Supporting Information

CONFORMATIONAL CONTROL OF A PYRROLE-BASED AMIDE PENTAMER BY DIHYDROGEN PHOSPHATE ANION BINDING

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CHART OF CONTENTS

Structural studies

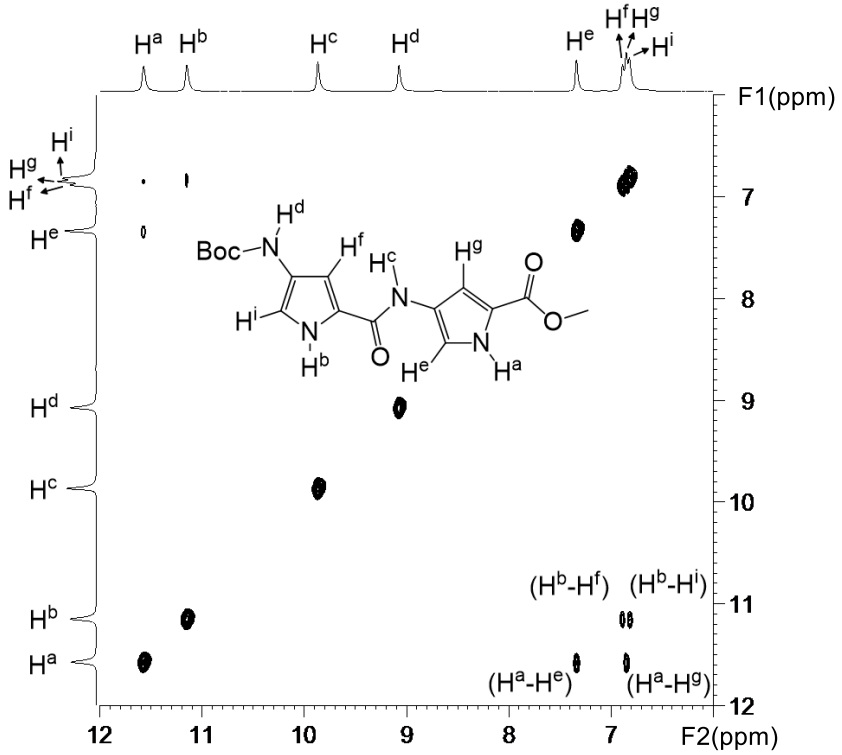


Figure S1. Part of the ^{1}H-^{1}H COSY spectra (400MHz, 298K) of **4** (10 mM) in DMSO-d_6.

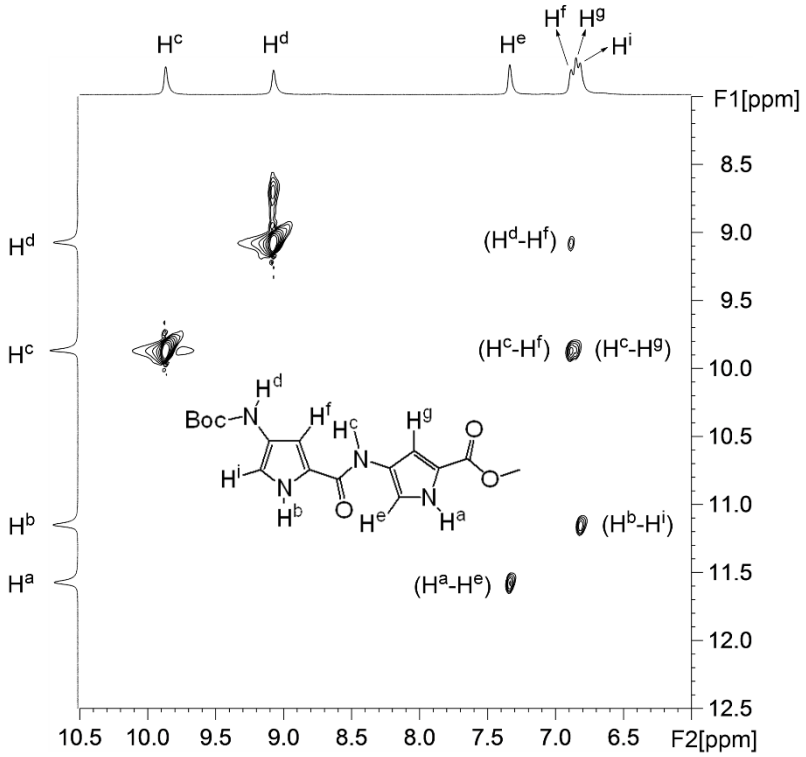


Figure S2. Part of the 2D NOESY NMR spectrum (400 MHz, 298 K) of **4** (10 mM) in DMSO-d_6.

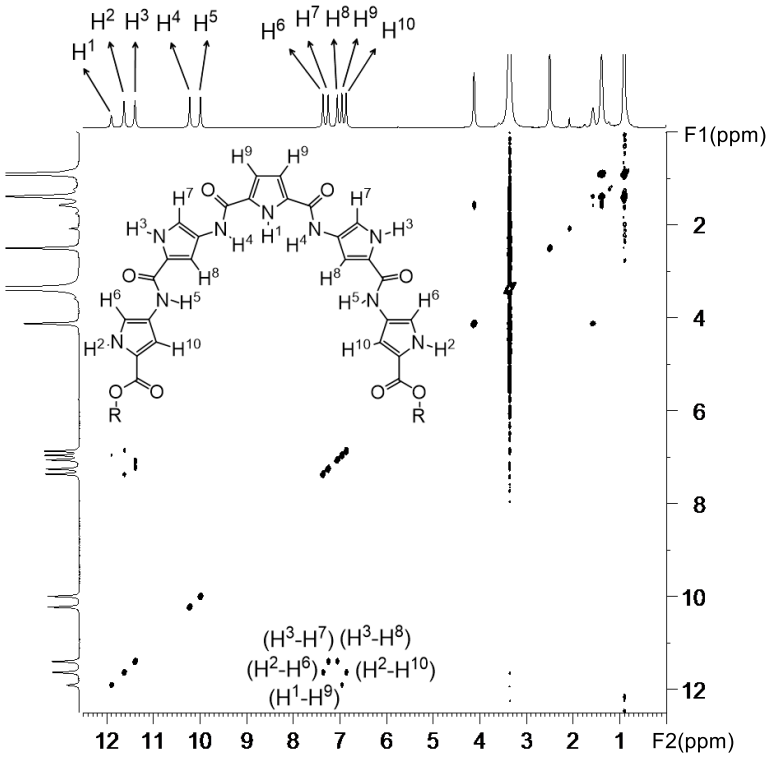


Figure S3. ^{1}H-^{1}H COSY spectra (400MHz, 298K) of **1** (10 mM) in DMSO-d_6 (R=C_6H_{13}).

The detection of various anions of tetrabutylammonium salts was determined by ^{1}HNMR spectroscopy in DMSO-d_6.

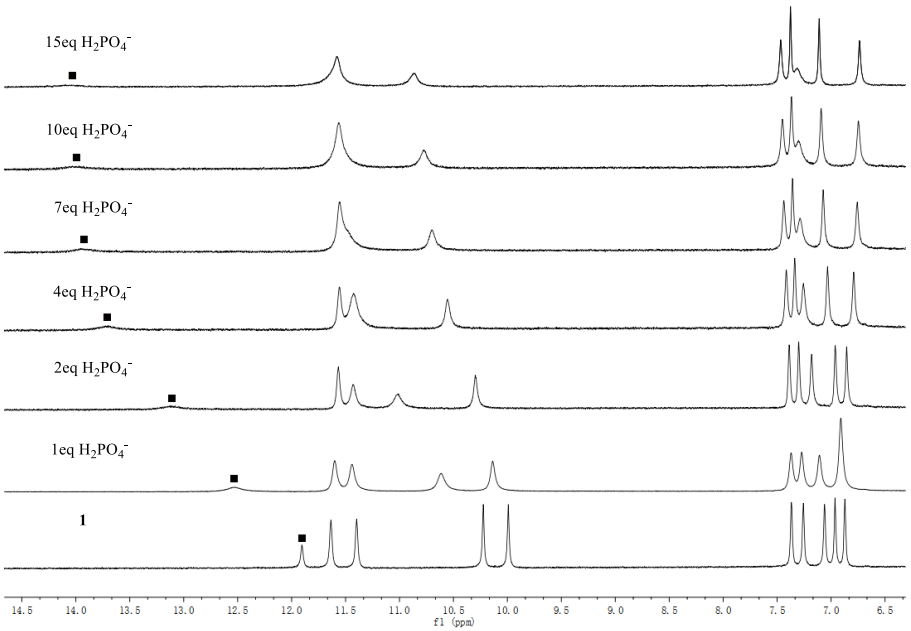


Figure S4. Partial ^{1}H NMR spectra (400 MHz, 298 K) changes of **1** (1 mM) in DMSO-d_6 upon titration with nBu$_4$N$^+$H$_2$PO$_4^-$.

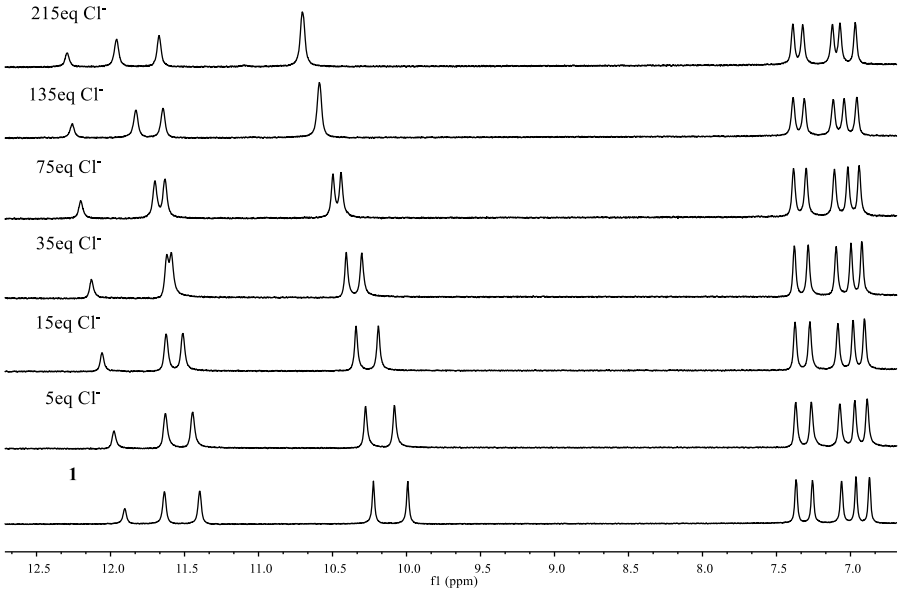


Figure S5. Partial ^{1}H NMR spectra (400 MHz, 298 K) changes of **1** (1 mM) in DMSO-d_6 upon titration with nBu$_4$N$^+$Cl$^-$.

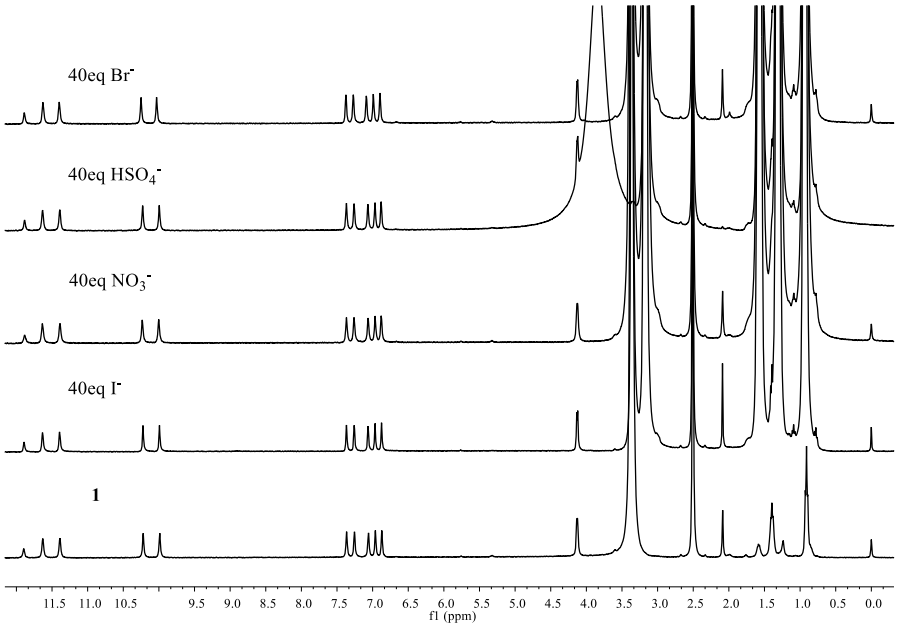


Figure S6. Partial ^{1}H NMR spectra (400 MHz, 298 K) changes of **1** (1 mM) in DMSO-d_6 upon titration with 40 eq bisulfate, nitrate, bromide and iodine, respectively. The ^{1}H chemical shift of **1** were not more than 0.03 ppm.

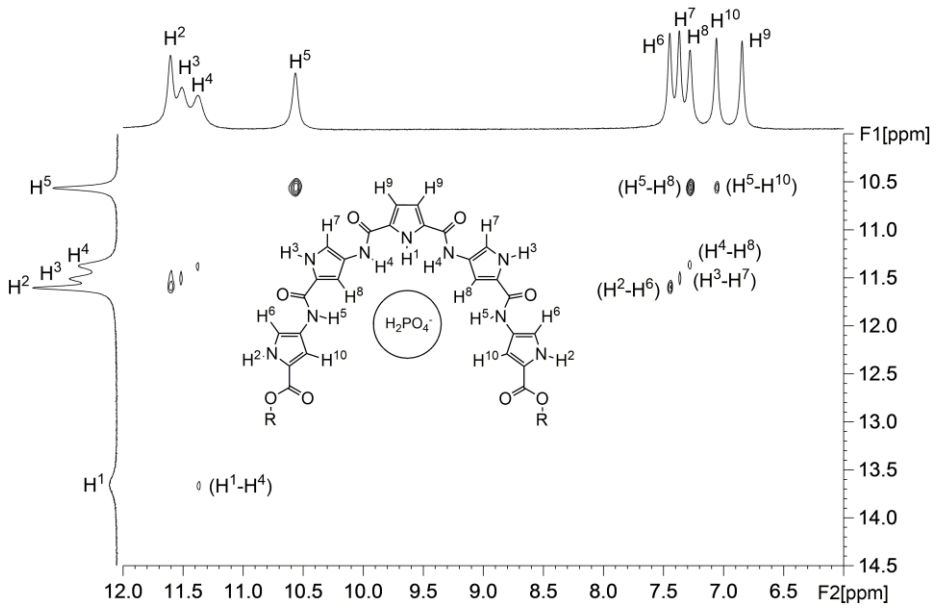


Figure S7. Part of the 2D NOESY NMR spectrum (400 MHz, 298 K) of **1** (1 mM) in DMSO-d_6 upon addition with 3 eq nBu$_4$N$^+$H$_2$PO$_4^-$ (R=C$_6$H$_{13}$).

X-Ray crystallography

Single crystal compound **4** was grown by slow diffusion of n-hexane in its ethyl acetate solutions. A single crystal of compound **4** in mother liquor was pipetted onto a glass containing Paratone-N oil. The crystal was quickly mounted onto a nylon loop and immediately flash cooled in liquid nitrogen.

Crystallographic data were all collected at the the Analysis and Testing Center in Huazhong University of Science and Technology (HUST) on a Rigaku MM007 HF rotating anode (0.8 kW). Data were diffracted at the CuK**α** wavelength, and data-collection strategies were based on Omega scans at 100(2) K. The Rigaku CrystalClear suite versions 2.0 were used to index, integrate and scale the data with a multi-scan absorption correction.

The structures were solved by direct methods using SHELXT[S1] and refined against F^2 on all data by full-matrix least squares with SHELXL[S2] following established refinement strategies.[S3] The non-H atoms were refined with anisotropic temperature parameters. All hydrogen atoms, were included into the model at geometrically calculated positions and refined using a riding model. Crystallographic data have been deposited with the CCDC, under deposition number CCDC 1891957, compound **4**.

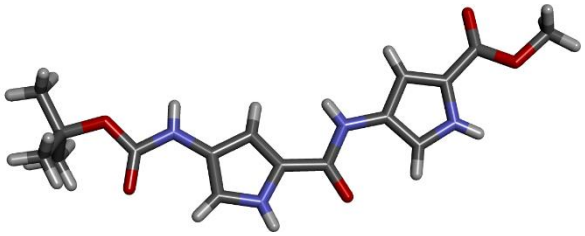


Figure S8. Crystal structure of compound **4**.

Table S1: Crystal data and structure refinement for the interpenetrated helicate

Formula	C16 H20 N4 O5
M	348.36
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	23.2765(3)
b/Å	6.93108(7)
c/Å	10.15585(11)
α/°	90
β/°	96.7519(10)
γ/ °	90
V/Å^3	1627.10(3)
T /K	100.01(10)
Z	4
ρ/g cm^{-1}	1.422
size (mm)	0.02 × 0.01 × 0.01
λ/ Å	1.54184
μ/mm^{-1}	0.901
Independent reflections	3208
measured reflections	9545
parameters/restraints	230/0
*R*1, *wR*2	0.0353/0.0931
goodness of fit	1.062

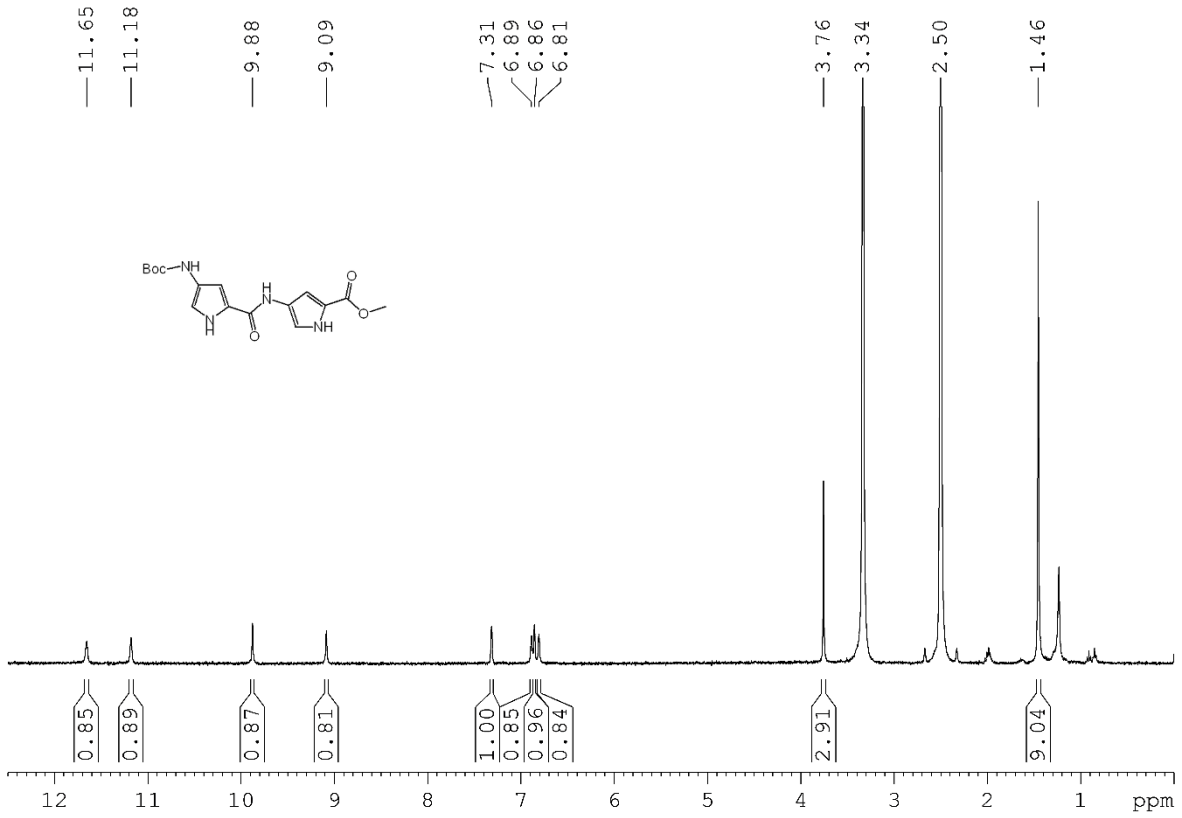
References

S1. G. M. Sheldrick, *Acta Crystallogr., Sect. A: Found. Adv.*, 2015, **71**, 3.

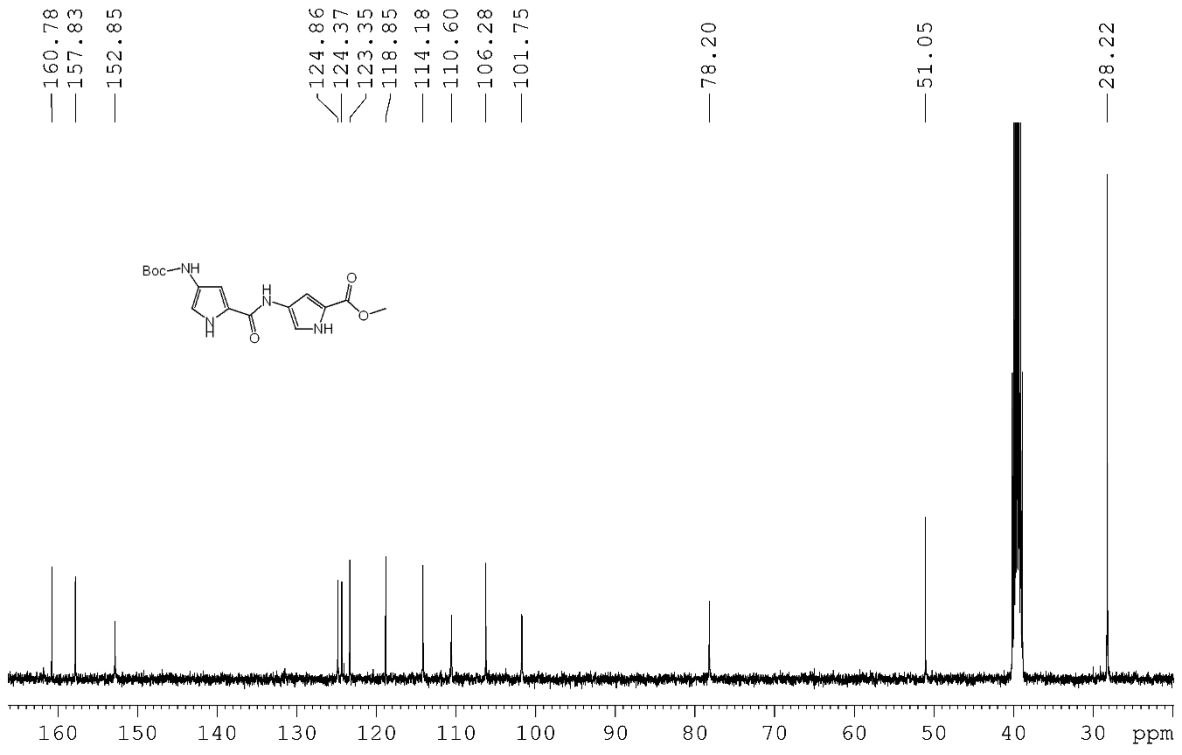
S2. G. M. Sheldrick, *Acta Crystallogr., Sect. C: Cryst. Struct. Commun.,* 2015, **71**, 3.

S3. P. Müller, *Cryst. Rev.*, 2009, **15**, 57.

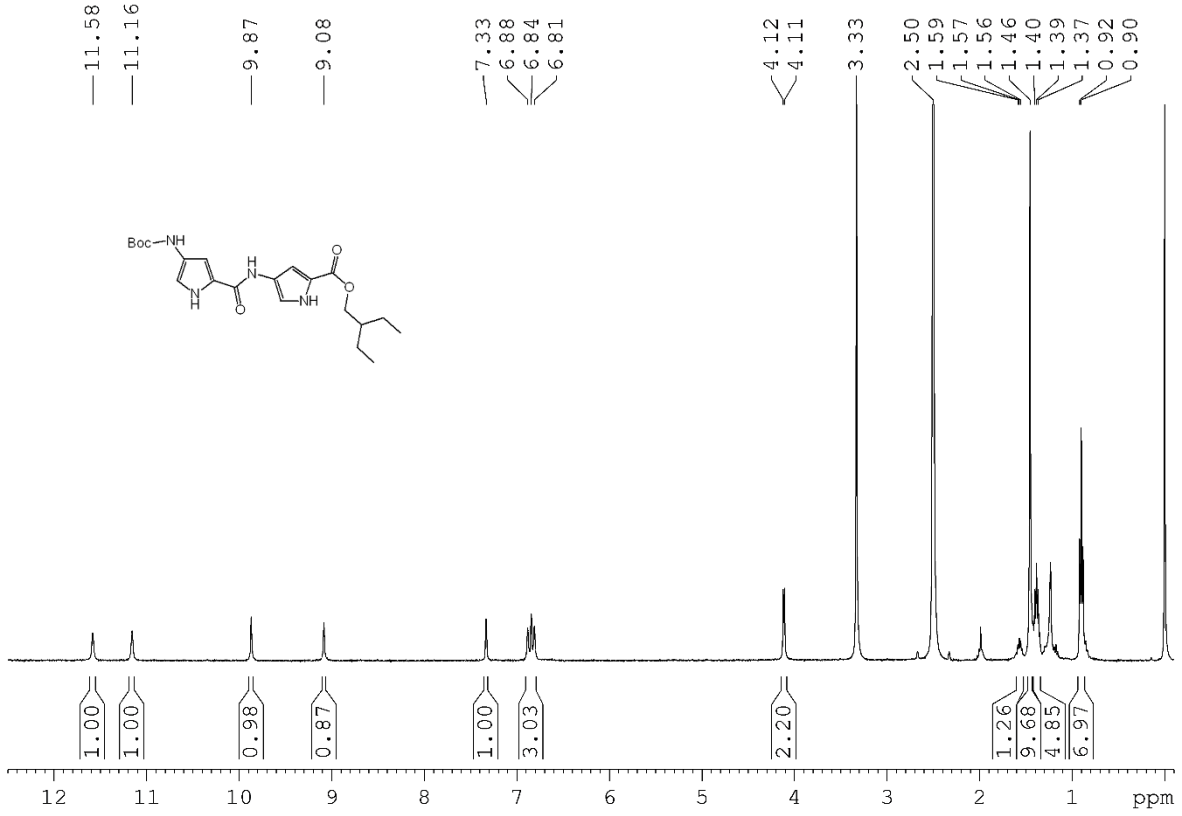
Copies of ^{1}H and ^{13}C NMR spectra



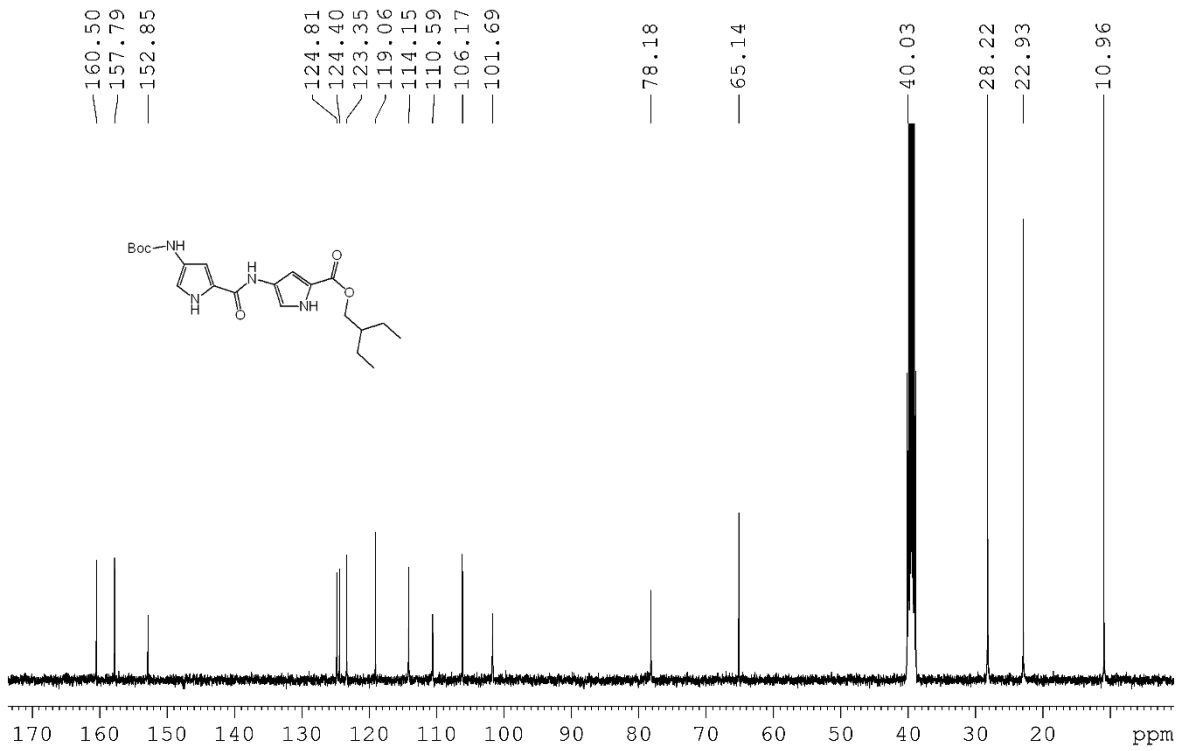
^{1}H spectra of compound **4**



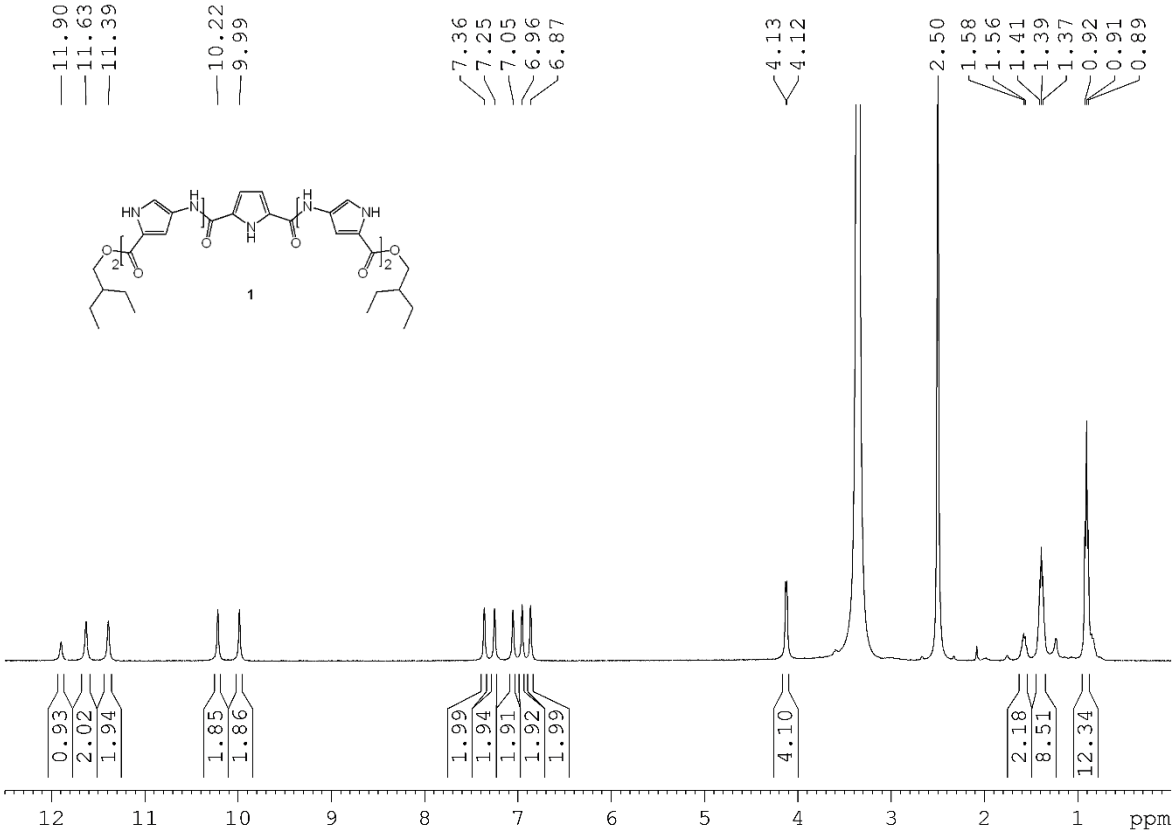
^{13}C spectra of compound **4**



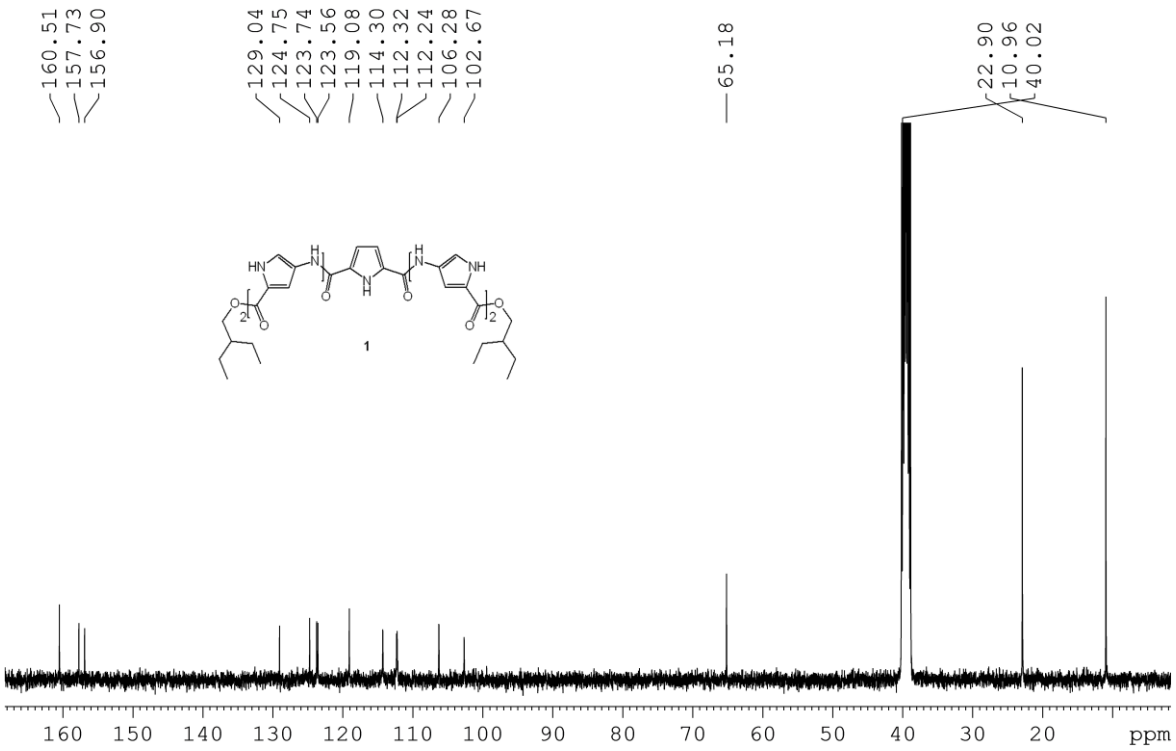
^{1}H spectra of compound **5**



^{13}C spectra of compound **5**



^{1}H spectra of compound **1**



^{13}C spectra of compound **1**