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[3+2] Cycloaddition Reaction for the Stereoselective Synthesis of a New Spirooxindole Compound Grafted Imidazo[2,1-*b*]thiazole Scaffold: Crystal Structure and Computational Study

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Supplementary data

General

All chemical and reagents obtained from commercial suppliers (sigma Aldrich and TCI companies) were used without further purification. Reactions were monitored by thin layer chromatography using Merck silica gel 60 Kieselgel F254 TLC (Merck, Kenilworth, NJ, USA), and column chromatography was performed on silica gel 100–200 (40–63 μm, ASTM) from Merck using the indicated solvents. ^{1}H and ^{13}C-NMR spectra were recorded in $CDCl_3$ on a Jeol Spectrometer (Jeol, Tokyo, Japan) (400 MHz). The chemical shifts are reported in ppm. Infrared spectra were recorded on a Thermo Scientific Nicolet iS10 FT-IR spectrometer (Thermo Fisher Scientific, Waltham, MA, USA). Melting points (m.p.) were recorded on a Thomas-Hoover capillary melting point apparatus (Thomas-Hoover, Texas City, USA) and were not corrected. Elemental analyses were performed on Perkin-Elmer PE 2400 CHN Elemental Analyzer with autosampler, CHN mode. X-ray diffraction data were collected on a Rigaku Oxford Diffraction Supernova diffractometer and processed with CrysAlisPro software v. 1.171.41.93a (Rigaku Oxford Diffraction, Yarnton, UK, 2020) using Cu K_ radiation.

1. X-Ray single crystal measurements of **4**

The crystal of 4 was immersed in cryo-oil, mounted in a loop, and measured at a temperature of 120 K. The X-ray diffraction data was collected on a Rigaku Oxford Diffraction Supernova diffractometer using Cu Kα radiation. The *CrysAlisPro*[1] software package was used for cell refinement and data reduction. A multi-scan absorption correction (*CrysAlisPro*[1]) was applied to the intensities before structure solution. The structure was solved by intrinsic phasing (*SHELXT*[2]) method. Structural refinement was carried out using *SHELXL*[3] software with *SHELXLE*[4] graphical user interface. The NH hydrogen atom was located from the difference Fourier map and refined isotropically. All other hydrogen atoms were positioned geometrically and constrained to ride on their parent atoms, with C-H = 0.95-0.99 Å and U_{iso} = 1.2· U_{eq}(parent atom).

2. *Hirshfeld surface analysis*

   The topology analyses were performed using Crystal Explorer 17.5 program [5].

3. *Computational methods*

   All DFT calculations were performed using Gaussian 09 software package [6] utilizing B3LYP/6-31G(d,p) method. Natural charge calculations were performed using NBO 3.1 program as implemented in the Gaussian 09W package [7]. The self-consistent reaction filed (SCRF) method [8, 9] was used to model the solvent effects when calculated the optimized geometries in solution. Then the NMR chemical shifts for the protons and carbons were computed using GIAO method [10]. Also, the UV-Vis electronic spectra of the compound were calculated using TD-DFT method.

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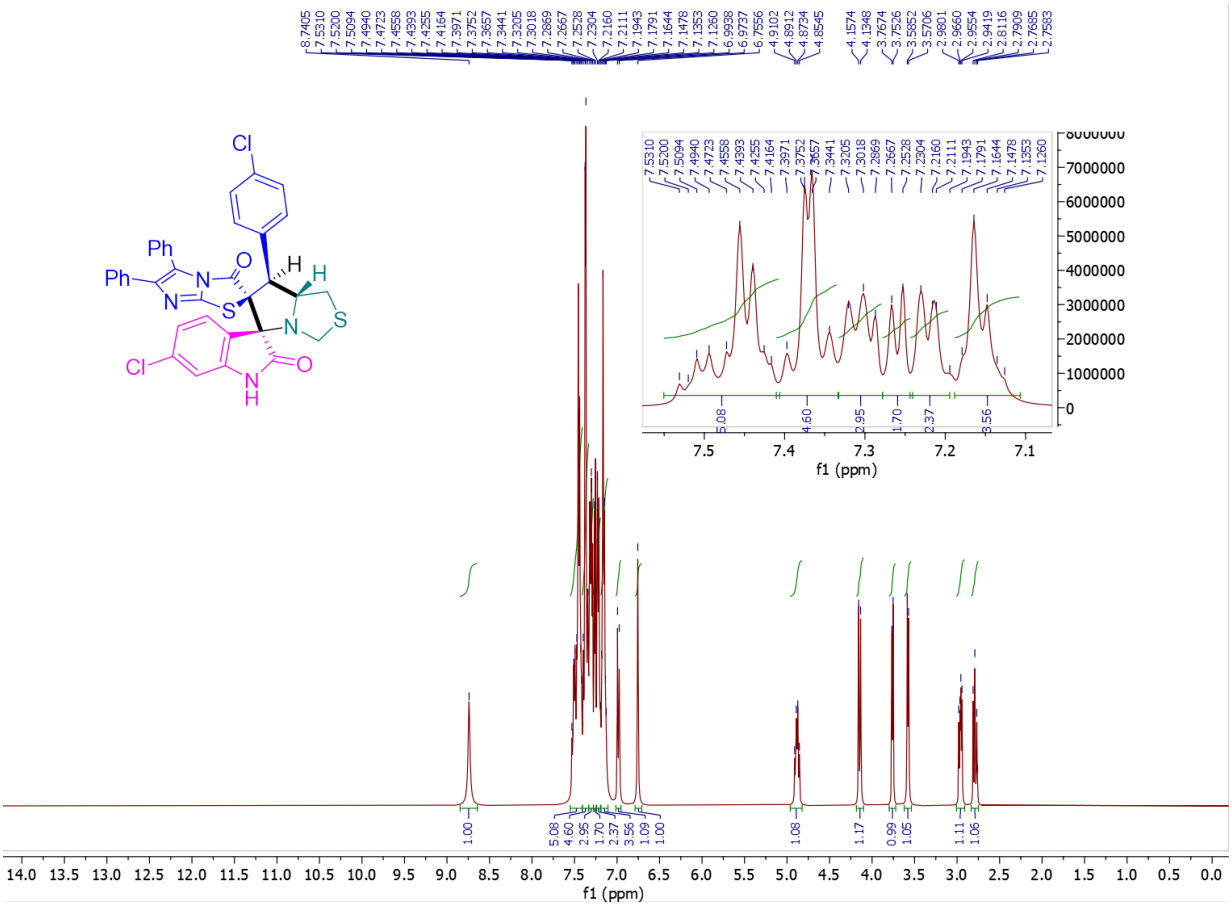

Figure S1: [1]HNMR spectrum for the synthesized compound **4.**

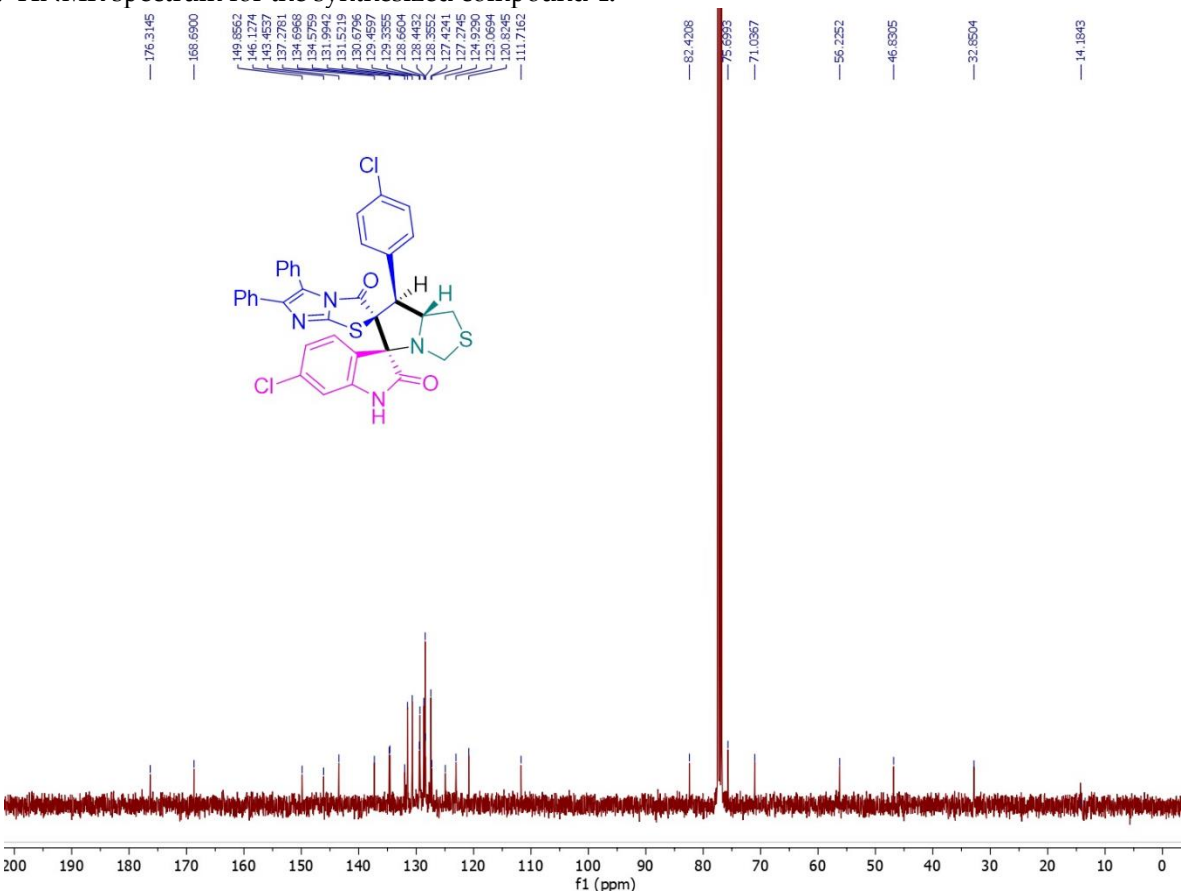

Figure S2: ^{13}C-NMR spectrum for the synthesized compound **4.**

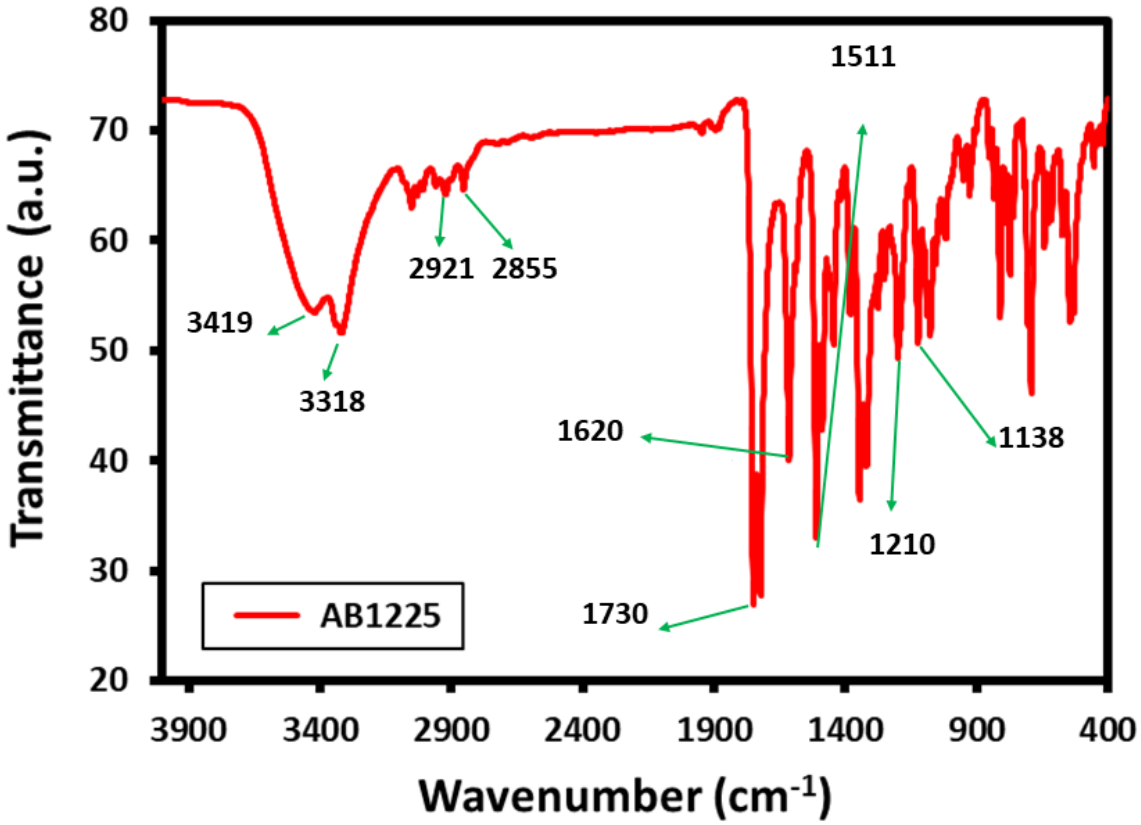

Figure S3: IR spectrum for the synthesized compound **4.**

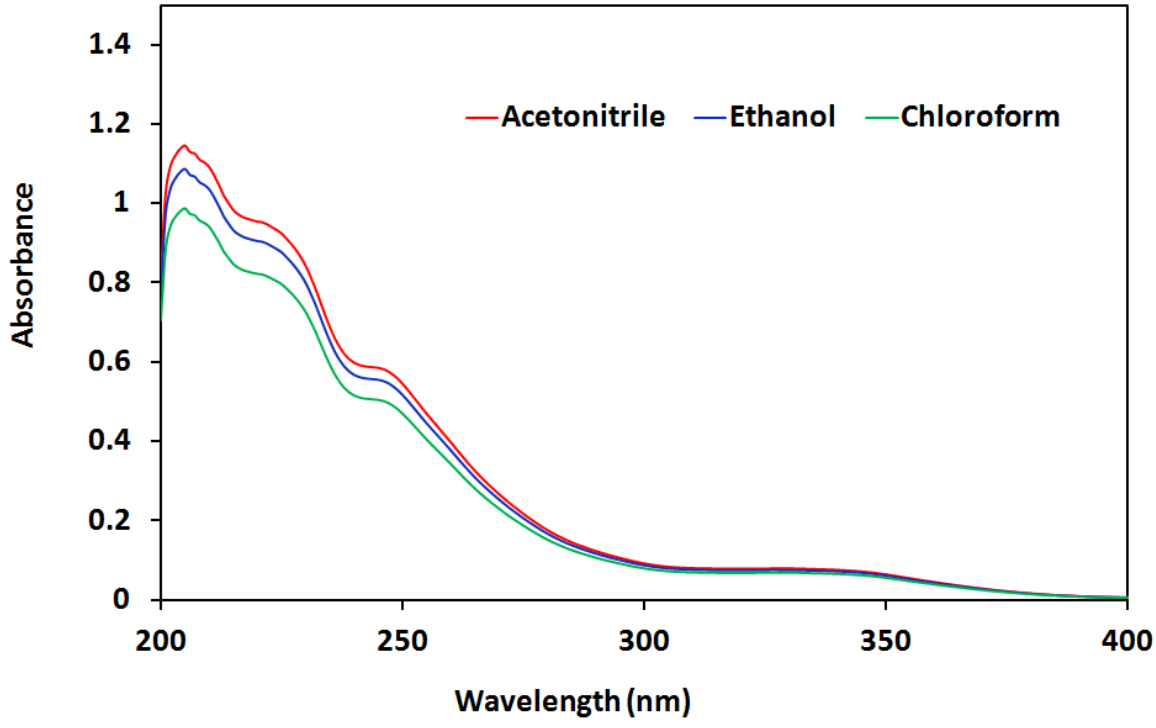

Figure S4. Electronic spectra of **4** in different solvents.

Table S1 The calculated geometric parameters of **4**[a].

Parameter	Calc	Exp	Parameter	Calc	Exp
R(1-15)	1.759	1.744	A(1-15-13)	119.536	119.010
R(2-36)	1.756	1.737	A(1-15-16)	119.503	119.406
R(3-25)	1.855	1.846	A(2-36-34)	118.381	118.035
R(3-28)	1.861	1.828	A(2-36-37)	119.252	118.996
R(4-42)	1.864	1.840	A(25-3-28)	92.683	93.412
R(4-68)	1.755	1.741	A(3-25-23)	104.037	105.048
R(5-32)	1.219	1.222	A(3-25-26)	109.100	110.742
R(6-43)	1.212	1.198	A(3-25-27)	109.299	110.735
R(7-23)	1.459	1.473	A(3-28-7)	103.680	101.817
R(7-28)	1.449	1.449	A(3-28-29)	110.163	111.425
R(7-31)	1.448	1.467	A(3-28-30)	108.282	111.420
R(8-32)	1.374	1.352	A(42-4-68)	90.568	91.240
R(8-33)	1.401	1.404	A(4-42-21)	116.502	117.525
R(9-43)	1.388	1.387	A(4-42-31)	111.865	111.523
R(9-44)	1.415	1.406	A(4-42-43)	106.197	106.660
R(9-68)	1.383	1.376	A(4-68-9)	113.963	113.691
R(10-56)	1.407	1.417	A(4-68-10)	132.688	132.977
R(10-68)	1.292	1.294	A(5-32-8)	125.693	126.909
R(11-13)	1.394	1.393	A(5-32-31)	126.949	125.358
R(11-20)	1.404	1.402	A(6-43-9)	125.593	125.532
R(13-15)	1.394	1.378	A(6-43-42)	123.710	124.091
R(15-16)	1.392	1.372	A(23-7-28)	110.687	108.879
R(16-18)	1.395	1.393	A(23-7-31)	109.413	105.299
R(18-20)	1.400	1.392	A(7-23-21)	103.040	99.914
R(20-21)	1.514	1.508	A(7-23-24)	111.893	109.793
R(21-23)	1.545	1.538	A(7-23-25)	104.176	106.551
R(21-42)	1.589	1.589	A(28-7-31)	122.133	119.579
R(23-25)	1.527	1.527	A(7-28-29)	111.156	111.402
R(31-32)	1.575	1.563	A(7-28-30)	113.667	111.394
R(31-41)	1.520	1.515	A(7-31-32)	113.226	111.328
R(31-42)	1.605	1.579	A(7-31-41)	114.108	112.983
R(33-34)	1.388	1.386	A(7-31-42)	100.680	99.778
R(33-41)	1.404	1.394	A(32-8-33)	112.329	110.914
R(34-36)	1.399	1.387	A(32-8-69)	122.063	120.788
R(36-37)	1.394	1.387	A(8-32-31)	107.298	107.704
R(37-39)	1.401	1.396	A(33-8-69)	125.536	124.252
R(39-41)	1.389	1.389	A(8-33-34)	127.807	126.482
R(42-43)	1.555	1.541	A(8-33-41)	109.701	110.423
R(44-45)	1.476	1.478	A(43-9-44)	136.903	135.910

R(44-56)	1.384	1.368	A(43-9-68)	116.543	116.199
R(45-46)	1.404	1.391	A(9-43-42)	110.697	110.329
R(45-54)	1.405	1.397	A(44-9-68)	106.554	106.907
R(46-48)	1.394	1.387	A(9-44-45)	123.421	121.021
R(48-50)	1.396	1.382	A(9-44-56)	103.934	104.113
R(50-52)	1.396	1.391	A(9-68-10)	113.314	113.315
R(52-54)	1.394	1.386	A(56-10-68)	104.952	104.172
R(56-57)	1.472	1.473	A(10-56-44)	111.239	111.492
R(57-58)	1.406	1.393	A(10-56-57)	118.623	119.586
R(57-66)	1.406	1.398	A(13-11-20)	120.929	120.096
R(58-60)	1.393	1.393	A(11-13-15)	119.380	119.556
R(60-62)	1.396	1.387	A(11-20-18)	118.341	118.740
R(62-64)	1.396	1.380	A(11-20-21)	122.264	123.061
R(64-66)	1.393	1.393	A(13-15-16)	120.961	121.560
			A(15-16-18)	118.944	119.005
			A(16-18-20)	121.444	121.017
			A(18-20-21)	119.379	118.019
			A(20-21-22)	108.077	106.155
			A(20-21-23)	114.756	114.635
			A(20-21-42)	117.851	119.927
			A(22-21-23)	106.683	106.133
			A(22-21-42)	104.319	106.141
			A(23-21-42)	104.188	102.867
			A(21-23-25)	118.397	120.211
			A(21-42-31)	104.591	104.526
			A(21-42-43)	108.508	108.912
			A(32-31-41)	101.481	101.185
			A(32-31-42)	109.789	110.047
			A(41-31-42)	118.021	121.756
			A(31-41-33)	109.132	108.024
			A(31-41-39)	131.335	132.659
			A(31-42-43)	109.018	107.304
			A(34-33-41)	122.455	123.088
			A(33-34-36)	116.699	116.093
			A(33-41-39)	119.439	119.116
			A(34-36-37)	122.365	122.959
			A(36-37-39)	119.479	119.404
			A(37-39-41)	119.517	119.313
			A(45-44-56)	132.363	134.390
			A(44-45-46)	121.789	120.868
			A(44-45-54)	119.414	119.744

			A(44-56-57)	130.133	128.921
			A(46-45-54)	118.794	119.323
			A(45-46-48)	120.461	120.020
			A(45-54-52)	120.592	120.406
			A(45-54-55)	119.222	119.793
			A(46-48-50)	120.299	120.479
			A(48-50-52)	119.716	119.925
			A(50-52-54)	120.137	119.821
			A(56-57-58)	122.376	121.396
			A(56-57-66)	118.976	119.465
			A(58-57-66)	118.627	119.138
			A(57-58-60)	120.504	120.478
			A(57-66-64)	120.634	119.995
			A(57-66-67)	118.735	120.009
			A(58-60-62)	120.407	119.890
			A(60-62-64)	119.524	120.072
			A(62-64-66)	120.293	120.409

[a]Atom numbering refer to **Figure 1 (main text)**

Table S2 The calculated natural charges of **4**[a].

Atom	Charge	Atom	Charge	Atom	Charge
Cl1	-0.0040	H24	0.2484	H47	0.2485
Cl2	0.0075	C25	-0.5828	C48	-0.2355
S3	0.1929	H26	0.2646	H49	0.2438
S4	0.3759	H27	0.2531	C50	-0.2303
O5	-0.6032	C28	-0.3843	H51	0.2420
O6	-0.5536	H29	0.2562	C52	-0.2386
N7	-0.5147	H30	0.2230	H53	0.2438
N8	-0.6231	C31	0.0659	C54	-0.2099
N9	-0.4406	C32	0.7016	H55	0.2513
N10	-0.4840	C33	0.1890	C56	0.1493
C11	-0.2221	C34	-0.2878	C57	-0.0824
H12	0.2671	H35	0.2625	C58	-0.2124
C13	-0.2407	C36	-0.0201	H59	0.2468
H14	0.2593	C37	-0.2685	C60	-0.2369
C15	-0.0398	H38	0.2641	H61	0.2408
C16	-0.2452	C39	-0.1912	C62	-0.2328
H17	0.2581	H40	0.2744	H63	0.2395
C18	-0.2105	C41	-0.0965	C64	-0.2365
H19	0.2432	C42	-0.2321	H65	0.2409
C20	-0.0519	C43	0.7303	C66	-0.2045
C21	-0.2688	C44	0.1261	H67	0.2570
H22	0.2814	C45	-0.0980	C68	0.2450

C23	-0.0596	C46	-0.1988	H69	0.4481

[a]Atom numbering refer to **Figure 1 (main text)**

Table S3 The calculated chemical shifts of **4**[a].

Atom	Calc	Exp.	Atom	Calc.	Exp.
C 11	119.340	129.46	H 12	8.548	7.455
C 13	117.341	129.34	H 14	7.723	7.439
C 15	130.884	134.70	H 17	7.532	7.375
C 16	116.241	124.93	H 19	7.500	7.365
C 18	120.290	131.52	H 22	4.171	4.157
C 20	126.323	134.58	H 24	4.959	4.891
C 21	51.339	46.83	H 26	2.845	2.790
C 23	65.688	56.23	H 27	3.073	2.966
C 25	30.787	14.18	H 29	3.522	3.585
C 28	41.121	32.85	H 30	3.696	3.767
C 31	69.558	71.04	H 35	6.941	6.755
C 32	162.241	176.31	H 38	6.909	6.993
C 33	131.664	137.28	H 40	7.252	7.230
C 34	99.108	82.42	H 47	8.055	7.531
C 36	134.008	143.45	H 49	7.987	7.509
C 37	110.797	111.72	H 51	7.885	7.494
C 39	116.117	120.82	H 53	7.752	7.425
C 41	110.492	111.72	H 55	7.384	7.344
C 42	85.062	82.42	H 59	7.210	7.126
C 43	158.635	168.69	H 61	7.319	7.164
C 44	117.063	128.44	H 63	7.489	7.211
C 45	116.993	128.36	H 65	7.647	7.286
C 46	120.519	130.68	H 67	8.147	7.301
C 48	115.748	127.27	H 69	6.870	8.740
C 50	116.703	127.42			
C 52	116.130	123.07			
C 54	118.637	128.66			
C 56	137.224	146.13			
C 57	121.113	131.99			
C 58	115.043	111.72			
C 60	114.795	127.27			
C 62	115.283	111.50			
C 64	115.887	111.72			
C 66	116.577	127.27			
C 68	142.660	149.86			

[a]Atom numbering refer to **Figure 1 (main text).**