

– **Electronic Supplementary Information (ESI)** –

**Unusual truncation of *N*-acylated peptoids under acidic conditions**

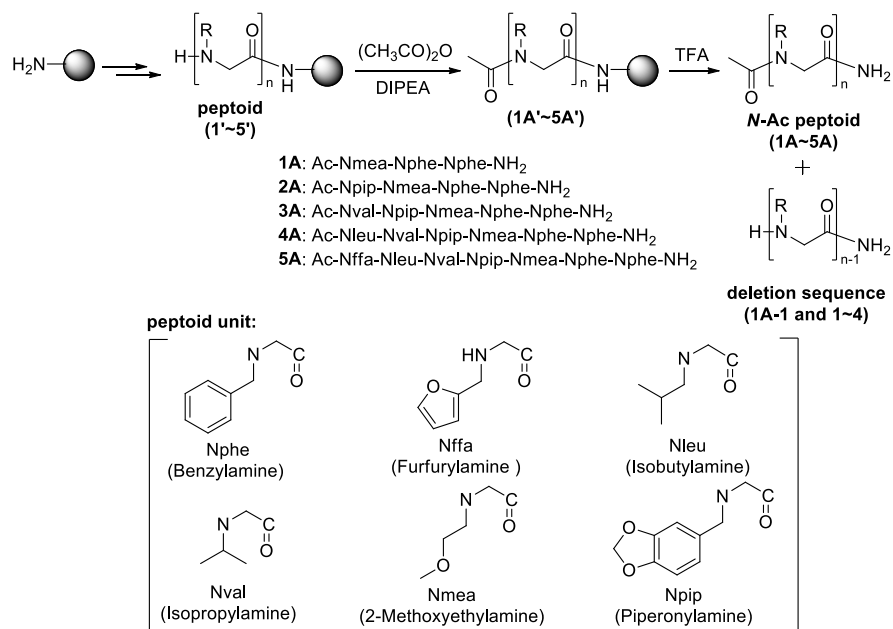
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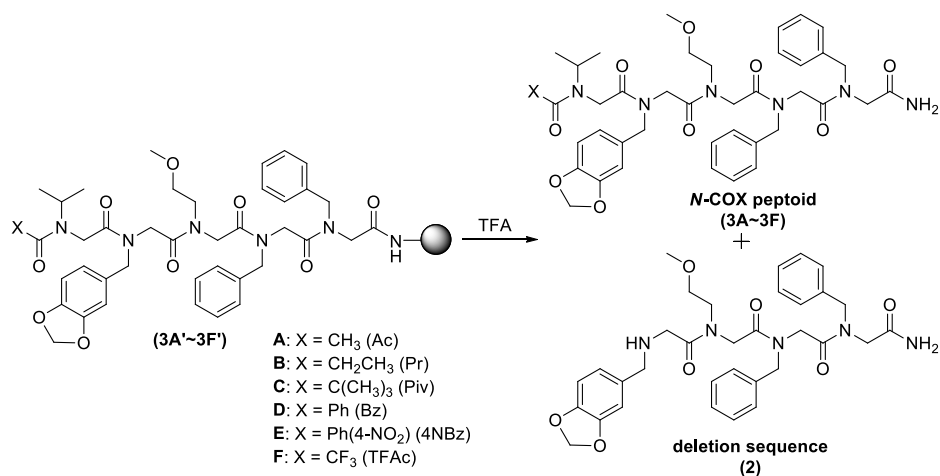
**Table S1.** TFA-assisted cleavage of *N*-acetylated model peptoids



Entry	Peptoid	Cleavage conditions <sup>a</sup>	Product (%) <sup>b</sup>	
			<i>N</i> -Ac peptoid	Deletion sequence
1		A	<b>1A</b> (67.0)	<b>1A-1</b> (33.0)
2	<b>1A'</b>	B	<b>1A</b> (70.7)	<b>1A-1</b> (29.3)
3		A	<b>2A</b> (80.9)	<b>1</b> (19.1)
4	<b>2A'</b>	B	<b>2A</b> (84.6)	<b>1</b> (15.4)
5		A	<b>3A</b> (0)	<b>2</b> (100)
6	<b>3A'</b>	B	<b>3A</b> (0)	<b>2</b> (100)
7		A	<b>4A</b> (74.3)	<b>3</b> (25.7)
8	<b>4A'</b>	B	<b>4A</b> (82.7)	<b>3</b> (17.3)
9		A	<b>5A</b> (93.8)	<b>4</b> (6.2)
10	<b>5A'</b>	B	<b>5A</b> (94.5)	<b>4</b> (5.5)

<sup>a</sup> Conditions A: 92% TFA/3% TIS/5% H<sub>2</sub>O, 1 h; B: 50% TFA/3% TIS/5% H<sub>2</sub>O/42% CH<sub>2</sub>Cl<sub>2</sub>, 1 h.

<sup>b</sup> Relative yields of *N*-Ac peptoids and deletion sequences were determined by HPLC analysis.

**Table S2.** TFA-assisted cleavage of various *N*-acylated peptoids

Entry	Acyl group	Cleavage conditions <sup>a</sup>	Product (%) <sup>b</sup>	
			<i>N</i> -COX peptoid	Deletion sequence
1		A	<b>3A</b> (0)	<b>2</b> (100)
2	Ac	B	<b>3A</b> (0)	<b>2</b> (100)
3		A	<b>3B</b> (0)	<b>2</b> (100)
4	Pr	B	<b>3B</b> (0)	<b>2</b> (100)
5		A	<b>3C</b> (0)	<b>2</b> (100)
6	Piv	B	<b>3C</b> (0)	<b>2</b> (100)
7		A	<b>3D</b> (17.5)	<b>2</b> (82.5)
8	Bz	B	<b>3D</b> (32.5)	<b>2</b> (67.5)
9		A	<b>3E</b> (96.1/89.2 <sup>c</sup> )	<b>2</b> (3.9/10.8 <sup>c</sup> )
10	4-NBz	B	<b>3E</b> (96.4/92.7 <sup>c</sup> )	<b>2</b> (3.6/7.3 <sup>c</sup> )
11		A	<b>3F</b> (100/100 <sup>c</sup> )	<b>2</b> (0/0 <sup>c</sup> )
12	TFAc	B	<b>3F</b> (100/100 <sup>c</sup> )	<b>2</b> (0/0 <sup>c</sup> )

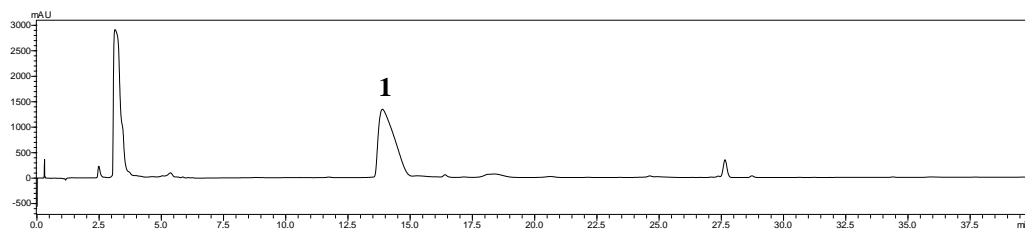
<sup>a</sup> Conditions A: 92% TFA/3% TIS/5% H<sub>2</sub>O, 1 h; B: 50% TFA/3% TIS/5% H<sub>2</sub>O/42% CH<sub>2</sub>Cl<sub>2</sub>, 1 h. <sup>b</sup> Relative yields of *N*-COX peptoids and deletion sequences were determined by HPLC analysis. <sup>c</sup> Cleavage time: 3 h.

**Table S3.** HPLC retention time and MALDI-TOF data of peptoids

Entry	Peptoid	HPLC Retention time (min) <sup>a</sup>	MALDI-TOF ( <i>m/z</i> )	
			observed	calculated
1	<b>1</b>	13.8	427.2	427.2 (M+H)
2	<b>1A</b>	18.0	491.4	491.2 (M+Na)
3	<b>1A-1</b>	10.4	312.4	312.1 (M+H)
4	<b>2</b>	19.5	618.2	618.3 (M+H)
5	<b>2A</b>	23.2	682.3	682.3 (M+Na)
6	<b>3</b>	19.9	717.8	717.4 (M+H)
7	<b>4</b>	22.9	830.5	830.4 (M+H)
8	<b>4A</b>	26.9	895.0	894.5 (M+Na)
9	<b>5</b>	21.0	967.9	967.5 (M+H)
10	<b>5A</b>	29.2	1031.4	1031.5 (M+Na)
11	<b>3D</b>	29.1	843.9	843.4 (M+Na)
12	<b>3E</b>	30.5	888.9	888.4 (M+Na)
13	<b>3F</b>	31.7	835.5	835.3 (M+Na)

<sup>a</sup> HPLC conditions: 20% ACN/H<sub>2</sub>O to 80% ACN/H<sub>2</sub>O with 0.05% TFA for 40 min.

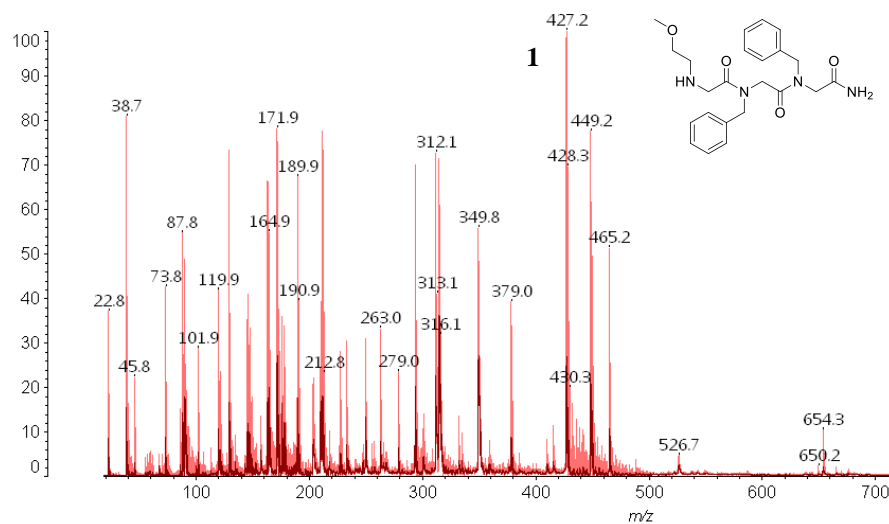
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(b)

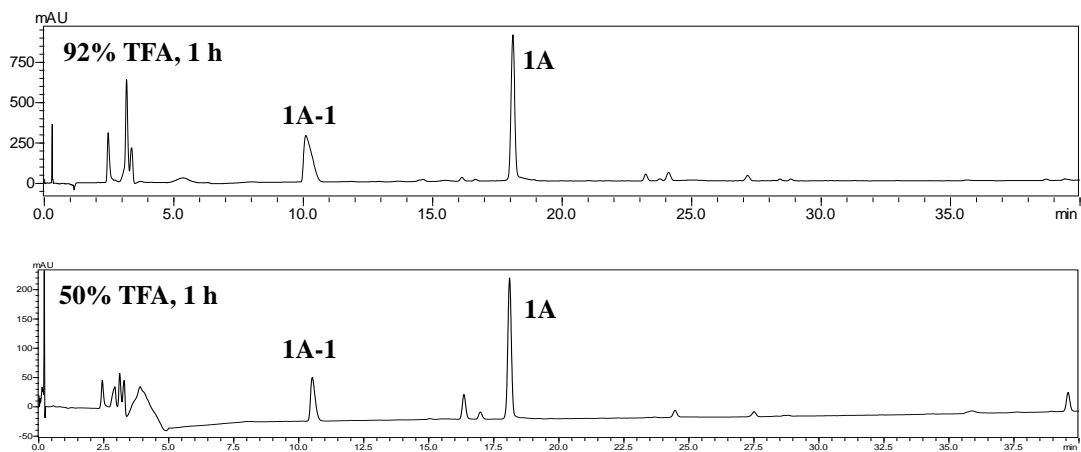
Ewha Univ.

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%Int. 1417 mV[sum= 72274 mV] Profiles 1-51 Unsmoothed



**Figure S1.** TFA-assisted cleavage of peptoid **1'**: (a) HPLC chromatogram and (b) MALDI-TOF spectrum.

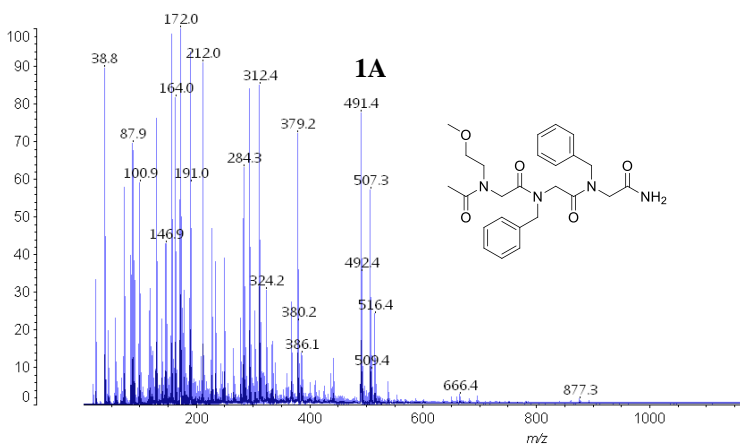
(a)



(b)

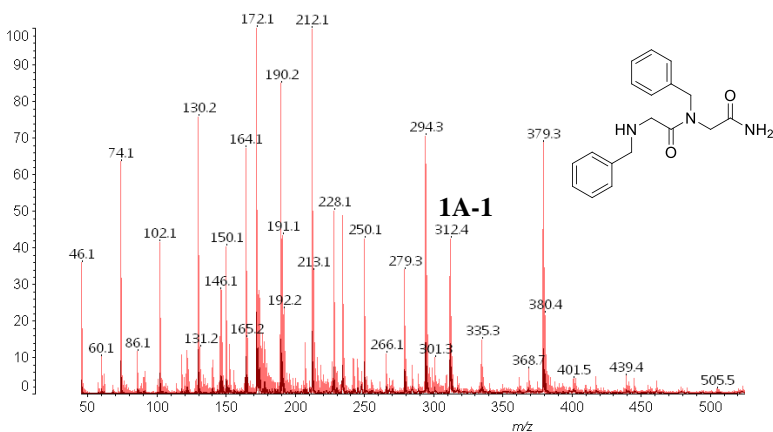
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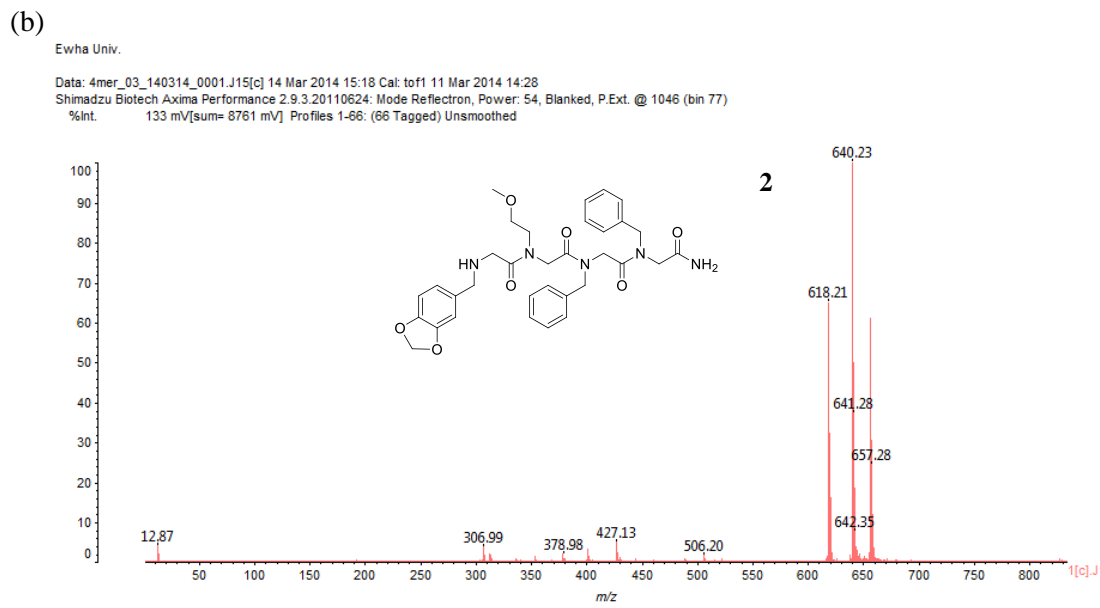
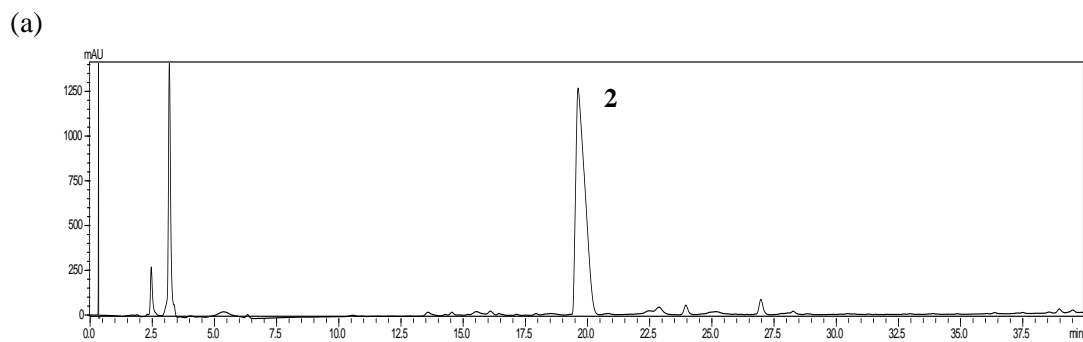


Ewha Univ.

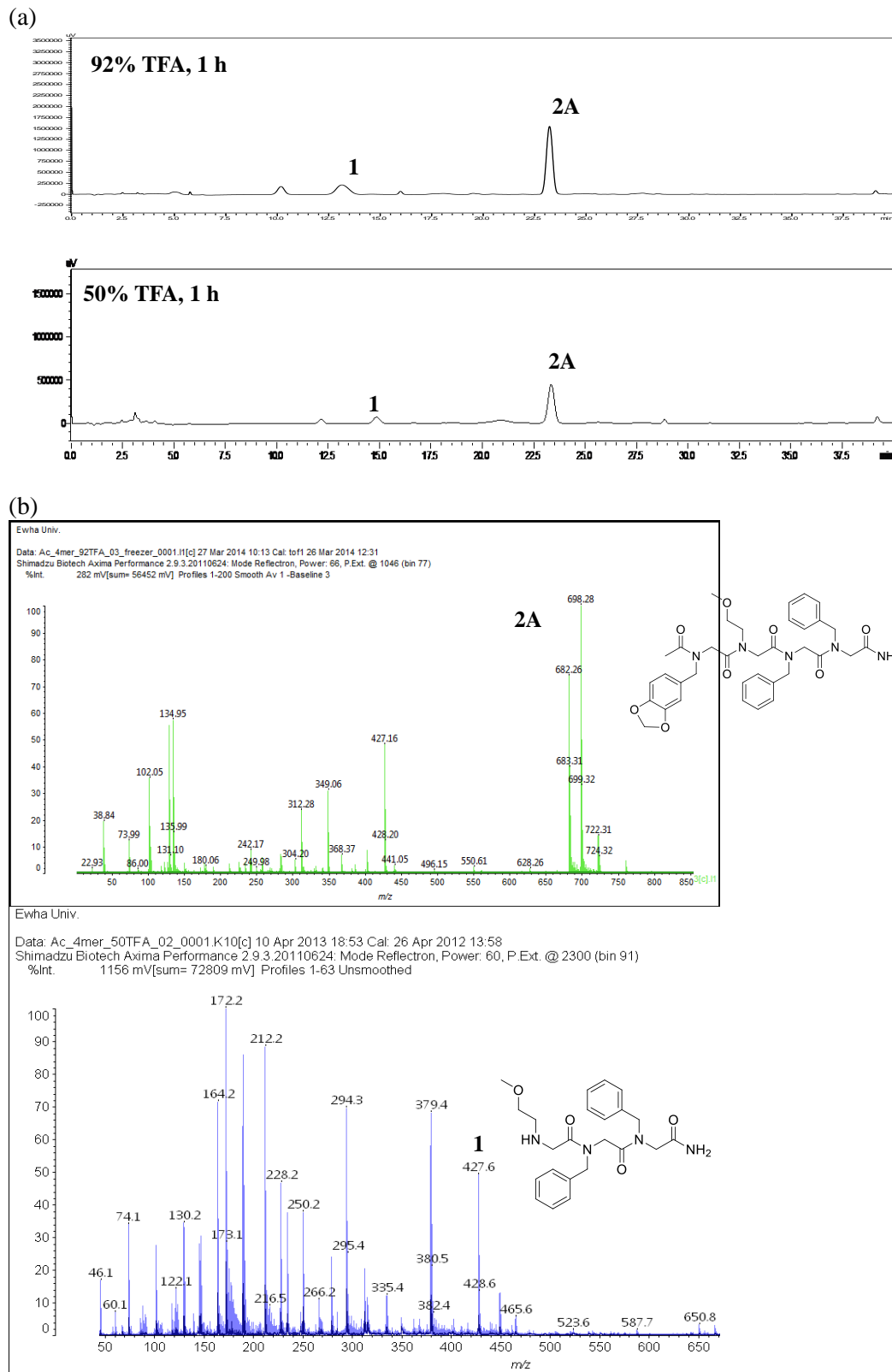
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**Figure S2.** TFA-assisted cleavage of peptoid 1A': (a) HPLC chromatograms and (b) MALDI-TOF spectra.

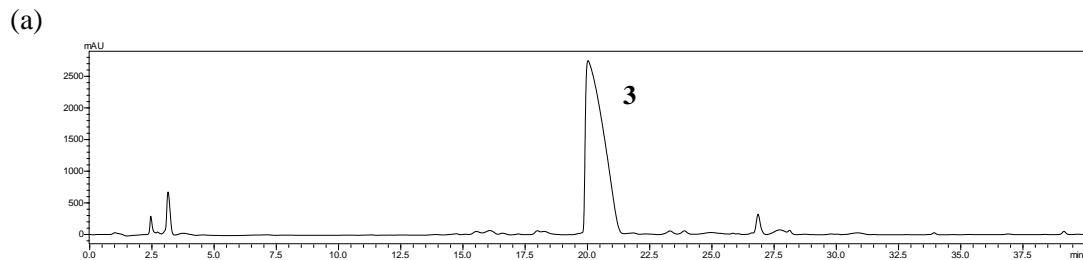


**Figure S3.** TFA-assisted cleavage of peptoid **2'**: (a) HPLC chromatogram and (b) MALDI-TOF spectrum.



**Figure S4.** TFA-assisted cleavage of peptoid **2A'**: (a) HPLC chromatograms and (b) MALDI-TOF spectra.

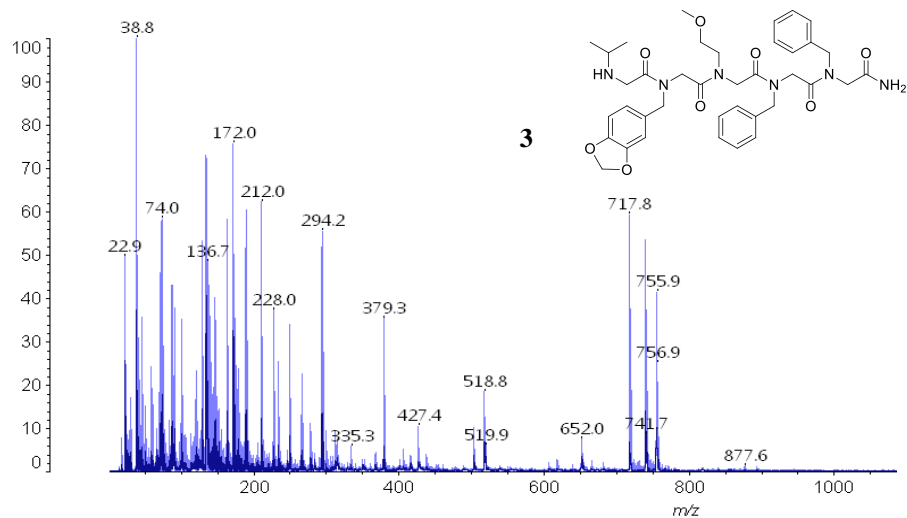




(b)

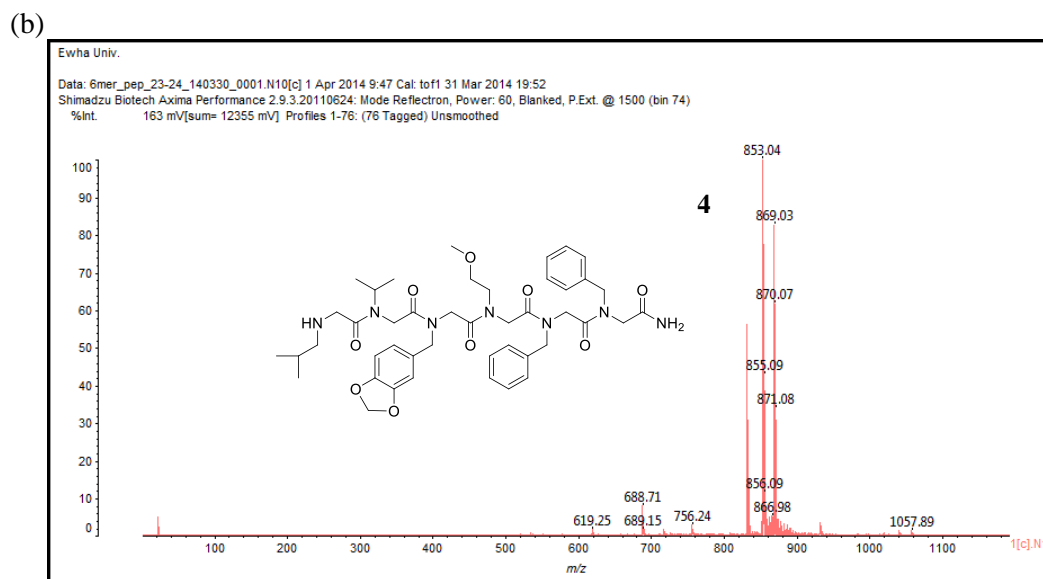
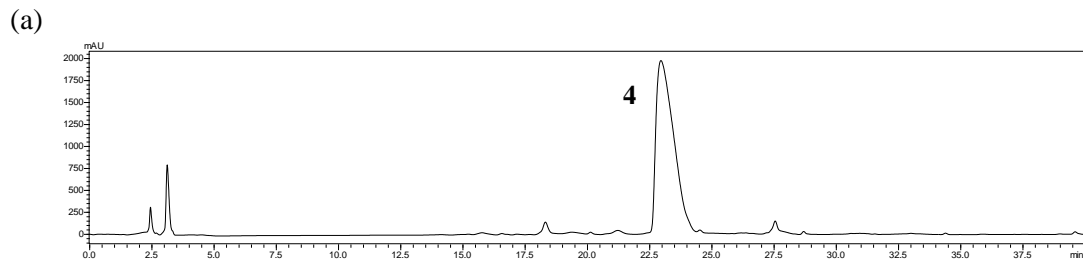
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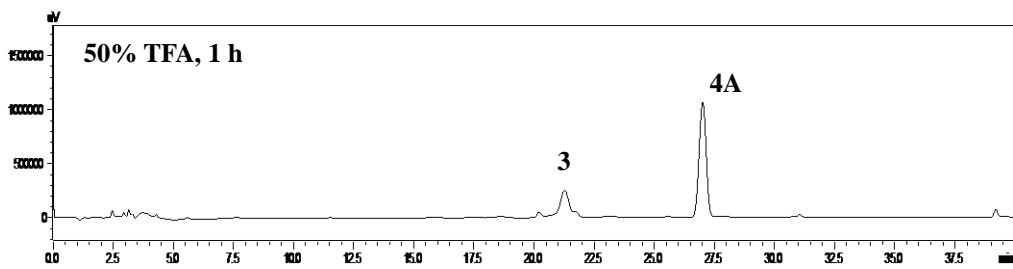
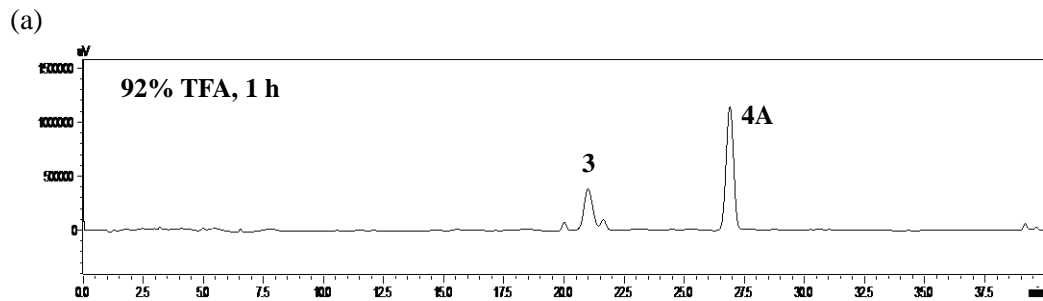


**Figure S5.** TFA-assisted cleavage of peptoid **3'**: (a) HPLC chromatogram and (b) MALDI-TOF spectrum.





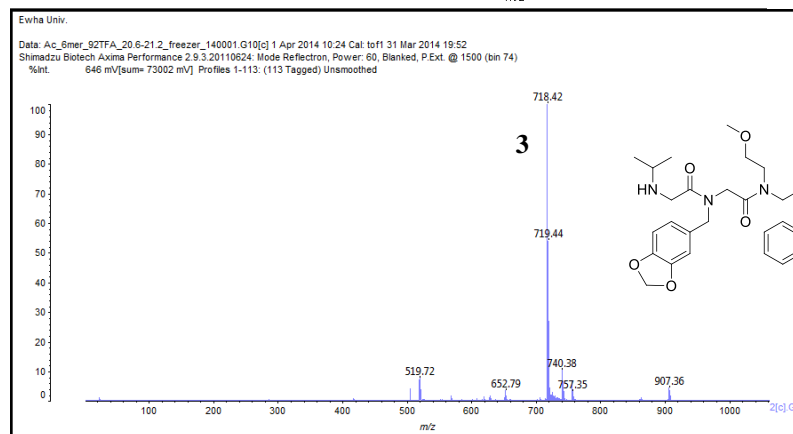
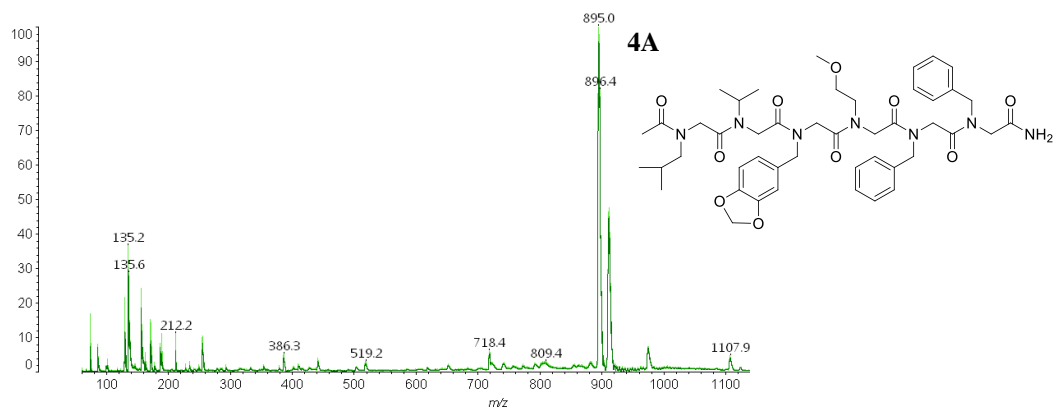
**Figure S7.** TFA-assisted cleavage of peptoid **4'**: (a) HPLC chromatogram and (b) MALDI-TOF spectrum.



(b)

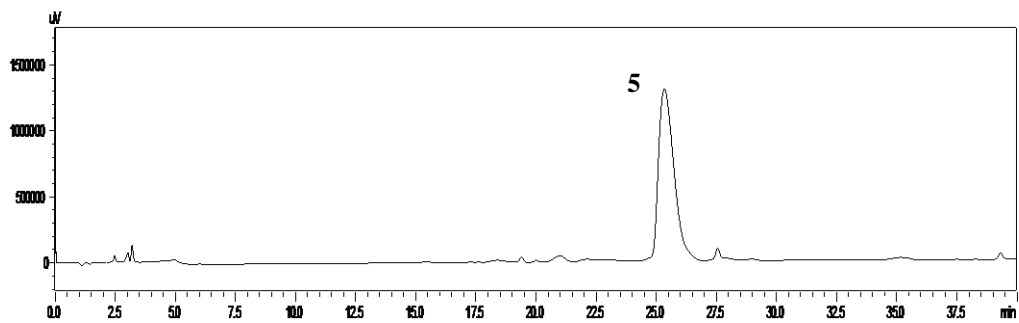
Ewha Univ.

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 %Int. 907 mV[sum= 80755 mV] Profiles 1-89 Unsmoothed



**Figure S8.** TFA-assisted cleavage of peptoid **4A'**: (a) HPLC chromatograms and (b) MALDI-TOF spectra.

(a)



(b)

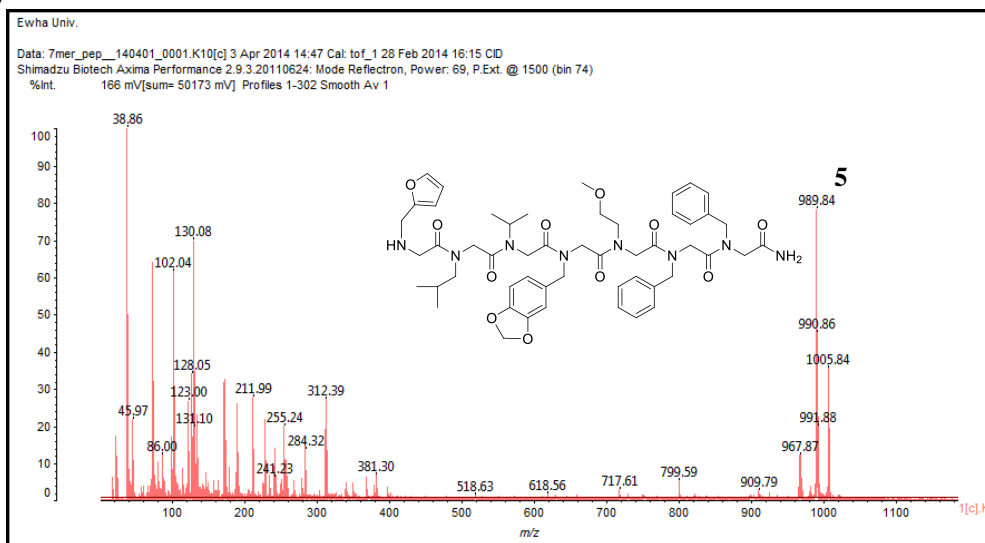
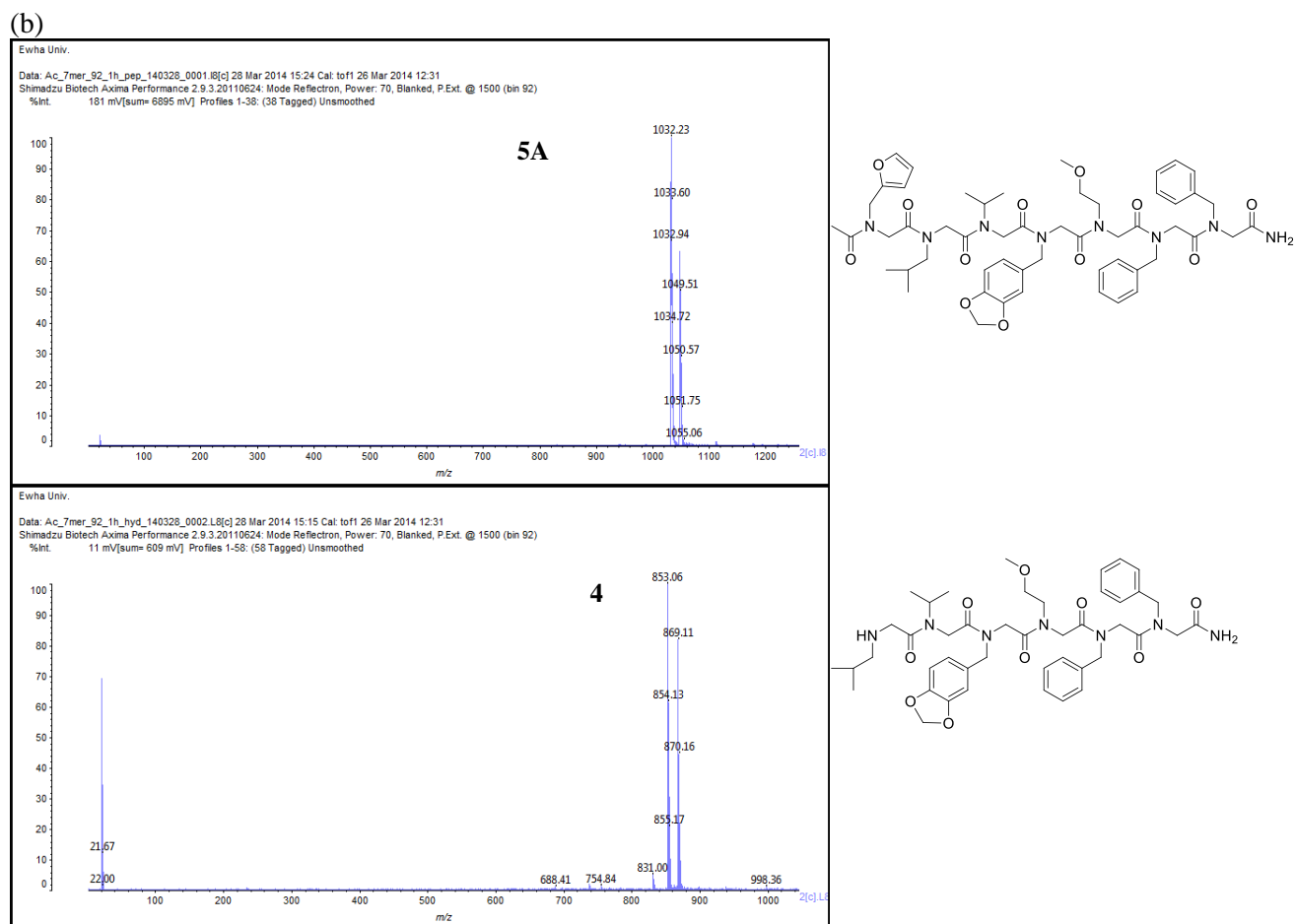
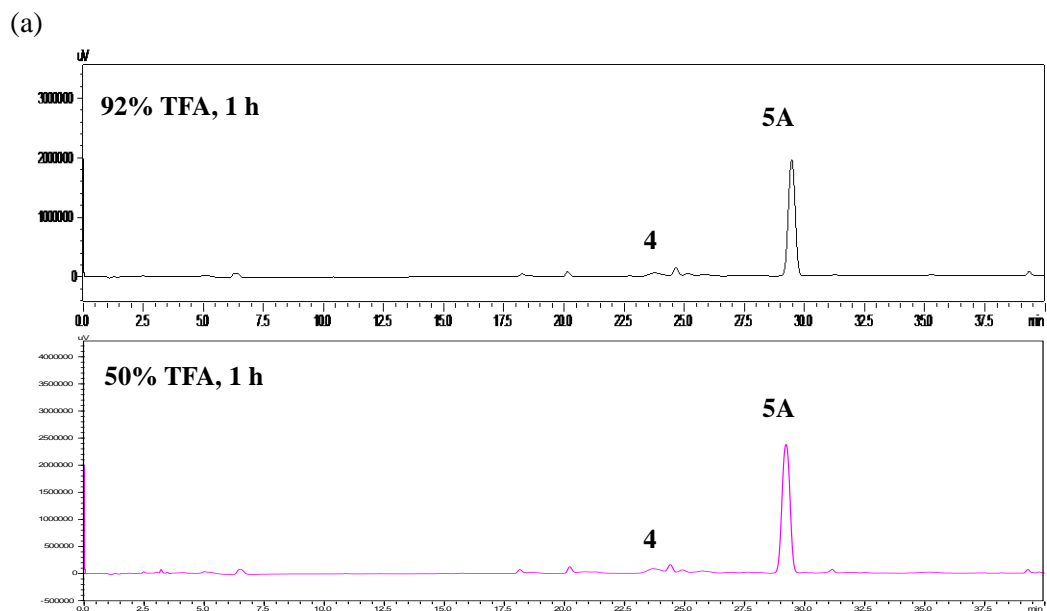
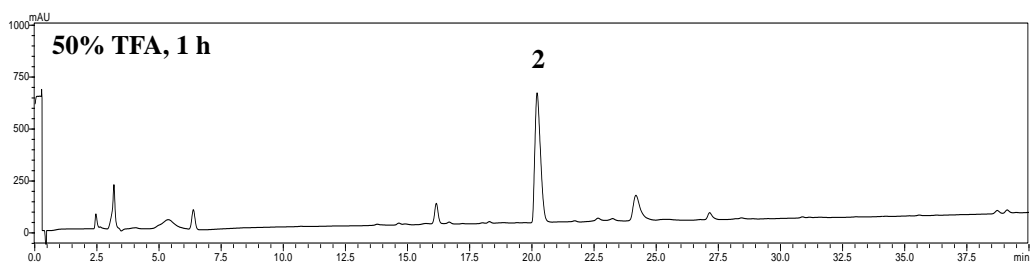
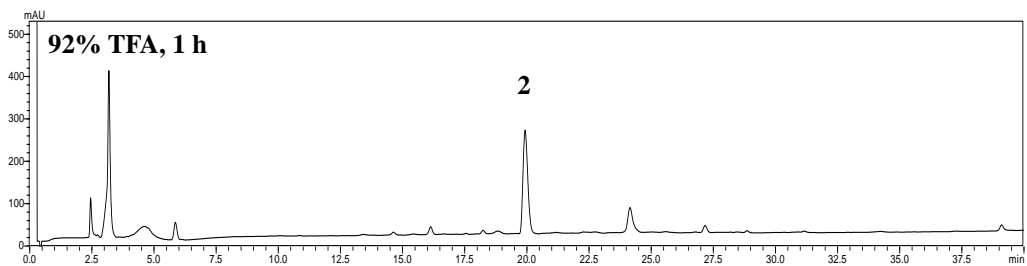


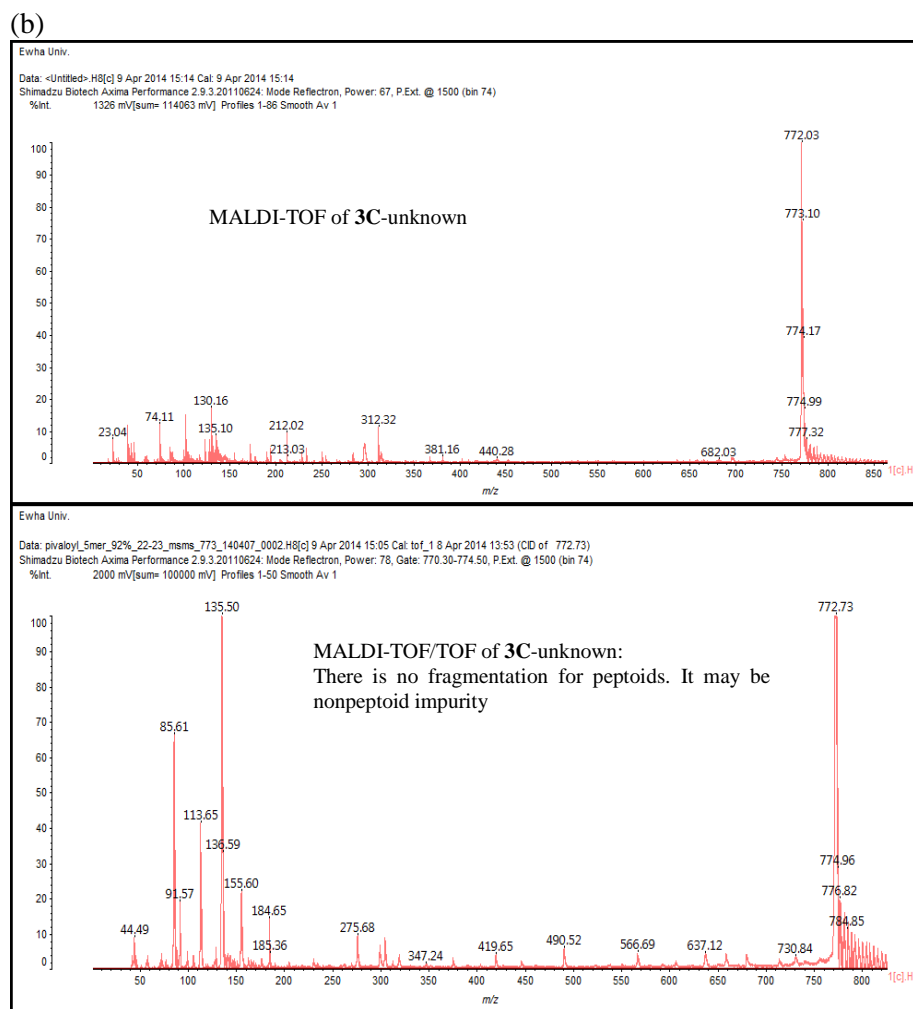
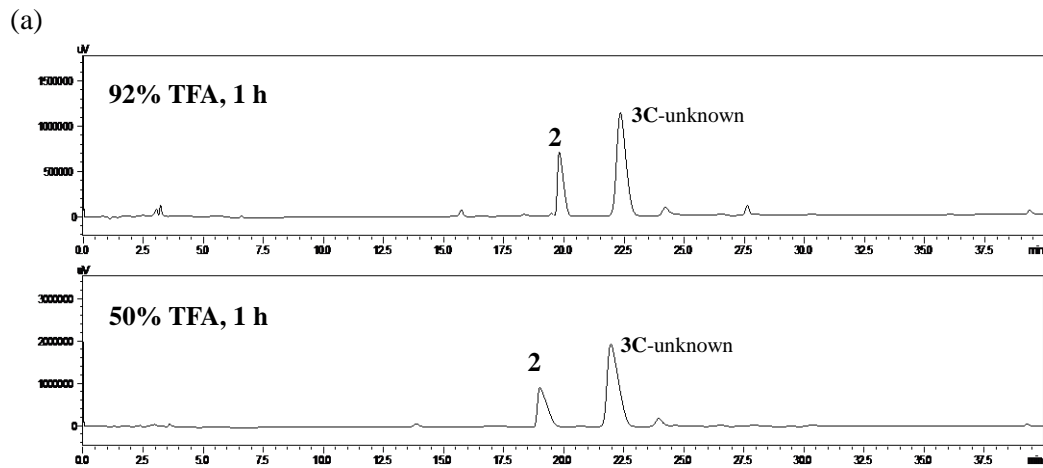
Figure S9. TFA-assisted cleavage of peptoid 5': (a) HPLC chromatogram and (b) MALDI-TOF spectrum.



**Figure S10.** TFA-assisted cleavage of peptoid 5A': (a) HPLC chromatograms and (b) MALDI-TOF spectra.

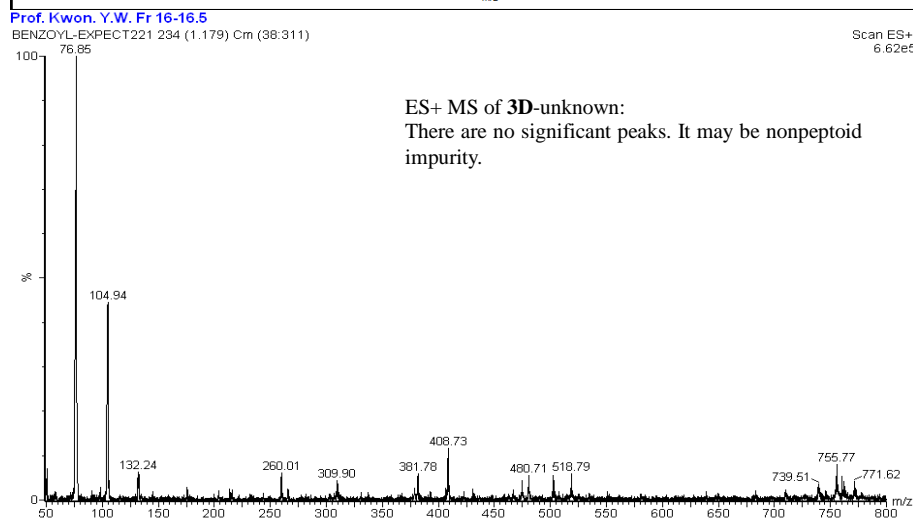
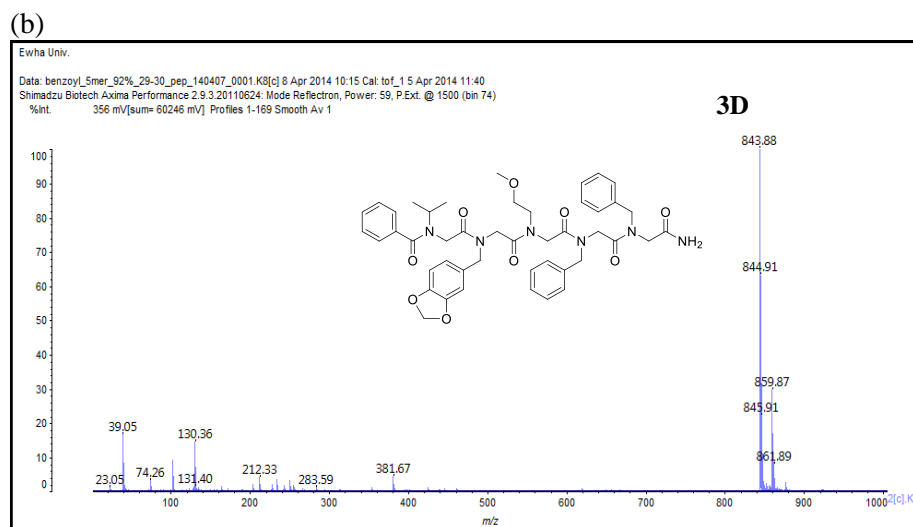
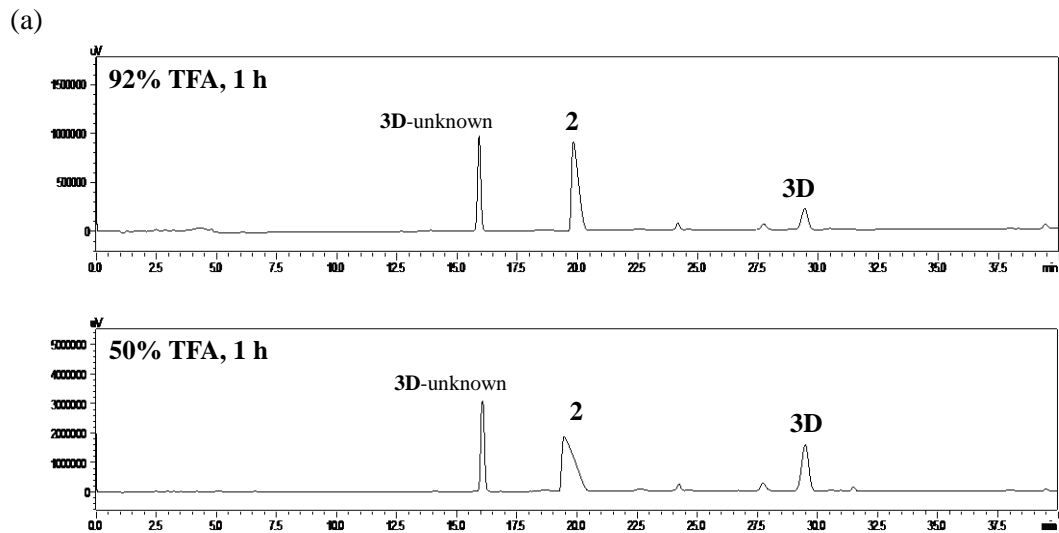


**Figure S11.** TFA-assisted cleavage of peptoid **3B'**: HPLC chromatograms.



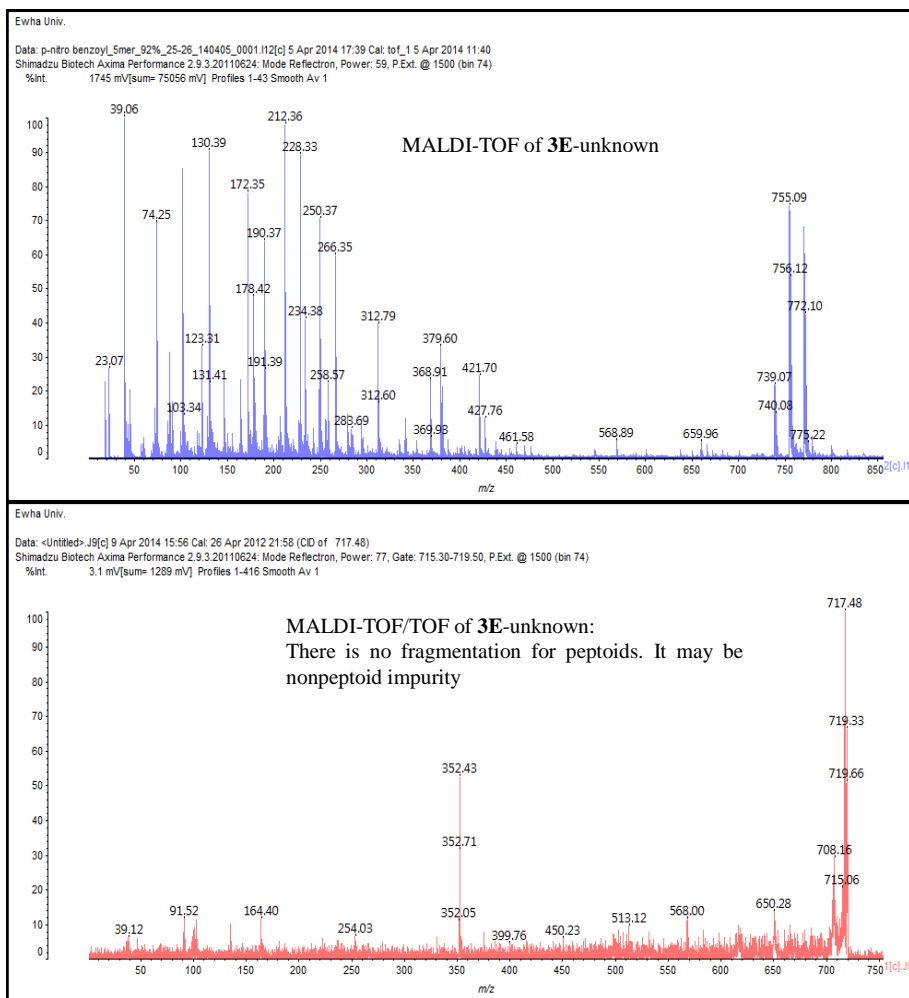
**Figure S12.** TFA-assisted cleavage of peptoid **3C'**: (a) HPLC chromatograms and (b) MALDI-TOF and MALDI-TOF/TOF spectra.





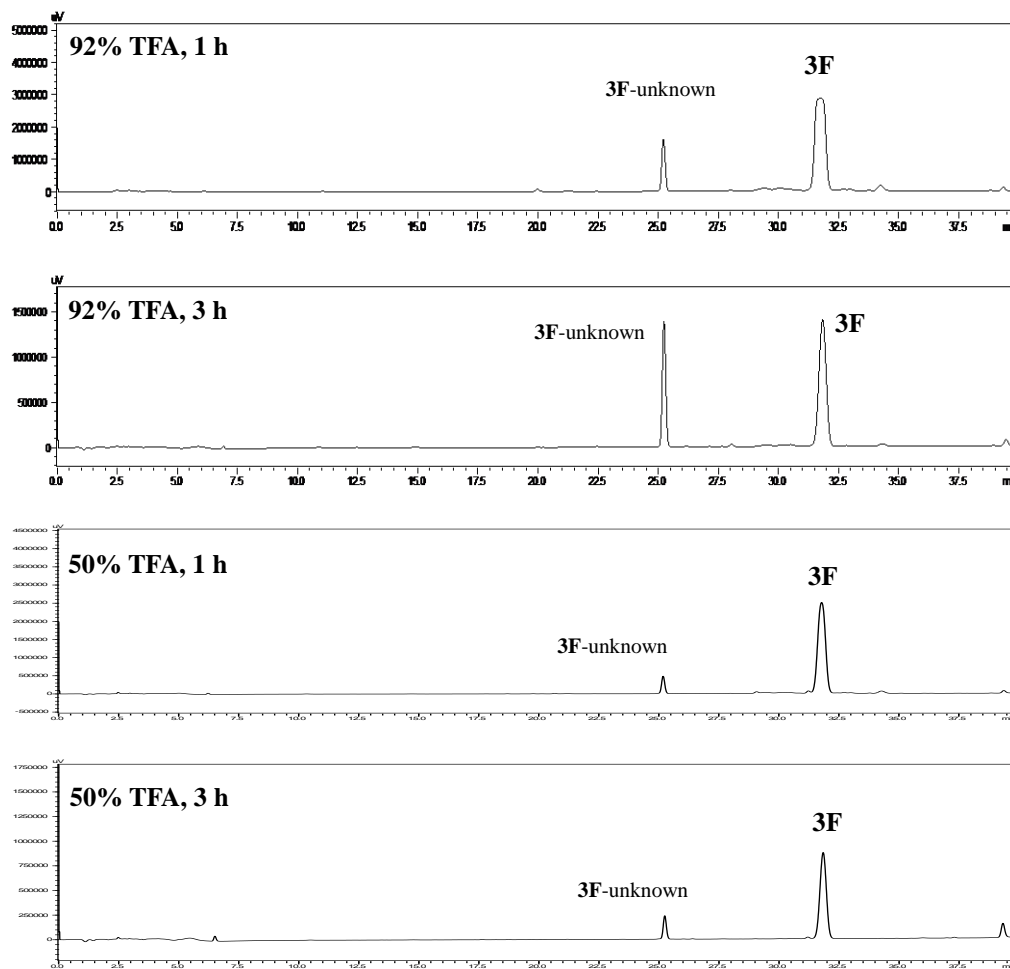
**Figure S13.** TFA-assisted cleavage of peptoid **3D'**: (a) HPLC chromatograms and (b) MALDI-TOF and ES+ spectra.



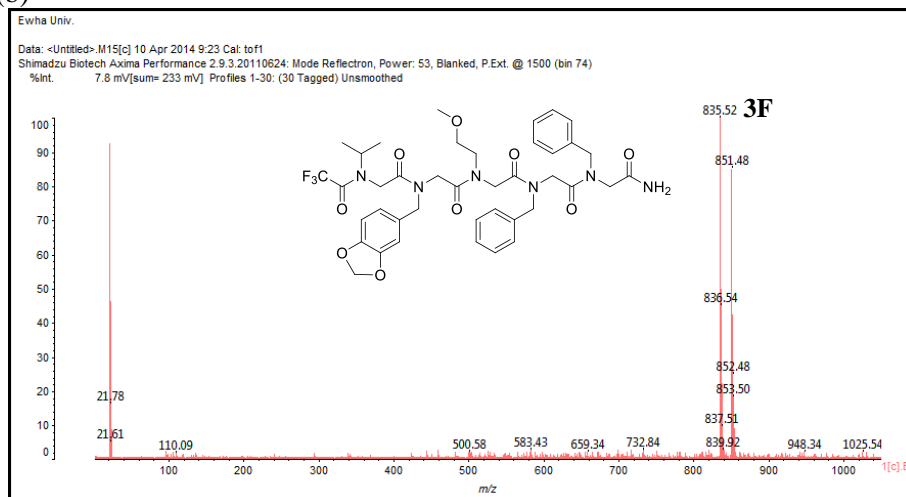


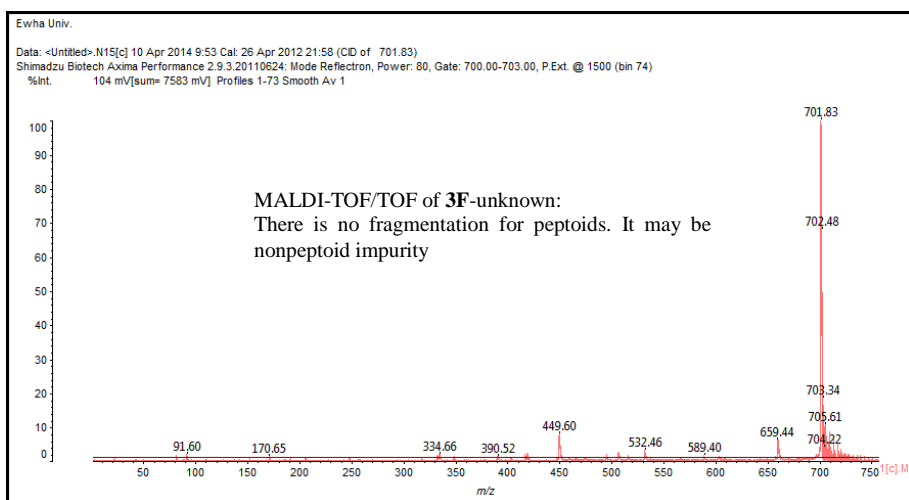
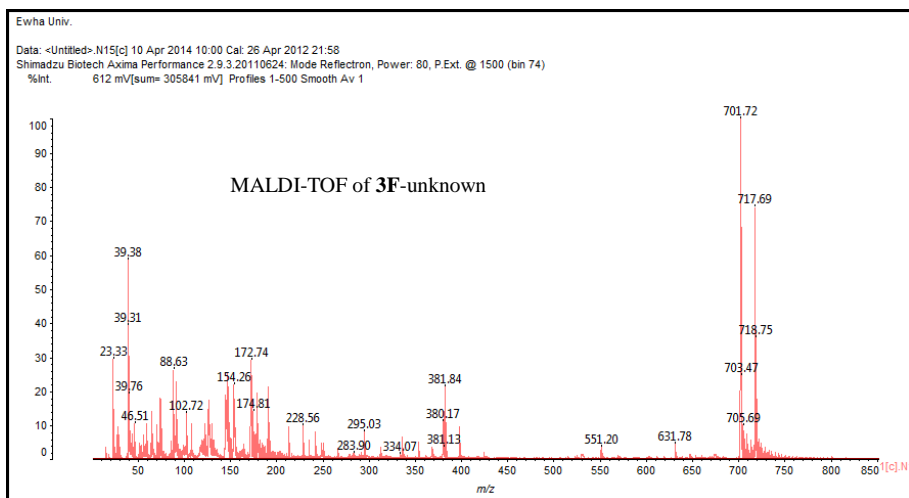
**Figure S14.** TFA-assisted cleavage of peptoid **3E'**: (a) HPLC chromatograms and (b) MALDI-TOF and MALDI-TOF/TOF spectra.

(a)



(b)





**Figure S15.** TFA-assisted cleavage of peptoid **3F'**: (a) HPLC chromatograms and (b) MALDI-TOF and MALDI-TOF/TOF spectra.