Structural Optimization of Reversible Dibromomaleimide Peptide Stapling

Ayanna Lindsey-Crosthwait,[a,b] Diana Rodriguez-Lema,[a,b] Martin Walko,[a,b] Christopher M. Pask[a] and Andrew J Wilson[a,b]

[a]School of Chemistry, University of Leeds, Leeds, LS2 9JT (UK) E-mail: a.j.wilson@leeds.ac.uk

[b]Astbury Centre for Structural Molecular Biology, University of Leeds, Leeds, LS2 9JT (UK).

SUPPORTING INFORMATION

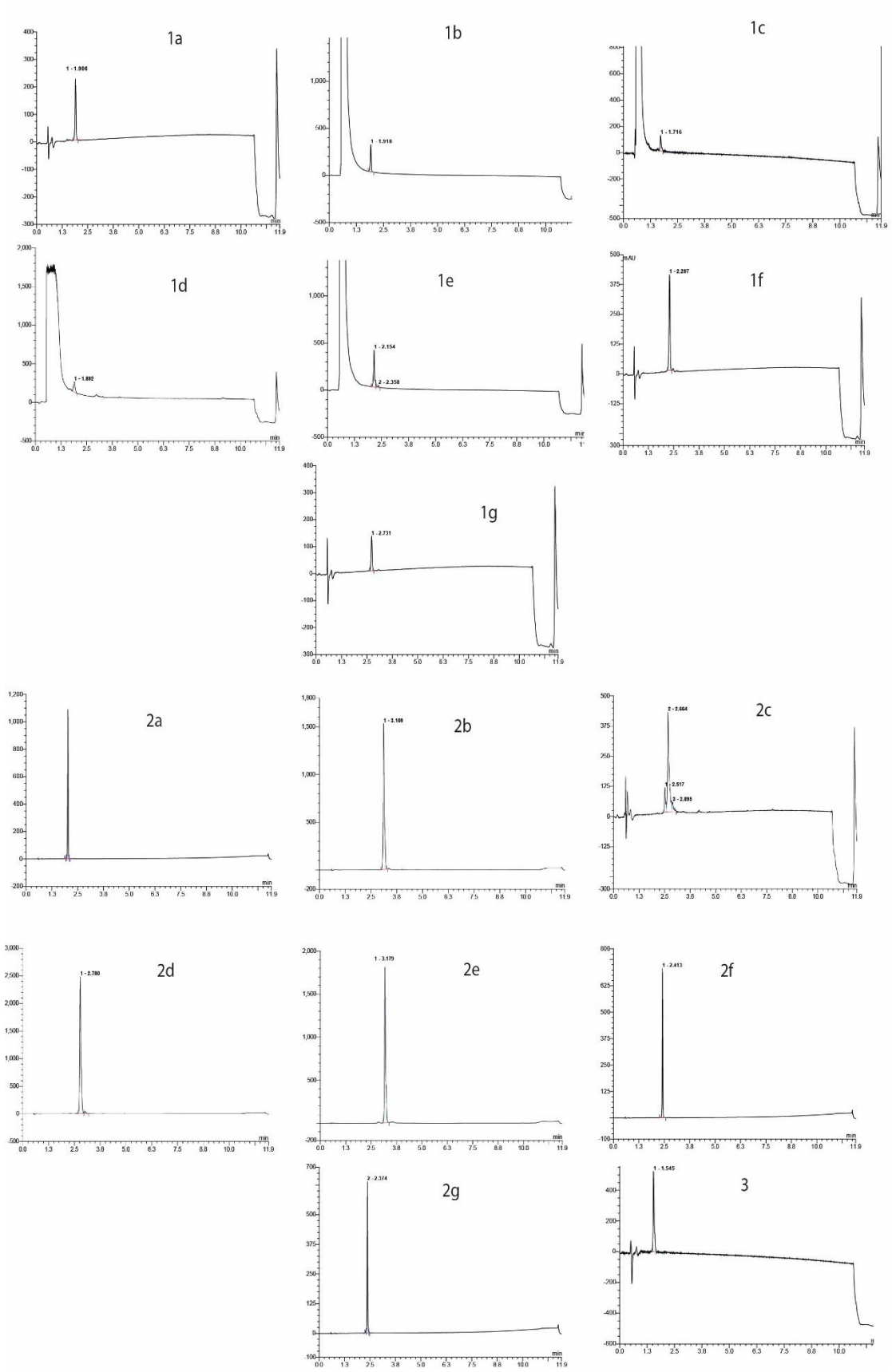
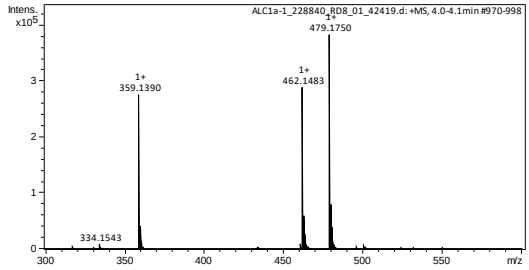


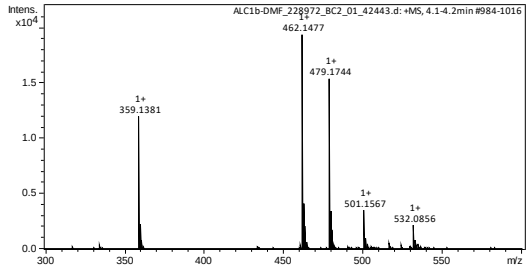
Figure S1. HPLC traces for synthetic peptides

1b

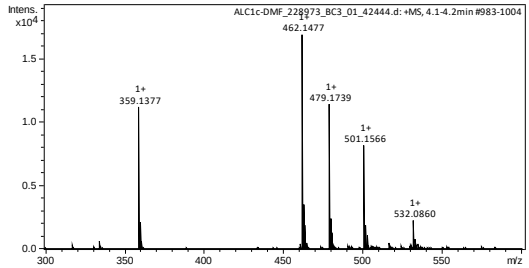
1a



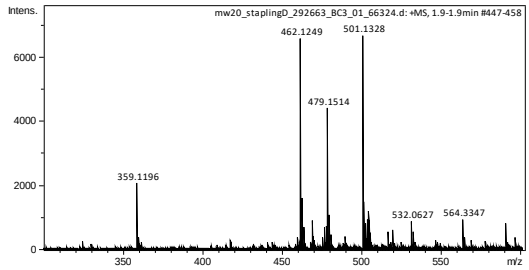
1b



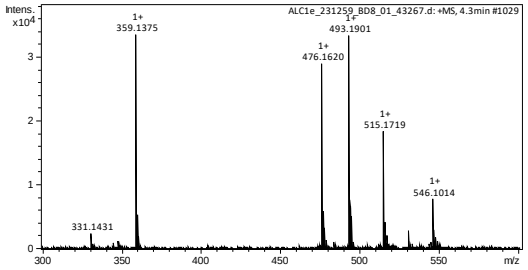
1c



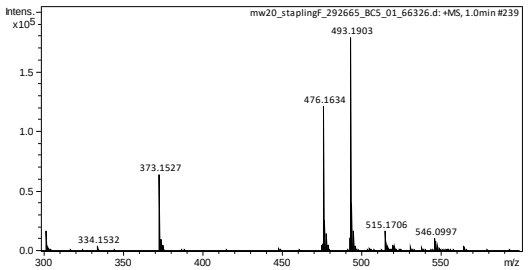
1d



1e



1f



1g

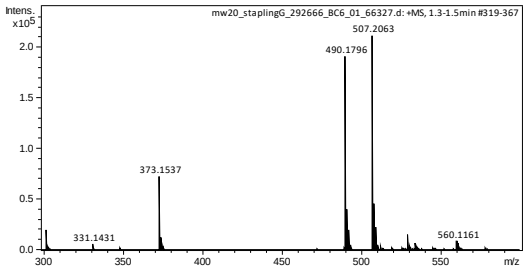
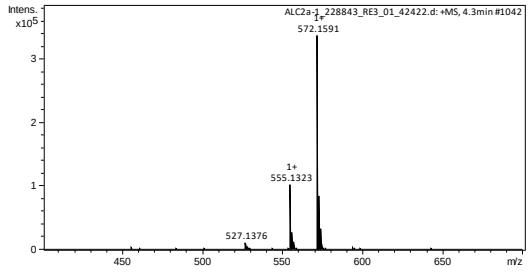


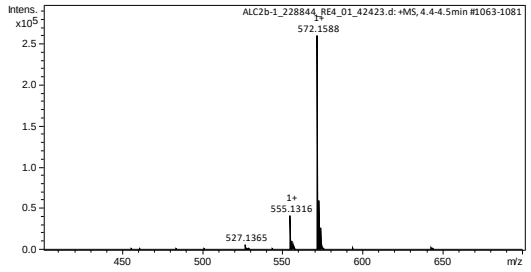
Figure S2. MS spectra for linear synthetic peptides

Except for $M+H^+$ in most of the MS spectra additional peaks corresponding to $M+Na^+$ and $[M-NH_2]^+$ can be observed

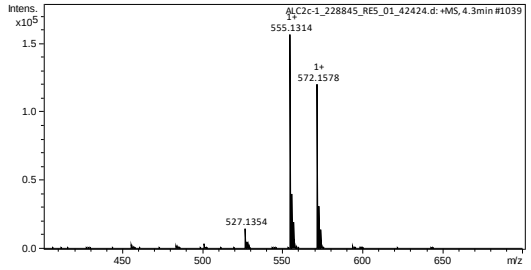
2a



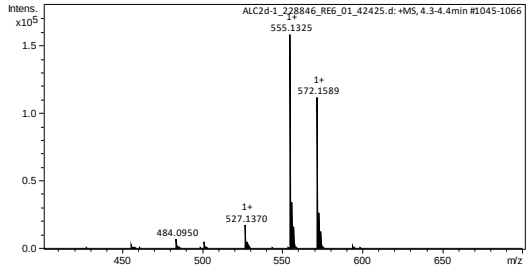
2b



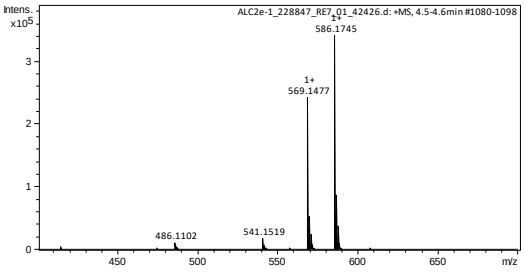
2c



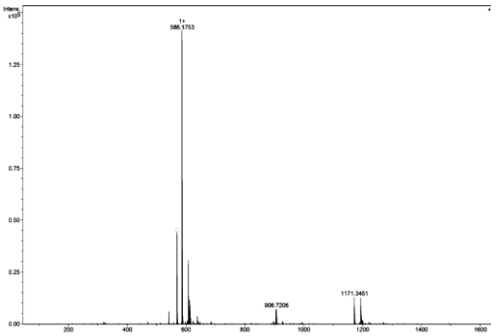
2d



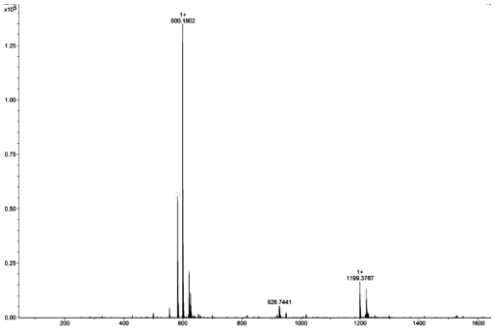
2e



2f



2g



3

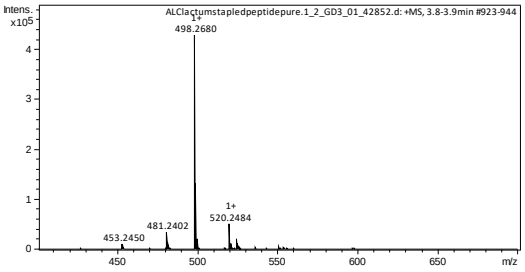


Figure S3. MS spectra for cyclic synthetic peptides.

Except for $M+H^+$ in most of the MS spectra additional peaks corresponding to $[M-NH_2]^+$ can be observed

Empirical formula	$C_{21}H_{33}N_7O_{10}S_2$
Formula weight	607.66
Temperature/K	120(2)
Crystal system	triclinic
Space group	P1
a/Å	4.9248(3)
b/Å	12.6348(9)
c/Å	12.6584(7)
α/°	115.628(6)
β/°	96.817(5)
γ/°	95.044(6)
Volume/Å^3	696.56(8)
Z	1
ρ_{calc}g/cm^3	1.449
μ/mm^{-1}	2.311
F(000)	320.0
Crystal size/mm^3	0.14 × 0.04 × 0.02
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.856 to 147.64
Index ranges	-6 ≤ h ≤ 6, -15 ≤ k ≤ 15, -14 ≤ l ≤ 15
Reflections collected	7059
Independent reflections	4047 [R_{int} = 0.0447, R_{sigma} = 0.0602]
Data/restraints/parameters	4047/3/413
Goodness-of-fit on F^2	1.073
Final R indexes [I>=2σ (I)]	R_1 = 0.0402, wR_2 = 0.0945
Final R indexes [all data]	R_1 = 0.0448, wR_2 = 0.0973
Largest diff. peak/hole / e Å^{-3}	0.34/-0.27
Flack parameter	0.03(2)

Table S1 Experimental data for the crystal structure determination of **2a**. CCDC-1976850 contains additional information in cif format and can be obtained from the CCDC via www.ccdc.cam.ac.uk/structures/.