Supporting Information for

A One-dimensional Array with Controlled Length from a Pybox Dimer with Flexible Oligo(sec-dialkylammioum cations)

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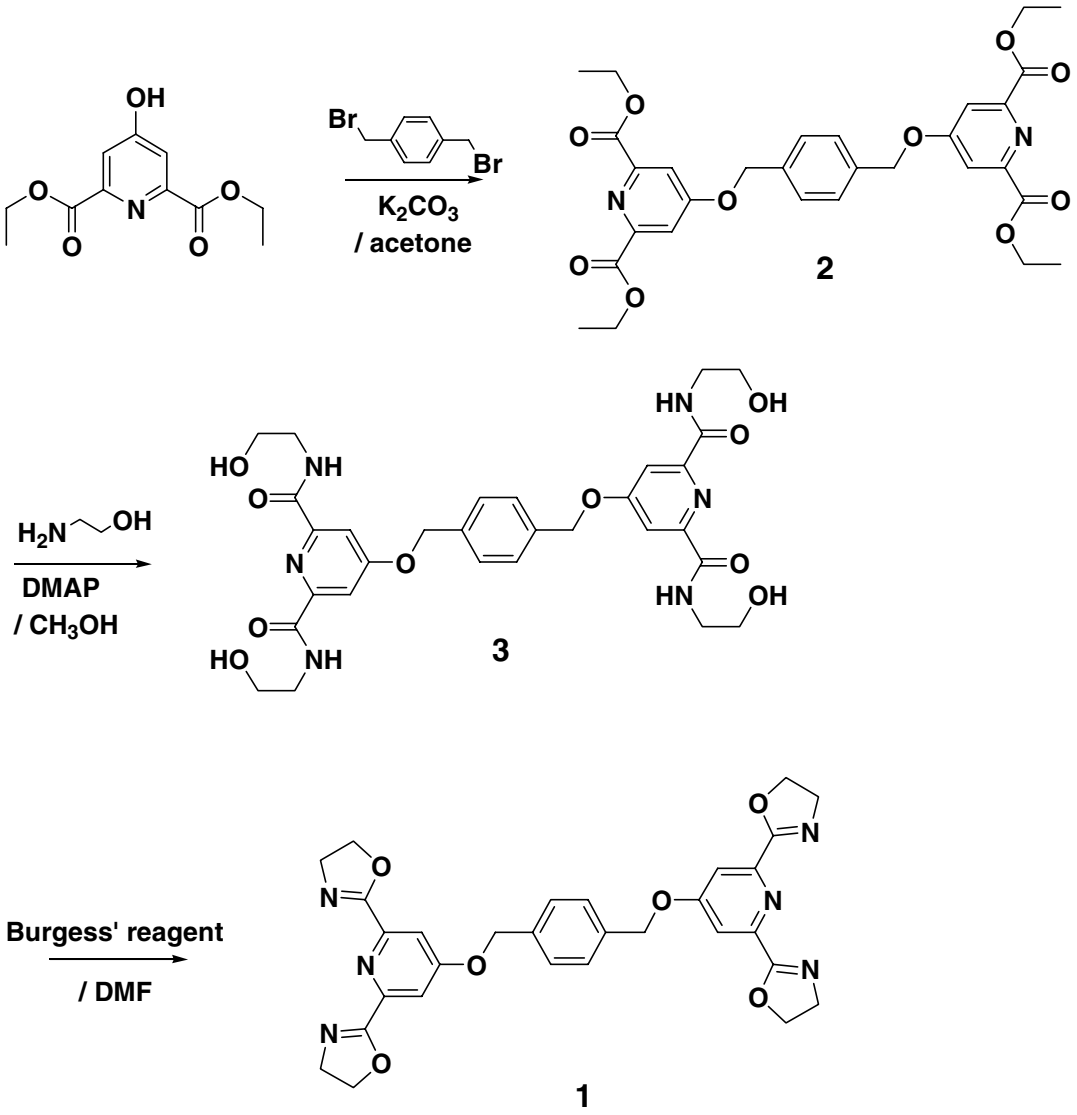
Preparation of 1.



General. Melting points were determined on a Micro Melting Point Apparatus (Yanaco MP-500D) and uncorrected. ^{1}H NMR spectra were measured on a Bruker DRX 600 spectrometer. MALDI-TOF Mass spectra were performed with a PerSeptive Voyager RP spectrometer. ESI mass spectra were performed with a PE Biosystems Mariner spectrometer.

1,4-Bis(2,6-bis(ethoxycarbonyl)-pyridin-4-yloxymethyl)benzene (**2**). A mixture of diethyl 4-hydroxy-pyridine-2,6-dicarboxylate[1] (3.63 g, 15.2 mmol), p-xylylene dibromide (1.10 g, 4.17 mmol), and well pulverized K_2CO_3 (2.09 g, 15.2 mmol) in 30 mL of dry acetone was refluxed for 12 h. The solvent was evaporated under reduced pressure and the residue was extracted with chloroform. The solution was washed

with 0.01 M hydrochloric acid, aqueous 5 % $NaHCO_3$, and water and then was dried over Na_2SO_4. After the solvent was removed under reduced pressure, the solid residue was further purified through chromatography (silica gel, CH_2Cl_2: MeOH = 45: 1 (*v/v*)) to give **3** in 94 % (2.29 g); mp 154.5-156.7 °C; ^{1}H NMR (250 MHz, $CDCl_3$, TMS, r.t.) 1.46 (t, *J* = 7.1, 12H), 4.48 (q, J = 7.1, 8H), 5.25 (s, 4H) 7.50 (s, 4H) 7.87 (s, 4H); MS [dithranol] m/z: calcd: 580.6, found: 581.7; Anal. Calcd for $C_{30}H_{32}N_2O_{10}\cdot 0.5\ H_2O$: C, 61.11; H, 5.64; N, 4.75; found: C, 61.14; H, 5.46; N, 4.73.

1,4-Bis(2,6-bis(2-hydroxy-ethylcarbamoyl)-pyridin-4-yloxymethyl)benzene (**3**). A suspension of **3** (3.00 g, 5.17 mmol), 4-(*N,N*-dimethylamino)pyridine (2.52 g, 20.7 mmol), and ethanolamine (30 mL) in 30 mL of methanol was refluxed for 48 h. The resulting precipitate was filtered off and then washed well with methanol to give **4** in 96 % (3.17 g); mp 292.0-292.8 °C; ^{1}H NMR (600 MHz, DMSO-d_6, TMS, r.t.) 3.41 (q, *J* = 6.2, 8H), 3.55 (q, *J* = 6.0, 8H), 4.83 (t, *J* = 5.4, 4H), 5.38 (s, 4H), 7.52 (s, 4H) 7.74 (s, 4H), 9.33 (t, *J* = 6.1, 4H); MS [dithranol] m/z: calcurated: 640.6, found: 641.8. This material was used without further purification.

1,4-Bis(2,6-bis(4,5-dihydro-oxazol-2-yl)-pyridin-4-yloxymethyl)benzene (**1**). A mixture of (**4**; 500 mg, 0.78 mmol), and Burgess reagent (1.10 g, 4.63 mmol) in 25 mL of dry DMF was warmed at 60 °C for 4 h. The solvent was evaporated under reduced pressure, and the solid residue was purified through chromatography (Merk alumina, activity III, CH_2Cl_2: MeOH = 40: 1 (*v/v*)) to give **1** in 52 % (230 mg); mp 201.2 °C(decomp.); ^{1}H NMR (600 MHz, $CDCl_3$, TMS, r.t.) d 4.11 (t, *J* = 9.7, 8H), 4.53 (t, *J* = 9.7, 8H), 5.22 (s, 4H), 7.46 (s, 4H), 7.77 (s, 4H); MS [dithranol] m/z: calcd: 568.6, found: 569.8.

Reference

(1) Froidevaux, P.; Harrowfield, J. M.; Sobolev, A. N. *Inorg. Chem.* **2000**, 39, 4678.

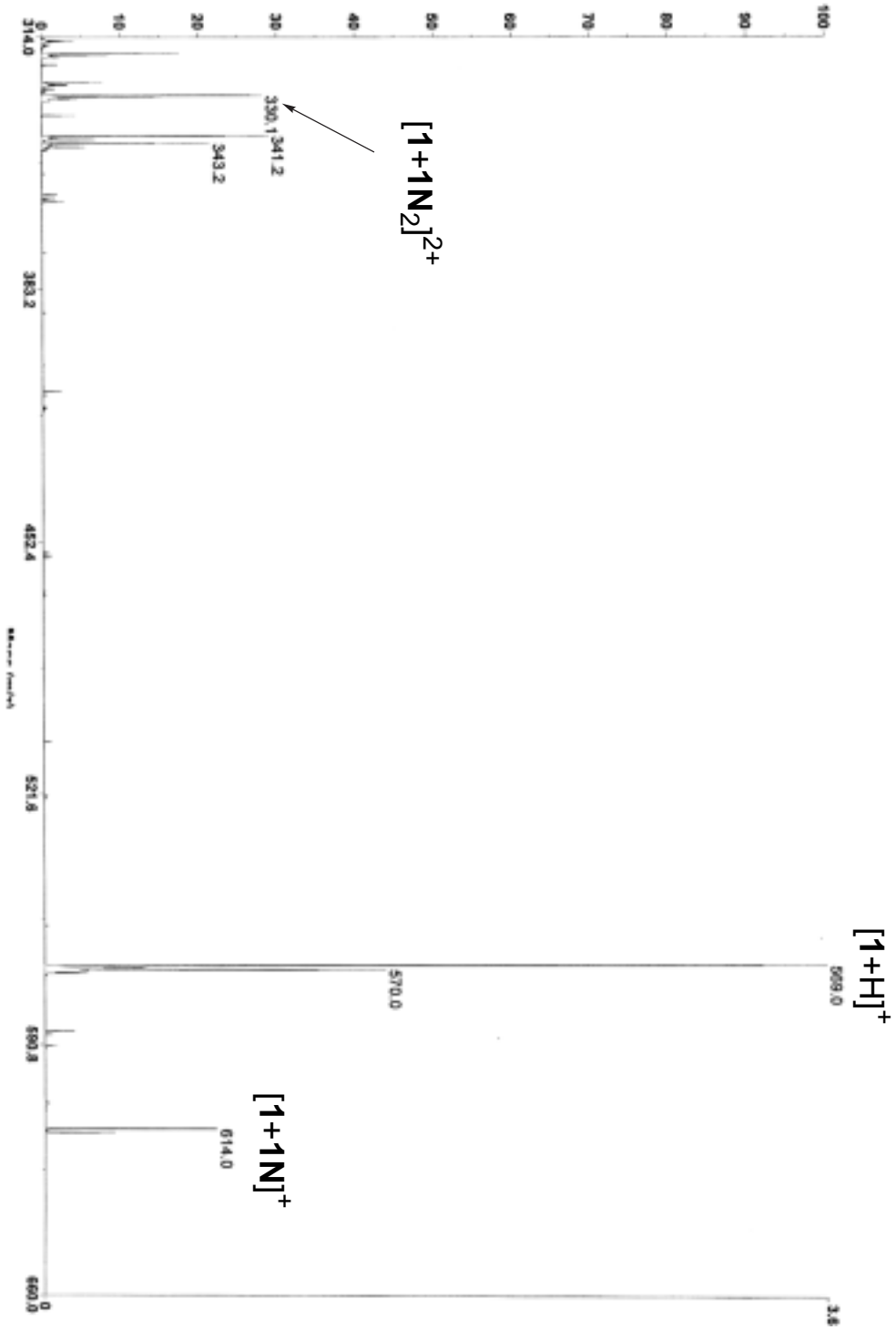


Figure S1 ESI mass spectrum of **1+1n** (1:2)

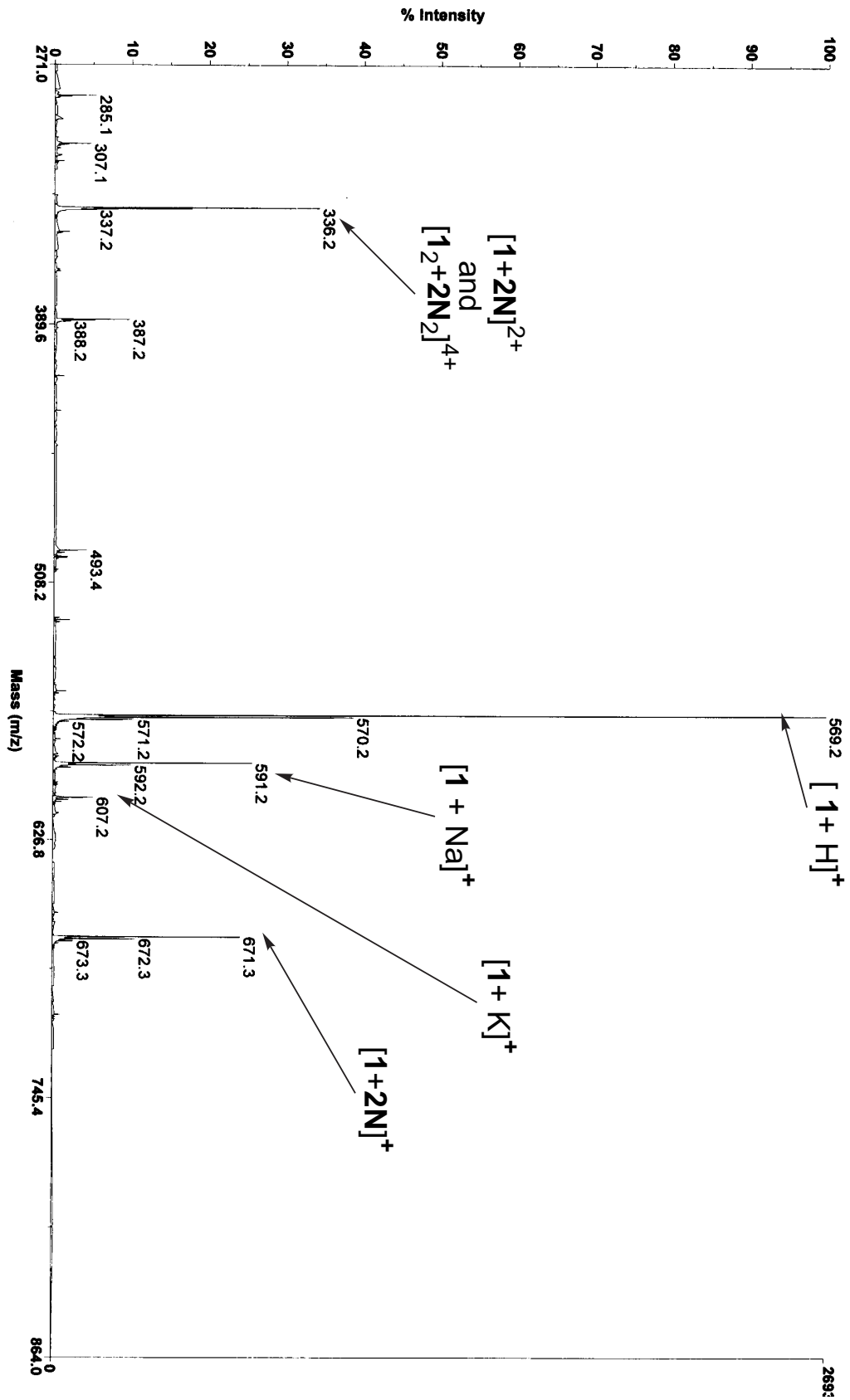


Figure S2 ESI mass spectrum of **1+2n** (1:2).

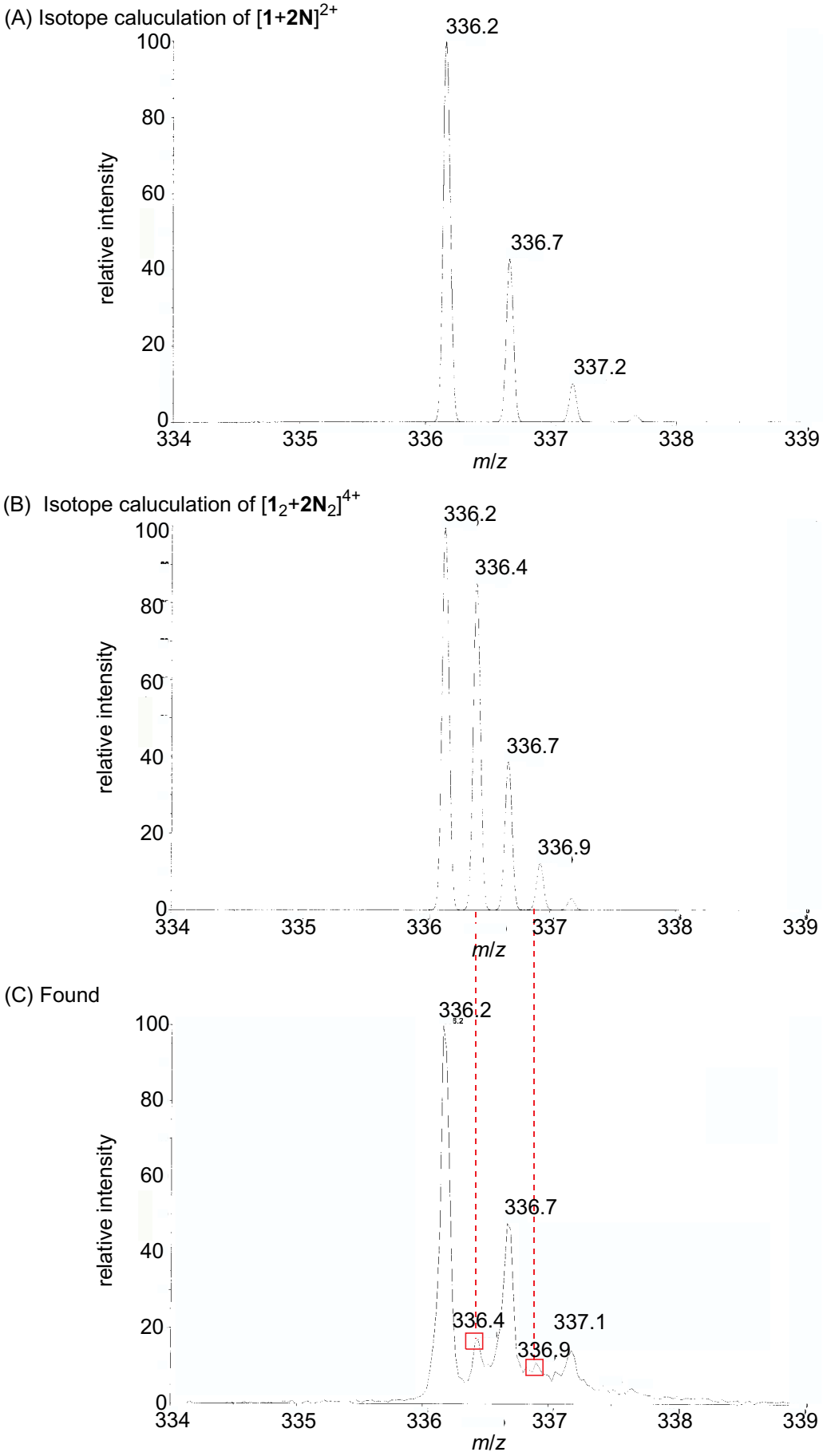


Figure S3 Detail of ESI mass spectrum of **1+2n** (1:1) around $[\mathbf{1+2n}]^{2+}$.

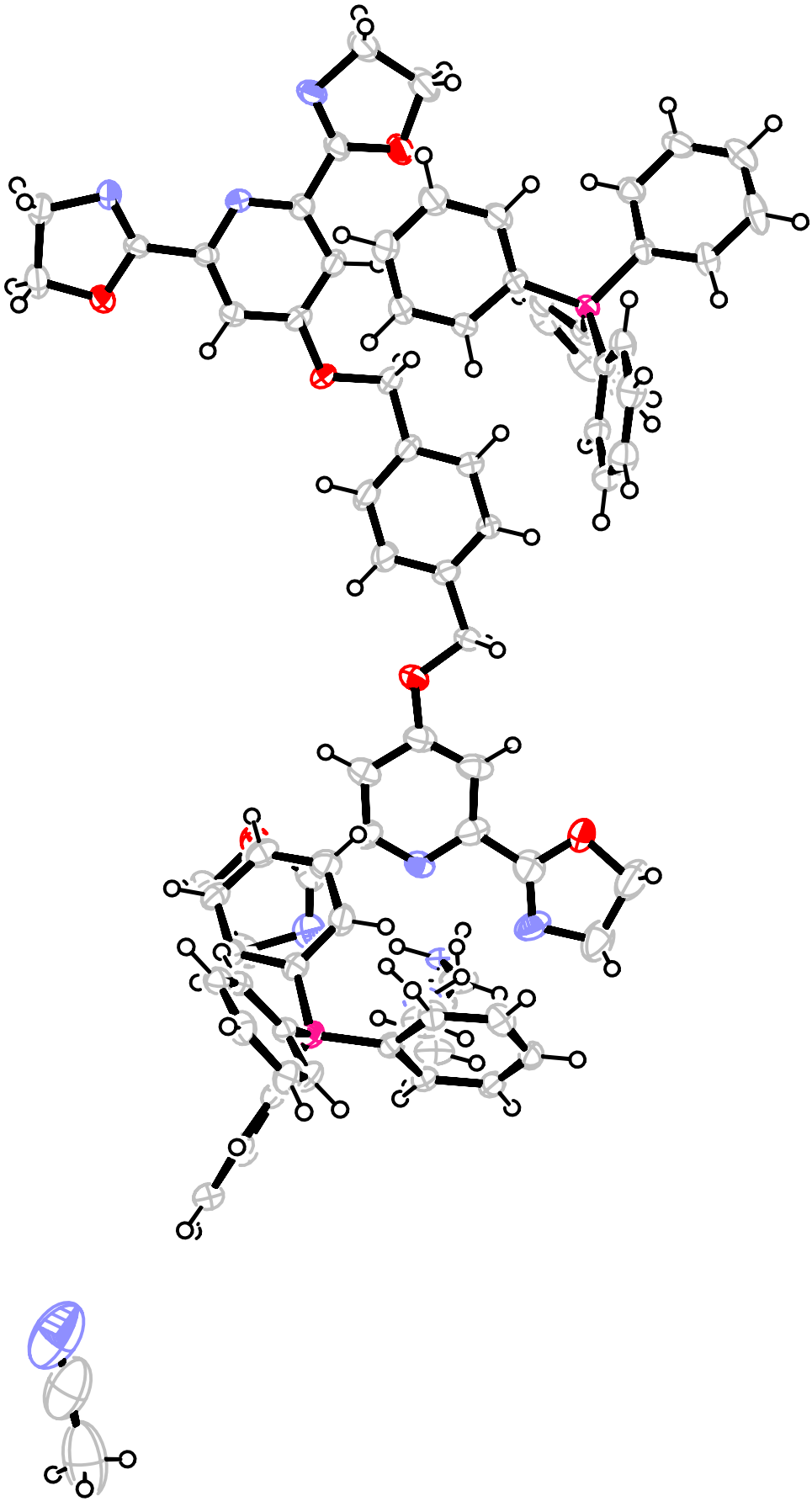


Figure S4 ORTEP drawing of **1+2n** (1:1).

^{1}H NMR titration