Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supplementary Information

Clickity click: highly functionalized peptoid oligomers generated by sequential conjugation reactions on solid phase support

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Characterization of *N*-substituted glycine peptoid oligomers

1. Mass spectrometry of peptoids (LC/MS)

For characterization by mass spectrometry, all compounds were analyzed using an Agilent 1100 Series LC/MSD Trap XCT equipped with an electrospray ion source. All LC/MS experiments were preformed in positive ion mode. Unless otherwise stated, all analyses were performed on peptoids cleaved from resin without further purification.

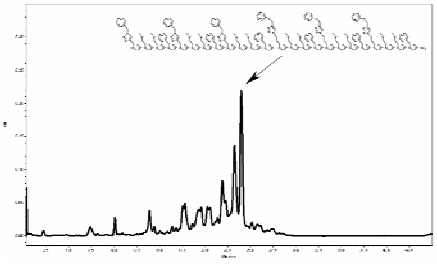
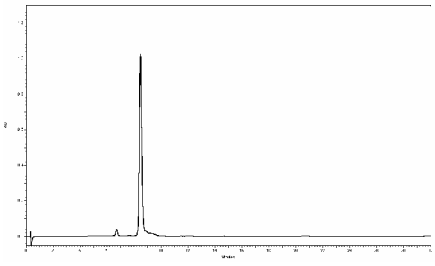
Table S-1.

Entry	Charge	Formula	Calc. m/z	Obs. m/z
1	+1	$C_{74}H_{113}N_{22}O_{18}$	1569.9	1570.4
	+2	$C_{74}H_{114}N_{22}O_{18}$	785.4	785.8
2	+1	$C_{99}H_{133}N_{22}O_{21}$	1966.2	1966.7
	+2	$C_{99}H_{134}N_{22}O_{21}$	984.1	984.1
3	+2	$C_{171}H_{229}N_{34}O_{39}$	1692.9	1692.7
	+3	$C_{171}H_{230}N_{34}O_{39}$	1128.9	1129.0
4	+2	$C_{192}H_{250}N_{43}O_{39}$	1891.4	1892.1
	+3	$C_{192}H_{251}N_{43}O_{39}$	1261.3	1262.1
5	+1	$C_{19}H_{30}N_7O_4$	419.5	420.4
6	+1	$C_{28}H_{38}N_7O_5$	552.3	552.5
7	+1	$C_{47}H_{61}N_{10}O_9$	909.5	909.5
	+2	$C_{47}H_{62}N_{10}O_9$	455.7	453.7
8	+1	$C_{54}H_{68}N_{13}O_9$	1042.5	1042.6
	+2	$C_{54}H_{69}N_{13}O_9$	522.1	521.9
9	+1	$C_{73}H_{94}N_{19}O_{13}$	1444.6	1444.7
	+2	$C_{73}H_{95}N_{19}O_{13}$	723.3	723.2
10	+1	$C_{82}H_{101}FN_{19}O_{13}$	1578.8	1579.5
	+2	$C_{82}H_{102}FN_{19}O_{13}$	790.3	790.4
11	+1	$C_{101}H_{127}FN_{25}O_{17}$	1982.2	1982.2
	+2	$C_{101}H_{128}FN_{25}O_{17}$	991.2	991.7
12	+1	$C_{114}H_{143}FN_{25}O_{17}$	2153.5	2154.2
	+2	$C_{114}H_{144}FN_{25}O_{17}$	1077.6	1077.8
13	+1	$C_{62}H_{90}FeN_{13}O_{12}$	1264.3	1264.9
	+2	$C_{62}H_{91}FeN_{13}O_{12}$	633.2	633.2
14	+1	$C_{42}H_{66}FeN_{13}O_{10}$	967.9	968.3
	+2	$C_{42}H_{67}FeN_{13}O_{10}$	484.9	484.6

2. Reversed-Phase High Performance Liquid Chromatography on Peptoid Oligomers

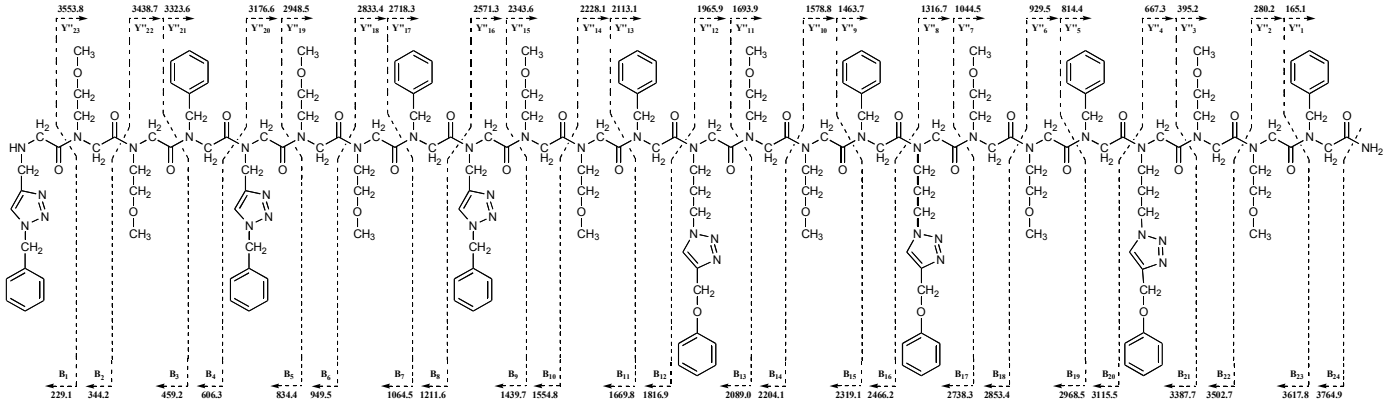
Peptoid oligomers were characterized by analytical Reversed-Phase High Performance Liquid Chromatography (RP-HPLC) using a C4 column as described in the Experimental section. Products were detected by UV absorbance at 214 nm and data were analyzed with Beckman Coulter 32 Karat software version 5.0. Linear gradients were conducted from 5% to 95% solvent B (0.1% TFA in HPLC grade acetonitrile) over solvent A (0.1% TFA in HPLC grade water) with a flow rate of 0.7 mL min^{-1}.

Figure S-1. HPLC traces of selected peptoid oligomers

Compound	RP-HPLC traces	Analytical data
4		Gradient = 5-95% solvent B over solvent A in 32 min. Flow rate = 0.7 mL min^{-1} Retention time = 24.02 min
13		Gradient = 5-95% solvent B over solvent A in 30 min. Flow rate = 0.7 mL min^{-1} Retention time = 8.32 min

3. Sequencing of Peptoid Oligomers by Ion Trap LC/MS/MS

Figure S-2. MS/MS sequencing of crude peptoid **4**. 1261.3 m/z of $(M+2H^{+})(z)^{-1}$ as doubly charged parent molecular ion was trapped and subjected to fragmentation. Upper figure represents calculated masses of ions in sequence from peptoid **4**. Lower figure shows mass spectra containing observed masses of **4** upon MS/MS fragmentation. Target mass was set to 1261.3 m/z to enhance low range mass intensity.



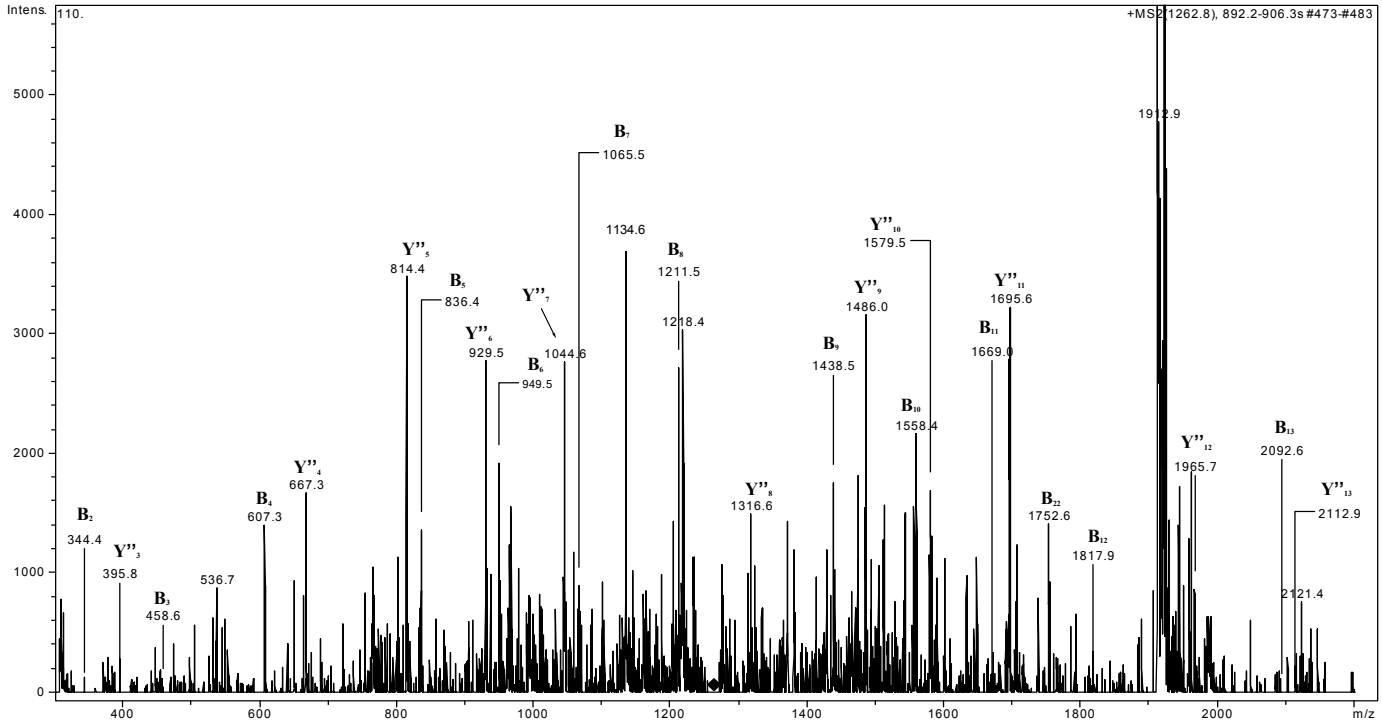
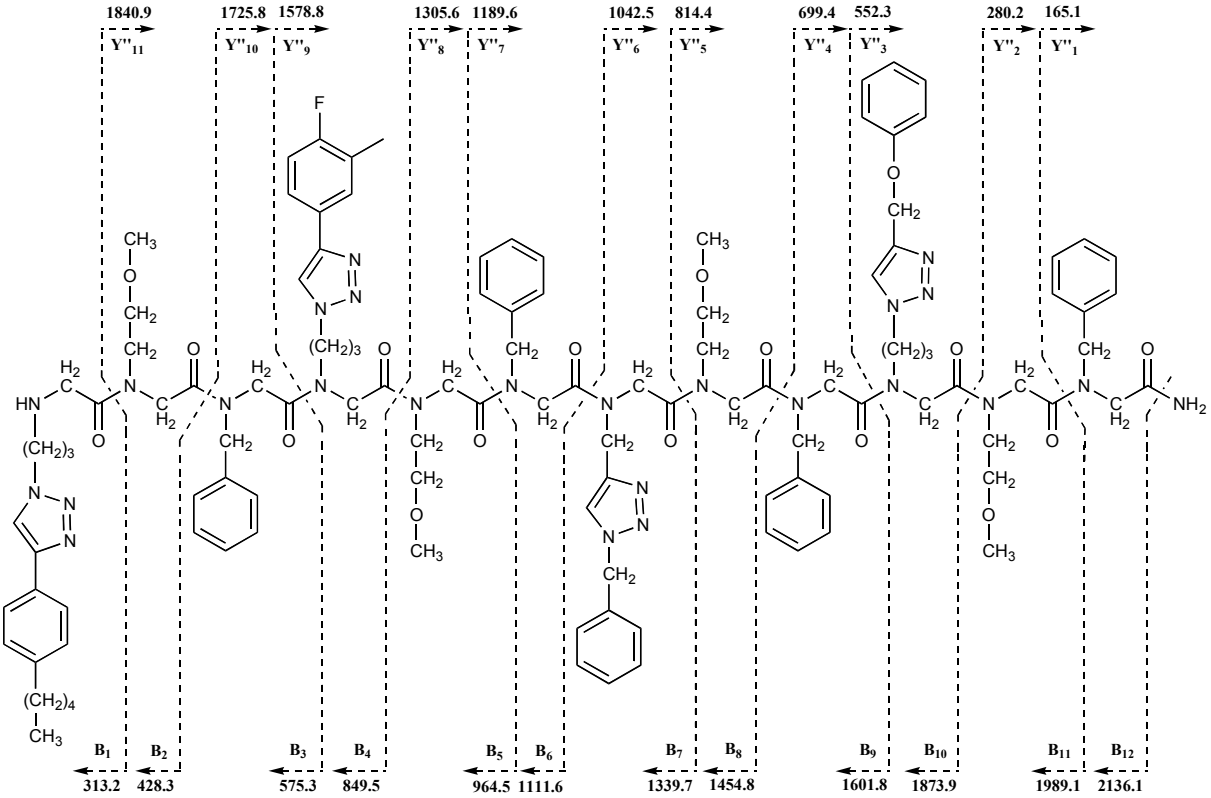
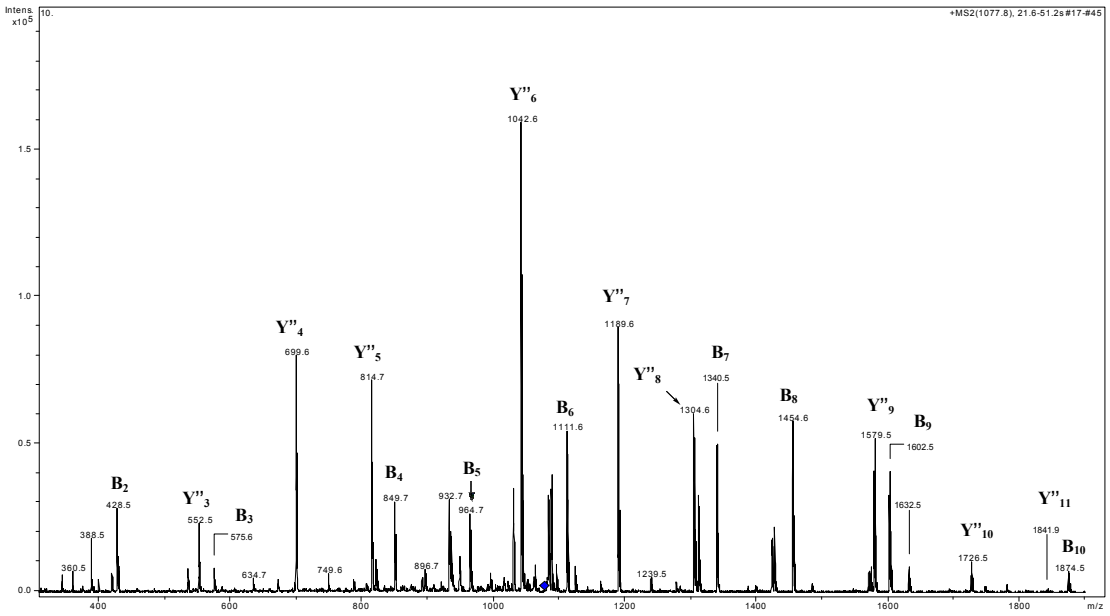


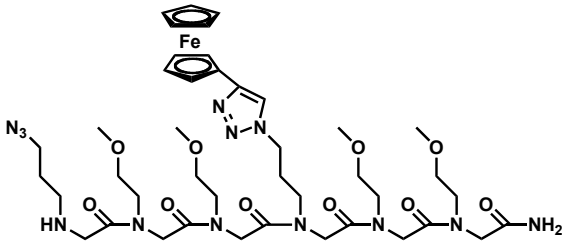
Figure S-3. MS/MS sequencing of crude peptoid **12**. 1078.8 m/z of $(M+2H^{+})(z)^{-1}$ as doubly charged parent molecular ion was trapped and subjected to fragmentation. Upper figure represents calculated masses of ions in sequence from peptoid **12**. Lower figure shows mass spectra containing observed masses of **12** upon MS/MS fragmentation. Target mass was set to 1078.8 m/z to enhance low range mass intensity.





4. Peptoid Redox Sensor

Figure S-4. Peptoid oligomer **14**.



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