Supplementary Data

Adaptable synthesis of *C*-lactosyl glycoclusters and their binding properties with galectin-3

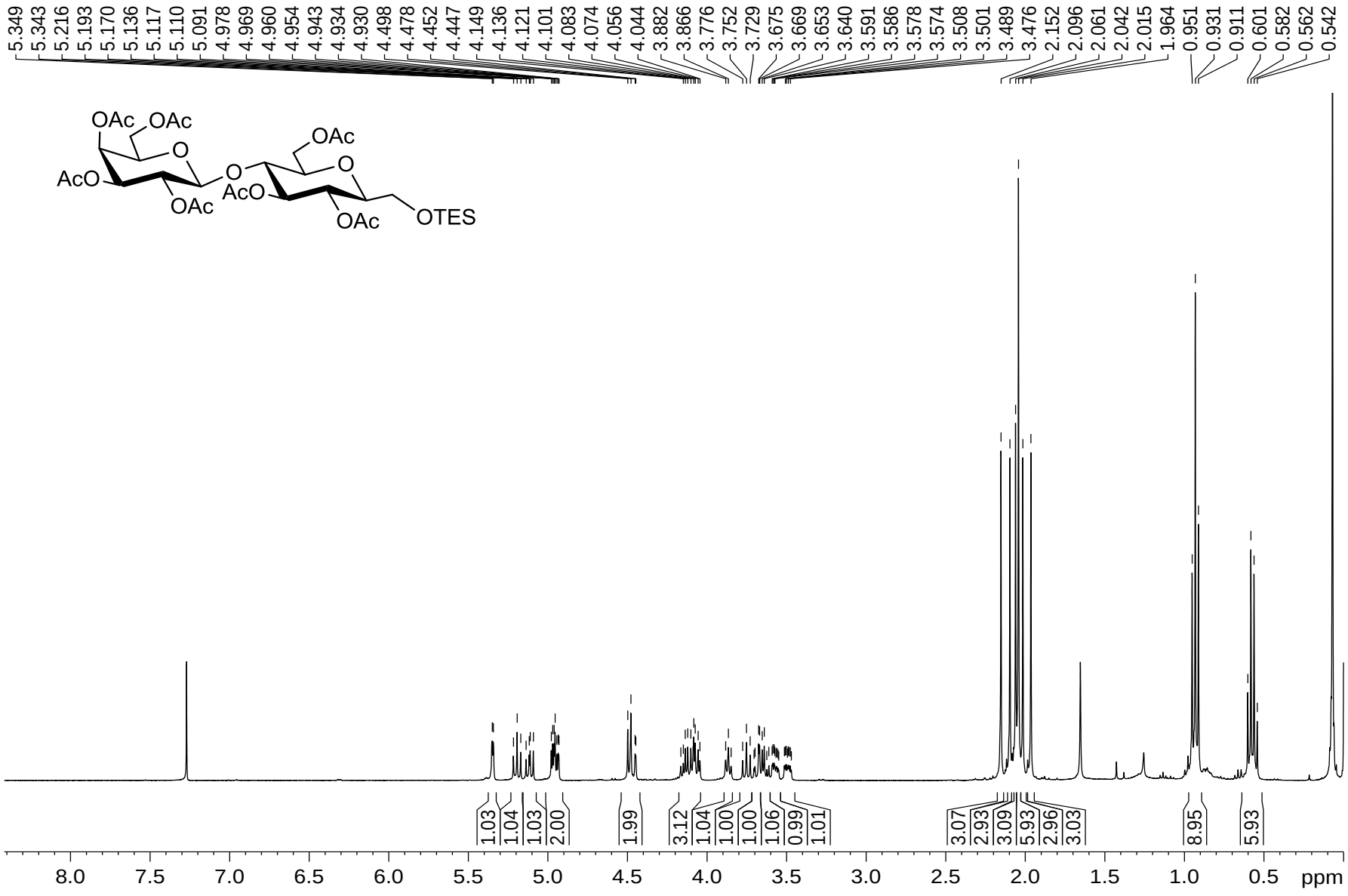
Wang Yao,[a] Meng-jie Xia[a], Xiang-bao Meng,[a] Qing Li*[a] and Zhong-jun Li*[a]

[a.] The State Key Laboratory of Natural and Biomimetic Drugs, School of Pharmaceutical Science, Peking University, Beijing 100191, P R China.

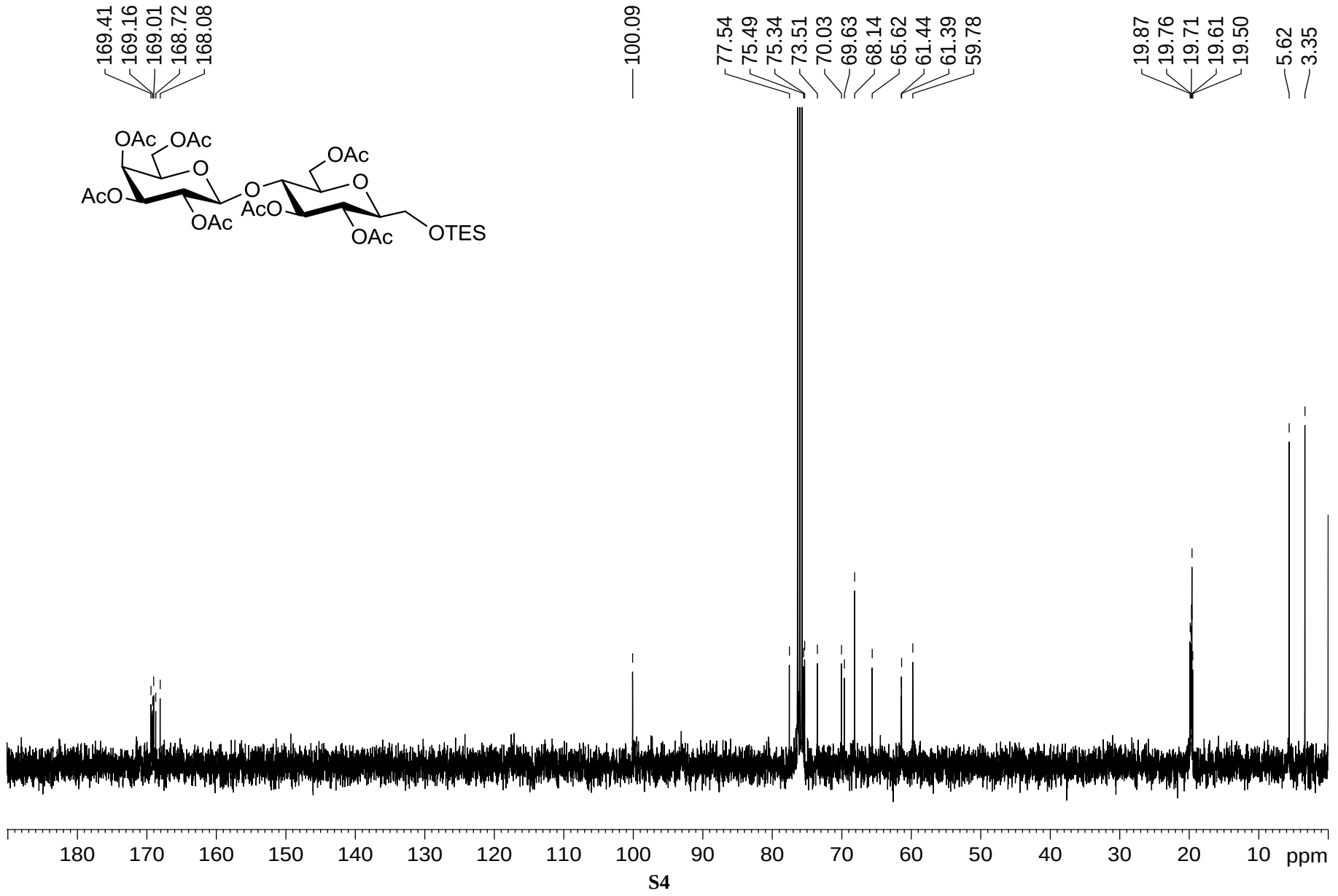
Content

Part I ^{1}H, ^{13}C, Dept 135 and 2D NMR spectrum

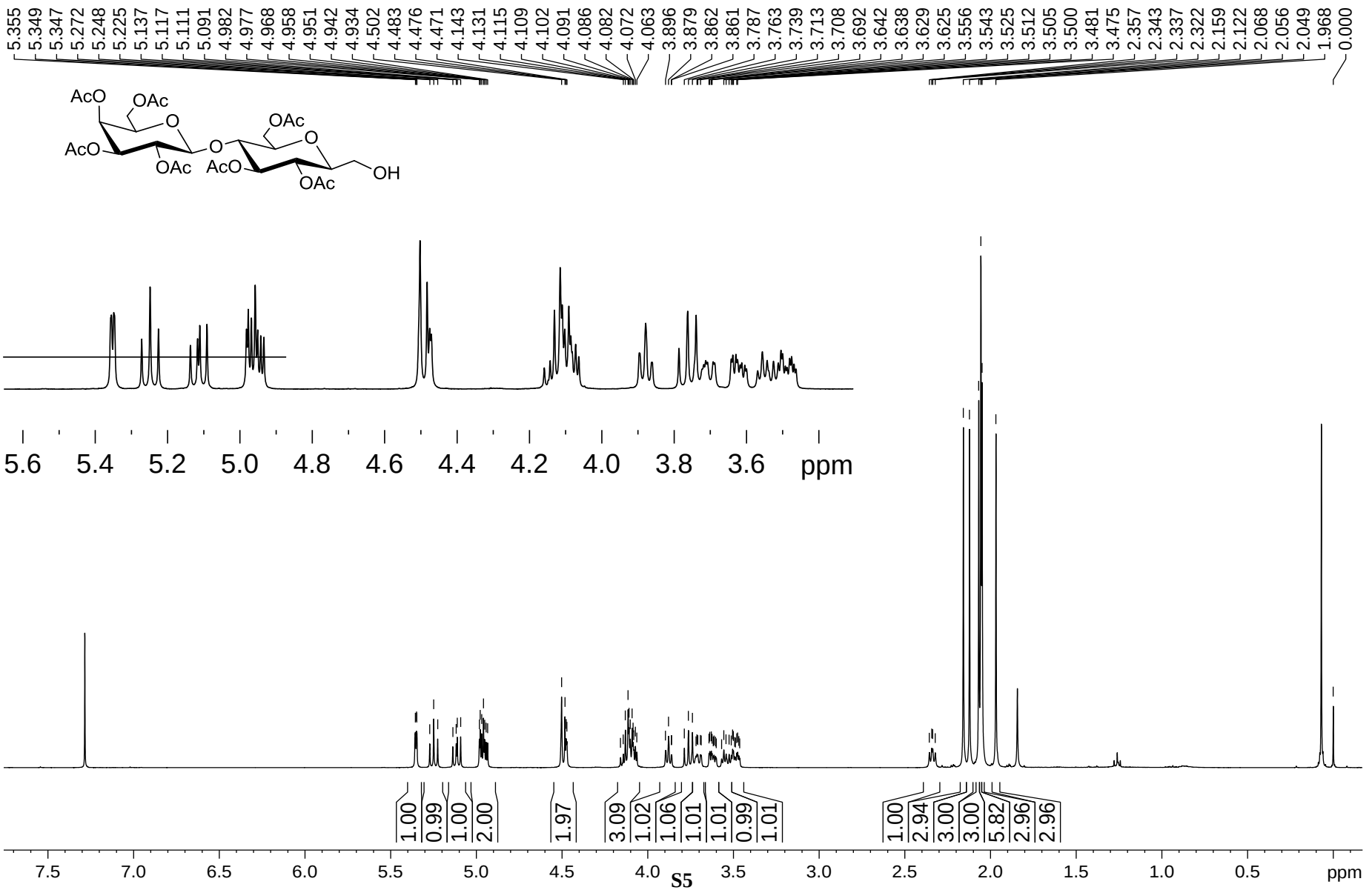
^{1}H NMR (400MHz, CDCl3) spectrum of **2**



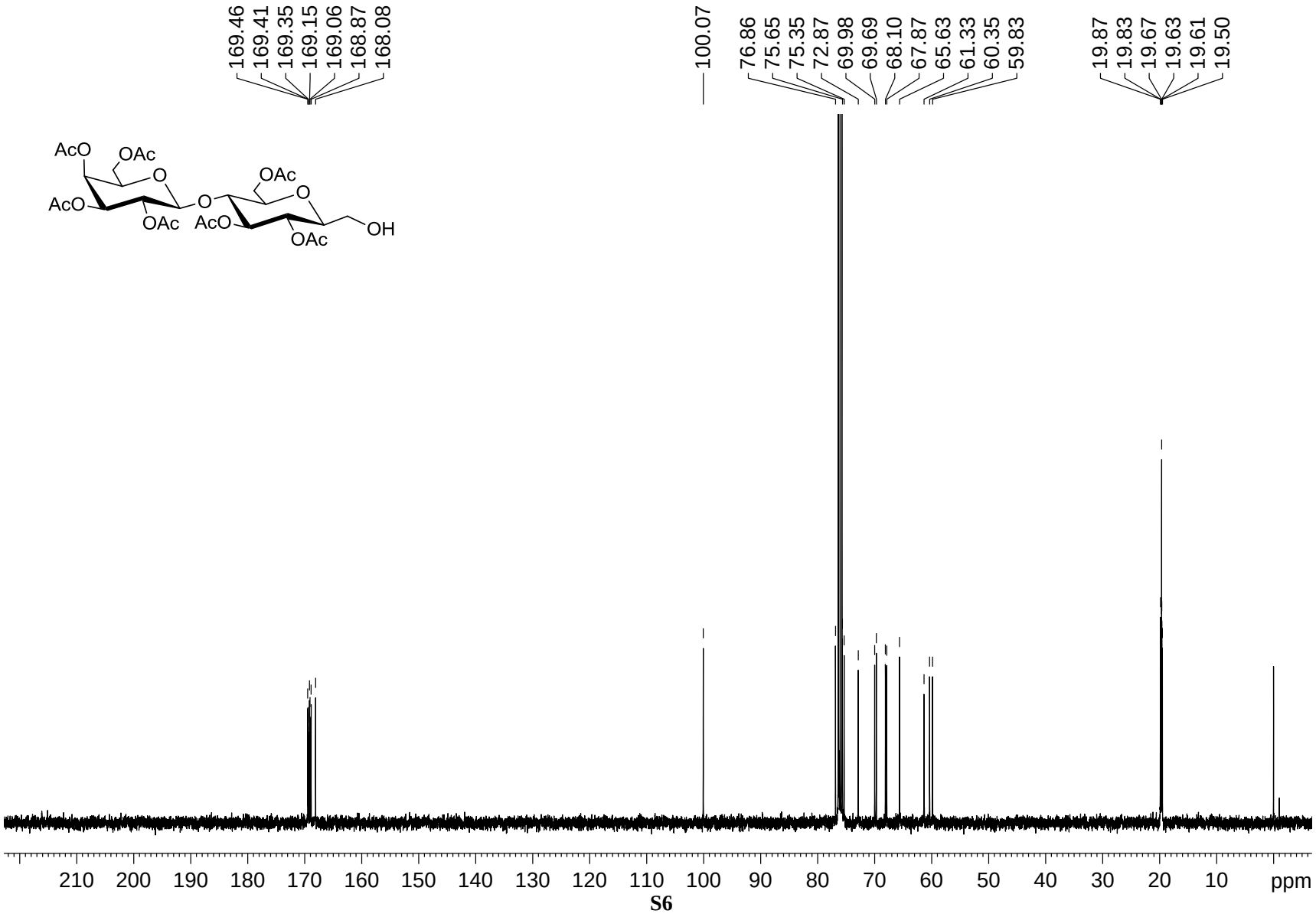
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **2**



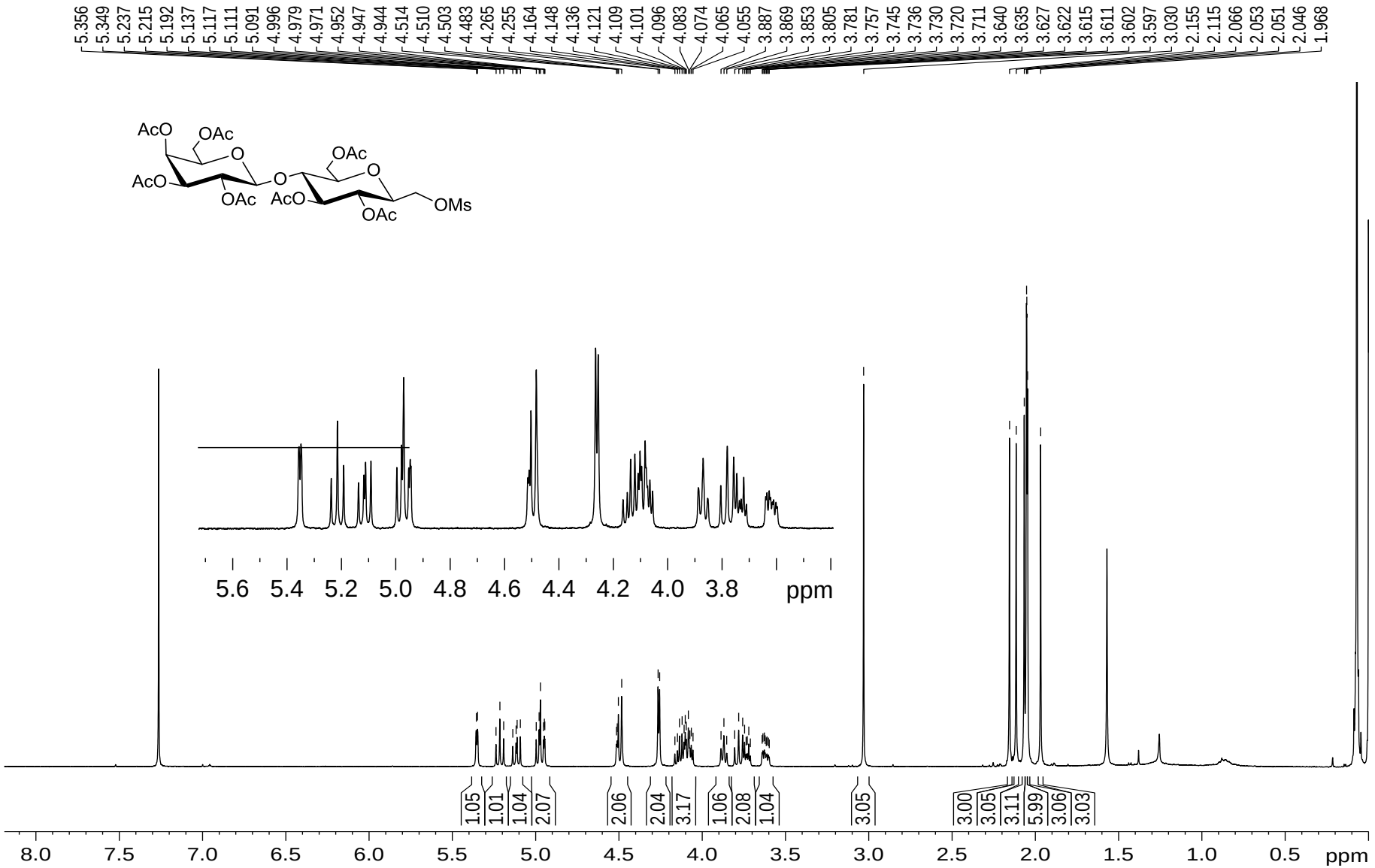
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **3**



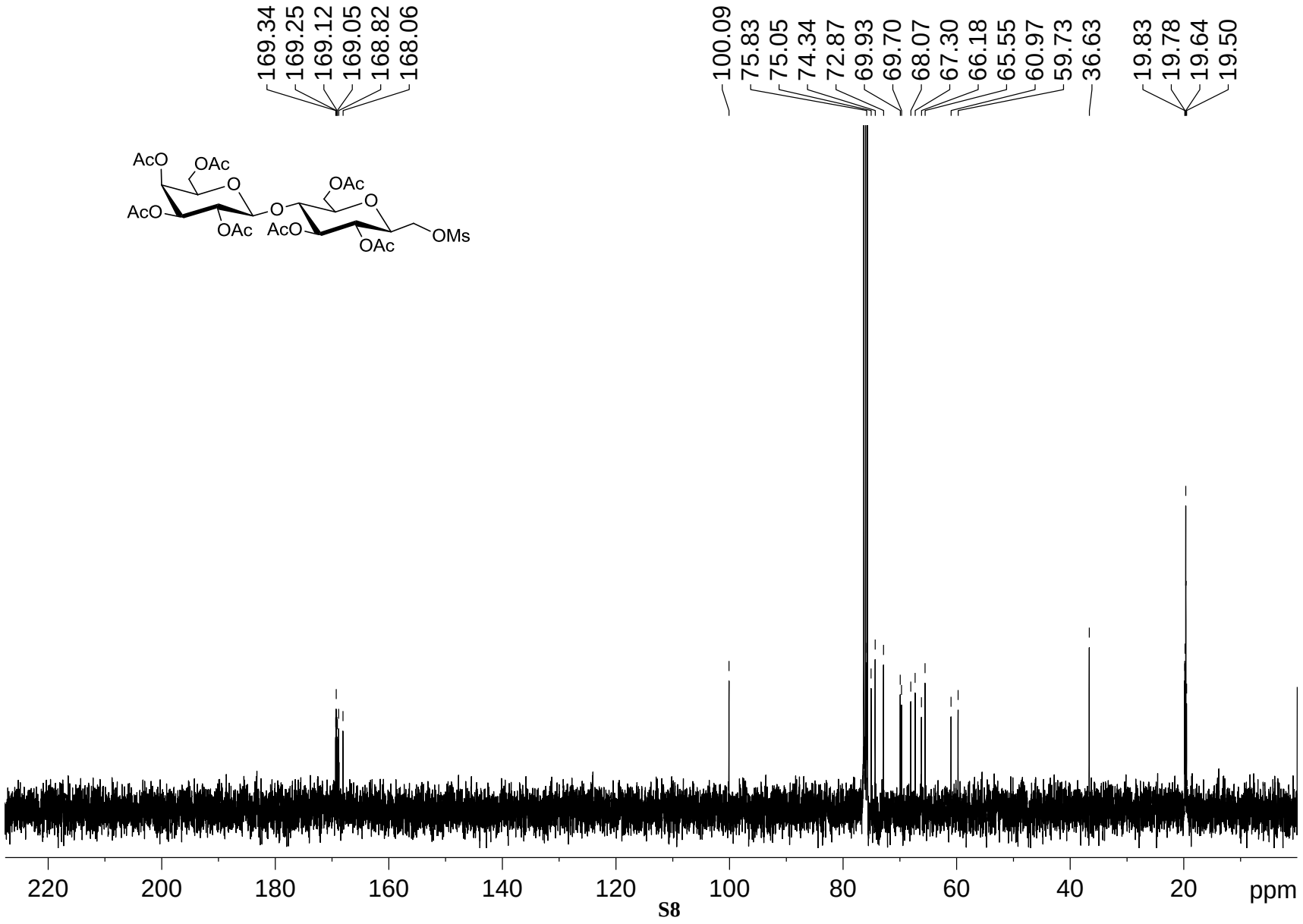
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **3**



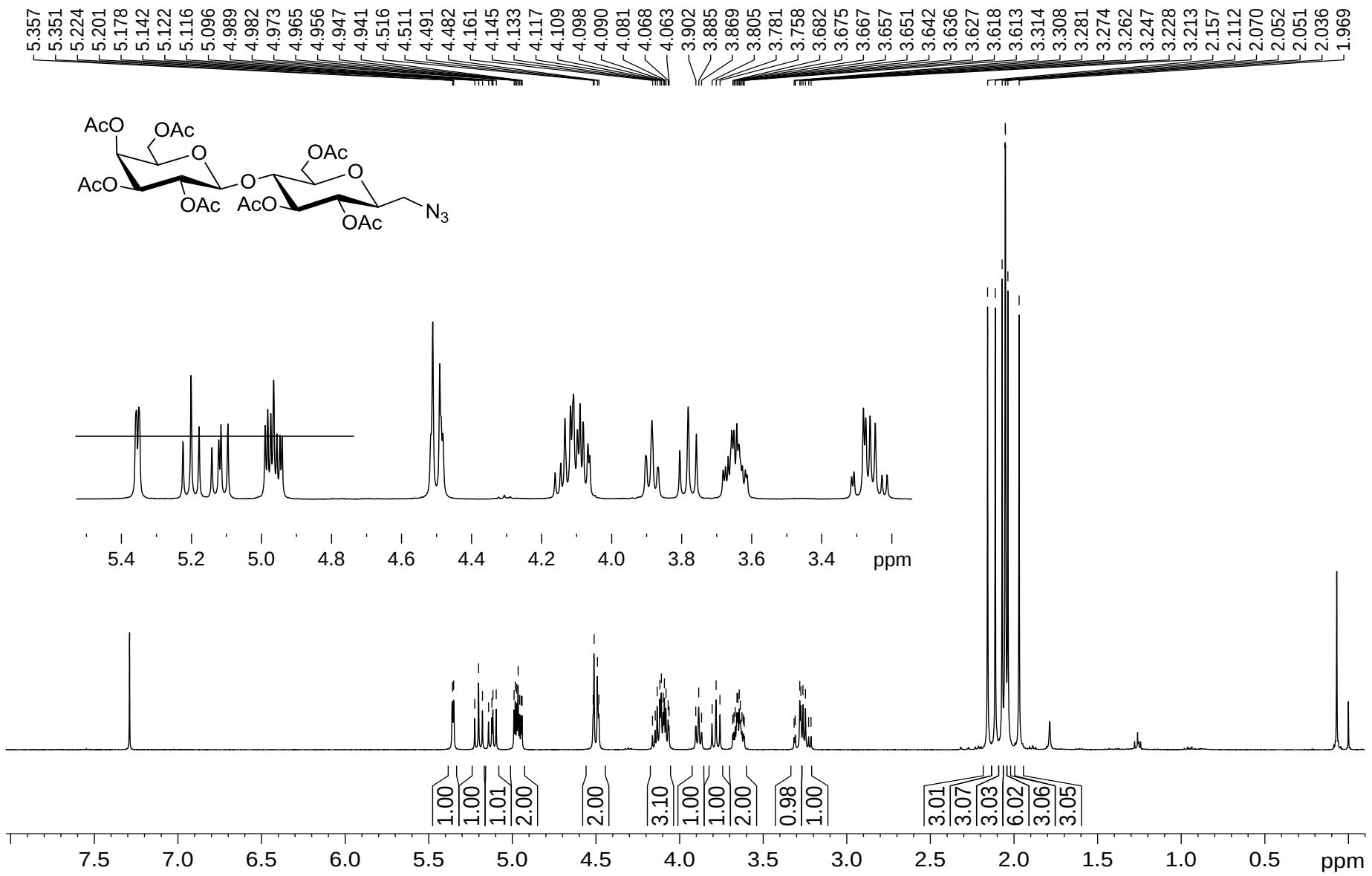
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **4**



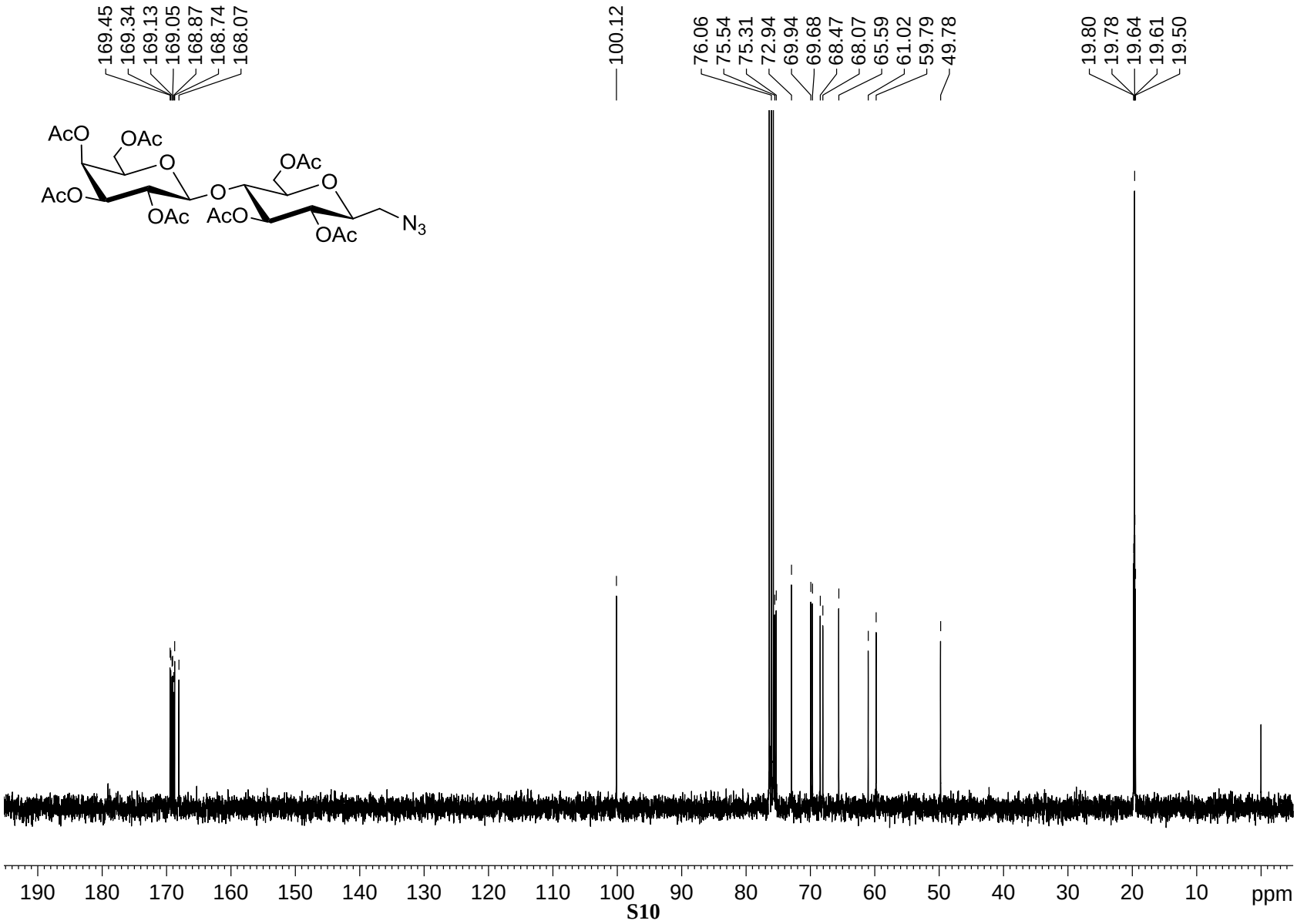
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **4**



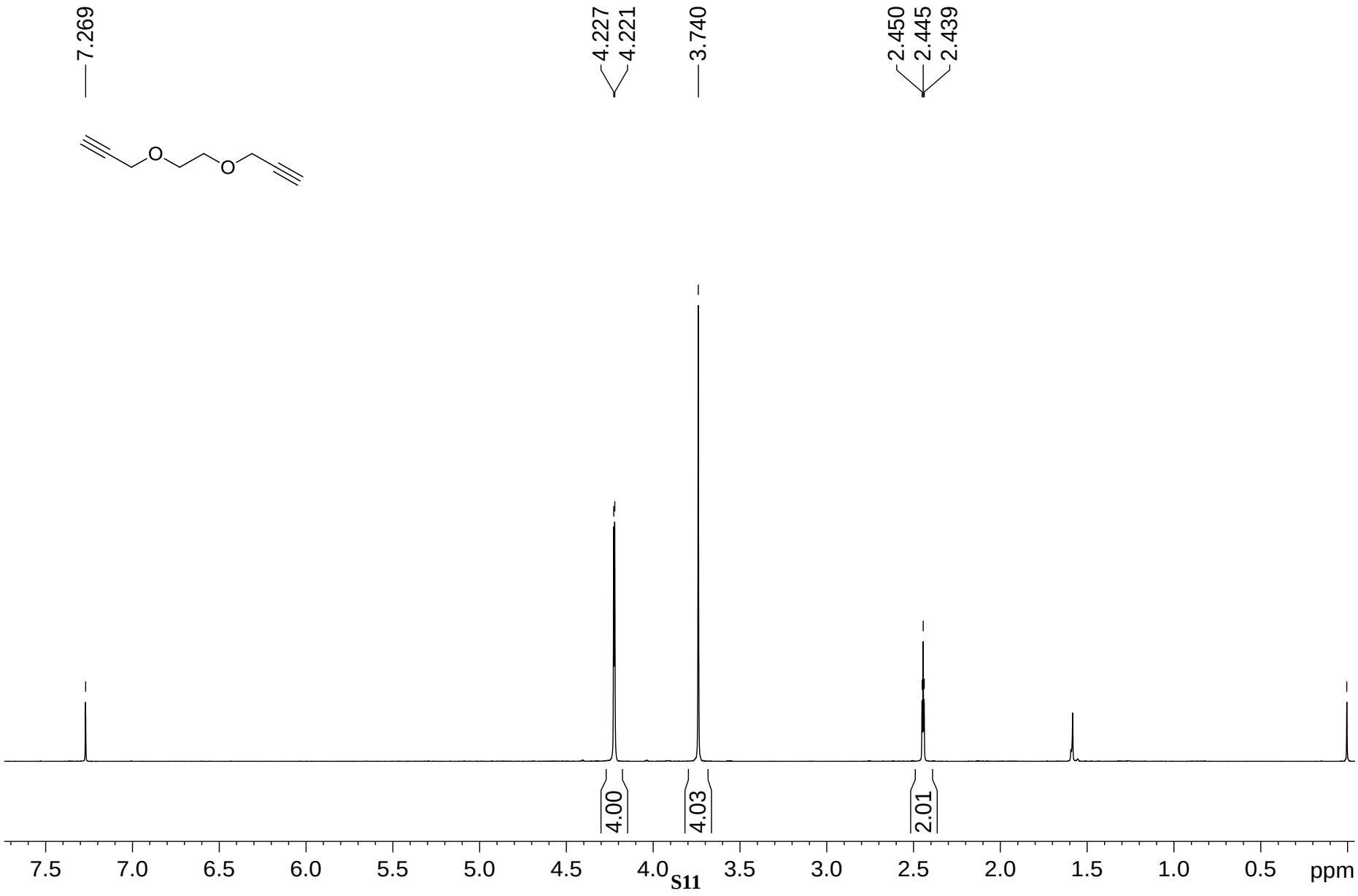
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **5**



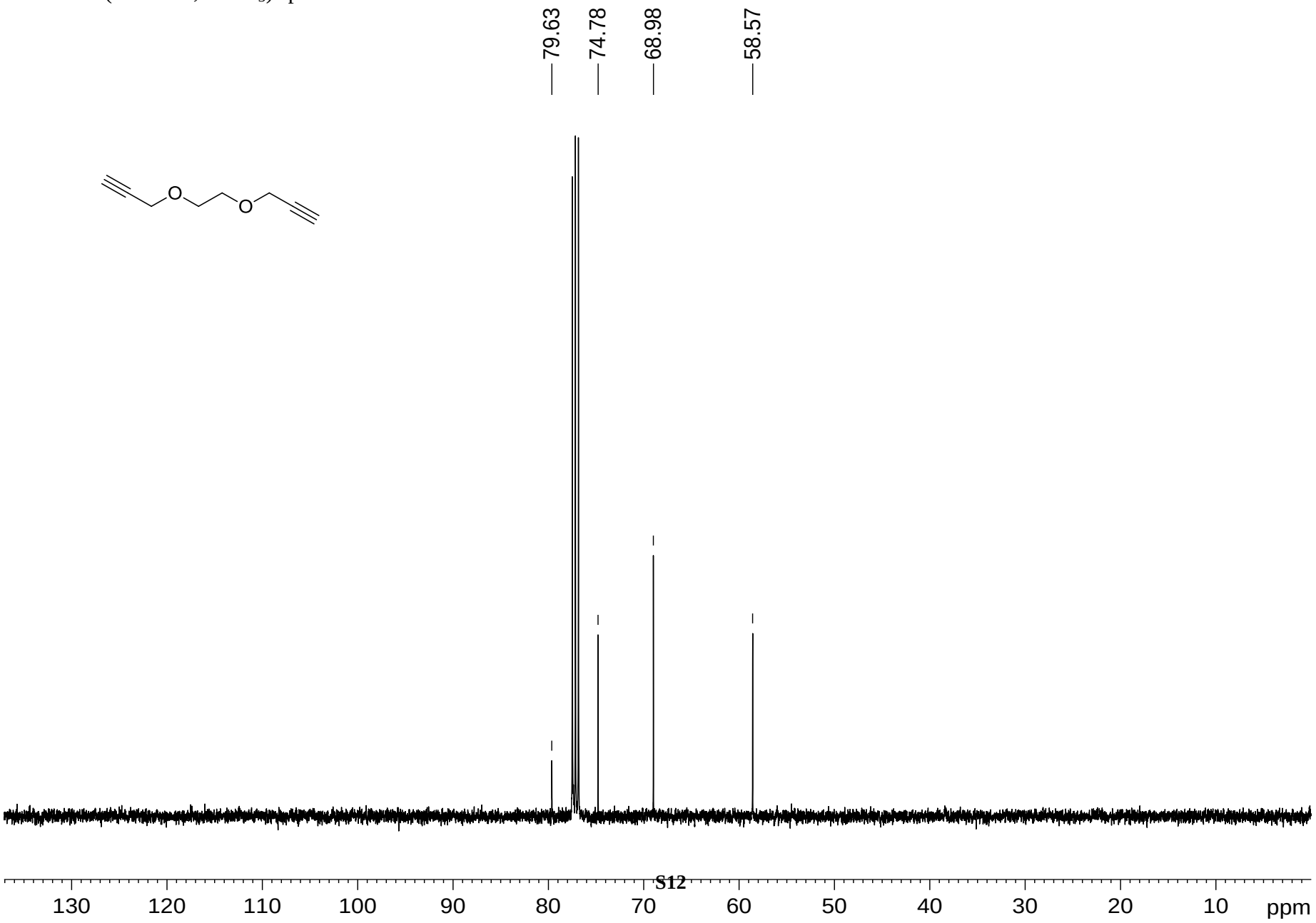
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **5**



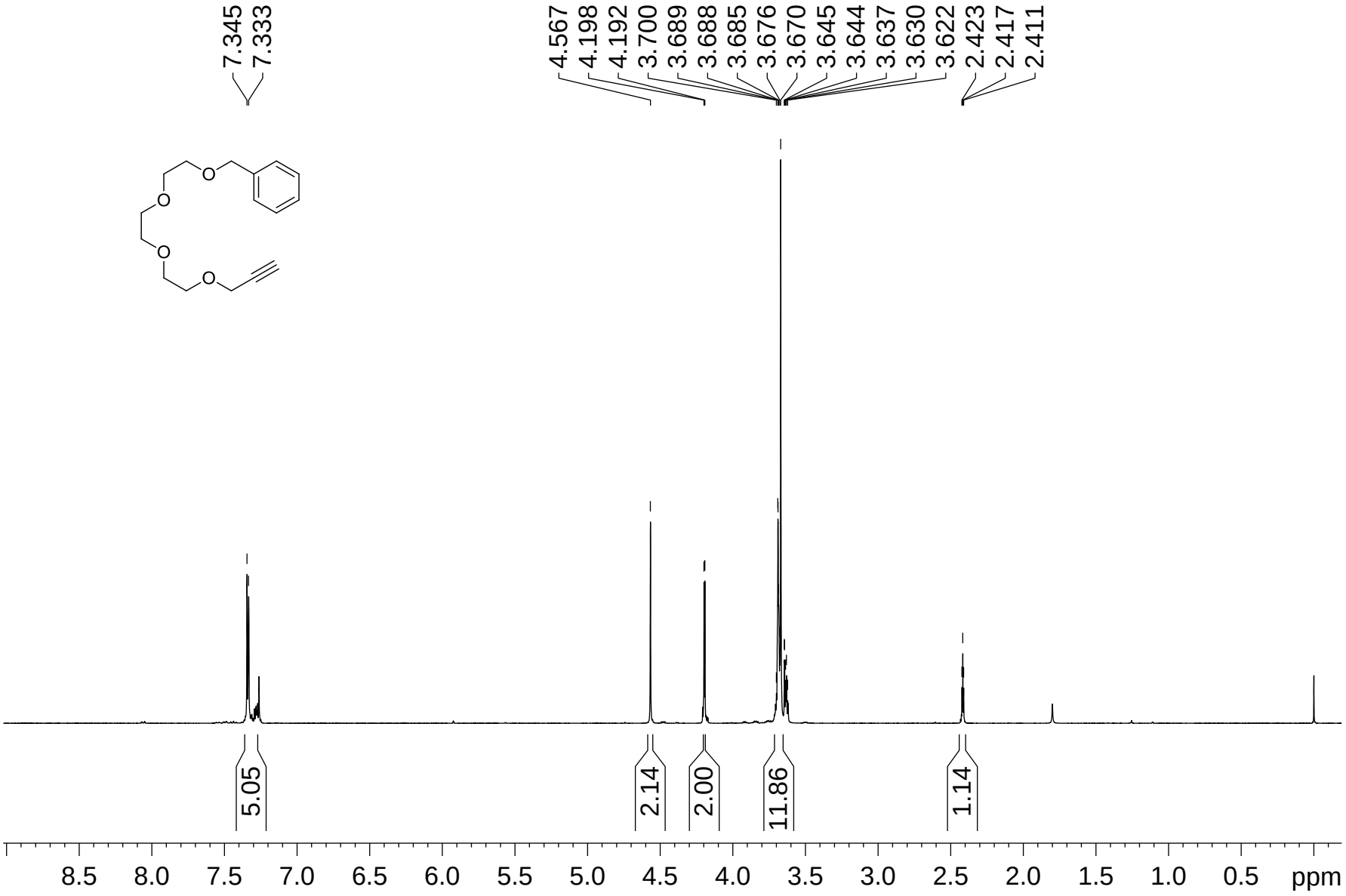
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **6a**



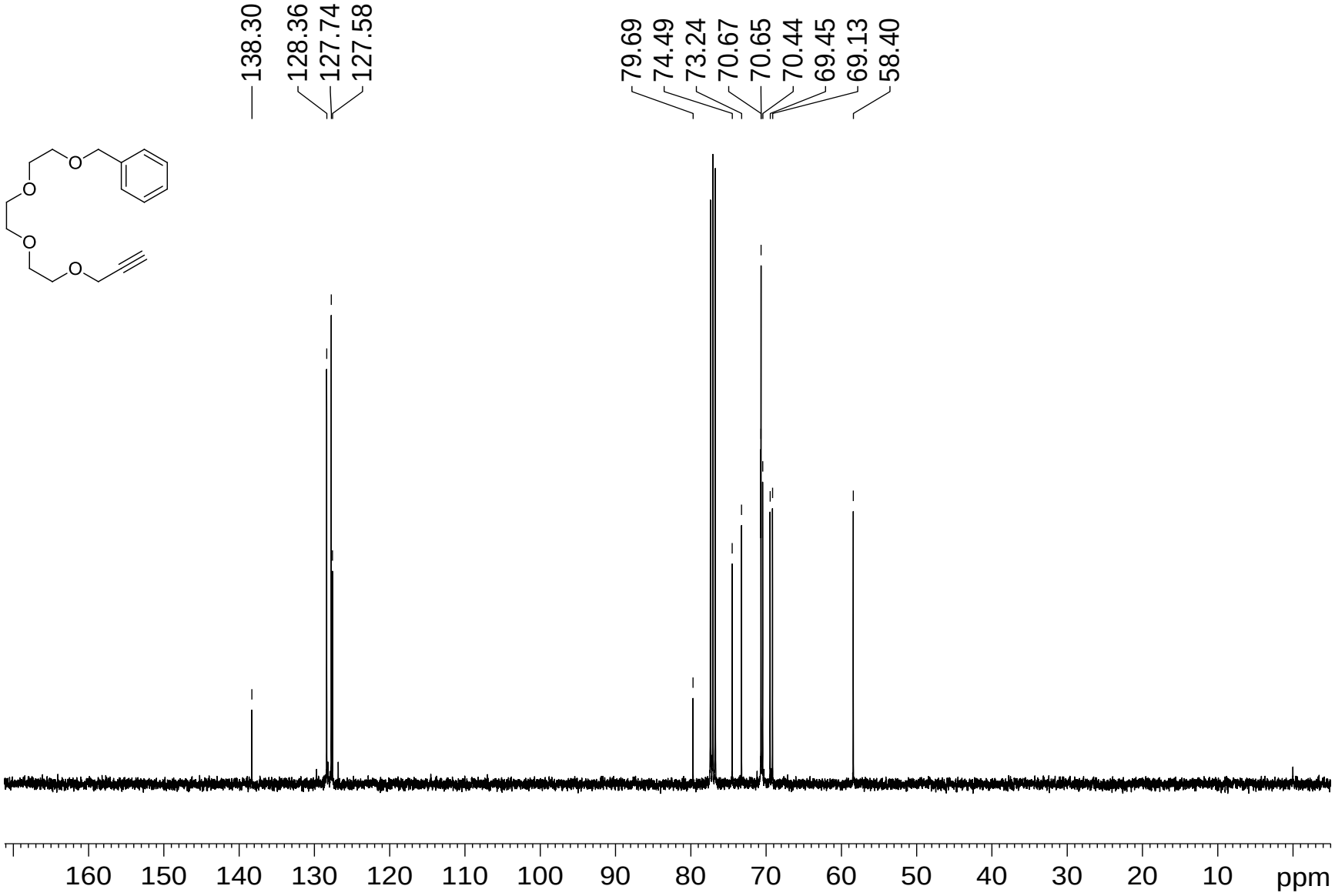
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **6a**



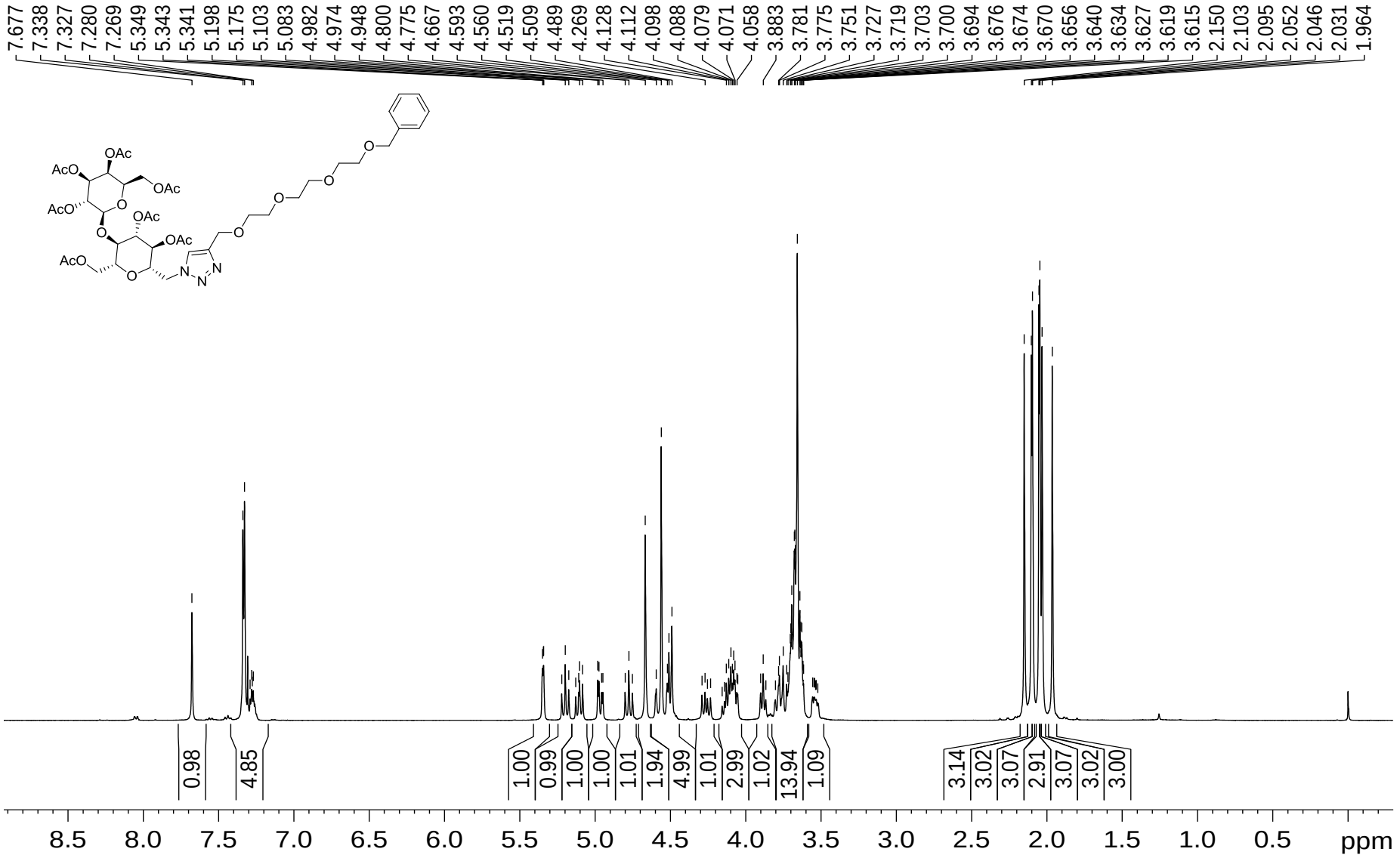
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **6c**



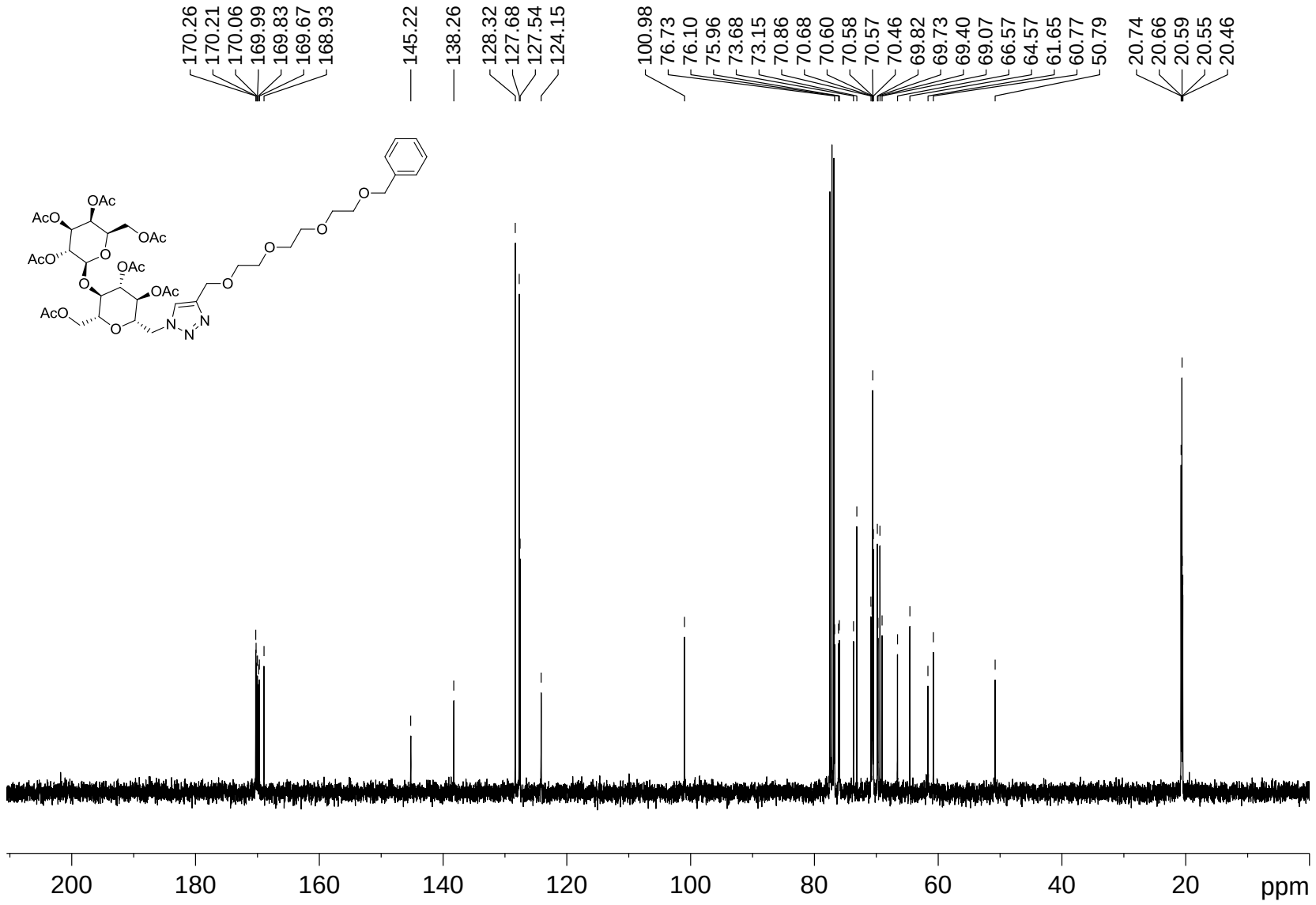
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **6c**



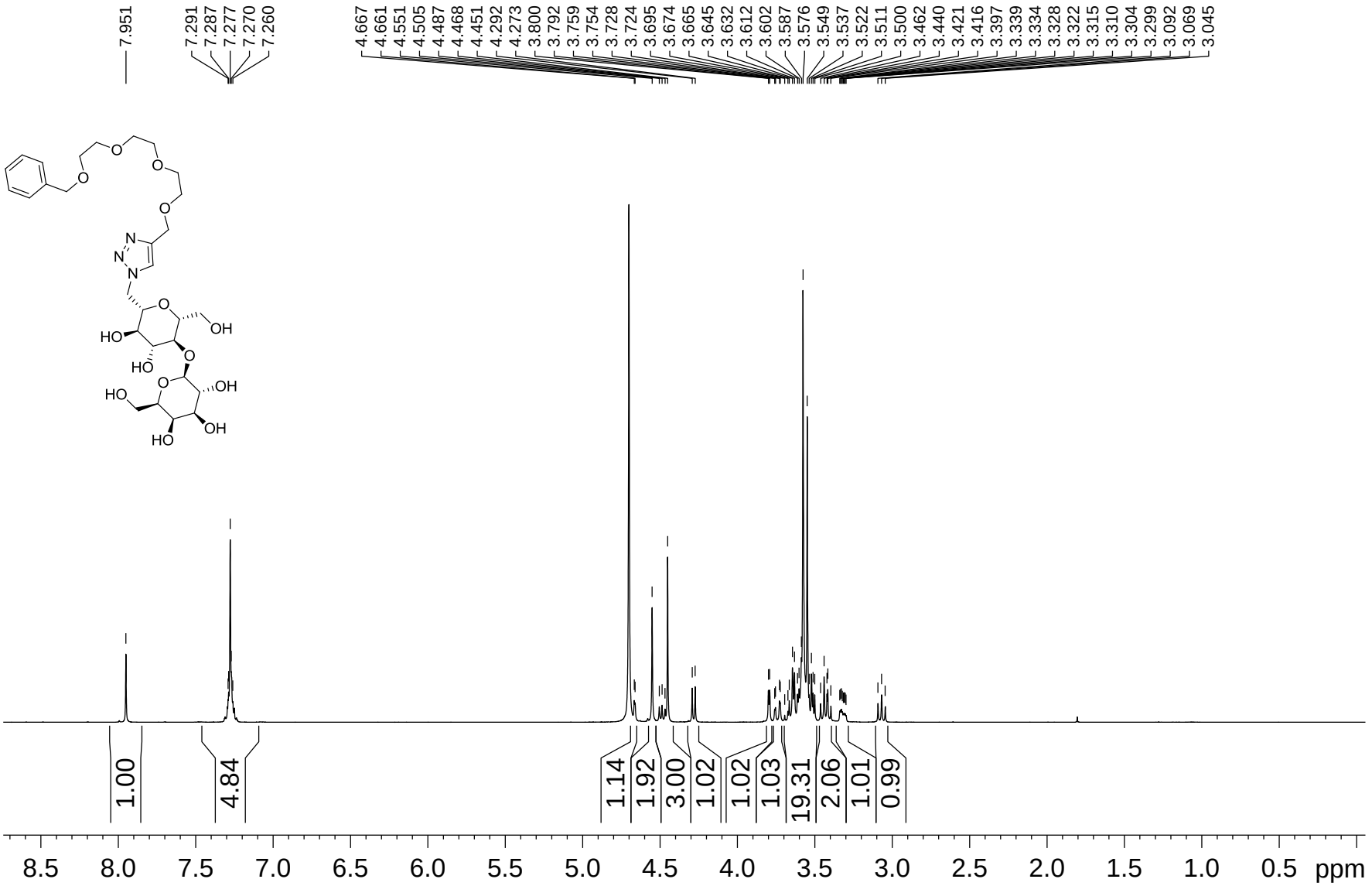
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7c**



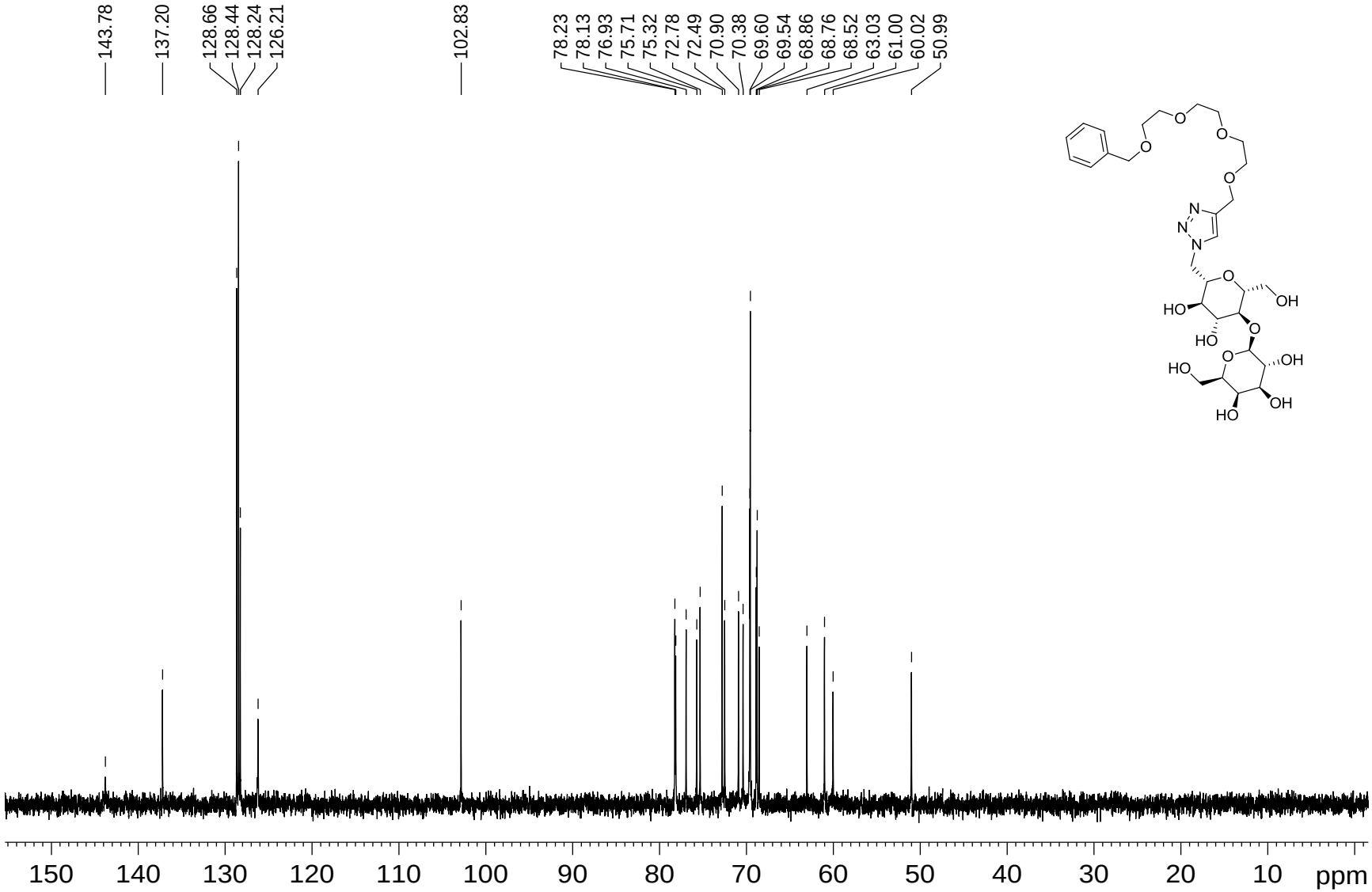
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7c**



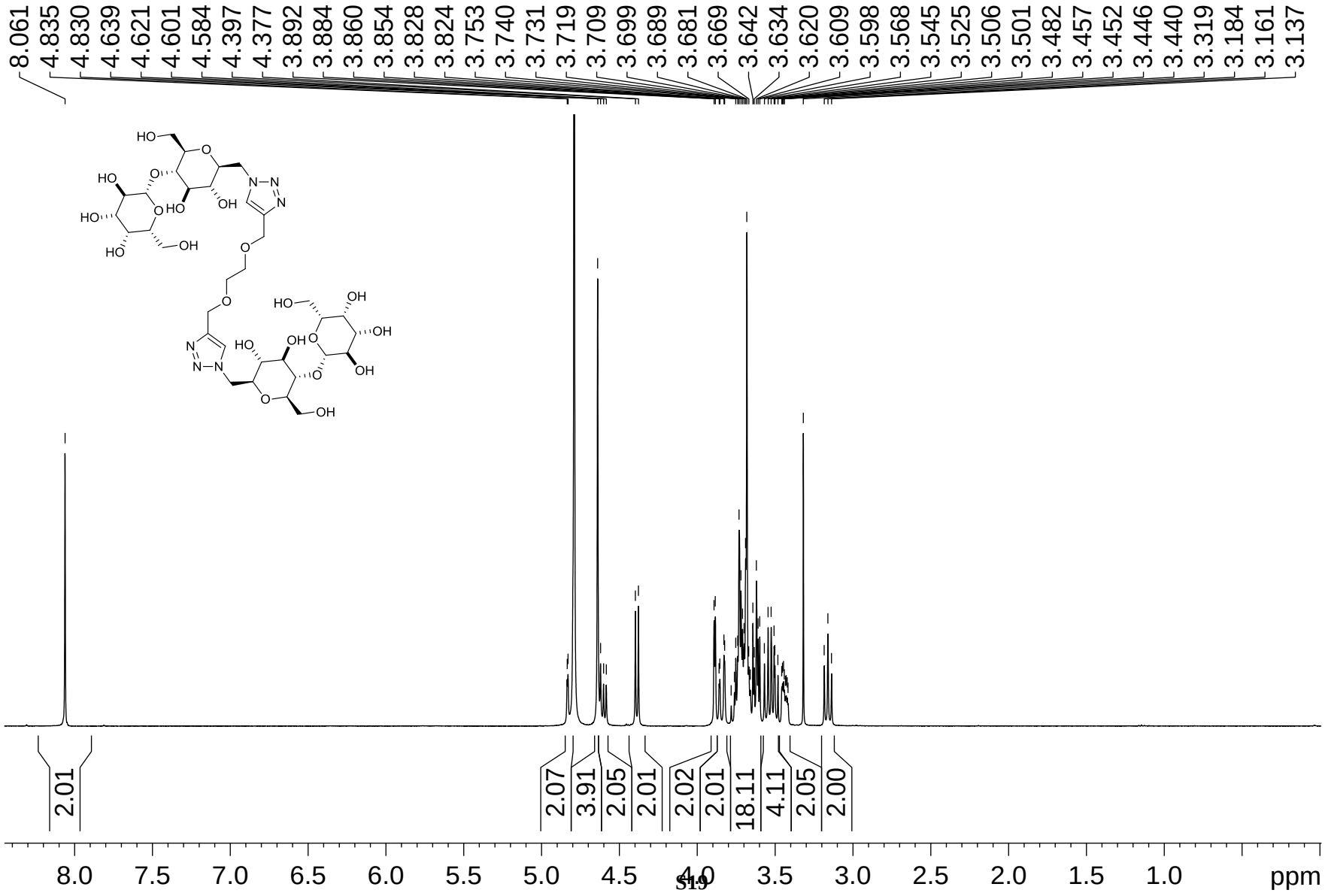
^{1}H NMR (400MHz, D_2O) spectrum of **8c**



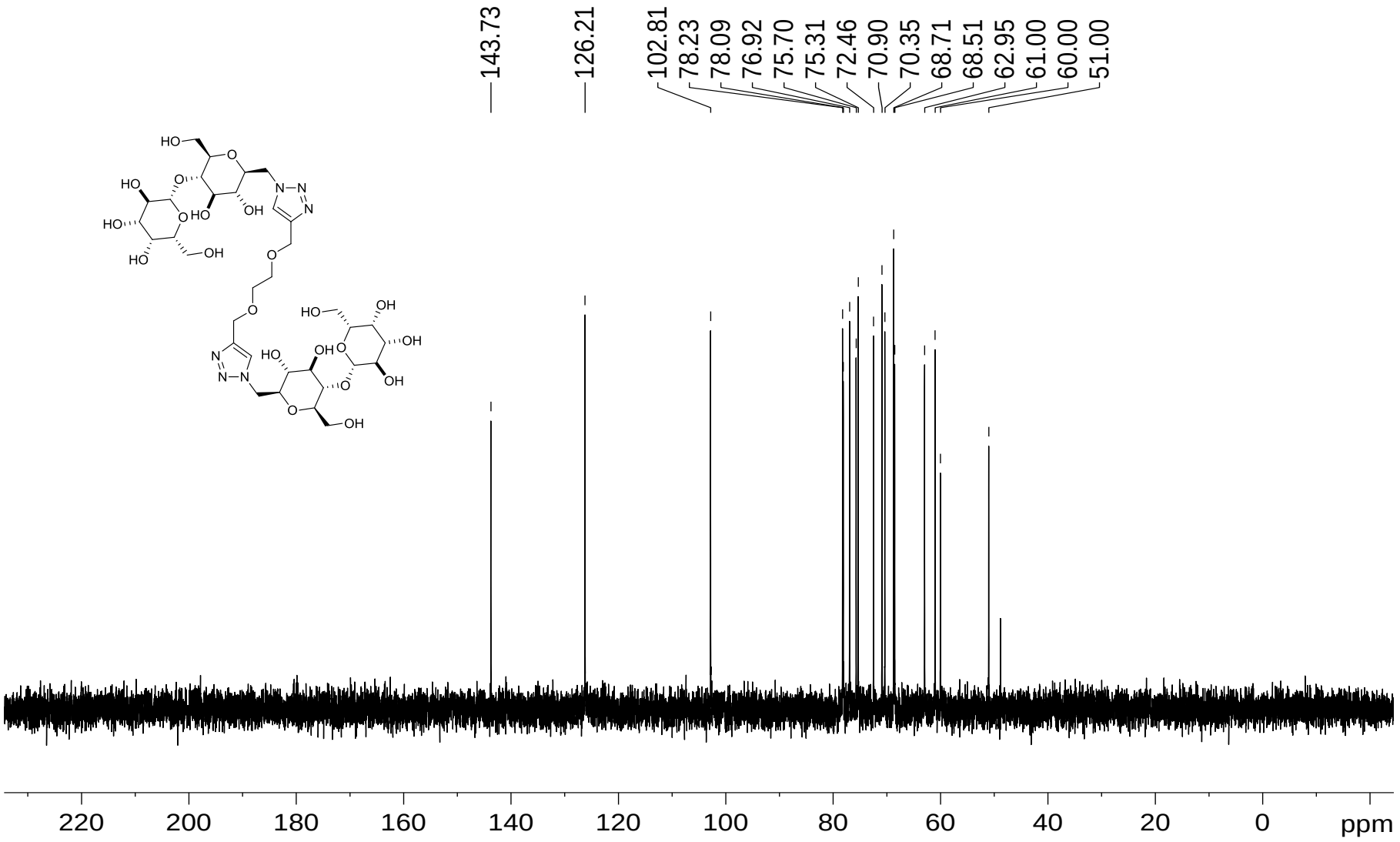
^{13}C NMR (100MHz, D_2O) spectrum of **8c**



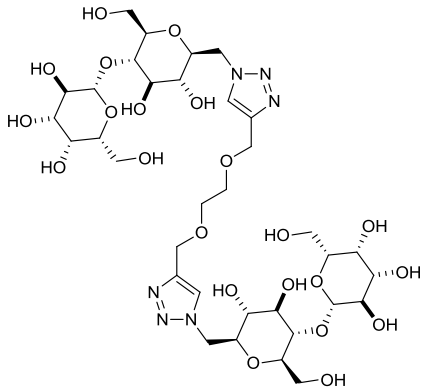
^{1}H NMR (400MHz, D_2O) spectrum of **8d**

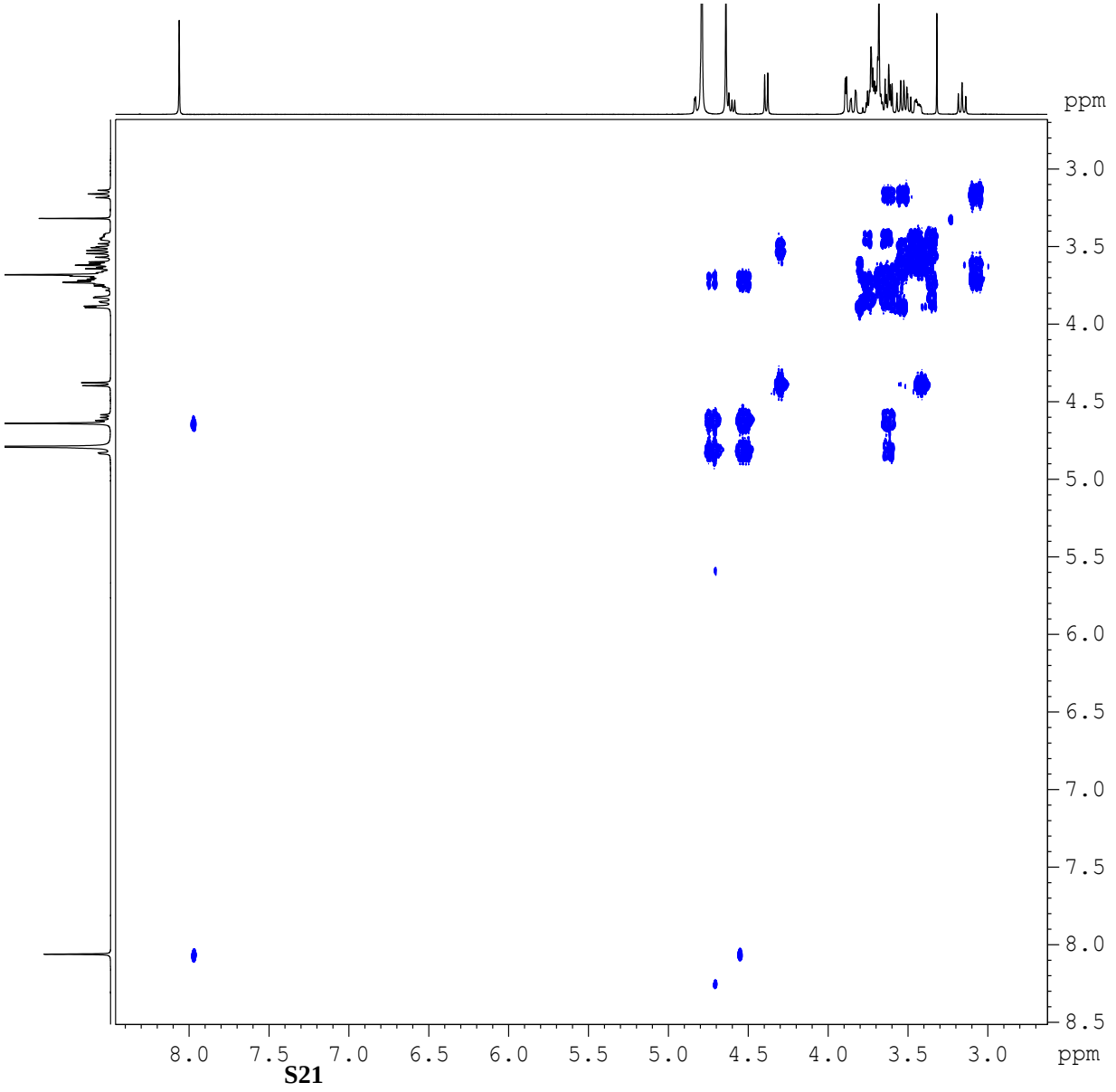


^{13}C NMR (100MHz, D_2O) spectrum of **8d**

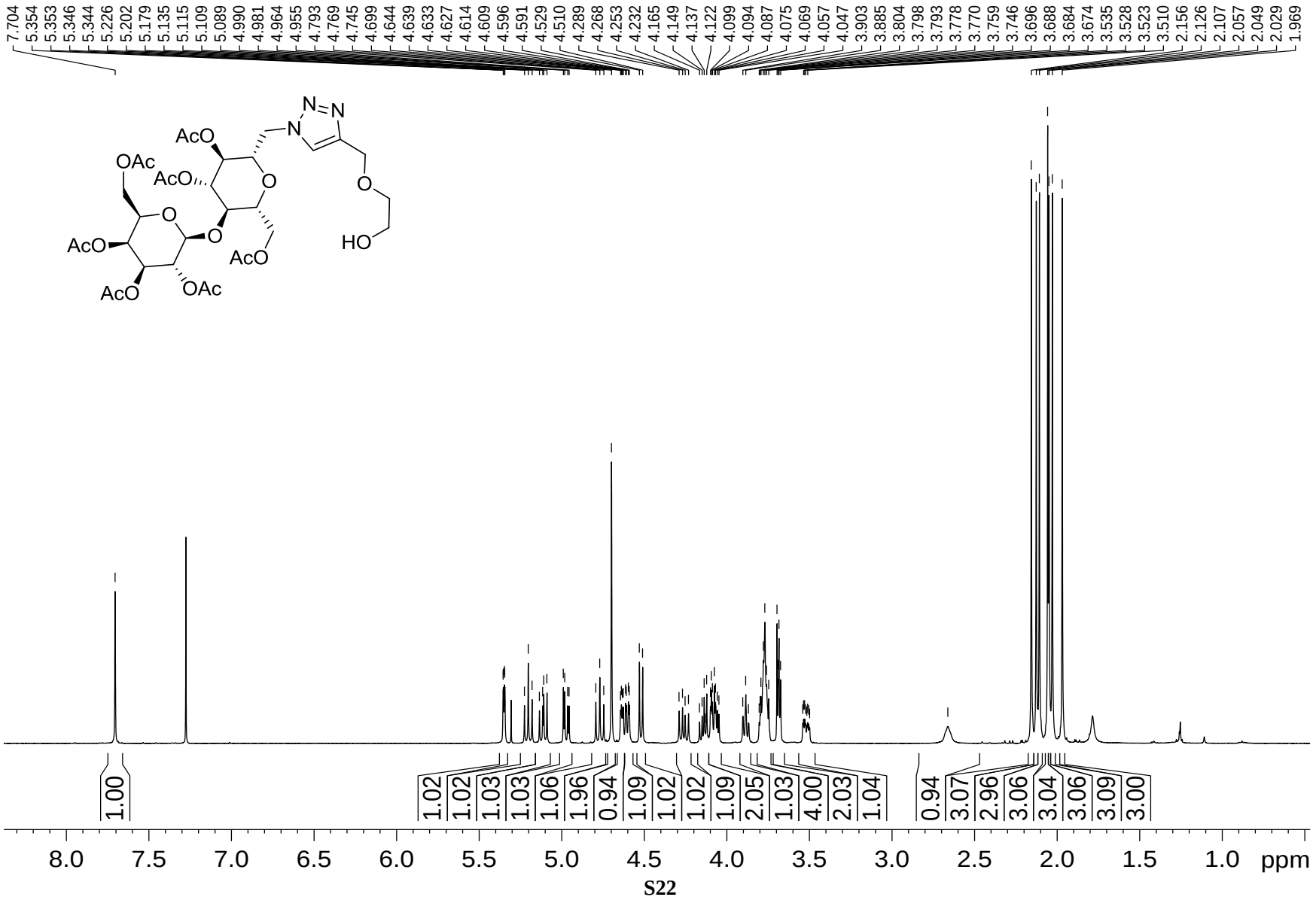


H-H COSY (400MHz, $CDCl_3$) spectrum of **8d**

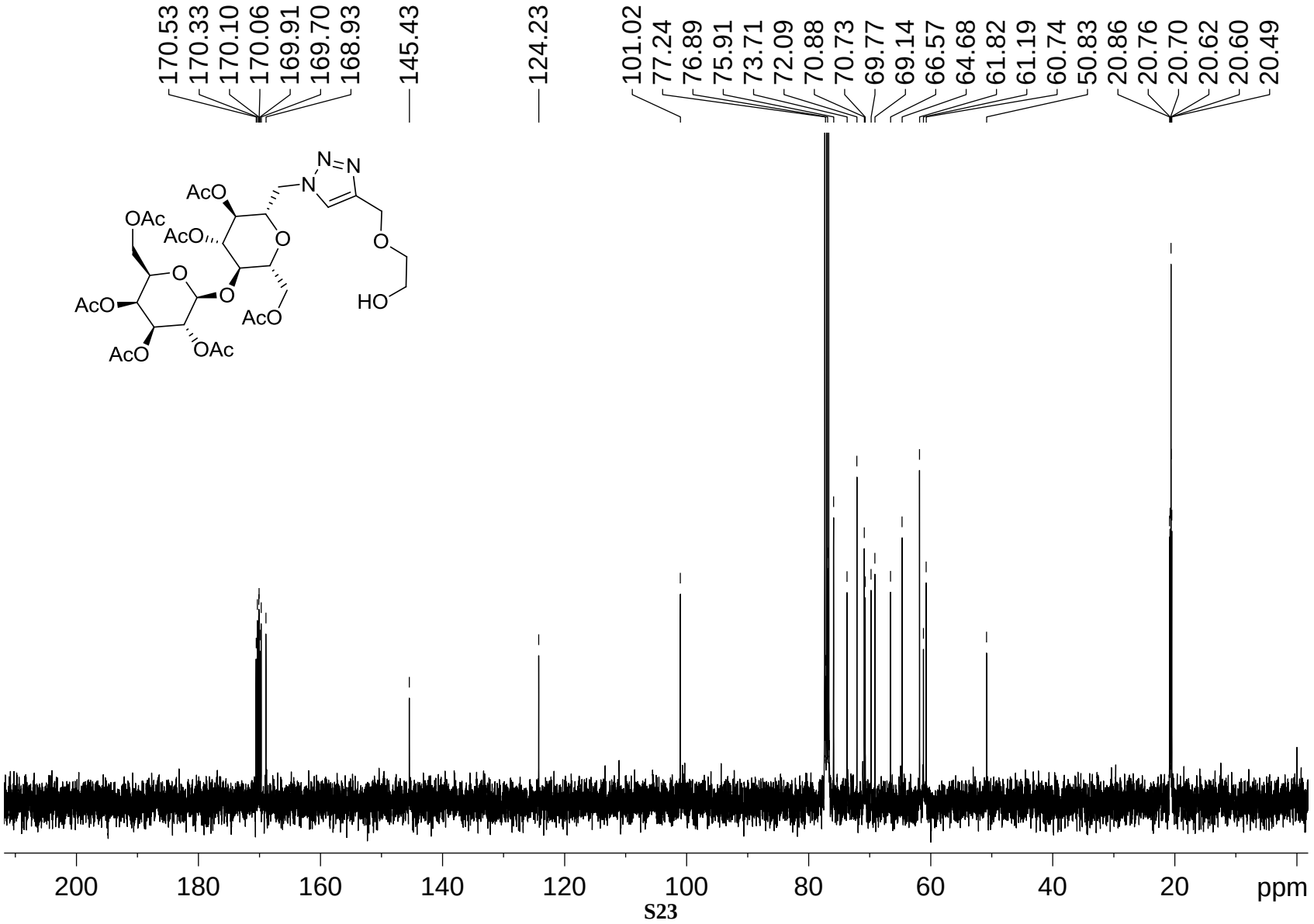




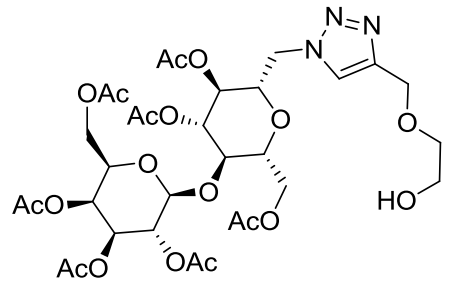
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7a**

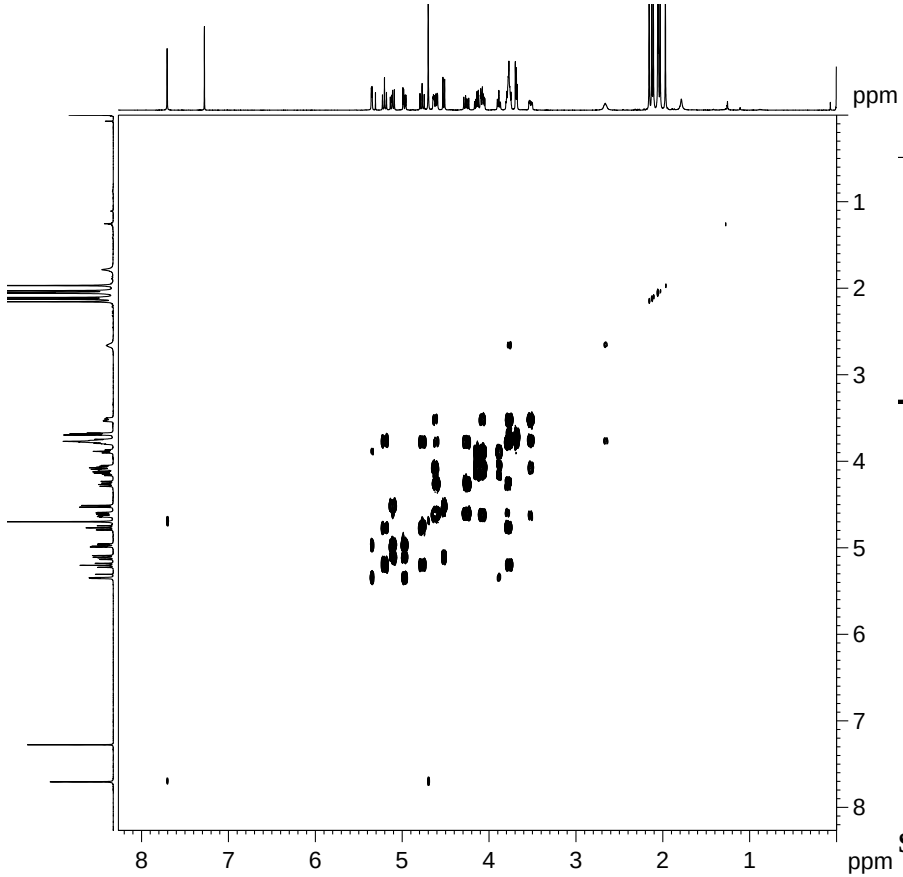


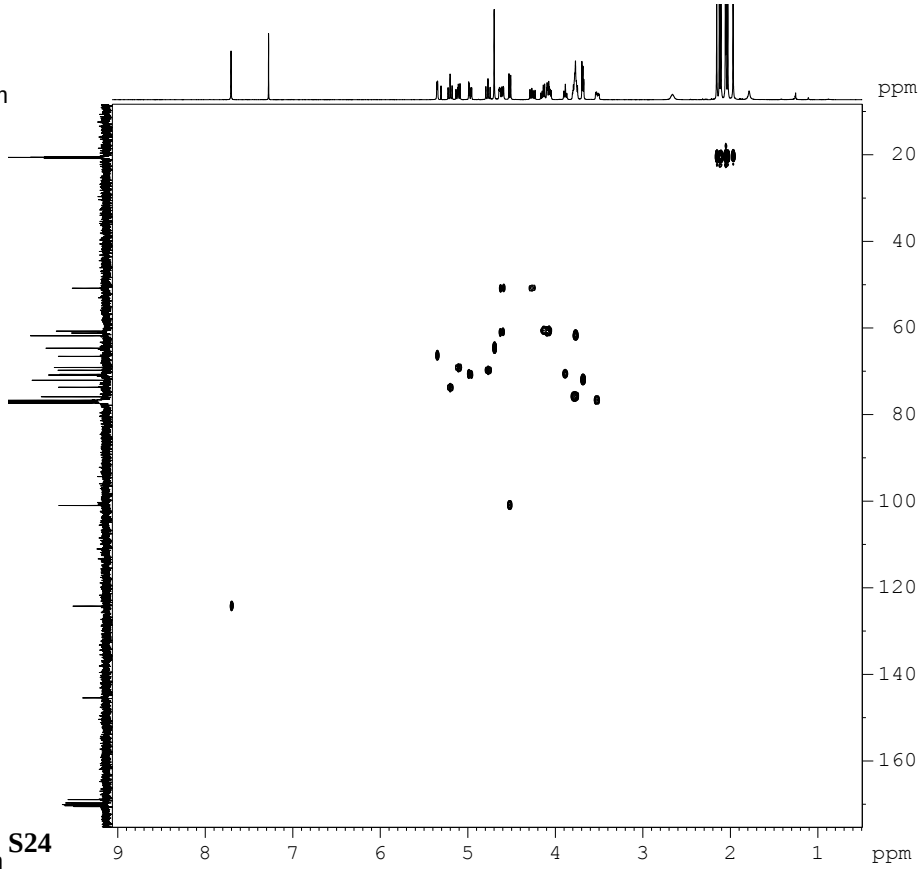
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7a**



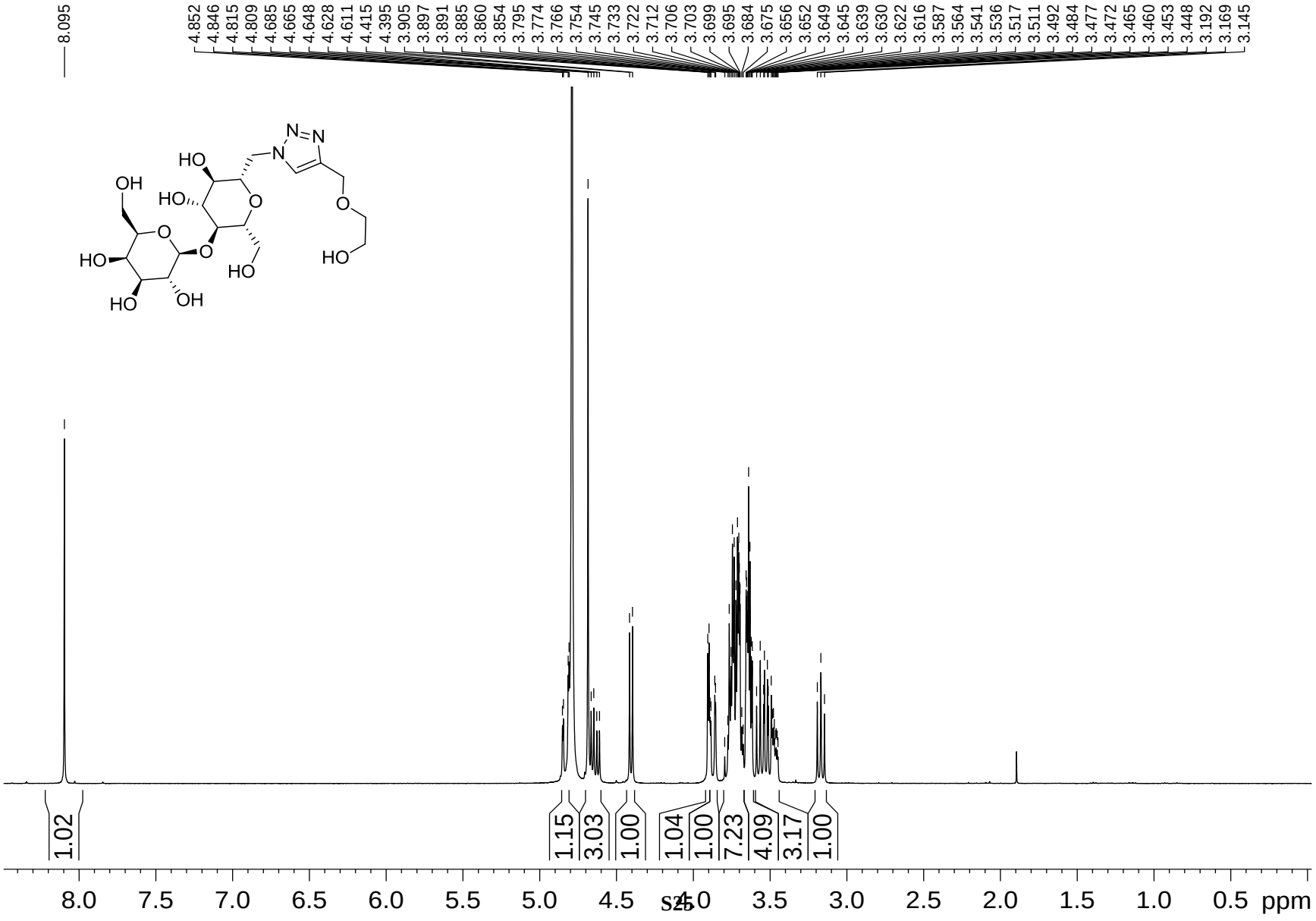
H-H COSY and HSQC (400MHz, $CDCl_3$) spectrum of **7a**



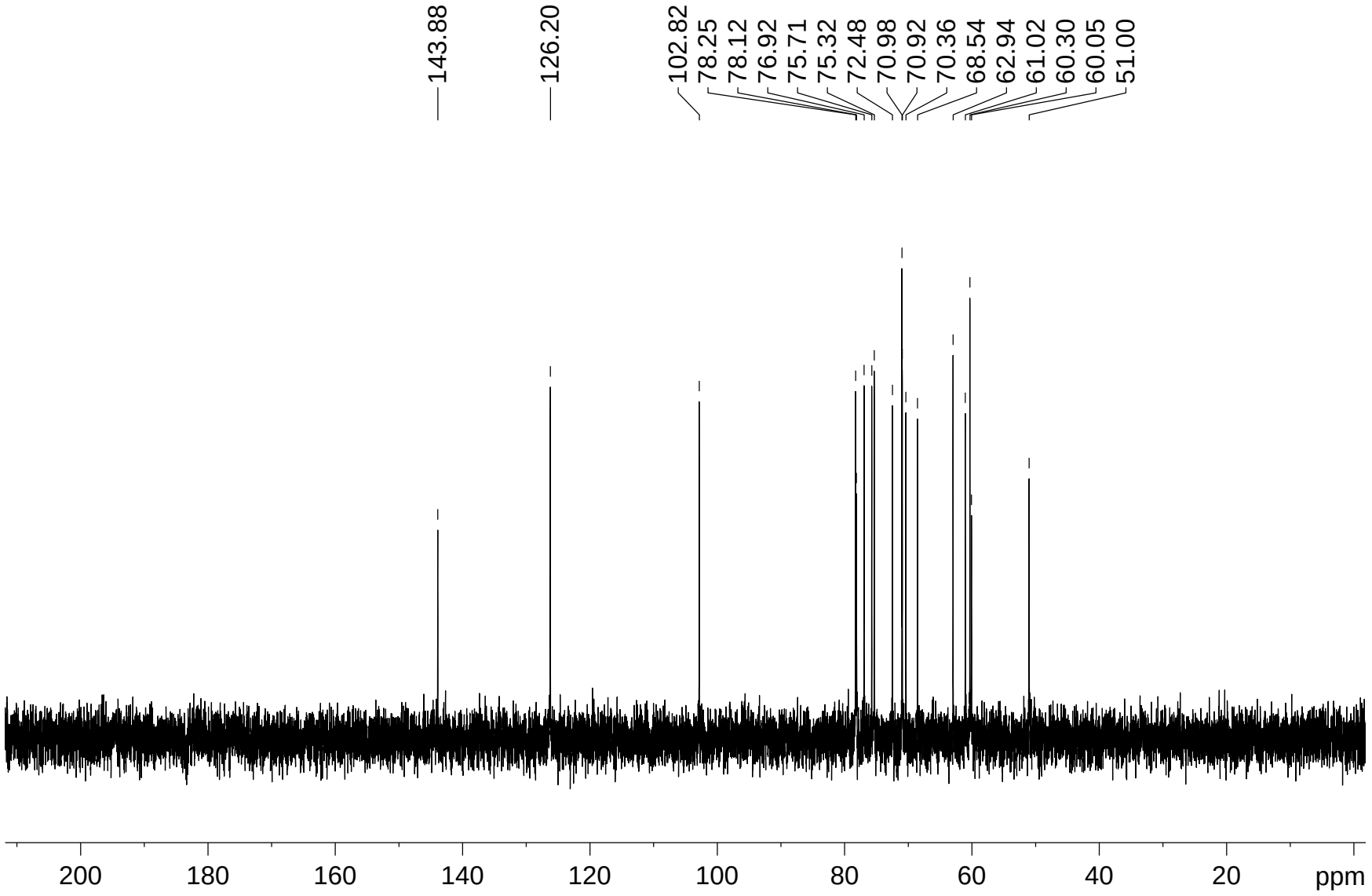




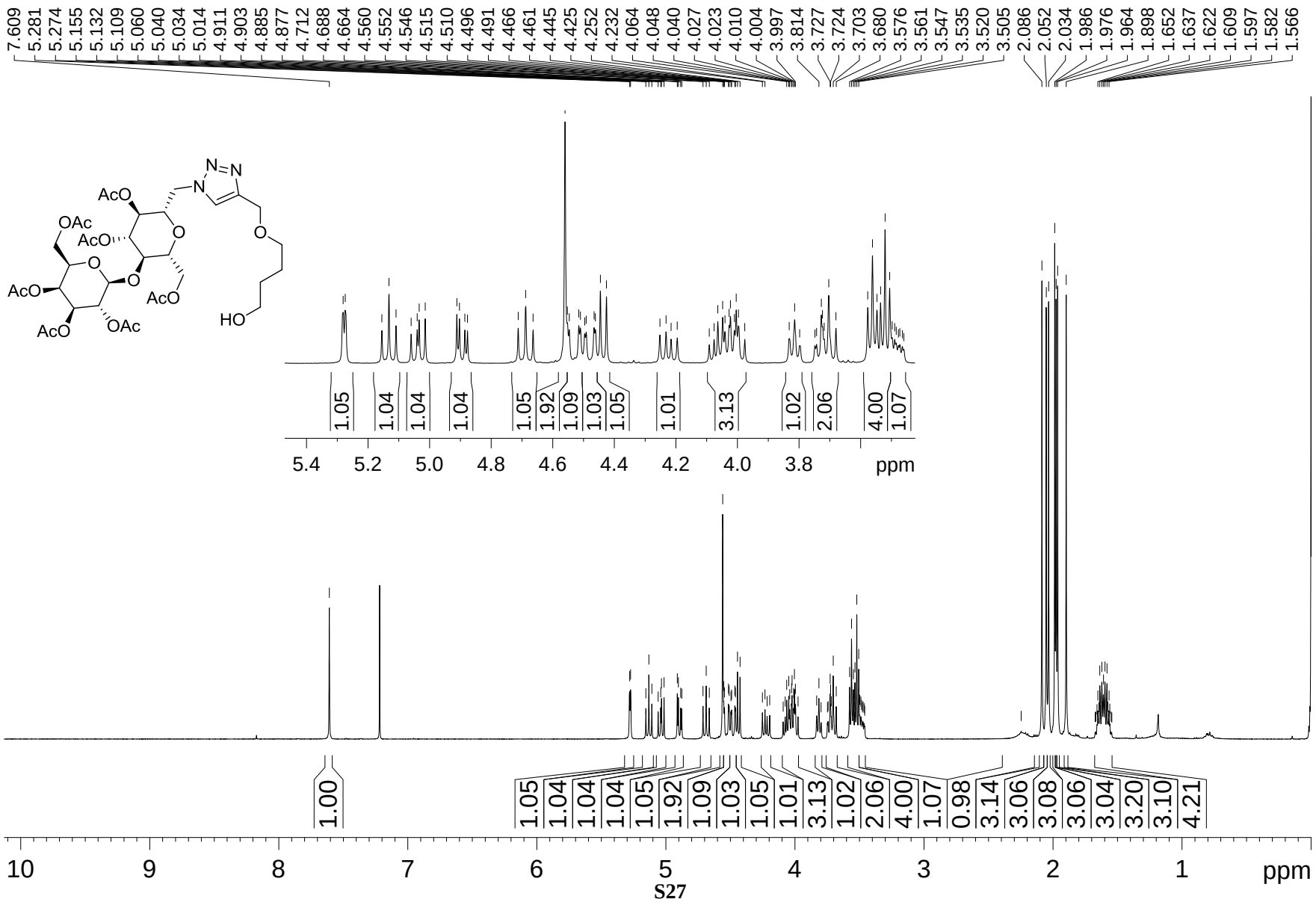
^{1}H NMR (400MHz, D_2O) spectrum of **8a**



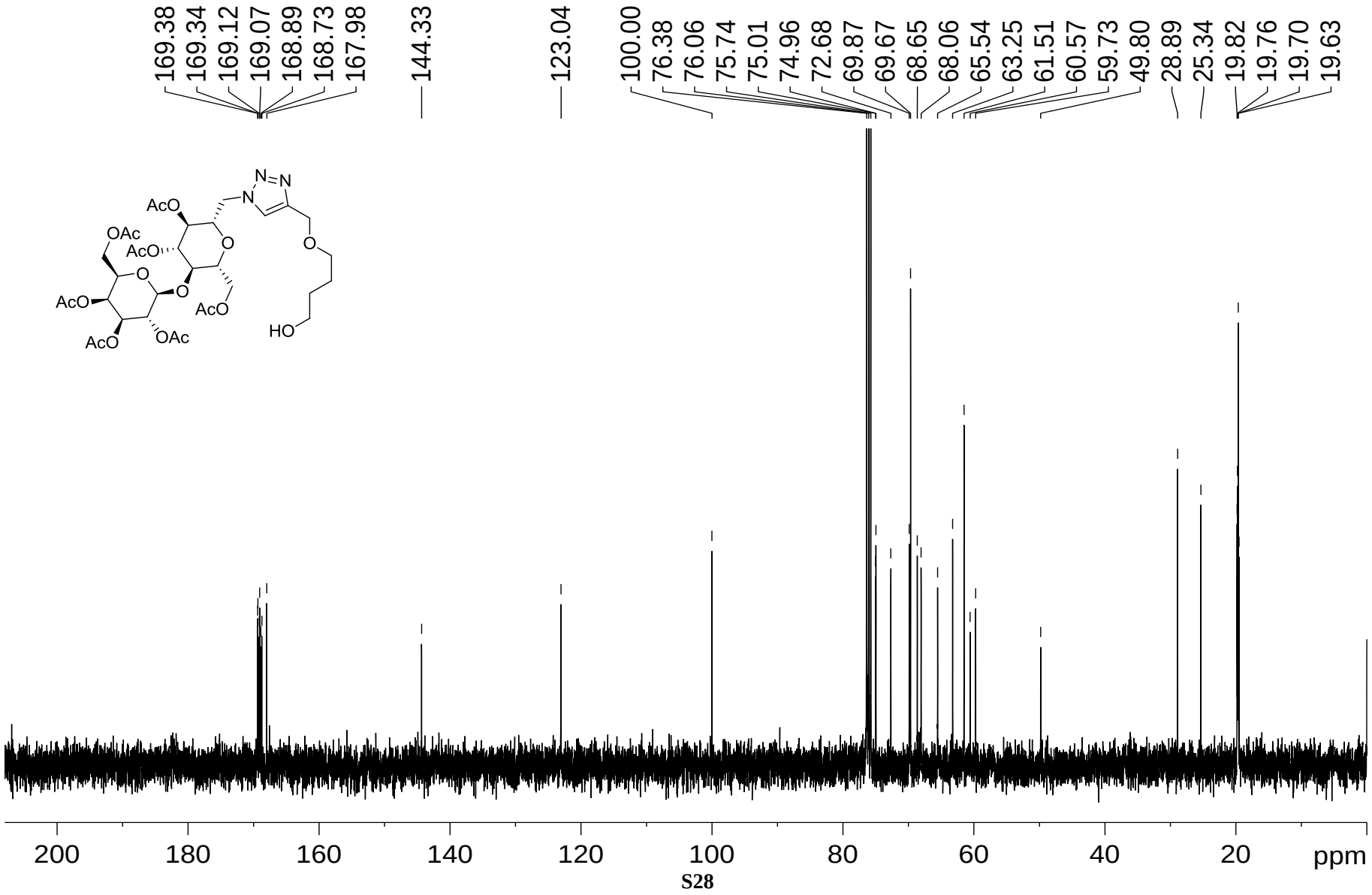
^{13}C NMR (100MHz, D_2O) spectrum of **8a**



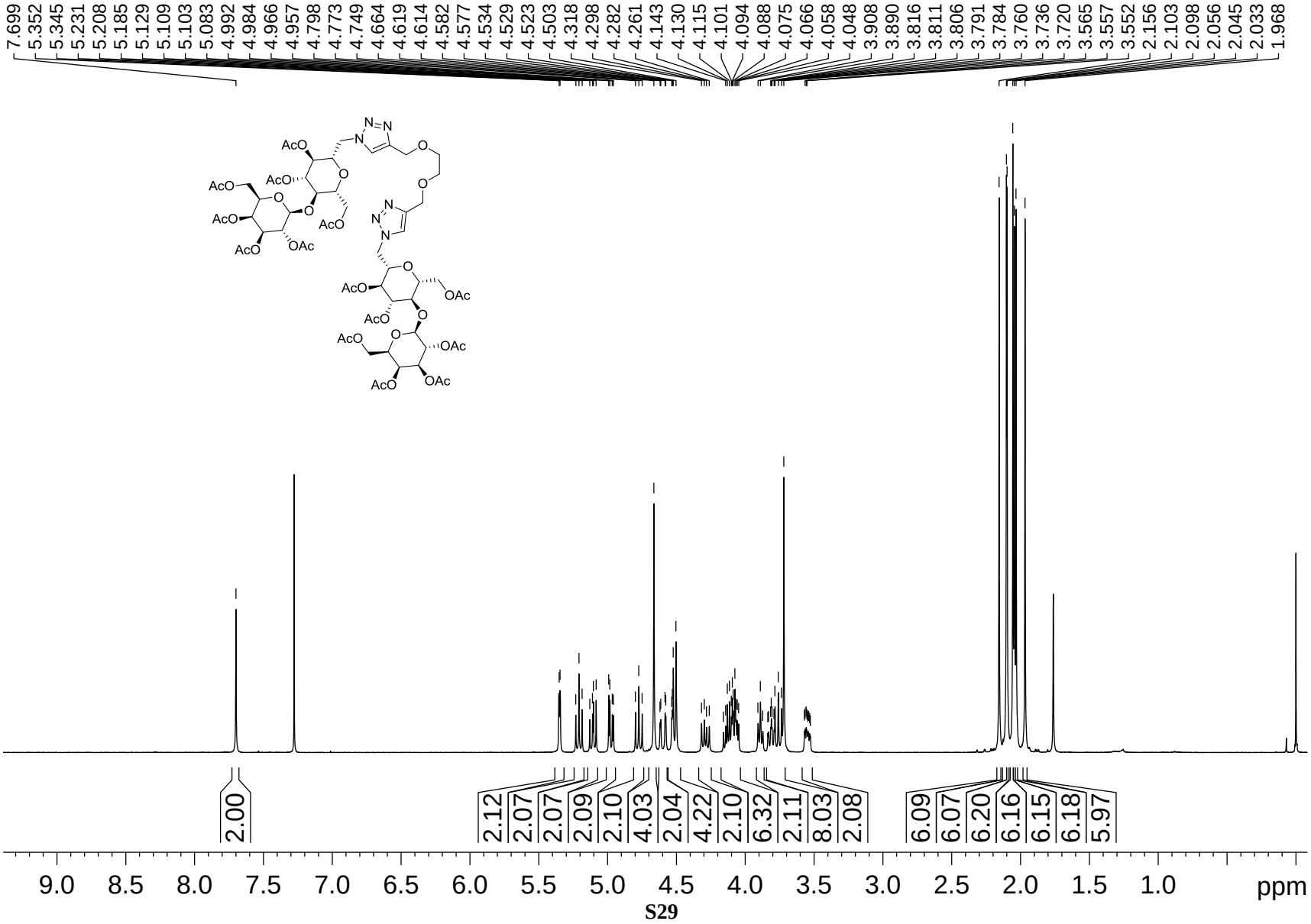
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7b**



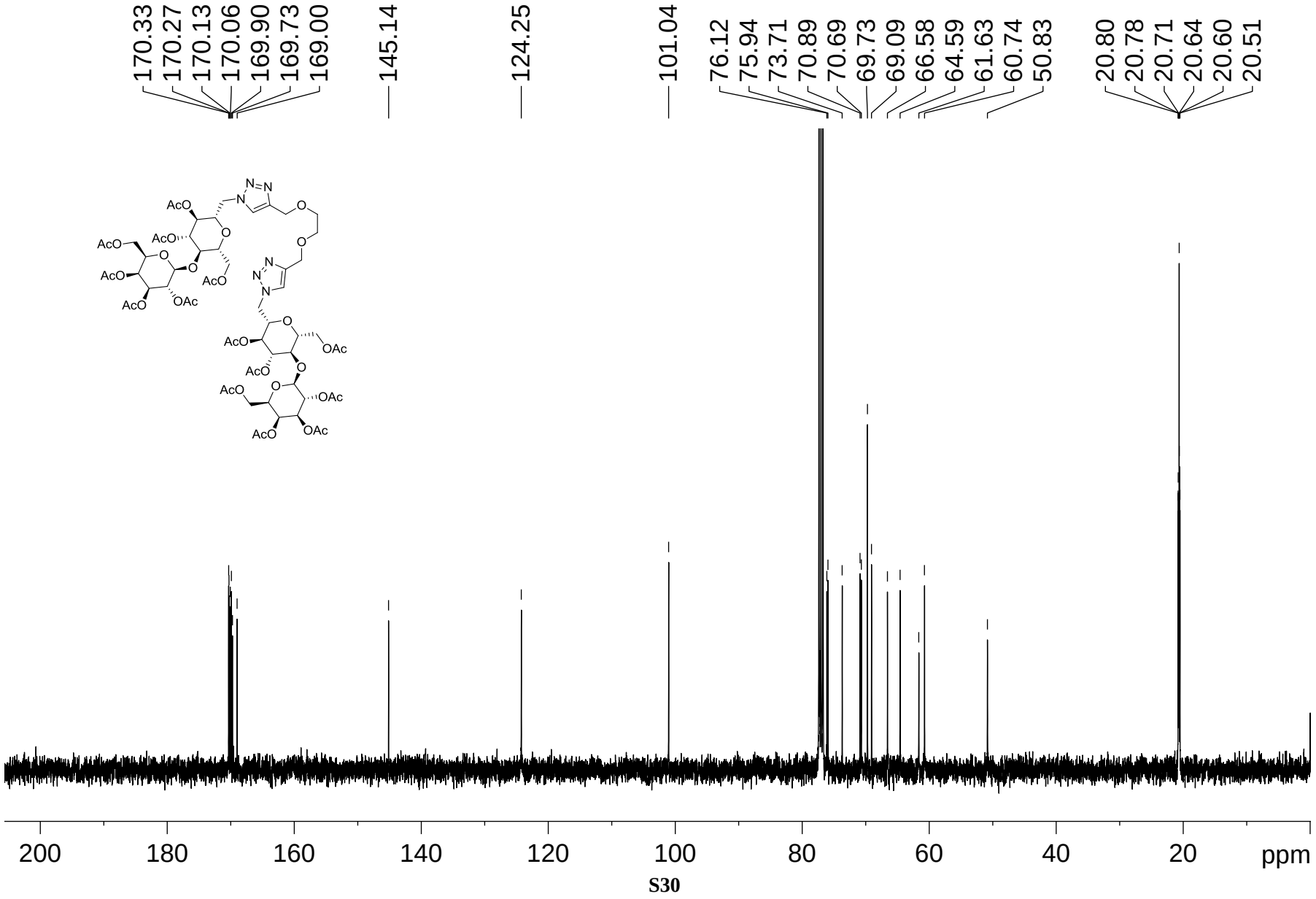
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7b**



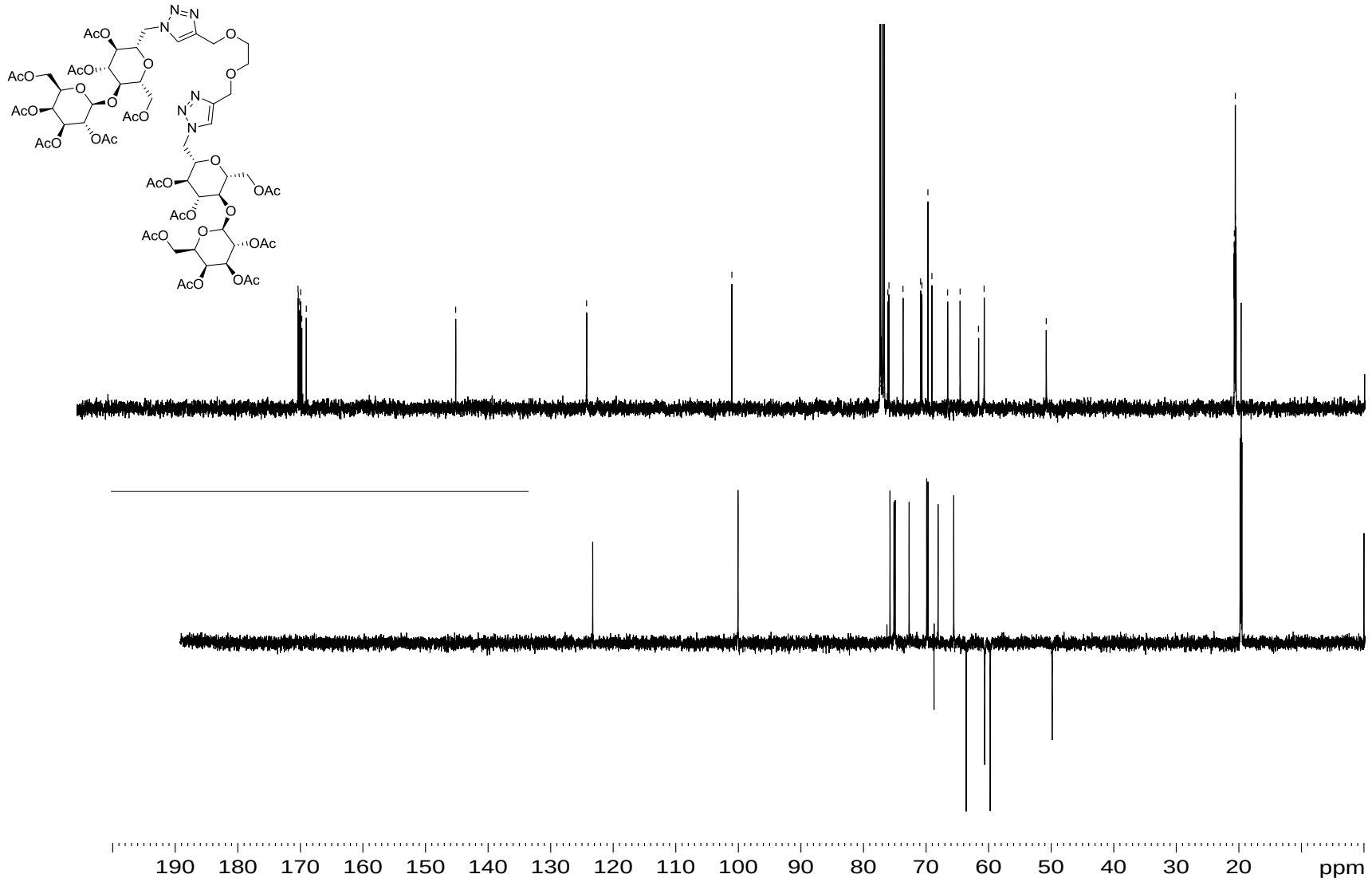
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7d**



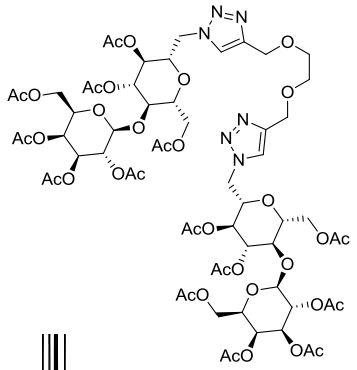
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7d**

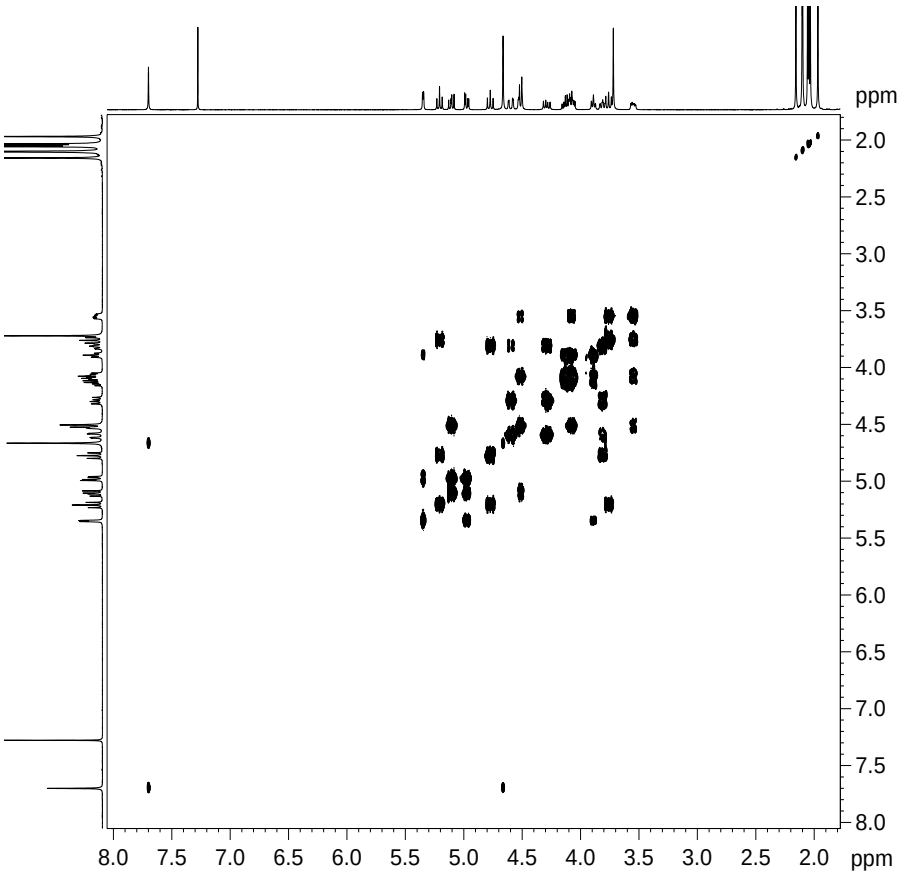


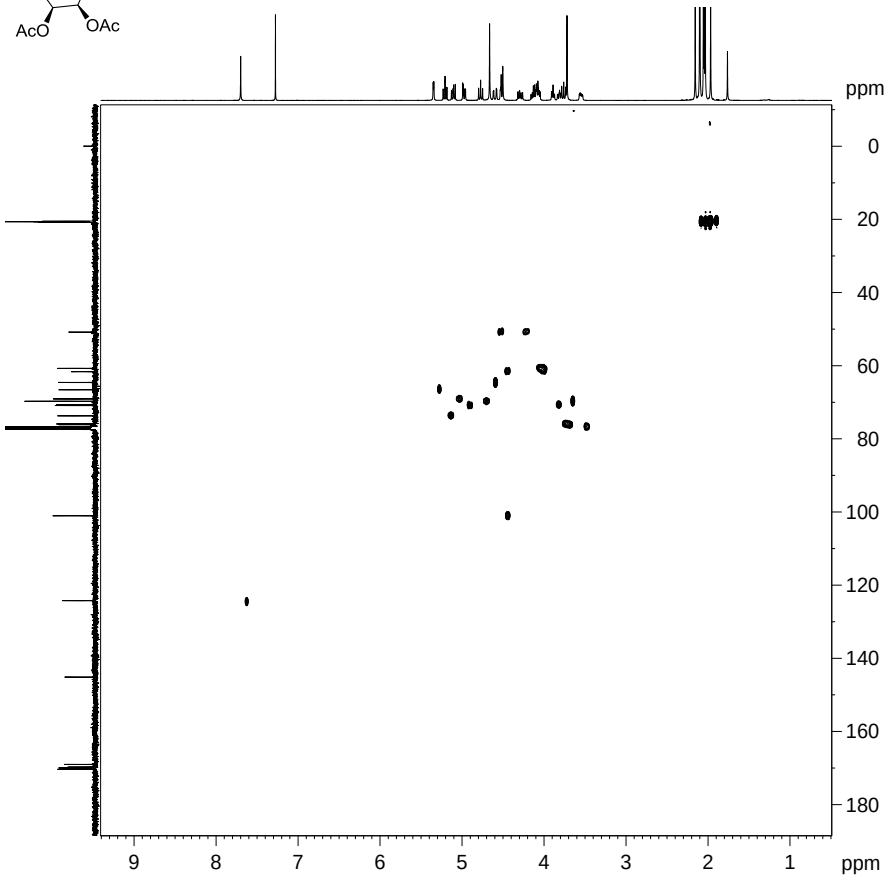
The comparison of ^{13}C NMR and DEPT135 (100MHz, $CDCl_3$) spectrum of **7d**



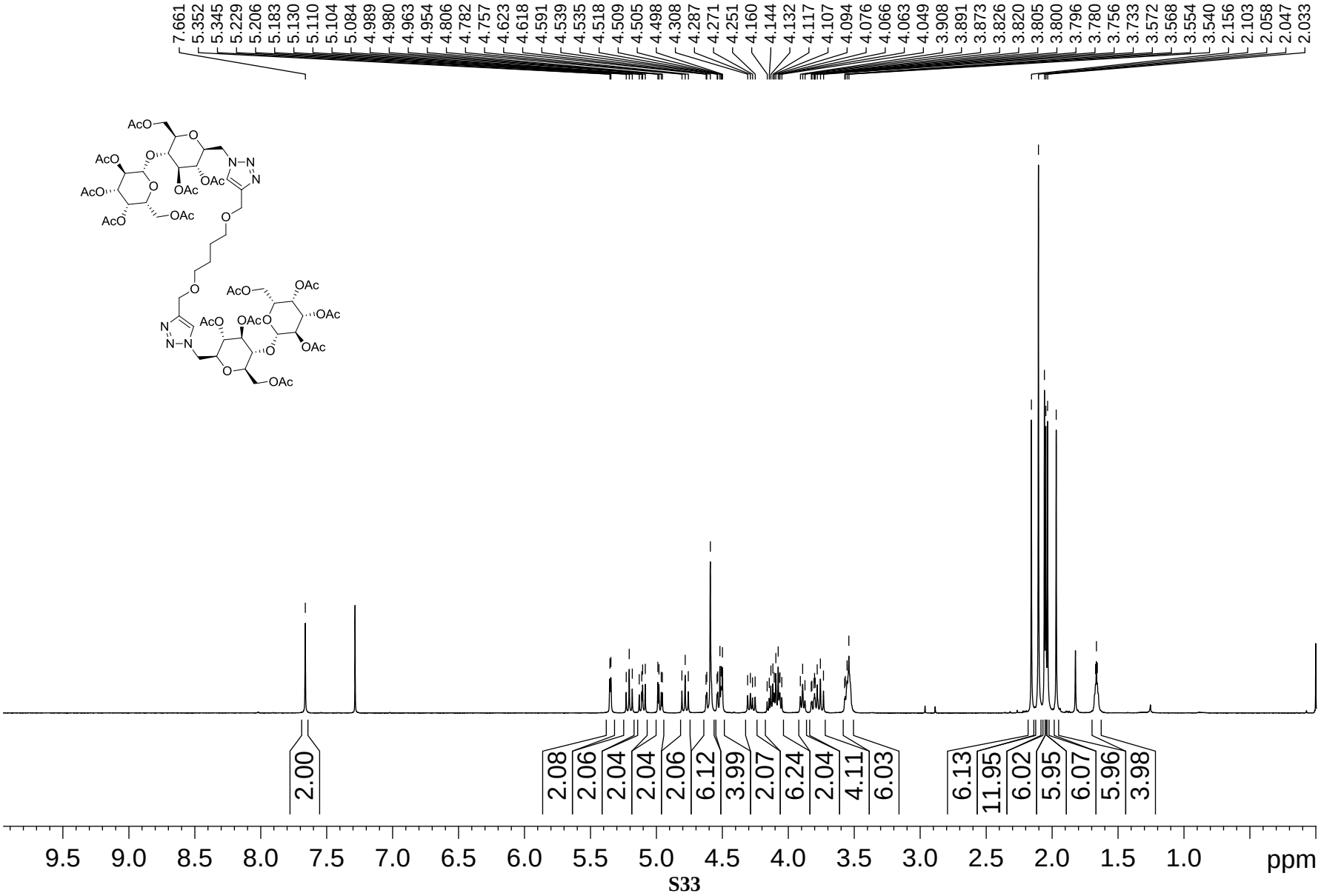
H-H COSY and HSQC (400MHz, $CDCl_3$) spectrum of **7d**



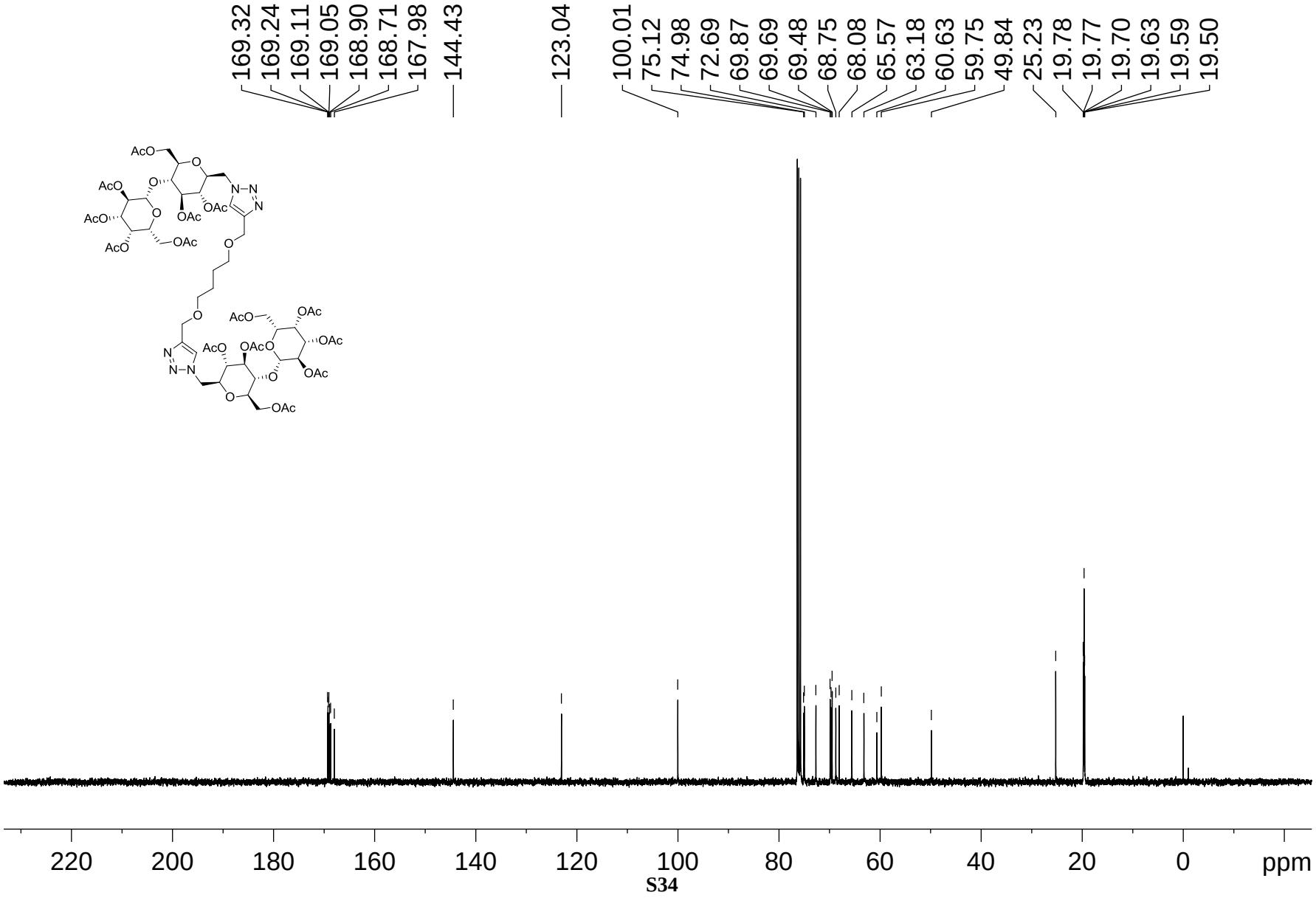




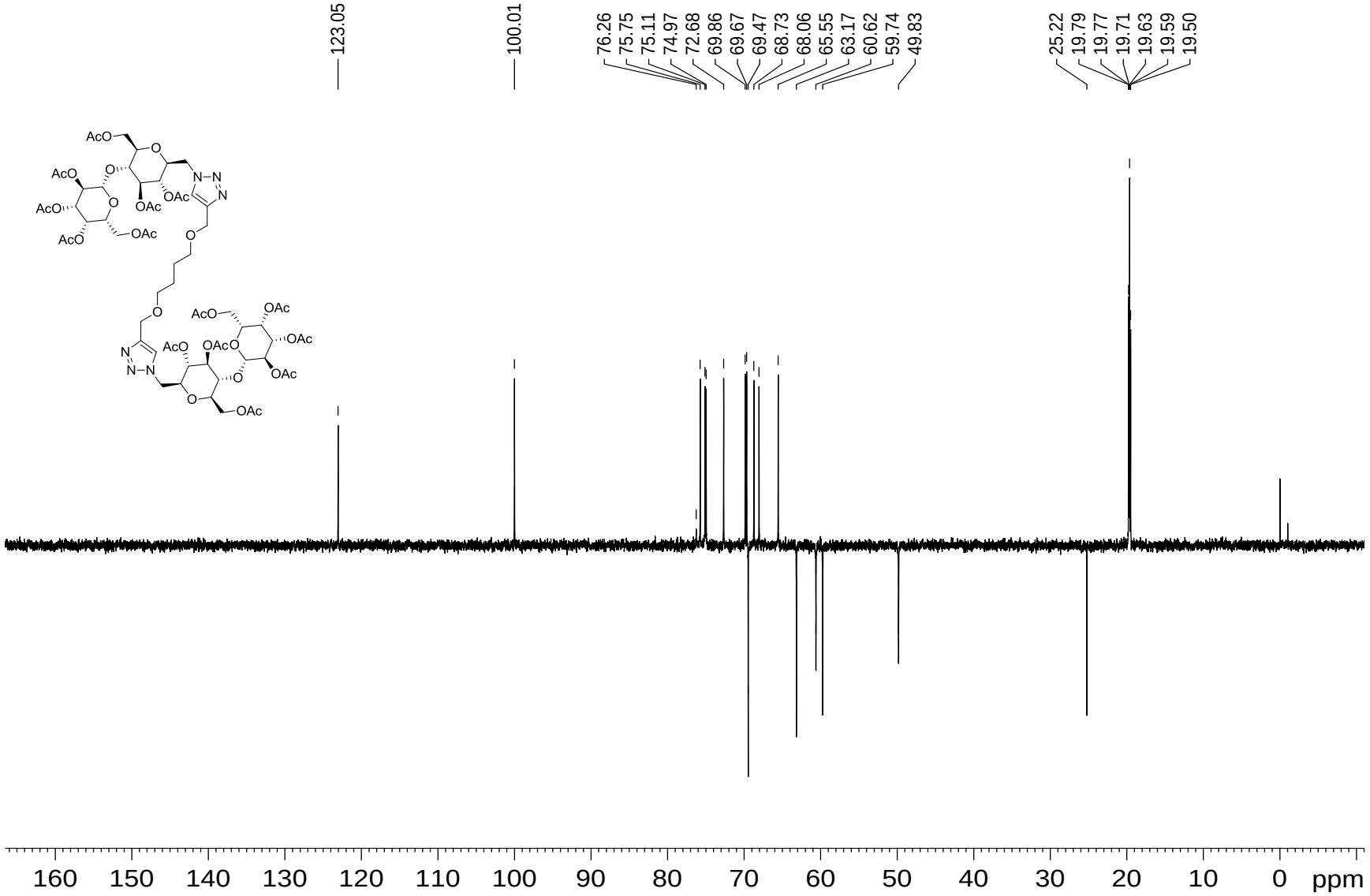
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7f**



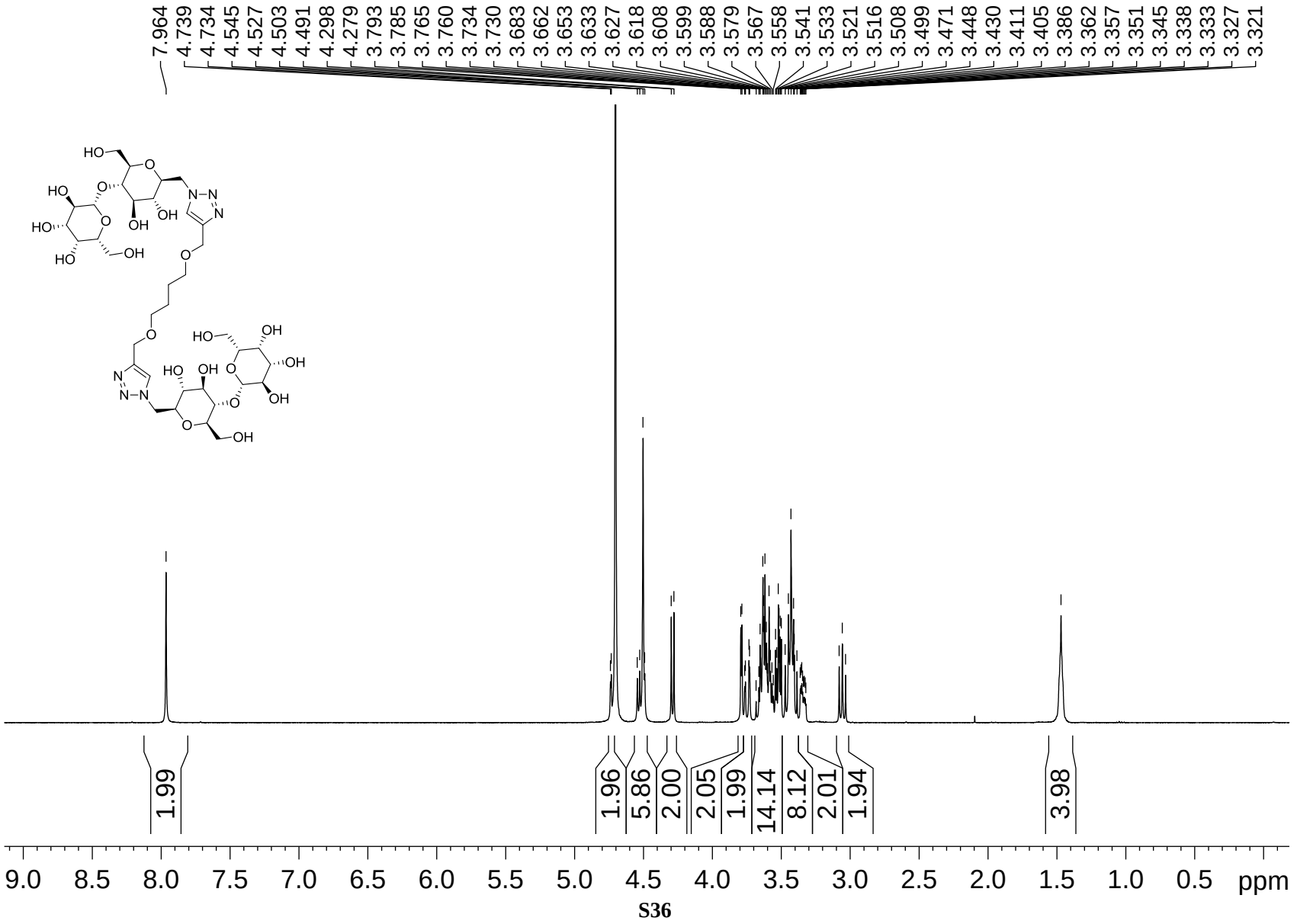
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7f**



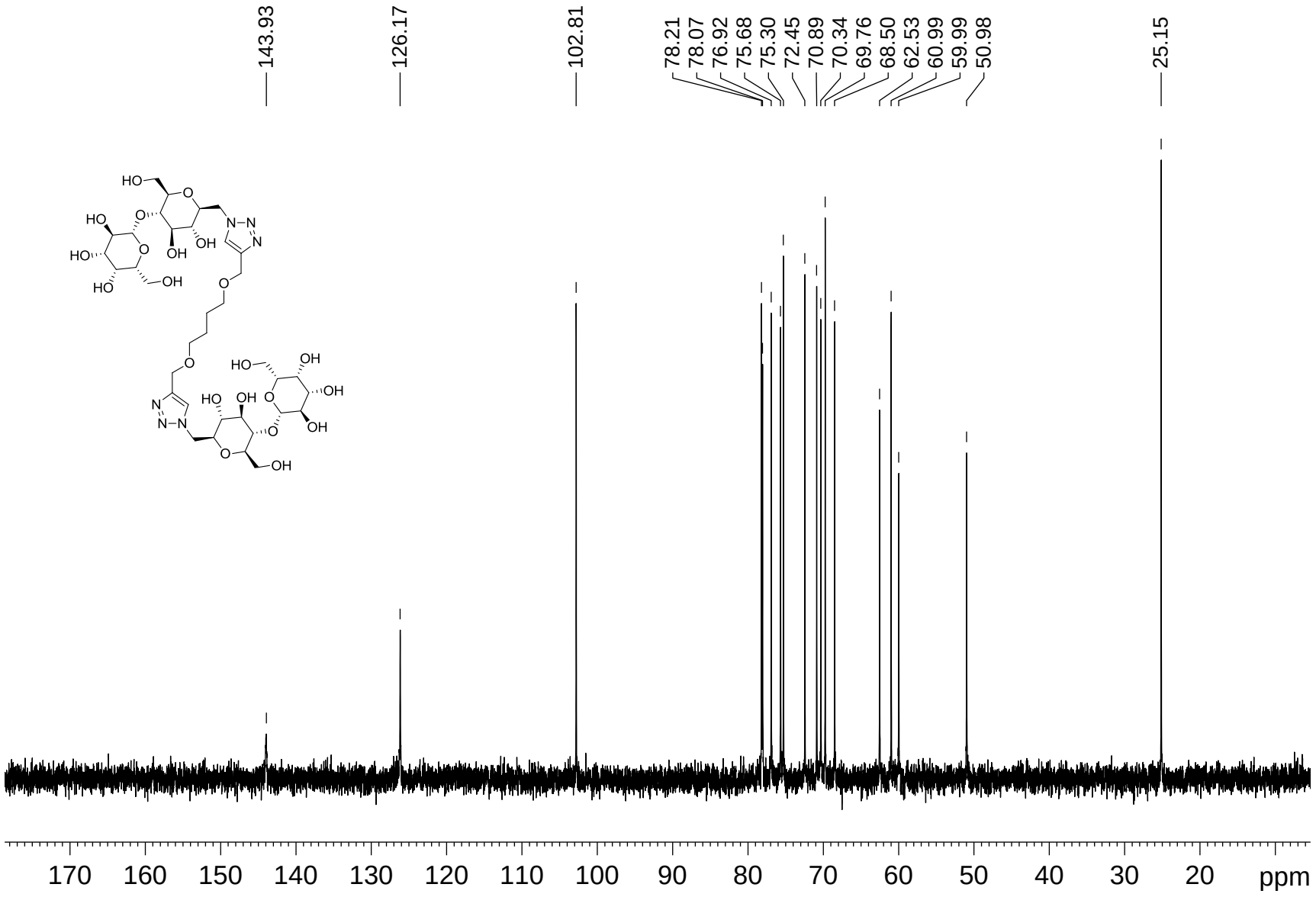
DEPT135 (100MHz, $CDCl_3$) spectrum of **7f**



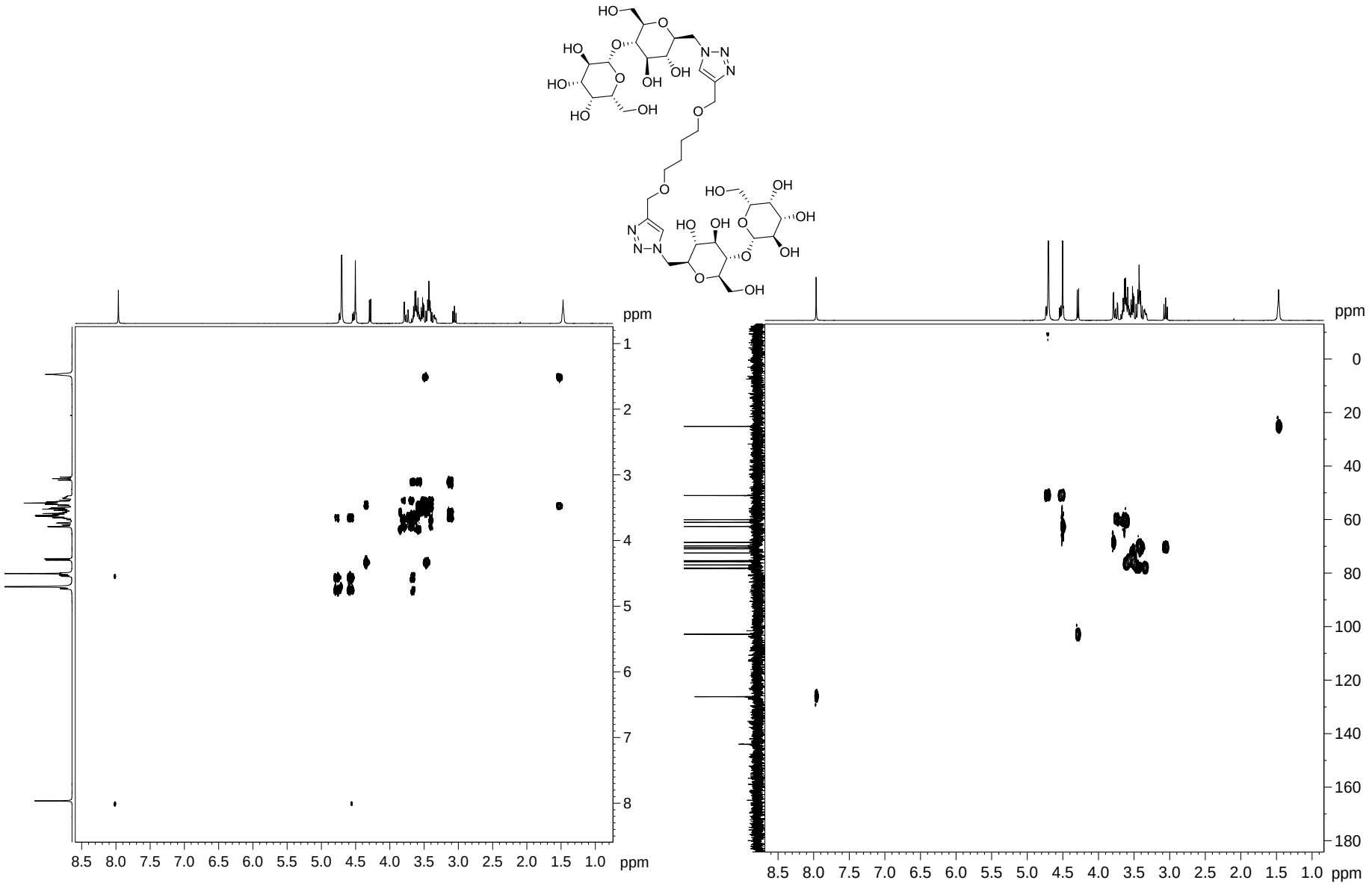
^{1}H NMR (400MHz, D_2O) spectrum of **8f**



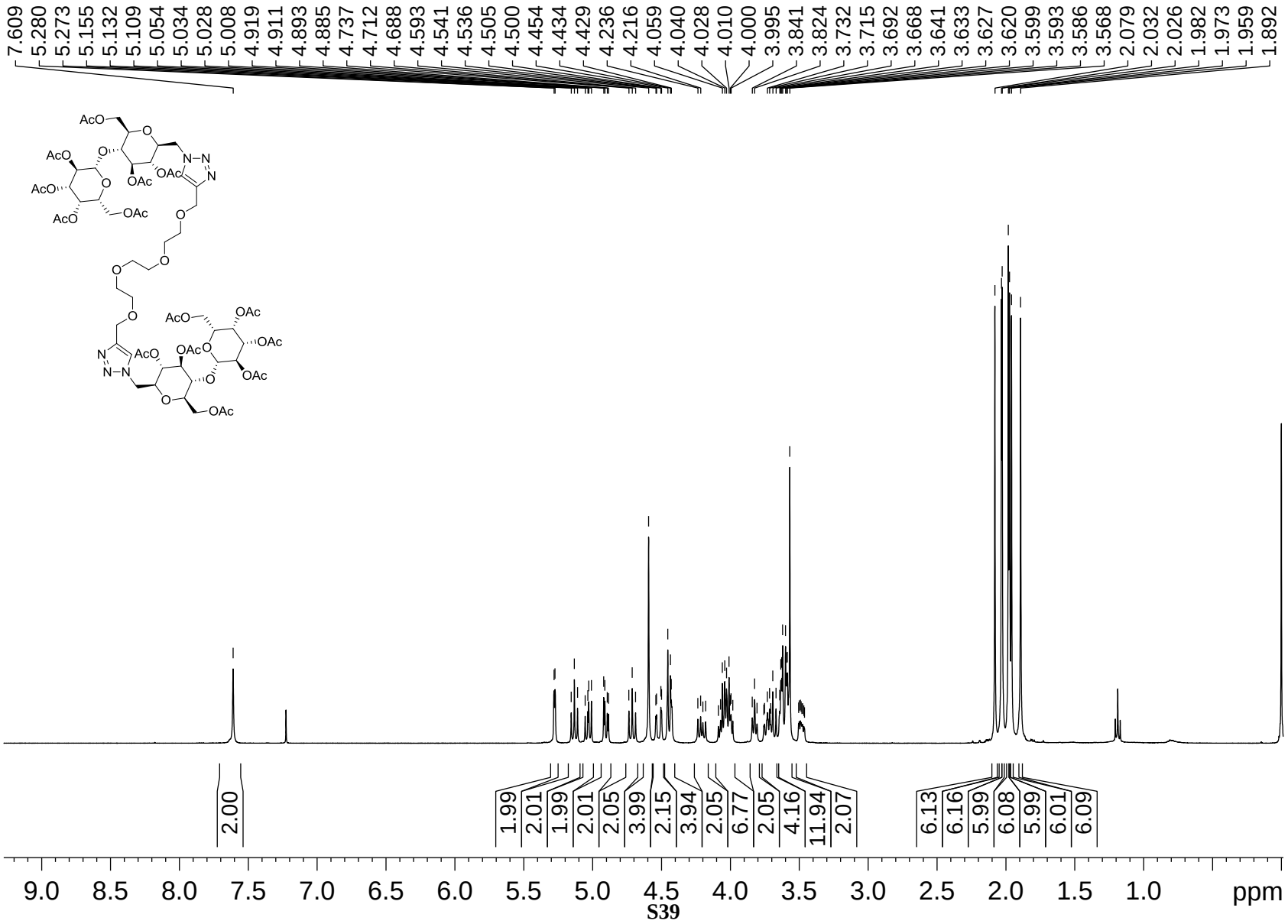
^{13}C NMR (100MHz, D_2O) spectrum of **8f**



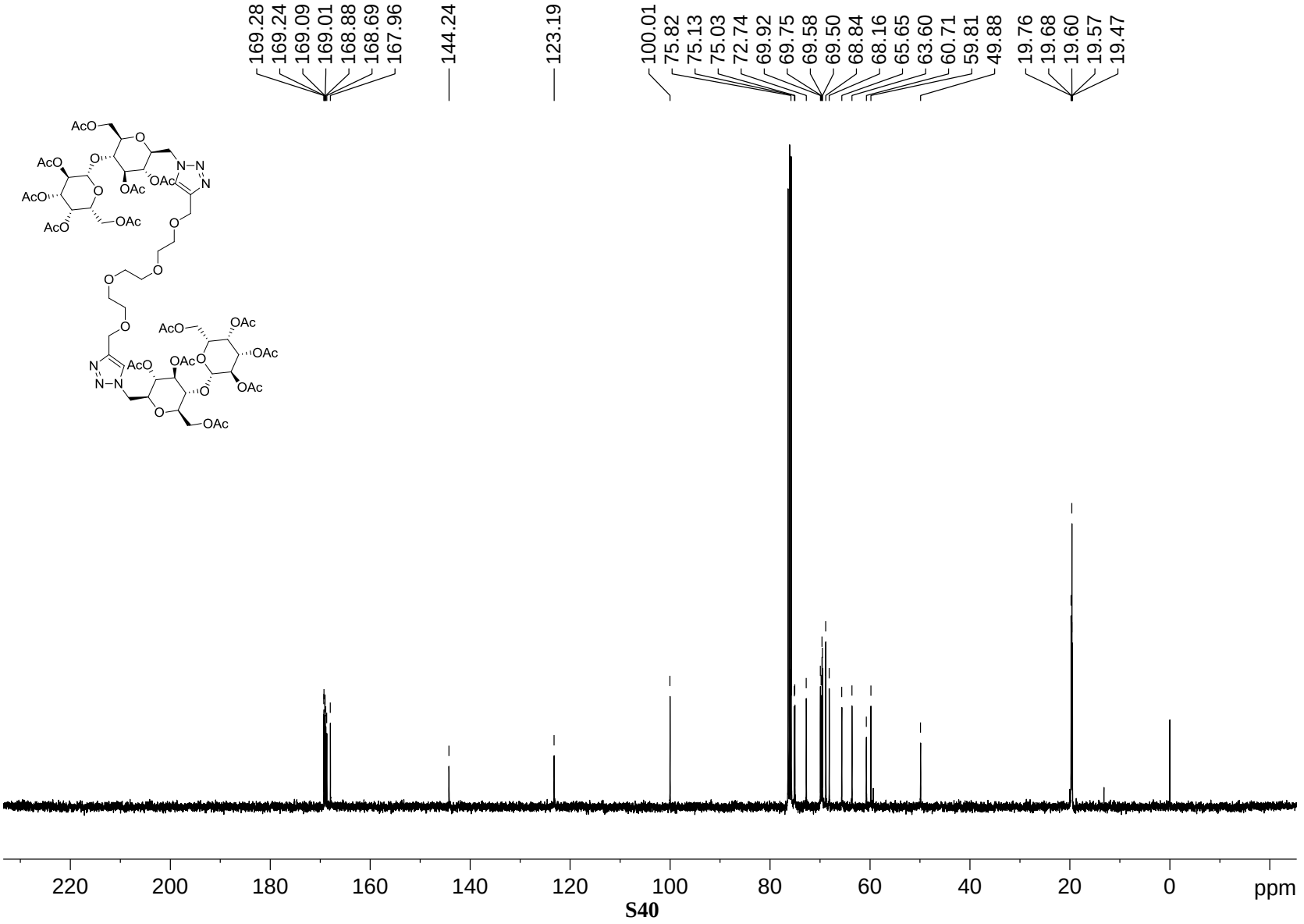
H-H COSY and HSQC (400MHz, D_2O) spectrum of **8f**



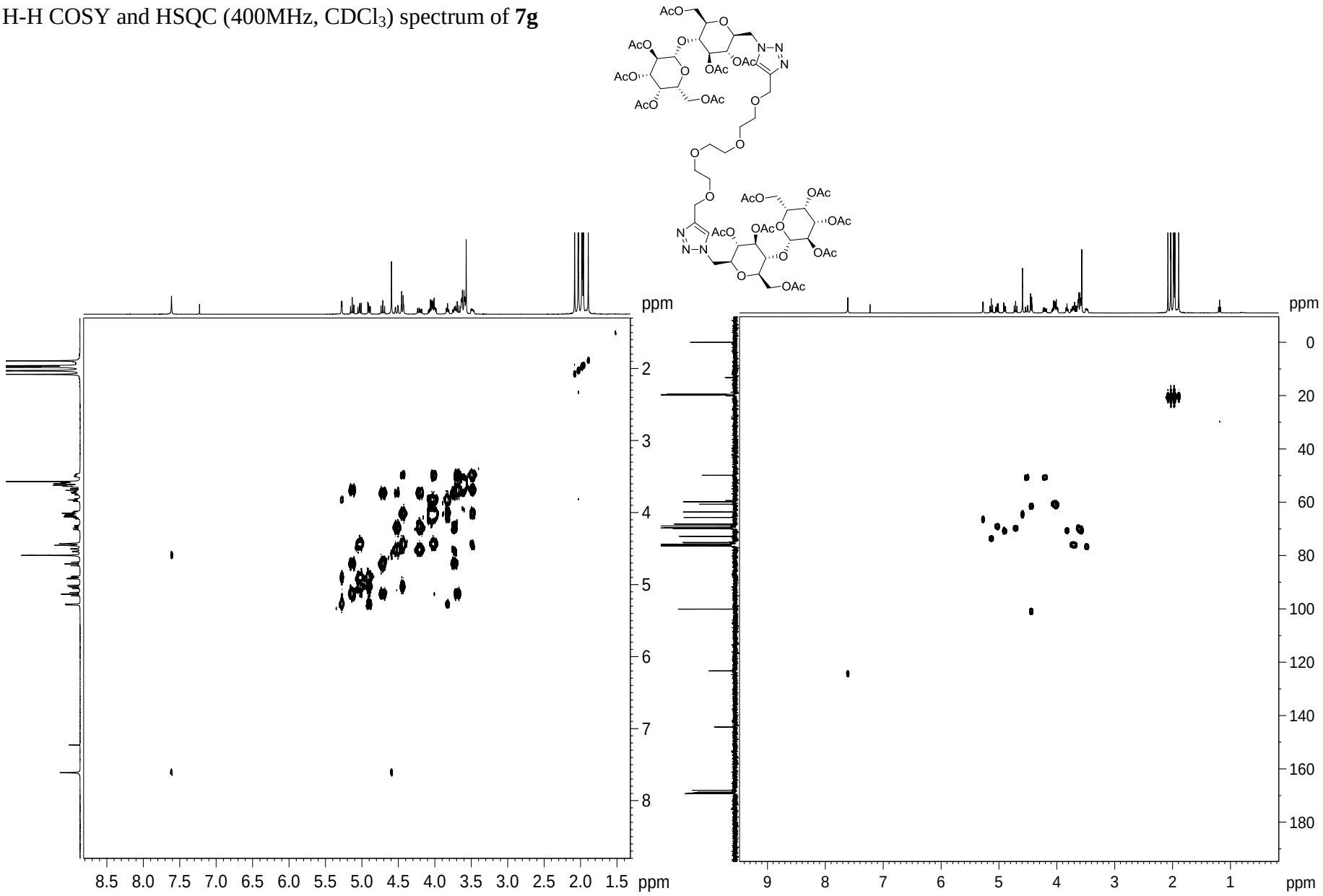
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7g**



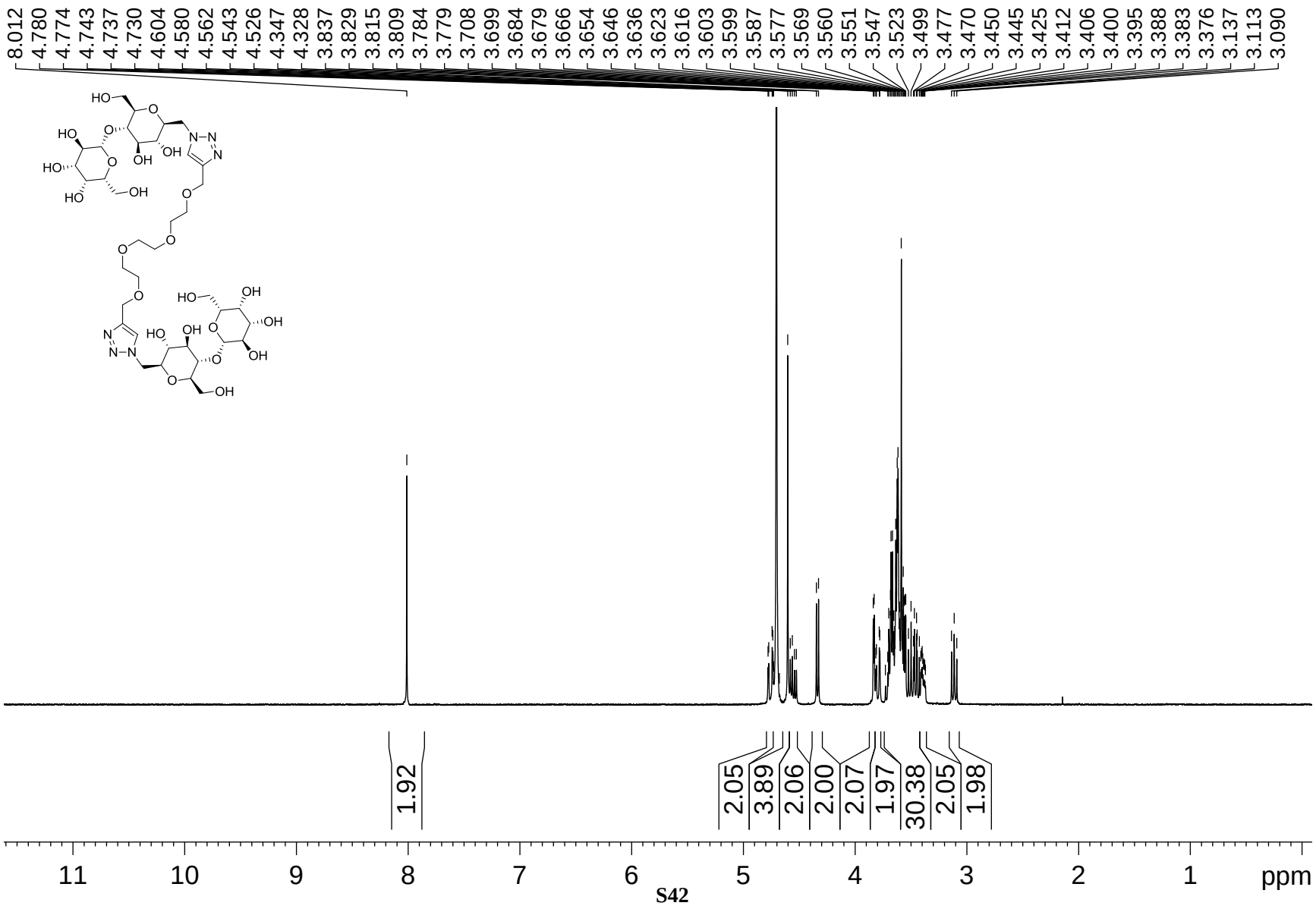
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7g**



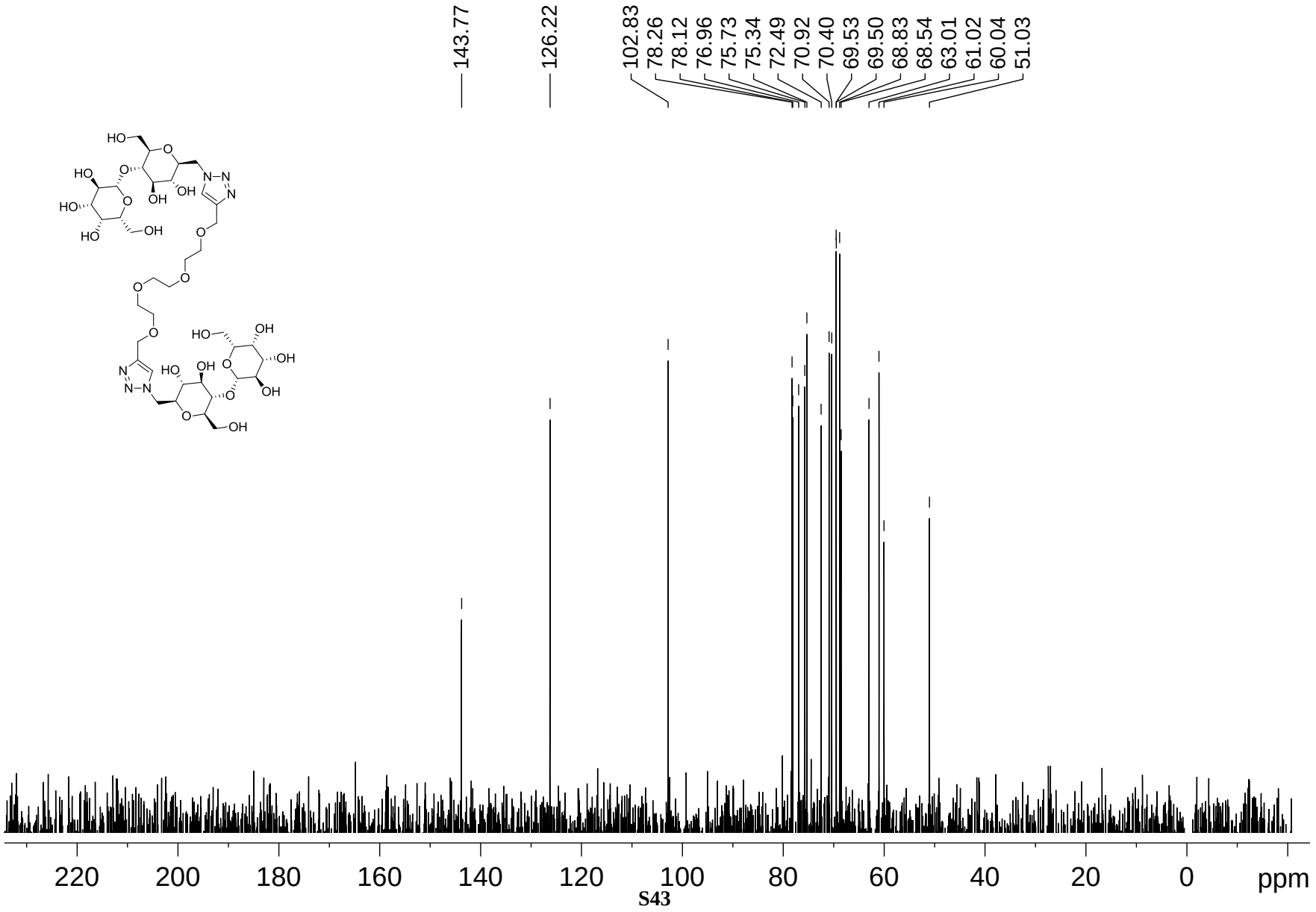
H-H COSY and HSQC (400MHz, $CDCl_3$) spectrum of **7g**



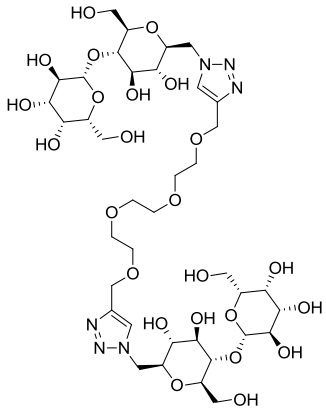
^{1}H NMR (400MHz, D_2O) spectrum of **8g**

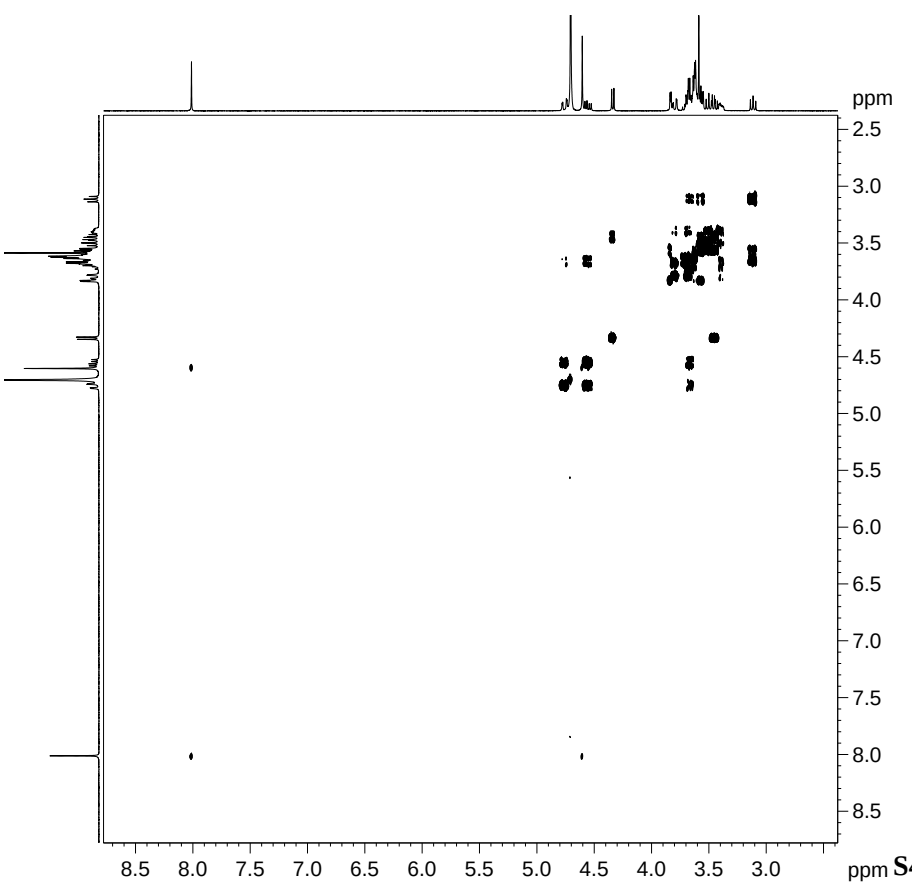


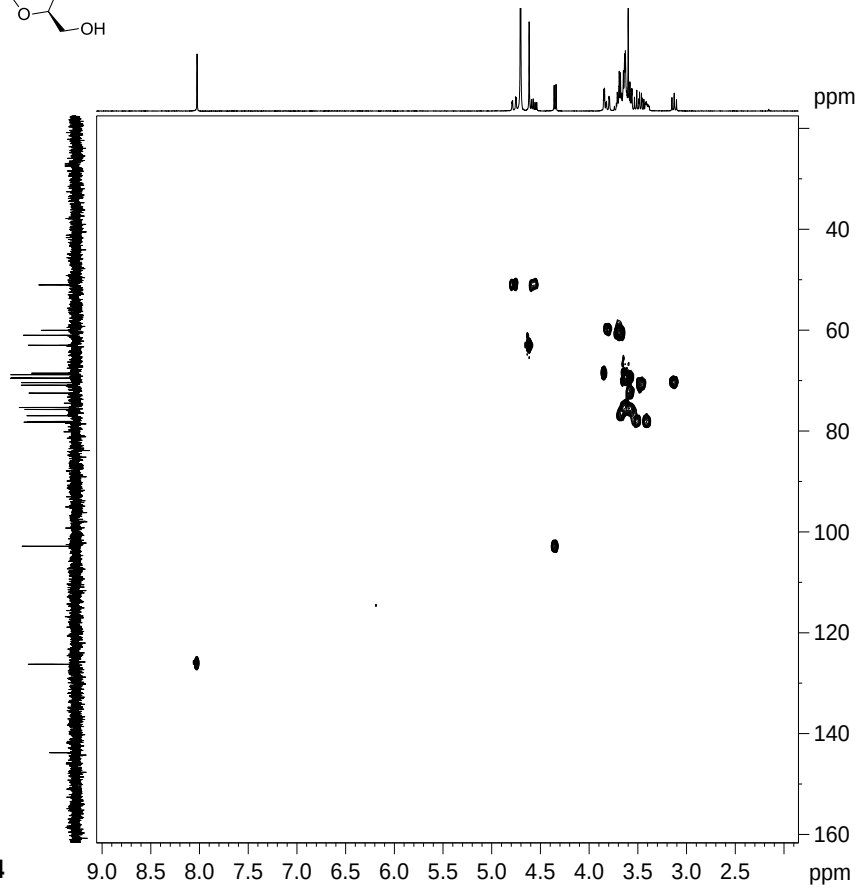
^{13}C NMR (100MHz, D_2O) spectrum of **8g**



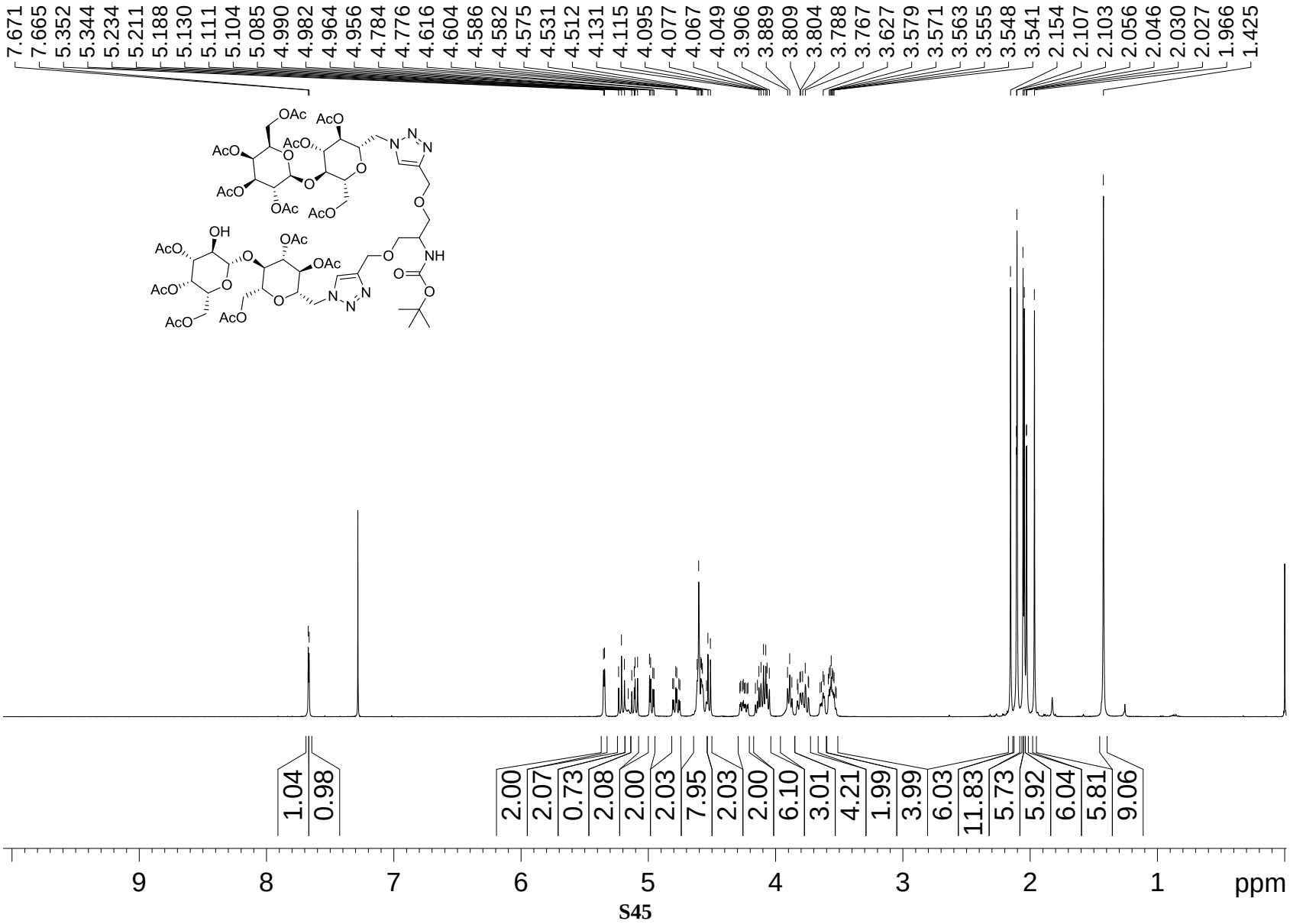
H-H COSY and HSQC (400MHz, D_2O) spectrum of **8g**



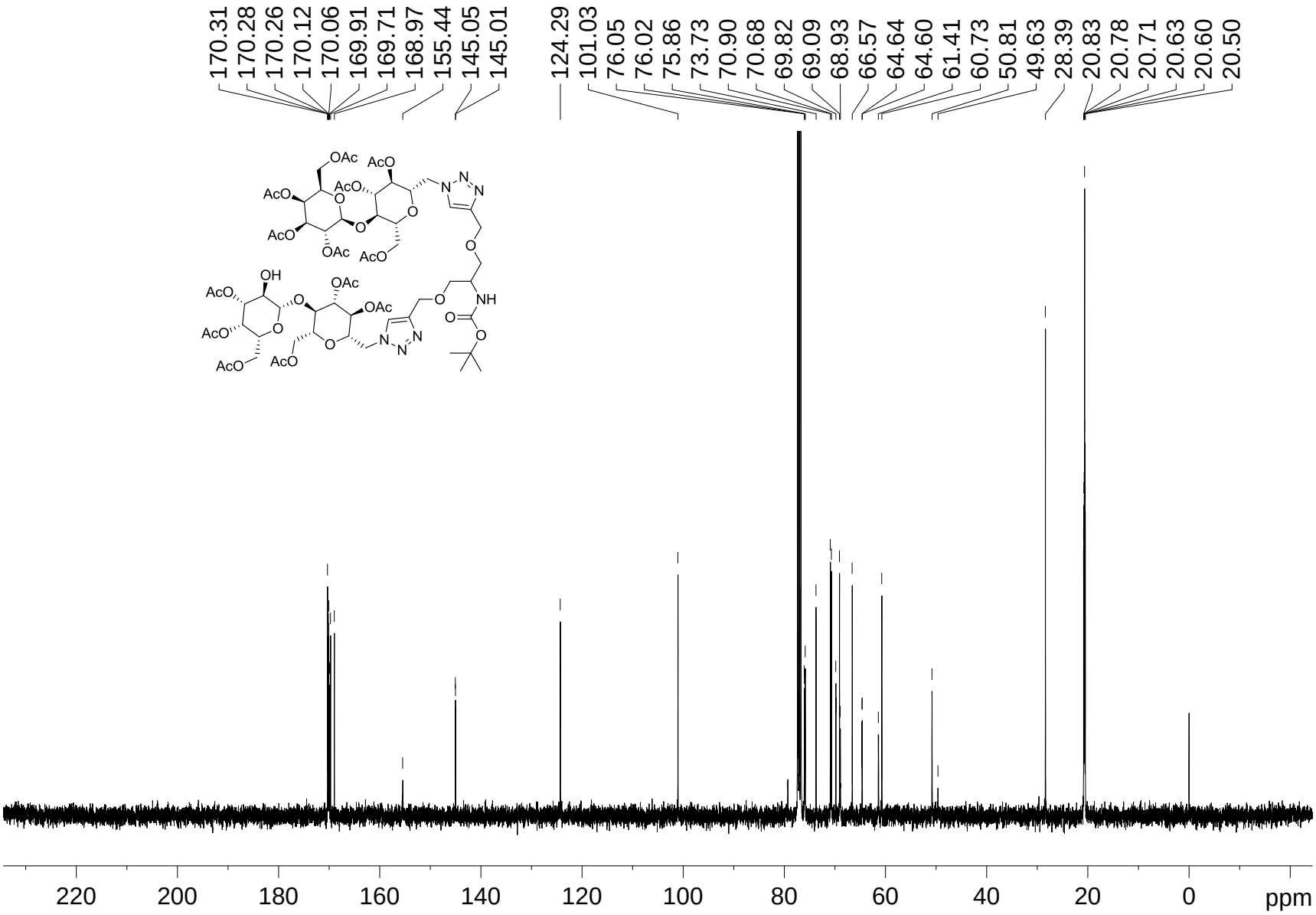




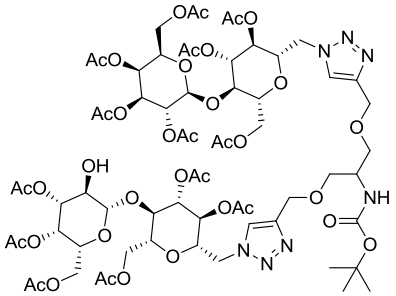
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7e**

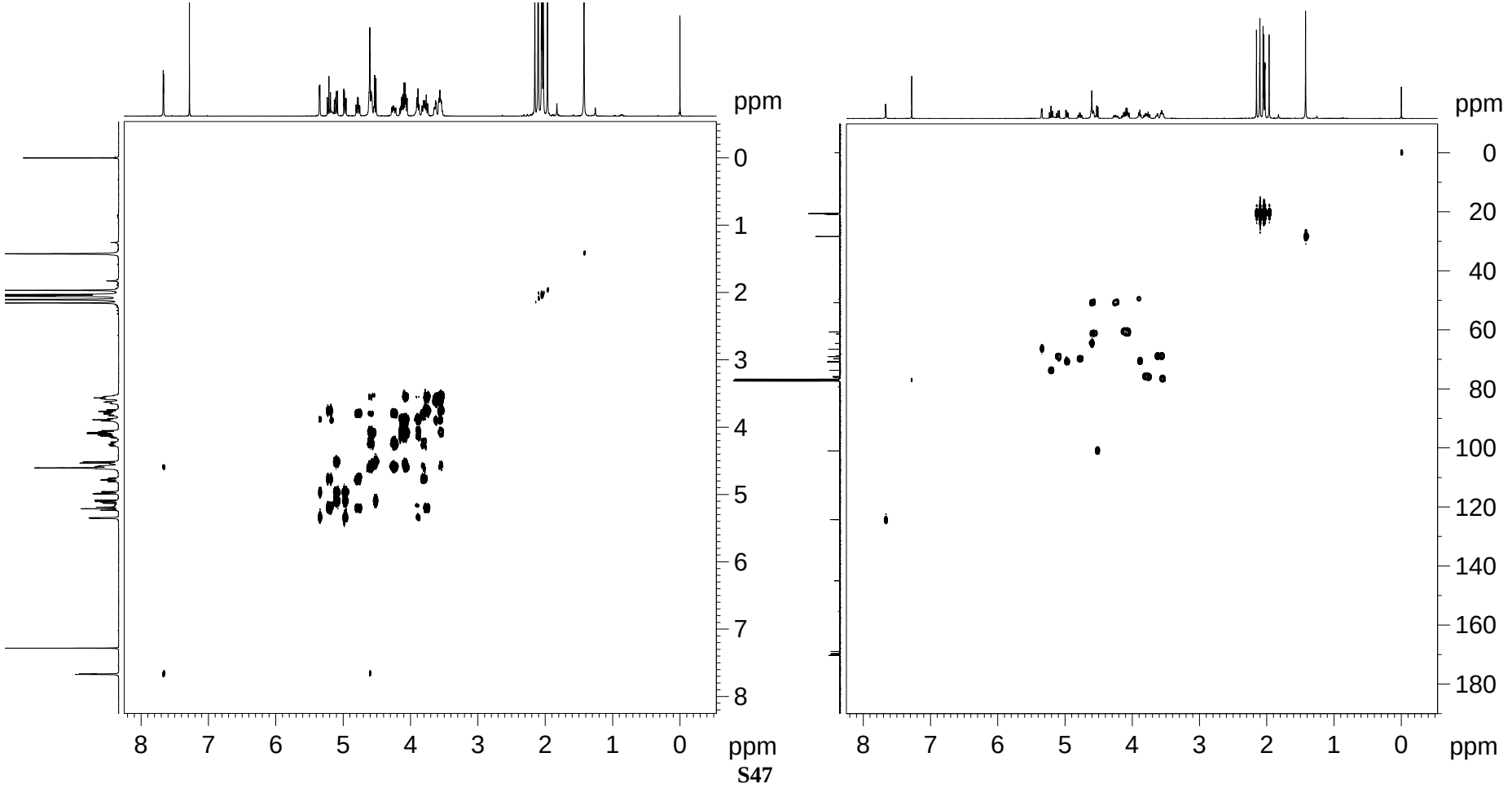


^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7e**

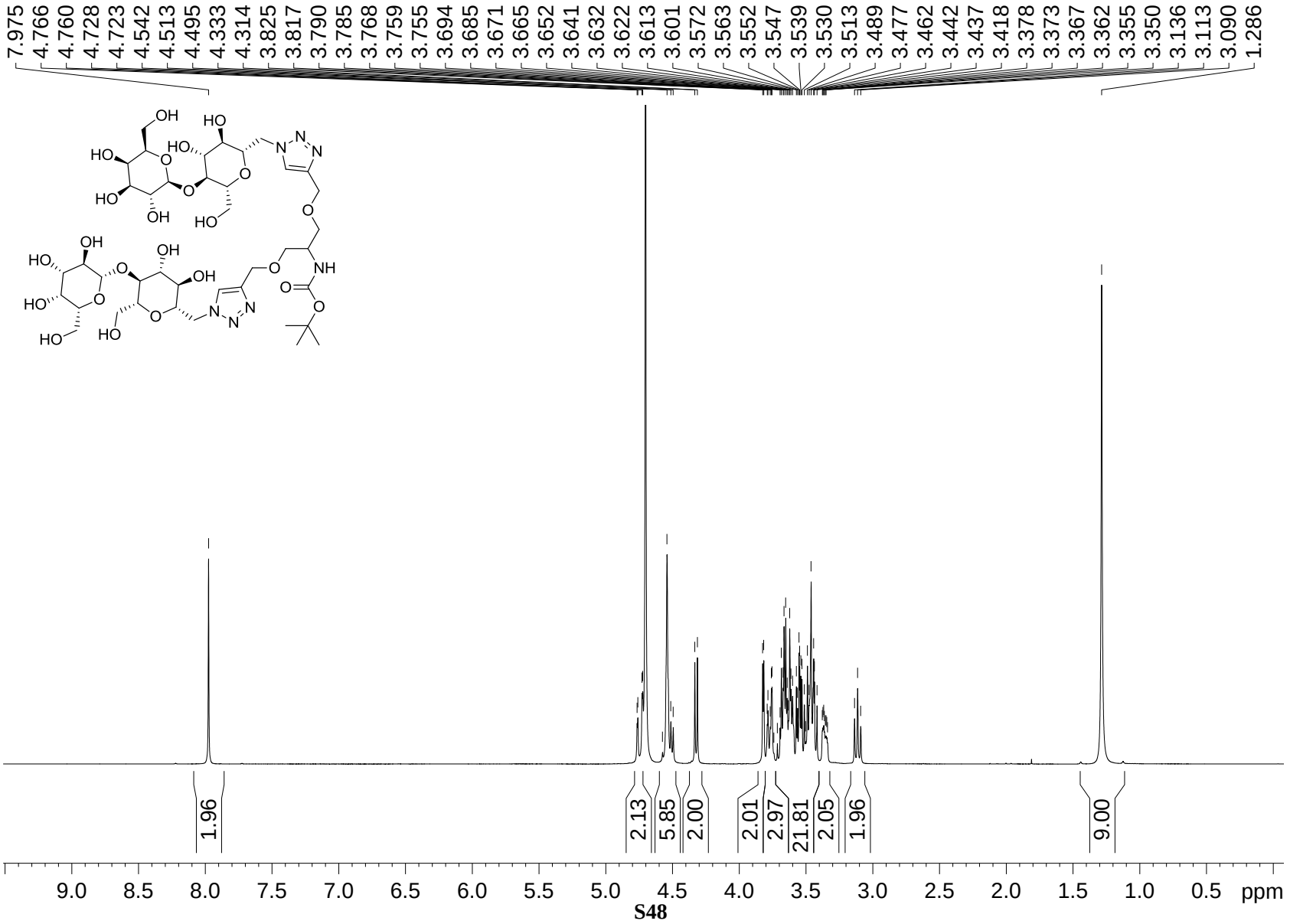


H-H COSY and HSQC (400MHz, $CDCl_3$) spectrum of **7e**

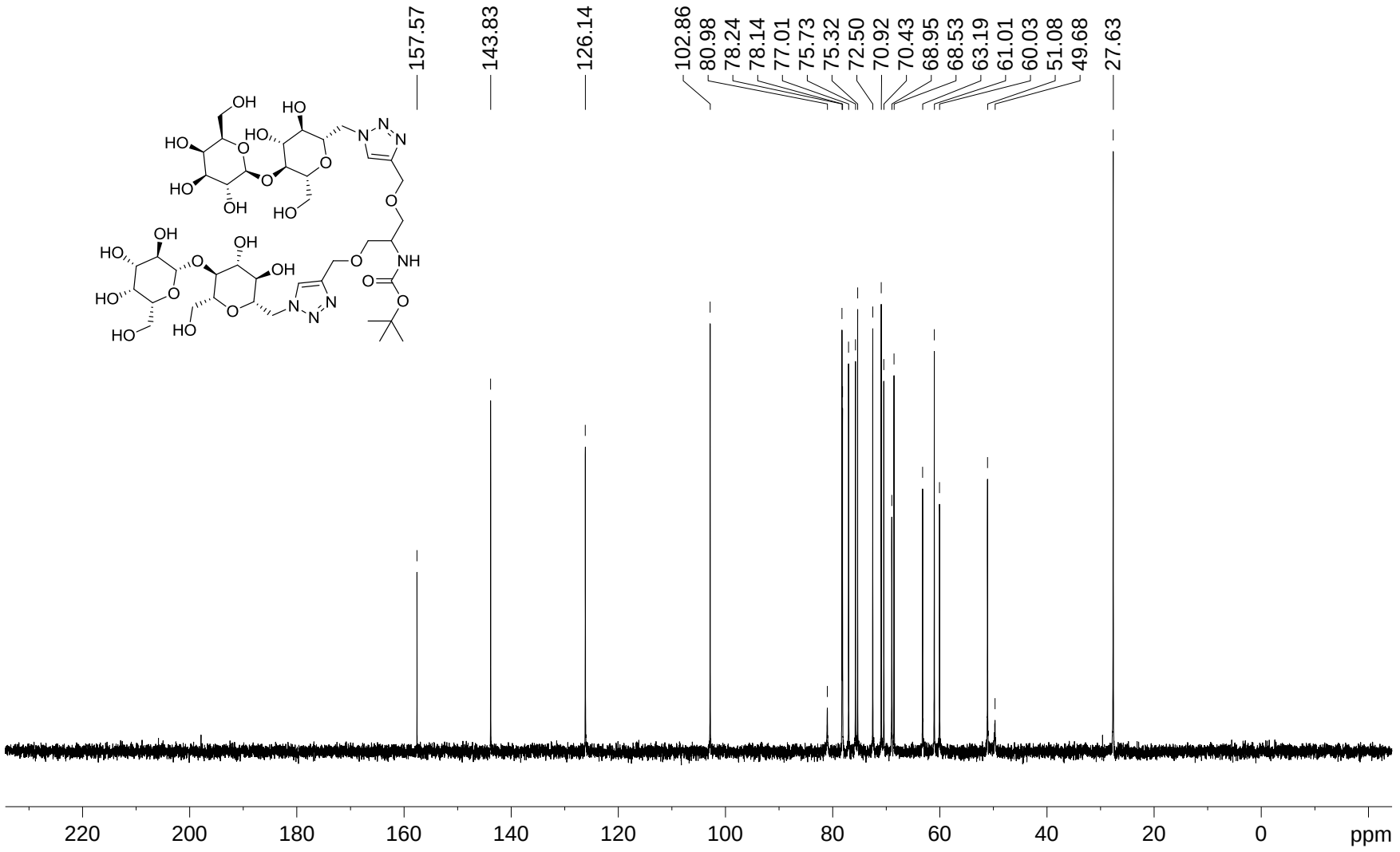




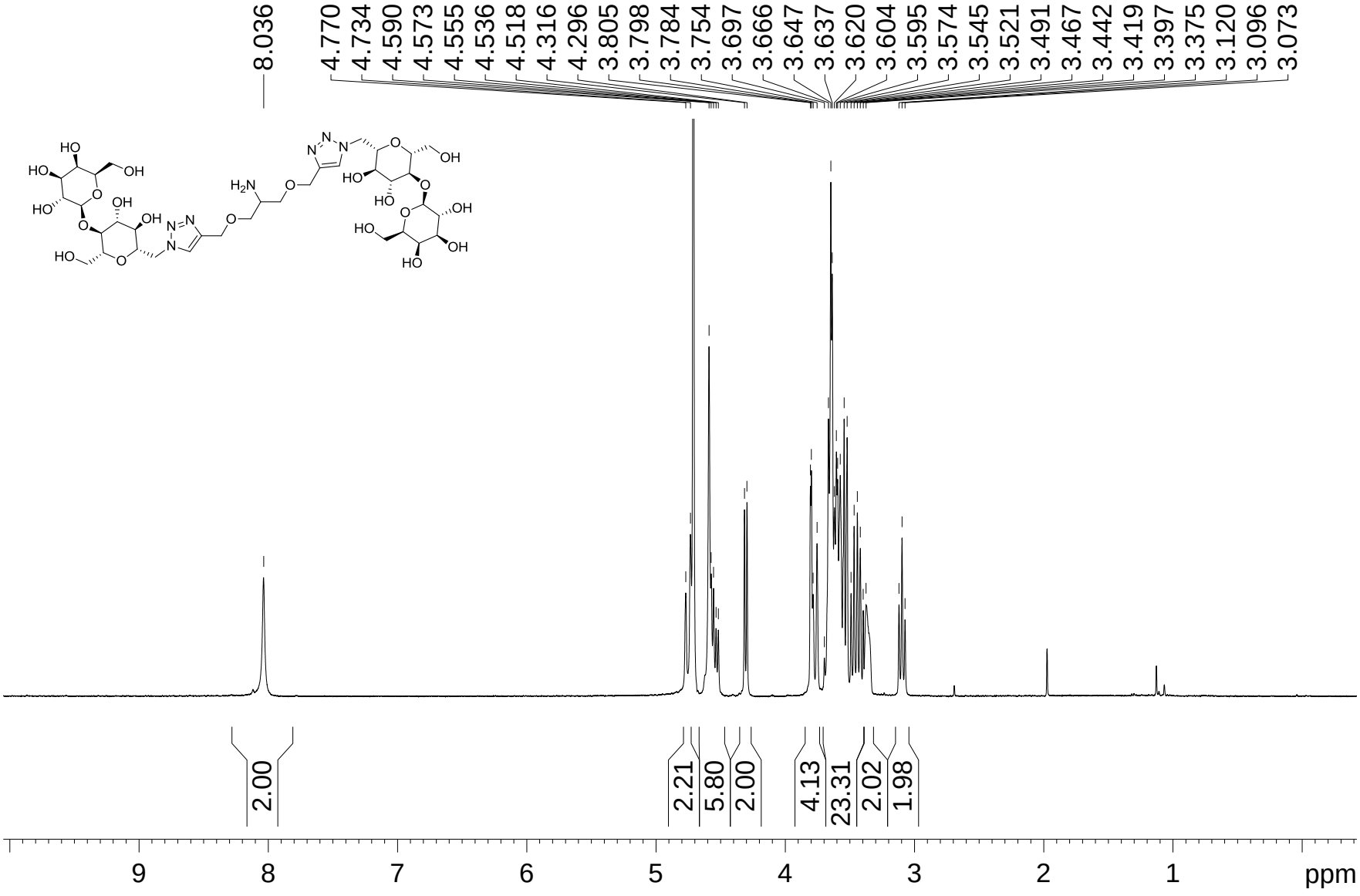
^{1}H NMR (400MHz, D_2O) spectrum of **8e**



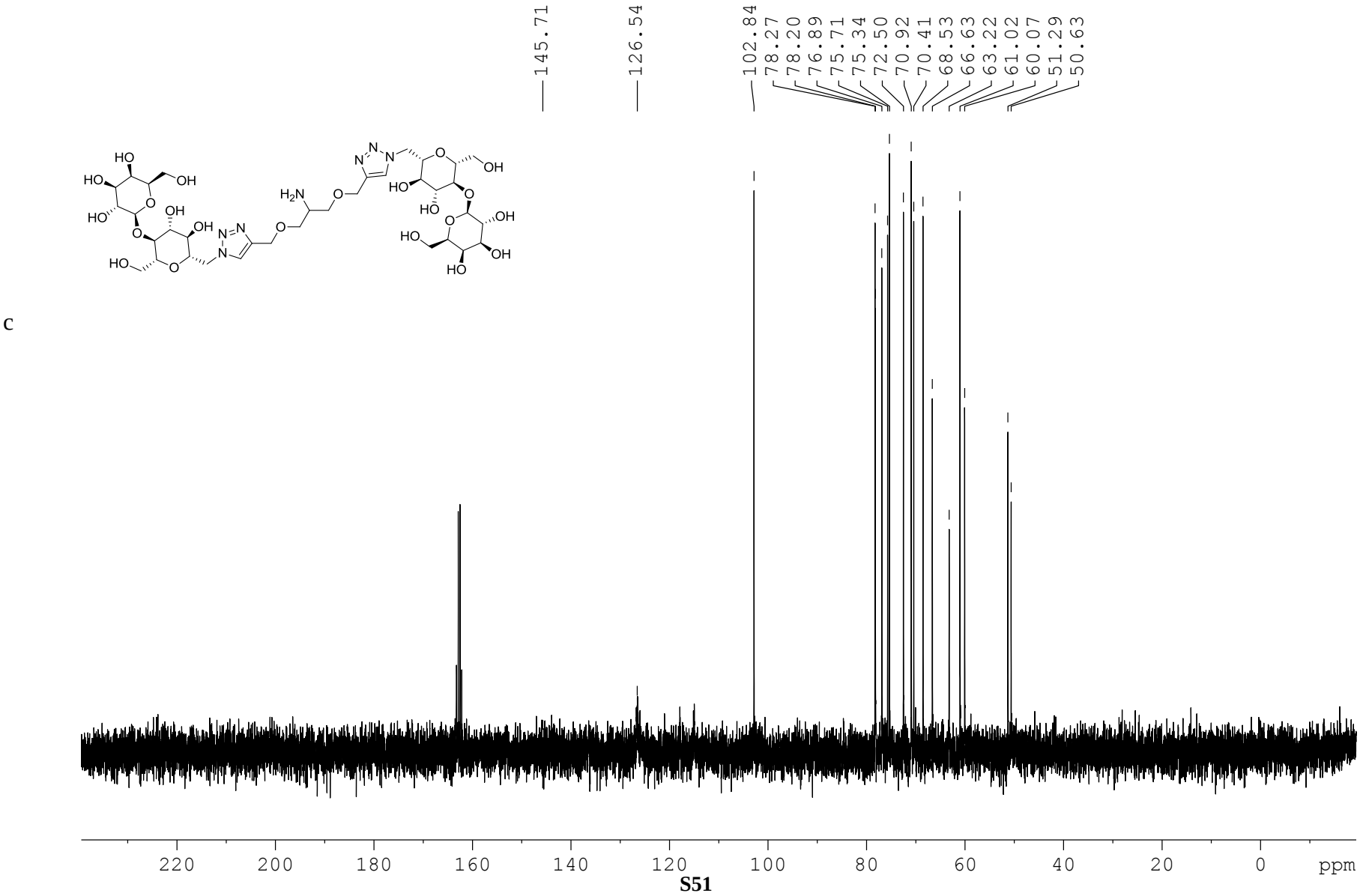
^{13}C NMR (100MHz, D_2O) spectrum of **8e**



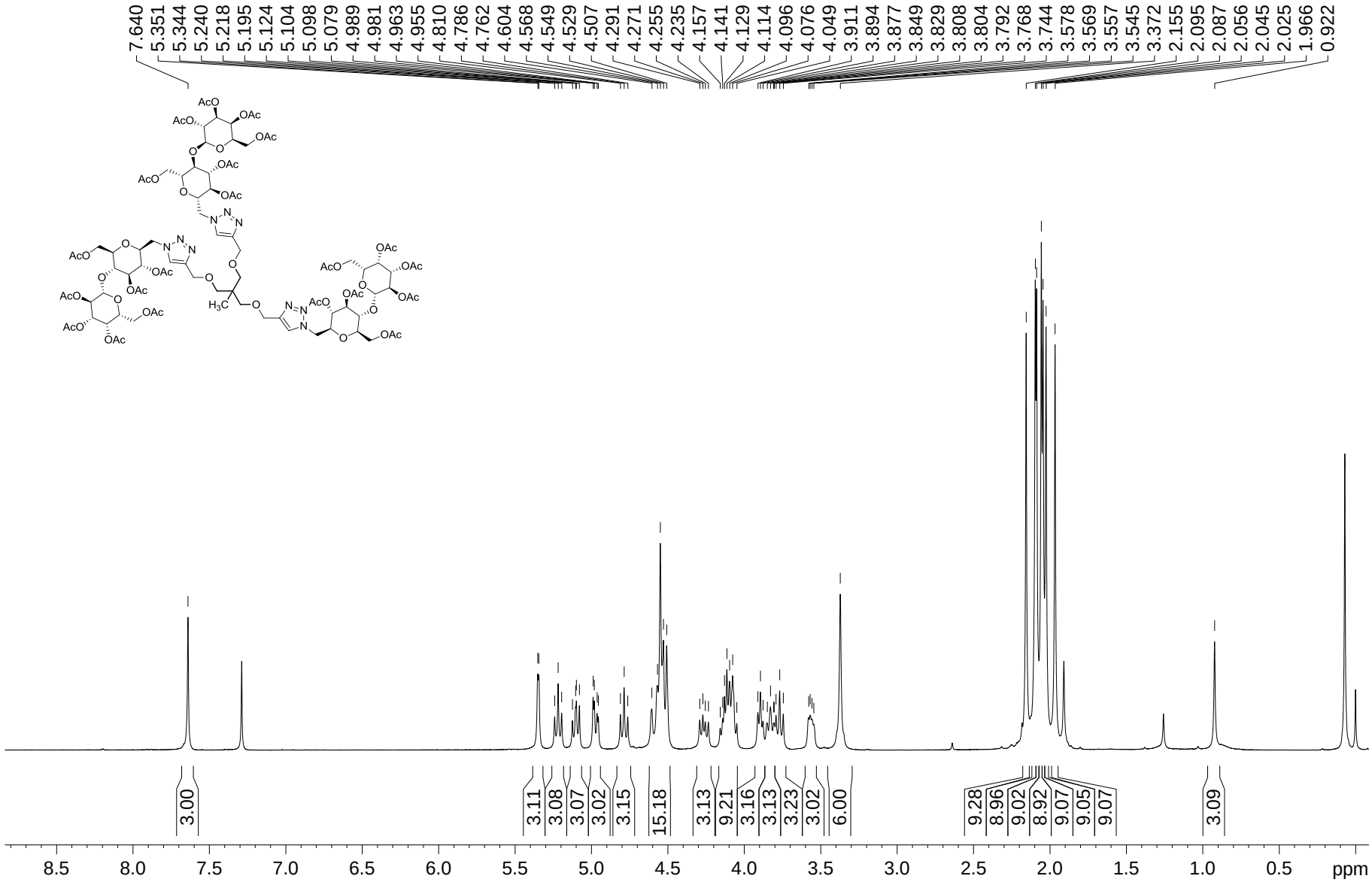
^{1}H NMR (400MHz, D_2O, TFA salt) spectrum of **8q**



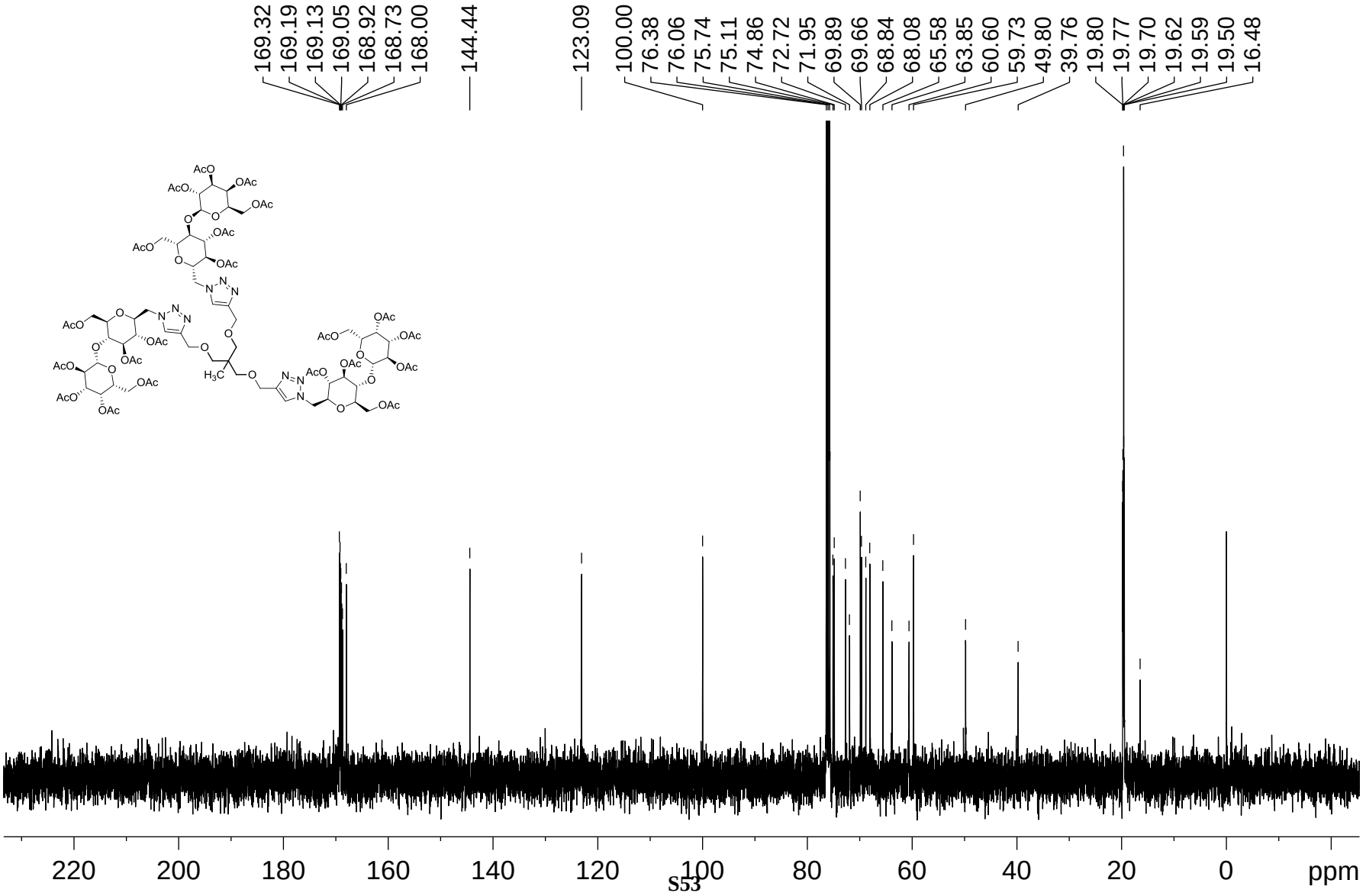
^{13}C NMR (100MHz, D_2O, TFA salt) spectrum of **8q**



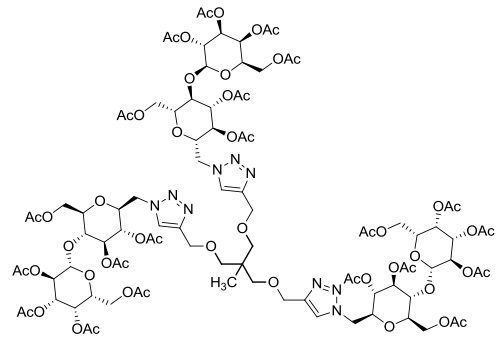
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7h**

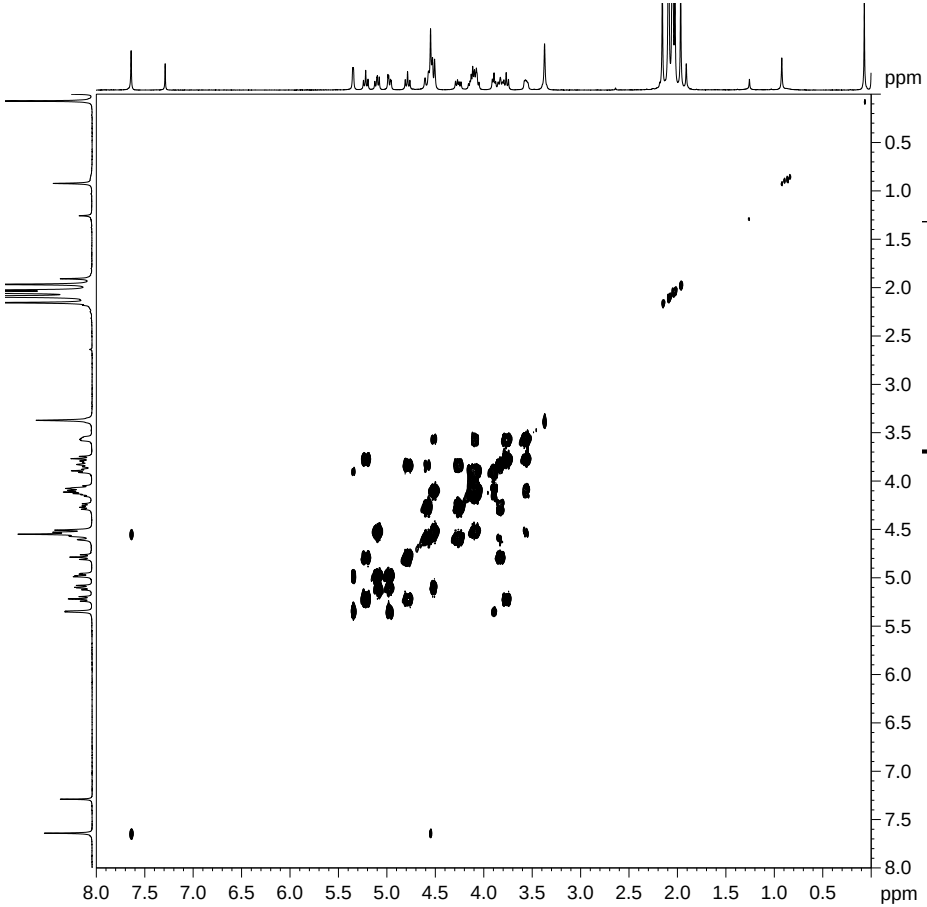


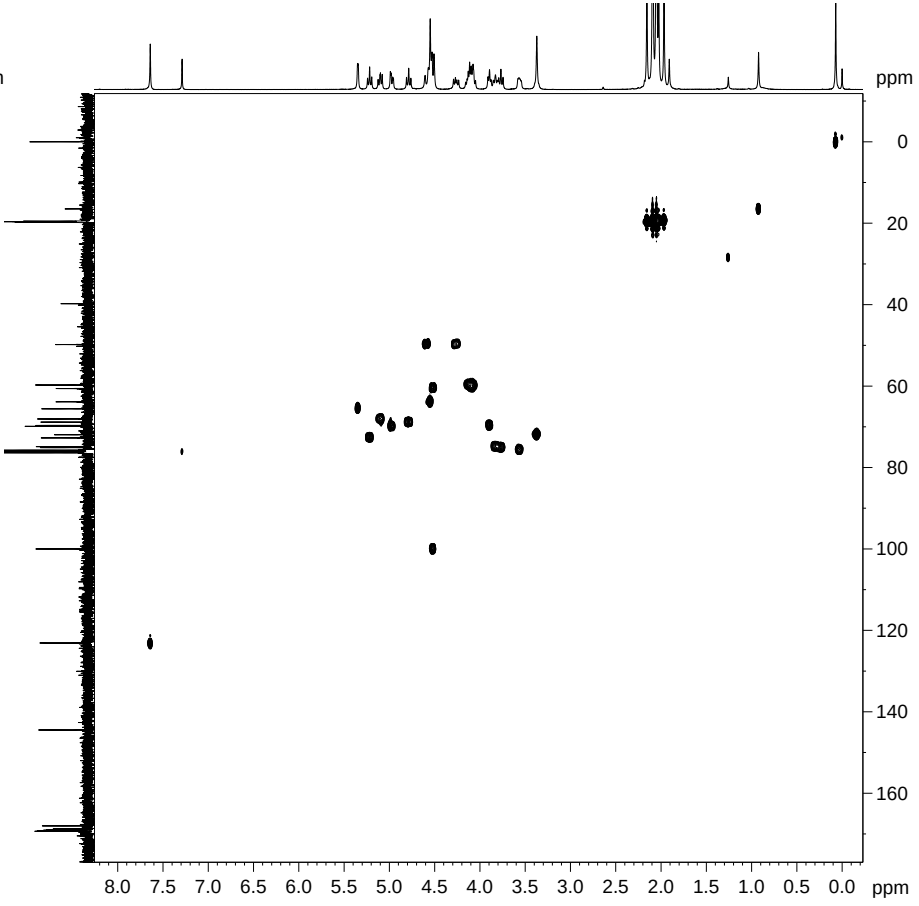
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7h**



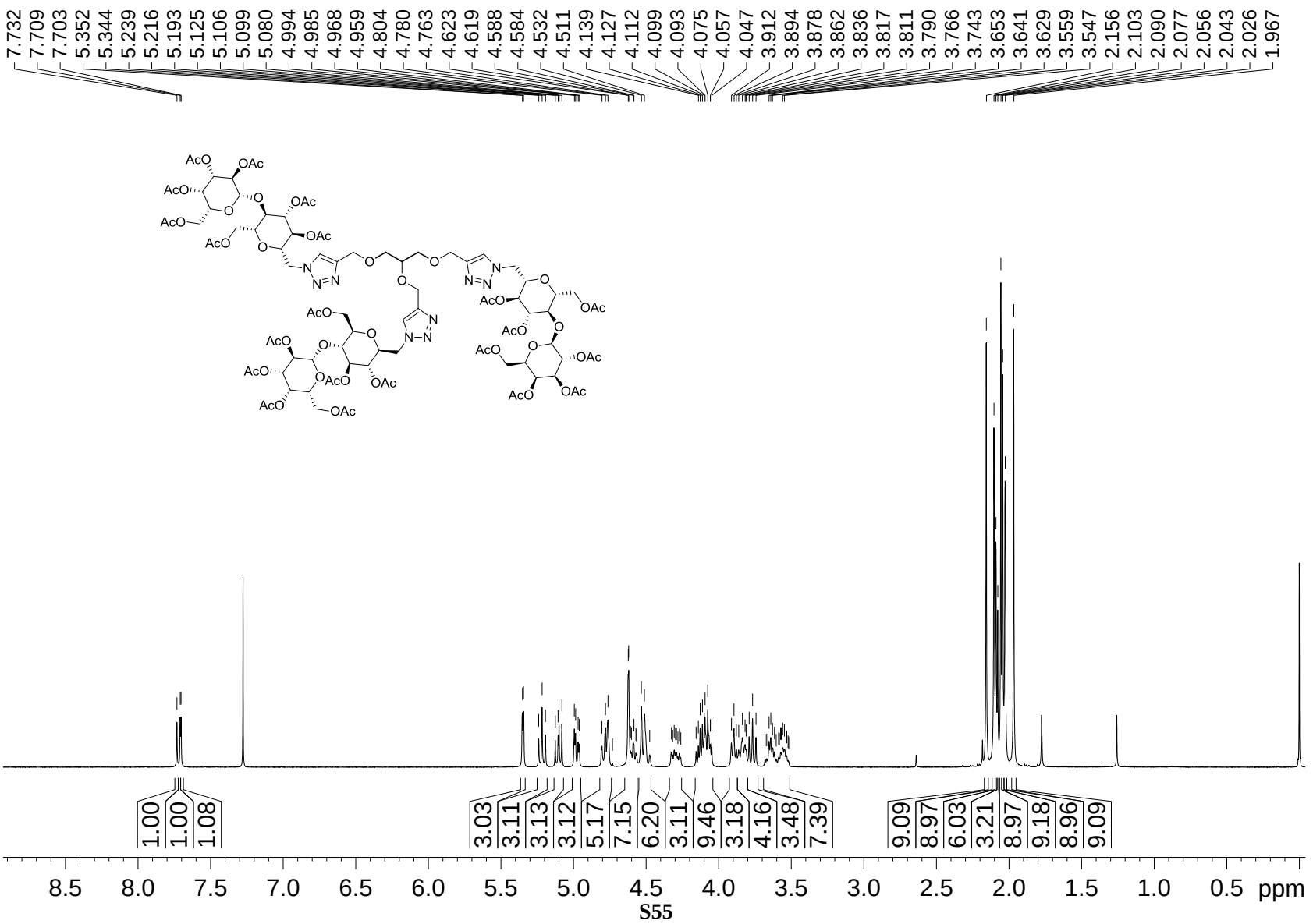
H-H COSY and HSQC (400MHz, $CDCl_3$) spectrum of **7h**



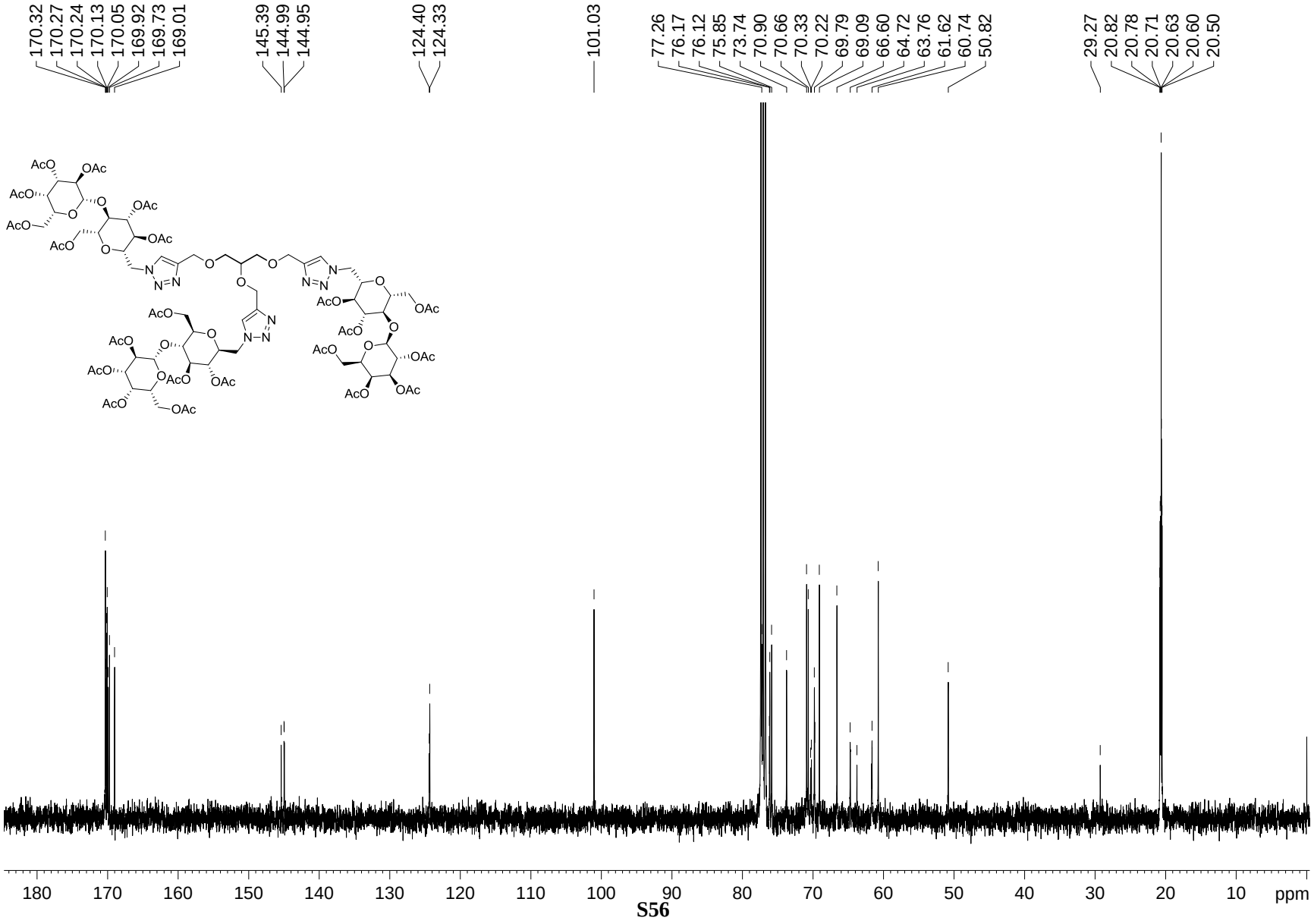




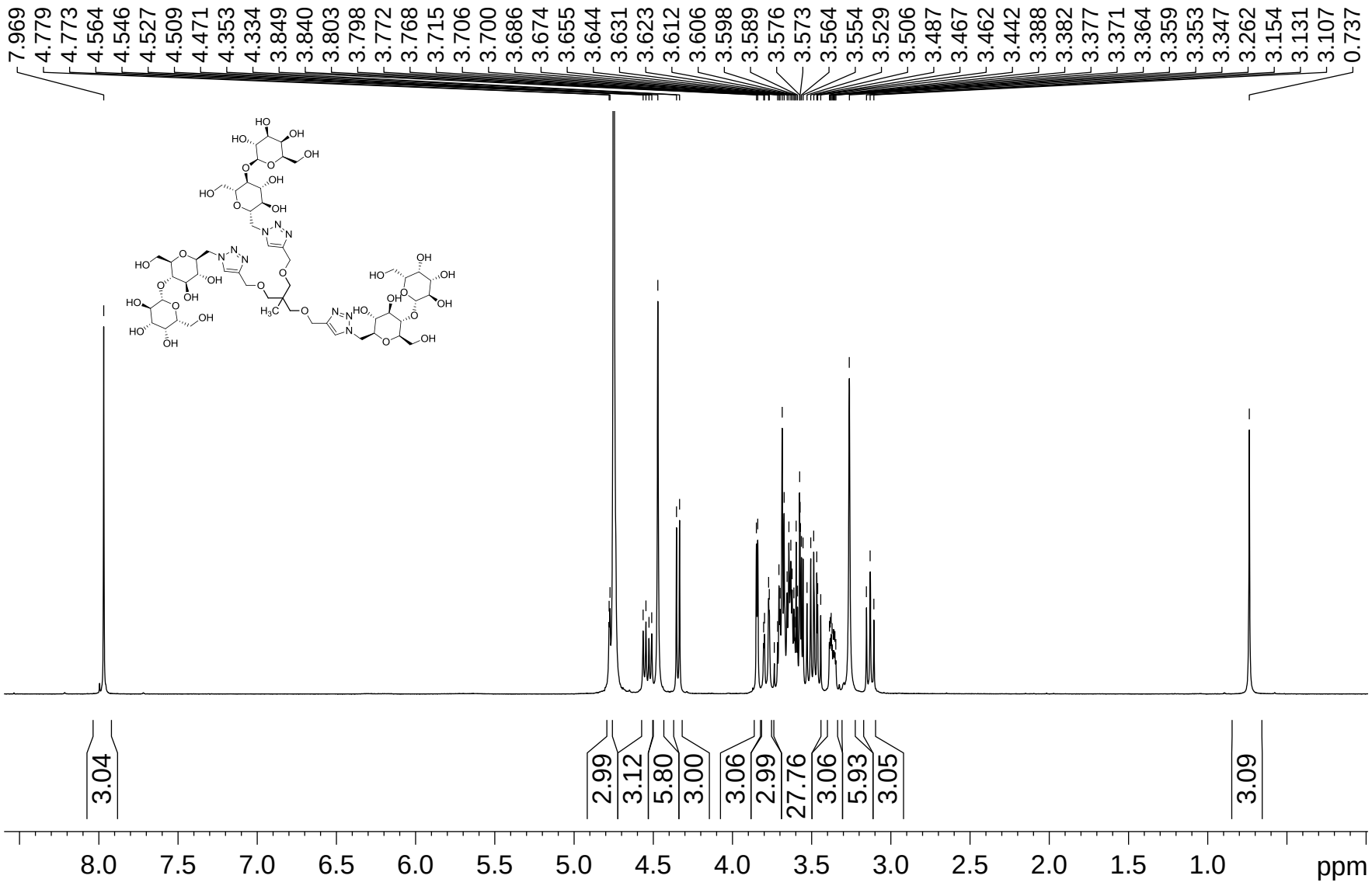
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7i**



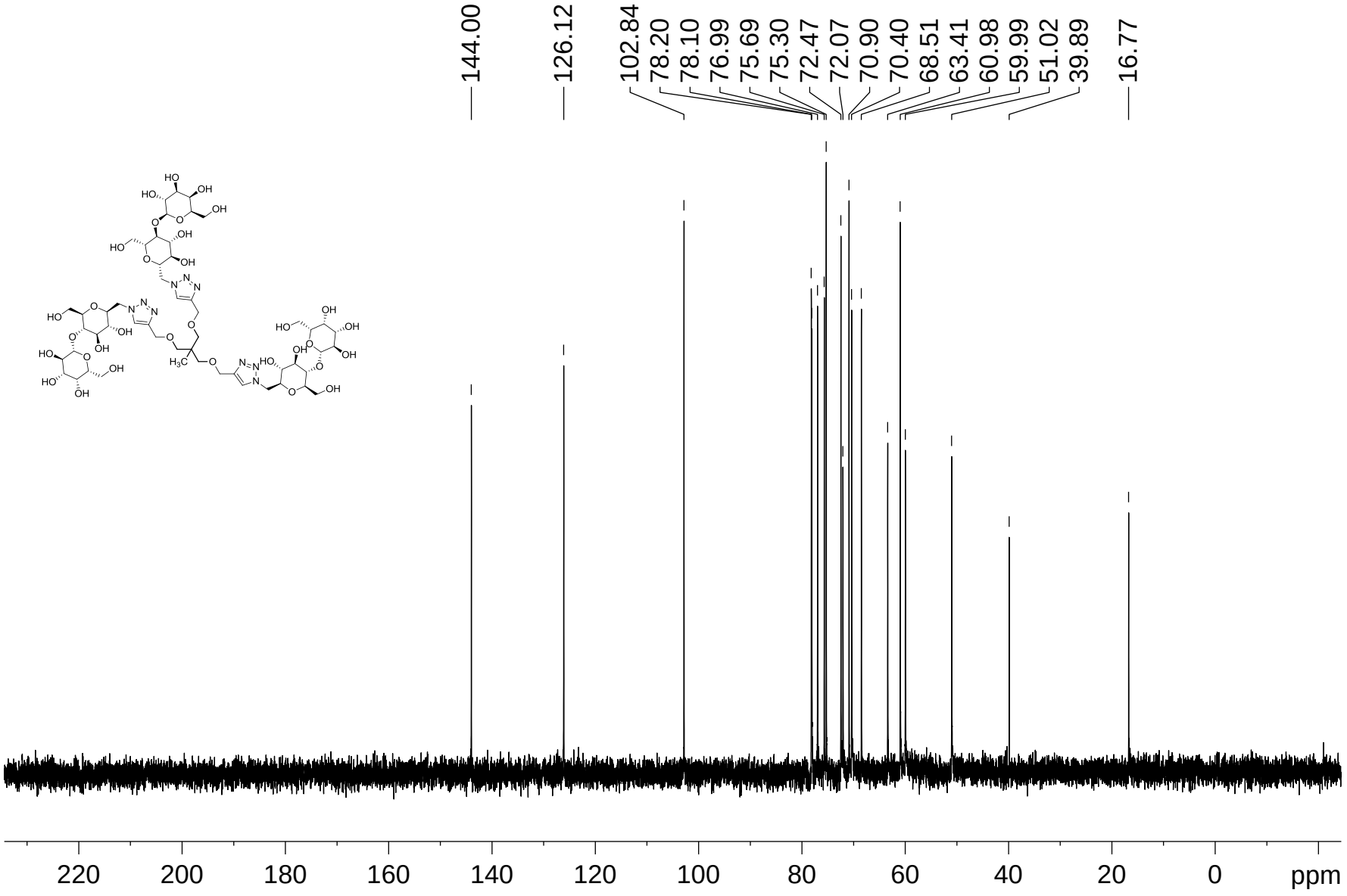
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7i**



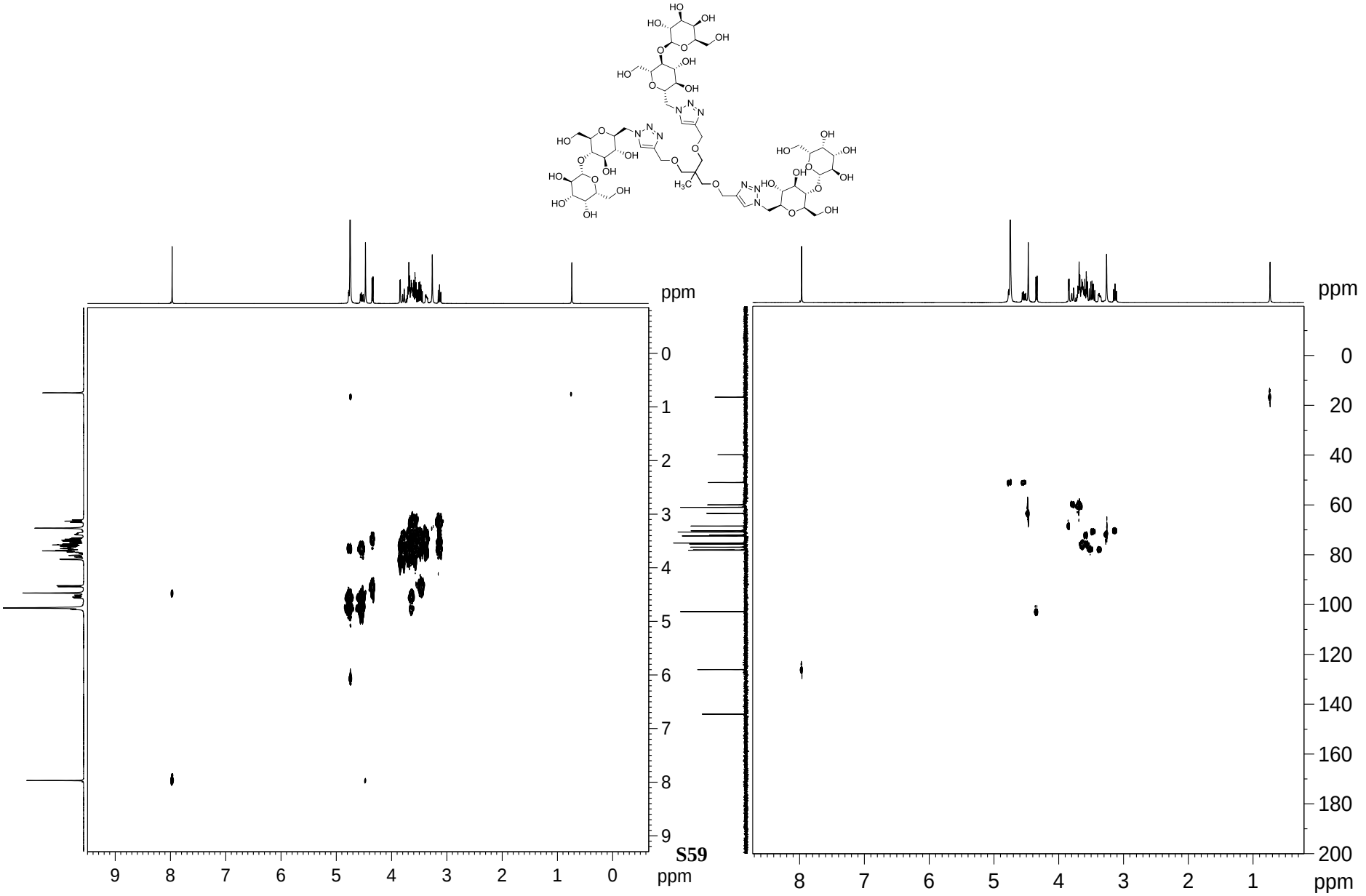
^{1}H NMR (400MHz, D_2O) spectrum of **8h**



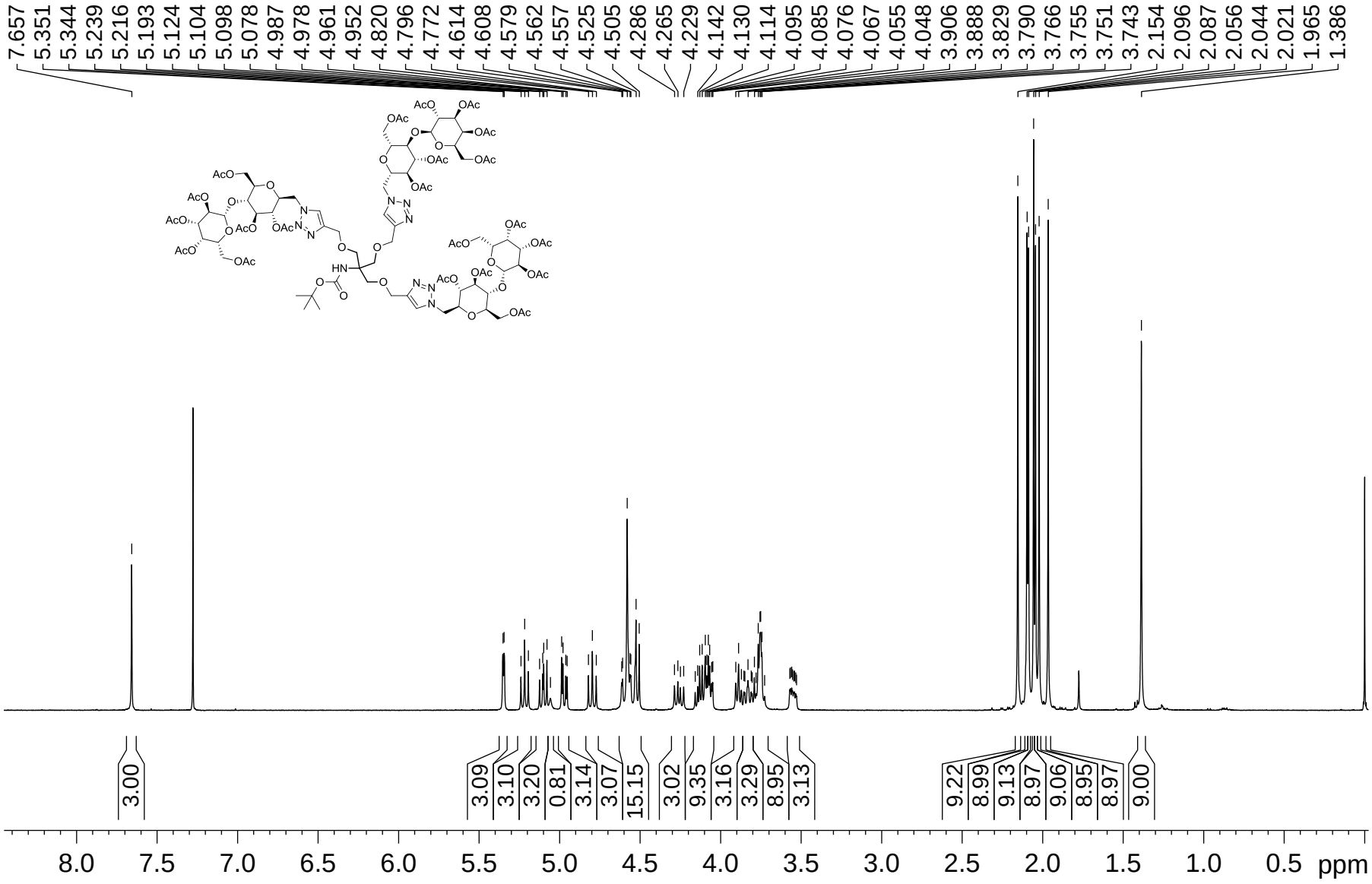
^{13}C NMR (100MHz, D_2O) spectrum of **8h**



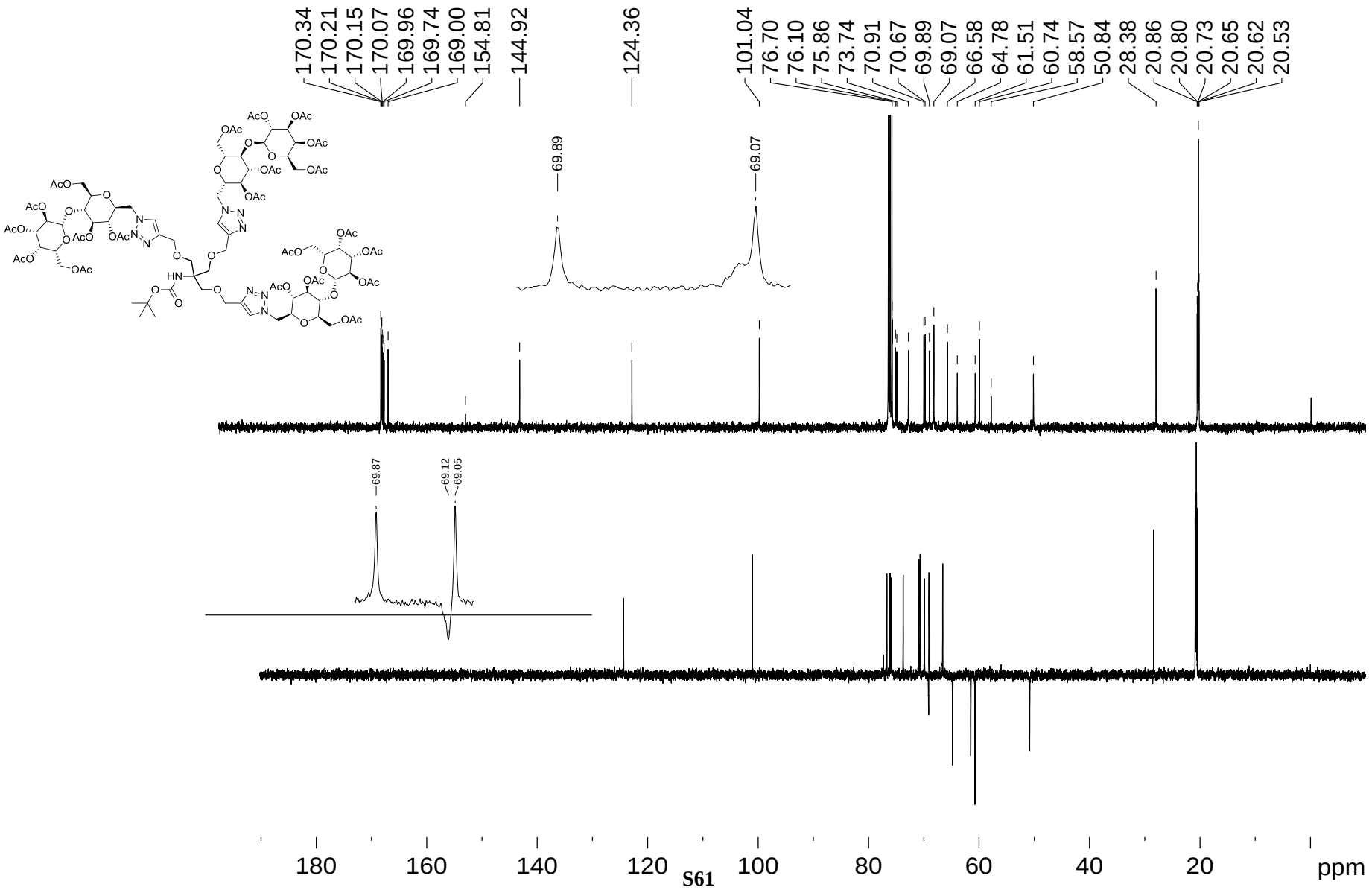
H-H COSY and HSQC (400MHz, D_2O) spectrum of **8h**



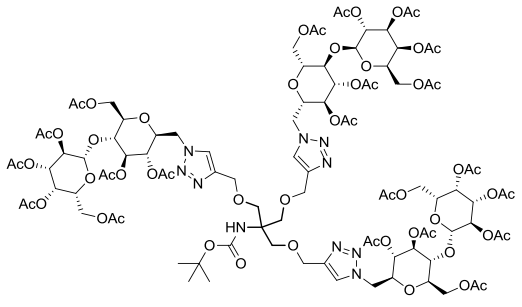
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7j**

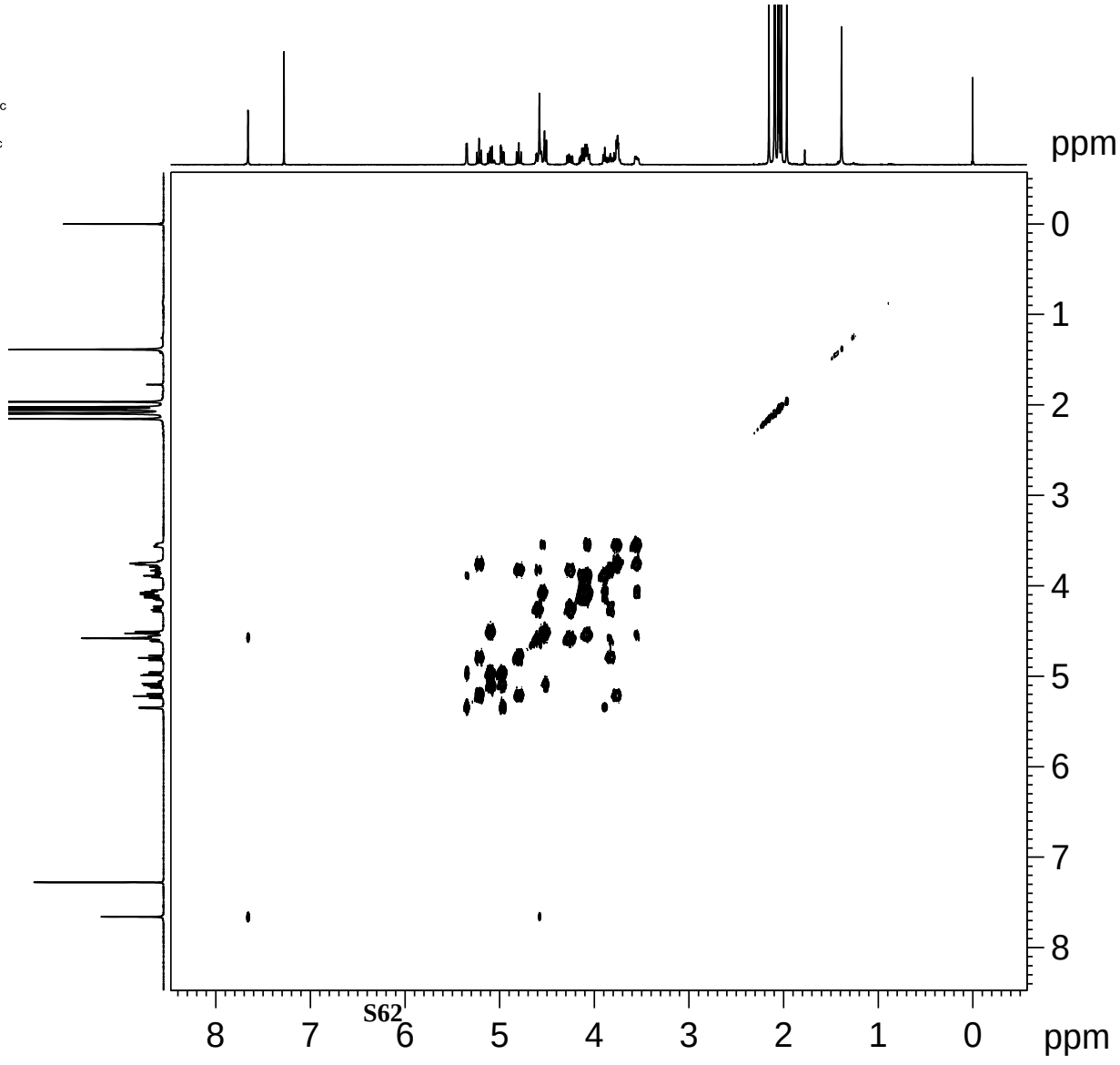


^{13}C NMR and DEPT135 (100MHz, $CDCl_3$) spectrum of **7j**

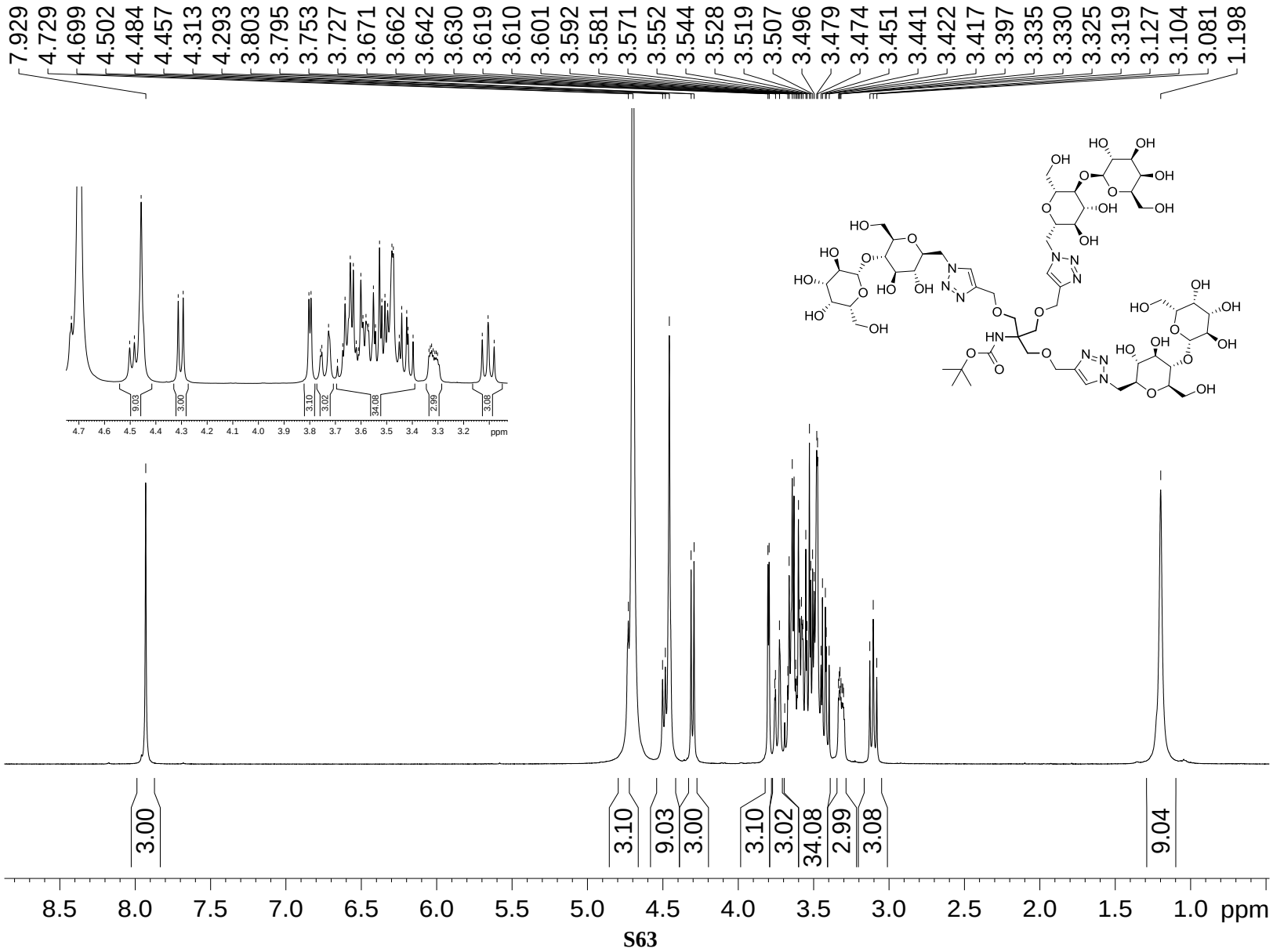


H-H COSY (400MHz, $CDCl_3$) spectrum of **7j**

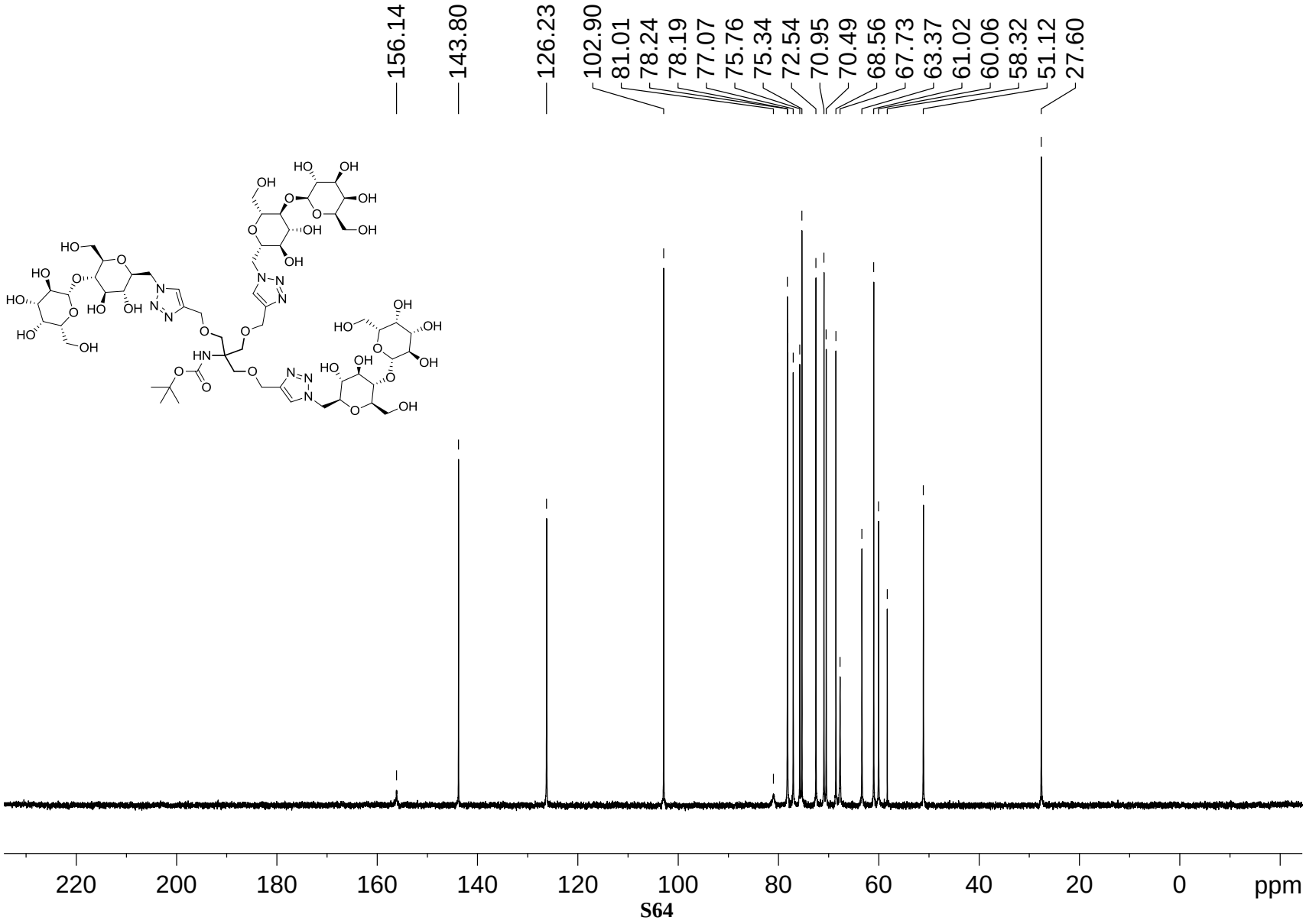




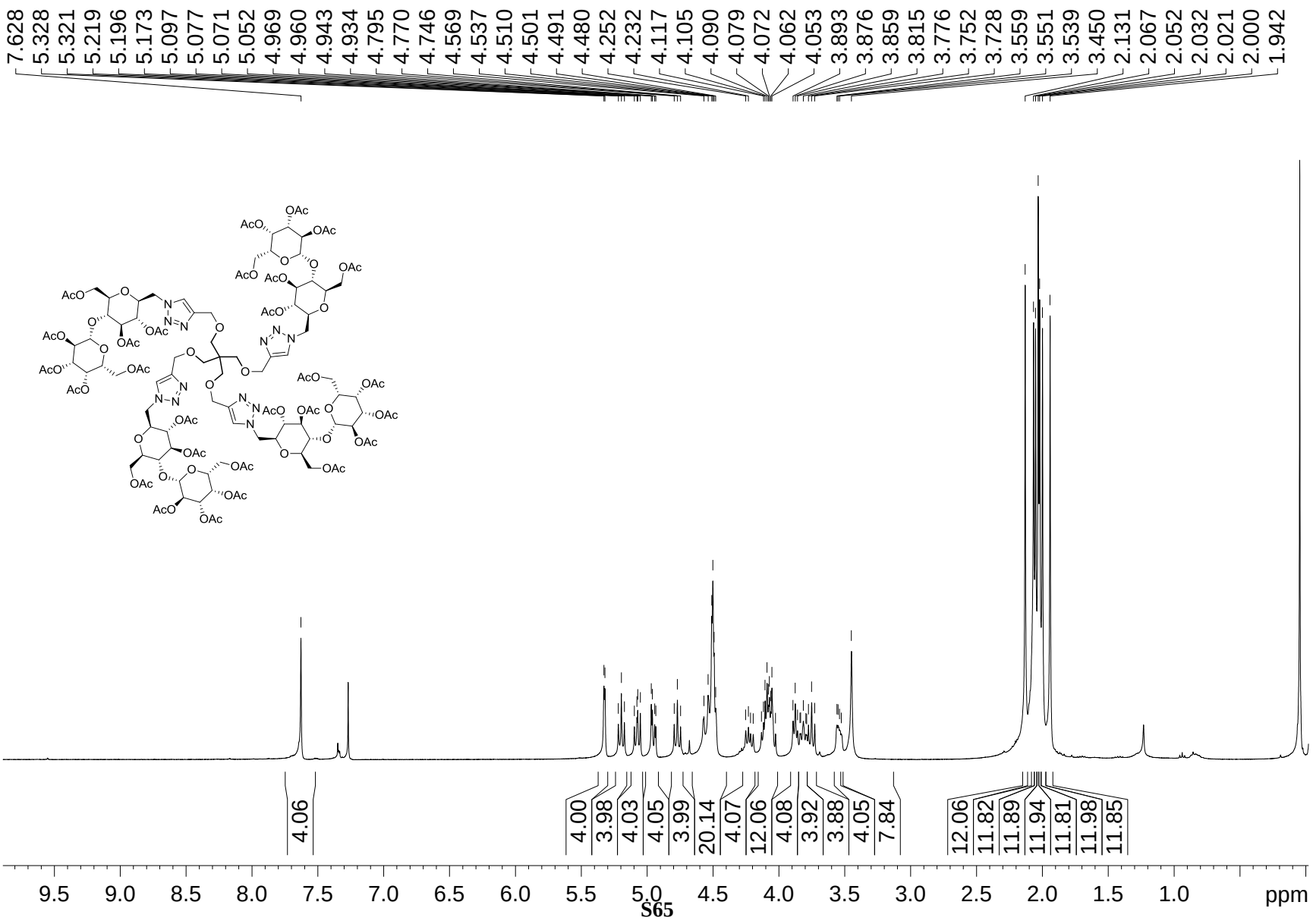
^{1}H NMR (400MHz, D_2O) spectrum of **8j**



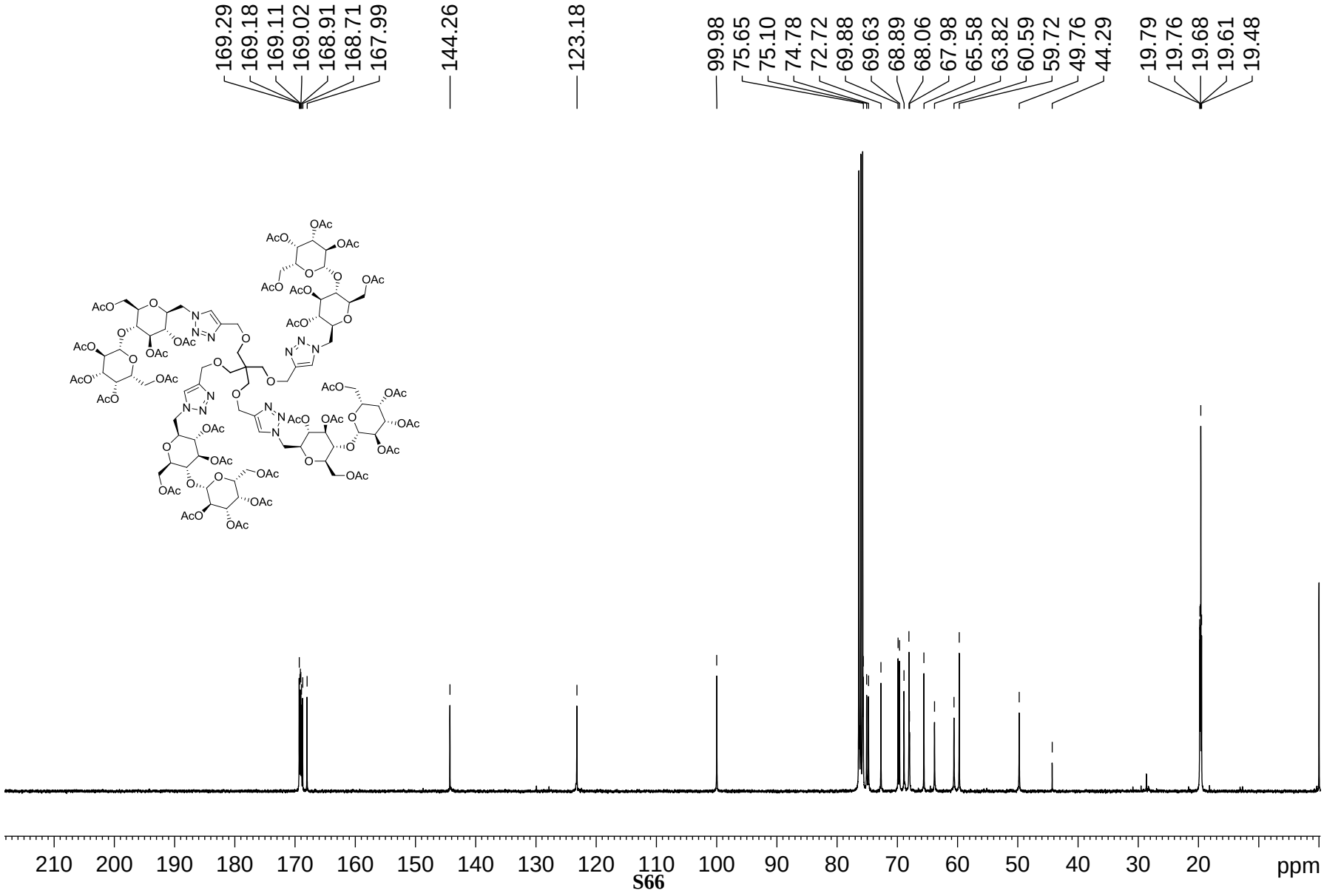
^{13}C NMR (400MHz, D_2O) spectrum of **8j**



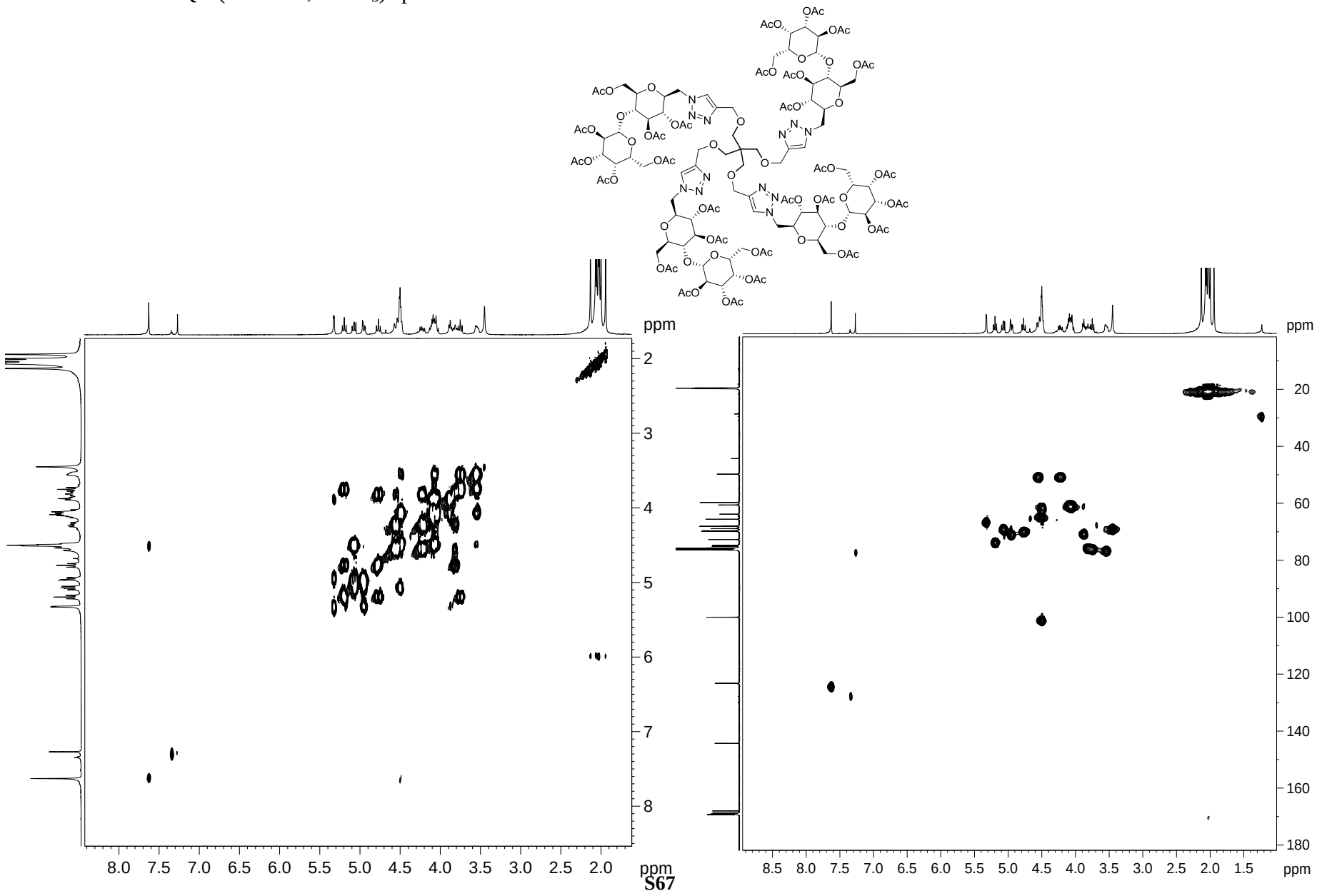
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7k**



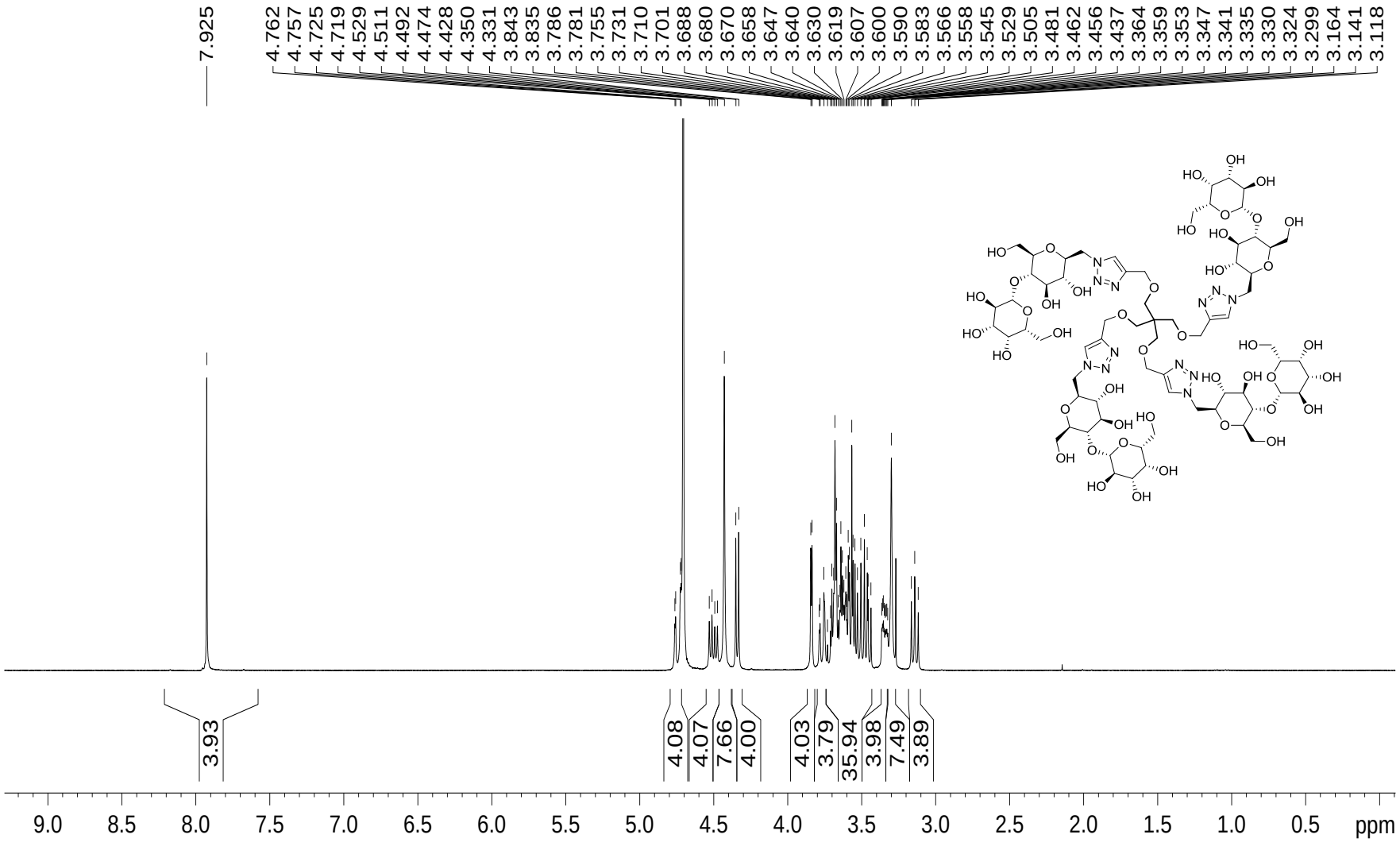
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7k**



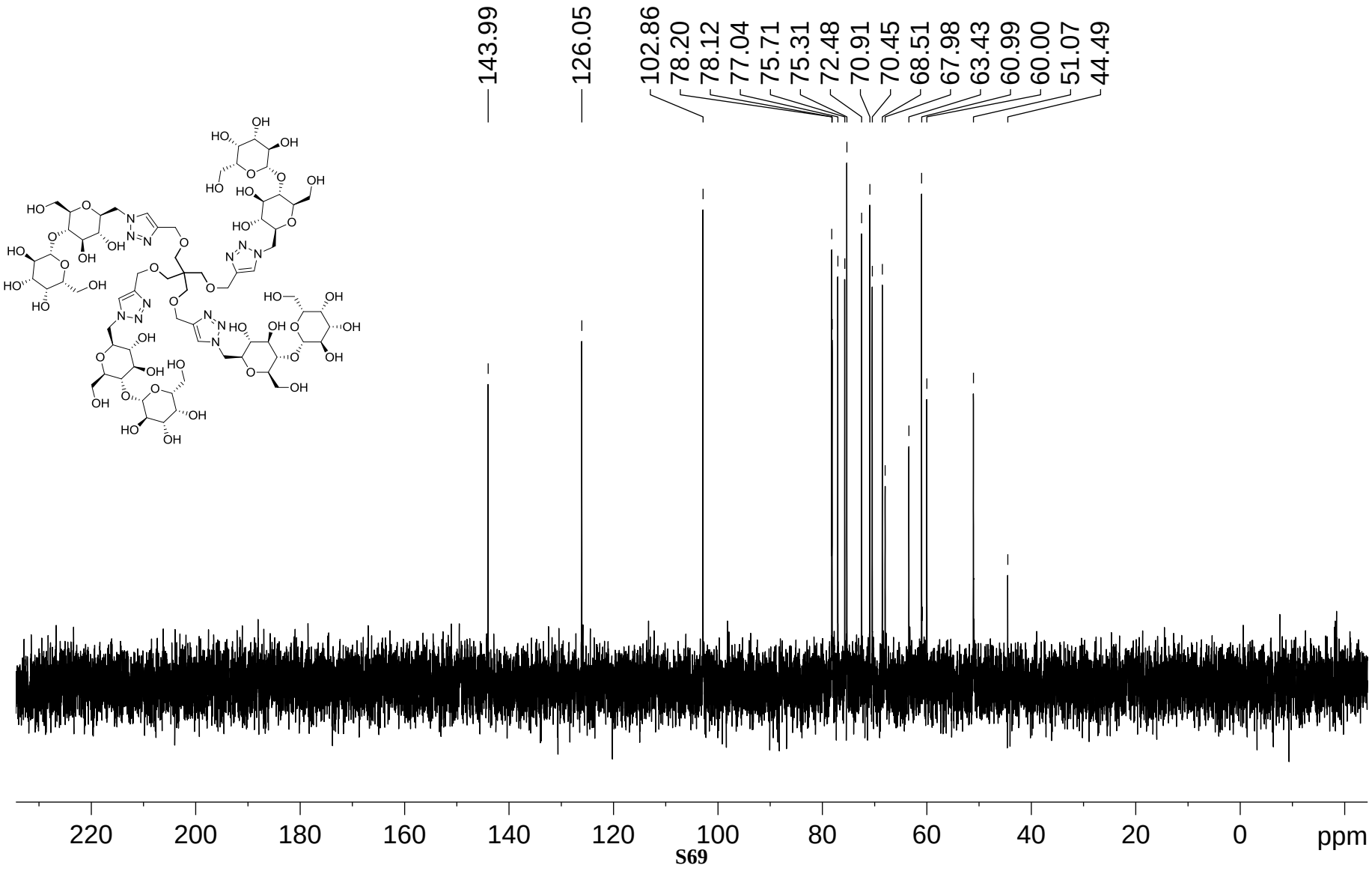
H-H COSY and HSQC (400MHz, $CDCl_3$) spectrum of **7k**



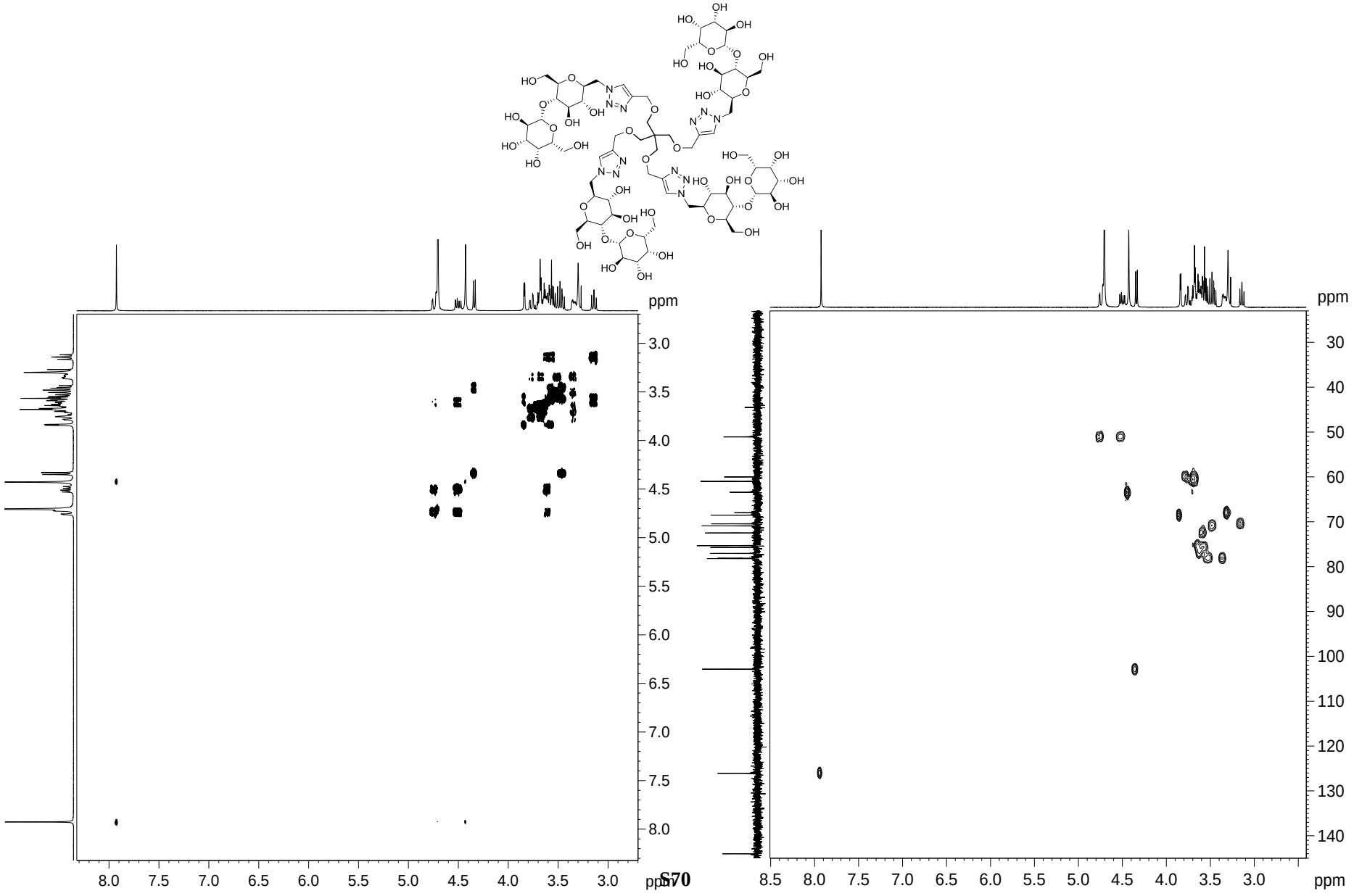
^{1}H NMR (400MHz, D_2O) spectrum of **8k**



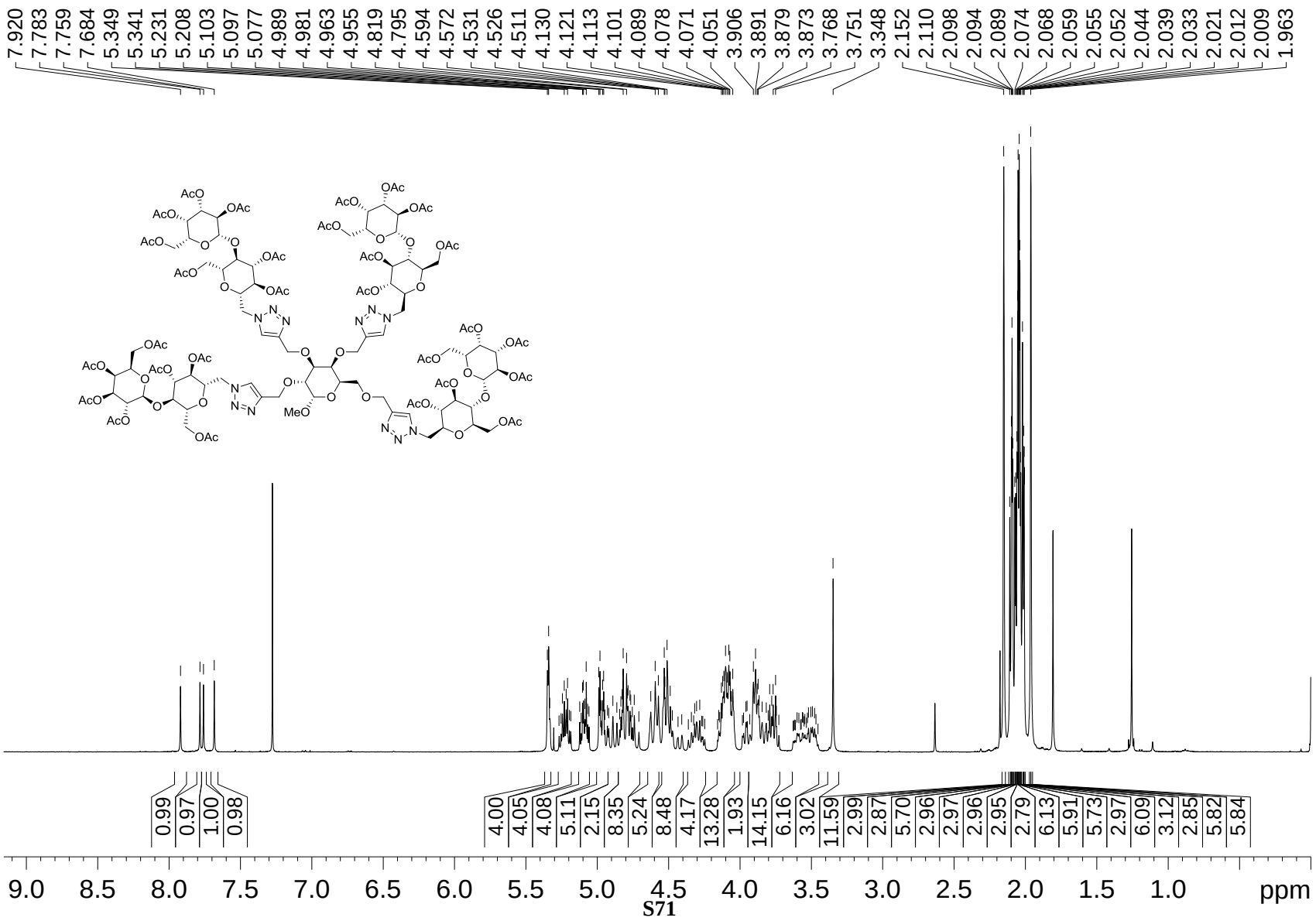
^{13}C NMR (100MHz, D_2O) spectrum of **8k**



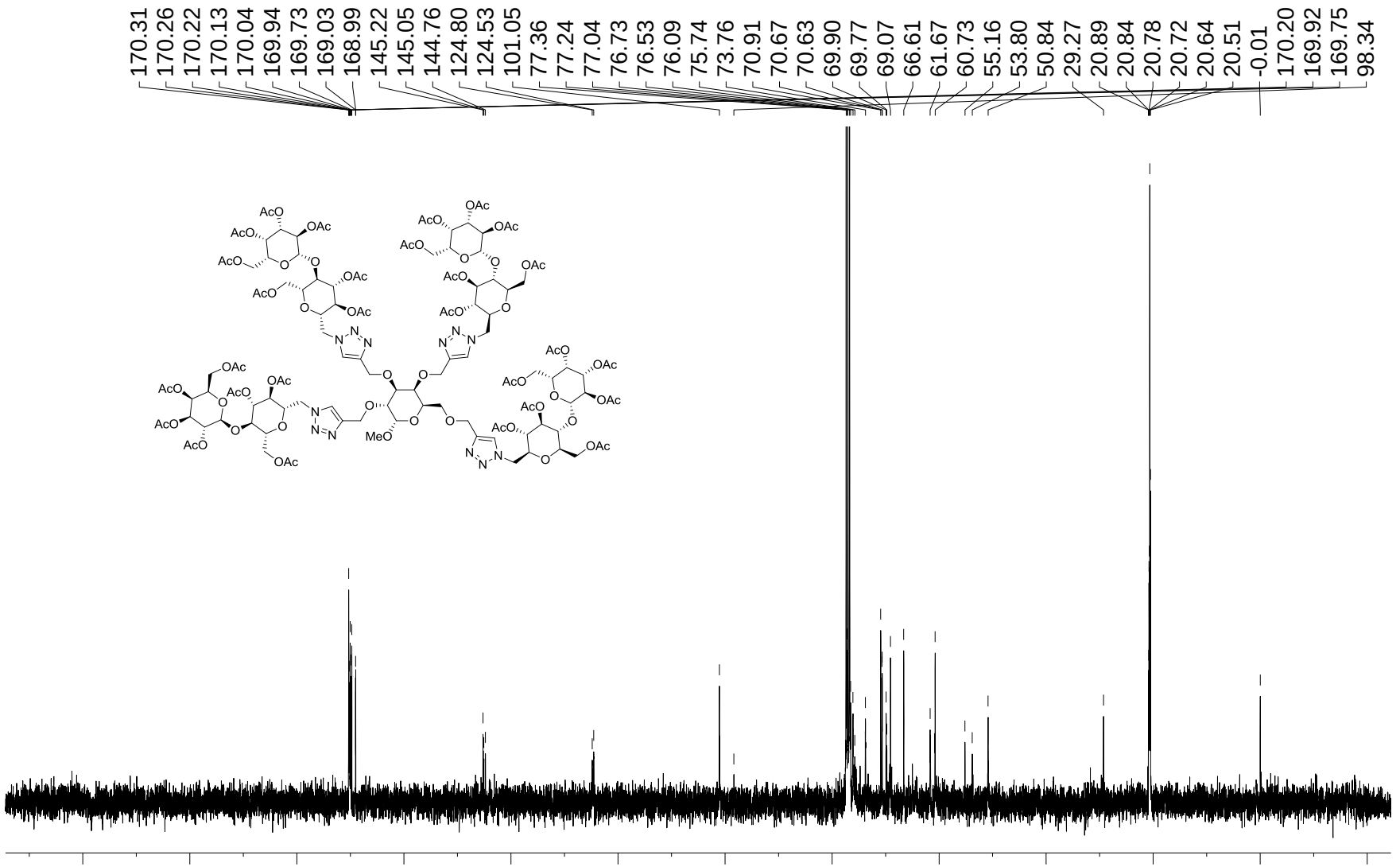
H-H COSY and HSQC (400MHz, D_2O) spectrum of **8k**



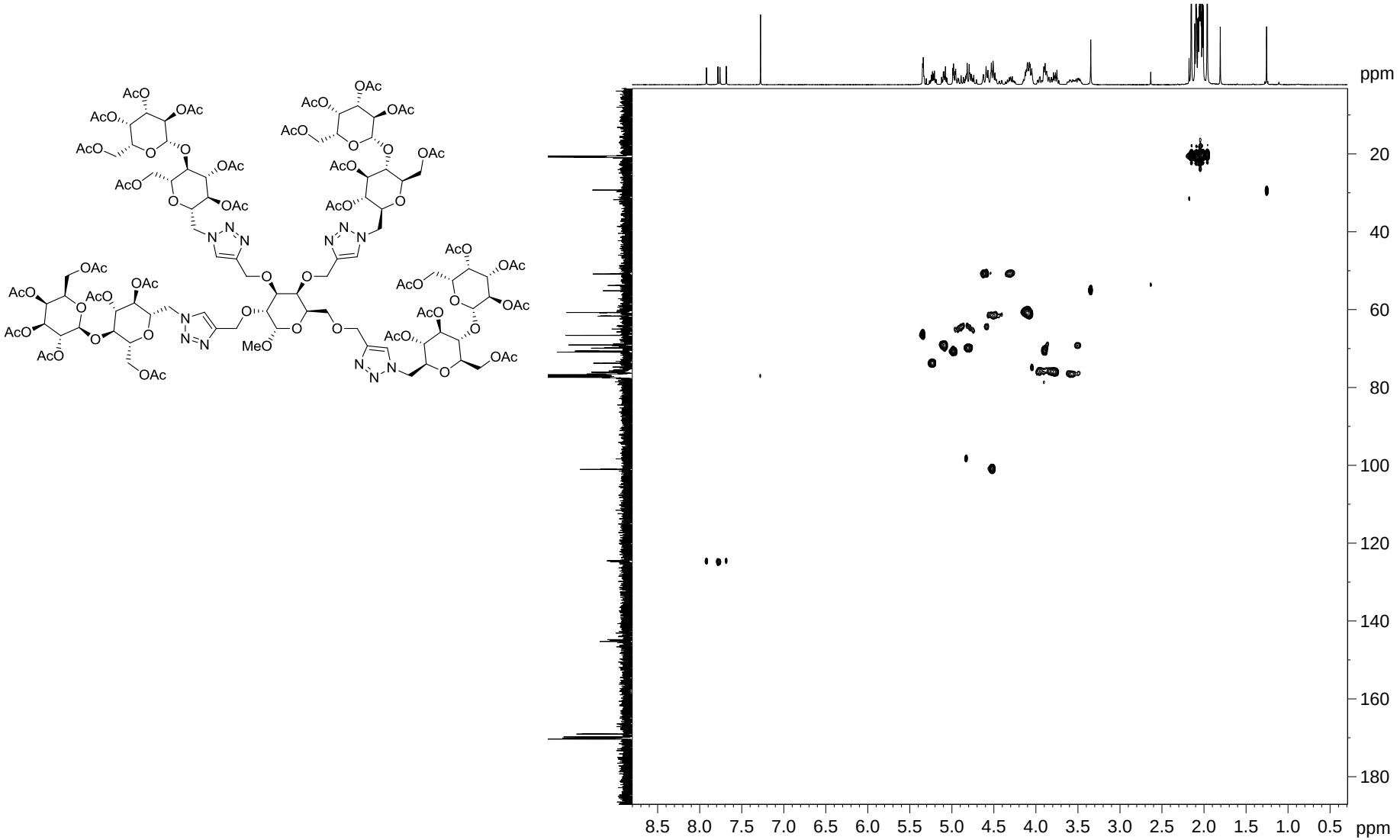
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7m**



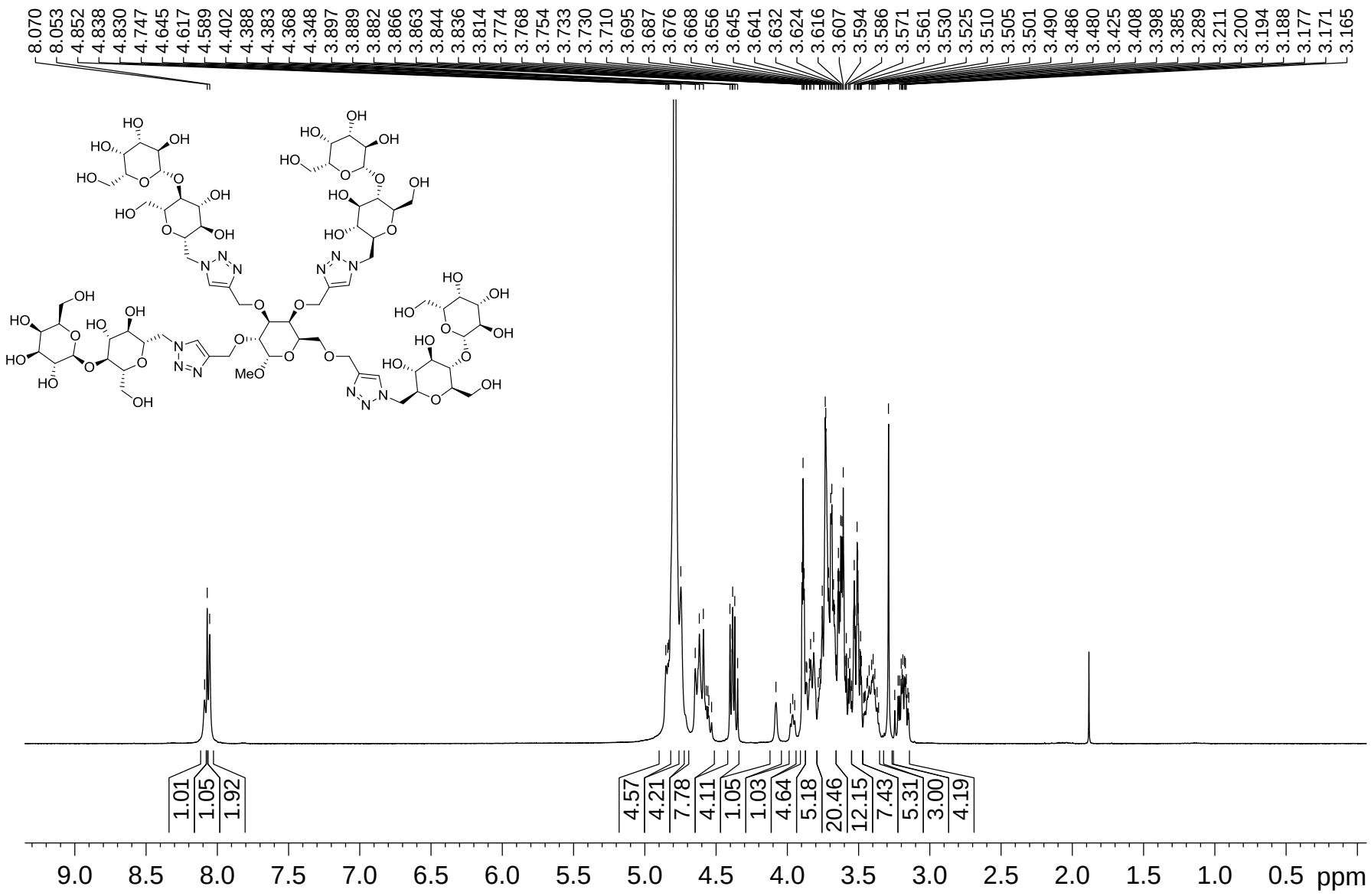
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7m**



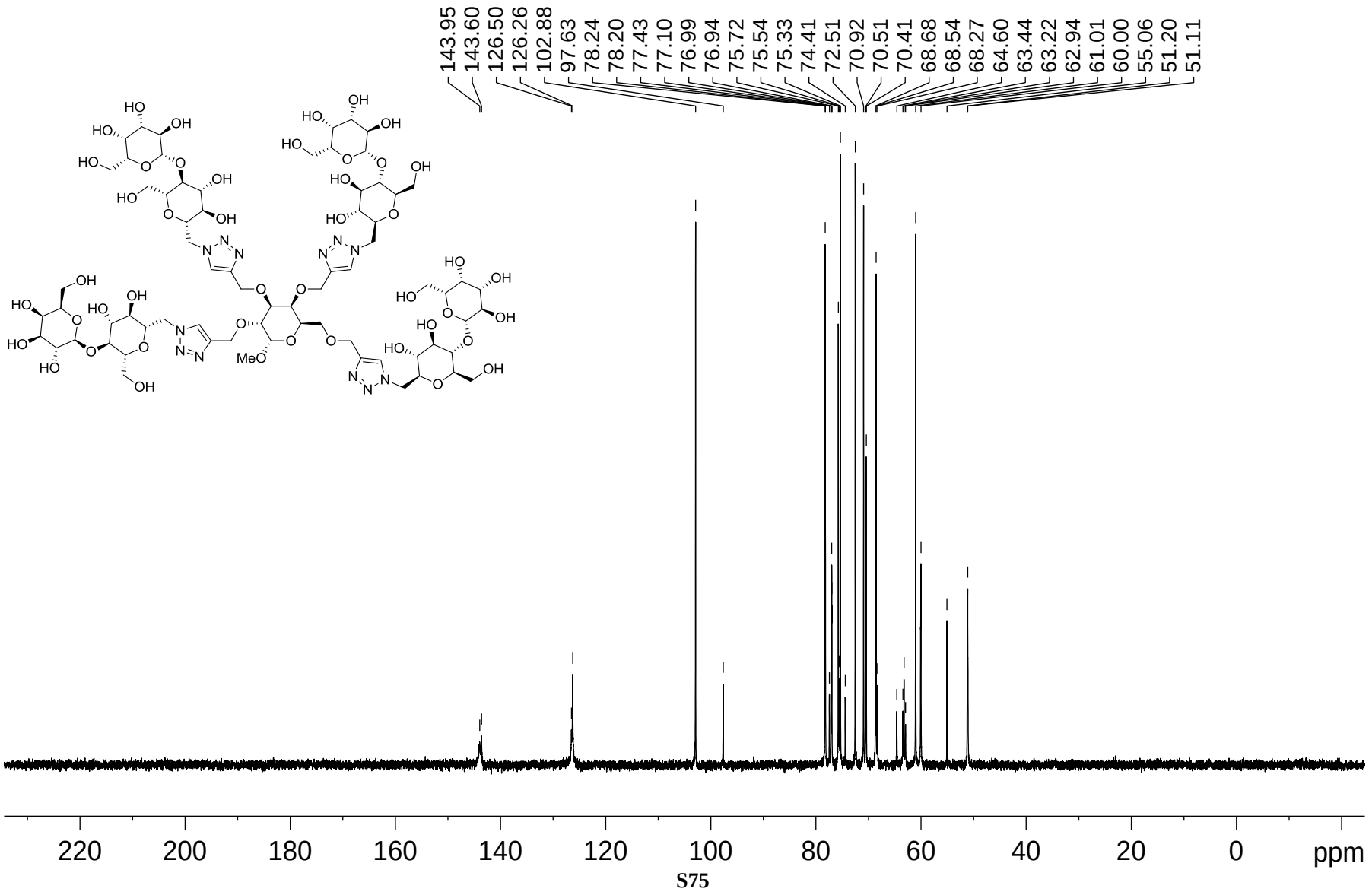
HSQC (400MHz, $CDCl_3$) spectrum of **7m**



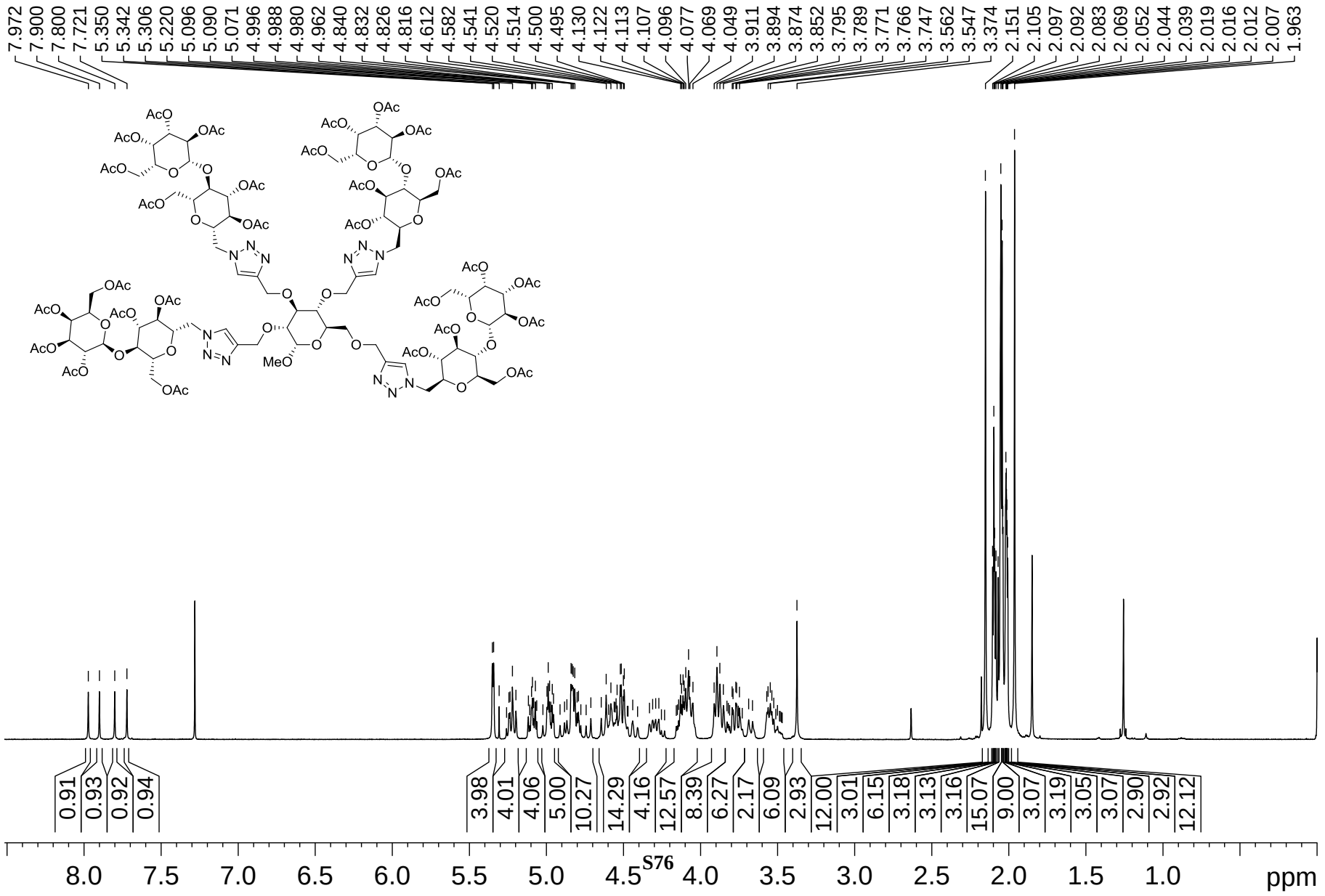
^{1}H NMR (400MHz, D_2O) spectrum of **8m**



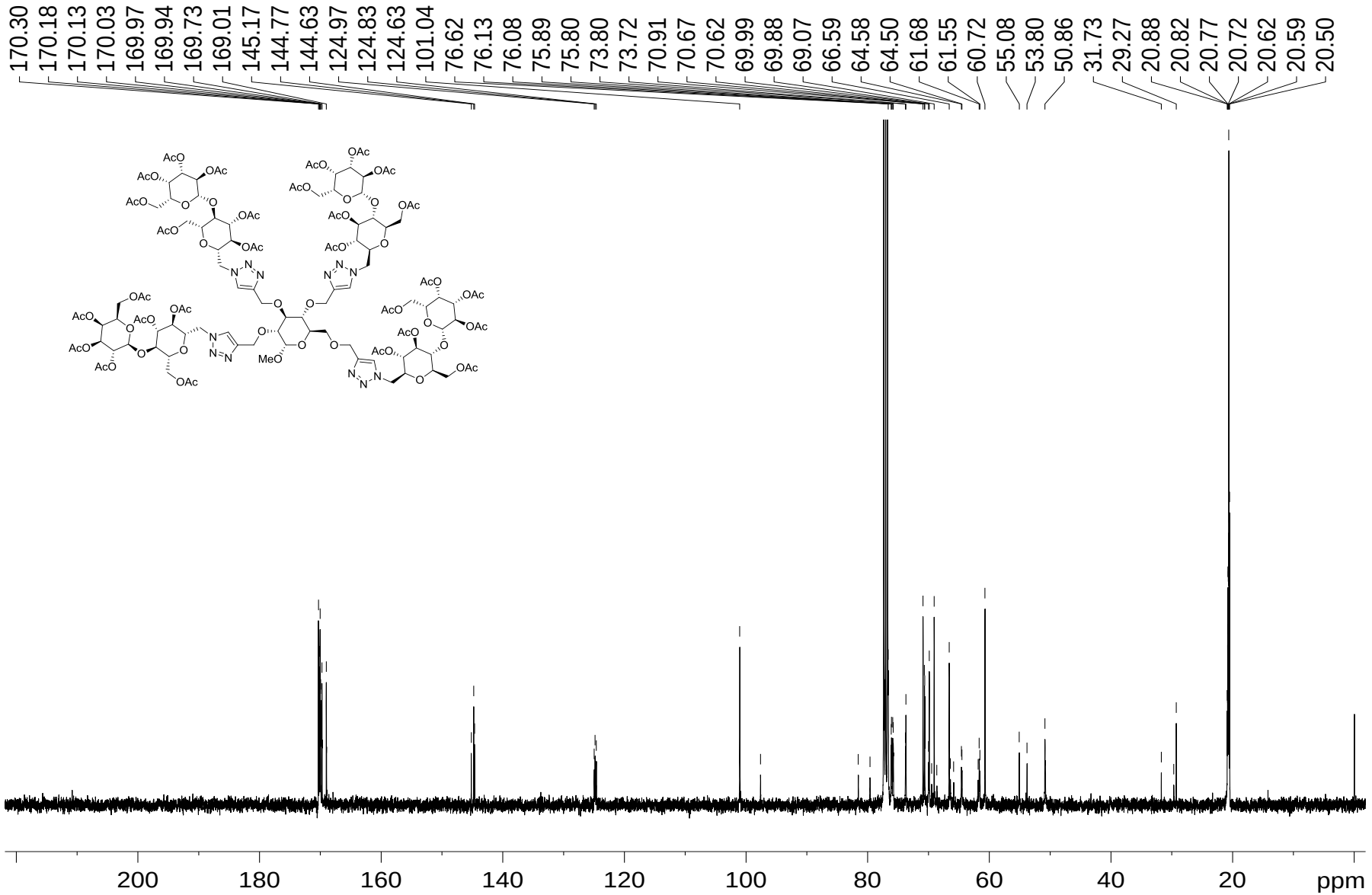
^{13}C NMR (100MHz, D_2O) spectrum of **8m**



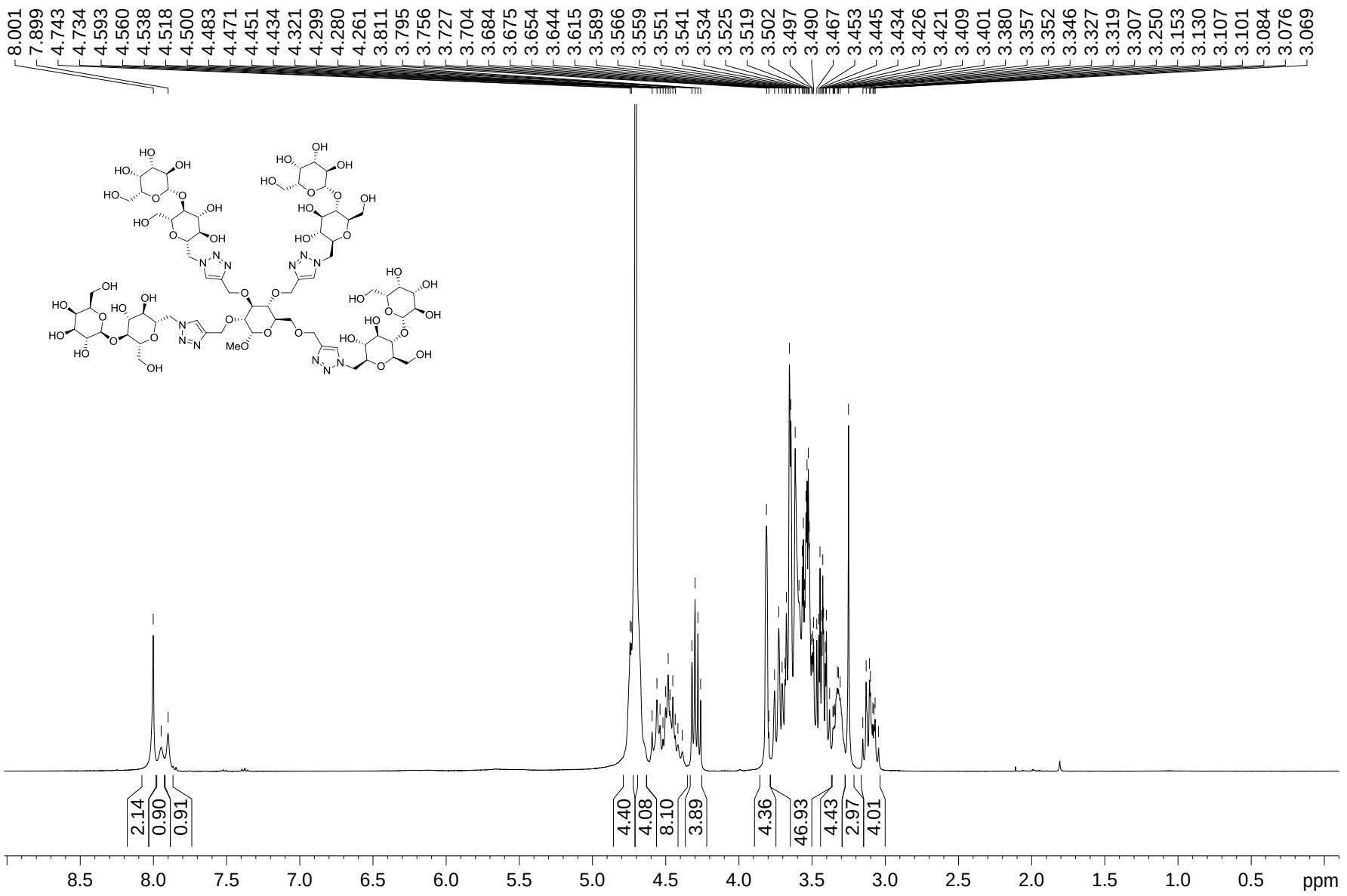
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7l**



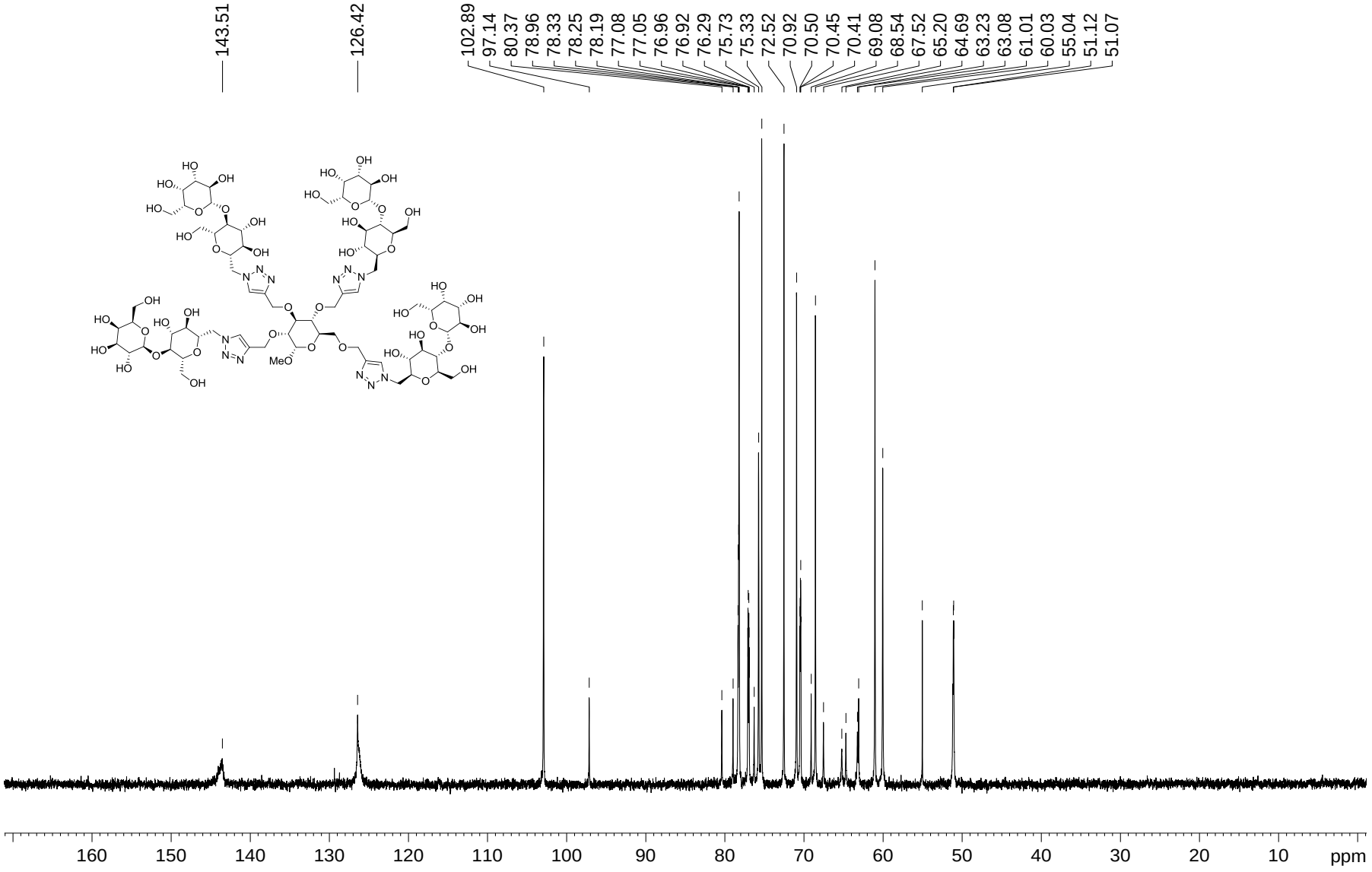
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7l**



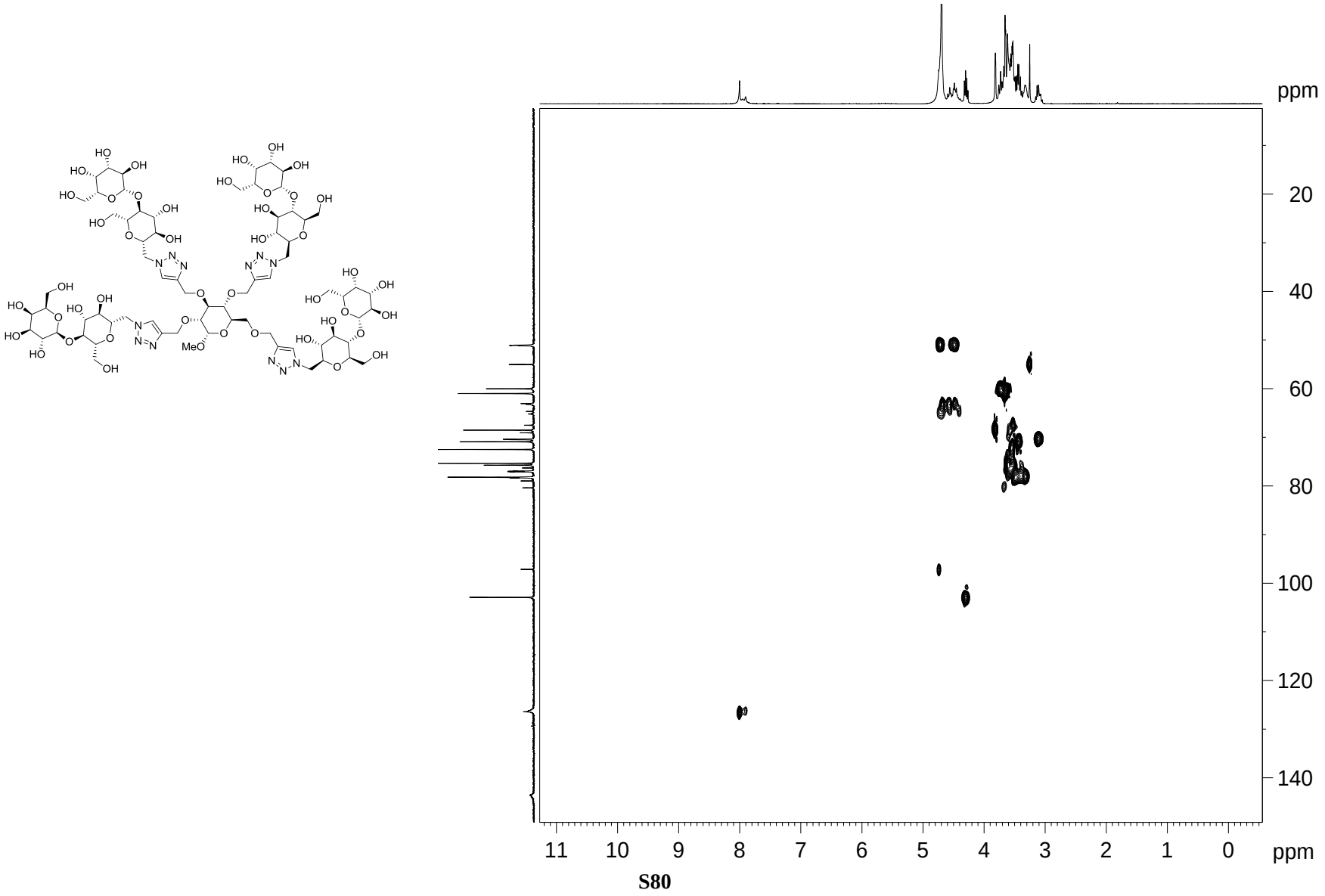
^{1}H NMR (400MHz, D_2O) spectrum of **8l**



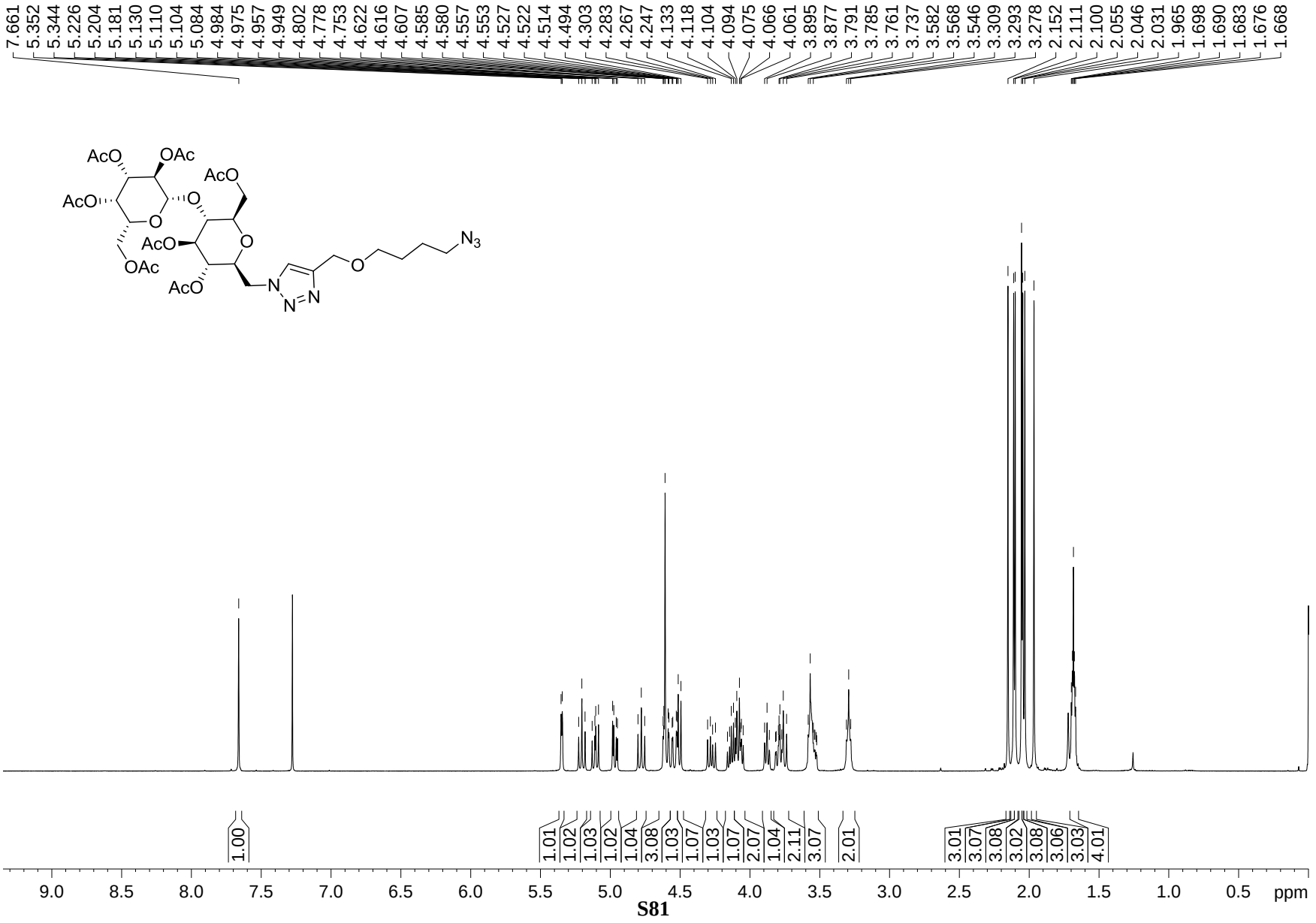
^{13}C NMR (100MHz, D_2O) spectrum of **8l**



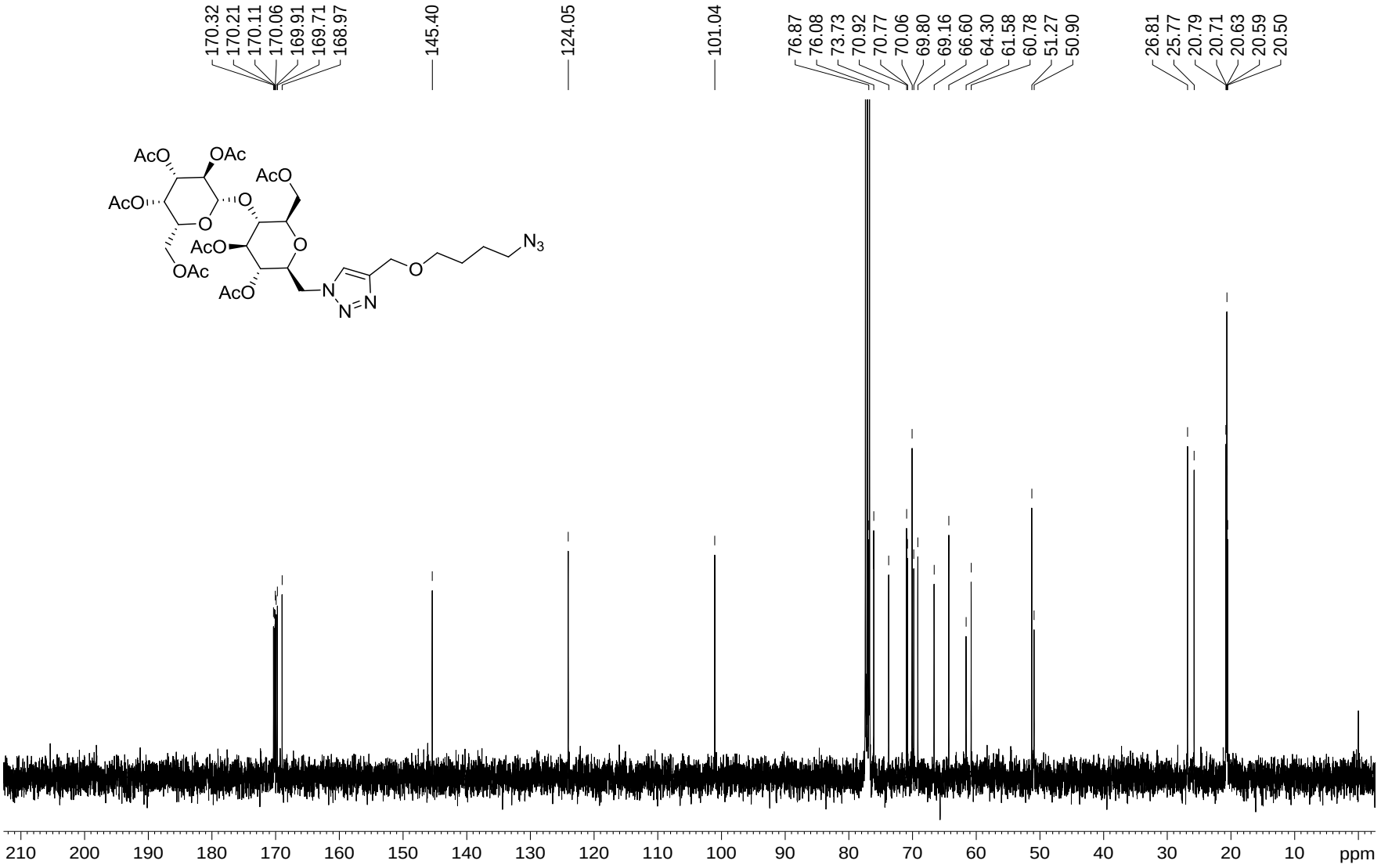
HSQC (400MHz, D_2O) spectrum of **8l**



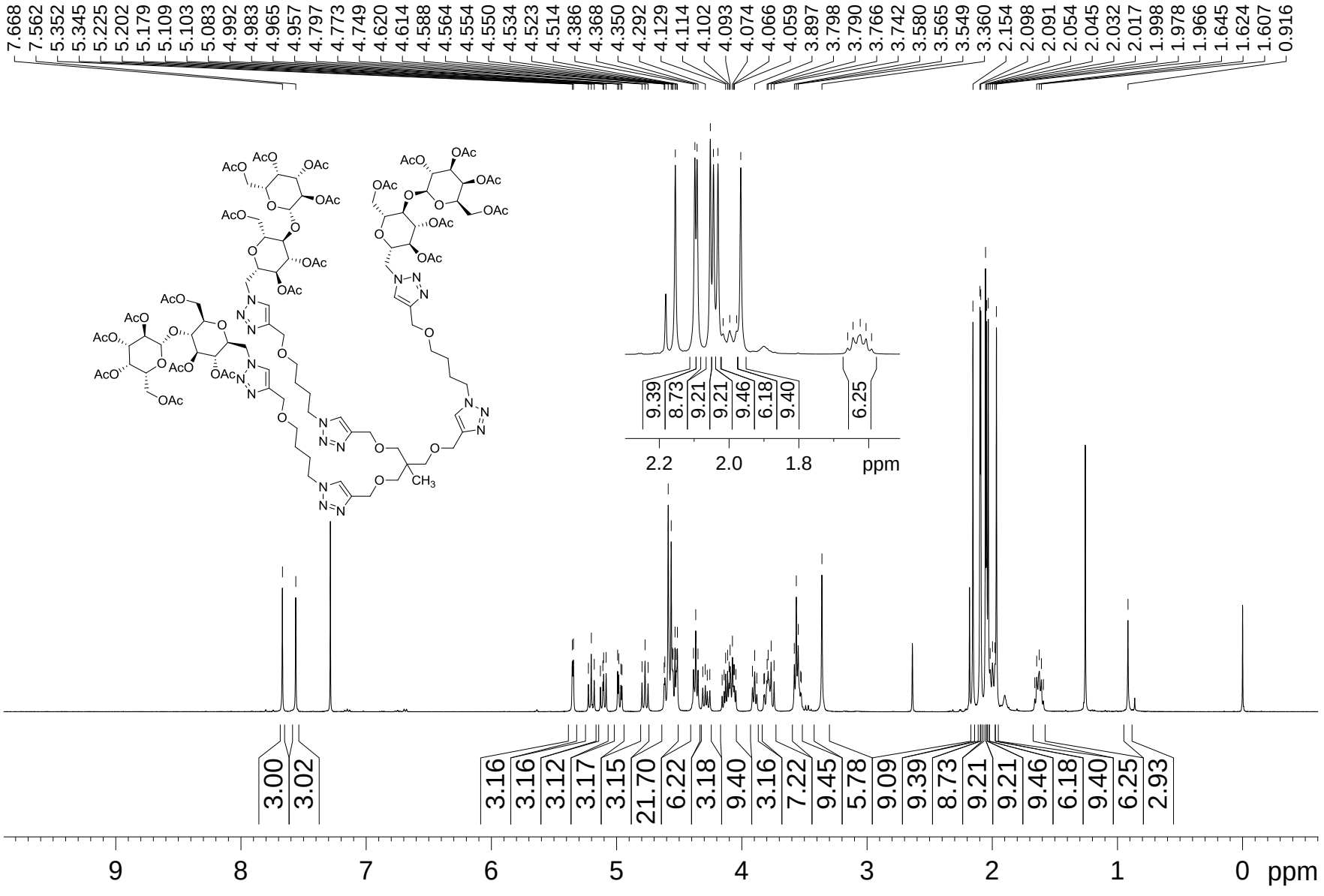
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7n**



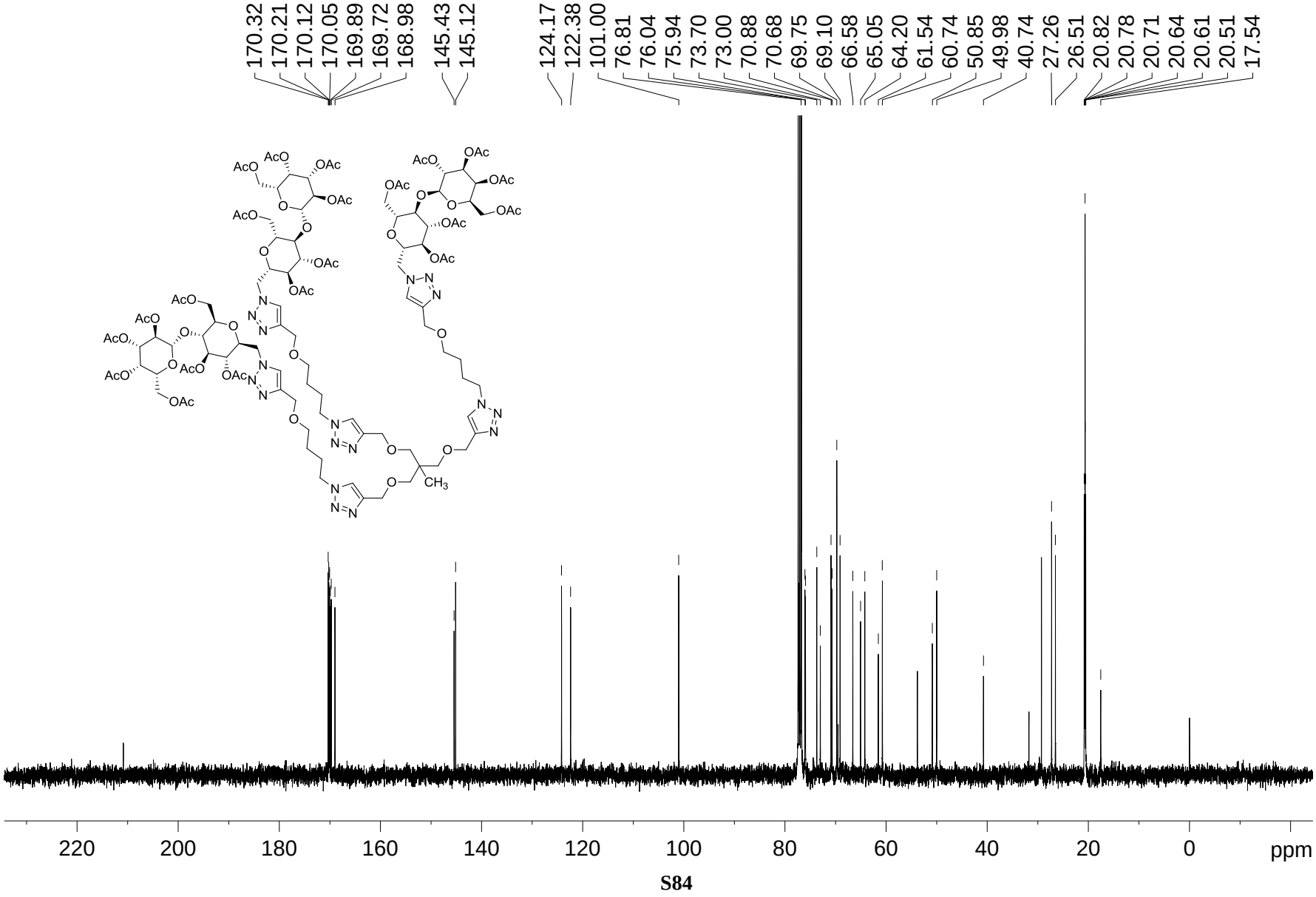
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7n**



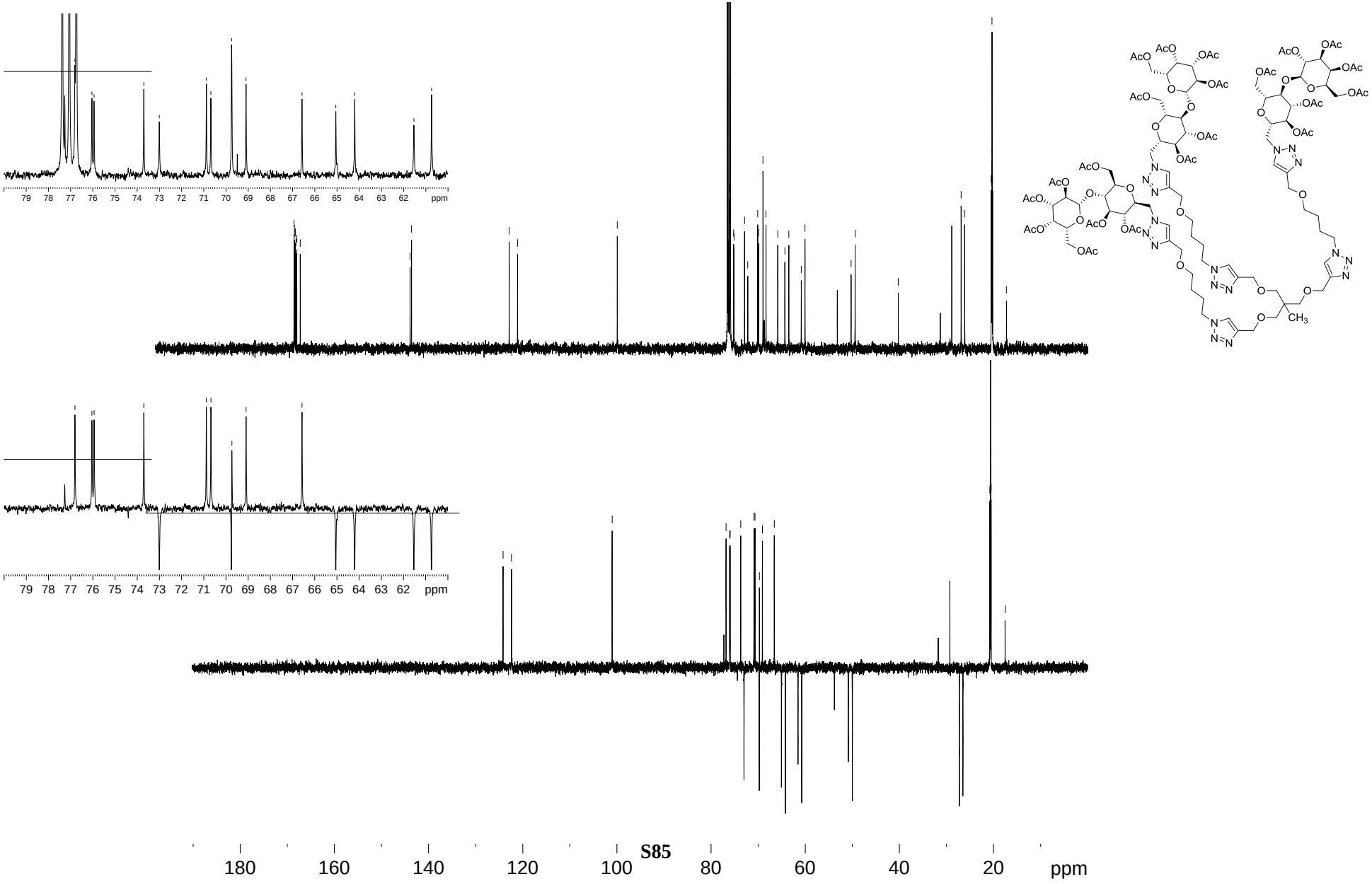
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7o**



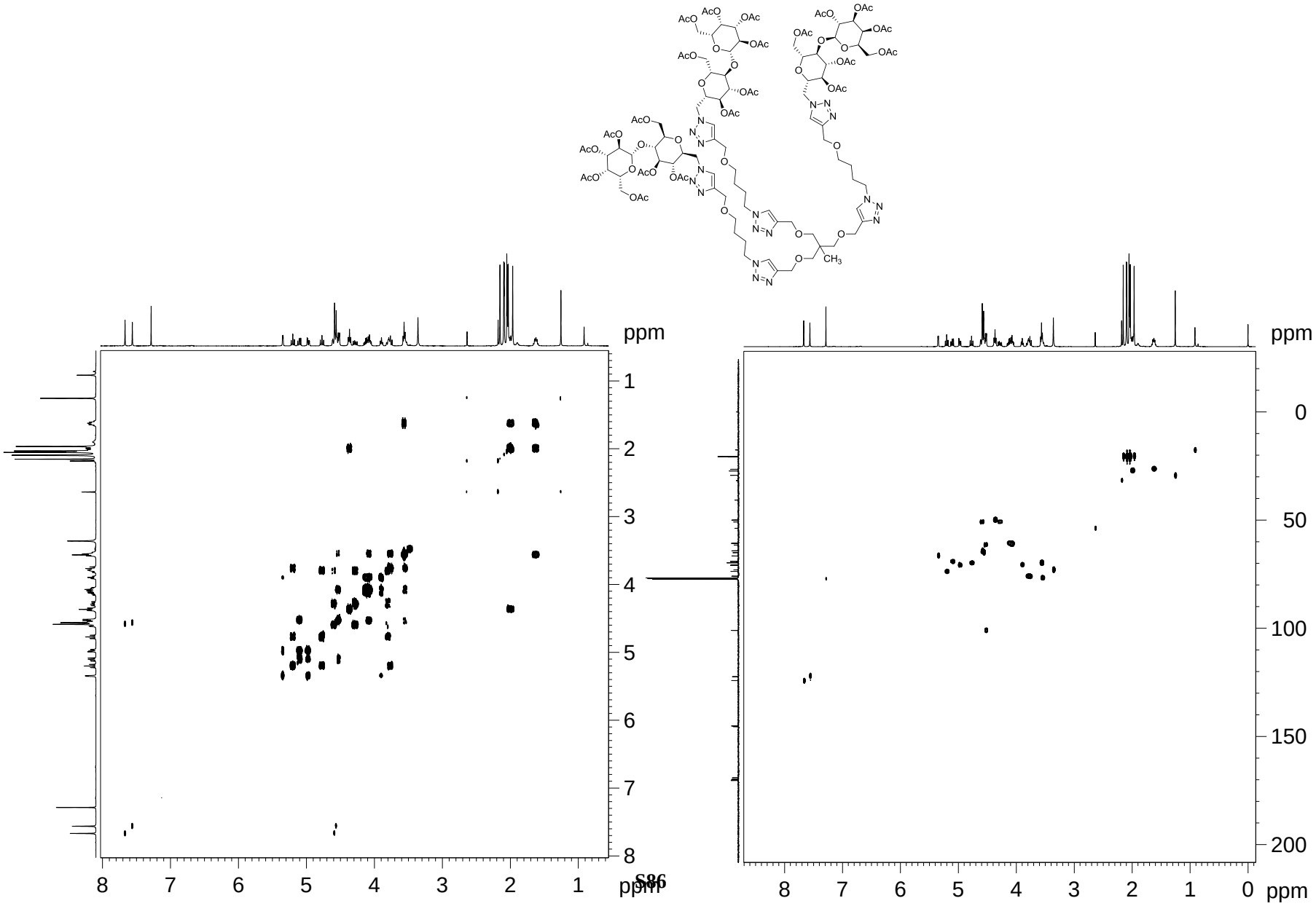
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7o**



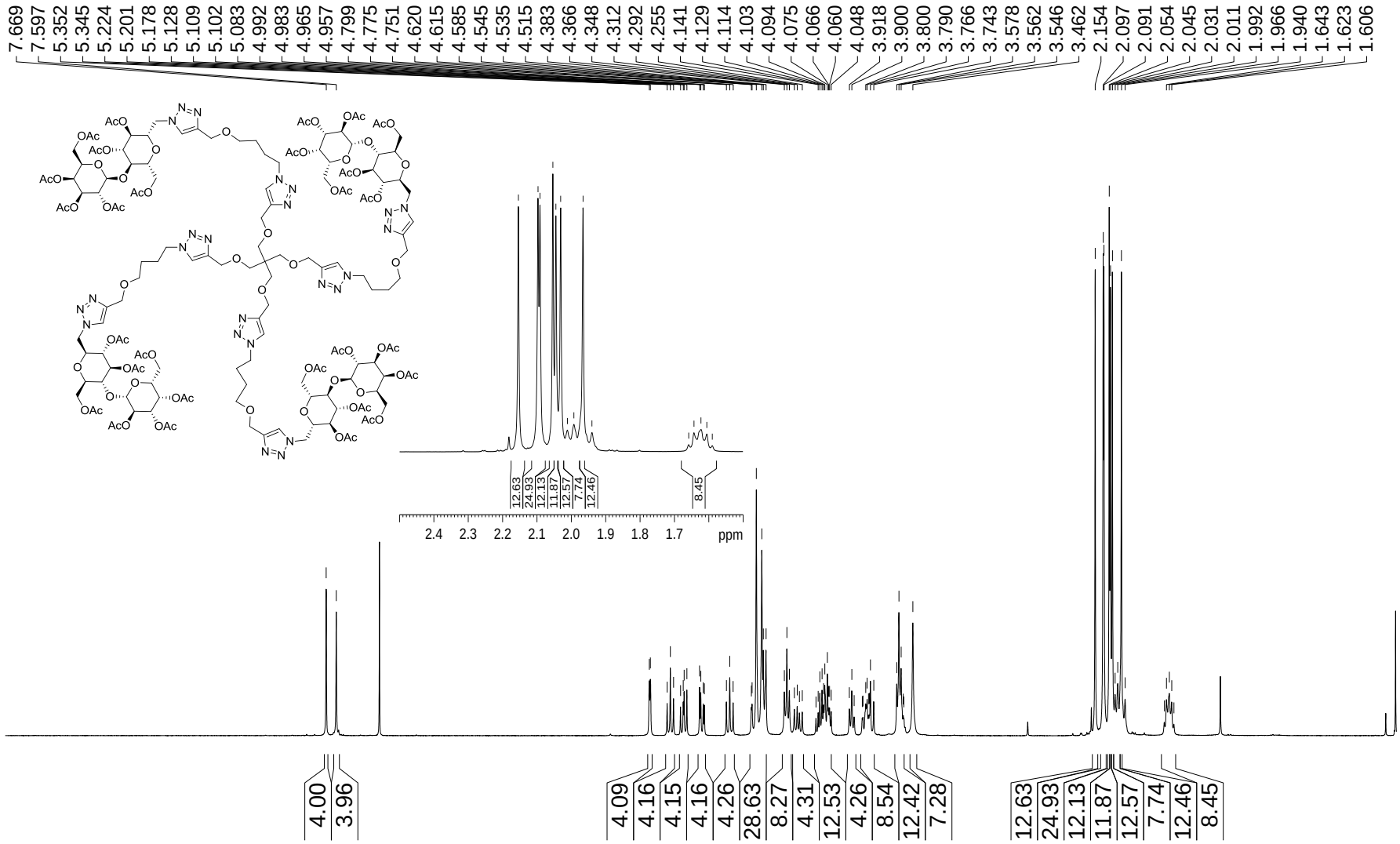
Comparison of ^{13}C NMR and DEPT135 (100MHz, $CDCl_3$) spectrum of **7o**



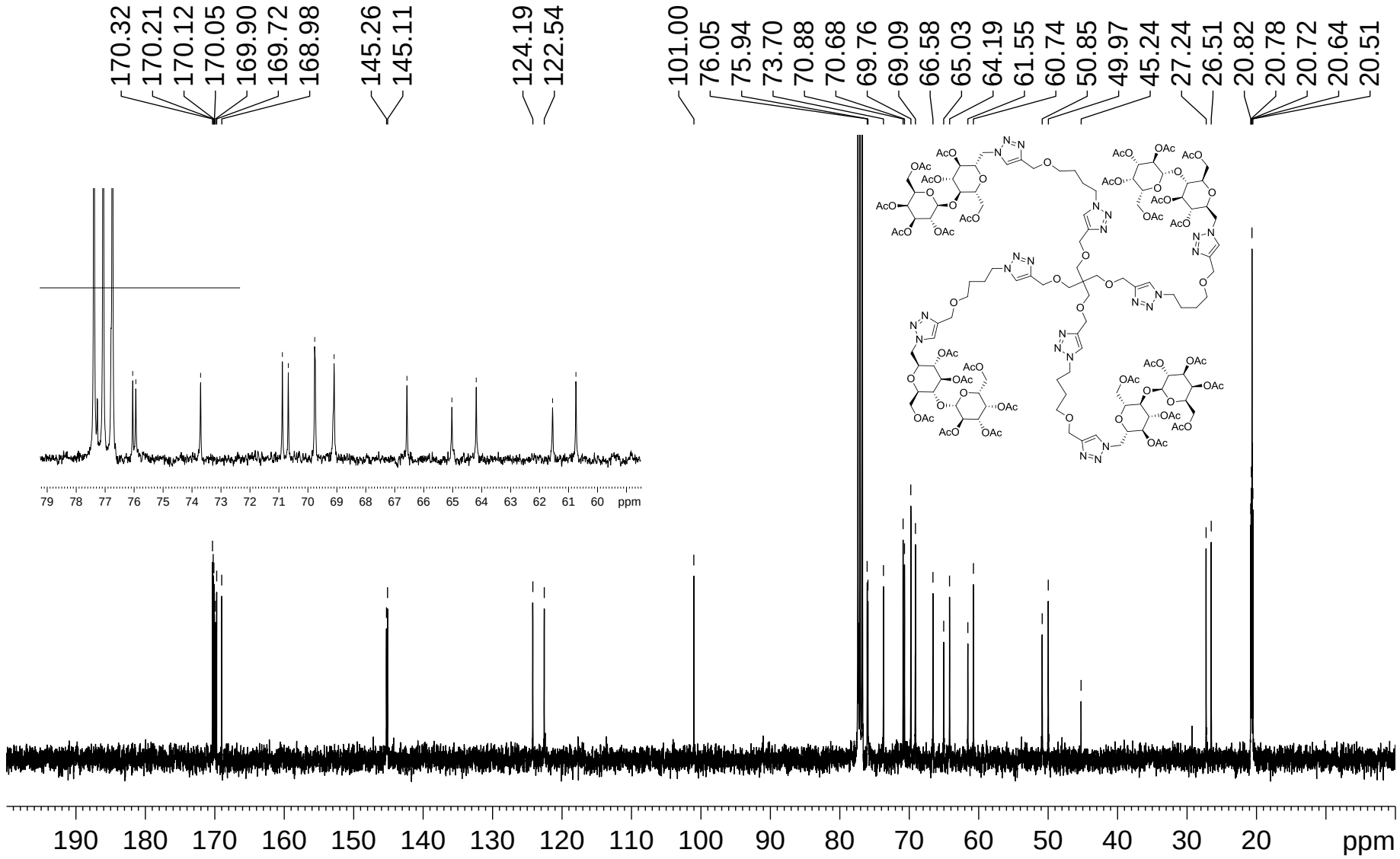
H-H COSY and HSQC (400MHz, $CDCl_3$) spectrum of **7o**



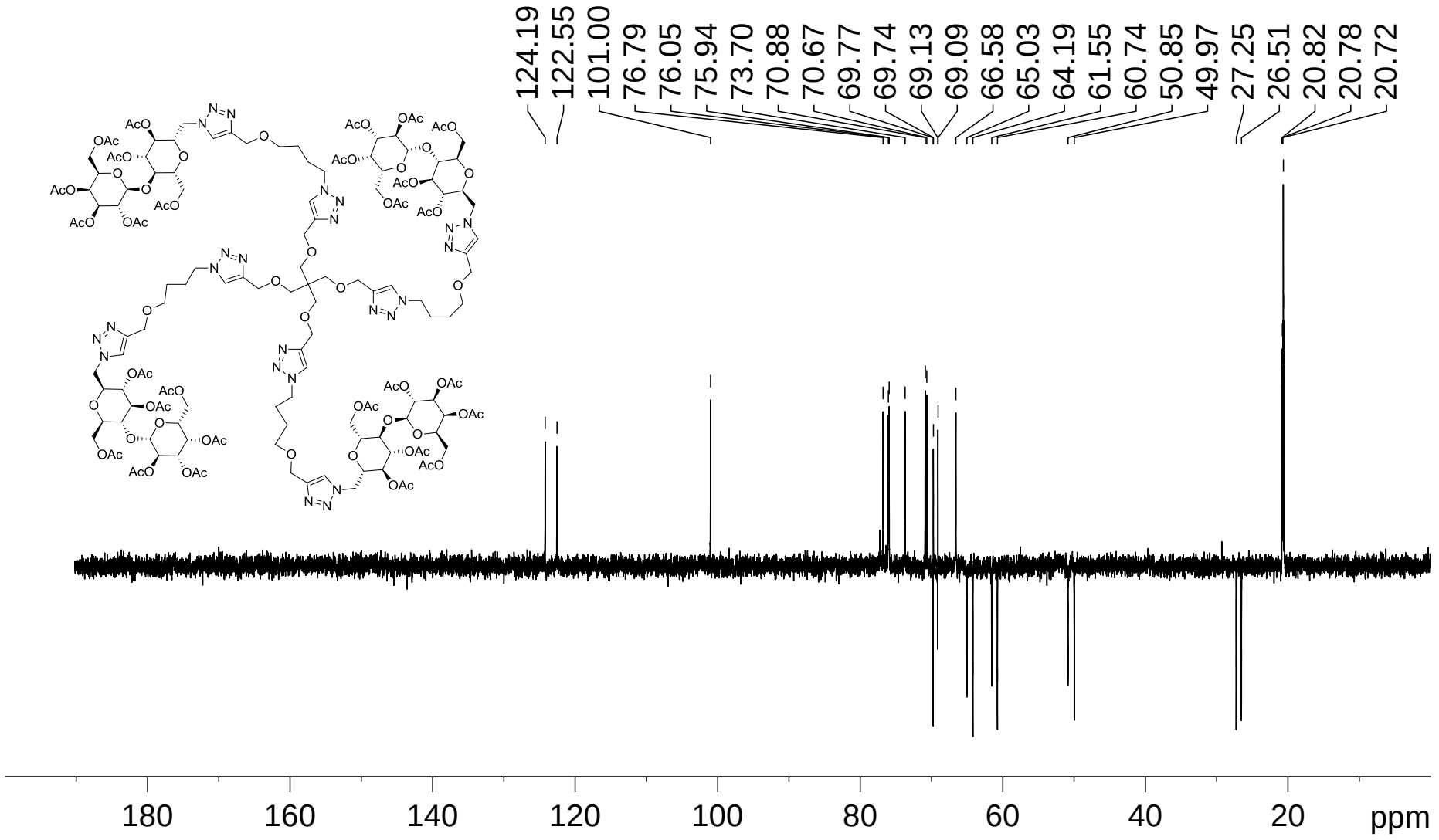
^{1}H NMR (400MHz, $CDCl_3$) spectrum of **7p**



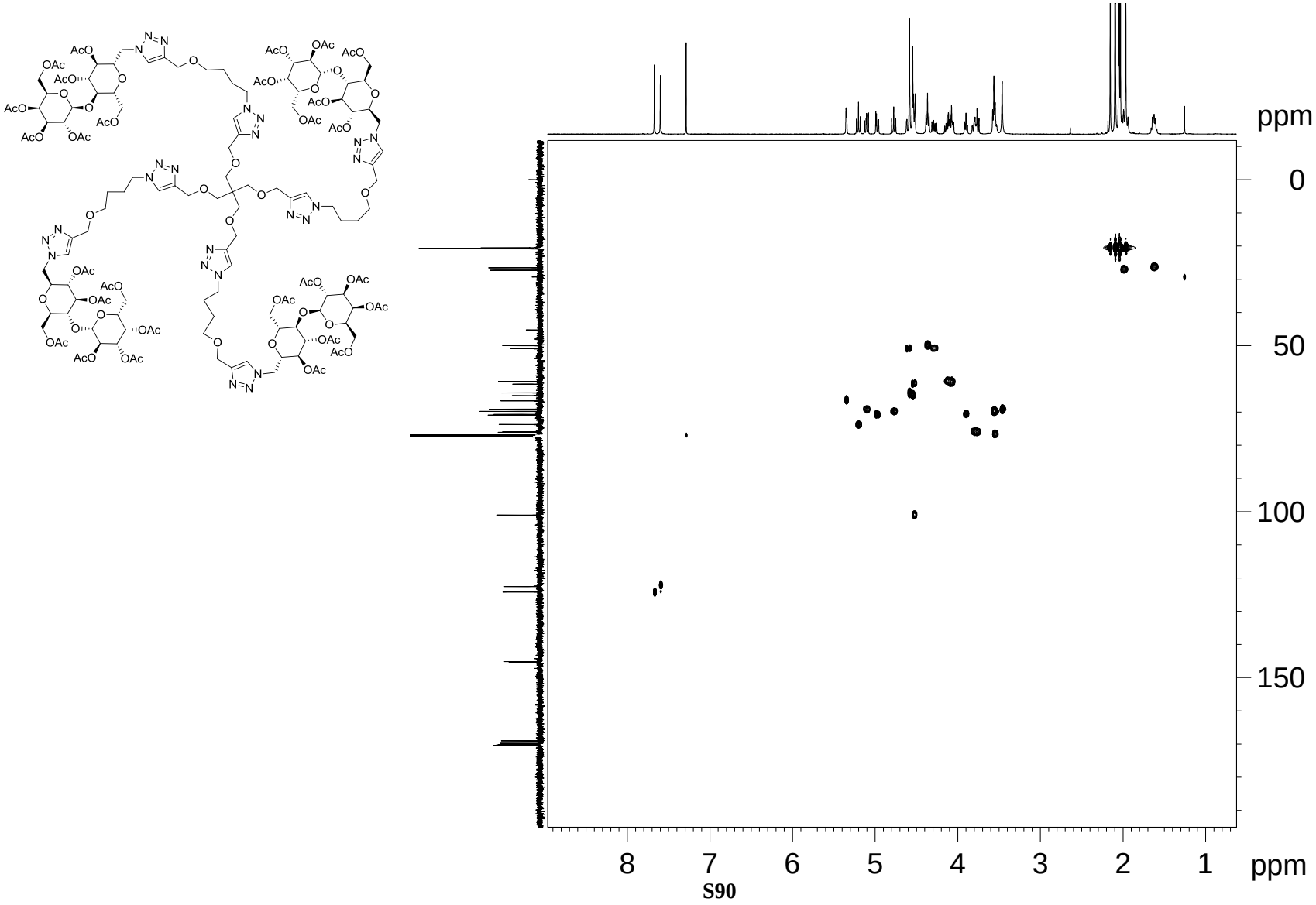
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **7p**



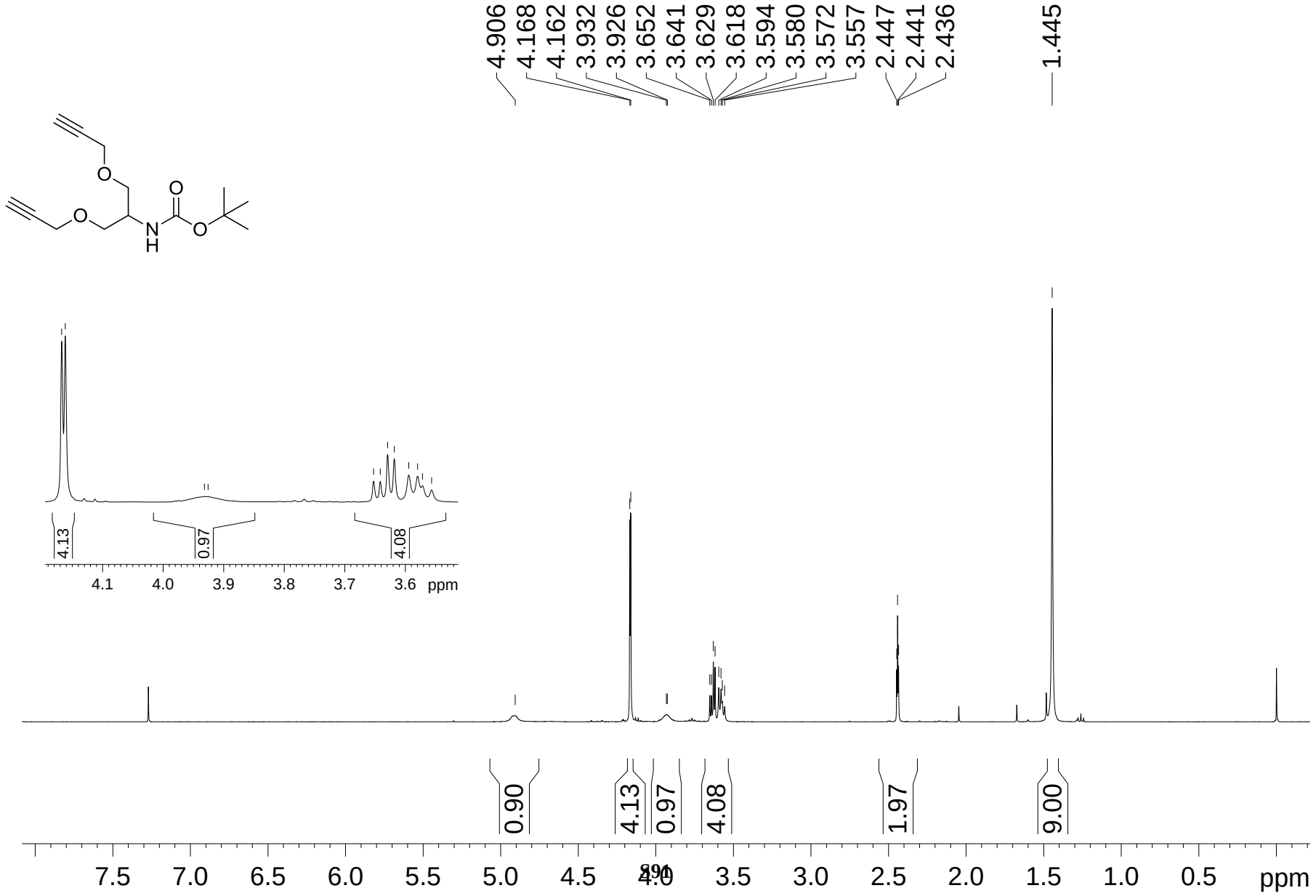
DEPT135 (100MHz, $CDCl_3$) spectrum of **7p**



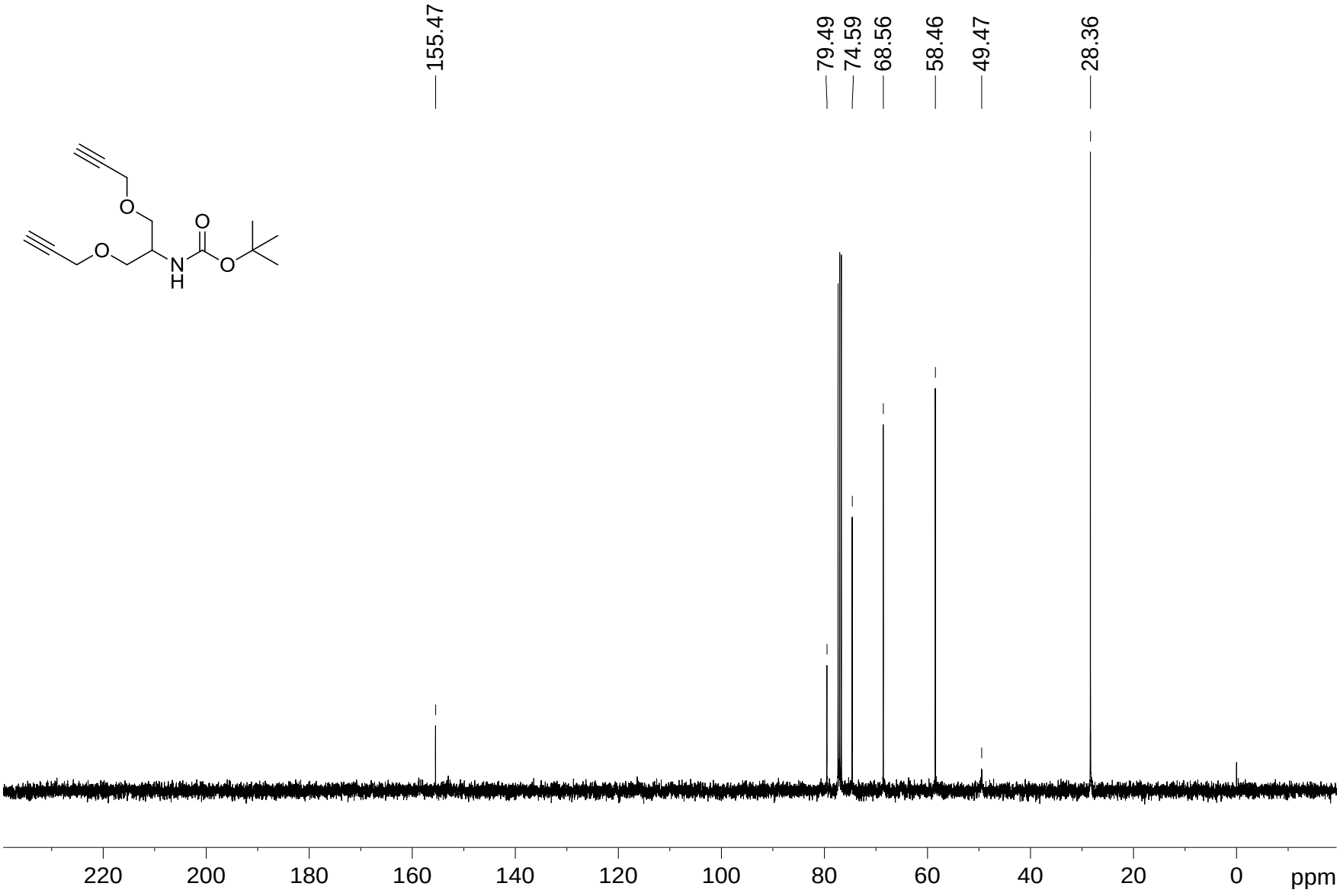
HSQC (400MHz, $CDCl_3$) spectrum of **7p**



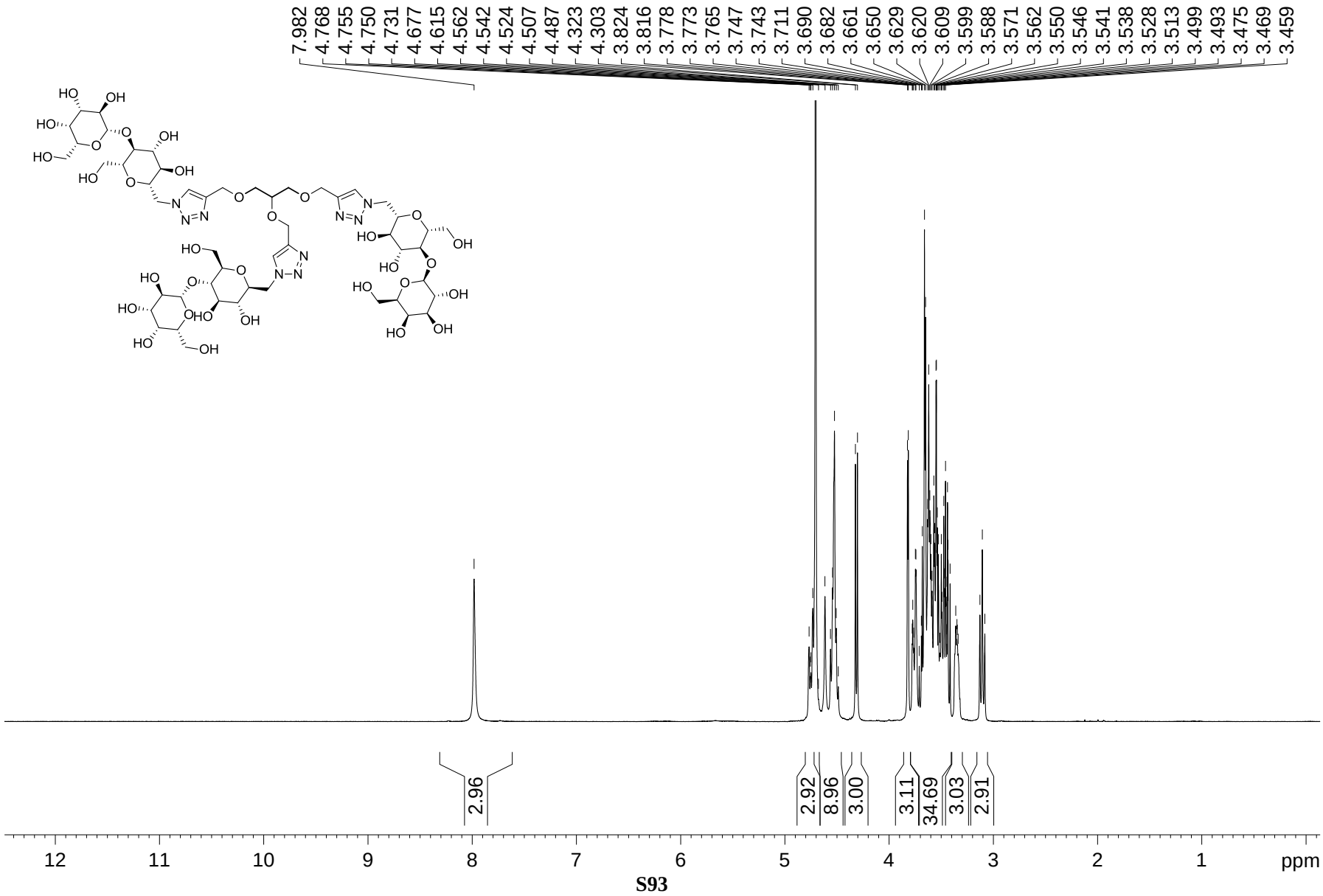
^{1}H NMR(400MHz, $CDCl_3$) spectrum of **6e**



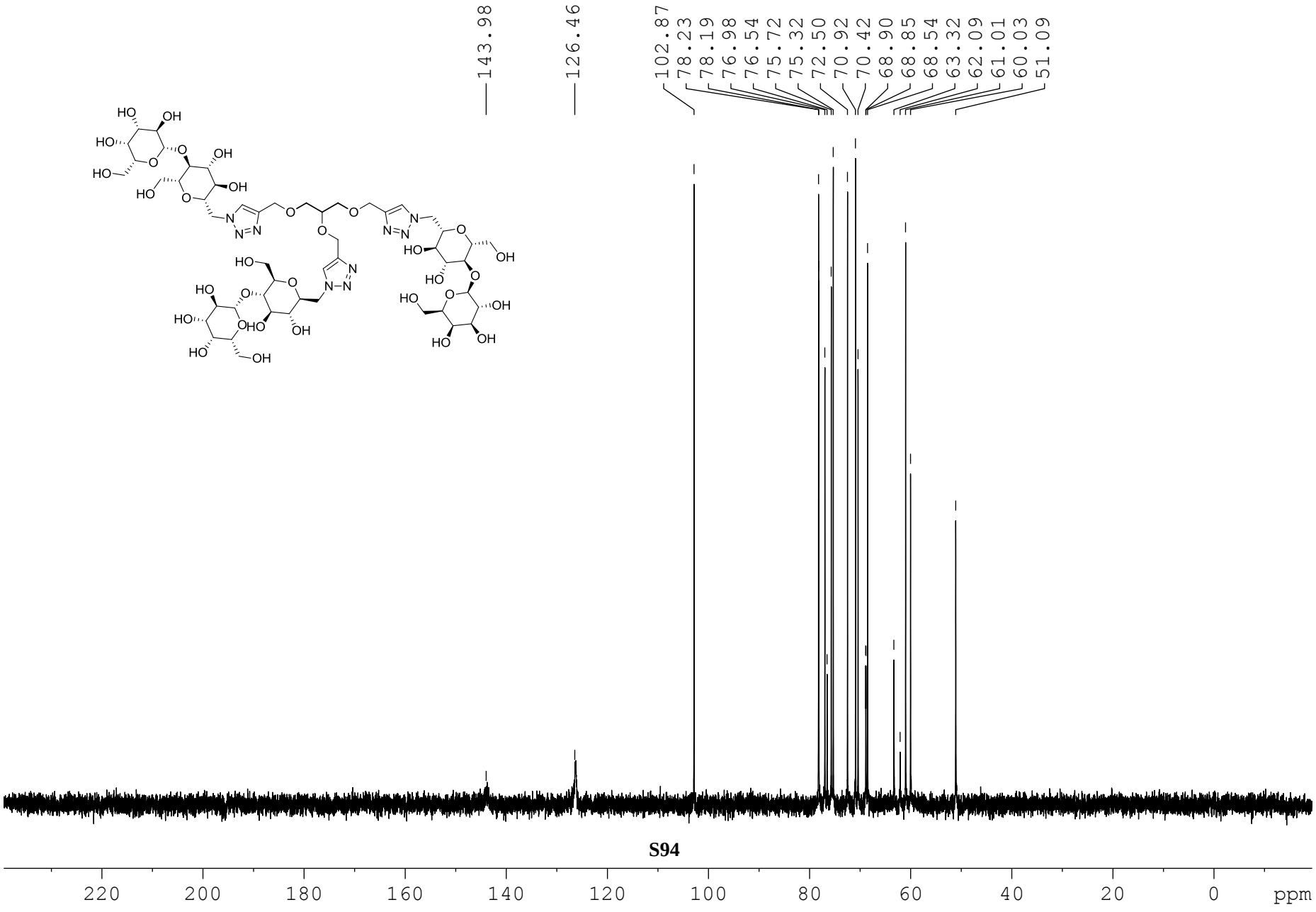
^{13}C NMR (100MHz, $CDCl_3$) spectrum of **6e**



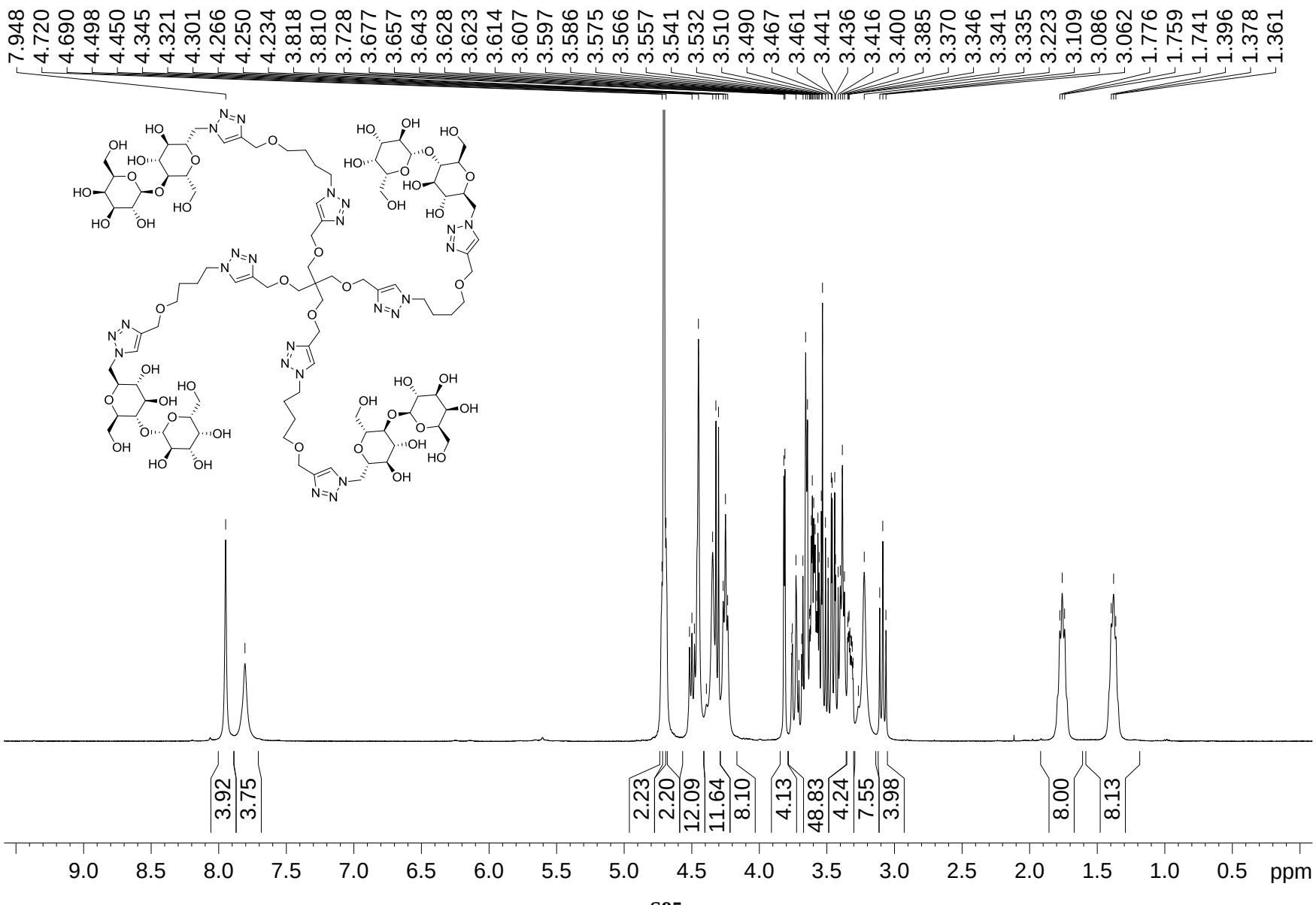
^{1}H NMR (400MHz, D_2O) spectrum of **8i**



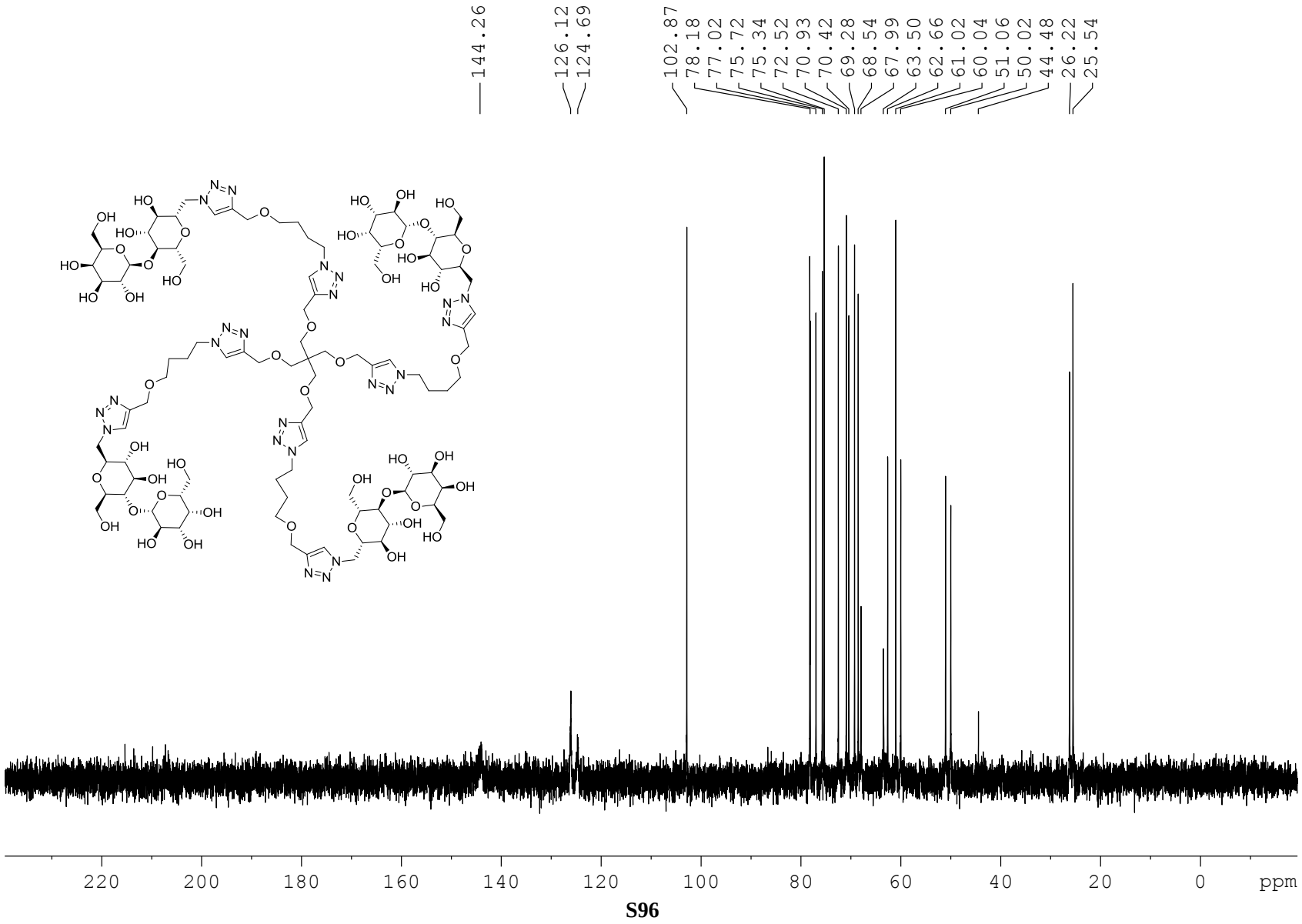
^{13}C NMR (100MHz, D_2O) spectrum of **8i**



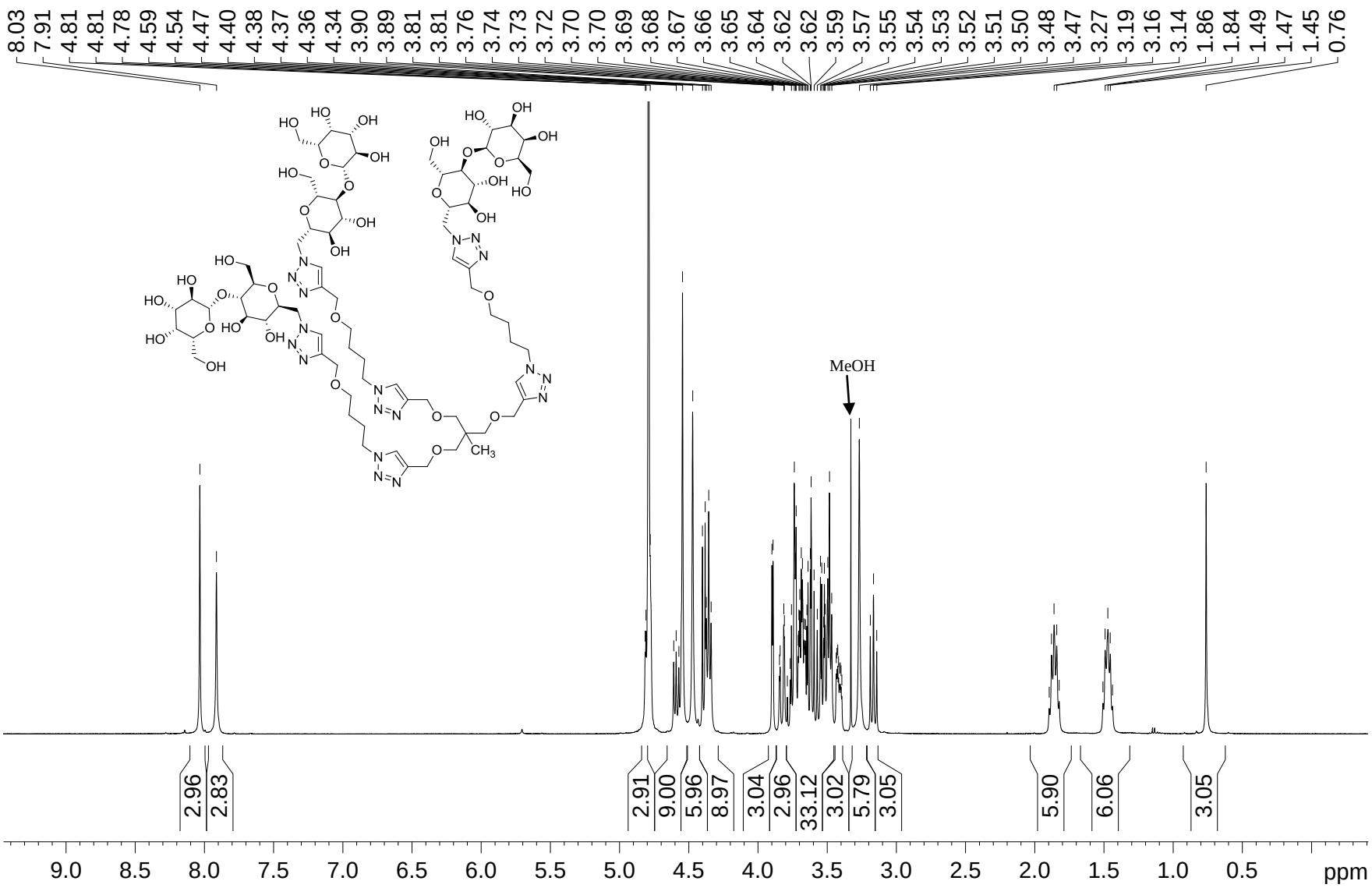
^{1}H NMR (400MHz, D_2O) spectrum of **8p**



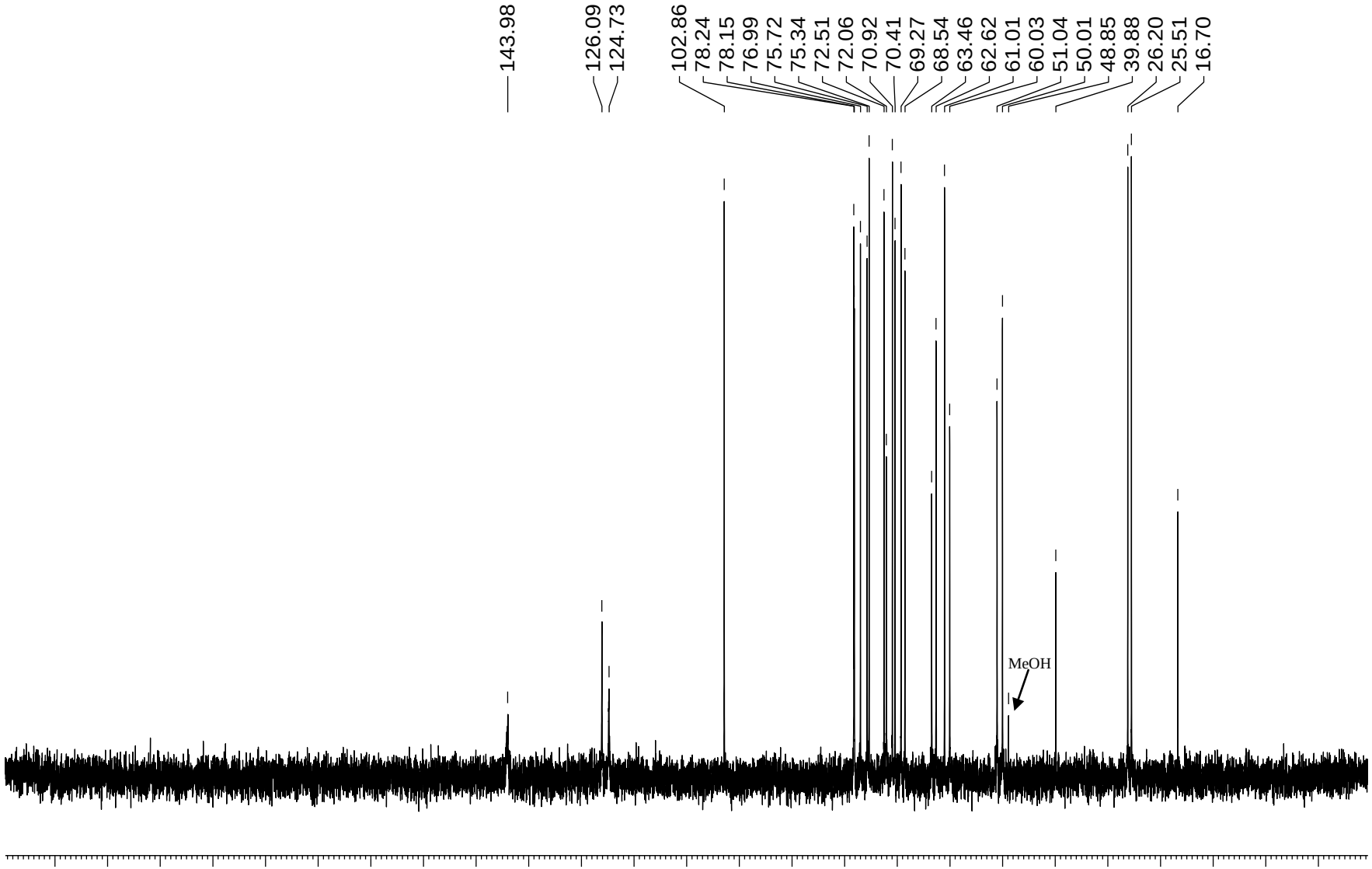
^{13}C NMR (100MHz, D_2O) spectrum of **8p**



^{1}H NMR (400MHz, D_2O, internal MeOH) spectrum of **8o**



^{13}C NMR (100MHz, D_2O, internal MeOH) spectrum of **8o**



Part II Sensorgrams and corresponding kinetic/steady state analysis

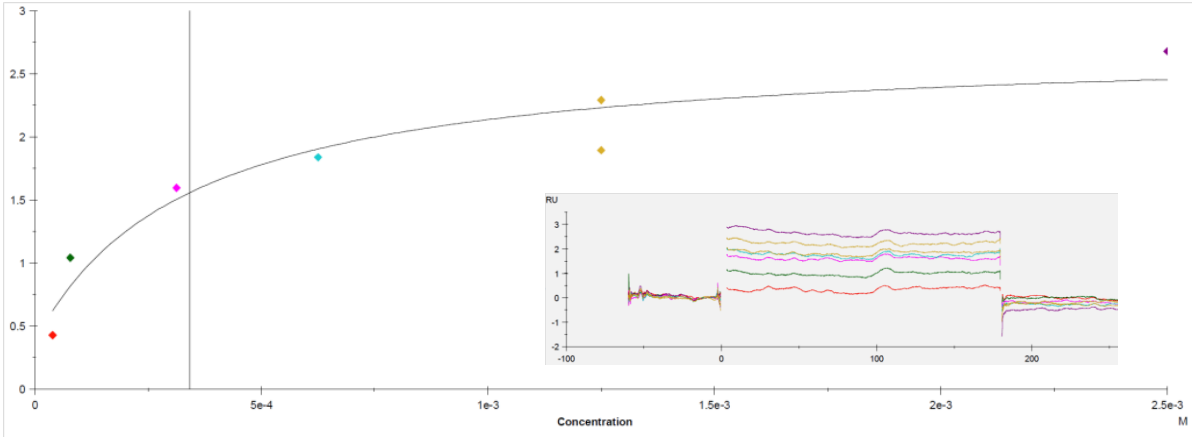


Figure S1. Sensorgrams and steady state analysis of the binding between Lactose and Galectin-3 (K_D = 342 μM; Chi^2 = 0.0669 RU^2)

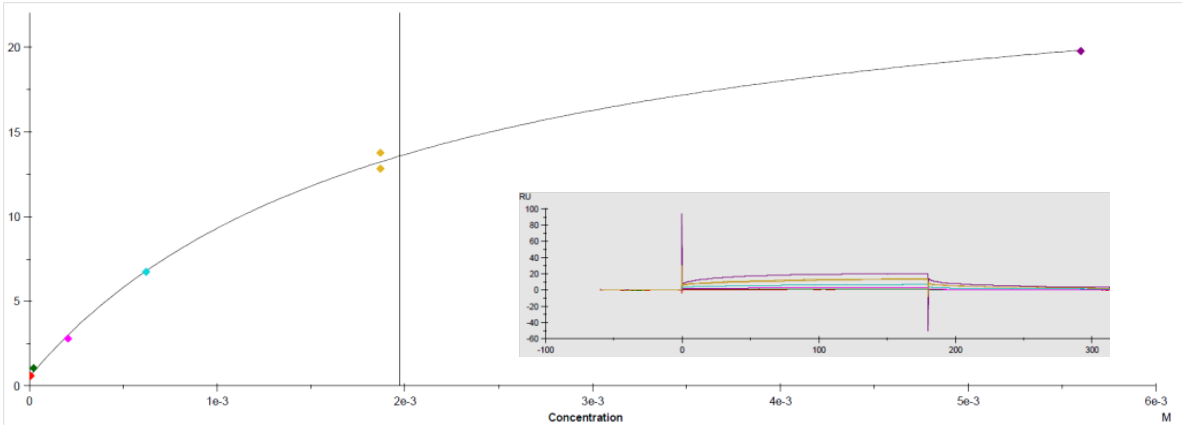


Figure S2. Sensorgrams and steady state analysis of the binding between **8a** and Galectin-3 (K_D = 1970 μM; Chi^2 = 0.137 RU^2)

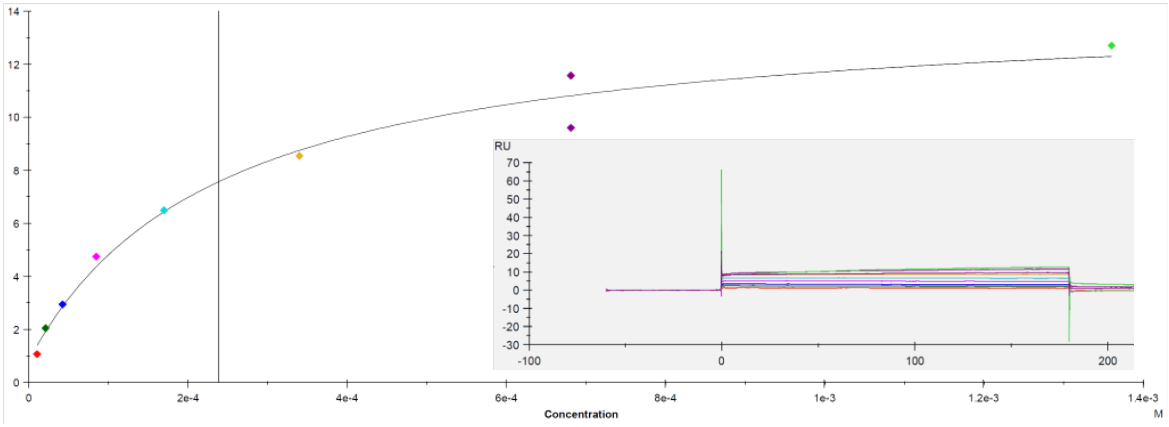


Figure S3. Sensorgrams and steady state analysis of the binding between **8h** and Galectin-3 (K_D = 239 μM; Chi^2 = 0.421 RU^2)

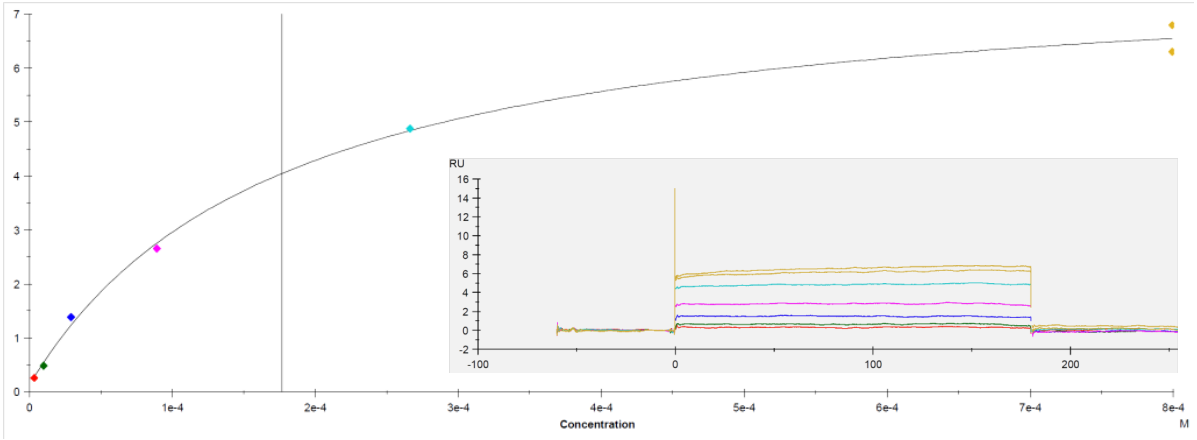
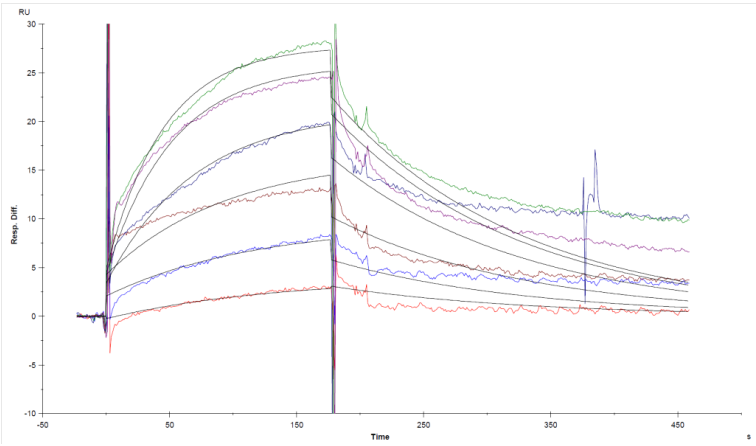


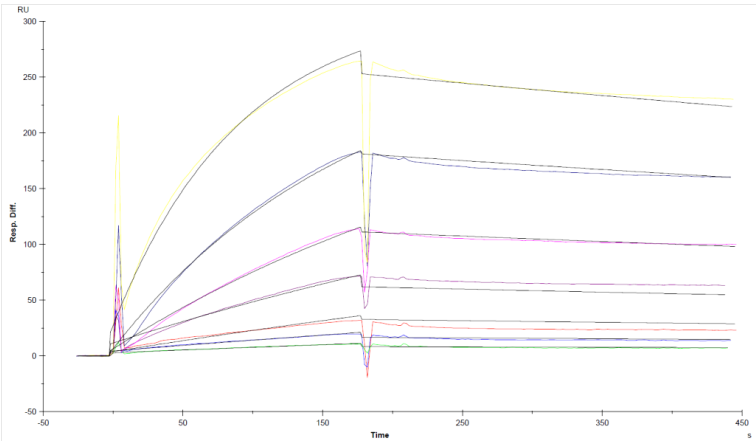
Figure S4. Sensorgrams and steady state analysis of the binding between **8e** and Galectin-3 (K_D = 176 μM; Chi^2 = 0.039 RU^2)



Kinetic affinity

Model: Langmuir 1:1
k_{on} = 12.8 (1/Ms)
k_{off} = 3.91 ×10^{-3}(1/s)
K_D = 306μM
Chi^2 = 0.845 RU^2

Figure S5. Sensorgrams and kinetic analysis of the binding between **8g** and Galectin-3 (100μM, 200μM, 400μM, 800μM, 1.6mM and 2.8mM)



Kinetic affinity

Model: Langmuir 1:1
k_{on} = 6.57 (1/Ms)
k_{off} = 4.57 (1/s)
K_D = 69.3 μM
Chi^2 = 14.6 RU^2

Figure S6. Sensorgrams and kinetic analysis of the binding between **8f** and Galectin-3 (25μM, 50μM, 100μM, 200μM, 400μM, 800μM and 1.6mM)

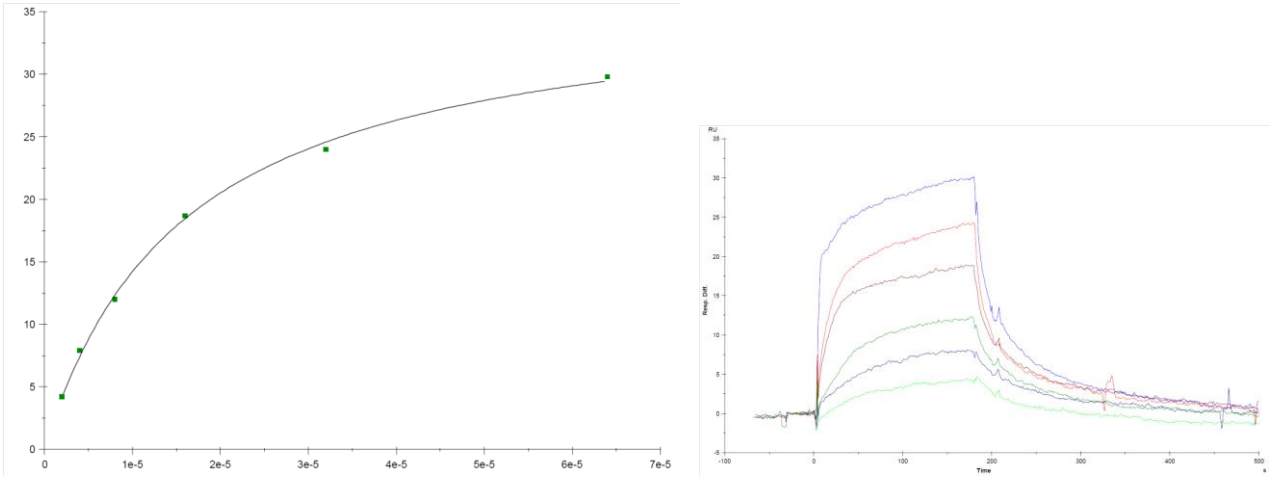


Figure S7. Sensorgrams and state steady analysis of the binding between **8q** and Galectin-3 (K_D = 15.8 µM; Chi^2 = 0.233 RU^2

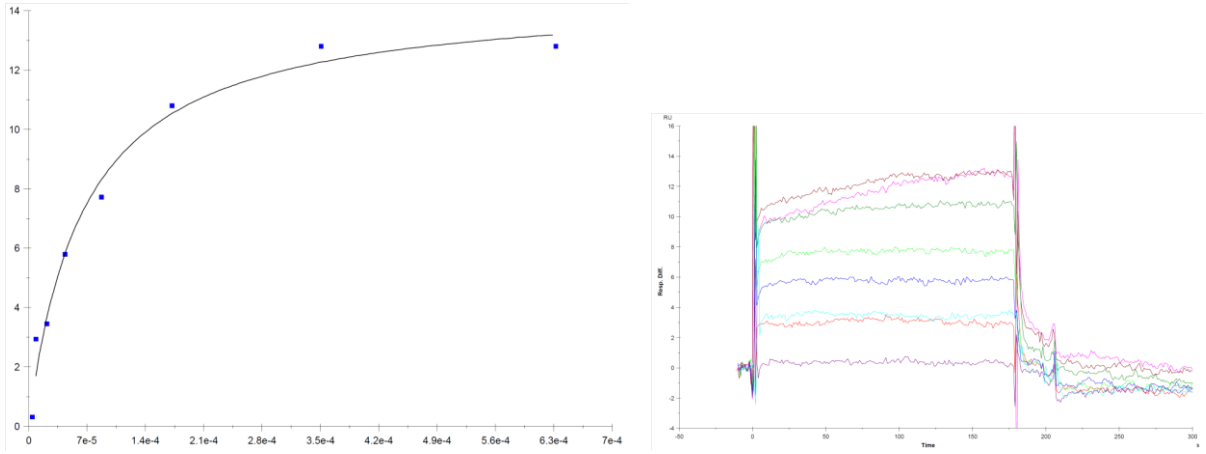


Figure S8. Sensorgrams and state steady analysis of the binding between **8j** and Galectin-3 (K_D = 65.9 µM; Chi^2 = 0.445 RU^2

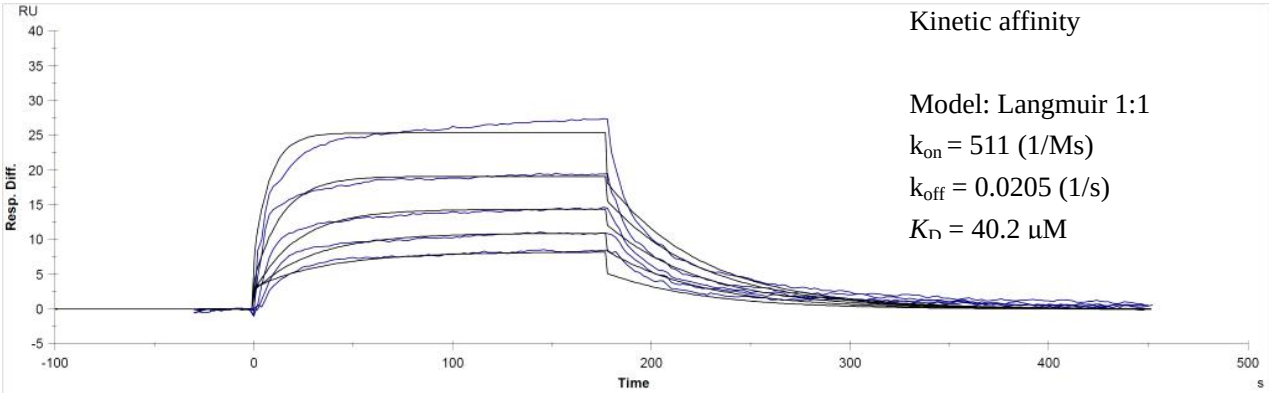


Figure S9. Sensorgrams and kinetic analysis of the binding between **8i** and Galectin-3 (12.5µM, 25µM, 50µM, 100µM, 200µM)

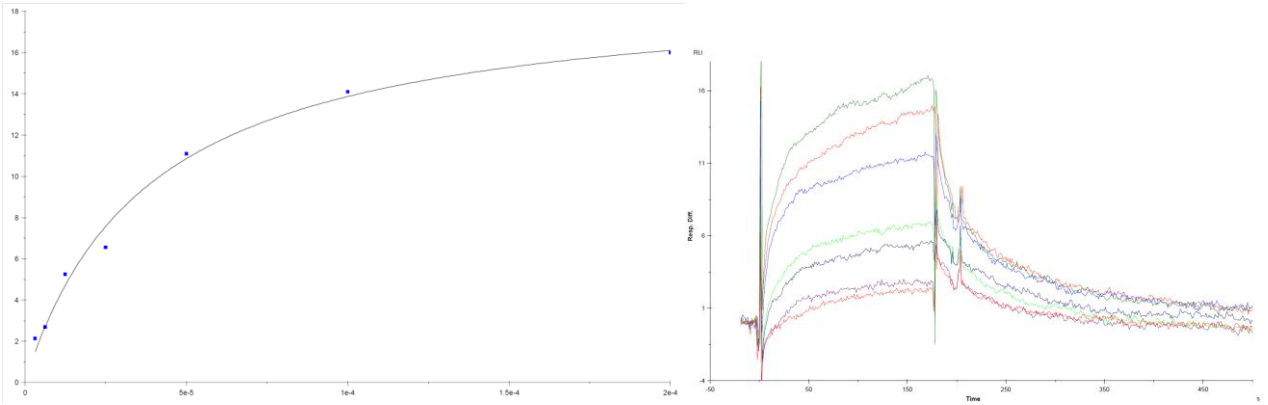


Figure S10. Sensorgrams and state steady analysis of the binding between **8o** and Galectin-3 (K_D = 38.5 μM; Chi^2 = 0.386 RU^2)

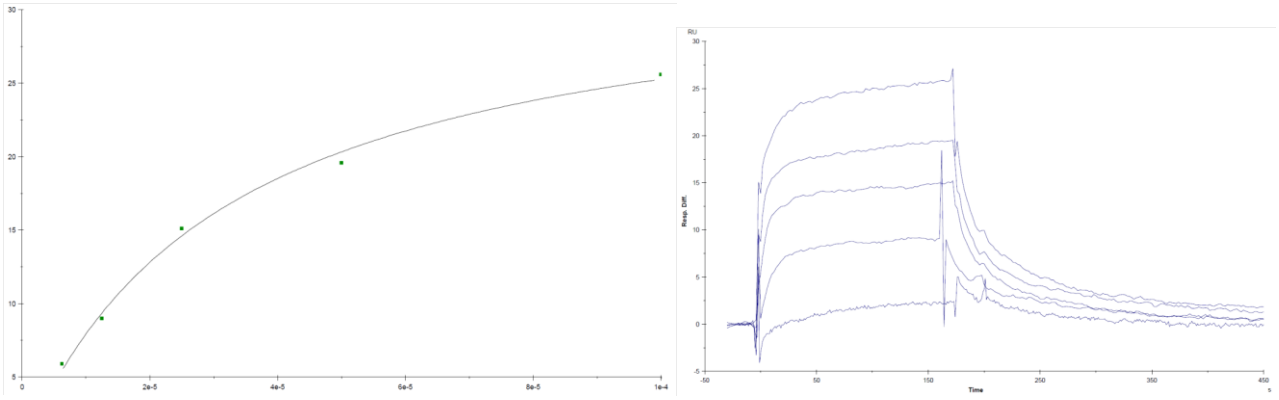


Figure S11. Sensorgrams and state steady analysis of the binding between **8p** and Galectin-3 (K_D = 32.1μM; Chi^2 = 0.413 RU^2)

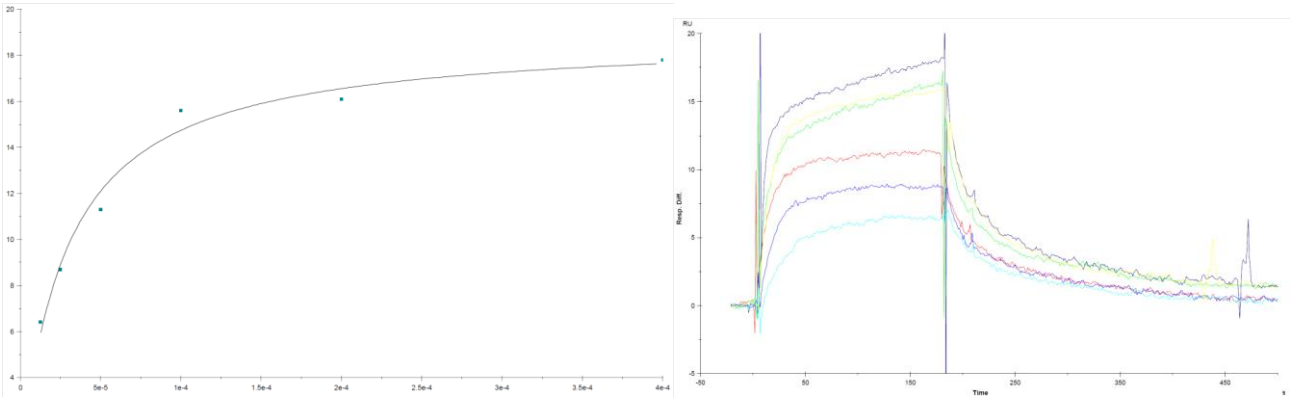


Figure S12. Sensorgrams and state steady analysis of the binding between **8l** and Galectin-3 (K_D = 28.0 μM; Chi^2 = 0.497 RU^2)

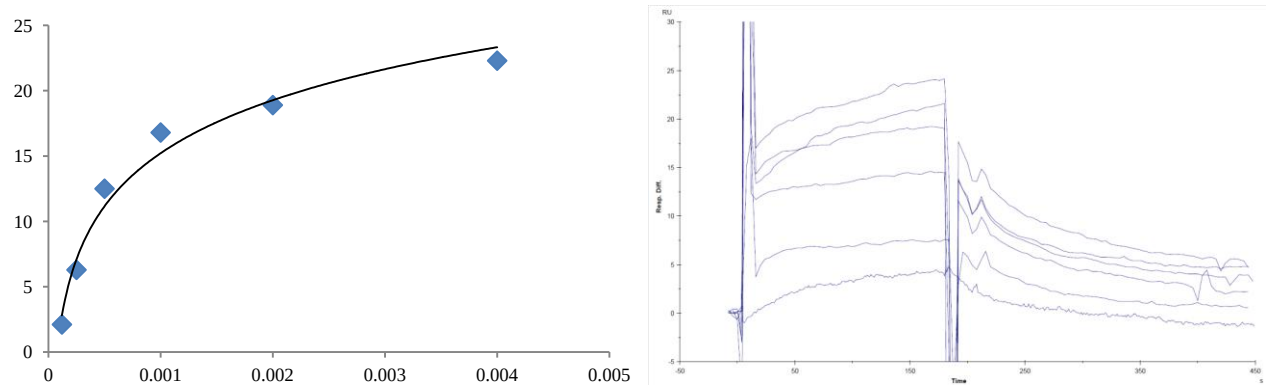


Figure S13. Sensorgrams and state steady analysis of the binding between **8m** and Galectin-3 (K_D = 41.3 μM; Chi^2 = 0.689RU^2)

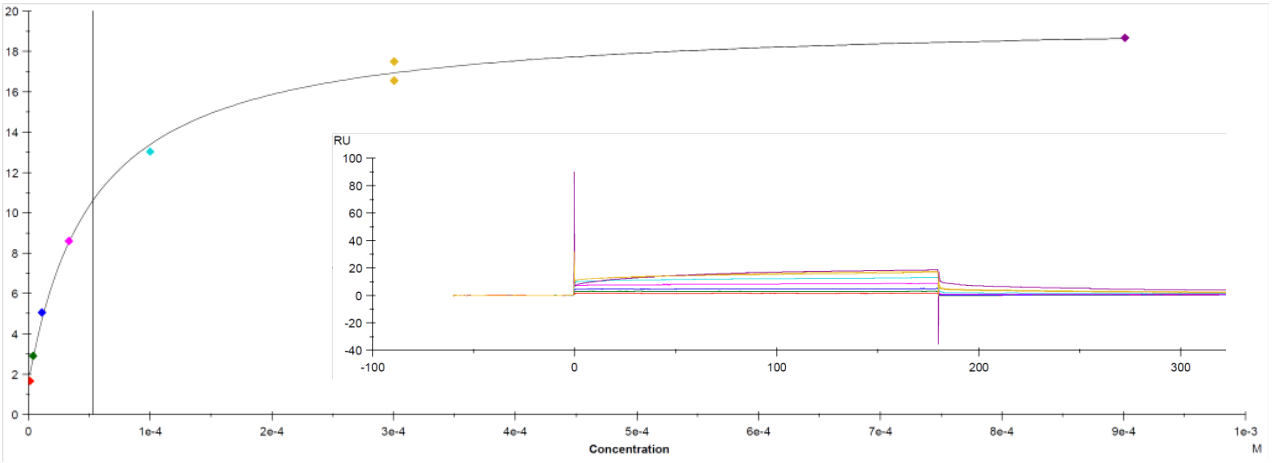


Figure S14. Sensorgrams and state steady analysis of the binding between **8k** and Galectin-3 (K_D = 53.1 μM; Chi^2 = 0.165 RU^2)