Supporting Information

Stimuli responsive AIE active positional isomers of phenanthroimidazole as non- doped emitters in OLEDs

Anupama Ekbote[1], Si Hyun Han[2], Thaksen Jadhav[1], Shaikh M. Mobin[1], Jun Yeob Lee[2] and Rajneesh Misra[1*]

[1]Department of Chemistry, Indian Institute of Technology Indore, 452 020, India

[2]School of Chemical Engineering, Sungkyunkwan University, Suwon, Gyeonggi, 446-740, Korea

*** Corresponding author**

Email: rajneeshmisra@iiti.ac.in

Table of Contents

Introduction

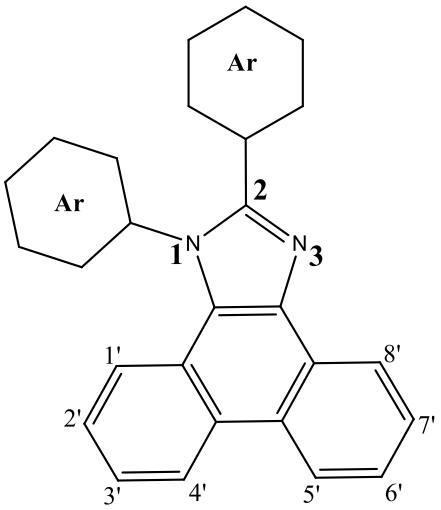
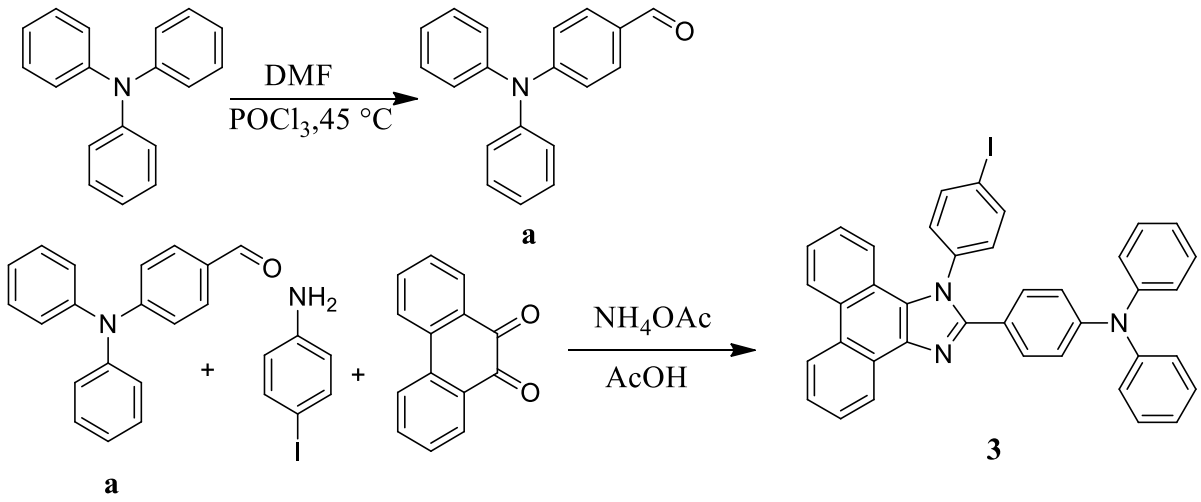


Fig. S1 Chemical structure of phenanthroimidazole

Experimental section

Scheme S1. Synthetic scheme for intermediates 3 and 4



Compounds **a** and **b** were prepared according to the synthetic route reported in the literature as shown in Scheme S1.

Thermogravimetric analysis

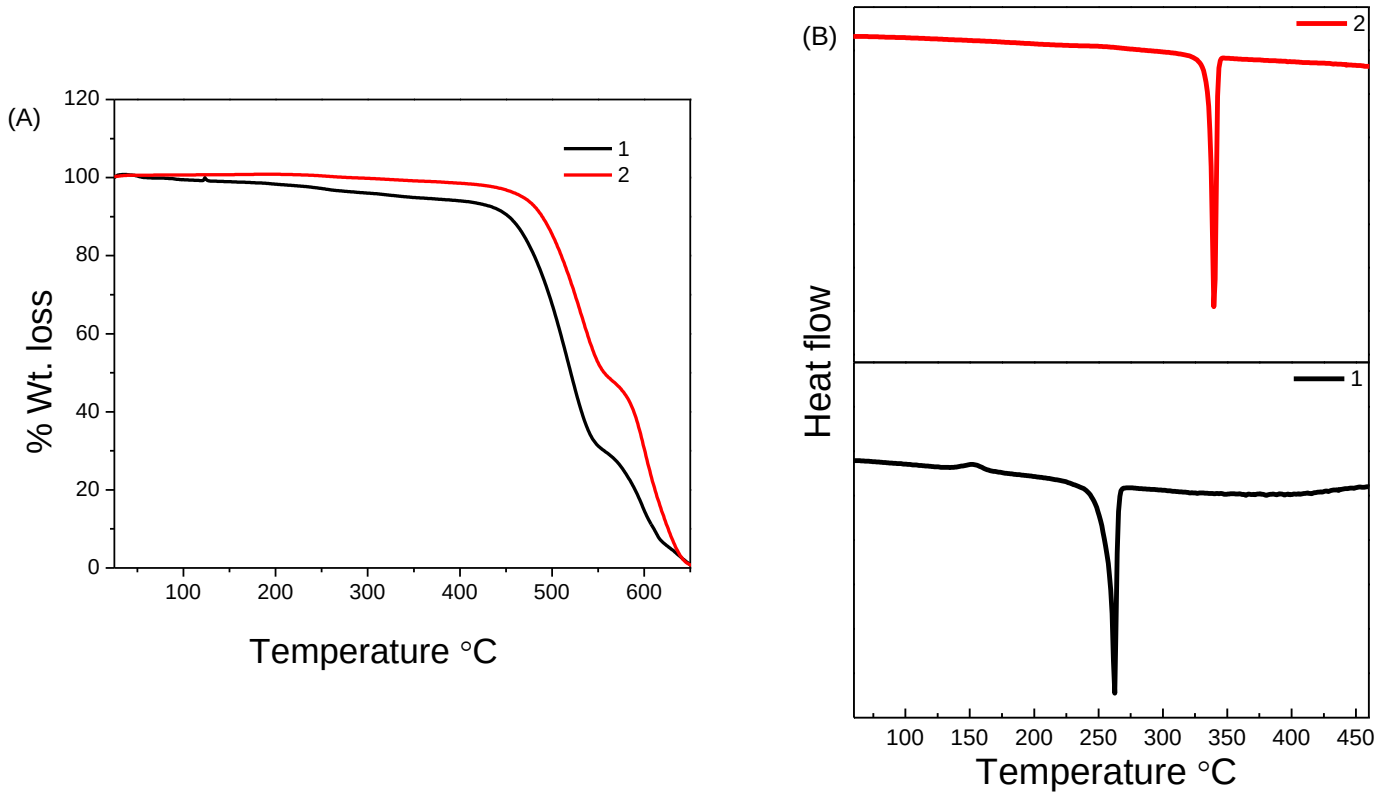


Fig. S2 Thermogravimetric analysis (A) and differential scanning calorimetry (B) curves of luminophores **1** and **2**.

Photophysical properties

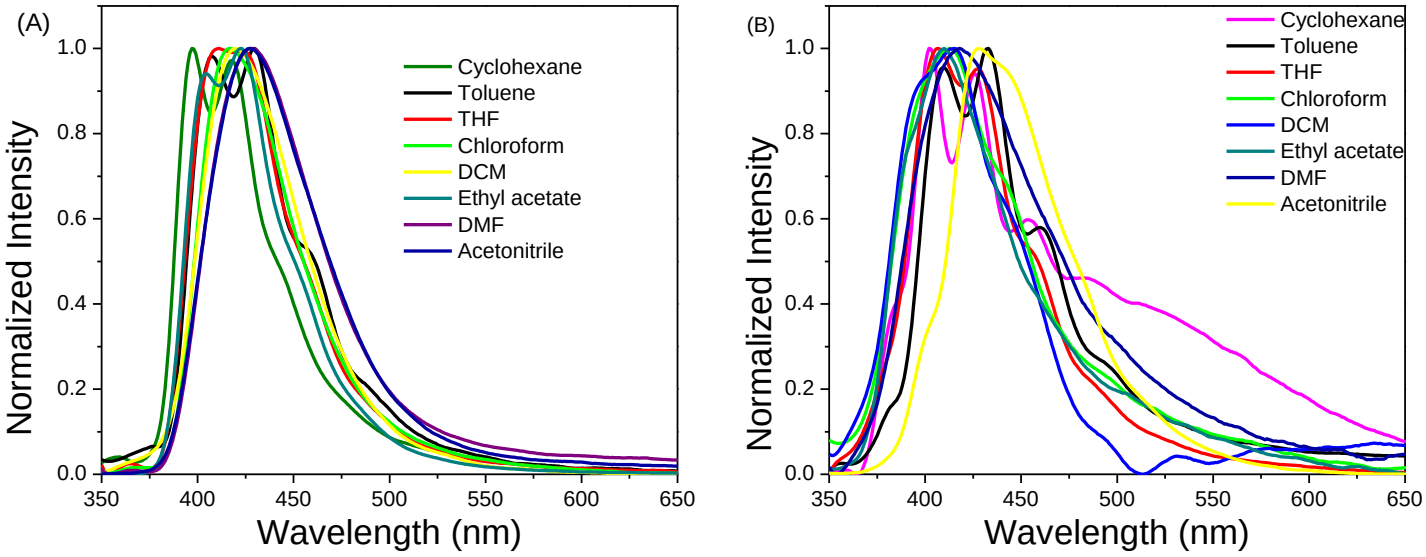


Fig. S3 (A) Fluorescence spectra of **1** (A) and **2** (B) in different solvents of varying polarities. (excitation wavelength or λ_{ex} = 340 nm).

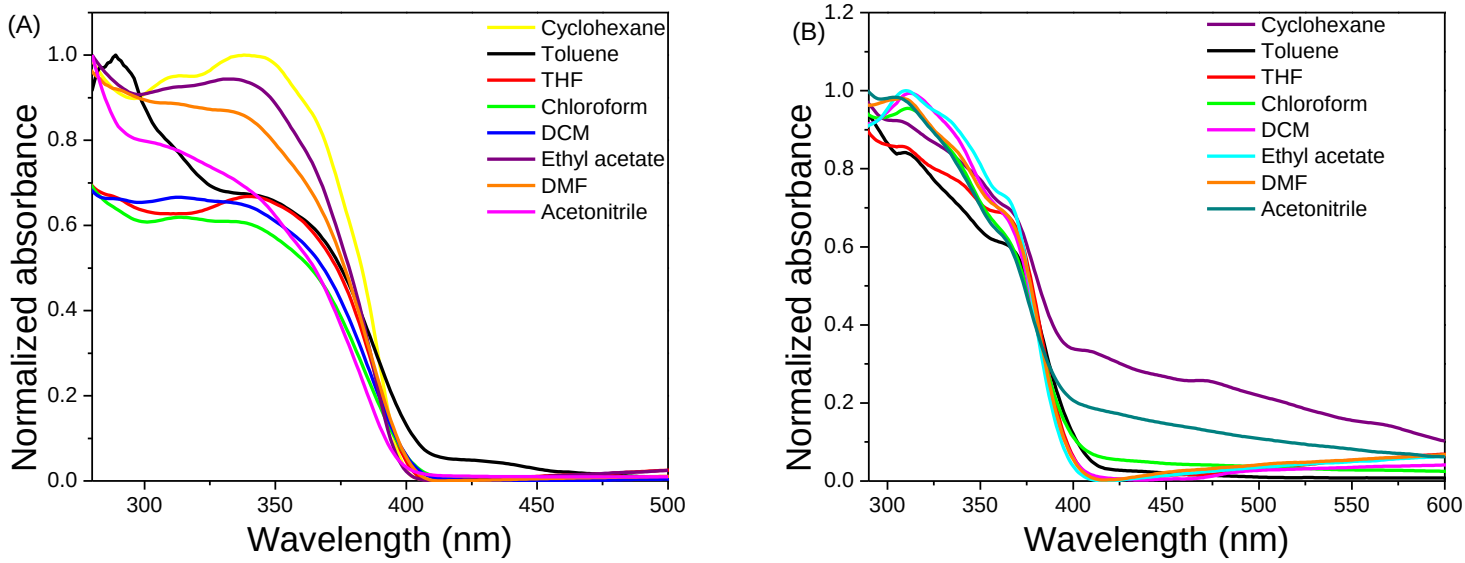


Fig. S4 UV-vis absorption spectra of 1 (A) and 2 (B) in different solvents of varying polarities.

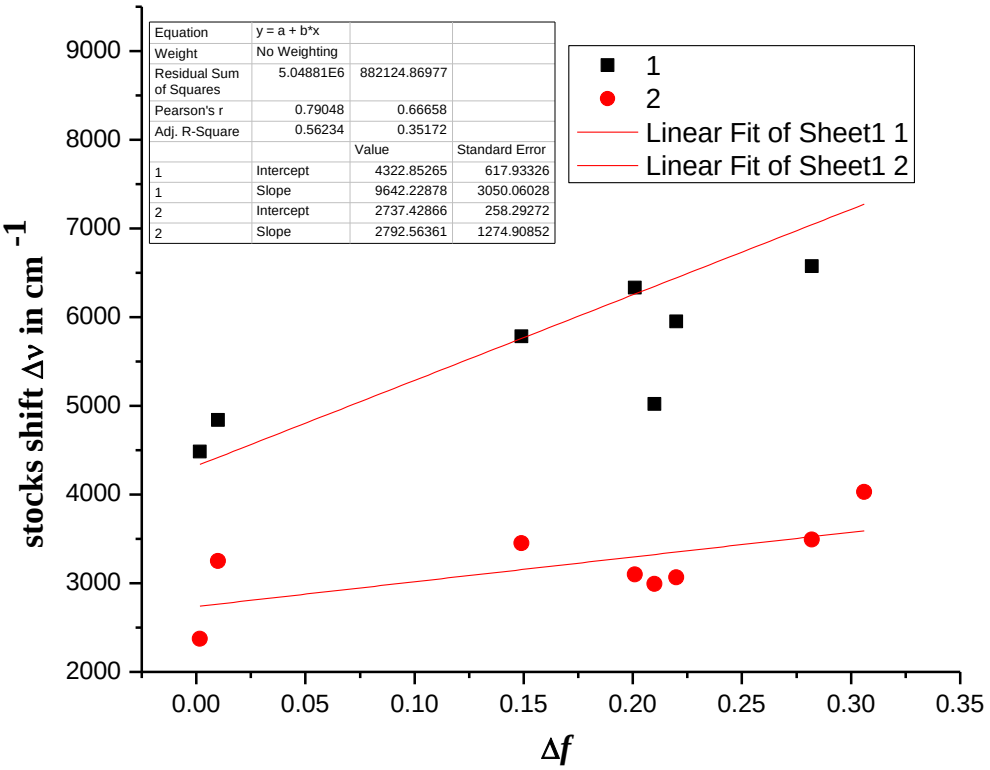


Fig. S5 Stokes Shift ($\Delta\nu$) of **1** and **2** as a function of the solvent polarity parameter (Δf).

Table S1 Solvatochromic properties of luminophores **1** and **2**.

Compounds	**Solvent**	**Δ*f***	**λ_{ab}(nm)**	**λ_{em}(nm)**	**Stokes shift (cm^{-1})**	**Φ_f[a]**
1	Cyclohexane	0.014	337	397	4484.67	0.0187
	Toluene		340	407	4841.74	0.0438
	Chloroform	0.15	337	417	5782.38	0.0153
	DCM		336	420	5952.38	0.0207
	Ethyl acetate	0.21	333	422	6333.35	0.01931
	DMF	0	334	428	6575.63	0.0208
	Acetonitrile		310	428	8893.57	0.0115
2	Cyclohexane	0.014	367	402	2372.34	0.0081
	Toluene		361	409	3250.95	0.0276
	Chloroform	0.15	365	411	3451.82	0.0100
	DCM		363	415	3066.36	0.0111
	Ethyl acetate	0.21	363	409	3098.33	0.0059
	DMF	0.26	364	417	3491.71	0.0118
	Acetonitrile		365	428	4026.77	0.0284

[a] The fluorescence quantum yields were recorded using 9,10-diphenylanthracene as a standard in ethanol solution.

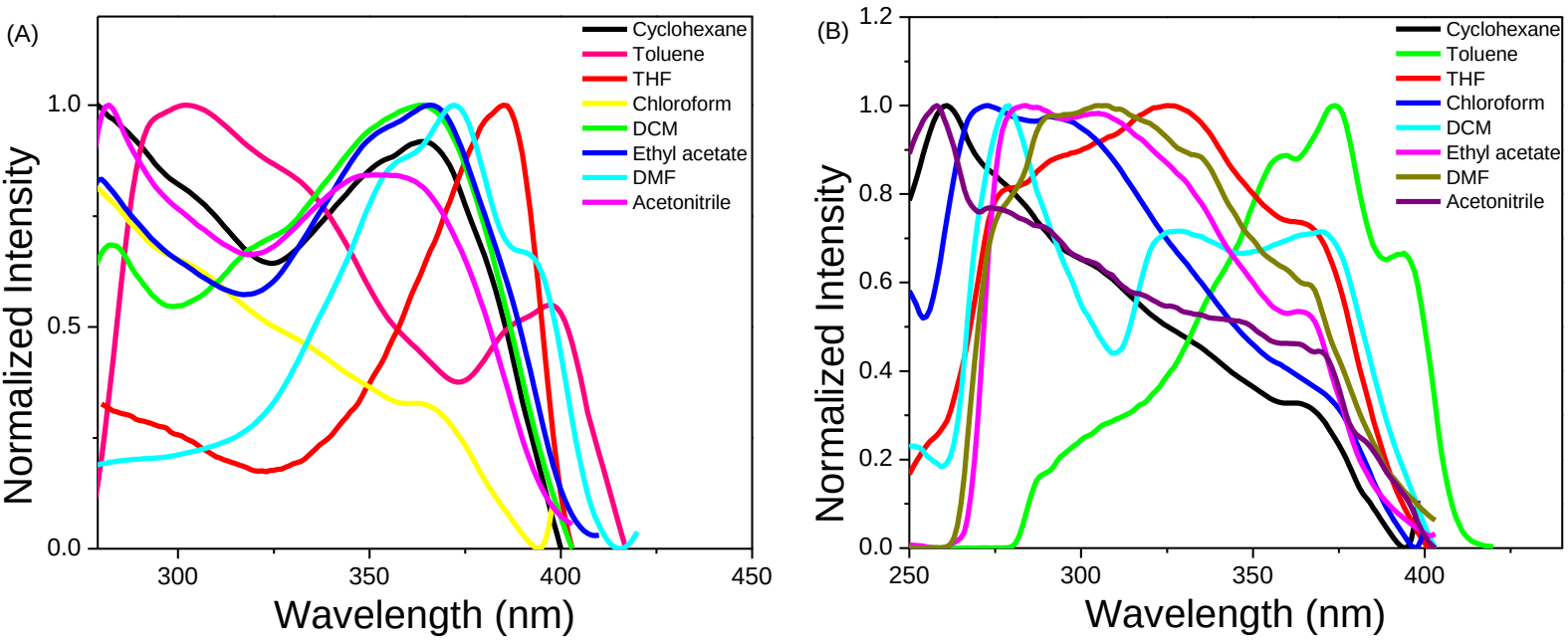


Fig. S6 Normalized excitation spectra of **1** (A) and **2** (B) in different solvents of varying polarities.

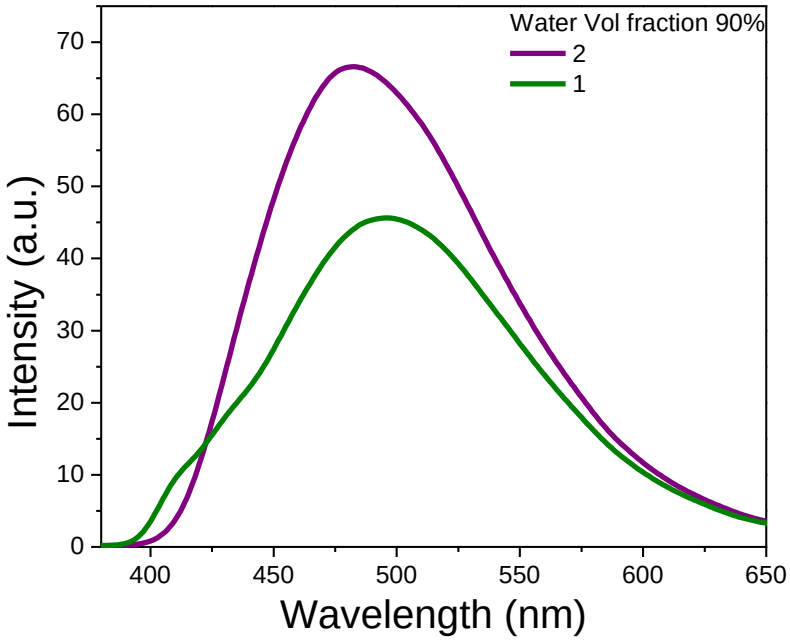


Fig. S7 Emission spectra of **1** and **2** at 90% THF-water mixture.

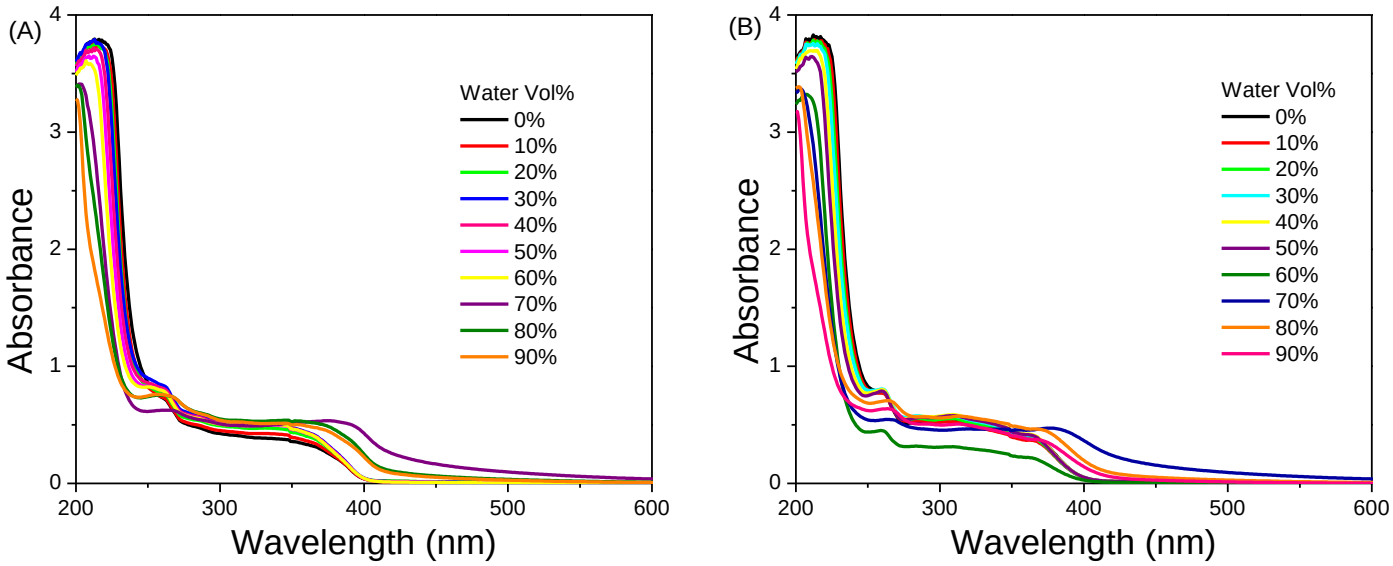


Fig. S8 UV-vis absorption spectra of **1** (A) and **2** (B) in THF–water mixtures with different water fractions.

Mechanochromism

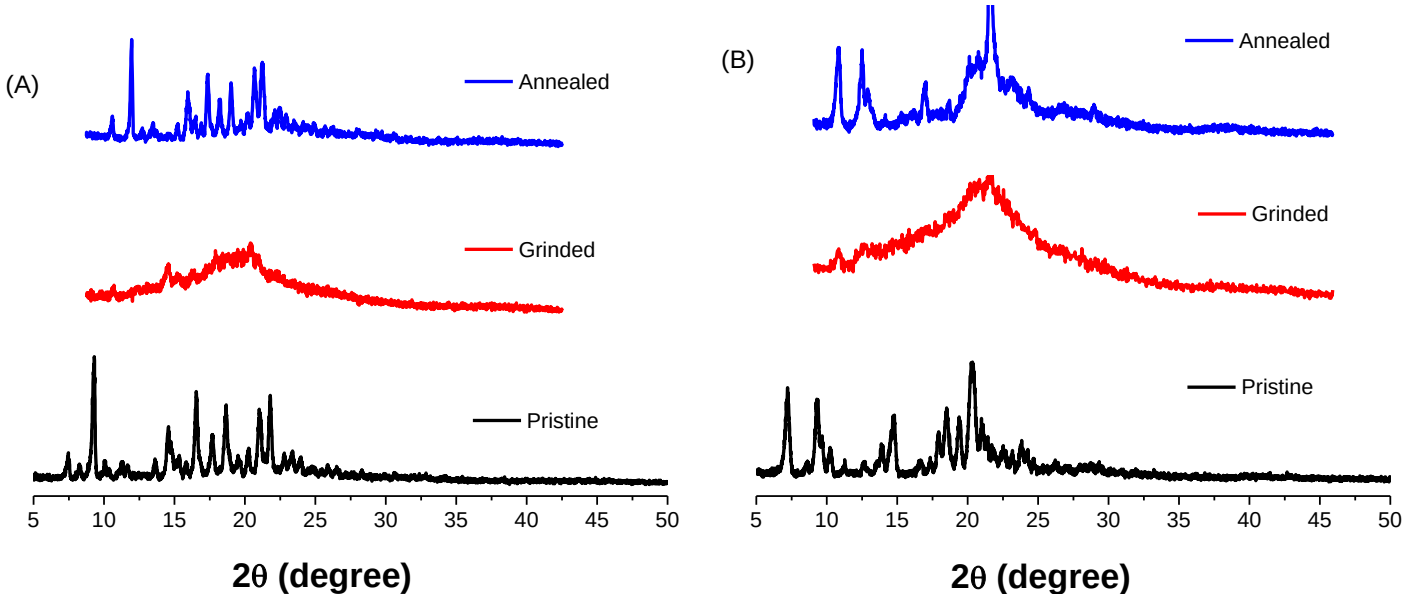


Fig. S9 Powder X-ray diffraction curves of **1** (A) and **2** (B) in pristine, ground and annealed form.

Crystallographic data

Single crystal X-ray structures of **1** and **4** were performed on a CCD Agilent Technologies (Oxford Diffraction) SUPER NOVA diffractometer. The strategy for the Data collection was evaluated by using the CrysAlisPro CCD software. The data were collected by the standard 'phi omega scan techniques, and were scaled and reduced using CrysAlisPro RED software. The structures were solved by direct methods using SHELXS-97, and refined by full matrix least-squares with SHELXL-97, refining on F2. The positions of all the atoms were obtained by direct methods. All non hydrogen atoms were refined anisotropically. The remaining hydrogen atoms were placed in geometrically constrained positions, and refined with isotropic temperature factors, generally 1.2Ueq of their parent atoms. SQUEEZE was applied to omit disordered DCM molecule. The crystal and refinement data are summarized in Table S1. The CCDC number 1571257, 1571869 and 1571258 contains the supplementary crystallographic data for **1**, **3** and **4**. These data can be obtained free of charge via www.ccdc.cam.ac.uk (or from the Cambridge Crystallographic Data Centre, 12 union Road, Cambridge CB21 EZ, UK; Fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

Table S2. Crystal data and structure refinement for 1, 3 and 4 .

Identification code	rm224	rm230	rm193
Empirical formula	$C_{65}H_{45}N_3$	$C_{39}H_{27}IN_3$	$C_{39}H_{26}BrN_3$
Formula weight	868.04	664.53	616.54
Temperature	293(2) K	293(2) K	293(2) K
Wavelength	1.54184 A	0.71073 A	0.71073 A
Crystal system, space group	Triclinic, P -1	Triclinic, P -1	Monoclinic, P 21/n
a/(Å)	11.0325(4)	8.1689(12) A	16.0838(5)
b/(Å)	13.8922(5)	11.2594(9) A	6.9446(2)
c/(Å)	17.6967(8)	17.439(2) A	26.8363(7)
Alpha/(º)	100.024(4)	96.245(9)	90

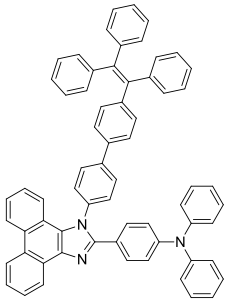
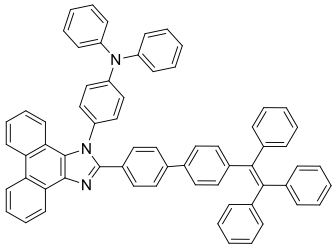
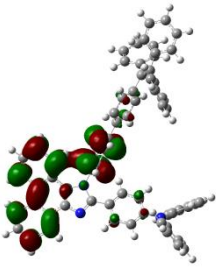
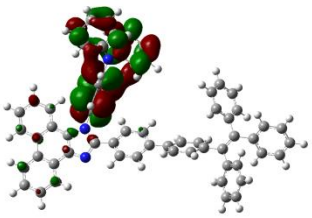
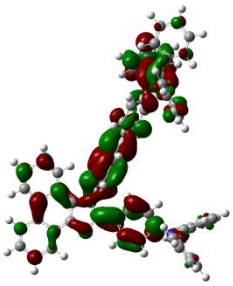
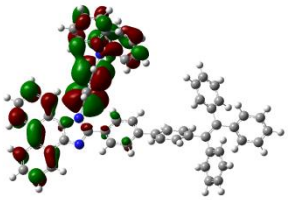
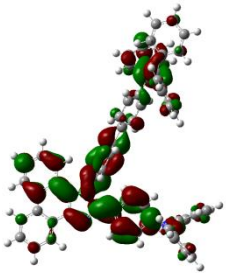
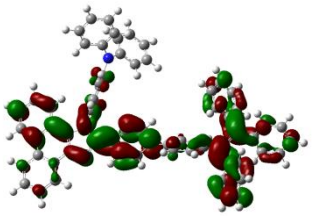
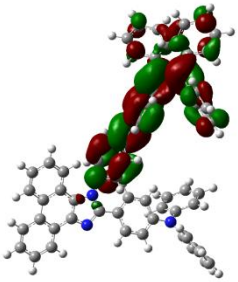
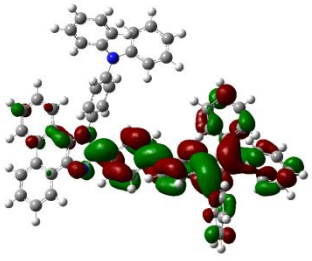
Beta/(º)	94.455(4)	91.993(13)	91.926(3)
Gamma/(º)	109.496(4)	101.903(10)	90
Volume	2490.68(18) $Å^3$	1557.4(3) Å3	2995.80(15) $Å^3$
Z, Calculated density	2, 1.157 mg/m^{-3}	2, 1.417 mg/m^{-3}	4, 1.367 Mg/m^{-3}
Absorption coefficient	0.513 mm^{-1}	1.060 mm^{-1}	1.405 mm^{-1}
F(000)	912	670	1264
Crystal size	0.230 x 0.180 x 0.120 mm	0.230 x 0.180 x 0.130 mm	0.260 x 0.210 x 0.180 mm
Θ range for data collection/(º)	3.454 to 71.472	3.032 to 28.846	2.911 to 32.252
Reflections collected / unique	16838 / 9463 [R(int) = 0.0683]	13182 / 7155 [R(int) = 0.0921]	37707 / 9935 [R(int) = 0.0923]
Completeness to theta	Θ = 67.684 99.8 %	Θ = 25.242 99.8 %	Θ = 25.242 99.9 %
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.70721	1.00000 and 0.48753	1.00000 and 0.78417
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F2	Full-matrix least-squares on F^2
Data / restraints / parameters	9463 / 630 / 669	7155 / 0 / 389	9935 / 0 / 389
Goodness-of-fit on F^2	1.072	1.316	1.005
Final R indices [I>2sigma(I)]	R1 = 0.0780, wR2 = 0.2241	R1 = 0.1382, wR2 = 0.3736	R1 = 0.0630, wR2 = 0.1208
R indices (all data)	R1 = 0.0889, wR2 = 0.2348	R1 = 0.1809, wR2 = 0.4207	R1 = 0.1815, wR2 = 0.1661
Extinction coefficient	n/a	0.072(10)	0.0014(3)
Largest diff. peak and hole ($e.Å^{-3}$)	0.348 and -0.387	1.848 and -1.929	0.351 and -0.531

DFT calculations

TDDFT data

Table S3. Computed vertical transitions, their oscillator strengths and configurations of phenanthroimidazoles 1 and 2.

Compound	λ_{max} (nm)	f^a	Configuration
1	369.88nm	0.7027	HOMO-2-LUMO(0.22724) HOMO-LUMO+1(0.60262) HOMO-LUMO+2(0.24335)
	360.33	0.8086	HOMO-2-LUMO(0.63456) HOMO-1-LUMO(0.17404) HOMO-LUMO+1(-0.21985)
2	387.10	1.0332	HOMO-2-LUMO(0.13012) HOMO-LUMO (0.68217)
	337.70	0.1898	HOMO-LUMO+1(0.62732) HOMO-LUMO+2(0.22827) HOMO-LUMO+3(0.12819)
	314.88	0.3462	HOMO-3-LUMO (-0.16557) HOMO-2-LUMO+3(-0.10500) HOMO-1-LUMO (0.39406) HOMO-1-LUMO+2 (0.25834) HOMO-1-LUMO+3(0.45047)

Phenanthroimidazole		
	1	**2**
LUMO+3		
LUMO+2		
LUMO+1		
LUMO		

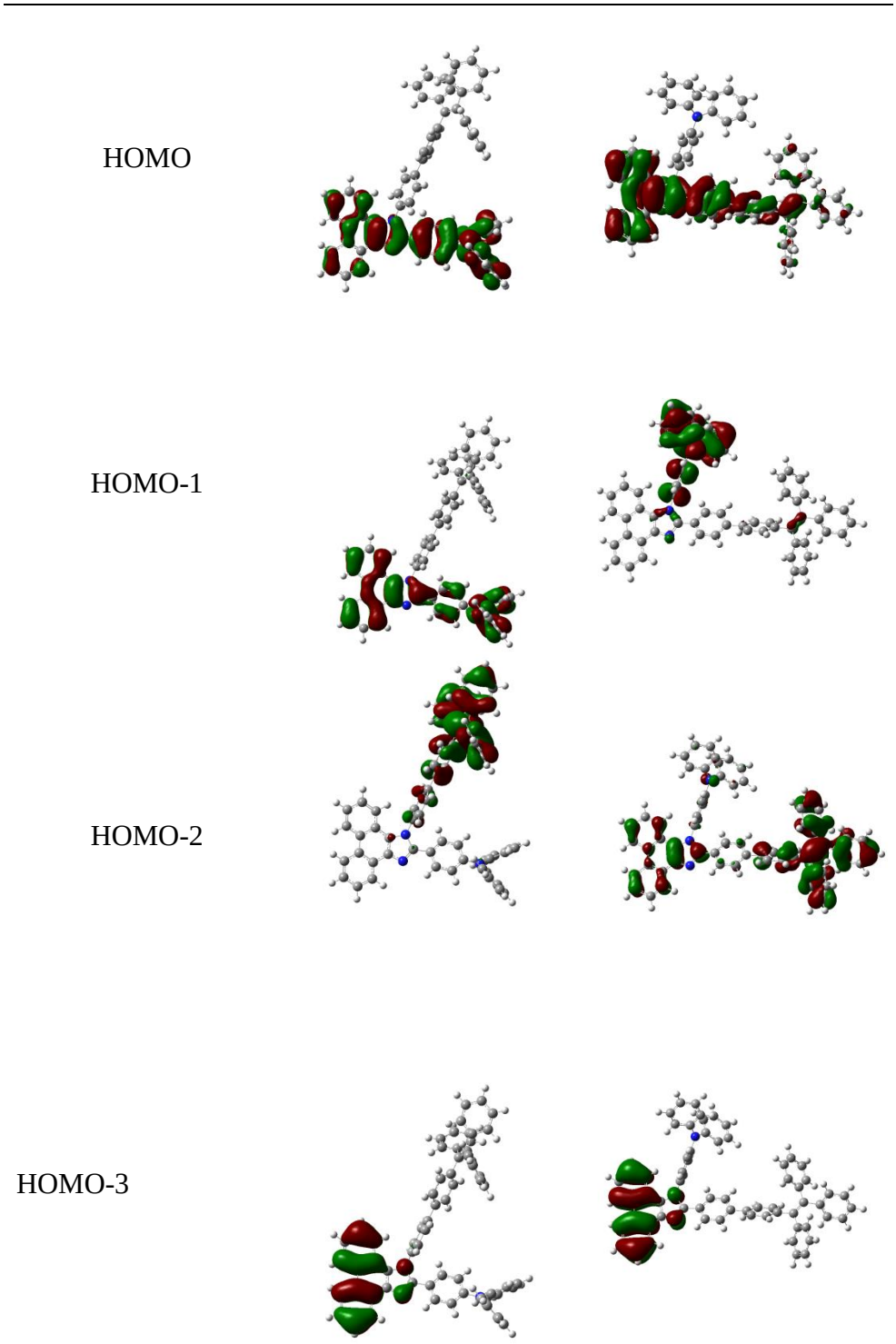


Fig. S10 Frontier molecular orbitals of phenanthroimidazoles **1** and **2** at the B3LYP/ 6-31G(d,p) level.

DFT calculation data of 1 and 2

Calculation method: B3LYP/6-31G(d,p) with Gaussian 09

1:

Standard orientation:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	6	0	-4.363180	-3.213541	-0.092273
2	6	0	-3.914290	-4.576649	-0.197945
3	6	0	-2.557099	-4.975748	-0.211334
4	1	0	-1.779294	-4.227848	-0.147936
5	6	0	-2.199957	-6.307712	-0.312403
6	1	0	-1.149911	-6.584926	-0.320535
7	6	0	-3.193019	-7.292639	-0.407200
8	1	0	-2.920409	-8.340877	-0.485042
9	6	0	-4.526965	-6.925377	-0.409871
10	1	0	-5.273306	-7.706018	-0.495247
11	6	0	-4.938653	-5.577930	-0.312284
12	6	0	-6.360697	-5.212315	-0.339417
13	6	0	-7.396792	-6.169828	-0.441759
14	1	0	-7.159451	-7.226055	-0.492949
15	6	0	-8.729083	-5.795533	-0.478268
16	1	0	-9.498126	-6.558466	-0.556978
17	6	0	-9.089424	-4.438364	-0.413718
18	1	0	-10.136210	-4.150171	-0.442862
19	6	0	-8.104633	-3.474454	-0.310023
20	1	0	-8.349314	-2.419122	-0.253187
21	6	0	-6.745195	-3.843999	-0.270225
22	6	0	-5.706502	-2.861527	-0.151344
23	6	0	-4.657181	-0.997432	0.051021
24	7	0	-3.682900	-1.998811	0.047783
25	7	0	-5.870963	-1.505059	-0.067900
26	6	0	-2.264449	-1.803765	0.087347
27	6	0	-1.587323	-1.365042	-1.052138
28	6	0	-1.554676	-2.042829	1.268391
29	1	0	-2.145111	-1.172448	-1.962596
30	6	0	-0.210095	-1.164960	-1.007844
31	6	0	-0.176690	-1.849076	1.302303
32	1	0	-2.086029	-2.393653	2.147335
33	1	0	0.300987	-0.795947	-1.891213
34	6	0	0.524374	-1.404436	0.166447
35	1	0	0.367583	-2.067268	2.215538
36	6	0	1.991991	-1.193983	0.204334
37	6	0	2.796771	-1.495183	-0.907444
38	6	0	2.626711	-0.691567	1.352827
39	1	0	2.344318	-1.925291	-1.795966
40	6	0	4.171508	-1.289111	-0.877945
41	6	0	4.003465	-0.497123	1.385909
42	1	0	2.032228	-0.426169	2.222006
43	1	0	4.768317	-1.539351	-1.748434
44	6	0	4.805788	-0.773478	0.265031

45	1	0	4.466428	-0.109421	2.288095
46	6	0	6.285653	-0.580218	0.317733
47	6	0	6.980924	0.050893	-0.675467
48	6	0	6.943902	-1.126815	1.543894
49	6	0	6.308224	0.832487	-1.757904
50	6	0	8.472442	0.017166	-0.764654
51	6	0	7.828072	-0.344528	2.305715
52	6	0	6.645395	-2.423607	1.997662
53	6	0	5.345339	1.811069	-1.458755
54	6	0	6.668541	0.645025	-3.103999
55	6	0	9.200102	1.197122	-0.999736
56	6	0	9.181156	-1.193129	-0.677473
57	1	0	8.056707	0.664835	1.980149
58	6	0	8.408581	-0.847911	3.468511
59	6	0	7.234492	-2.931378	3.154268
60	1	0	5.949509	-3.036597	1.432685
61	1	0	5.070122	1.983950	-0.423538
62	6	0	4.747241	2.559847	-2.470864
63	6	0	6.061242	1.384436	-4.117306
64	1	0	7.427323	-0.090881	-3.352545
65	1	0	8.668215	2.139399	-1.090670
66	6	0	10.589043	1.172063	-1.111250
67	6	0	10.569022	-1.219975	-0.801628
68	1	0	8.635292	-2.116457	-0.514399
69	1	0	9.085555	-0.223366	4.044353
70	6	0	8.118636	-2.145029	3.895365
71	1	0	6.999346	-3.940920	3.479259
72	1	0	4.009629	3.315371	-2.215702
73	6	0	5.097598	2.345457	-3.804995
74	1	0	6.345097	1.213701	-5.151878
75	1	0	11.131907	2.097746	-1.279913
76	6	0	11.279805	-0.037332	-1.013515
77	1	0	11.095576	-2.167874	-0.736843
78	1	0	8.572428	-2.537258	4.800789
79	1	0	4.630298	2.927809	-4.593724
80	1	0	12.361523	-0.058768	-1.108472
81	6	0	-4.420578	0.450712	0.155086
82	6	0	-3.330874	1.044899	0.814073
83	6	0	-5.392881	1.305722	-0.399166
84	6	0	-3.206267	2.427747	0.894519
85	1	0	-2.567757	0.433404	1.277889
86	6	0	-5.280685	2.684996	-0.308729
87	1	0	-6.253779	0.860982	-0.885125
88	6	0	-4.177756	3.273544	0.335922
89	1	0	-2.350725	2.858725	1.402665
90	1	0	-6.050168	3.317603	-0.737561
91	7	0	-4.054268	4.681642	0.425971
92	6	0	-4.453677	5.508541	-0.661029
93	6	0	-5.178426	6.687864	-0.427268
94	6	0	-4.124951	5.160905	-1.980925
95	6	0	-5.557098	7.502080	-1.492587
96	1	0	-5.440356	6.958912	0.589929
97	6	0	-4.524250	5.972218	-3.040993
98	1	0	-3.558476	4.254713	-2.167299
99	6	0	-5.237938	7.148844	-2.805164
100	1	0	-6.118027	8.410908	-1.294226
101	1	0	-4.262393	5.688134	-4.056232
102	1	0	-5.541545	7.781891	-3.633152

103	6	0	-3.530972	5.280931	1.606226
104	6	0	-3.940480	4.837262	2.873725
105	6	0	-2.601714	6.329463	1.518453
106	6	0	-3.420766	5.425222	4.025025
107	1	0	-4.664708	4.033015	2.948080
108	6	0	-2.101564	6.923236	2.675531
109	1	0	-2.278290	6.673749	0.541836
110	6	0	-2.502574	6.473368	3.934958
111	1	0	-3.748780	5.070343	4.997871
112	1	0	-1.383590	7.733915	2.589883
113	1	0	-2.105686	6.934050	4.834288

Total Energy (HF) = -2668.4004814Hartree

2:

Standard orientation:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	6	0	-5.484541	-2.258965	-0.205866
2	6	0	-6.868273	-1.863387	-0.186193
3	6	0	-7.318668	-0.523349	-0.133722
4	1	0	-6.597646	0.281310	-0.100975
5	6	0	-8.667241	-0.218070	-0.118555
6	1	0	-8.984772	0.819825	-0.078926
7	6	0	-9.617828	-1.247735	-0.152514
8	1	0	-10.678622	-1.015801	-0.141968
9	6	0	-9.201162	-2.566422	-0.193942
10	1	0	-9.957021	-3.342342	-0.211169
11	6	0	-7.835398	-2.925816	-0.208967
12	6	0	-7.419016	-4.334052	-0.237397
13	6	0	-8.341790	-5.405526	-0.268309
14	1	0	-9.407090	-5.206947	-0.280678
15	6	0	-7.921225	-6.724370	-0.284530
16	1	0	-8.658383	-7.521675	-0.308449
17	6	0	-6.550403	-7.034856	-0.270689
18	1	0	-6.225951	-8.071289	-0.283818
19	6	0	-5.619159	-6.014114	-0.242671
20	1	0	-4.554033	-6.219672	-0.235133
21	6	0	-6.035837	-4.668001	-0.227036
22	6	0	-5.087541	-3.591416	-0.204440
23	6	0	-4.152124	-0.106254	-0.128911
24	6	0	-3.864680	0.509689	1.091223
25	1	0	-3.745219	-0.099697	1.980989
26	6	0	-3.709847	1.890279	1.163493
27	1	0	-3.469559	2.355744	2.112464
28	6	0	-3.859572	2.690003	0.015723
29	6	0	-4.162189	2.060195	-1.207066
30	1	0	-4.289929	2.660593	-2.100537
31	6	0	-4.295251	0.677979	-1.278274
32	1	0	-4.529829	0.200237	-2.224252
33	6	0	-3.256783	-2.468197	-0.185820
34	6	0	-1.813764	-2.178570	-0.148791
35	6	0	-1.213590	-1.015477	-0.659616
36	1	0	-1.816517	-0.238847	-1.111446
37	6	0	0.166268	-0.843759	-0.604303

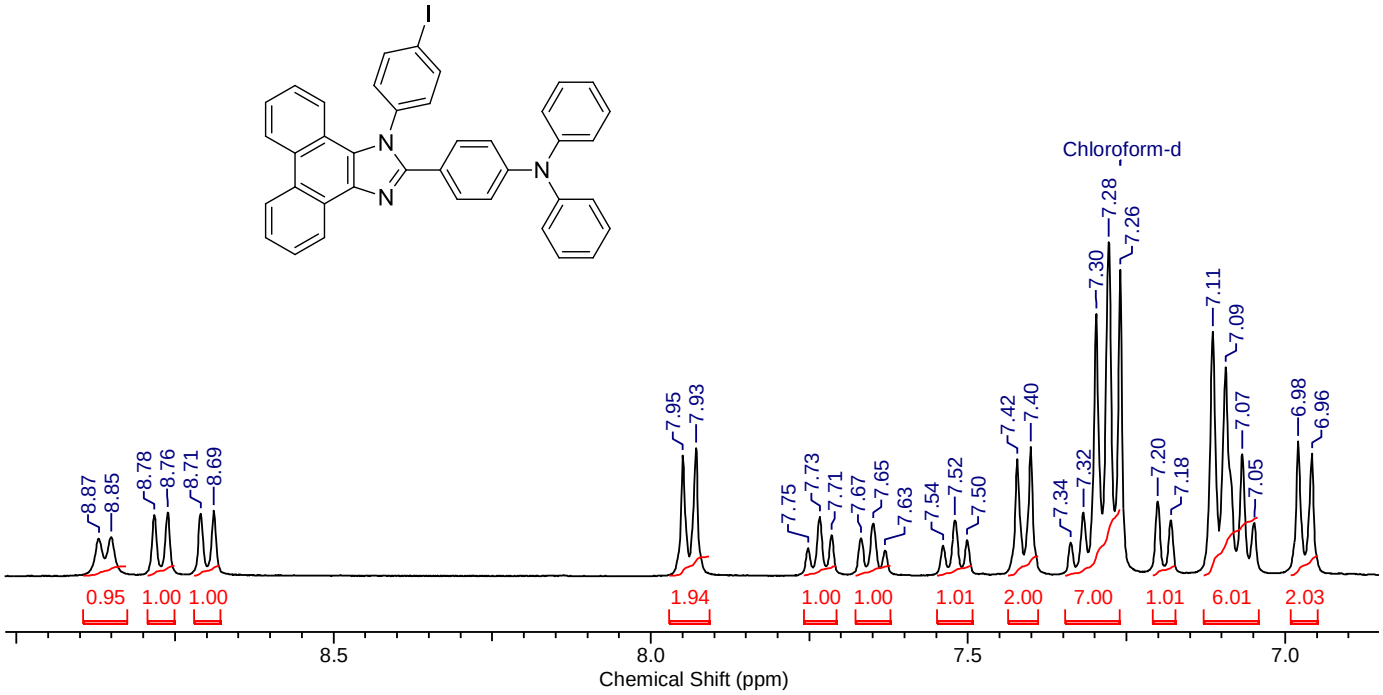
38	1	0	0.596308	0.077324	-0.985764
39	6	0	1.007926	-1.819466	-0.046495
40	6	0	0.402865	-2.988729	0.449379
41	1	0	1.023432	-3.780009	0.858652
42	6	0	-0.972686	-3.165656	0.399591
43	1	0	-1.423999	-4.078315	0.771543
44	6	0	2.475535	-1.623783	0.014189
45	6	0	3.162901	-0.942716	-1.005568
46	1	0	2.616487	-0.589013	-1.874567
47	6	0	4.538113	-0.747497	-0.943723
48	1	0	5.041528	-0.225614	-1.750688
49	6	0	5.293317	-1.227758	0.139353
50	6	0	4.610980	-1.933592	1.145746
51	1	0	5.169133	-2.328939	1.989058
52	6	0	3.233350	-2.115996	1.091228
53	1	0	2.733057	-2.628194	1.907571
54	6	0	6.775056	-1.053467	0.197630
55	6	0	7.384562	0.149457	-0.025317
56	6	0	7.538652	-2.297603	0.520962
57	6	0	8.519514	-2.311024	1.526944
58	1	0	8.743432	-1.393175	2.060345
59	6	0	9.201525	-3.483869	1.845961
60	1	0	9.952326	-3.471798	2.630934
61	6	0	8.918910	-4.669611	1.165598
62	1	0	9.451683	-5.583028	1.413532
63	6	0	7.939404	-4.674810	0.170656
64	1	0	7.708212	-5.593047	-0.361782
65	6	0	7.249321	-3.504452	-0.139914
66	1	0	6.479699	-3.518669	-0.905808
67	6	0	6.629205	1.438644	-0.079320
68	6	0	5.719723	1.793164	0.931539
69	1	0	5.544074	1.104115	1.750995
70	6	0	5.048327	3.013973	0.894526
71	1	0	4.354201	3.269226	1.690229
72	6	0	5.269859	3.907450	-0.154942
73	1	0	4.745808	4.858398	-0.183887
74	6	0	6.178053	3.573898	-1.161722
75	1	0	6.361849	4.263899	-1.980462
76	6	0	6.858364	2.358303	-1.117870
77	1	0	7.573651	2.110846	-1.896407
78	6	0	8.859204	0.282275	-0.231386
79	6	0	9.590518	1.264347	0.459793
80	1	0	9.076169	1.911076	1.164288
81	6	0	10.961439	1.411969	0.257348
82	1	0	11.508735	2.168724	0.812299
83	6	0	11.628128	0.596158	-0.658803
84	1	0	12.695060	0.715967	-0.822981
85	6	0	10.911247	-0.368580	-1.368820
86	1	0	11.417592	-1.000187	-2.093195
87	6	0	9.542496	-0.524450	-1.156923
88	1	0	8.991487	-1.276363	-1.712093
89	7	0	-4.291354	-1.530405	-0.202646
90	7	0	-3.723918	-3.704165	-0.186563
91	7	0	-3.708563	4.092161	0.084349
92	6	0	-3.120505	4.805854	-1.001152
93	6	0	-3.704056	5.994464	-1.464274
94	6	0	-1.946888	4.336455	-1.610401
95	6	0	-3.118663	6.699639	-2.514119

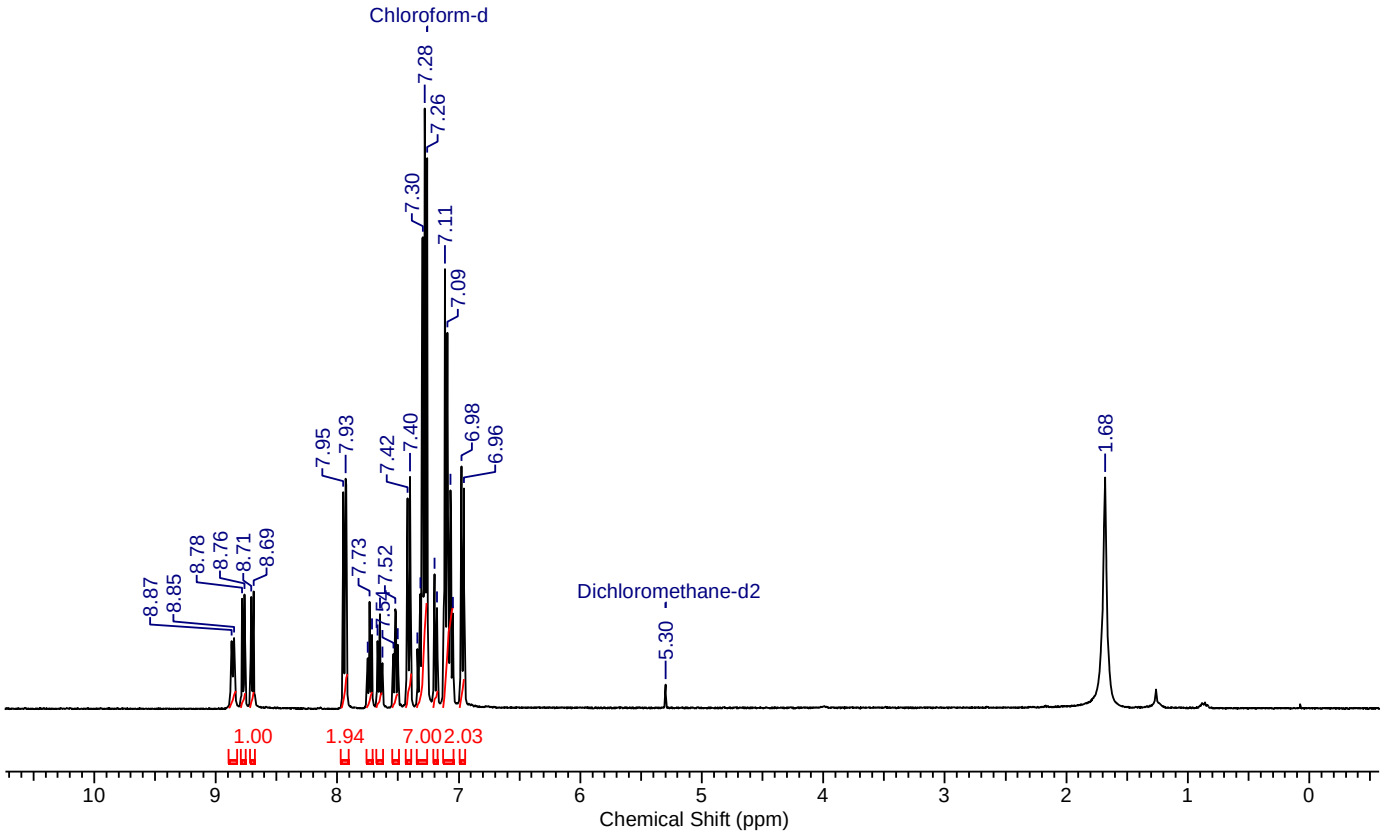
96	1	0	-4.612069	6.358965	-0.995613
97	6	0	-1.378964	5.038394	-2.671860
98	1	0	-1.485307	3.424208	-1.247053
99	6	0	-1.957646	6.224627	-3.127255
100	1	0	-3.581606	7.618486	-2.862028
101	1	0	-0.470681	4.662656	-3.133940
102	1	0	-1.508609	6.772884	-3.949538
103	6	0	-4.121496	4.810158	1.244404
104	6	0	-5.359214	4.540377	1.848300
105	6	0	-3.297485	5.806352	1.789381
106	6	0	-5.754236	5.245978	2.983140
107	1	0	-6.005231	3.778424	1.424963
108	6	0	-3.709630	6.519942	2.913117
109	1	0	-2.338976	6.016623	1.326905
110	6	0	-4.935800	6.241744	3.519870
111	1	0	-6.714537	5.024901	3.439874
112	1	0	-3.060741	7.288306	3.323279
113	1	0	-5.250390	6.794509	4.399535

Total Energy (HF) = -2668.4004181Hartree

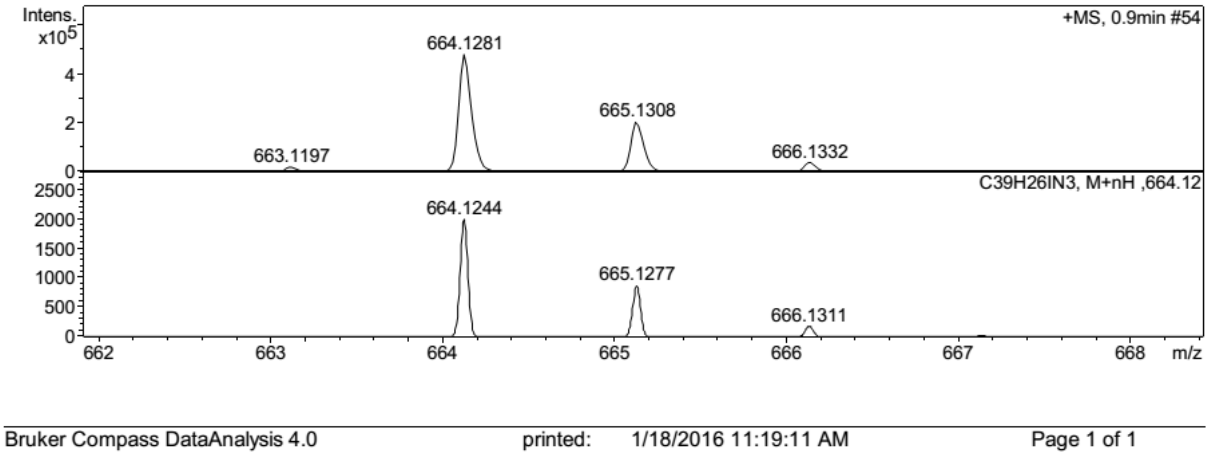
Copies of NMR and HRMS spectra of the new compounds

^{1}H NMR of 3:

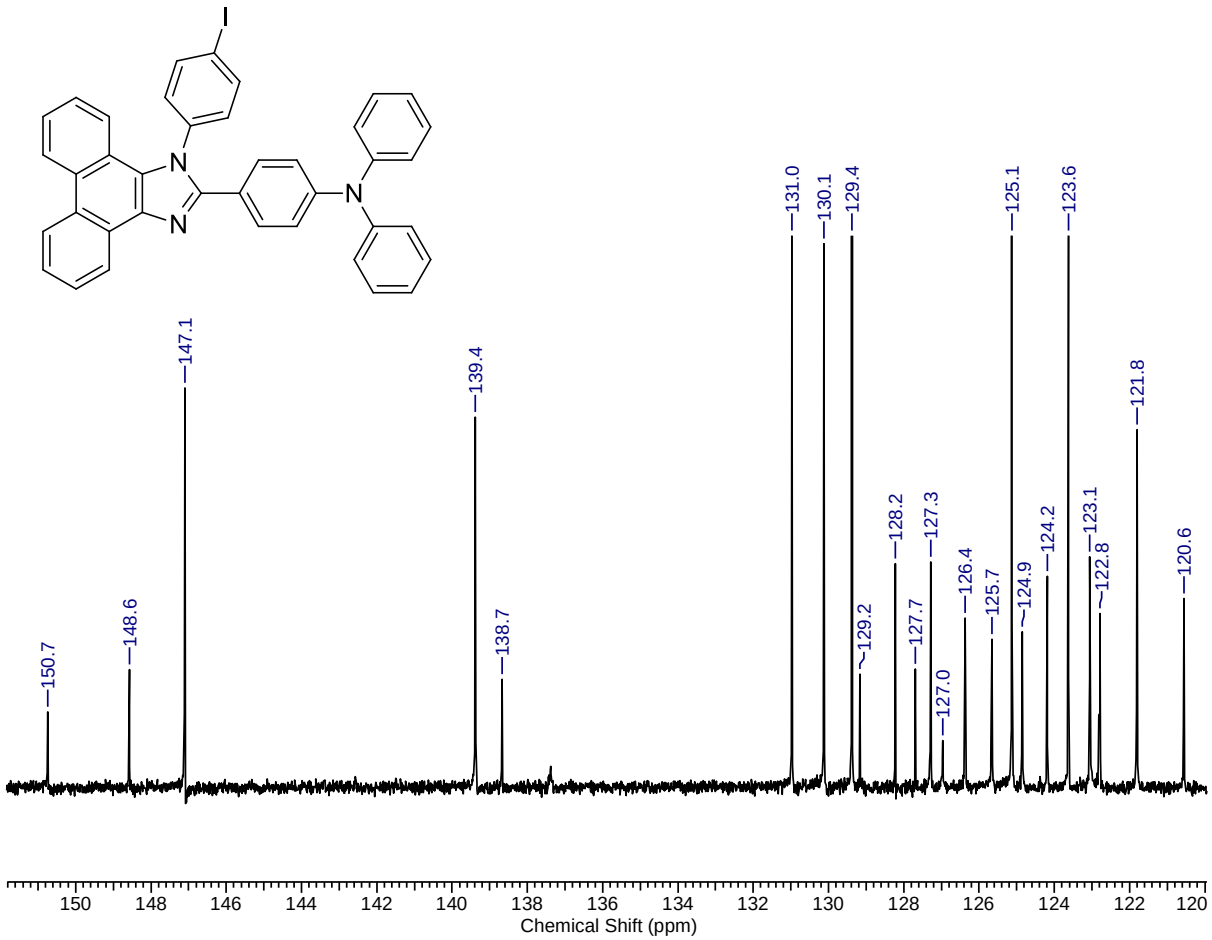


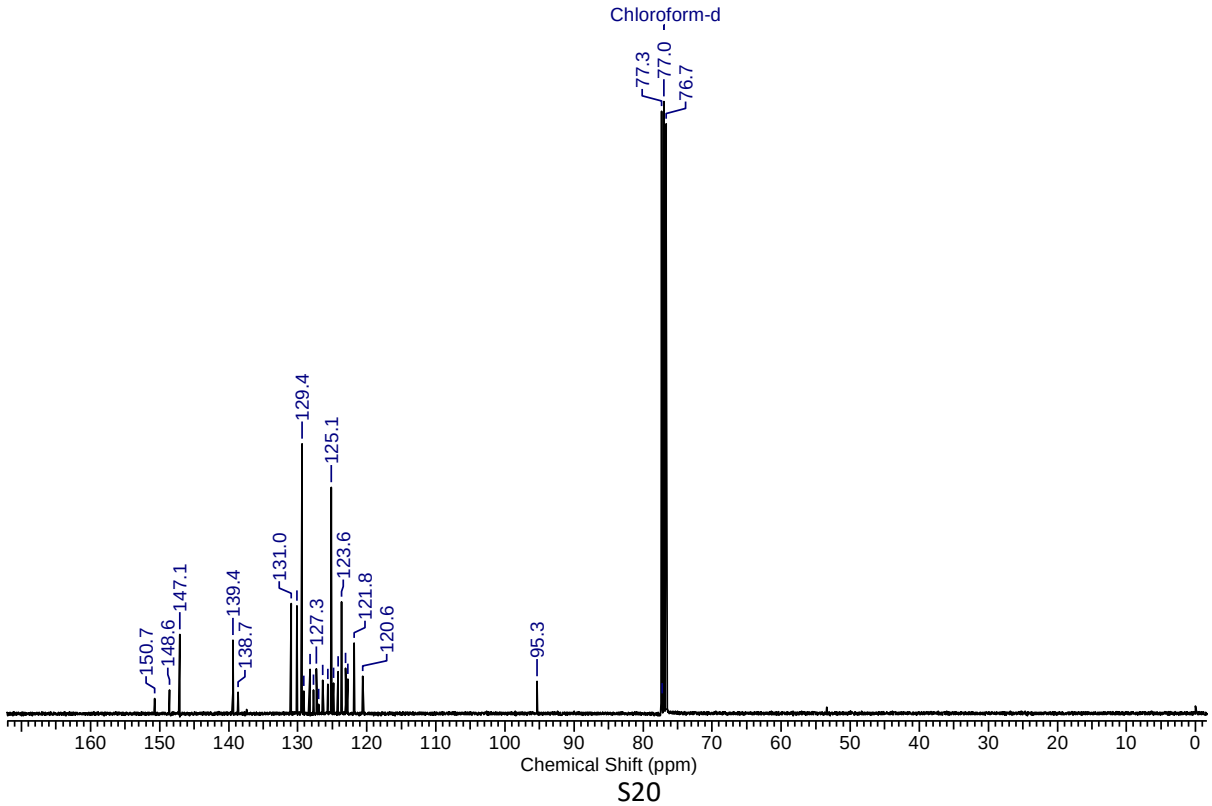


HRMS of 3:

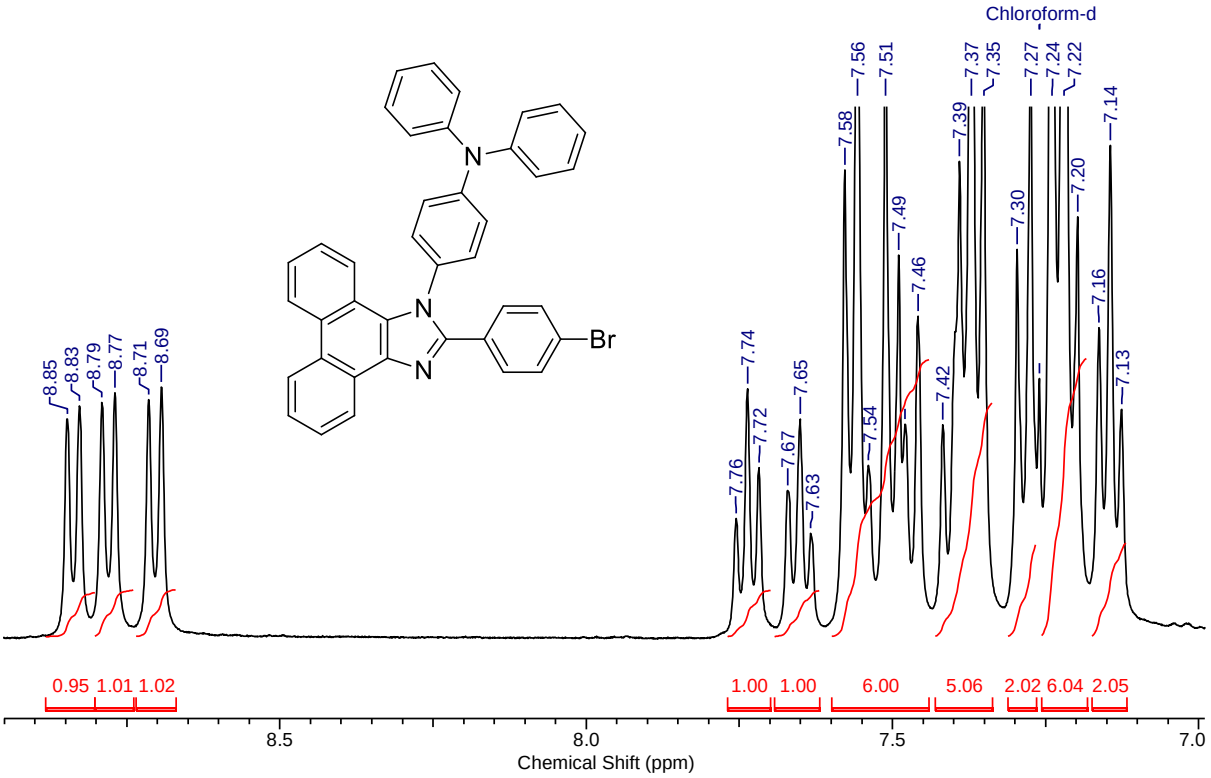

Intens.
x10^5
+MS, 0.9min #54
664.1281
665.1308
663.1197
666.1332
C39H26IN3, M+nH ,664.12
664.1244
665.1277
666.1311
662
663
664
665
666
667
668
m/z
Bruker Compass DataAnalysis 4.0
printed: 1/18/2016 11:19:11 AM
Page 1 of 1

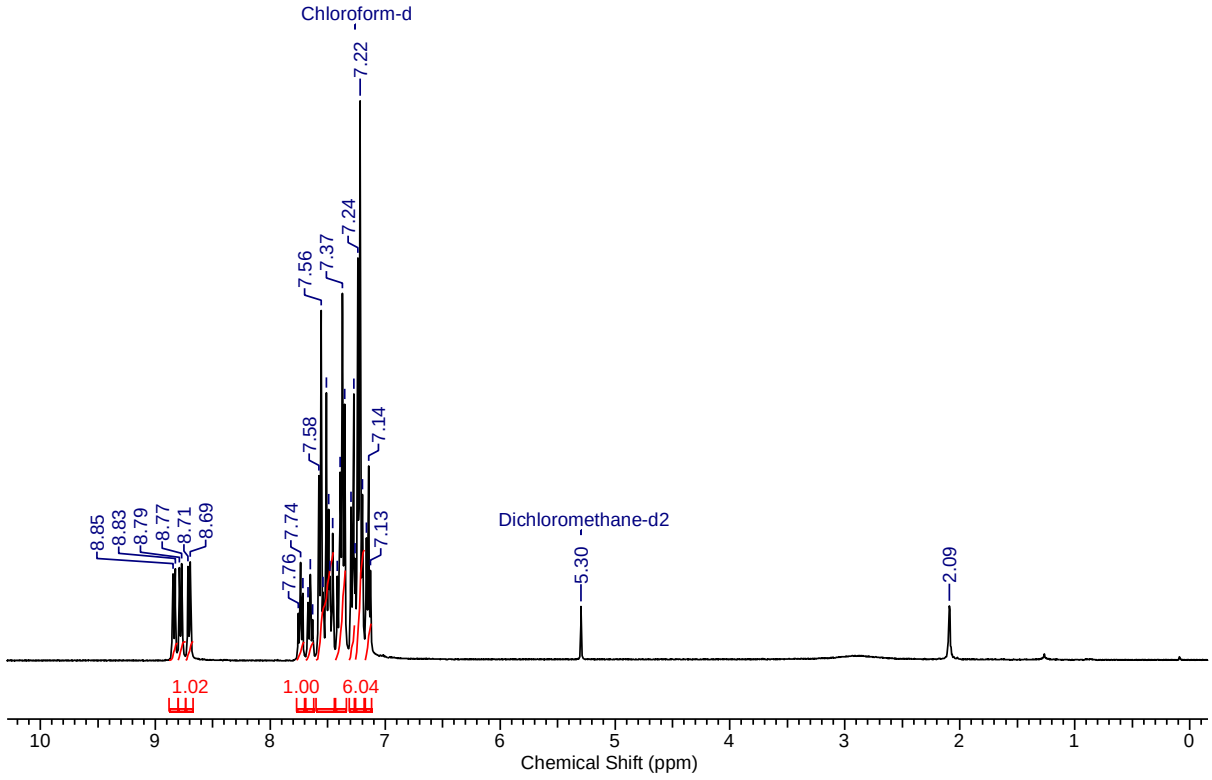
^{13}C NMR of 3:


I
N
N
N
150.7
148.6
147.1
139.4
138.7
131.0
130.1
129.4
129.2
128.2
127.7
127.3
127.0
126.4
125.7
125.1
124.9
124.2
123.6
123.1
122.8
121.8
120.6
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
120
Chemical Shift (ppm)

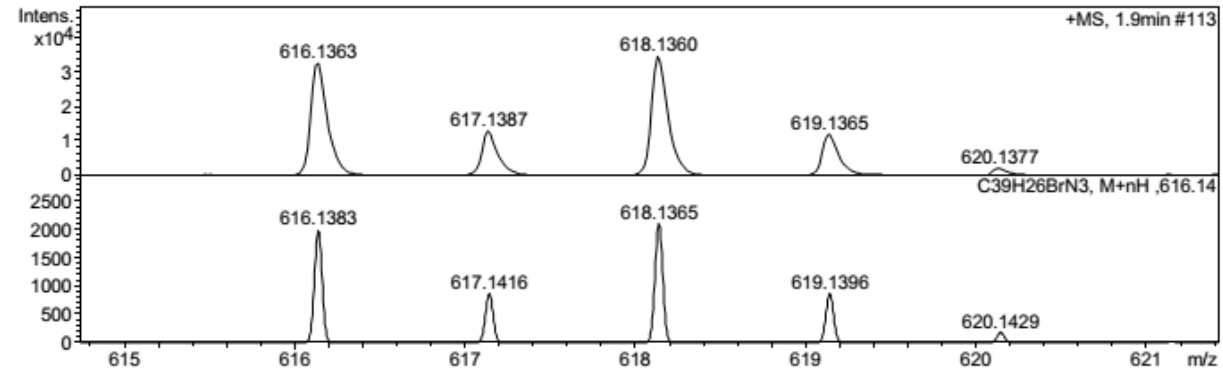

Chloroform-d
77.3
77.0
76.7
150.7
148.6
147.1
139.4
138.7
131.0
129.4
127.3
125.1
123.6
121.8
120.6
95.3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
Chemical Shift (ppm)

^{1}H NMR of 4:


Chloroform-d
8.85
8.83
8.79
8.77
8.71
8.69
7.76
7.74
7.72
7.67
7.65
7.63
7.58
7.56
7.54
7.51
7.49
7.46
7.42
7.39
7.37
7.35
7.30
7.27
7.24
7.22
7.20
7.16
7.14
7.13
Br
N
N
0.95 1.01 1.02
1.00 1.00 6.00 5.06 2.02 6.04 2.05
8.5
8.0
7.5
7.0
Chemical Shift (ppm)

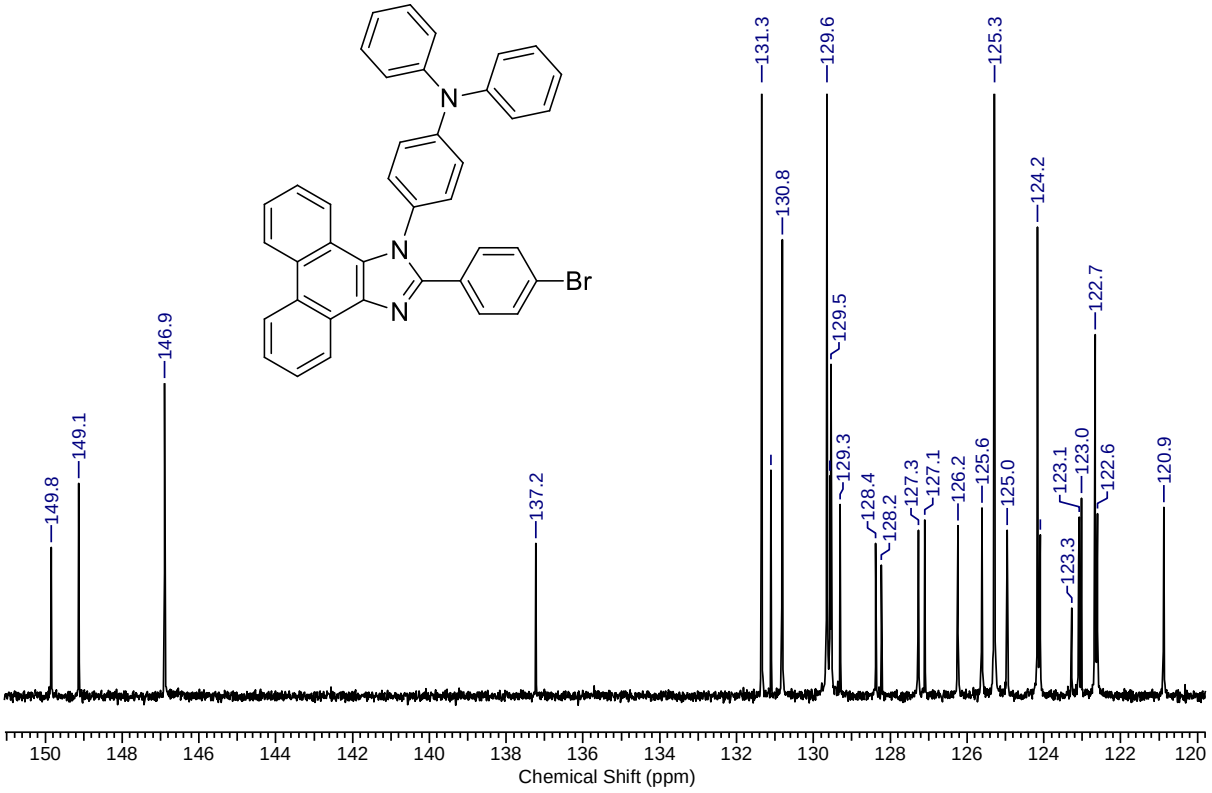

Chloroform-d
7.22
7.56
7.37
7.24
7.58
7.14
8.85
8.83
8.79
8.77
8.71
8.69
7.76
7.74
7.13
Dichloromethane-d2
5.30
2.09
1.02
1.00
6.04
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

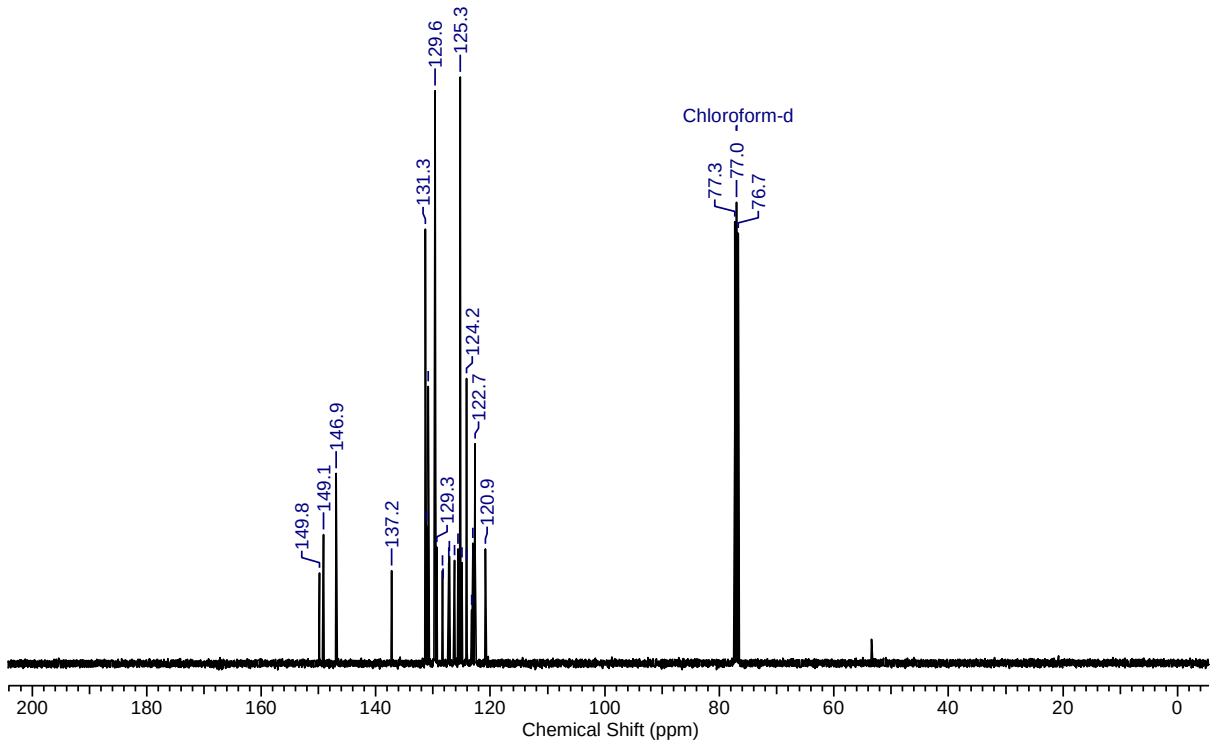
HRMS of 4:



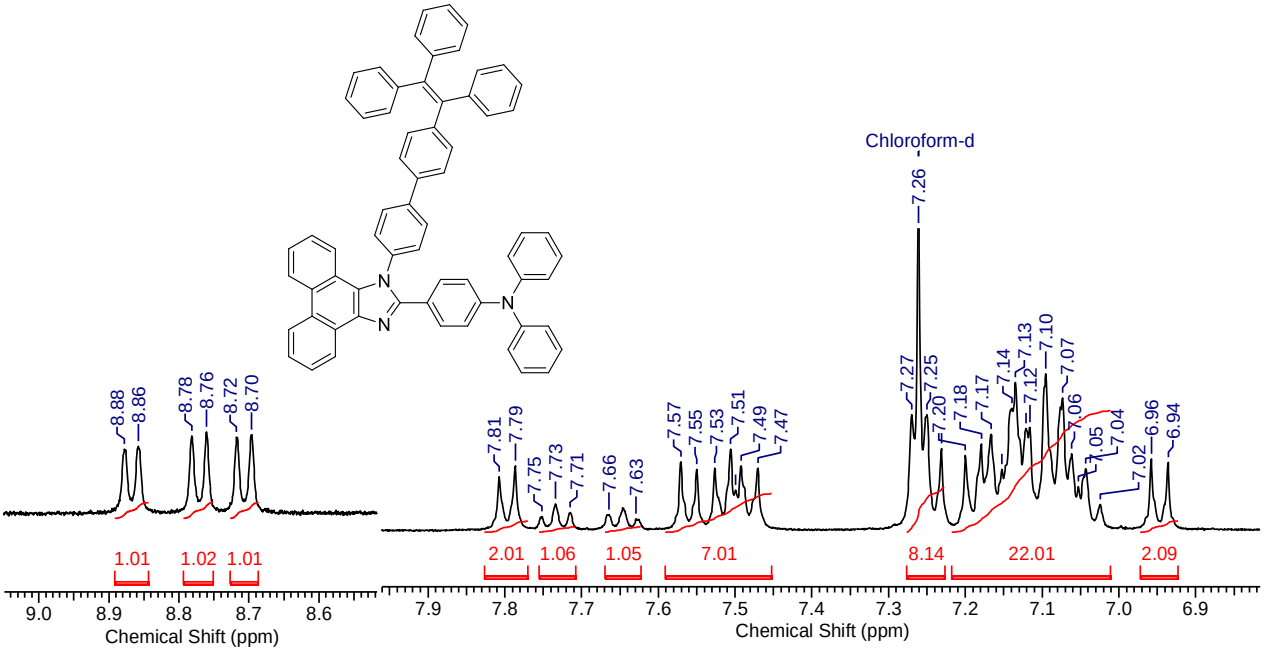
Bruker Compass DataAnalysis 4.0 printed: 4/21/2017 9:54:14 AM Page 1 of 1

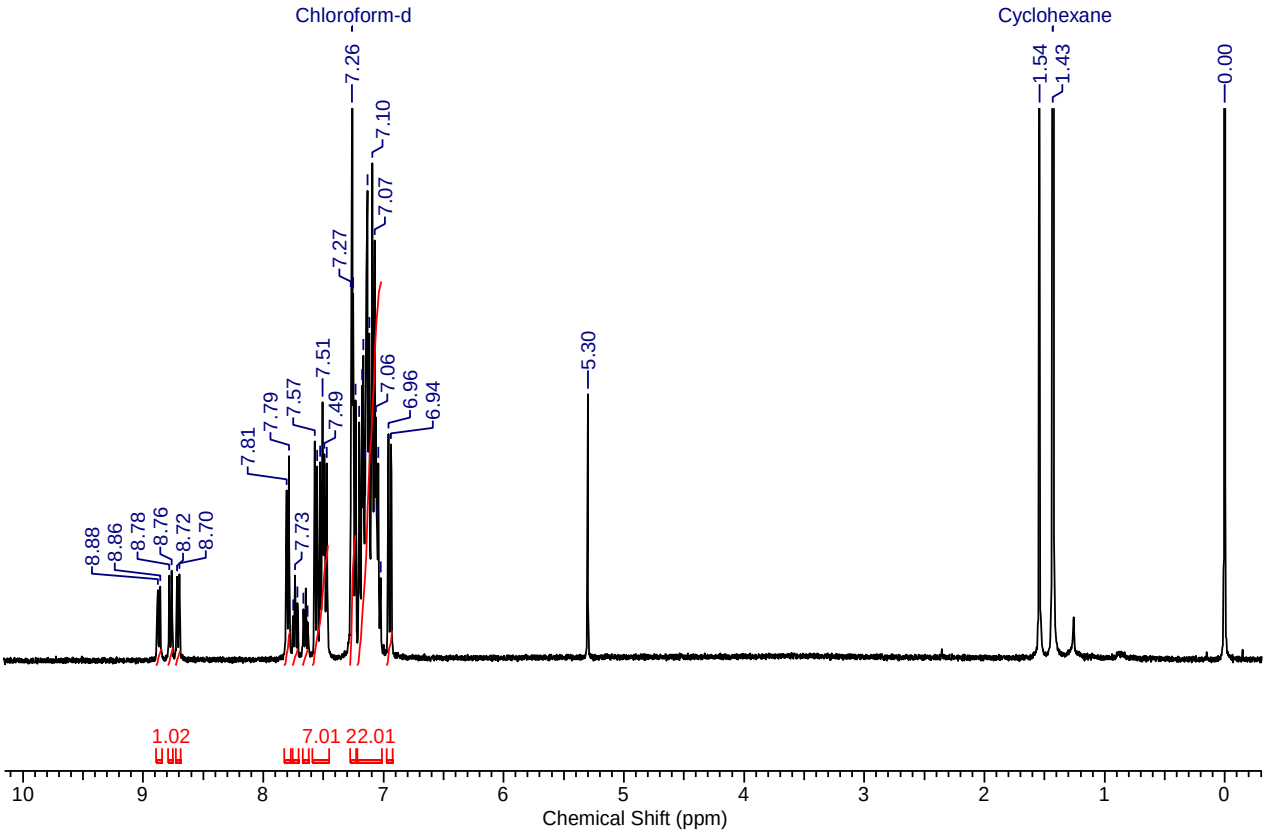
^{13}C NMR of 4:


N
N
N
Br
149.8
149.1
146.9
137.2
131.3
130.8
129.6
129.5
129.3
128.4
128.2
127.3
127.1
126.2
125.6
125.3
125.0
124.2
123.3
123.1
123.0
122.7
122.6
120.9
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
120
Chemical Shift (ppm)

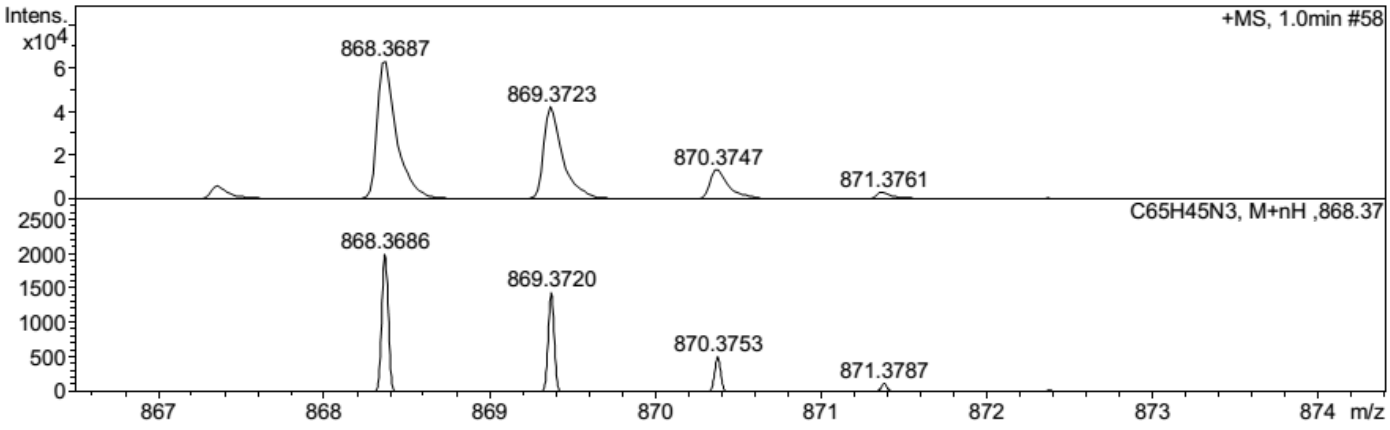

Chloroform-d
77.3
77.0
76.7
149.8
149.1
146.9
137.2
131.3
129.6
129.3
125.3
124.2
122.7
120.9
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

^{1}H NMR of 1:


Chloroform-d
7.26
8.88
8.86
8.78
8.76
8.72
8.70
7.81
7.79
7.75
7.73
7.71
7.66
7.63
7.57
7.55
7.53
7.51
7.49
7.47
7.27
7.25
7.20
7.18
7.17
7.14
7.13
7.12
7.10
7.07
7.06
7.05
7.04
7.02
6.96
6.94
1.01
1.02
1.01
2.01
1.06
1.05
7.01
8.14
22.01
2.09
Chemical Shift (ppm)
Chemical Shift (ppm)

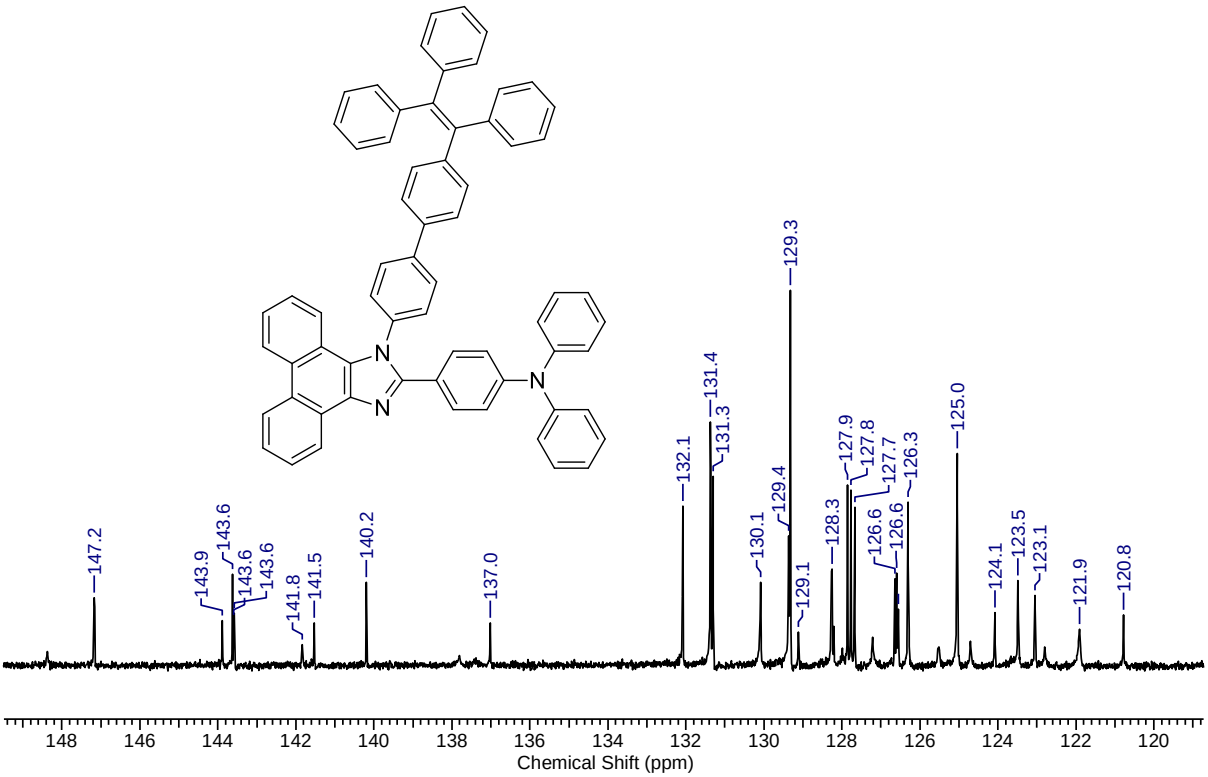

Chloroform-d
Cyclohexane
7.26
1.54
1.43
0.00
7.10
7.07
7.27
5.30
7.51
7.06
7.79
7.57
7.49
6.96
6.94
7.81
7.73
8.88
8.86
8.78
8.76
8.72
8.70
1.02
7.01
22.01
Chemical Shift (ppm)

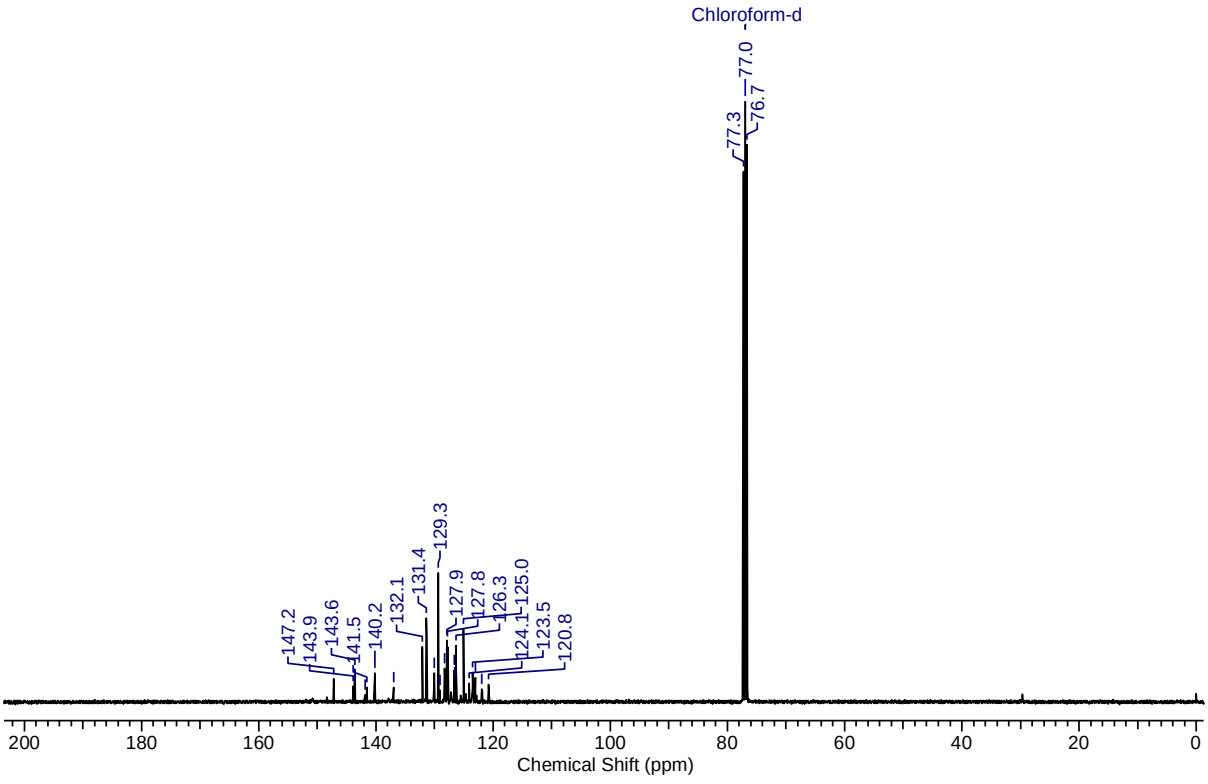
HRMS of 1:



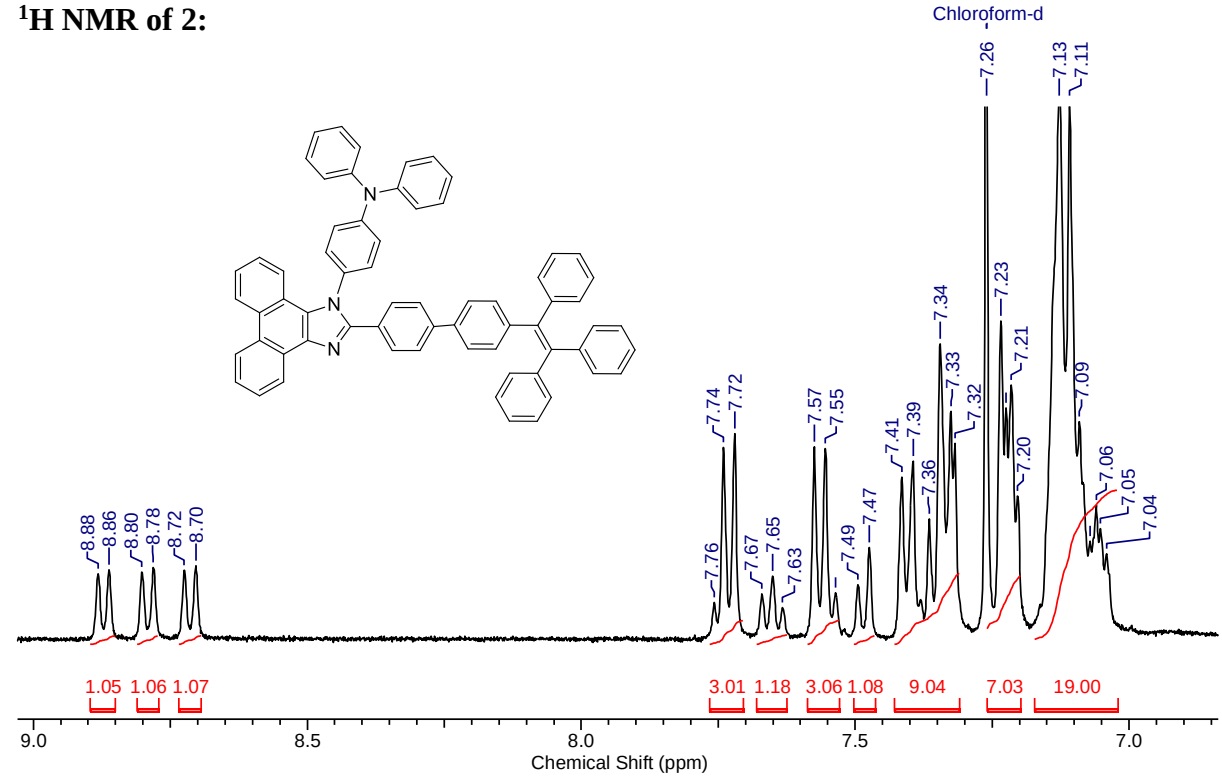
Bruker Compass DataAnalysis 4.0 printed: 10/19/2016 2:56:24 PM Page 1 of 1

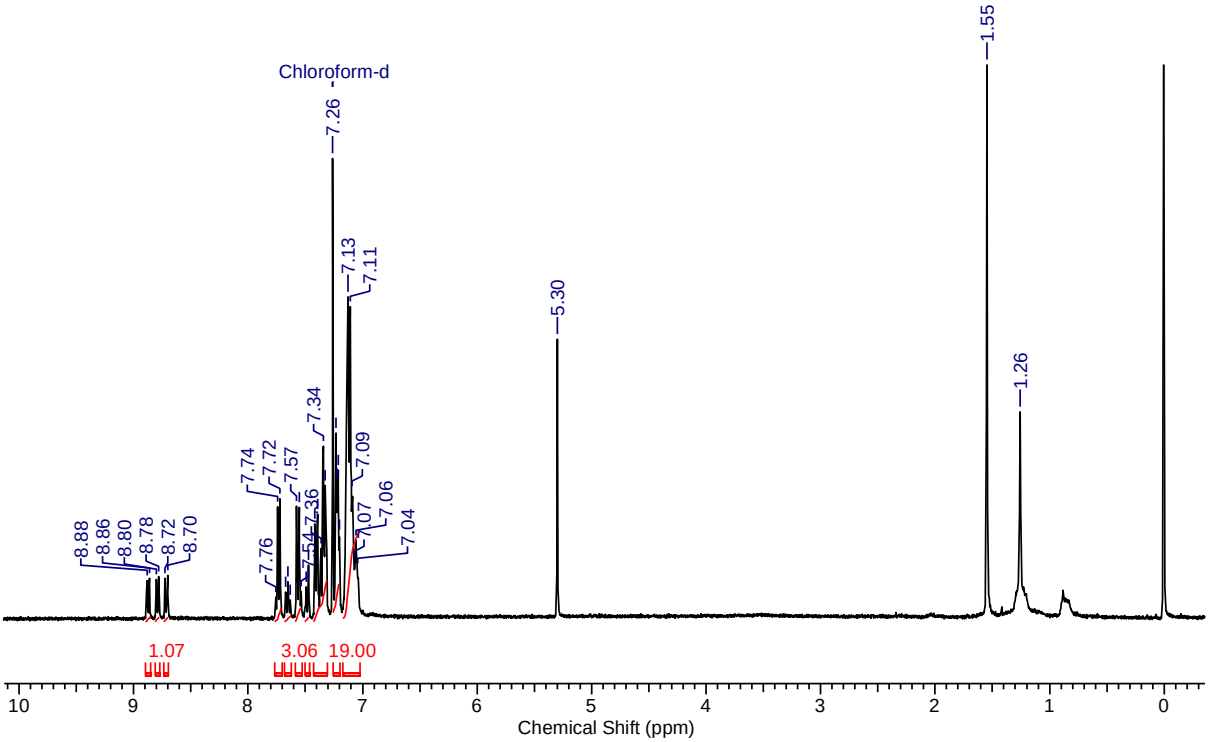
^{13}C NMR of 1:


N
N
N
147.2
143.9
143.6
143.6
143.6
141.8
141.5
140.2
137.0
132.1
131.4
131.3
130.1
129.4
129.3
129.1
128.3
127.9
127.8
127.7
126.6
126.6
126.3
125.0
124.1
123.5
123.1
121.9
120.8
148
146
144
142
140
138
136
134
132
130
128
126
124
122
120
Chemical Shift (ppm)

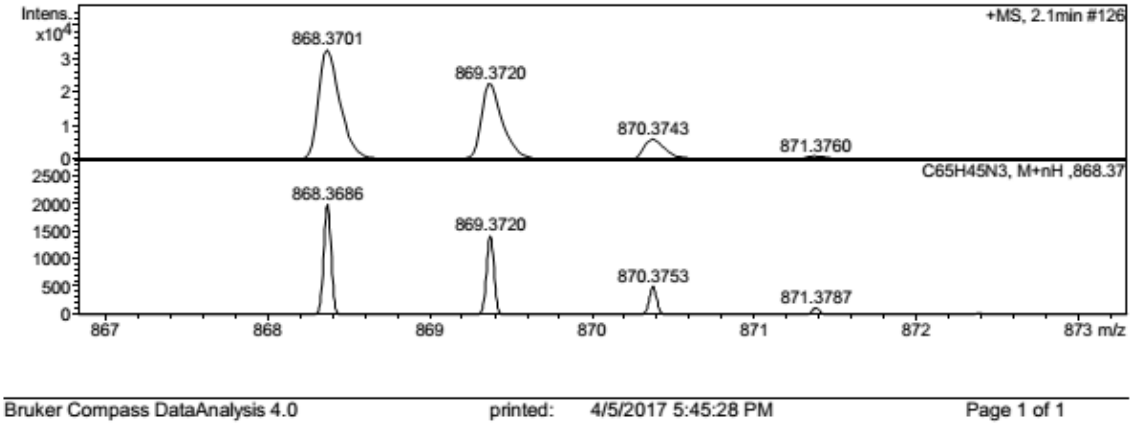

Chloroform-d
77.3
77.0
76.7
147.2
143.9
143.6
141.5
140.2
132.1
131.4
129.3
127.9
127.8
126.3
125.0
124.1
123.5
120.8
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

^{1}H NMR of 2:


Chloroform-d
7.26
7.13
7.11
7.34
7.23
7.21
7.09
7.33
7.32
7.74
7.72
7.57
7.55
7.41
7.39
7.36
7.20
7.06
7.05
7.04
7.47
7.49
7.76
7.67
7.65
7.63
8.88
8.86
8.80
8.78
8.72
8.70
1.05 1.06 1.07
3.01 1.18 3.06 1.08 9.04 7.03 19.00
9.0
8.5
8.0
7.5
7.0
Chemical Shift (ppm)

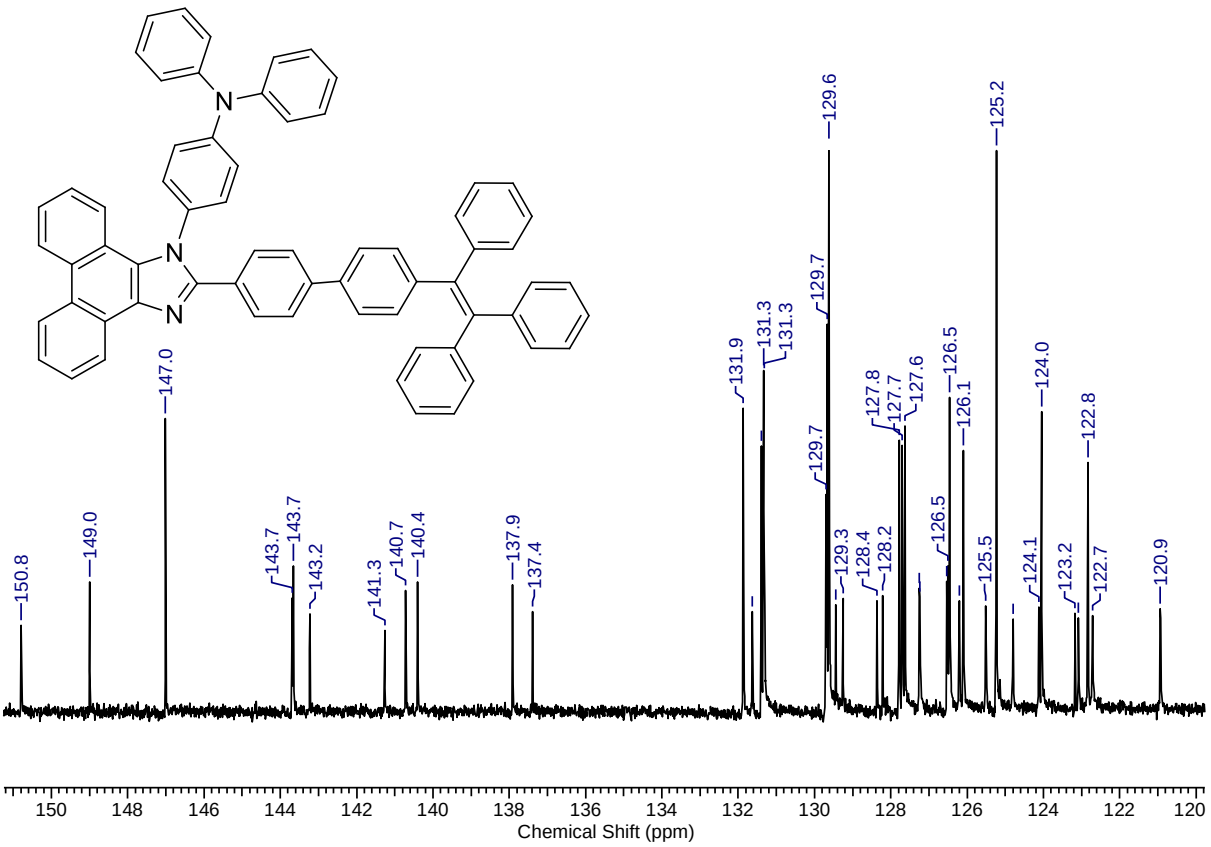

Chloroform-d
1.55
7.26
7.13
7.11
5.30
1.26
7.34
7.74
7.72
7.57
7.36
7.09
7.06
7.07
7.04
7.76
8.88
8.86
8.80
8.78
8.72
8.70
1.07
3.06 19.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

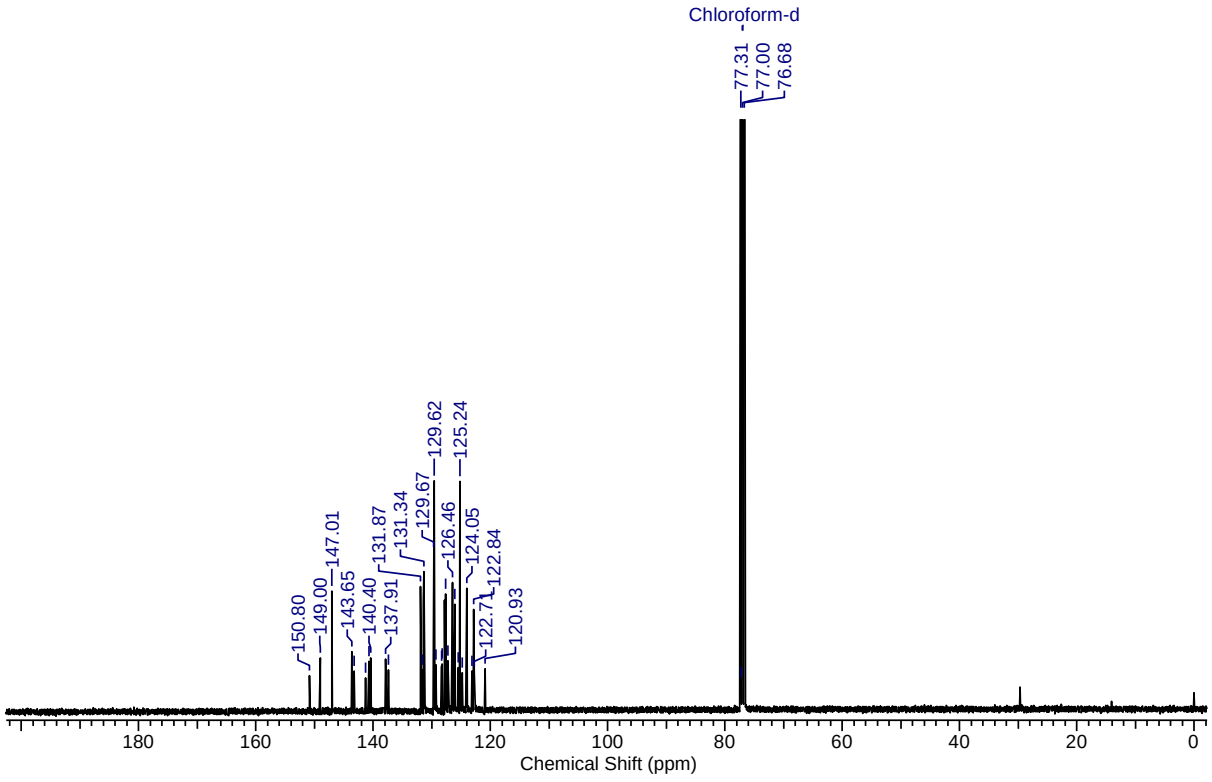
HRMS of 2:



Bruker Compass DataAnalysis 4.0 printed: 4/5/2017 5:45:28 PM Page 1 of 1

^{13}C NMR of 2:


150.8
149.0
147.0
143.7
143.7
143.2
141.3
140.7
140.4
137.9
137.4
131.9
131.3
131.3
129.7
129.7
129.6
129.3
128.4
128.2
127.8
127.7
127.6
126.5
126.1
126.5
125.5
125.2
124.1
124.0
123.2
122.8
122.7
120.9
150
148
146
144
142
140
138
136
134
132
130
128
126
124
122
120
Chemical Shift (ppm)


Chloroform-d
77.31
77.00
76.68
150.80
149.00
147.01
143.65
140.40
137.91
131.87
131.34
129.67
129.62
126.46
125.24
124.05
122.84
122.71
120.93
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)