Supporting Information

The outside knows the difference inside: trapping helium by immediate reduction of orifice size of an open-cage fullerene and the effect of encapsulated helium and hydrogen upon the NMR of proton directly attached to the outside

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Content

[1] M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, J. A. Montgomery, Jr., T. Vreven, K. N. Kudin, J. C. Burant, J. M. Millam, S. S. Iyengar, J. Tomasi, V. Barone, B. Mennucci, M. Cossi, G. Scalmani, N. Rega, G. A. Petersson, H. Nakatsuji, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, M. Klene, X. Li, J. E. Knox, H. P. Hratchian, J. B. Cross, C. Adamo, J. Jaramillo, R. Gomperts, R. E. Stratmann, O. Yazyev, A. J. Austin, R. Cammi, C. Pomelli, J. W. Ochterski, P. Y. Ayala, K. Morokuma, G. A. Voth, P. Salvador, J. J. Dannenberg, V. G. Zakrzewski, S. Dapprich, A. D. Daniels, M. C. Strain, O. Farkas, D. K. Malick, A. D. Rabuck, K. Raghavachari, J. B. Foresman, J. V. Ortiz, Q. Cui, A. G. Baboul, S. Clifford, J. Cioslowski, B. B. Stefanov, G. Liu, A. Liashenko, P. Piskorz, I. Komaromi, R. L. Martin, D. J. Fox, T. Keith, M. A. Al-Laham, C. Y. Peng, A. Nanayakkara, M. Challacombe, P. M. W. Gill, B. Johnson, W. Chen, M. W. Wong, C. Gonzalez, and J. A. Pople, Gaussian 03, Revision B.05, Gaussian, Inc., Pittsburgh PA, 2003.

Experimental Section

General method.

Dry ODCB was distilled from CaH_2 and THF from Na/benzophenone under argon. Sodium Boronhydride (Aldrich) were used as received.

Synthesis of compound 2

To a solution of compound **1**[2] (1.011 g, 0.9484 mmol) in 100 mL dry ODCB was added 15 equivalents of $NaBH_4$ (0.532 g, 0.0141 mmol) at room temperature. THF (20 mL) was added slowly to the solution and stirred at room temperature for 3 h. The whole solution was stirred and monitored by HPLC (Buckyprep column). An additional 10 mL of THF was added after 3 h and further stirred for 2 h. Upon completion of the reaction, the mixture was passed through a short pad of Al_2O_3 gel to remove insoluble materials and suspension of excess $NaBH_4$. 1 M HCl (20 mL) was slowly added to the filtrate. The organic layer was separated and dried with Na_2SO_4. Evaporation of solvents at reduced pressure formed some brown solids. The mixture was precipitated with pentane and centrifuged to give 874 mg compound **2**. Yield: 86%. ^{1}H NMR ($CDCl_3/CS_2$ = 1:1, 400 MHz) δ 5.27 (s, 1H), 6.45 (s, 1H), 6.88-6.97 (m, 2H), 7.04–7.07 (m, 1H), 7.18–7.28 (m, 4H), 7.52 (dt, J = 2.0, 7.9 Hz, 1H), 7.95 (br, 1H), 7.96–7.99 (m, 2H), 8.07 (d, J = 7.9 Hz, 1H), 8.36 (br, 1H), 8.43–8.45 (m, 1H); ^{13}C NMR (ODCB-d_4/CS_2 = 1:1, 100 MHz) δ 51.20, 69.71, 84.10, 94.44, 121.44, 122.15, 122.92, 125.66, 130.25, 130.69, 131.04, 131.59, 132.16, 132.43, 132.57, 132.88, 132.96, 133.15, 135.96, 136.12, 136.85, 137.04, 137.94, 138.00, 138.39, 138.67, 139.24, 139.34, 139.69, 139.81, 140.28, 140.40, 140.58, 140.96, 141.34, 141.57, 142.03, 142.32, 142.56, 144.18, 144.59, 145.33, 146.38, 146.61, 147.31, 147.51, 147.54, 147.56, 147.63, 147.65, 147.82, 148.10, 148.21, 148.24, 148.30, 148.32, 148.67, 148.70, 149.13, 149.73, 149.90, 149.98, 150.44, 151.54, 152.93, 155.00, 162.63, 198.62; FT-IR ν (cm^{-1}) (C=O) 1707. UV-vis ($CHCl_3$, 5.0 × 10^{-5} M) λ_{max} (ε)

[2] Murata, Y.; Murata, M.; Komatsu, K. *Chem. Eur. J.* 2003, **9**, 1600.

231 (38802), 262 (8687), 316 (3701, sh), 390 (1412, sh). HRMS (FAB-positive mode) calcd for $C_{80}H_{17}O_2N_2S$ ($M + H^+$): 1069.1011; found: 1069.1005.

Synthesis of compound H_2@2

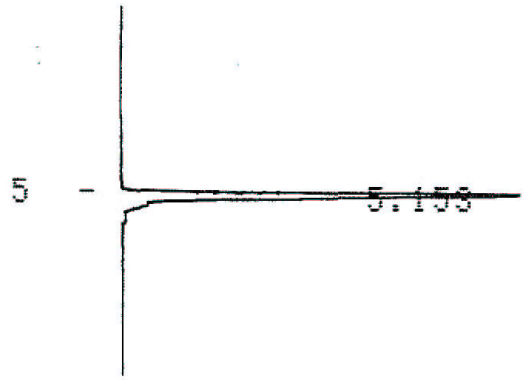
To a solution of compound H_2@**1** (136 mg, 0.127 mmol), including 25% empty **1**, in 45 mL dry ODCB was added 15 equiv of $NaBH_4$ (72.4 mg, 1.91 mmol) at ambient temperature. A total of 15 mL of THF was slowly added to the solution in 1.5 h. The whole solution was stirred and monitored by HPLC (Buckyprep column). Upon completion of the reaction, the mixture was passed through a short pad of Al_2O_3 gel to remove insoluble materials. Evaporation of solvents to about 5 mL at reduced pressure formed brown solids. The mixture was precipitated with pentane and centrifuged to give 113 mg compound H_2@**2**. Yield: 83%. ^{1}H NMR (ODCB-d_4, 300 MHz) δ –7.83 (s, 1.5H), 5.54 (s, 1H), 6.56 (s, 1H), 6.99–7.40 (m, 7H), 7.60 (dt, J = 1.8, 7.8 Hz, 1H), 8.11–8.15 (br, 1H), 8.17–8.20 (m, 2H), 8.22–8.25 (m, 1H), 8.54–8.56 (m, 1H), 8.58–8.66 (br, 1H); APCI-MS (positive mode): calcd for $C_{80}H_{18}O_2N_2SNa$ ($M + Na^+$): 1093.1; found: 1093.3.

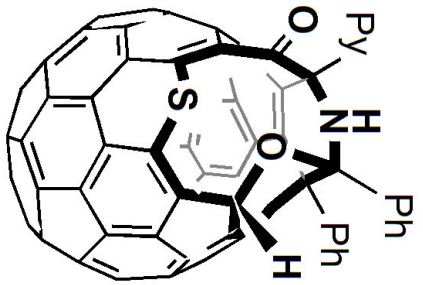
Synthesis of compound He@2

The insertion of He into **1** (101 mg, 0.095 mmol) was carried out at a relatively lower temperature at 90 ^{o}C (650 atm) with reaction time of 24 hr inside a stainless autoclave. Immediate cooling of the autoclave from 90 ^{o}C to room temperature (15 ^{o}C) and further down to –78 ^{o}C (dry ice/isopropanol) was carried out in 10 minutes long. The autoclave was kept at –78 ^{o}C for one additional hour and opened quickly to transfer sample in 30 ml of a pre-cooled ODCB at –25 ^{o}C. The whole mixture was sonicated for 5 min and added 15 equiv of $NaBH_4$ (54 mg, 1.43 mmol) in one portion at –20 to –25 ^{o}C. A total of 6 mL of THF was slowly added to the solution in 1 h. The whole solution was stirred, sonicated and monitored by HPLC (Buckyprep column). Upon completion of the reaction, the mixture was added 5 mL of 1 M HCl and passed through a short pad of Al_2O_3 gel to remove insoluble materials. Then 20 mL of saturated brine solution was added and the organic layer was separated. The organic layer was dried with Na_2SO_4 and solvents were evaporated under vacuum. CS_2/ODCB (1:1) was added to dissolve

solids and the solution was divided into four portions for silica gel chromatography. After chromatography using toluene/ethyl acetate (20/1 to 5/1), a total of 90.9 mg of He@**2** was collected. The product was determined to contain >35% He incorporation by APCI-MS (positive mode). Yield: 90%. ^{1}H NMR (ODCB-d_4, 300 MHz) δ 5.54 (s, 1H), 6.56 (s, 1H), 6.90–7.40 (m, 7H), 7.60 (dt, J = 1.8, 7.8 Hz, 1H), 8.08–8.15 (br, 1H), 8.17–8.20 (m, 2H), 8.22–8.25 (m, 1H), 8.54–8.56 (m, 1H), 8.58–8.66 (br, 1H); APCI-MS (positive mode): calcd for $C_{80}H_{16}O_2N_2SHeNa$ (M + Na+): 1095.1; found: 1095.3.

Figure S1. HPLC of compound **2**.





analytical Buckyprep (4.6 mm x 250 mm)
(detection at 326 nm)
eluent: toluene
rate: 1mL/min

CHROMATOGRAM 1 MEMORIZED

CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 7165

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	2.967	1341			0.0358	
2	3.163	10247	V		0.2738	
3	3.683	12844			0.3432	
4	4.442	16854	V		0.4503	
5	4.697	14337	V		0.383	
6	5.153	3657622	SV		97.721	
7	5.74	21558	T		0.576	
8	7.39	3966	T		0.1059	
9	7.982	2670	TV		0.0713	
10	8.495	1484	TV		0.0396	
	TOTAL	3742922			100	

Figure S2. FAB MS spectrum of compound **2** ($M + H^+ = 1069$)

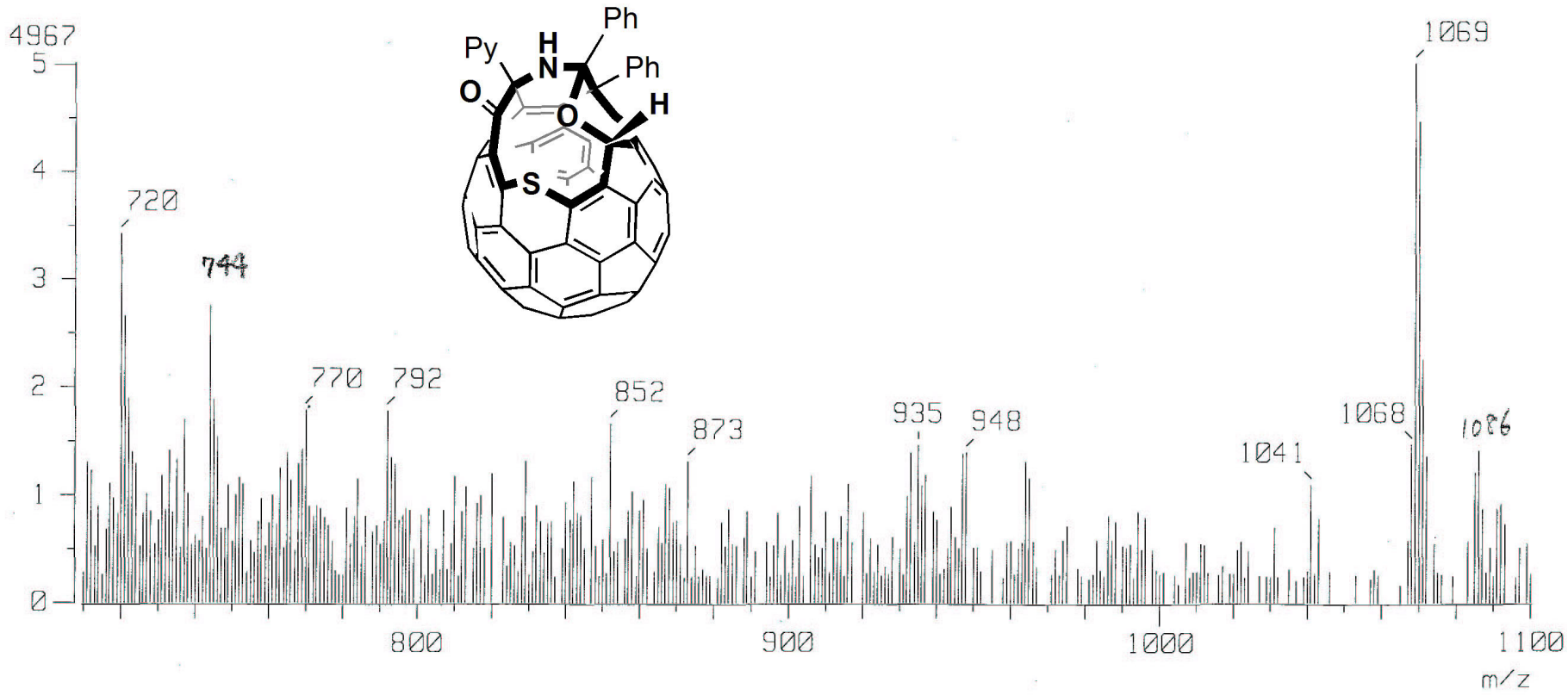
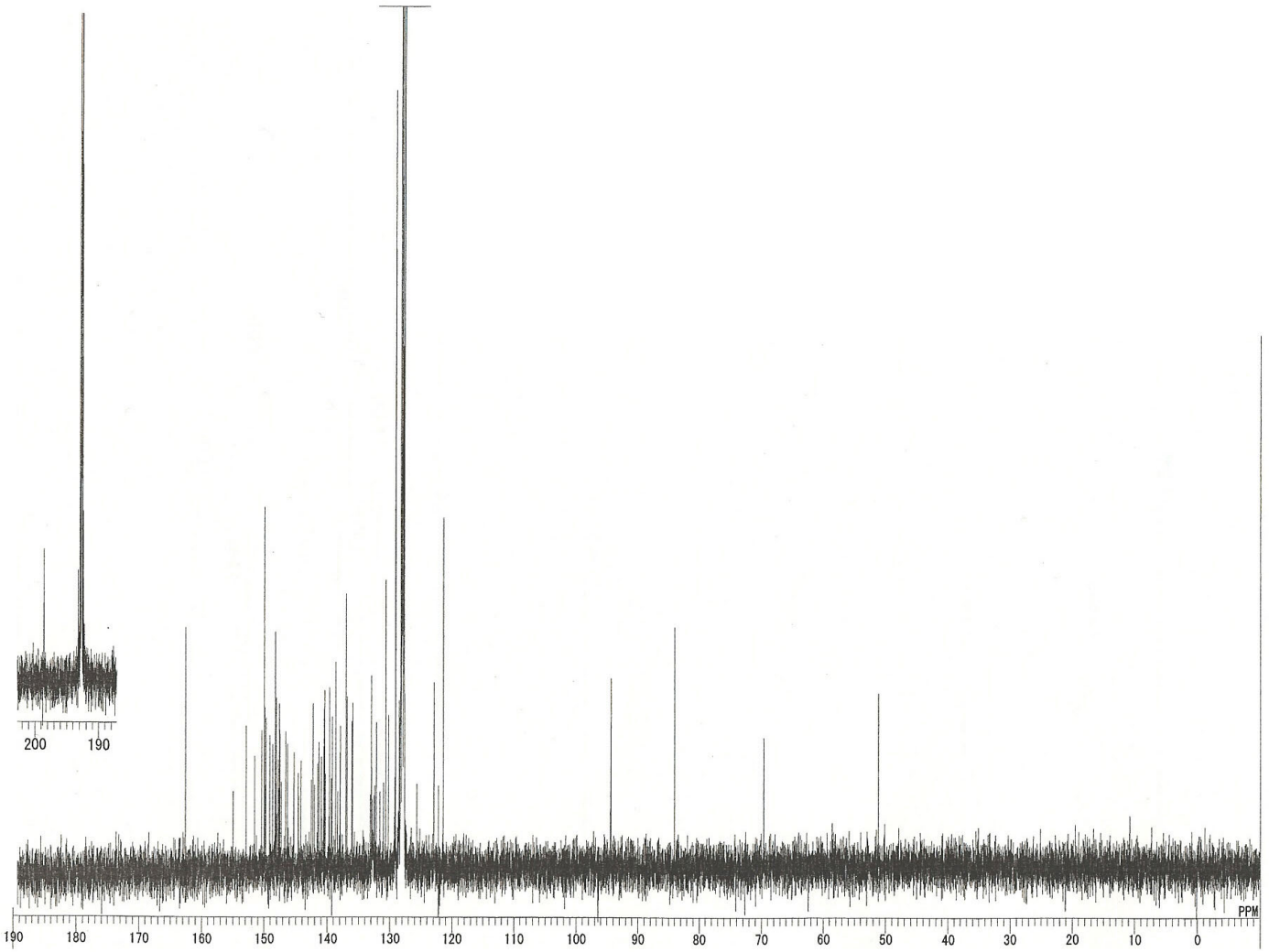


Figure S3. ^{13}C NMR spectrum of compound **2** (100 MHz, ODCB-d_4/CS_2 = 1:1)



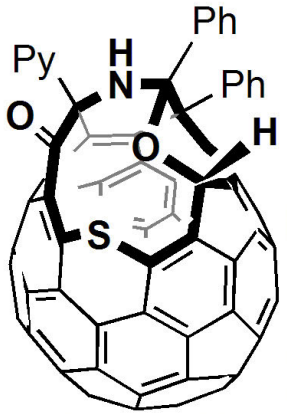
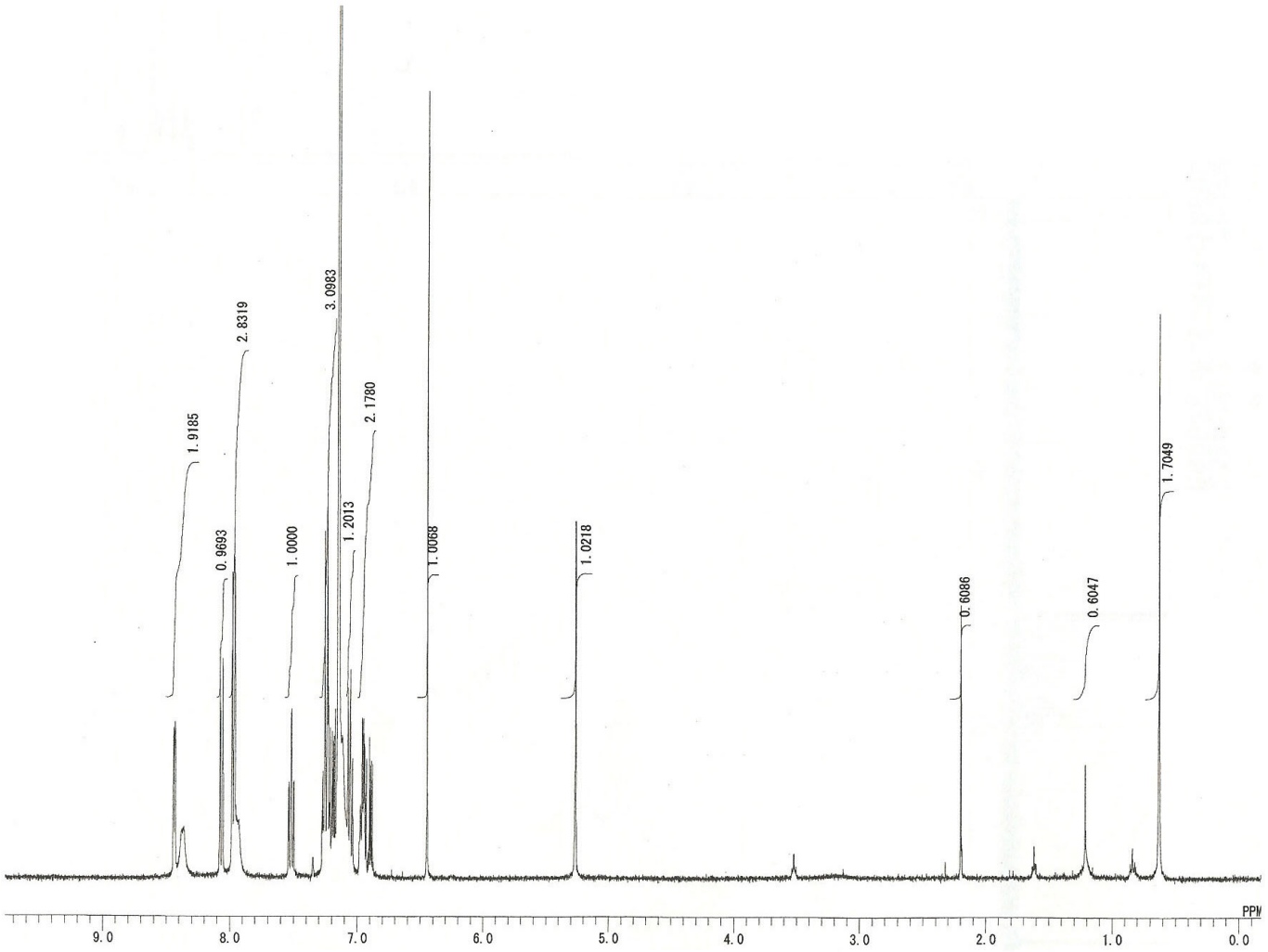


Figure S4. ^{1}H NMR spectrum of compound **2** (400 MHz, $CDCl_3/CS_2$ = 1:1)



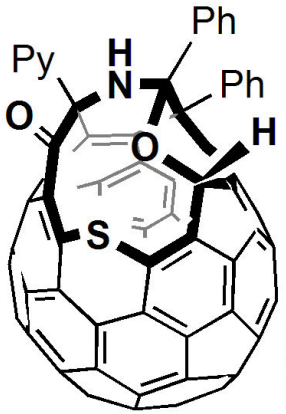
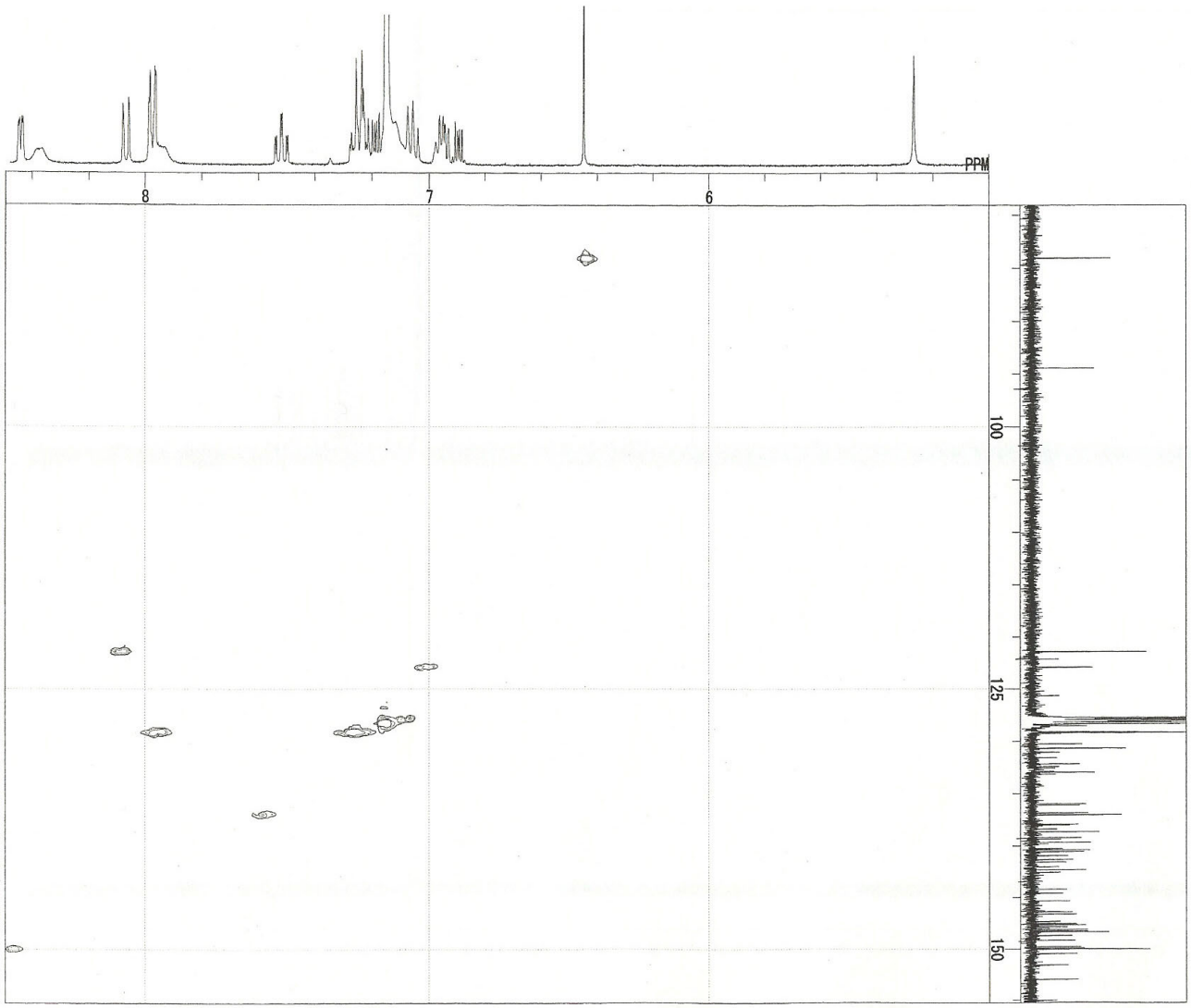


Figure S5. 2D-HMQC spectrum of compound **2** (400 MHz, $CDCl_3/CS_2$ = 1:1)



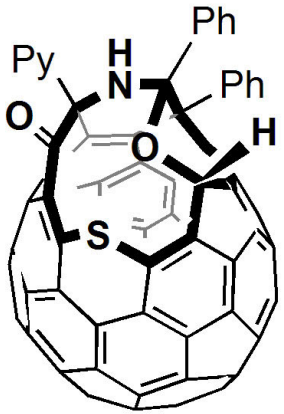
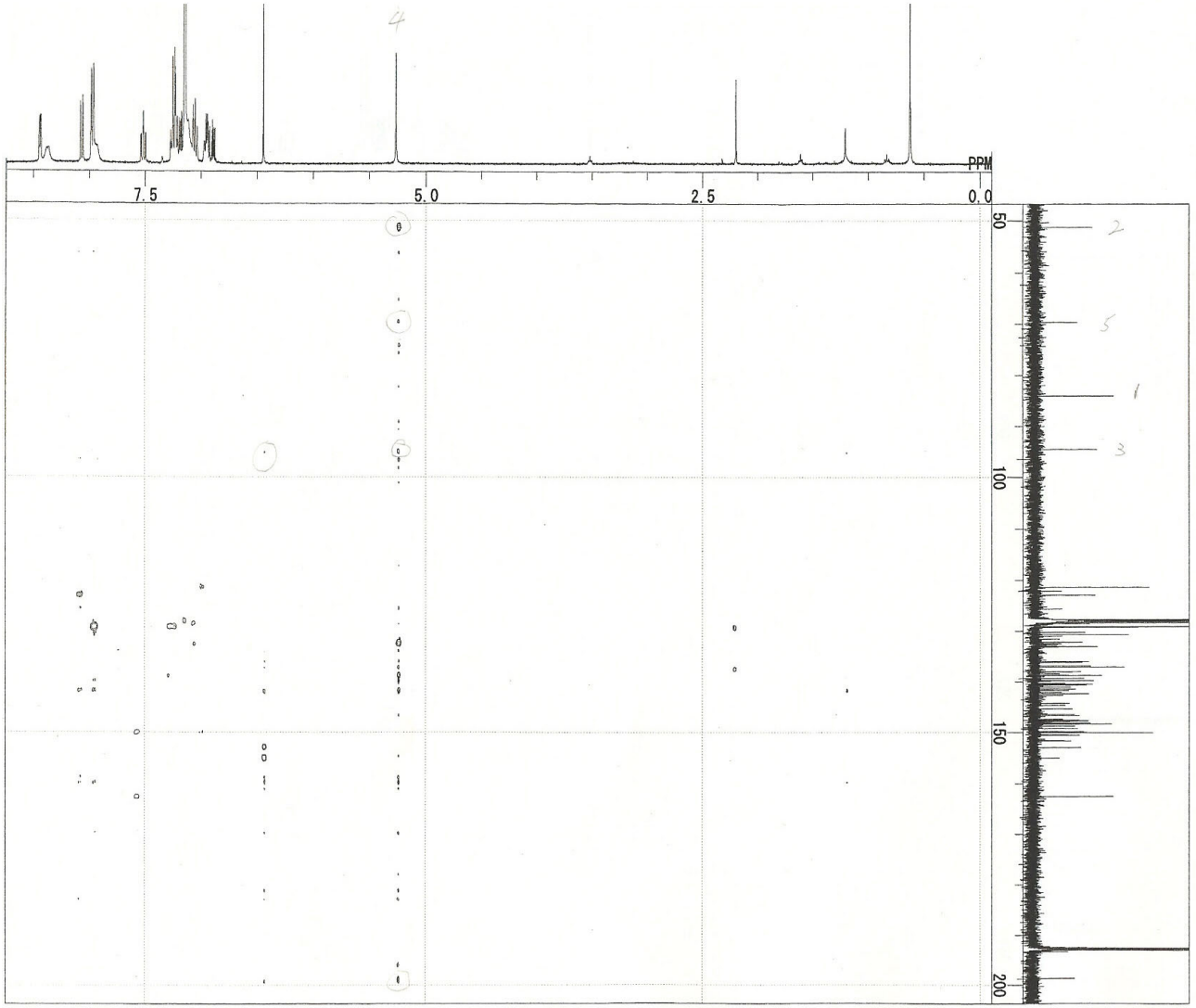


Figure S6. 2D-HMBC spectrum of compound **2** (400 MHz, $CDCl_3/CS_2$ = 1:1)



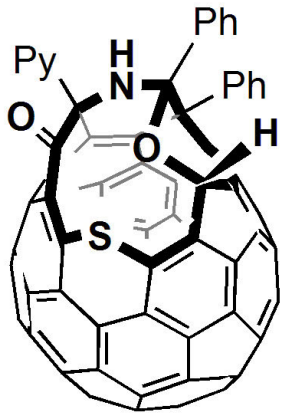


Figure S7. IR spectrum of compound **2** (KBr)

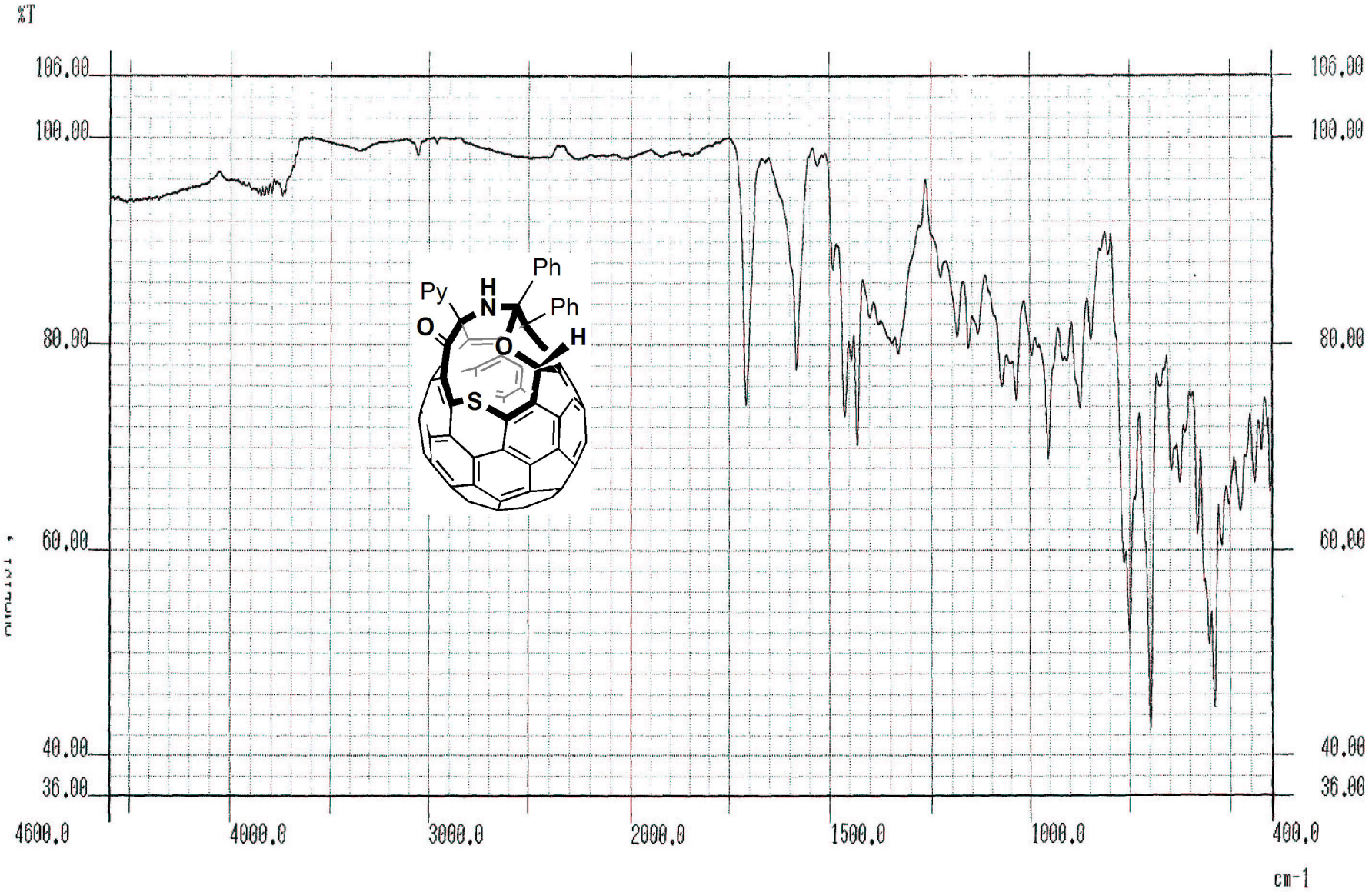
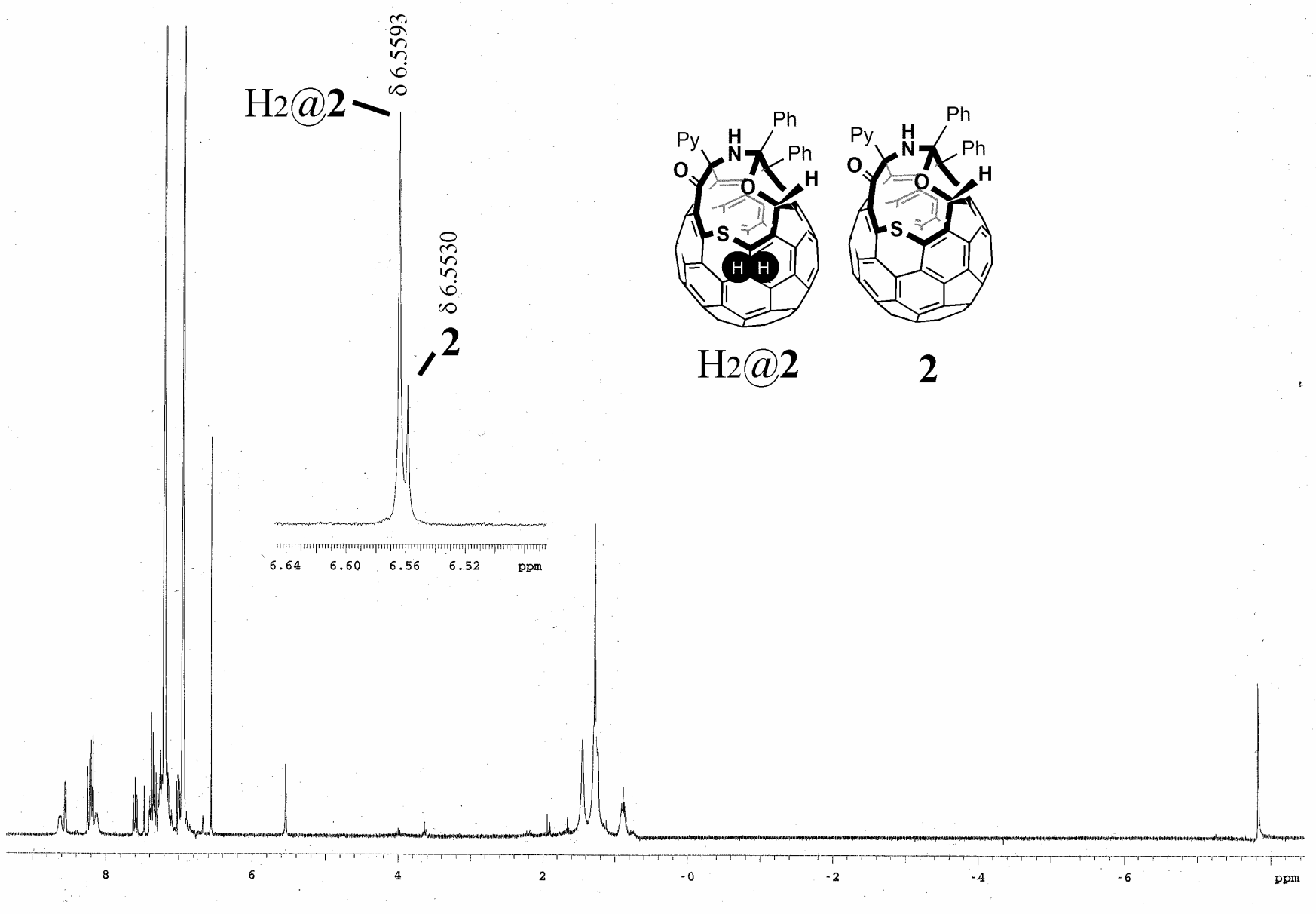


Figure S8. ^{1}H NMR spectrum of compound H_2@**2** (300 MHz, ODCB-d_4)



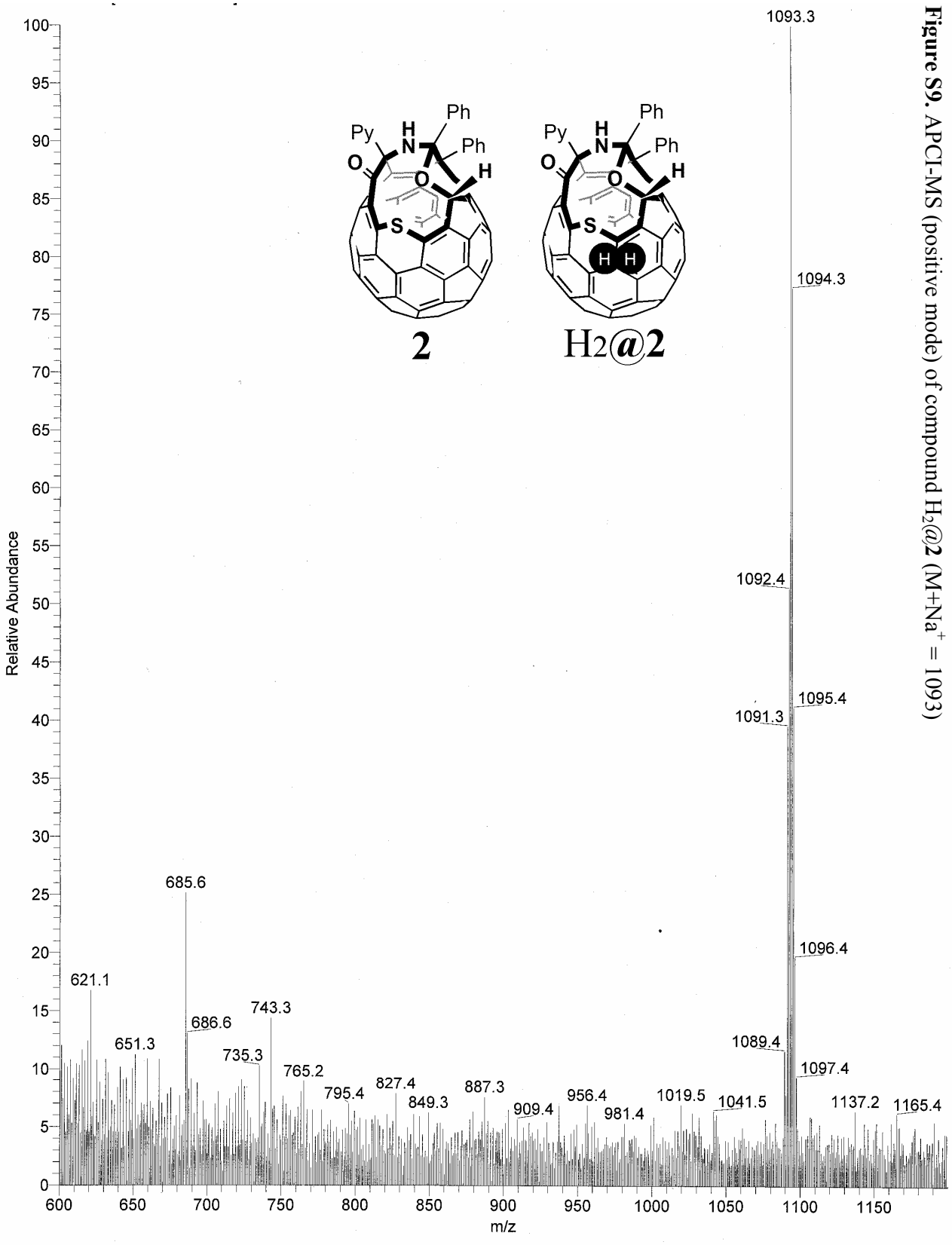
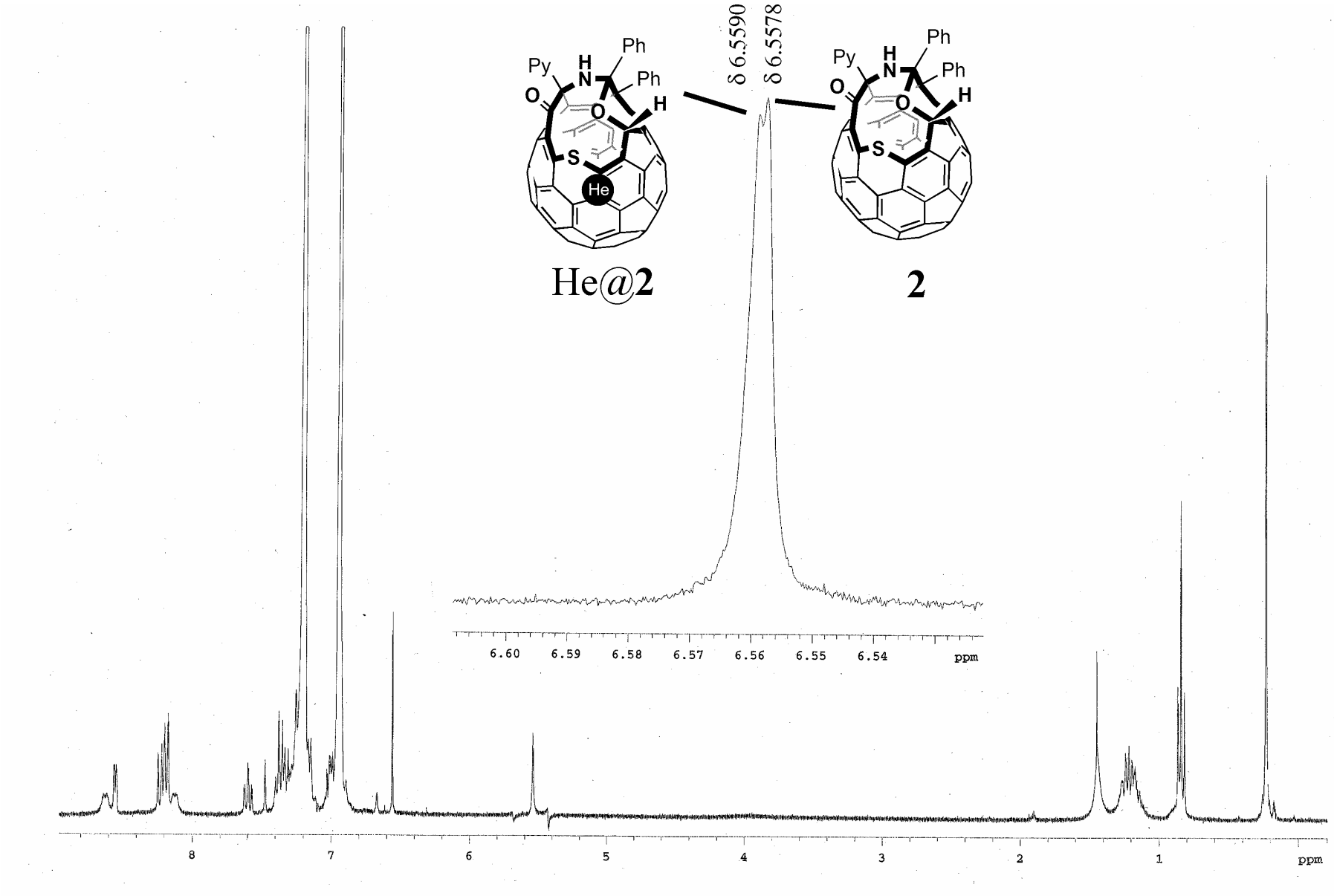


Figure S9. APCI-MS (positive mode) of compound H_2@**2** ($M+Na^+$ = 1093)

Figure S10. ^{1}H NMR spectrum of compound He@**2** (300 MHz, ODCB-d_4)



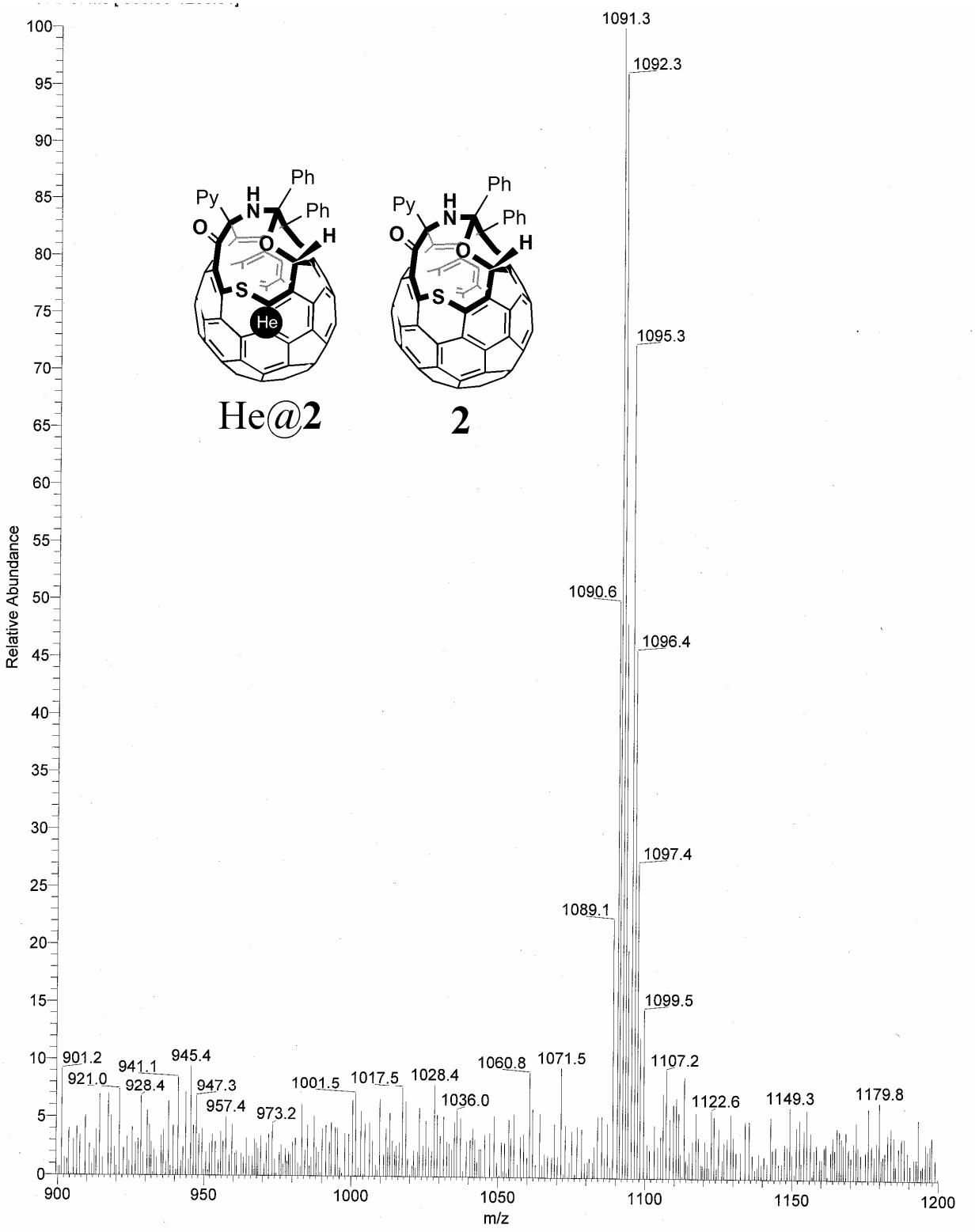


Figure S11. APCI-MS (positive mode) of compound He@**2** ($M+Na^+$ = 1095)

Optimized structure of compound **2**

B3LYP/3-21G

0 1

6 0	3.550943	-0.124104	3.237879
6 0	2.122779	-0.026211	3.512519
6 0	1.467557	1.174291	3.344463
6 0	2.190329	2.344908	2.856436
6 0	3.534031	2.235424	2.514642
6 0	4.234974	0.974451	2.720341
6 0	1.477501	-1.263563	3.096484
6 0	0.133007	1.185188	2.787116
6 0	1.273814	3.084144	2.008355
6 0	4.004487	2.812628	1.257161
6 0	5.165357	0.783704	1.610656
6 0	3.809163	-1.457827	2.723153
6 0	3.104595	3.463881	0.419598
6 0	1.717018	3.632280	0.813131
6 0	3.108042	3.172959	-1.008505
6 0	0.881042	3.507672	-0.369944
6 0	0.214320	-1.266708	2.468812
6 0	-0.010650	2.413853	2.030497
6 0	1.743389	3.143261	-1.479164
6 0	-0.882826	2.449672	0.954383
6 0	-0.496625	0.011851	2.331103
6 0	4.994234	1.905009	0.690610
6 0	5.411365	-0.492366	1.117152
6 0	-0.064360	-2.305641	1.464203
6 0	4.741623	-1.649194	1.705494
6 0	2.539254	-2.173085	2.686684
6 0	5.503559	-0.718327	-0.322848
6 0	4.471237	-2.608049	0.642849
6 0	2.296755	-3.102167	1.697970
6 0	-0.420550	3.016025	-0.314435
6 0	5.023733	1.675301	-0.683768
6 0	-1.603104	0.163746	1.401133
6 0	4.942677	-2.032350	-0.612845
6 0	3.293923	-3.351091	0.658209
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6 0	1.149189	-3.611195	-0.263718
6 0	-1.137551	-2.069725	0.506346
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6 0	-2.416639	2.083116	-1.522248
6 0	2.574523	-3.622776	-0.572545
6 0	3.021461	-3.066869	-1.772558
6 0	2.059040	-2.476145	-2.698102
6 0	4.210078	-2.232153	-1.778716
6 0	4.022262	2.292849	-1.543378
6 0	5.281888	0.342516	-1.198364
6 0	4.441769	0.151200	-2.374232
6 0	-0.948290	-2.478448	-0.792854
6 0	-0.982443	2.478178	-1.530774
6 0	-0.133549	2.084977	-2.549689
6 0	0.242079	-3.152318	-1.195375
6 0	0.706430	-2.550417	-2.428415

6	0	3.939880	-1.099115	-2.661043			
6	0	2.597766	-1.218505	-3.177723			
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6	0	3.598040	1.326943	-2.551362			
6	0	1.299525	2.236130	-2.457271			
6	0	0.325851	-0.354373	-3.377063			
6	0	2.257007	1.235113	-2.986883			
6	0	1.755681	-0.106156	-3.366601			
6	0	-2.291011	-1.118558	0.870476			
6	0	-2.983342	1.661316	-0.104367			
7	0	-3.810693	0.472155	-0.222709			
6	0	-3.123743	-0.760738	-0.434382			
6	0	-3.938136	2.804142	0.308856			
6	0	-4.122153	-1.767192	-1.016079			
6	0	-3.182390	-1.677389	2.008403			
6	0	-4.665656	-1.388301	-2.259539			
6	0	-5.576394	-2.214282	-2.911947			
6	0	-5.948652	-3.435466	-2.340112			
6	0	-5.402158	-3.818825	-1.116498			
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6	0	-4.281339	-0.925406	2.452902			
6	0	-5.095696	-1.396260	3.482130			
6	0	-4.829007	-2.624503	4.090969			
6	0	-3.739590	-3.379176	3.656929			
6	0	-2.922359	-2.909904	2.625003			
6	0	-3.588444	3.879414	1.124935			
6	0	-4.539230	4.872060	1.371835			
6	0	-5.804553	4.767896	0.793522			
6	0	-6.073395	3.659985	-0.009298			
7	0	-5.161954	2.697874	-0.247704			
8	0	-3.137597	2.041768	-2.517372			
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16	0	-0.781610	1.013933	-3.897730			
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1	0	-4.345180	-0.452341	-2.705741			
1	0	-5.989353	-1.910888	-3.866876			
1	0	-6.656300	-4.080958	-2.847275			
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1	0	-4.094525	-3.289959	0.504261			
1	0	-4.503553	0.011930	1.963116			
1	0	-5.941583	-0.801237	3.807018			
1	0	-5.461259	-2.987444	4.892857			
1	0	-3.518878	-4.334367	4.119519			
1	0	-2.082418	-3.509629	2.302353			
1	0	-4.291395	5.712661	2.008648			
1	0	-6.563269	5.521152	0.961396			
1	0	-7.043787	3.534180	-0.475574			
1	0	-2.229009	-2.301284	-2.571127			
1	0	-4.657452	0.655607	-0.763413			
1	0	-2.604439	3.937567	1.564342			

Single point energy = -3716.09566842 hartrees

(SPE, B3LYP/6-31G**//B3LYP/3-21G)

Optimized TS structure coordinate of He-**2** (B3LYP/3-21G); Nimag = 1

0 1

6	0	-3.363462	-0.422794	-3.343226
6	0	-1.922020	-0.297935	-3.517197
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6	0	-2.098365	2.106103	-3.017382
6	0	-3.461503	1.976509	-2.770645
6	0	-4.111256	0.683421	-2.944818
6	0	-1.278995	-1.489737	-2.985762
6	0	-0.030674	1.011001	-2.702500
6	0	-1.268925	2.928727	-2.155693
6	0	-4.042798	2.617480	-1.592099
6	0	-5.113837	0.531285	-1.893457
6	0	-3.624274	-1.731952	-2.769552
6	0	-3.229009	3.355203	-0.739978
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6	0	0.027063	2.291405	-2.023110
6	0	-2.002323	3.223368	1.270086
6	0	0.807327	2.422952	-0.887122
6	0	0.580788	-0.111792	-2.122452
6	0	-5.042395	1.712908	-1.037635
6	0	-5.357111	-0.720028	-1.337760
6	0	0.170382	-2.401579	-1.199138
6	0	-4.619322	-1.889616	-1.806903
6	0	-2.343158	-2.408253	-2.605280
6	0	-5.538156	-0.860335	0.104559
6	0	-4.396933	-2.774763	-0.671487
6	0	-2.146583	-3.275348	-1.552114
6	0	0.237641	3.095416	0.279540
6	0	-5.159067	1.566759	0.344098
6	0	1.598125	0.122399	-1.103859
6	0	-4.961435	-2.135282	0.513840
6	0	-3.206758	-3.487772	-0.566371
6	0	-0.865281	-3.305120	-0.878329
6	0	-1.126634	-3.646413	0.508524
6	0	1.196530	-2.109401	-0.195168
6	0	1.785302	1.394646	-0.615832
6	0	2.248240	2.571571	1.619946
6	0	-2.564104	-3.664693	0.723186
6	0	-3.090808	-3.036418	1.853379
6	0	-2.195305	-2.348971	2.779668
6	0	-4.297530	-2.237539	1.732674
6	0	-4.239333	2.272693	1.227238
6	0	-5.407100	0.258884	0.922632
6	0	-4.634907	0.168726	2.156062
6	0	0.936071	-2.483875	1.096544
6	0	0.743691	2.703938	1.577685
6	0	-0.161932	2.328420	2.554159
6	0	-0.290090	-3.111943	1.460661
6	0	-0.829856	-2.400127	2.598691
6	0	-4.111126	-1.041351	2.550463
6	0	-2.794055	-1.080712	3.139186
6	0	0.049612	-1.302810	2.960704
6	0	-3.841368	1.379124	2.311239
6	0	-1.589739	2.390755	2.322083

6	0	-0.540978	-0.139537	3.419475	7	0	5.098218	2.341035	-0.508276
6	0	-2.525477	1.361868	2.828070	8	0	2.967622	3.028976	2.497930
6	0	-1.989611	0.057935	3.293726	8	0	2.509848	-0.895078	2.092581
6	0	2.329451	-1.120319	-0.533391	16	0	0.309494	1.388418	4.087105
6	0	2.866574	1.758392	0.363379	6	0	1.480986	-1.809588	2.348187
7	0	3.667112	0.605005	0.725665	1	0	5.136743	-0.246703	2.507711
6	0	3.223143	-0.759515	0.759706	1	0	7.085993	-1.590510	3.203311
6	0	3.839769	2.768216	-0.321244	1	0	7.472847	-3.836622	2.198244
6	0	4.455686	-1.653573	1.027953	1	0	5.879529	-4.698444	0.494748
6	0	3.224412	-1.608364	-1.708463	1	0	3.998145	-3.327993	-0.251200
6	0	5.332484	-1.197997	2.031354	1	0	4.502477	0.131128	-1.629893
6	0	6.418128	-1.968622	2.437861	1	0	5.921820	-0.561728	-3.507213
6	0	6.630731	-3.232583	1.881075	1	0	5.484139	-2.703177	-4.695598
6	0	5.738482	-3.713830	0.925367	1	0	3.591417	-4.140956	-3.961089
6	0	4.656289	-2.934264	0.504867	1	0	2.170871	-3.453094	-2.078461
6	0	4.291327	-0.802410	-2.133580	1	0	4.083860	5.906005	-1.581797
6	0	5.099873	-1.197577	-3.198939	1	0	6.431093	5.091908	-1.940102
6	0	4.856400	-2.399760	-3.866043	1	0	7.005650	2.773010	-1.201712
6	0	3.795207	-3.204808	-3.454047	1	0	1.826602	-2.577681	3.055754
6	0	2.985828	-2.812150	-2.385197	1	0	4.666658	0.775047	0.604419
6	0	3.441803	4.055335	-0.694647	1	0	2.428835	4.389564	-0.524126
6	0	4.373598	4.905146	-1.286007	2	0	1.830237	0.851027	2.409250
6	0	5.682374	4.456062	-1.486389	Single point energy = -3718.92169650 hartrees				
6	0	6.003838	3.166533	-1.078663	(SPE, B3LYP/6-31G**//B3LYP/3-21G)				

Optimized structure of He@**2**

B3LYP/3-21G

0 1

6 0	3.543979	-0.128203	3.235771
6 0	2.116365	-0.030891	3.511830
6 0	1.460763	1.169838	3.346465
6 0	2.182987	2.341582	2.859995
6 0	3.526375	2.232361	2.516728
6 0	4.227750	0.971309	2.720372
6 0	1.471046	-1.267703	3.094852
6 0	0.125710	1.181430	2.790098
6 0	1.265888	3.081104	2.012682
6 0	3.996130	2.811693	1.260006
6 0	5.156796	0.782129	1.609260
6 0	3.802537	-1.461237	2.719107
6 0	3.095373	3.463423	0.423664
6 0	1.708142	3.631569	0.818143
6 0	3.098496	3.175534	-1.004996
6 0	0.871572	3.508420	-0.364765
6 0	0.208337	-1.269199	2.466632
6 0	-0.018045	2.410261	2.034142
6 0	1.733594	3.146548	-1.475172
6 0	-0.891349	2.448174	0.959039
6 0	-0.504048	0.008739	2.332544
6 0	4.985583	1.904995	0.691431
6 0	5.404406	-0.493117	1.114282
6 0	-0.071215	-2.306486	1.460978
6 0	4.734767	-1.651072	1.700886
6 0	2.532823	-2.176758	2.683109
6 0	5.494654	-0.716340	-0.326291
6 0	4.464485	-2.608597	0.637079
6 0	2.289790	-3.103635	1.692522
6 0	-0.430126	3.016848	-0.309310
6 0	5.014572	1.677639	-0.683339
6 0	-1.611208	0.161792	1.403322
6 0	4.934757	-2.030330	-0.618135
6 0	3.286579	-3.350974	0.651949
6 0	0.968535	-3.195121	1.107841
6 0	1.141007	-3.610464	-0.269224
6 0	-1.145343	-2.069677	0.504476
6 0	-1.877851	1.404351	0.861942
6 0	-2.426009	2.084275	-1.517790
6 0	2.566339	-3.620527	-0.578794
6 0	3.012364	-3.062402	-1.777974
6 0	2.049544	-2.470199	-2.701790
6 0	4.200521	-2.227283	-1.783301
6 0	4.012507	2.296201	-1.541577
6 0	5.273006	0.345839	-1.200167
6 0	4.432363	0.156309	-2.375981
6 0	-0.956956	-2.476571	-0.795441
6 0	-0.992115	2.480348	-1.526282
6 0	-0.143470	2.089415	-2.546284
6 0	0.233607	-3.149168	-1.199419
6 0	0.697300	-2.544820	-2.431283
6 0	3.930492	-1.093642	-2.664404
6 0	2.587963	-1.212027	-3.179944
6 0	-0.198352	-1.485922	-2.807302
6 0	3.588440	1.332007	-2.551131
6 0	1.289586	2.241161	-2.454720

6 0 0.315918 -0.347949 -3.377537
6 0 2.247188 1.241043 -2.986537
6 0 1.745802 -0.099623 -3.367122
6 0 -2.299007 -1.119755 0.870732
6 0 -2.992246 1.661110 -0.100004
7 0 -3.819701 0.472077 -0.219369
6 0 -3.132255 -0.760134 -0.433330
6 0 -3.947063 2.803806 0.313323
6 0 -4.129808 -1.766246 -1.017026
6 0 -3.190320 -1.681020 2.007482
6 0 -4.673514 -1.385308 -2.259787
6 0 -5.582347 -2.211399 -2.914679
6 0 -5.952507 -3.434734 -2.346075
6 0 -5.405883 -3.820046 -1.123132
6 0 -4.495813 -2.991285 -0.457102
6 0 -4.290498 -0.930871 2.452037
6 0 -5.105507 -1.404248 3.479589
6 0 -4.838212 -2.633206 4.086690
6 0 -3.747428 -3.385965 3.652724
6 0 -2.929541 -2.914187 2.622461
6 0 -3.597259 3.879633 1.128566
6 0 -4.547713 4.872971 1.374060
6 0 -5.812776 4.768919 0.795156
6 0 -6.081763 3.660375 -0.006745
7 0 -5.170681 2.697622 -0.243768
8 0 -3.147501 2.044016 -2.512554
8 0 -2.111363 -0.475539 -1.530051
16 0 -0.791907 1.020766 -3.896016
6 0 -1.515217 -1.702430 -2.003359
1 0 -4.354388 -0.447823 -2.703768
1 0 -5.995318 -1.906482 -3.869118
1 0 -6.658492 -4.080422 -2.855300
1 0 -5.687059 -4.766278 -0.675872
1 0 -4.099798 -3.292816 0.499443
1 0 -4.513213 0.006927 1.963356
1 0 -5.952432 -0.810671 3.804422
1 0 -5.471075 -2.998203 4.887160
1 0 -3.526238 -4.341665 4.114034
1 0 -2.088572 -3.512450 2.299788
1 0 -4.299780 5.714104 2.010123
1 0 -6.571182 5.522753 0.961826
1 0 -7.051963 3.534614 -0.473444
1 0 -2.238028 -2.296170 -2.573233
1 0 -4.666383 0.656204 -0.760039
1 0 -2.613382 3.937759 1.568288
2 0 2.248786 -0.120811 0.080531

Single point energy = -3719.00197411 hartrees

(SPE, B3LYP/6-31G**//B3LYP/3-21G)

Single point energy for He = -2.90704885073 hartrees (B3LYP/6-31G**//B3LYP/3-21G)

Table 1. Crystal data and structure refinement for compound **2**.

Empirical formula	$C_{82}H_{16}N_2O_2S_5$
Formula weight	1221.27
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	P2(1)2(1)2(1)
Unit cell dimensions	a = 15.7008(9) Å α= 90°.
	b = 16.1532(9) Å β= 90°.
	c = 19.0307(11) Å γ = 90°.
Volume	4826.5(5) $Å^3$
Z	4
Density (calculated)	1.681 Mg/m^3
Absorption coefficient	0.308 mm^{-1}
F(000)	2472
Crystal size	0.18 x 0.11 x 0.09 mm^3
Theta range for data collection	1.65 to 25.00° .
Index ranges	-16<=h<=18, -19<=k<=18, -20<=l<=22
Reflections collected	25316
Independent reflections	8471 [R(int) = 0.0442]
Completeness to theta = 25.00-	99.9 %
Absorption correction	Empirical (SADABS)
Max. and min. transmission	0.9728 and 0.9466
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8471 / 0 / 824
Goodness-of-fit on F^2	1.036
Final R indices [I>2sigma(I)]	R1 = 0.0430, wR2 = 0.0666
R indices (all data)	R1 = 0.0618, wR2 = 0.0696
Absolute structure parameter	0.17(5)
Largest diff. peak and hole	0.408 and -0.318 e.$Å^{-3}$