Novel reaction of 3,4-dibromofuran with azo diesters to give tetrahydropyridazinones

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Supplementary Information

Experimental procedures

Preparation of diisopropyl 3,5-dibromo-4-oxo-1,2,3,4-tetrahydropyridazine-1,2-dicarboxylate 6

A mixture of 3,4-dibromofuran (2.26 g, 10 mmol) and diisopropyl azodicarboxylate (2.02 g, 10 mmol) was stirred at RT in the absence of solvent for 7 days. Column chromatography of the mixture (SiO_2, diethyl ether–hexane, 1:2) gave the title product (1.71 g, 40%) as colourless crystals.

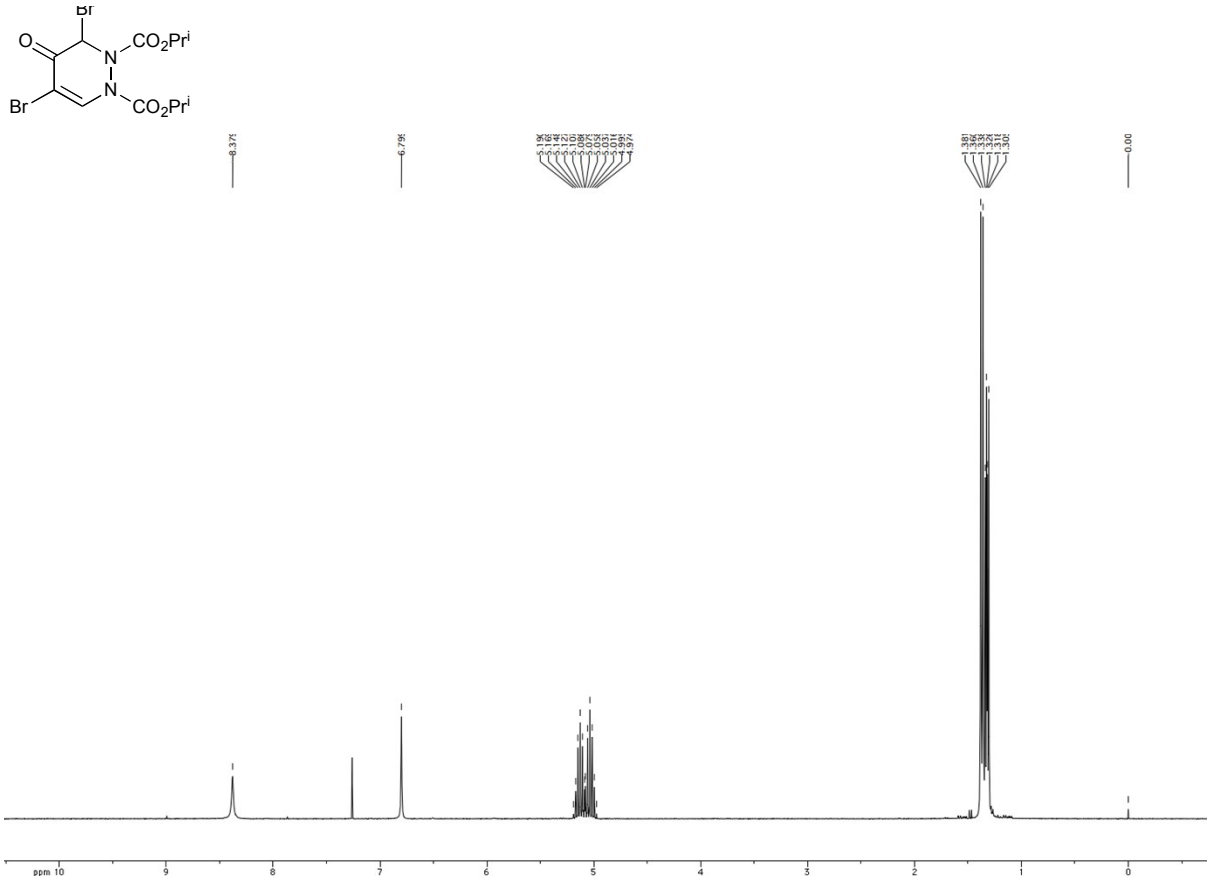
Preparation of diethyl 3,5-dibromo-4-oxo-1,2,3,4-tetrahydropyridazine-1,2-dicarboxylate 7

A mixture of 3,4-dibromofuran (2.26 g, 10 mmol) and diethyl azodicarboxylate (1.74 g, 10 mmol) was stirred at RT in the absence of solvent for 7 days. Column chromatography of the mixture (SiO_2, diethyl ether–hexane, 1:2) gave the title product (2.72 g, 68%) as colourless crystals.

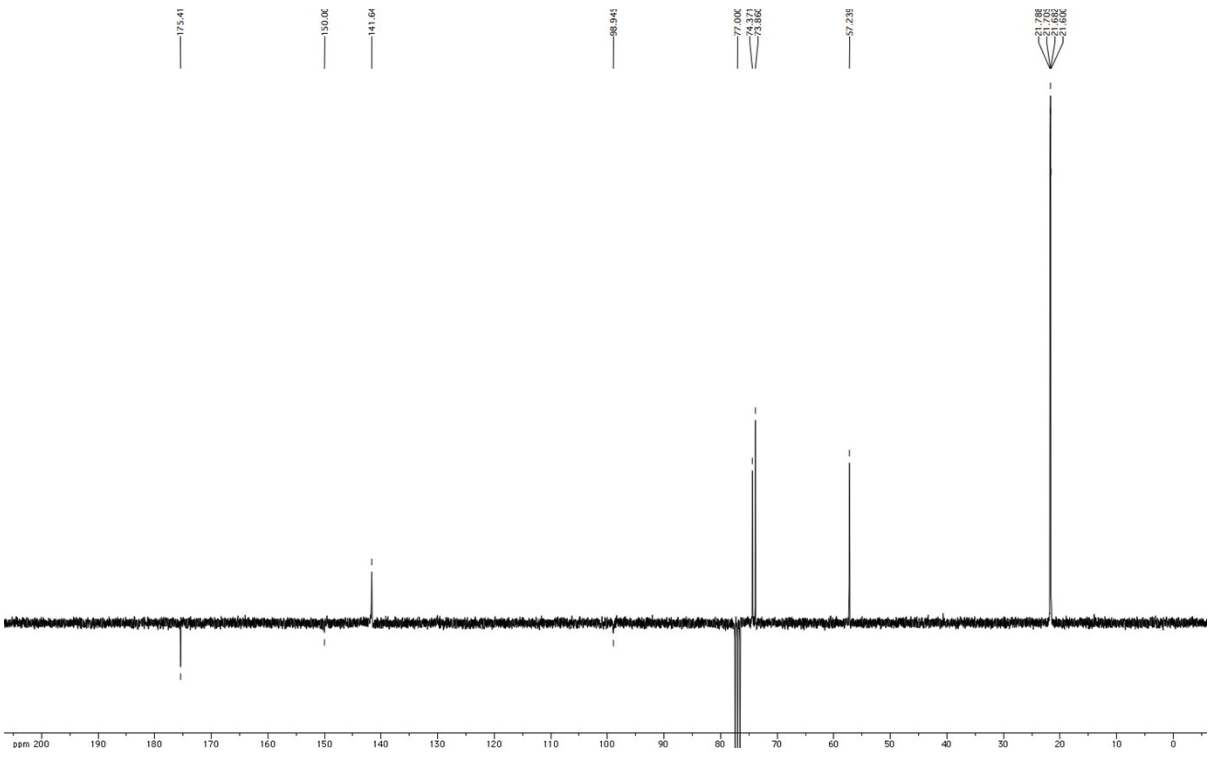
Table: Hydrogen bonding parameters for **11** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(2)-H(2N)...Br(1)	0.98(3)	2.33(4)	3.272(3)	162(3)
O(5)-H(5O)...O(10)	0.98(2)	1.57(2)	2.544(4)	177(5)
O(10)-H(10A)...Br(1)#1	0.98(4)	2.35(5)	3.324(3)	175(5)
O(10)-H(10B)...Br(1)#2	0.98(5)	2.31(6)	3.274(3)	168(6)

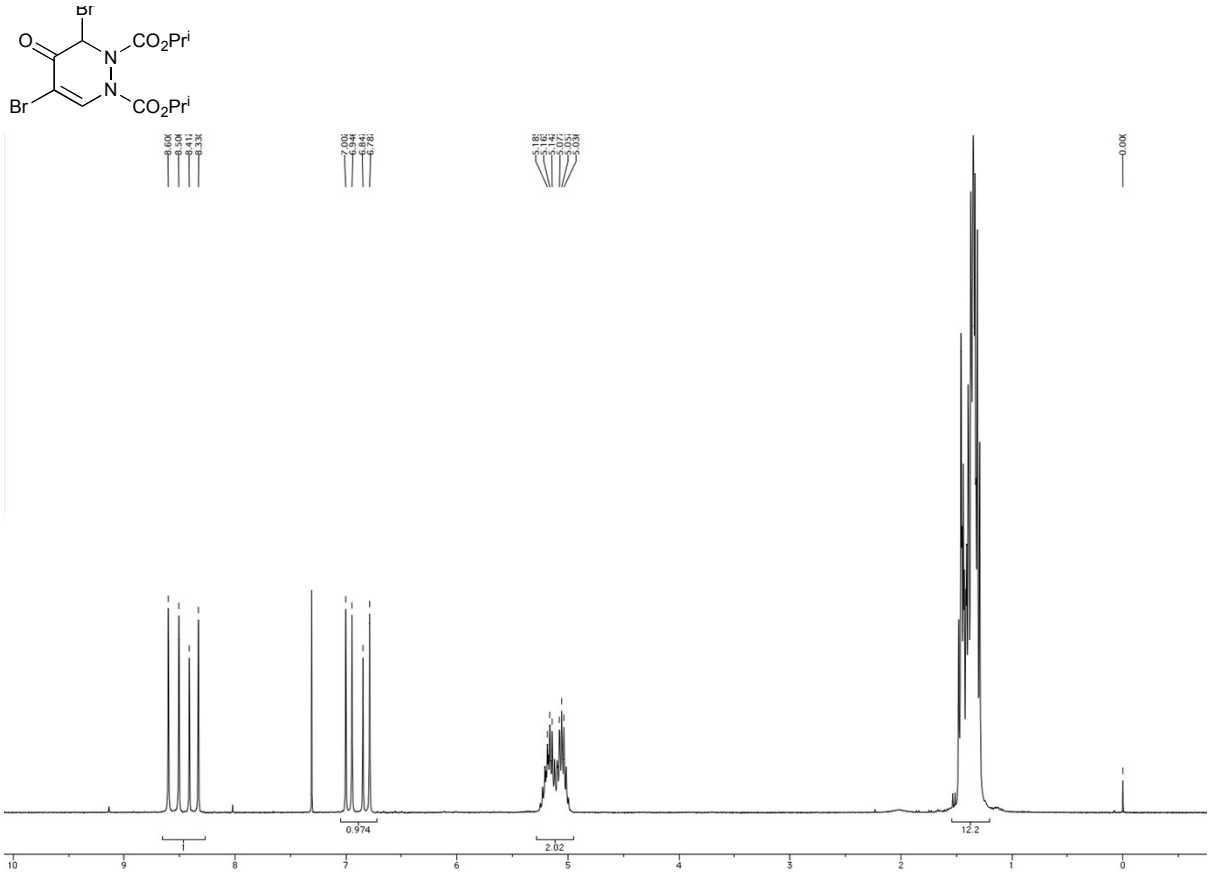
^{1}H NMR spectrum of **6** at 55 °C ($CDCl_3$)



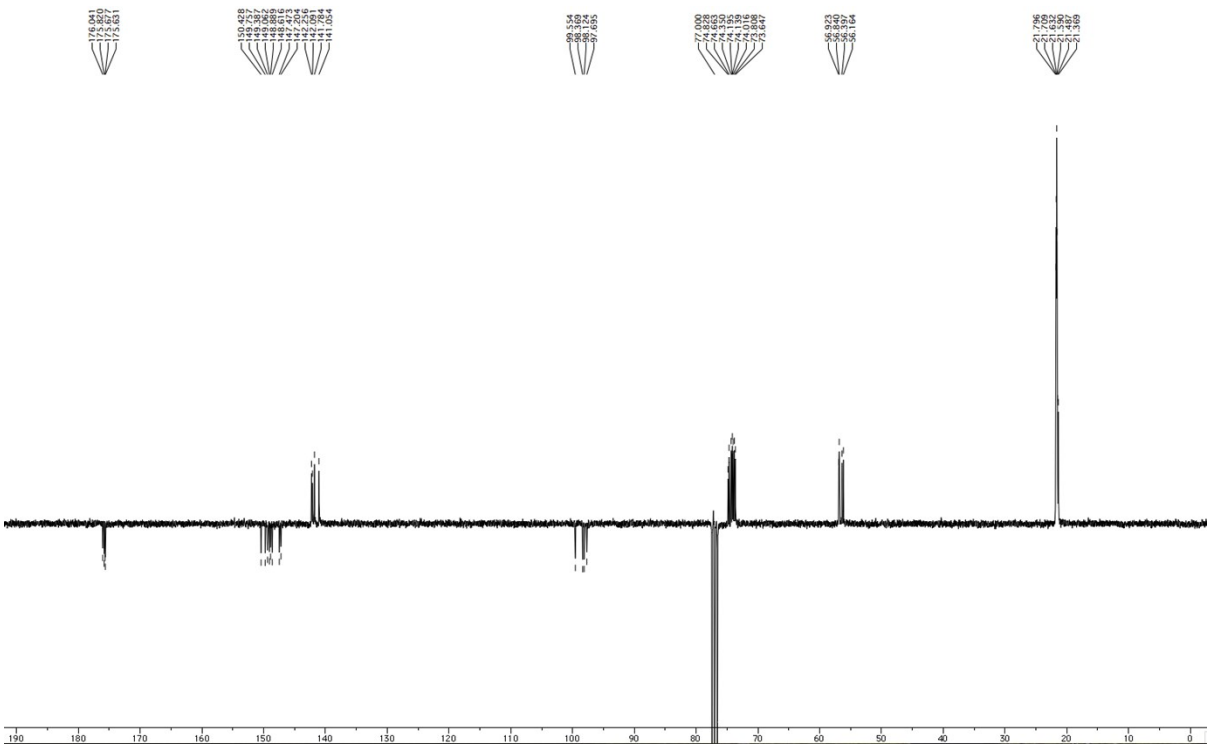
^{13}C DEPTQ NMR spectrum of **6** at 55 °C ($CDCl_3$)



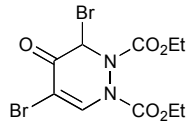
^{1}H NMR spectrum of **6** at –30 °C ($CDCl_3$)

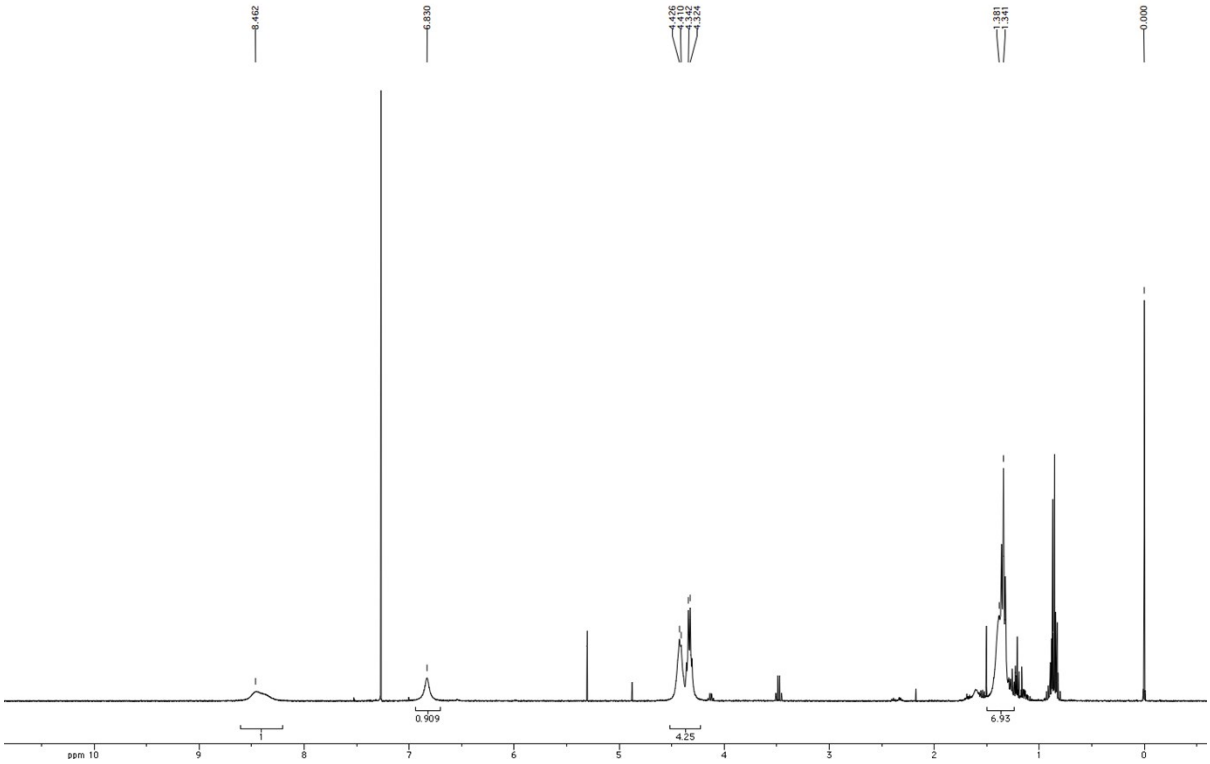


^{13}C DEPTQ NMR spectrum of **6** at –30 °C ($CDCl_3$)

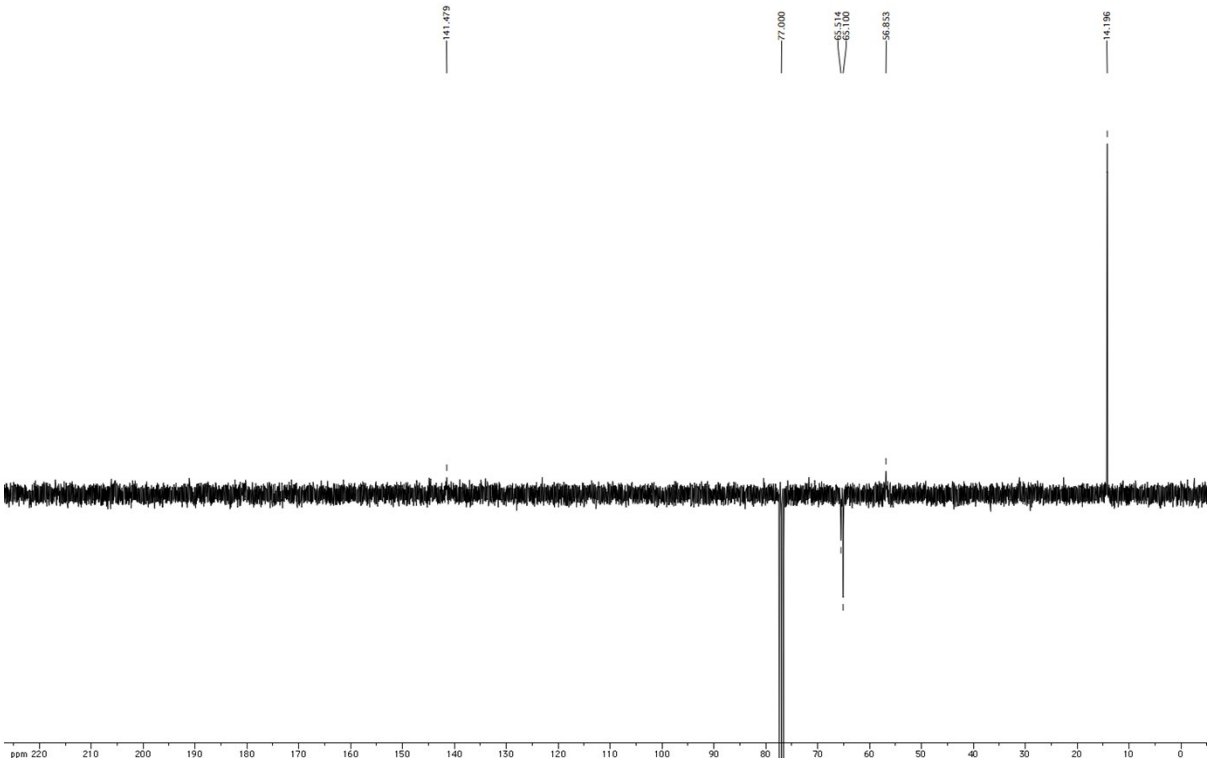


^{1}H NMR spectrum of **7** at 55 °C ($CDCl_3$)

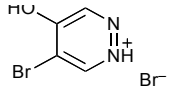


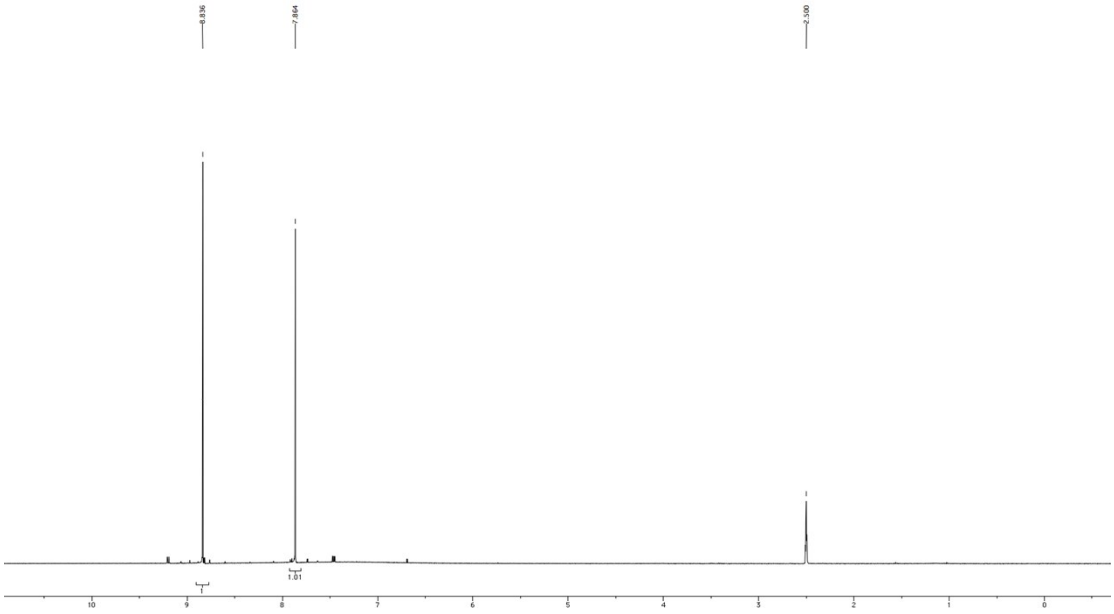


^{13}C DEPTQ NMR spectrum of **7** at 55 °C ($CDCl_3$)



^{1}H NMR spectrum of **11** (CD_3SOCD_3)





^{13}C DEPTQ NMR spectrum of **11** (CD_3SOCD_3)