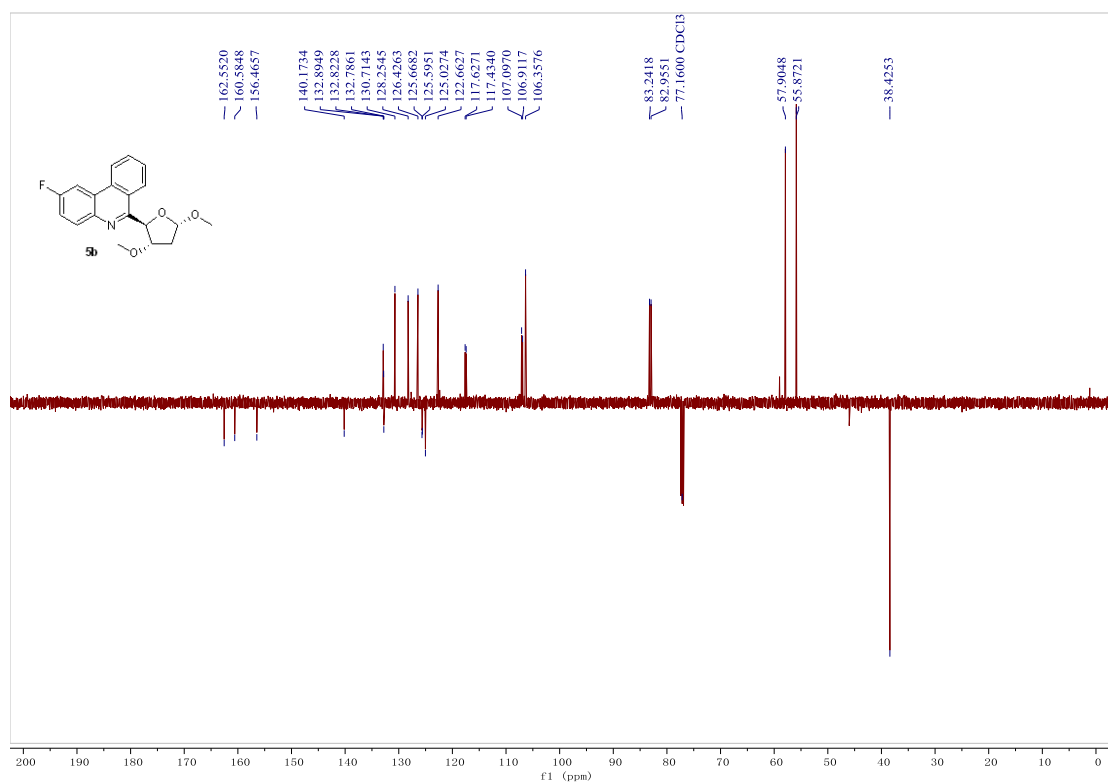
¹H NMR spectrum for 5a (500 MHz, CDCl₃)



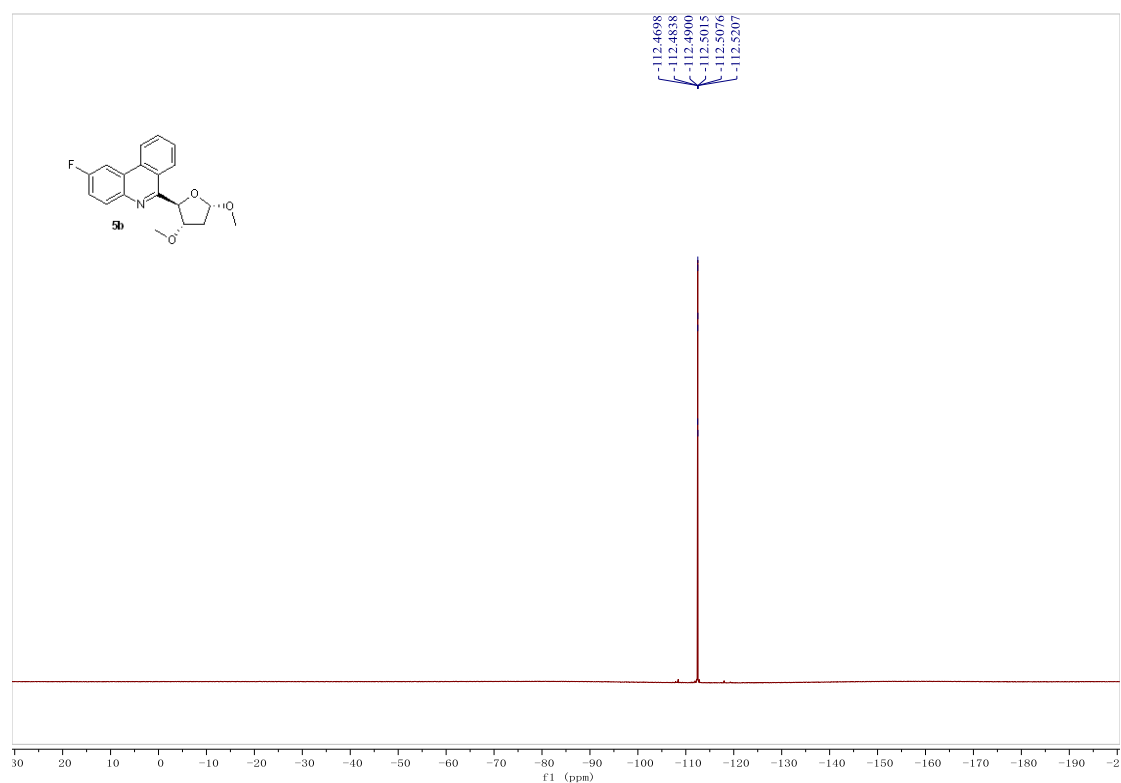
DEPT-Q spectrum for 5a (126 MHz, CDCl₃)



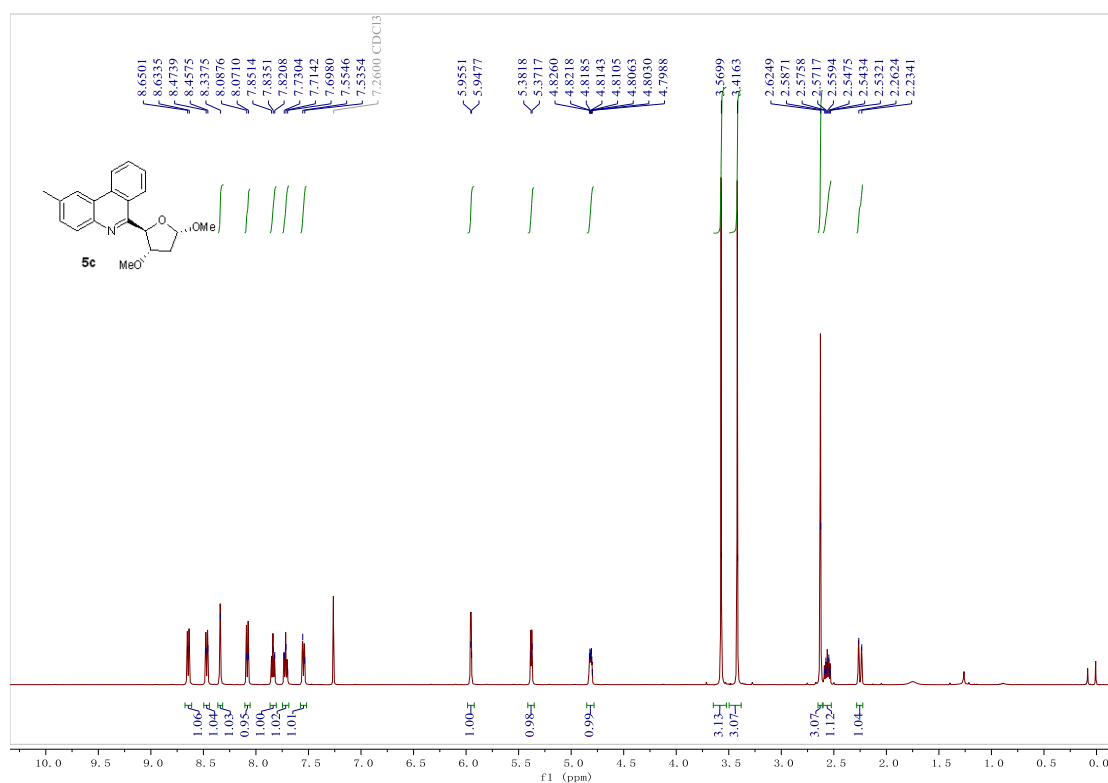
¹H NMR spectrum for 5b (500 MHz, CDCl₃)



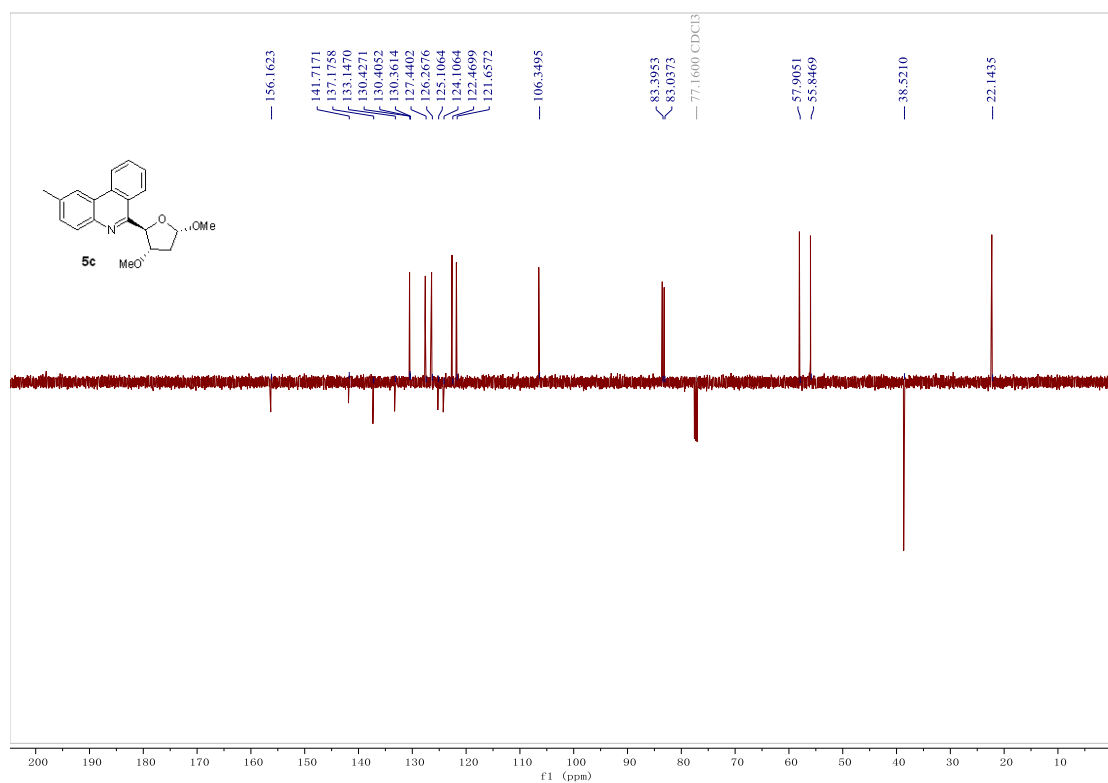
DEPT-Q spectrum for 5b (126 MHz, CDCl₃)



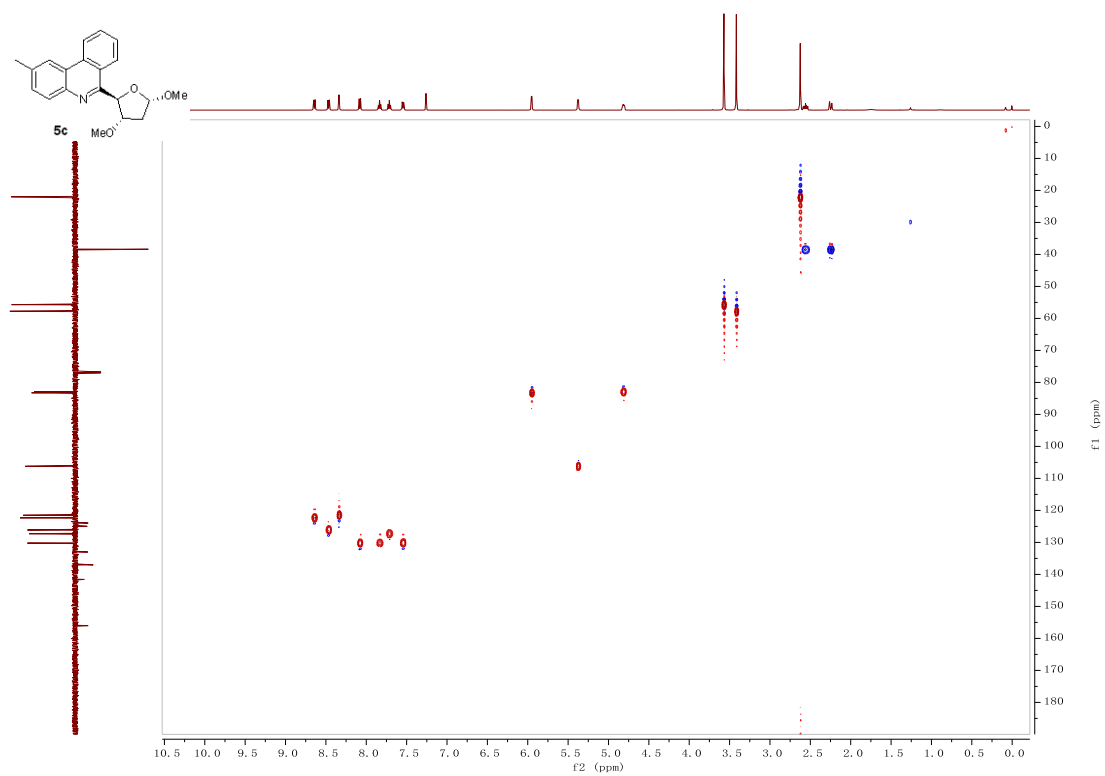
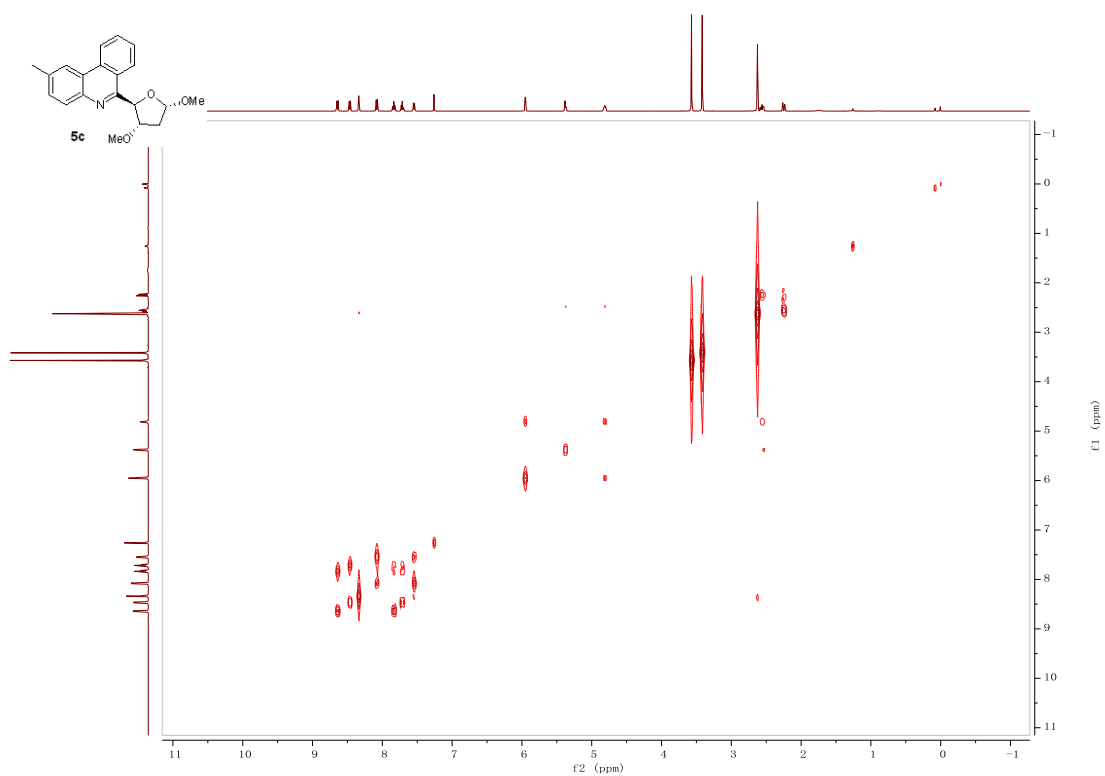
^{19}F NMR spectrum for **5b**

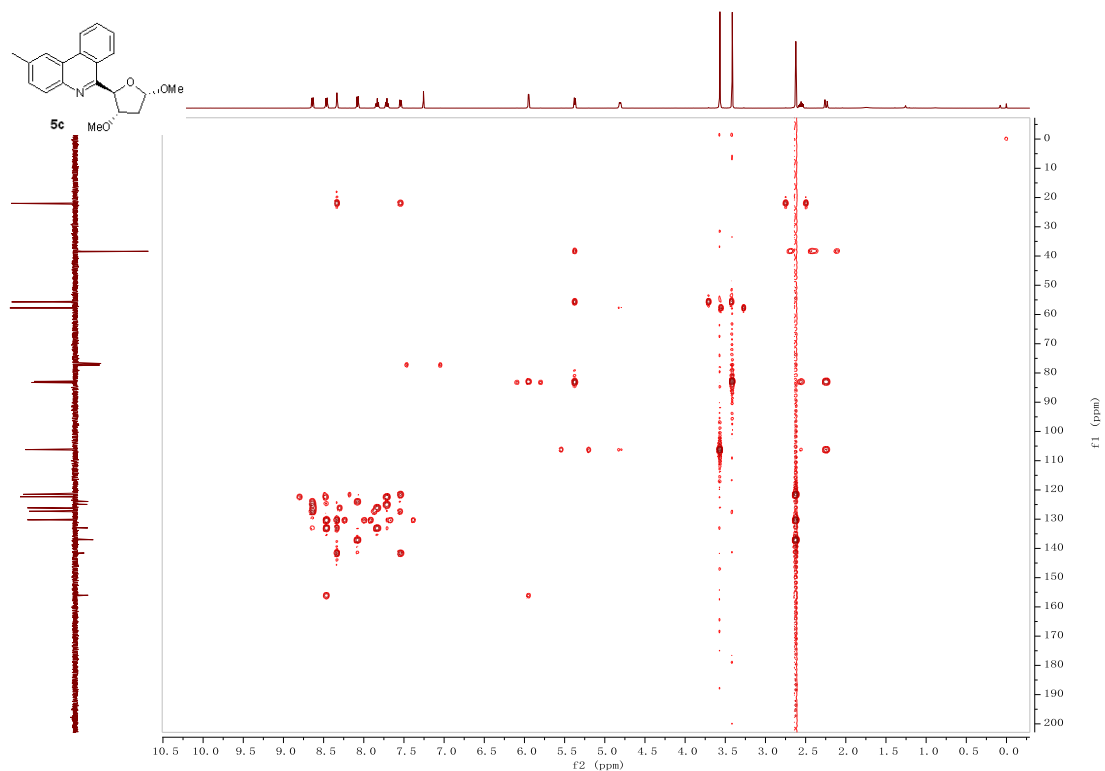


¹H NMR spectrum for 5c (500 MHz, CDCl₃)

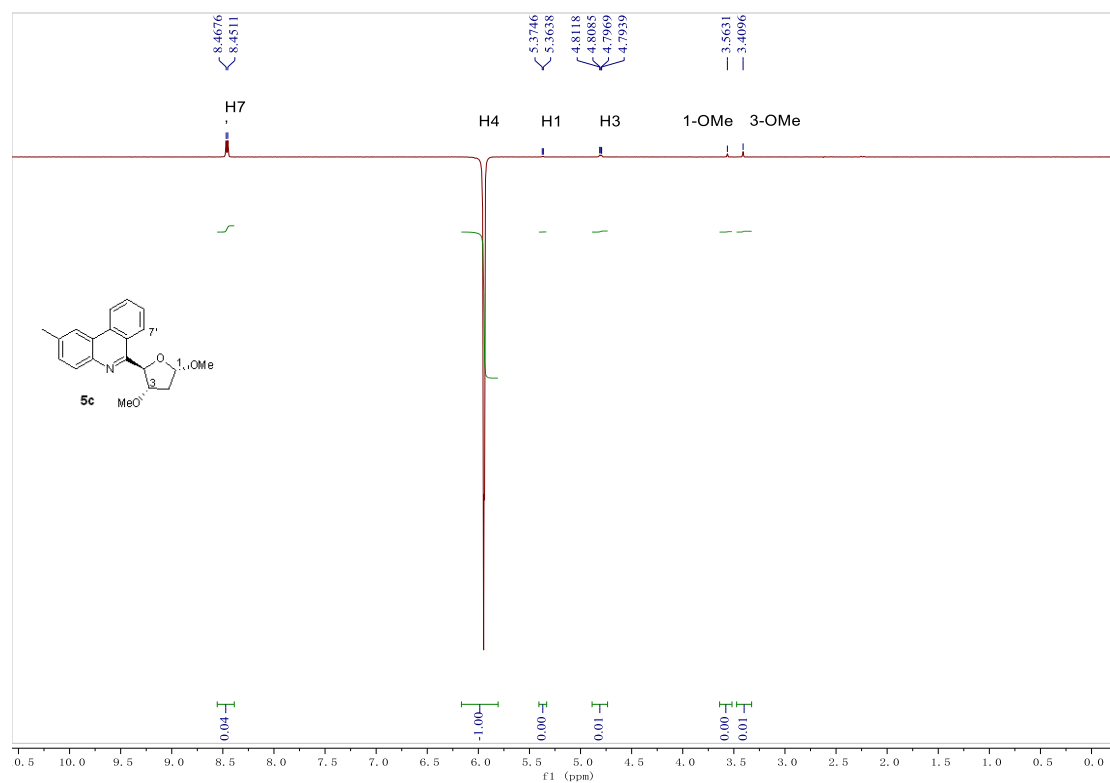


DEPT-Q spectrum for 5c (126 MHz, CDCl₃)

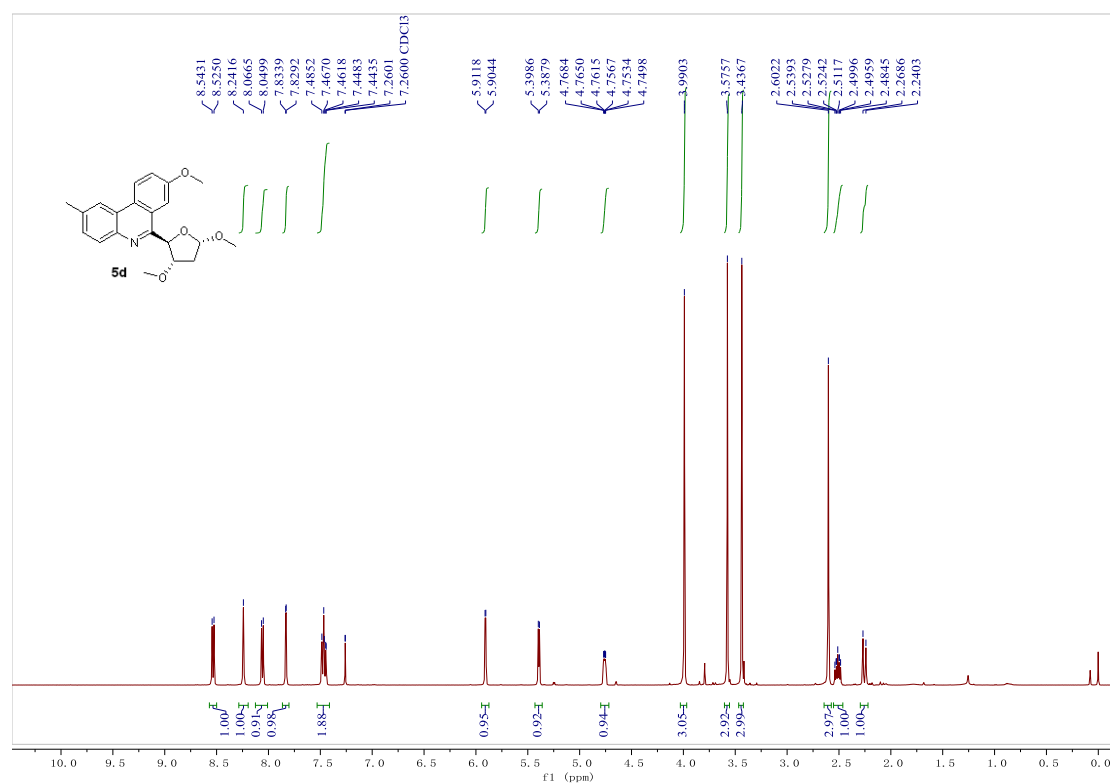




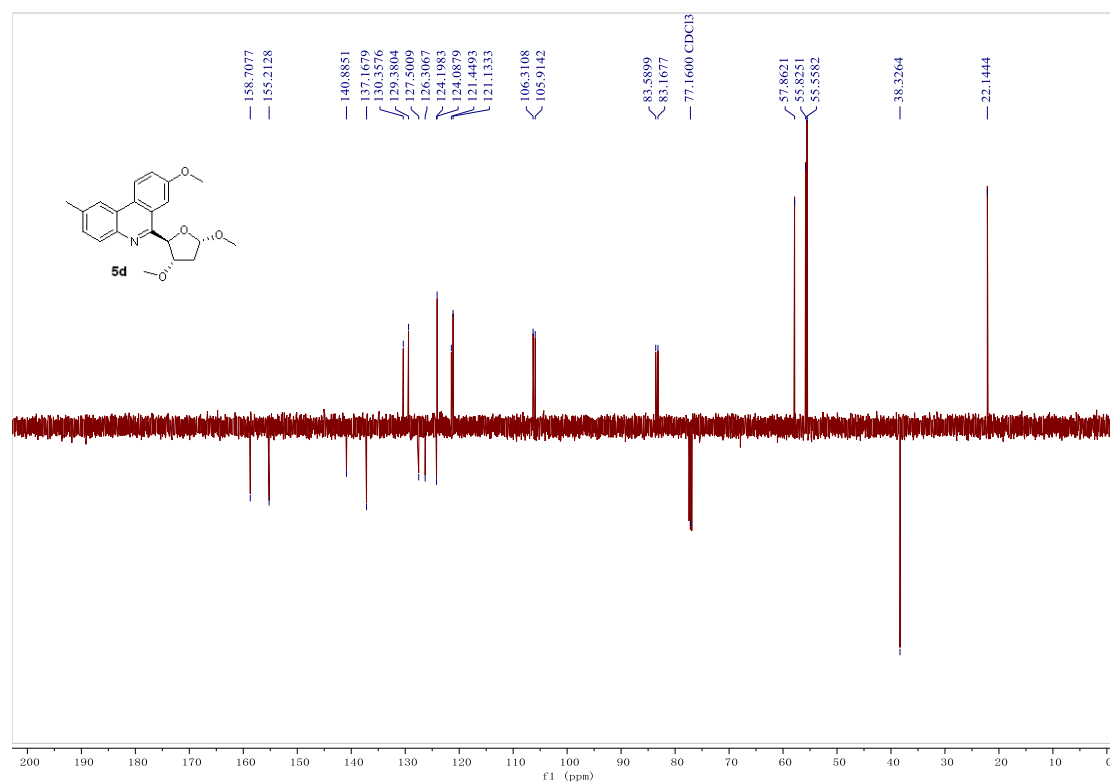
HMBC spectrum for 5c



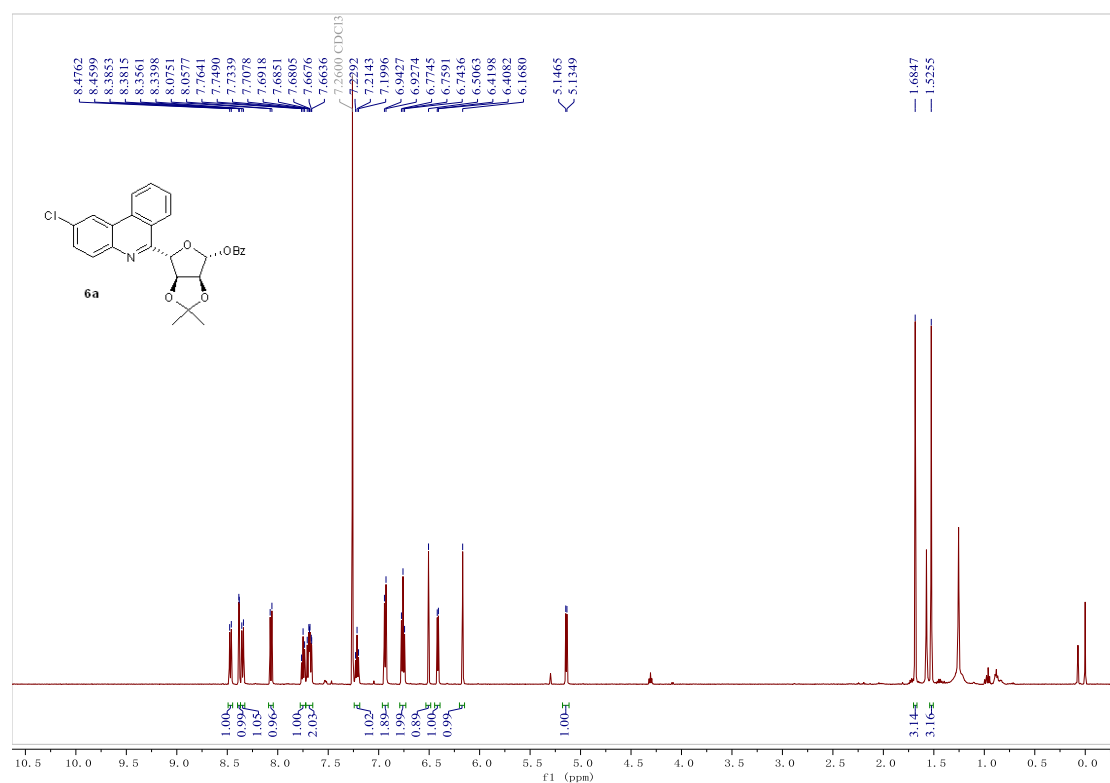
NOE difference spectrum for 5c



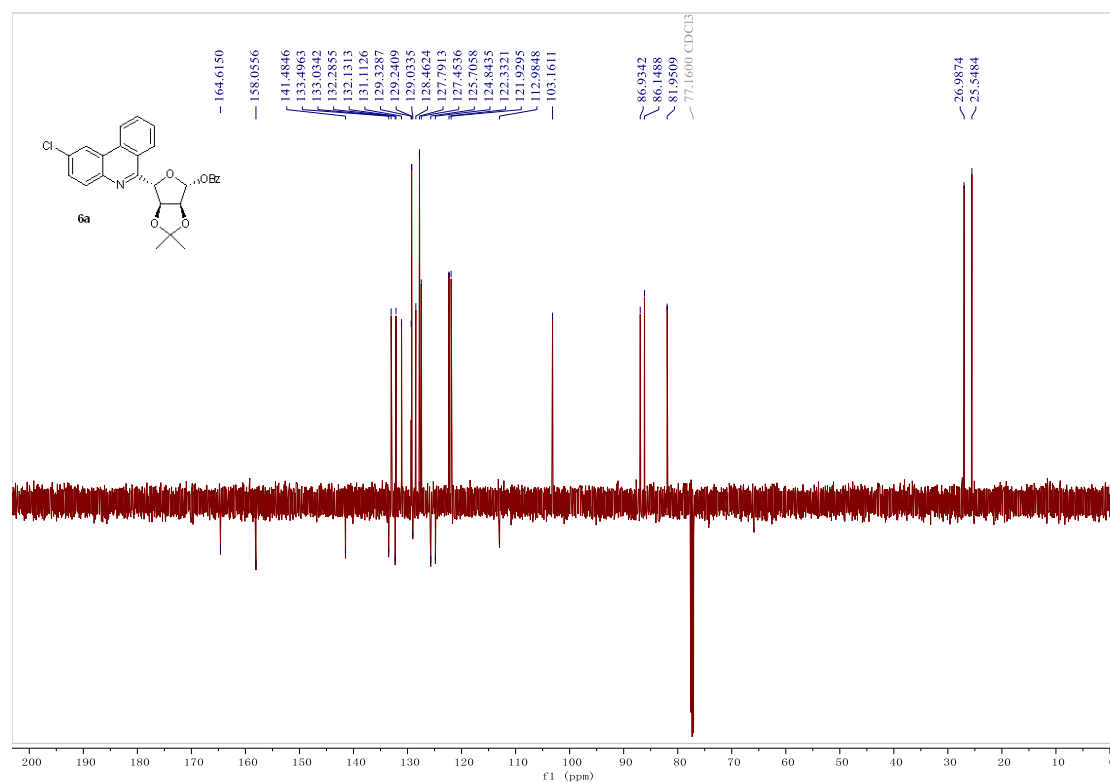
¹H NMR spectrum for 5d (500 MHz, CDCl₃)



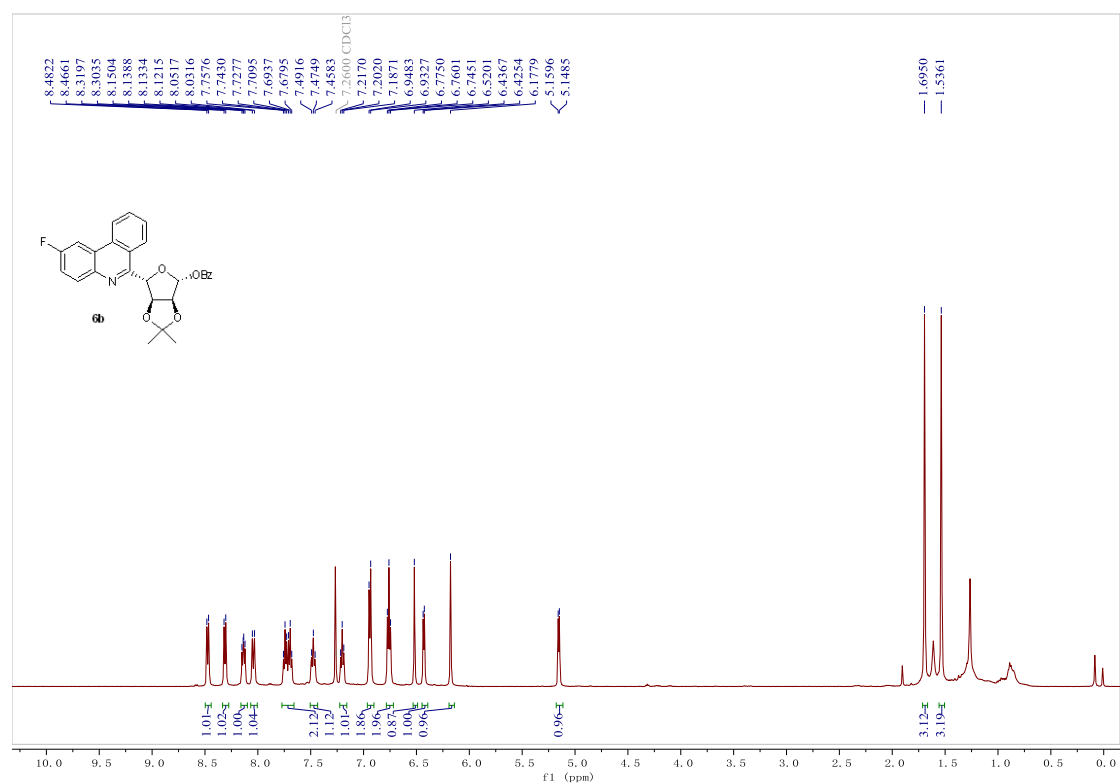
DEPT-Q spectrum for 5d (126 MHz, CDCl₃)



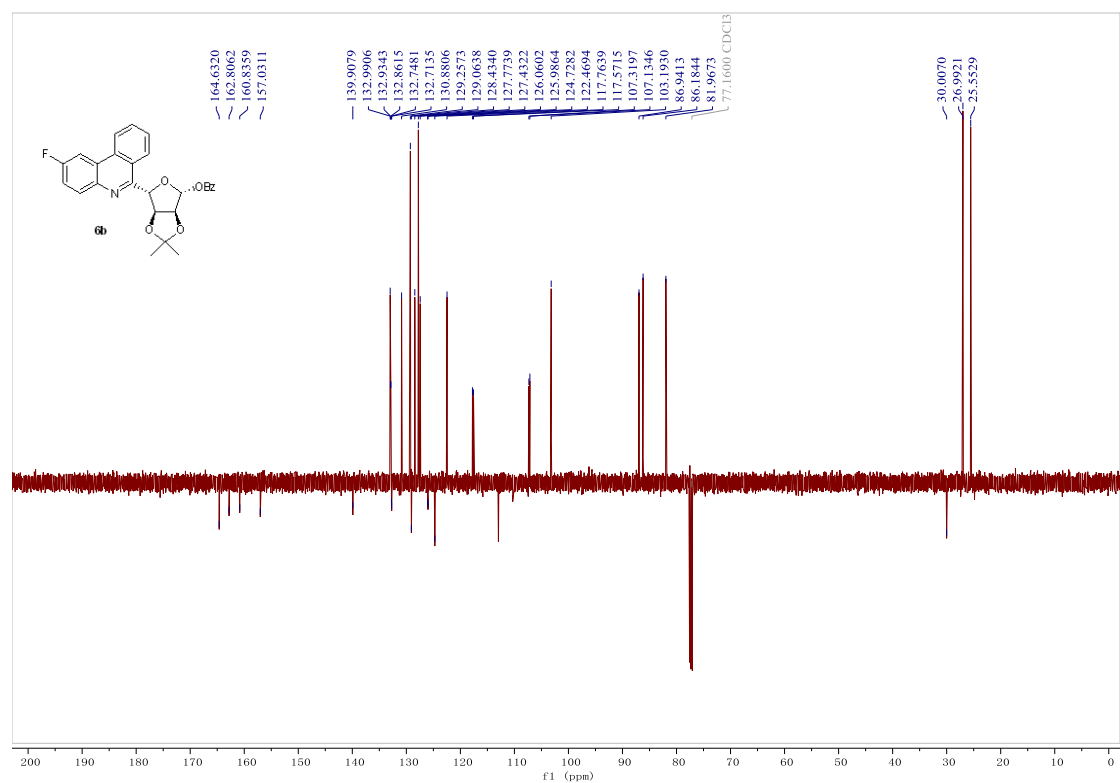
¹H NMR spectrum for **6a** (500 MHz, CDCl₃)



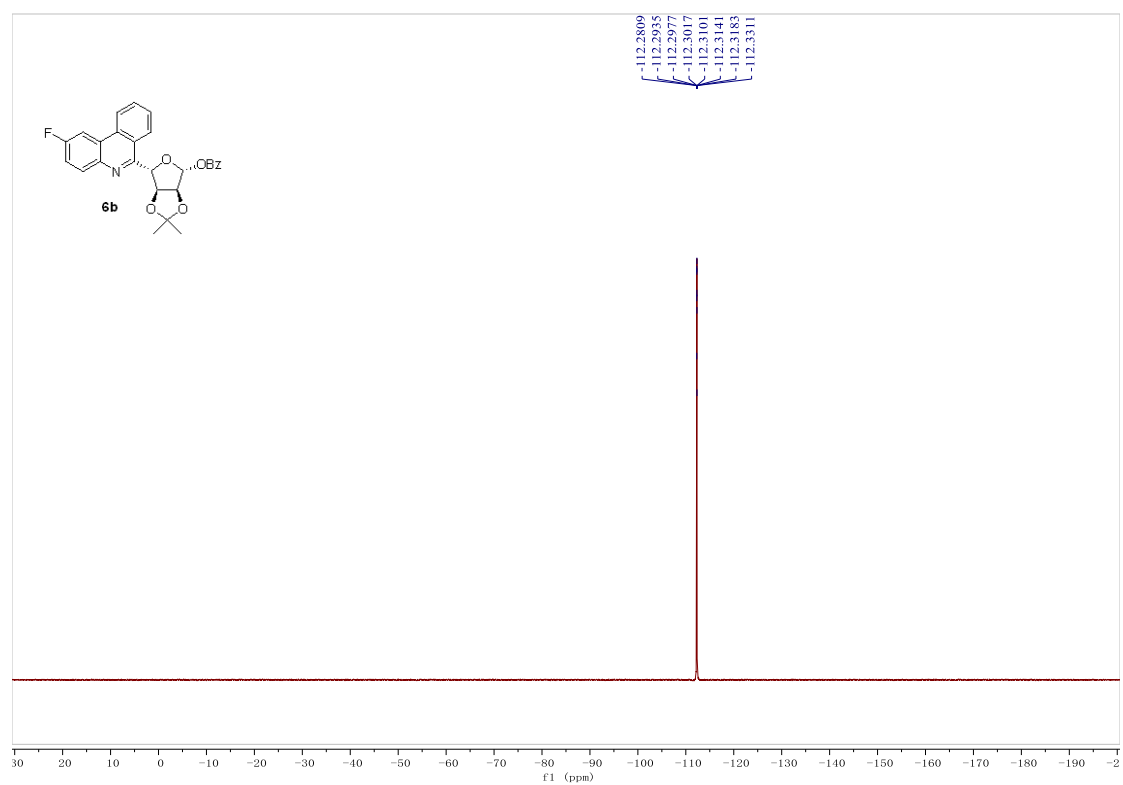
DEPT-Q spectrum for **6a** (126 MHz, CDCl₃)



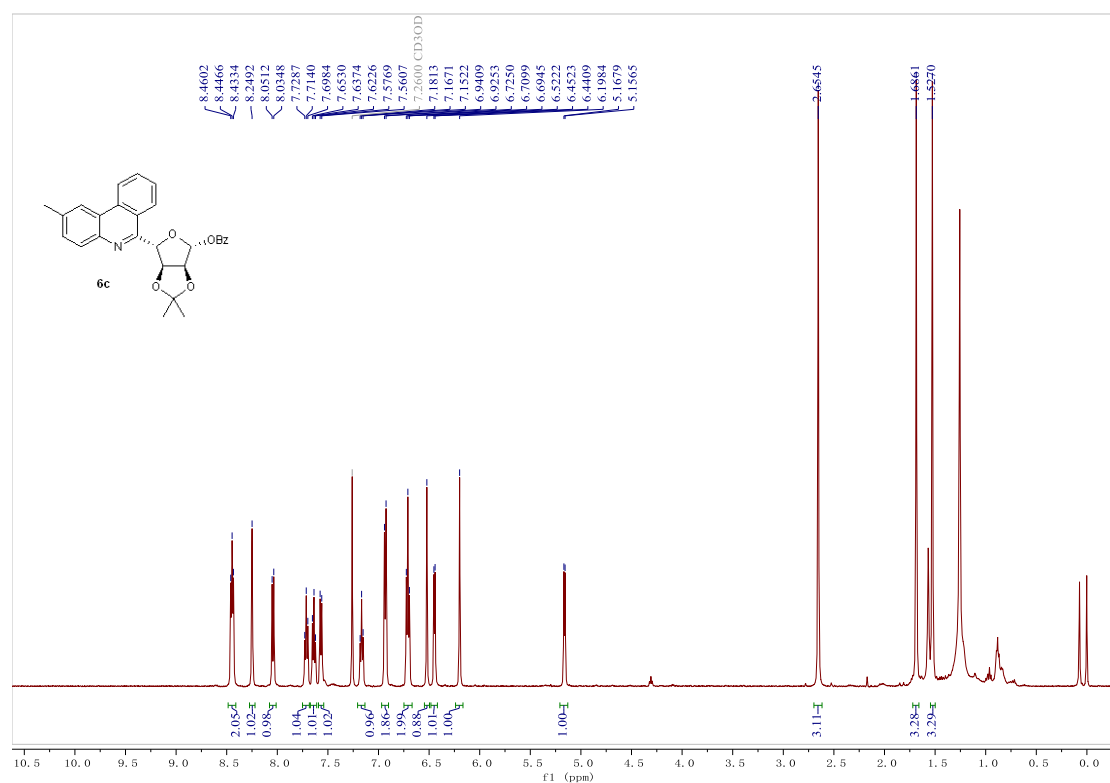
¹H NMR spectrum for **6b (500 MHz, CDCl₃)**



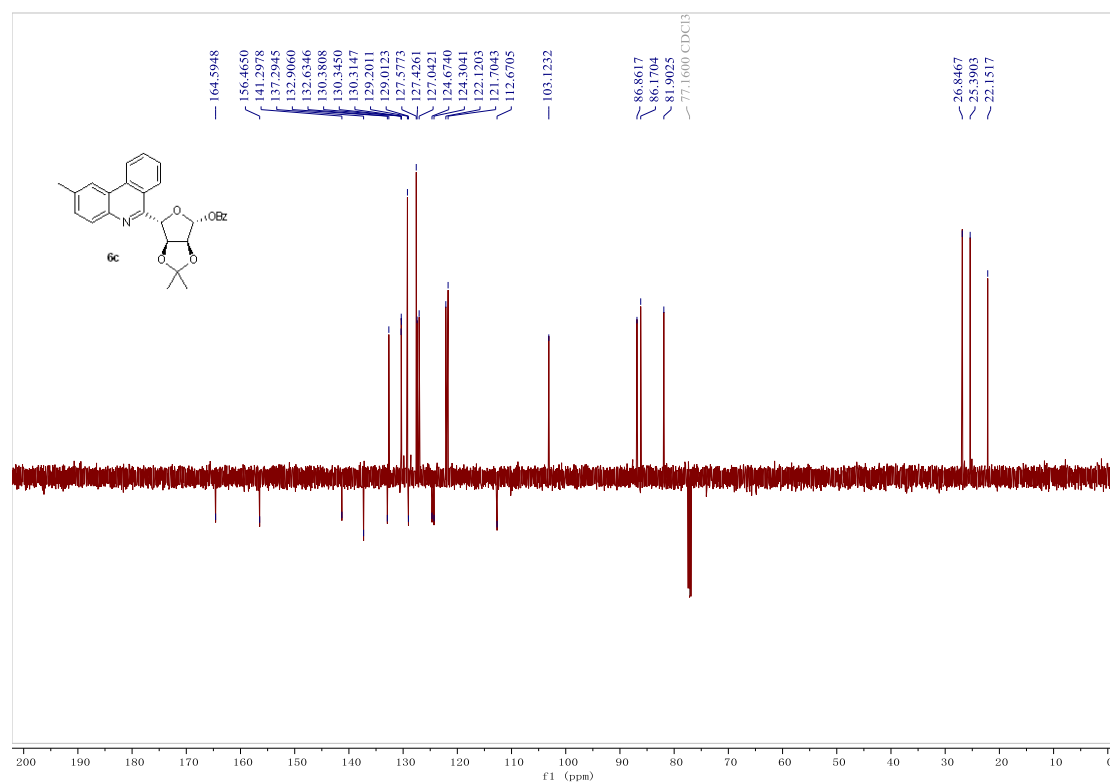
DEPT-Q spectrum for **6b (126 MHz, CDCl₃)**



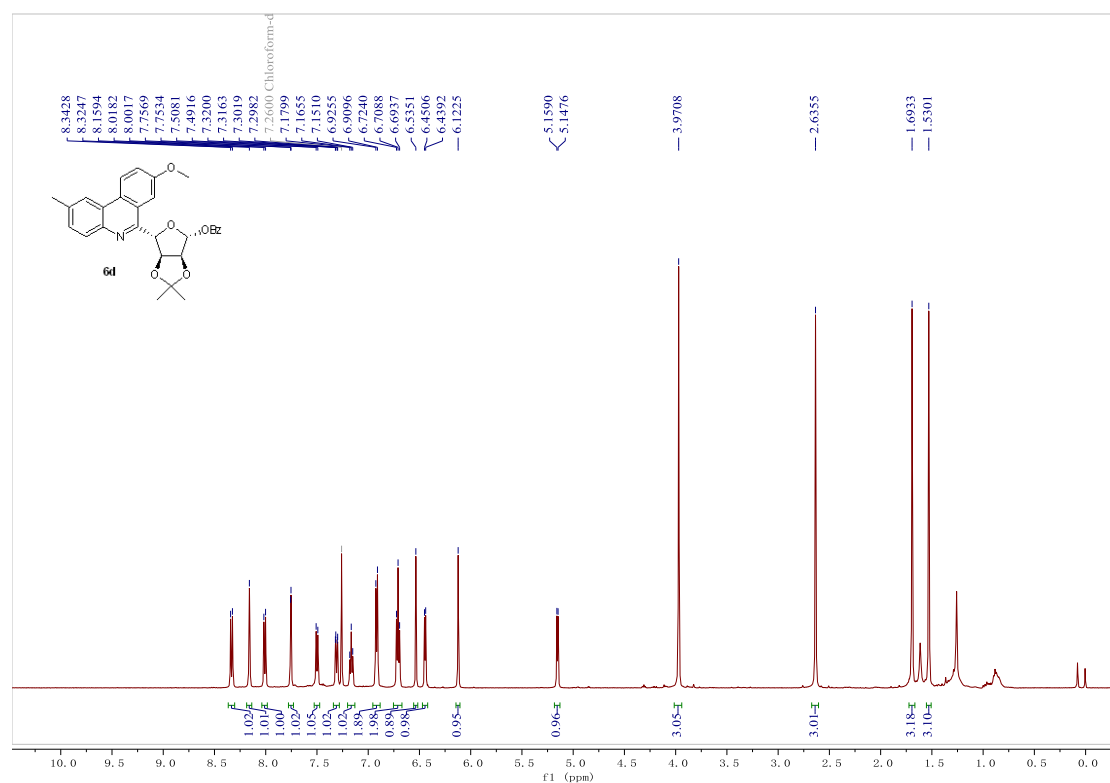
^{19}F NMR spectrum for **6b**



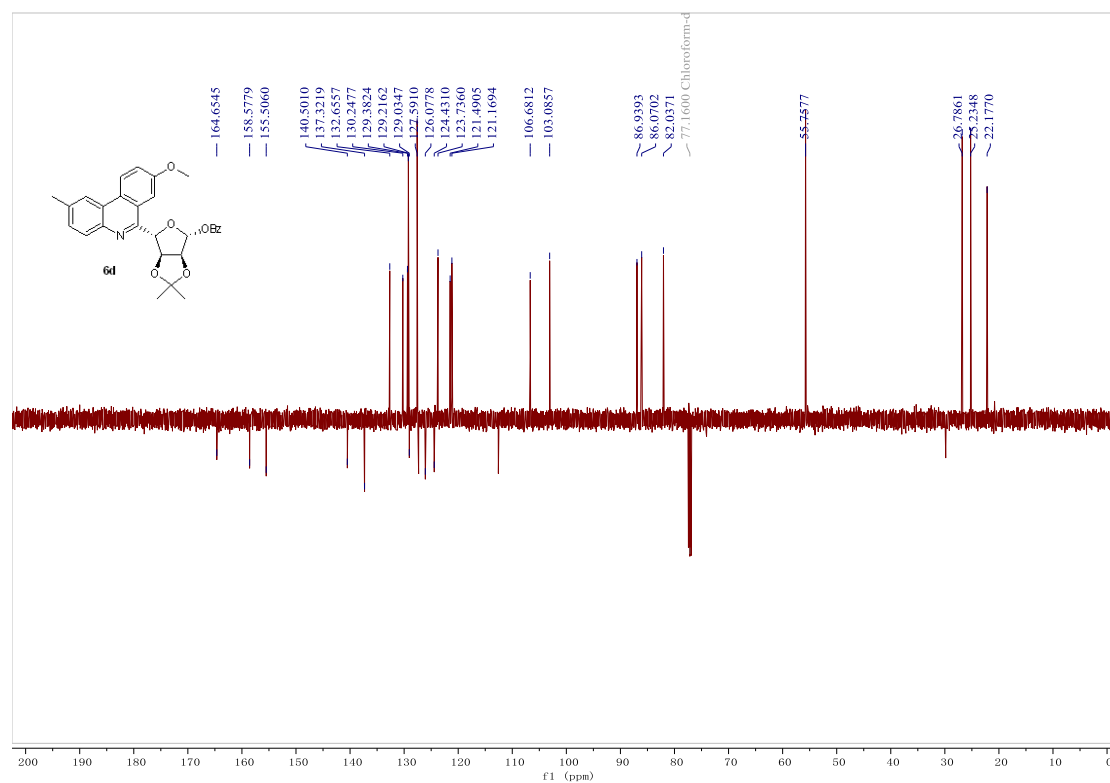
¹H NMR spectrum for **6c** (500 MHz, CDCl₃)



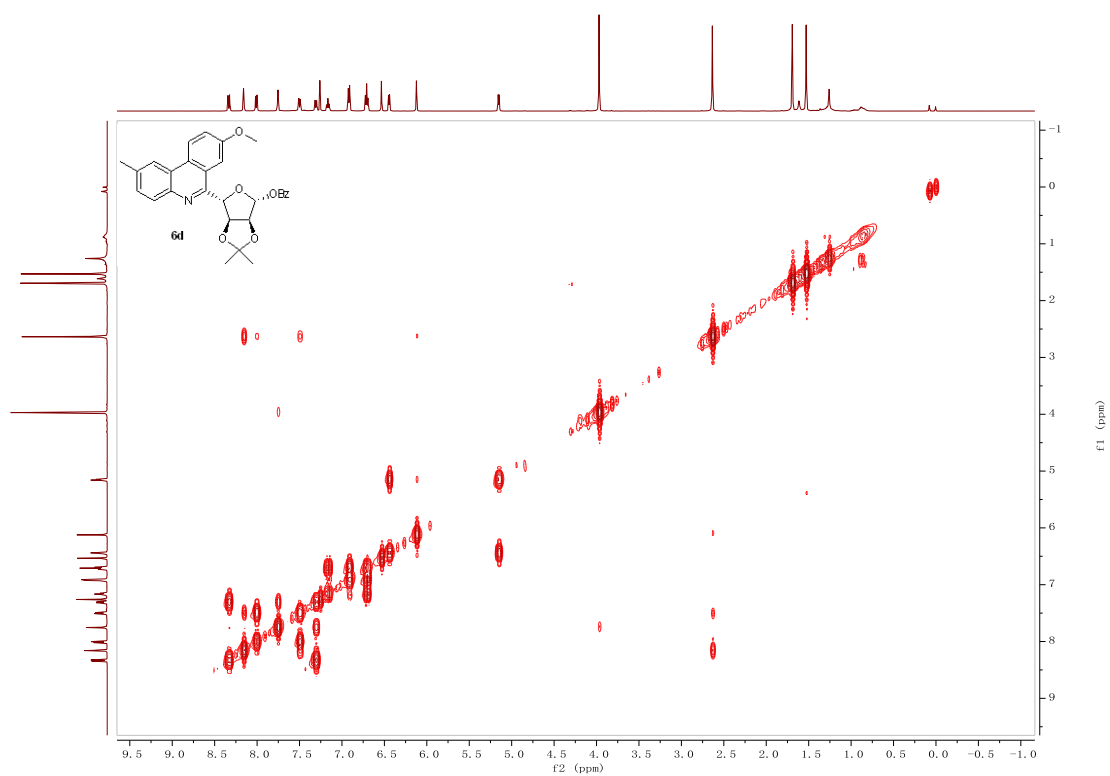
DEPT-Q spectrum for **6c** (126 MHz, CDCl₃)



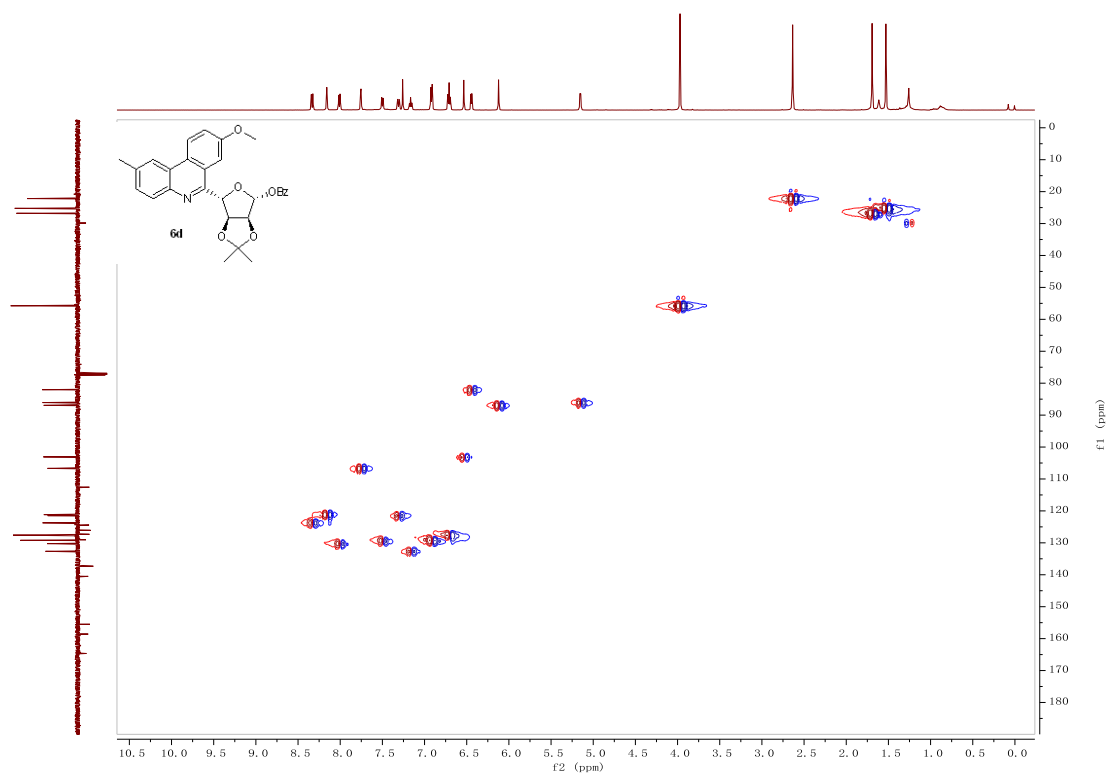
¹H NMR spectrum for 6d (500 MHz, CDCl₃)



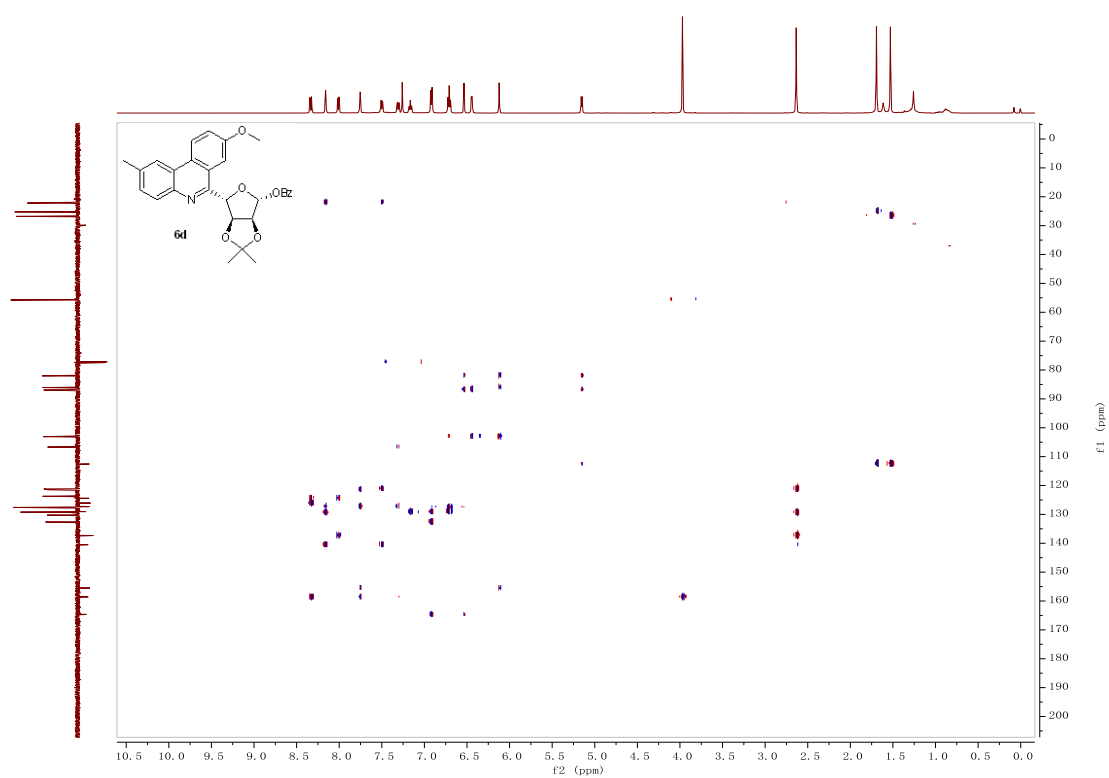
DEPT-Q spectrum for 6d (126 MHz, CDCl₃)



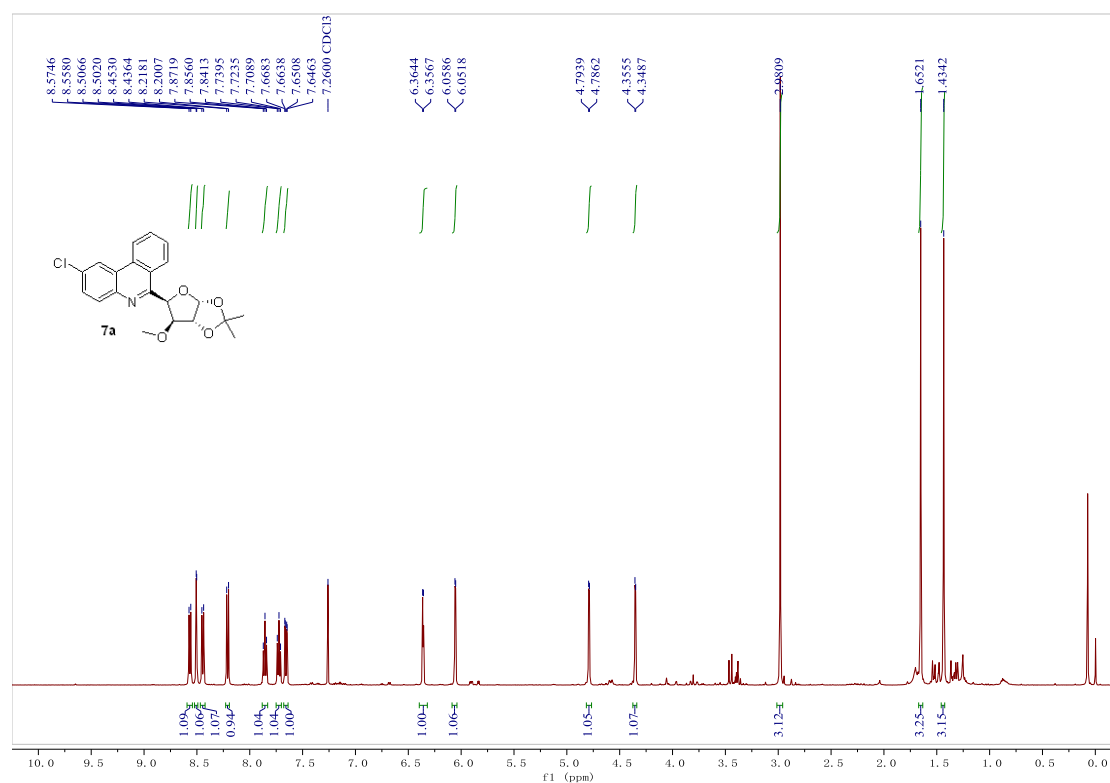
COSY spectrum for **6d**



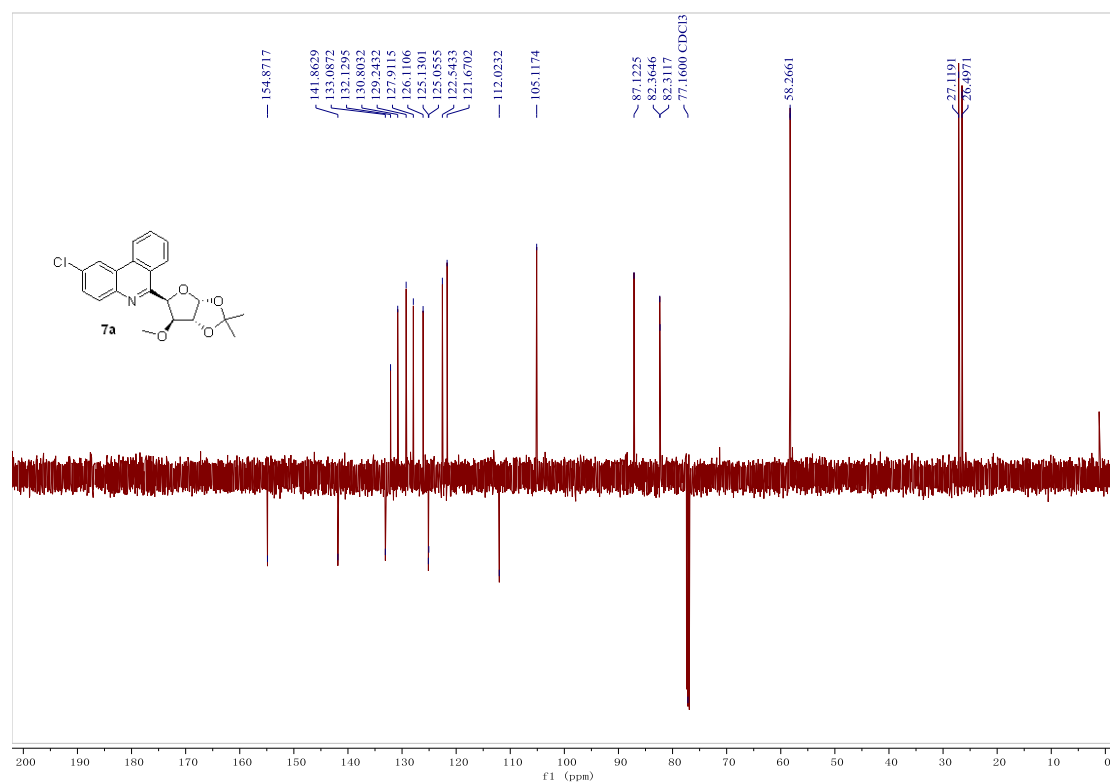
HSQC spectrum for **6d**



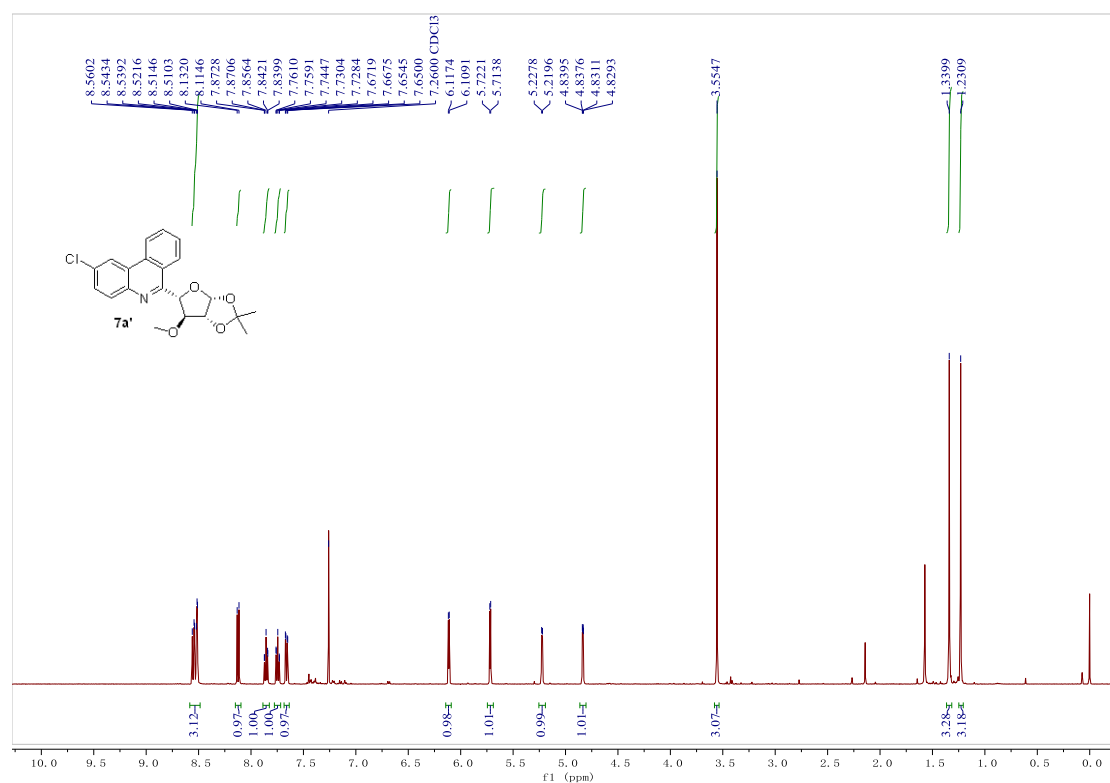
HMBC spectrum for **6d**



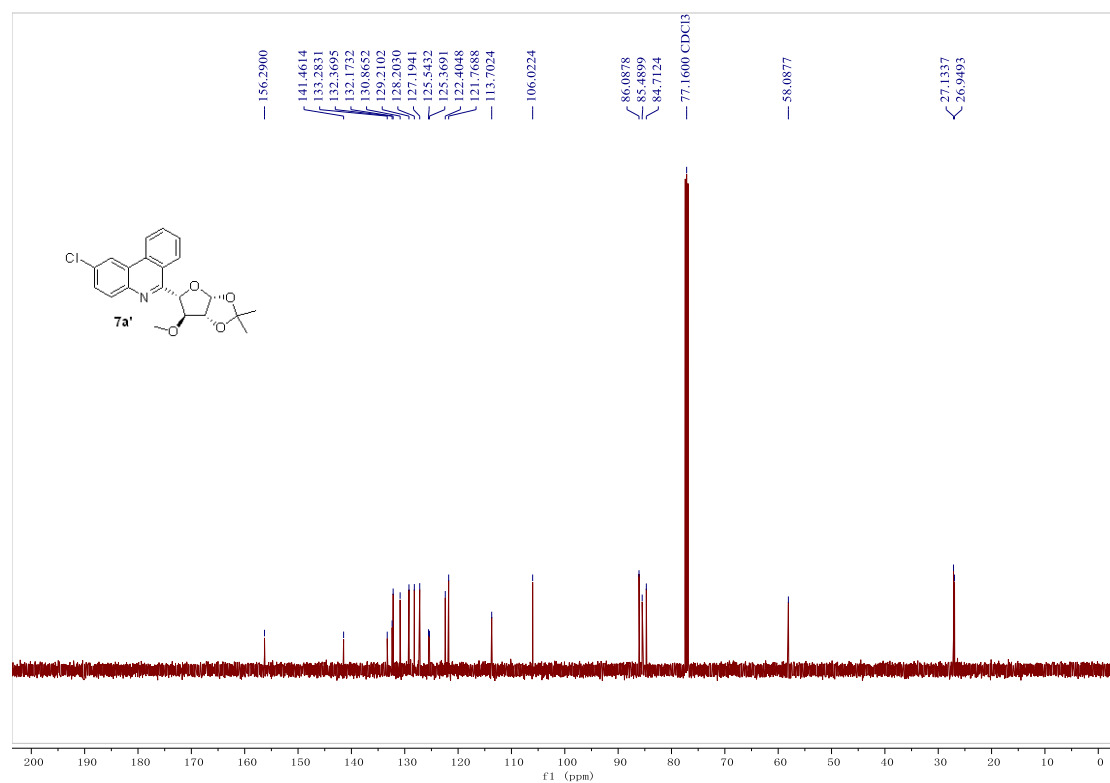
¹H NMR spectrum for 7a (500 MHz, CDCl₃)



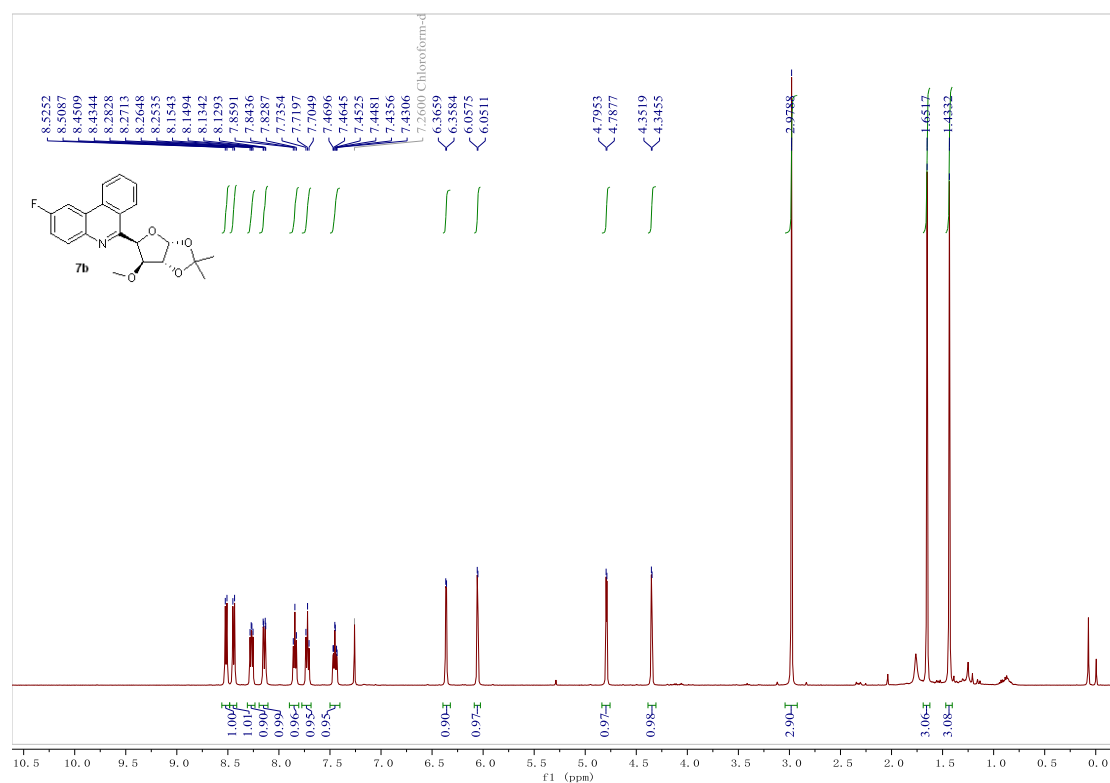
¹³C NMR spectrum for 7a (126 MHz, CDCl₃)



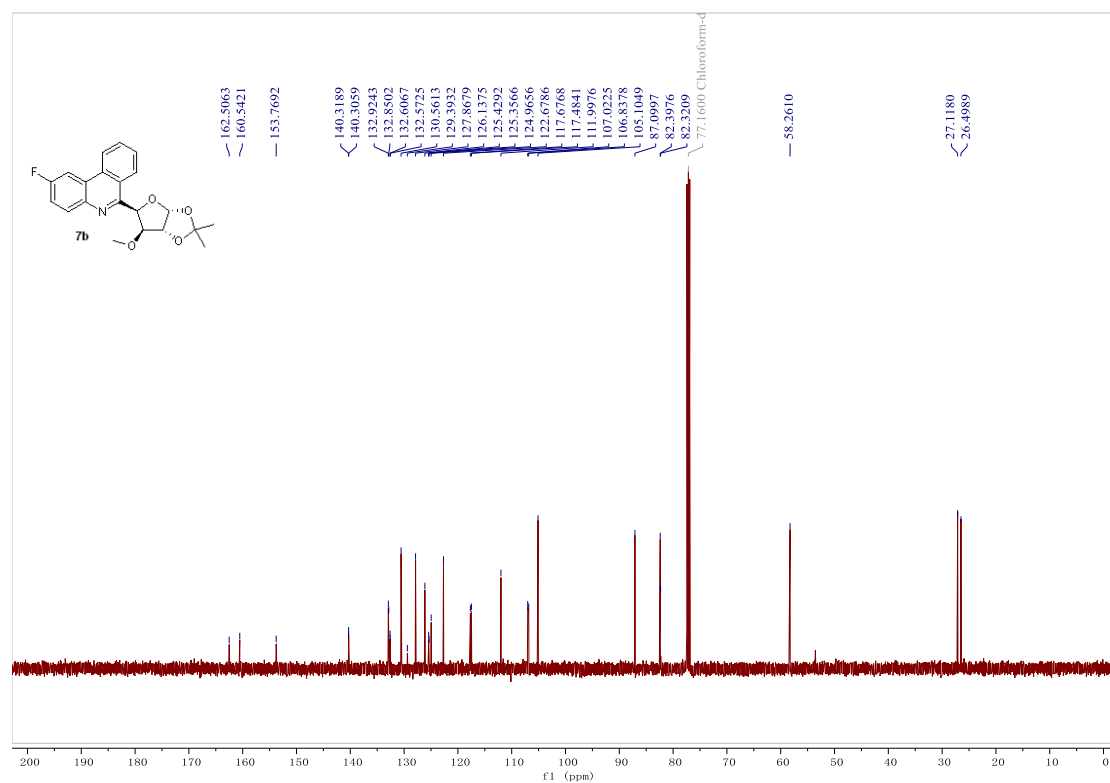
¹H NMR spectrum for 7a' (500 MHz, CDCl₃)



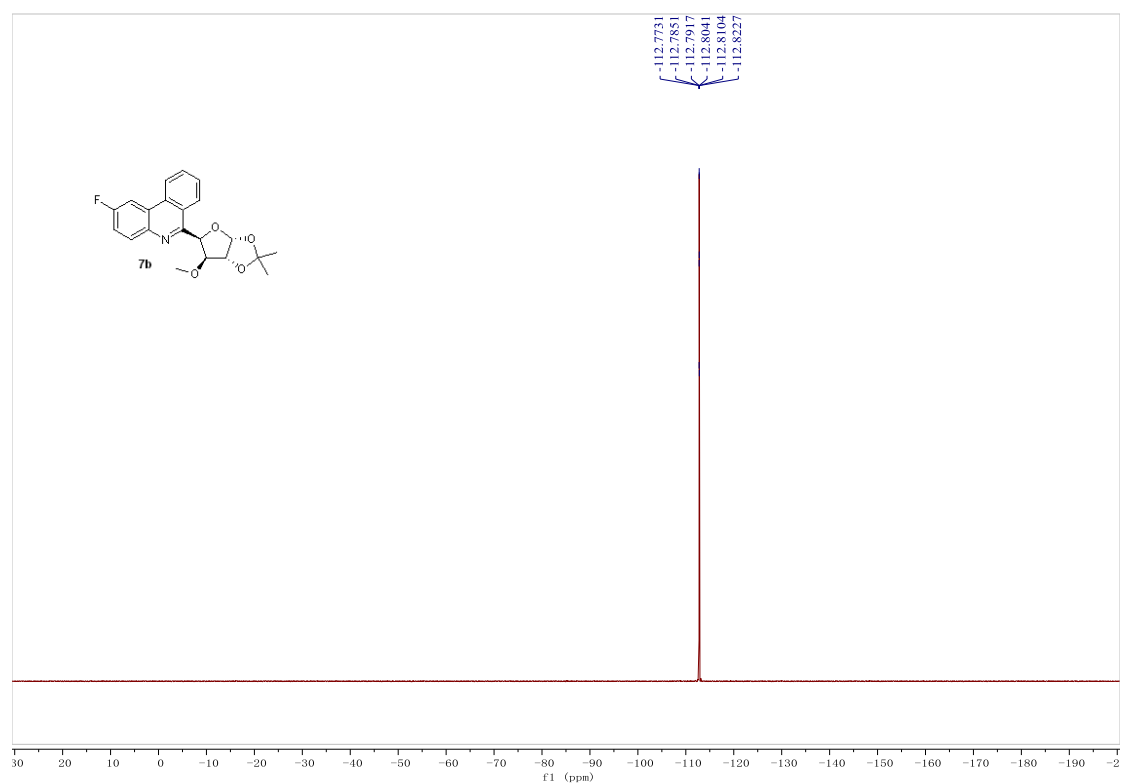
DEPT-Q spectrum for 7a' (126 MHz, CDCl₃)



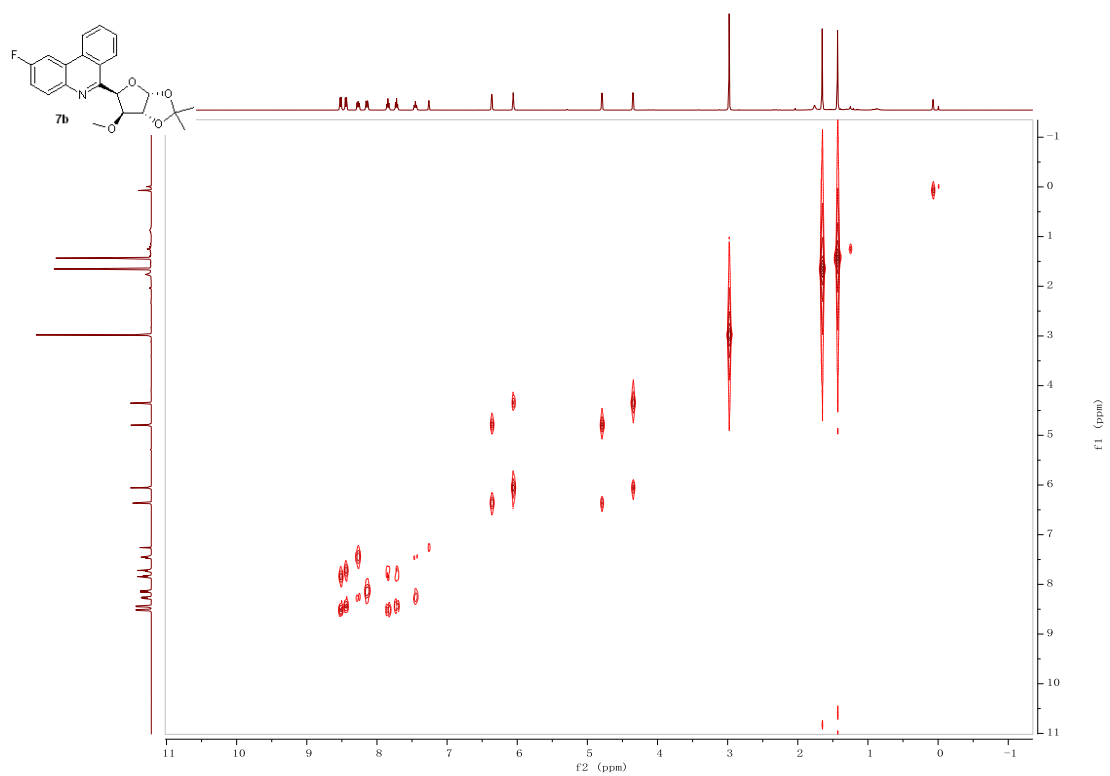
¹H NMR spectrum for 7b (500 MHz, CDCl₃)



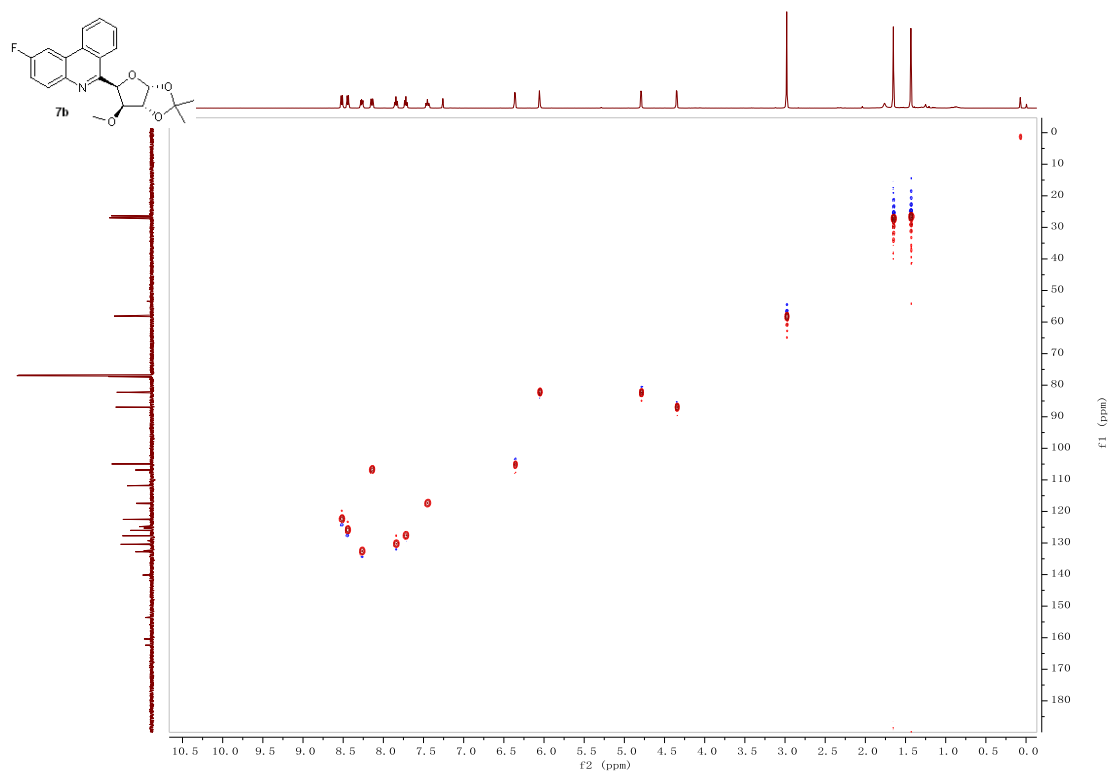
¹³C NMR spectrum for 7b (126 MHz, CDCl₃)



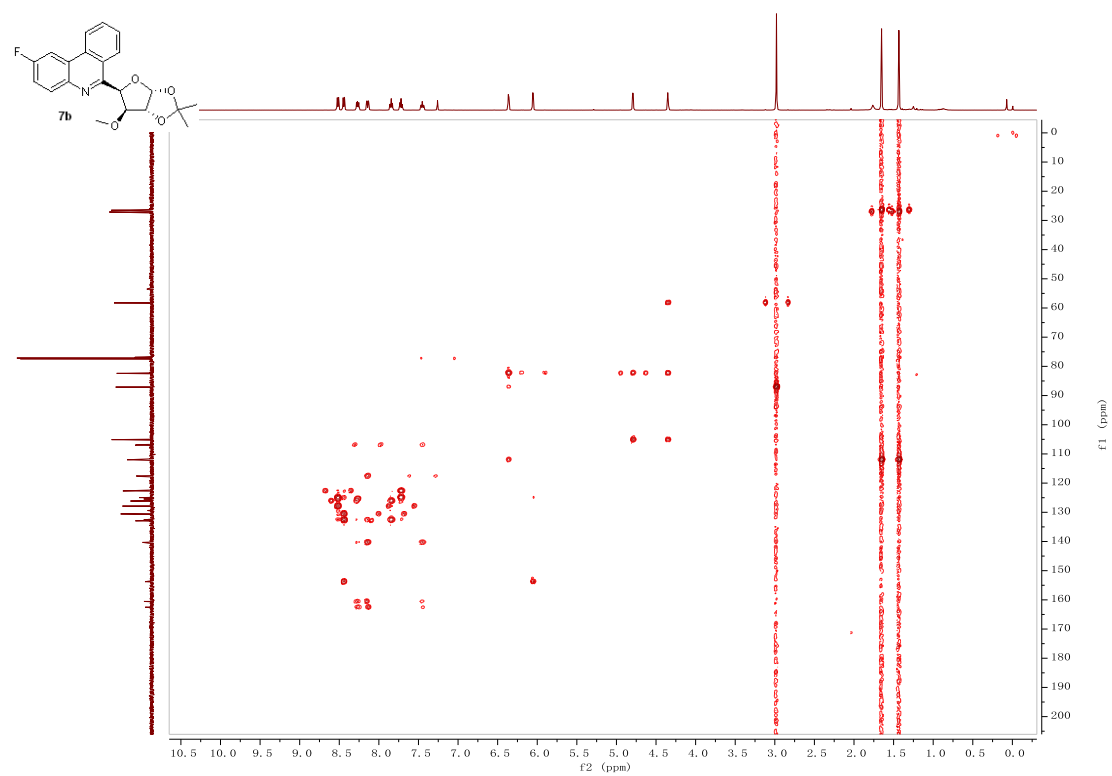
¹⁹F NMR spectrum for **7b**



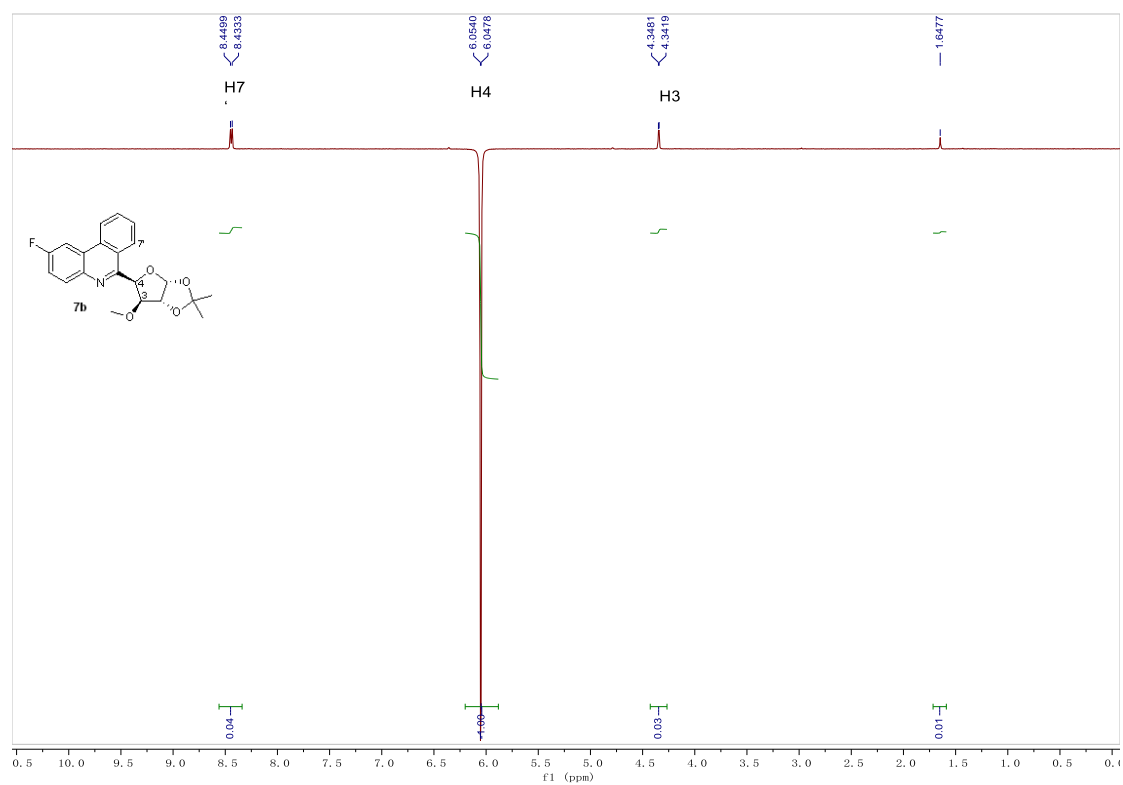
COSY spectrum for **7b**



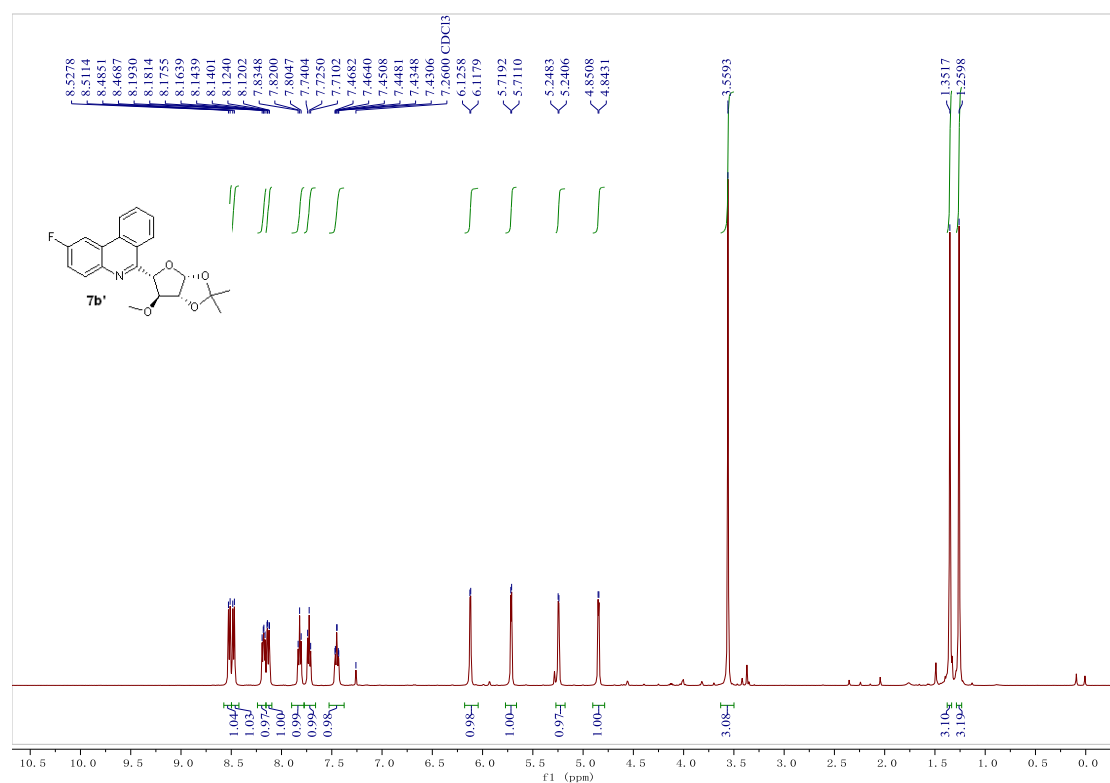
HSQC spectrum for **7b**



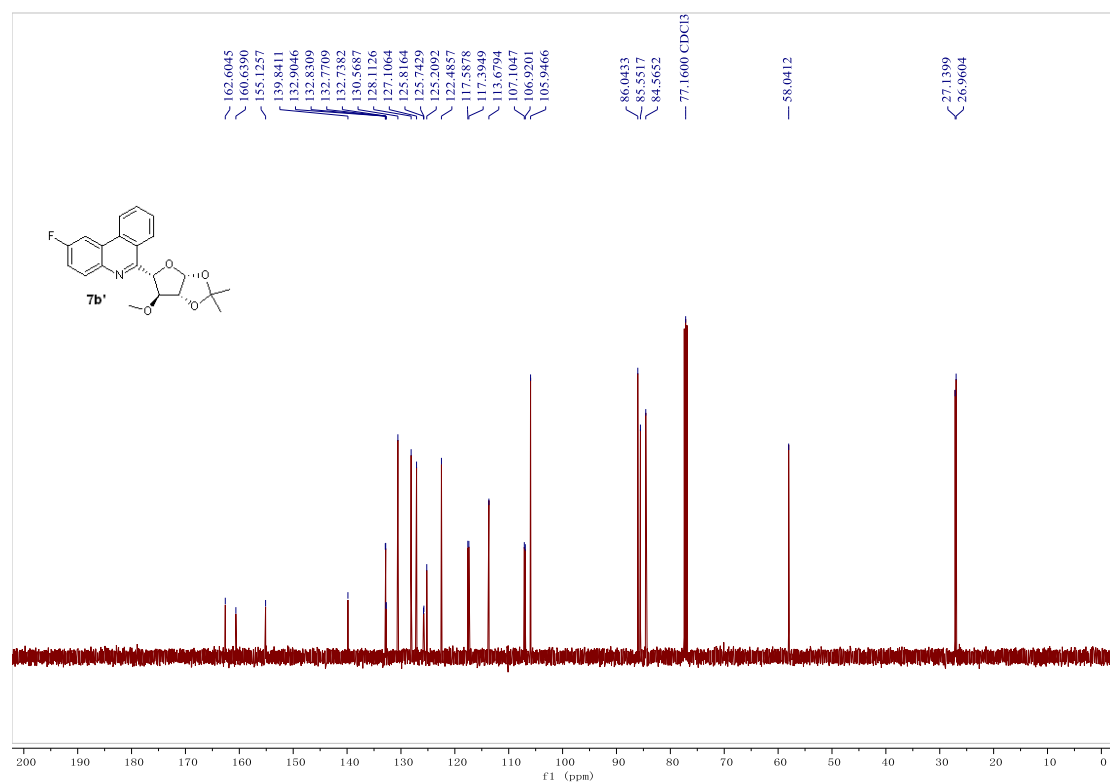
HMBC spectrum for **7b**



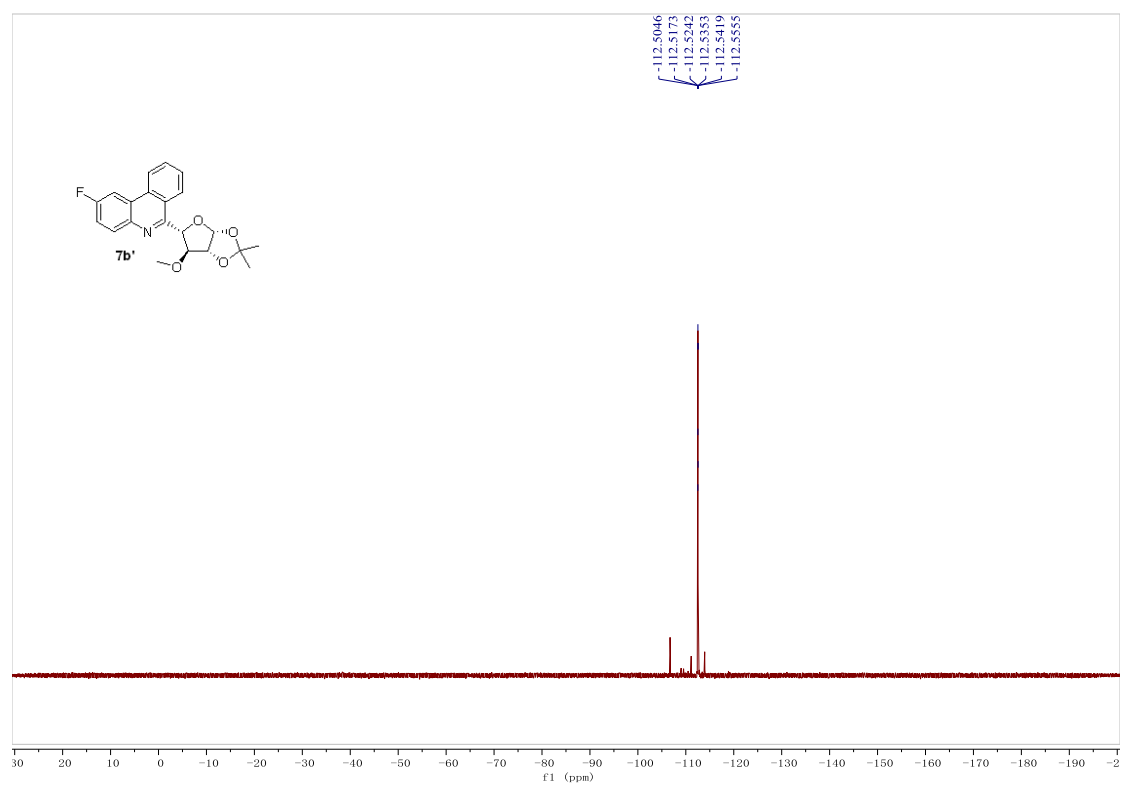
NOE difference spectrum for **7b**



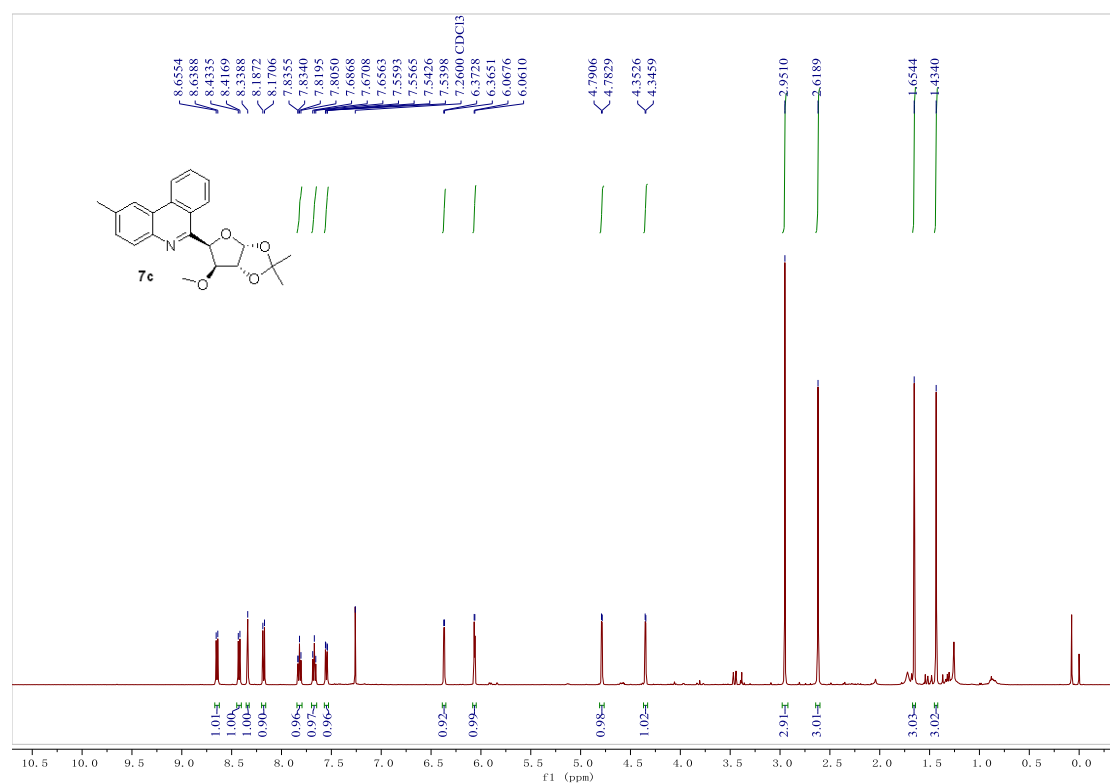
¹H NMR spectrum for 7b' (500 MHz, CDCl₃)



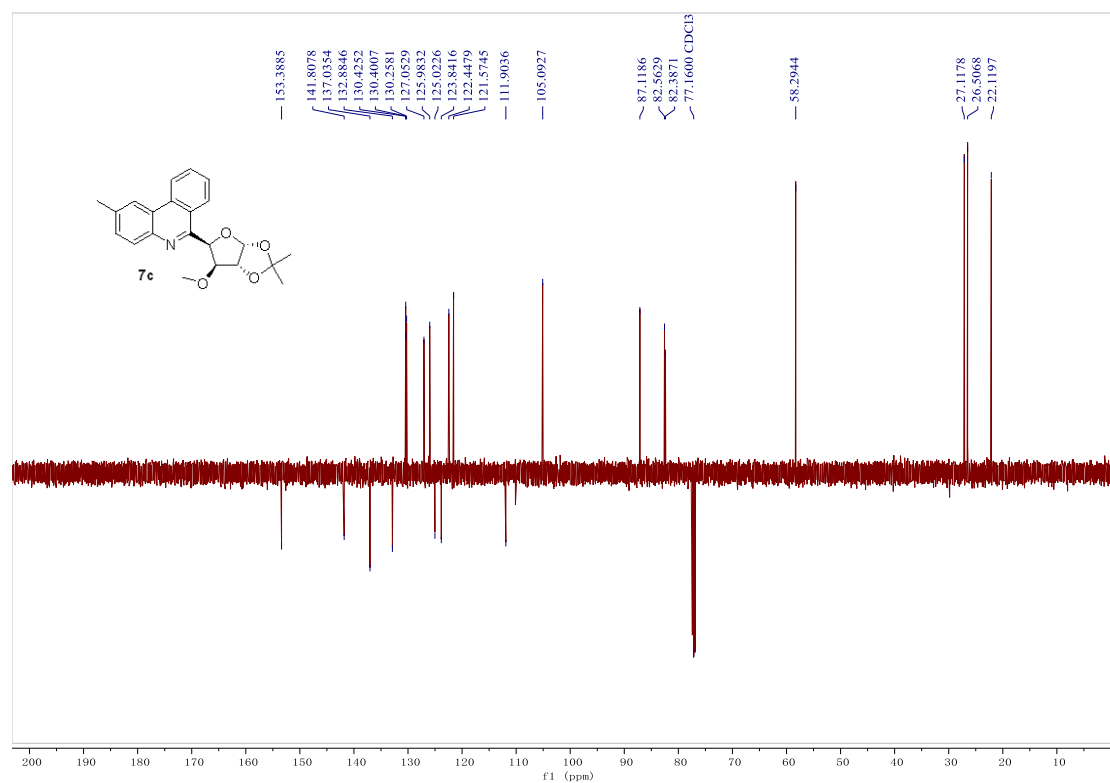
¹³C NMR spectrum for 7b' (126 MHz, CDCl₃)



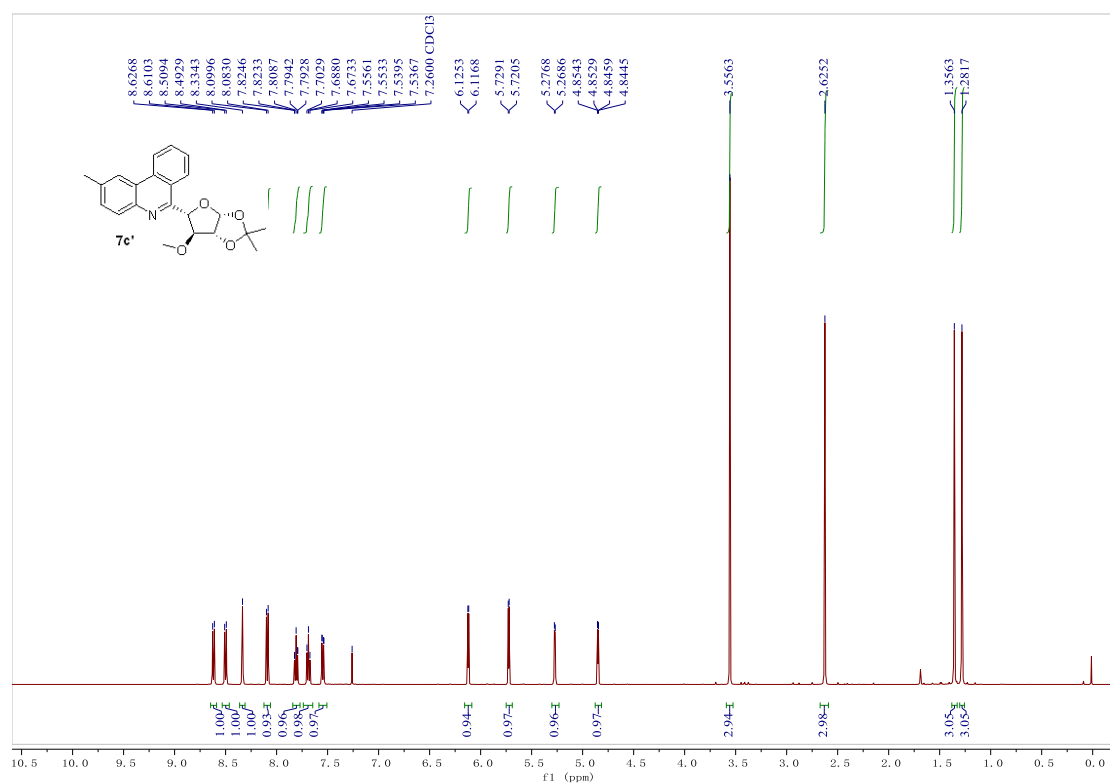
^{19}F NMR spectrum for **7b'**



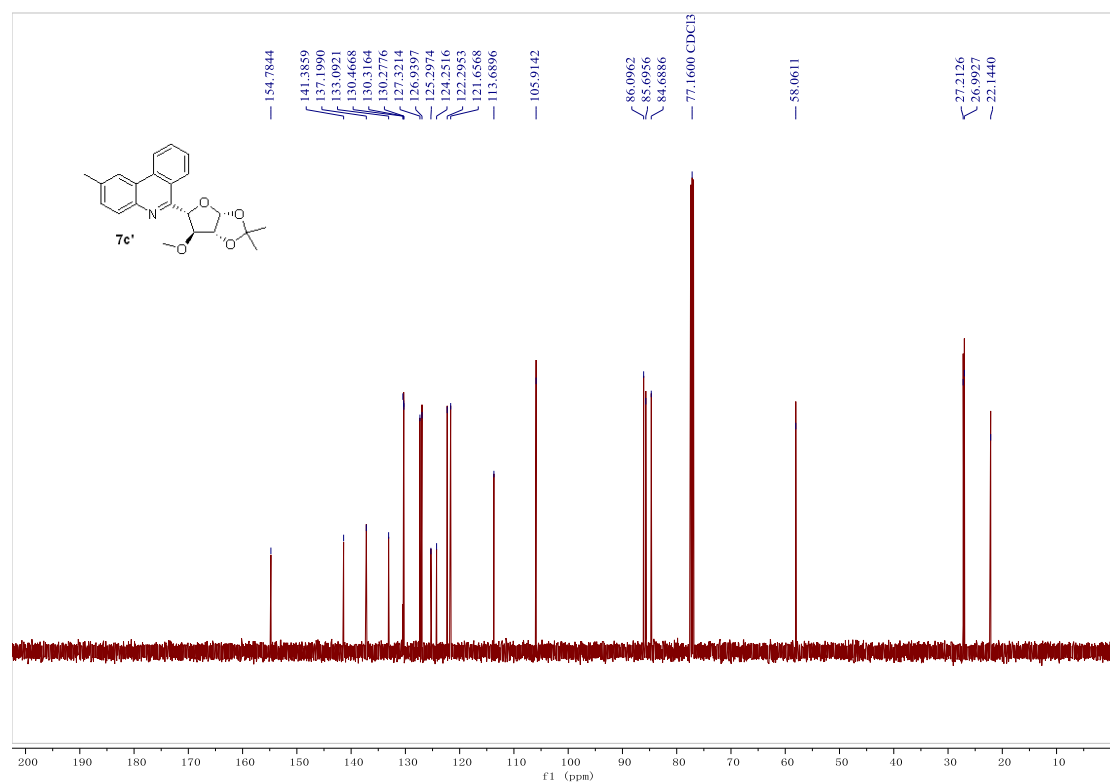
¹H NMR spectrum for 7c (500 MHz, CDCl₃)



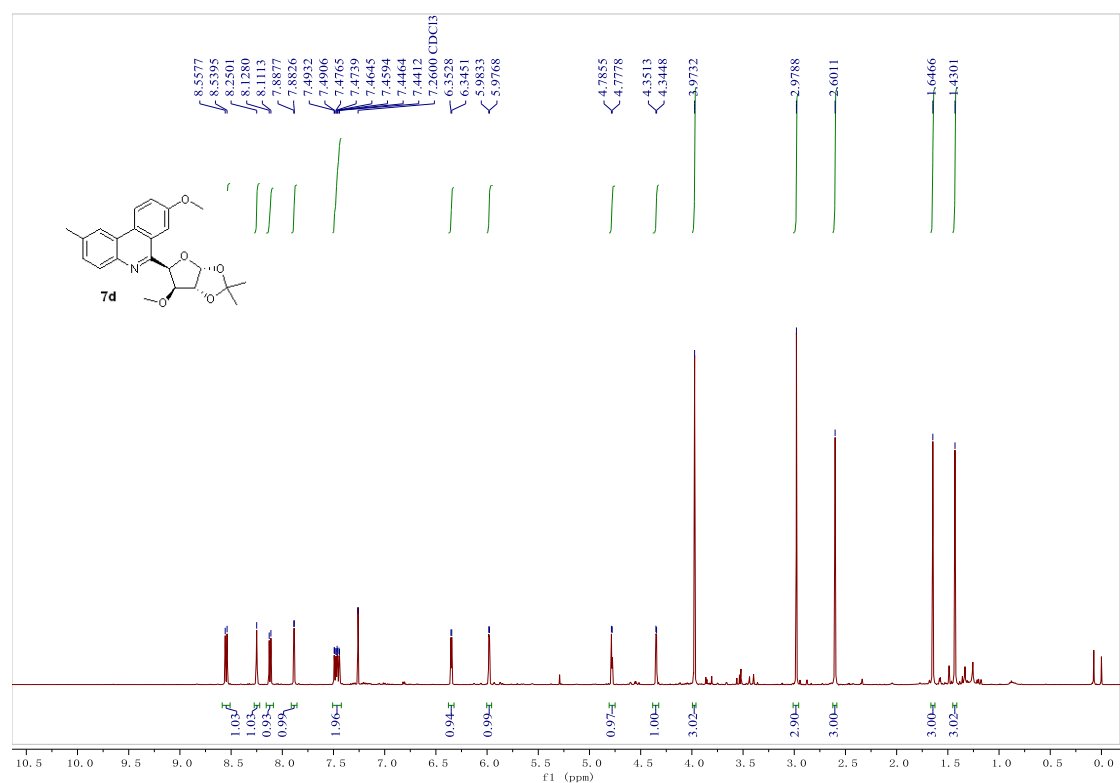
DEPT-Q spectrum for 7c (126 MHz, CDCl₃)



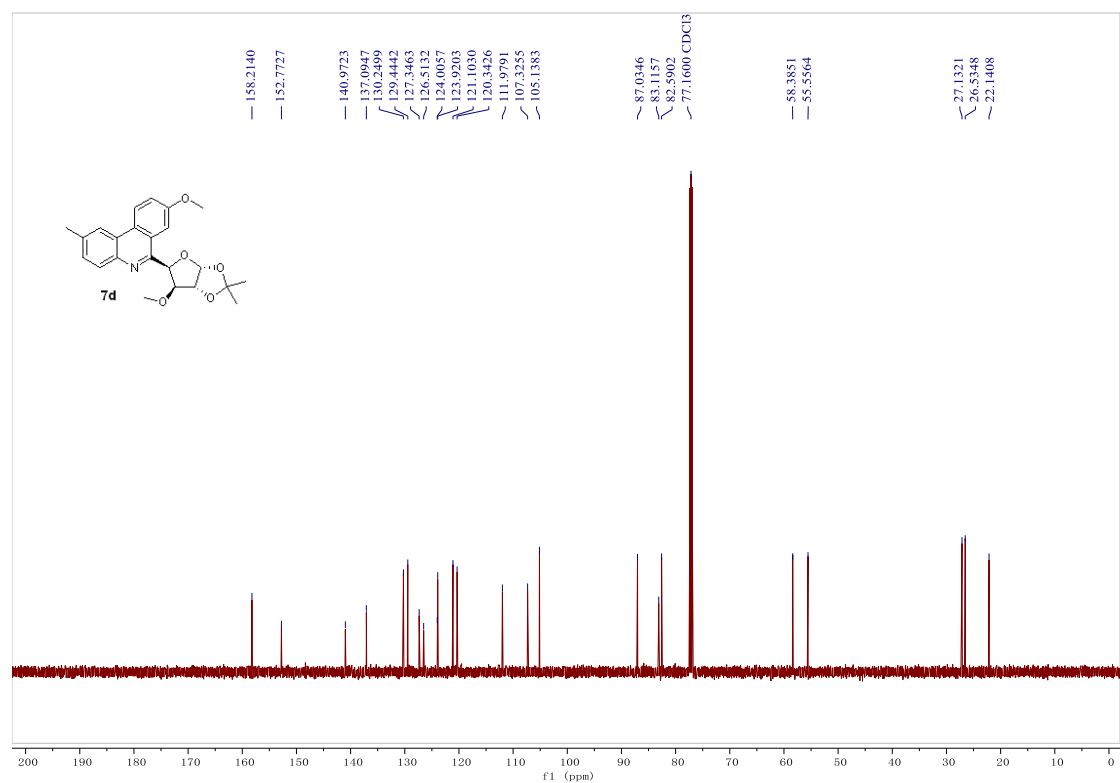
¹H NMR spectrum for 7c' (500 MHz, CDCl₃)



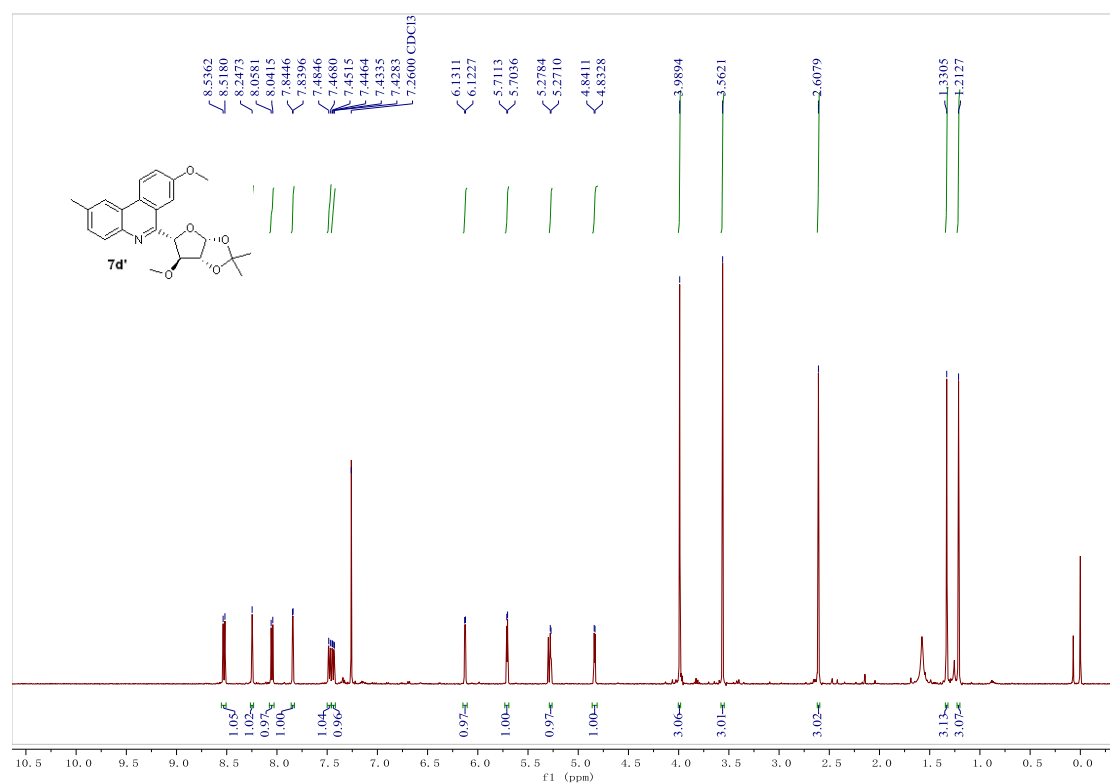
¹³C NMR spectrum for 7c' (126 MHz, CDCl₃)



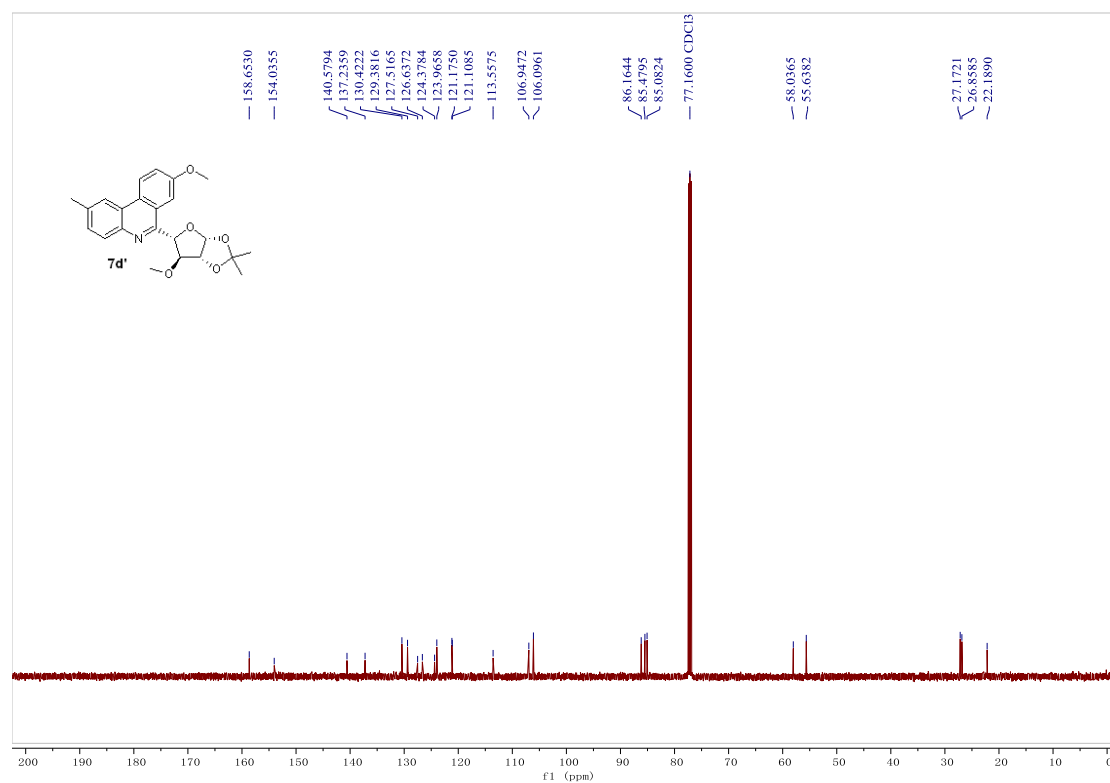
¹H NMR spectrum for 7d (500 MHz, CDCl₃)



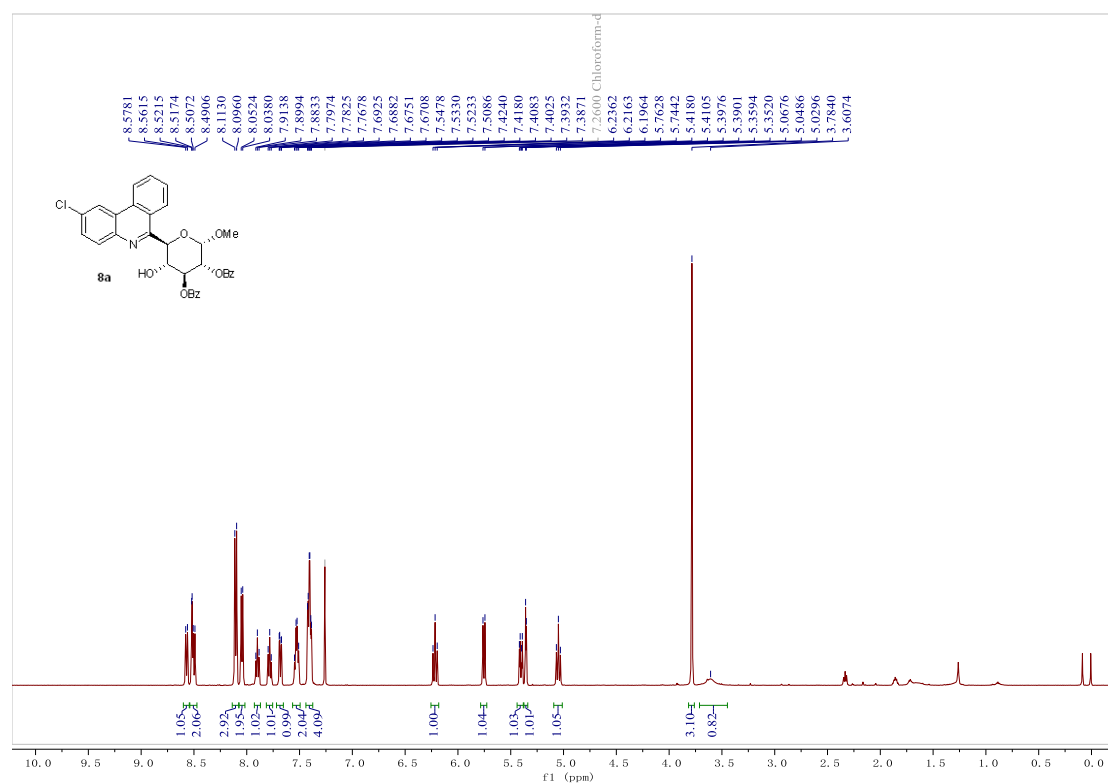
¹³C NMR spectrum for 7d (126 MHz, CDCl₃)



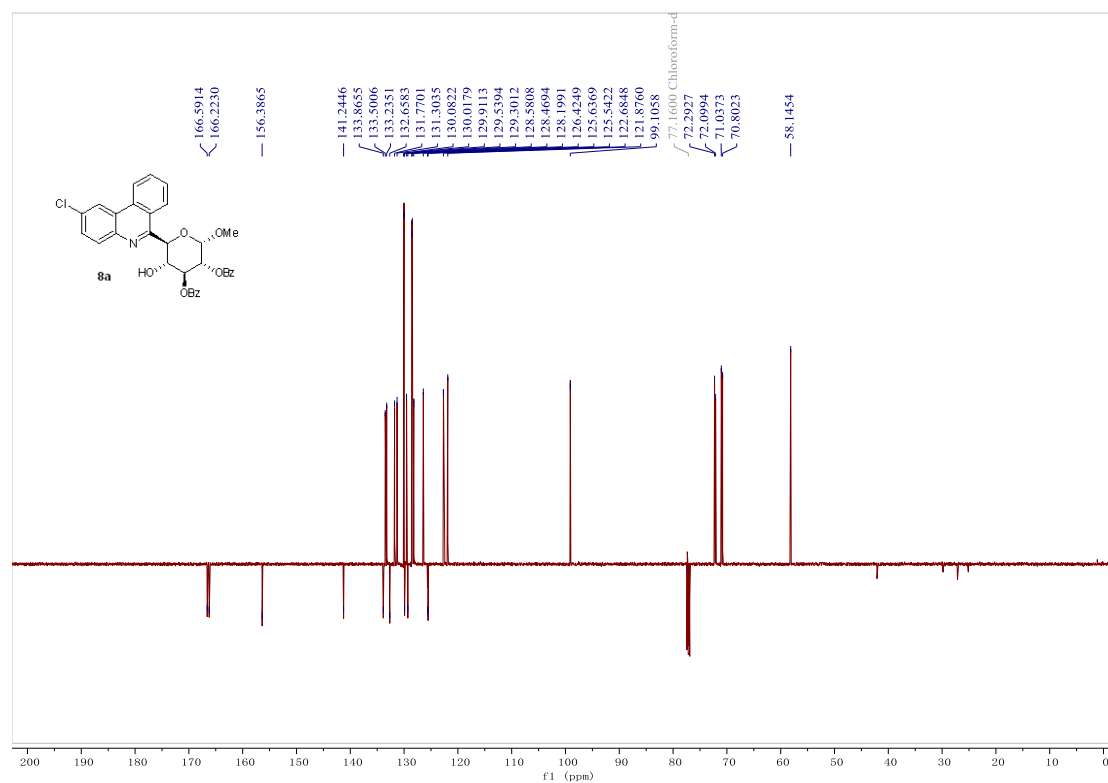
¹H NMR spectrum for 7d' (500 MHz, CDCl₃)



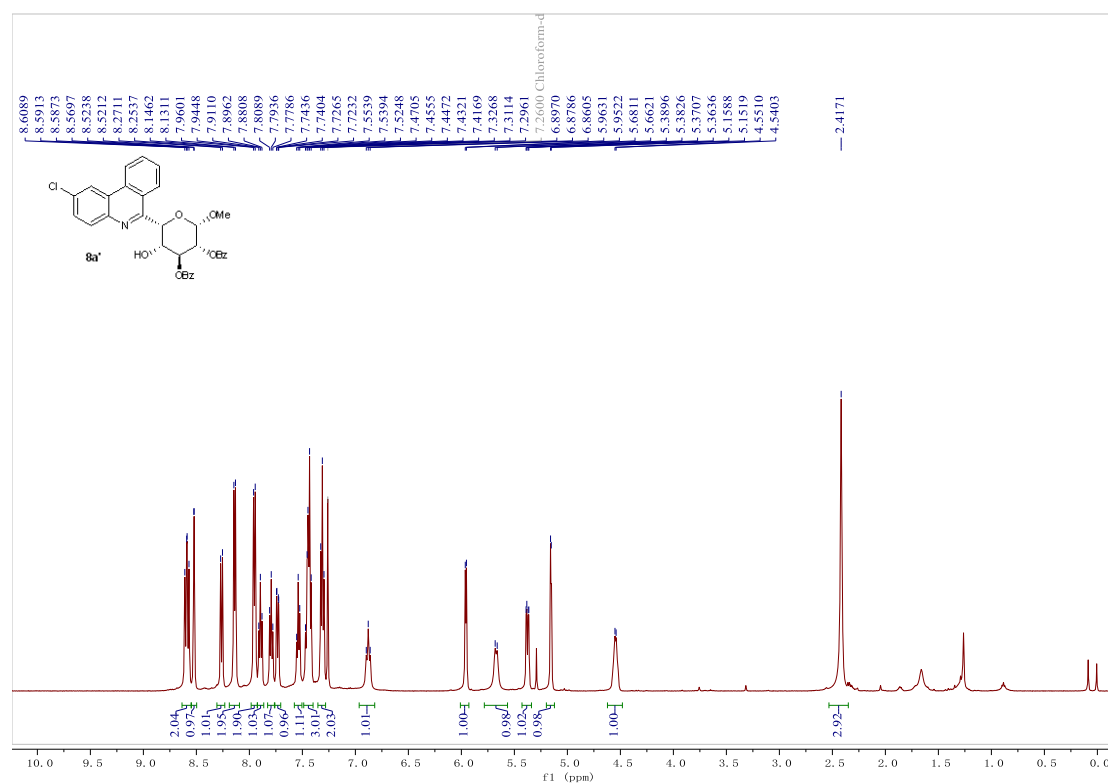
¹³C NMR spectrum for 7d' (126 MHz, CDCl₃)



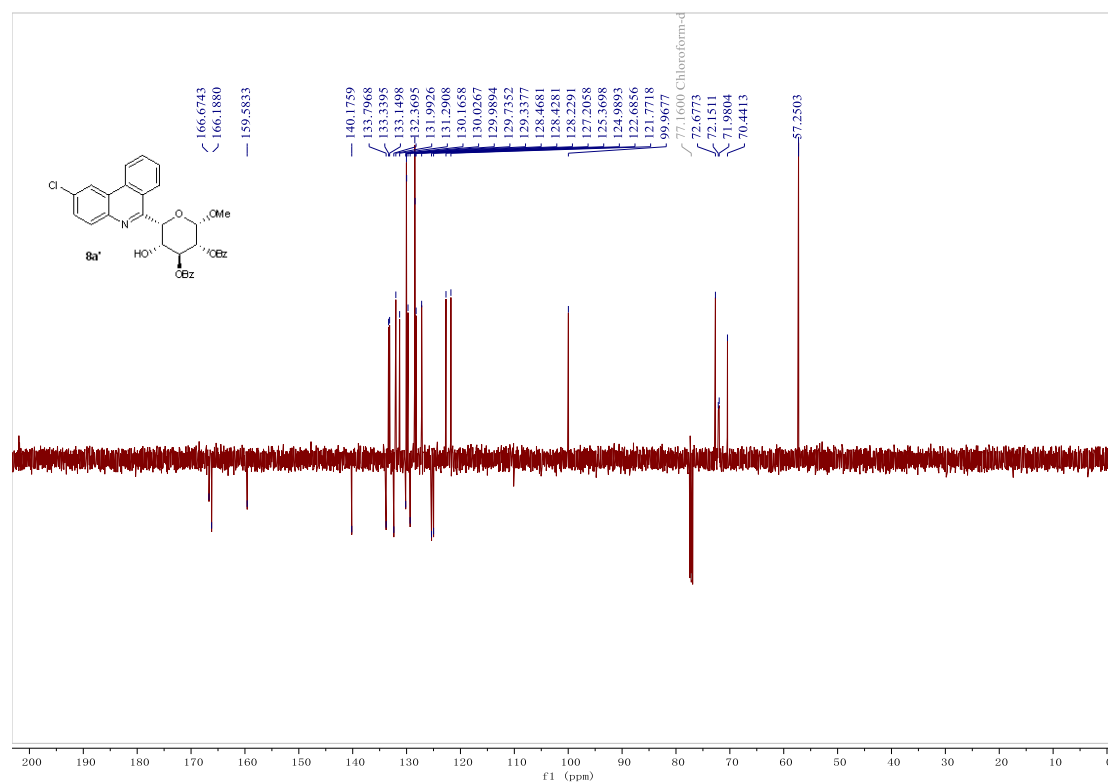
¹H NMR spectrum for **8a** (500 MHz, CDCl₃)



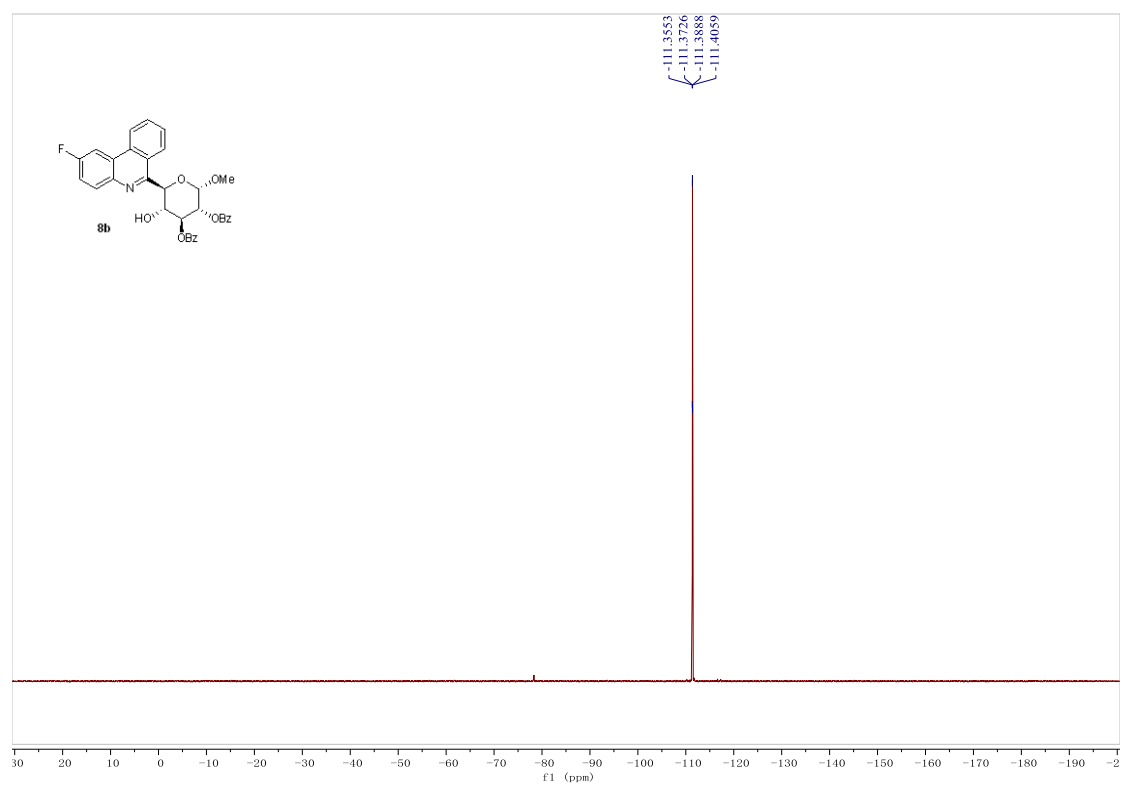
DEPT-Q spectrum for **8a** (126 MHz, CDCl₃)



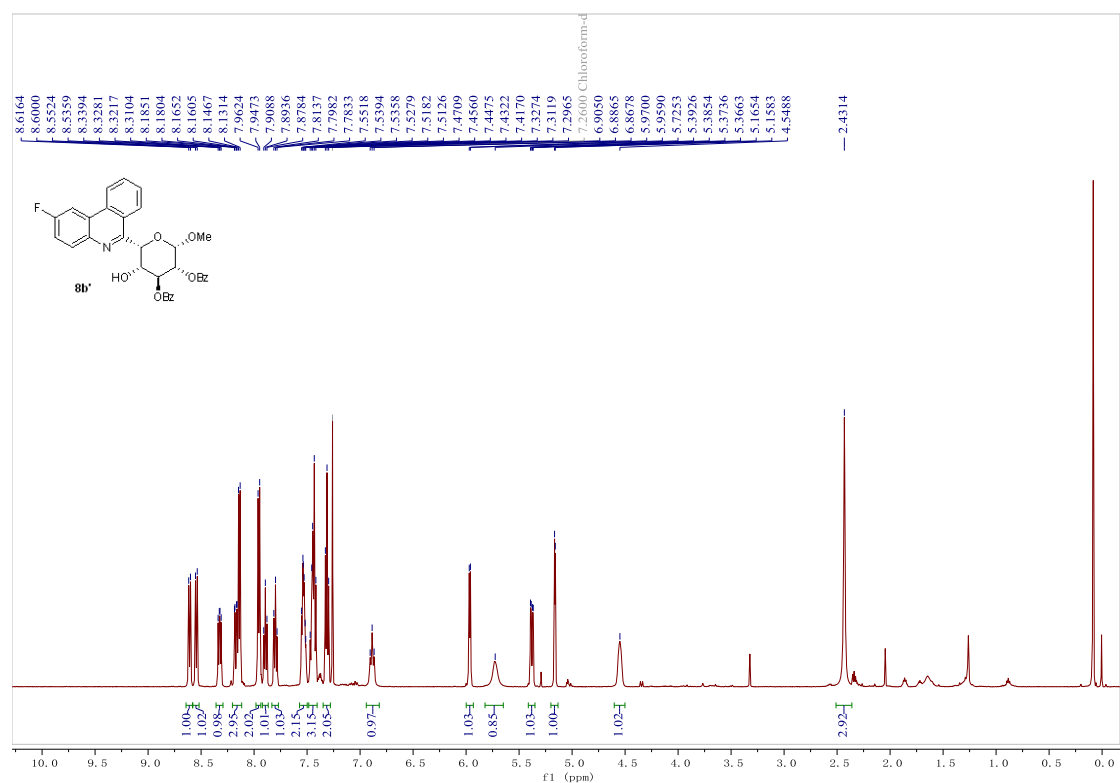
¹H NMR spectrum for **8a'** (500 MHz, CDCl₃)



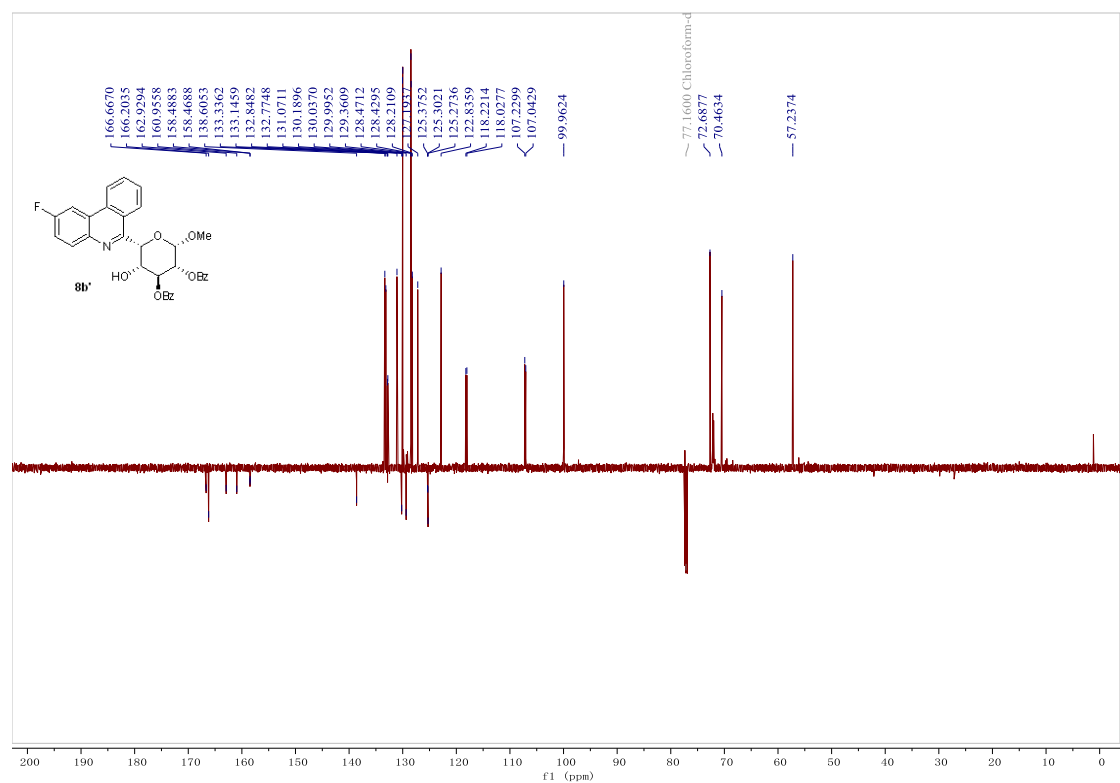
DEPT-Q spectrum for **8a'** (126 MHz, CDCl₃)



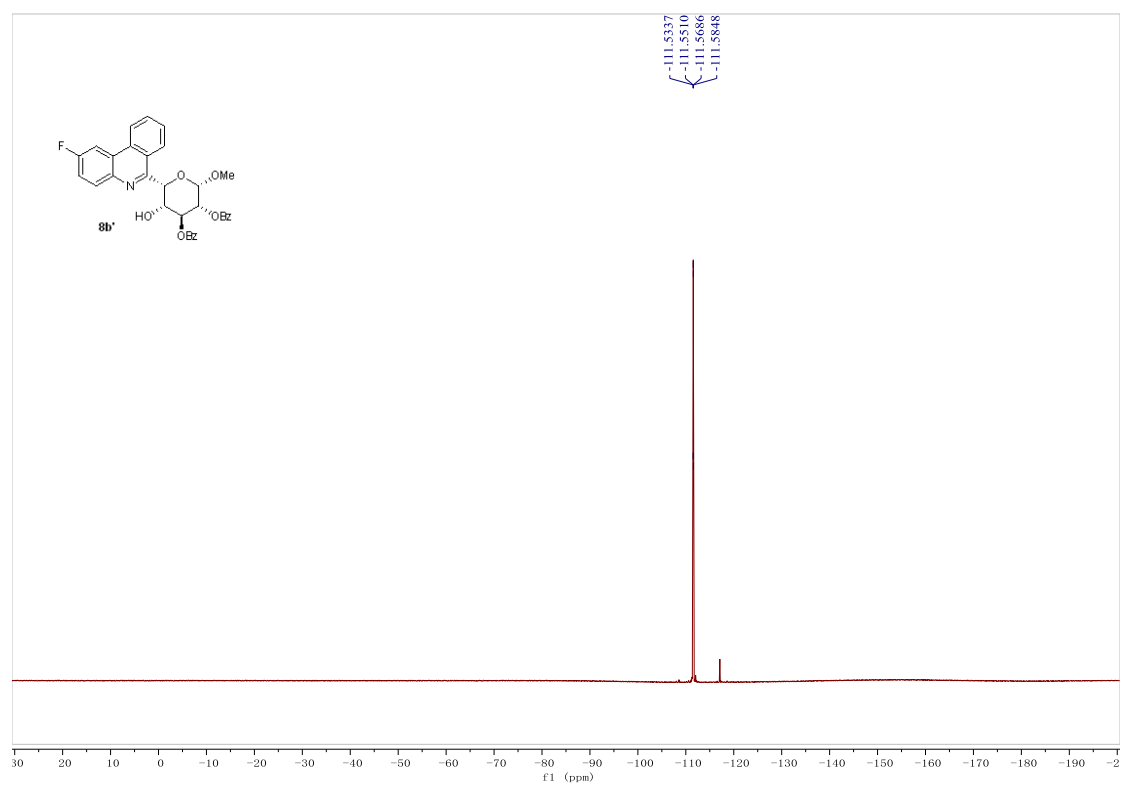
^{19}F NMR spectrum for **8b**



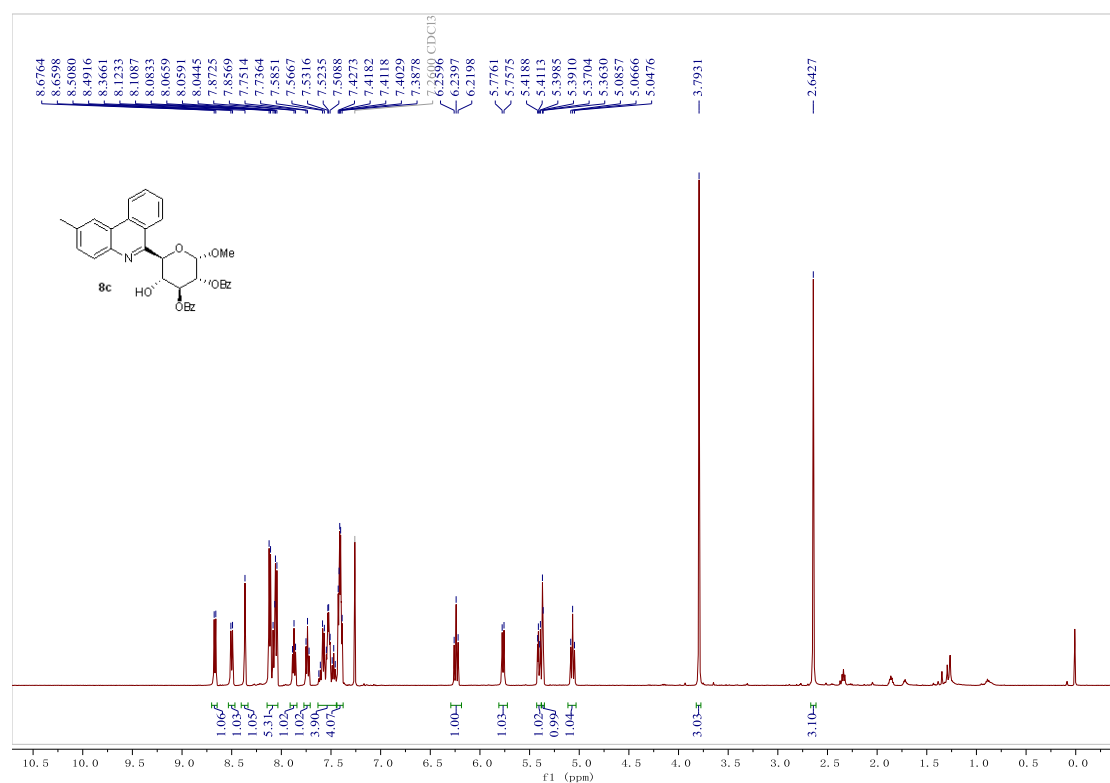
¹H NMR spectrum for **8b' (500 MHz, CDCl₃)**



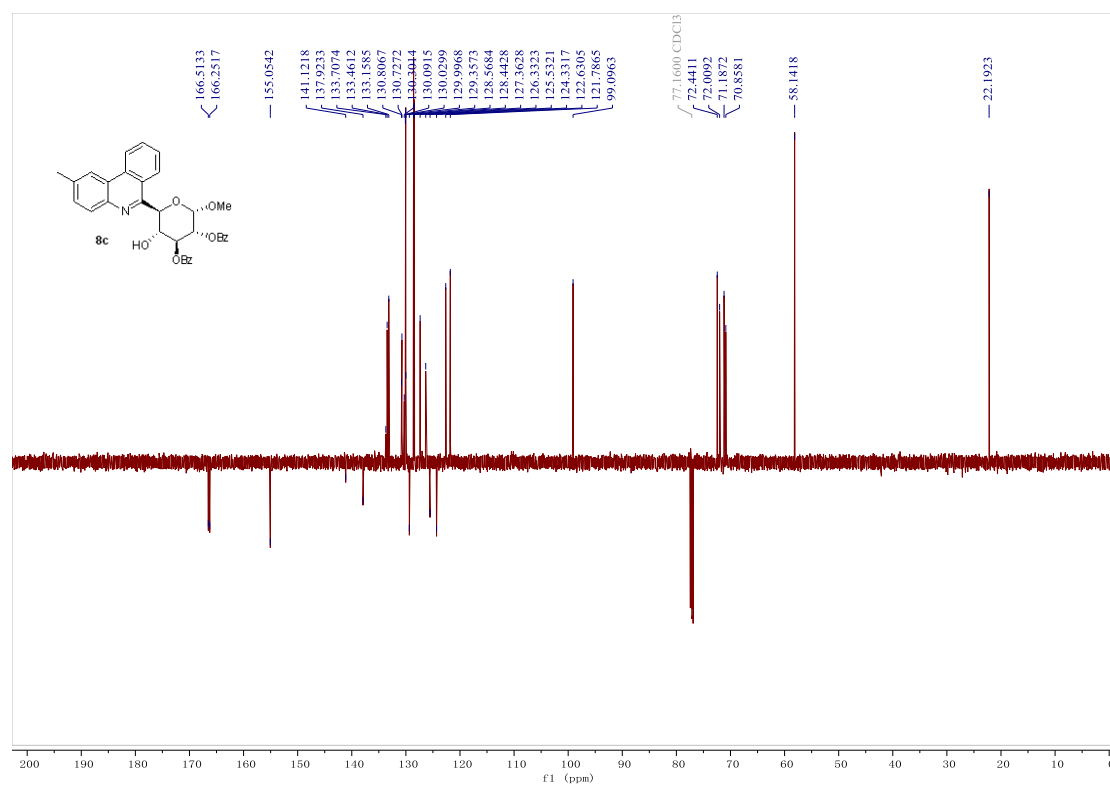
DEPT-Q spectrum for **8b' (126 MHz, CDCl₃)**



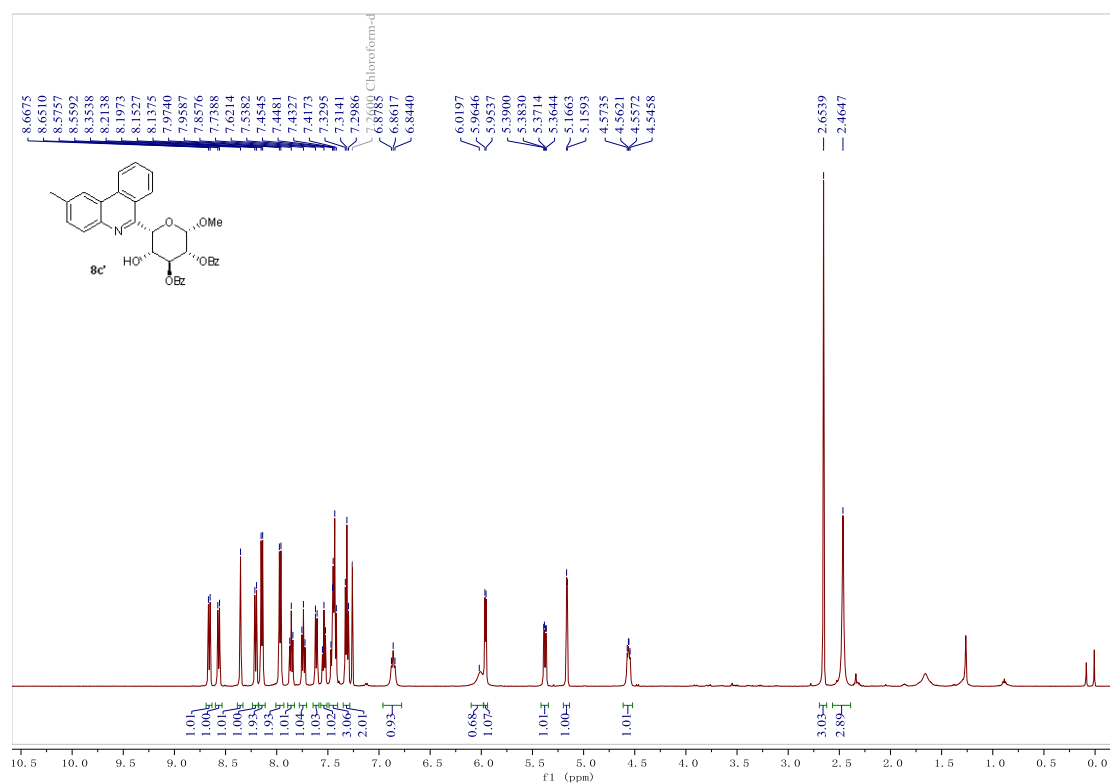
^{19}F NMR spectrum for **8b'**



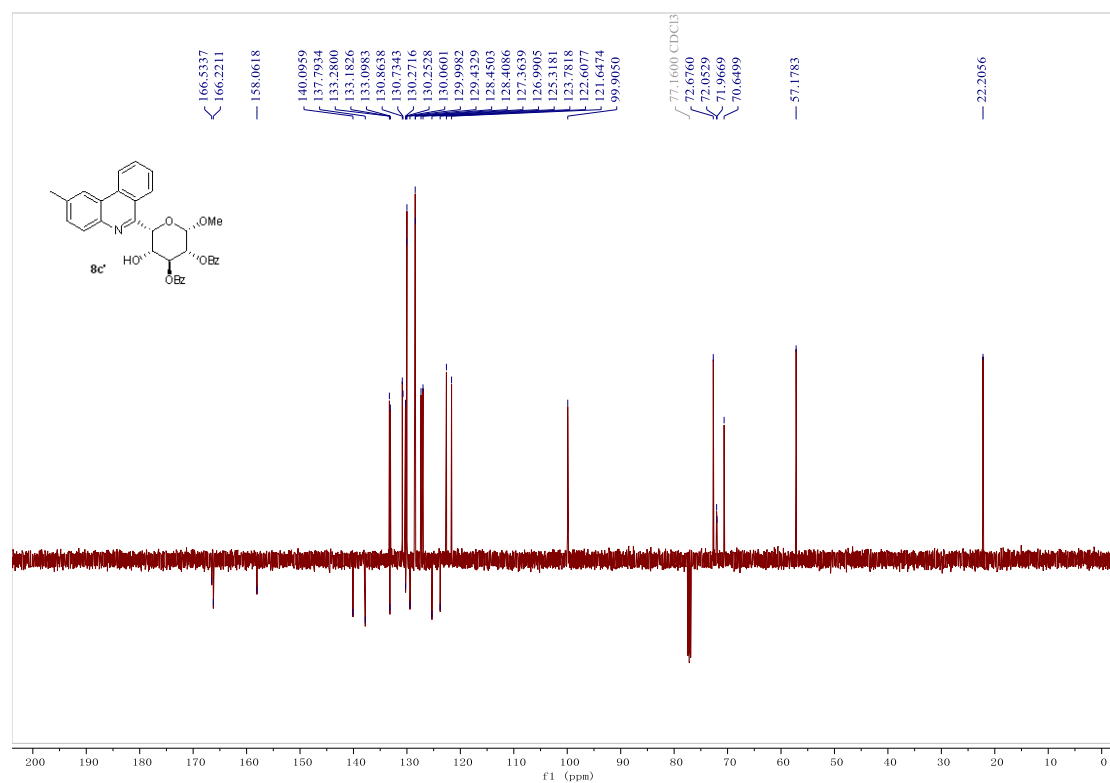
¹H NMR spectrum for 8c (500 MHz, CDCl₃)



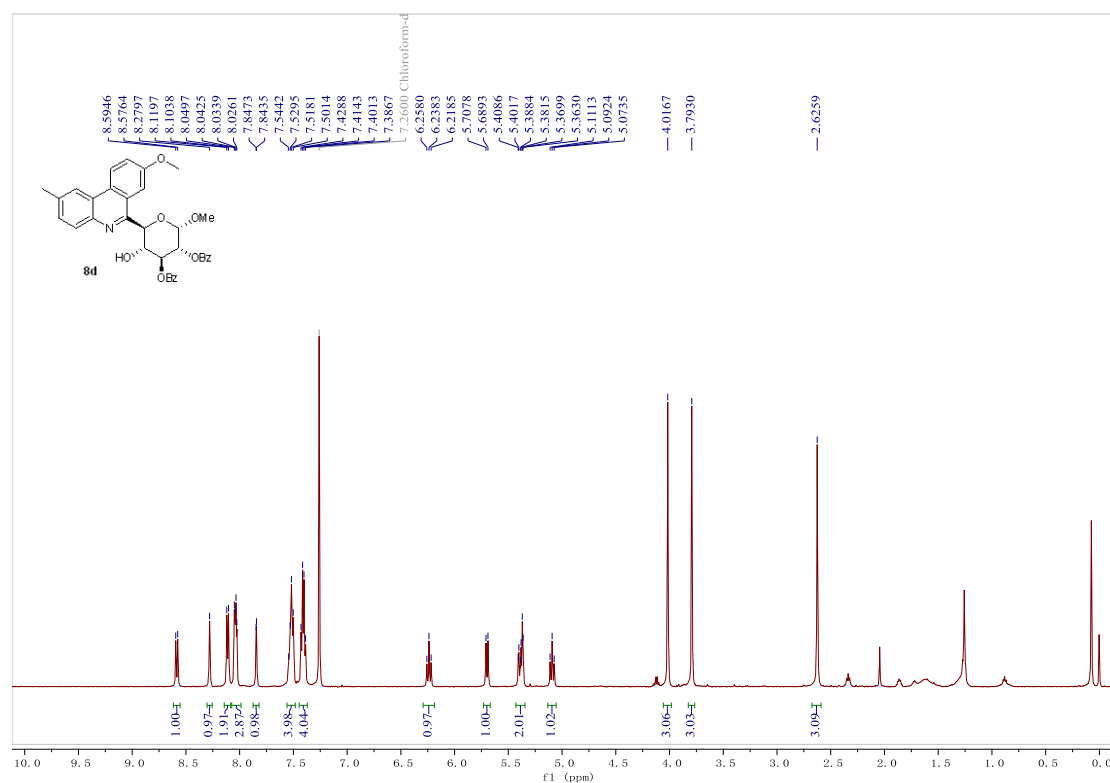
DEPT-Q spectrum for 8c (126 MHz, CDCl₃)



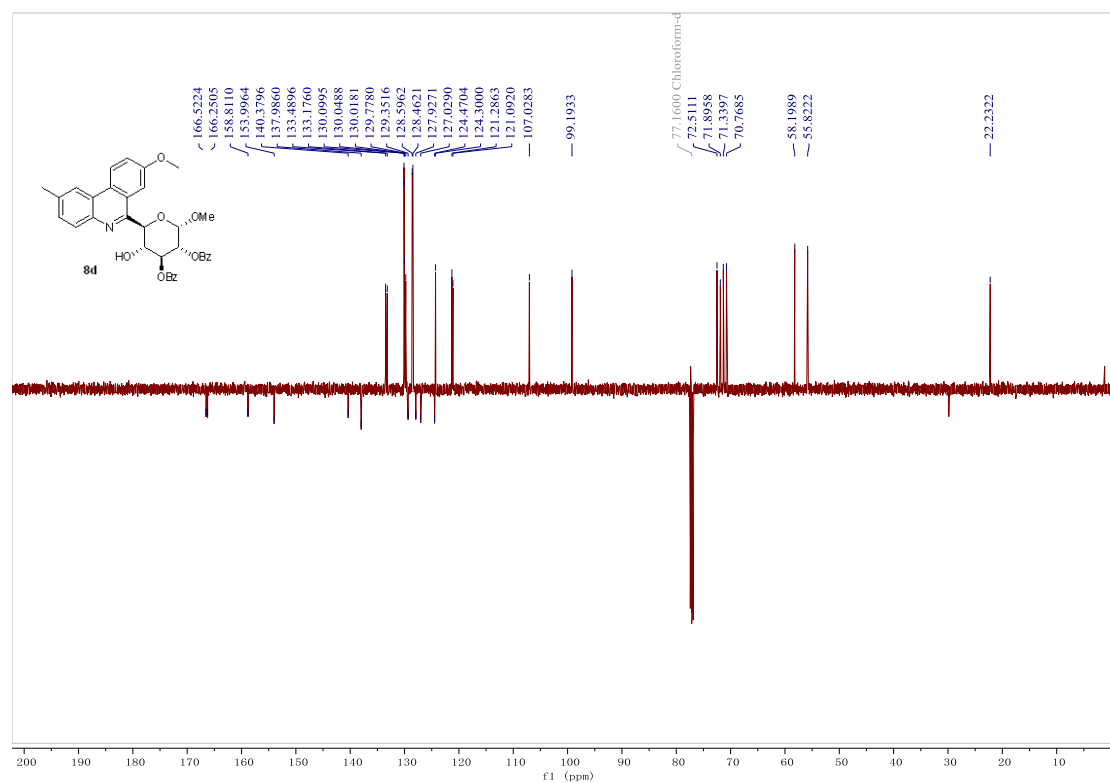
¹H NMR spectrum for 8c' (500 MHz, CDCl₃)



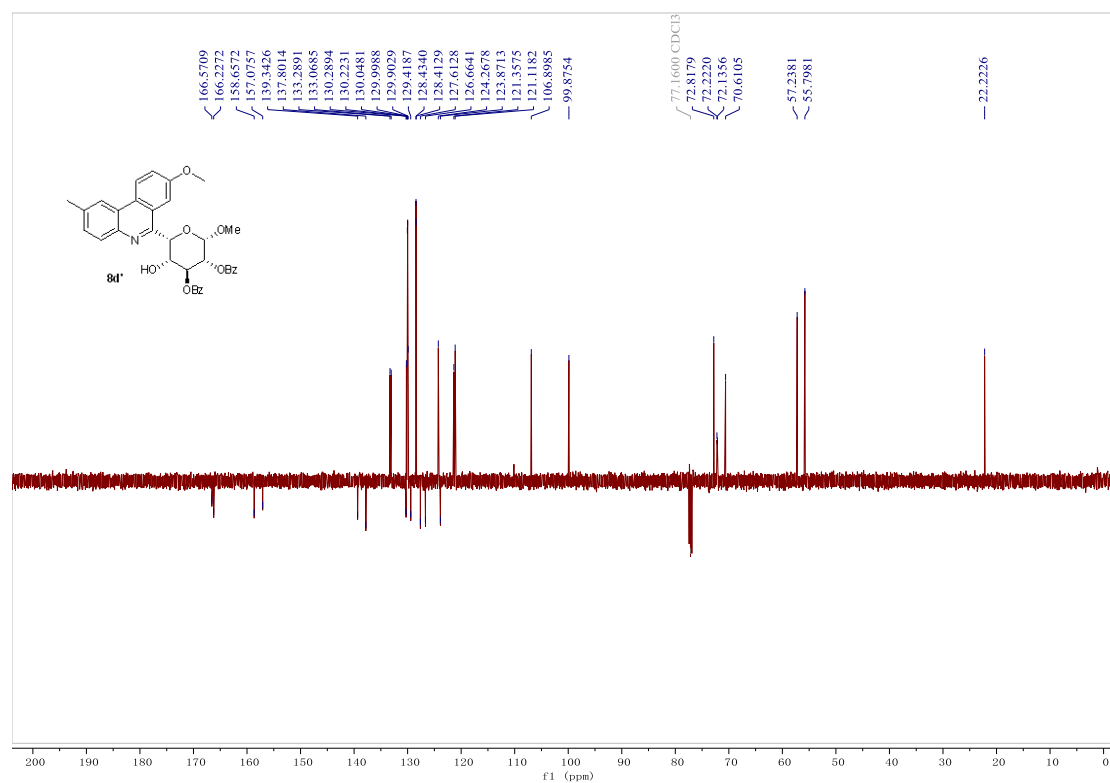
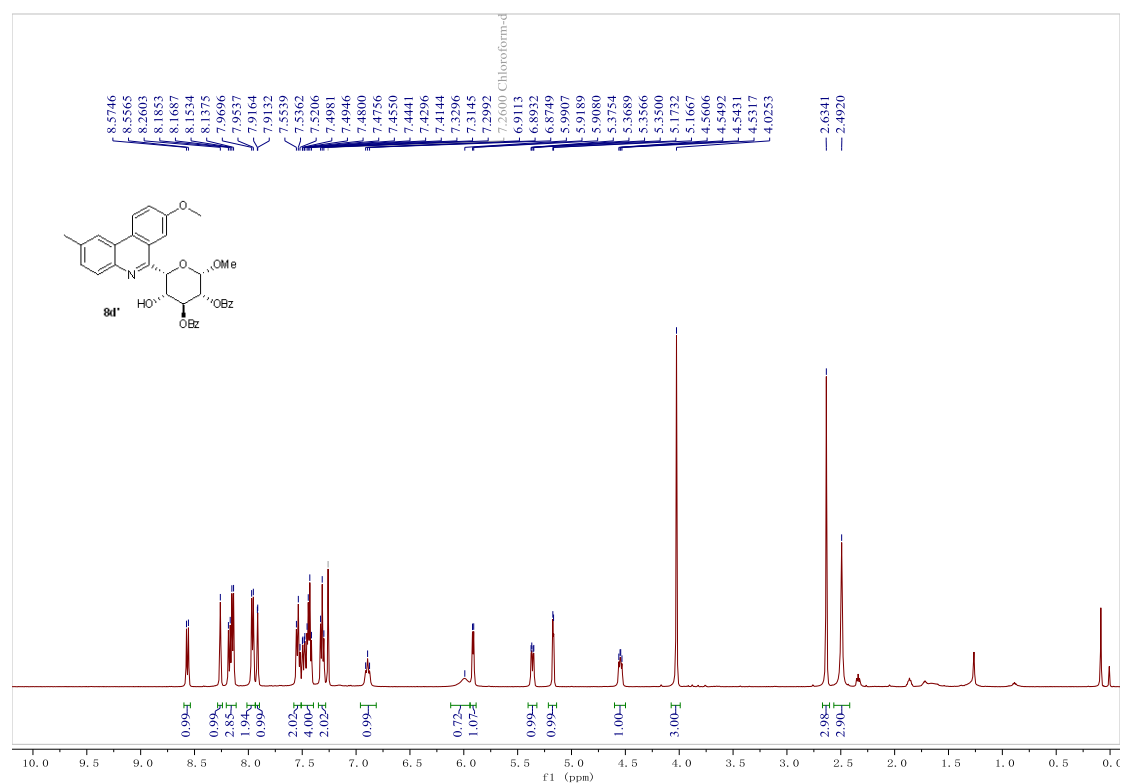
DEPT-Q spectrum for 8c' (126 MHz, CDCl₃)

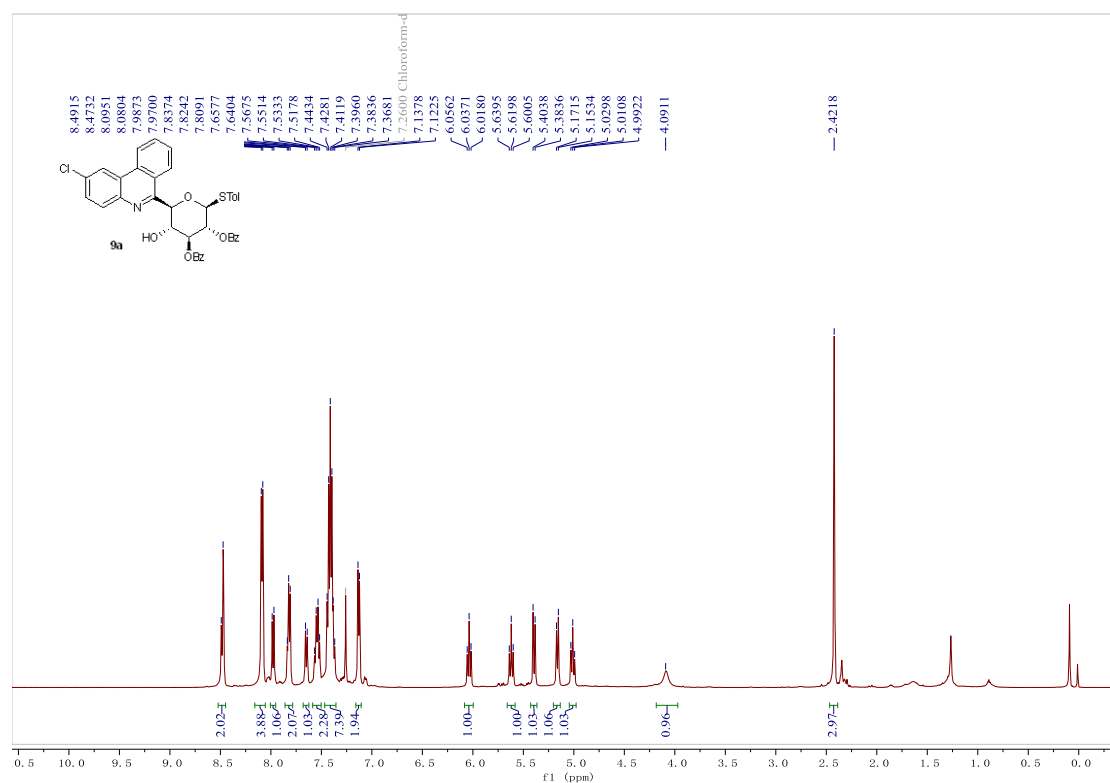


¹H NMR spectrum for 8d (500 MHz, CDCl₃)

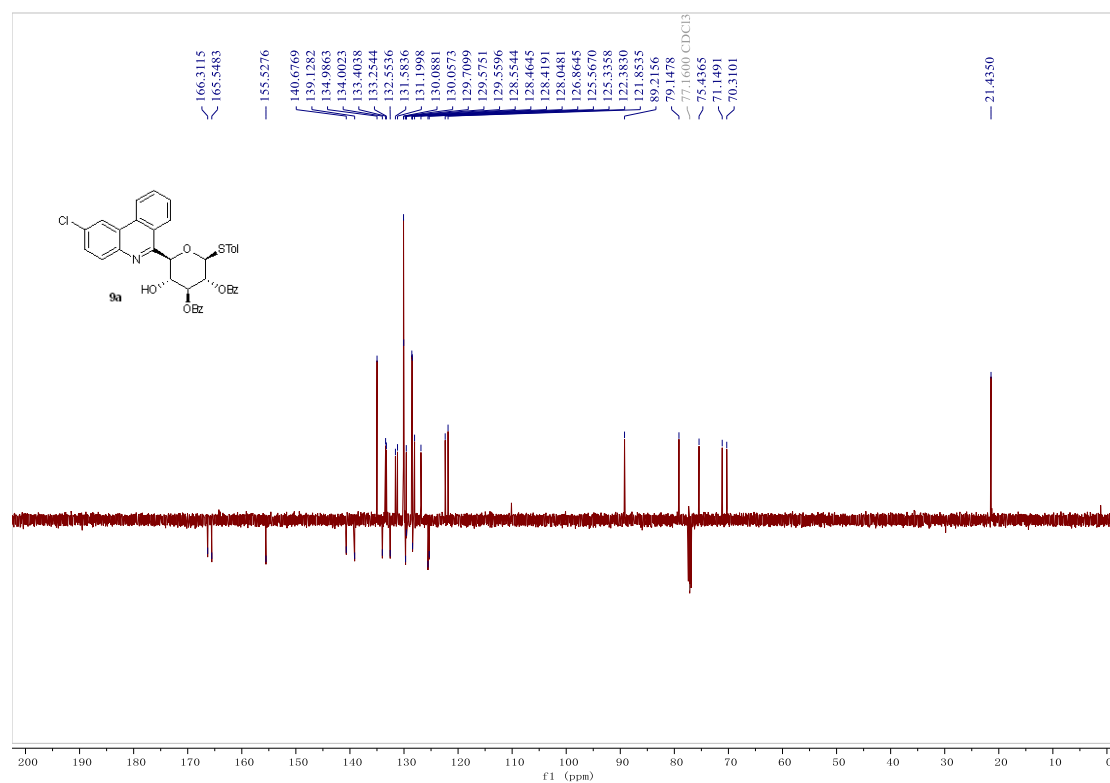


DEPT-Q spectrum for 8d (126 MHz, CDCl₃)

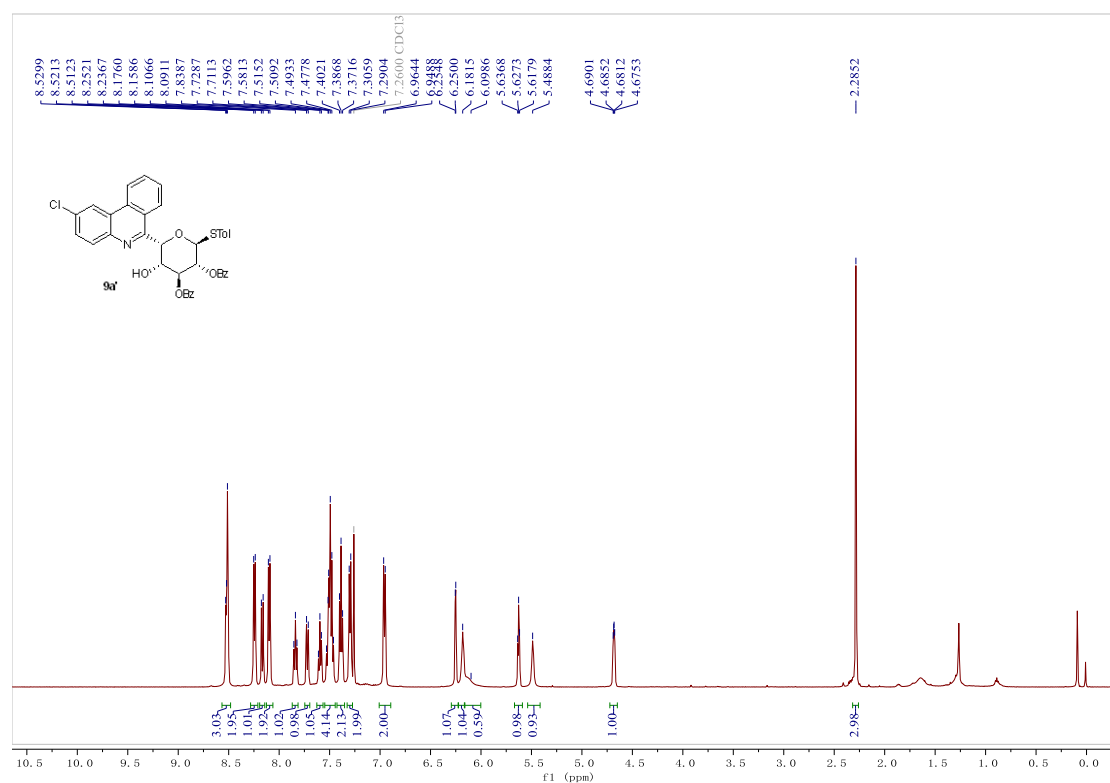




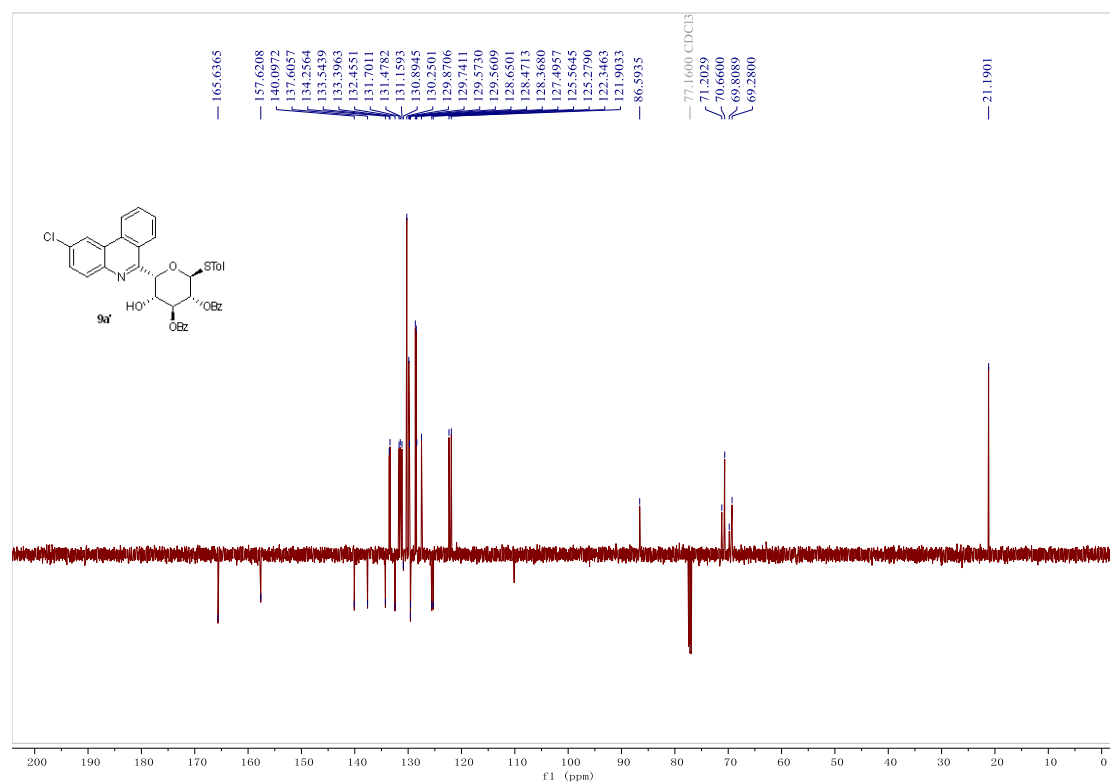
¹H NMR spectrum for 9a (500 MHz, CDCl₃)



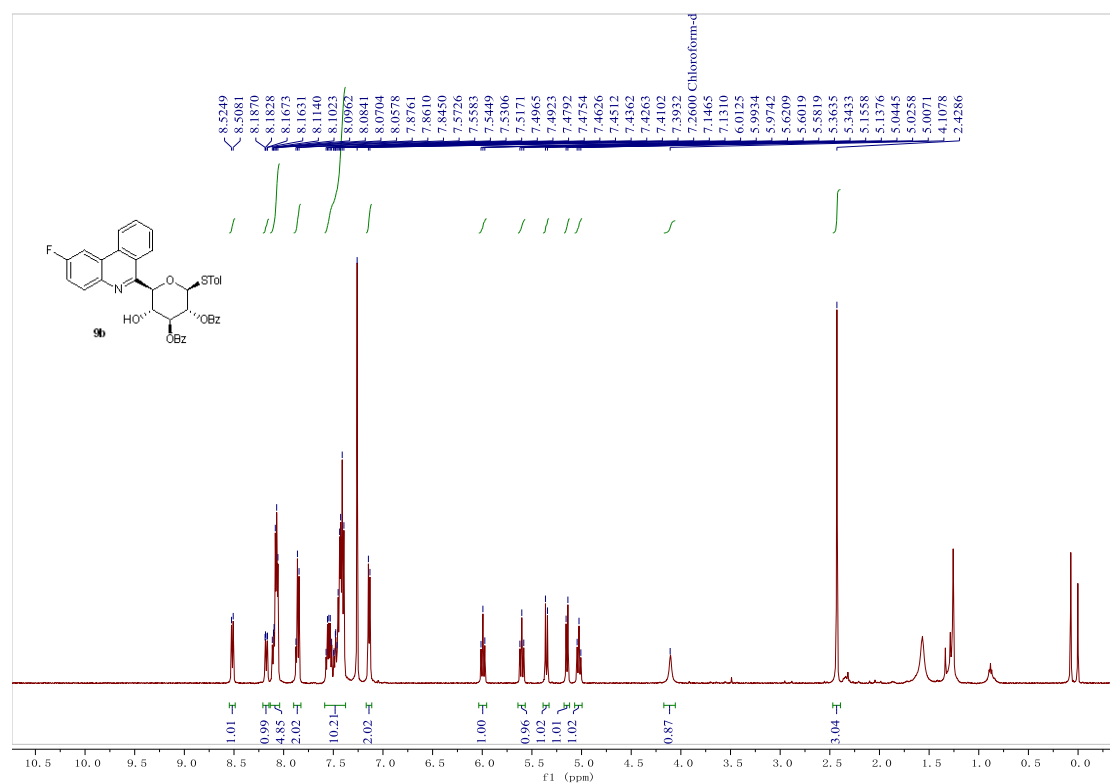
DEPT-Q spectrum for 9a (126 MHz, CDCl₃)



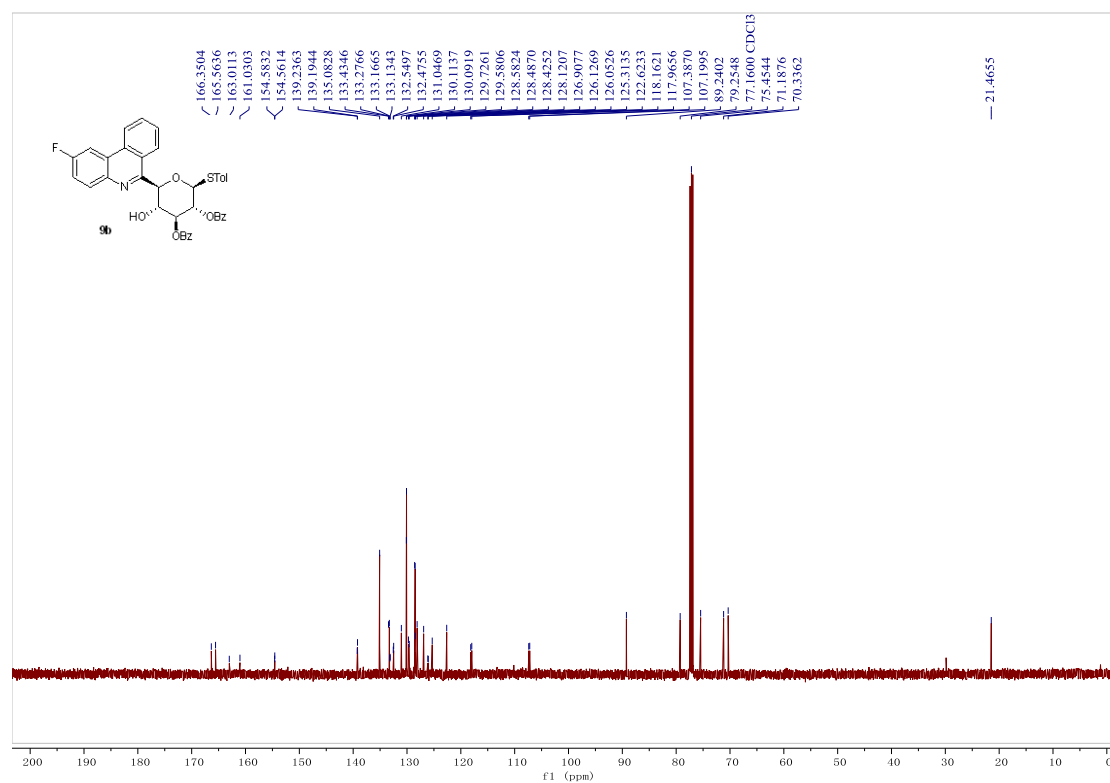
¹H NMR spectrum for 9a' (500 MHz, CDCl₃)



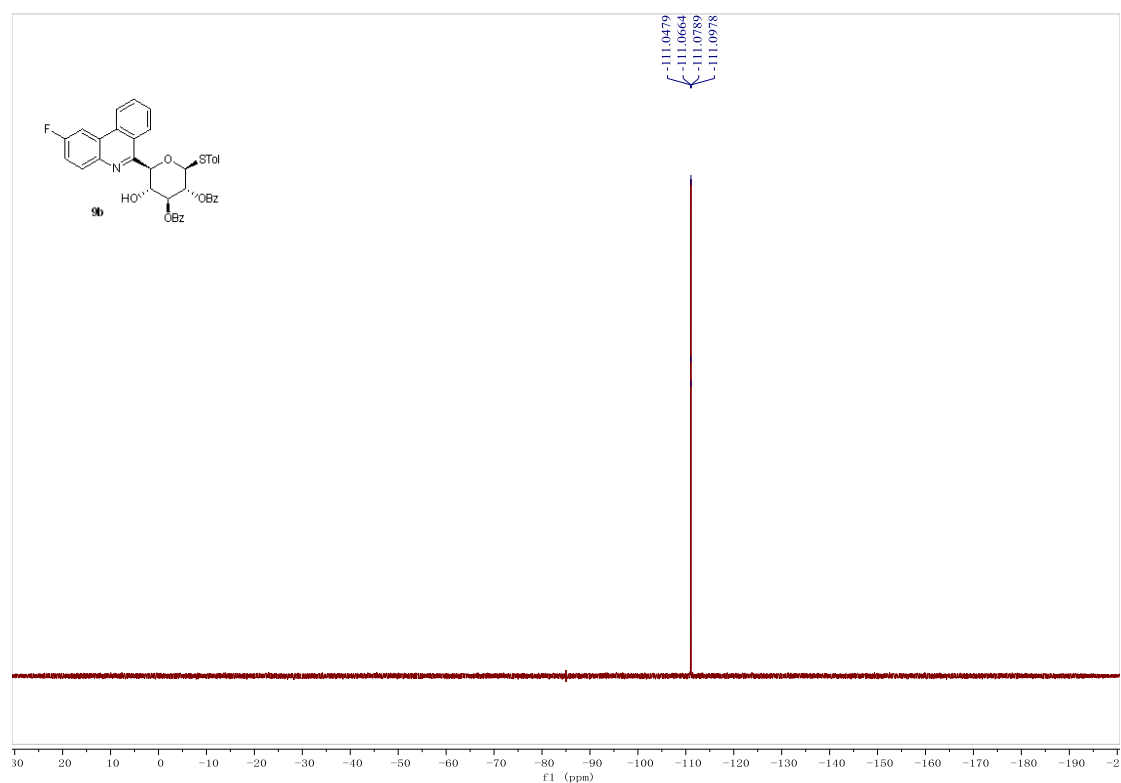
DEPT-Q spectrum for 9a' (126 MHz, CDCl₃)



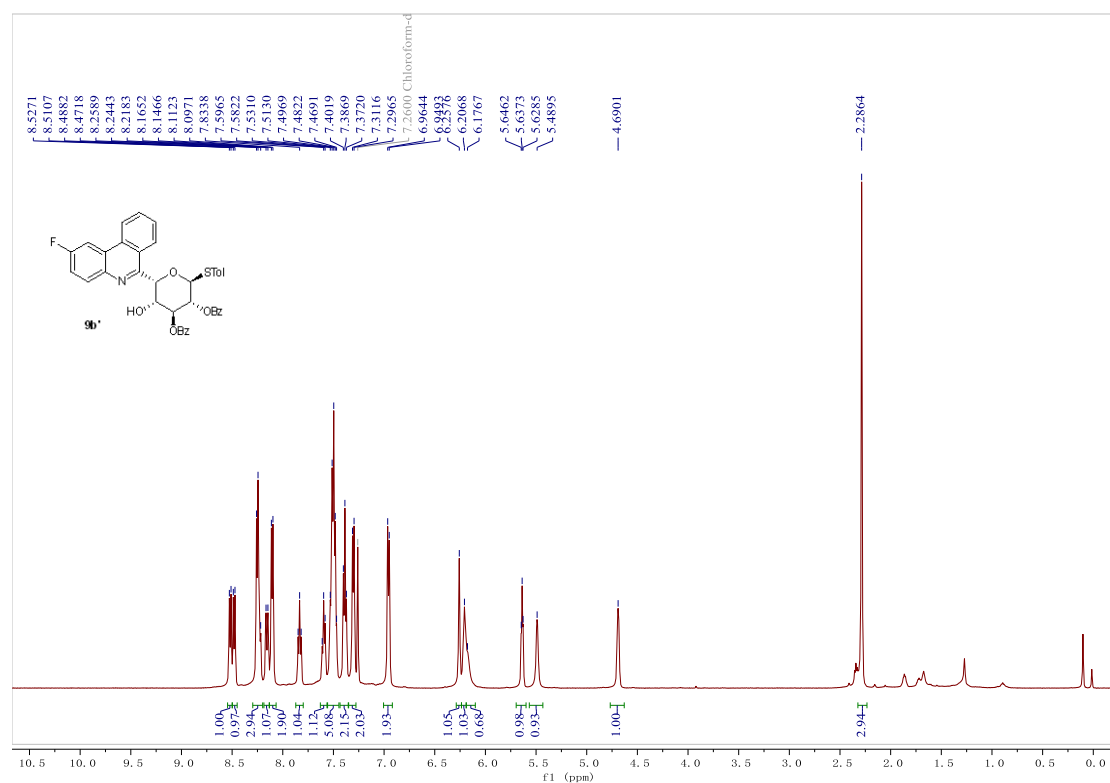
¹H NMR spectrum for **9b (500 MHz, CDCl₃)**



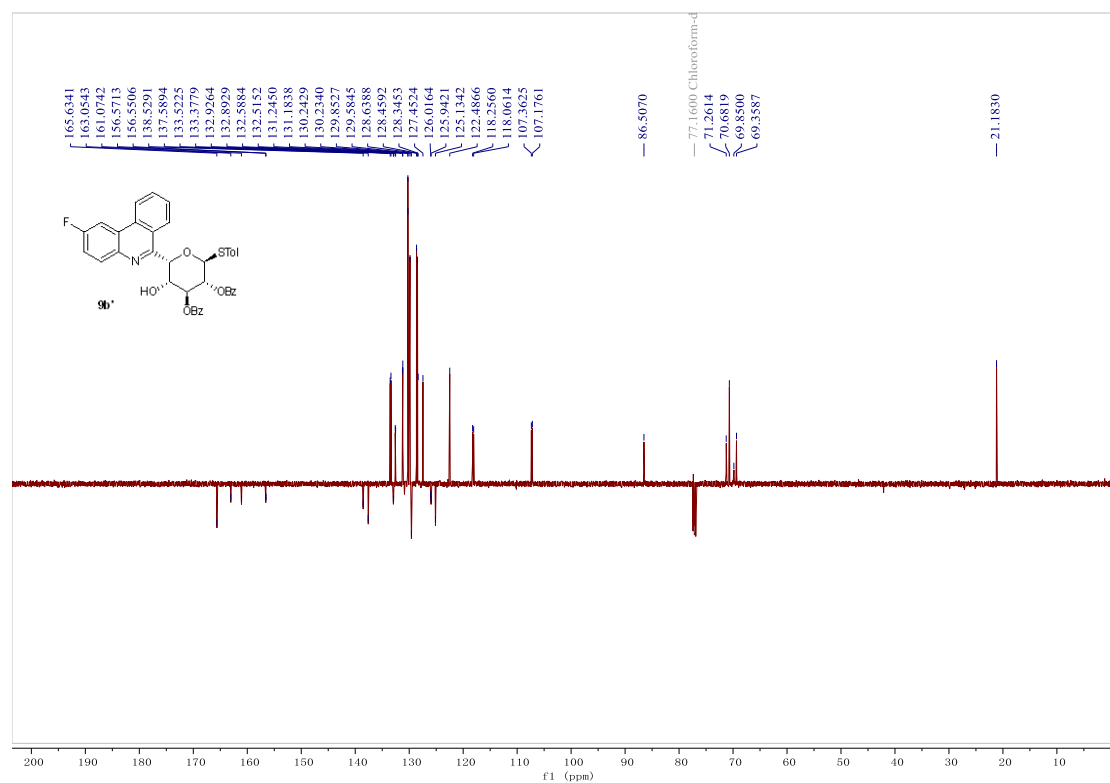
¹³C NMR spectrum for **9b (126 MHz, CDCl₃)**



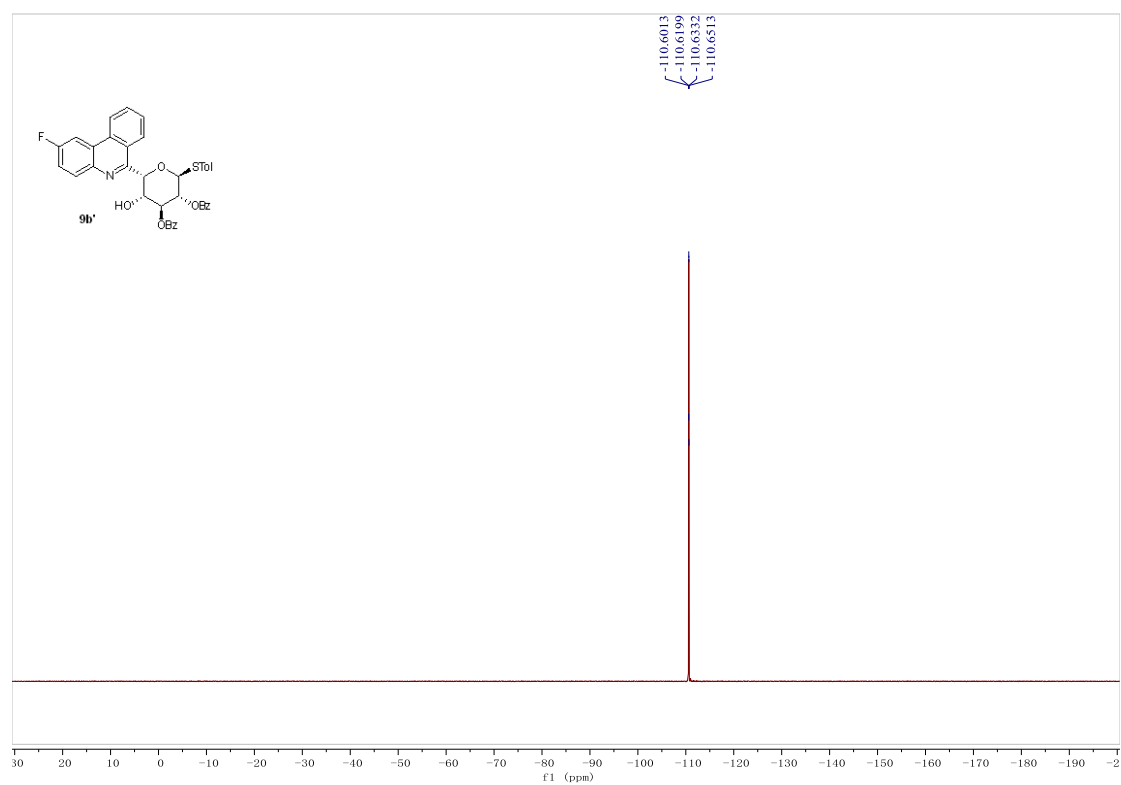
^{19}F NMR spectrum for **9b**



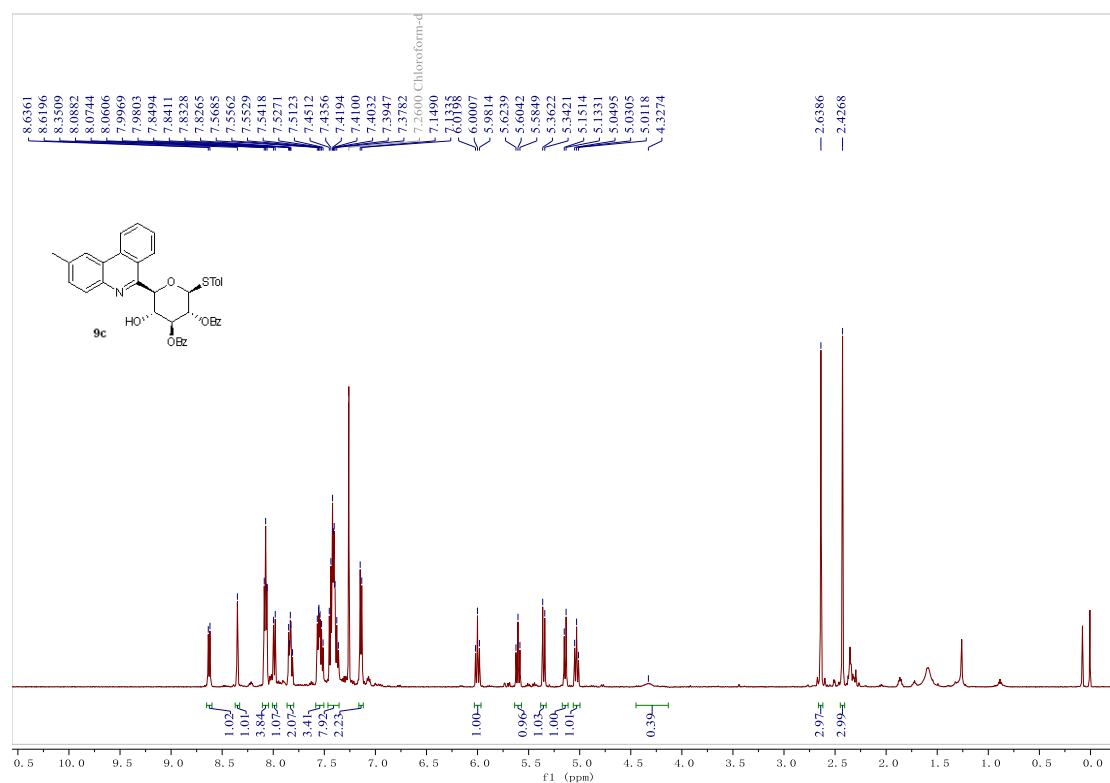
¹H NMR spectrum for **9b' (500 MHz, CDCl₃)**



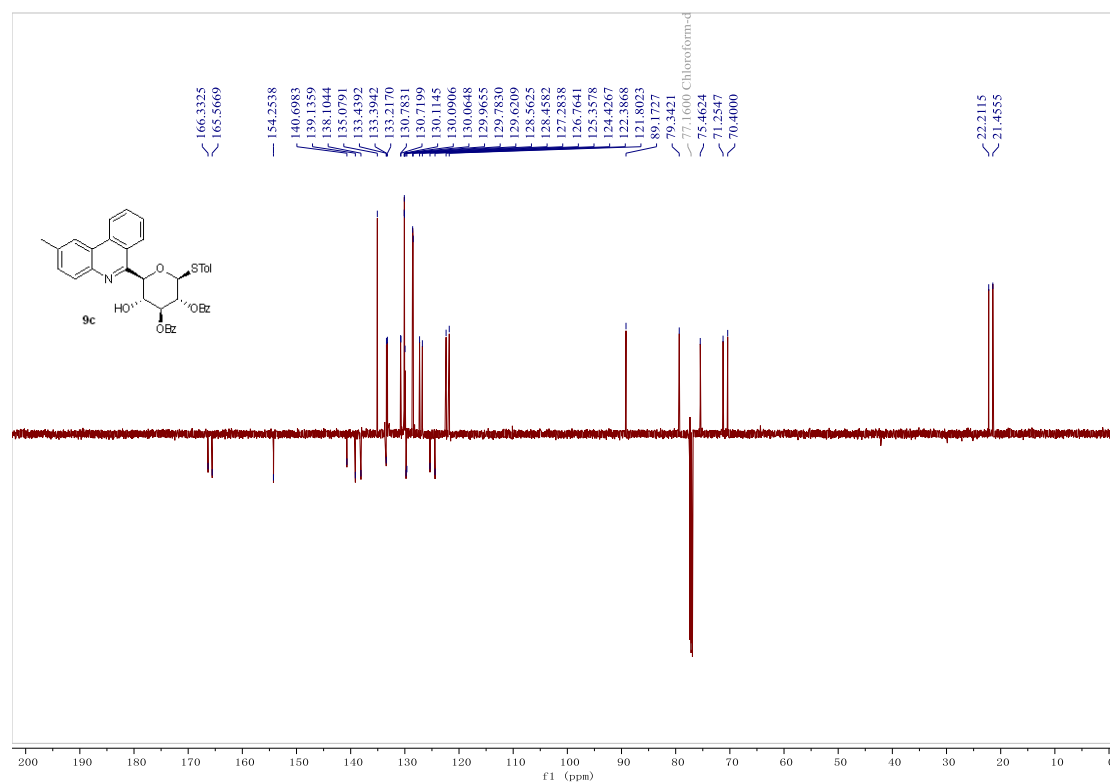
DEPT-Q spectrum for **9b' (126 MHz, CDCl₃)**



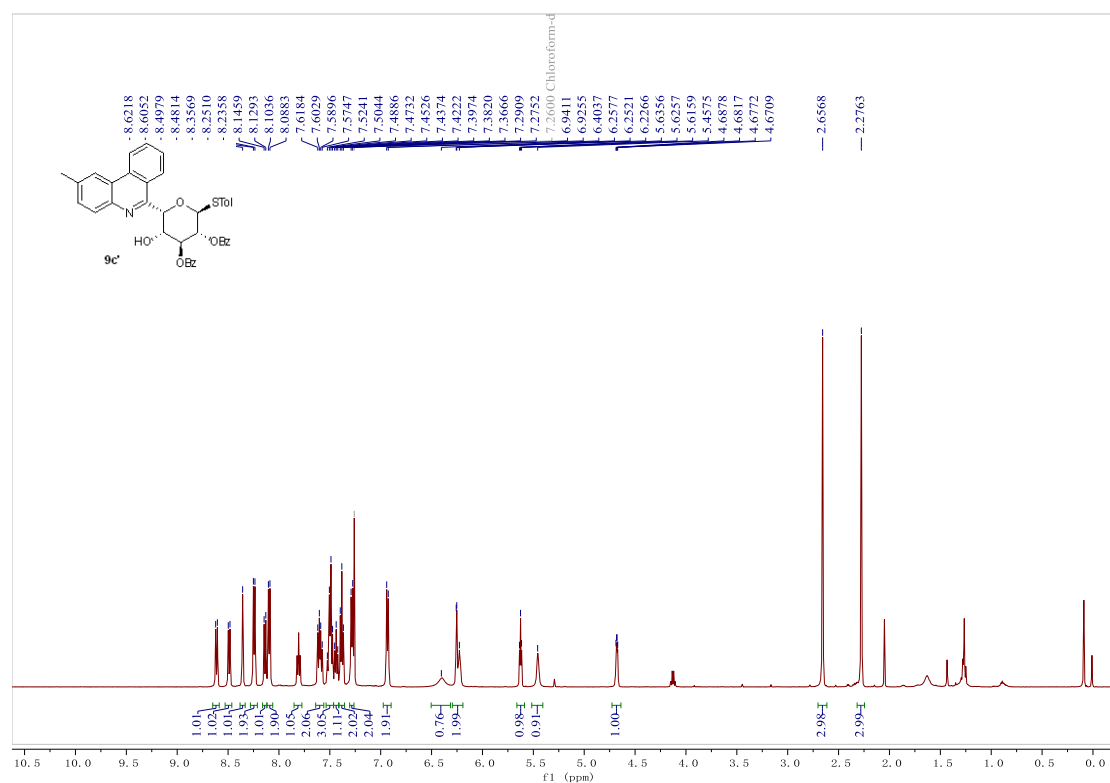
^{19}F NMR spectrum for **9b'**



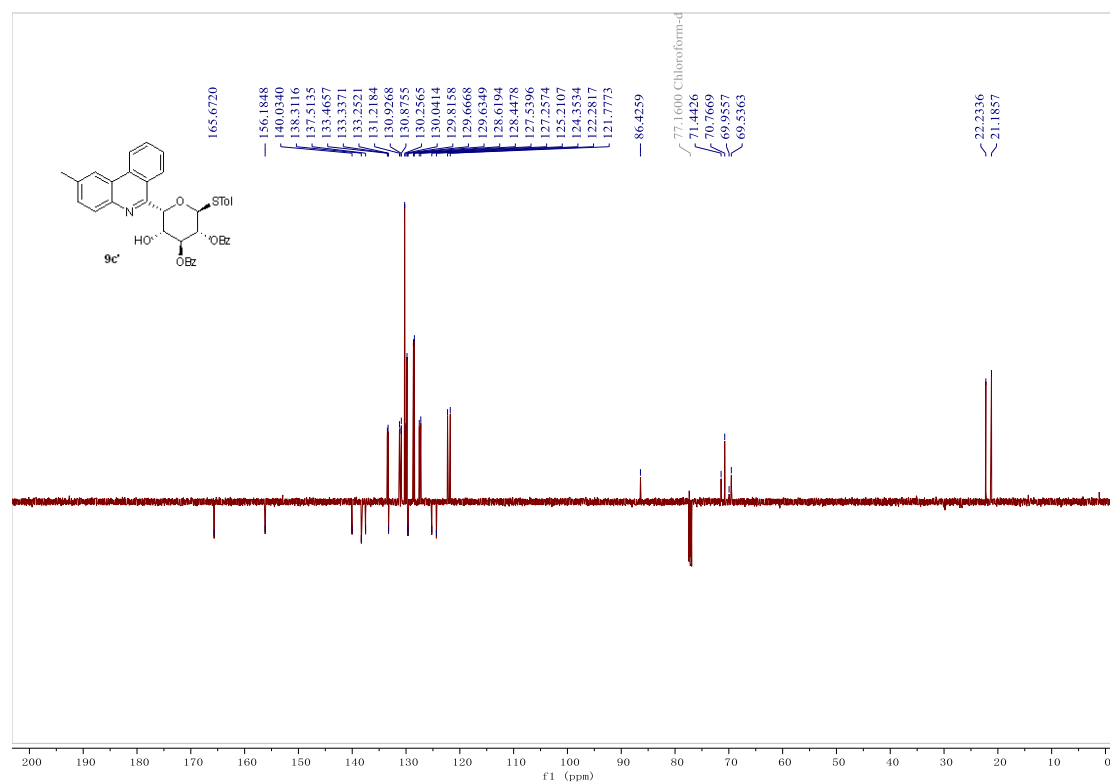
¹H NMR spectrum for 9c (500 MHz, CDCl₃)



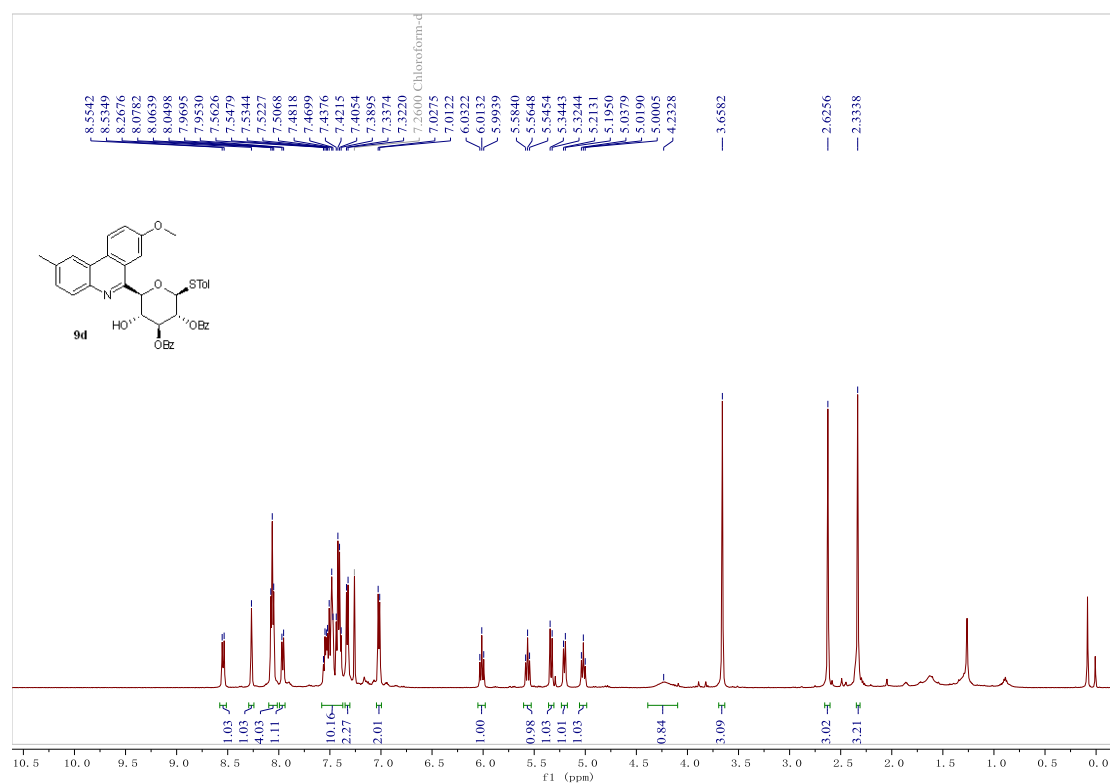
DEPT-Q spectrum for 9c (126 MHz, CDCl₃)



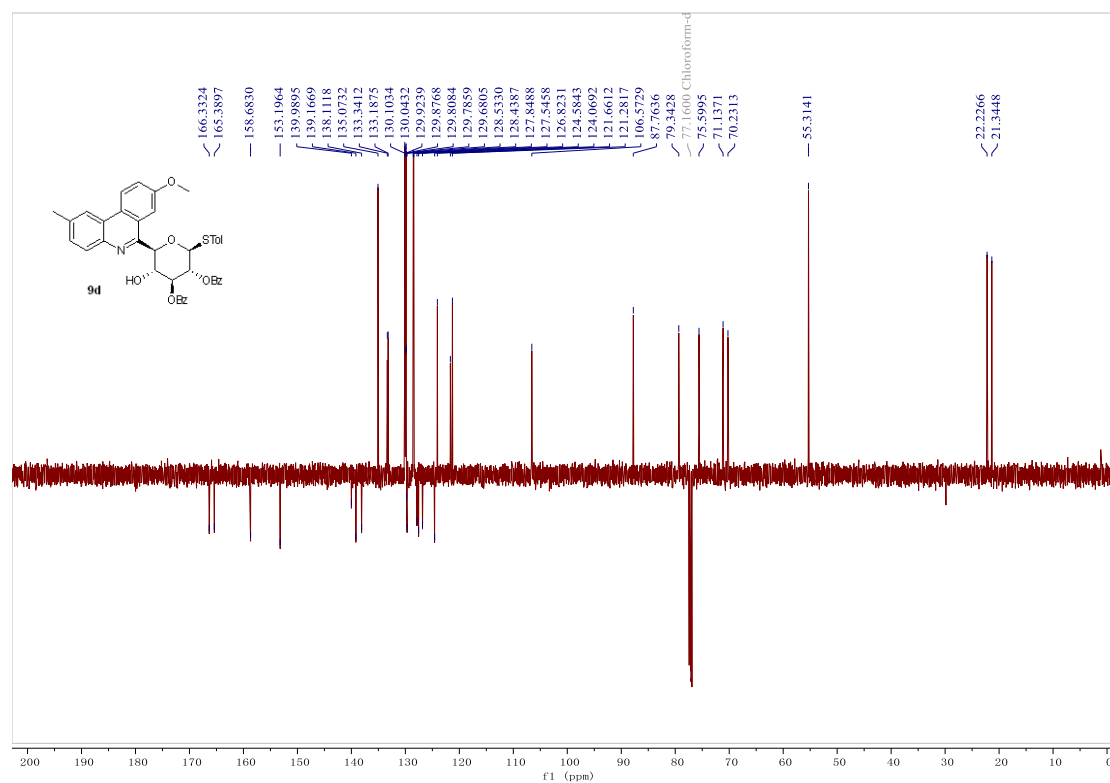
¹H NMR spectrum for 9c' (500 MHz, CDCl₃)



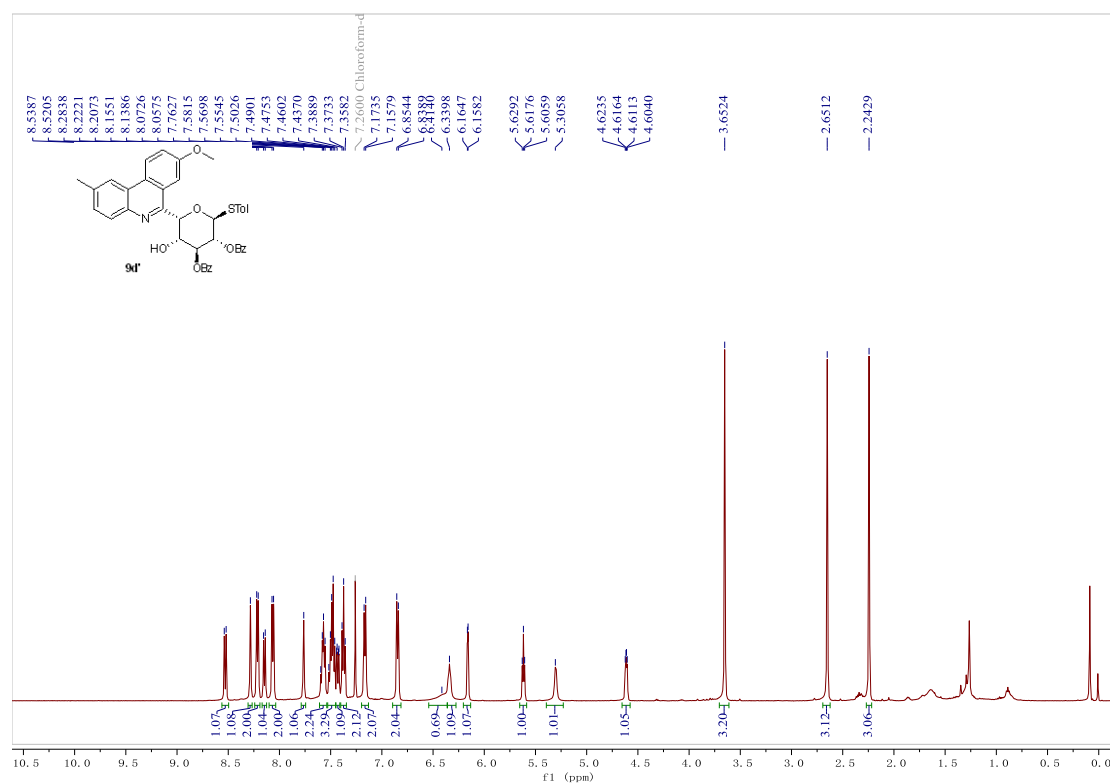
DEPT-Q spectrum for 9c' (126 MHz, CDCl₃)



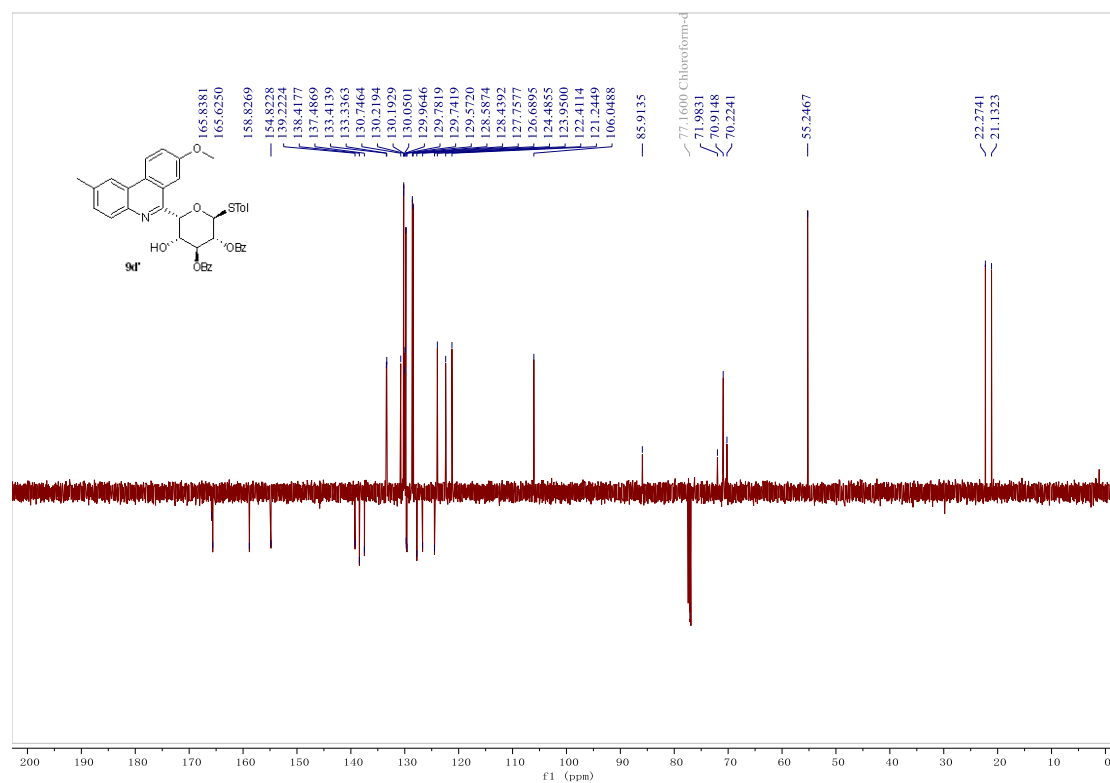
¹H NMR spectrum for 9d (500 MHz, CDCl₃)



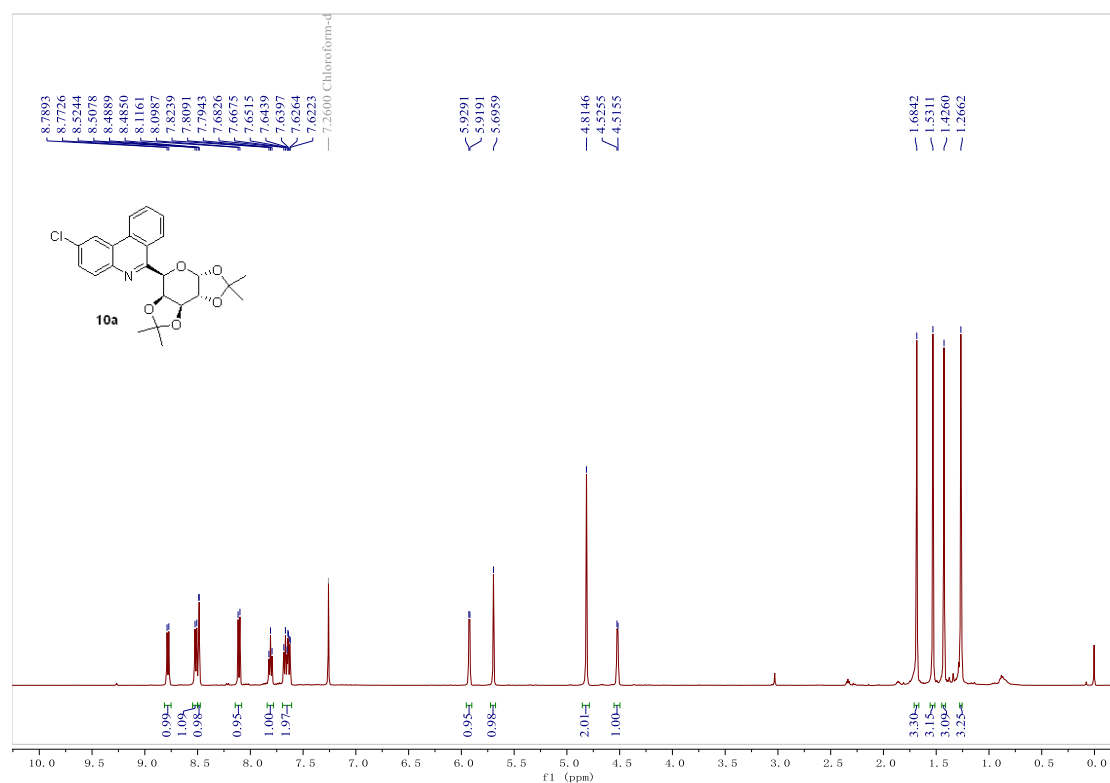
DEPT-Q spectrum for 9d (126 MHz, CDCl₃)



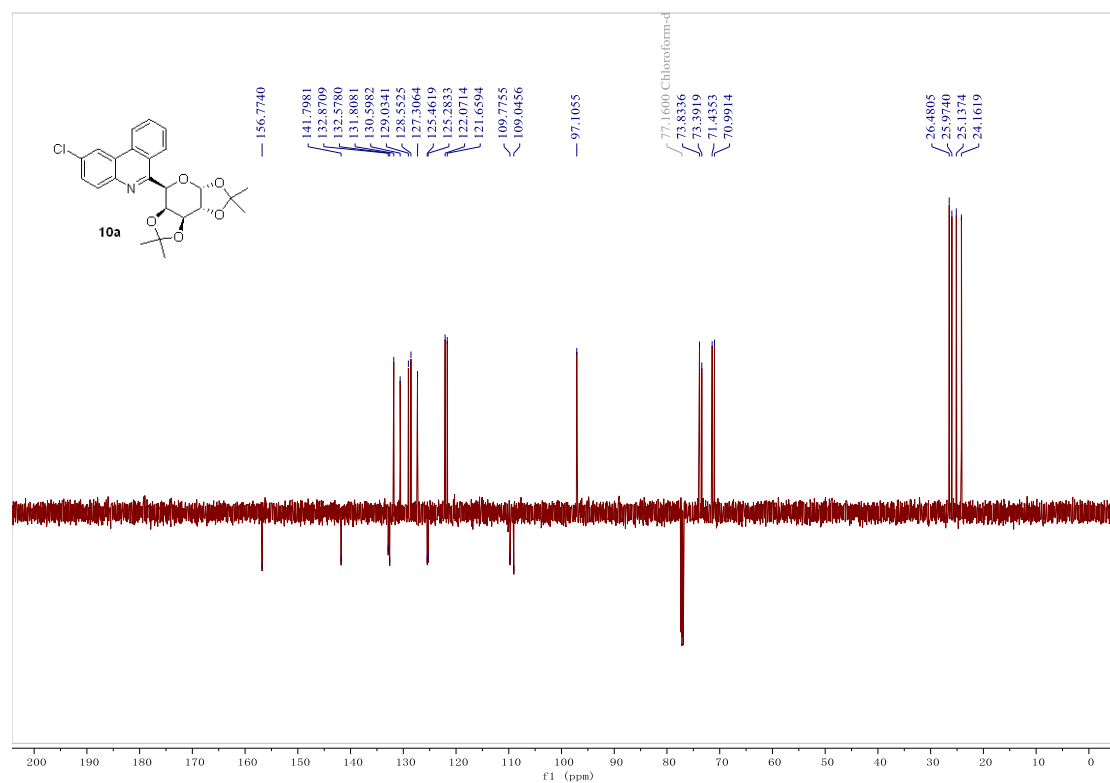
¹H NMR spectrum for 9d' (500 MHz, CDCl₃)



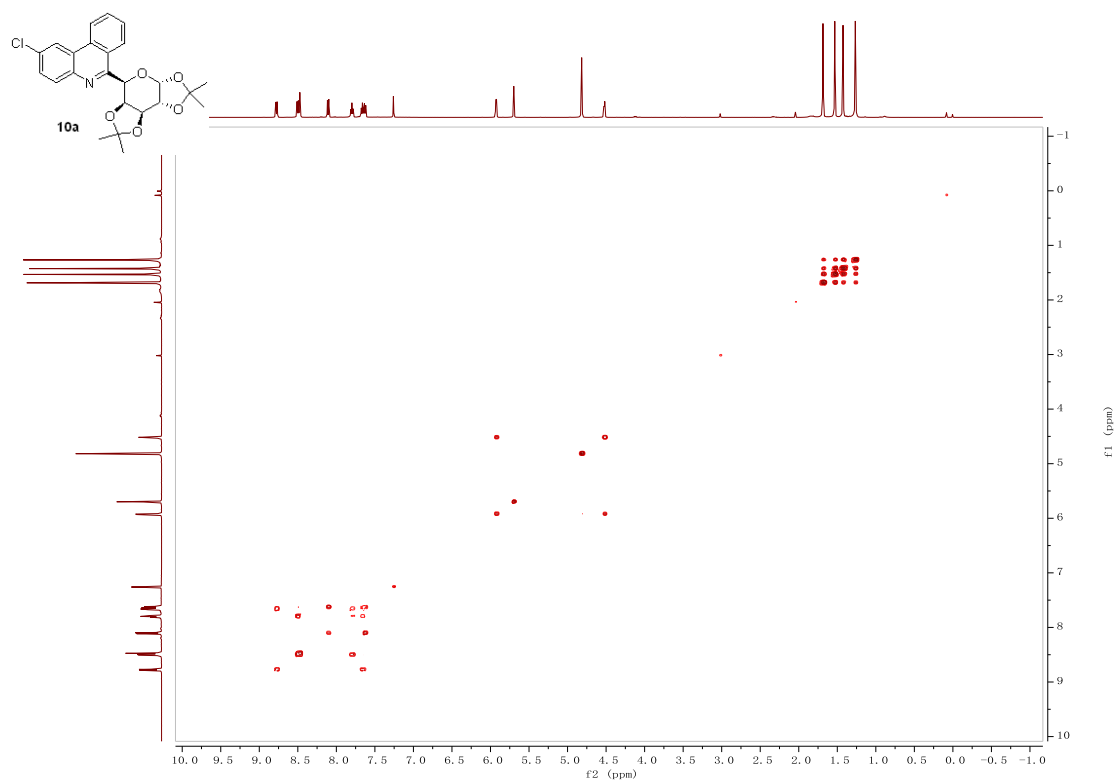
DEPT-Q spectrum for 9d' (126 MHz, CDCl₃)



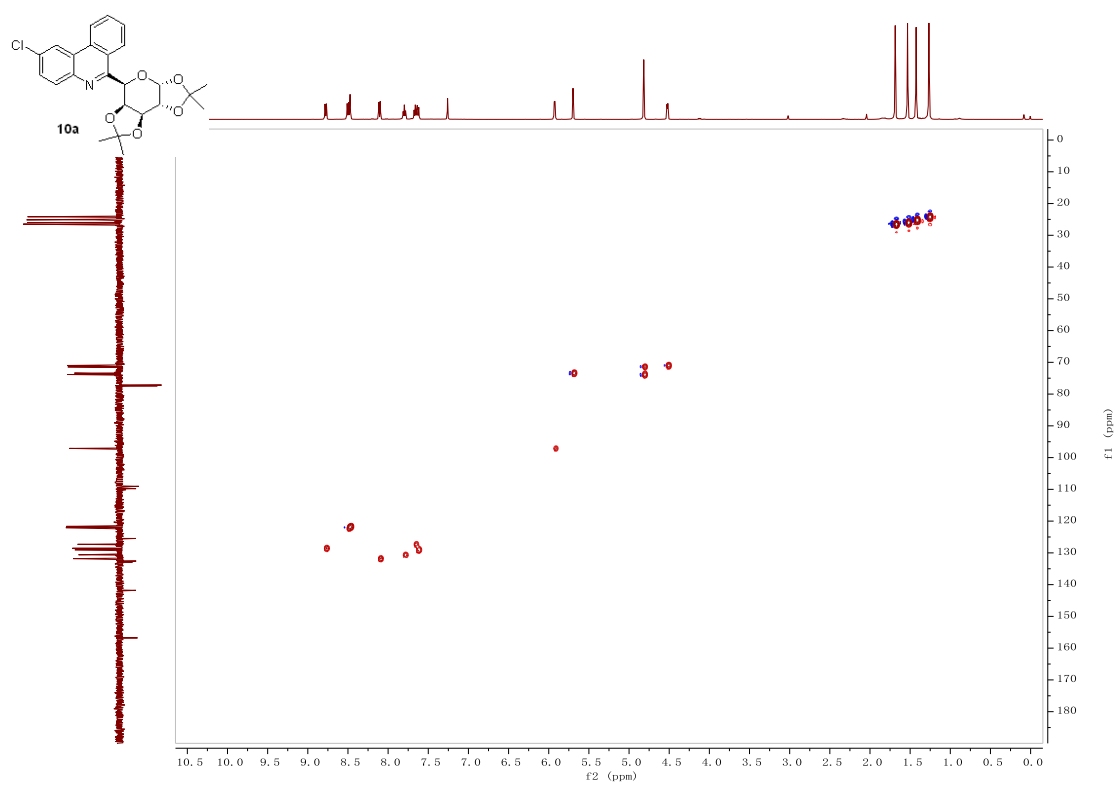
¹H NMR spectrum for 10a (500 MHz, CDCl₃)



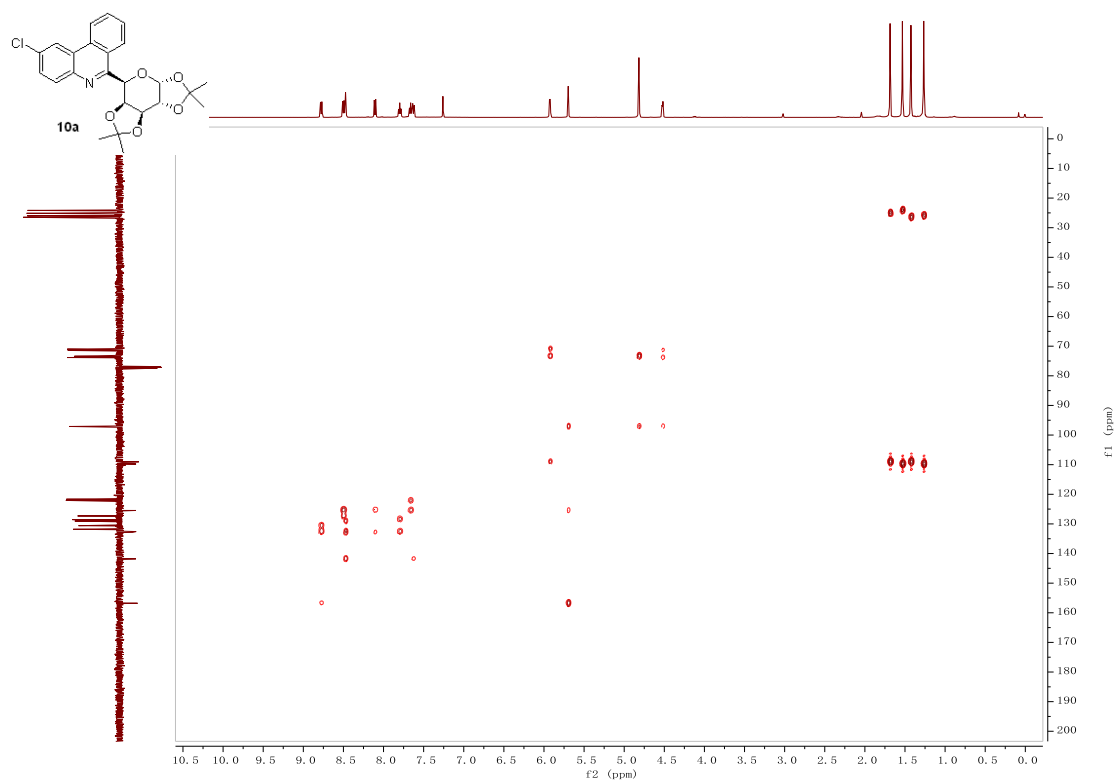
DEPT-Q spectrum for 10a (126 MHz, CDCl₃)



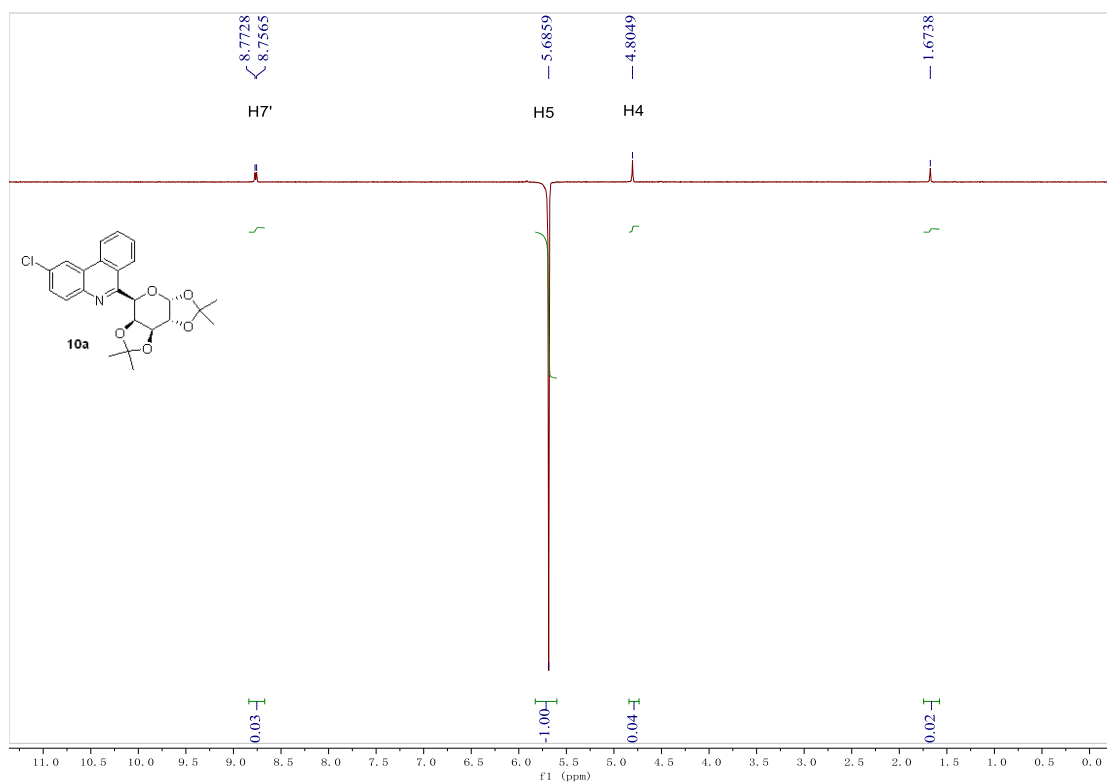
COSY spectrum for **10a**



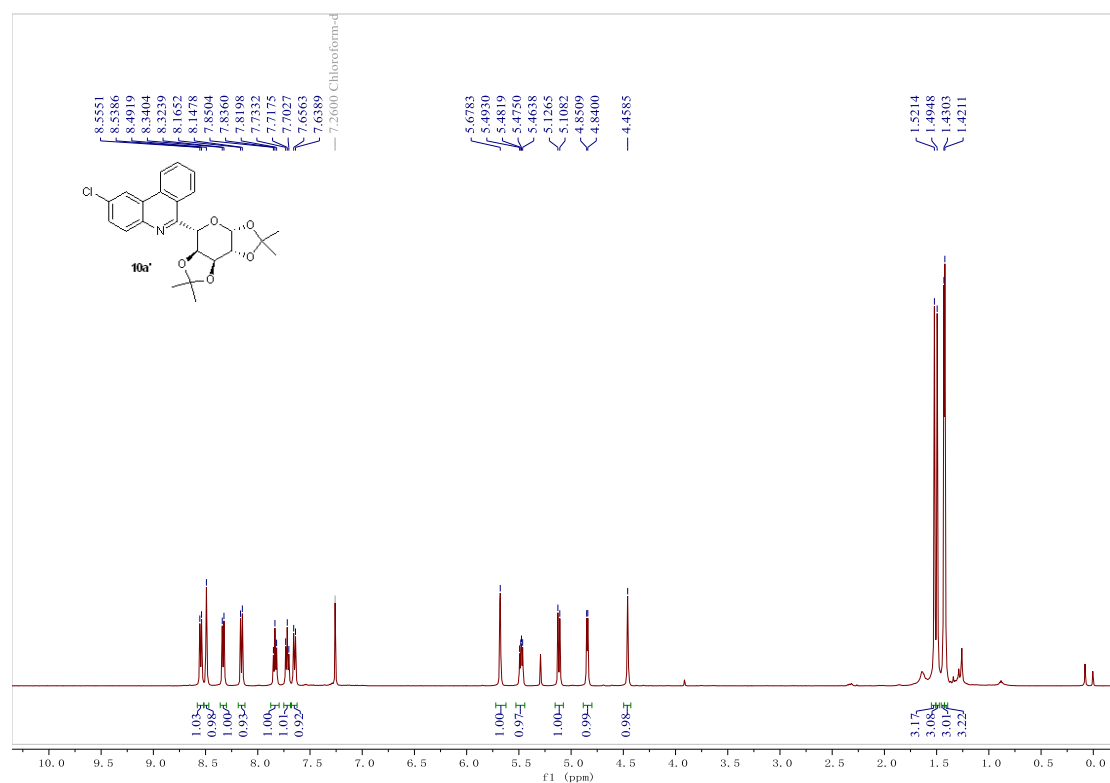
HSQC spectrum for **10a**



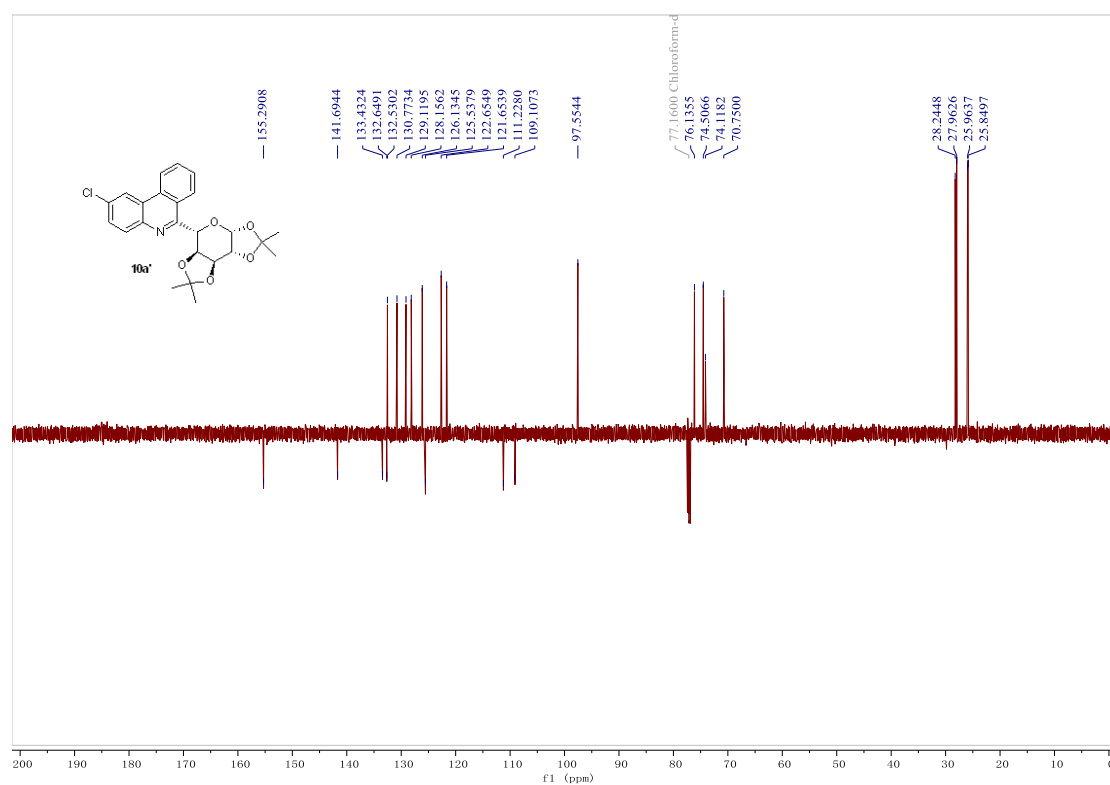
HMBC spectrum for **10a**



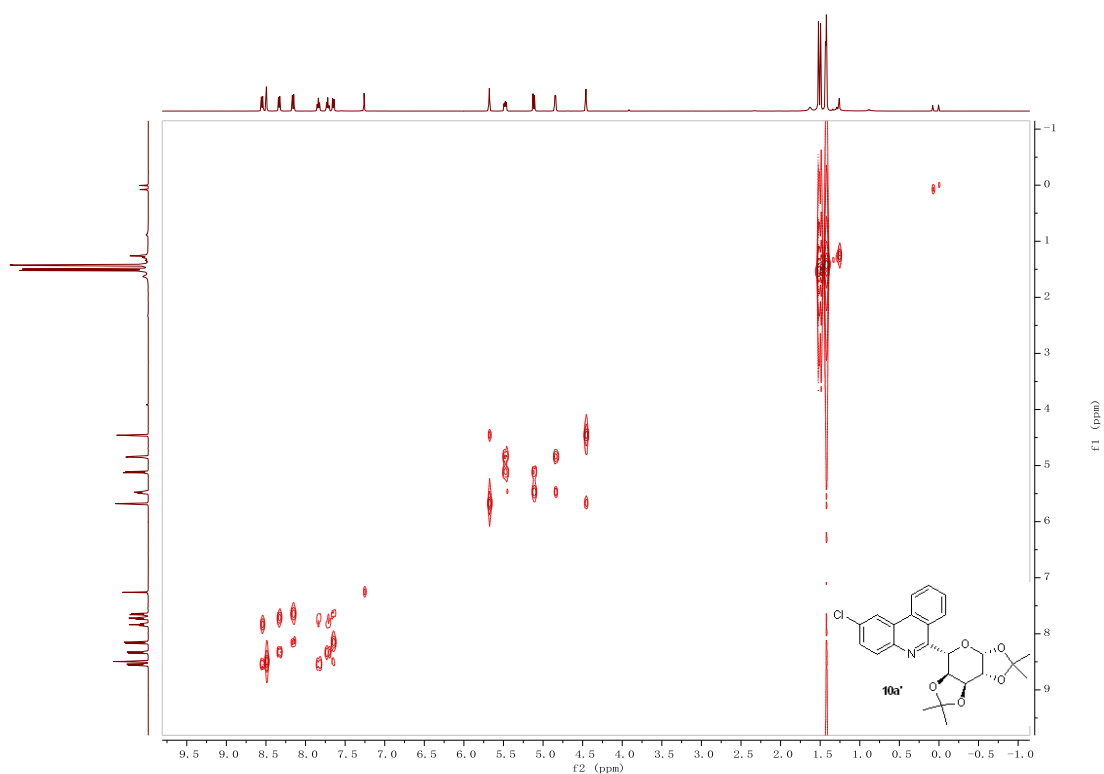
NOE difference spectrum for **10a**



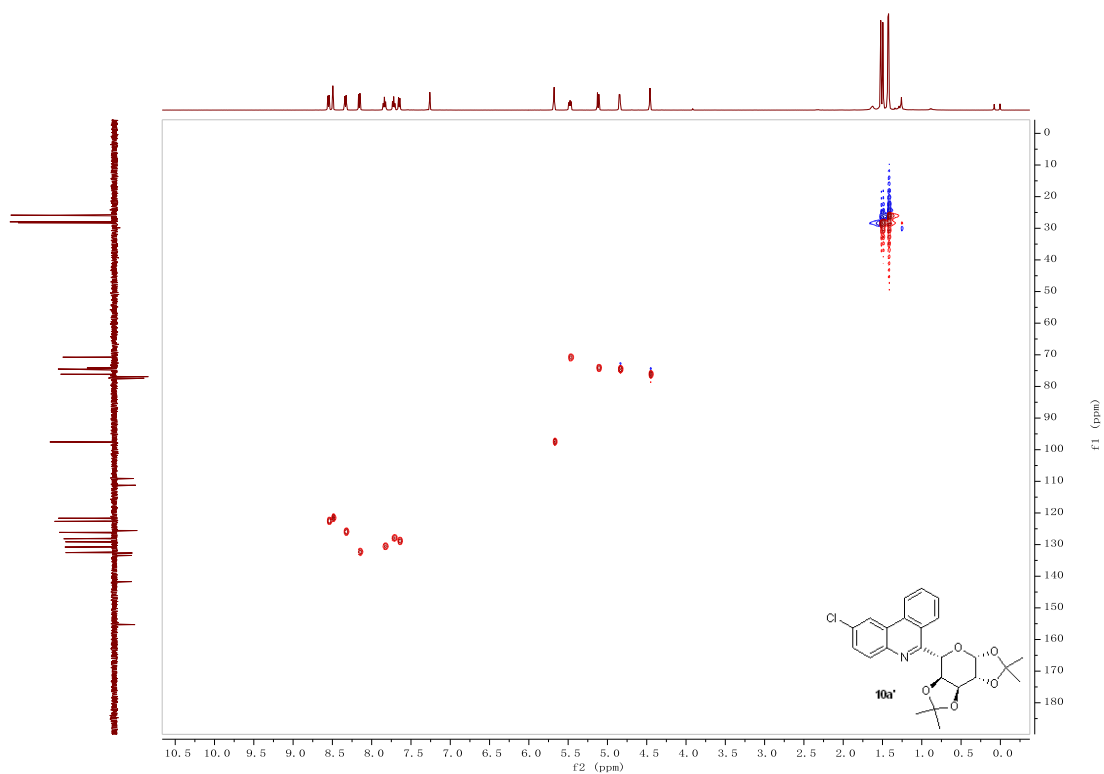
¹H NMR spectrum for 10a' (500 MHz, CDCl₃)



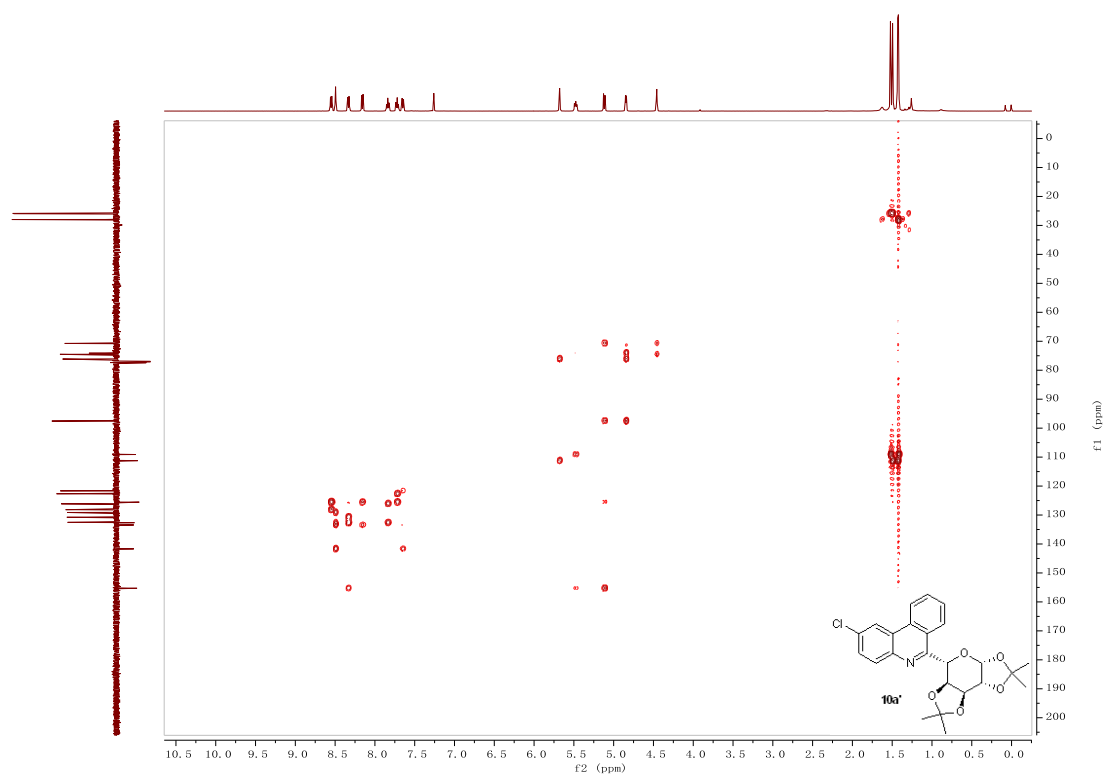
DEPT-Q spectrum for 10a' (126 MHz, CDCl₃)



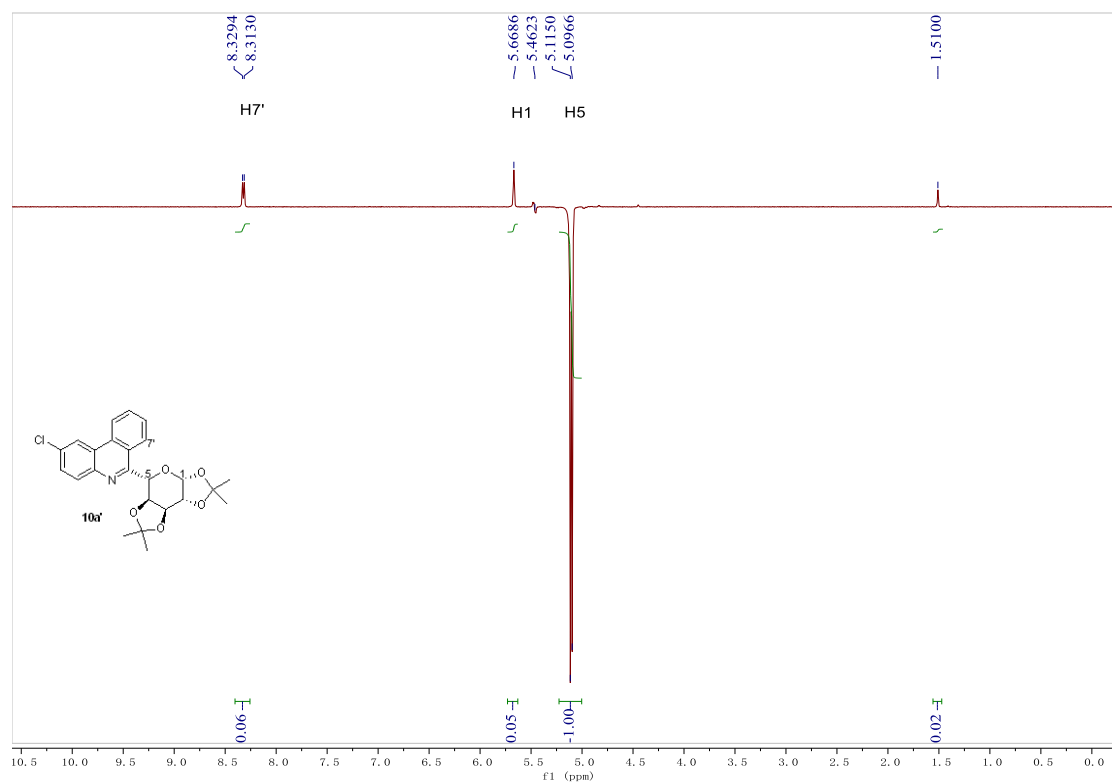
COSY spectrum for **10a'**



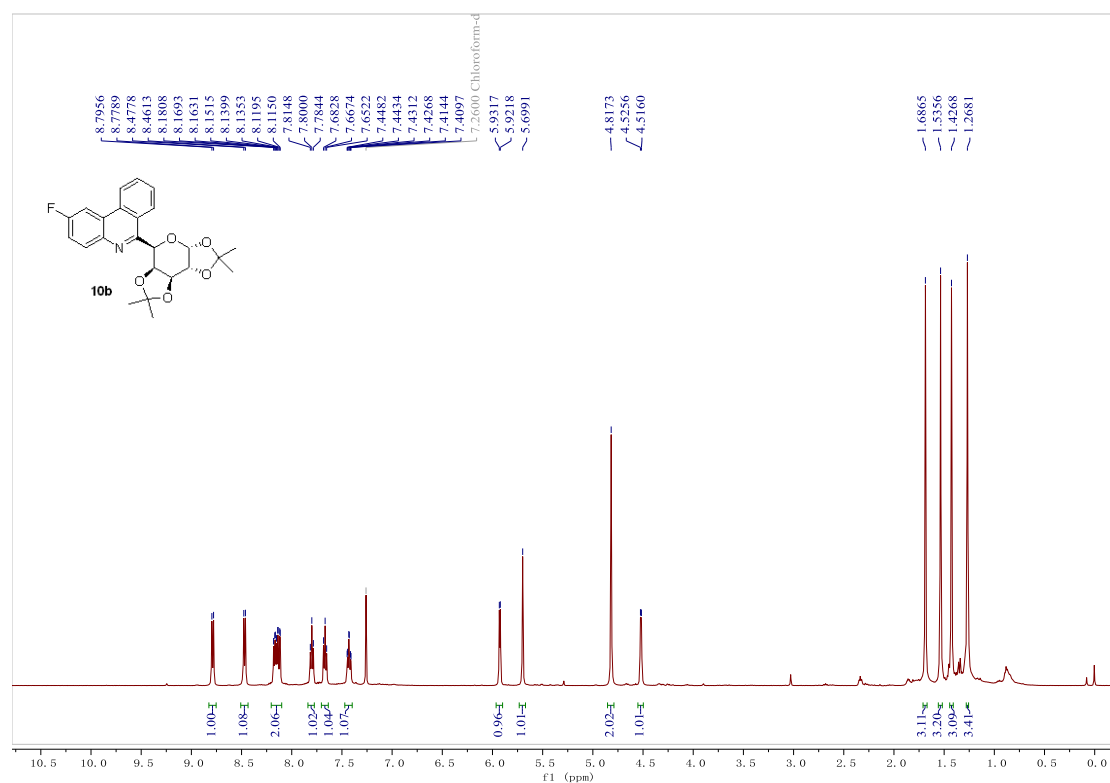
HSQC spectrum for **10a'**



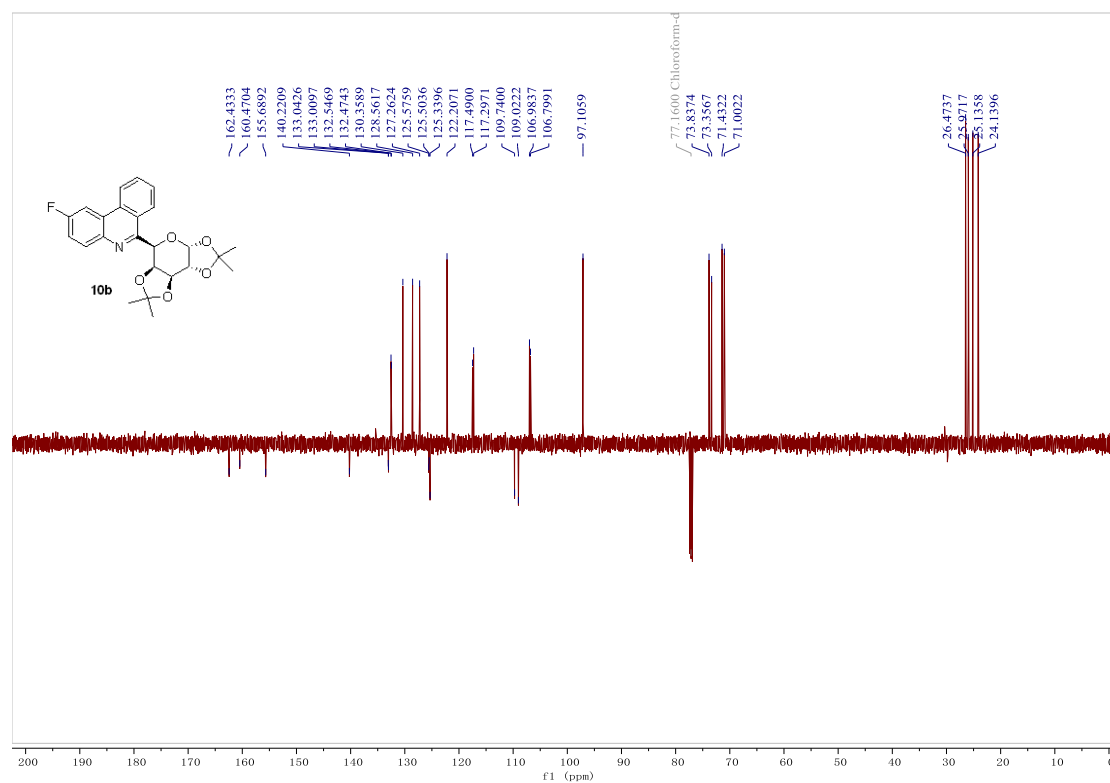
HMBC spectrum for 10a'



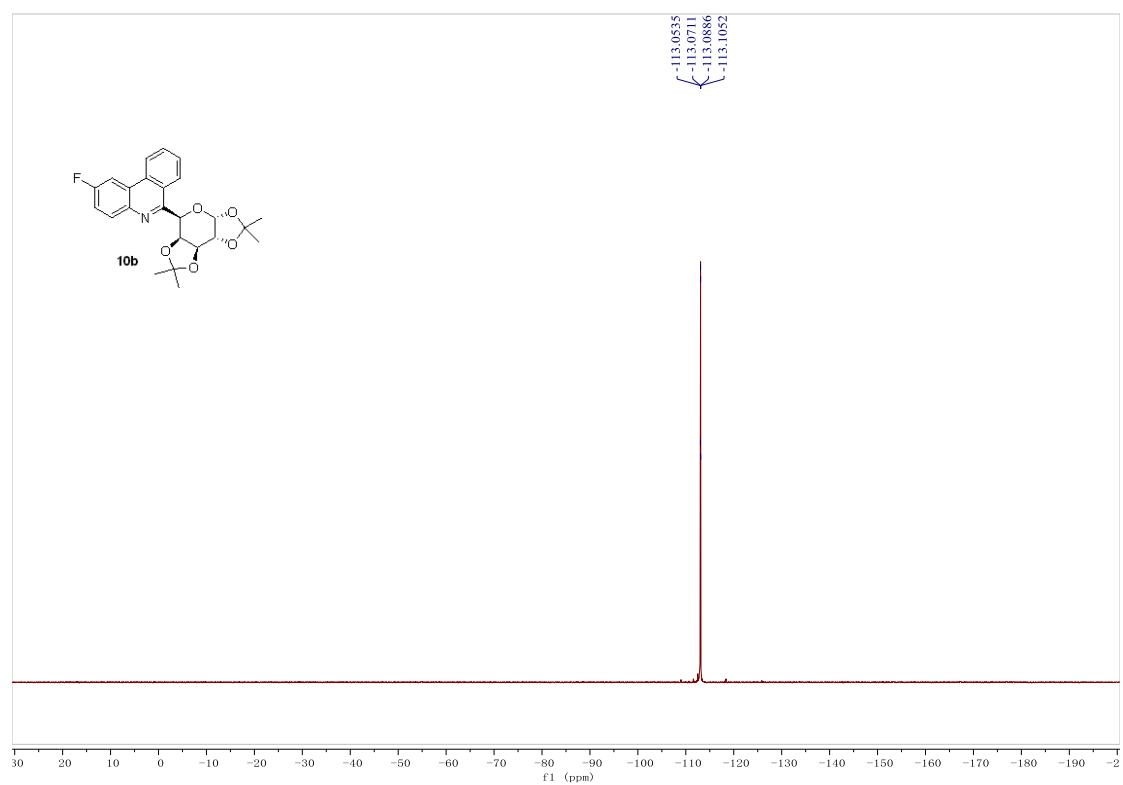
NOE difference spectrum for 10a'



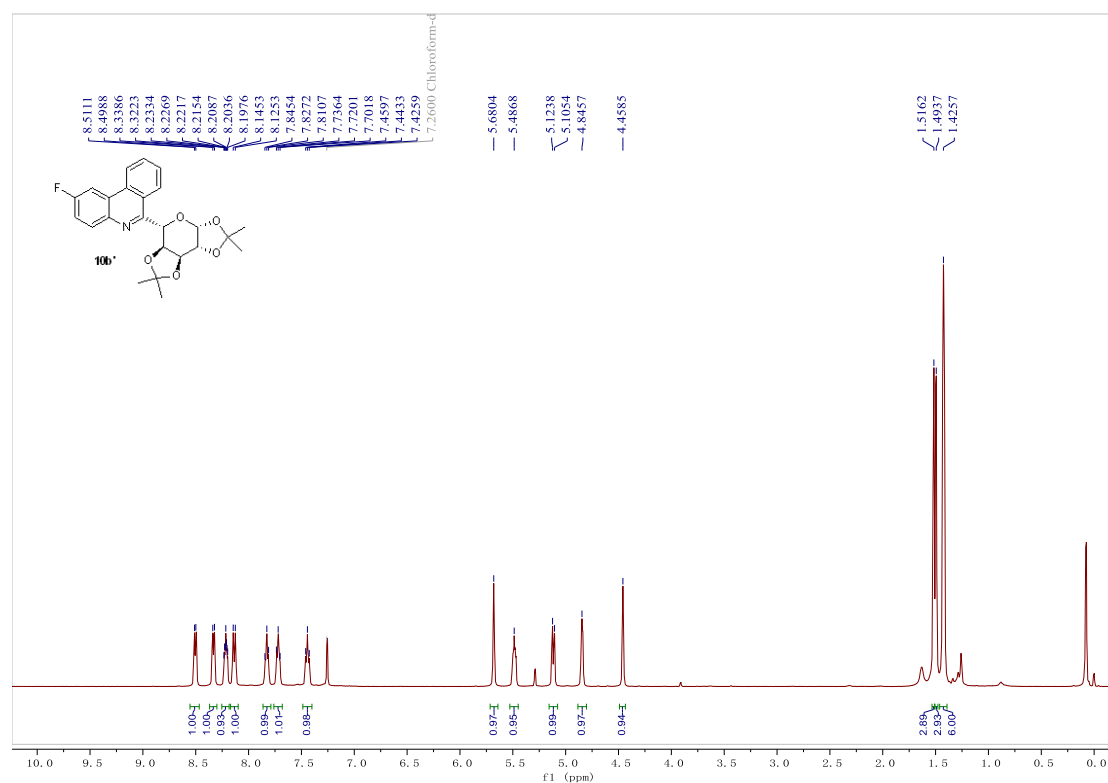
¹H NMR spectrum for 10b (500 MHz, CDCl₃)



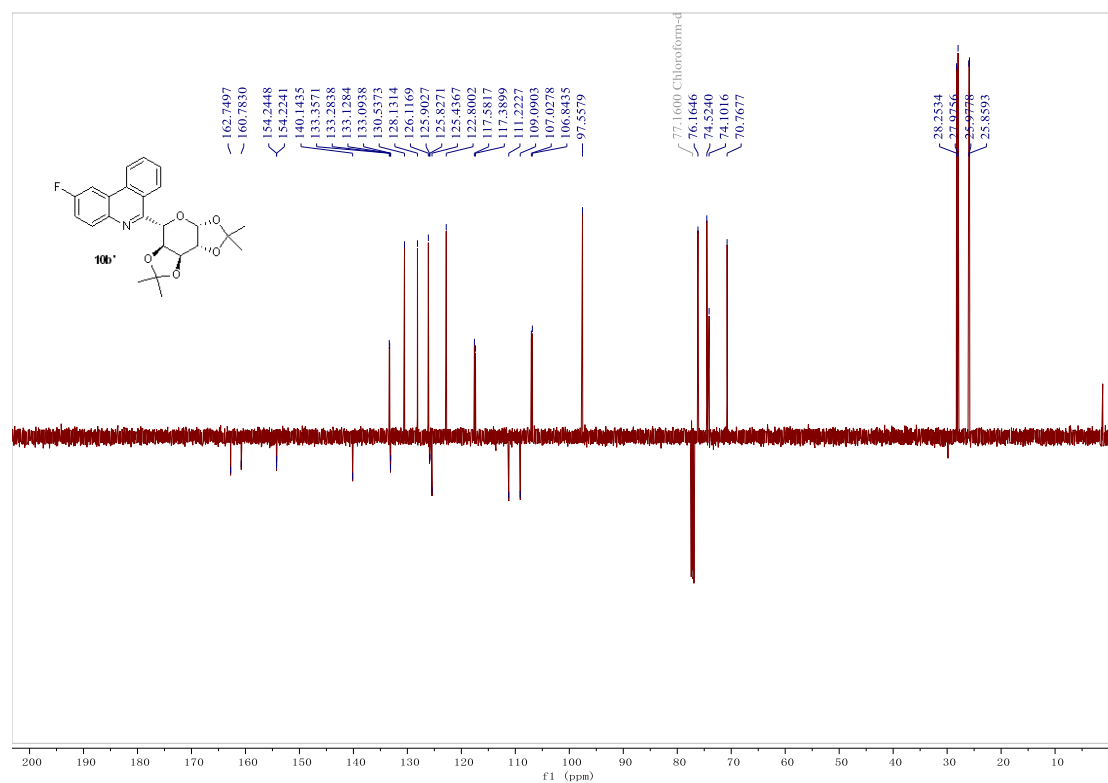
DEPT-Q spectrum for 10b (126 MHz, CDCl₃)



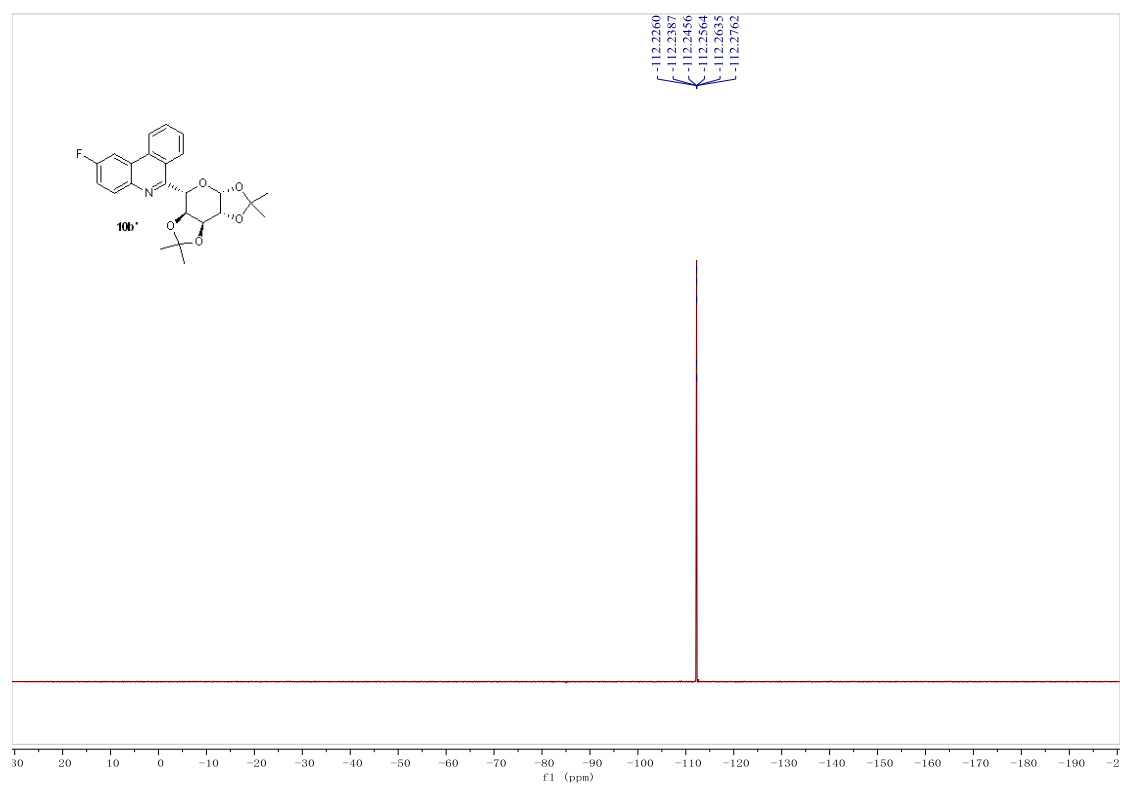
^{19}F NMR spectrum for **10b**



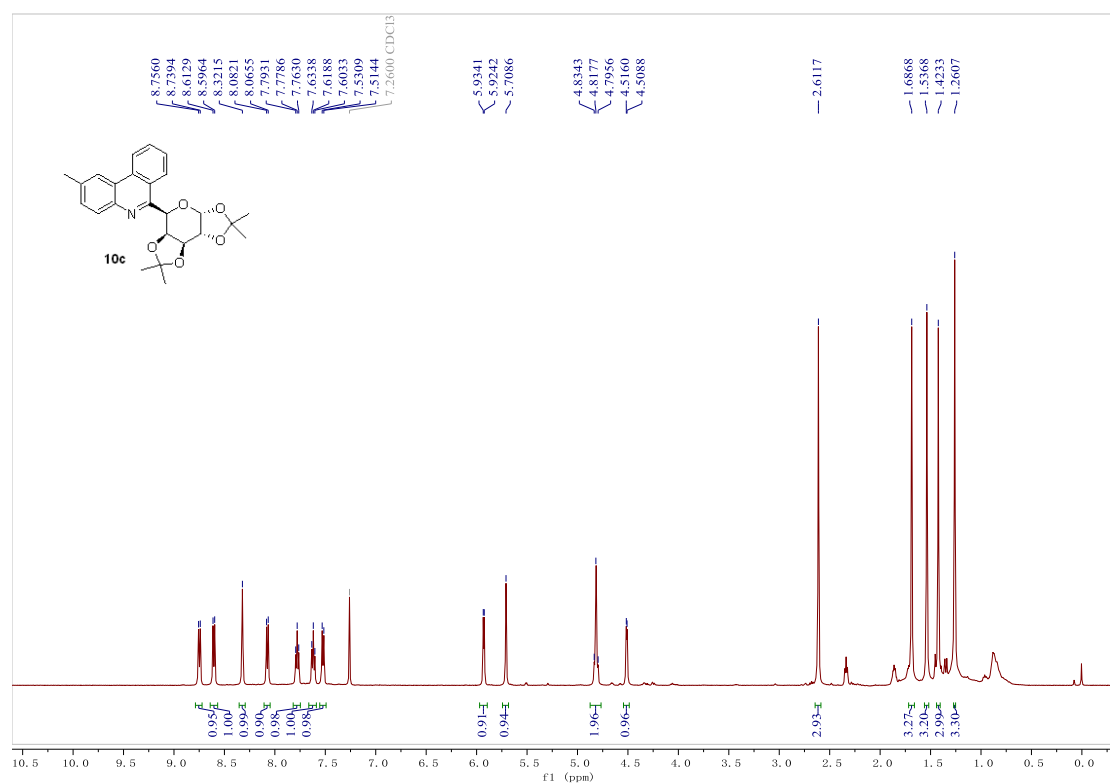
¹H NMR spectrum for 10b' (500 MHz, CDCl₃)



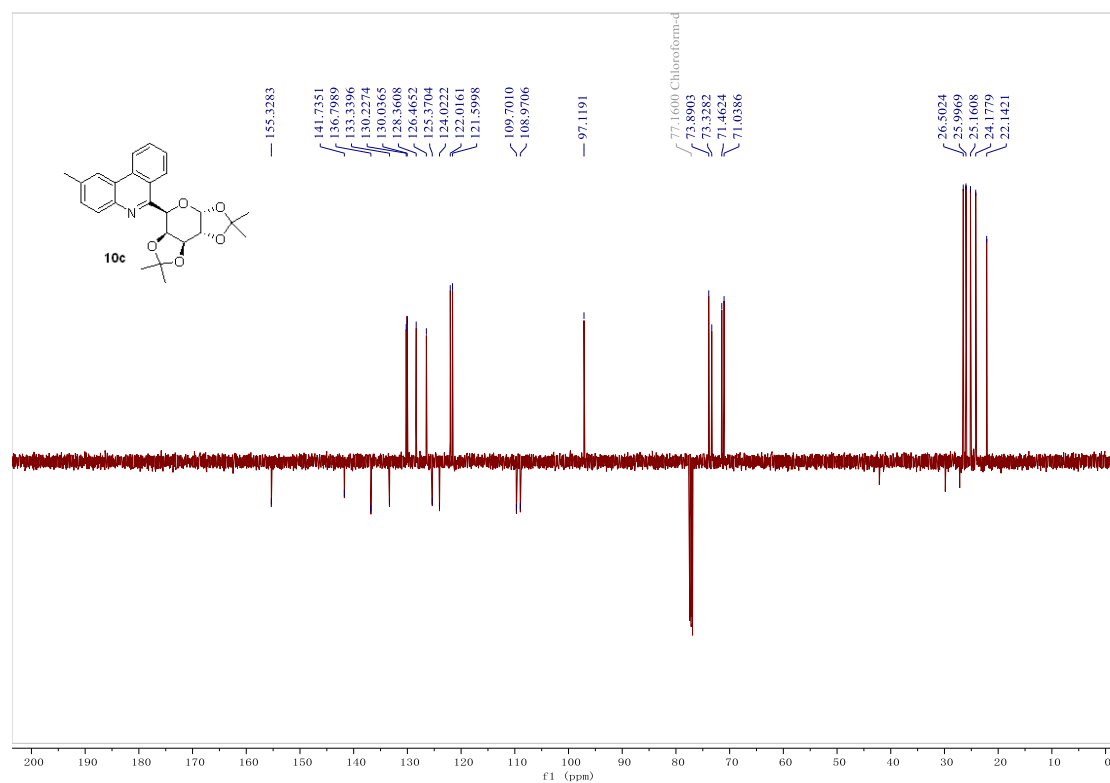
DEPT-Q spectrum for 10b' (126 MHz, CDCl₃)



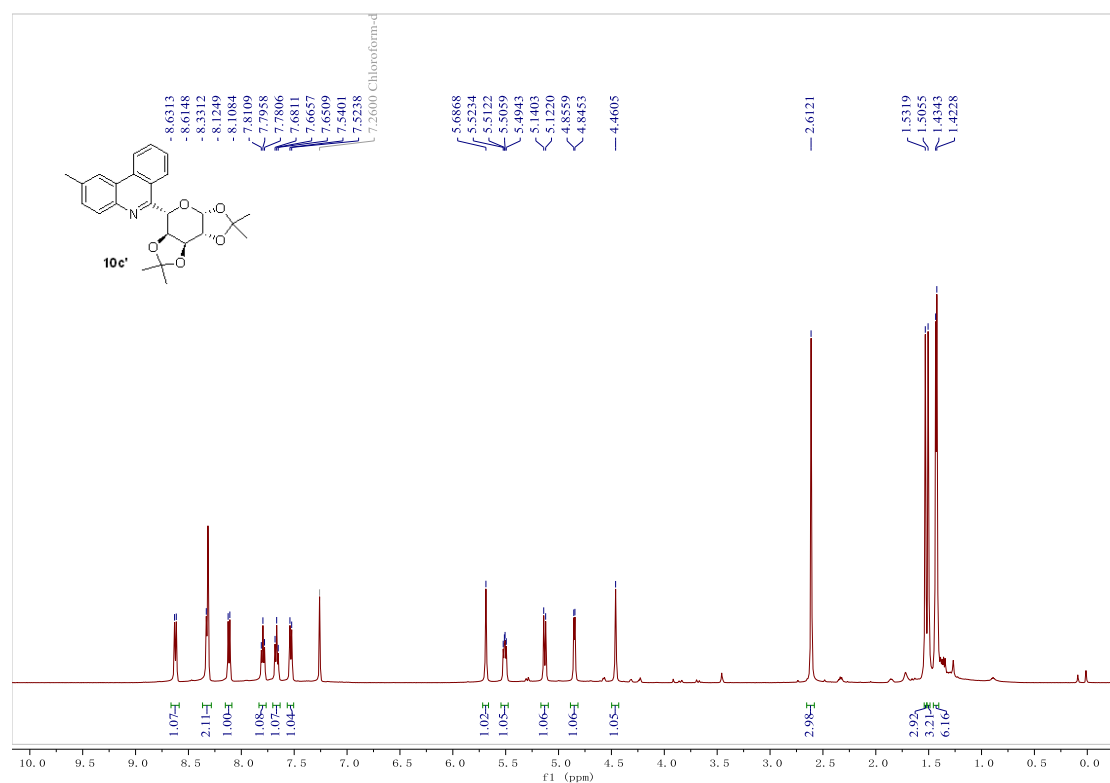
^{19}F NMR spectrum for **10b'**



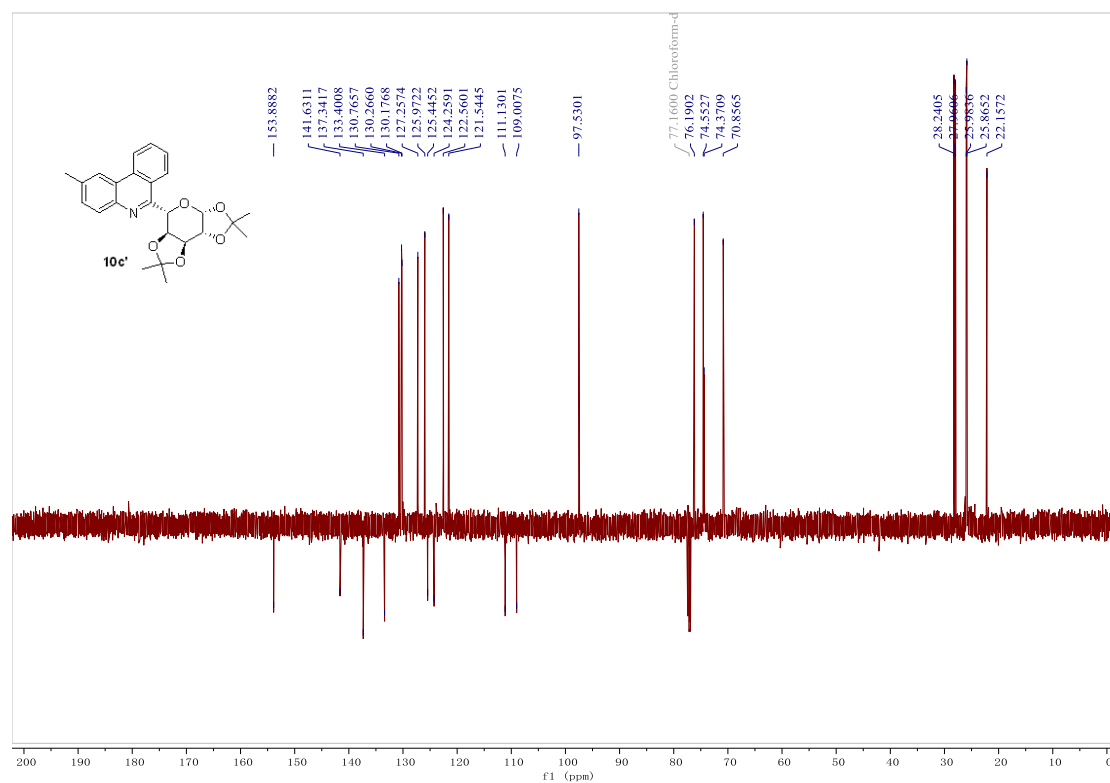
¹H NMR spectrum for 10c (500 MHz, CDCl₃)



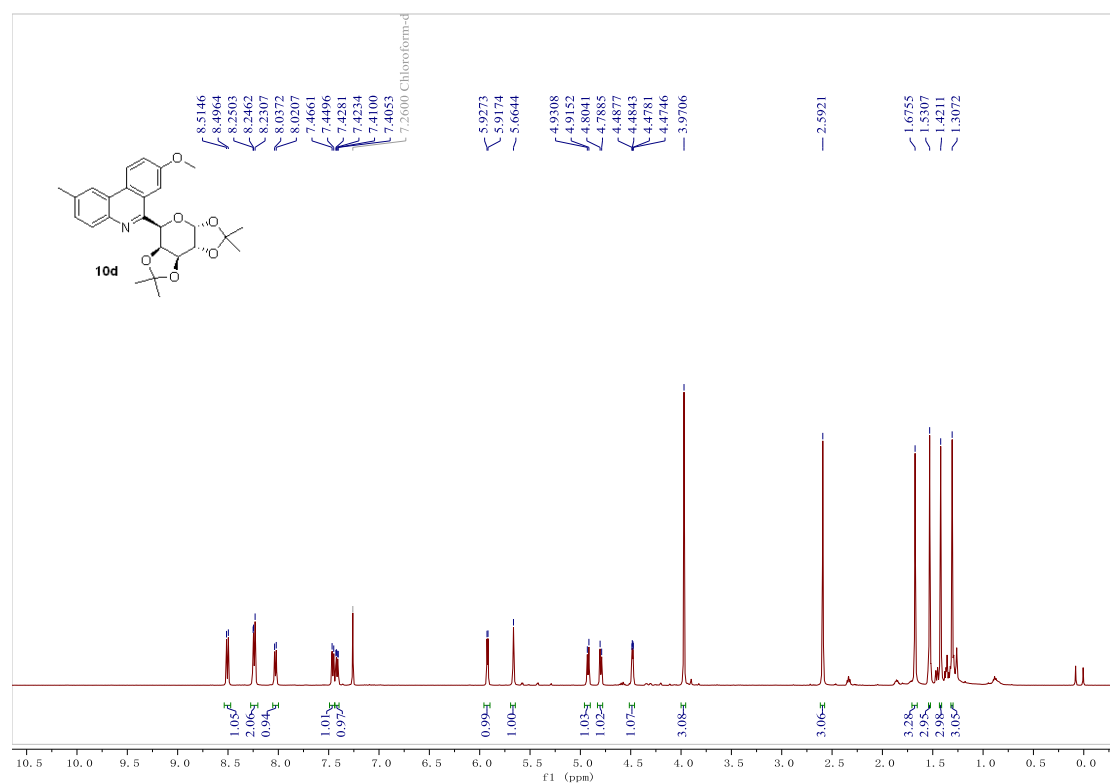
DEPT-Q spectrum for 10c (126 MHz, CDCl₃)



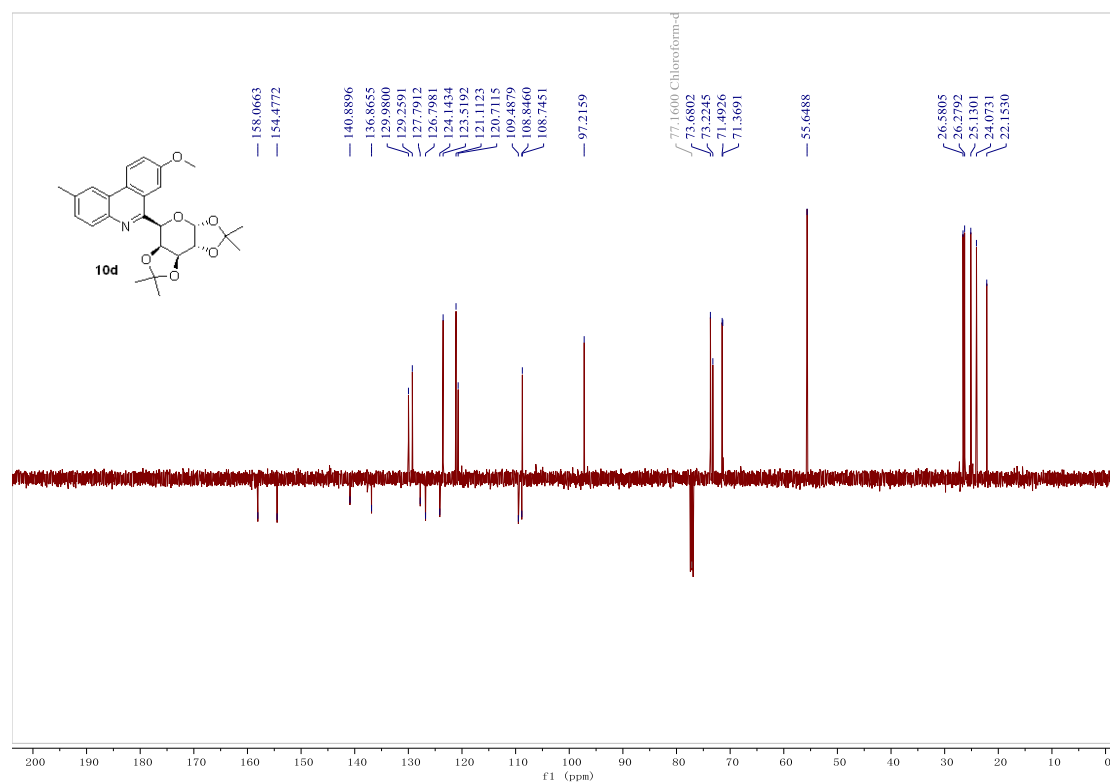
¹H NMR spectrum for 10c' (500 MHz, CDCl₃)



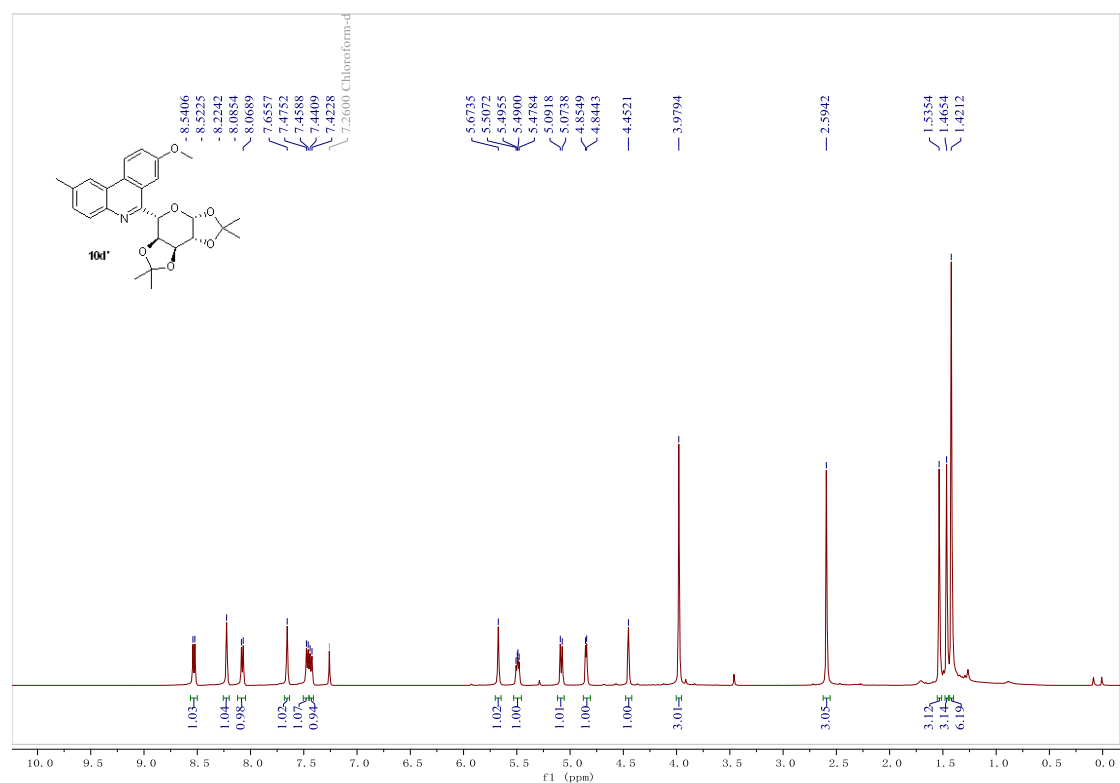
DEPT-Q spectrum for 10c' (126 MHz, CDCl₃)



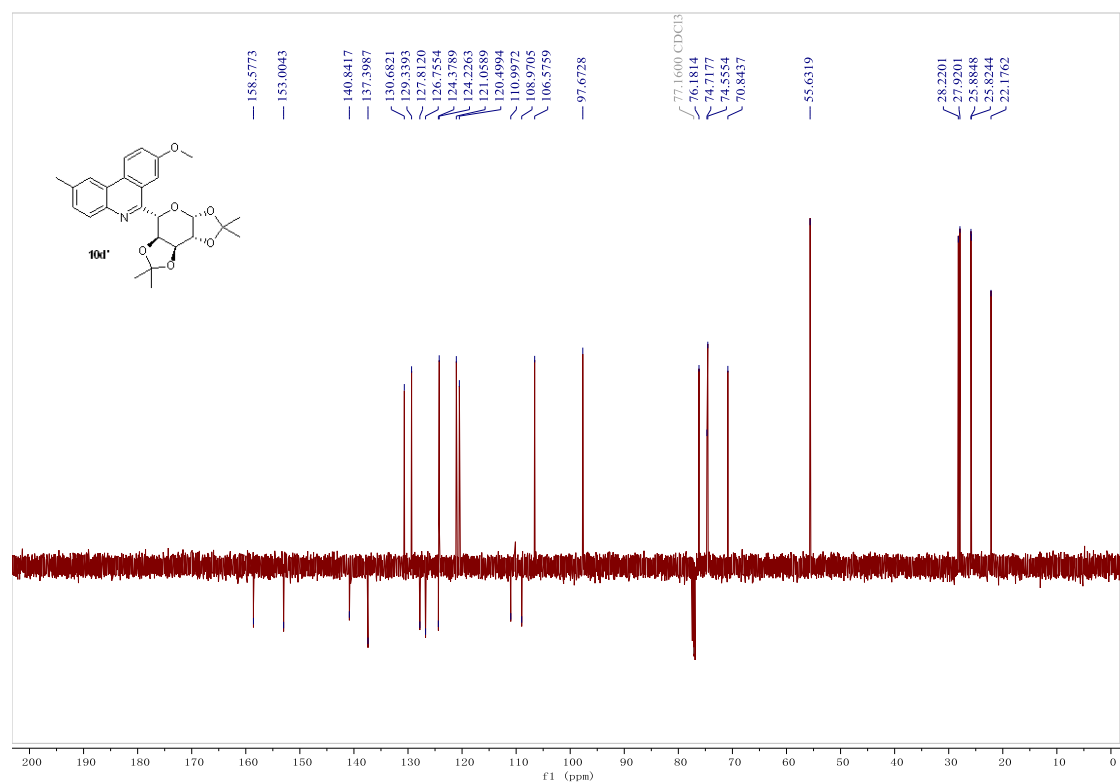
¹H NMR spectrum for 10d (500 MHz, CDCl₃)



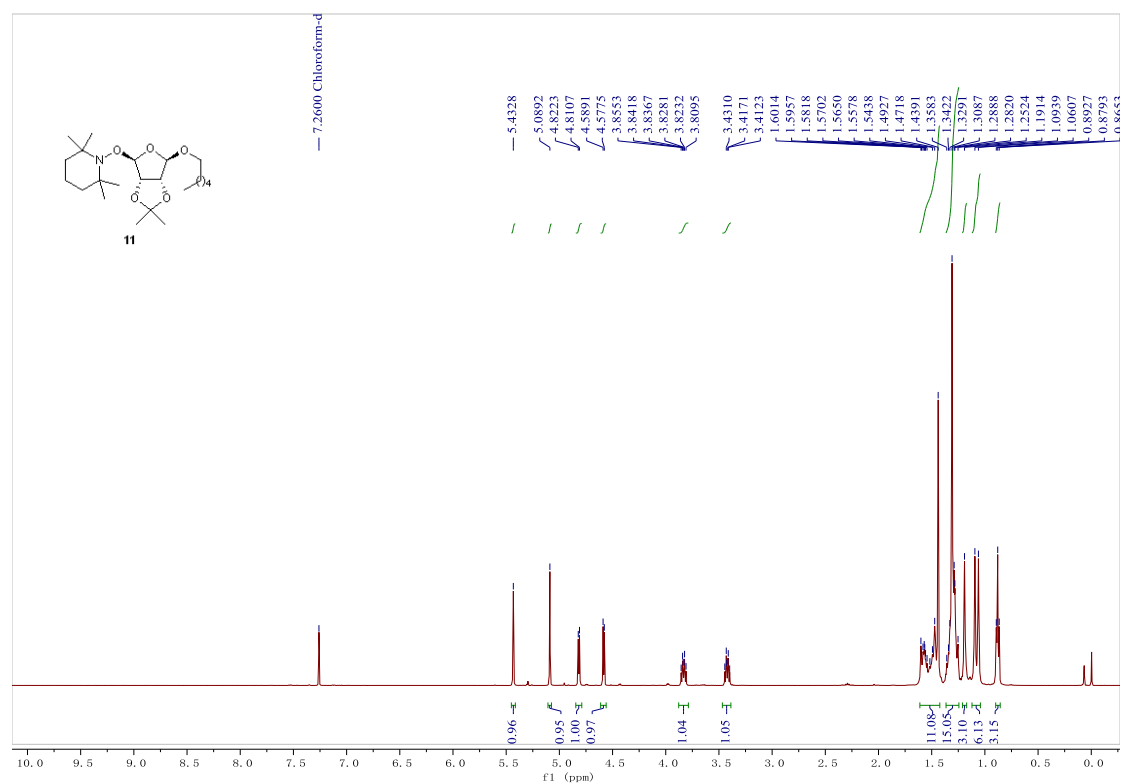
DEPT-Q spectrum for 10d (126 MHz, CDCl₃)

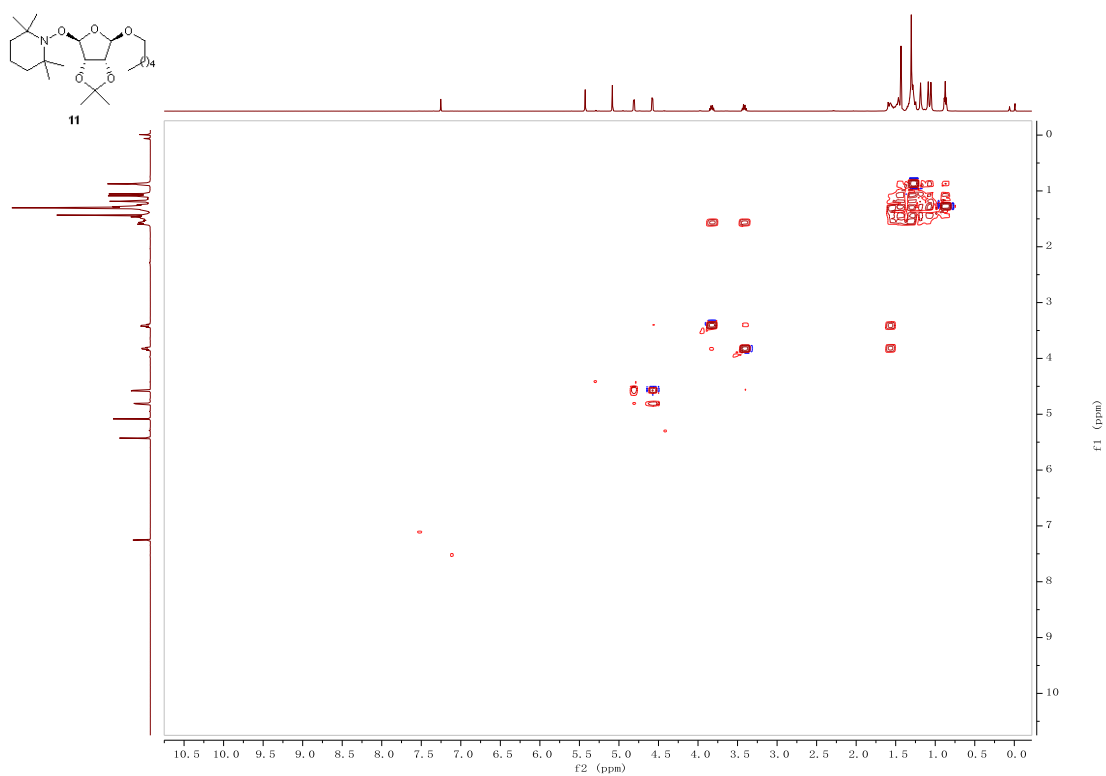


¹H NMR spectrum for 10d' (500 MHz, CDCl₃)

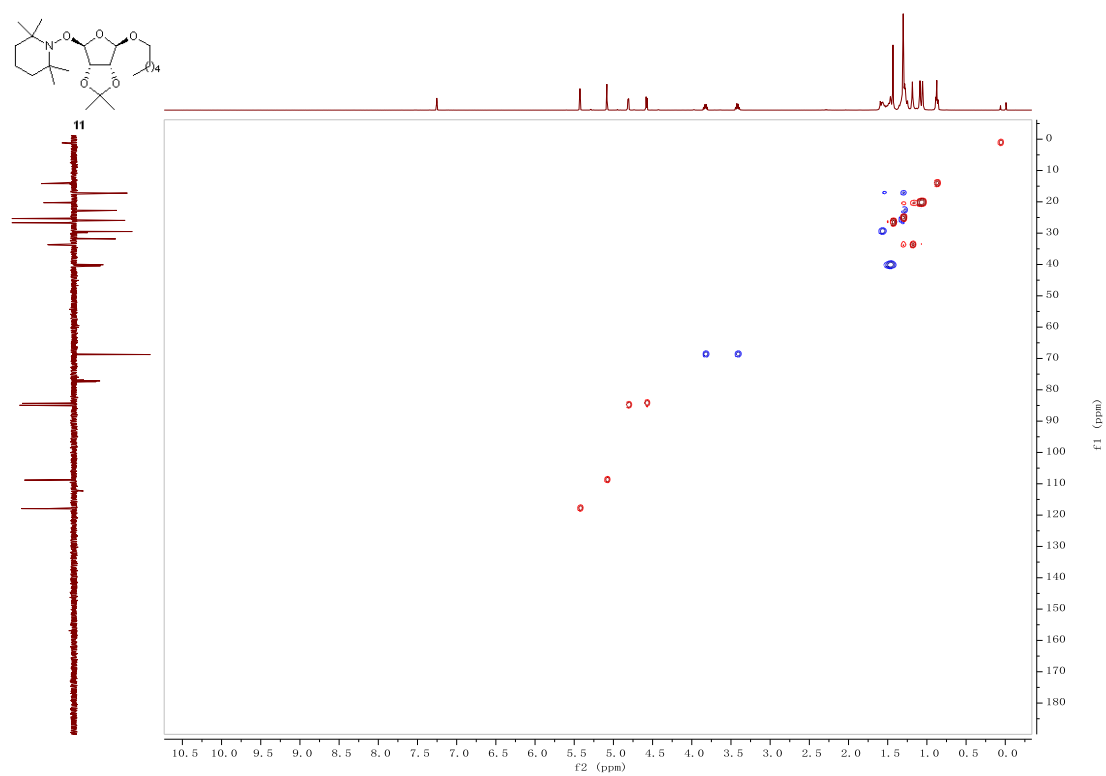


DEPT-Q spectrum for 10d' (126 MHz, CDCl₃)

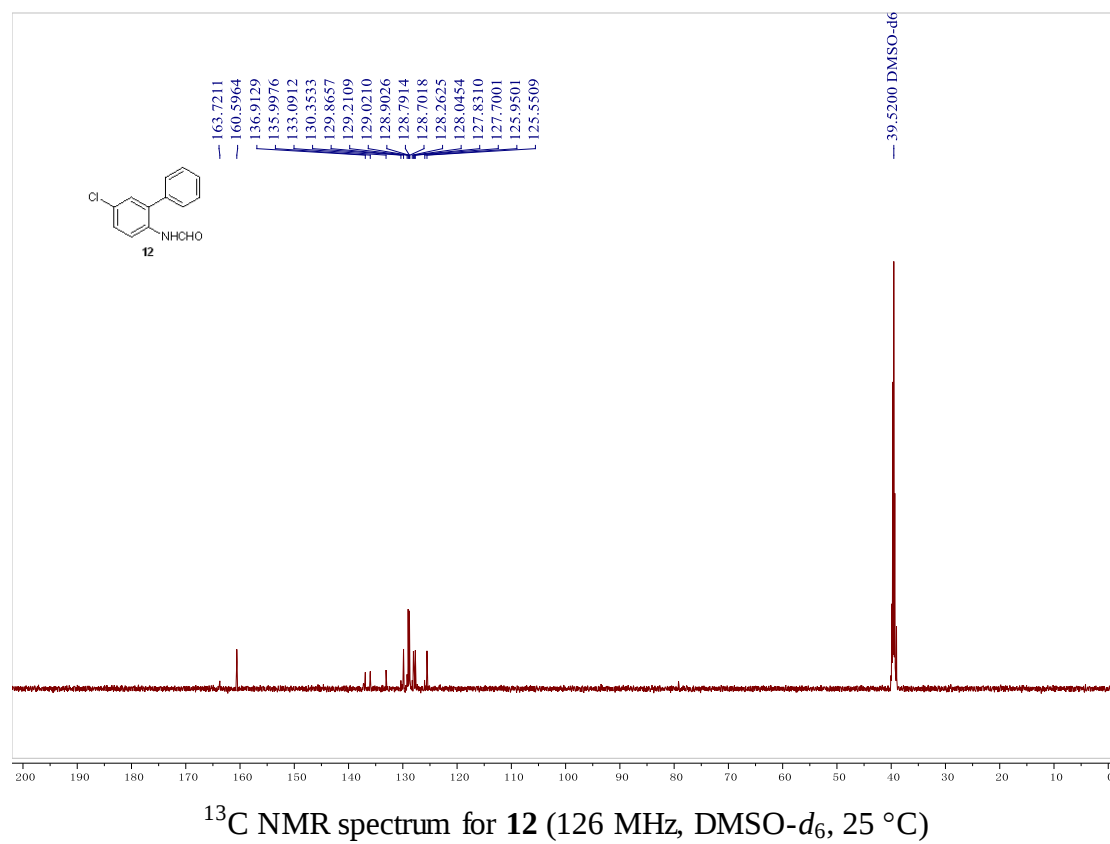
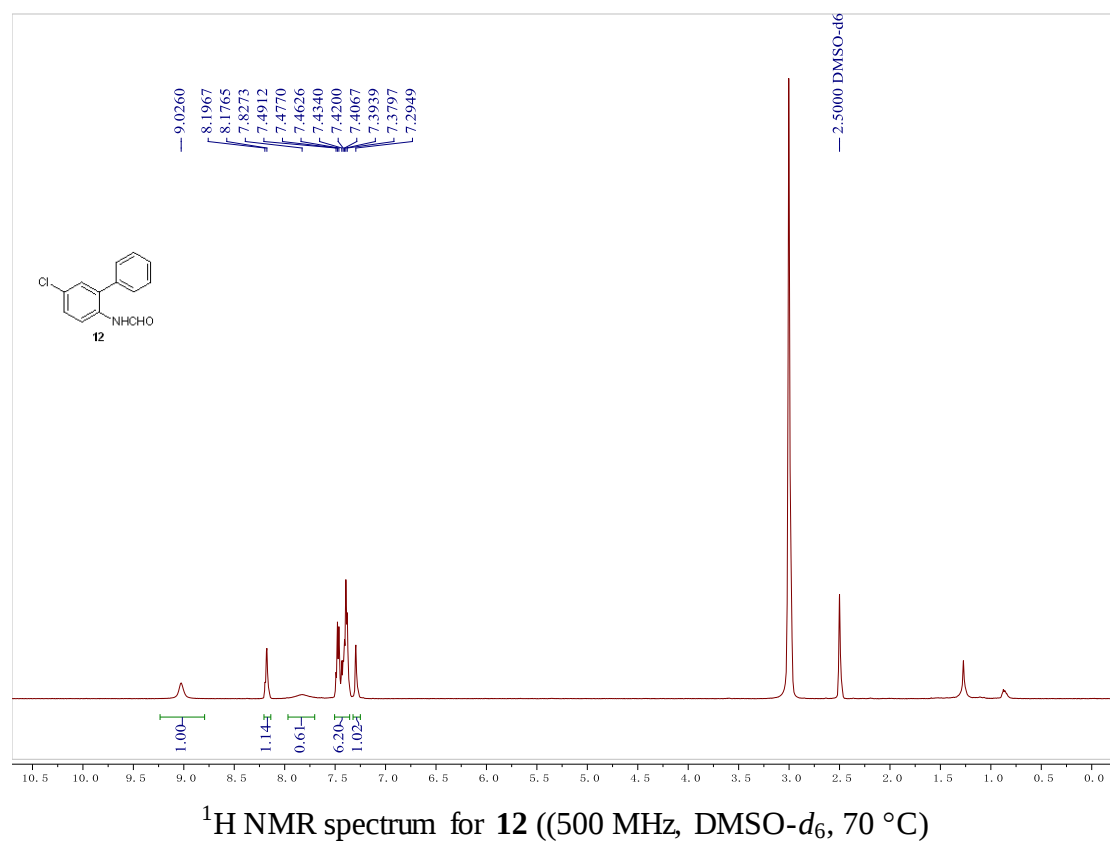


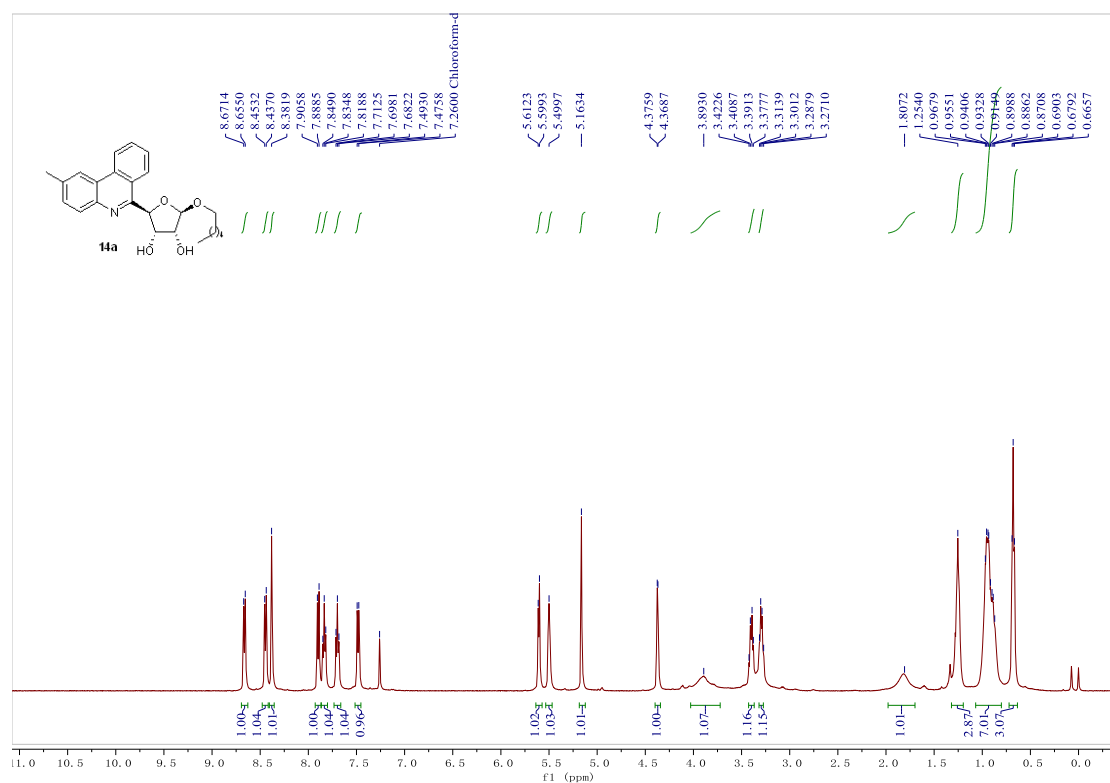


COSY spectrum for **11**

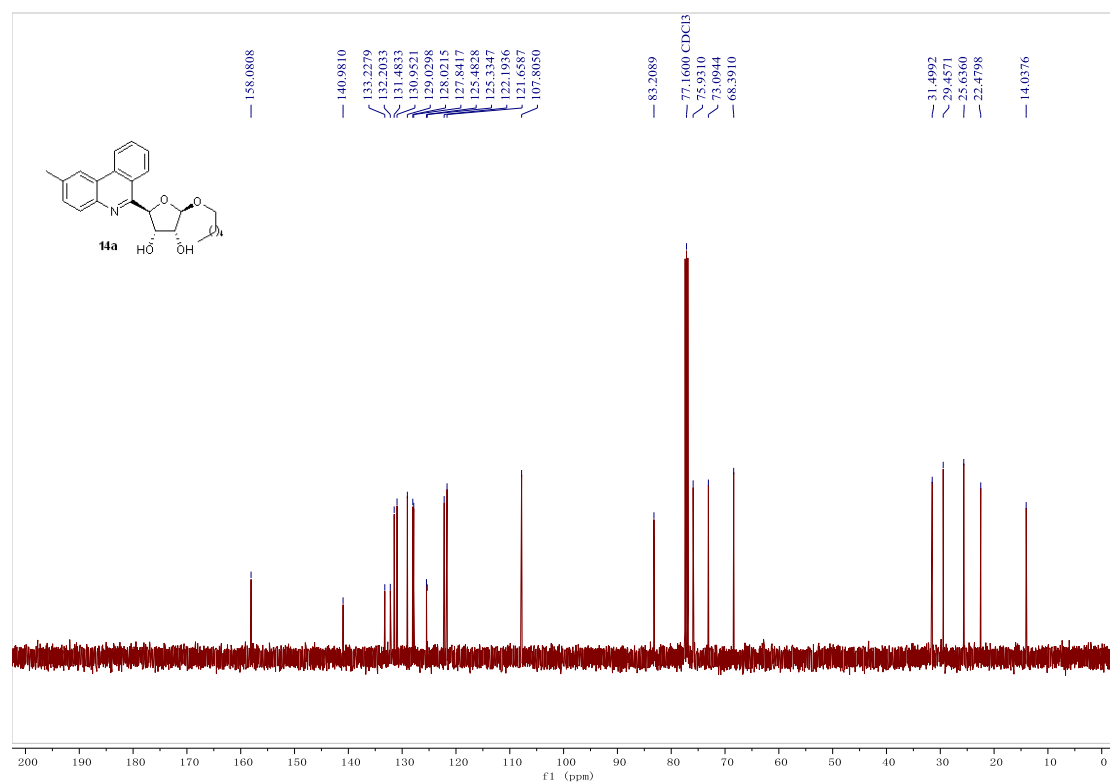


HSQC spectrum for **11**

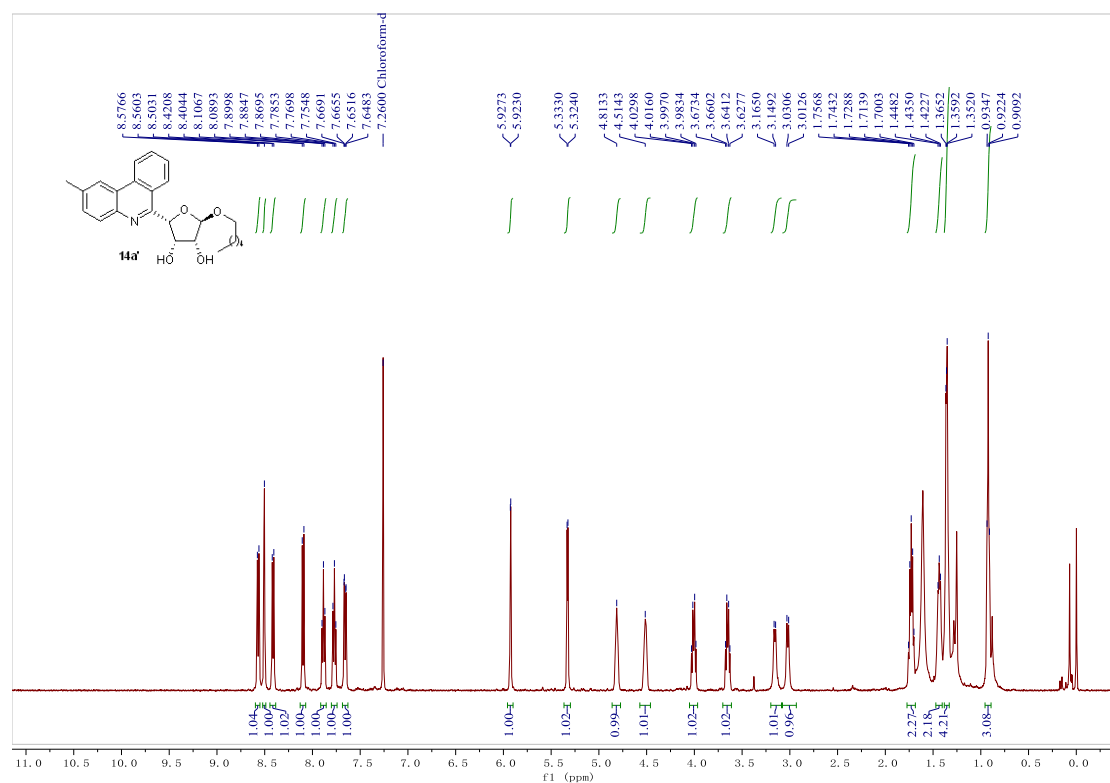




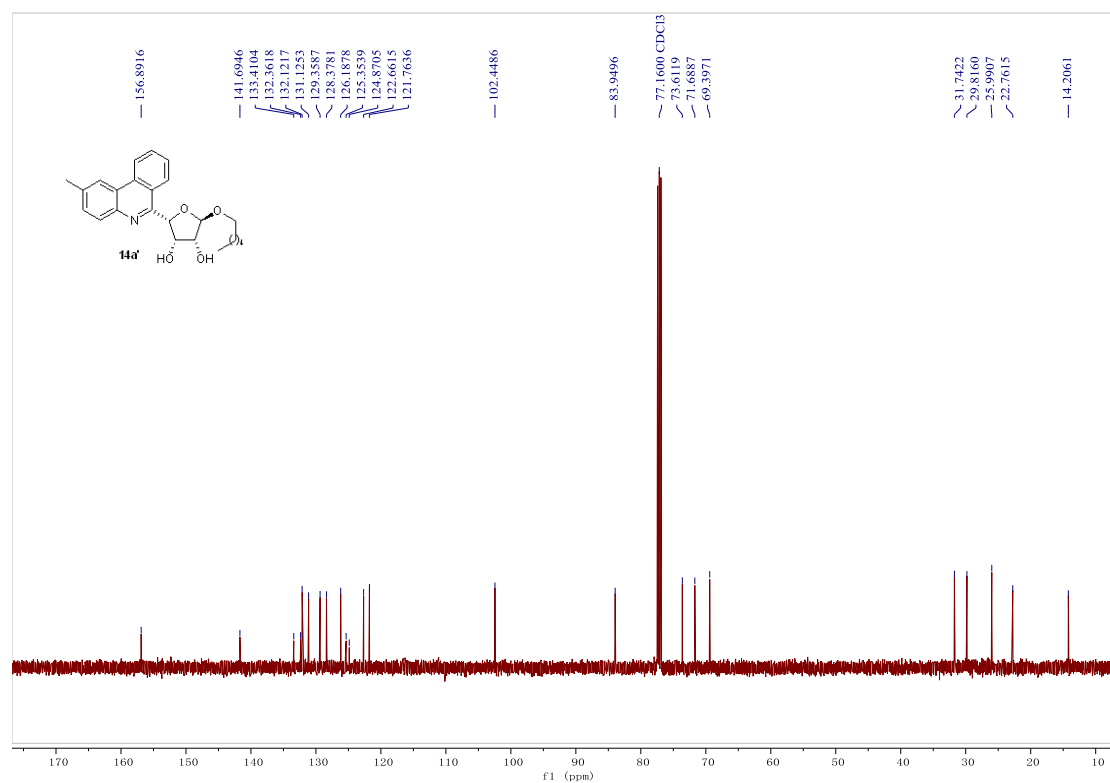
¹H NMR spectrum for 14a (500 MHz, CDCl₃)



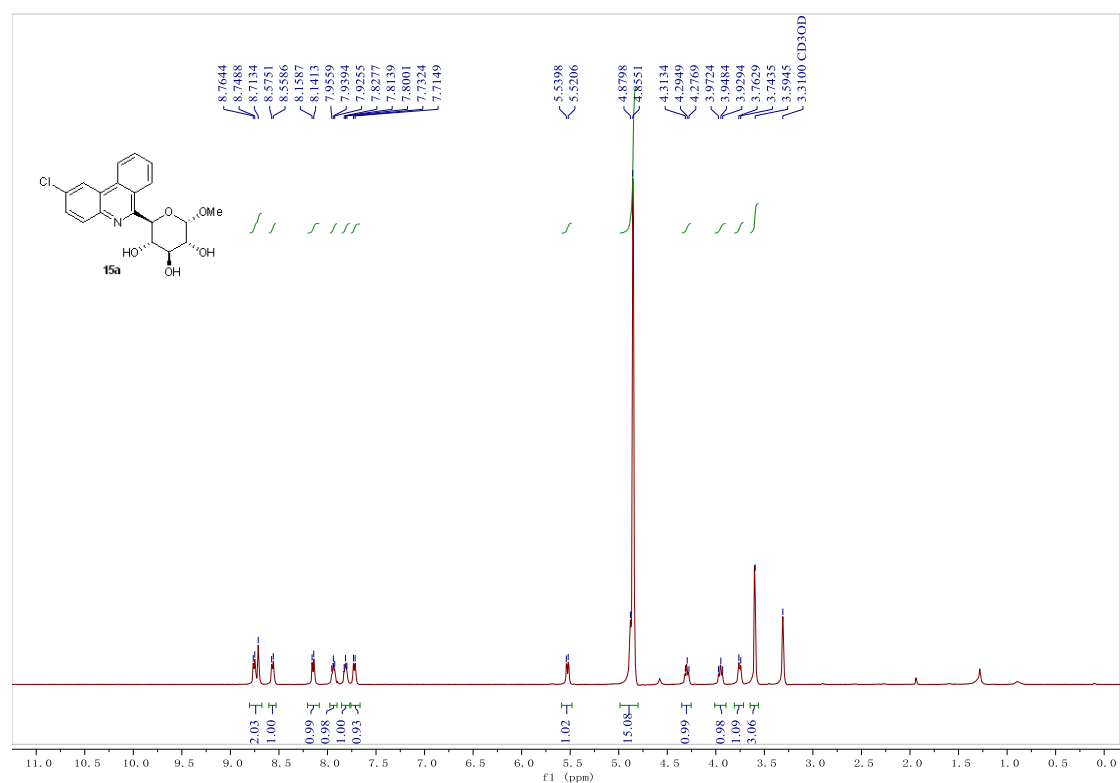
¹³C NMR spectrum for 14a (126 MHz, CDCl₃)



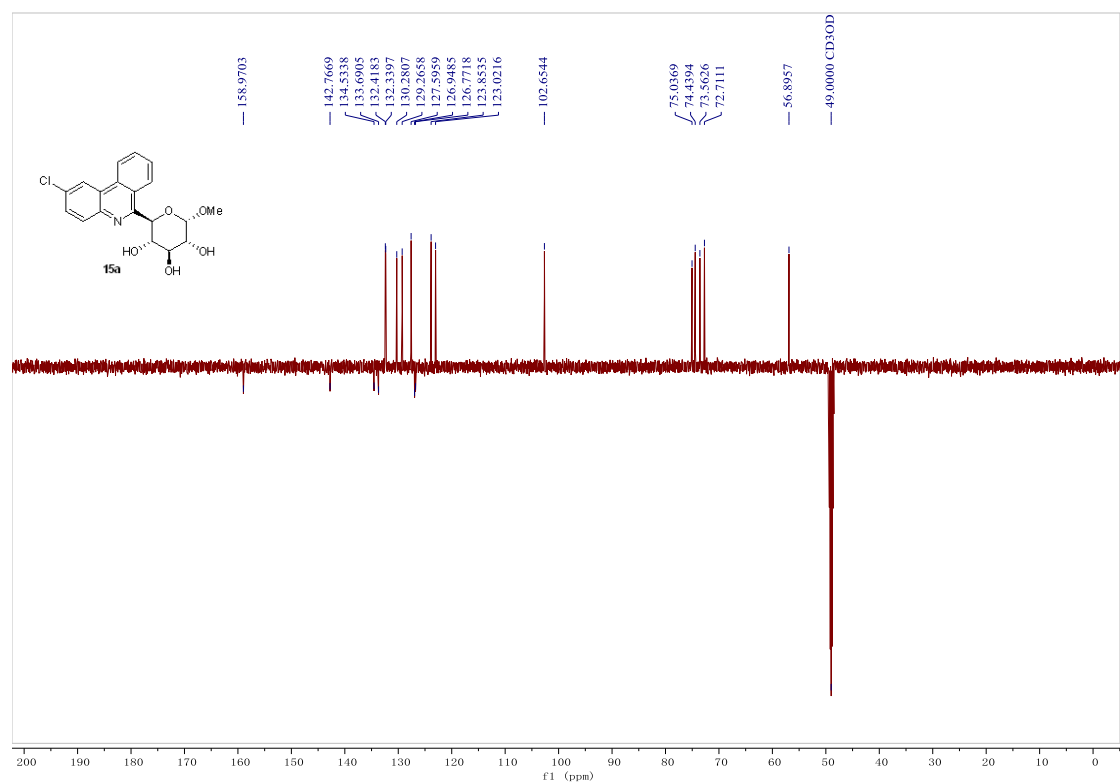
¹H NMR spectrum for **14a' (500 MHz, CDCl₃)**



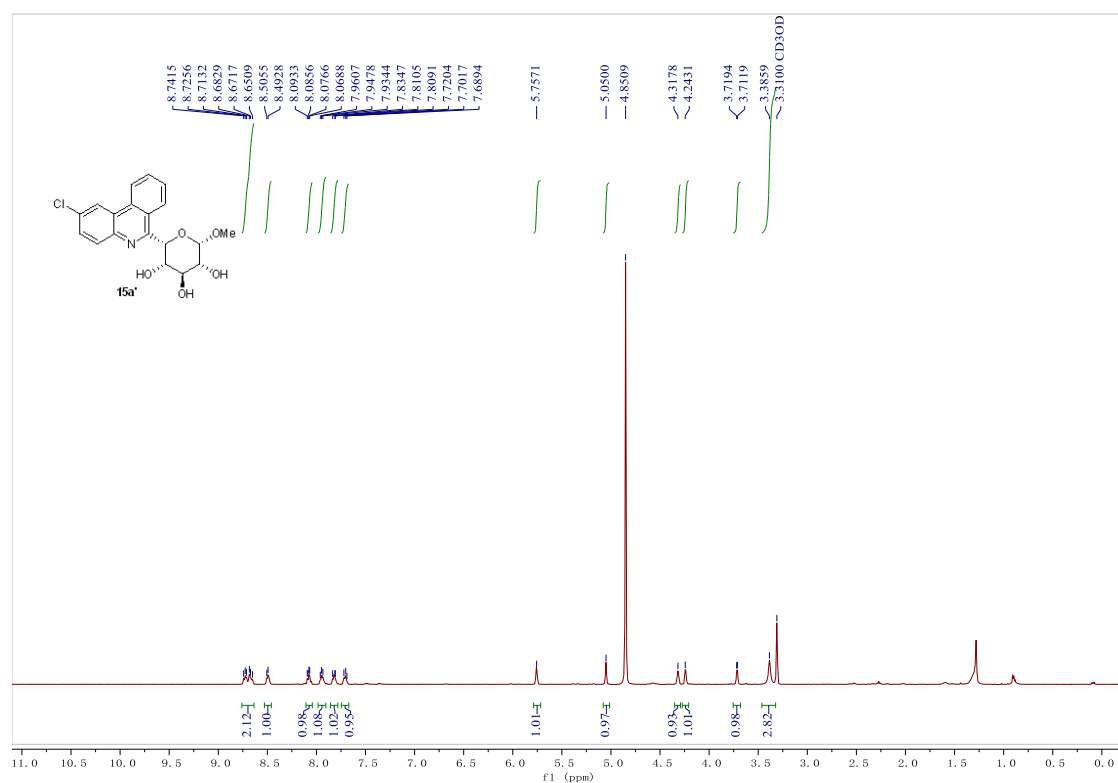
¹³C NMR spectrum for **14a' (126 MHz, CDCl₃)**



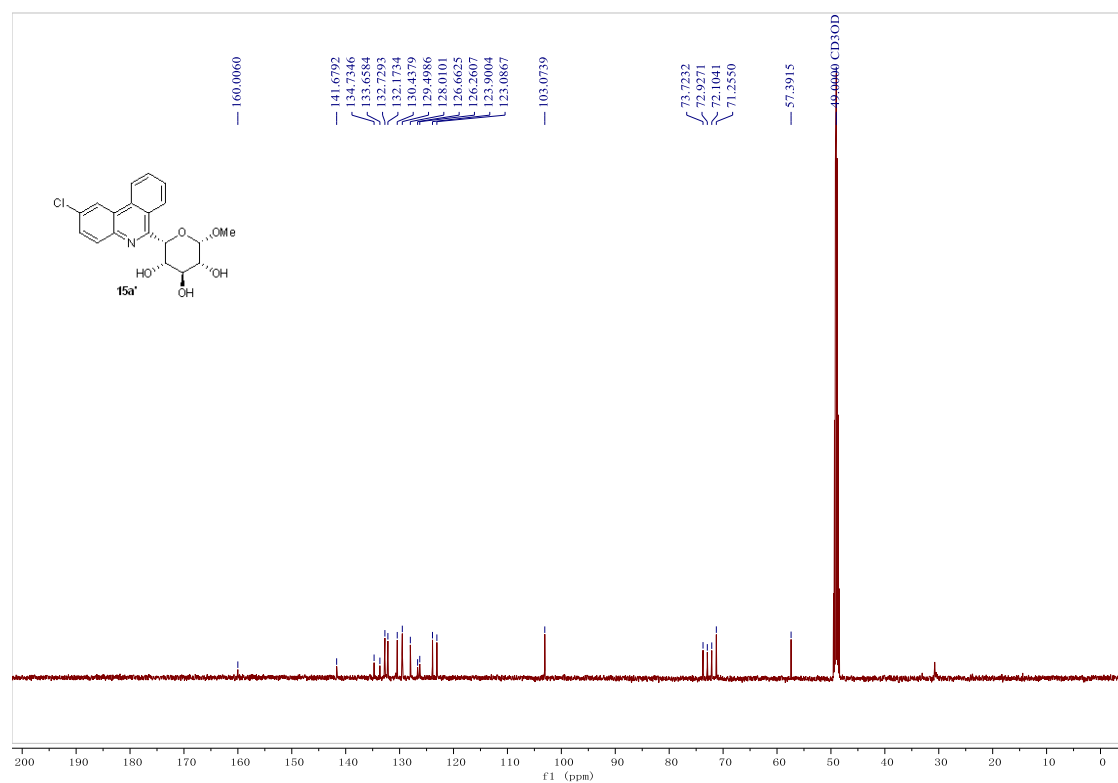
¹H NMR spectrum for 15a (500 MHz, CD₃OD)



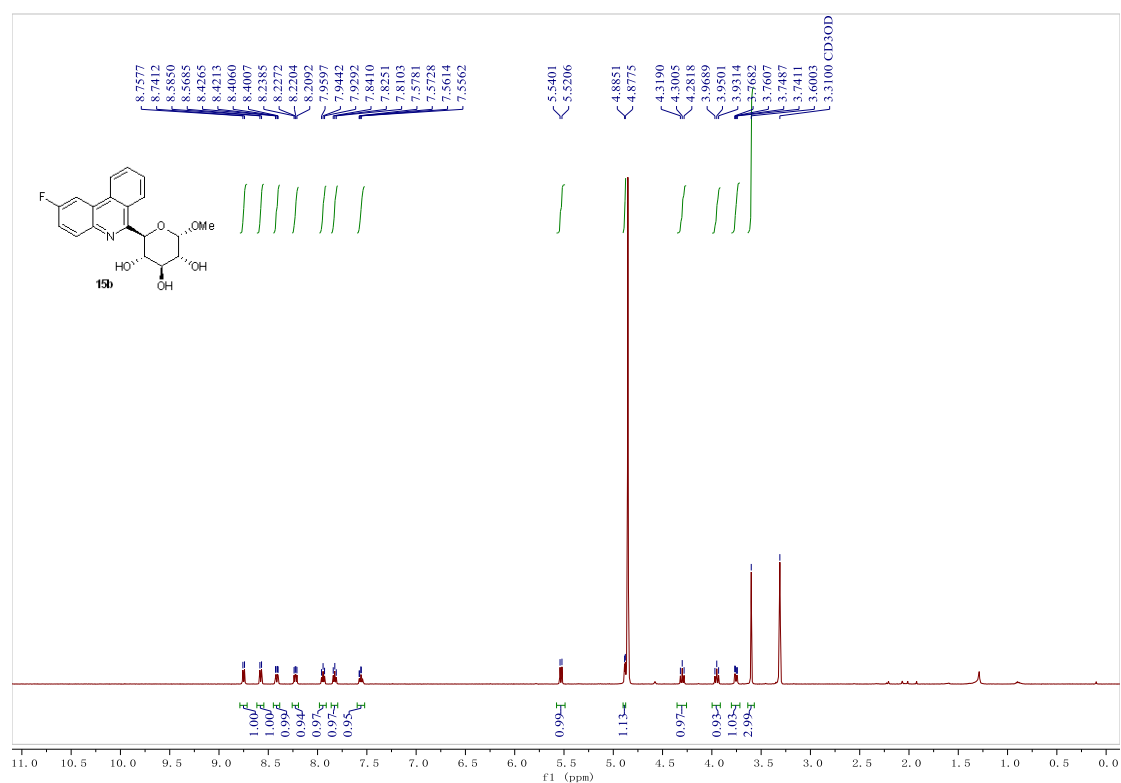
DEPT-Q spectrum for 15a (126 MHz, CD₃OD)



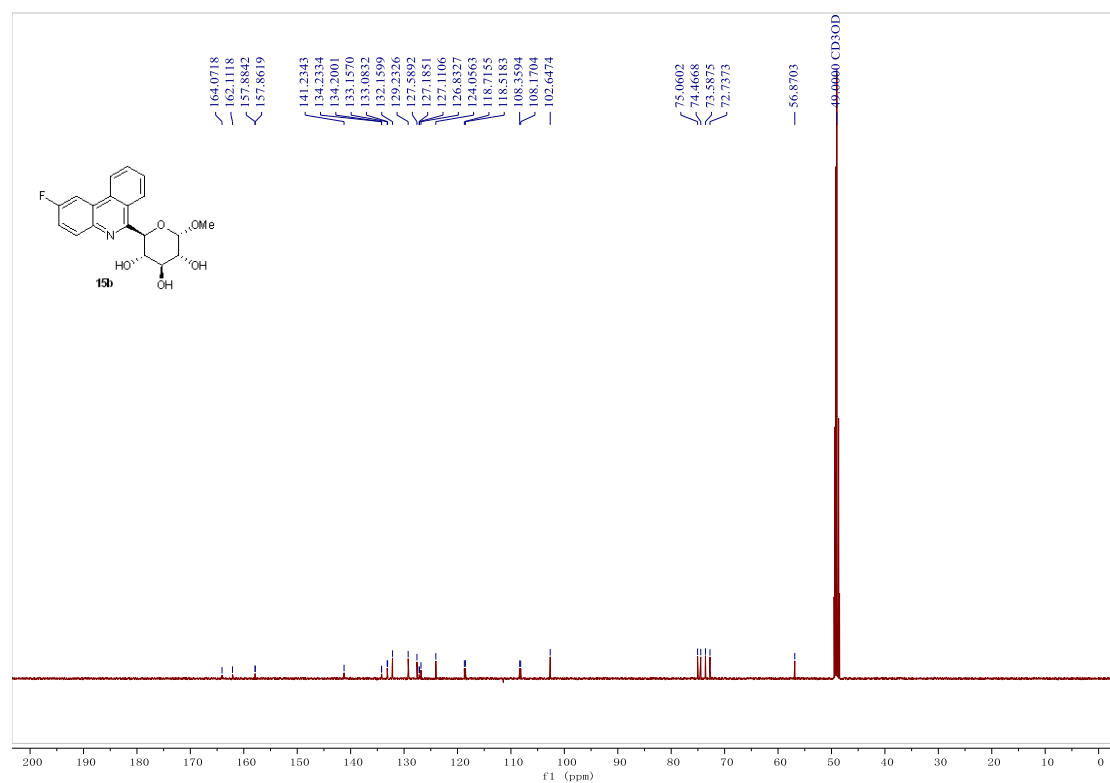
¹H NMR spectrum for 15a' (500 MHz, CD₃OD)



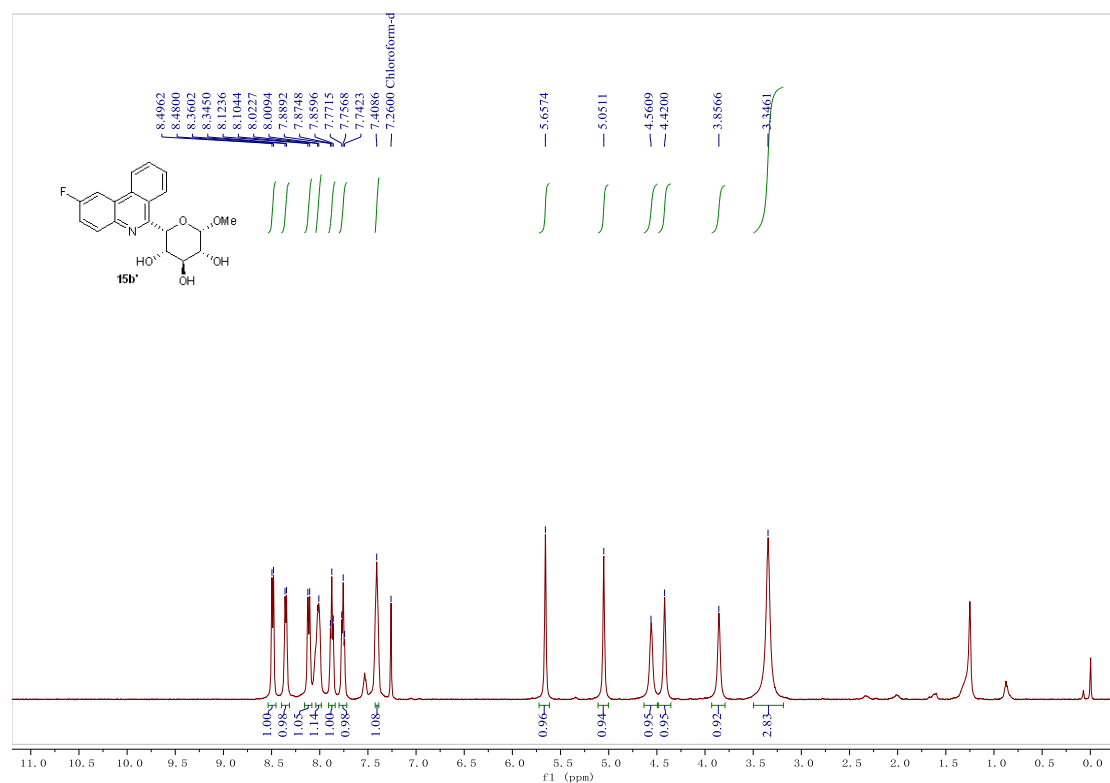
¹³C NMR spectrum for 15a' (126 MHz, CD₃OD)



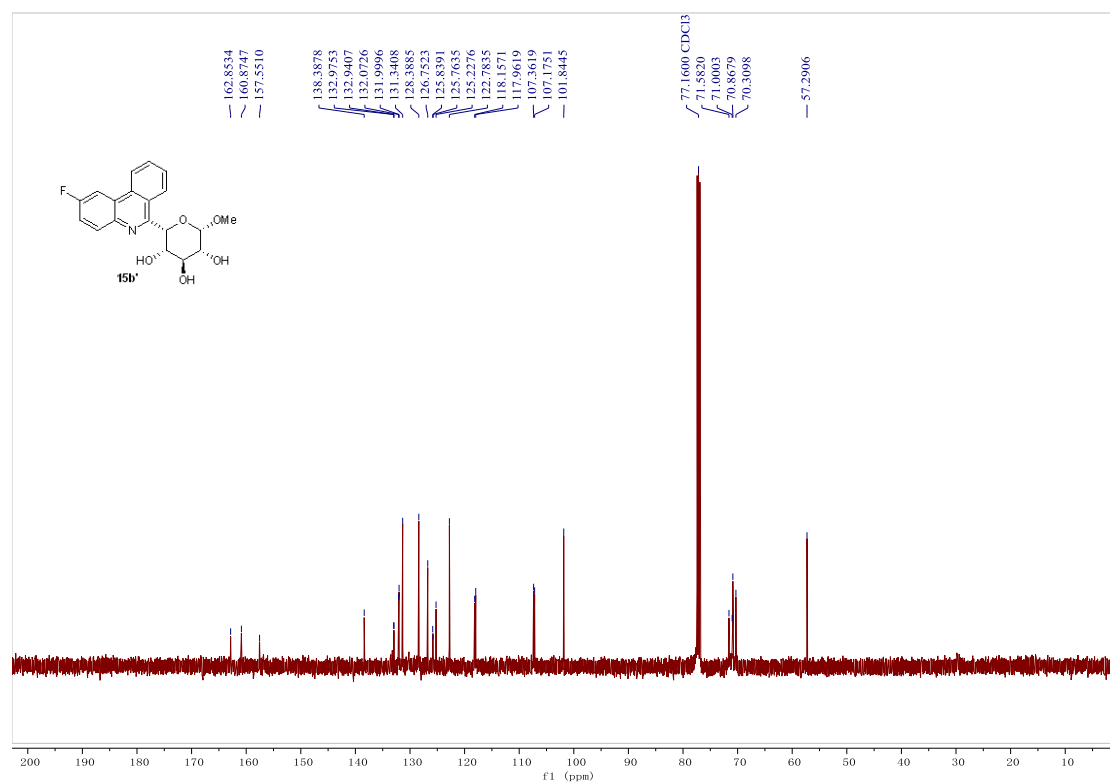
¹H NMR spectrum for 15b (500 MHz, CD₃OD)



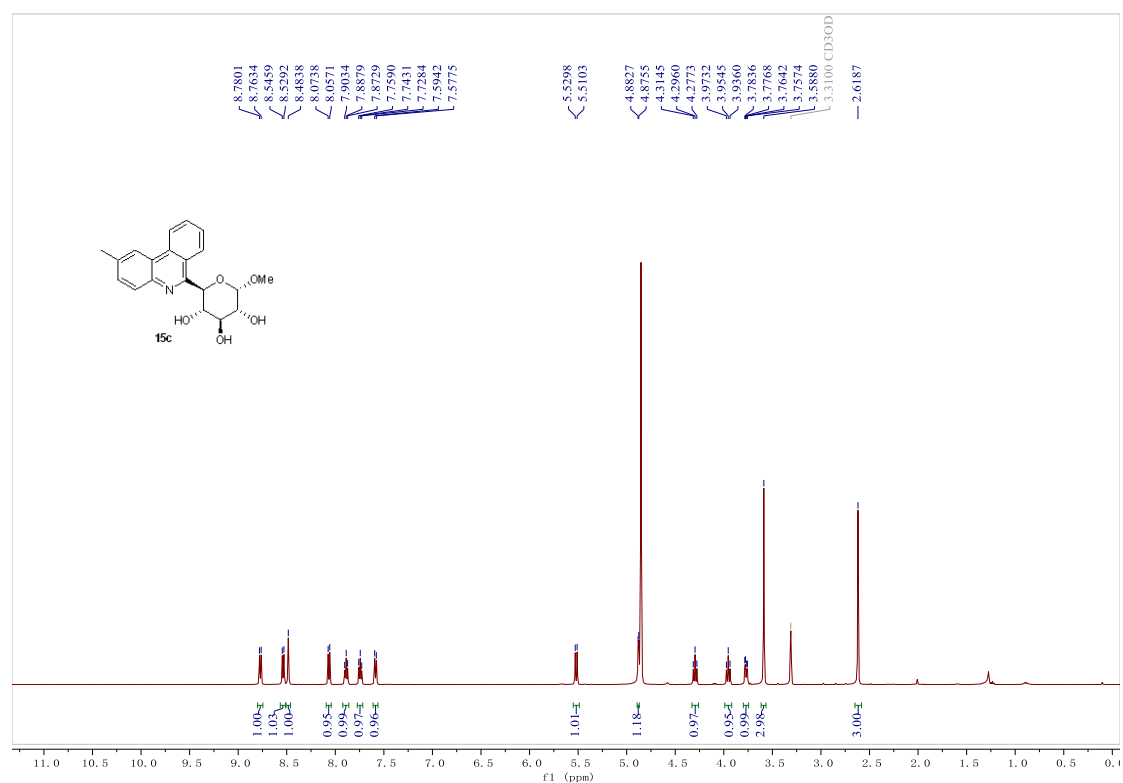
¹³C NMR spectrum for 15b (126 MHz, CD₃OD)



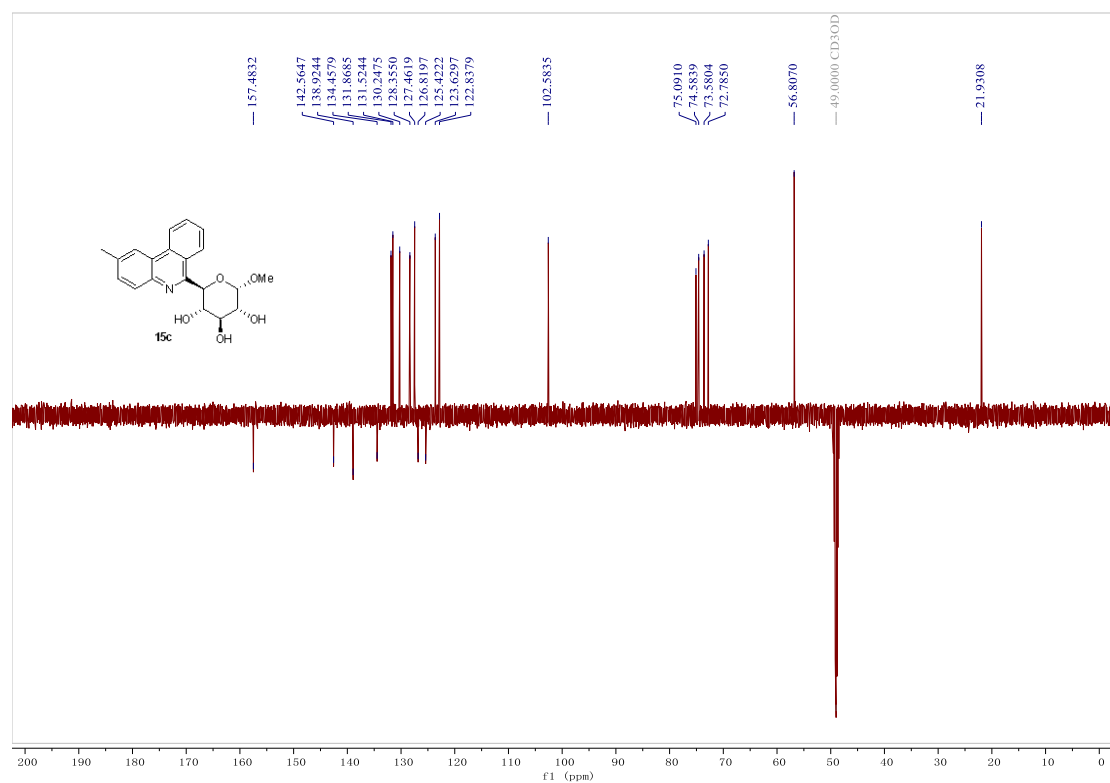
^1H NMR spectrum for **15b'** (500 MHz, CDCl_3)



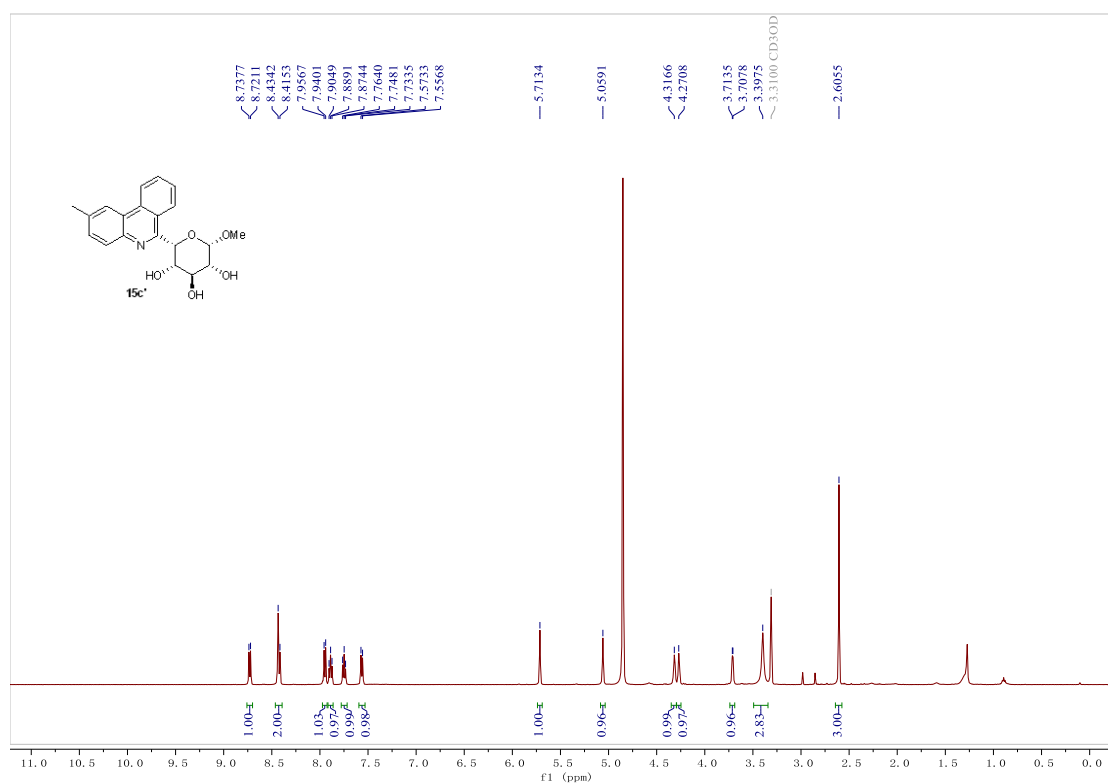
^{13}C NMR spectrum for **15b'** (126 MHz, CDCl_3)



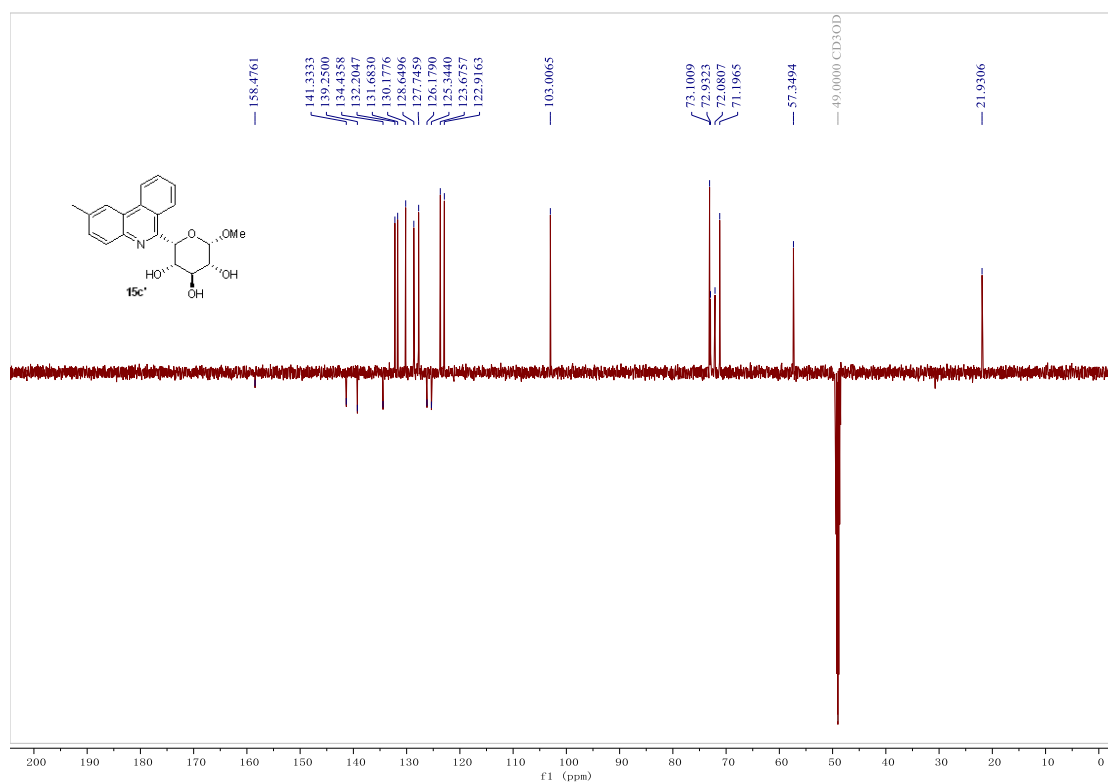
¹H NMR spectrum for **15c** (500 MHz, CD₃OD)



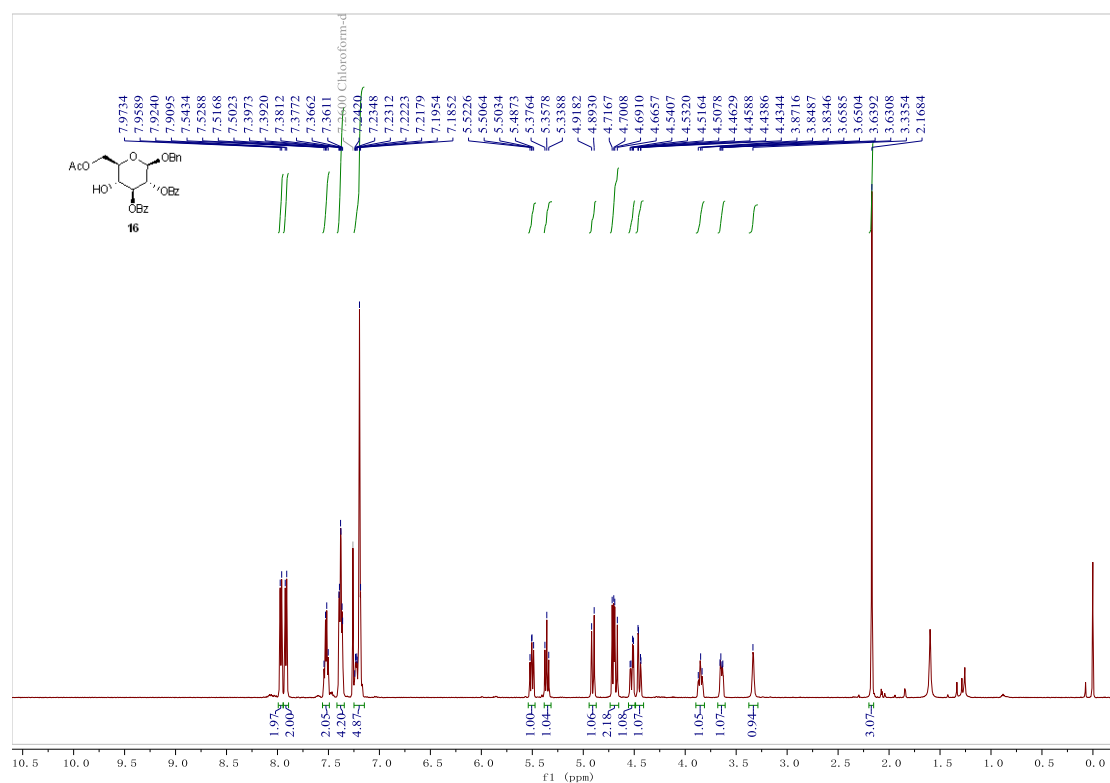
DEPT-Q spectrum for **15c** (126 MHz, CD₃OD)



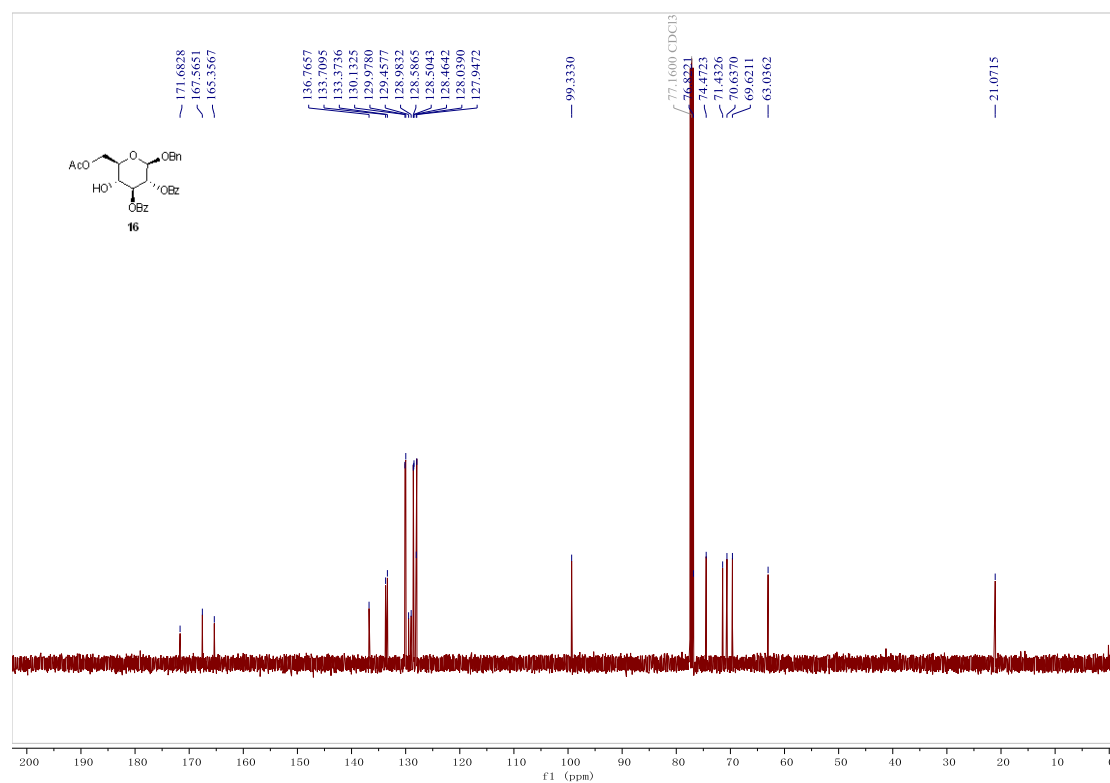
¹H NMR spectrum for 15c' (500 MHz, CD₃OD)



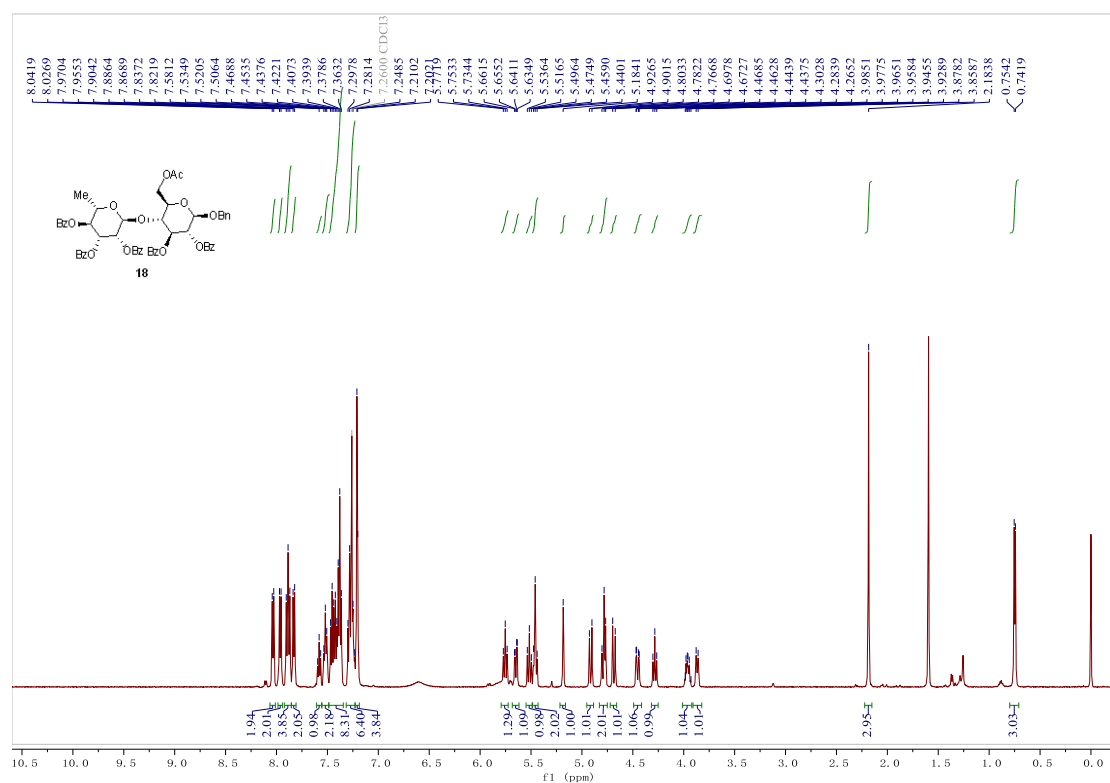
DEPT-Q spectrum for 15c' (126 MHz, CD₃OD)



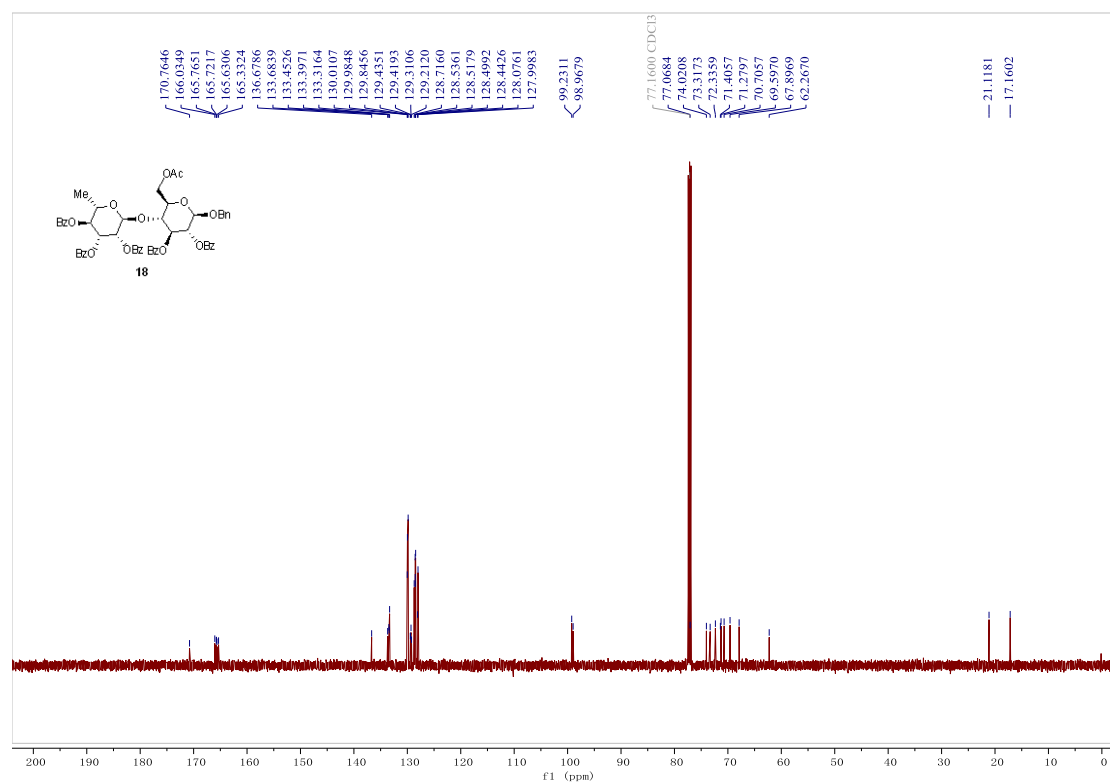
¹H NMR spectrum for **16** (500 MHz, CDCl₃)



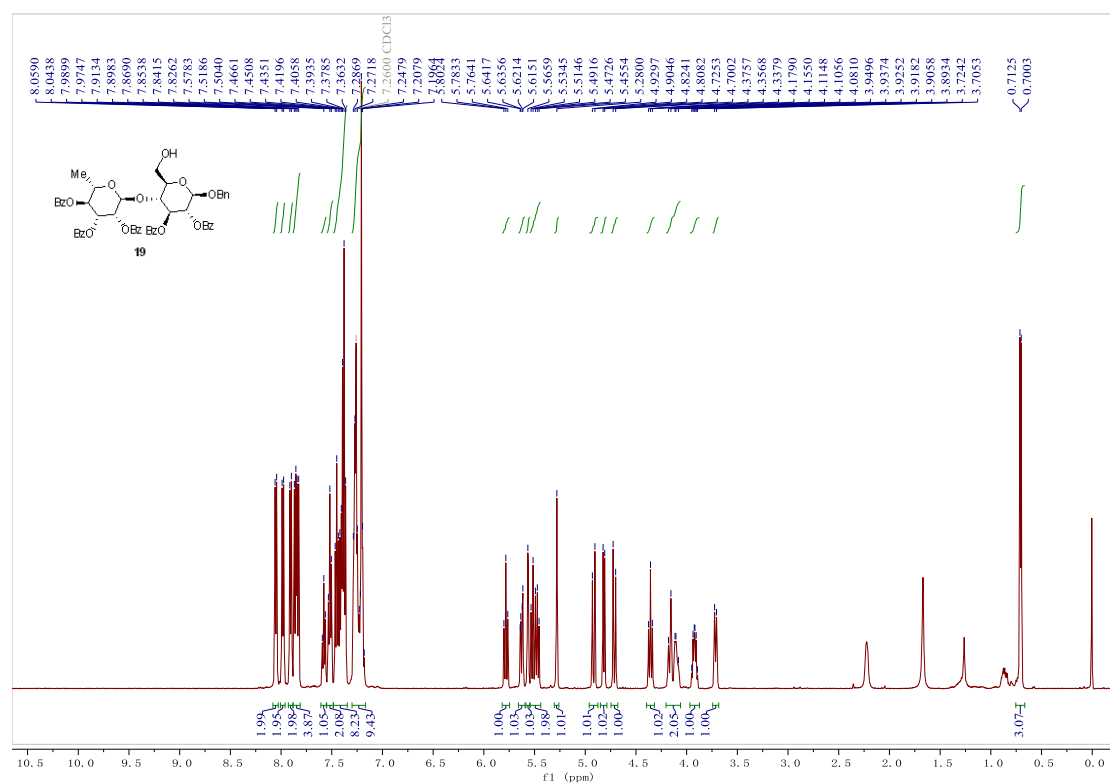
¹³C NMR spectrum for **16** (126 MHz, CDCl₃)



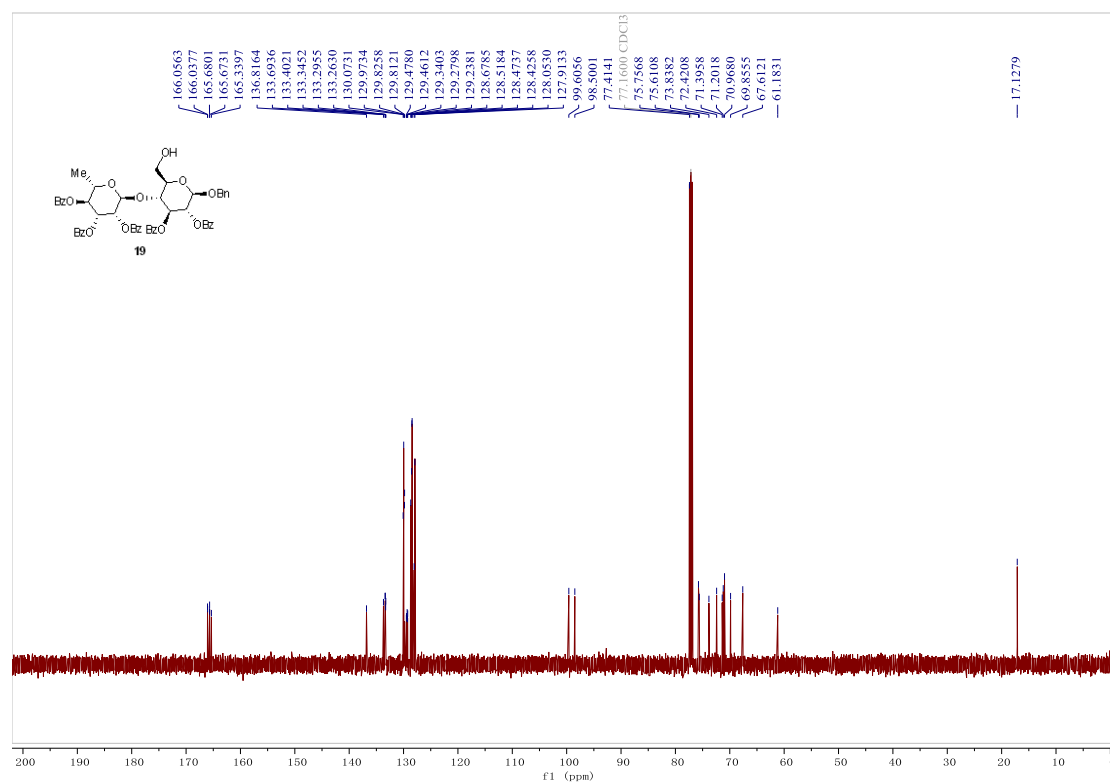
¹H NMR spectrum for **18** (500 MHz, CDCl₃)



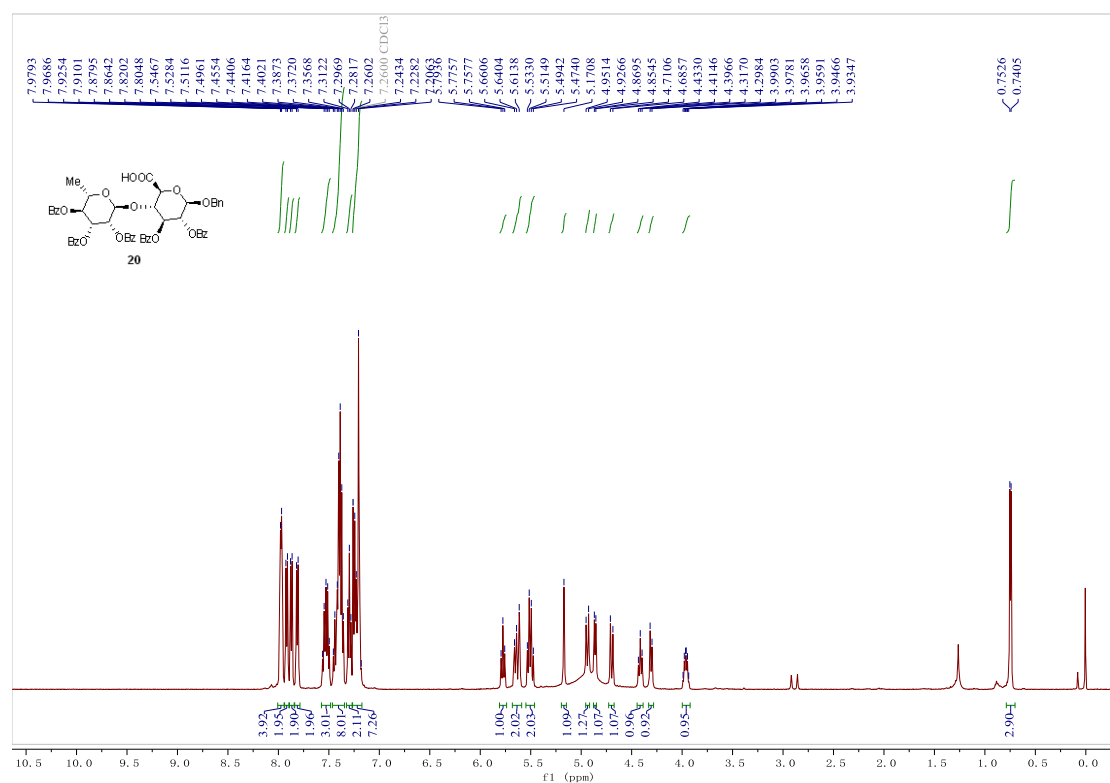
¹³C NMR spectrum for **18** (126 MHz, CDCl₃)



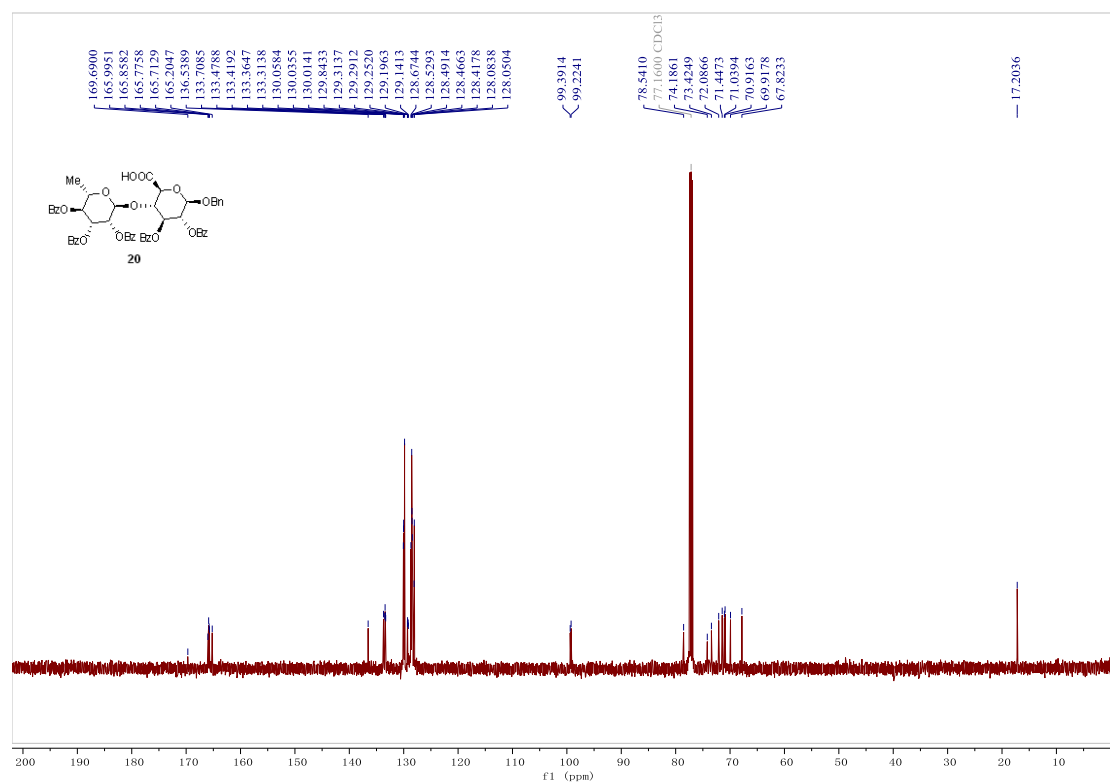
¹H NMR spectrum for 19 (500 MHz, CDCl₃)



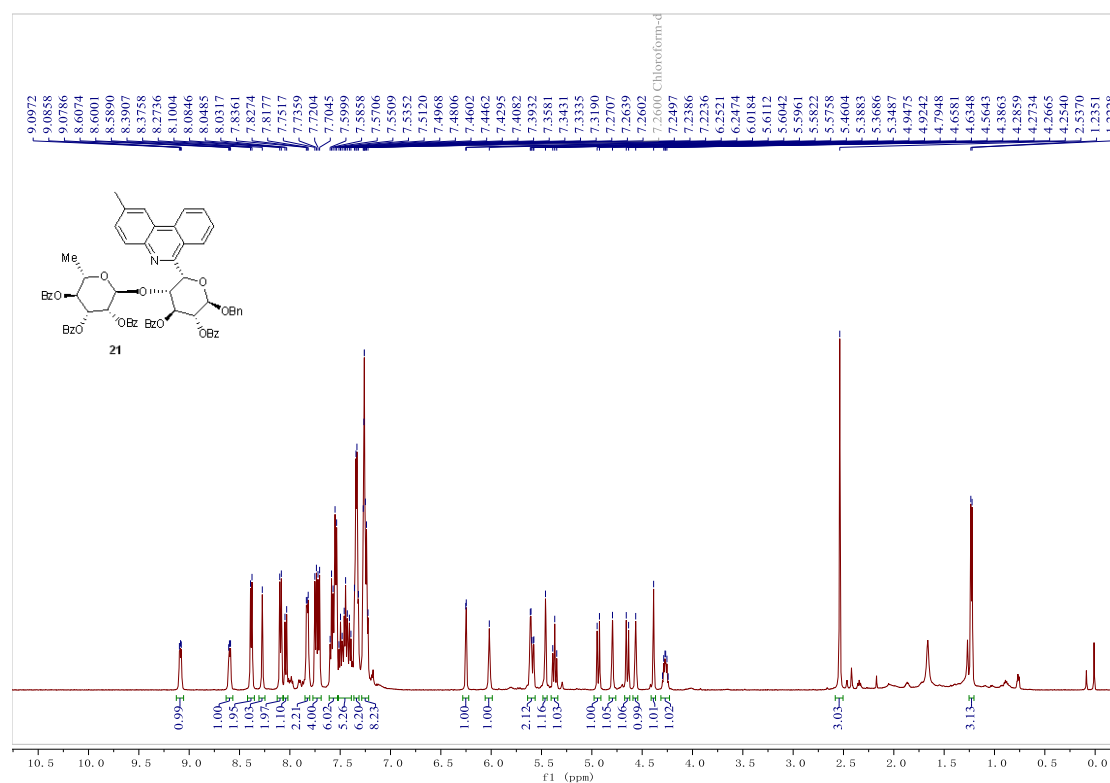
¹³C NMR spectrum for 19 (126 MHz, CDCl₃)



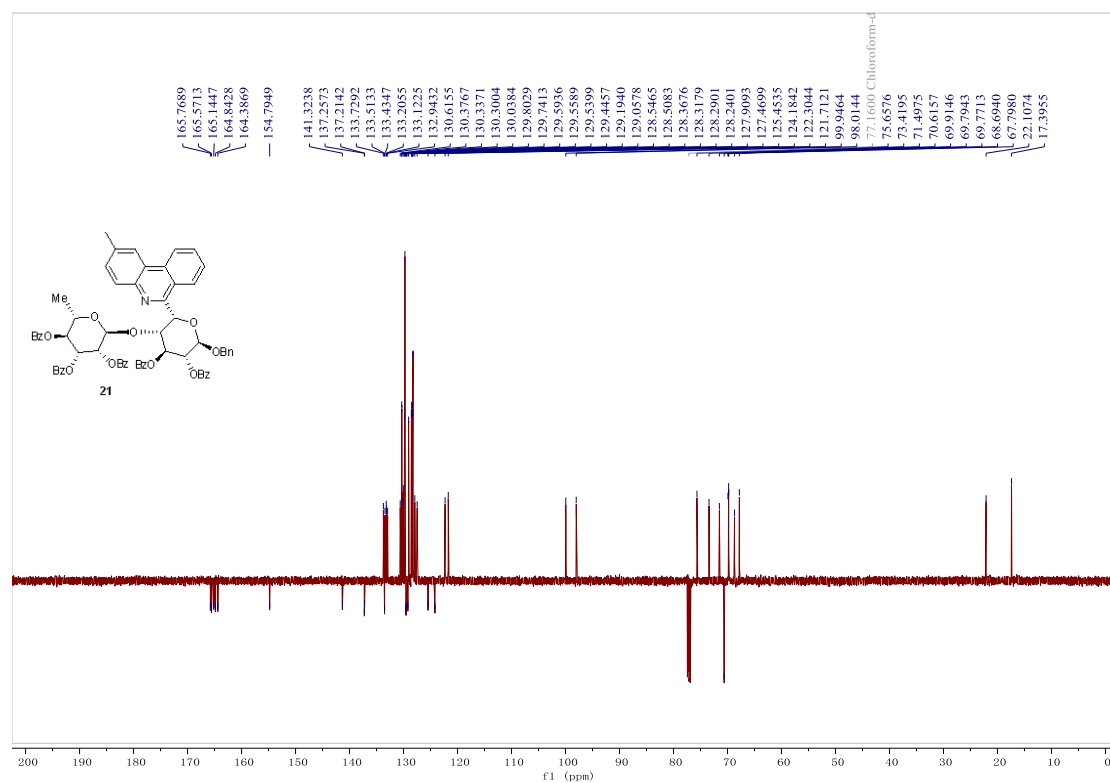
¹H NMR spectrum for 20 (500 MHz, CDCl₃)



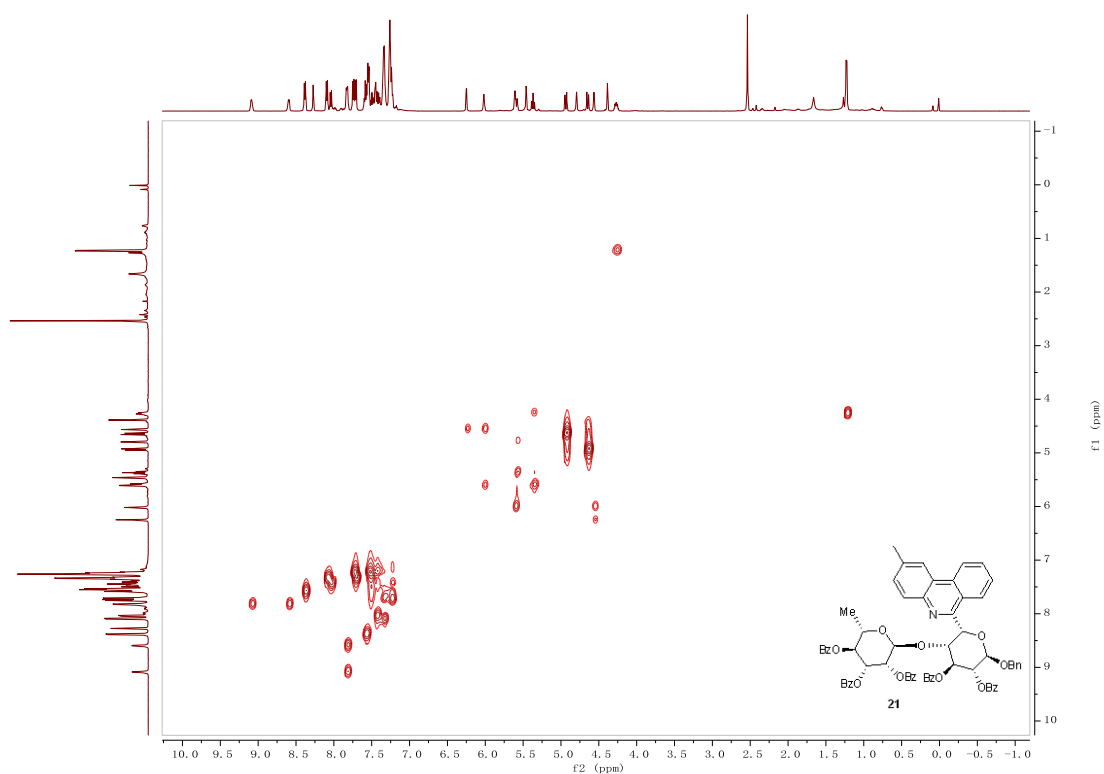
¹³C NMR spectrum for 20 (126 MHz, CDCl₃)



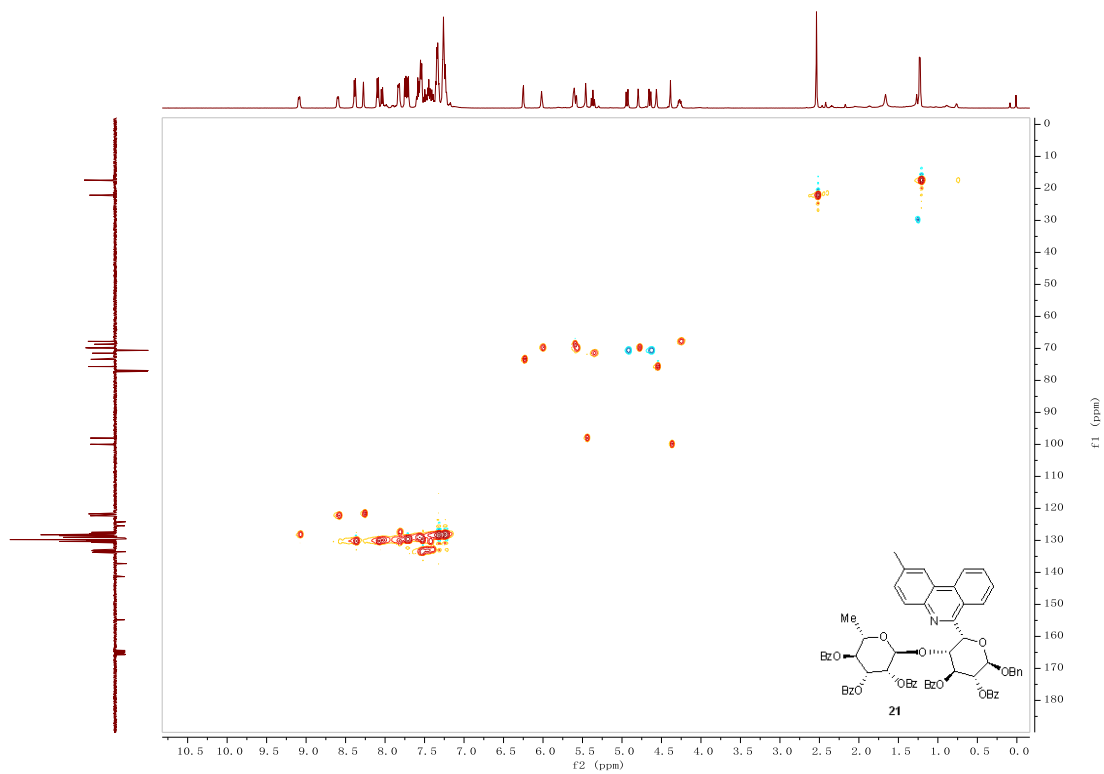
¹H NMR spectrum for **21** (500 MHz, CDCl₃)



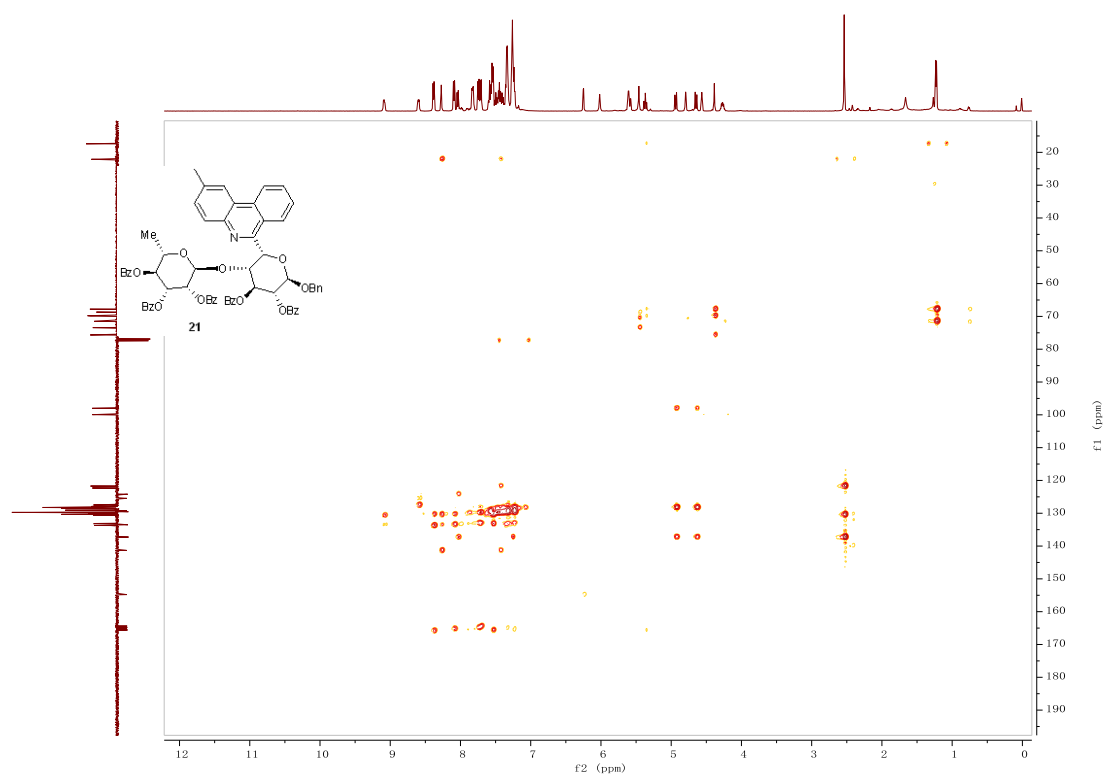
DEPT-Q spectrum for **21** (126 MHz, CDCl₃)



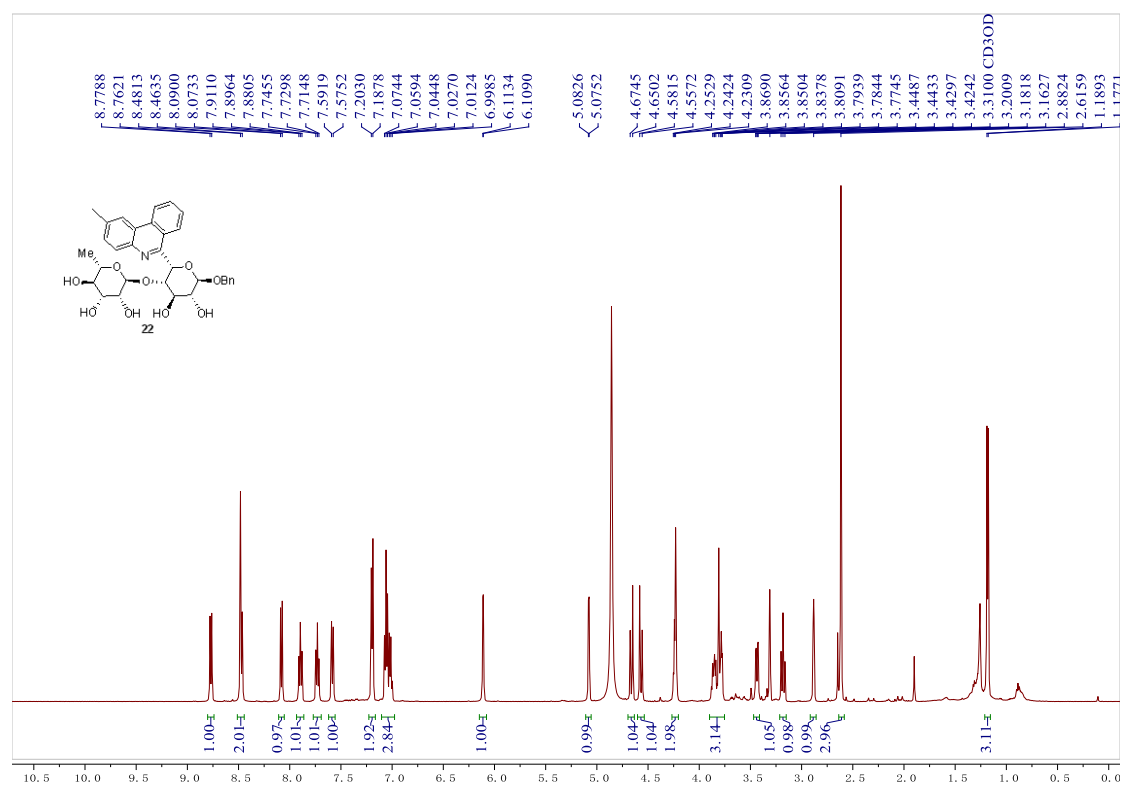
COSY spectrum for **21**



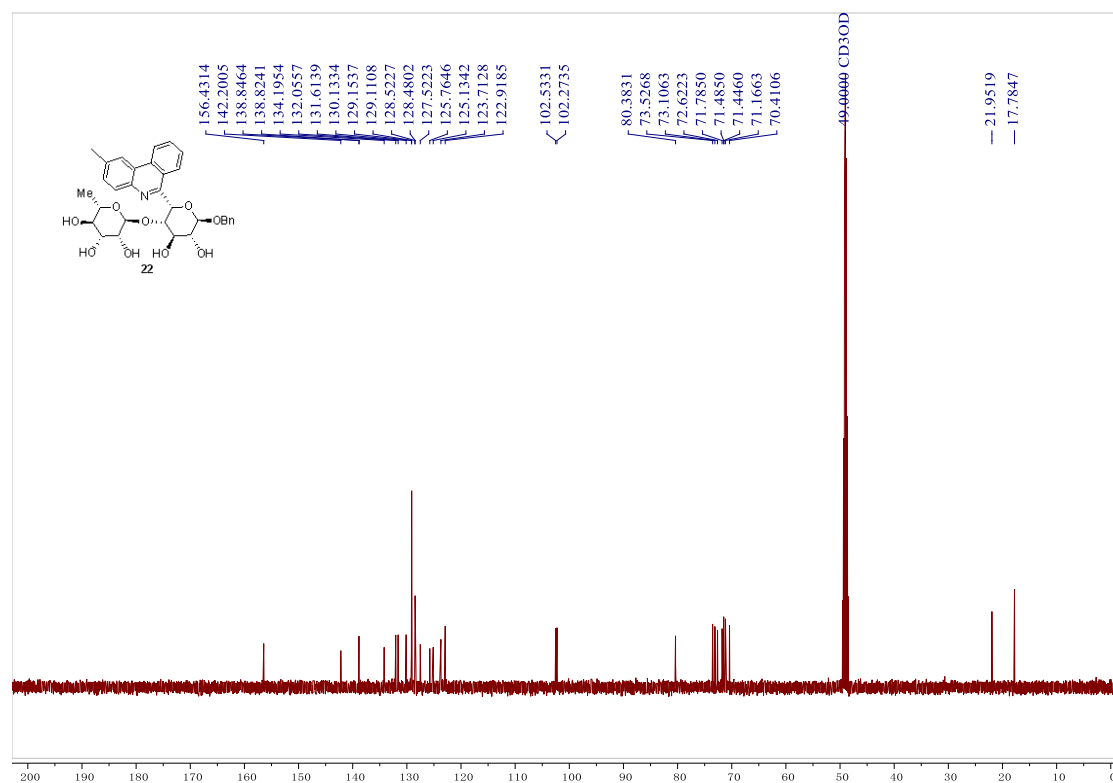
HSQC spectrum for **21**



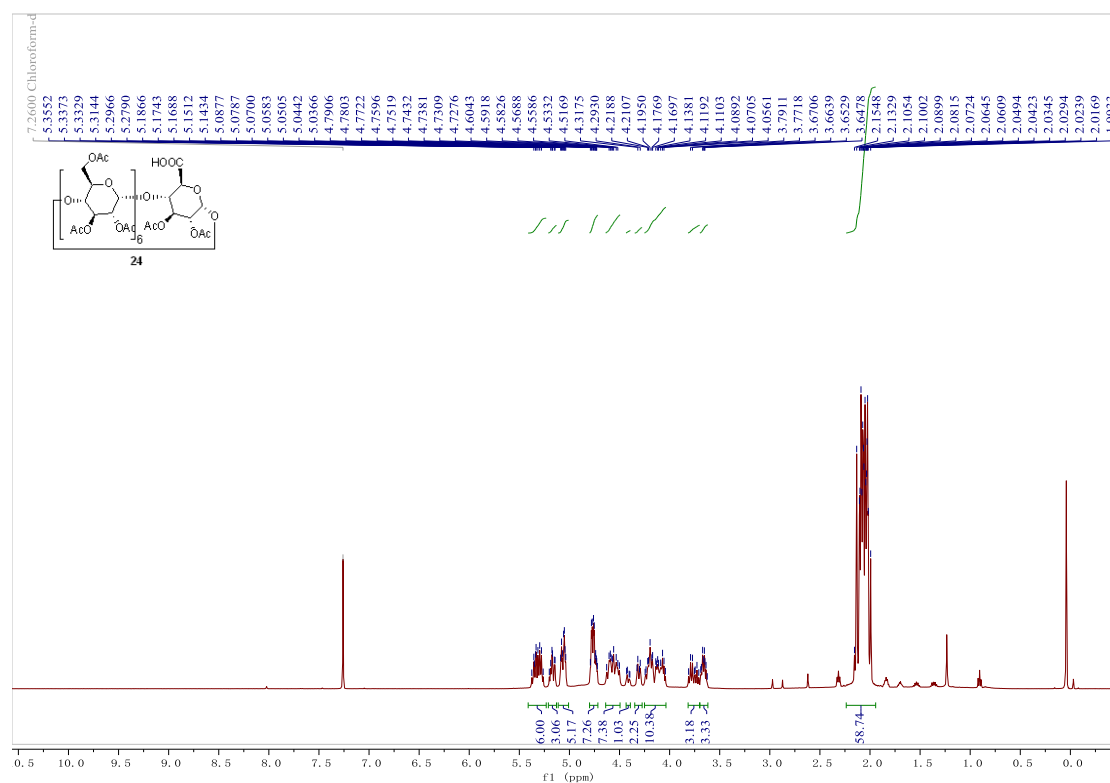
HMBC spectrum for **21**



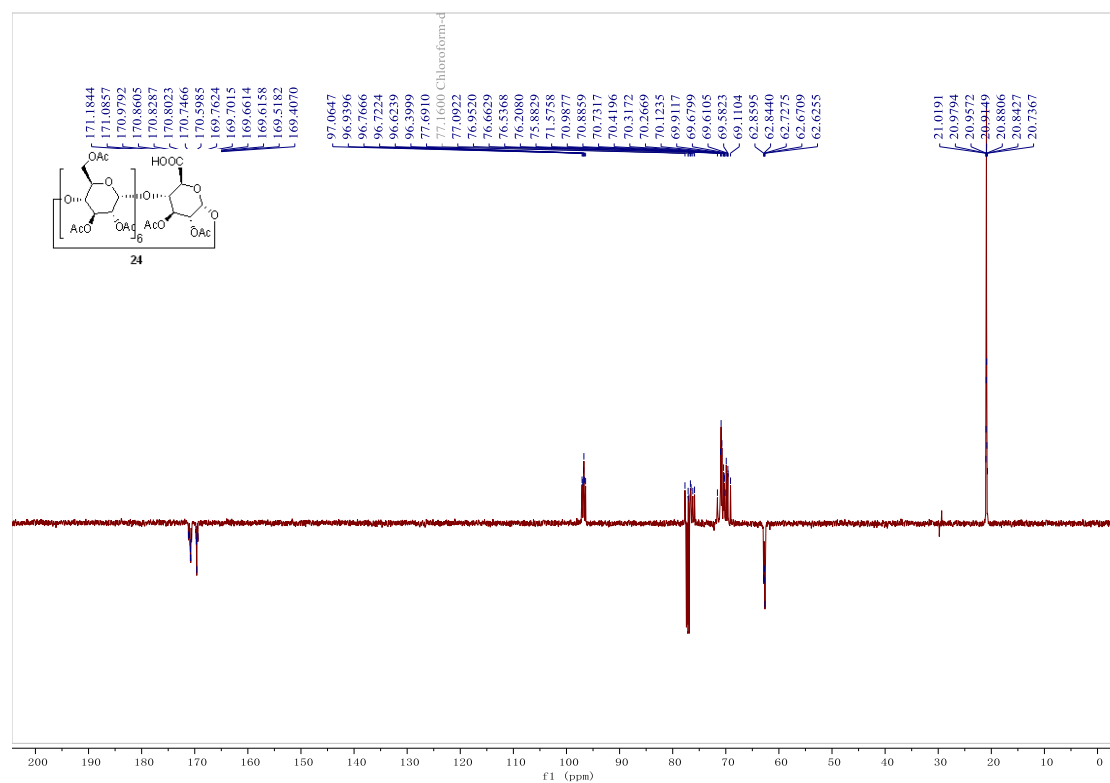
¹H NMR spectrum for 22 (500 MHz, CD₃OD)



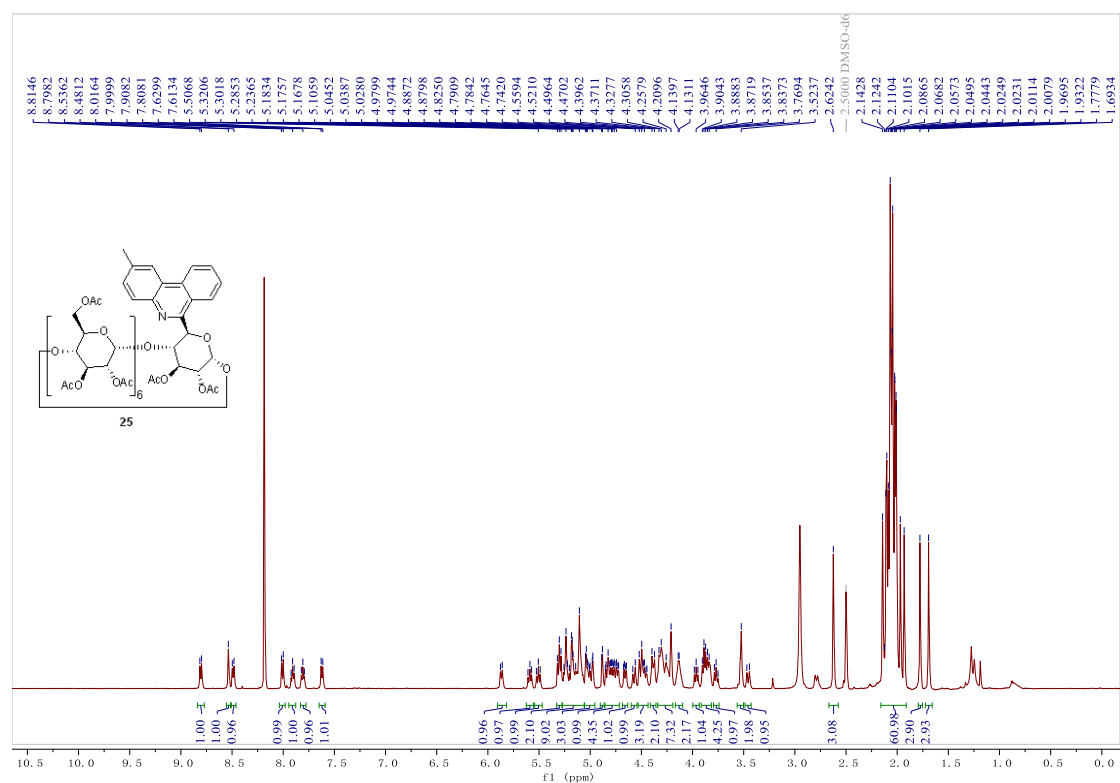
¹³C NMR spectrum for 22 (126 MHz, CD₃OD)



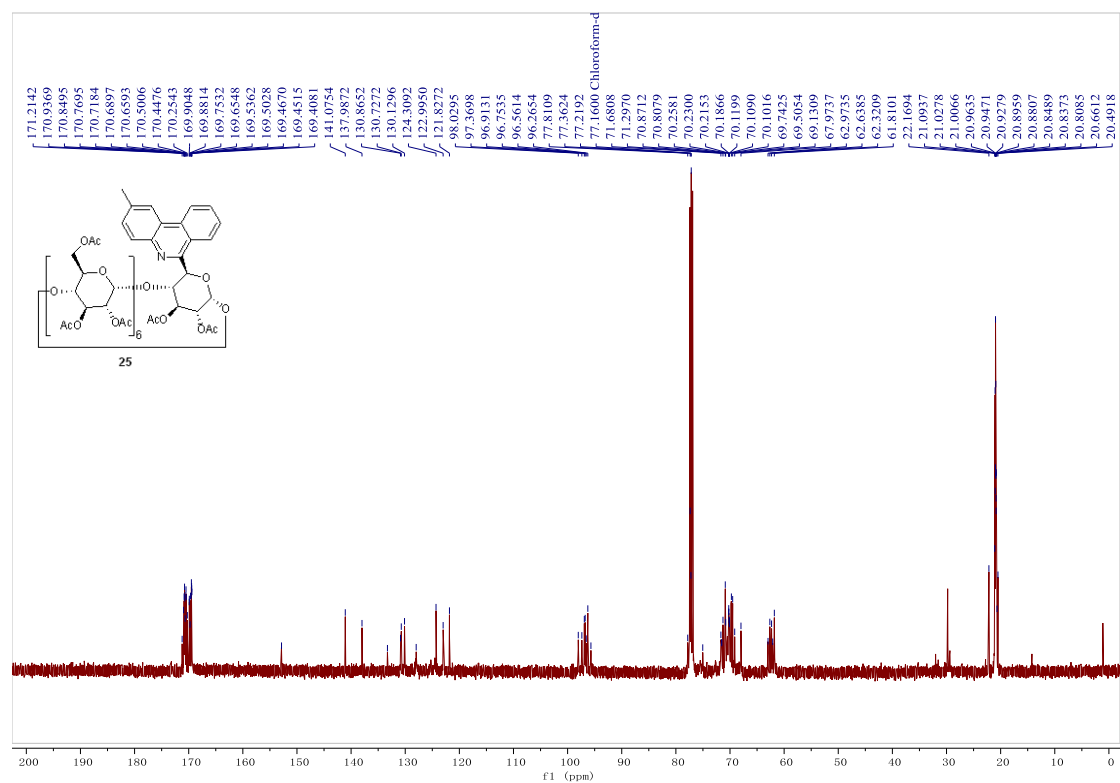
¹H NMR spectrum for **24** (500 MHz, CDCl₃)



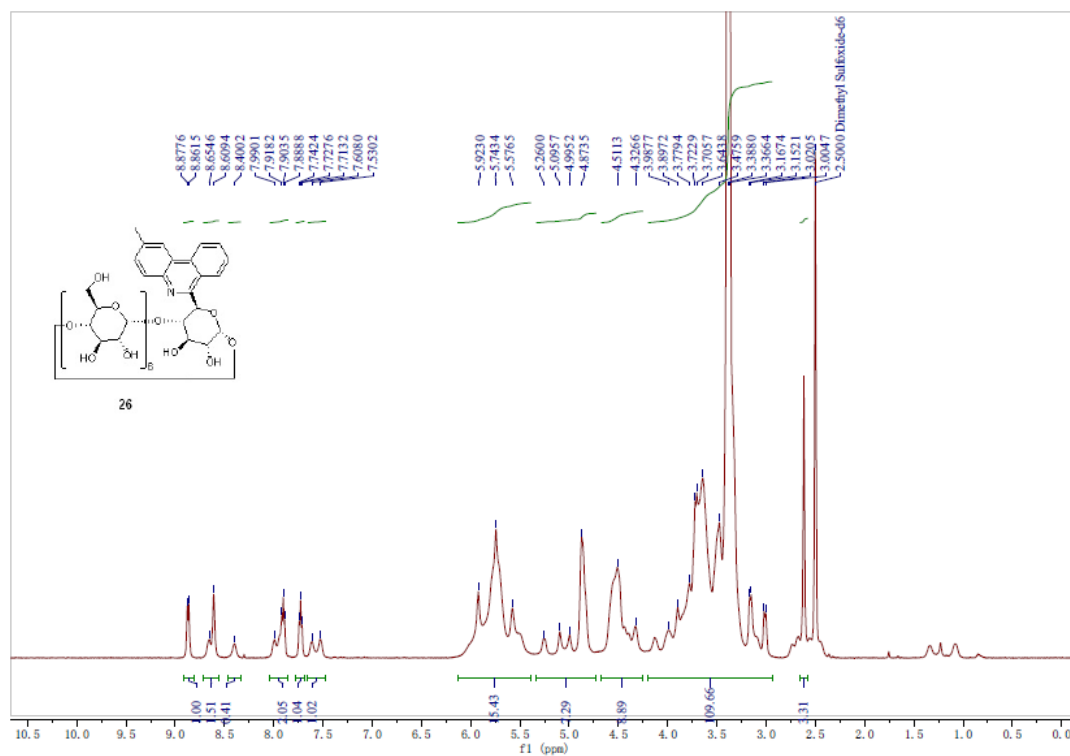
DEPT-Q spectrum for **24** (126 MHz, CDCl₃)



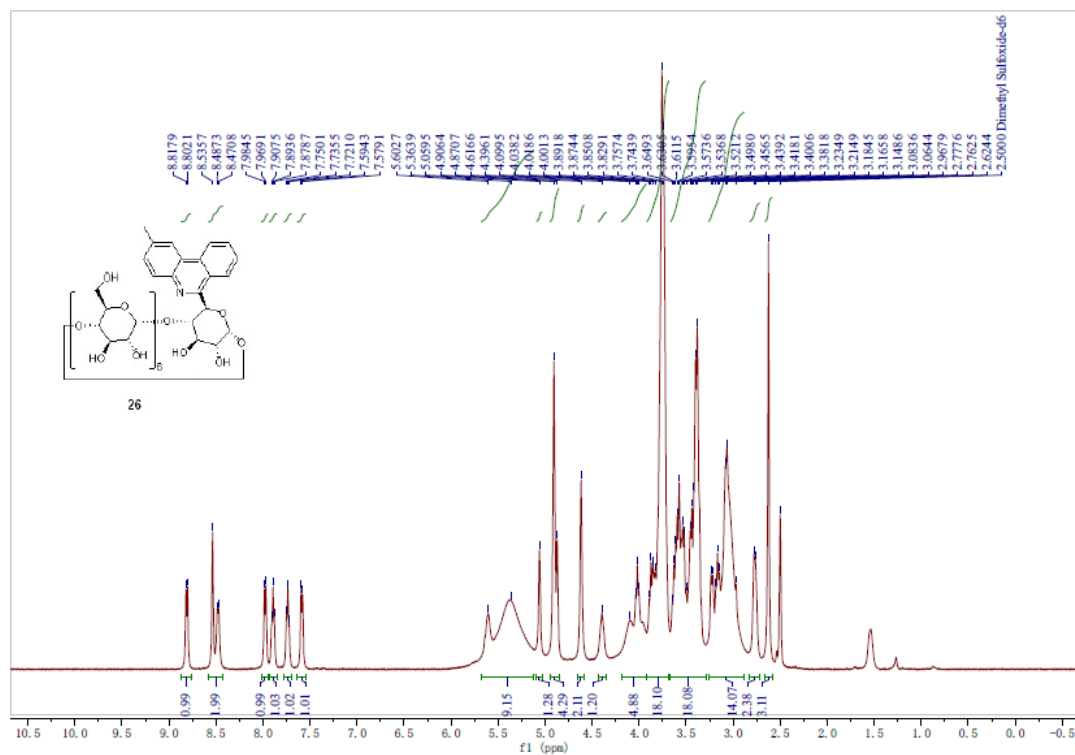
¹H NMR spectrum for 25 (500 MHz, DMSO-*d*₆, 70 °C)



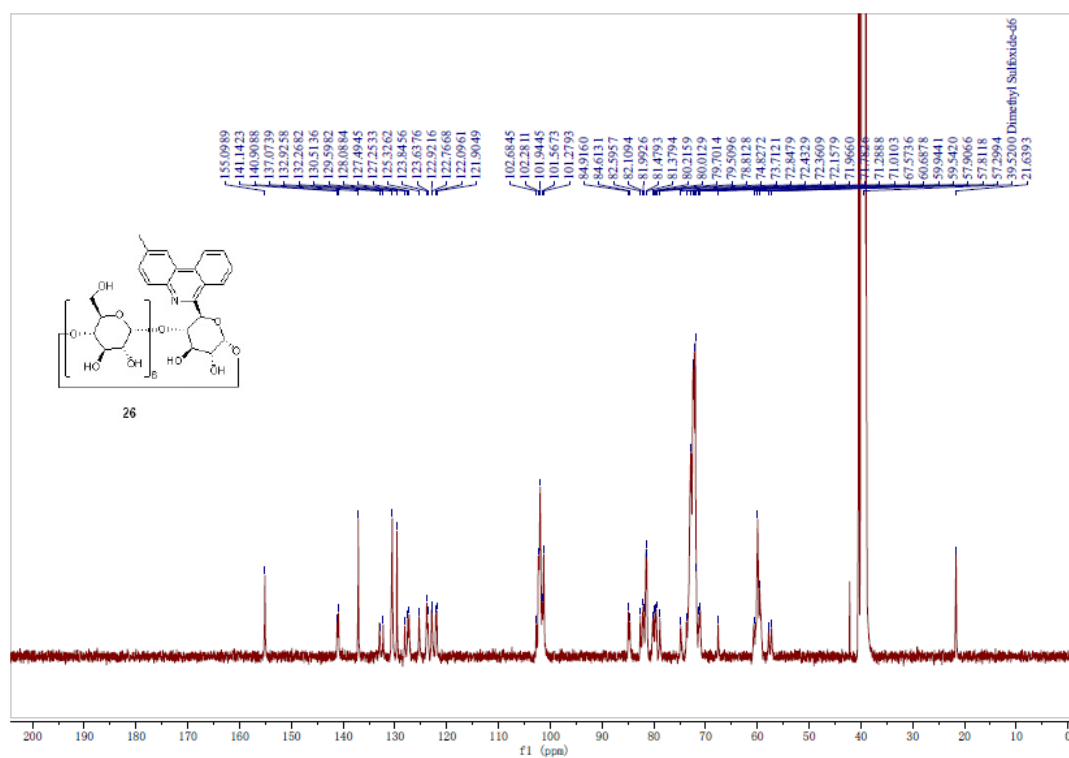
¹³C NMR spectrum for 25 (500 MHz, CDCl₃, 25 °C)



^1H NMR spectrum for **26** (500 MHz, $\text{DMSO}-d_6$, 25 °C)



^1H NMR spectrum for **26** (500 MHz, $\text{DMSO}-d_6$, 70 °C)



^{13}C NMR spectrum for **26** (500 MHz, $\text{DMSO}-d_6$, 25 °C)