

Supporting Information

for

S-Fluorenylmethyl protection of the cysteine side chain upon N^α -Fmoc deprotection

Johannes W. Wehner and Thisbe K. Lindhorst*

Address: Otto Diels Institute of Organic Chemistry, Christiana Albertina University of Kiel, Otto-Hahn-Platz 3/4, 24098 Kiel, Germany

Email: Thisbe K. Lindhorst* - tklind@oc.uni-kiel.de

* Corresponding author

**Analytical material: NMR and mass spectra of products 3-dimer, 6,
7, 8, 9 and 10**

L-Cystine-bis[2-(α -D-mannopyranosyloxy)ethyl]amide (3-dimer)

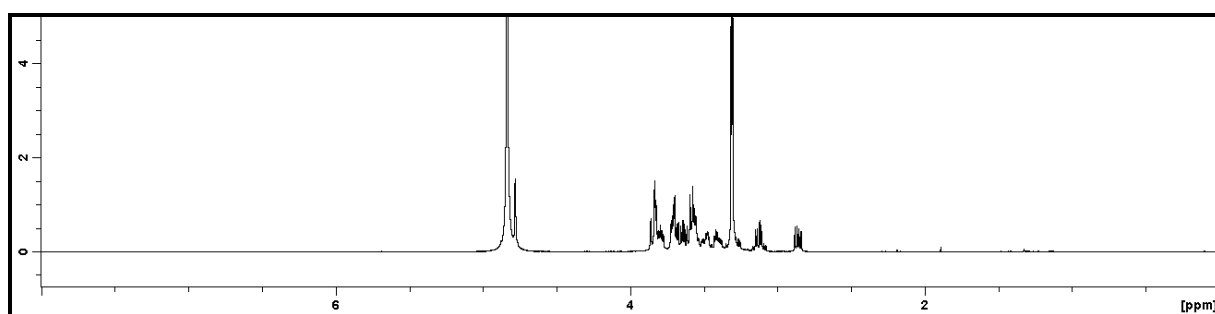
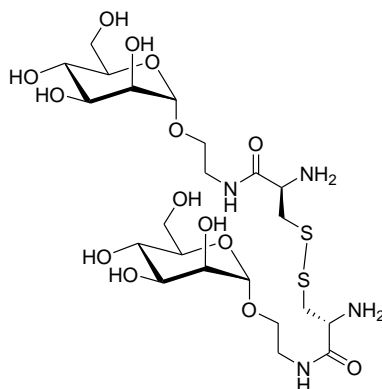


Figure S1: ^1H NMR spectrum (500 MHz, CD_3OD) of compound **3-dimer**.

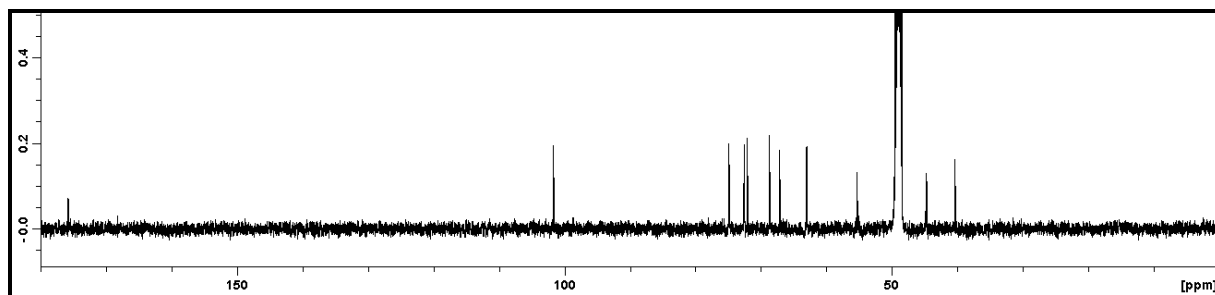


Figure S2: ^{13}C NMR spectrum (125 MHz, CD_3OD) of compound **3-dimer**.

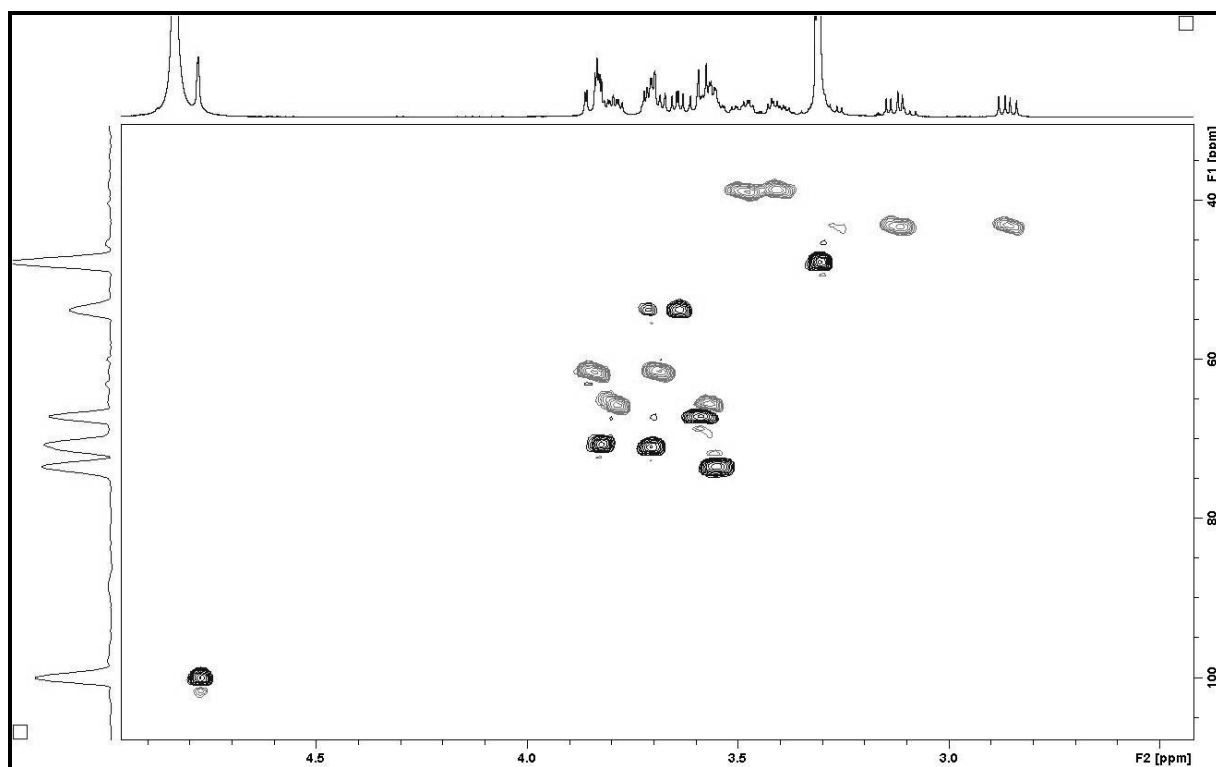


Figure S3: $^1\text{H}/^{13}\text{C}$ HSQC NMR spectrum (125 MHz, CD_3OD) of compound **3-dimer**.

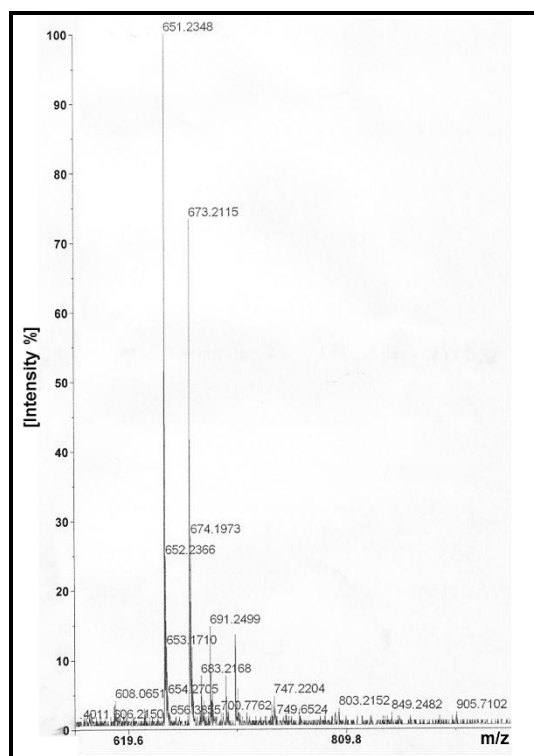


Figure S4: ESI mass spectrum of compound **3-dimer** (m/z 651.2348 $[\text{M} + \text{H}]^+$ and m/z 673.2115 $[\text{M} + \text{Na}]^+$).

L-Cysteine-[2-(2,3,4,6-tetra-*O*-acetyl- α -D-mannopyranosyloxy)ethyl]amide (6)

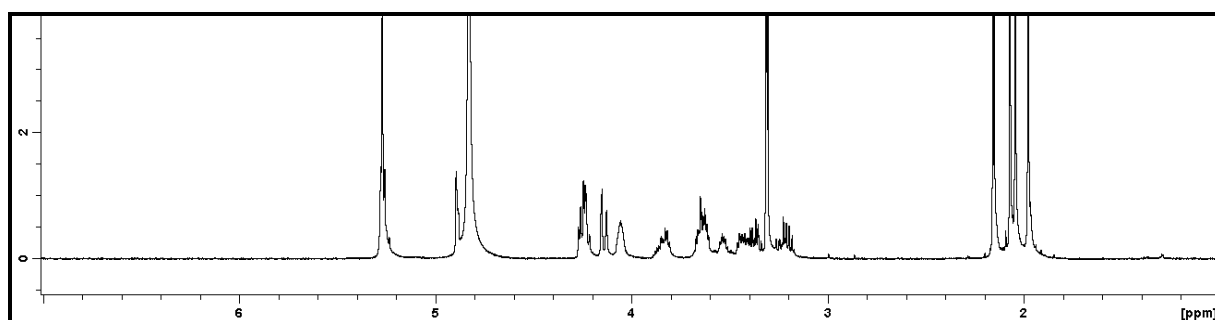
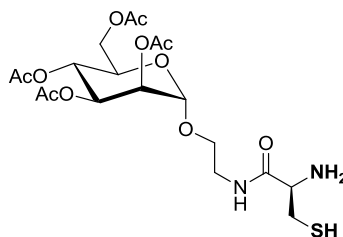


Figure S5: ^1H NMR spectrum (500 MHz, CD_3OD) of compound **6**.

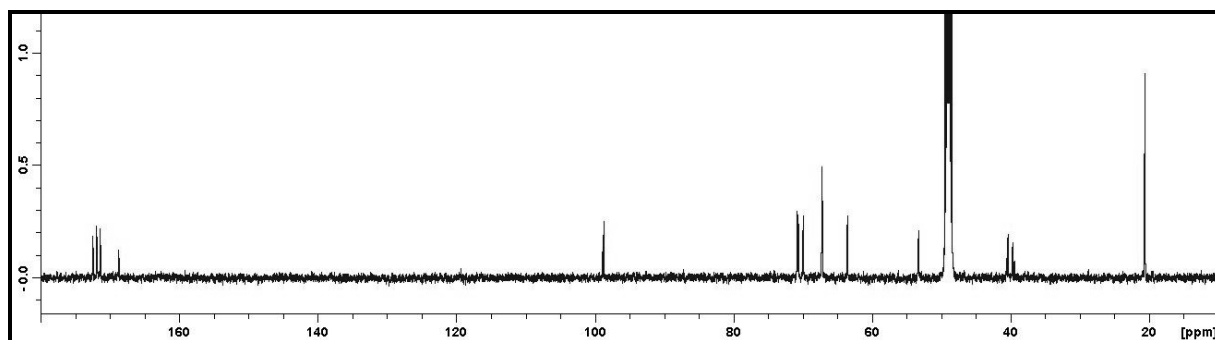


Figure S6: ^{13}C NMR spectrum (125 MHz, CD_3OD) of compound **6**.

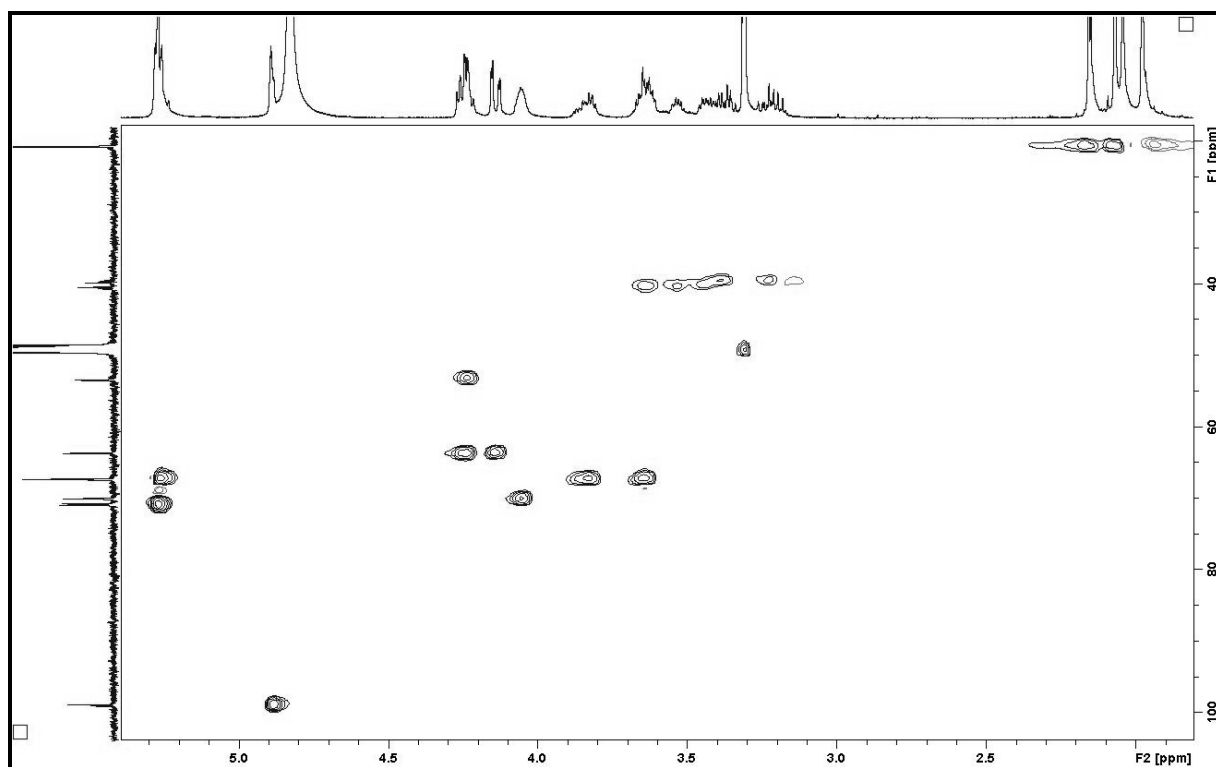


Figure S7: $^1\text{H}/^{13}\text{C}$ HSQC NMR spectrum (125 MHz, CD_3OD) of compound **6**.

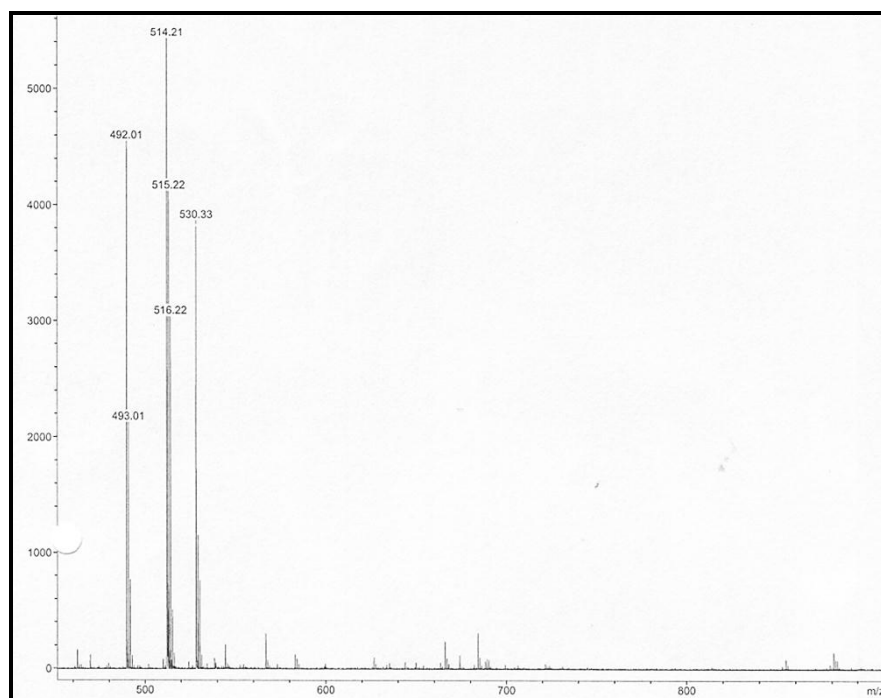


Figure S8: MALDI-TOF mass spectrum of compound **6** (m/z 492.01 $[\text{M} + \text{H}]^+$, m/z 514.21 $[\text{M} + \text{Na}]^+$ and m/z 530.33 $[\text{M} + \text{K}]^+$).

***N*-(Fluoren-9-ylmethoxycarbonyl)-L-cysteine-[2-(2,3,4,6-tetra-*O*-acetyl- α -D-mannopyranosyloxy)ethyl]amide (7)**

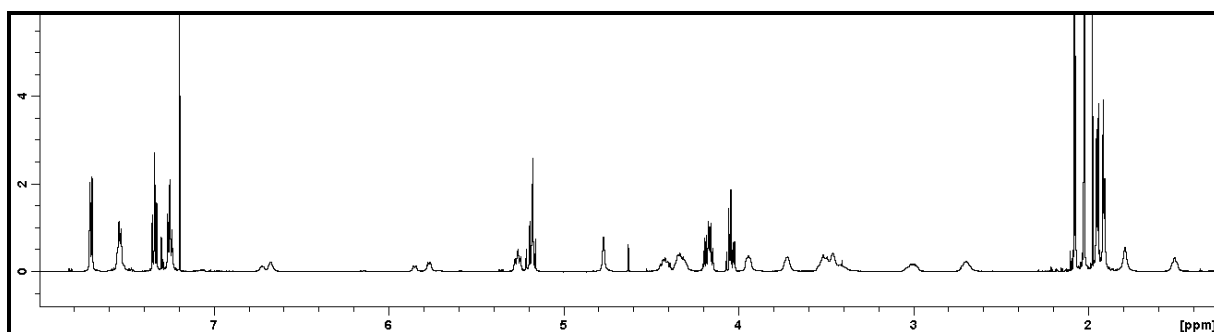
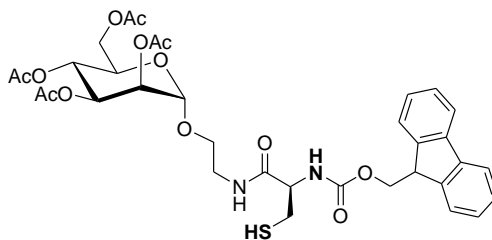


Figure S9: ¹H NMR spectrum (600 MHz, CDCl₃) of compound 7.

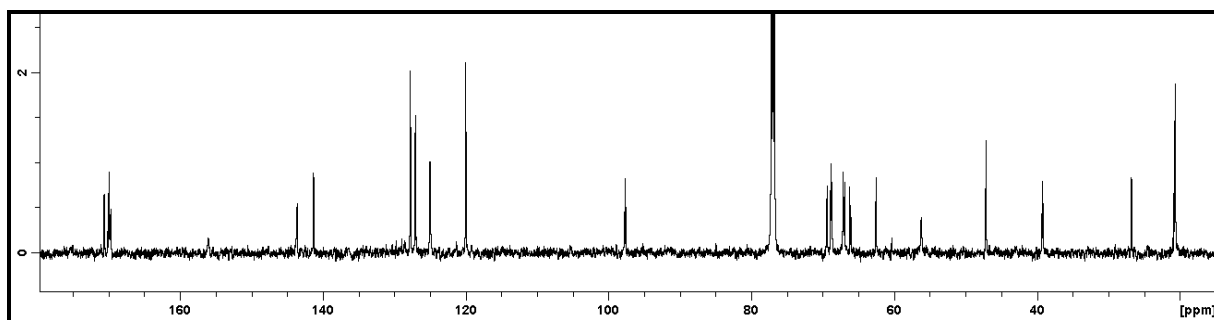


Figure S10: ¹³C NMR spectrum (150 MHz, CDCl₃) of compound 7.

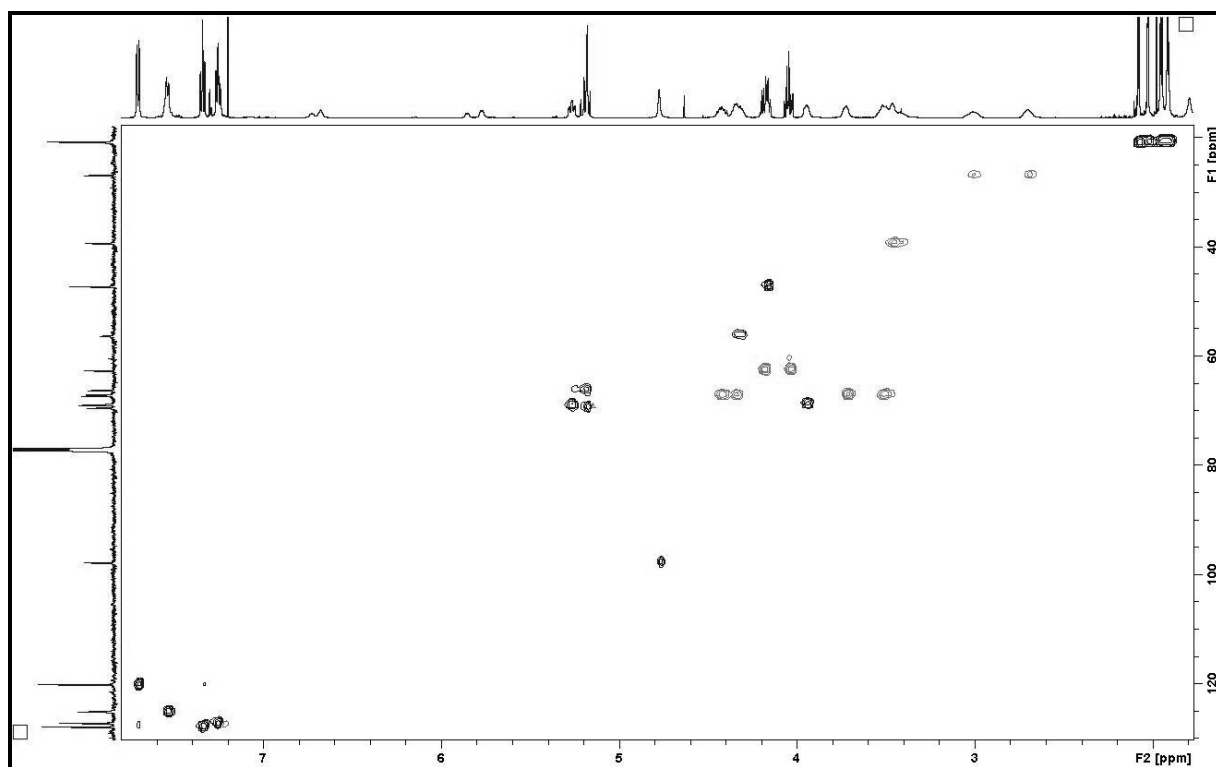


Figure S11: $^1\text{H}/^{13}\text{C}$ HSQC NMR spectrum (150 MHz, CDCl_3) of compound **7**.

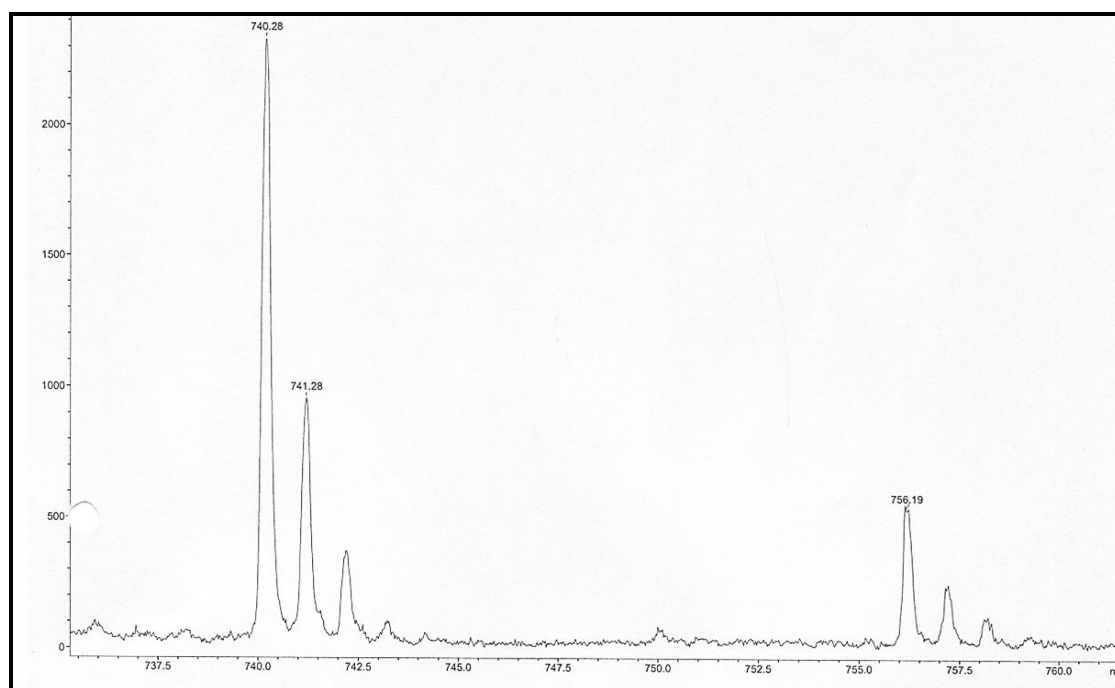


Figure S12: MALDI-TOF mass spectrum of compound **7** (m/z 740.28 $[\text{M} + \text{Na}]^+$ and m/z 756.19 $[\text{M} + \text{K}]^+$).

S-(Fluoren-9-yl)-L-cysteine-[2-(2,3,4,6-tetra-O-acetyl- α -D-mannopyranosyloxy)ethyl]amide (8)

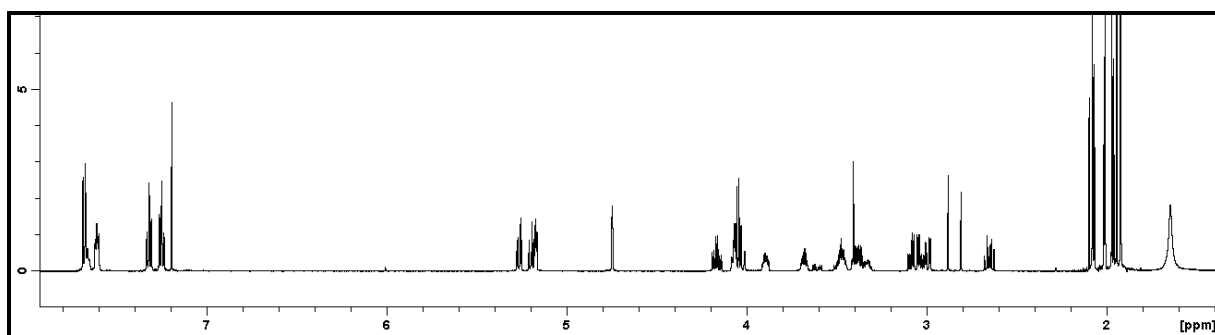
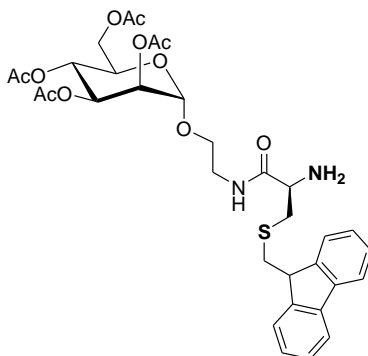


Figure S13: ^1H NMR spectrum (600 MHz, CDCl_3) of compound 8.

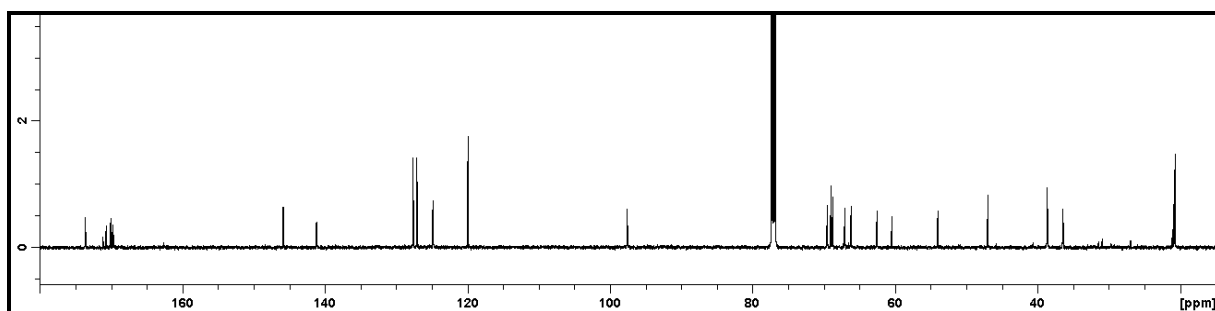


Figure S14: ^{13}C NMR spectrum (150 MHz, CDCl_3) of compound 8.

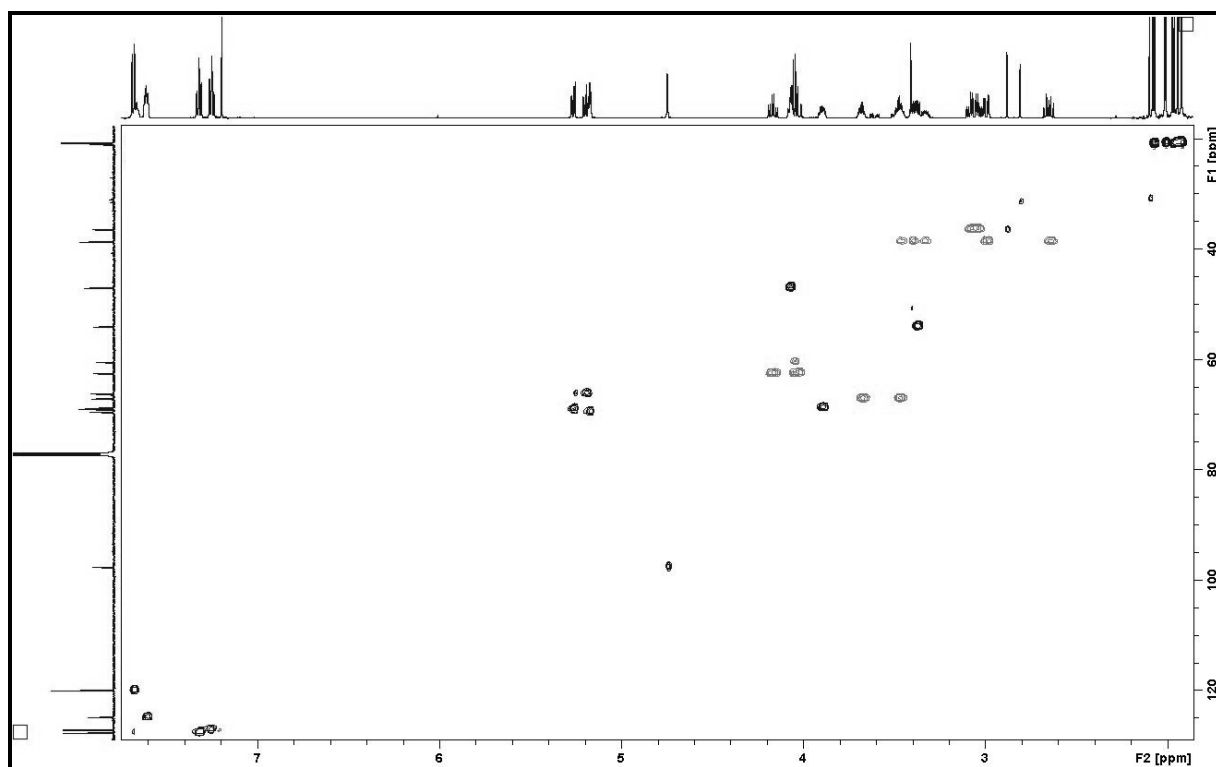


Figure S15: $^1\text{H}/^{13}\text{C}$ HSQC NMR spectrum (150 MHz, CDCl_3) of compound **8**.

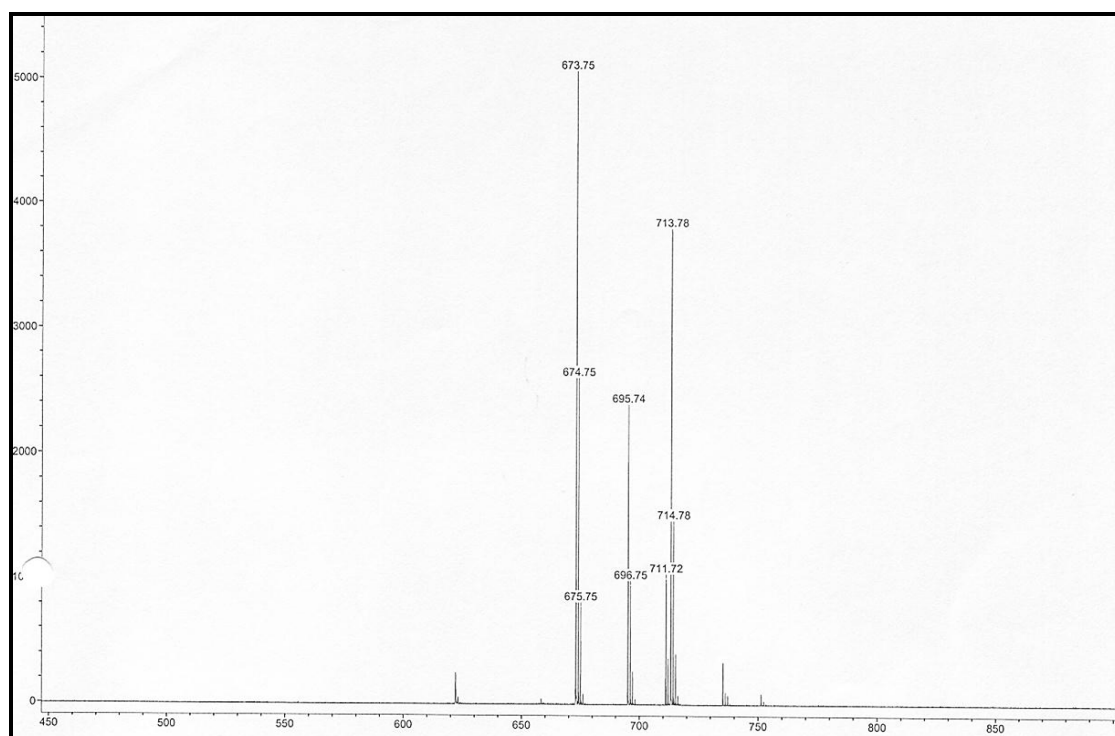


Figure S16: MALDI-TOF mass spectrum of compound **8** (m/z 673.75 $[\text{M} + \text{H}]^+$, m/z 695.74 $[\text{M} + \text{Na}]^+$ and m/z 713.78 $[\text{M} + \text{K}]^+$).

S-(Fluoren-9-yl)-L-cysteine-[2-(α -D-mannopyranosyloxy)ethyl]amide (9)

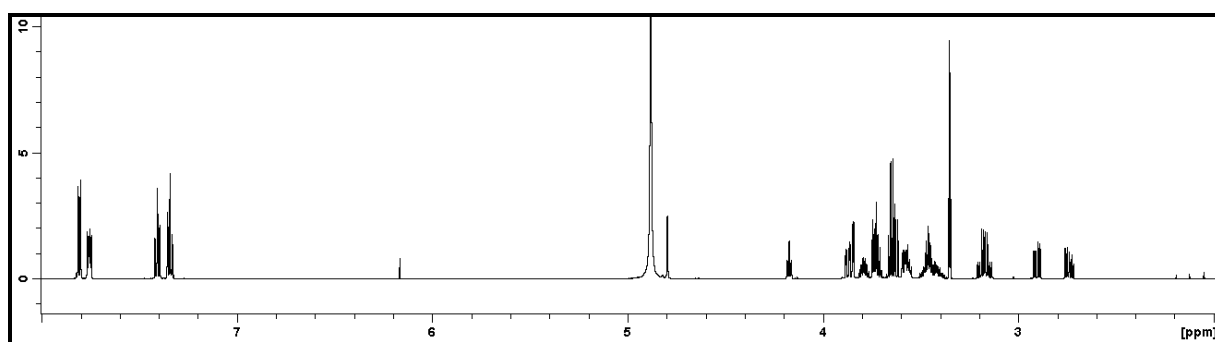
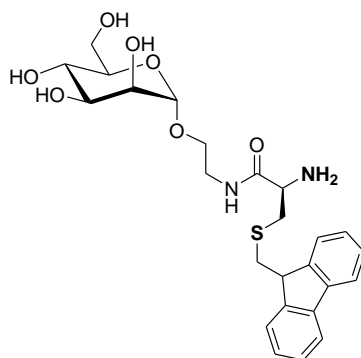


Figure S17: ^1H NMR spectrum (600 MHz, CD_3OD) of compound 9.

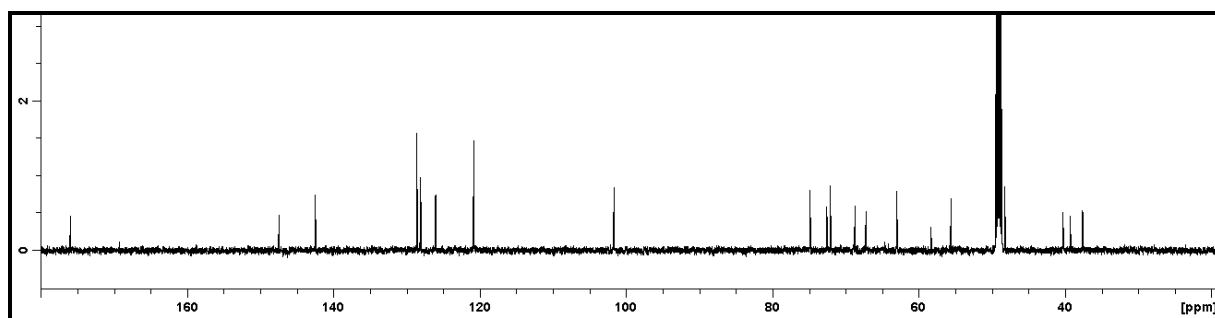


Figure S18: ^{13}C NMR spectrum (150 MHz, CD_3OD) of compound 9.

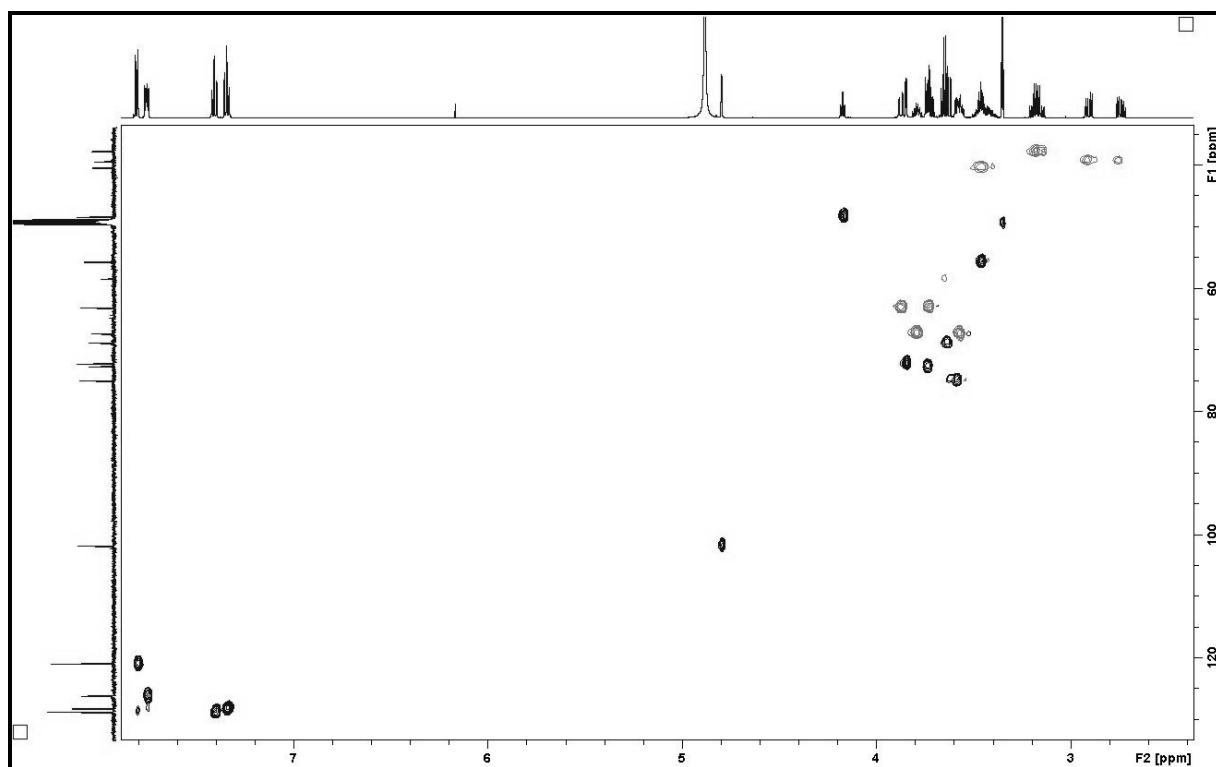


Figure S19: $^1\text{H}/^{13}\text{C}$ HSQC NMR spectrum (150 MHz, CD_3OD) of compound **9**.

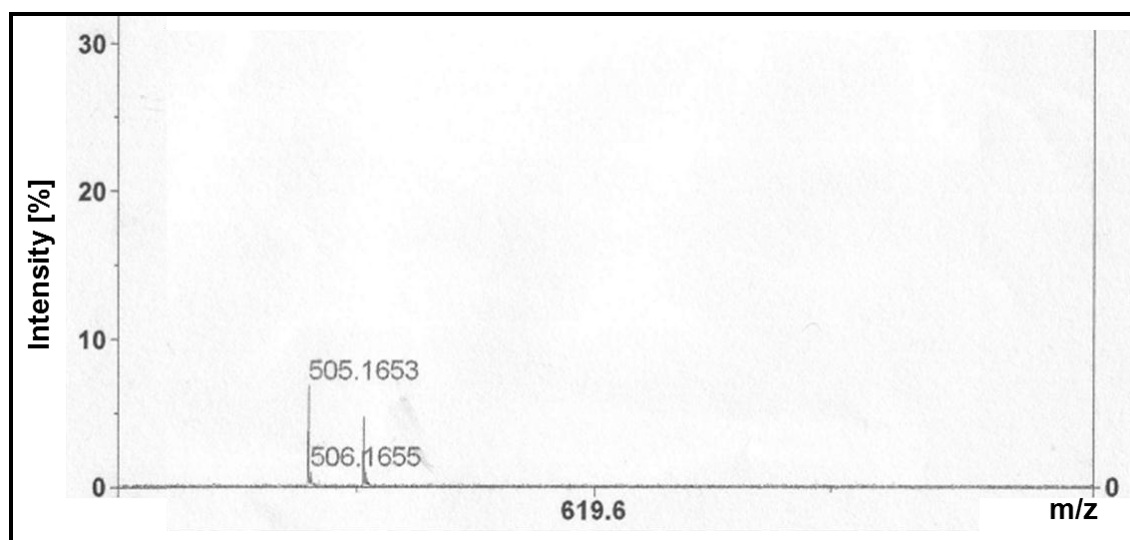


Figure S20: ESI mass spectrum of compound **9** (m/z 505.1653 [$\text{M} + \text{H}$] $^+$).

***N*-(Fluoren-9-ylmethoxycarbonyl)-*S*-(fluoren-9-yl)-*L*-cysteine-[2-(2,3,4,6-tetra-*O*-acetyl- α -*D*-mannopyranosyloxy)ethyl]amide (10)**

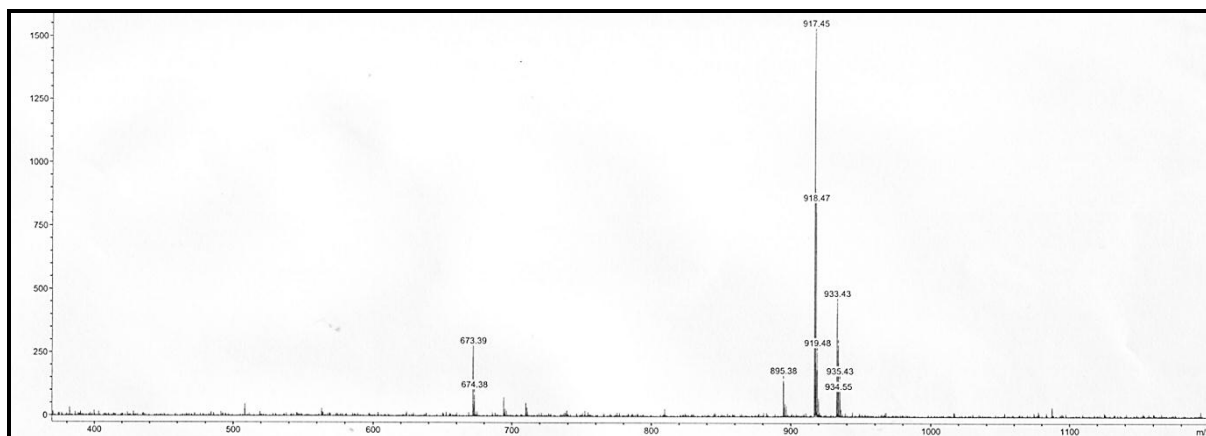
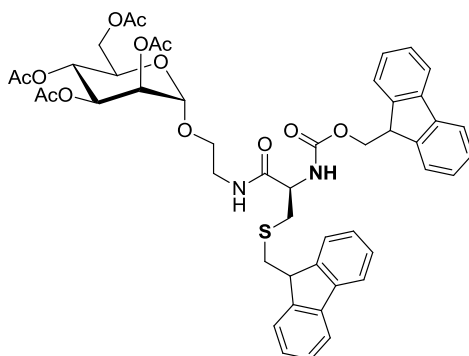


Figure S21: MALDI-TOF mass spectrum of compound **10** (m/z 895.38 $[M + H]^+$, m/z 917.45 $[M + Na]^+$ and m/z 933.43 $[M + K]^+$); m/z 673.39 is caused by the loss of the *N*-Fmoc-protecting group during ionization.