Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting information

Synthesis of a Tethered Dibenzotetramethyltetraaza[14]annulene Macrocycle and The Di-nickel(II) Derivative

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Contents

Mass Spectrometry

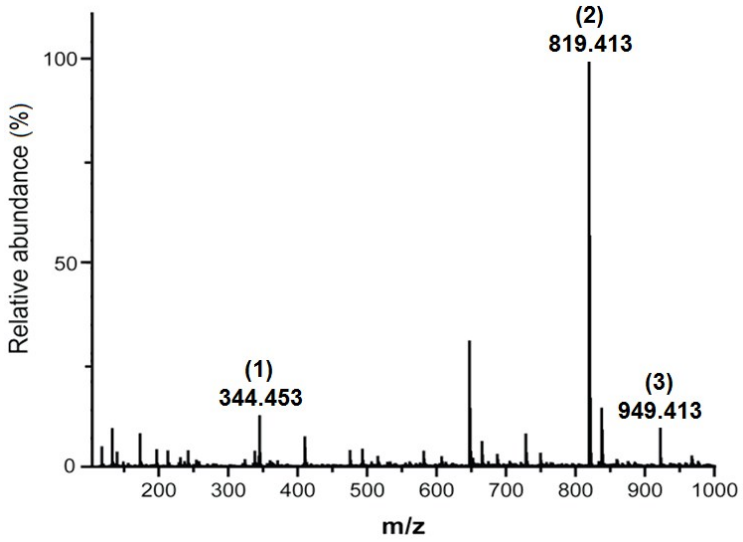

Fig. S1. ESI mass spectrum of **2** (crude product).

UV-Visible Absorption Spectroscopy

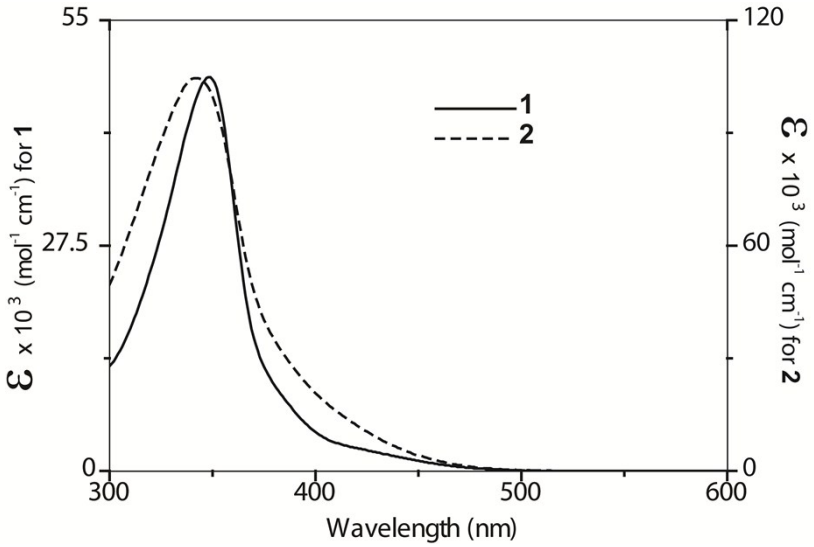

Fig. S2. UV-visible spectra of 1.16×10^{-5} (M) **1** and 4.9×10^{-6} (M) **2** in benzene at 295 K.

Selected parameters and metrics from X-ray Diffraction

Table S1. Crystallographic Data and Data Collection Parameters

	2
T, K	100(2)
Formula	$C_{54.5}H_{56}N_8O_2$
Formula weight	855.07
Crystal system	triclinic
Space group	Pī
a, Å	11.74(2)
b, Å	14.45(3)
c, Å	15.40(3)
α , deg	62.50(5)
β, deg	82.90(3)
γ, deg	73.44(3)
V, Å3	2221(7)
Radiation (λ, Å)	Mo Kα (0.71073)
Z	2
No. of unique data	5073
No. of params. Refined	600
GOF on F^2	0.965
R1[a] [I > 2σ(I)]	0.0875
R1[a] (all data)	0.2028
wR2[b] (all data)	0.2453

[a] $$R1 = \frac{\sum ||Fo| - |Fc||}{\sum |Fo|}$$

[b] $$wR2 = \sqrt{\frac{\sum \left[w\left(Fo^2 - Fc^2\right)^2\right]}{\sum \left[w\left(Fo^2\right)^2\right]}}$$

Table S2. **Some selected bond distances and angles**

Bond length, (Å)	**2**	
	Ring-I	Ring-II
Ct_{14}-N1	1.841	1.968
Ct_{14}-N2	1.968	1.844
Ct_{14}-N3	1.838	1.942
Ct_{14}-N4	1.968	1.848
Bond angles,(deg)		
N1-Ct_{14}-N2	90.24	90.89
N1-Ct_{14}-N3	161.32	164.27
N1-Ct_{14}-N4	85.32	84.62
N2-Ct_{14}-N3	87.13	88.65
N2-Ct_{14}-N4	155.75	162.21
N3-Ct_{14}-N4	89.51	91.06

Unit cell packing

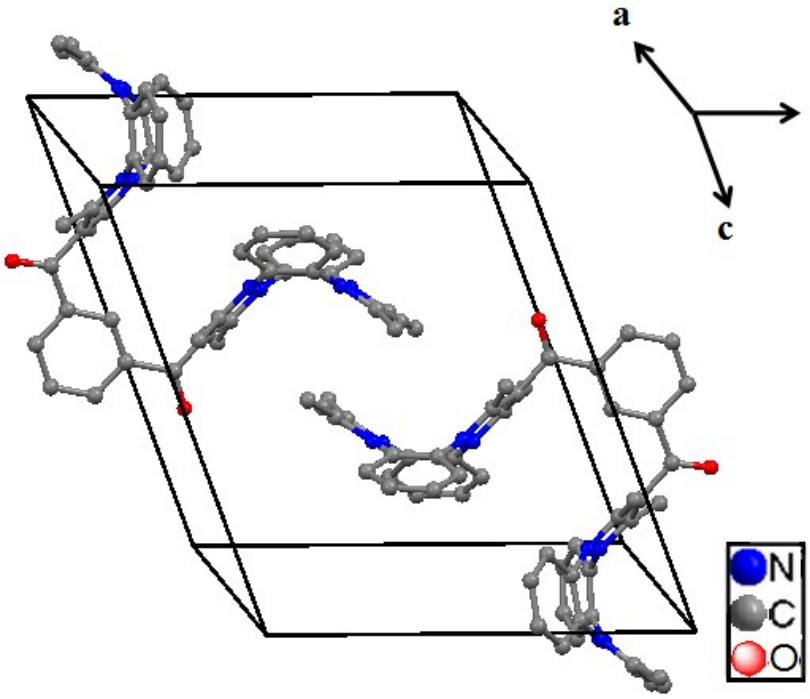

Fig. S3. Diagram illustrating the packing of the **2** in the unit cell at 100 K (H-atoms and non-coordinating solvent molecule have been omitted for clarity).

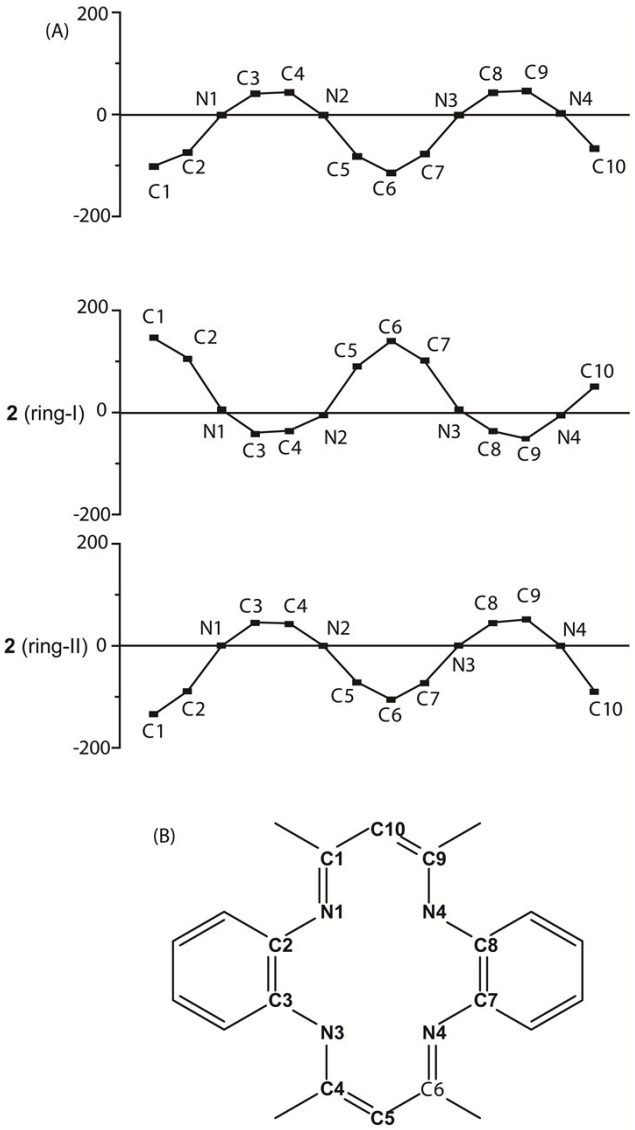

Fig. S4. (A) Out-of-plane displacements (in unit of 0.01 Å) of [14]annulene core atoms of **1** and **2** from least-squares planes of four 'N' ring atoms. The horizontal axes show the bond connectivity between atoms. (B) The corresponding atom numbers of $C_{10}N_4$ [14]annulene core atoms.

^{13}C NMR Spectra

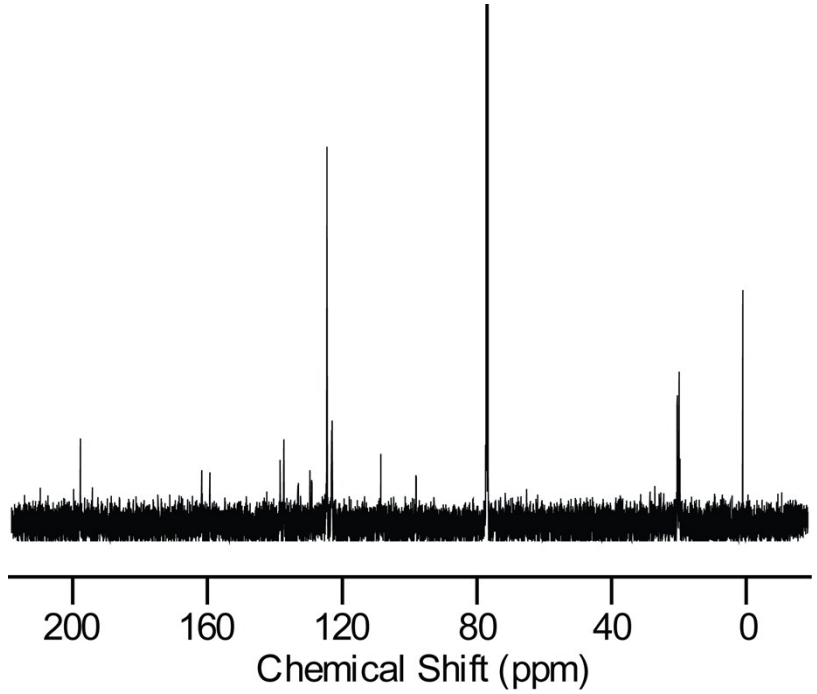

Fig. S5. 500 MHz ^{13}C NMR spectra in $CDCl_3$ (at 295 K) of **2**.

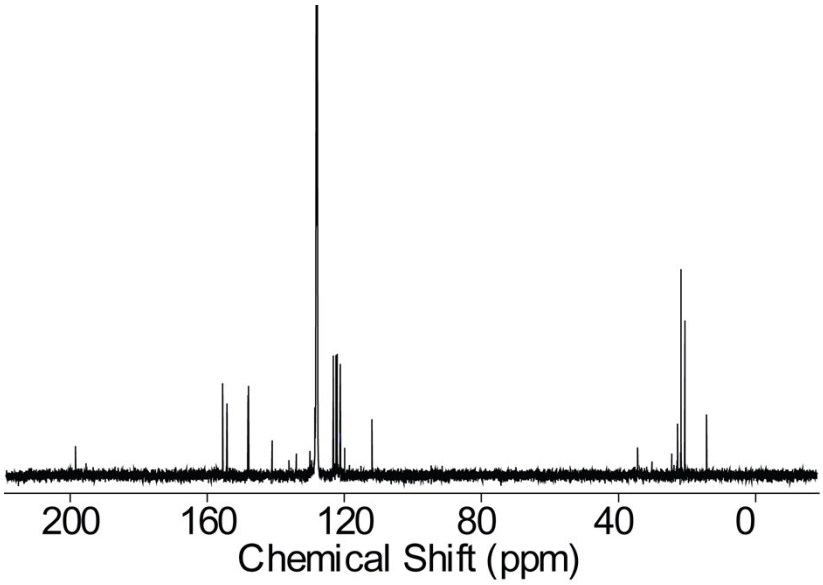

Fig. S6. 500 MHz ^{13}C NMR spectra in C_6D_6 (at 295 K) of **2**-Ni.

Results from density functional theoretical calculations.

Selected figures of molecular and orbital geometries.

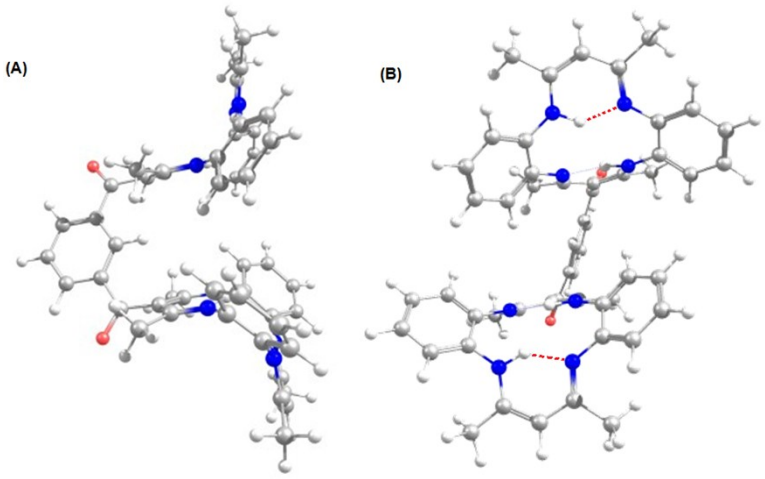

Fig. S7. (A) Top and (B) front views of B3LYP optimized geometries of **2** as obtained using 6-31G basis set for all atoms. Red dotted lines indicate H-bonds.

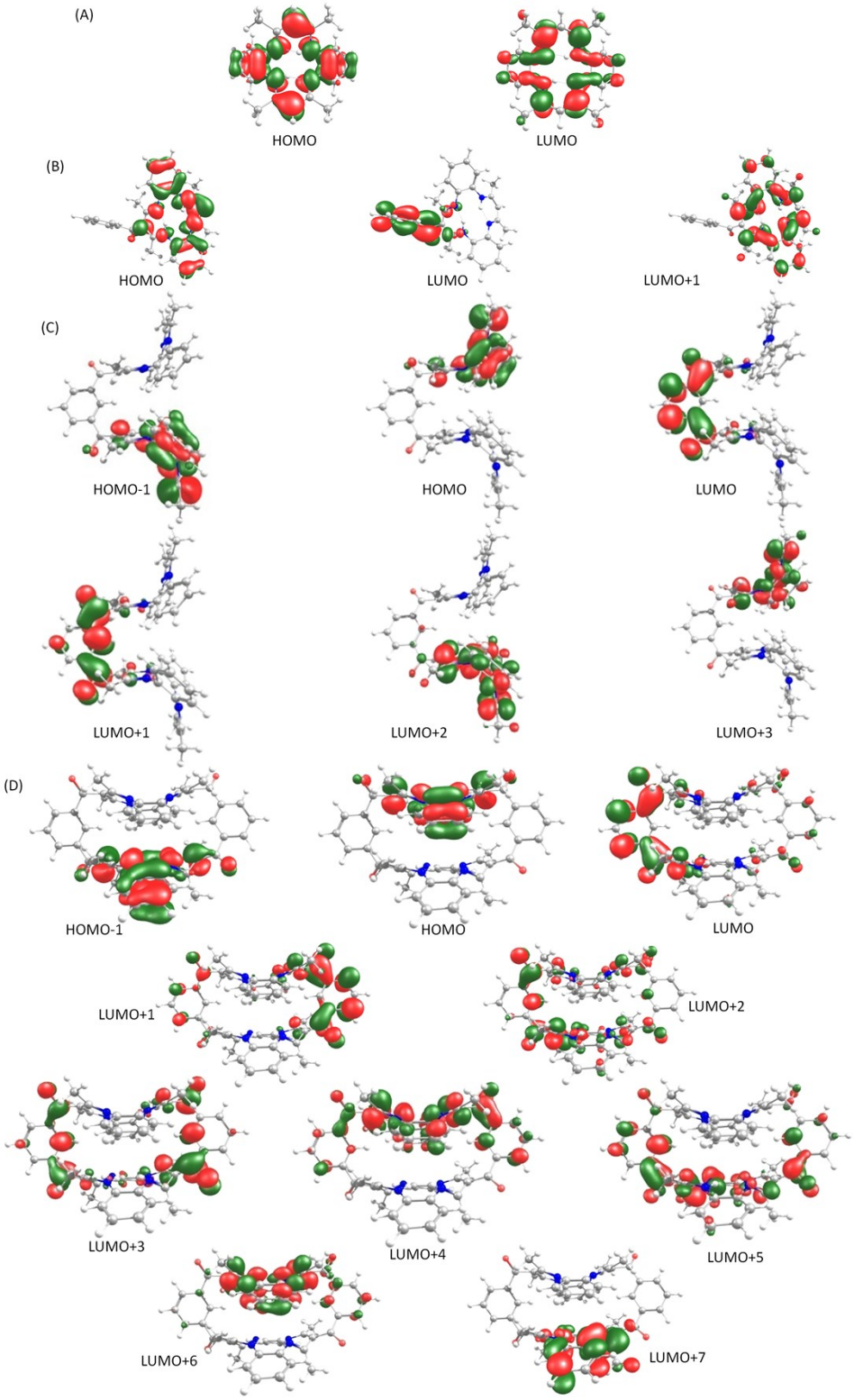

Fig. S8. (A) HOMO, LUMO of **1**, (B) HOMO, LUMO, LUMO+1 of **4**, (C) HOMO-1, HOMO, LUMO, LUMO+1, LUMO+2, LUMO+3 of **2** and (d) HOMO-1, HOMO, LUMO, LUMO+1, LUMO+2, LUMO+3, LUMO+4, LUMO+5, LUMO+6, LUMO+7 of **3** calculated by using DFT at the B3LYP/6-31G level.

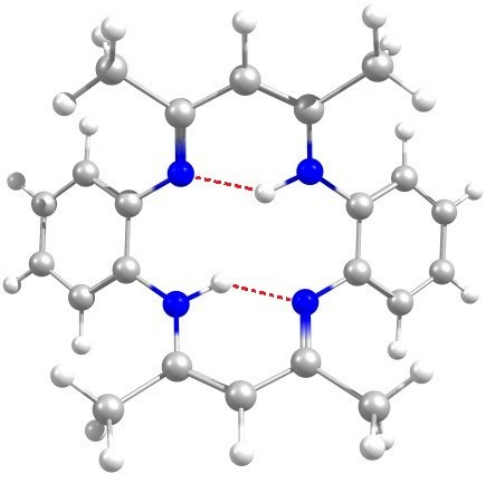

Fig. S9. B3LYP optimized geometries of 1 as obtained using 6-31G basis set for all atoms. Red dotted lines indicate H-bonds.

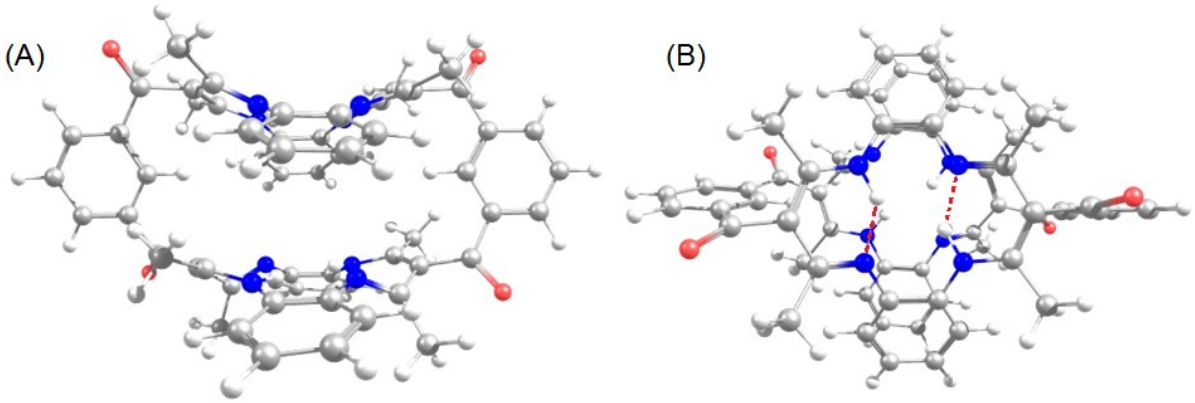

Fig. S10. (A) Side and (B) top views of B3LYP optimized geometry of **3** as obtained using 6-31G basis set for all atoms. Red dotted lines indicate H-bonds.

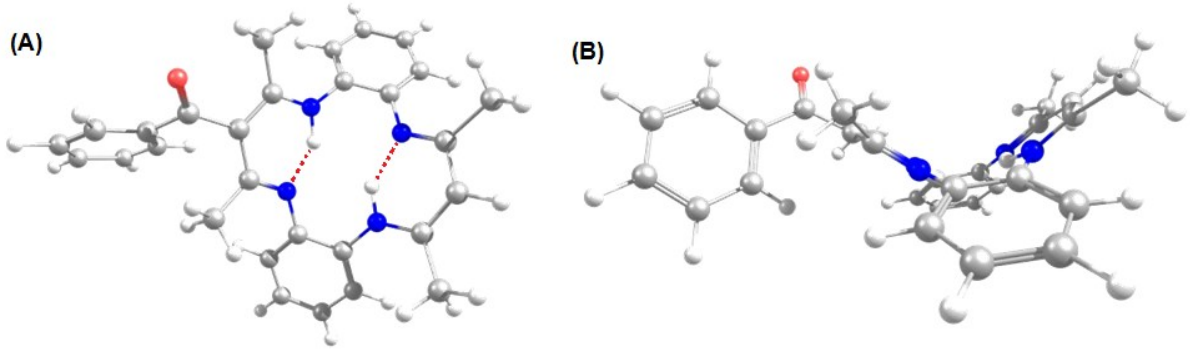

Fig. S11. (A) Top and (B) side views of B3LYP optimized geometry of **4** as obtained using 6-31G basis set for all atoms. Red dotted lines indicate H-bonds.

Electrochemistry

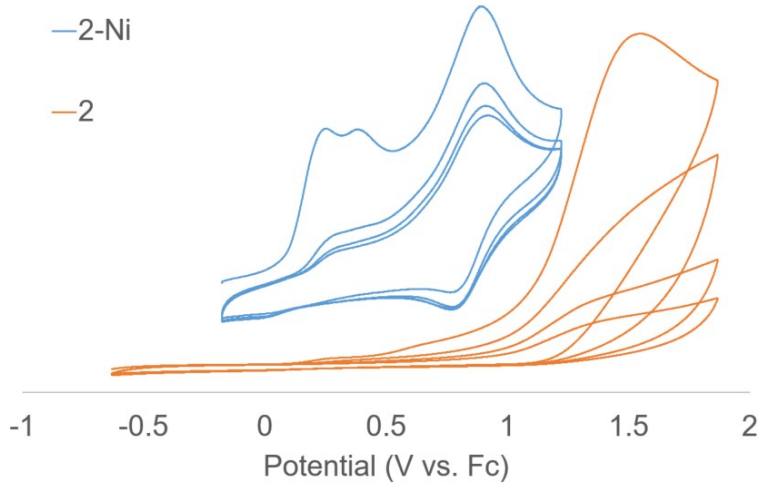

Fig. S12. Cyclic voltammetric repeat scans on **2** and **2-Ni** showing loss of the oxidation wave upon repeat scans. For **2-Ni** a quasi-reversible wave belonging to a decomposition product appears at 0.9 V after the first scan and persists upon repeat scans, even as the peak at 0.23 V disappears. The additional signal at 0.39 V is an additional decomposition product not present in fresh solution samples of **2-Ni**.

Cartesian coordinates of optimized geometries

Compound 1

7	1.392236698	1.378616580	0.034403976	6	4.642411711	-0.798114774	-1.558787527	1	-2.303032289	-3.877947800	2.089377097
7	-1.290448713	1.341344351	0.048550503	6	4.697677596	0.599229442	-1.548073630	6	2.445793643	-3.159522077	1.347013268
7	-1.392331622	-1.378694031	0.034673036	6	3.631423048	1.333059970	-1.016749614	1	2.761628851	-3.985709122	0.697357028
7	1.290280646	-1.341578396	0.047957996	6	2.519385630	0.685202959	-0.458317884	1	3.296137127	-2.482903759	1.461068169
6	1.297358112	2.523428031	0.769329876	1	0.465185046	0.948179551	-0.126034652	1	2.205336094	-3.590075477	2.323572072
6	0.046200952	3.008659840	1.132634177	1	-0.465401962	-0.948062090	-0.125351113	1	-5.451377640	1.375622554	-1.994618047
6	-1.226485630	2.443810541	0.783237548	1	0.030015009	3.895009993	1.755140010	1	-5.548336565	-1.119741959	-1.975265537
6	-2.463638616	0.742427547	-0.443397692	6	2.542158873	3.236629203	1.237928210	1	-3.476374114	2.537942347	-1.068499186
6	-3.531829588	1.456898670	-1.021844099	1	3.320016155	2.528213926	1.539573783	1	-3.649589825	-2.414314513	-1.066864891
6	-4.642330906	0.798230939	-1.558701514	1	2.967385798	3.875270338	0.454495486	1	-0.029826699	-3.894856225	1.755425252
6	-4.697565347	-0.599123279	-1.548288740	1	2.303242690	3.877474848	2.089488626	1	3.476450644	-2.537929228	-1.069138506
6	-3.631398563	-1.333066474	-1.016947749	6	-2.445749319	3.160308942	1.346384992	1	5.451566239	-1.375442850	-1.994596938
6	-2.519511833	-0.685313706	-0.458098120	1	-2.760375965	3.986971813	0.696707215	1	5.548558290	1.119922996	-1.974736725
6	-1.297332552	-2.523687624	0.769282842	1	-3.296717454	2.484379634	1.459587129	1	3.649602901	2.414329277	-1.066357320
6	-0.046139089	-3.008654119	1.132718713	1	-2.205690277	3.590406255	2.323251882				
6	1.226455965	-2.443746219	0.783102994	6	-2.542014609	-3.237160338	1.237793217				
6	2.463423845	-0.742565831	-0.444001110	1	-3.320037184	-2.528907957	1.539419147				
6	3.531797880	-1.456893230	-1.022291418	1	-2.967071133	-3.875882538	0.454340990				

Compound 2

7	3.513016554	-0.295954178	0.841801361	1	6.923423438	2.549098853	-2.674167970	6	0.138233680	0.937722718	-2.530744060
7	5.177917026	1.722575230	0.049113573	6	4.794582476	2.240717599	-2.787172718	1	-0.536782145	0.200571875	-2.110801642
1	4.319244626	1.595340091	-0.522319539	6	4.228193690	0.399511232	1.838470546	6	1.489349057	0.915056982	-2.161440148
7	3.689456668	1.915654964	-2.133240271	6	4.047036976	0.166637996	3.214369801	6	1.287243968	-2.017010145	-2.530986089
7	1.994446763	-0.047061421	-1.245128603	1	3.366712126	-0.619262378	3.524807163	1	1.537344032	-1.428995032	-3.419539181
1	2.578375732	0.278512925	-0.442272603	6	4.719869851	0.930621202	4.173271843	1	0.195568208	-2.034225895	-2.452306577
7	-2.096223963	-0.162588048	1.240842347	1	4.568935106	0.726501714	5.228345400	1	1.627262375	-3.040167043	-2.688895877
7	-3.657506137	1.831820402	2.305067288	6	5.576280352	1.958760351	3.767190441	6	4.291997734	-2.544448205	1.556020930
1	-3.832731096	1.613401064	1.304413769	1	6.091780327	2.566760721	4.503218774	1	4.969888860	-1.984482112	2.199208068
7	-5.079152945	1.742059806	0.074919850	6	5.746160060	2.227931491	2.405846031	1	4.862993246	-3.219810955	0.918106371
7	-3.529035347	-0.273999632	-0.915007906	1	6.363979049	3.062058984	2.098059694	1	3.657143587	-3.175732575	2.190480787
1	-3.037332669	0.178361937	-0.109038110	6	5.090160250	1.459054406	1.431408768	6	7.620150745	2.194677072	-0.134809589
8	3.536004754	-4.387252321	-0.467946443	6	2.379624627	1.923792371	-2.631413512	1	7.801178419	1.406853193	0.603307790
8	-3.607887562	-4.457902511	0.124191270	6	1.853307554	2.931264889	-3.467689511	1	7.776431022	3.156911249	0.367897241
6	1.944064326	-1.397604185	-1.314201940	1	2.500766671	3.737428184	-3.791096833	1	8.369841446	2.111988423	-0.925145365
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6	3.458821967	-1.608371514	0.694970122	1	0.131717296	3.721790504	-4.485749056	1	4.021578930	2.028728002	-4.811504168
6	6.236703719	2.086747202	-0.728400984	6	-0.353890918	1.930717063	-3.382941479	1	5.791709521	2.145622818	-4.695958114
6	6.045212570	2.309593669	-2.087020962	1	-1.404931554	1.939671541	-3.650121537	1	4.795662749	3.588481616	-4.497609151

6	2.505508496	-3.668899709	-0.410050427	6	-5.025594635	1.603746214	-1.317088718	1	-4.151940783	1.717987549	4.950488455
6	-2.586383936	-3.724404460	0.155848807	6	-5.628805533	2.494390620	-2.230153094	1	-5.835829409	2.270772549	4.819340907
6	-3.462043629	-1.624904245	-0.852630921	1	-6.205216561	3.328043599	-1.847193536	1	-4.509182597	3.428179191	4.692960738
6	-2.690872762	-2.234807271	0.165483698	6	-5.466673244	2.340874077	-3.608972709	6	-7.542588601	2.210783116	0.240367776
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6	0.373129791	1.490332105	3.522150338	1	-4.617418939	-3.336846748	-1.435499795	6	1.172751962	-5.771845477	-0.335920977
1	1.423377869	1.415759881	3.782853145	1	-3.572534183	-2.721795742	-2.720439683	1	2.118237831	-6.278579737	-0.490305176
6	-0.425746422	2.495096288	4.076827565	6	-1.556319465	-2.258216596	2.460654713	6	1.175283429	-4.363513615	-0.299174585
1	-0.003390567	3.206117060	4.779260721	1	-1.644131842	-1.646359895	3.361787999	6	-0.039644072	-3.687219191	-0.098606477
6	-1.764997825	2.613159273	3.694987296	1	-0.503072245	-2.536646455	2.354866762	1	-0.049282963	-2.605290904	-0.069293259
1	-2.361028528	3.432074454	4.077522311	1	-2.125305629	-3.179245862	2.609313424				
6	-2.334192971	1.713446658	2.779909549	6	-4.823441200	2.410063008	4.432952831				

Compound 3

6	-0.513673855	-0.464205077	-3.115113332	6	2.497675969	-3.336629516	-0.228378266	1	-2.241284507	-1.569377586	-4.803640996
6	0.907868644	-0.439843166	-3.045228755	6	0.613458553	-3.042017152	1.253528815	6	2.800631643	1.211168293	1.962320189
6	1.612932985	0.612195248	-3.654633461	6	0.545107839	-3.232698517	3.696823165	6	2.391852569	3.122962919	0.409525248
1	2.683151754	0.685634826	-3.533328795	1	1.075245026	-3.306527607	4.640322689	6	3.261387602	1.991852894	0.909667460
6	-0.448615959	1.595148814	-4.443904783	6	-0.810307362	-2.995157057	1.235186024	6	3.678769780	0.248520241	2.724334841
1	-0.980475810	2.382802172	-4.967056497	6	1.261699910	-3.135765617	2.503356063	1	4.171547842	0.741255689	3.569954825
6	-3.311145287	1.769717975	1.115497661	1	2.342911399	-3.103152539	2.537436670	1	4.469447323	-0.139790191	2.088730262
6	-1.163764055	0.580208081	-3.806542649	6	-1.517563815	-3.054737478	2.448930346	1	3.097490787	-0.591326720	3.110491132
1	-2.245232846	0.613699471	-3.811464256	1	-2.591217144	-2.943845067	2.455429680	6	2.799167764	4.515309231	0.839415756
6	0.946818862	1.610261958	-4.369196338	6	-0.851358154	-3.198181978	3.668166973	1	3.728104018	4.805785847	0.341686311
1	1.515885556	2.411005922	-4.828417969	1	-1.423364158	-3.241861856	4.588398505	1	3.012903429	4.495574481	1.915020634
6	3.106337223	-4.507231113	0.533333651	6	-3.161784818	-2.637026490	-1.671046188	1	2.023427631	5.256273275	0.638369365
1	3.885291764	-4.191626721	1.238353218	6	-2.699105564	-3.069024189	-0.423696758	6	-1.004227540	5.680704539	-2.331011335
1	3.583421078	-5.188270193	-0.173652639	6	-3.600190922	-3.936263048	0.430693170	1	-1.614167333	6.430622626	-2.823642794
1	2.340684018	-5.043647787	1.095629389	1	-3.015947679	-4.670167460	0.991098155	6	1.147459009	4.748052286	-1.782481593
6	3.279622870	-2.791453794	-1.324761021	1	-4.304578304	-4.479244410	-0.201559188	1	2.227368653	4.743645605	-1.879138701
6	2.800681899	-1.916899372	-2.311612766	1	-4.185007201	-3.353167950	1.151033120	6	0.546687581	3.777812536	-0.959728443
6	3.684642119	-1.595110237	-3.499254580	6	-2.386705240	-1.923432139	-2.669719018	6	-1.621536418	4.712861258	-1.534831196
1	3.083795336	-1.469049061	-4.403477039	6	-2.999689455	-1.820914435	-4.061109550	1	-2.692018764	4.757053909	-1.402161306
1	4.388572492	-2.411104032	-3.671542796	1	-3.791372571	-1.063087205	-4.116076493	6	0.385610904	5.685158041	-2.482125736
1	4.268784971	-0.680047868	-3.356611714	1	-3.458616614	-2.776099573	-4.324730119	1	0.872513946	6.417399969	-3.116959948

6	-0.873372293	3.707017971	-0.892394786	6	-0.531484065	0.135964595	5.883214004	6	4.767183647	-2.901177119	-1.186503479
6	-2.523918271	1.749606700	2.389646783	1	-1.058034981	-0.239553823	6.753903146	1	2.580173464	0.952894076	4.826118387
6	-2.786464457	2.280768935	-0.066553450	6	0.849651029	0.345119224	5.917383667	6	5.459962218	-1.624521750	-0.792295136
6	-3.145001871	2.430824330	3.598523871	1	1.413298577	0.170813237	6.827753377	6	4.724273685	-0.501738560	-0.362637530
1	-3.930188326	1.833023772	4.069885992	6	1.517138335	0.778194447	4.770449591	6	6.865147052	-1.556514673	-0.800117572
1	-3.628131090	3.354536276	3.273353300	6	-1.236254189	0.435448450	4.717615699	6	5.366355261	0.685940331	0.014302586
1	-2.382005141	2.657896140	4.345988693	1	-2.304798180	0.266497013	4.687525125	1	3.643606979	-0.541946957	-0.327929864
6	-3.636124899	2.478397423	-1.305285954	6	-4.649094791	-2.629955205	-1.874900134	6	7.514006256	-0.378201630	-0.420342325
1	-4.283799471	3.358723396	-1.227323889	8	-5.258294398	-3.353583841	-2.696092254	1	7.428513067	-2.428850658	-1.112183583
1	-4.289534239	1.620949698	-1.457962126	6	-5.396480898	-1.591550345	-1.085098979	6	6.771835005	0.743502068	-0.034868564
1	-3.010177869	2.588338669	-2.191075053	6	-4.717784226	-0.606297298	-0.338945803	1	8.597486513	-0.327611209	-0.436470902
7	1.484361435	1.373737772	2.370161223	6	-6.802805580	-1.564416300	-1.136714853	1	7.268133996	1.671090216	0.226106593
7	-1.205390805	-1.423941575	-2.351233002	6	-5.424412143	0.387116823	0.354971320	6	4.598705718	1.950937640	0.293734396
7	1.335592152	2.811241313	-0.283179269	1	-3.635987054	-0.607136906	-0.296137594	8	5.421437585	-3.967705305	-1.272634638
7	-1.325440908	1.215283541	2.375365284	6	-7.514005250	-0.582378512	-0.443170263	8	5.131395720	3.019636876	-0.135355369
7	1.311422997	-2.822137449	0.049598990	1	-7.316474708	-2.317508960	-1.723736568	1	-0.903835212	-2.241464967	-0.714852200
7	-1.464859613	2.689583649	-0.115533752	6	-6.832278160	0.382547745	0.304123804	1	1.012146899	-1.778016998	-1.434459322
7	-1.435382001	-2.779535383	-0.012766675	1	-8.597868738	-0.568170876	-0.482453619	1	-0.860865157	2.212222796	0.544339516
7	1.536509360	-1.426693968	-2.252409982	1	-7.374563076	1.140448458	0.857009095	1	0.870557007	1.748286767	1.658283169
6	-0.596503624	0.923922802	3.559741894	6	-4.752722730	1.398275981	1.245506541				
6	0.830080482	1.005089172	3.562343610	8	-5.453147050	1.877753823	2.178708577				

Compound 4

7	0.482106474	1.492513469	-0.335022803	6	2.380171849	4.286254896	-1.955040467	1	4.479853551	2.396972951	1.657616728
7	-0.248232782	-1.001038120	-0.344479726	6	1.310830493	3.510655127	-1.495134256	1	5.087228354	1.120085612	2.734368459
7	2.304034191	-1.869913766	0.202713326	6	1.540254305	2.338551741	-0.761440002	1	-1.087141973	-5.110430718	-2.288203357
7	3.037516627	0.675923741	0.151071093	1	0.576269219	0.465065982	-0.502527659	1	1.228354642	-5.901754893	-1.786915791
6	-0.668973284	1.799287400	0.303468409	1	2.210916178	-0.865326772	-0.044779051	1	-1.777708181	-2.827696805	-1.614088481
6	-1.608327900	0.771205634	0.570134473	6	-0.912085239	3.237046253	0.702671132	1	2.824239182	-4.396635375	-0.656218911
6	-1.342400600	-0.638809456	0.305462061	1	0.032235445	3.775646656	0.795150434	1	4.793506193	-1.166179903	2.314051538
6	0.094234409	-2.329245910	-0.680044222	1	-1.521303743	3.758065192	-0.047010412	1	4.953629032	2.357726658	-0.842789251
6	-0.783498639	-3.185543976	-1.370870653	1	-1.445480115	3.276426227	1.649212356	1	4.529631085	4.452583613	-2.080679619
6	-0.389606981	-4.469005714	-1.759702999	6	-2.273379588	-1.678900888	0.920775266	1	2.186274542	5.191249525	-2.520645786
6	0.906523777	-4.913024669	-1.477272656	1	-3.105571557	-1.938501556	0.259809420	1	0.292885274	3.801390325	-1.726278159
6	1.806349937	-4.067636058	-0.820898940	1	-1.719011132	-2.596746041	1.130855369	6	-2.910449675	1.142113451	1.189061591
6	1.419426597	-2.781502473	-0.409725573	1	-2.702003915	-1.313626999	1.856691442	6	-4.185803020	0.709892317	0.530309466
6	3.289443982	-2.060090941	1.127233881	6	3.517153693	-3.420197631	1.739622834	6	-4.243399979	0.388648104	-0.838768795
6	4.055624919	-0.976696252	1.544232739	1	2.572438083	-3.943714875	1.916831462	6	-5.374364094	0.701382713	1.285341819
6	3.950256955	0.371152986	1.063668438	1	4.132001336	-4.061113107	1.096503828	6	-5.462397386	0.052111423	-1.436502888
6	2.873285287	1.910464592	-0.493279206	1	4.041155954	-3.313555033	2.691956231	1	-3.336833921	0.419144134	-1.433437396
6	3.934716686	2.693394080	-0.993895534	6	4.893241627	1.385962307	1.691104731	6	-6.589063005	0.354765428	0.691252217
6	3.693112728	3.870581563	-1.707239335	1	5.861824607	1.403356537	1.175259992	1	-5.325055199	0.968318561	2.335026166

6	-6.635846877	0.028378384	-0.672055318	1	-7.497580329	0.339848666	1.284607379	8	-2.993075390	1.857921594	2.225078400
1	-5.497550101	-0.185816794	-2.494706470	1	-7.580756886	-0.237718360	-1.135070149				

Compound 2-Ni

7	3.550698382	-0.673739816	0.182038803	6	0.546361650	-2.098216548	-2.712377197	1	-5.842015809	2.739016525	-2.033930878
7	4.535527241	1.590656729	-0.188729272	1	0.351557381	-1.392592702	-3.522374392	6	-5.576790531	1.114671213	-3.408455231
7	2.704707503	2.058928497	-2.068764050	1	-0.410866826	-2.372408268	-2.257954966	1	-6.065832067	1.636775273	-4.222251918
7	1.660719749	-0.164595384	-1.581180615	1	0.983745718	-3.006108486	-3.135767998	6	-5.026624403	-0.148731724	-3.614570430
7	-1.660292947	-0.164418368	1.580598763	6	4.194500953	-3.056304514	0.586145656	1	-5.081785025	-0.618115376	-4.589351230
7	-2.704863991	2.058597915	2.069085637	1	5.093284697	-2.629591544	1.030052956	6	-4.363656599	-0.801357688	-2.569274545
7	-4.535179053	1.590897627	0.188302032	1	4.480613767	-3.776314457	-0.181375413	1	-3.889061840	-1.754201096	-2.753947118
7	-3.550762042	-0.673646480	-0.182212278	1	3.637487028	-3.597293407	1.361816880	6	-4.271204613	-0.201920700	-1.312271993
8	3.115243567	-4.560176138	-1.779657749	6	6.670534234	2.919446652	-0.338072098	6	-4.195264550	-3.056075524	-0.585502198
8	-3.115458911	-4.560018327	1.779062690	1	7.173235952	2.107239988	0.192530686	1	-5.094158671	-2.629205266	-1.029036200
6	1.523883615	-1.494273819	-1.705913915	1	6.604715221	3.783005490	0.334752097	1	-4.481119808	-3.775929909	0.182254205
6	2.366343246	-2.389238592	-1.007966373	1	7.281356018	3.214717952	-1.195133191	1	-3.638722324	-3.597261148	-1.361373938
6	3.344367950	-1.976591423	-0.075707795	6	3.304487977	3.757300610	-3.828071056	6	-0.545847687	-2.098036310	2.711650113
6	5.296225887	2.494180330	-0.836022767	1	2.595624773	3.257765103	-4.492385849	1	-0.351165472	-1.392572937	3.521837261
6	4.872472578	3.063572642	-2.037945265	1	4.240896161	3.922765504	-4.367921312	1	0.411424915	-2.371836820	2.257105541
1	5.560487586	3.726069479	-2.544876320	1	2.897600135	4.740380280	-3.562500696	1	-0.982947237	-3.006143015	3.134827311
6	3.595062888	2.918635407	-2.591020000	6	2.230312223	-3.870518210	-1.238690781	6	-3.305462912	3.755865005	3.829135030
6	4.271066921	-0.201891285	1.312084707	6	-2.230312953	-3.870292420	1.238467817	1	-2.596842113	3.255976188	4.493449490
6	4.363239485	-0.801110309	2.569218511	6	-3.344592882	-1.976447973	0.075766633	1	-4.242080756	3.921049171	4.368702955
1	3.888327908	-1.753767546	2.754022780	6	-2.366293514	-2.389085744	1.007748431	1	-2.898463297	4.739085938	3.564258226
6	5.026291101	-0.148487014	3.614458081	6	-1.523530288	-1.494074875	1.705368688	6	-6.670517036	2.919103713	0.337901680
1	5.081254191	-0.617705941	4.589328486	6	-3.595523563	2.917868560	2.591507631	1	-7.173059789	2.106938685	-0.192932584
6	5.576831754	1.114720612	3.408145355	6	-4.872790476	3.062885246	2.038144193	1	-6.604725751	3.782791205	-0.334750312
1	6.065981401	1.636835387	4.221872911	1	-5.561038565	3.725017036	2.545226038	1	-7.281470850	3.214115611	1.194951180
6	5.466590639	1.735106375	2.158642832	6	-5.296193660	2.493940893	0.835882011	6	-1.039828632	-4.550674522	0.627137806
1	5.842614520	2.738745411	2.033392139	6	-0.733512381	0.771022121	2.123316097	6	-1.022435777	-5.954232343	0.638832824
6	4.824107984	1.085329197	1.102101100	6	0.642786344	0.605277404	2.274927723	1	-1.835377992	-6.463494112	1.141787282
6	1.328967546	2.021734184	-2.413970267	1	1.123302401	-0.310544513	1.965767859	6	0.000293187	-6.653259296	0.000658300
6	0.530212600	3.070949474	-2.871833717	6	1.431072228	1.656500609	2.757894812	1	0.000378895	-7.736946461	0.000943507
1	0.954423150	4.048816281	-3.037366605	1	2.498846753	1.515292993	2.865842880	6	1.022906380	-5.954399245	-0.637874426
6	-0.843082822	2.882480372	-3.057614413	6	0.842622465	2.882587038	3.058721198	1	1.835903041	-6.463796801	-1.140609450
1	-1.453267816	3.709167611	-3.401588515	1	1.452557956	3.709266752	3.403156266	6	1.040082166	-4.550832037	-0.626857167
6	-1.431162833	1.656049718	-2.757458627	6	-0.530723553	3.070769003	2.872991642	6	0.000068134	-3.858452011	-0.000050106
1	-2.498900189	1.514605814	-2.865477552	1	-0.955213740	4.048426146	3.039030423	1	-0.000017043	-2.777794242	-0.000374083
6	-0.642577727	0.604792090	-2.275071802	6	-1.329166697	2.021592084	2.414495820	28	-3.106742139	0.707362299	0.908535894
1	-1.122844721	-0.311319462	-1.966376995	6	-4.823935134	1.085448902	-1.102467666	28	3.107272936	0.707045370	-0.909230238
6	0.733686687	0.770838142	-2.123389970	6	-5.466291813	1.735245475	-2.159065364				

Full crystallographic tables

Crystal data and structure refinement for **2**.

Identification code	**2**
Empirical formula	$C_{54.5}H_{56}N_8O_2$
Formula weight	855.07
Temperature/K	100.0
Crystal system	triclinic
Space group	P-1
a/Å	11.74(2)
b/Å	14.45(3)
c/Å	15.40(3)
α/°	62.50(5)
β/°	82.90(3)
γ/°	73.44(3)
Volume/Å^3	2221(7)
Z	2
ρ_{calc}g/cm^3	1.279
μ/mm^{-1}	0.080
F(000)	910.0
Crystal size/mm^3	0.18 × 0.11 × 0.06
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.29 to 46.512
Index ranges	$-13 \leq h \leq 12$, $-15 \leq k \leq 16$, $-12 \leq l \leq 17$
Reflections collected	6369
Independent reflections	5073 [R_{int} = 0.1086, R_{sigma} = 0.2102]
Data/restraints/parameters	5073/548/613
Goodness-of-fit on F^2	0.965
Final R indexes [I>=2σ (I)]	R_1 = 0.0875, wR_2 = 0.1863
Final R indexes [all data]	R_1 = 0.1988, wR_2 = 0.2453
Largest diff. peak/hole / e Å^{-3}	0.41/-0.37

Bond Lengths for **2**

Atom	Atom	Length/Å	Atom	Atom	Length/Å
N1	C2AA	1.271(10)	C15	C16	1.352(11)
N1	C7	1.436(10)	C16	C17	1.389(10)
N2	C3AA	1.342(10)	C17	C18	1.374(10)
N2	C12	1.398(10)	C23	C51	1.478(11)
N3	C6	1.286(9)	C24	C26	1.467(12)
N3	C13	1.396(9)	C24	C47	1.476(11)
N4	C0AA	1.317(10)	C25	C26	1.432(10)
N4	C18	1.425(9)	C25	C43	1.502(11)
N5	C27	1.299(10)	C26	C27	1.428(11)
N5	C31	1.444(11)	C27	C44	1.492(9)
N6	C28	1.284(9)	C28	C29	1.408(10)
N6	C36	1.395(9)	C28	C45	1.525(12)
N7	C30	1.323(10)	C29	C30	1.384(11)
N7	C37	1.417(10)	C30	C46	1.477(10)
N8	C25	1.290(10)	C31	C32	1.385(10)
N8	C42	1.423(9)	C31	C36	1.360(11)
O1	C23	1.208(8)	C32	C33	1.384(12)
O2	C24	1.209(9)	C33	C34	1.347(12)
C0AA	C1AA	1.380(9)	C34	C35	1.377(10)
C0AA	C19	1.526(11)	C35	C36	1.423(12)
C1AA	C2AA	1.481(11)	C37	C38	1.412(10)
C1AA	C23	1.469(12)	C37	C42	1.366(11)
C2AA	C20	1.494(10)	C38	C39	1.386(11)
C3AA	C5	1.370(12)	C39	C40	1.332(11)
C3AA	C21	1.499(10)	C40	C41	1.375(10)
C5	C6	1.427(11)	C41	C42	1.374(11)
C6	C22	1.511(12)	C47	C48	1.356(11)
C7	C8	1.345(11)	C47	C52	1.380(10)
C7	C12	1.380(11)	C48	C49	1.362(10)
C8	C9	1.379(12)	C49	C50	1.377(11)
C9	C10	1.347(12)	C50	C51	1.389(11)
C10	C11	1.361(12)	C51	C52	1.368(10)
C11	C12	1.428(12)	C2	C3	1.57(2)
C13	C14	1.376(10)	C3	C4	1.56(2)
C13	C18	1.373(11)	C4	C1	1.57(2)
C14	C15	1.383(10)	C1	C1A	1.59(2)

Bond Angles for **2**

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2AA	N1	C7	122.3(7)	N8	C25	C26	120.2(7)

C3AA	N2	C12	128.2(7)	N8	C25	C43	123.1(7)
C6	N3	C13	124.4(7)	C26	C25	C43	116.7(7)
C0AA	N4	C18	127.6(7)	C25	C26	C24	120.1(7)
C27	N5	C31	128.7(6)	C27	C26	C24	117.6(7)
C28	N6	C36	125.9(7)	C27	C26	C25	121.9(8)
C30	N7	C37	131.3(7)	N5	C27	C26	120.9(7)
C25	N8	C42	123.2(6)	N5	C27	C44	118.2(7)
N4	C0AA	C1AA	123.1(7)	C26	C27	C44	120.5(8)
N4	C0AA	C19	117.6(7)	N6	C28	C29	121.6(8)
C1AA	C0AA	C19	119.1(8)	N6	C28	C45	123.7(7)
C0AA	C1AA	C2AA	121.7(8)	C29	C28	C45	114.6(8)
C0AA	C1AA	C23	120.1(7)	C30	C29	C28	126.1(8)
C23	C1AA	C2AA	118.1(7)	N7	C30	C29	119.2(7)
N1	C2AA	C1AA	118.5(7)	N7	C30	C46	120.4(8)
N1	C2AA	C20	124.1(7)	C29	C30	C46	120.4(8)
C1AA	C2AA	C20	117.5(8)	C32	C31	N5	120.3(8)
N2	C3AA	C5	120.9(7)	C36	C31	N5	117.2(7)
N2	C3AA	C21	118.8(8)	C36	C31	C32	122.2(9)
C5	C3AA	C21	120.1(9)	C33	C32	C31	120.1(9)
C3AA	C5	C6	124.2(8)	C34	C33	C32	118.6(8)
N3	C6	C5	121.2(8)	C33	C34	C35	122.3(9)
N3	C6	C22	123.6(7)	C34	C35	C36	119.8(8)
C5	C6	C22	115.1(8)	N6	C36	C35	122.8(8)
C8	C7	N1	123.5(8)	C31	C36	N6	119.9(8)
C8	C7	C12	121.3(8)	C31	C36	C35	116.9(7)
C12	C7	N1	115.1(7)	C38	C37	N7	121.0(8)
C7	C8	C9	121.1(8)	C42	C37	N7	119.0(7)
C10	C9	C8	119.0(8)	C42	C37	C38	119.7(8)
C9	C10	C11	121.9(8)	C39	C38	C37	118.2(9)
C10	C11	C12	119.3(9)	C40	C39	C38	121.1(8)
N2	C12	C11	122.0(8)	C39	C40	C41	120.9(8)
C7	C12	N2	120.5(7)	C42	C41	C40	119.9(8)
C7	C12	C11	117.2(8)	C37	C42	N8	117.1(7)
C14	C13	N3	124.1(8)	C37	C42	C41	120.1(7)
C18	C13	N3	117.8(7)	C41	C42	N8	122.8(8)
C18	C13	C14	117.5(7)	C48	C47	C24	120.2(7)
C13	C14	C15	121.1(8)	C48	C47	C52	118.4(8)
C16	C15	C14	120.1(7)	C52	C47	C24	121.5(8)
C15	C16	C17	120.5(7)	C47	C48	C49	120.3(8)
C18	C17	C16	118.2(8)	C48	C49	C50	121.6(9)
C13	C18	N4	117.3(6)	C49	C50	C51	119.0(8)
C13	C18	C17	122.7(7)	C50	C51	C23	117.9(8)
C17	C18	N4	120.0(7)	C52	C51	C23	124.0(8)

O1	C23	C1AA	121.8(7)	C52	C51	C50	118.0(7)
O1	C23	C51	120.4(8)	C51	C52	C47	122.7(8)
C1AA	C23	C51	117.7(7)	C4	C3	C2	106.1(17)
O2	C24	C26	120.6(8)	C3	C4	C1	109(2)
O2	C24	C47	119.1(9)	C4	C1	C1A	104.8(18)
C26	C24	C47	120.3(7)				

Experimental
Single crystals of $C_{54.5}H_{56}N_8O_2$ were mounted on a MiTeGen loop with paratone-N oil. The crystal was kept at 100.0 K during data collection. The structure was solved with the XS and refined with XL.

Crystal Data for $C_{54.5}H_{56}N_8O_2$ (M =855.07 g/mol): triclinic, space group P-1 (no. 2), a = 11.74(2) Å, b = 14.45(3) Å, c = 15.40(3) Å, α = 62.50(5)°, β = 82.90(3)°, γ = 73.44(3)°, V = 2221(7) Å^3, Z = 2, T = 100.0 K, μ(MoKα) = 0.080 mm^{-1}, *Dcalc* = 1.279 g/cm^3, 6369 reflections measured (3.29° ≤ 2Θ ≤ 46.512°), 5073 unique (R_{int} = 0.1086, R_{sigma} = 0.2102) which were used in all calculations. The final R_1 was 0.0875 (I > 2σ(I)) and wR_2 was 0.2453 (all data).
Refinement model description
Number of restraints - 548, number of constraints - 0
Details:
1. Fixed Uiso
At 1.2 times of:
All C(H) groups, All C(H,H) groups, All N(H) groups
At 1.5 times of:
All C(H,H,H) groups
2. Restrained distances
C2-C3 ≈ C3-C4 ≈ C4-C1 ≈ C1-C1A
with sigma of 0.02
C2-C4 ≈ C4-C1A ≈ C3-C1
with sigma of 0.04
3. Restrained planarity
C2, C3, C4, C1, C1A
with sigma of 0.1
4. Uiso/Uaniso restraints and constraints
C2 ≈ C3 ≈ C4 ≈ C1 ≈ C1A: within 2A with sigma of 0.04 and sigma
for terminal atoms of 0.08
5. Rigid body (RIGU) restrains
C51, N1, C2AA, C7, C1AA, C20, C8, C12, C0AA, C23, C9, N2, C11, N4, C19, O1,
C10, C3AA, C18, C50, C52, C5, C21, C13, C17, C49, C47, C6, N3, C14, C16, C48,
C24, C22, C15, O2, C26, C25, C27, N8, C43, N5, C44, C42, C31, C37, C41, C32,
C36, N7, C38, C40, C33, N6, C35, C30, C39, C34, C28, C29, C46, C45
with sigma for 1-2 distances of 0.01 and sigma for 1-3 distances of 0.01
C2, C3, C4, C1, C1A
with sigma for 1-2 distances of 0.004 and sigma for 1-3 distances of 0.004
C4, C2, C3, C1, C1A
with sigma for 1-2 distances of 0.001 and sigma for 1-3 distances of 0.001
C43, C25, N8, C26
with sigma for 1-2 distances of 0.001 and sigma for 1-3 distances of 0.001
6. Others
Fixed Sof: C2(0.5) H2A(0.5) H2B(0.5) H2C(0.5) C3(0.5) H3A(0.5) H3B(0.5)
C4(0.5) H4A(0.5) H4B(0.5) C1(0.5) H1A(0.5) H1B(0.5) C1A(0.5) H1AA(0.5)
H1AB(0.5) H1AC(0.5)
7.a Secondary CH2 refined with riding coordinates:
C3(H3A,H3B), C4(H4A,H4B), C1(H1A,H1B)

7.b Me refined with riding coordinates:
C2(H2A,H2B,H2C), C1A(H1AA,H1AB,H1AC)
7.c Aromatic/amide H refined with riding coordinates:
N2(H2), N4(H4), N5(H5), N7(H7), C5(H5A), C8(H8), C9(H9), C10(H10), C11(H11), C14(H14), C15(H15), C16(H16), C17(H17), C29(H29), C32(H32), C33(H33), C34(H34), C35(H35), C38(H38), C39(H39), C40(H40), C41(H41), C48(H48), C49(H49), C50(H50), C52(H52)
7.d Idealised Me refined as rotating group:
C19(H19A,H19B,H19C), C20(H20A,H20B,H20C), C21(H21A,H21B,H21C), C22(H22A,H22B, H22C), C43(H43A,H43B,H43C), C44(H44A,H44B,H44C), C45(H45A,H45B,H45C), C46(H46A, H46B,H46C)
This report has been created with Olex2, compiled on 2018.05.29 svn.r3508 for OlexSys. Please let us know if there are any errors or if you would like to have additional features.