Figure S1. ESI-MS spectrum of compound 5

Peaks:
- m/z 249.1, z 1, Abund 104946.4
- m/z 341.1, z 1, Abund 494078.5
- m/z 342.1, z 1, Abund 108131.1
- m/z 359.1, z 1, Abund 109946.5
- m/z 379, z 1, Abund 68316.6
- m/z 381.1, z 1, Abund 870123.6
- m/z 382.1, z 1, Abund 173339.7
- m/z 739.1, z 1, Abund 188025.3
- m/z 740.1, z 1, Abund 91163.2
- m/z 755, z 1, Abund 58995.4

+ESI Scan:1 (0.159-0.505 min, 24 Scans) Frag=135.0V 2595_20141218_TIC.d Subtract
Figure S2. IR spectrum of compound 5 ($c = 5 \times 10^{-2}$M)
Figure S3. $^1$H NMR spectrum of compound 5 ($c = 2.5 \times 10^{-2}$M)
Figure S4. $^{13}$C NMR spectrum of compound 5 ($c = 5 \times 10^{-2}$M)
Figure S5. COSY NMR spectrum of compound 5 ($c = 2.5 \times 10^{-2}$M)
**Compound 7**

*Figure S6.* ESI-MS spectrum of compound 7

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Figure S7. IR spectrum of compound 7 (c = 5 x 10^{-2} M)
Figure S8. $^1$H NMR spectrum of compound 7 ($c = 2.5 \times 10^{-2}$M)
Figure S9. $^{13}$C NMR spectrum of compound 7 ($c = 5 \times 10^{-2}$M)
Figure S10. COSY NMR spectrum of compound 7 ($c = 2.5 \times 10^{-2}$M)
Figure S11. NOESY NMR spectrum of compound 7 ($c = 2.5 \times 10^{-2}$M)
Compound 3b

*Figure S12. HRMS spectrum of compound 3b*
**Figure S13.** LC-MS assessment of purity of 3b (97%) dissolved in MeOH. Elution conditions: 30-70% gradient elution system with H2O + 0.1% FA (solvent A) and MeOH + 0.1% FA (solvent B) over 20 min at 0.5 mL/min.

Column used: Zorbax C18 XDB 3.5 um, 4.6x75 mm (Agilent, Palo Alto, CA, USA).
Figure S14. Concentration-dependent IR spectra of compound 3b
Figure S15. $^1$H NMR spectrum of compound 3b ($c = 5 \times 10^{-2}$M)
Figure S16. $^{13}$C NMR spectrum of compound 3b ($c = 5 \times 10^{-2}$M)
Figure S17. Concentration-dependent NH chemical shifts of compound 3b
Figure S18. Temperature-dependent NH chemical shifts of compound 3b ($c = 2.5 \times 10^{-2}$M)
Figure S19. Solvent dependence of NH chemical shifts of compound 3b at varying concentrations of $d_6$-DMSO in CDCl$_3$ ($c = 2.5 \times 10^{-2}$M)
Figure S20. COSY NMR spectrum of compound 3b (c = 5 × 10^{-2} M)
Figure S21. NOESY NMR spectrum of compound 3b ($c = 5 \times 10^{-2} \text{M}$)
Figure S22. HRMS spectrum of compound 3c
Figure S23. LC-MS assessment of purity of 3c (95%) dissolved in MeOH. Elution conditions: 30-70% gradient elution system with H2O + 0.1% FA (solvent A) and MeOH + 0.1% FA (solvent B) over 20 min at 0.5 mL/min. Column used: Zorbax C18 XDB 3.5 um, 4.6x75 mm (Agilent, palo Alto, CA, USA).
Figure S24. Concentration-dependent IR spectra of compound 3c
Figure S25. $^1$H NMR spectrum of compound 3c ($c = 5 \times 10^{-2}$M)
Figure S26. $^1$H NMR spectrum of compound 3c ($c = 2.5 \times 10^{-2}$M)
Figure S27. $^{13}$C NMR spectrum of compound 3c ($c = 5 \times 10^{-2}$M)
Figure S28. $^{13}$C NMR spectrum of compound 3c ($c = 2.5 \times 10^{-2} \text{M}$)
Figure S29. Concentration-dependent NH chemical shifts of compound 3c
Figure S30. Temperature-dependent NH chemical shifts of compound 3c (c = 2.5 × 10^{-2}M)
Figure S31. Solvent dependence of NH chemical shifts of compound 3c at varying concentrations of $d_6$-DMSO in CDCl$_3$ ($c = 2.5 \times 10^{-2}$M)
Figure S32. COSY NMR spectrum of compound 3c ($c = 5 \times 10^{-2}$M)
Figure S33. NOESY NMR spectrum of compound 3c (c = 5 × 10^{-2} M)