

Supplementary Information

Anion Receptors with Nitron C-H Hydrogen Bond Donors

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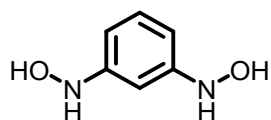
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S.1 General methods

All commercially available reagents and solvents were used without purification. ^1H NMR (400 MHz and 500 MHz) and ^{13}C NMR (100 MHz and 125 MHz) were recorded on a Bruker AVIII-400 MHz Nanobay and Bruker AVIII 500 MHz at rt (298 K). Chemical shifts (δ) were referenced to tetramethylsilane (TMS) or residual solvent peaks, chloroform (7.26 ppm for ^1H NMR, 77.16 ppm for ^{13}C NMR), acetone- d_6 (2.05 ppm for ^1H NMR, 29.84 ppm for ^{13}C NMR), DMSO- d_6 (2.50 ppm for ^1H NMR, 39.52 ppm for ^{13}C NMR), acetonitrile- d_3 (1.94 ppm for ^1H NMR, 1.32 ppm for ^{13}C NMR). High resolution electrospray ionization (ESI) mass spectrometry was performed on Agilent 6230 TOF LC/MS. Column chromatography was performed on the Biotage Isolera One system. Empty flash cartridge housing from Luknova were filled and packed with Silicycle F60 silica gel (40 – 63 μm , 60 \AA) for column chromatography purifications.

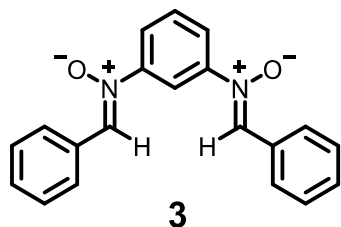
S.2 Synthesis and Characterization of Compounds



2

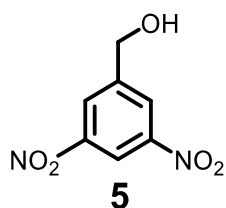
***m*-Phenylenedihydroxylamine (2)**: Synthesized by following a reported procedure using Rh/C.¹ To a mixture of 1,3-dinitrobenzene (0.15 g, 0.89 mmol) and rhodium 5% on carbon (Rh/C) (11 mg, 0.0054 mmol Rh, 0.006 equivalents) in THF (3 mL) was slowly added hydrazine monohydrate (0.107 g, 2.14 mmol, 2.4 equiv.) and stirred at 0 °C under N₂ atmosphere for 1 hour. The reaction was monitored by TLC. The reaction mixture was filtered through Celite to remove the residue of Rh/C. The filtrate was dried using a rotary evaporator and then high vacuum to provide a yellow-brown solid (0.107g 0.76 mmol, 86%) without further purification. ^1H NMR (400 MHz, Acetone- d_6): δ = 7.57 (s, 2H), 7.51 (s, 2H), 6.99 (t, J = 8 Hz, 1H), 6.66 (t, J = 2 Hz, 1H), 6.43 (dd, J = 8, 2 Hz, 2H). ^{13}C NMR

(125 MHz, Acetone- d_6): $\delta = 153.5, 129.3, 106.7, 99.9$. ESI: $C_6H_9N_2O_2$ $[M+H]^+$ calcd 141.06585, found 141.06581.

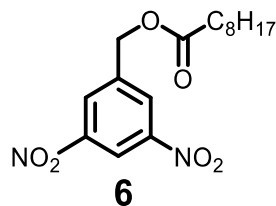


Dinitrone 3: Dihydroxylamine **2** (0.107 g, 0.77 mmol) and benzaldehyde (0.406 g, 3.83 mmol, 5 equiv) were dissolved with 5 mL ethanol in a vial. The reaction mixture was stirred at room temperature under N_2 atmosphere overnight. The milky suspension

was generated next day. 10 mL hexane was added to the milky suspension and then sonicated for 15 minutes. The mixture was filtered using a membrane filter and thoroughly washed with hexane (5 mL \times 3). The solid mixture was collected and transferred to a vial and dried completely under high vacuum to provide a light yellow solid product (90 mg, 0.28 mmol, 36%). 1H NMR (400 MHz, DMSO- d_6): $\delta = 8.66$ (s, 2H), 8.52-8.50 (m, 5H), 8.09 (dd, $J = 8, 2$ Hz, 2H), 7.74 (t, $J = 8, 1.03$ H), 7.55-7.51 (m, 6H). ^{13}C NMR (125 MHz, DMSO- d_6): $\delta = 148.7, 134.2, 130.9, 130.9, 129.9, 129.0, 128.5, 122.8, 115.1$. ESI: $C_{20}H_{17}N_2O_2$ $[M+H]^+$ calcd 317.12845, found 317.12842.

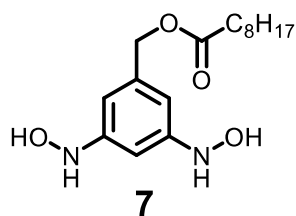


3,5-Dinitrobenzyl alcohol (**5**) was prepared from 3,5-Dinitrobenzoic acid following the published method.² The 1H NMR and ^{13}C NMR spectra matched that of the published results.²

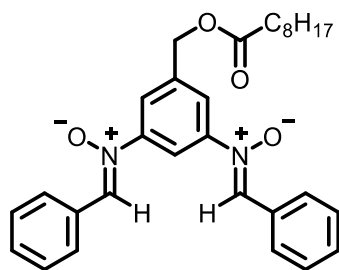


Compound 6: A solution of nonanoic acid (1.64 g, 11.4 mmol, 1.5 equiv.) and thionyl chloride (5.4 g, 45.4 mmol, 6 equiv.) in chloroform (8 mL) was stirred at 70 $^\circ C$ for 6 hours. Chloroform and excess thionyl chloride were removed by distillation to give nonanoyl chloride, which was used in the next step without

further purification. To a solution of compound **5** (1.5 g, 7.57 mmol) and triethyl amine (4.2 mL, 30 mmol, 4 equiv.) in DCM (50 mL) was dropwise added nonanoyl chloride (11.4 mmol) at 0 °C. The reaction was warmed to room temperature and stirred under nitrogen atmosphere for 16 hours. The reaction was quenched with water. The reaction mixture was extracted with DCM (3 × 50 mL) and the organic phase was washed with brine (1 × 50 mL). The organic phase was collected and dried with MgSO₄, filtered and dried by rotary evaporation. The crude mixture was purified by column chromatography (SiO₂, hexane:ethyl acetate 10:1) yielding the product as a light yellow solid (2.25 g, 6.66 mmol, 88%). ¹H NMR (400 MHz CDCl₃): δ = 9.00 (t, *J* = 2 Hz, 1H), 8.54 (d, *J* = 2 Hz, 2H), 5.30 (s, 2H), 2.44 (t, *J* = 8 Hz, 2H), 1.71-1.64 (m, 2H), 1.35-1.27 (m, 10H), 0.87 (t, *J* = 8, 3H). ¹³C NMR (125 MHz, CDCl₃): δ = 173.2, 148.7, 141.0, 127.8, 118.4, 63.7, 34.1, 31.8, 29.25, 29.18, 29.16, 24.9, 22.7, 14.1. ESI: C₁₆H₂₂N₂O₆ [M]⁻ calcd 338.14834, found 338.14695.



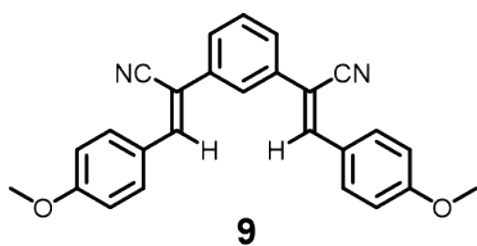
Dihydroxylamine 7: To a mixture of compound **6** (0.3 g, 0.89 mmol) and rhodium 5% on carbon (Rh/C) (18 mg, 0.0089 mmol, 0.01 equiv.) in THF (3 mL) was slowly added hydrazine monohydrate (0.11 mL, 2.2 mmol, 2.5 equiv.) and stirred at 0 °C under nitrogen atmosphere for 1 hour. The reaction was monitored by TLC. The reaction mixture was filtered through Celite. The filtrate was dried using a rotary evaporator, followed by high vacuum to provide **7** as a yellow solid (0.26g 0.84 mmol, 94%). ¹H NMR (400 MHz, Acetone-*d*₆): δ = 7.63 (s, 2H), 7.59 (s, 2H), 6.61 (s, 1H), 6.45 (s, 2H), 4.97 (s, 2H), 2.33 (t, *J* = 8 Hz, 2H), 1.64-1.58 (m, 2H), 1.34-1.27 (m, 10H), 0.87 (t, *J* = 4 Hz, 3H). ¹³C NMR (125 MHz Acetone-*d*₆): δ = 173.6, 153.5, 138.0, 106.1, 99.3, 66.7, 34.6, 32.5, 29.9, 29.8, 29.8, 25.6, 23.2, 14.3. ESI: C₁₆H₂₆N₂O₄ [M+H]⁺ calcd 311.19653, found 311.19631.



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Dinitrone 8: Dihydroxylamine **7** (0.088 g, 0.28 mmol) and benzaldehyde (0.14 mL, 1.42 mmol, 5 equiv.) were dissolved with 2 mL ethanol in a vial. The reaction mixture was stirred at room temperature under nitrogen atmosphere overnight. The solvent was removed by rotary evaporation. The crude mixture was purified by

column chromatography (SiO₂, hexane:ethyl acetate 2.5:1) yielding the product as blown sticky oil (76 mg, 0.16 mmol, 57%). ¹H NMR (400 MHz Acetone-*d*₆): δ = 8.56-8.54 (m, 4H), 8.52 (s, 2H), 8.43 (s, 1H), 8.10 (s, 2H), 7.51-7.50 (m, 6H), 5.29 (s, 2H), 2.43 (t, *J* = 8 Hz, 2H), 1.67-1.61 (m, 2H), 1.35-1.25 (m, 10H), 0.85 (t, *J* = 4 Hz, 3.00H). ¹³C NMR (125 MHz Acetone-*d*₆): δ = 173.5, 150.4, 140.3, 134.7, 132.2, 131.6, 129.9, 129.3, 122.7, 115.3, 65.1, 34.5, 32.5, 30.0 (overlap with the solvent peak), 29.9, 29.8, 25.6, 23.3, 14.3. ESI: C₂₀H₃₄N₂O₄ [M+H]⁺ calcd 487.25913, found 487.25850.



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Dicyanostilbene (**9**) was prepared from the condensation between *p*-Anisaldehyde and 1,3-Phenylenediacetonitrile following the published method.³ The ¹H NMR and ¹³C NMR spectra matched that of the published results.³

S.3 NMR Spectra of Compounds

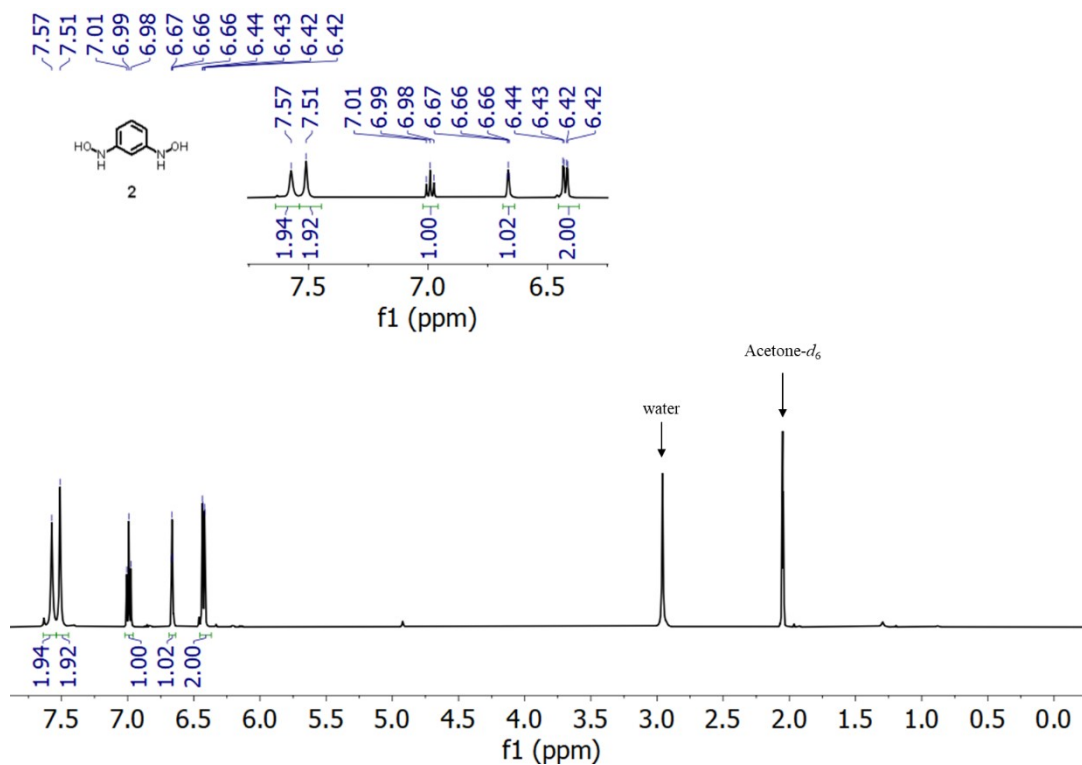


Figure S1. ¹H NMR spectrum of **2** (400 MHz, Acetone-*d*₆).

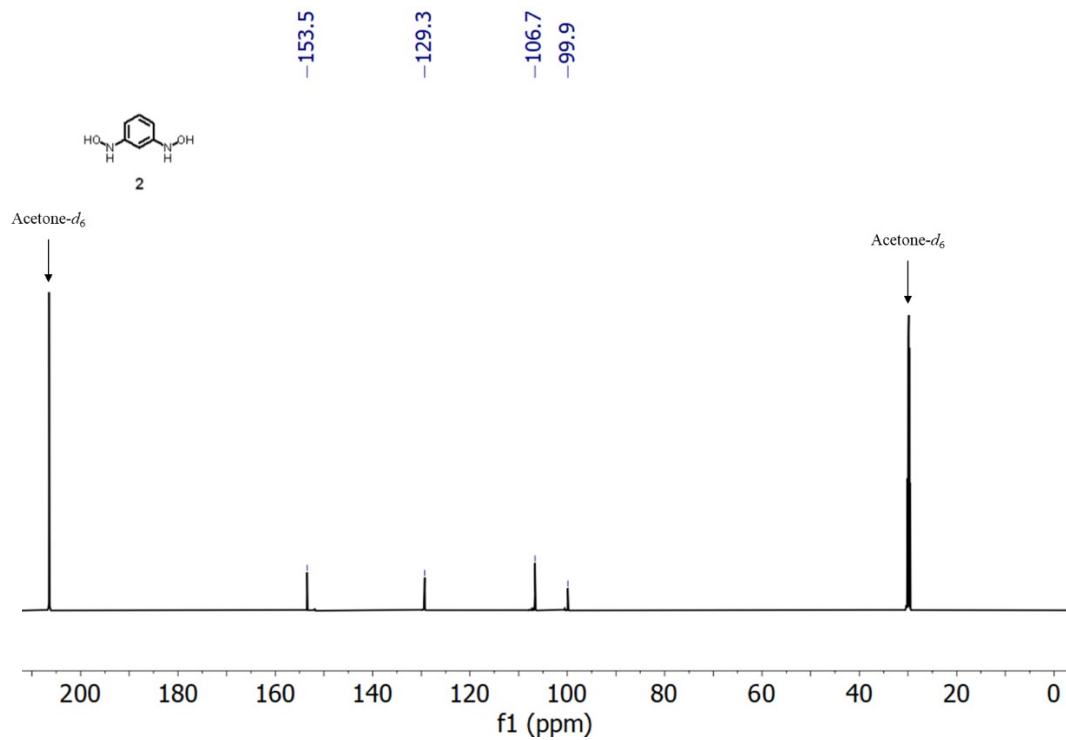


Figure S2. ¹³C NMR spectrum of **2** (125 MHz, Acetone-*d*₆).

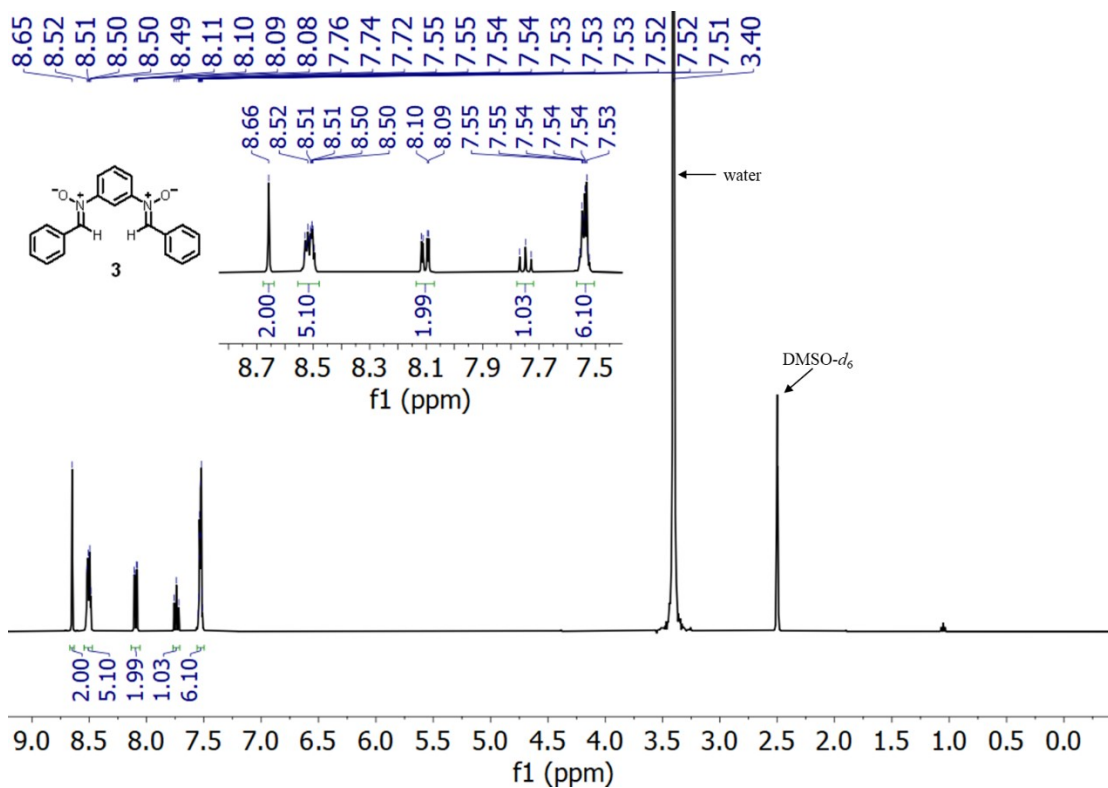


Figure S3. ^1H NMR spectrum of **3** (400 MHz, $\text{DMSO-}d_6$).

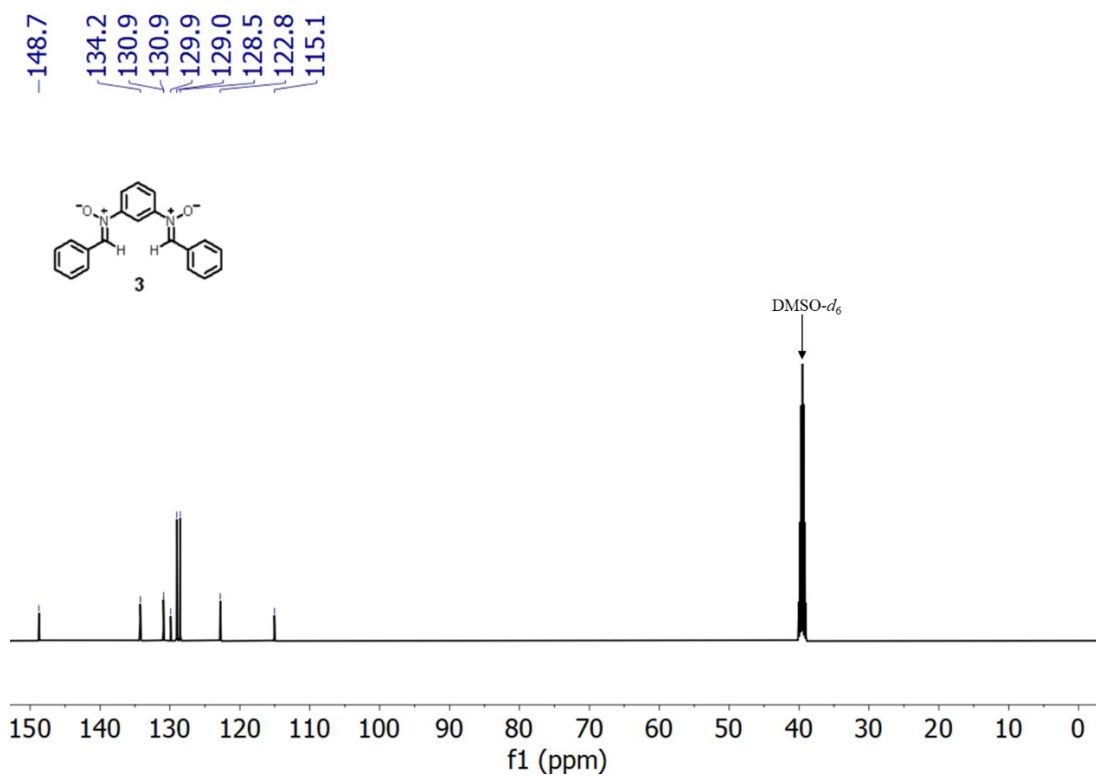


Figure S4. ^{13}C NMR spectrum of **3** (125 MHz, $\text{DMSO-}d_6$).

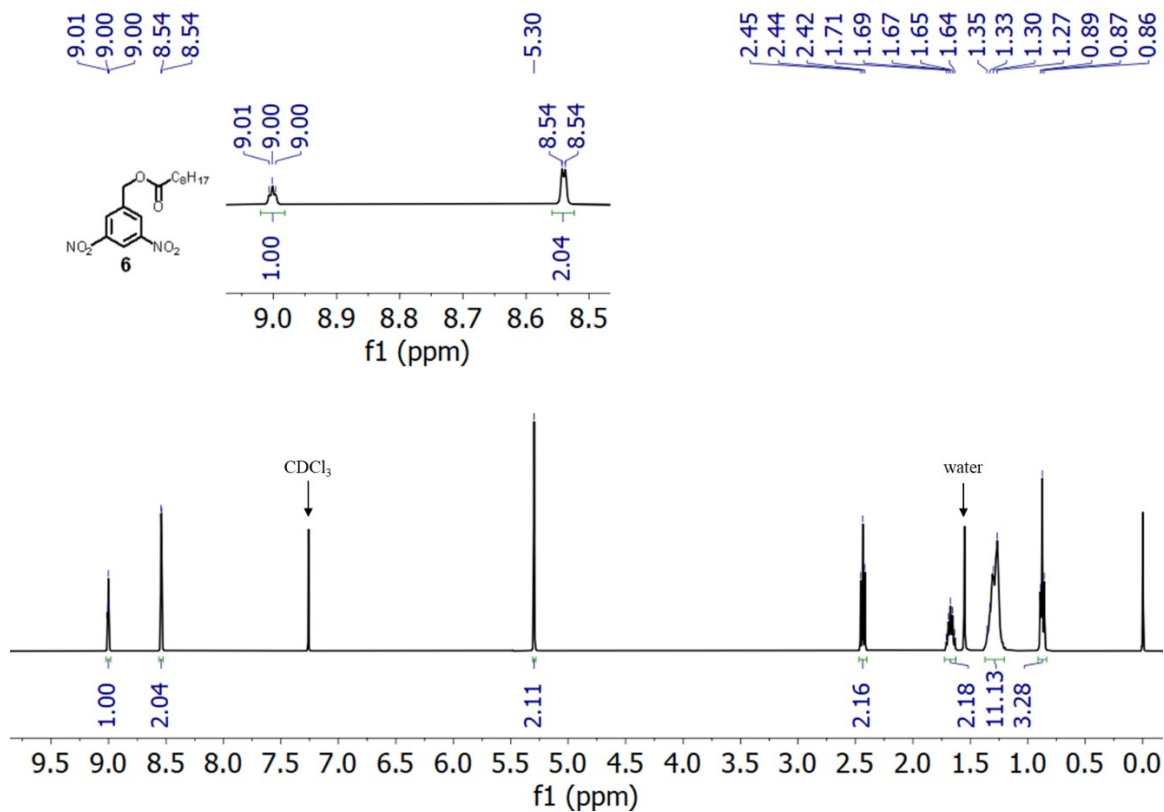


Figure S5. ¹H NMR spectrum of **6** (400 MHz, CDCl₃).

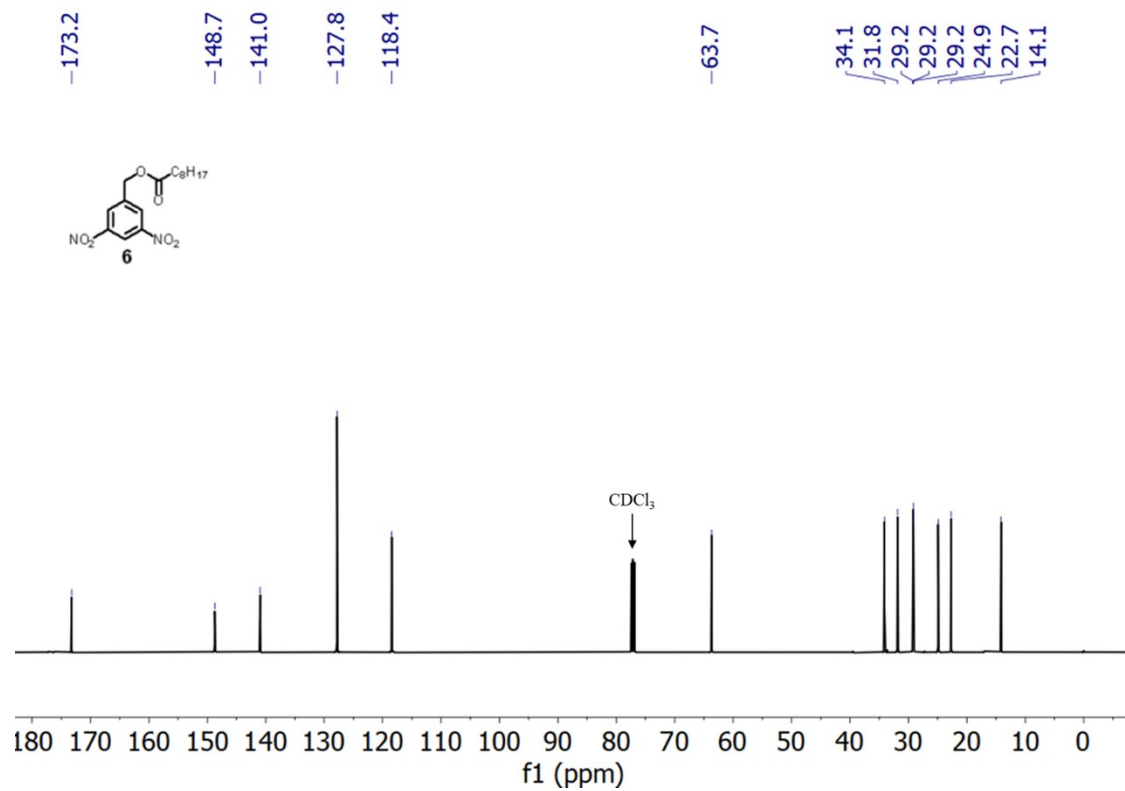


Figure S6. ¹³C NMR spectrum of **6** (125 MHz, CDCl₃).

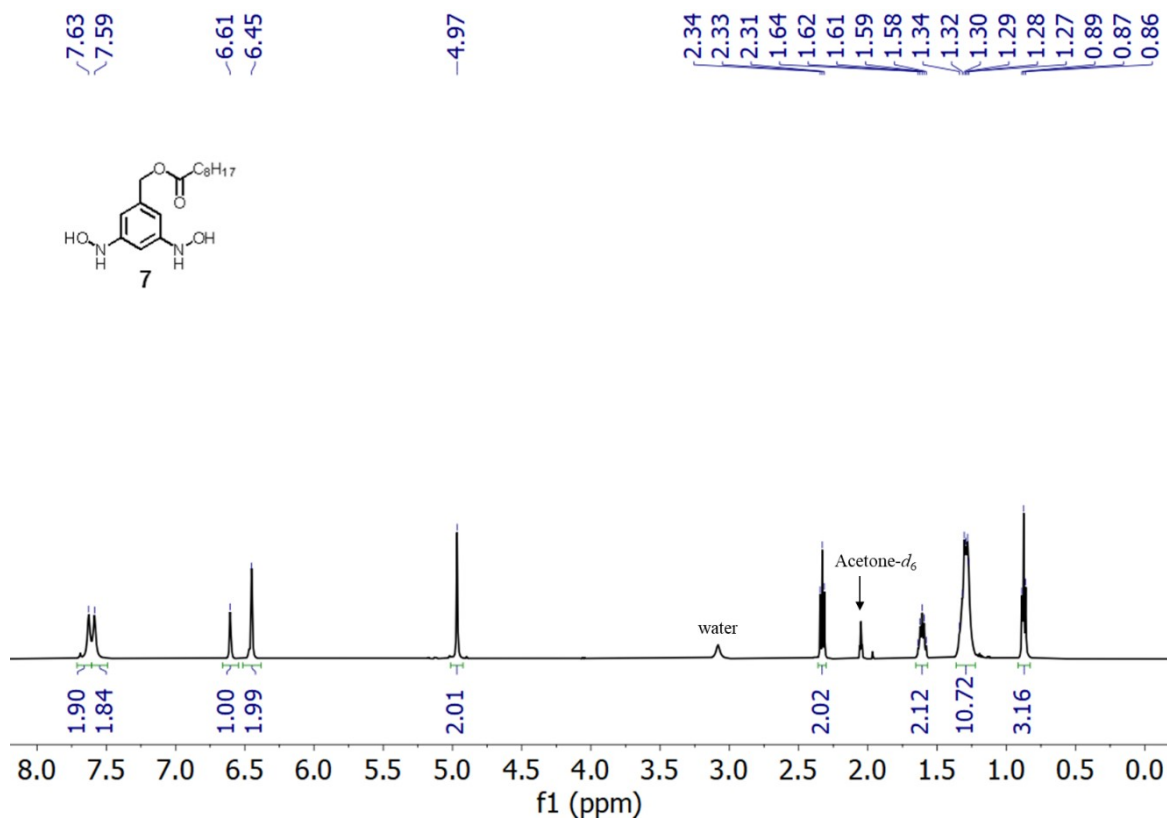


Figure S7. ¹H NMR spectrum of **7** (400 MHz, Acetone-*d*₆).

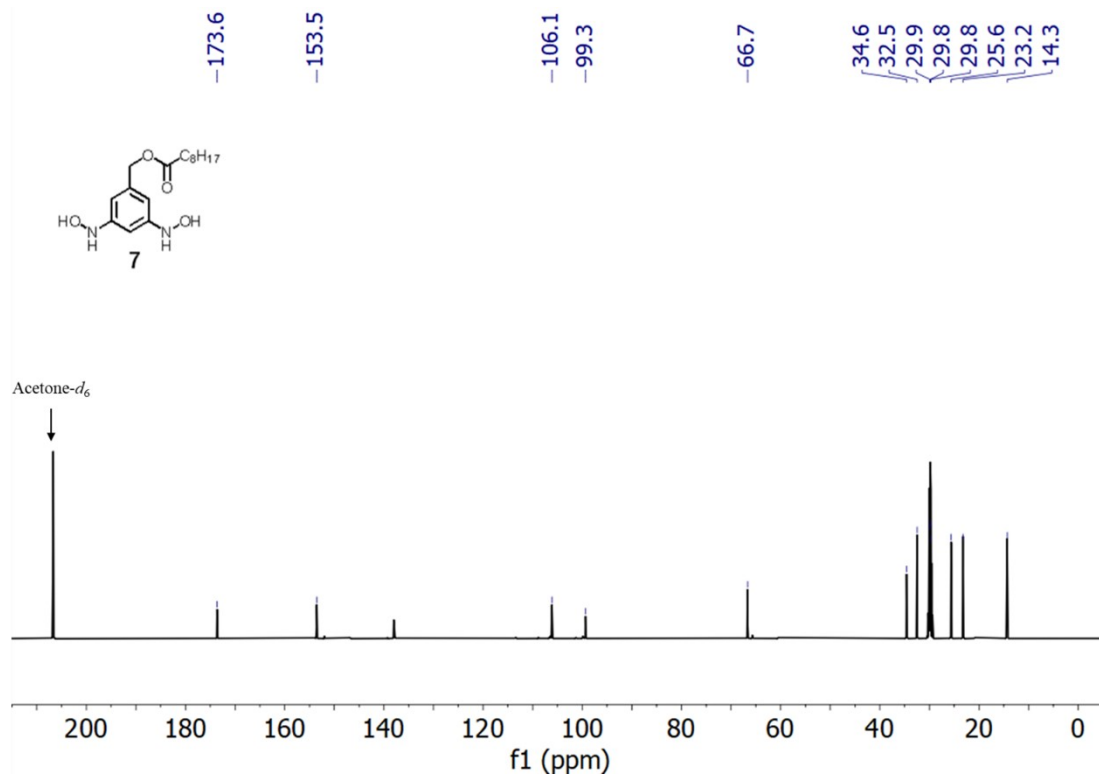


Figure S8. ¹³C NMR spectrum of **7** (125 MHz, Acetone-*d*₆).

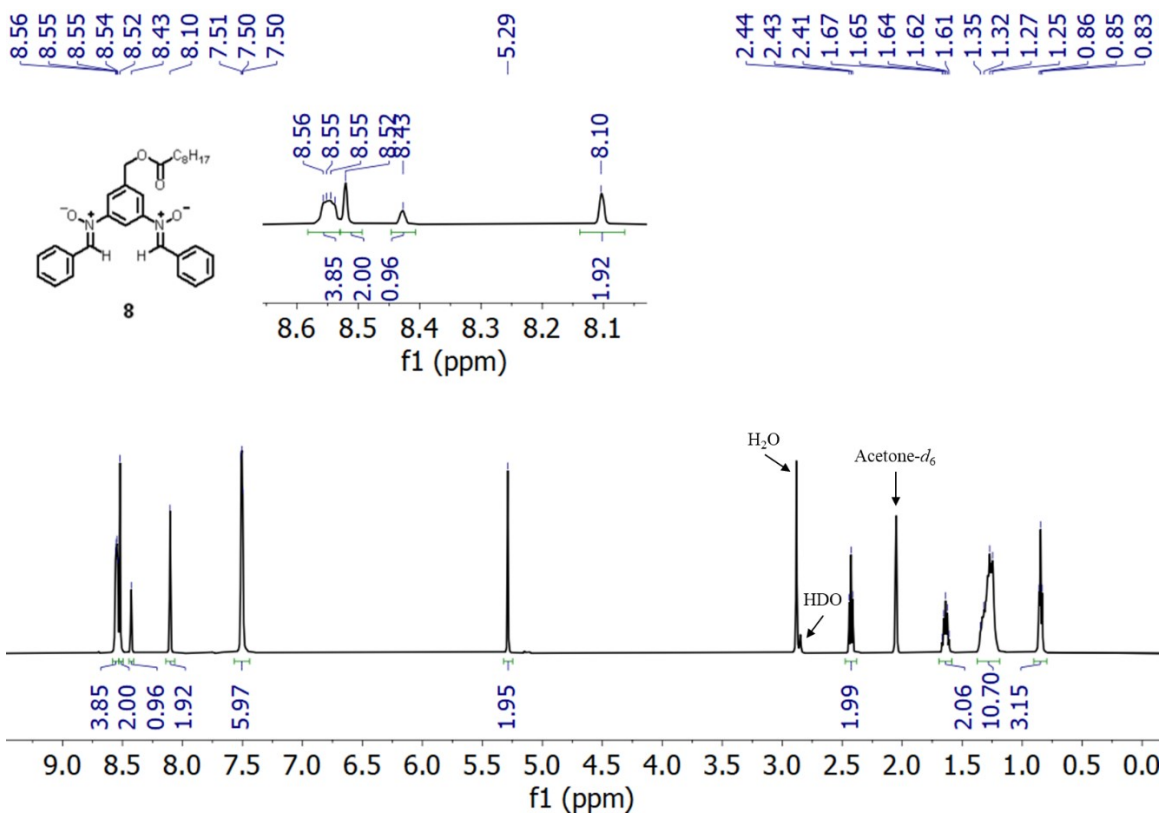


Figure S9. ¹H NMR spectrum of **8** (400 MHz, Acetone-*d*₆).

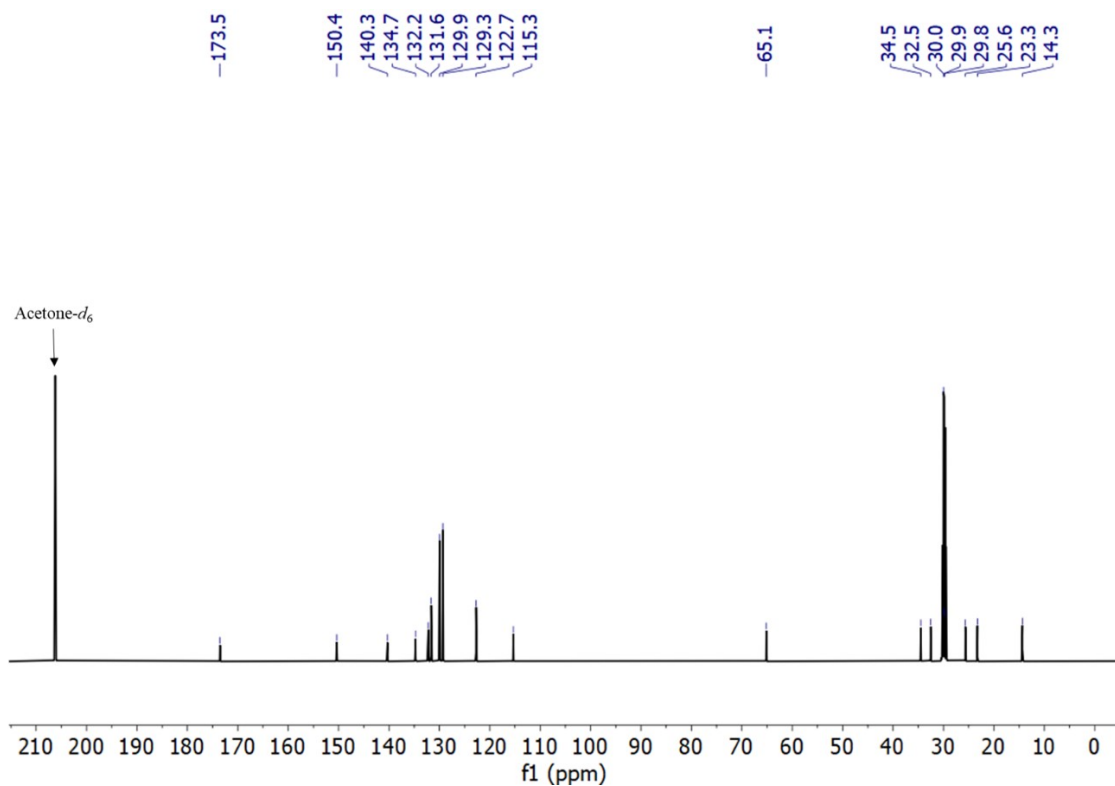


Figure S10. ¹³C NMR spectrum of **8** (125 MHz, Acetone-*d*₆).

S.4 Determining Anion Association Constant Using ^1H NMR Titration

A solution of dinitrone **8** in acetone- d_6 (5 mM, 0.5 mL) was loaded into an NMR tube capped with silicone/PTFE septum. Increasing equivalents of tetrabutylammonium chloride (TBACl), tetrabutylammonium bromide (TBABr) and tetrabutylammonium iodide (TBAI) were added as a concentrated solution (250 mM). Spectra were recorded after each addition.

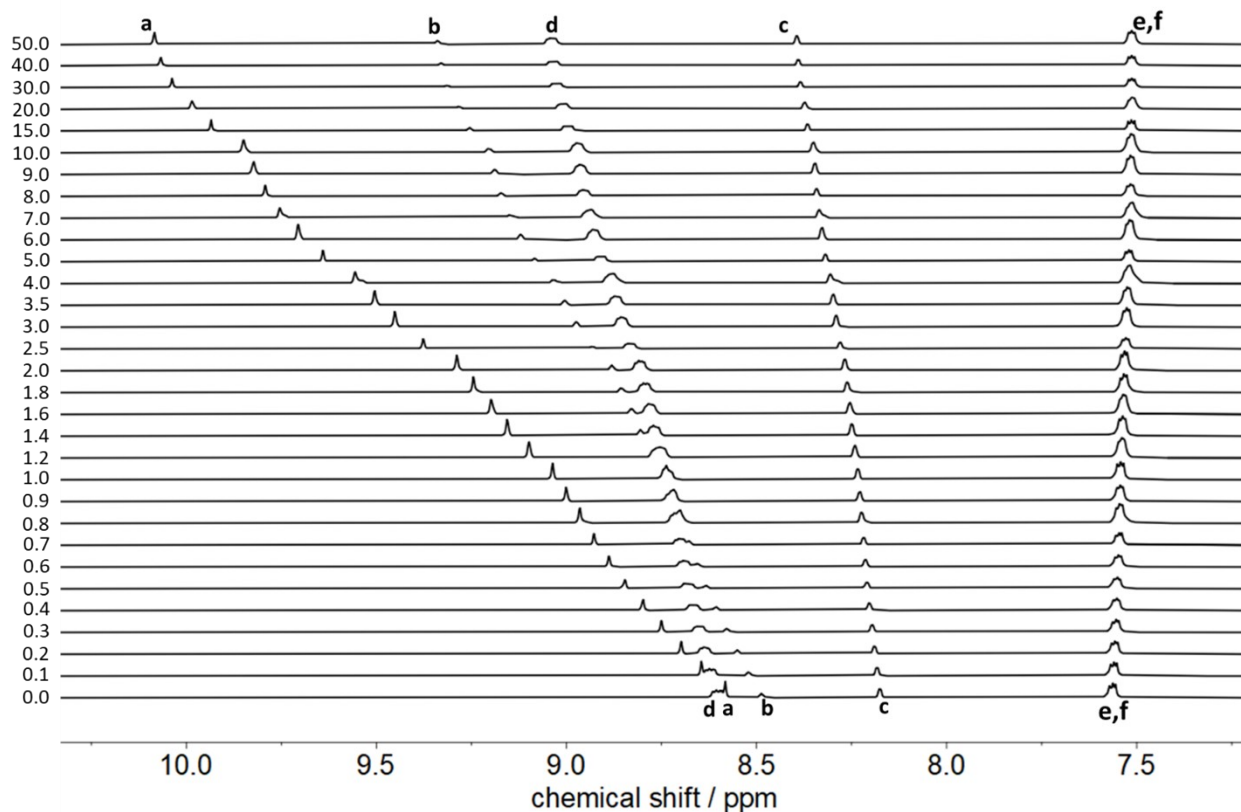
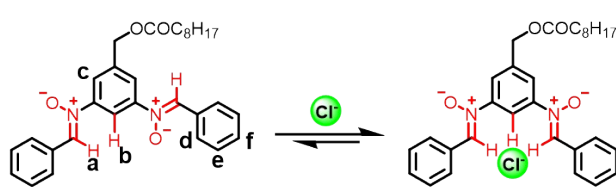


Figure S11. ^1H NMR titration of dinitrone **8** with TBACl (5 mM Acetone- d_6 , 298 K)



Parameters			
Parameter (bounds)	Optimised	Error	Initial
$K (0 \rightarrow \infty)$	107.16 M^{-1}	± 1.9341 %	100.00 M^{-1}

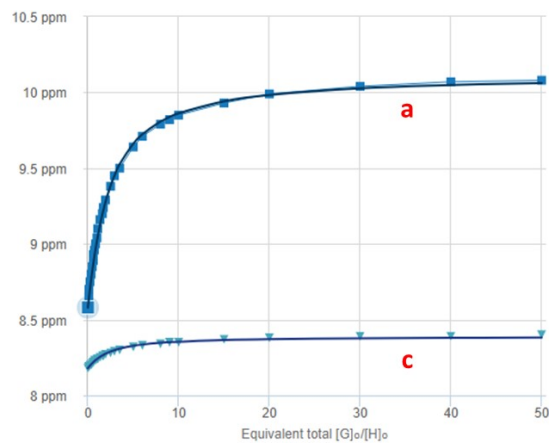


Figure S12. Result of data fitting of dinitrone **8** for proton H^a and H^c using online Bindfit platform

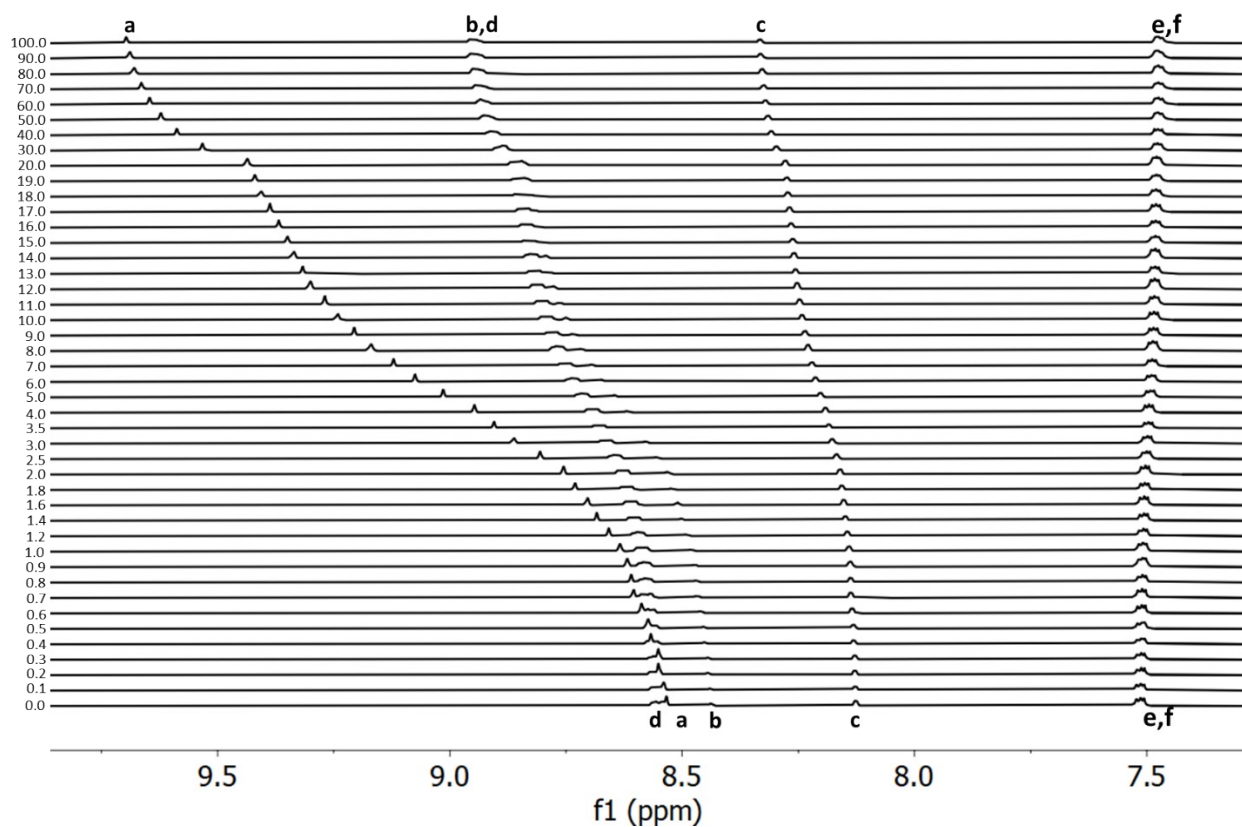


Figure S13. ^1H NMR titration of dinitrone **8** (5 mM Acetone- d_6 , 298 K) with TBABr.

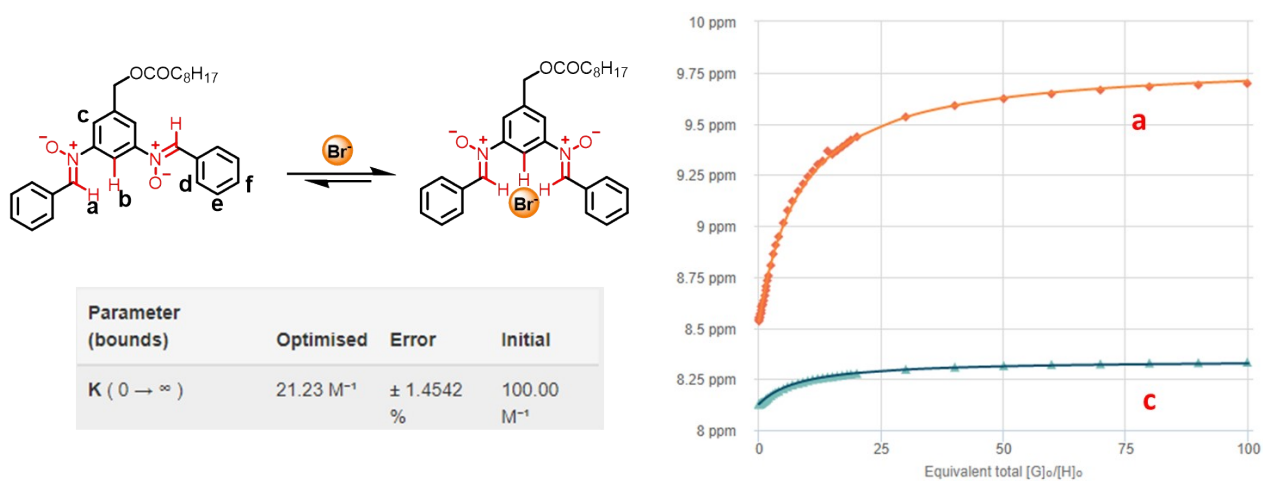


Figure S14. Result of data fitting of dinitrone **8** for proton H^a and H^c using online Bindfit platform

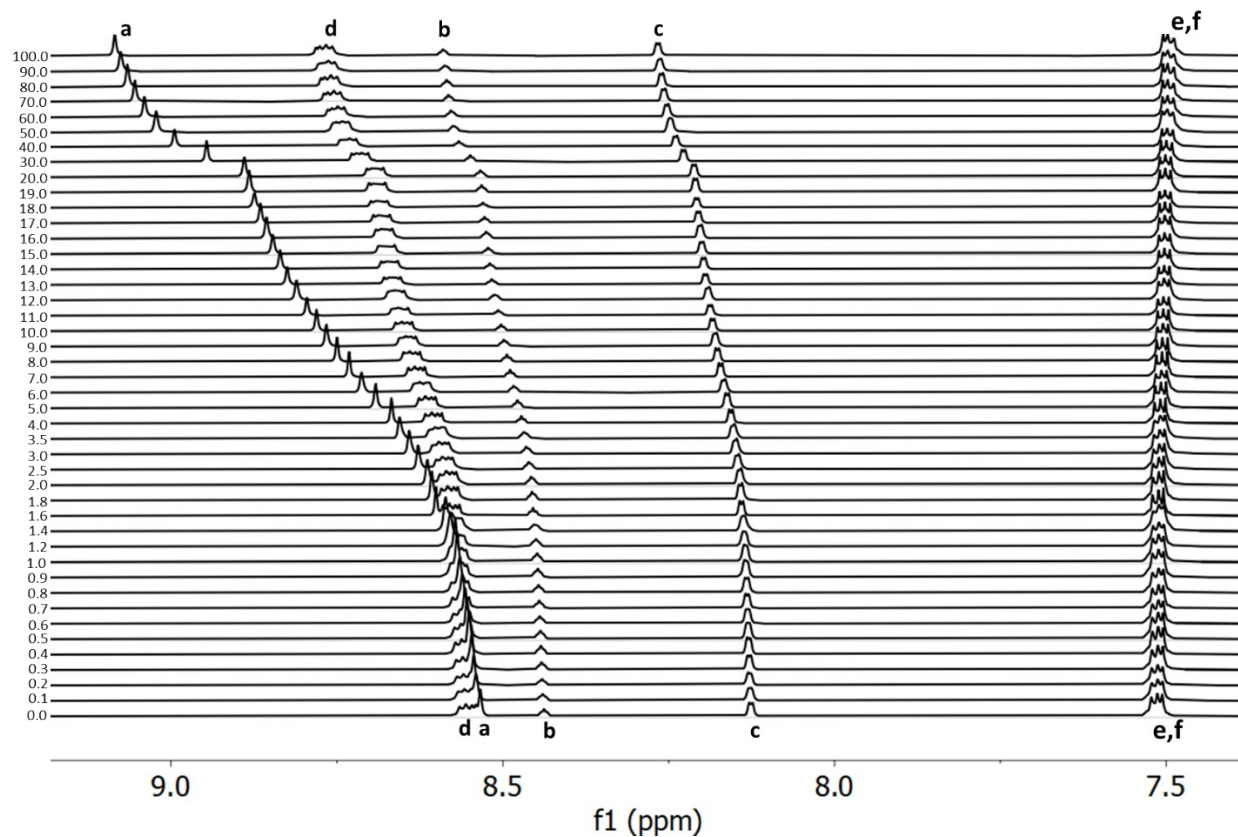


Figure S15. ^1H NMR titration of dinitrone **8** with TBAI (5 mM Acetone- d_6 , 298 K)

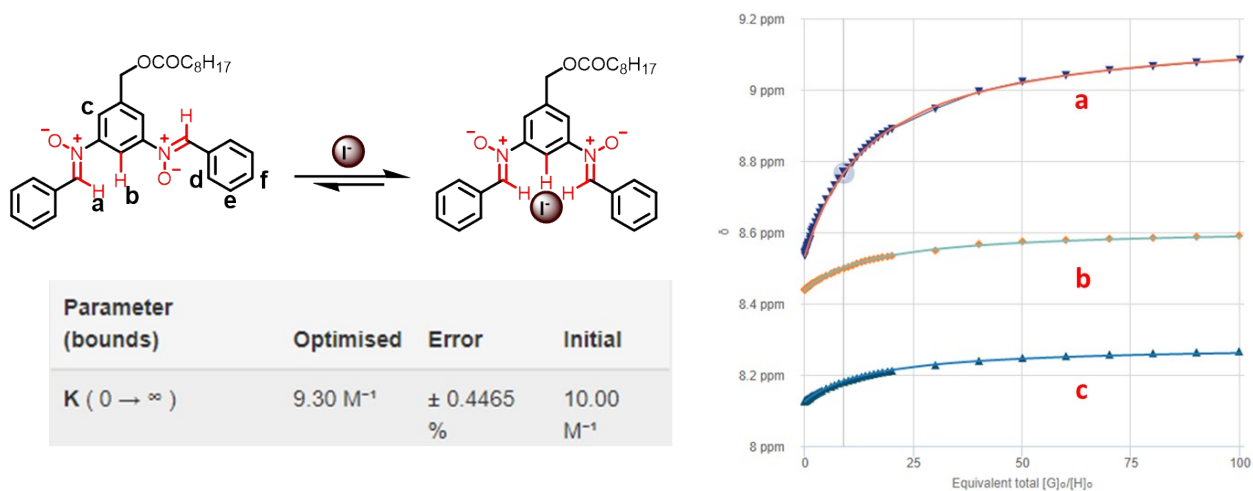


Figure S16. Result of data fitting of dinitrone **8** for proton H^a , H^b and H^c using online Bindfit platform.

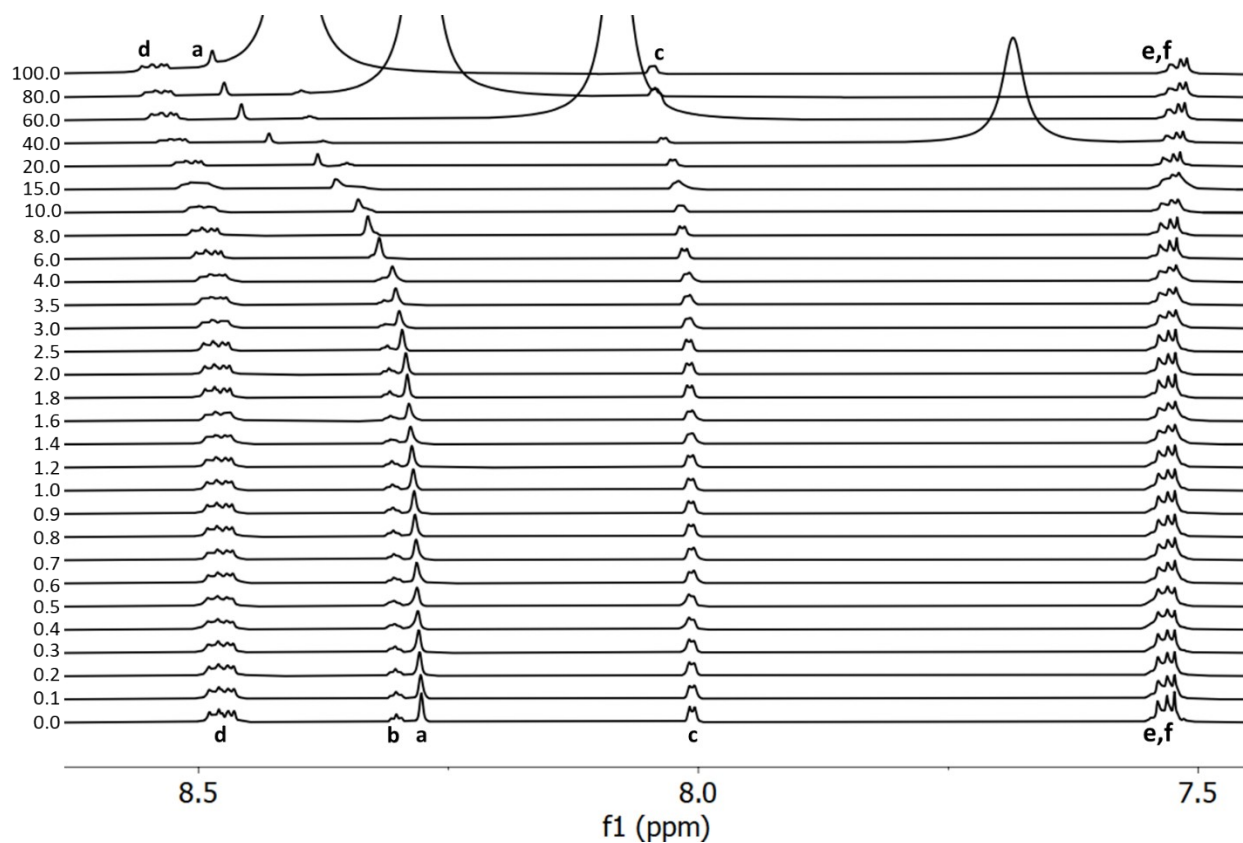


Figure S17. ^1H NMR titration of dinitrone **8** with TBAH_2PO_4 (5 mM CD_3CN , 298 K).

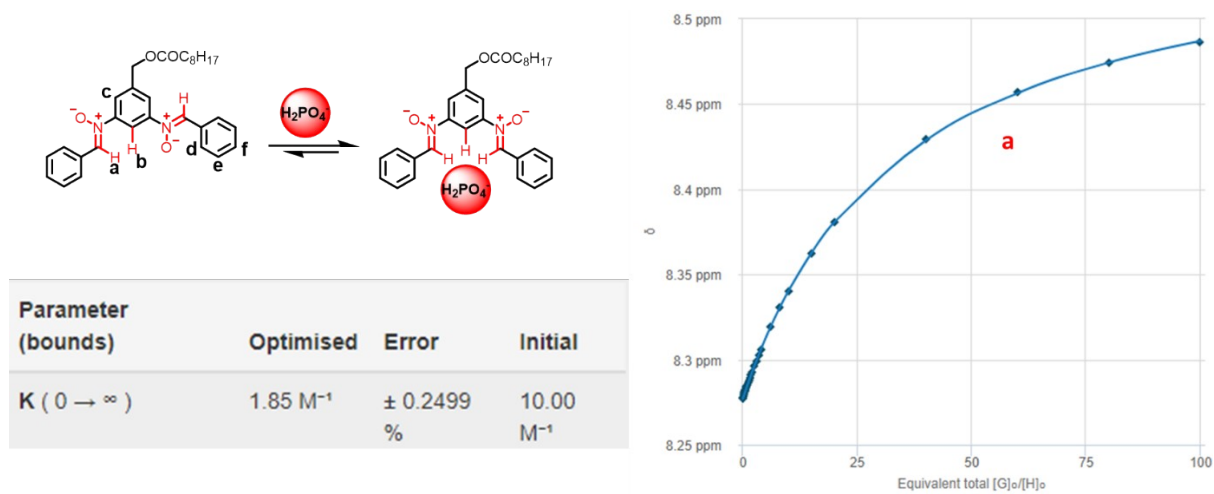


Figure S18. Result of data fitting of dinitrone **8** for proton H^a using online Bindfit platform.

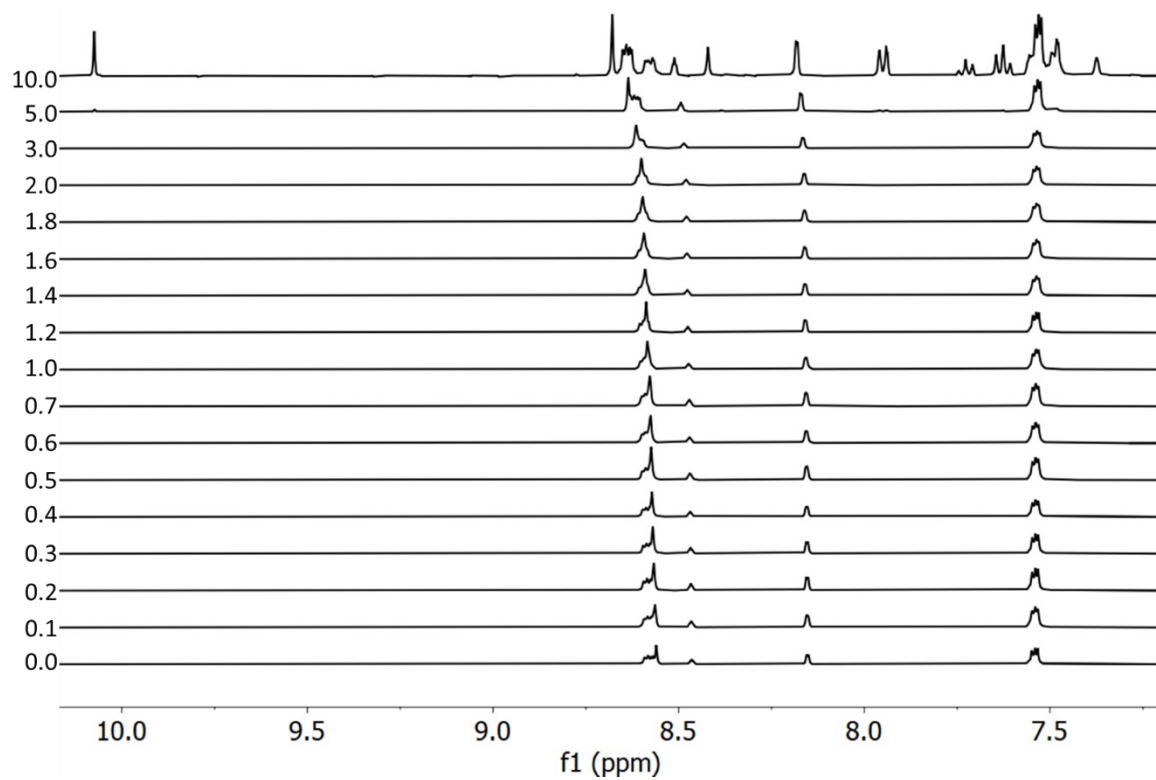


Figure S19. ¹H NMR titration of dinitrone **8** with TBAHSO₄ (5 mM Acetone-*d*₆, 298 K). Dinitrone **8** started to decompose when 10 equiv. of TBAHSO₄ is added.

To compare the binding constant of dinitrone **8** with dicyanostilbene **9**, we titrated dicyanostilbene **9** with TBACl in acetone (0.6 mM in acetone- d_6) and determined its binding constant.

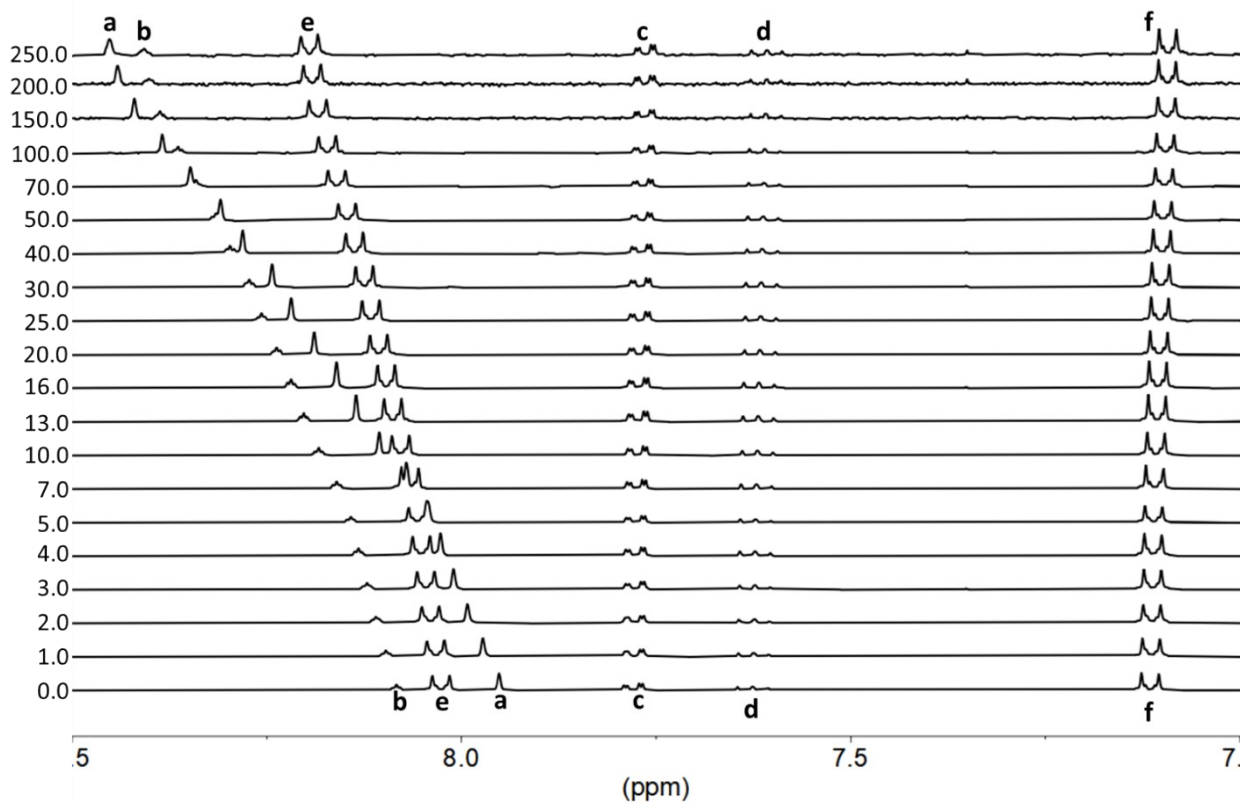


Figure S20. ^1H NMR titration of dicyanostilbene **9** with TBACl (0.6 mM acetone- d_6 , 298 K).

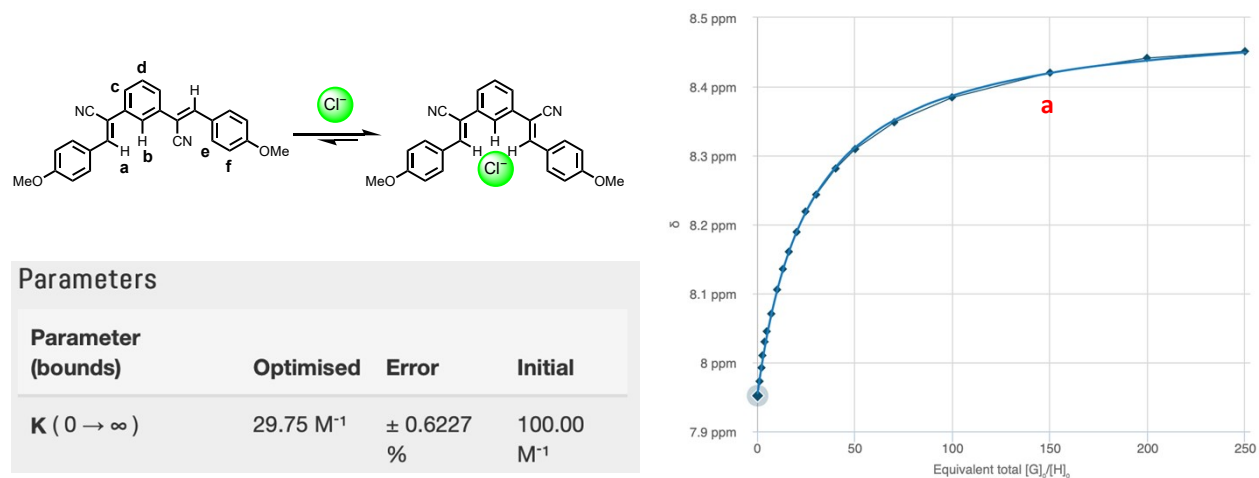
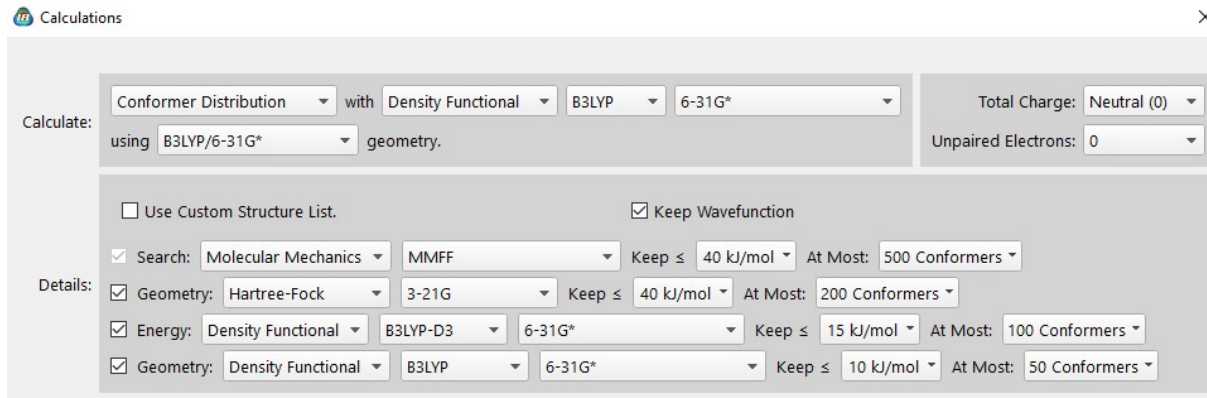


Figure S21. Result of data fitting of dicyanostilbene **9** for proton H^a using online Bindfit platform.

S.5 Computational Studies of Dinitrone 3

All structures were minimized using DFT B3LYP 6-31G* in Spartan '18 (version 1.1.0). All energies are given in kcal/mol. "Conformer Distribution" method (shown below) was used to identify viable conformations **I**, **II**, and **III**.



Calculations

Calculate: Conformer Distribution with Density Functional B3LYP 6-31G* Total Charge: Neutral (0)
using B3LYP/6-31G* geometry. Unpaired Electrons: 0

Details: Use Custom Structure List. Keep Wavefunction

Search: Molecular Mechanics MMFF Keep ≤ 40 kJ/mol At Most: 500 Conformers

Geometry: Hartree-Fock 3-21G Keep ≤ 40 kJ/mol At Most: 200 Conformers

Energy: Density Functional B3LYP-D3 6-31G* Keep ≤ 15 kJ/mol At Most: 100 Conformers

Geometry: Density Functional B3LYP 6-31G* Keep ≤ 10 kJ/mol At Most: 50 Conformers

Conformation I

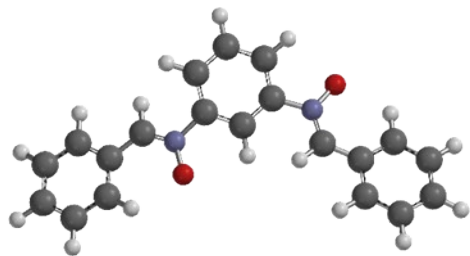


Figure S22. Conformation I, Energies = -647328.30 kcal/mol

	X	Y	Z
H	0.906834	-0.266087	-4.474763
C	0.676677	-0.287942	-3.413677
C	0.130458	-0.347621	-0.674416
C	0.402469	-1.503136	-2.792658
C	0.673971	0.903339	-2.685802
C	0.377278	0.861284	-1.323378
C	0.154237	-1.523177	-1.418643
H	0.380938	-2.437960	-3.338495
H	0.922376	1.841117	-3.170503

H	-0.089921	-0.324226	0.384925
N	-0.118961	-2.822715	-0.814603
C	0.233383	-3.044801	0.441315
H	0.754005	-2.230124	0.925336
C	0.010192	-4.255234	1.199768
C	-0.330910	-6.517943	2.840732
C	-0.653039	-5.402324	0.710227
C	0.495918	-4.272301	2.526967
C	0.328551	-5.388707	3.336047
C	-0.817855	-6.514411	1.532472
H	-1.024812	-5.396393	-0.305211
H	1.007745	-3.395617	2.916885
H	0.710017	-5.379680	4.354270
H	-1.331307	-7.389622	1.143829
H	-0.462713	-7.392715	3.471906
O	-0.669293	-3.681099	-1.592605
N	0.333897	2.061179	-0.496132
C	-0.089510	3.198570	-1.022776
H	-0.426203	3.131706	-2.048828
C	-0.164222	4.483224	-0.363186
C	-0.375545	7.066978	0.738611
C	0.222683	4.726635	0.974021
C	-0.660388	5.562664	-1.127478
C	-0.764401	6.837058	-0.584372
C	0.115874	6.009078	1.507360
H	0.600102	3.904379	1.564829
H	-0.963518	5.389760	-2.157220
H	-1.148398	7.653298	-1.191500

H	0.418759	6.182020	2.536630
H	-0.457385	8.063044	1.165269
O	0.692017	1.898502	0.724844

Conformation II

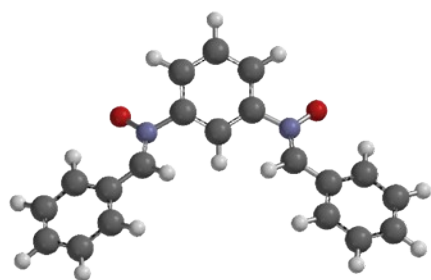


Figure S23. Conformation II, Energy = -647327.29 kcal/mol

	X	Y	Z
H	-5.185922	-0.000358	-0.486968
C	-4.117056	-0.000181	-0.293904
C	-1.370183	0.000218	0.225339
C	-3.440383	-1.211635	-0.166999
C	-3.440485	1.211490	-0.166068
C	-2.067500	1.199699	0.077460
C	-2.067381	-1.199576	0.076769
H	-3.948465	-2.165923	-0.232304
H	-3.948720	2.165610	-0.230478
H	-0.326689	0.000651	0.515886
N	-1.407647	-2.490477	0.234398
C	-0.155434	-2.635728	-0.170087
H	0.277967	-1.766673	-0.647122

C	0.656456	-3.826925	-0.058626
C	2.396255	-6.038539	0.073843
C	0.233469	-5.037262	0.534470
C	1.967406	-3.756286	-0.582926
C	2.825070	-4.847033	-0.519021
C	1.104311	-6.122738	0.596810
H	-0.769062	-5.099500	0.933665
H	2.307409	-2.830771	-1.043055
H	3.827720	-4.770532	-0.930816
H	0.766178	-7.046920	1.057811
H	3.064854	-6.893546	0.125530
O	-2.133700	-3.418741	0.736778
N	-1.407778	2.490300	0.236339
C	-0.157267	2.636911	-0.173062
H	0.274421	1.769032	-0.654532
C	0.655123	3.827661	-0.060778
C	2.395931	6.038209	0.074309
C	0.232005	5.038156	0.531434
C	1.966847	3.756055	-0.582730
C	2.825329	4.846043	-0.517149
C	1.103036	6.123494	0.594596
H	-0.770760	5.100498	0.929480
H	2.307134	2.830035	-1.041273
H	3.829325	4.768200	-0.925856
H	0.764806	7.048117	1.055292
H	3.065037	6.892767	0.127413
O	-2.131660	3.416197	0.746132

Conformation III

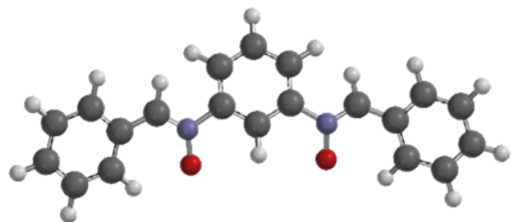


Figure S24. Conformation III, Energy = -647326.82 kcal/mol

	X	Y	Z
H	-3.364851	0.000054	-2.048724
C	-2.496936	0.000026	-1.396190
C	-0.336144	-0.000080	0.378779
C	-1.957926	1.211555	-0.965706
C	-1.958183	-1.211561	-0.965405
C	-0.868604	-1.196973	-0.090682
C	-0.868330	1.196870	-0.090983
H	-2.410663	2.148169	-1.272142
H	-2.411095	-2.148150	-1.271656
H	0.465975	-0.000084	1.104276
N	-0.270025	2.427832	0.416221
C	-0.222844	3.494765	-0.366214
H	-0.595807	3.344929	-1.370473
C	0.284035	4.802260	-0.014477
C	1.197796	7.419343	0.484825
C	0.806063	5.149952	1.251267
C	0.237085	5.794925	-1.019635
C	0.688019	7.085525	-0.773792
C	1.252174	6.448520	1.486816
H	0.849428	4.394193	2.022767

H	-0.160611	5.540476	-1.999789
H	0.642479	7.832773	-1.561530
H	1.648656	6.702995	2.466239
H	1.550457	8.428412	0.680323
O	0.197534	2.361307	1.605758
N	-0.270579	-2.428000	0.416688
C	-0.222982	-3.494766	-0.365912
H	-0.595550	-3.344724	-1.370216
C	0.284045	-4.802226	-0.014316
C	1.198514	-7.419024	0.484396
C	0.805171	-5.150223	1.251656
C	0.237778	-5.794569	-1.019798
C	0.689004	-7.085100	-0.774264
C	1.251723	-6.448657	1.486927
H	0.846746	-4.395033	2.023897
H	-0.159757	-5.539936	-1.999960
H	0.643651	-7.832247	-1.562033
H	1.646665	-6.703898	2.466842
H	1.551401	-8.428034	0.679748
O	0.196488	-2.361594	1.606469

Dinitrone **3**·Cl⁻ complex

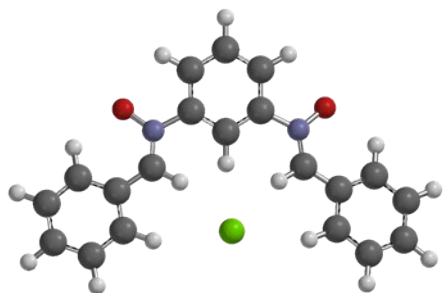


Figure S25. Dinitrone **3**·Cl⁻ complex, Energy = -936176.60 kcal/mol

	X	Y	Z
H	5.167029	-0.000000	1.097694
C	4.117983	-0.000000	0.812621
C	1.418605	0.000000	0.062876
C	3.454473	1.210064	0.627866
C	3.454473	-1.210064	0.627866
C	2.106333	-1.199273	0.265296
C	2.106333	1.199274	0.265296
H	3.947160	2.167065	0.741252
H	3.947160	-2.167065	0.741252
H	0.376036	0.000000	-0.241370
N	1.474493	2.503752	0.068955
C	0.159892	2.613713	0.132158
H	-0.406943	1.711501	0.353528
O	2.289840	3.483481	-0.136359
C	-0.602924	3.828131	-0.072250
C	-2.296297	6.047705	-0.435324
C	-0.061453	5.107401	-0.329104
C	-2.008846	3.684027	-0.003729
C	-2.840363	4.783968	-0.183351
C	-0.909571	6.199044	-0.507358
H	1.013110	5.213840	-0.382941
H	-2.421886	2.692922	0.180458
H	-3.918771	4.654044	-0.129302
H	-0.480766	7.179628	-0.704430
H	-2.948659	6.907210	-0.575834
N	1.474493	-2.503752	0.068956
C	0.159892	-2.613713	0.132157

H	-0.406943	-1.711501	0.353527
O	2.289840	-3.483481	-0.136358
C	-0.602924	-3.828131	-0.072250
C	-2.296297	-6.047705	-0.435324
C	-0.061453	-5.107401	-0.329104
C	-2.008846	-3.684027	-0.003729
C	-2.840363	-4.783969	-0.183351
C	-0.909571	-6.199044	-0.507359
H	1.013110	-5.213840	-0.382942
H	-2.421886	-2.692923	0.180459
H	-3.918771	-4.654044	-0.129301
H	-0.480766	-7.179628	-0.704430
H	-2.948659	-6.907210	-0.575834
Cl	-2.177298	0.000000	0.449115

S.6 X-ray Crystallographic Analysis of Dinitrone 3

Single crystals suitable for X-ray crystallography were prepared by slow diffusion of *n*-pentane into a solution of **3** in acetone. The crystal was placed MiTeGen pins, coated in oil. The X-ray intensity data collection was carried out on a Bruker APEXII DUO CCD area detector using graphite-monochromated Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$) at 90.0 K. CCDC 2201970 contain the supplementary crystallographic data for this paper.

Crystal data of dinitrone 3

$C_{20}H_{16}N_2O_2$	$D_x = 1.397 \text{ Mg m}^{-3}$
$M_r = 316.35$	Mo K α radiation, $\lambda = 0.71073 \text{ \AA}$
Tetragonal, $P4_1$	Cell parameters from 9912 reflections
$a = 5.3375 (6) \text{ \AA}$	$\theta = 3.1\text{--}28.1^\circ$
$c = 52.802 (9) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$V = 1504.3 (4) \text{ \AA}^3$	$T = 90 \text{ K}$
$Z = 4$	Triangular, colourless
$F(000) = 664$	$0.33 \times 0.17 \times 0.17 \text{ mm}$

Data collection

Bruker Kappa APEX-II DUO diffractometer	3562 independent reflections
Radiation source: fine-focus sealed tube	3508 reflections with $I > 2\sigma(I)$
TRIUMPH curved graphite monochromator	$R_{\text{int}} = 0.062$
ϕ and ω scans	$\theta_{\text{max}} = 30.2^\circ$, $\theta_{\text{min}} = 1.2^\circ$
Absorption correction: multi-scan <i>SADABS</i> (Krause <i>et al.</i> , 2015)	$h = -7 - 7$
$T_{\text{min}} = 0.921$, $T_{\text{max}} = 0.985$	$k = -7 - 7$
33370 measured reflections	$l = -68 - 67$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 1.4807P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.119$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.16$	$\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
3562 reflections	$\Delta\rho_{\min} = -0.41 \text{ e } \text{\AA}^{-3}$
218 parameters	Absolute structure: Flack x determined using 1632 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
1 restraint	Absolute structure parameter: 0.2 (4)

Refinement. Refined as a 2-component twin.

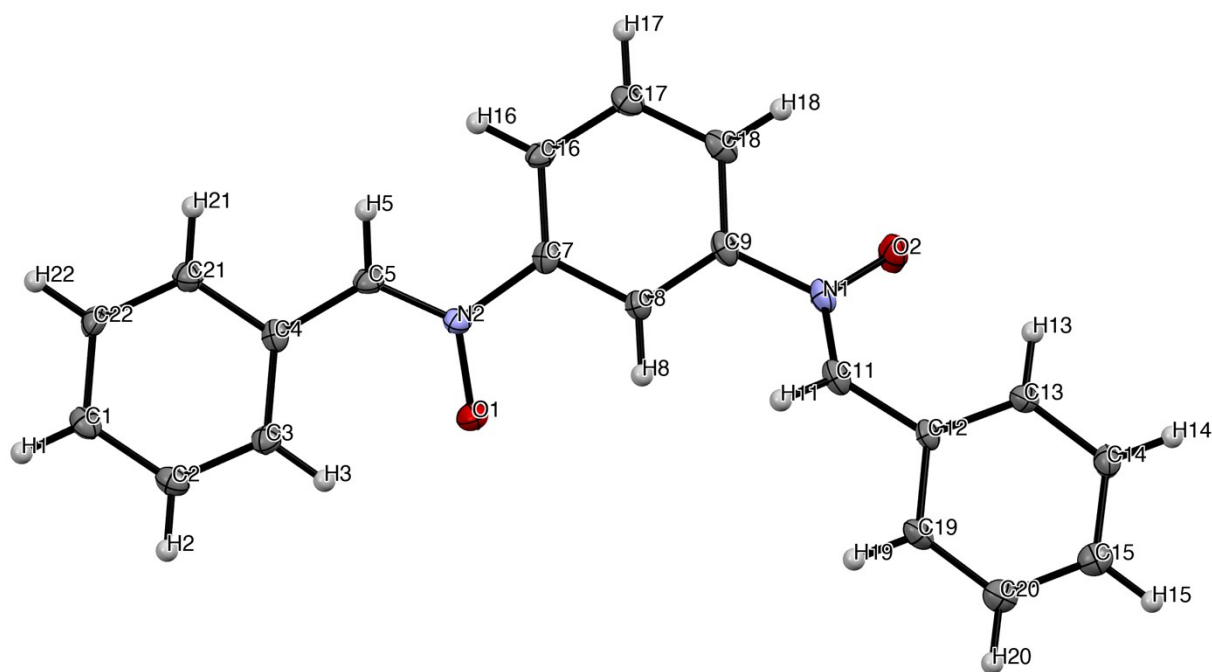
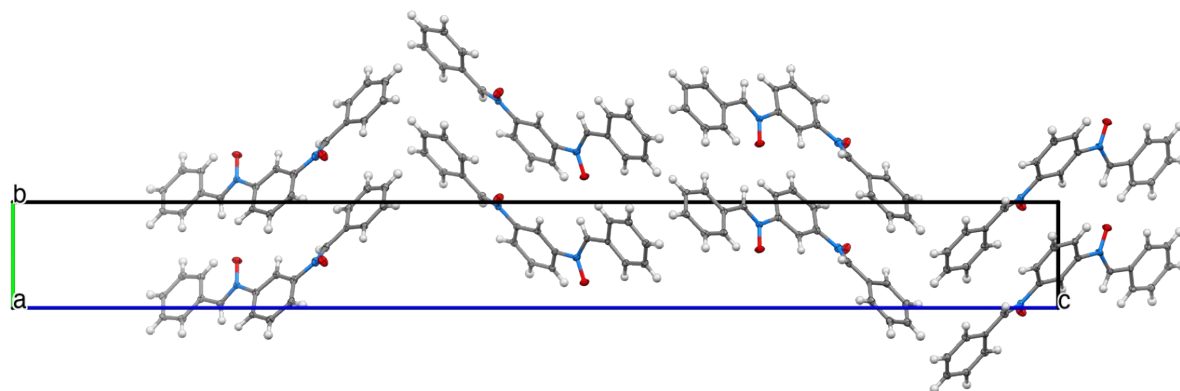


Figure S26. X-ray crystal structure of dinitrone **3**. Thermal ellipsoids at the 50% probability level.



Fi

Figure S27. Packing structure of **3** viewed along the a-axis.

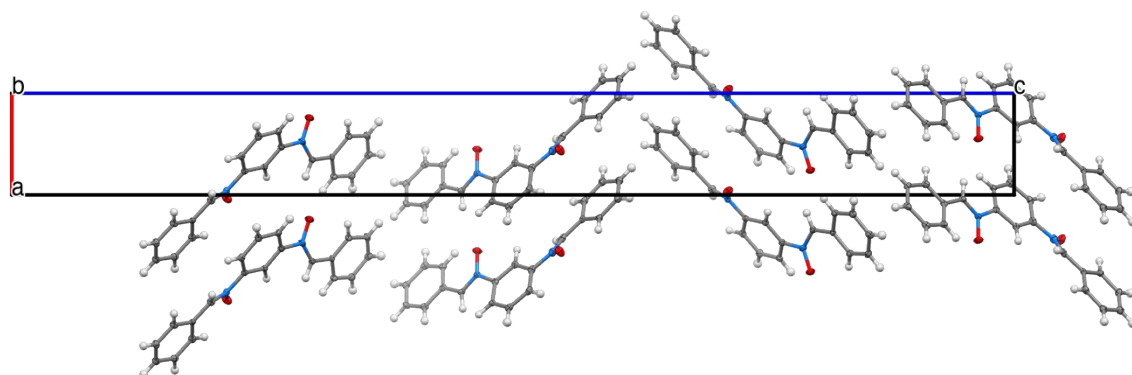


Figure S28. Packing structure of **3** viewed along the b-axis.

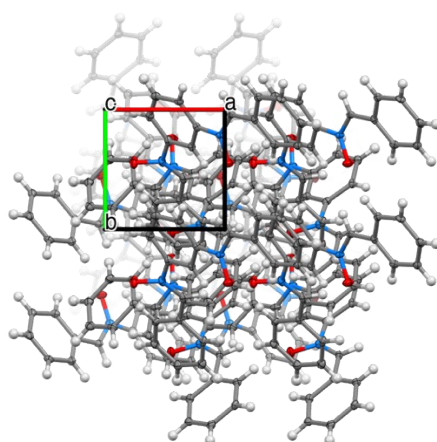


Figure S29. Packing structure of **3** viewed along the c-axis.

S.6 References

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2. Campbell, R. F.; Fitzpatrick, K.; Inghardt, T.; Karlsson, O.; Nilsson, K.; Reilly, J. E.; Yet, L. *Tetrahedron Lett.* **2003**, *44*, 5477-5481.
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