P-Stereogenic ligands with the *tert*-butylmethylphosphine fragment. Coordination chemistry and catalysis of their organometallic complexes

Guillem Vázquez Bigas

Chapter 2: Synthesis of emissive chelating peptides

3-(1H-Benzotriazol-1-ylcarbonyl)-2H-chromen-2-one

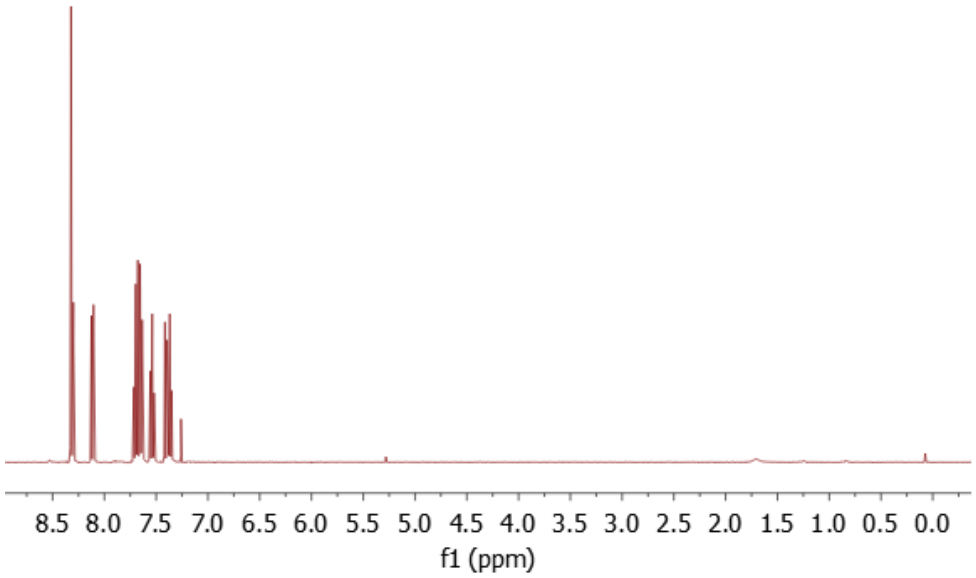

Figure S2.1: ^{1}H NMR spectrum of 3-(1*H*-Benzotriazol-1-ylcarbonyl)-2*H*-chromen-2-one in $CDCl_3$.

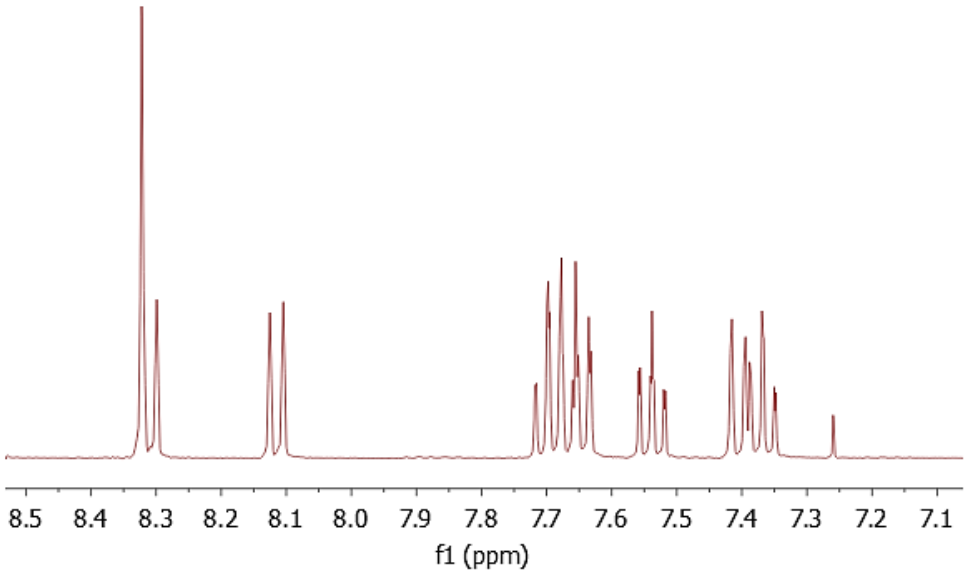

Figure S2.2: ^{1}H NMR spectrum of 3-(1*H*-Benzotriazol-1-ylcarbonyl)-2*H*-chromen-2-one in $CDCl_3$ (magnified).

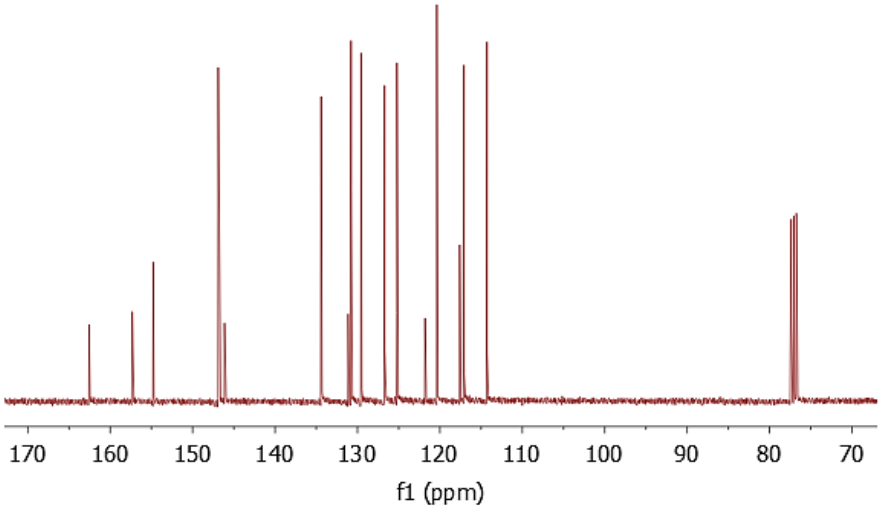

Figure S2.3: ^{13}C NMR spectrum of 3-(1*H*-Benzotriazol-1-ylcarbonyl)-2*H*-chromen-2-one in $CDCl_3$.

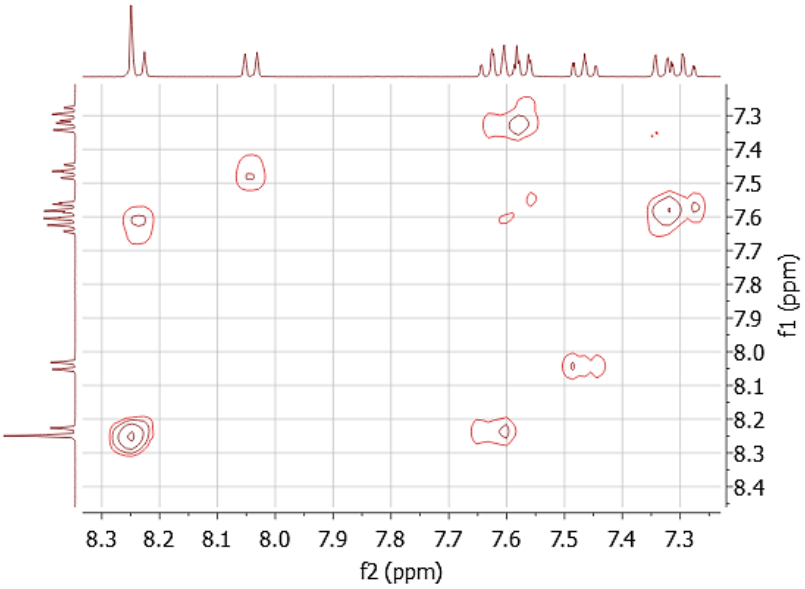

Figure S2.4: COSY NMR spectrum of 3-(1*H*-Benzotriazol-1-ylcarbonyl)-2*H*-chromen-2-one in $CDCl_3$.

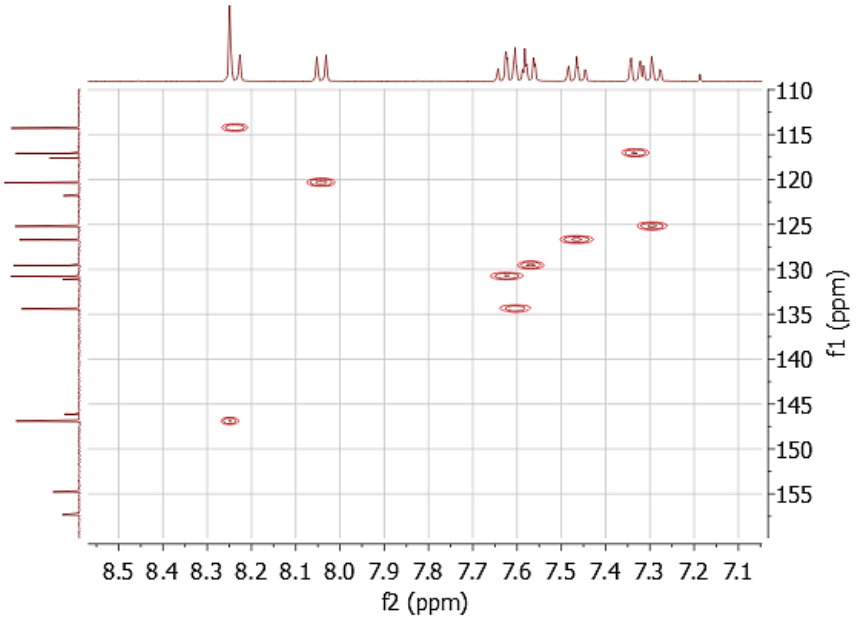

Figure S2.5: HSQC NMR spectrum of 3-(1*H*-Benzotriazol-1-ylcarbonyl)-2*H*-chromen-2-one in $CDCl_3$.

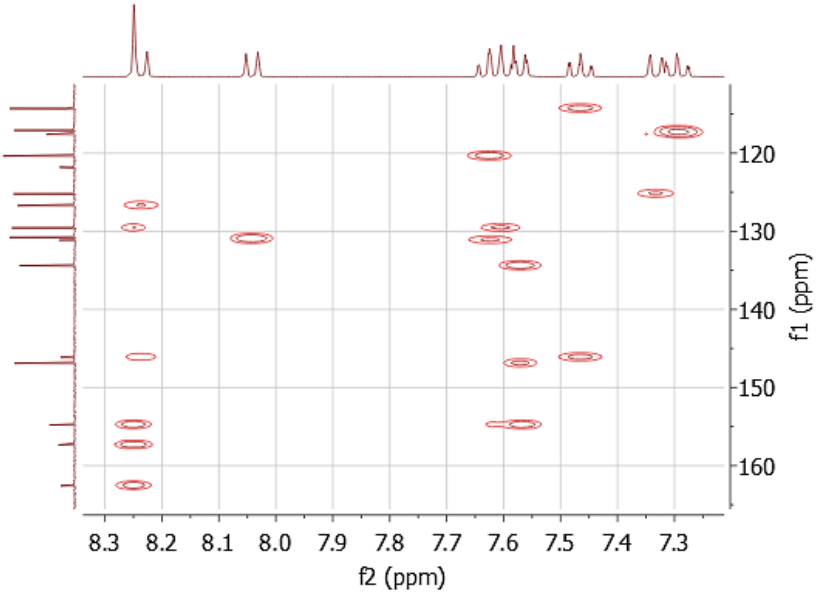

Figure S2.6: HMBC NMR spectrum of 3-(1*H*-Benzotriazol-1-ylcarbonyl)-2*H*-chromen-2-one in $CDCl_3$.

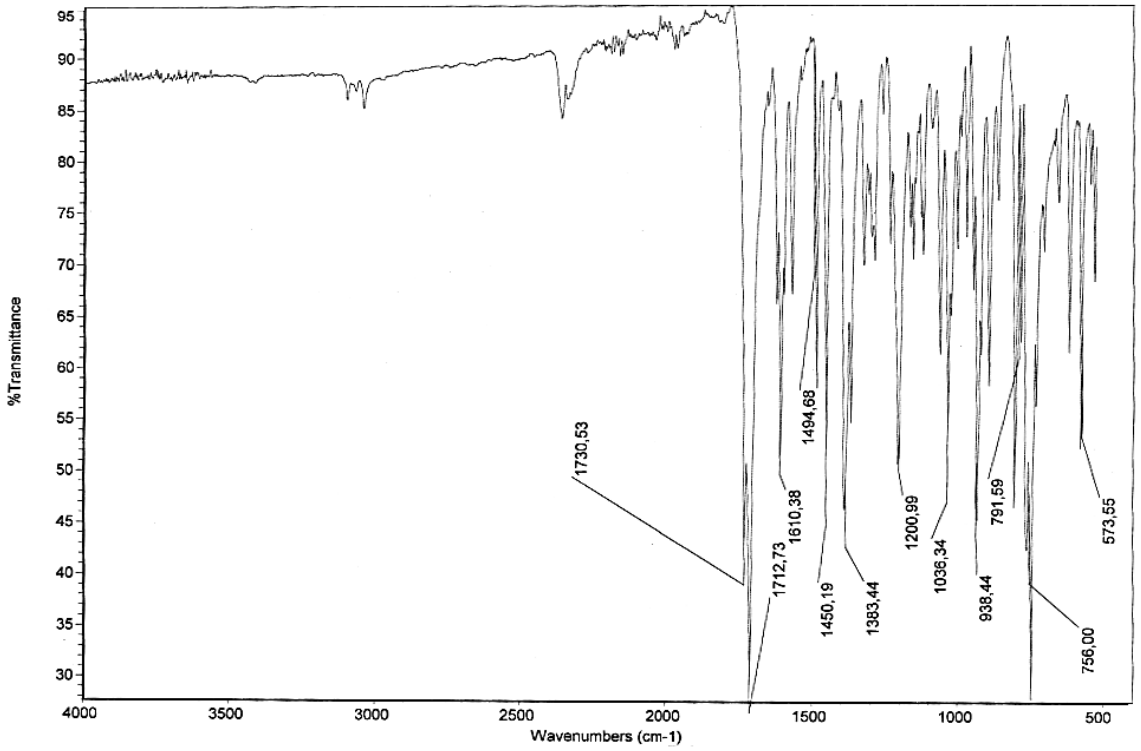

Figure S2.7: IR spectrum of 3-(1*H*-Benzotriazol-1-ylcarbonyl)-2*H*-chromen-2-one.

Fmoc-Lys(Coum)-OH

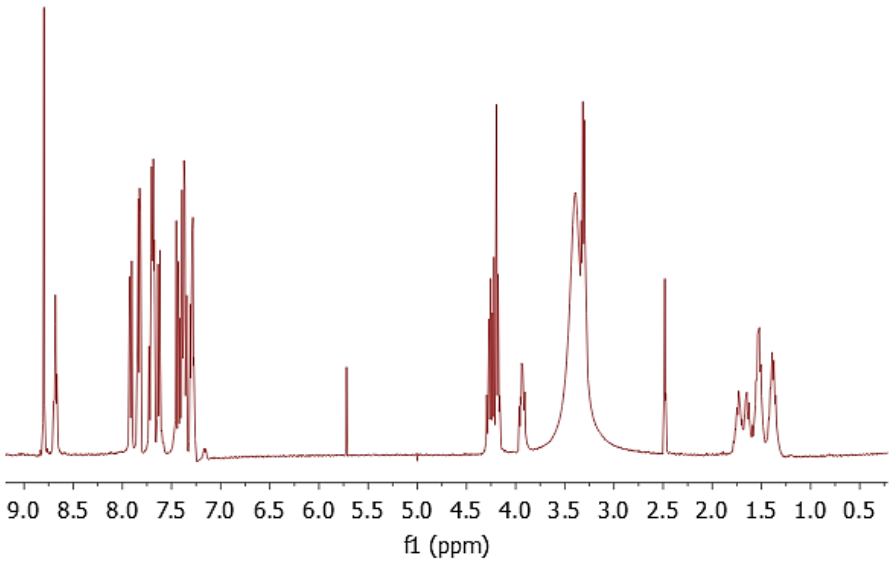

Figure S2.8: ^{1}H NMR spectrum of Fmoc-Lys(Coum)-OH in DMSO-d_6.

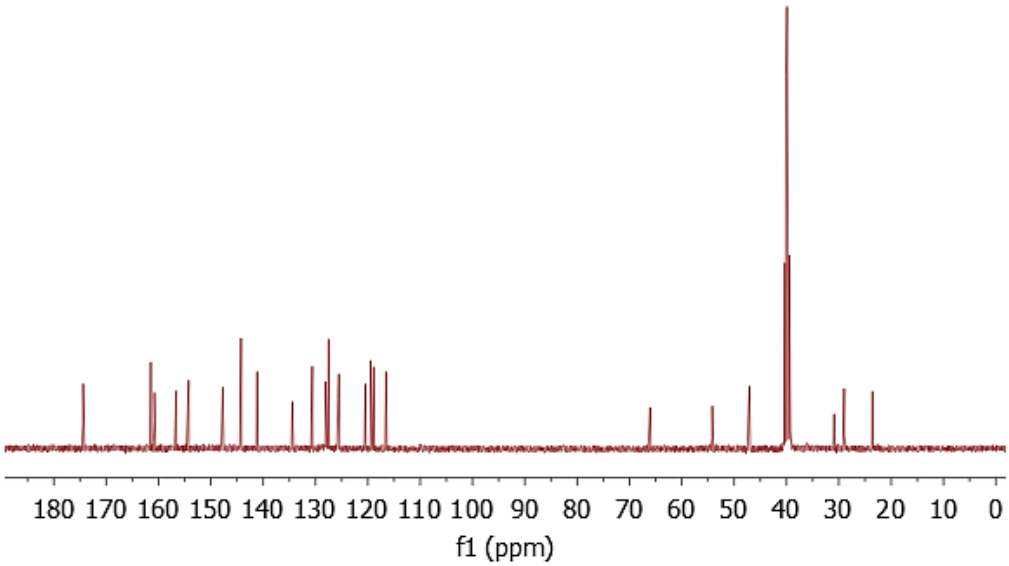

Figure S2.9: ^{13}C NMR spectrum of Fmoc-Lys(Coum)-OH in DMSO-d_6.

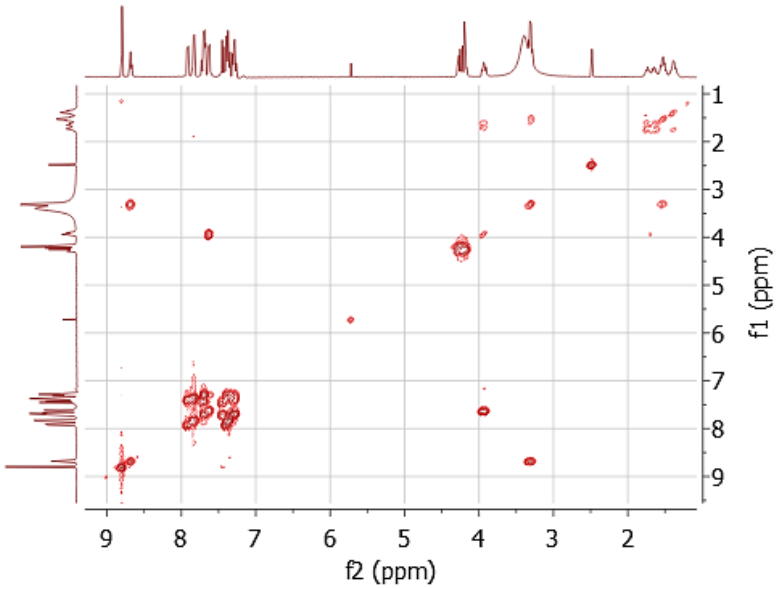

Figure S2.10: COSY NMR spectrum of Fmoc-Lys(Coum)-OH in DMSO-d_6.

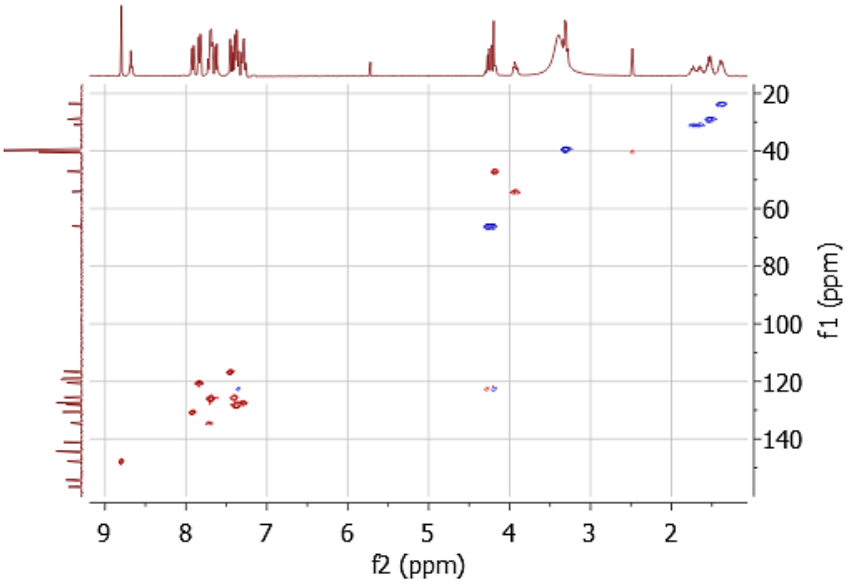

Figure S2.11: HSQC NMR spectrum of Fmoc-Lys(Coum)-OH in DMSO-d_6.

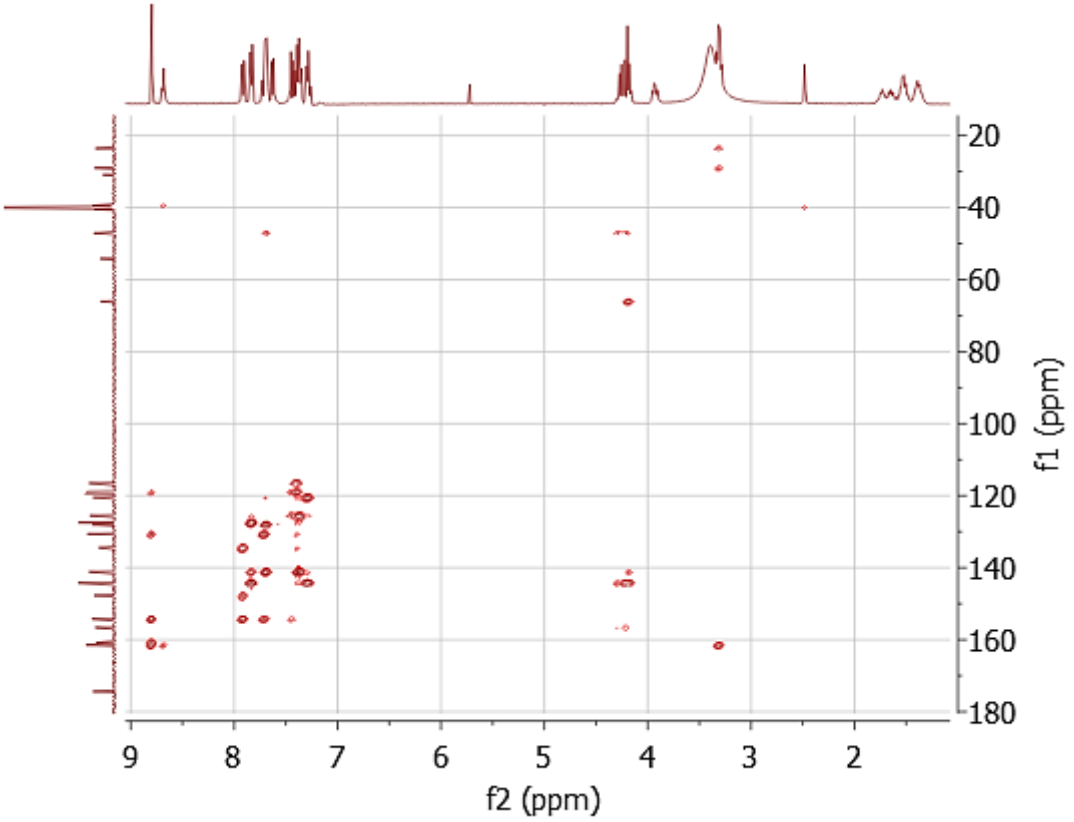

Figure S2.12: HMBC NMR spectrum of Fmoc-Lys(Coum)-OH in DMSO-d_6.

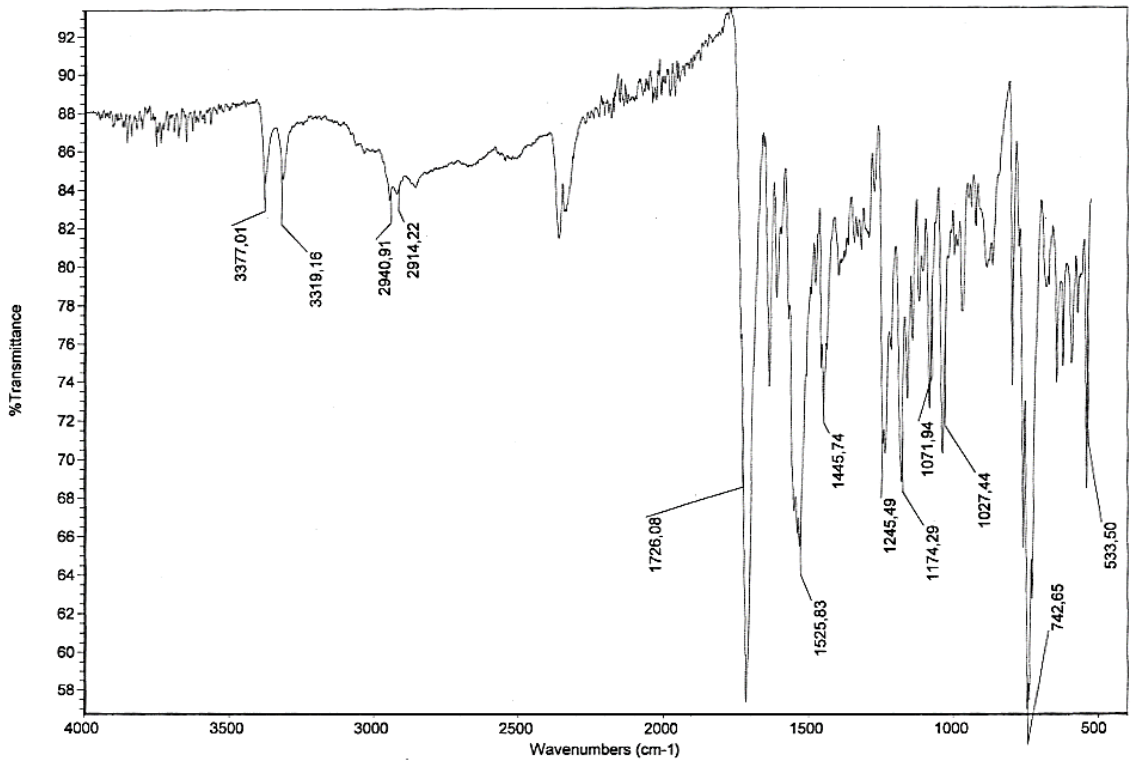

Figure S2.13: IR spectrum of Fmoc-Lys(Coum)-OH.

4-N,N-dimethylamino-1,8-naphthalic anhydride

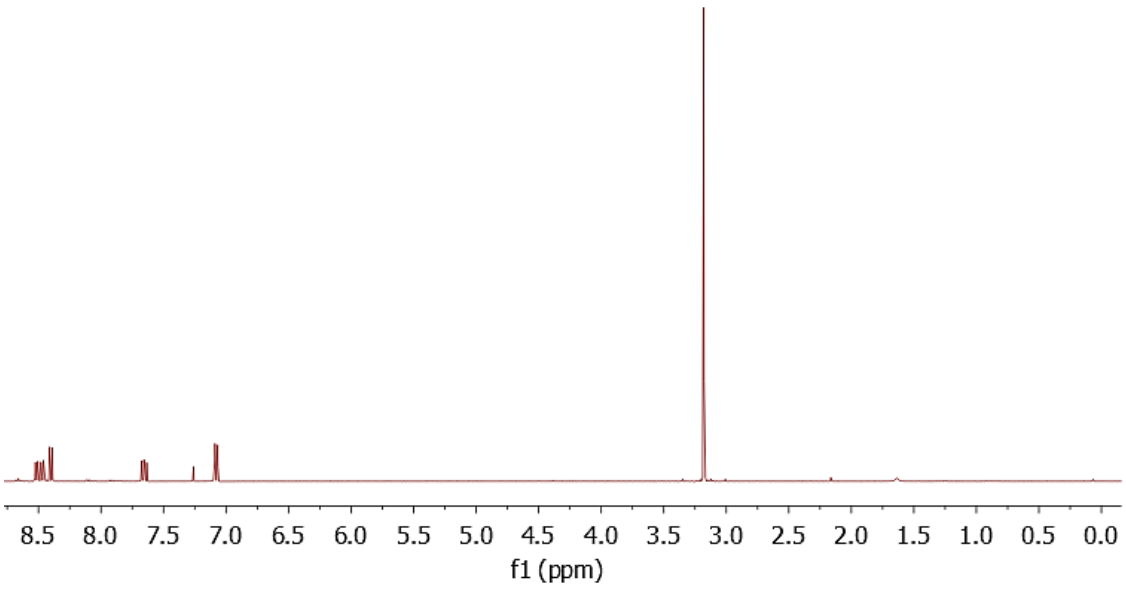

Figure S2.14: ^{1}H NMR spectrum of 4-*N,N*-dimethylamino-1,8-naphthalic anhydride in $CDCl_3$.

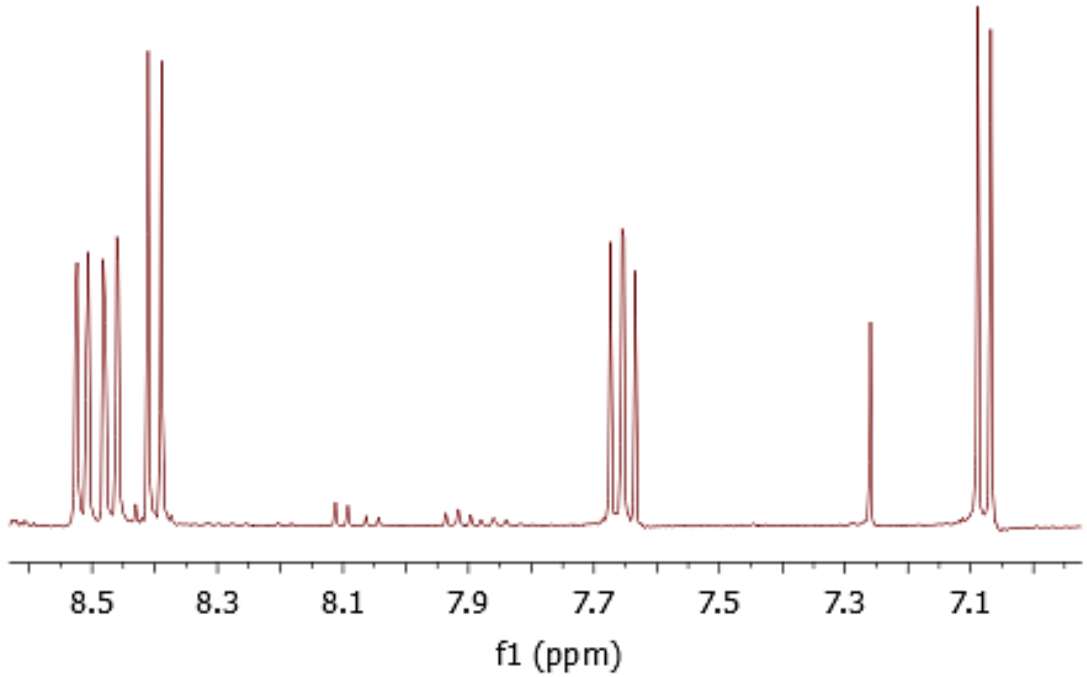

Figure S2.15: ^{1}H NMR spectrum of 4-*N,N*-dimethylamino-1,8-naphthalic anhydride in $CDCl_3$ (magnified).

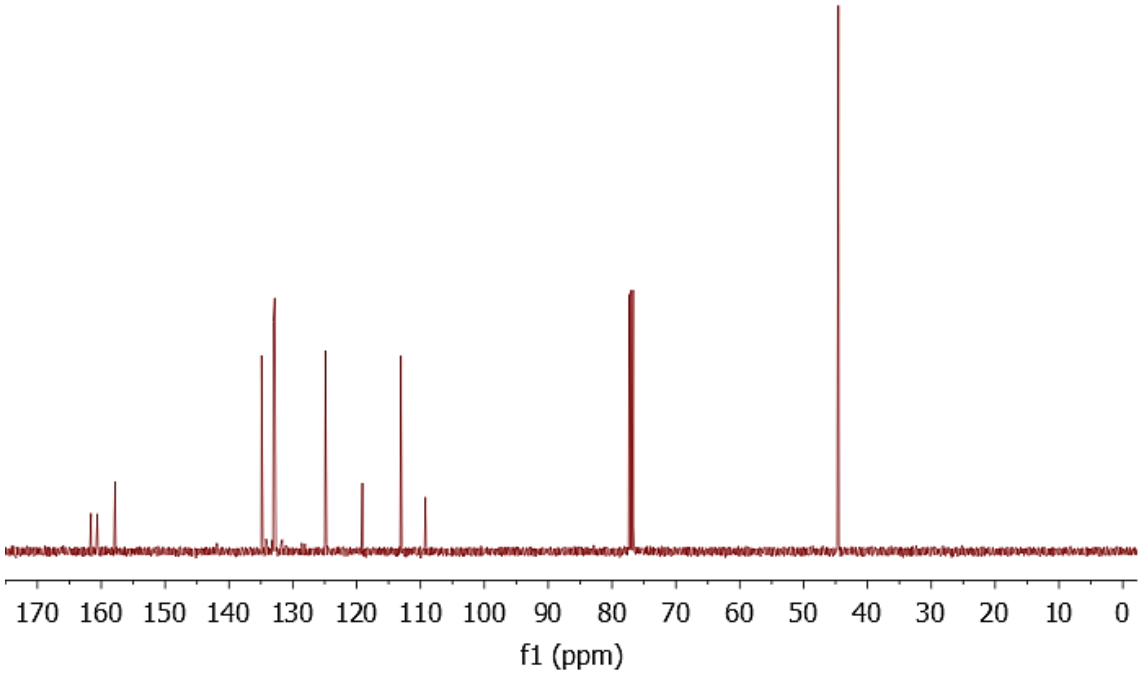

Figure S2.16: ^{13}C NMR spectrum of 4-*N,N*-dimethylamino-1,8-naphthalic anhydride in $CDCl_3$.

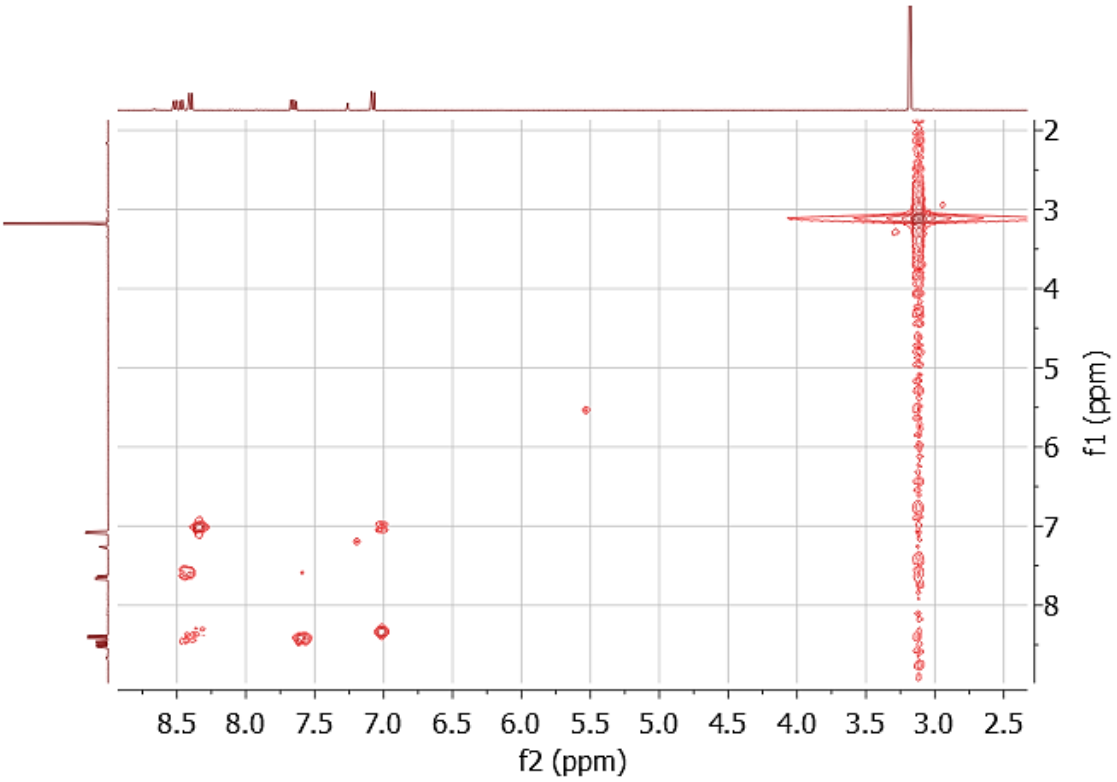

Figure S2.17: COSY NMR spectrum of 4-*N,N*-dimethylamino-1,8-naphthalic anhydride in $CDCl_3$.

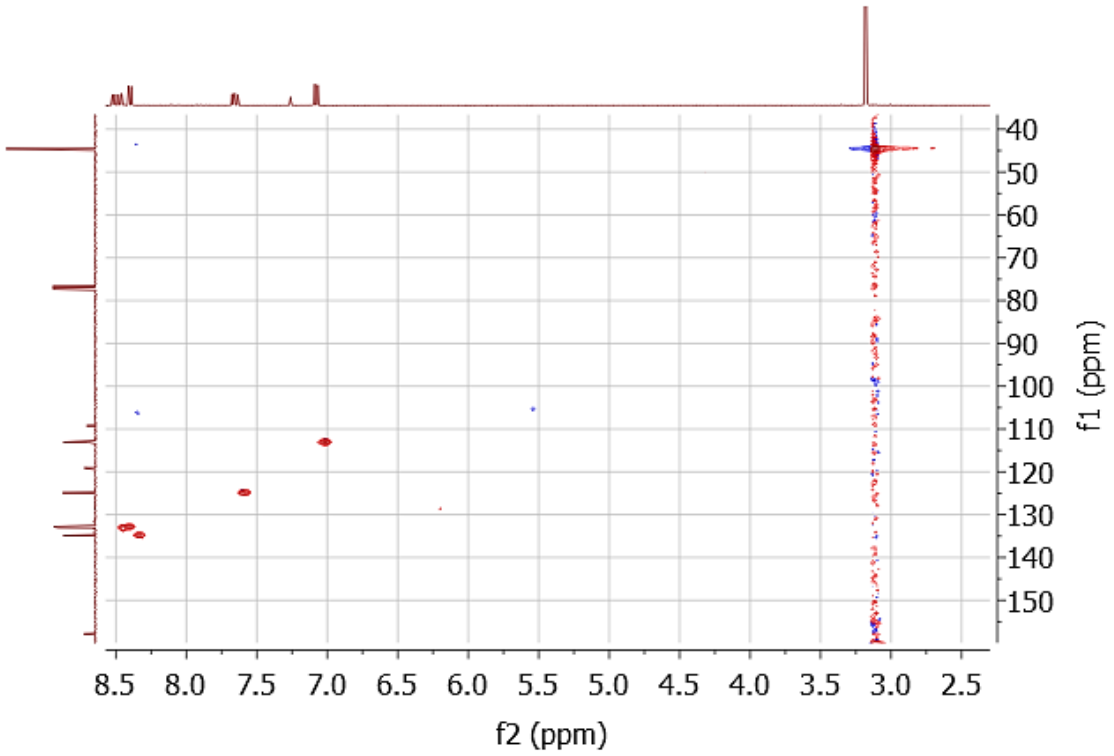

Figure S2.18: HSQC NMR spectrum of 4-*N,N*-dimethylamino-1,8-naphthalic anhydride in $CDCl_3$.

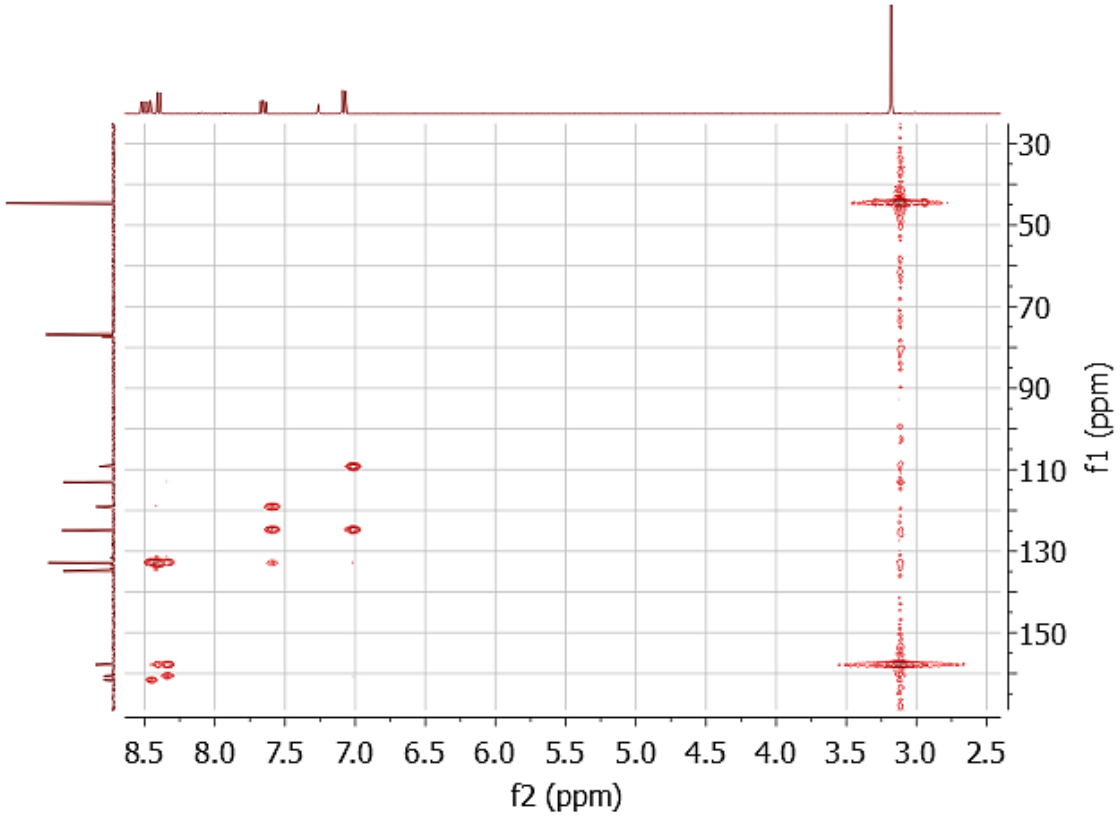

Figure S2.19: HMBC NMR spectrum of 4-*N,N*-dimethylamino-1,8-naphthalic anhydride in $CDCl_3$.

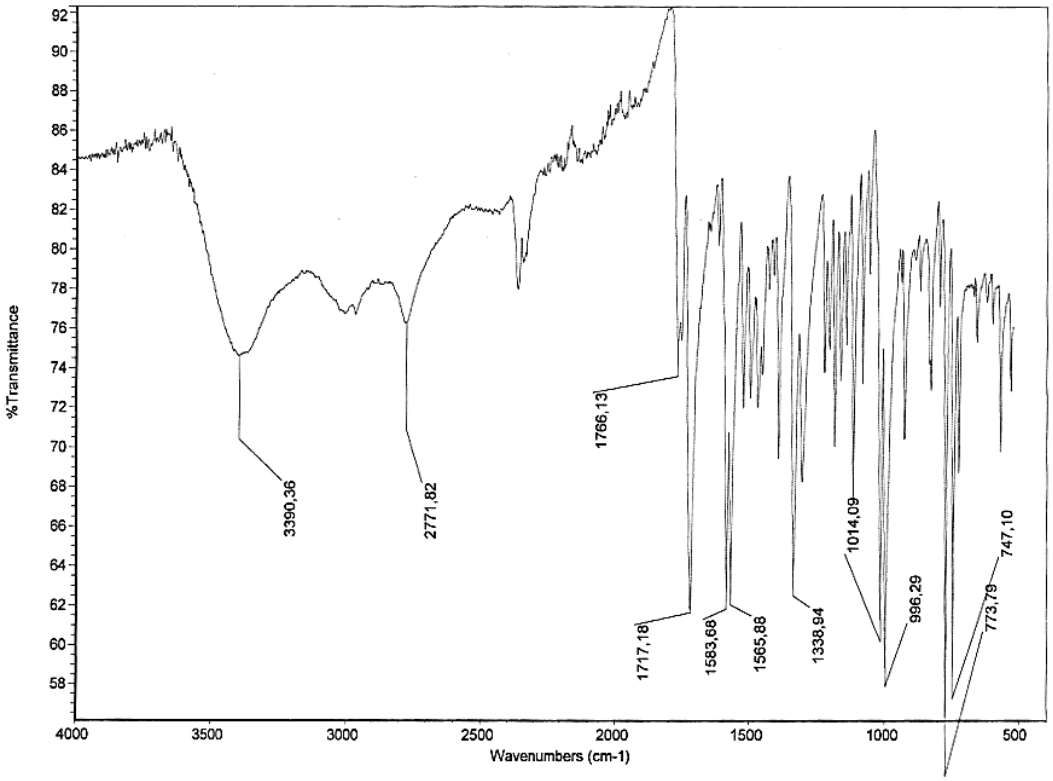

Figure S2.20: IR spectrum of 4-*N,N*-dimethylamino-1,8-naphthalic anhydride.

Boc-Lys(4DMN)-OH

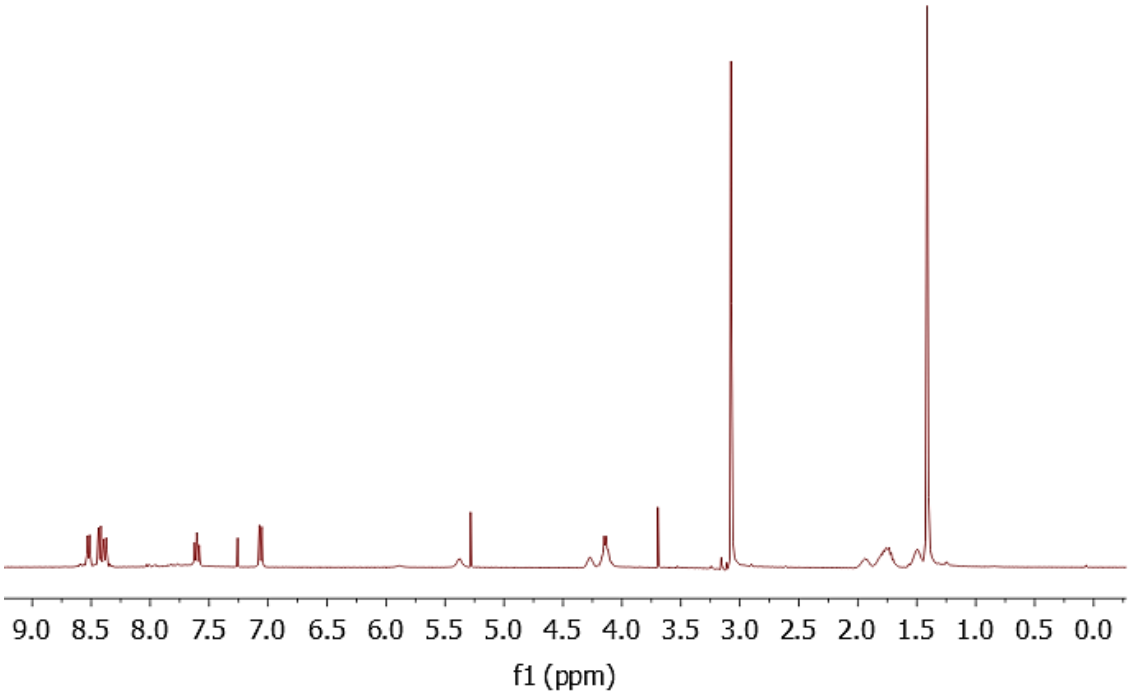

Figure S2.21: ^{1}H NMR spectrum of Boc-Lys(4DMN)-OH in $CDCl_3$.

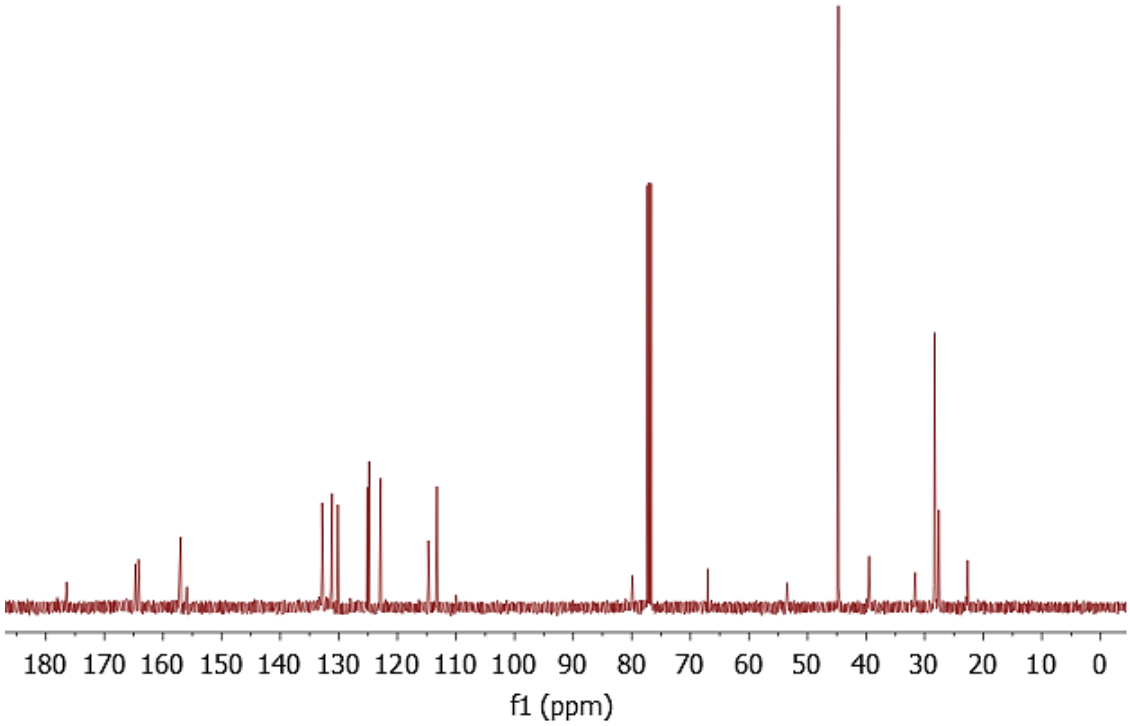

Figure S2.22: ^{13}C NMR spectrum of Boc-Lys(4DMN)-OH in $CDCl_3$.

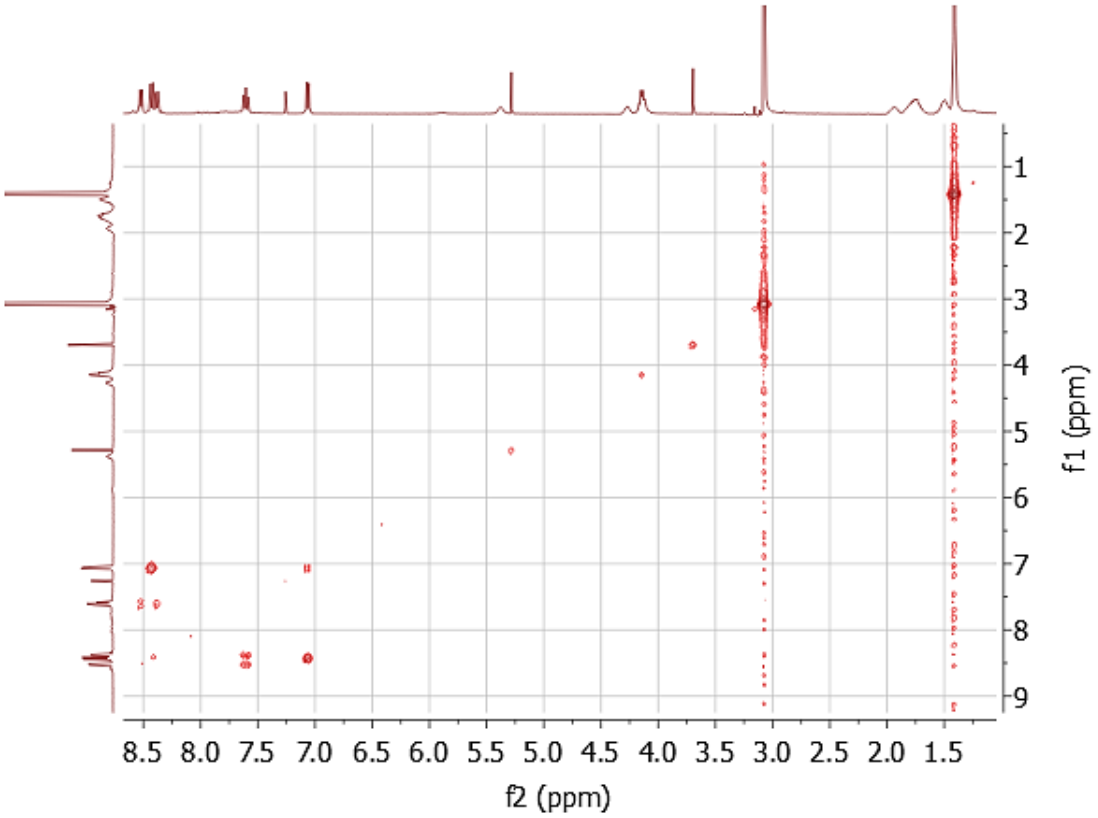

Figure S2.23: COSY NMR spectrum of Boc-Lys(4DMN)-OH in $CDCl_3$.

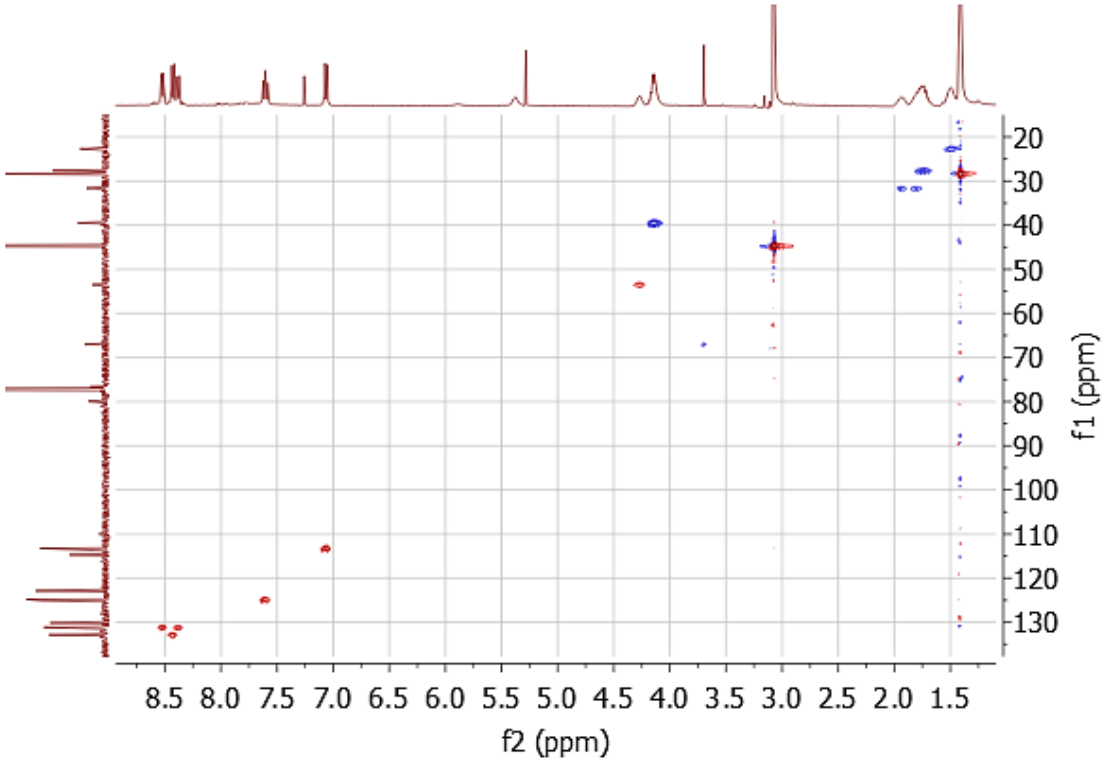

Figure S2.24: HSQC NMR spectrum of Boc-Lys(4DMN)-OH in $CDCl_3$.

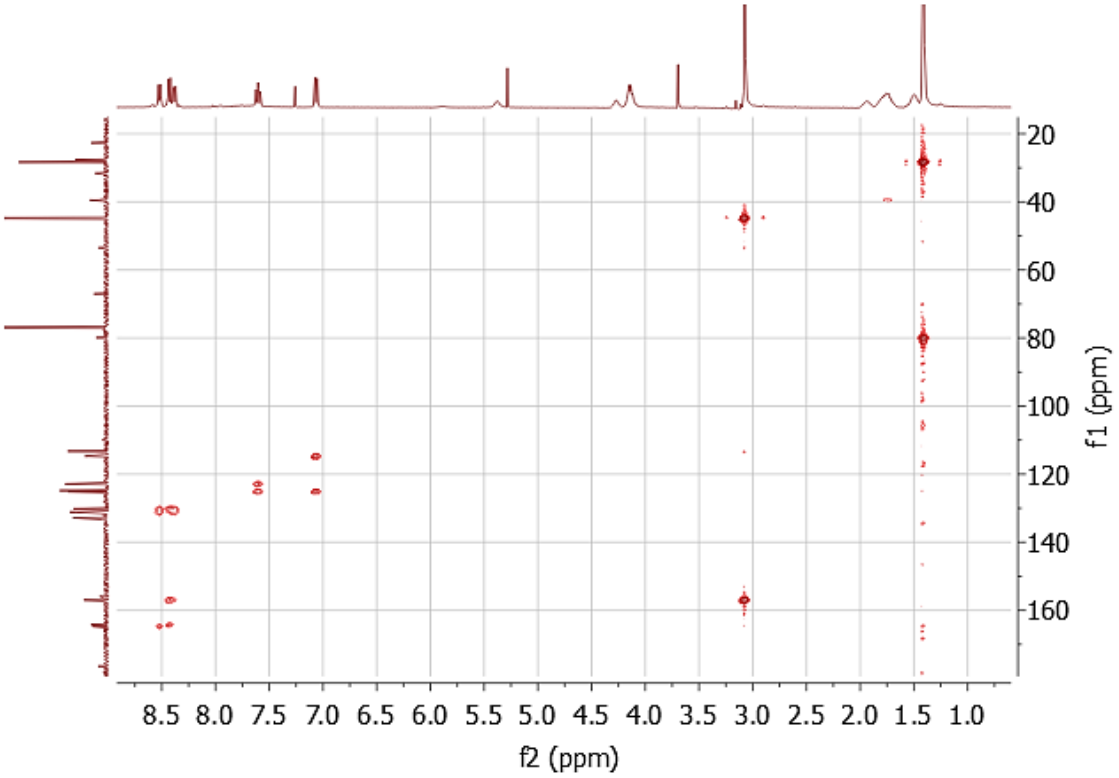

Figure S2.25: HMBC NMR spectrum of Boc-Lys(4DMN)-OH in $CDCl_3$.

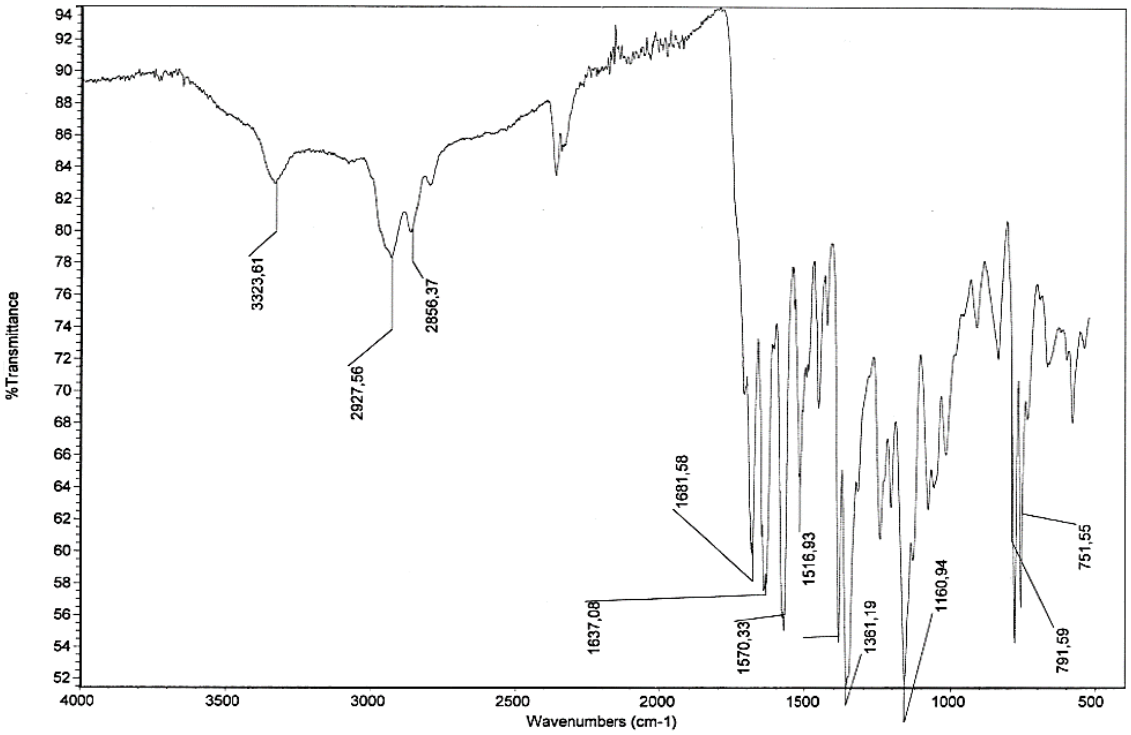

Figure S2.26: IR spectrum of Boc-Lys(4DMN)-OH.

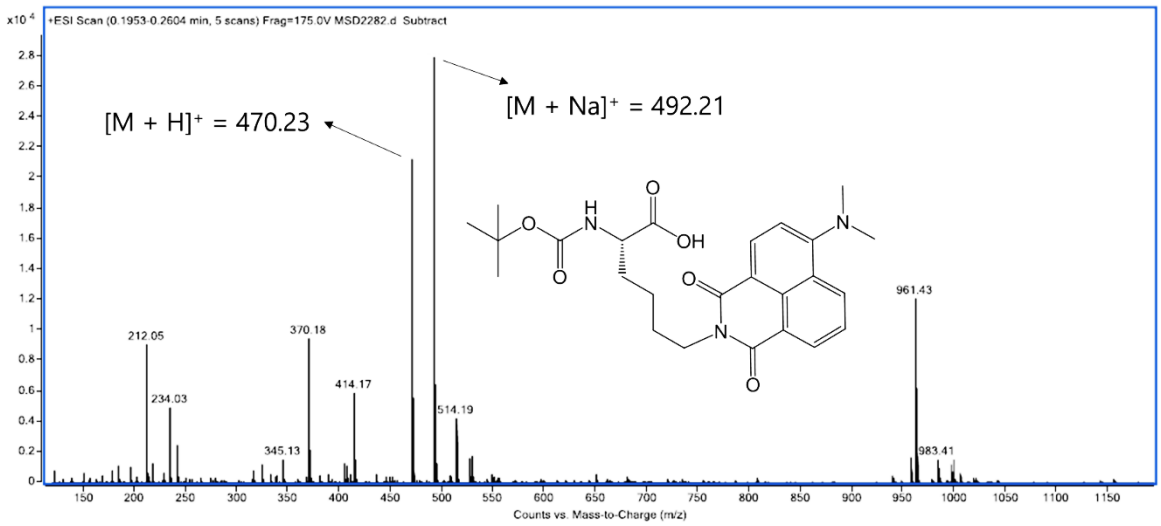

Figure S2.27: ESI mass spectrum of Boc-Lys(4DMN)-OH.

Fmoc-Lys(4DMN)-OH

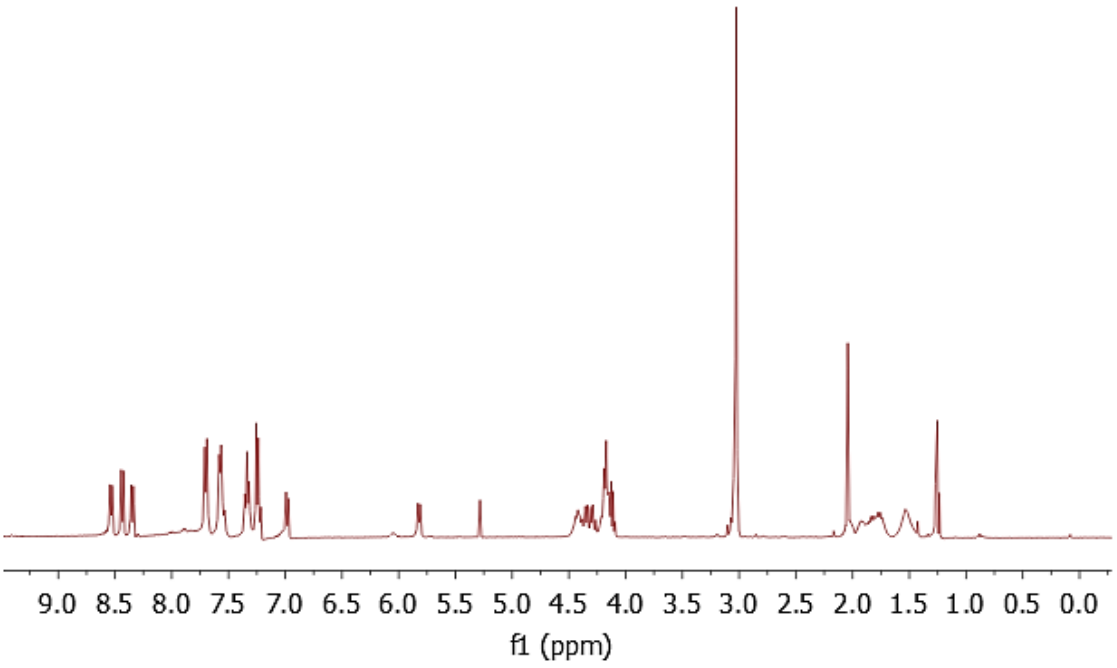

Figure S2.28: ^{1}H NMR spectrum of Fmoc-Lys(4DMN)-OH in $CDCl_3$.

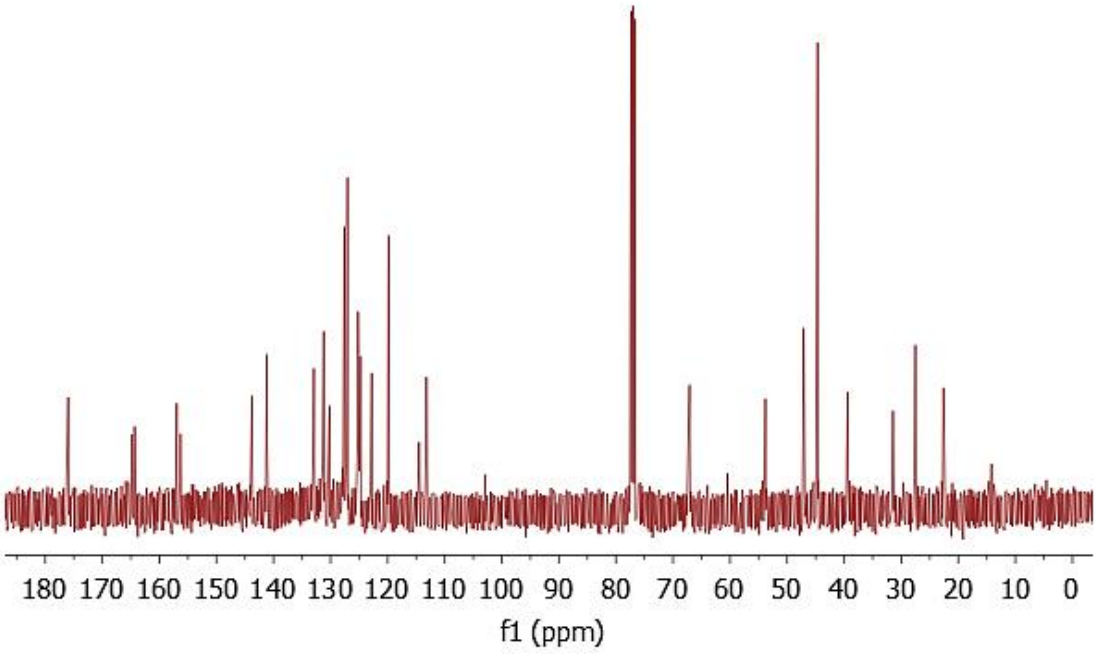

Figure S2.29: ^{13}C NMR spectrum of Fmoc-Lys(4DMN)-OH in $CDCl_3$.

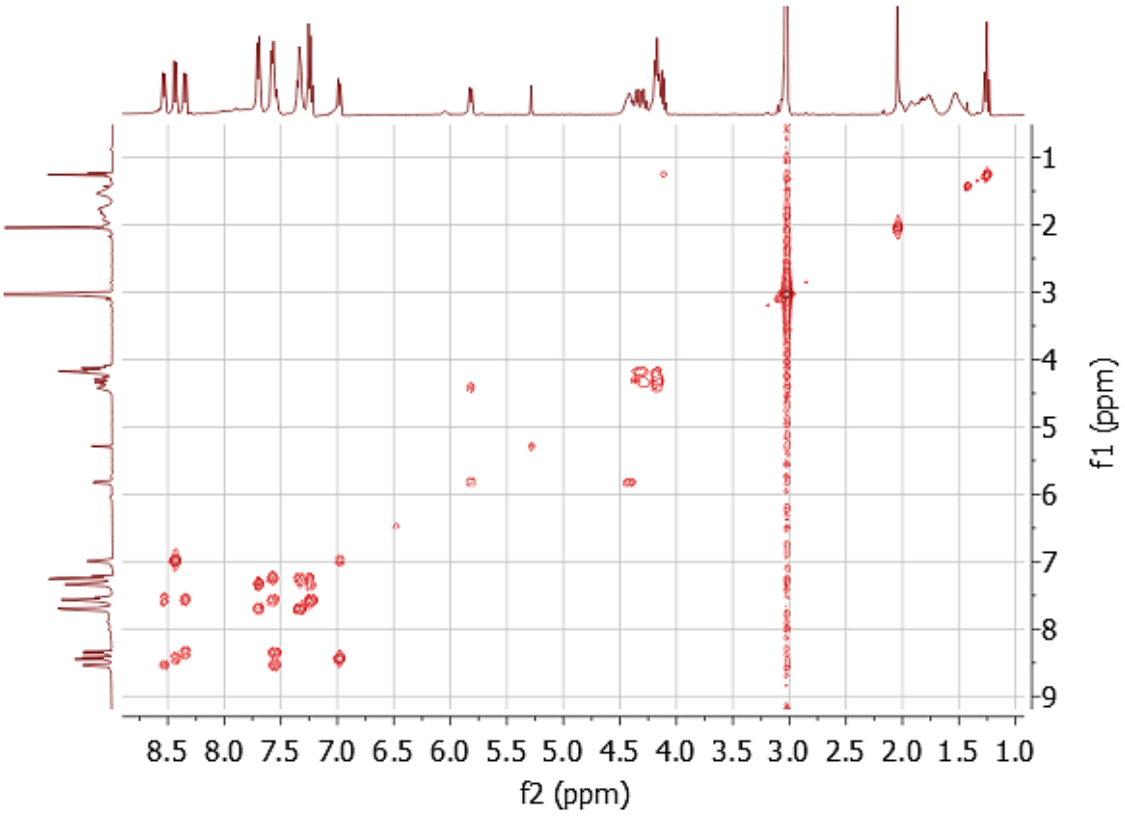

Figure S2.30: COSY NMR spectrum of Fmoc-Lys(4DMN)-OH in $CDCl_3$.

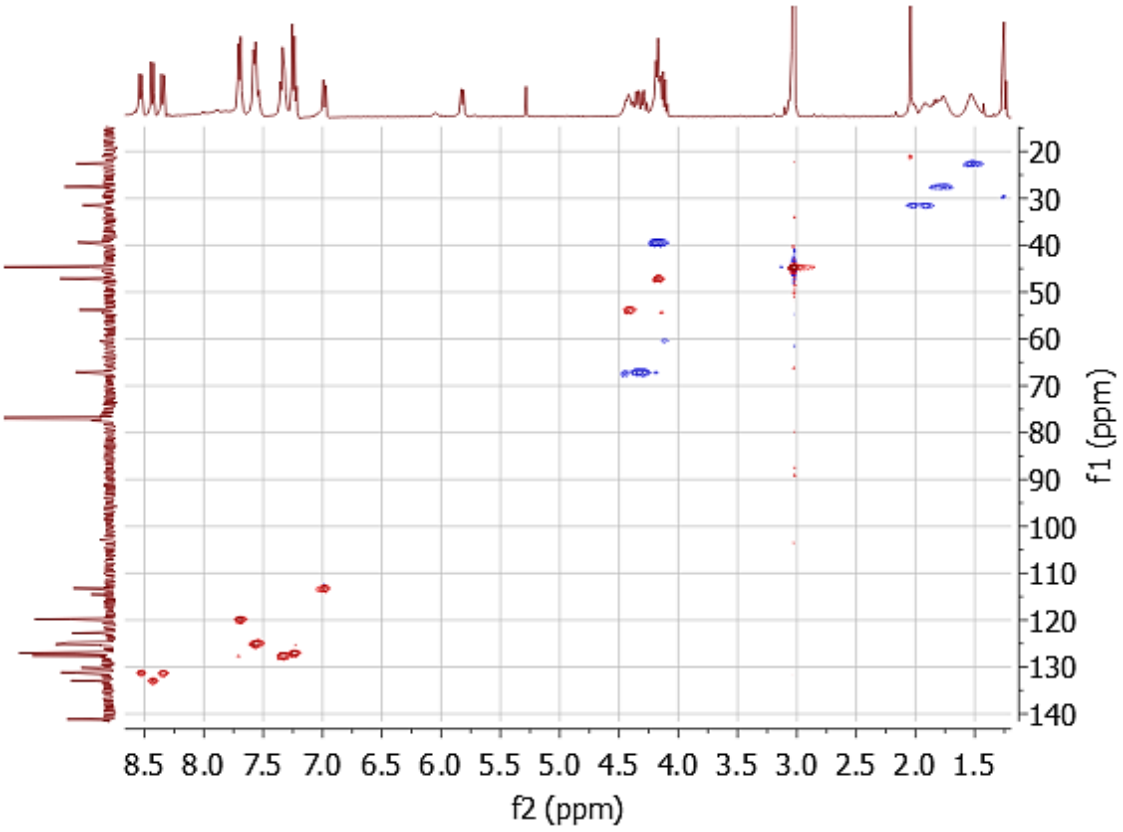

Figure S2.31: HSQC NMR spectrum of Fmoc-Lys(4DMN)-OH in $CDCl_3$.

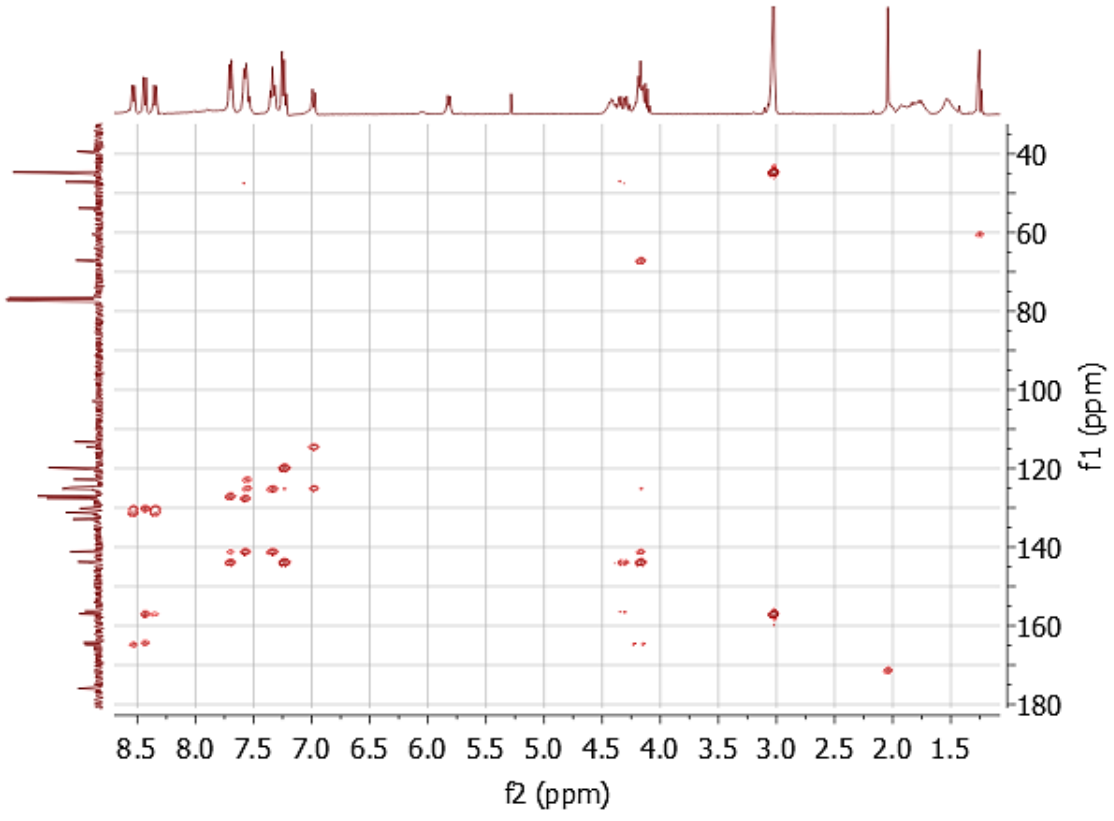

Figure S2.32: HMBC NMR spectrum of Fmoc-Lys(4DMN)-OH in $CDCl_3$.

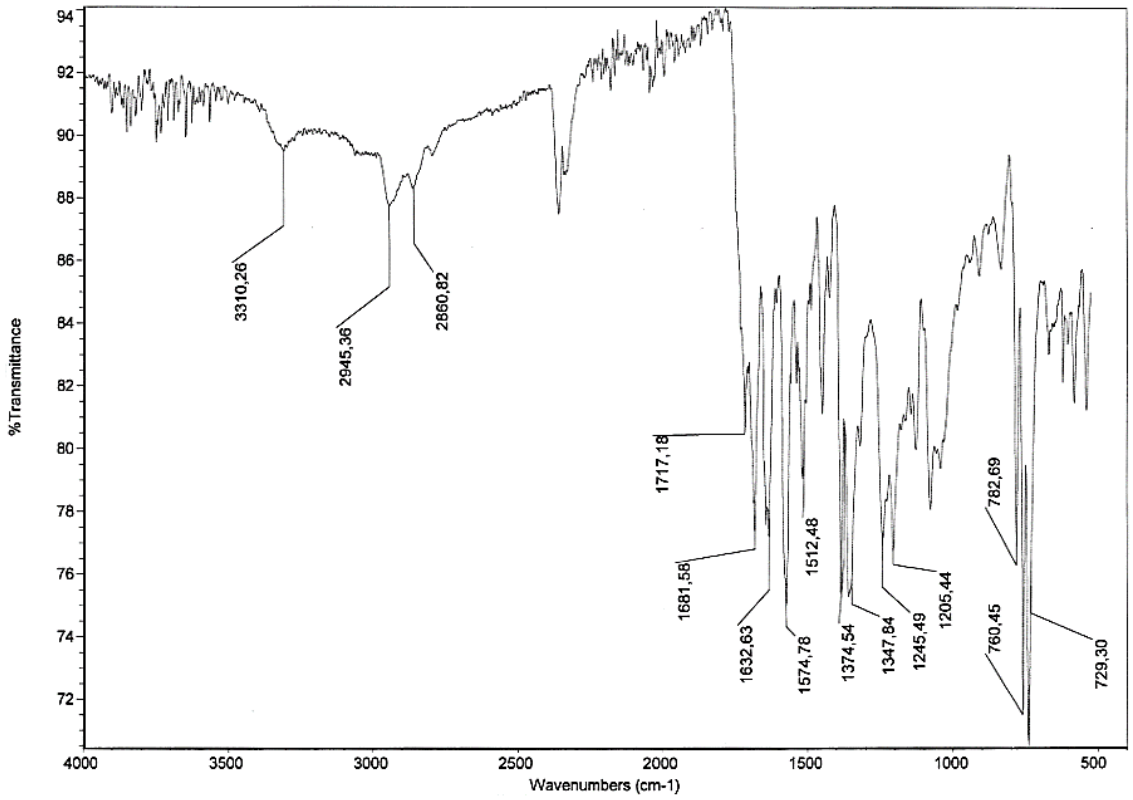

Figure S2.33: IR spectrum of Fmoc-Lys(4DMN)-OH.

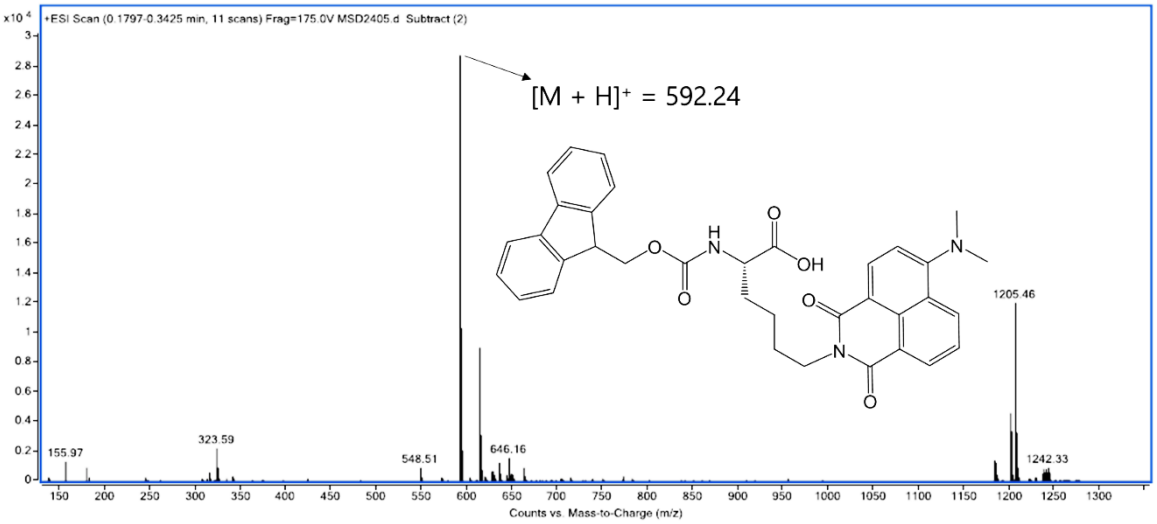

Figure S2.34: ESI mass spectrum of Fmoc-Lys(4DMN)-OH.

H-His-Lys(Coum)-His-OH (HK^{c}H)

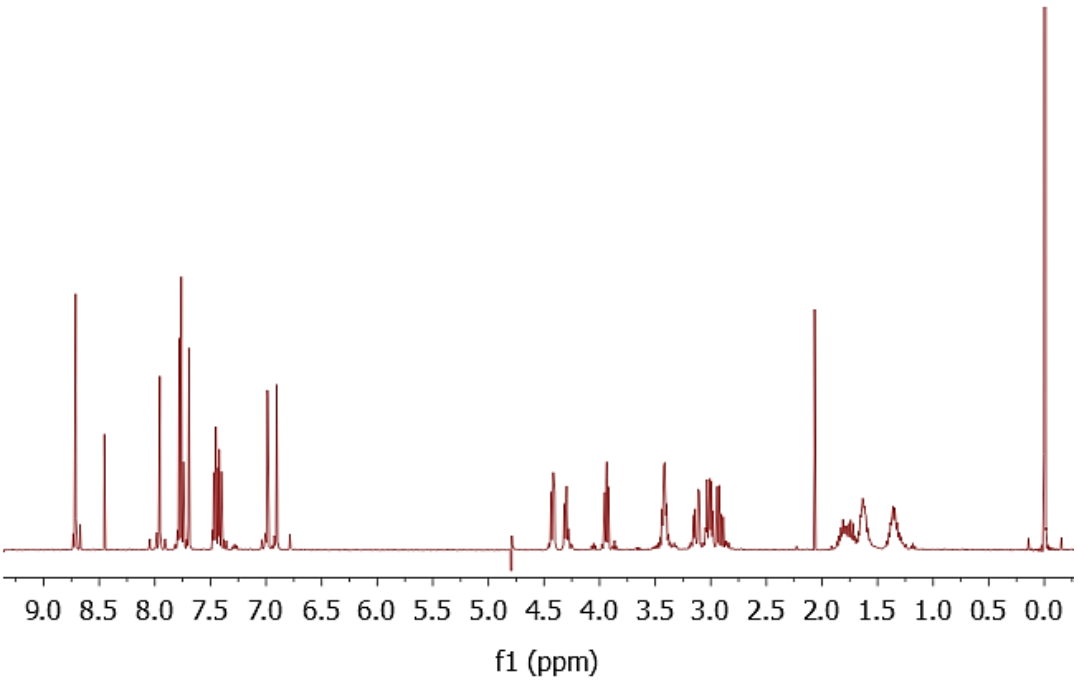

Figure S2.35: ^{1}H NMR spectrum of H-His-Lys(Coum)-His-OH (HK^{c}H) in D_2O (pH 7.4).

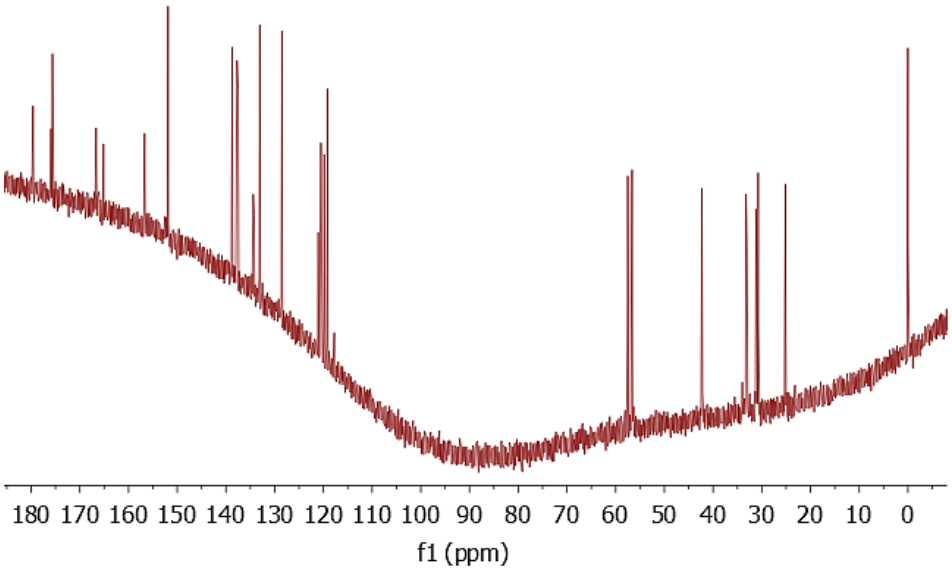

Figure S2.36: ^{13}C NMR spectrum of H-His-Lys(Coum)-His-OH (HK^{c}H) in D_2O (pH 7.4).

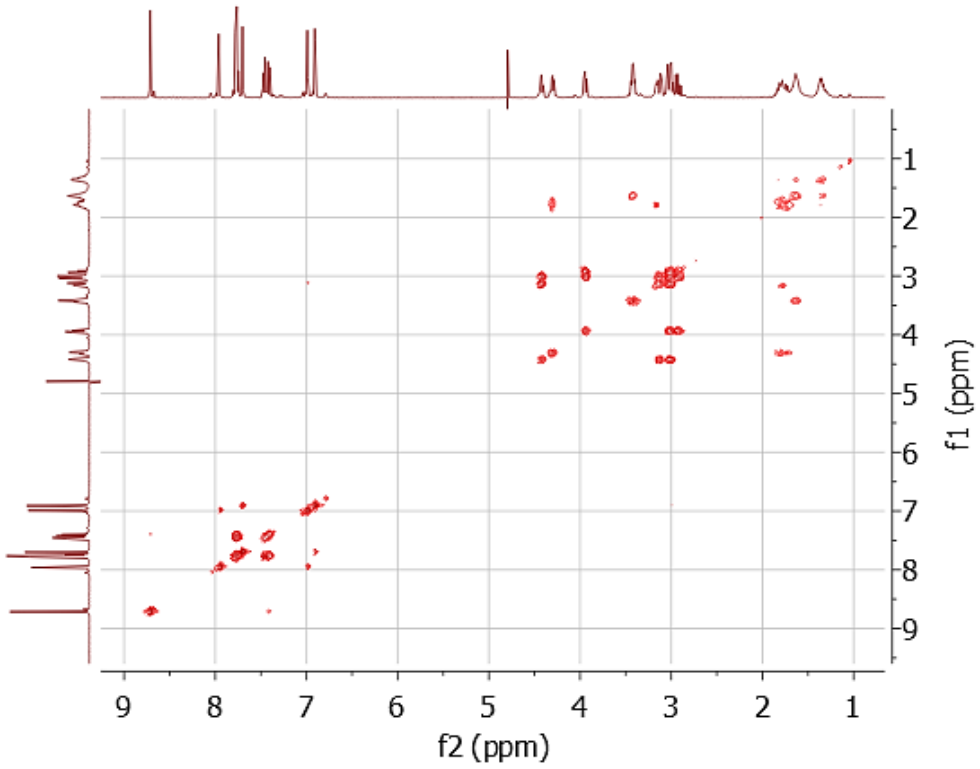

Figure S2.37: COSY NMR spectrum of H-His-Lys(Coum)-His-OH (HK^{c}H) in D_2O (pH 7.4).

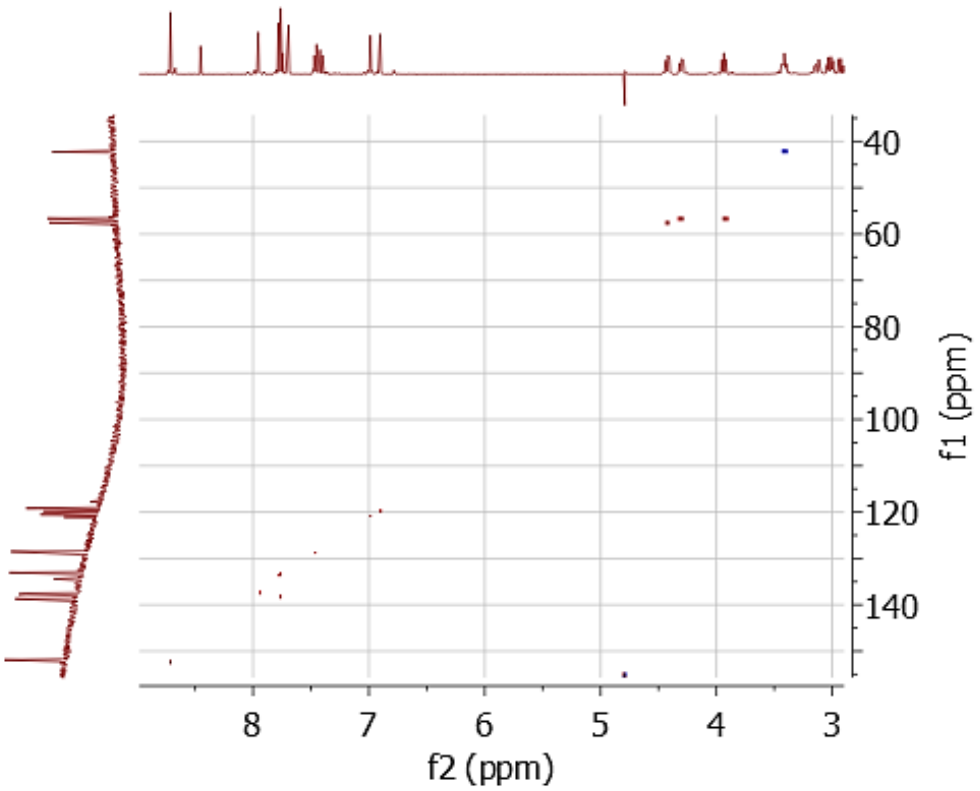

Figure S2.38: HSQC NMR spectrum of H-His-Lys(Coum)-His-OH (HK^{c}H) in D_2O (pH 7.4).

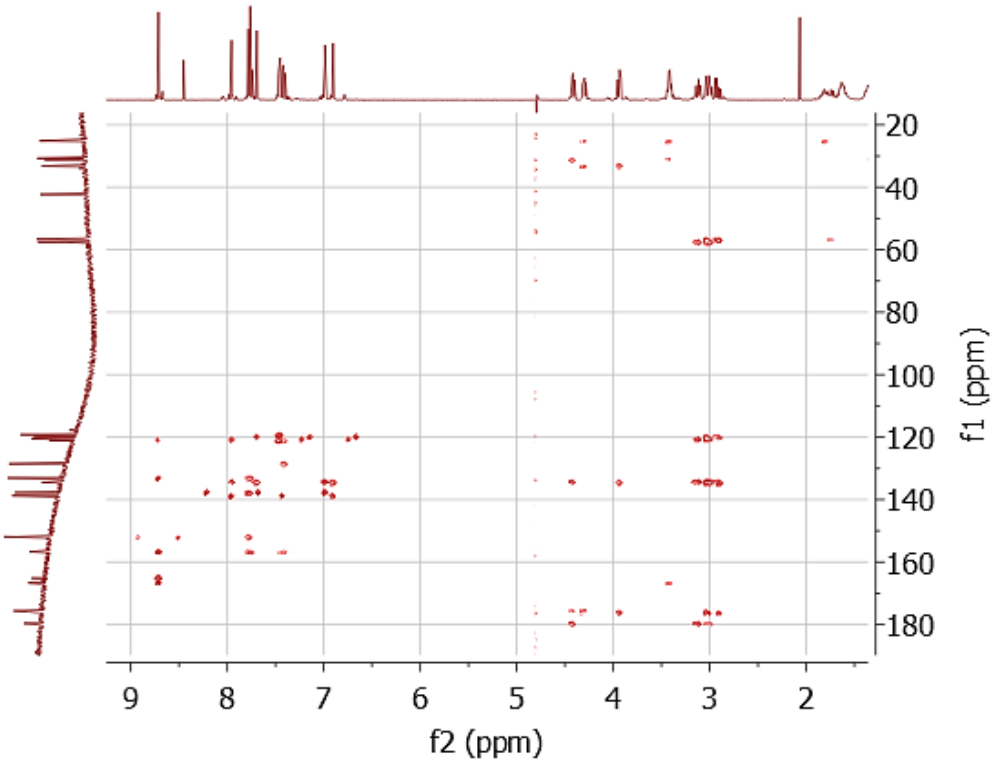

Figure S2.39: HMBC NMR spectrum of H-His-Lys(Coum)-His-OH (HK^{c}H) in D_2O (pH 7.4).

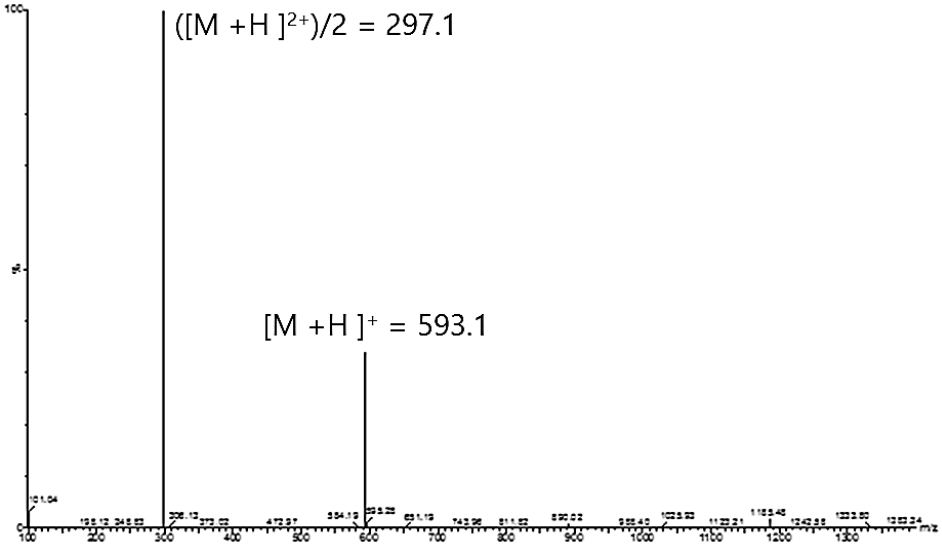

Figure S2.40: ESI mass spectrum of H-His-Lys(Coum)-His-OH (HK^{c}H).

Fluo-His-Nle-His-NH_2 (FluoHJH)

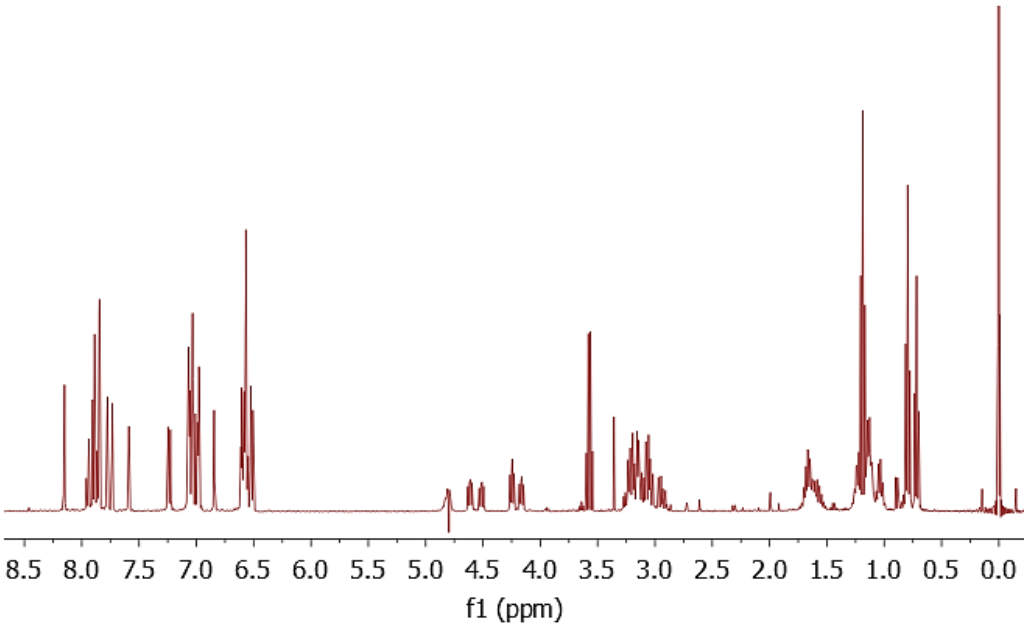

Figure S2.41: ^{1}H NMR spectrum of Fluo-His-Nle-His-NH_2 (FluoHJH) in D_2O (pH 7.4).

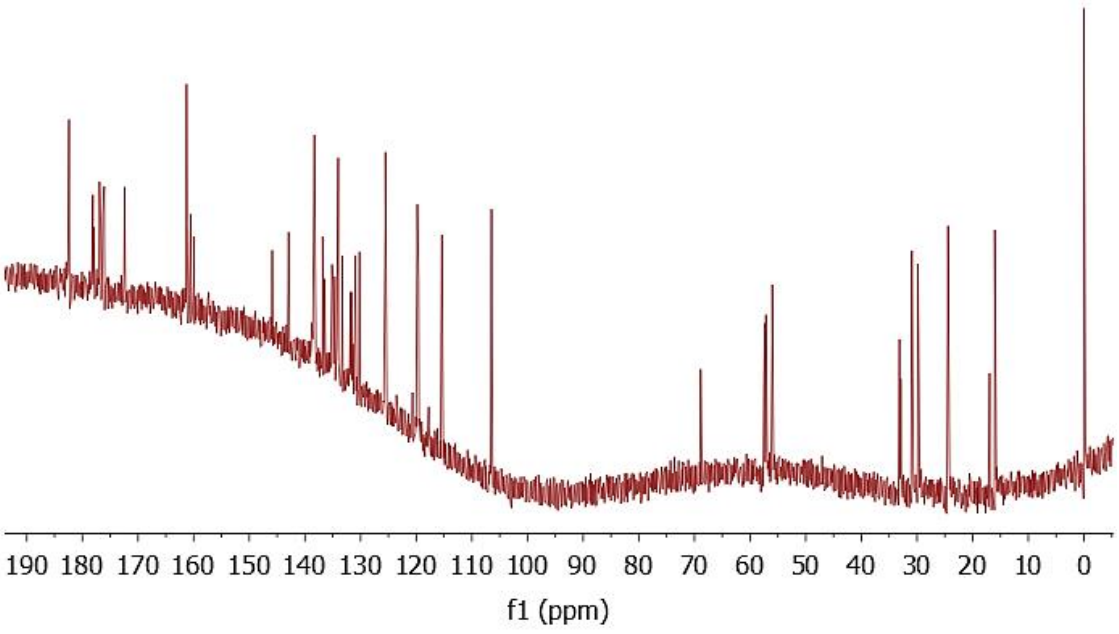

Figure S2.42: ^{13}C NMR spectrum of Fluo-His-Nle-His-NH_2 (FluoHJH) in D_2O (pH 7.4).

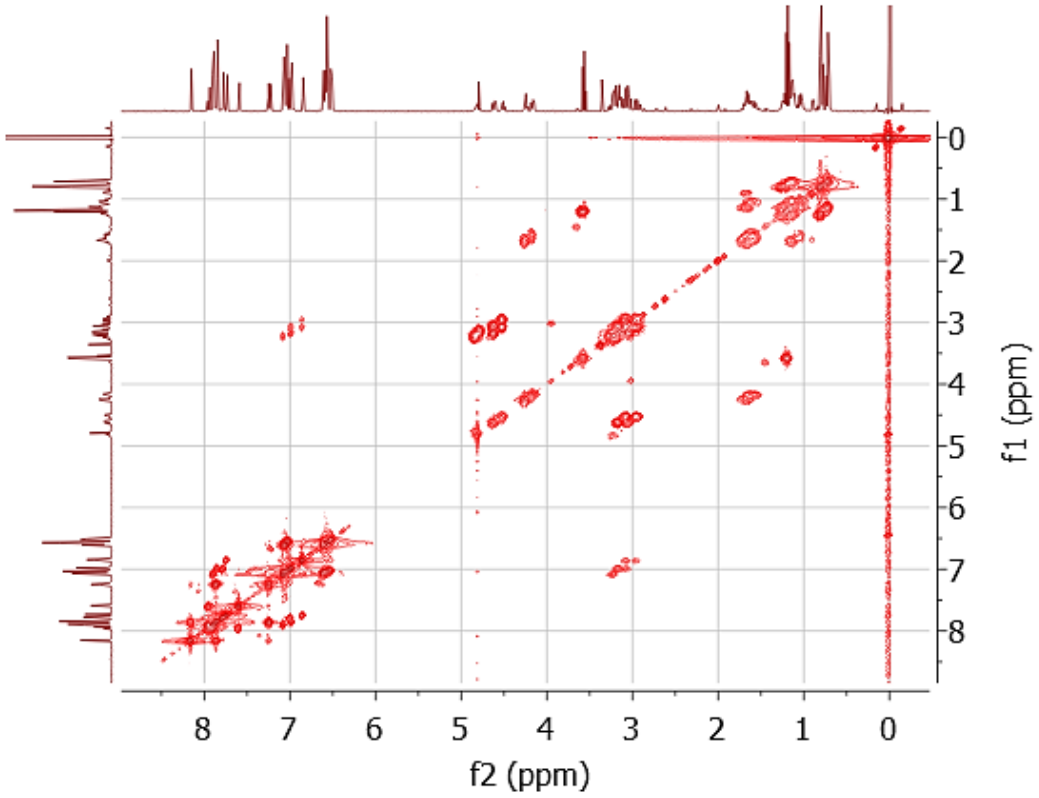

Figure S2.43: COSY NMR spectrum of Fluo-His-Nle-His-NH_2 (FluoHJH) in D_2O (pH 7.4).

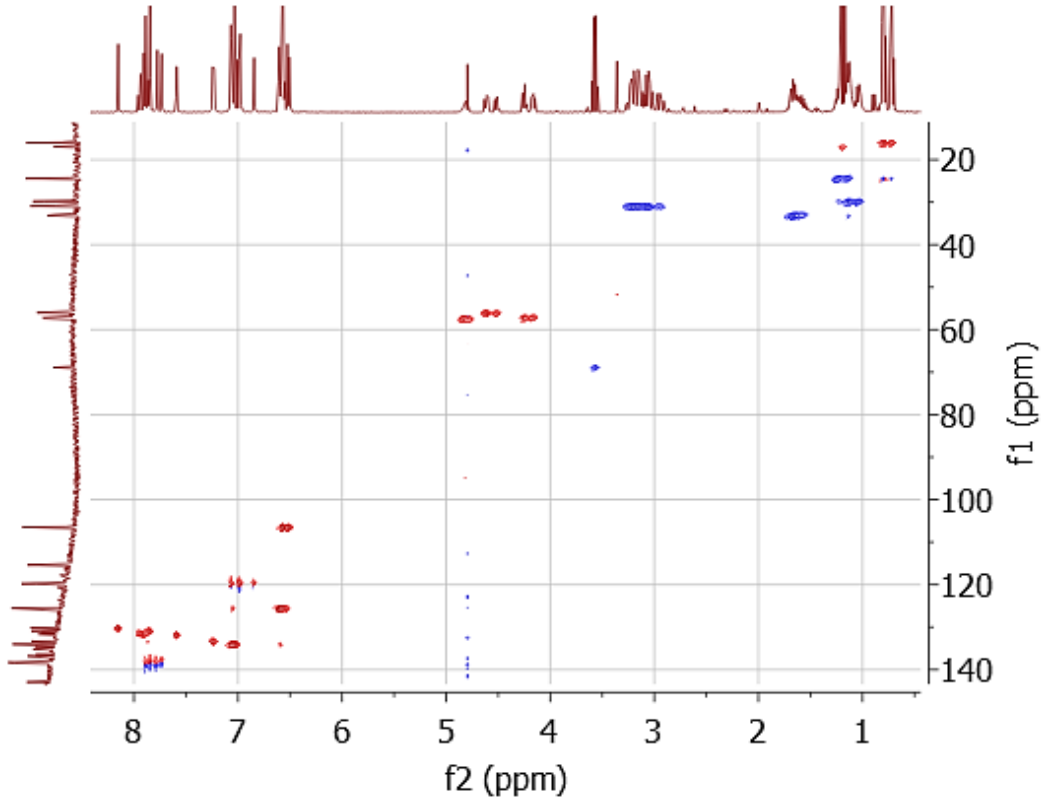

Figure S2.44: HSQC NMR spectrum of Fluo-His-Nle-His-NH_2 (FluoHJH) in D_2O (pH 7.4).

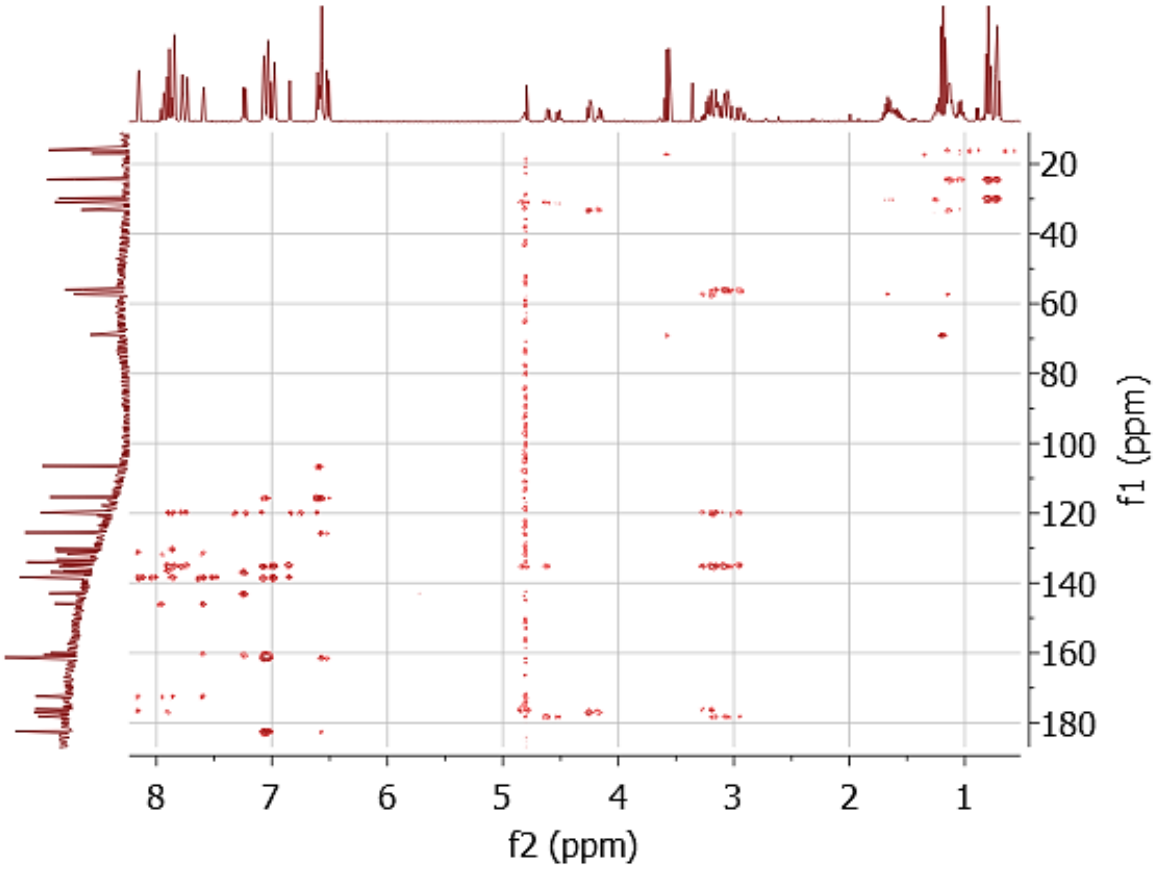

Figure S2.45: HMBC NMR spectrum of Fluo-His-Nle-His-NH_2 (FluoHJH) in D_2O (pH 7.4).

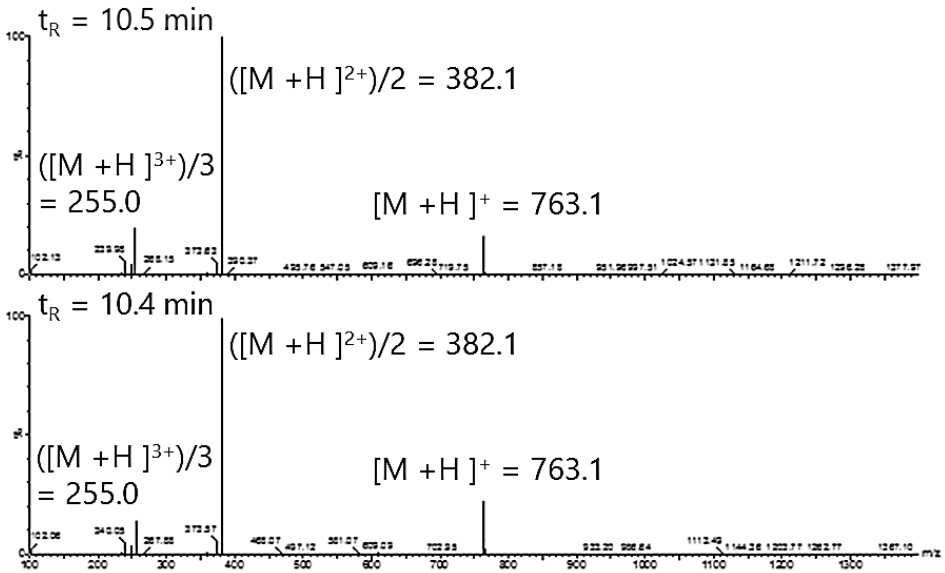

Figure S2.46: ESI mass spectrum of Fluo-His-Nle-His-NH_2 (FluoHJH).

Fluo-His-Lys(Coum)-His-NH_2 (FluoHK^{c}H)

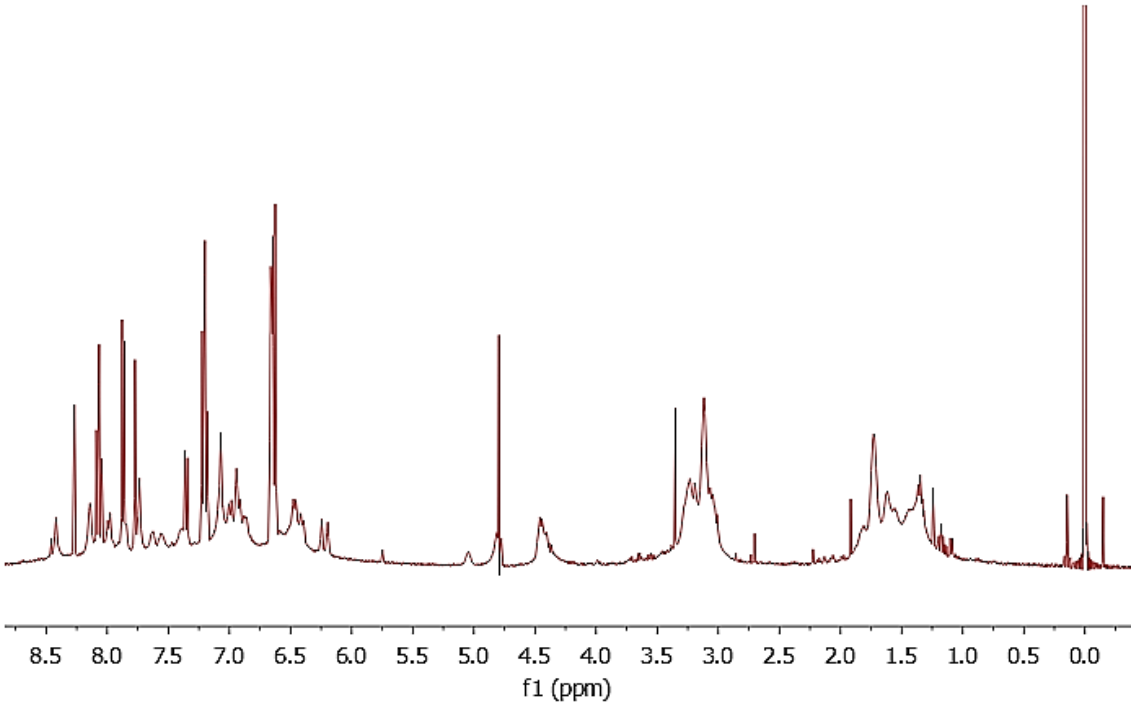

Figure S2.47: ^{1}H NMR spectrum of Fluo-His-Lys(Coum)-His-NH_2 (FluoHK^{c}H) in D_2O (pH 7.4).

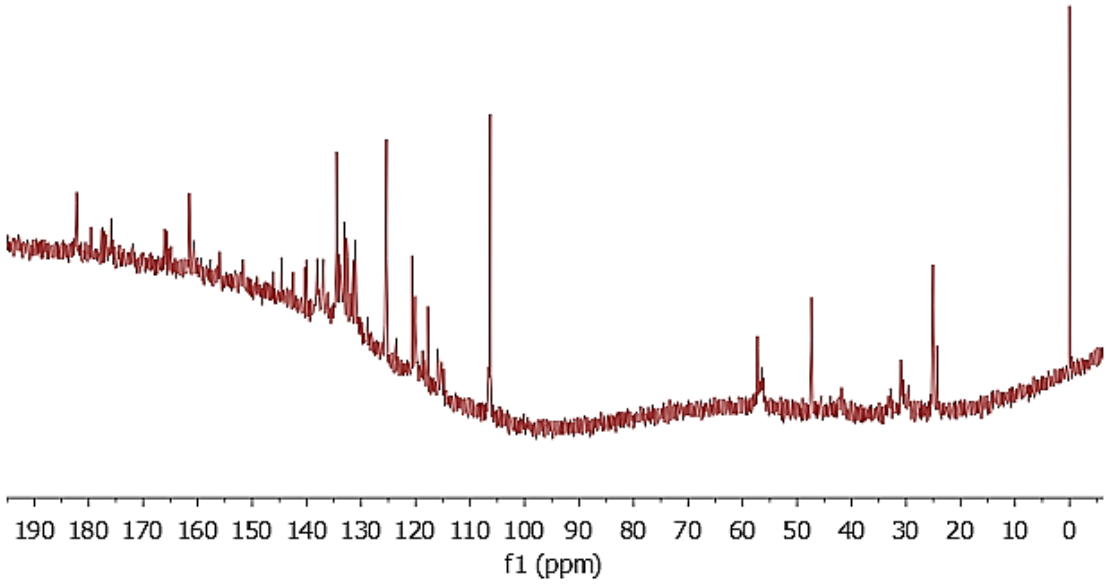

Figure S2.48: ^{13}C NMR spectrum of Fluo-His-Lys(Coum)-His-NH_2 (FluoHK^{c}H) in D_2O (pH 7.4).

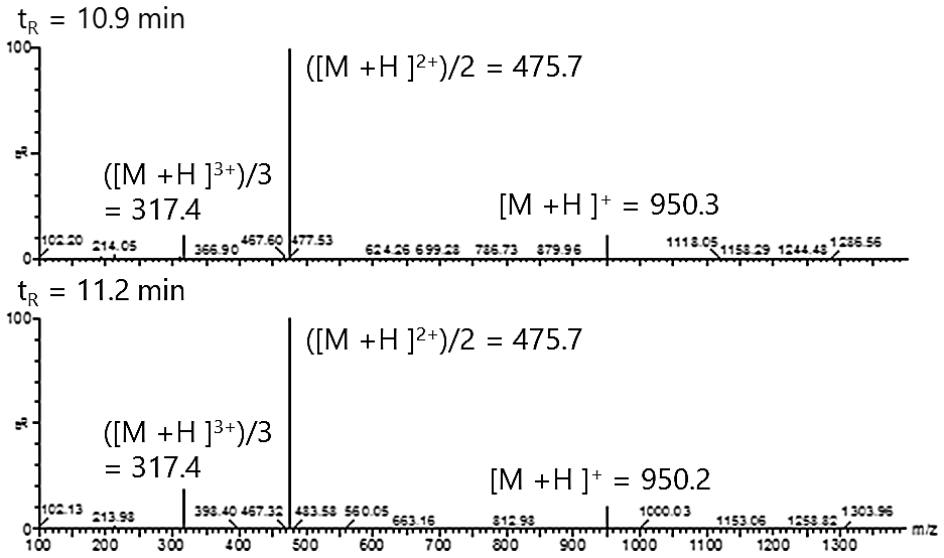

Figure S2.49: ESI mass spectrum of Fluo-His-Lys(Coum)-His-NH_2 (FluoHK^{c}H).

Ac-His-Lys(Coum)-His-OH (AcHK^{c}H)

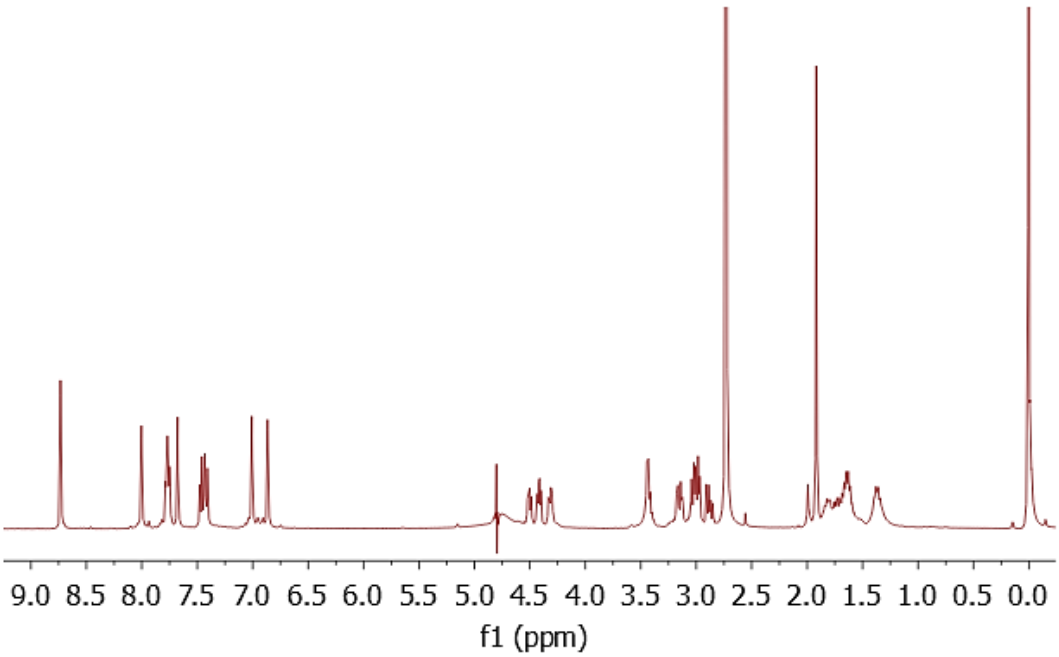

Figure S2.50: ^{1}H NMR spectrum of Ac-His-Lys(Coum)-His-OH (AcHK^{c}H) in D_2O (pH 7.4).

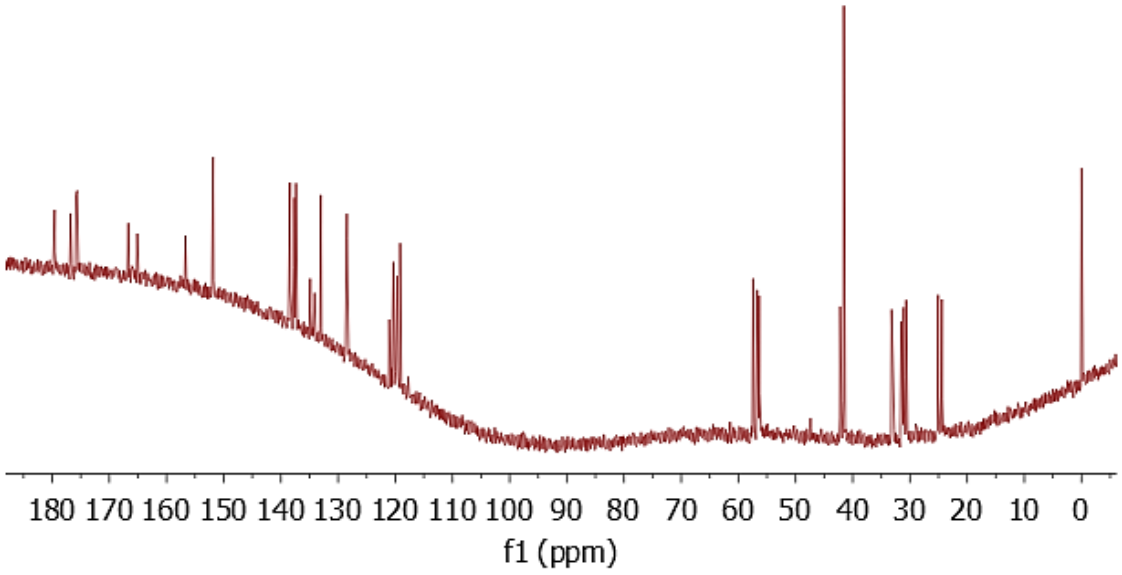

Figure S2.51: ^{13}C NMR spectrum of Ac-His-Lys(Coum)-His-OH (AcHK^{c}H) in D_2O (pH 7.4).

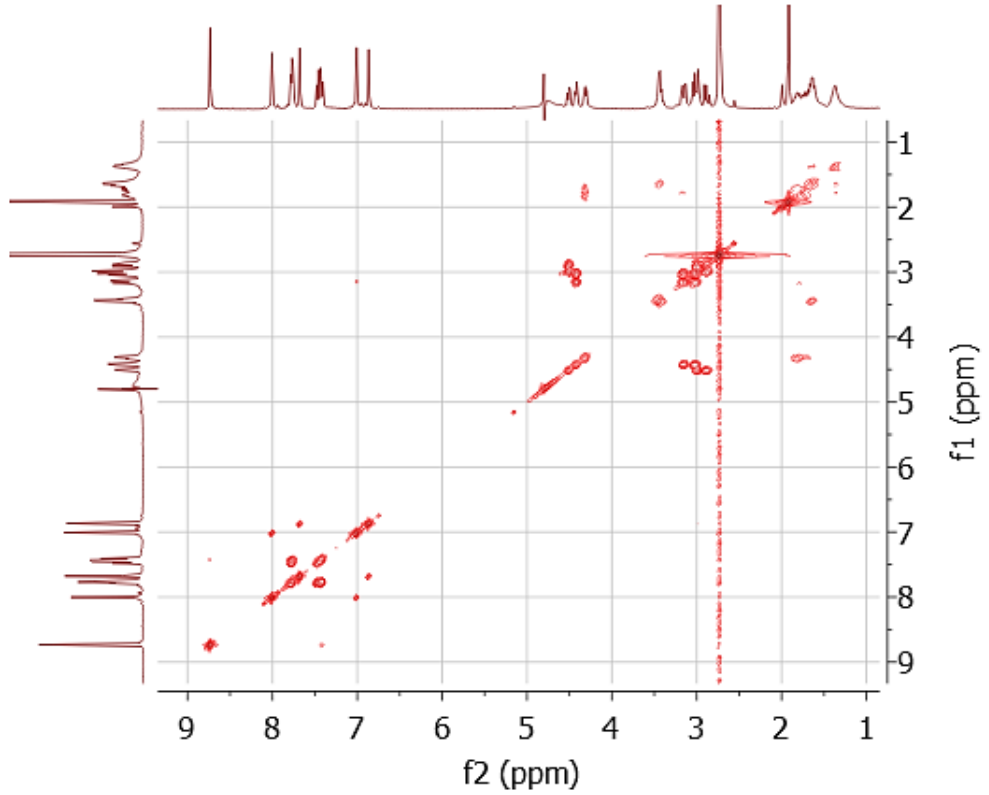

Figure S2.52: COSY NMR spectrum of Ac-His-Lys(Coum)-His-OH (AcHK^{c}H) in D_2O (pH 7.4).

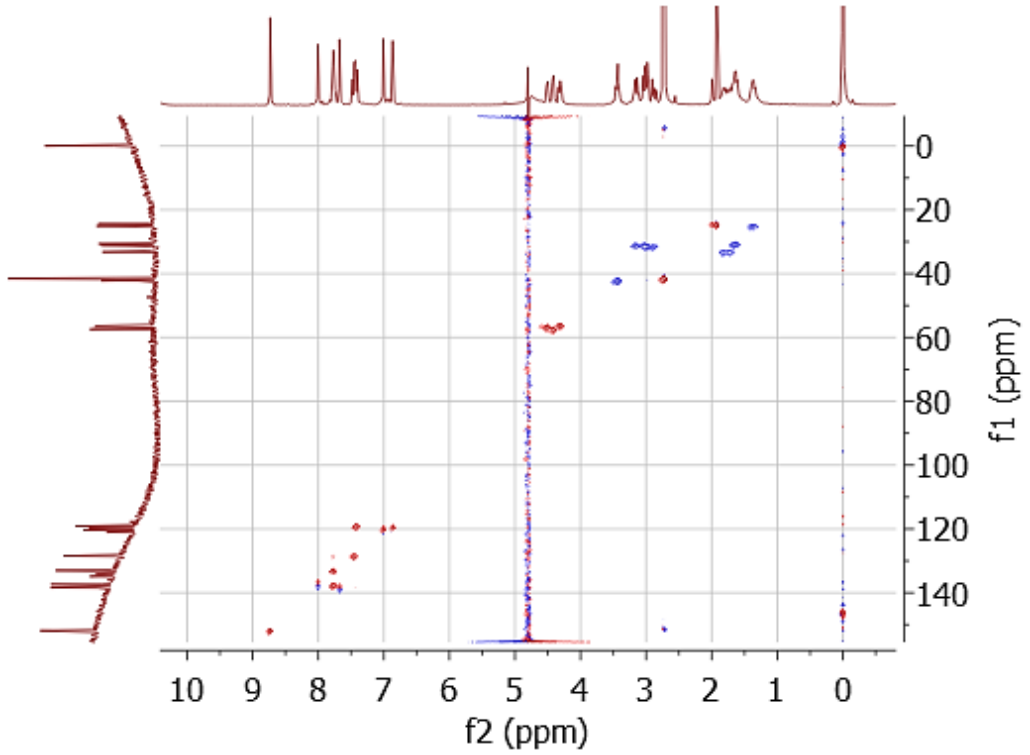

Figure S2.53: HSQC NMR spectrum of Ac-His-Lys(Coum)-His-OH (AcHK^{c}H) in D_2O (pH 7.4).

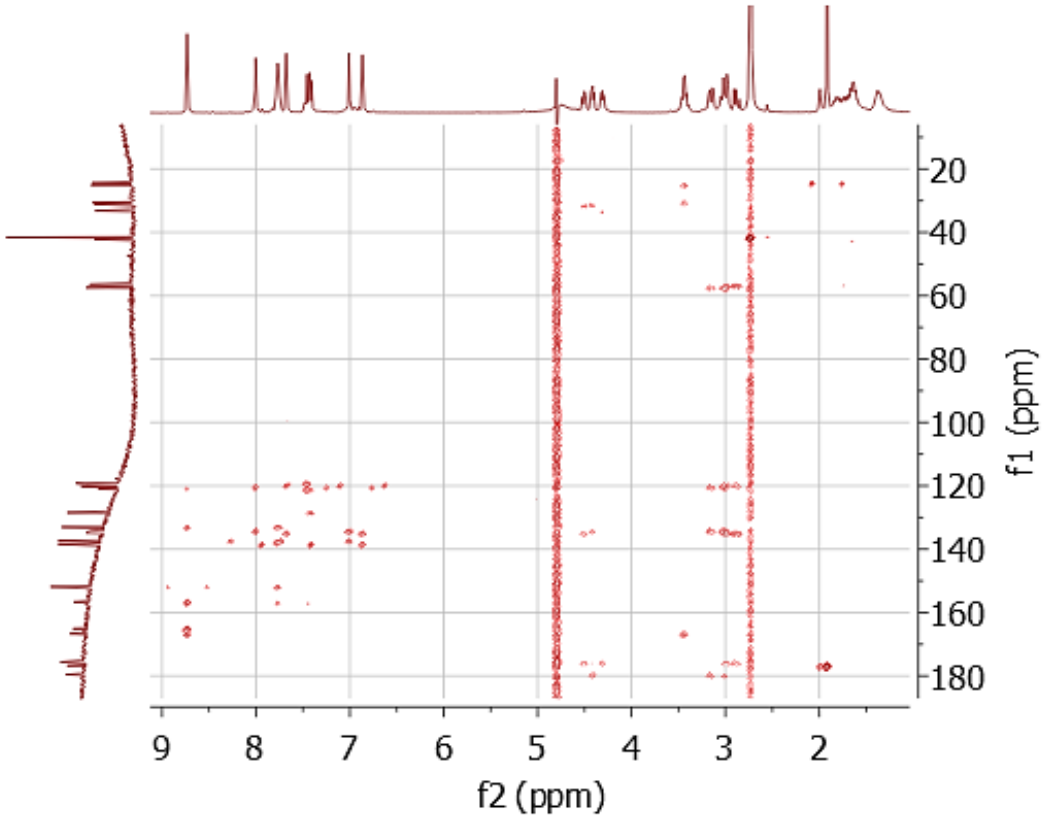

Figure S2.54: HMBC NMR spectrum of Ac-His-Lys(Coum)-His-OH (AcHK^{c}H) in D_2O (pH 7.4).

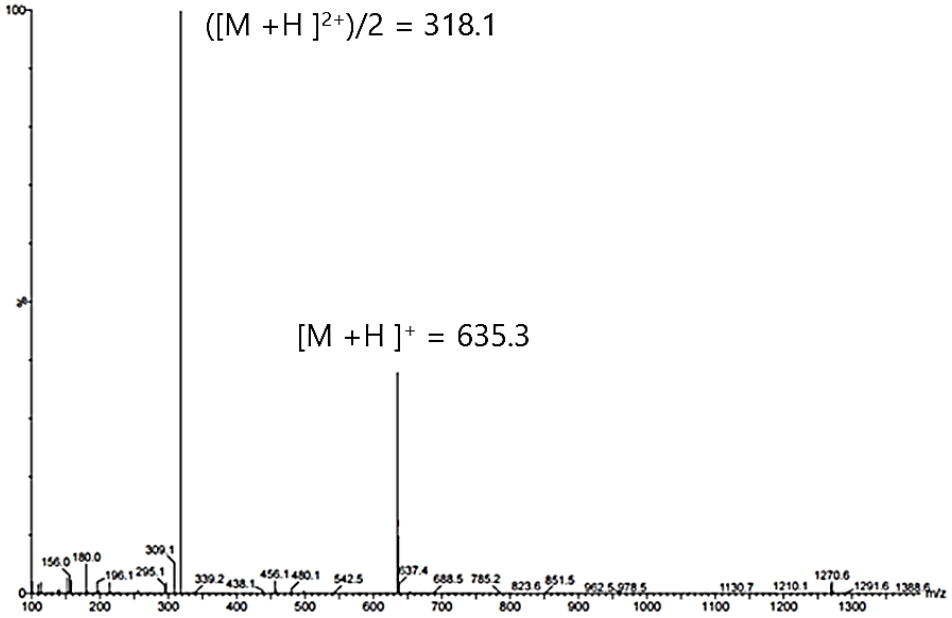

Figure S2.55: ESI mass spectrum of Ac-His-Lys(Coum)-His-OH (AcHK^{c}H).

Ac-His-Lys(4DMN)-His-OH (AcHK^{d}H)

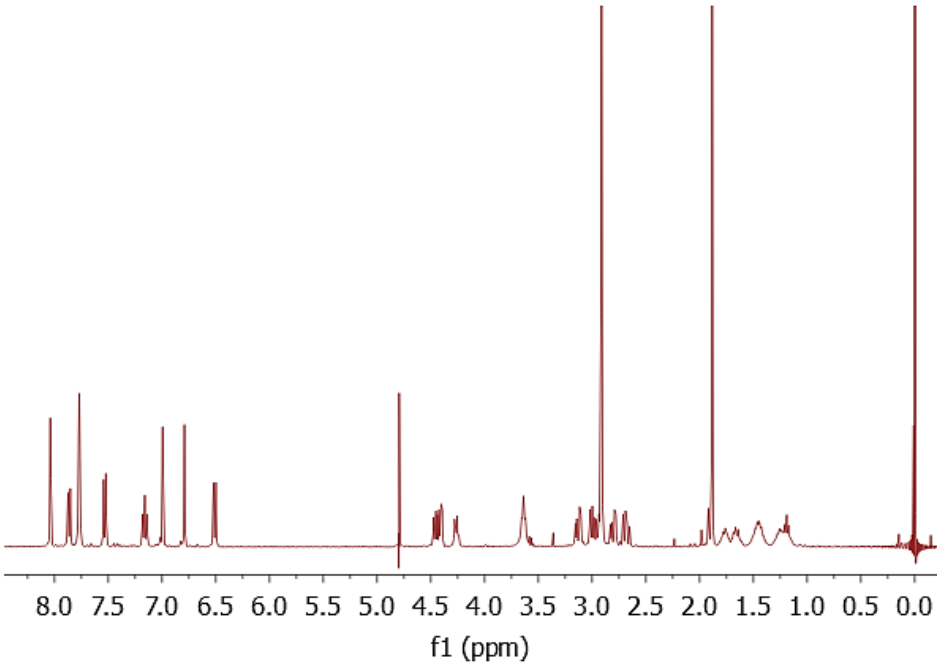

Figure S2.56: ^{1}H NMR spectrum of Ac-His-Lys(4DMN)-His-OH (AcHK^{d}H) in D_2O (pH 7.4).

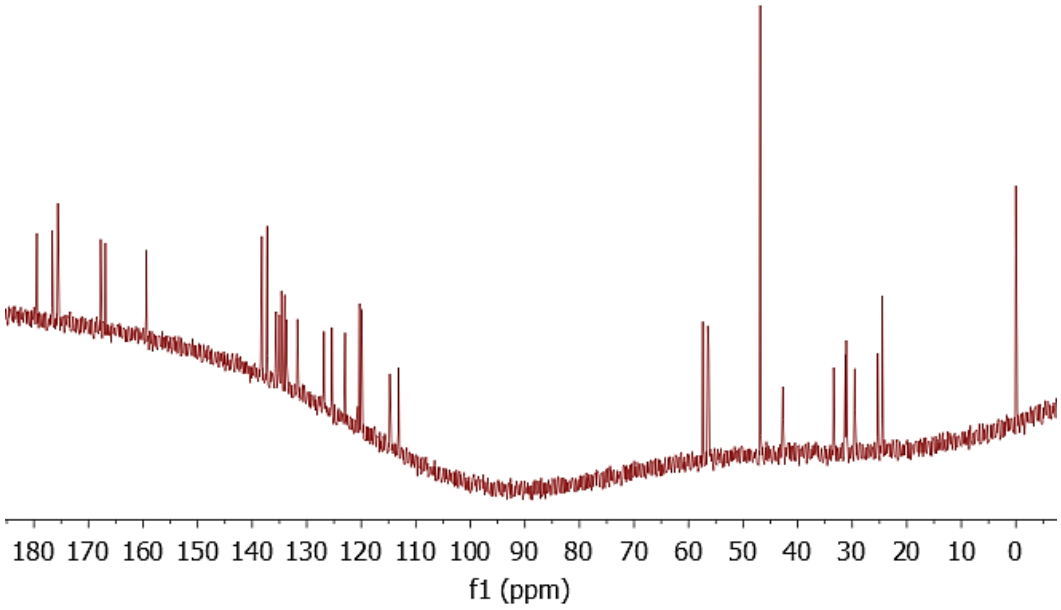

Figure S2.57: ^{13}C NMR spectrum of Ac-His-Lys(4DMN)-His-OH (AcHK^{d}H) in D_2O (pH 7.4).

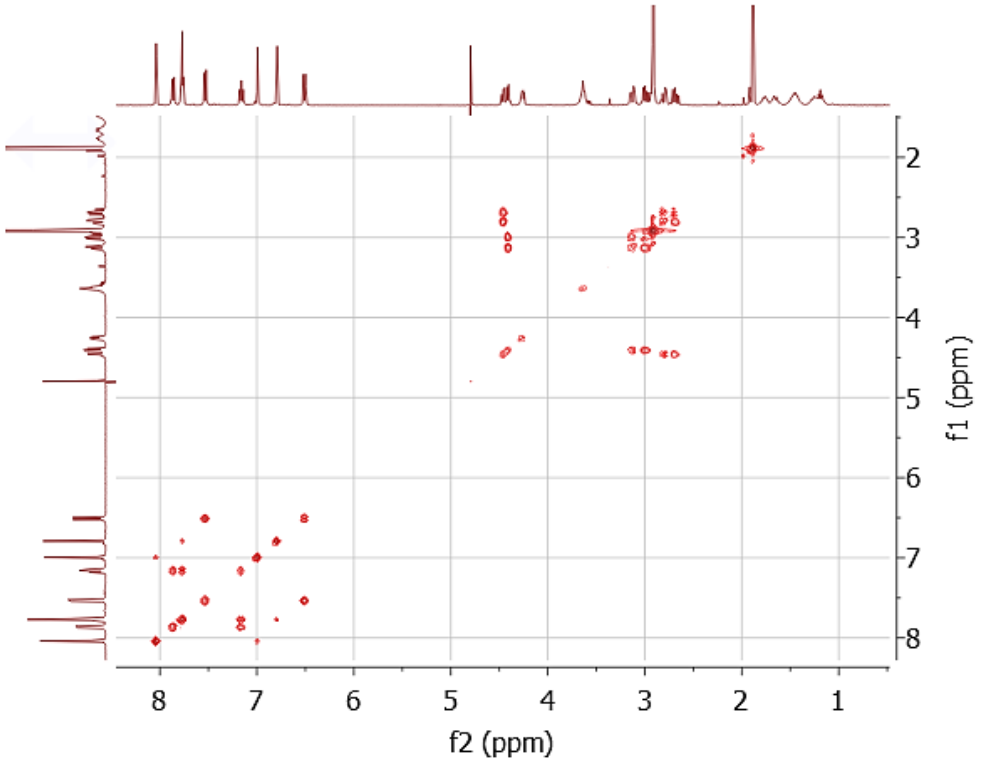

Figure S2.58: COSY NMR spectrum of Ac-His-Lys(4DMN)-His-OH (AcHK^{d}H) in D_2O (pH 7.4).

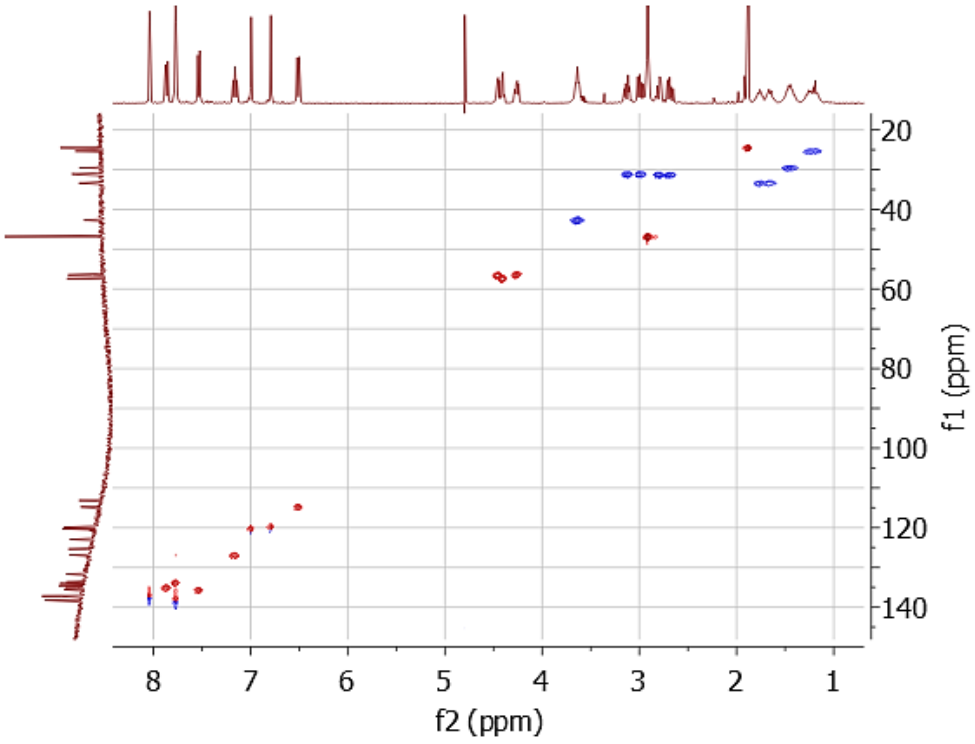

Figure S2.59: HSQC NMR spectrum of Ac-His-Lys(4DMN)-His-OH (AcHK^{d}H) in D_2O (pH 7.4).

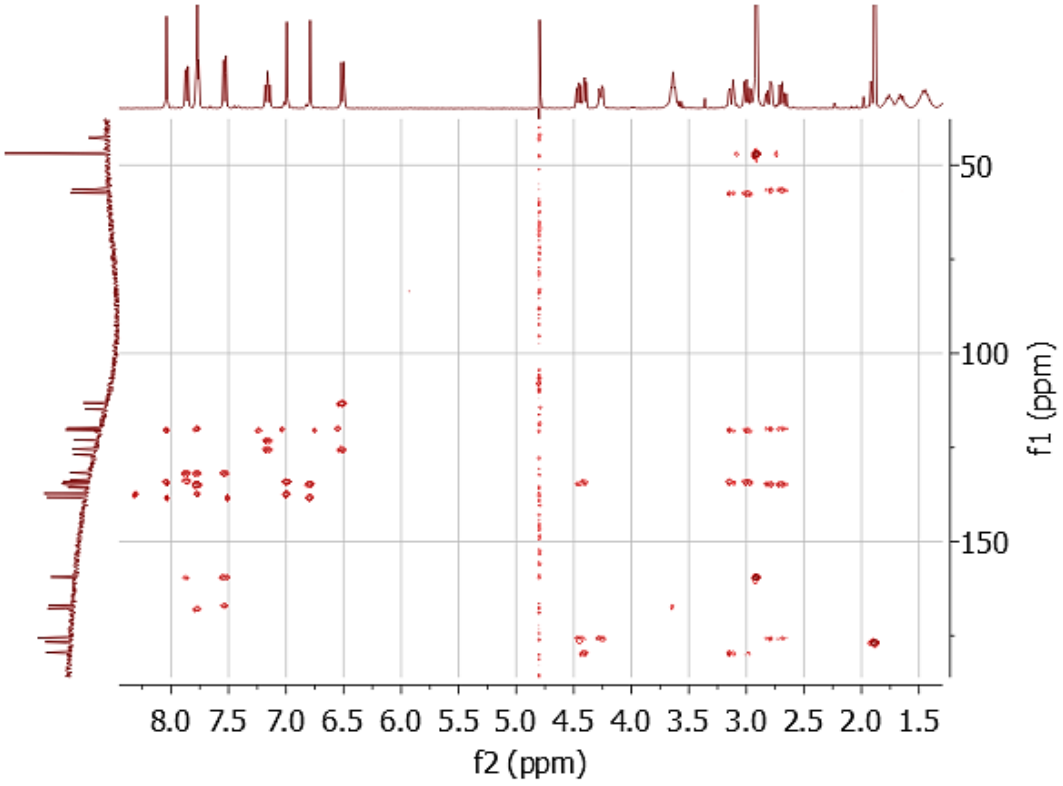

Figure S2.60: HMBC NMR spectrum of Ac-His-Lys(4DMN)-His-OH (AcHK^{d}H) in D_2O (pH 7.4).

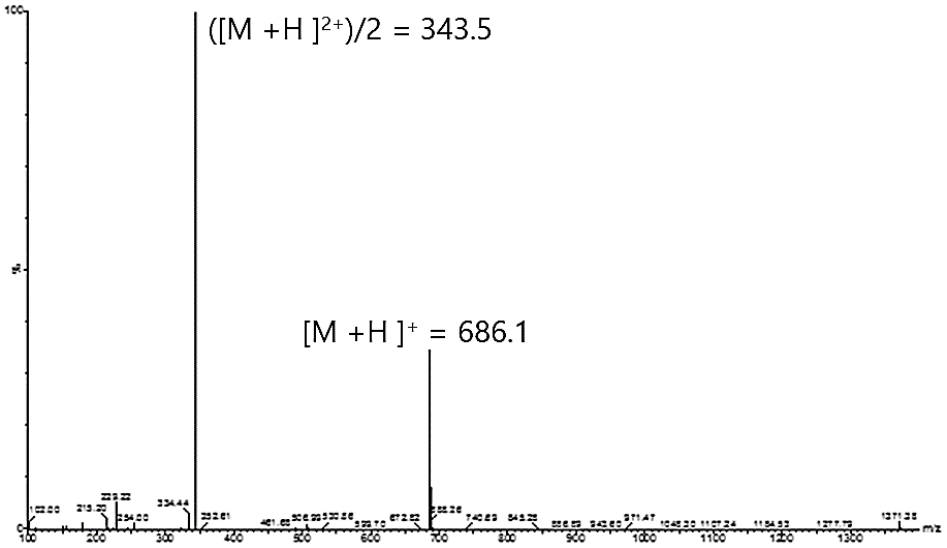

Figure S2.61: ESI mass spectrum of Ac-His-Lys(4DMN)-His-OH (AcHK^{d}H).

Ac-His-Lys(4DMN)-His-NH$_2$ (AcHK^{d}H-NH$_2$)

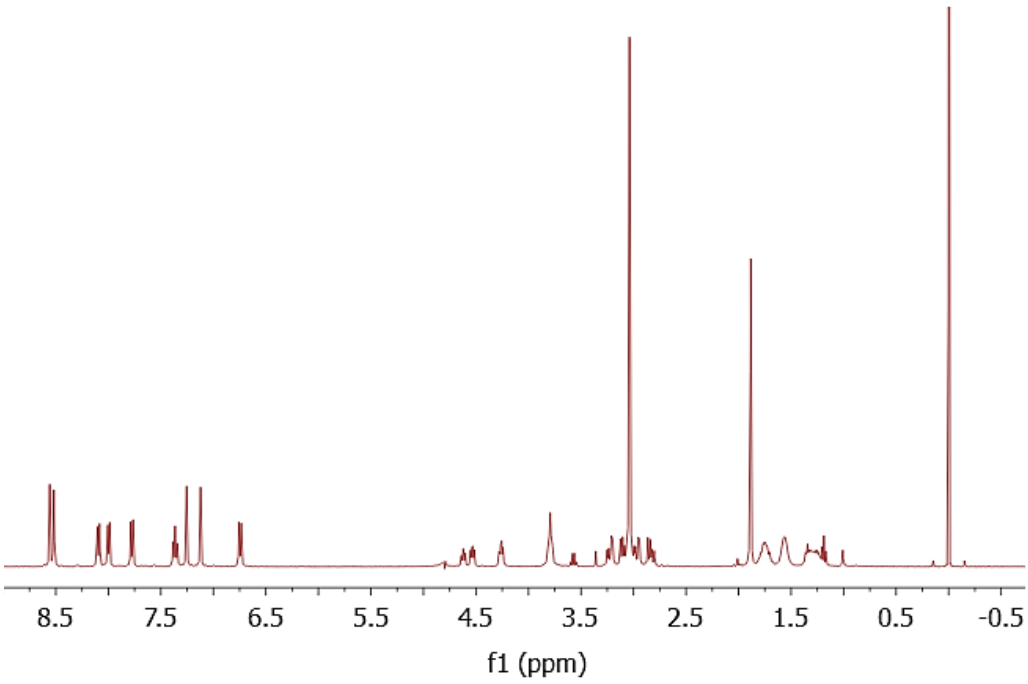

Figure S2.62: ^{1}H NMR spectrum of Ac-His-Lys(4DMN)-His-NH$_2$ (AcHK^{d}H-NH$_2$) in D$_2$O (pH 4.5).

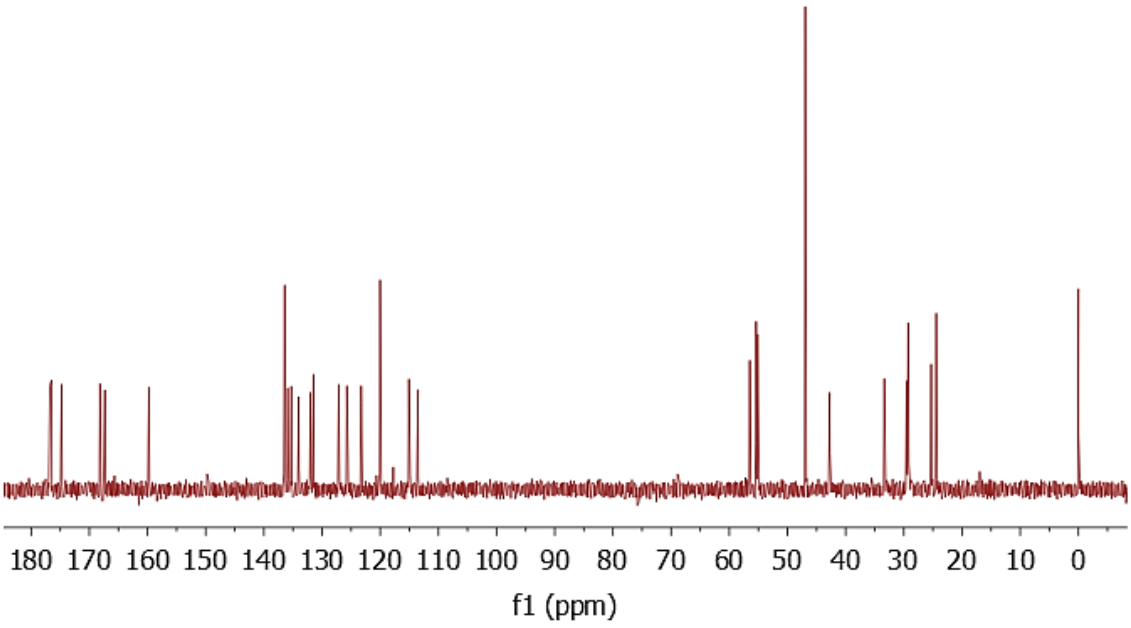

Figure S2.63: ^{13}C NMR spectrum of Ac-His-Lys(4DMN)-His-NH_2 (AcHK^{d}H-NH_2) in D_2O (pH 4.5).

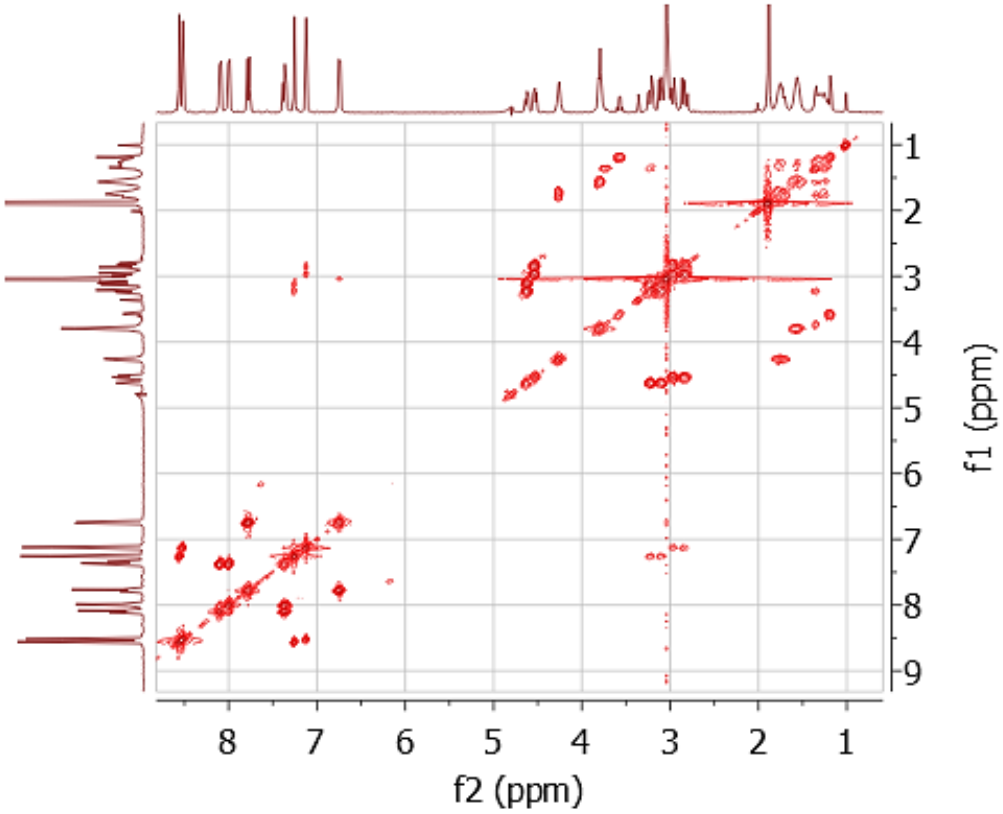

Figure S2.64: COSY NMR spectrum of Ac-His-Lys(4DMN)-His-NH_2 (AcHK^{d}H-NH_2) in D_2O (pH 4.5).

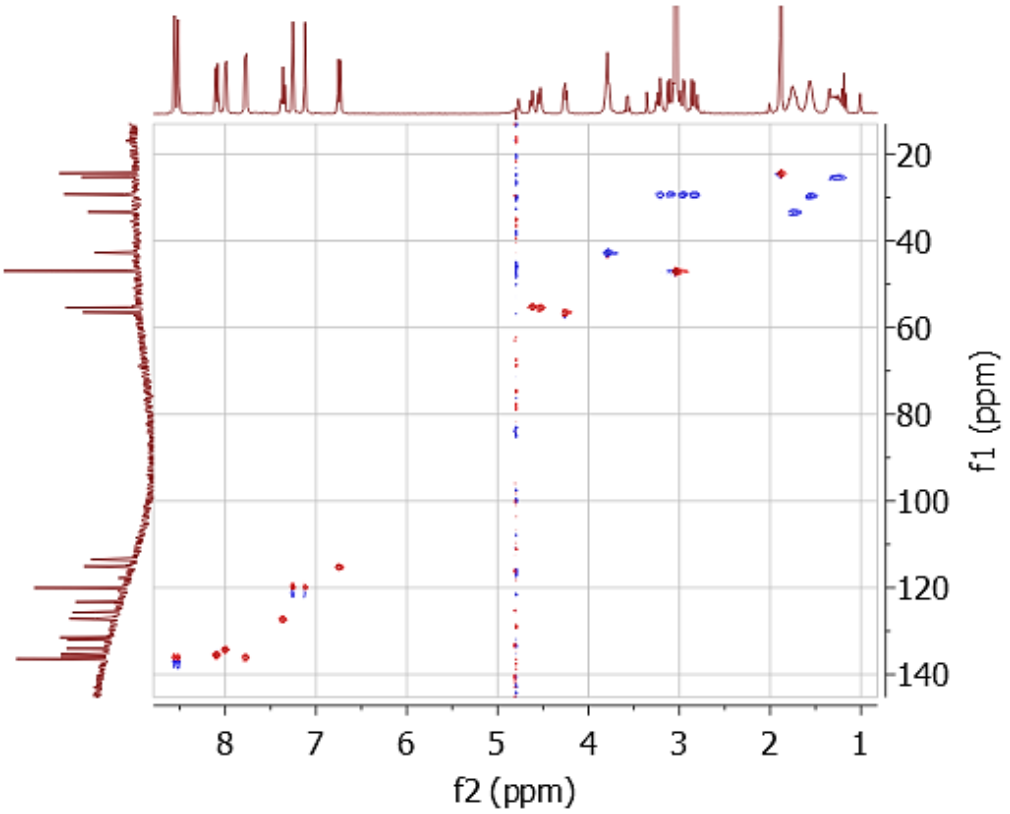

Figure S2.65: HSQC NMR spectrum of Ac-His-Lys(4DMN)-His-NH_2 (AcHK^{d}H-NH_2) in D_2O (pH 4.5).

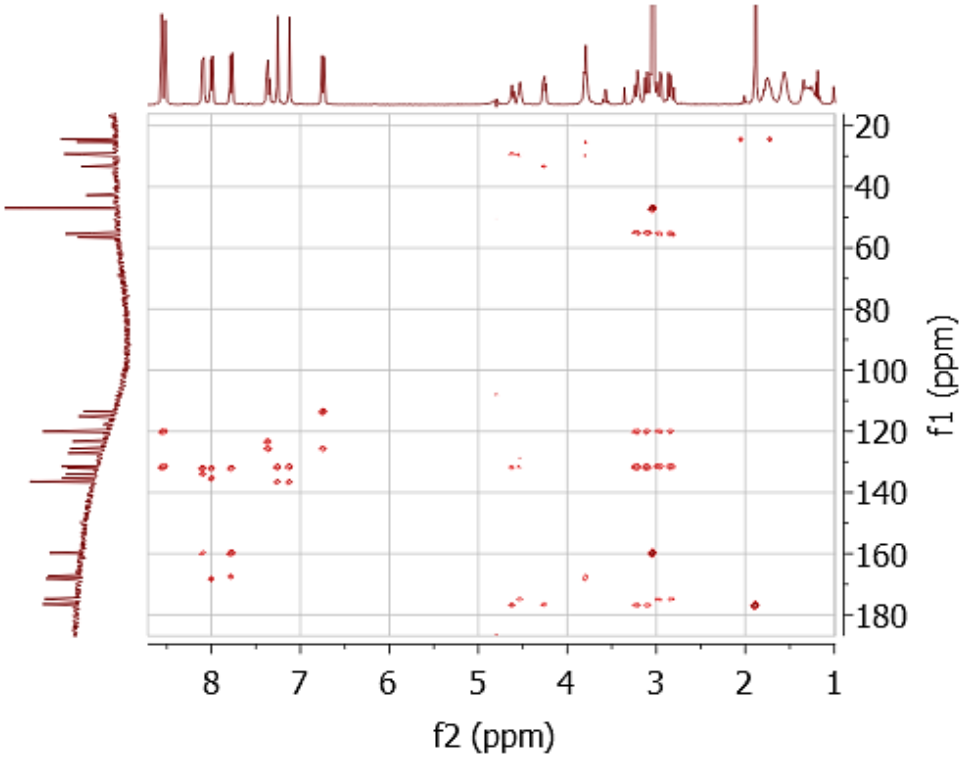

Figure S2.66: HMBC NMR spectrum of Ac-His-Lys(4DMN)-His-NH_2 (AcHK^{d}H-NH_2) in D_2O (pH 4.5).

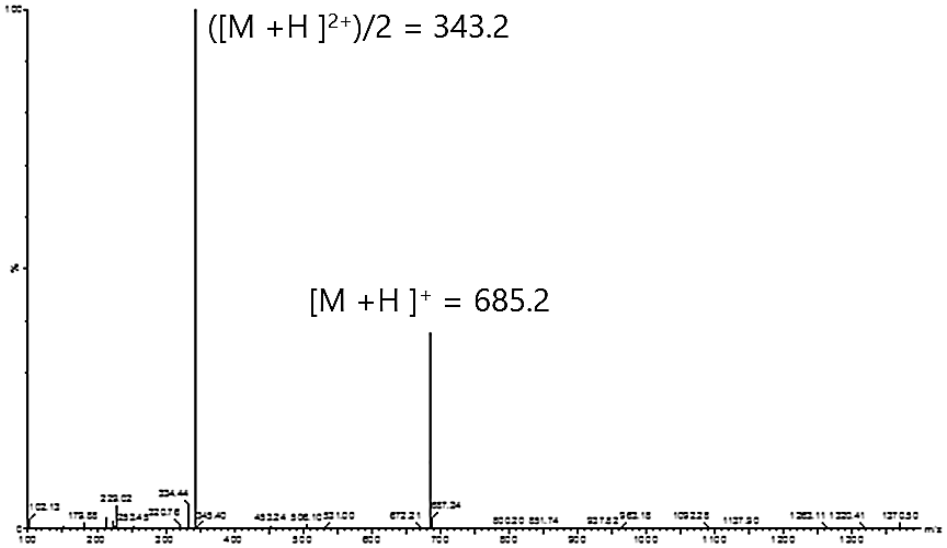

Figure S2.67: ESI mass spectrum of Ac-His-Lys(4DMN)-His-NH_2 (AcHK^{d}H-NH_2).

Ac-His-Lys(Coum)-His-Lys-NH_2 (AcHK^{c}HK)

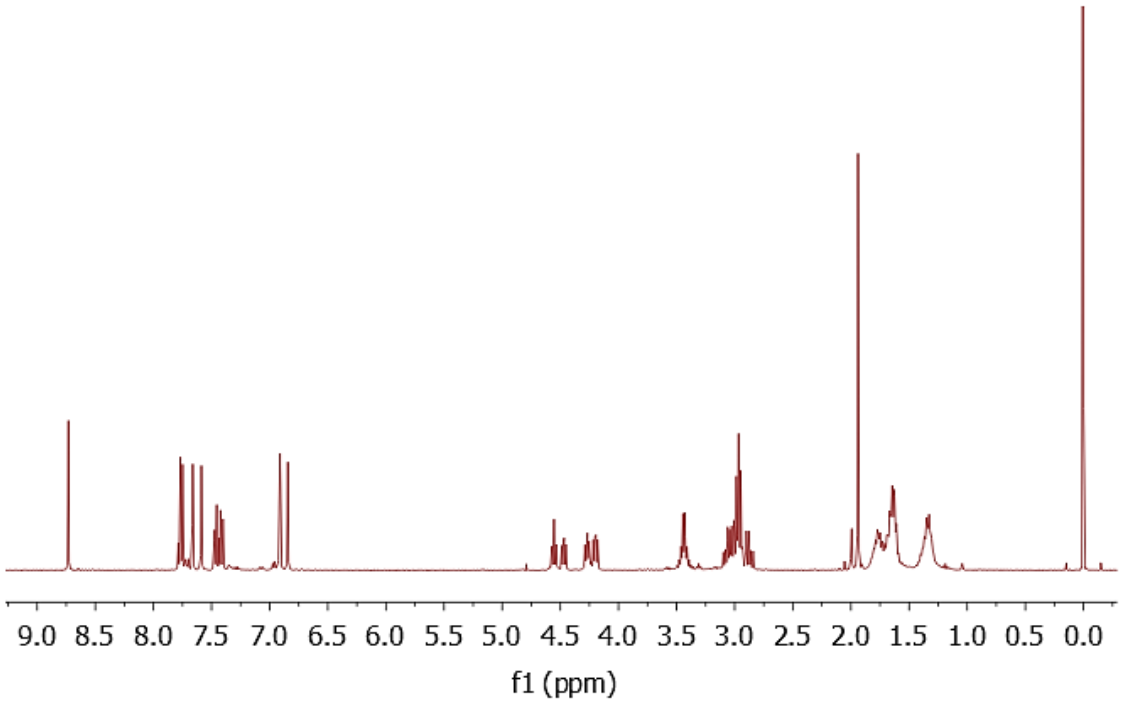

Figure S2.68: ^{1}H NMR spectrum of Ac-His-Lys(Coum)-His-Lys-NH_2 (AcHK^{c}HK) in D_2O (pH 7.4).

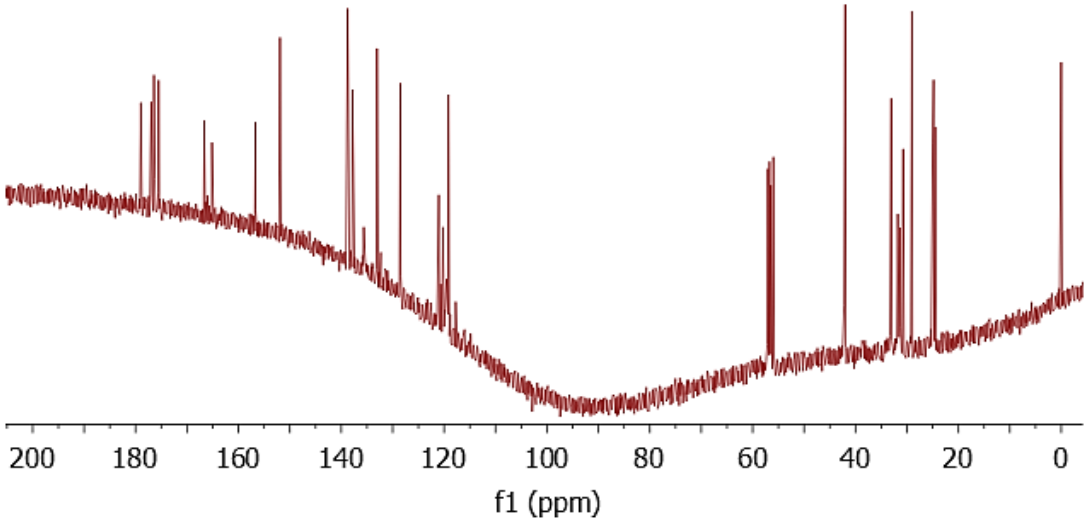

Figure S2.69: ^{13}C NMR spectrum of Ac-His-Lys(Coum)-His-Lys-NH_2 (AcHK^{c}HK) in D_2O (pH 7.4).

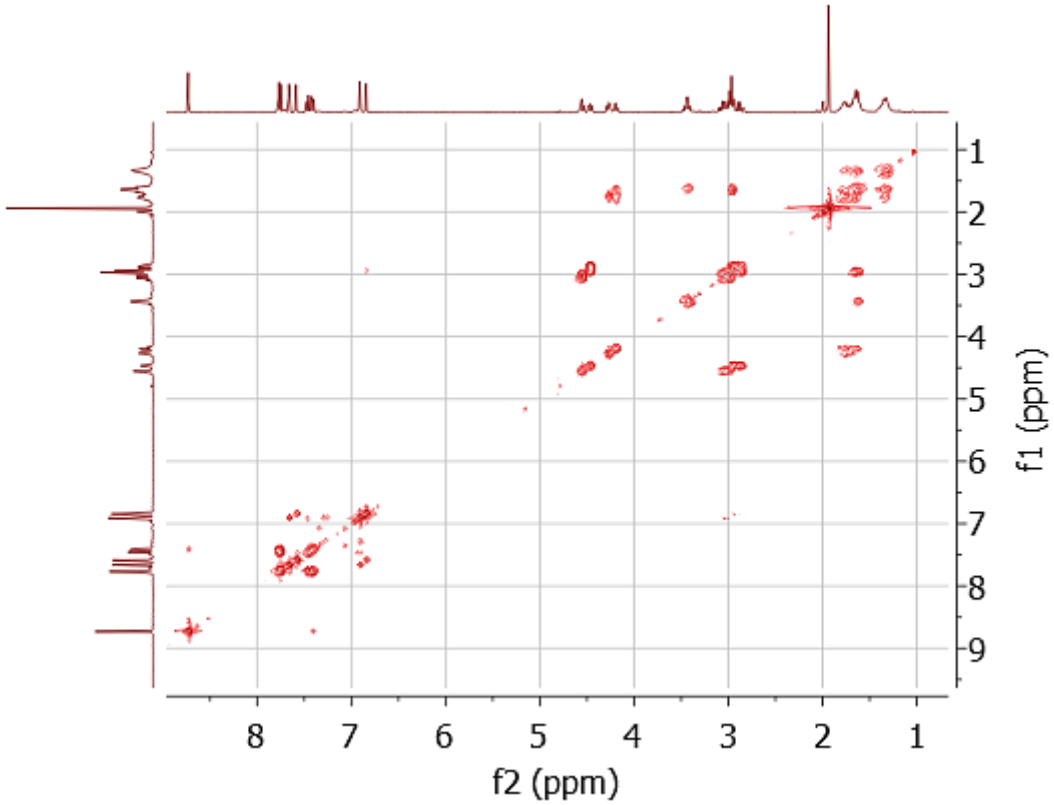

Figure S2.70: COSY NMR spectrum of Ac-His-Lys(Coum)-His-Lys-NH_2 (AcHK^{c}HK) in D_2O (pH 7.4).

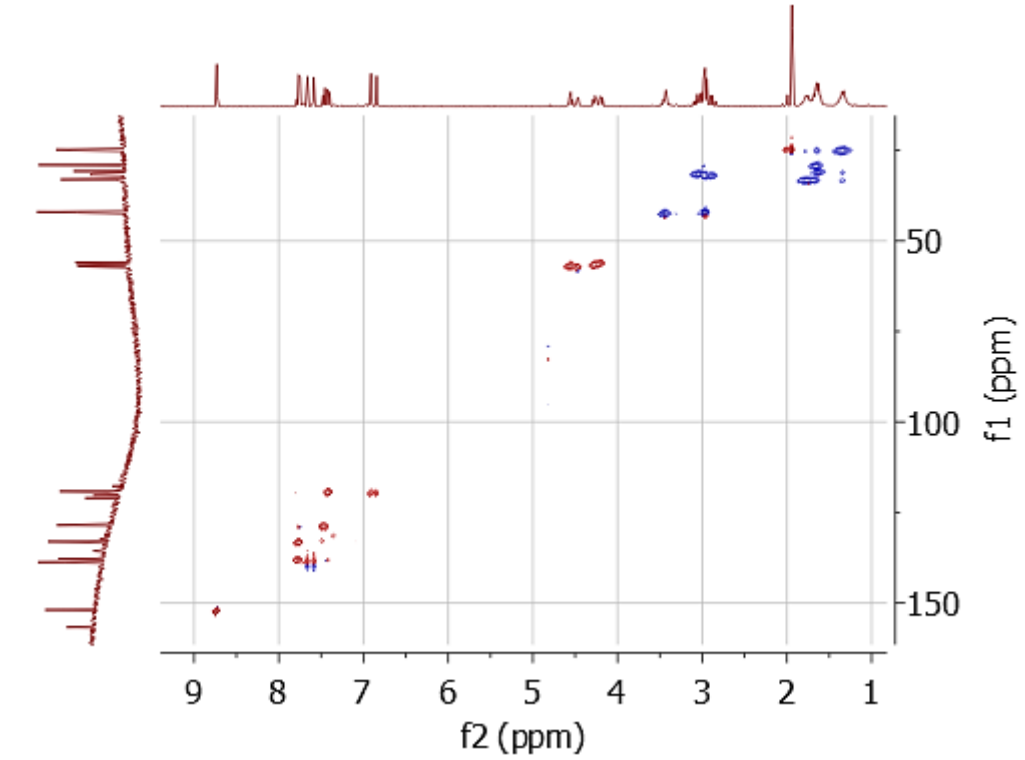

Figure S2.71: HSQC NMR spectrum of Ac-His-Lys(Coum)-His-Lys-NH_2 (AcHK^{c}HK) in D_2O (pH 7.4).

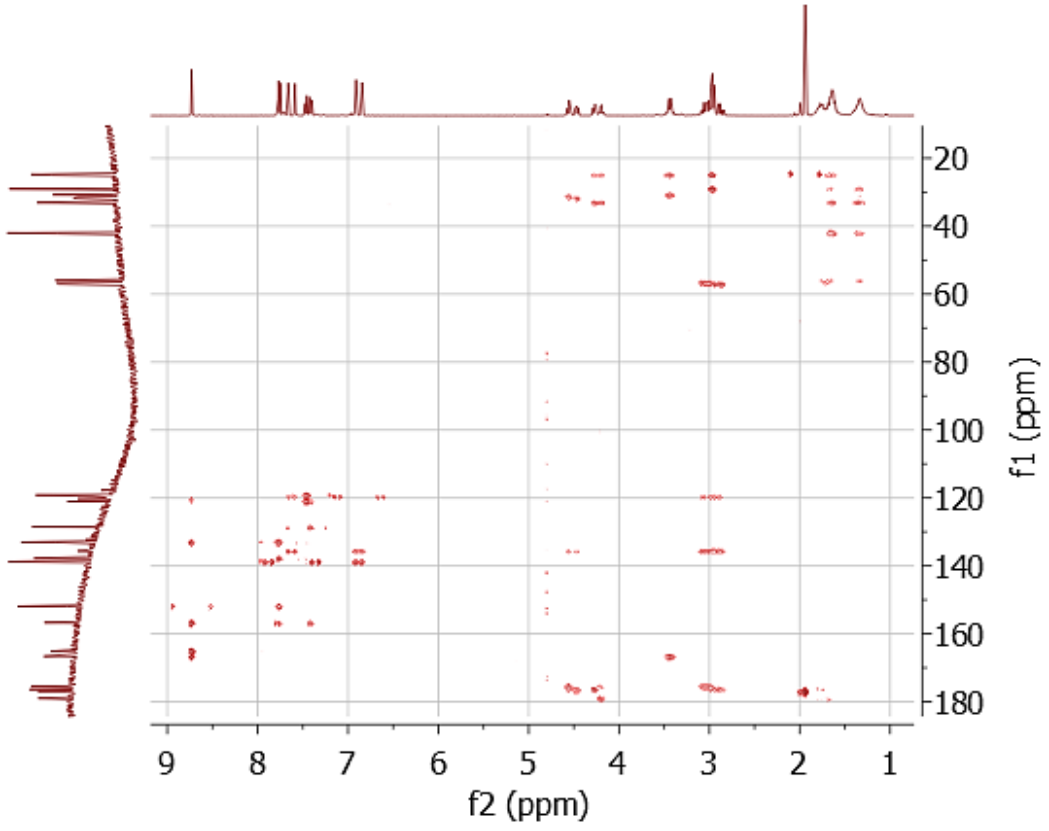

Figure S2.72: HMBC NMR spectrum of Ac-His-Lys(Coum)-His-Lys-NH_2 (AcHK^{c}HK) in D_2O (pH 7.4).

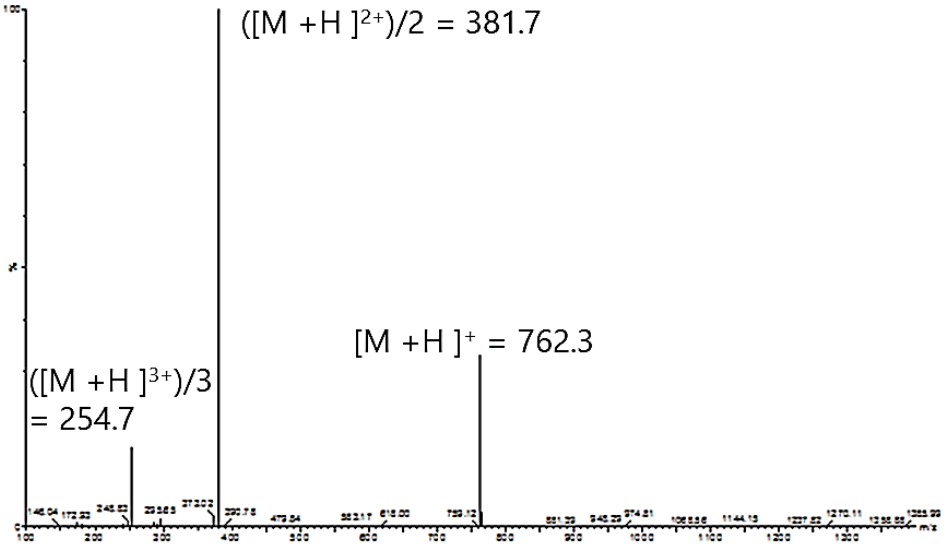

Figure S2.73: ESI mass spectrum of Ac-His-Lys(Coum)-His-Lys-NH_2 (AcHK^{c}HK).

Ac-His-Lys(4DMN)-His-Lys-NH_2 (AcHK^{d}HK)

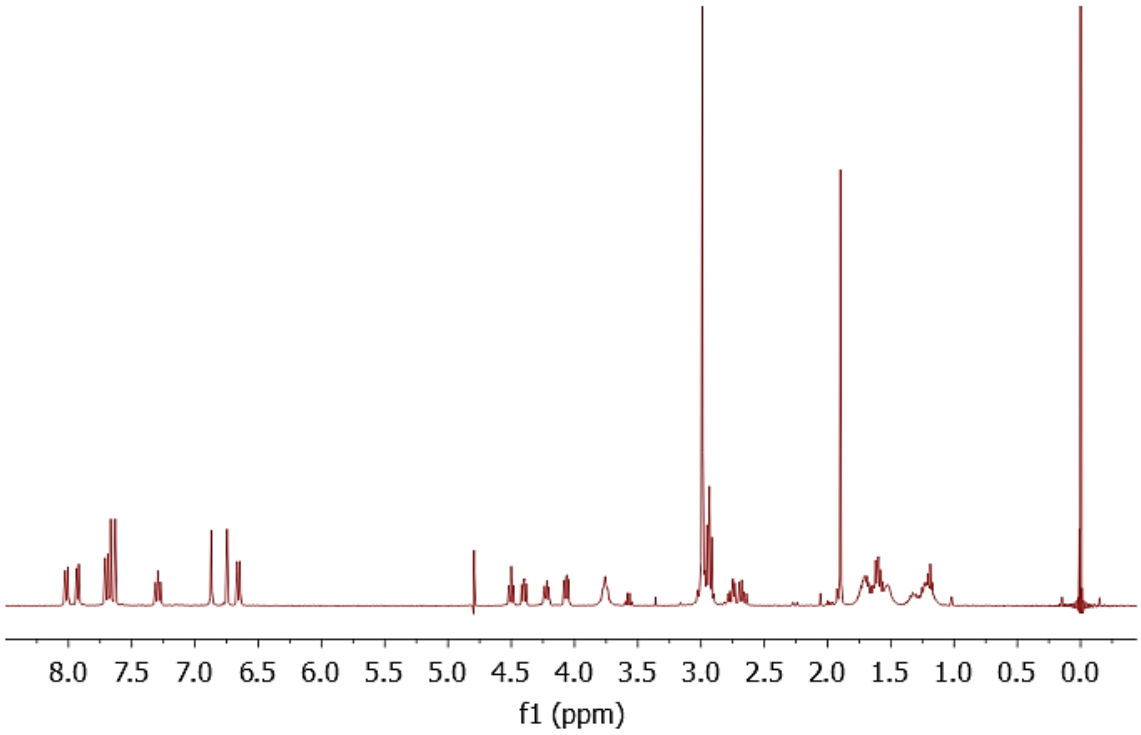

Figure S2.74: ^{1}H NMR spectrum of Ac-His-Lys(4DMN)-His-Lys-NH_2 (AcHK^{d}HK) in D_2O (pH 7.4).

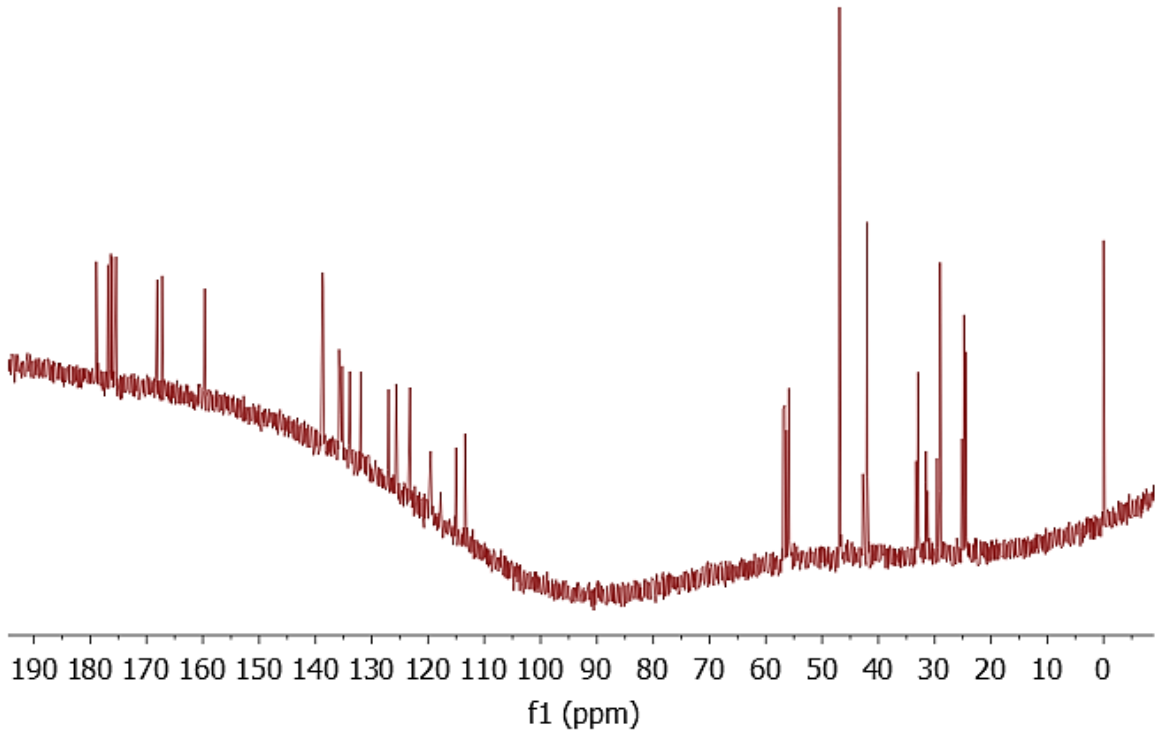

Figure S2.75: ^{13}C NMR spectrum of Ac-His-Lys(4DMN)-His-Lys-NH_2 (AcHK^{d}HK) in D_2O (pH 7.4).

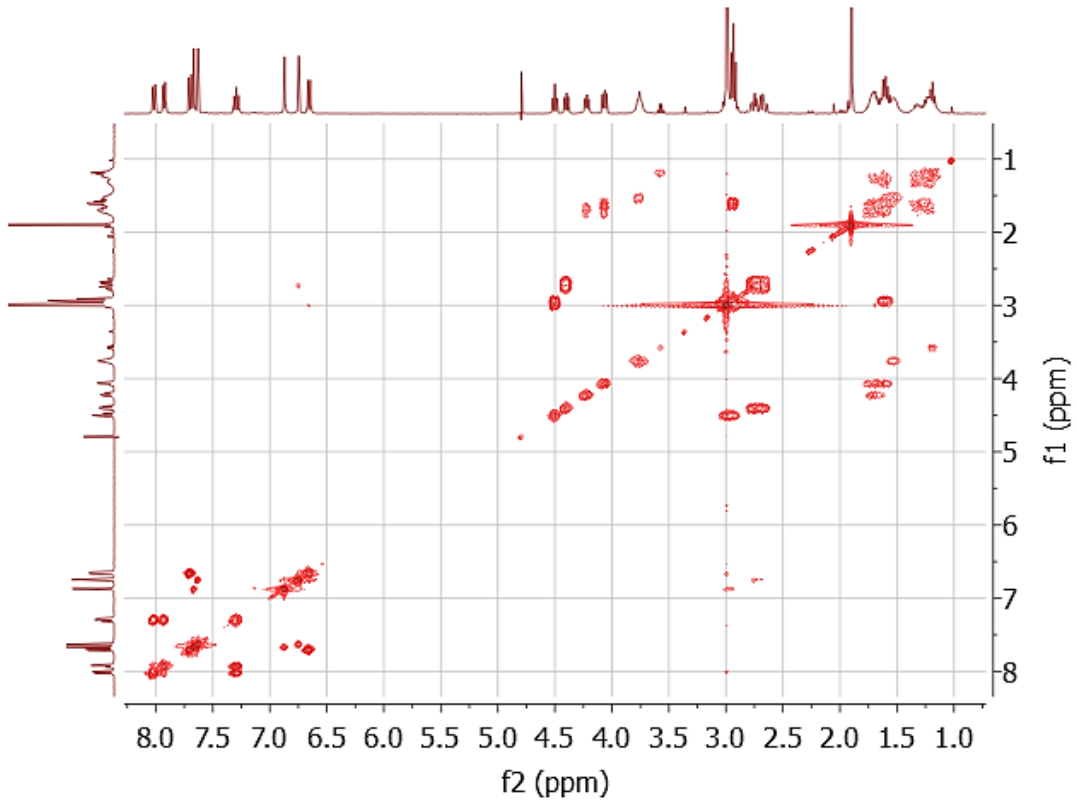

Figure S2.76: COSY NMR spectrum of Ac-His-Lys(4DMN)-His-Lys-NH_2 (AcHK^{d}HK) in D_2O (pH 7.4).

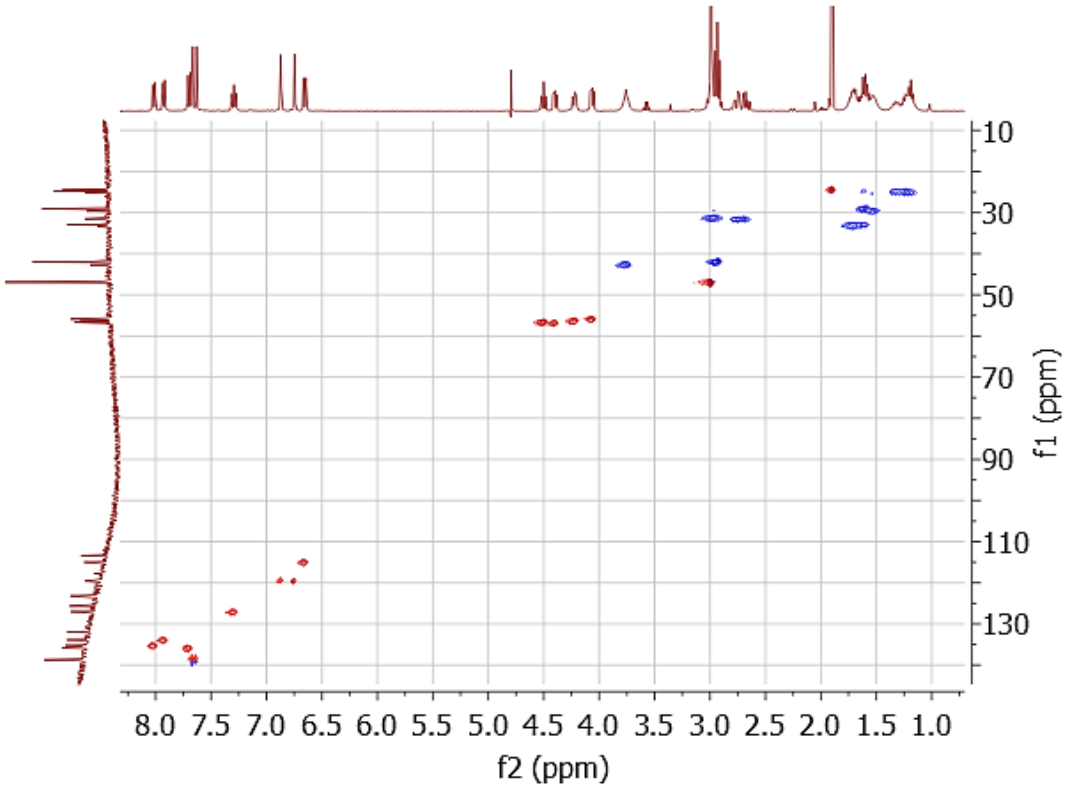

Figure S2.77: HSQC NMR spectrum of Ac-His-Lys(4DMN)-His-Lys-NH_2 (AcHK^{d}HK) in D_2O (pH 7.4).

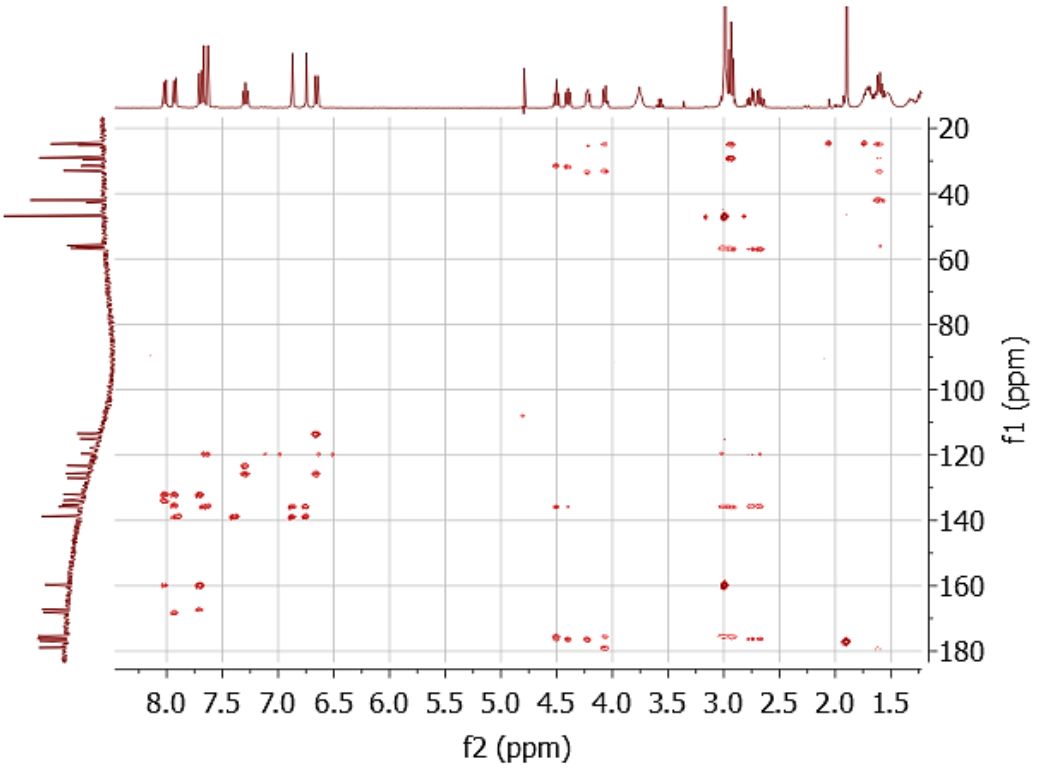

Figure S2.78: HMBC NMR spectrum of Ac-His-Lys(4DMN)-His-Lys-NH_2 (AcHK^{d}HK) in D_2O (pH 7.4).

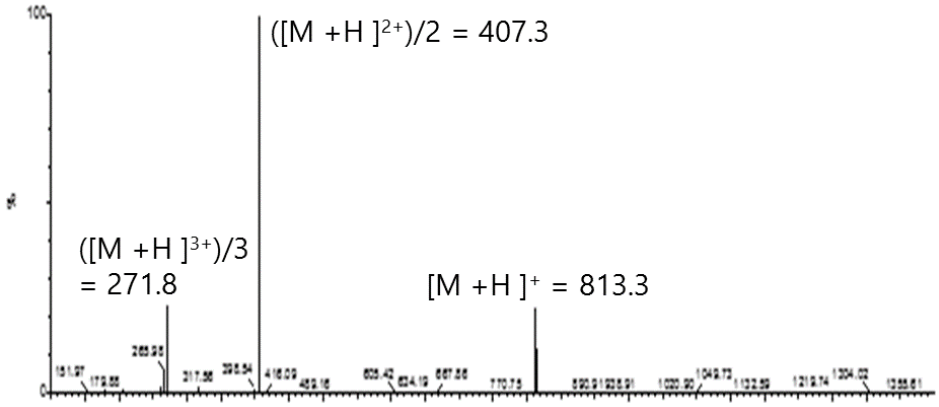

Figure S2.79: ESI mass spectrum of Ac-His-Lys(4DMN)-His-Lys-NH_2 (AcHK^{d}HK).