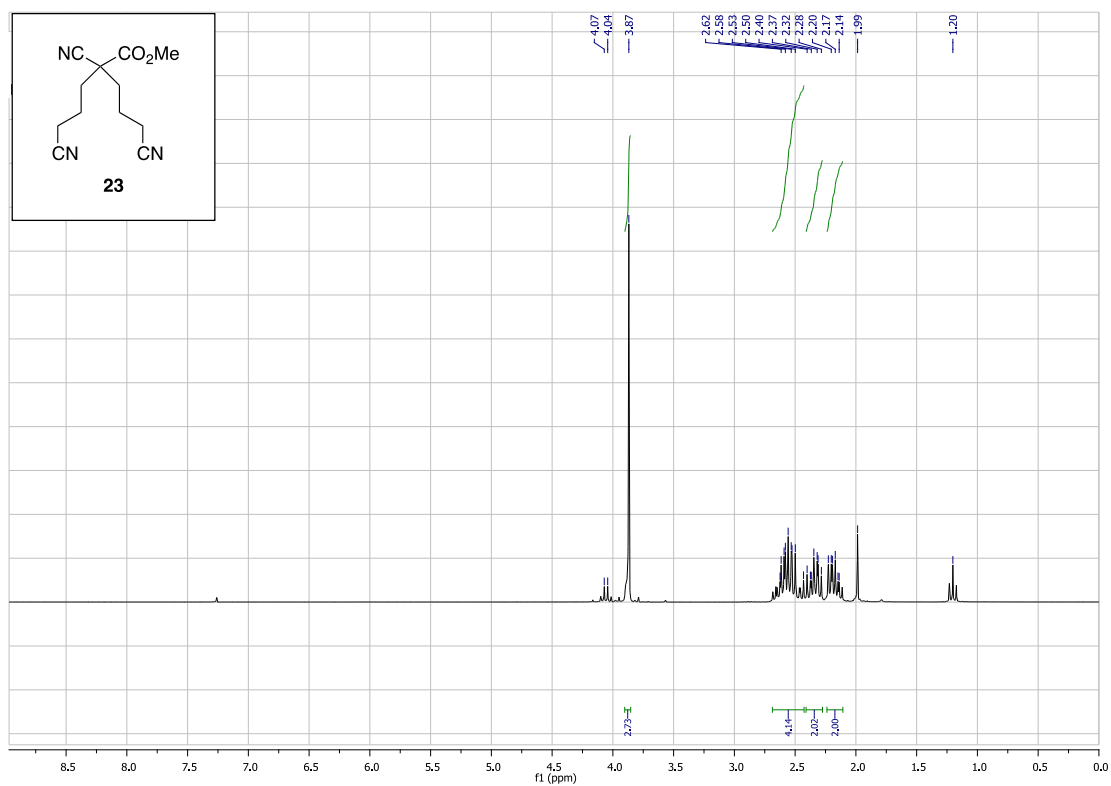
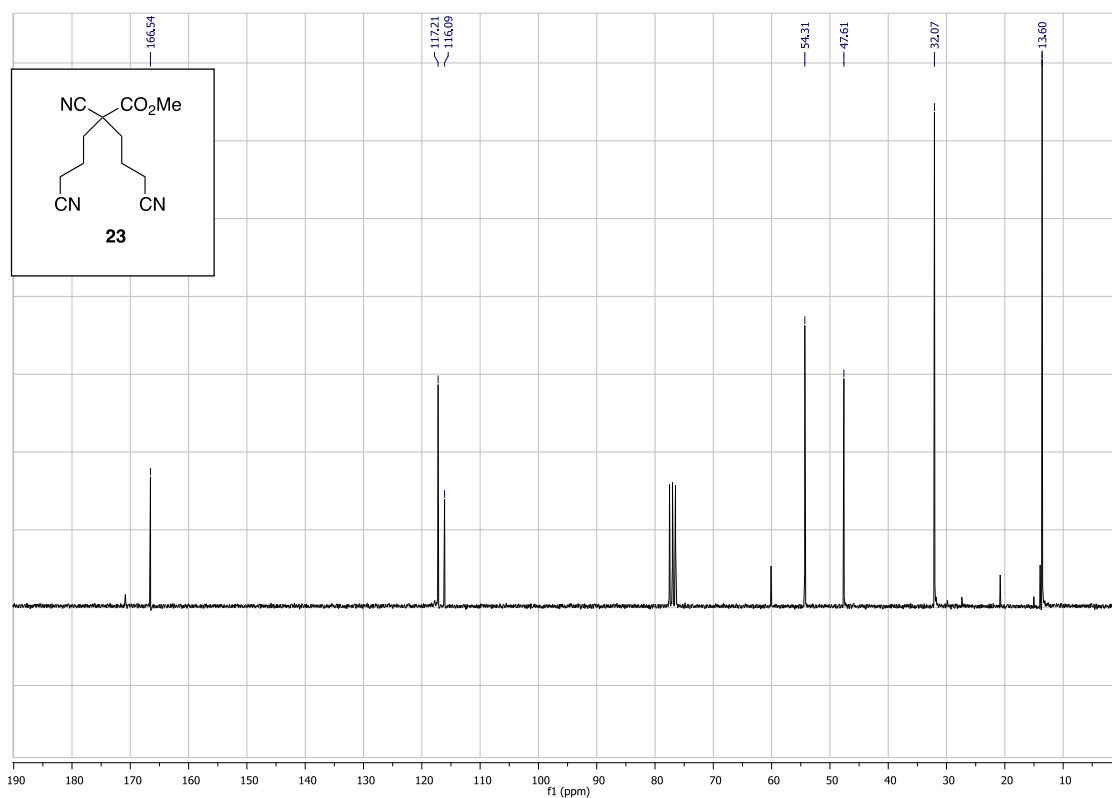


^1H and ^{13}C NMR spectra

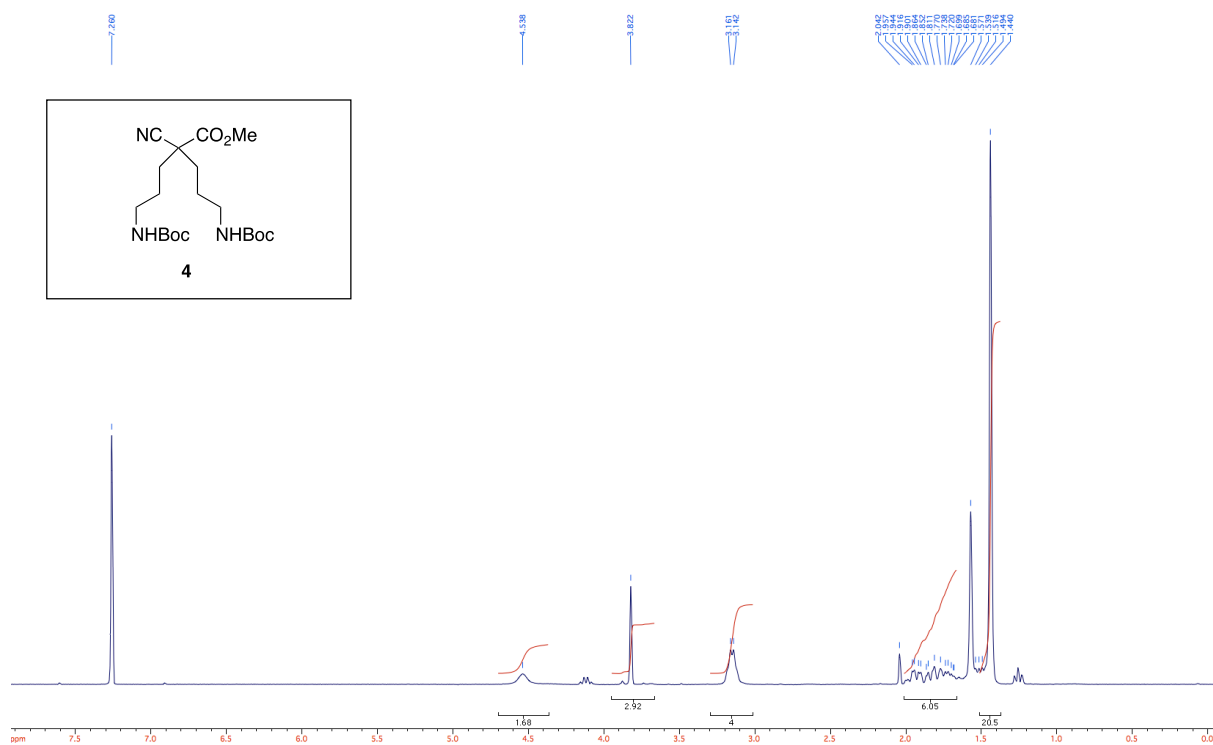
^1H NMR spectra of methyl 2,4-dicyano-2-(2-cyanoethyl)butanoate **23**, 250 MHz, CDCl_3



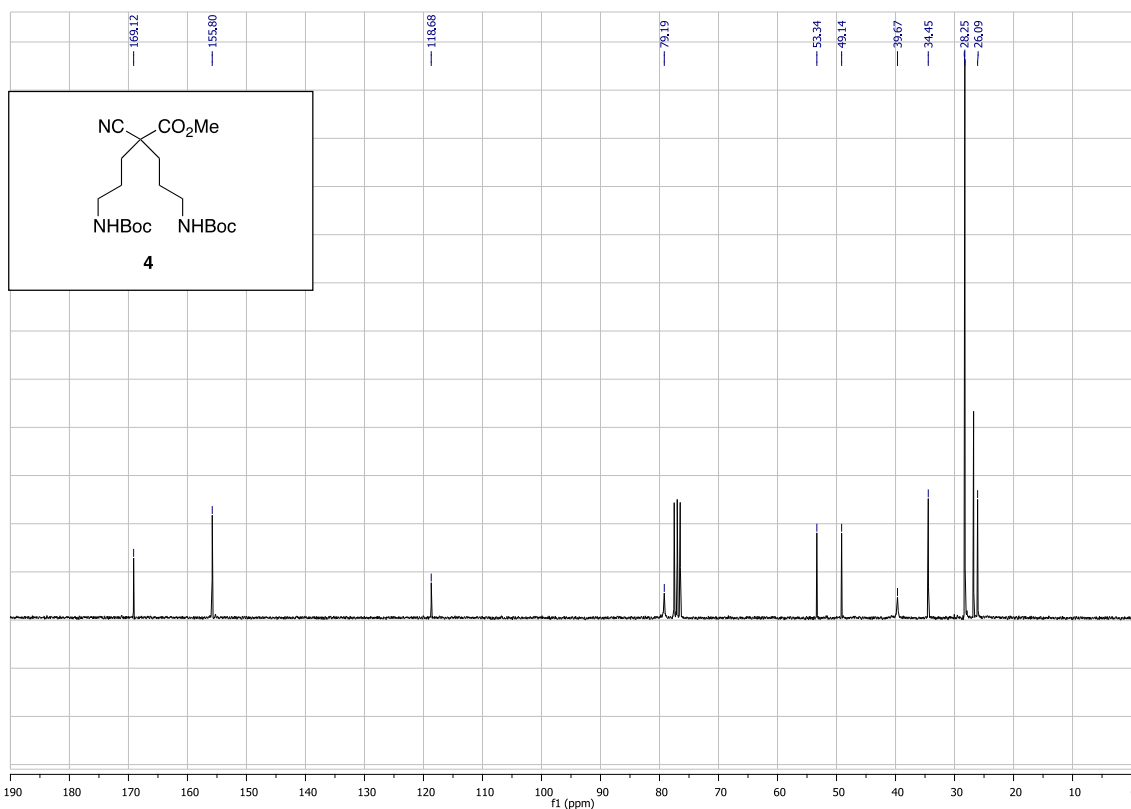
¹³C NMR spectra of methyl 2,4-dicyano-2-(2-cyanoethyl)butanoate **23**, 75 MHz, CDCl₃



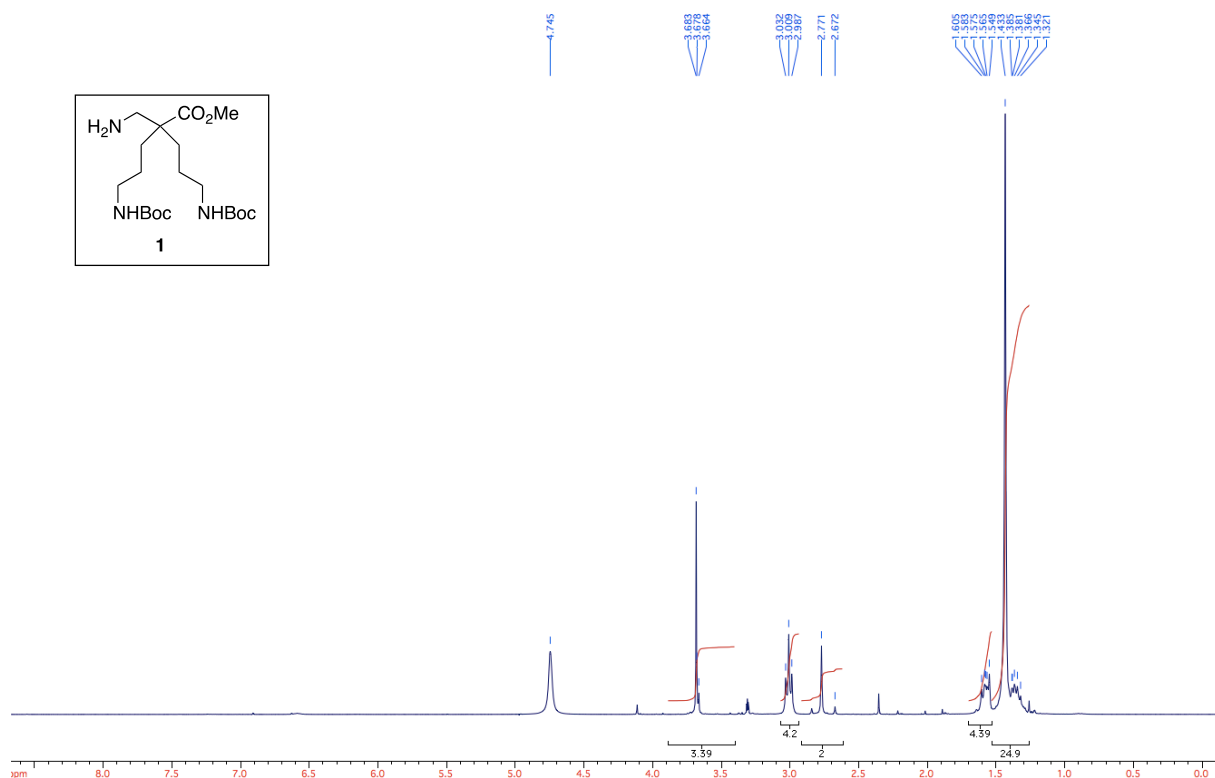
¹H NMR spectra of methyl 2-cyano-4-(Boc)amine-2-(3-(Boc)amine propyl)pentanoate **4**, 300 MHz, CDCl₃



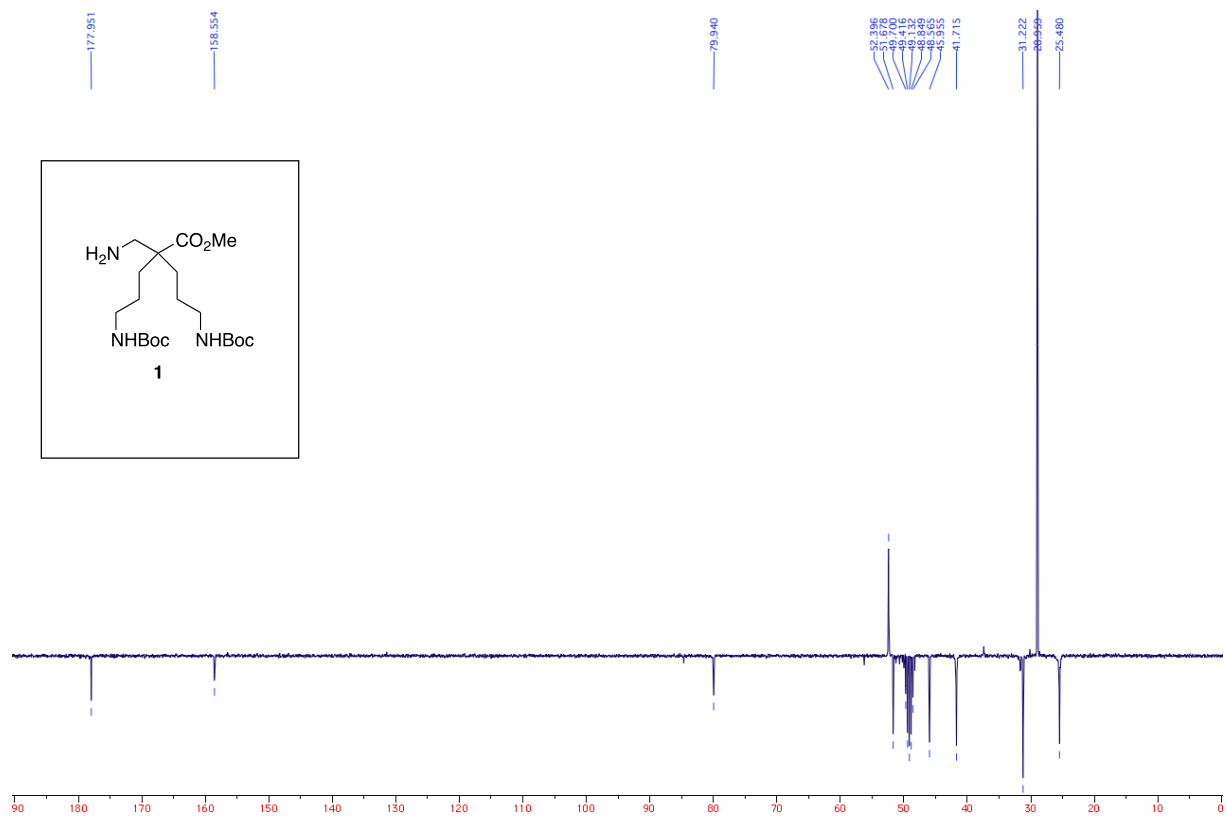
¹³C NMR spectra of methyl 2-cyano-4-(Boc)amine-2-(3-(Boc)amine propyl)pentanoate 4, 75 MHz, CDCl₃



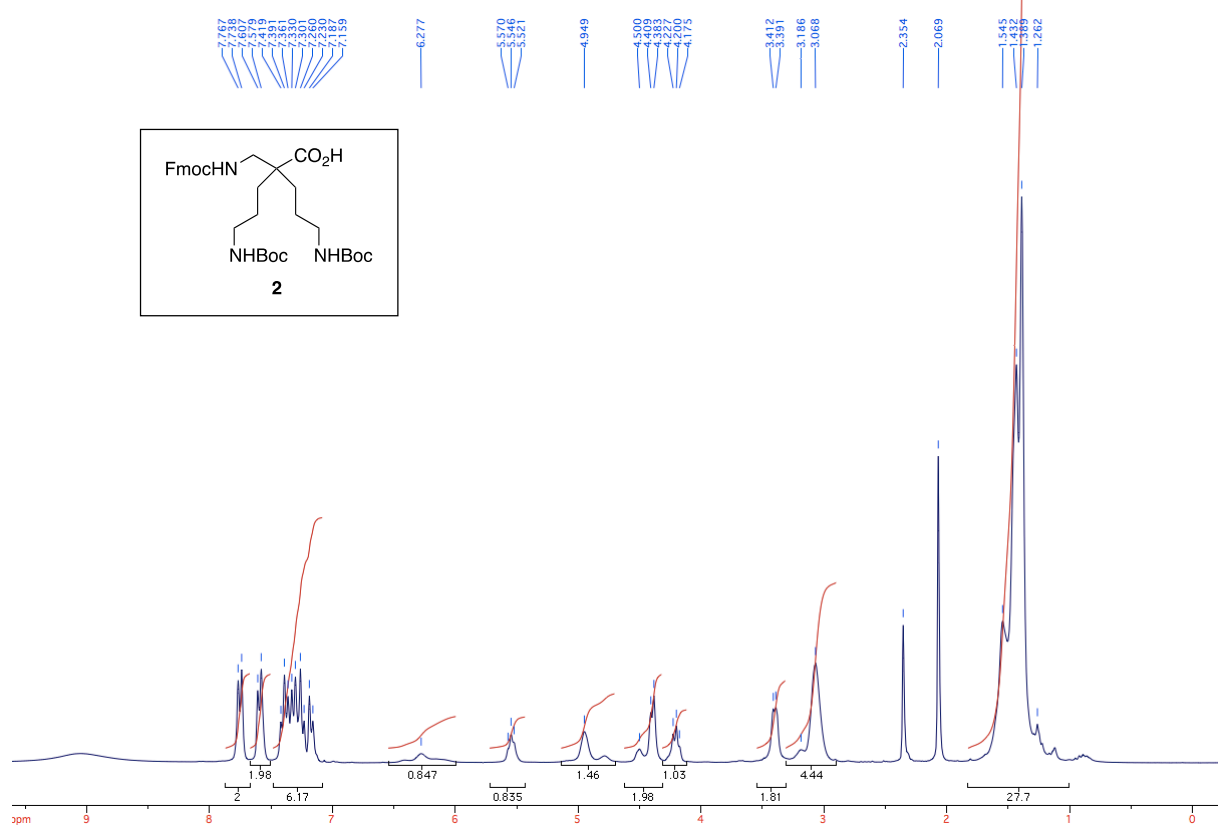
¹H NMR spectra of H-β^{2,2}h bis-Orn(Boc)₂OMe 1, 300 MHz, CD₃OD



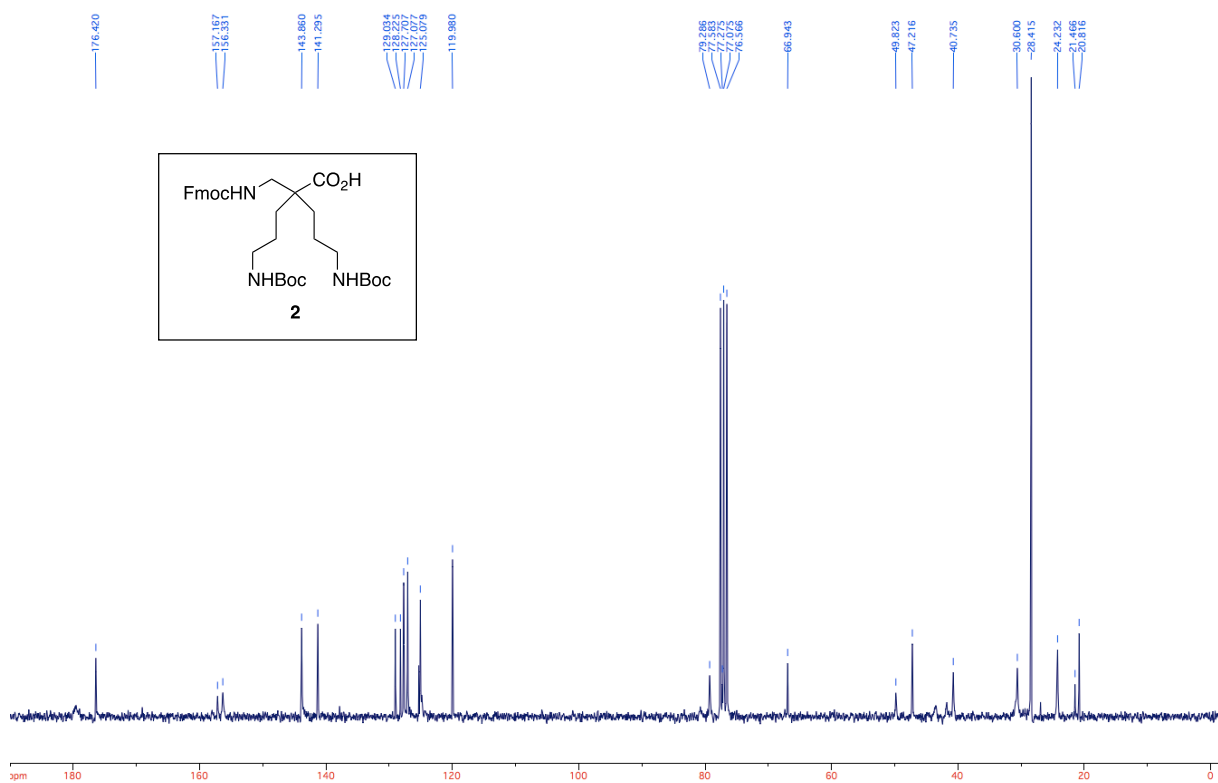
¹³C NMR spectra of H-β^{2,2}h bis-Orn(Boc)₂OMe 1, 75 MHz, CD₃OD



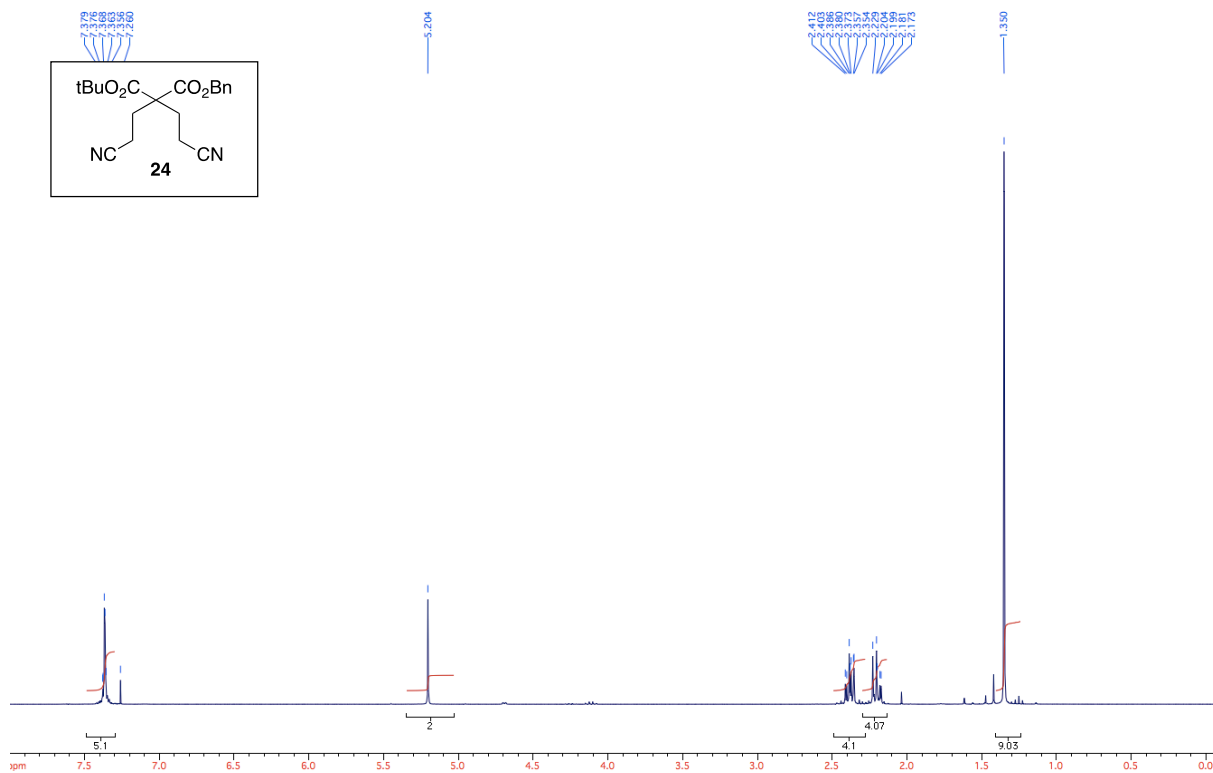
¹H NMR spectra of Fmoc β^{2,2}h bis-Orn(Boc)₂OH 2, 250 MHz, CDCl₃



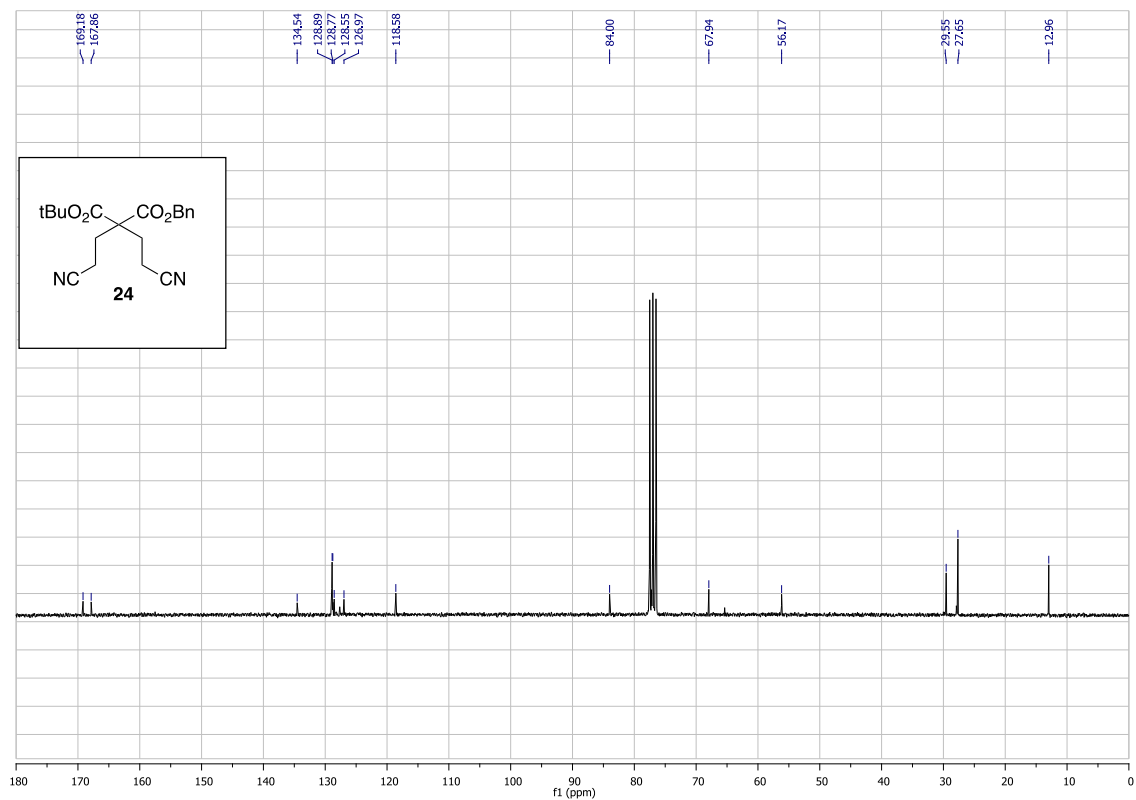
¹³C NMR spectra of Fmoc β^{2,2}h bis-Orn(Boc)₂OH 2, 75 MHz, CDCl₃



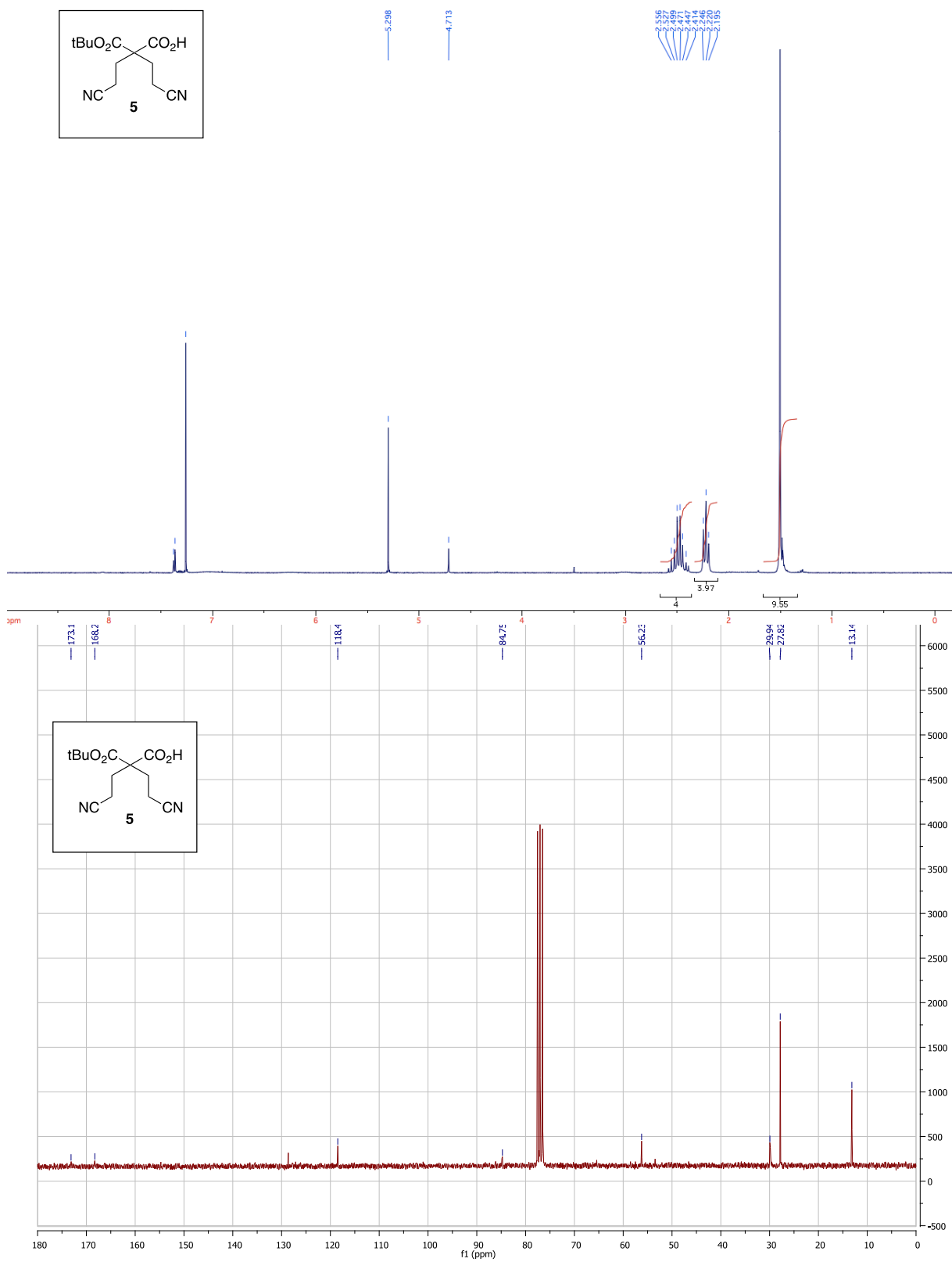
¹H NMR spectra of 1-benzyl 3-tert-butyl 2,2-bis(2-cyanoethyl)malonate 24, 250 MHz, CDCl₃



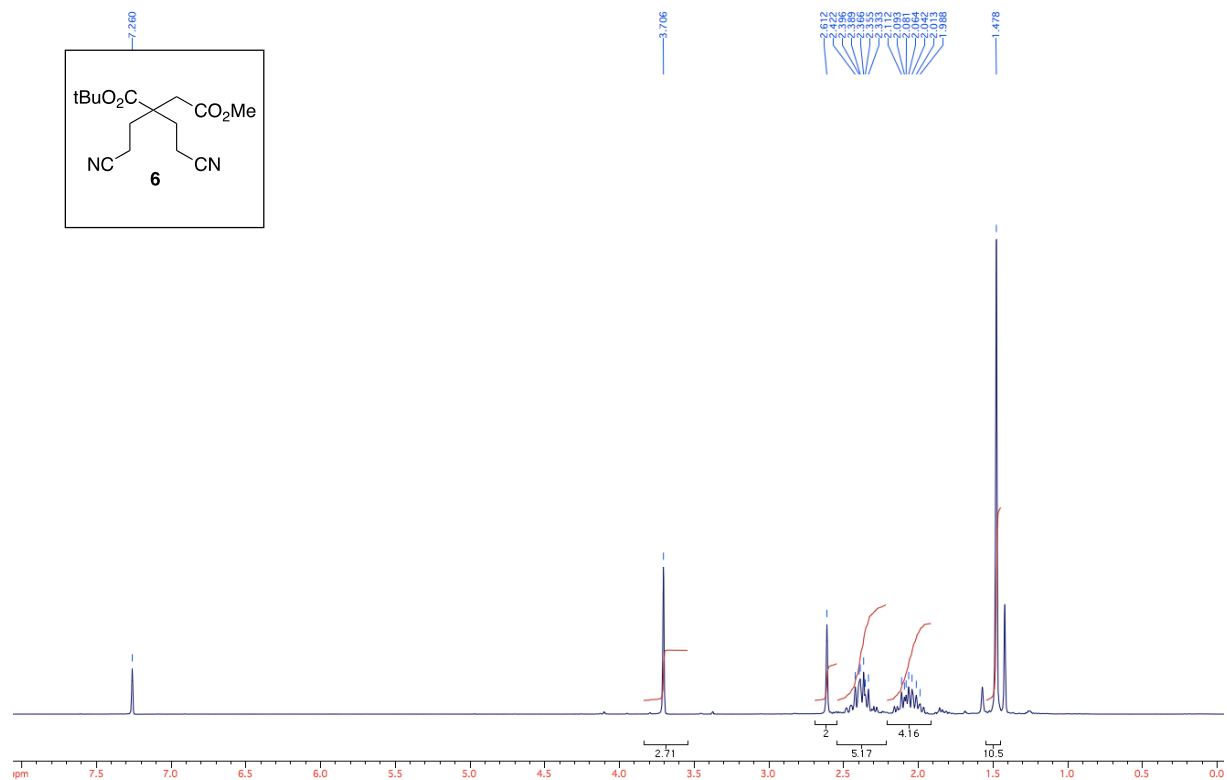
¹³C NMR spectra of 1-benzyl 3-*tert*-butyl 2,2-bis(2-cyanoethyl)malonate **24**, 62.5 MHz, CDCl₃



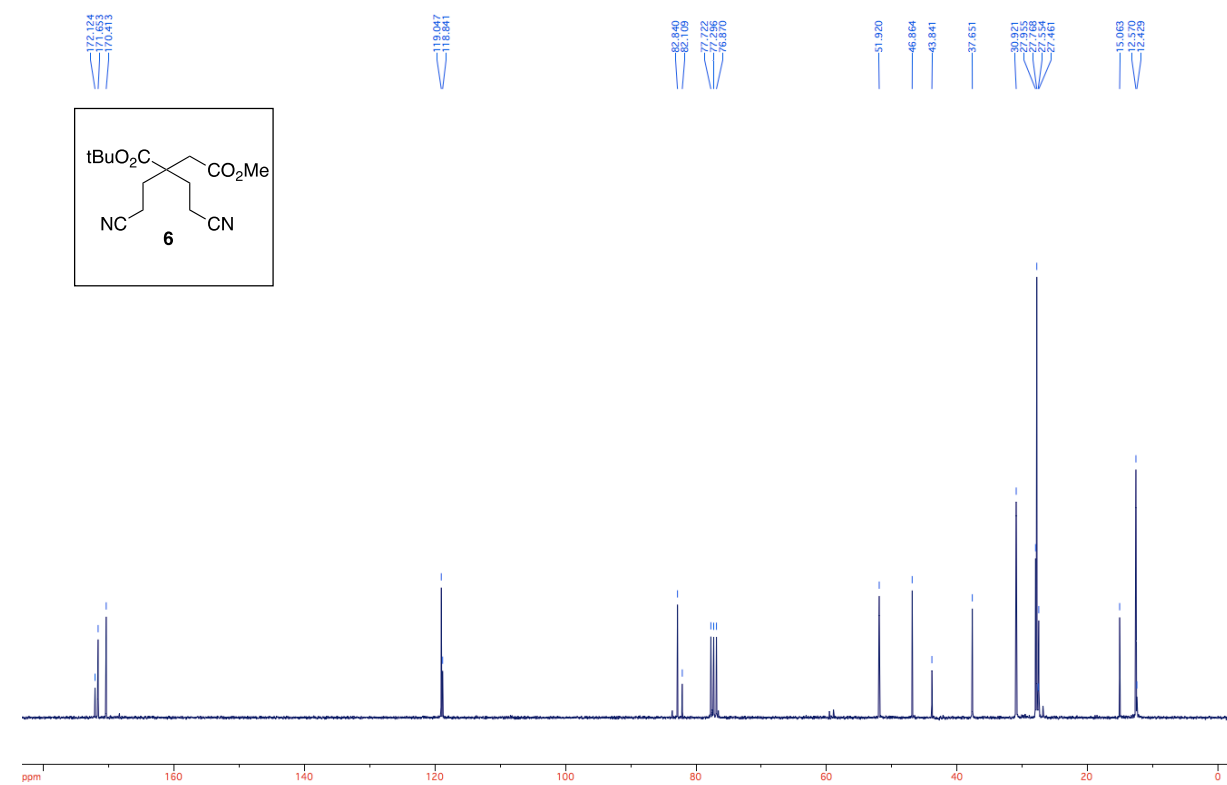
¹H NMR spectra of 2-(tert-butoxycarbonyl)-4-cyano-2-(2-cyanoethyl)butanoic acid **5**, 300 MHz, CDCl₃



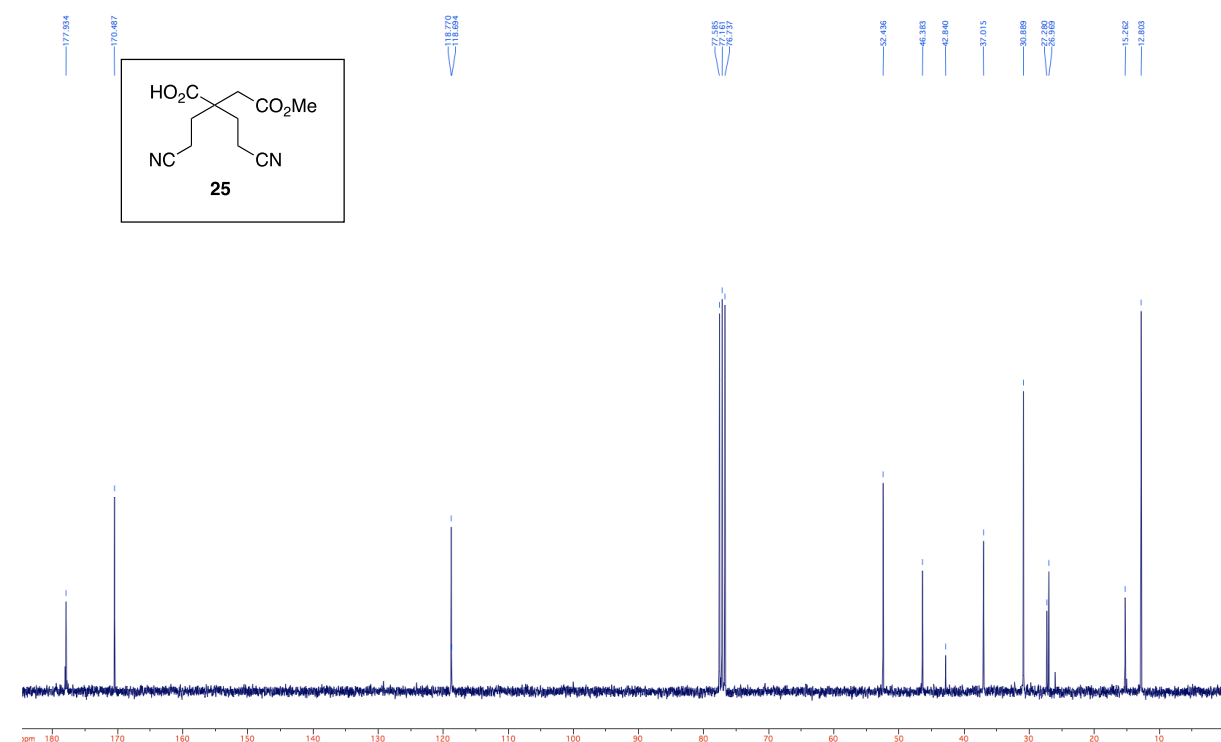
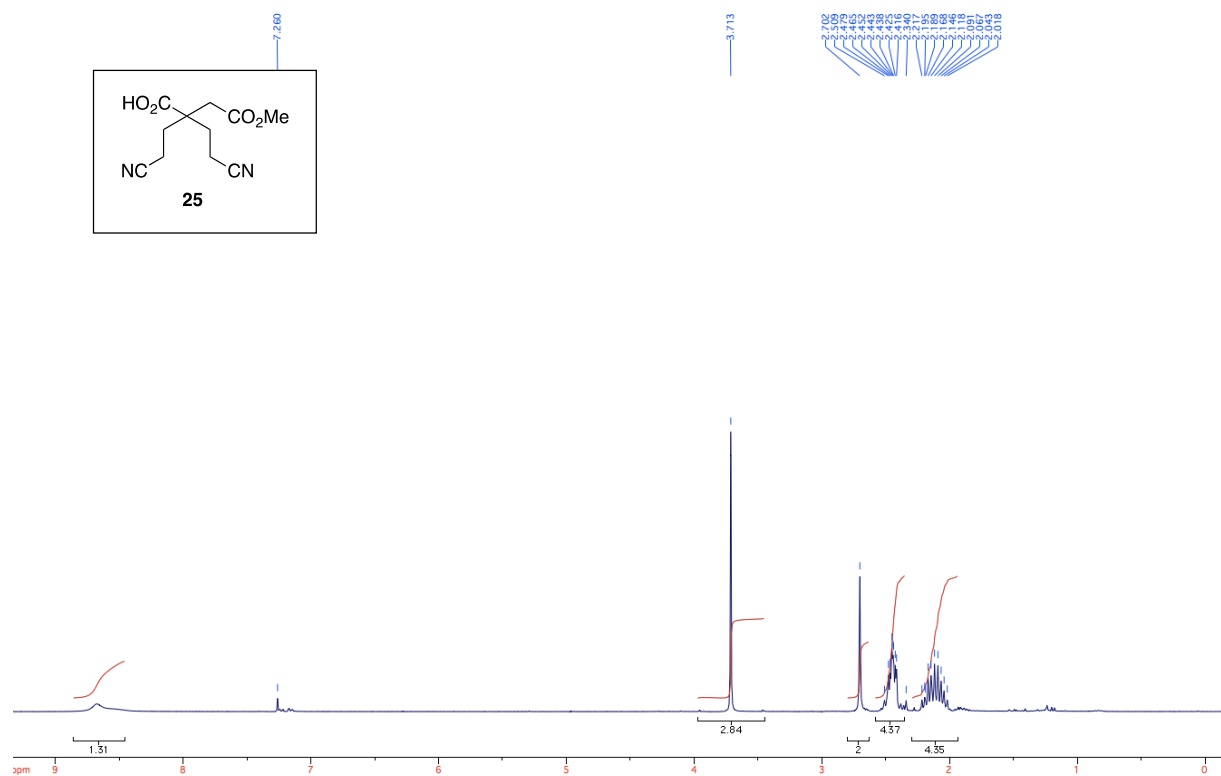
¹H NMR spectra of 1-tert-butyl 4-methyl 2,2-bis(2-cyanoethyl)succinate 6, 300 MHz, CDCl₃



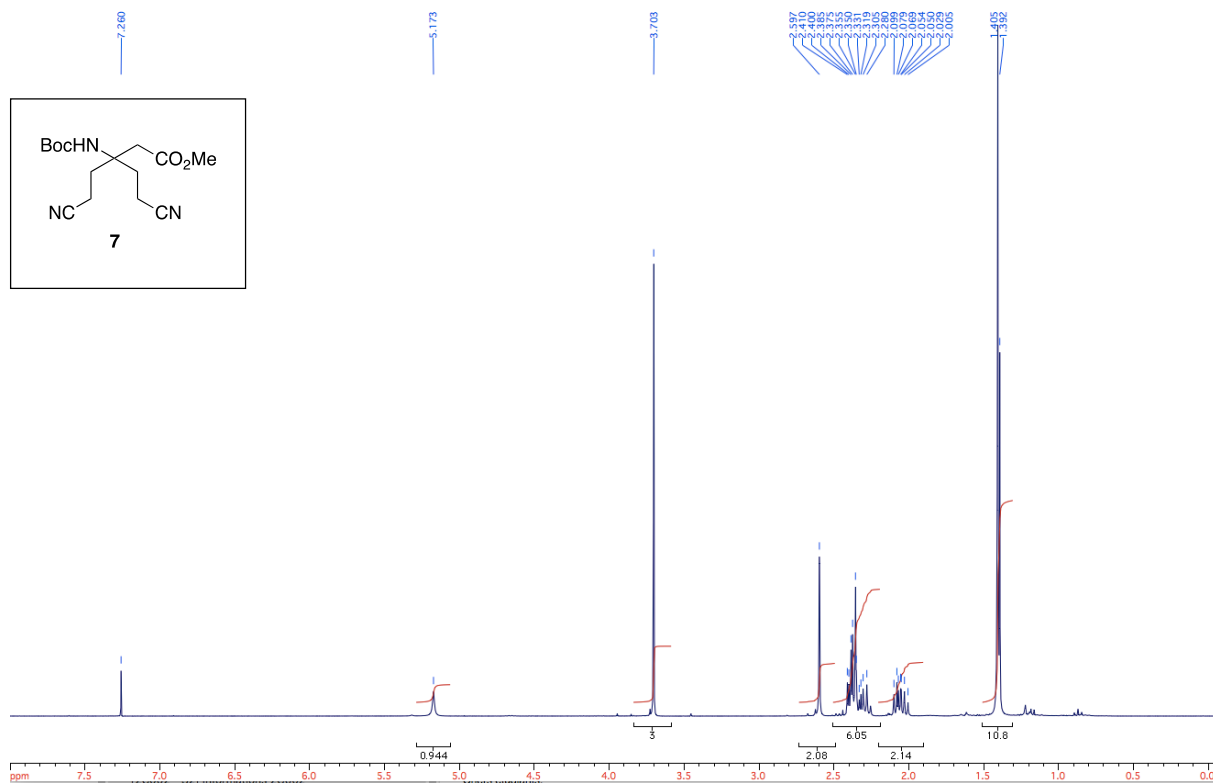
¹³C NMR spectra of 1-tert-butyl 4-methyl 2,2-bis(2-cyanoethyl)succinate 6, 75 MHz, CDCl₃



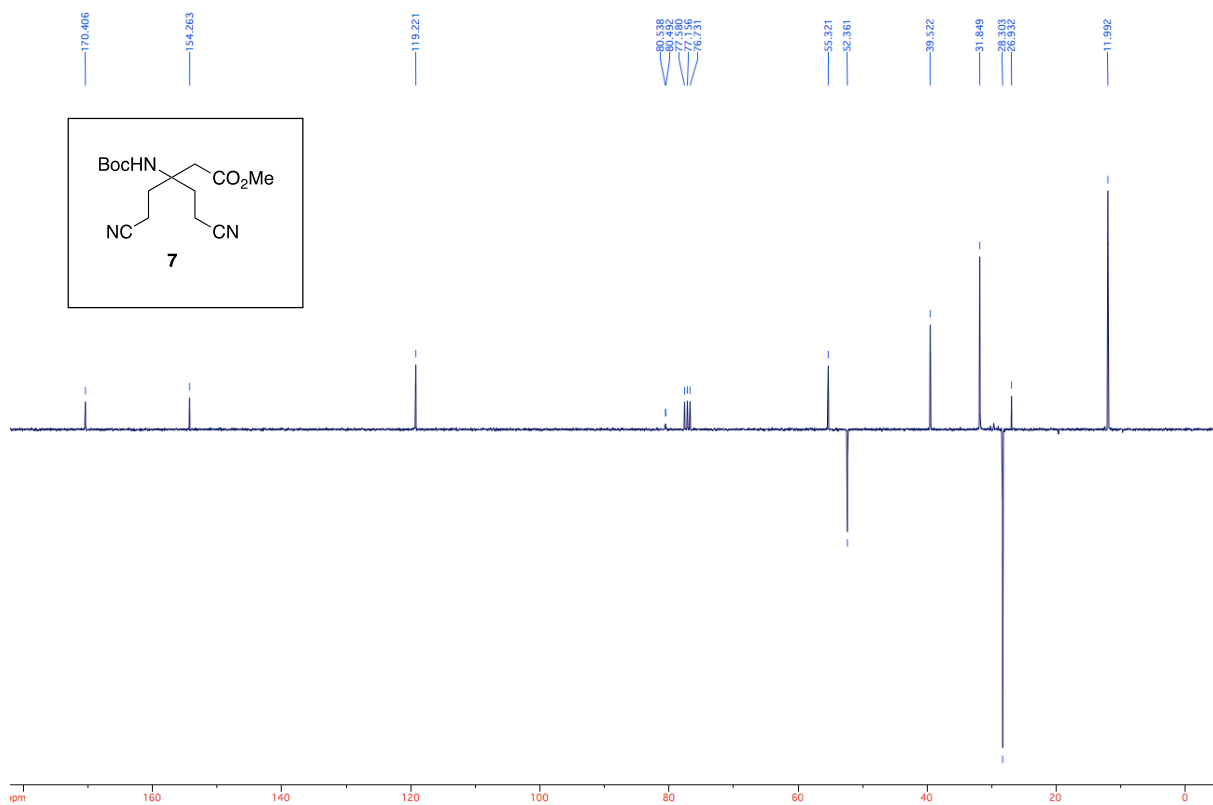
¹H NMR spectra of 2,2-bis(2-cyanoethyl)-4-methoxy-4-oxobutanoic acid **25**, 300 MHz, CDCl₃



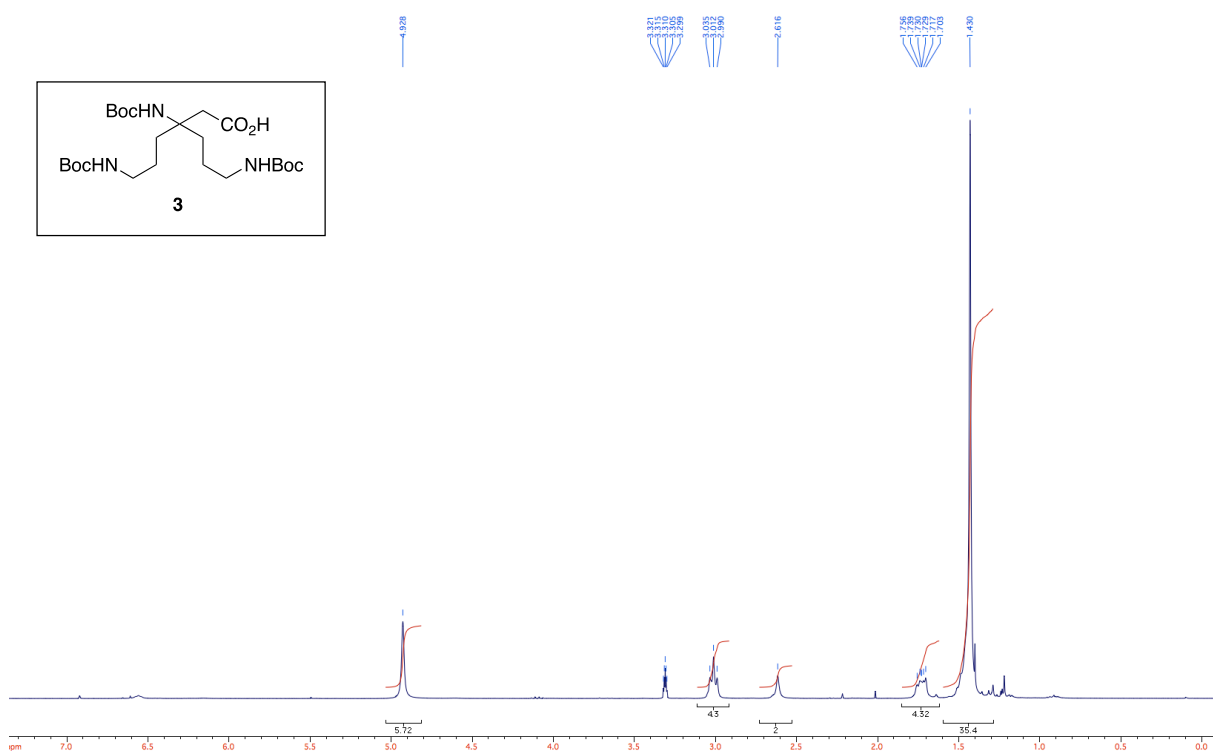
¹H NMR spectra of methyl 3-((tert-butoxycarbonyl)amino)-5-cyano-3-(2-cyanoethyl)pentanoate 7, 300 MHz, CDCl₃



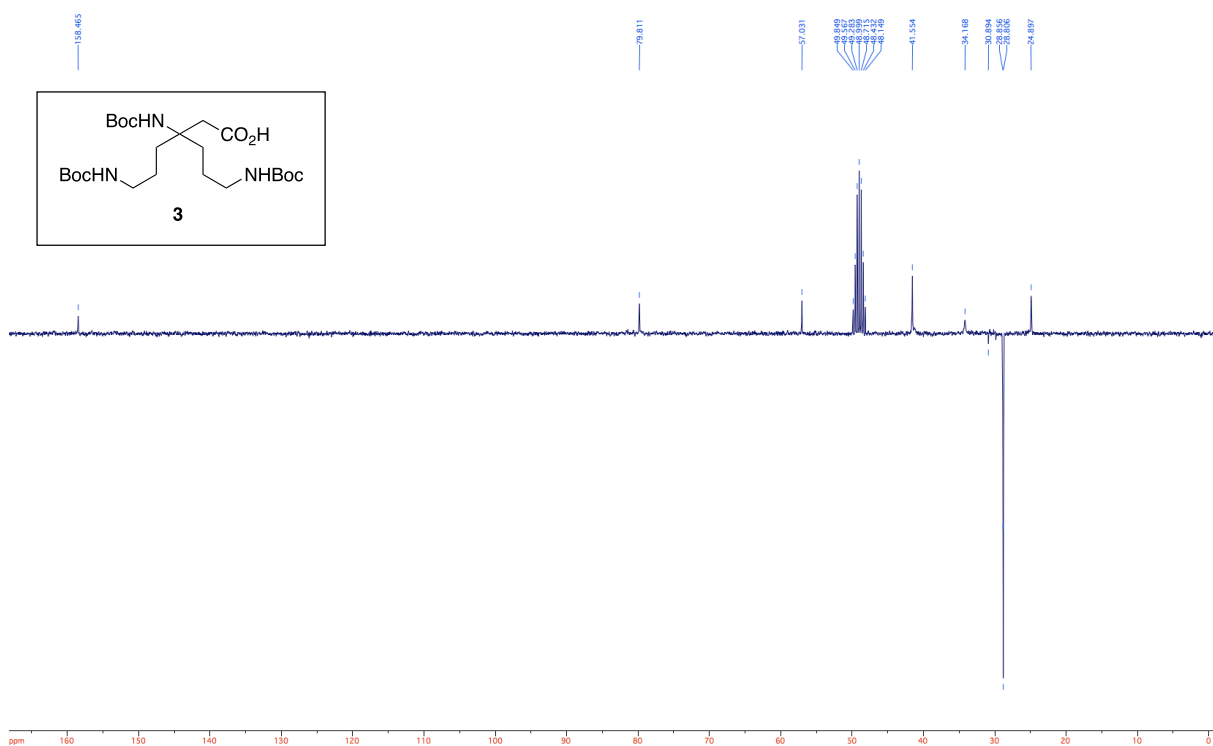
¹³C NMR spectra of methyl 3-((tert-butoxycarbonyl)amino)-5-cyano-3-(2-cyanoethyl)pentanoate 7, 75 MHz, CDCl₃



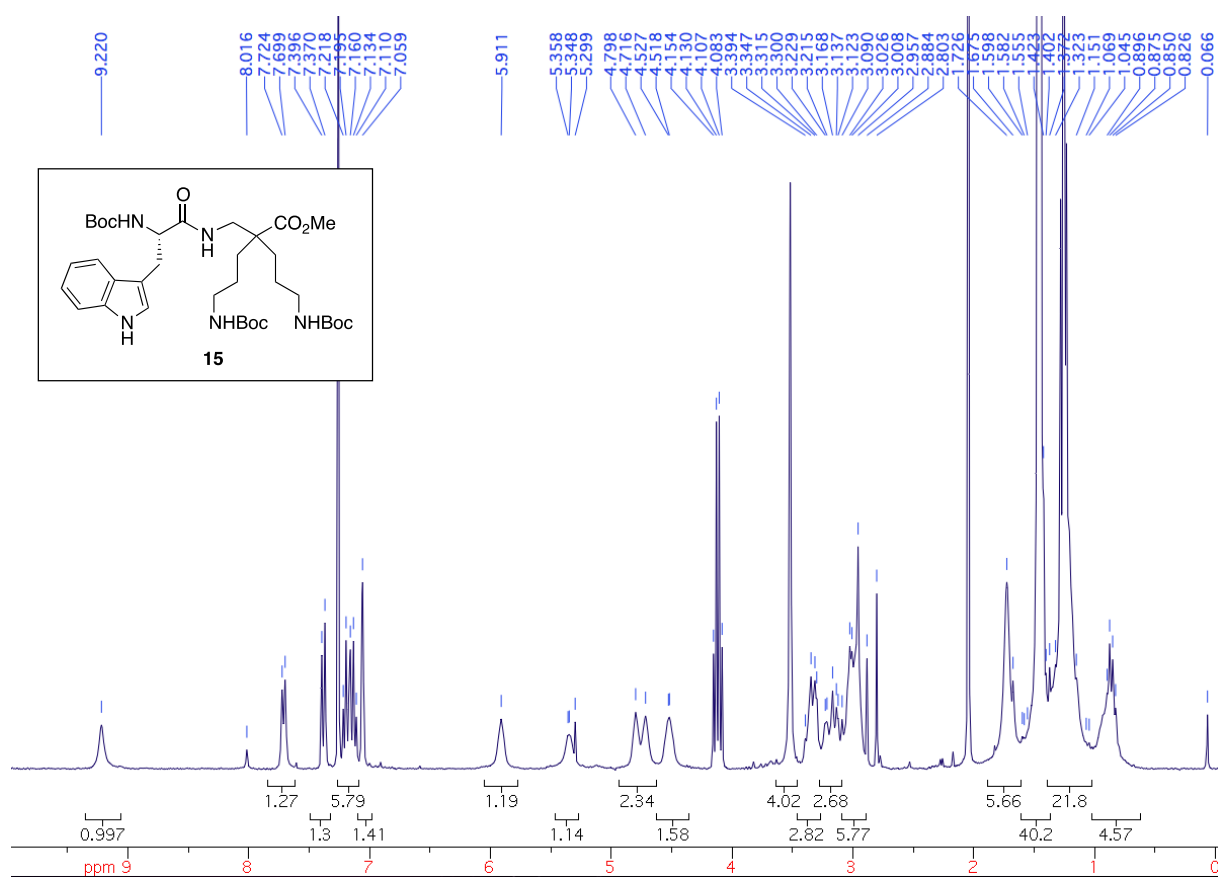
^1H NMR spectra of Boc- $\beta^{3,3}$ *h* bis-Orn(Boc) $_2$ OH 3, 300 MHz, CD $_3$ OD



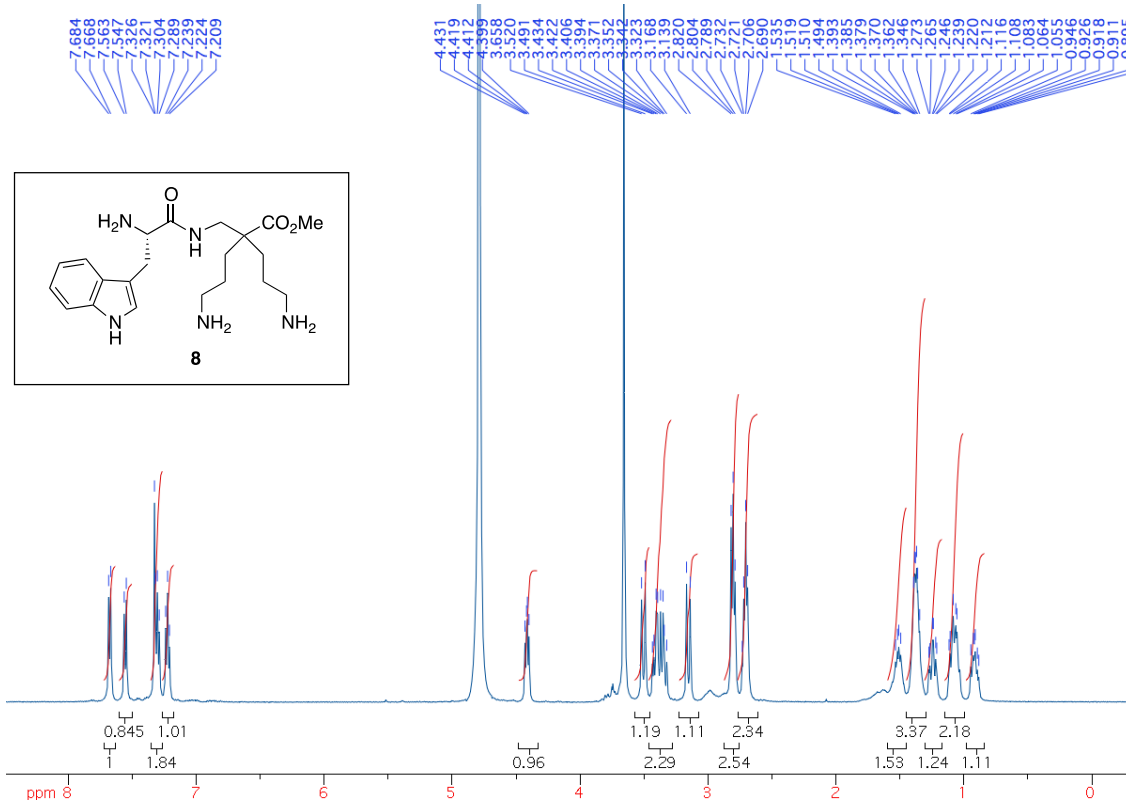
^{13}C NMR spectra of Boc- $\beta^{3,3}$ *h* bis-Orn(Boc) $_2$ OH 3, 75 MHz, CD $_3$ OD



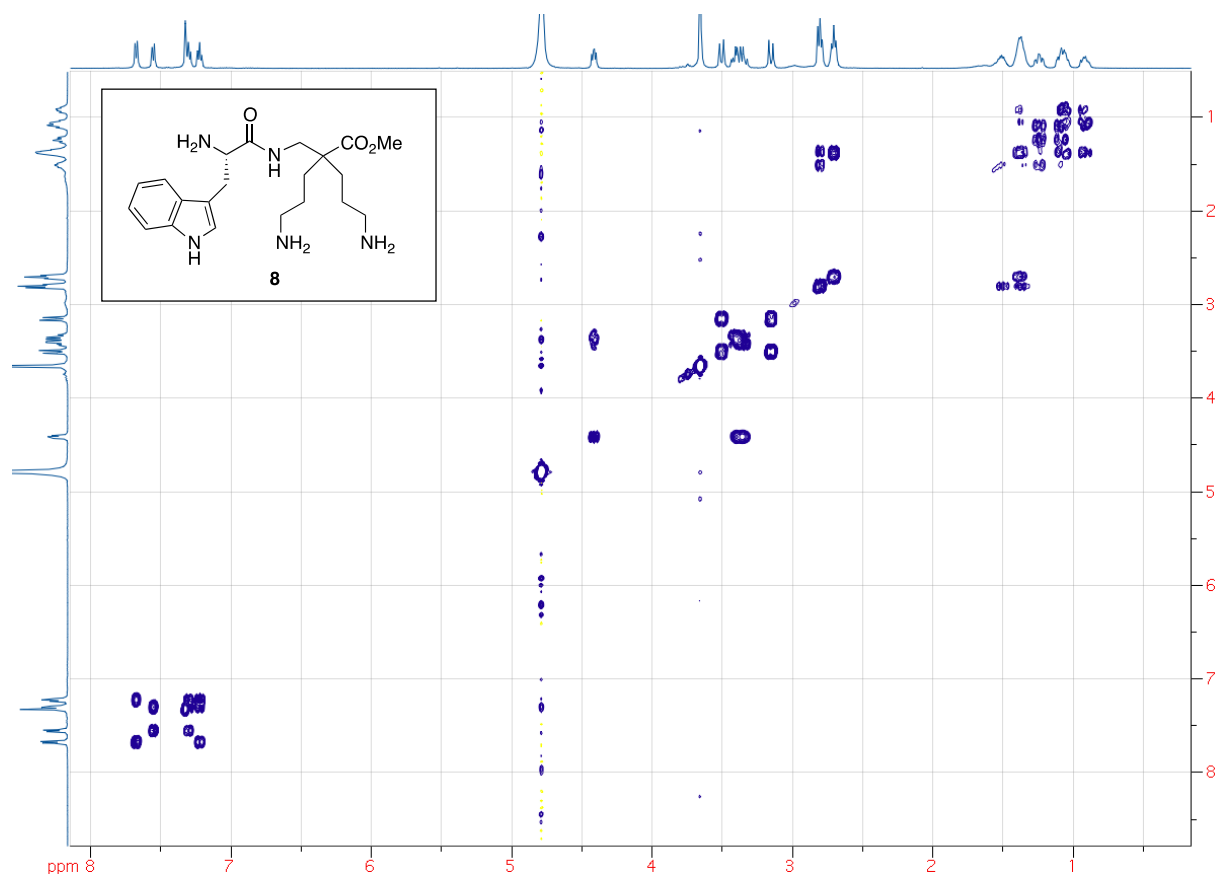
¹H NMR spectra of Boc Trp-β^{2,2}h bis-Orn(Boc)₂-OMe 15, 300MHz, CD₃OD



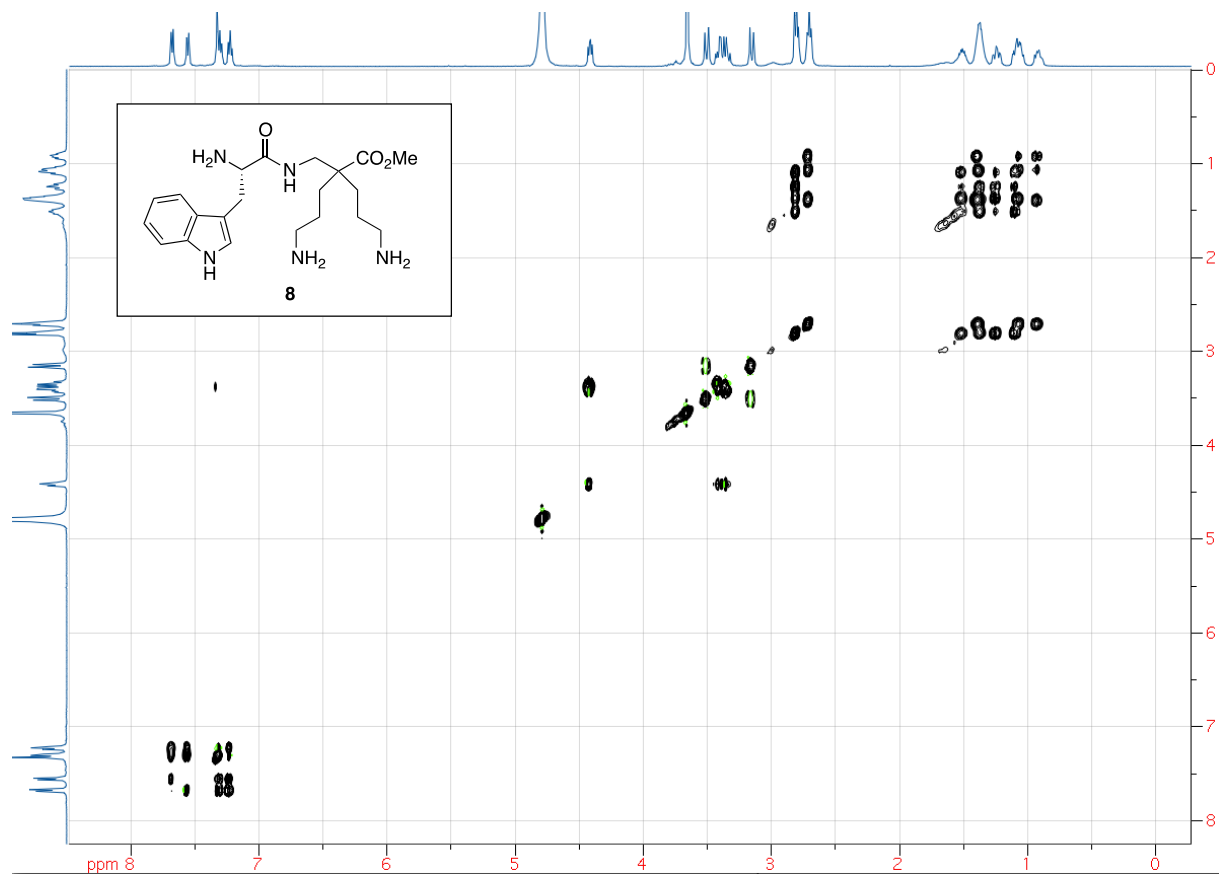
¹H NMR spectra of Trp-β^{2,2}h bis-Orn-OMe 8, 300MHz, D₂O



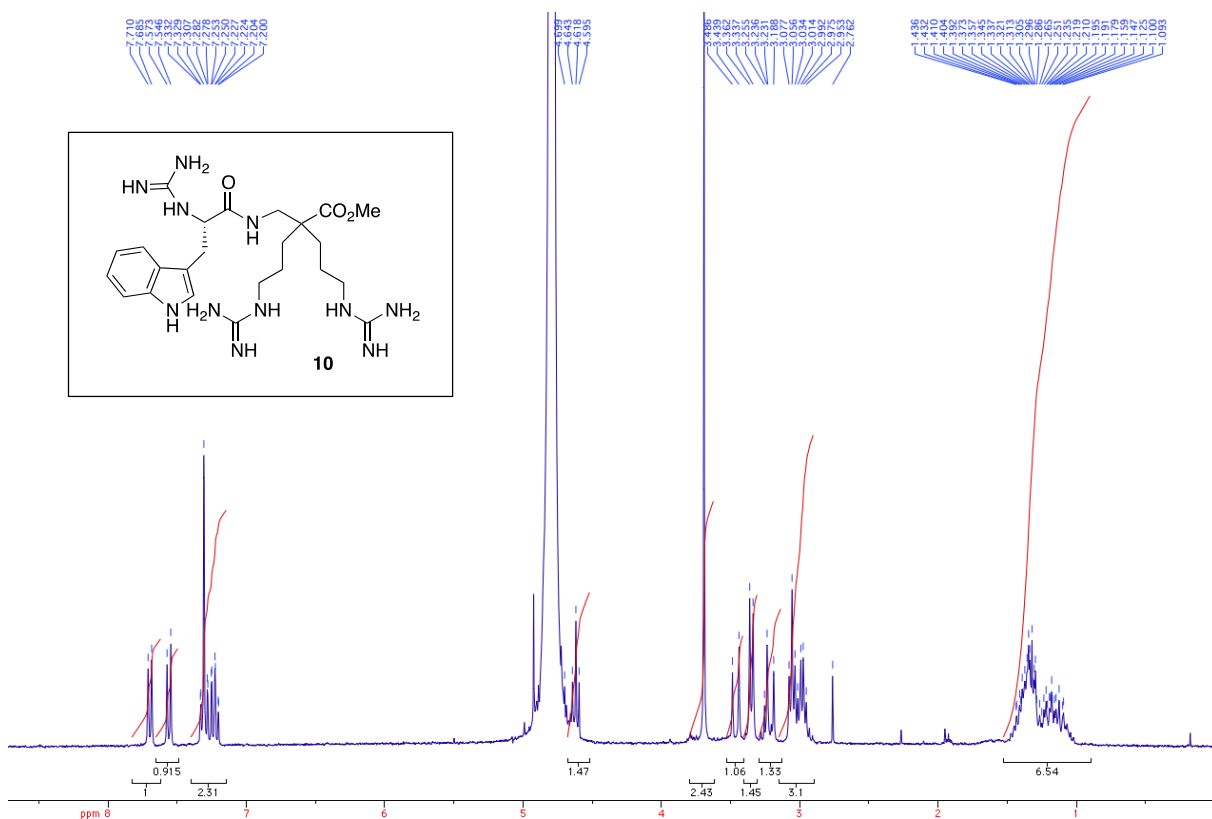
COSY NMR spectra of Trp- $\beta^{2,2}h$ bis-Orn-OMe 8, 300MHz, D₂O



TOCSY NMR spectra of Trp- $\beta^{2,2}h$ bis-Orn-OMe 8, 300MHz, D₂O

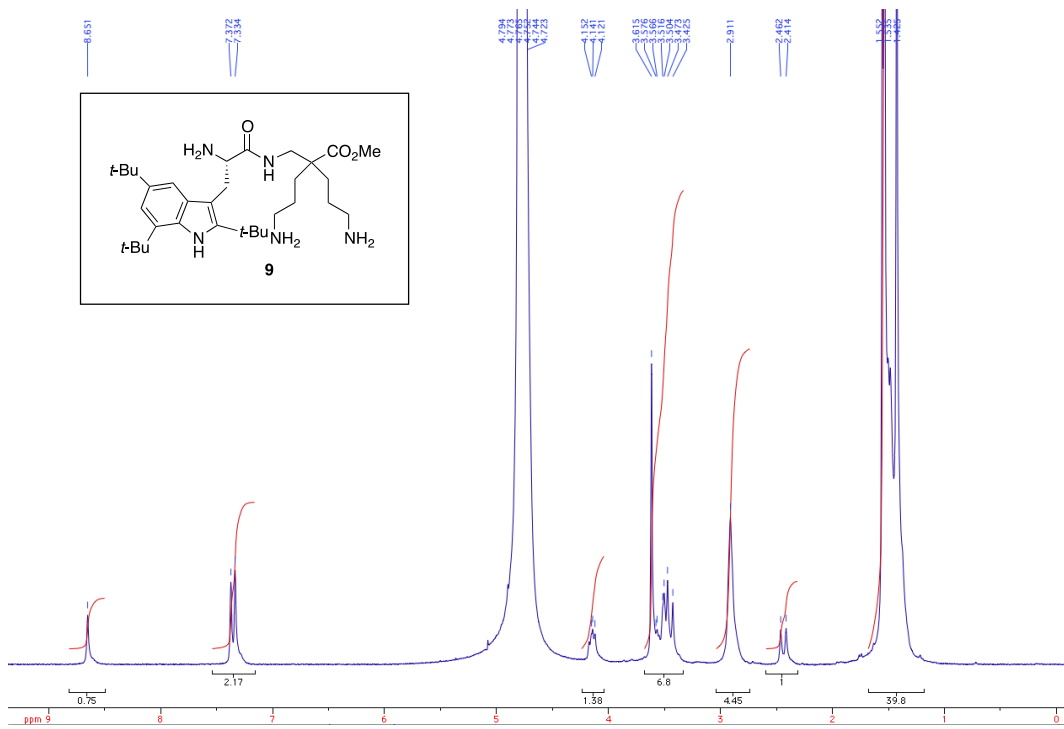


¹H NMR spectra of Gua-Trp-β^{2,2}h bis-Arg-OMe 10, 300MHz, D₂O

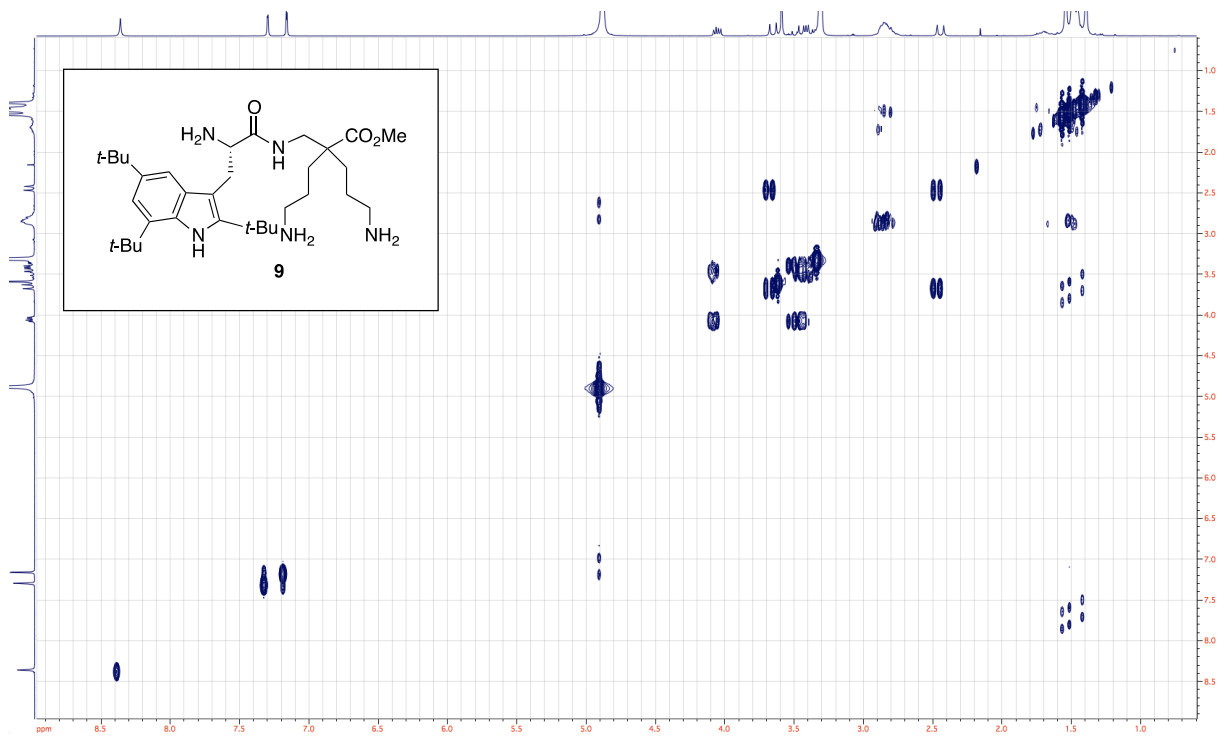


COSY NMR spectra of Gua-Trp-β^{2,2}h bis-Arg-OMe 10, 300MHz, CD₃OD

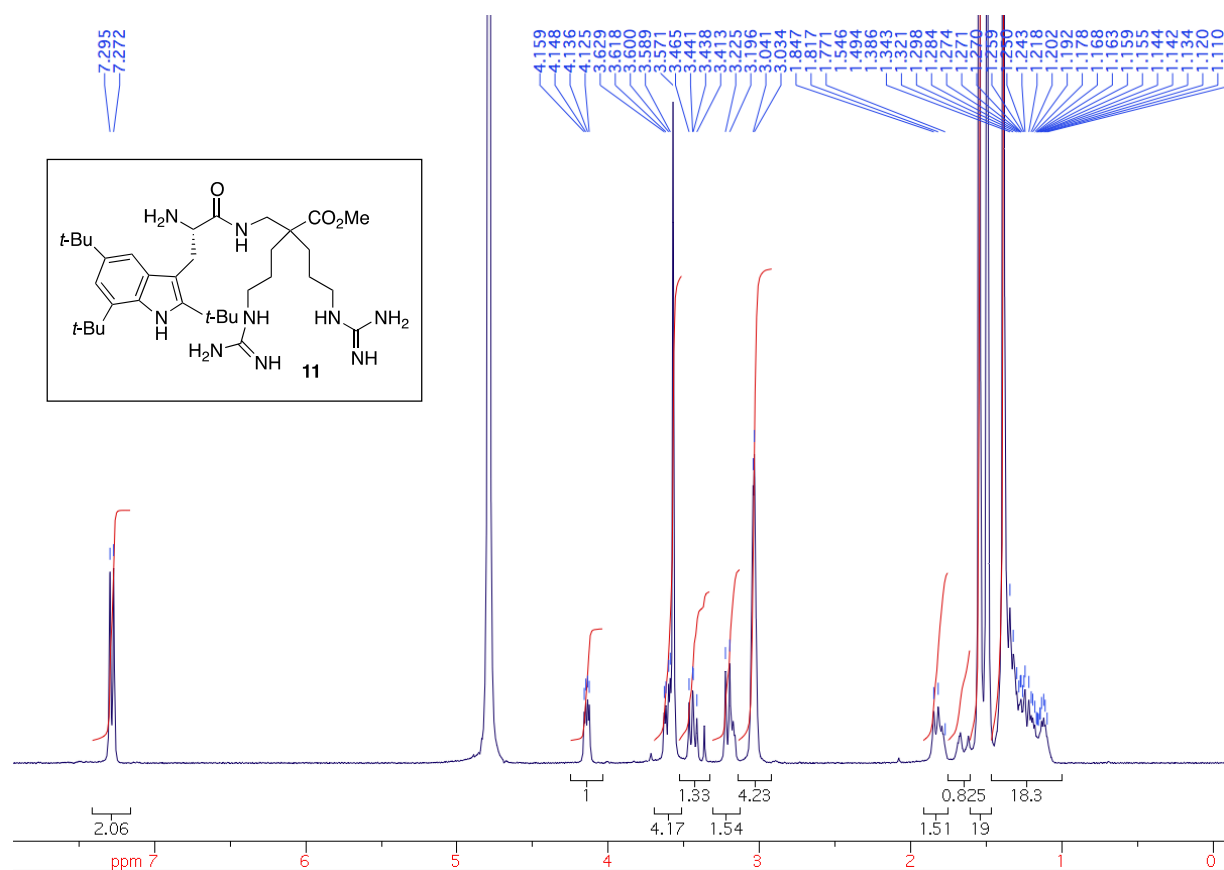




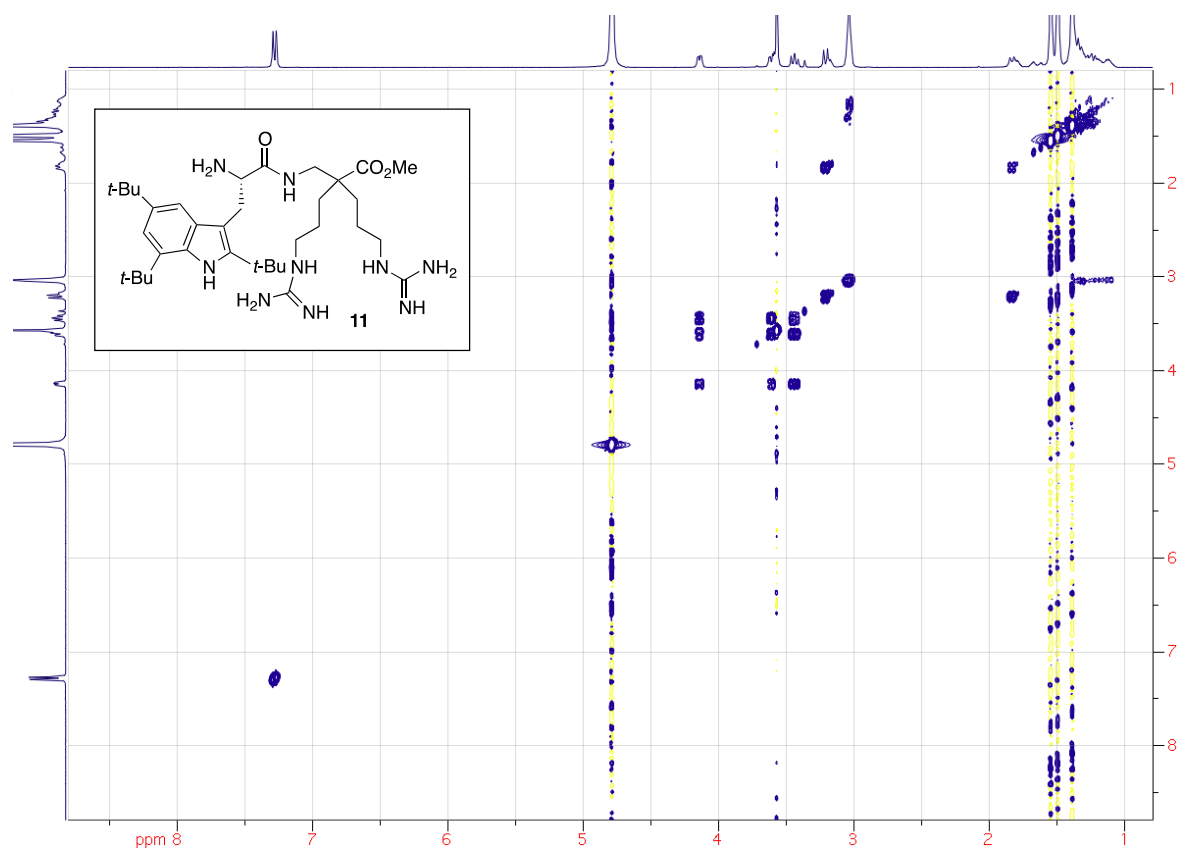
¹H NMR spectra of H-Tbt-β^{2.2}h bis-OrnOMe 9, 300MHz, D₂O

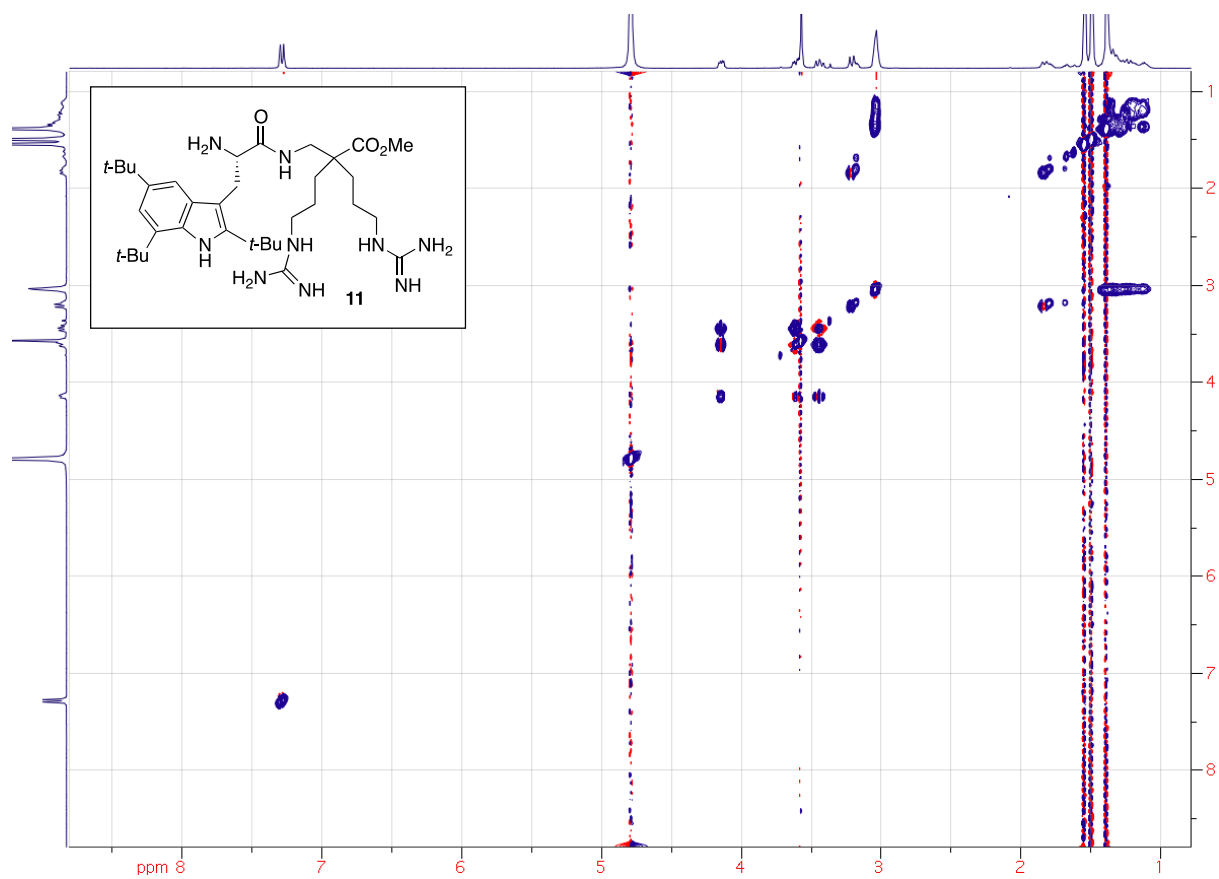


¹H NMR spectra of H-Tbt-β^{2,2}h bis-Arg-OMe 11, 500MHz, D₂O

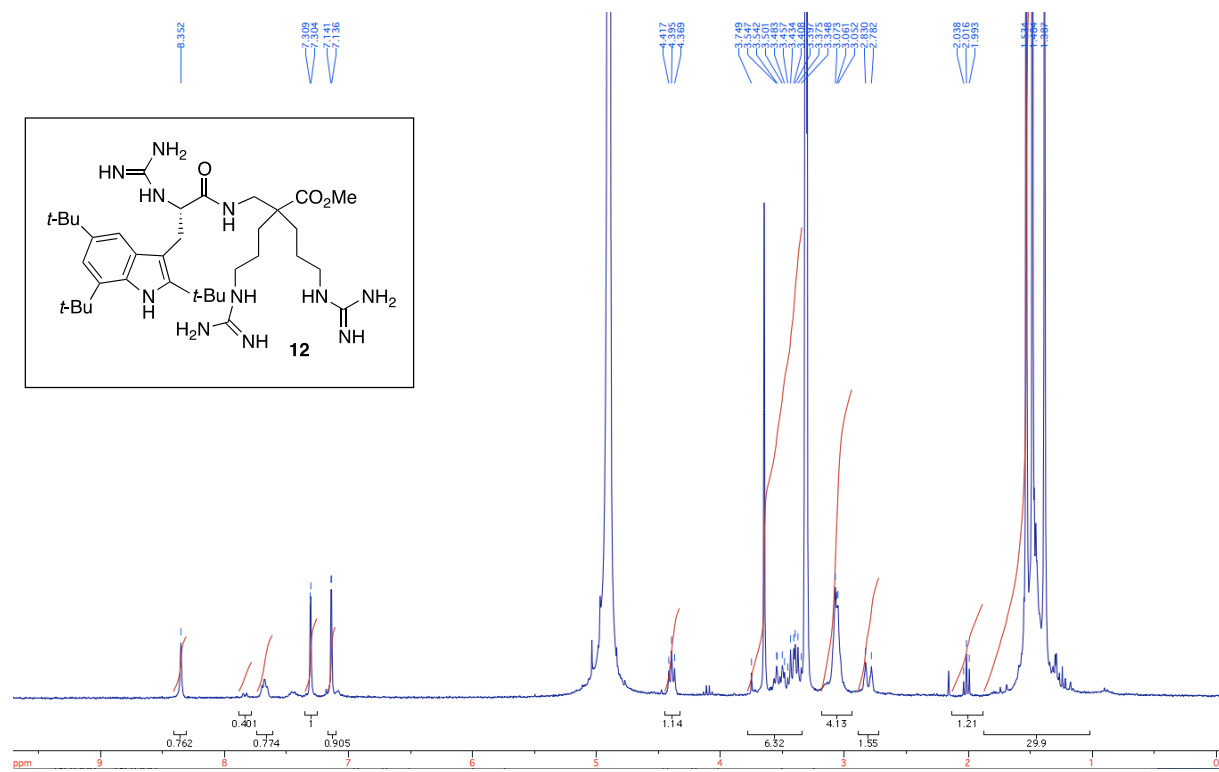


COSY NMR spectra of H-Tbt-β^{2,2}h bis-Arg-OMe 11, 500MHz, D₂O

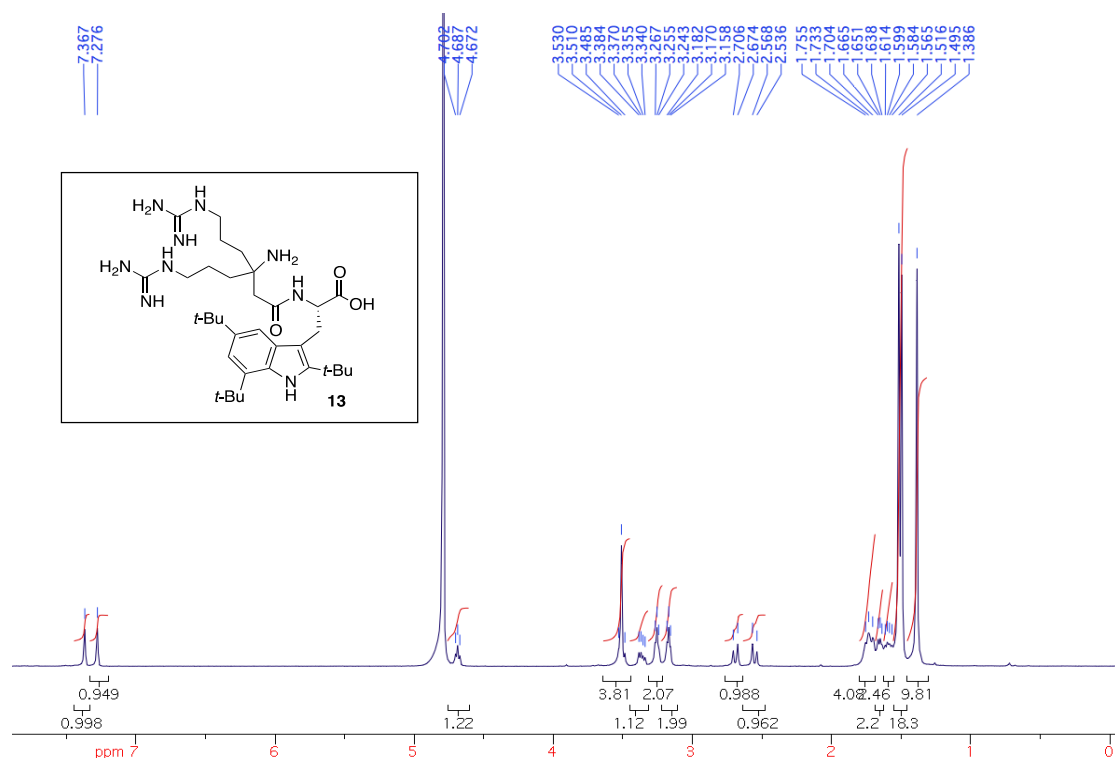




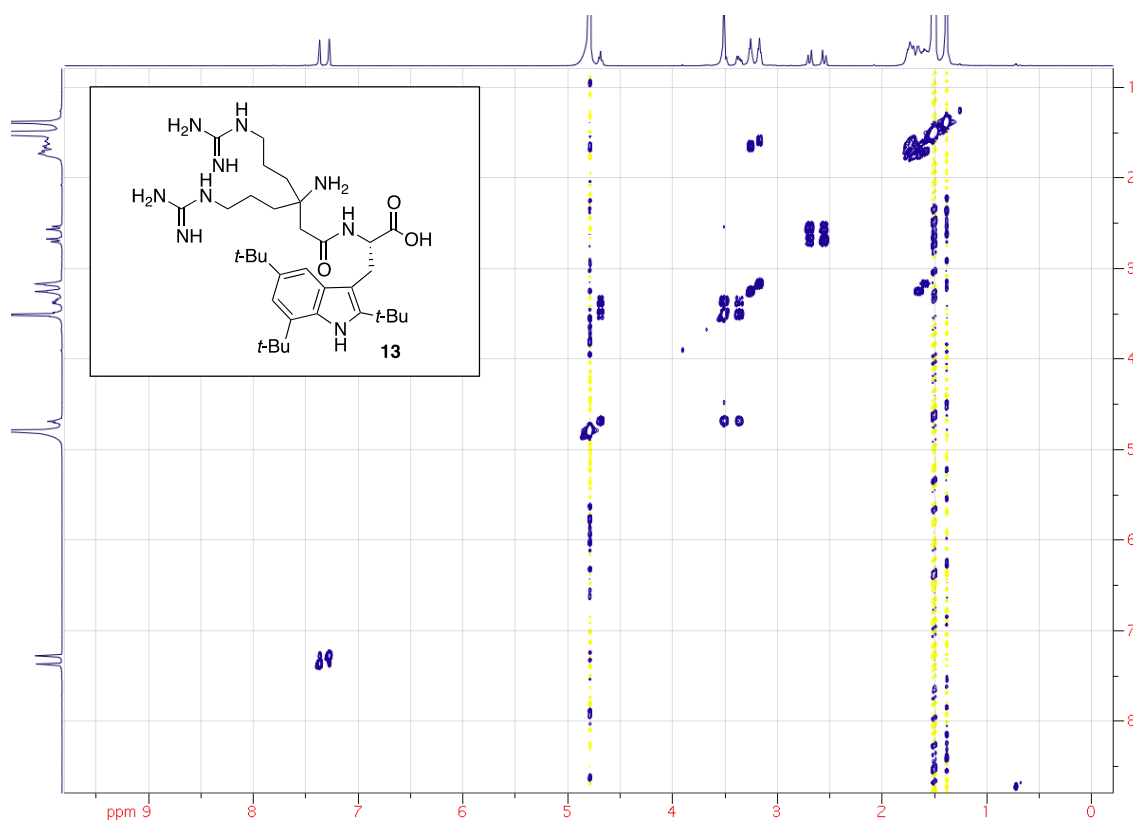
^1H NMR spectra of Gdm-Tbt- $\beta^{2,2}h$ bis-Arg-OMe 12, 400MHz, MeOD



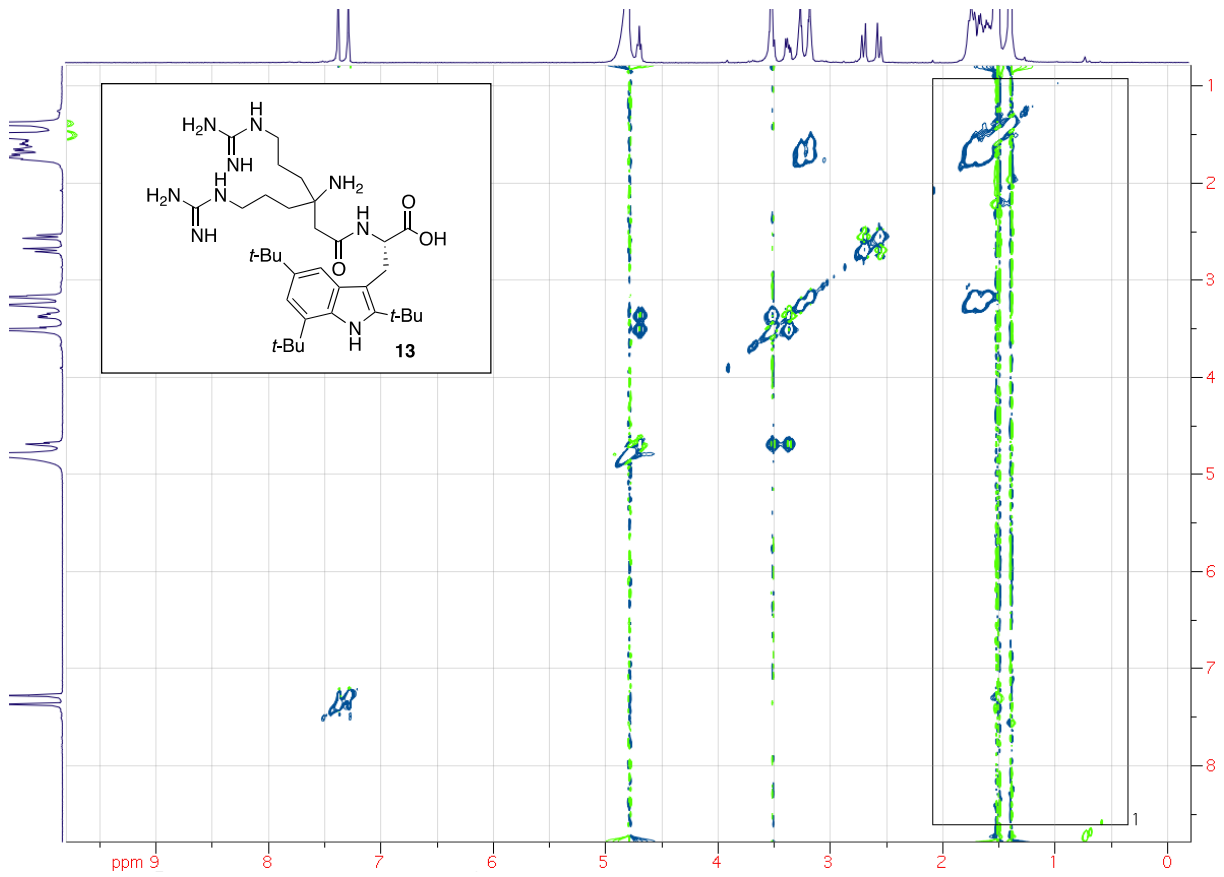
^1H NMR spectra of H- $\beta^{3,3}h$ bis-Arg-Tbt-OMe 13, 500MHz, D₂O

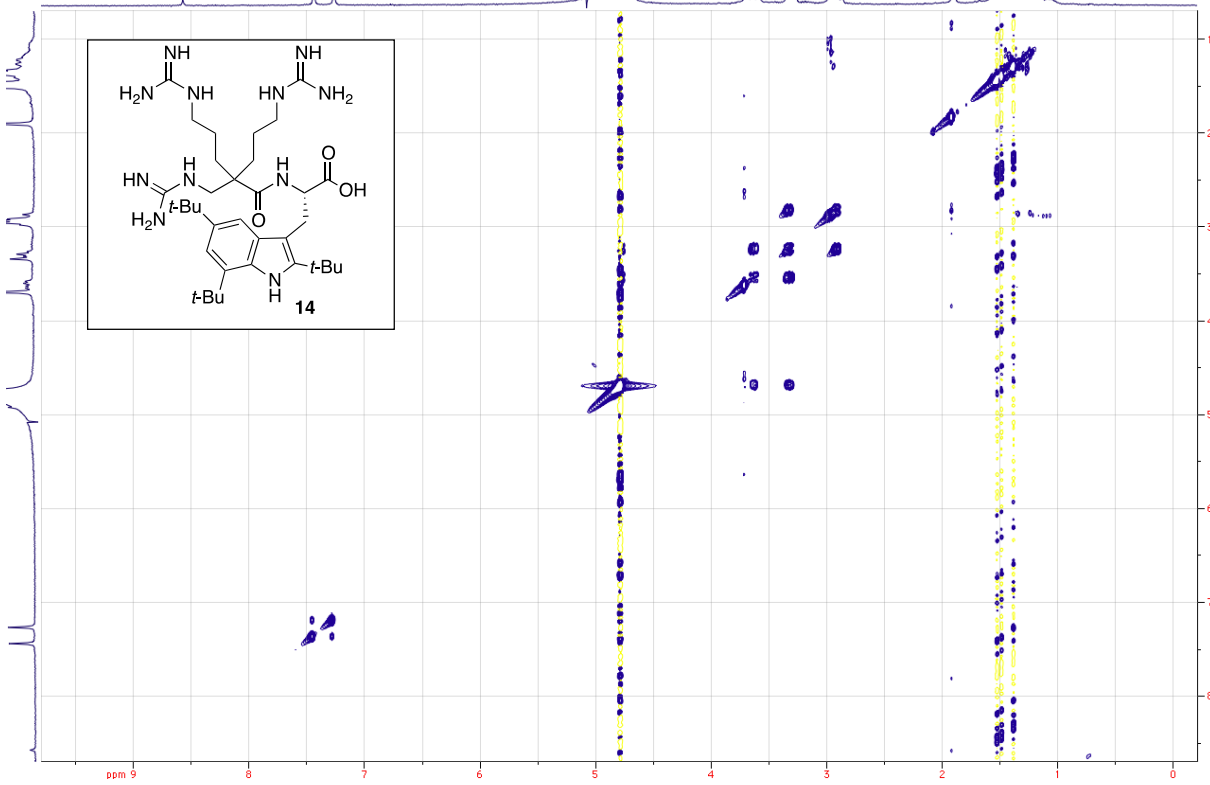
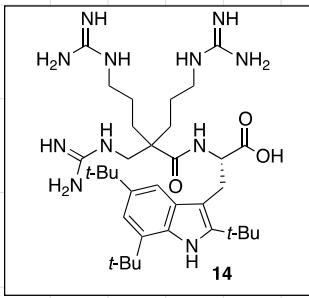
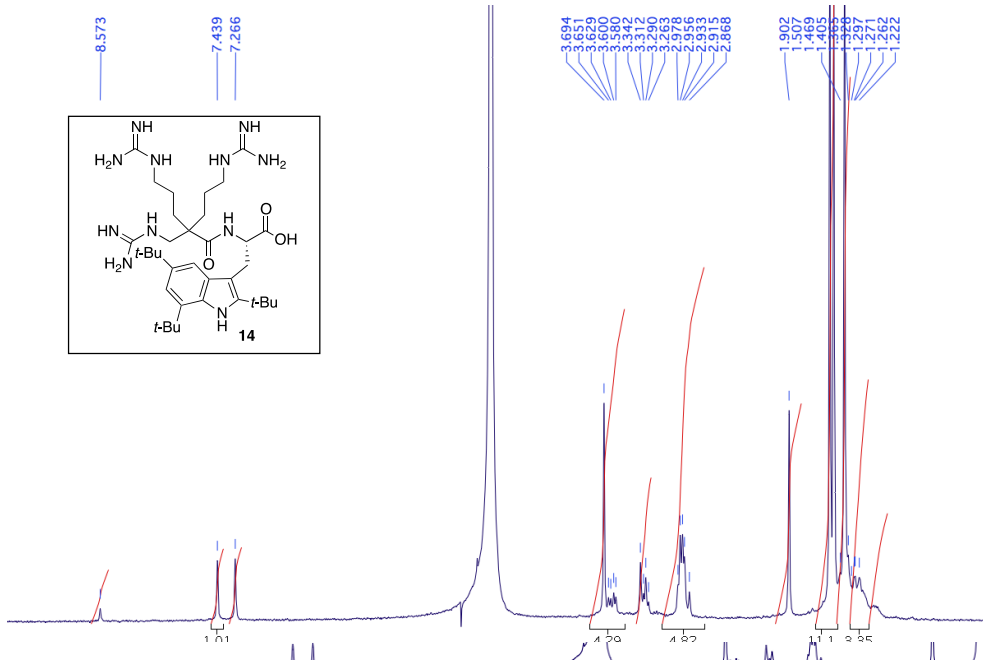
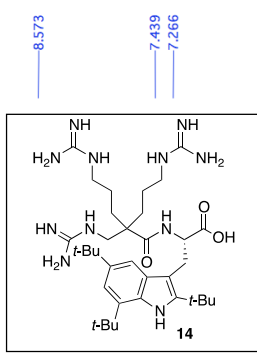


COSY NMR spectra of H- $\beta^{3,3}h$ bis-Arg-Tbt-OMe 13, 500MHz, D₂O

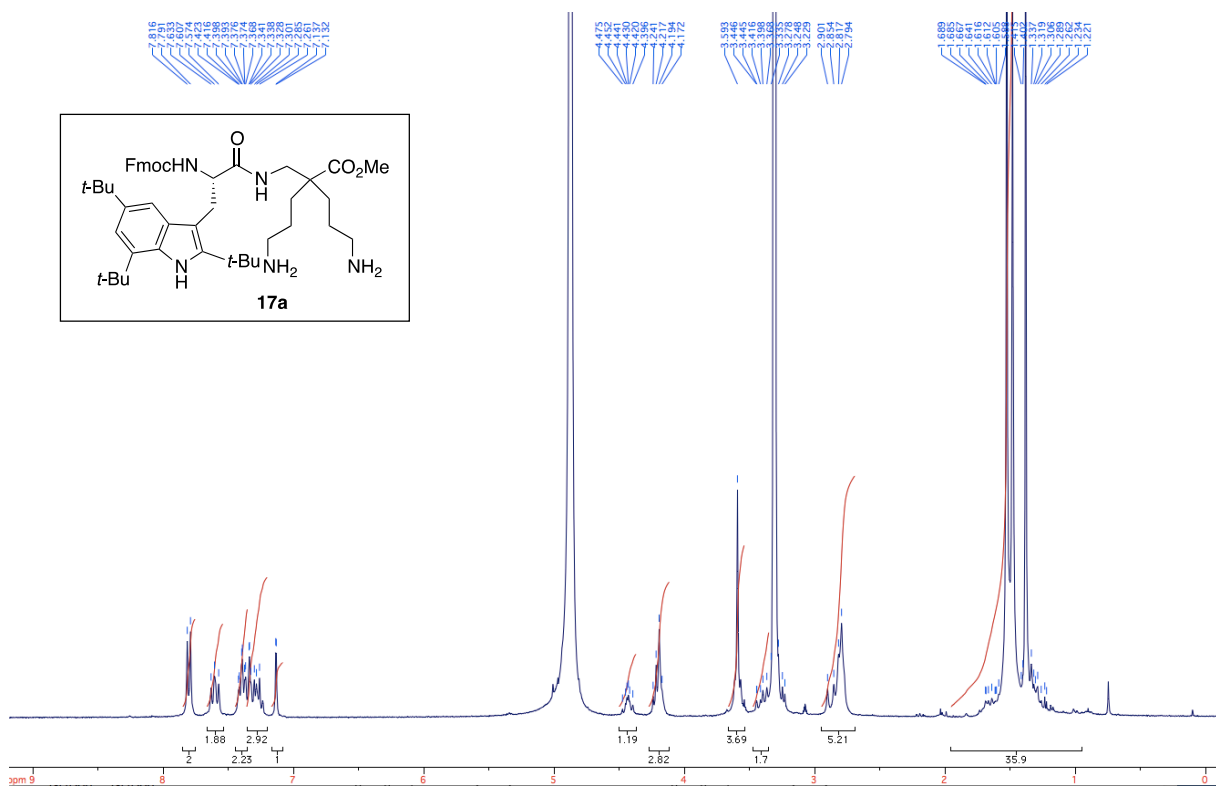
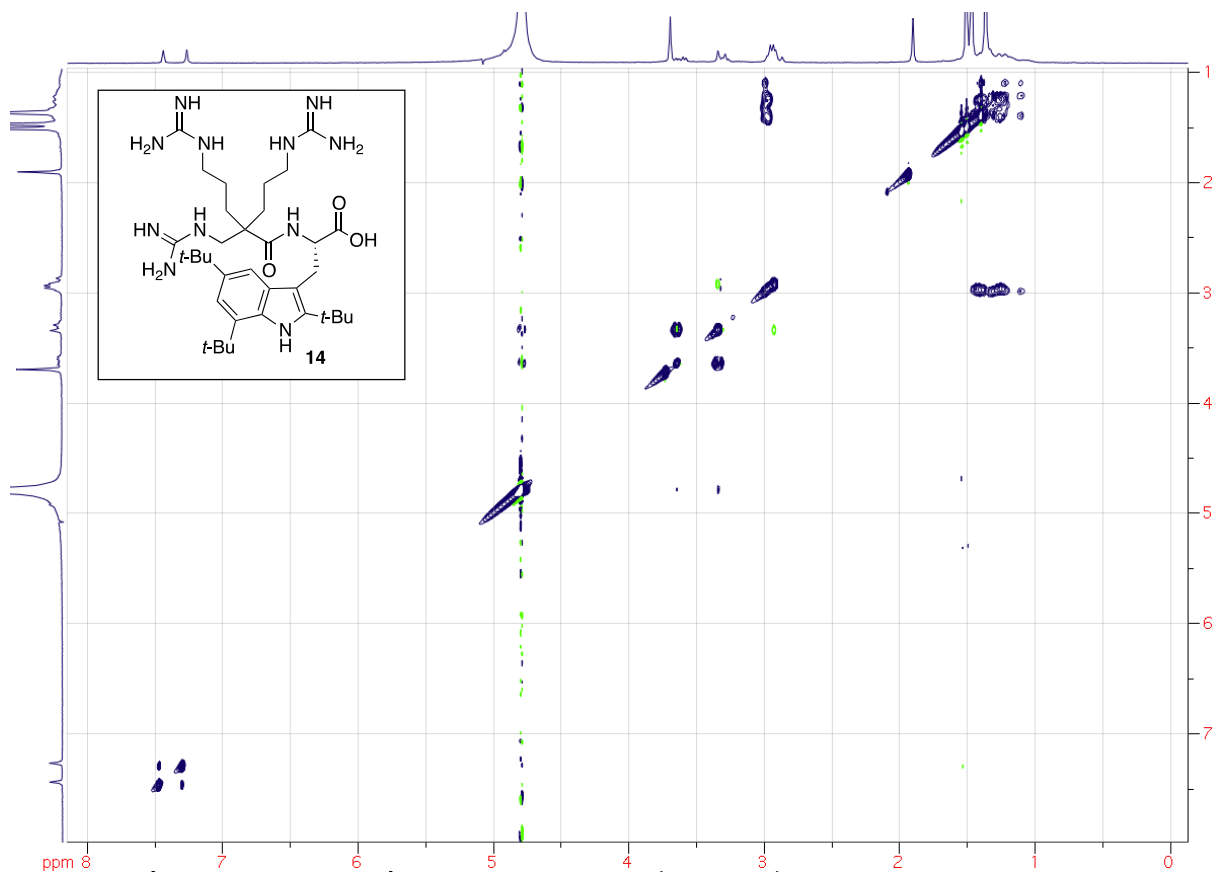


TOCSY NMR spectra of H- $\beta^{3,3}$ *h* bis-Arg-Tbt-OMe 13, 500MHz, D₂O

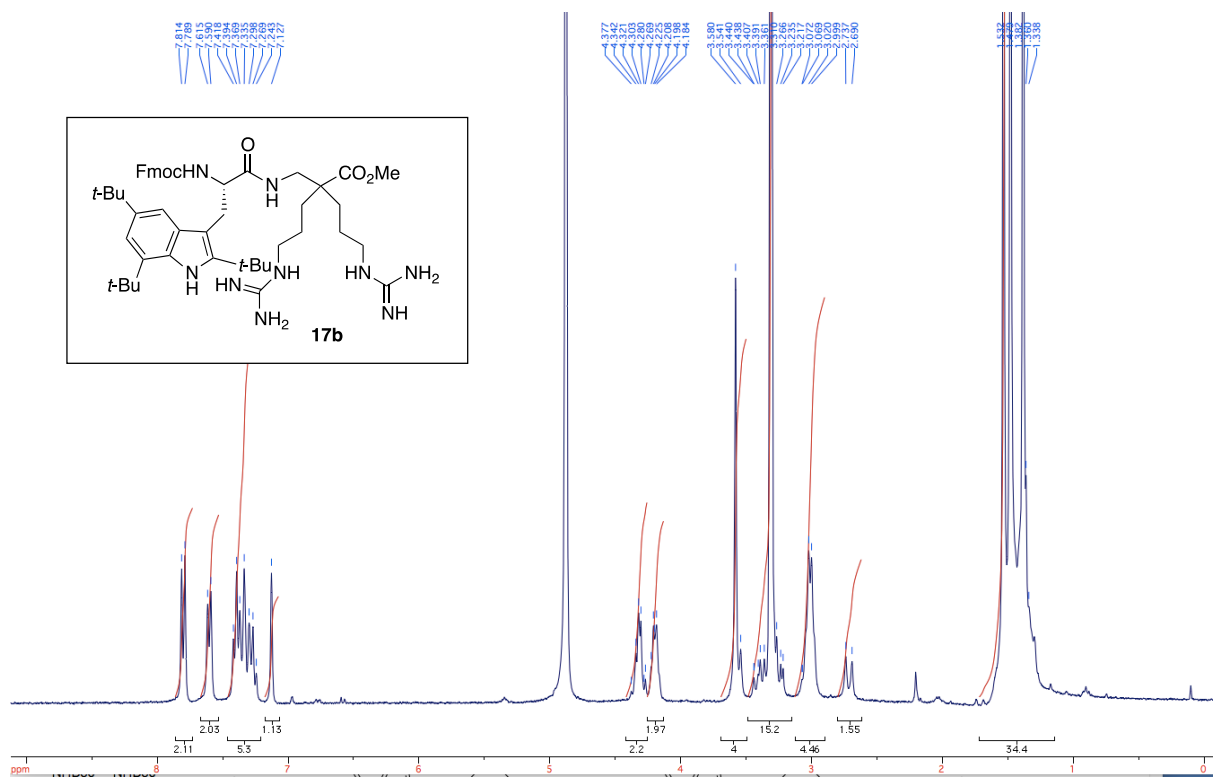




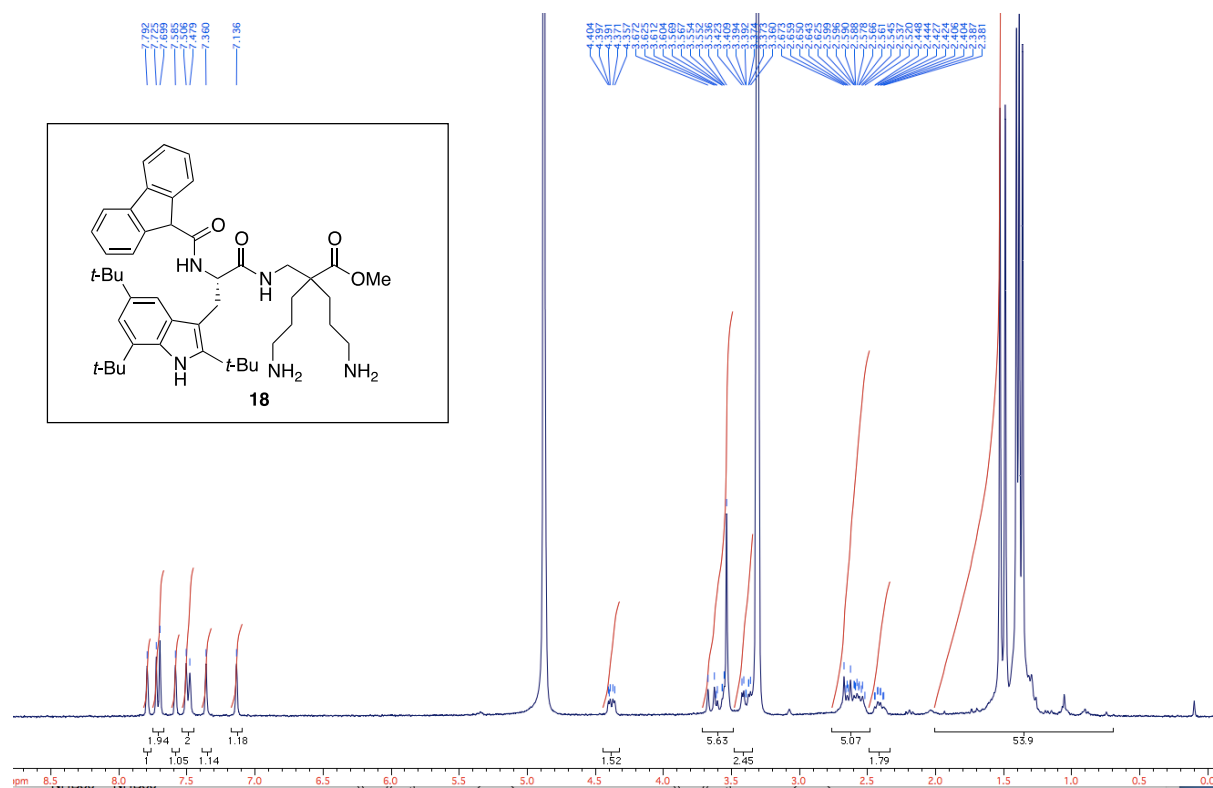
TOCSY NMR spectra of Gua- $\beta^{2,2}h$ bis-Arg-Tbt-OMe 14, 500MHz, D₂O



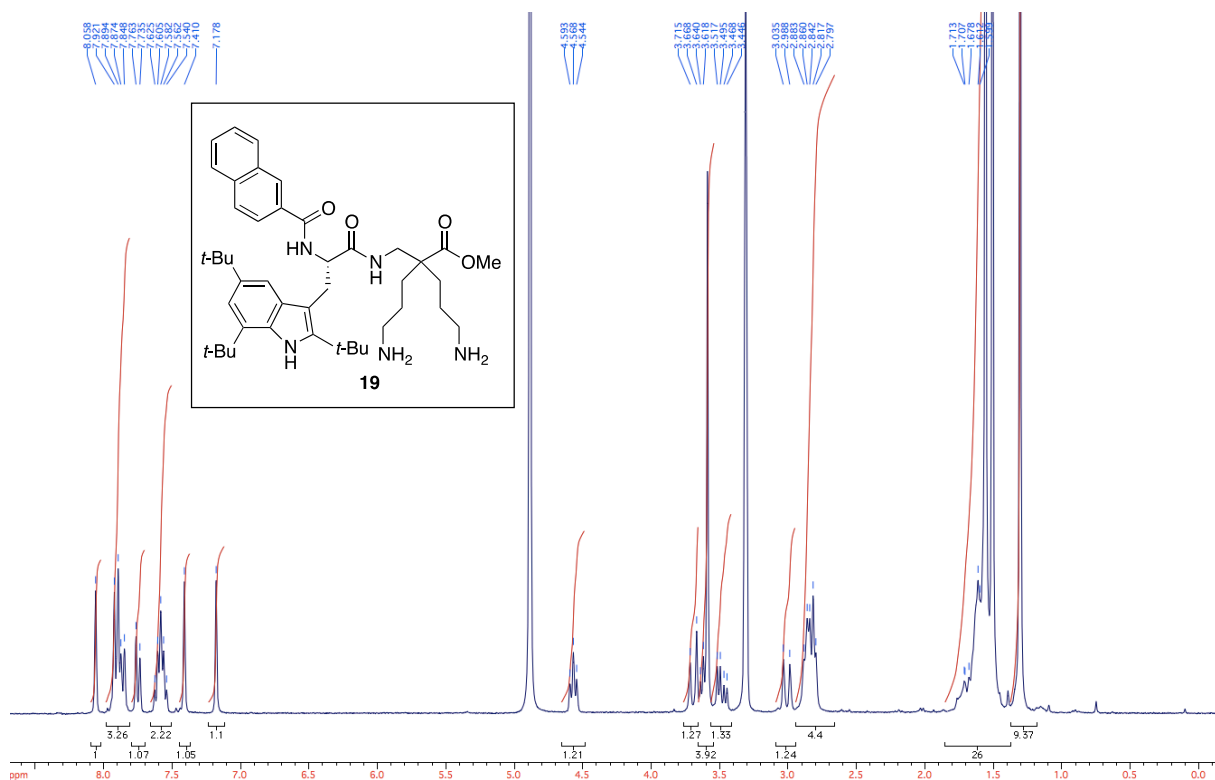
¹H NMR spectra of Fmoc-Tbt- $\beta^{2,2}h$ bis-Arg-OMe 17b, 300MHz, CD₃OD



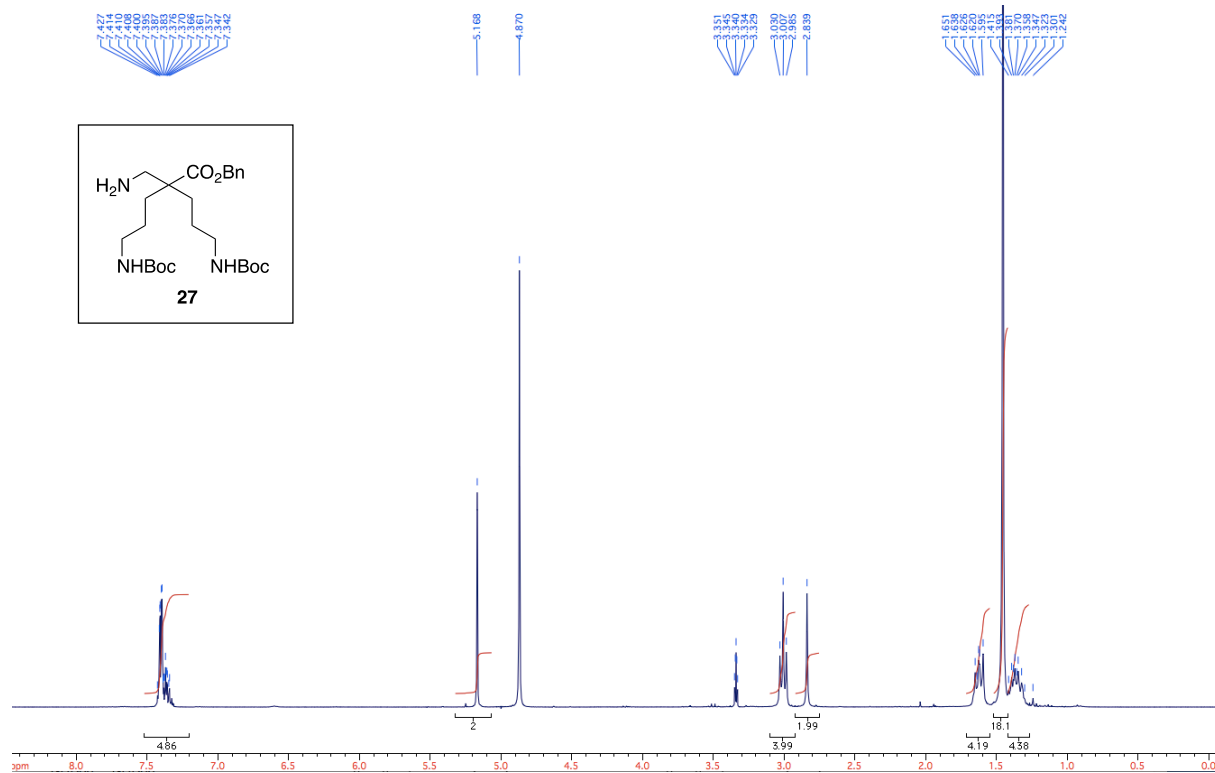
¹H NMR spectra of Fluo-Tbt-β^{2,2}h bis-Orn-OMe 18, 300MHz, CD₃OD



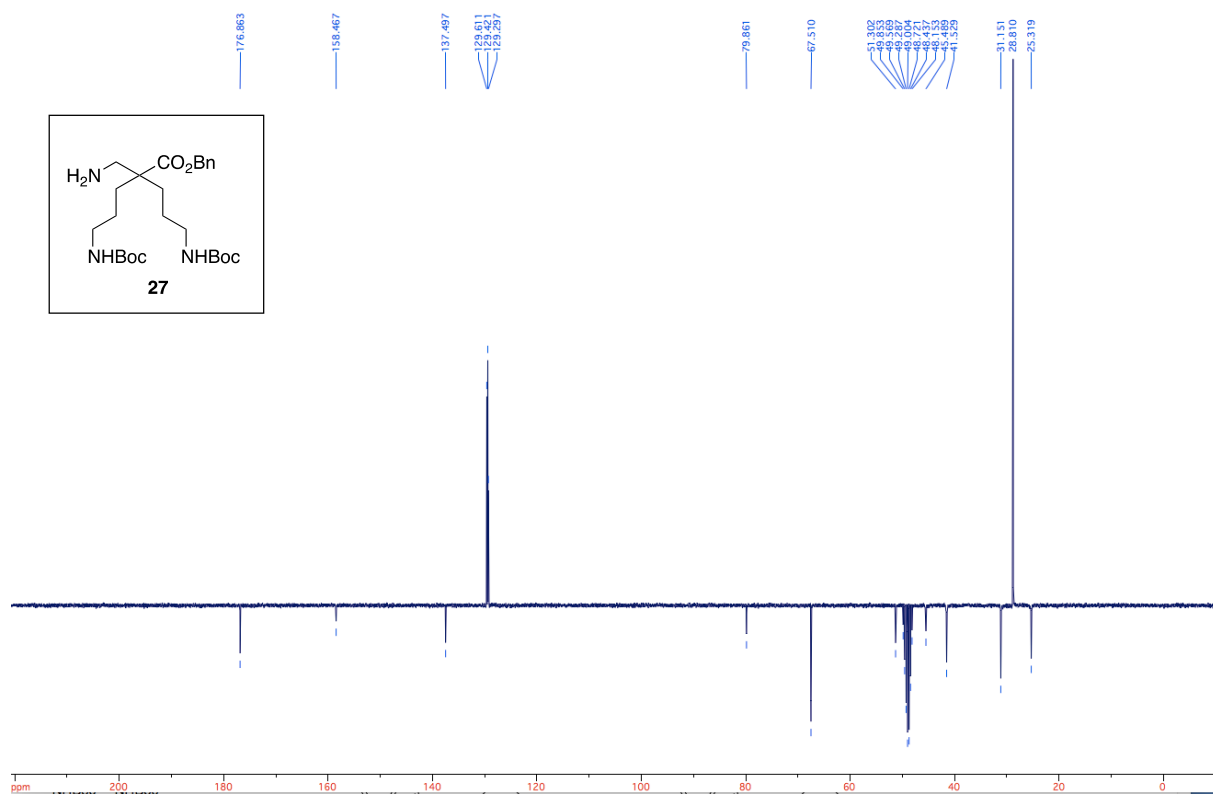
¹H NMR spectra of Np-Tbt-β^{2,2}h bis-Orn-OMe 19, 300MHz, CD₃OD



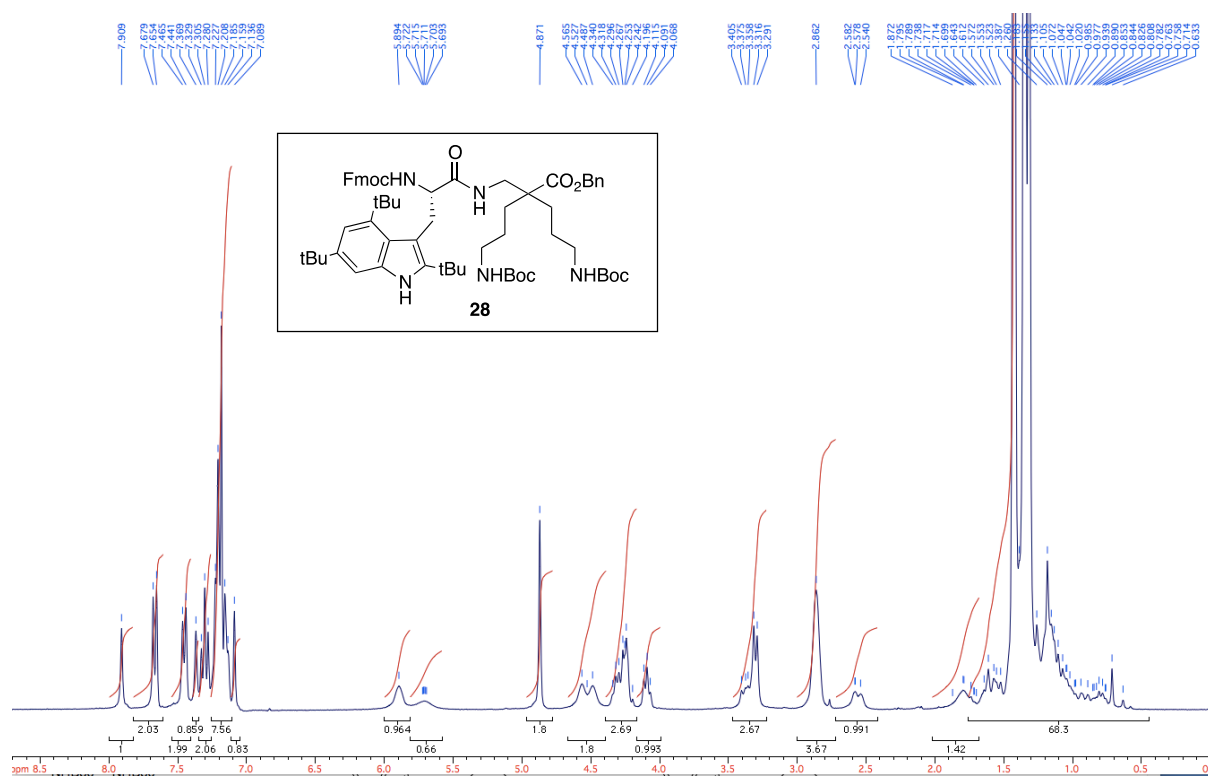
¹H NMR spectra of H-β^{2,2}h bis-Orn(Boc)₂OBn 27, 300 MHz, CD₃OD



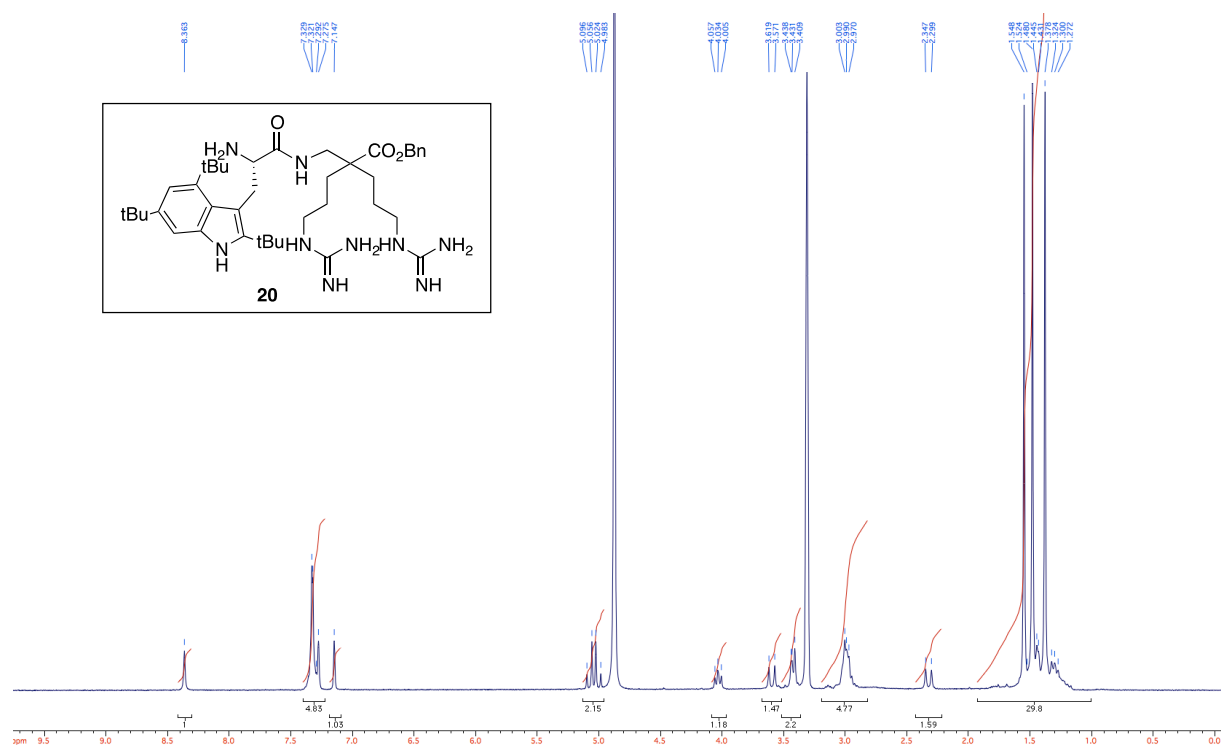
¹³C NMR spectra of H-β^{2,2}h bis-Orn(Boc)₂OBn 27, 300 MHz, CD₃OD



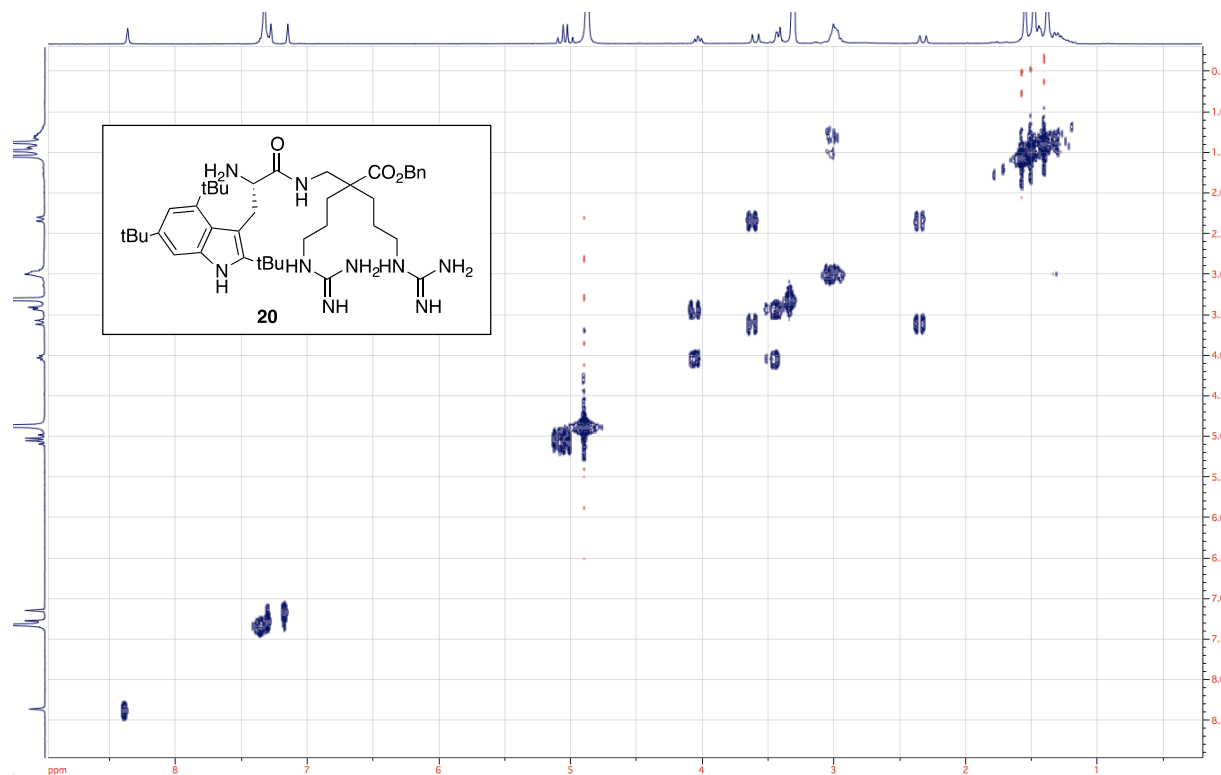
¹H NMR spectra of Fmoc-Tbt-β^{2,2}h bis-Orn(Boc)₂OBn 28, 300MHz, CD₃OD



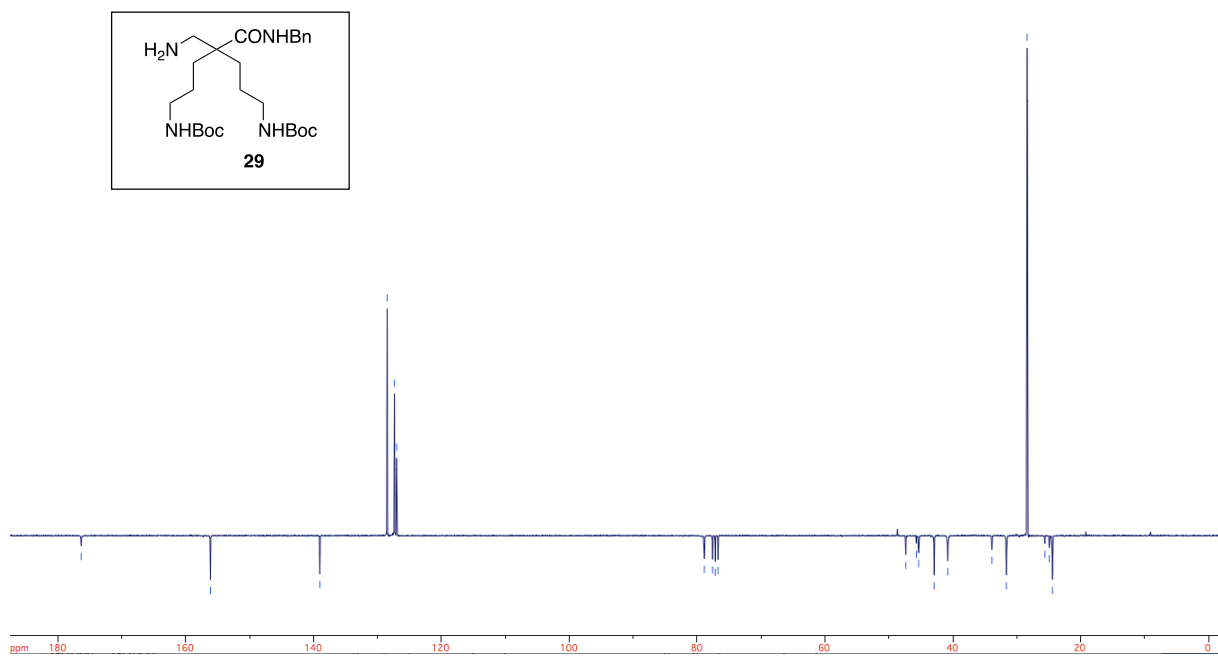
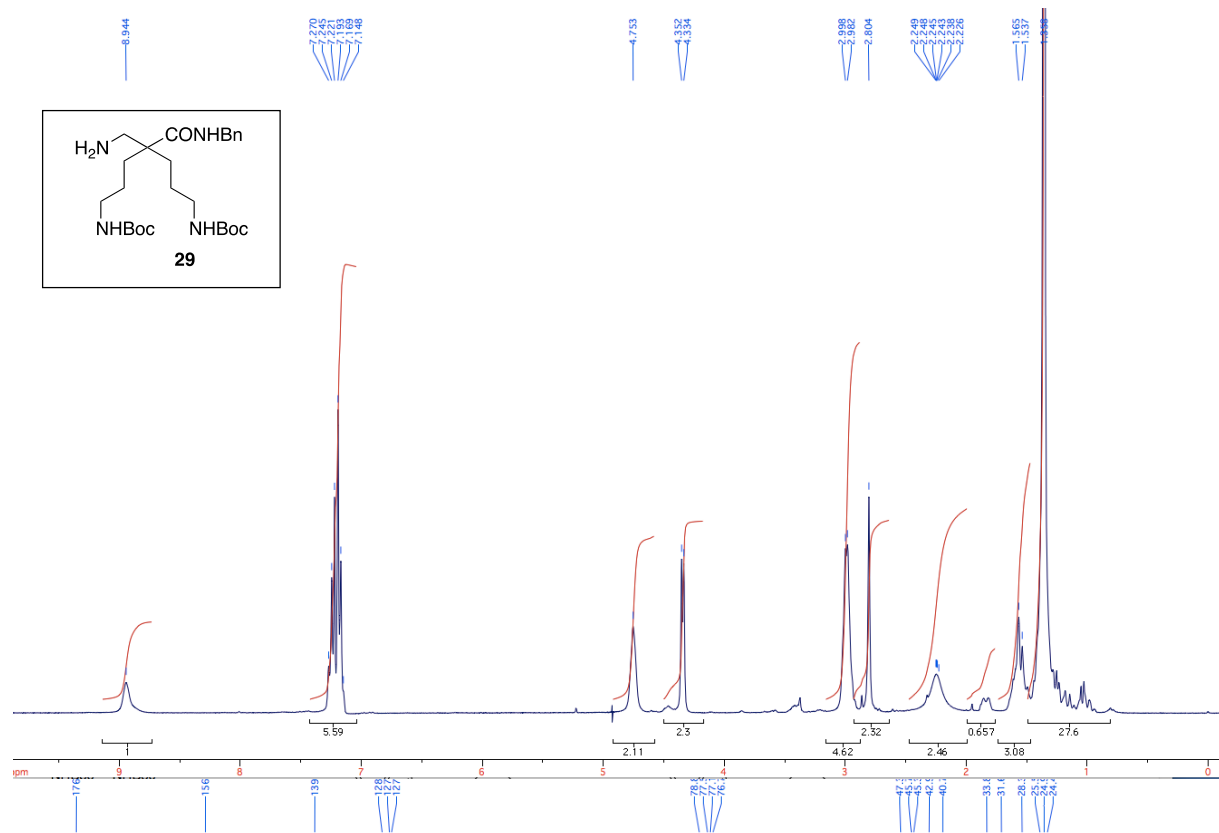
¹H NMR spectra of H-Tbt-β^{2,2}h bis-Arg-OBn 20, 300MHz, CD₃OD



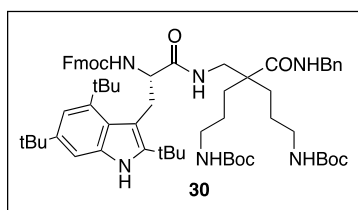
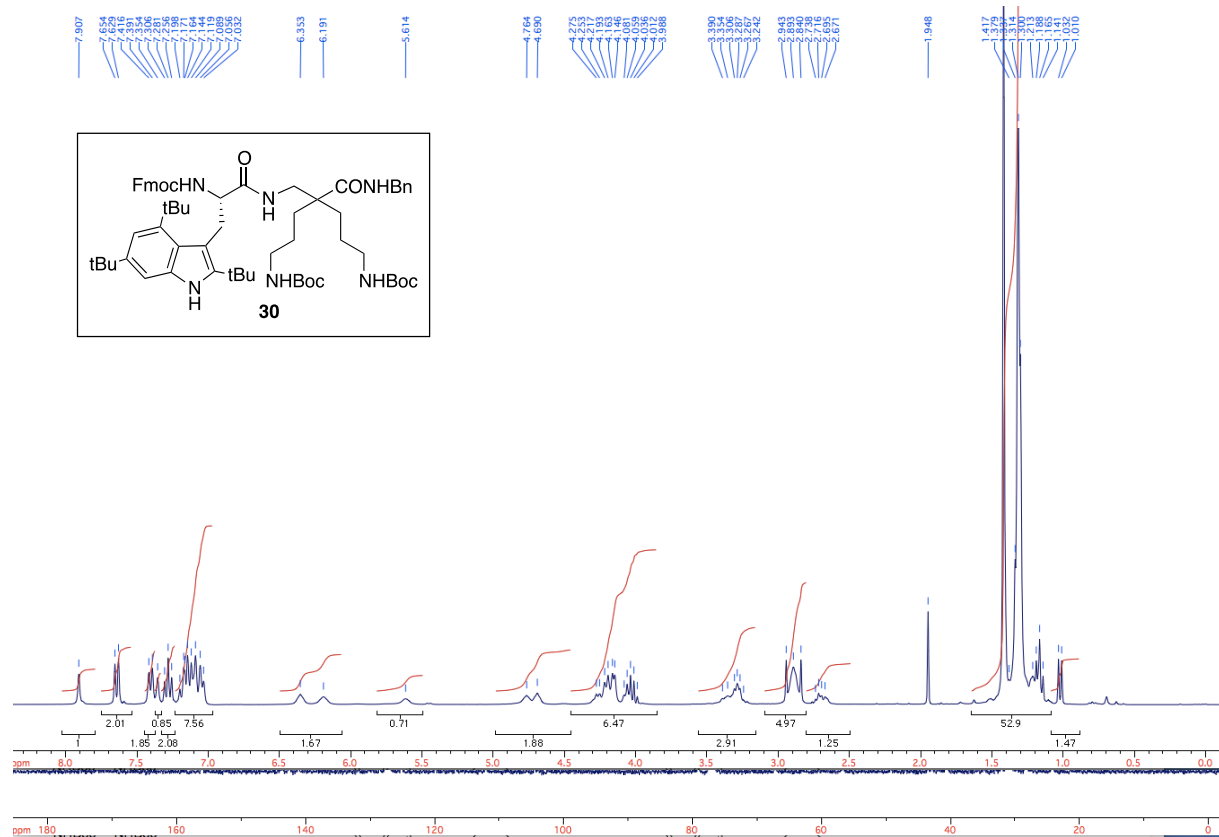
COSY NMR spectra of H-Tbt-β^{2,2}h bis-Arg-OBn 20, 300MHz, CD₃OD



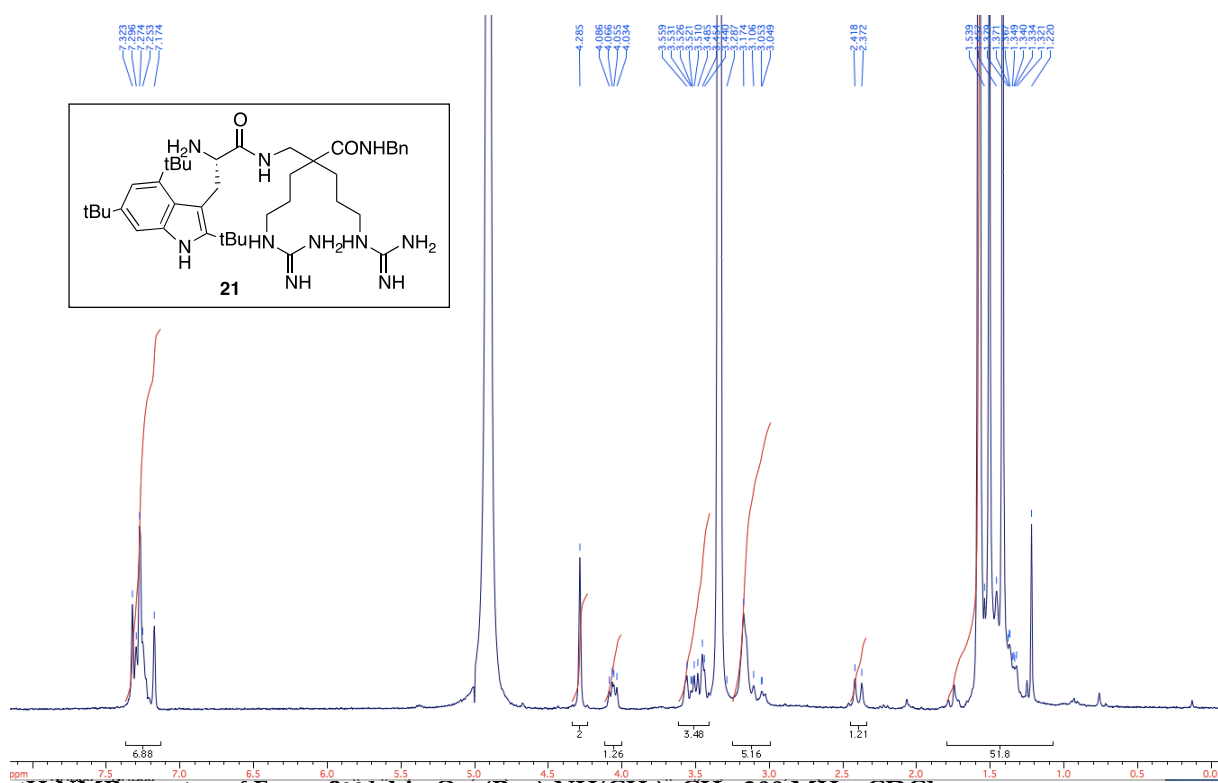
^1H NMR spectra of H- $\beta^{2,2}h$ bis-Orn(Boc) $_2$ NHBn **29**, 300 MHz, CD $_3$ OD



¹H NMR spectra of Fmoc-Tbt-β^{2,2}h bis-Orn(Boc)₂-NHBn 30, 300MHz, CD₃OD



¹H NMR spectra of H-Tbt-β^{2,2}h bis-Arg-NHBn 21, 300MHz, CD₃OD



¹H NMR spectra of Fmoc-β^{2,2}h bis-Orn(Boc)₂NH(CH₂)₁₃CH₃, 300 MHz, CDCl₃

