Electronic Supplementary Material (ESI) for ChemComm.

Electronic Supplementary Information

$AgNO_2$-Mediated Cleavage of the N−N Bond of Sulfonylhydrazones and Oxygen Transfer: Access to Fulleroisoxazolines via Radical Cyclization with [60]Fullerene

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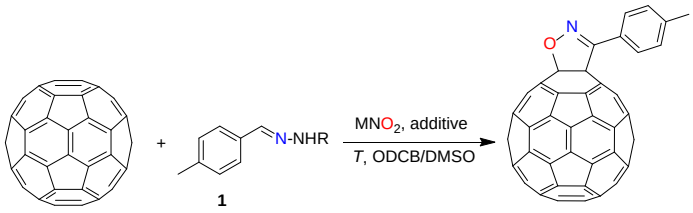
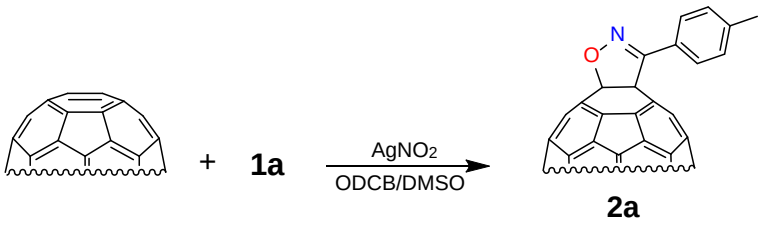
Table S1: Screening of the Reaction Conditions[a]



entry	**1**	MNO_2	additive	molar ratio[b]	T (^oC)[c]	yield (%)[d]
1	**1a**	$AgNO_2$	–	1:2:2:0	25	12 (84)
2	**1a**	$AgNO_2$	–	1:2:2:0	120	27 (73)
3	**1b**	$AgNO_2$	–	1:2:2:0	120	23 (70)
4	**1c**	$AgNO_2$	–	1:2:2:0	120	23 (62)
5	**1d**	$AgNO_2$	–	1:2:2:0	120	NR
6	**1a**	$AgNO_2$	–	1:2:1:0	120	12 (76)
7	**1a**	$AgNO_2$	–	1:2:3:0	120	26 (87)
8	**1a**	$NaNO_2$	–	1:2:2:0	120	15 (25)
9	**1a**	KNO_2	–	1:2:2:0	120	11 (34)
10	**1a**	$AgNO_2$	Cs_2CO_3	1:2:2:2	120	28 (82)
11	**1a**	$AgNO_2$	CF_3CO_2H	1:2:2:2	120	37 (73)
12	**1a**	$AgNO_2$	CF_3CO_2H	1:2:2:2	25	46 (90)
13	**1a**	$AgNO_2$	HOAc	1:2:2:2	25	18 (71)
14	**1a**	$AgNO_2$	CF_3SO_3H	1:2:2:2	25	36 (85)
15[e]	**1a**	$AgNO_2$	CF_3CO_2H	1:2:2:2	25	47 (88)
16	**1a**	$AgNO_2$	CF_3CO_2H	1:2:2:3	25	40 (80)
17[f]	**1a**	$AgNO_2$	CF_3CO_2H	1:2:2:2	25	41 (91)
18	**1a**	$NaNO_2$	CF_3CO_2H	1:2:2:2	25	9 (73)
19	**1a**	$NaNO_2$	CF_3CO_2H	1:2:2:2	40	21 (79)
20[g]	**1a**	$NaNO_2$	CF_3CO_2H	1:2:2:2	25	15 (72)
21[h]	**1a**	$NaNO_2$	CF_3CO_2H	1:2:2:2	25	28 (73)

[a]All reactions were carried out with C_{60}/**1**/MNO_2/additive in a designated molar ratio in co-solvent of anhydrous ODCB (5 mL) and DMSO (1 mL) for 2 h under air unless specified otherwise. [b]Molar ratio refers to C_{60}/**1**/MNO_2/additive. [c]Oil temperature. [d]Yields in parentheses were based on consumed C_{60}. [e]The reaction was carried out for 3 h. [f]The reaction was carried out under nitrogen atmosphere. [g]2 equiv of AgOAc was added. [h]2 equiv of $AgOOCCF_3$ was added.

Radical-Trapping Experiments:



without CF_3CO_2H: add 1,1-diphenylethylene (2 equiv) at 120 °C	5%
standard conditions: add 1,1-diphenylethylene (2 equiv) at 25 °C	4%
without CF_3CO_2H: add TEMPO (2 equiv) at 120 °C	trace
standard conditions: add TEMPO (2 equiv) at 25 °C	trace

General Procedure for the Synthesis of Products 2a, 2e–z, 2aa and 2bb from $AgNO_2$-Mediated Reaction of C_{60} with Substrates 1a, 1e–z, 1aa and 1bb: To a 15-mL tube equipped with a magnetic stirrer was charged with C_{60} (36.0 mg, 0.05 mmol), $AgNO_2$ (15.4 mg, 0.10 mmol), CF_3CO_2H (7.4 μL, 0.10 mmol) and **1a** (**1e–z** and **1aa**, 0.10 mmol; for **1bb**, 0.05 mmol). After they were dissolved in co-solvent of anhydrous ODCB (5 mL) and DMSO (1 mL) by sonication, and then the sealed mixture was stirred under room temperature for a desired time (monitored by TLC). The reaction mixture was filtered through a silica gel plug to remove any insoluble material. After the solvent was evaporated in vacuo, the residue was separated on a silica gel column with CS_2 as the eluent to give unreacted C_{60}, then with CS_2/DCM as the eluent to give product **2a** (**2e–v**, **2z**, **2aa** and **2bb**; for **2w–y**, with CS_2/DCM/EtOAc as the eluent). Among these compounds **2a**, **2e**, **2k**, **2n**, **2q**, **2r** and

2t are known compounds.

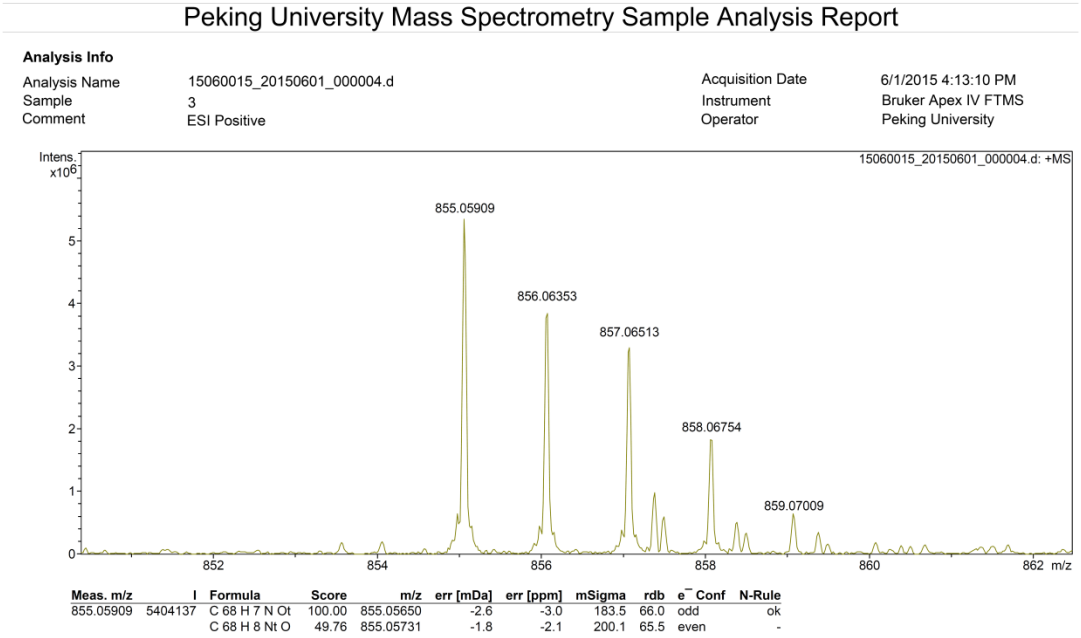
Researches on Intermediate Experiment: A mixture of C_{60} (36.0 mg, 0.05 mmol), $AgNO_2$ (15.4 mg, 0.10 mmol), CF_3CO_2H (7.4 μL, 0.10 mmol) and 4-methylbenzaldehyde oxime (13.5 mg, 0.10 mmol) in co-solvent of anhydrous ODCB (5 mL) and DMSO (1 mL) by sonication, and then the sealed mixture was stirred under room temperature for 2 h. The reaction mixture was filtered through a silica gel plug to remove any insoluble material. After the solvent was evaporated in vacuo, the residue was separated on a silica gel column with carbon disulfide as the eluent to give unreacted C_{60} (34.1 mg, 94%), then with CS_2/DCM as the eluent to give product **2a** (2.0 mg, 5%).

Carben or Metal Carben Experiments: A mixture of C_{60} (36.0 mg, 0.05 mmol), $AgNO_2$ (15.4 mg, 0.10 mmol), **1a** or **1r** (0.05 mmol), CF_3CO_2H (7.4 μL, 0.10 mmol) and ethyl 2-diazoacetate (0.05 mmol) in co-solvent of anhydrous ODCB (5 mL) and DMSO (1 mL) by sonication, and then the sealed mixture was stirred under room temperature for 2 h. The reaction mixture was filtered through a silica gel plug to remove any insoluble material. After the solvent was evaporated in vacuo, the residue was separated on a silica gel column with carbon disulfide as the eluent to give unreacted C_{60} (23.2 mg, 64% for **1a**; 22.1 mg, 62% for **1r**), then with CS_2/DCM as the eluent to give product **2a** (10.0 mg, 23%) or **2b** (6.1 mg, 15%), and other byproducts.

[$H_2{}^{18}O$]-Labeling Experiment: A mixture of C_{60} (36.0 mg, 0.05 mmol), $AgNO_2$ (15.4 mg, 0.10 mmol), **1a** (30.4 mg, 0.10 mmol), CF_3CO_2H (7.4 μL, 0.10 mmol) and $H_2{}^{18}O$ (0.10 mmol) in co-solvent of anhydrous ODCB (5 mL) and DMSO (1 mL) by sonication, and then the sealed mixture was stirred under room temperature for 2 h. The reaction mixture was filtered through a silica gel plug to remove any insoluble material. After the solvent was evaporated in vacuo, the residue was separated on a silica gel column with carbon disulfide as the eluent to give unreacted C_{60} (21.5 mg,

60%), then with CS_2/DCM as the eluent to give product **2a** (13.7 mg, 32%).

[$Na^{15}N^{18}O_2$]-Labeling Experiment: A mixture of C_{60} (36.0 mg, 0.05 mmol), ^{15}N,^{18}O-labeled $NaNO_2$ (90% ^{18}O, 95% ^{15}N specified by Sigma-Aldrich) (7.4 mg, 0.10 mmol), **1a** (30.4 mg, 0.10 mmol) and CF_3CO_2H (7.4 μL, 0.10 mmol) in co-solvent of anhydrous ODCB (5 mL) and DMSO (1 mL) by sonication, and then the sealed mixture was stirred at 40 ^oC for 2 h. The reaction mixture was filtered through a silica gel plug to remove any insoluble material. After the solvent was evaporated in vacuo, the residue was separated on a silica gel column with carbon disulfide as the eluent to give unreacted C_{60} (26.7 mg, 74%), then with CS_2/DCM as the eluent to give product **2a** (9.4 mg, 22%). The ^{18}O was determined in product **2a** by HRMS. HRMS *m/z* (ESI) calcd. for $C_{68}H_7N^{18}O$ $[M]^+$ 885.0565, found 885.0591.



Radical-Trapping Experiments: A mixture of C_{60} (36.0 mg, 0.05 mmol), $AgNO_2$ (15.4 mg, 0.10 mmol), **1a** (30.4 mg, 0.10 mmol), CF_3CO_2H (7.4 μL, 0.10 mmol) [and without CF_3CO_2H] and DPE or TEMPO (0.10 mmol) in co-solvent of anhydrous ODCB (5 mL) and DMSO (1 mL) by sonication, and then the sealed mixture was stirred under room temperature [120 ^oC in the absence of CF_3CO_2H] for 2 h. The

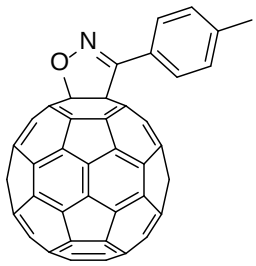
reaction mixture was filtered through a silica gel plug to remove any insoluble material. After the solvent was evaporated in vacuo, the residue was separated on a silica gel column with carbon disulfide as the eluent to give the following results:

For DPE, unreacted C_{60}: 32.6 mg, 92%, (33.6 mg, 93%, in the absence of CF_3CO_2H), then with CS_2/DCM = 5/1 as the eluent to give trace product **2a** (1.6 mg, 4%) (2.3 mg, 5%, in the absence of CF_3CO_2H).

For TEMPO, unreacted C_{60}: 34.8 mg, 97%, (35.1 mg, 98%, in the absence of CF_3CO_2H), then with CS_2/DCM = 5/1 as the eluent to give trace product **2a**.

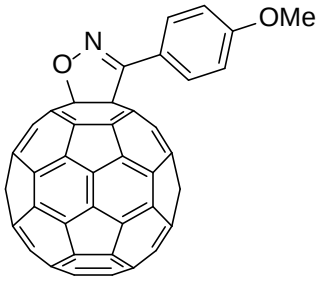
These results confirmed that DPE or TEMPO could severely retarded or almost completely suppressed the formation of **2a**, respectively.

Spectral data for Compounds:



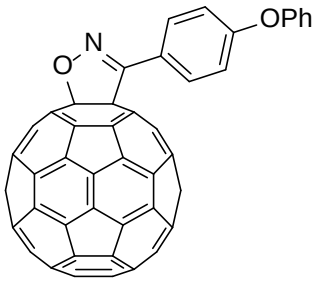
2a

Spectral data of **2a**: [1] ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.18 (d, J = 8.0 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 153.49 (1C), 147.67 (1C), 147.16 (1C), 146.29, 146.17, 146.14, 145.89, 145.84, 145.79, 145.50, 145.32, 145.12, 145.05, 144.73, 144.70, 144.41, 144.30, 144.00, 142.88, 142.74 (4C), 142.37 (4C), 142.22, 142.19, 141.98, 141.60, 140.87 (1C), 140.23, 140.17, 136.90, 136.63, 129.74, 129.72, 126.02 (1C), 103.95 (1C, sp^3-*C* of C_{60}), 79.21 (1C, sp^3-*C* of C_{60}), 21.54 (1C).



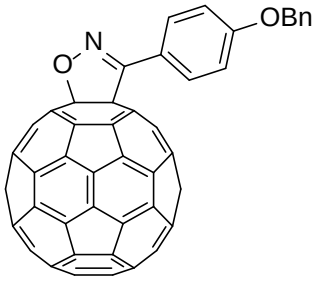
2e

Spectral data of **2e**:[2] ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.08 (d, J = 8.8 Hz, 2H), 6.97 (d, J = 8.8 Hz, 2H), 3.85 (s, 3H).



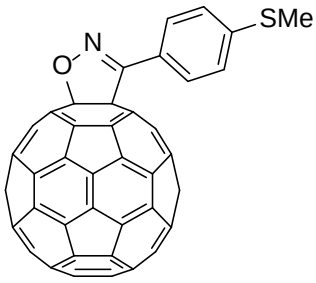
2f

Spectral data of **2f**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.12 (d, J = 8.4 Hz, 2H), 7.36−7.32 (m, 2H), 7.15−7.11 (m, 1H), 7.07−7.02 (m, 4H). ^{13}C NMR (150 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 159.92 (1C), 155.43 (1C), 152.21 (1C), 147.46 (1C), 146.98 (1C), 146.12, 145.98, 145.96, 145.71, 145.66, 145.55, 145.35, 145.12, 144.95, 144.86, 144.52, 144.50, 144.39, 144.16, 143.81, 142.73, 142.59 (4C), 142.22 (4C), 142.07, 142.01, 141.84, 141.44, 140.06, 140.04, 136.79, 136.35, 130.25, 129.79, 128.08 (1C), 124.14 (1C), 119.77, 118.00, 103.84 (1C, sp^3-*C* of C_{60}), 80.98 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm-1 (KBr) 1585, 1504, 1486, 1428, 1301, 1243, 1199, 1167, 1105, 981, 906, 873, 843, 770, 749, 727, 689, 635, 566, 527. λ_{max}/nm ($CHCl_3$) 256, 316, 427, 680. MALDI-TOF MS m/z calcd for $C_{73}H_{10}NO_2$ $[M+H]^+$ 932.0706, found 932.0707.



2g

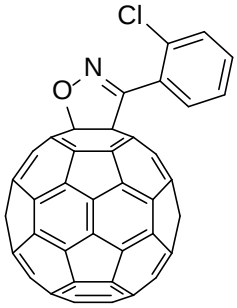
Spectral data of **2g**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.11 (d, J = 8.8 Hz, 2H), 7.41−7.31 (m, 5H), 7.08 (d, J = 8.8 Hz, 2H), 5.11 (s, 2H). ^{13}C NMR (100 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 160.38 (1C), 152.56 (1C), 147.56 (1C), 147.06 (1C), 146.20, 146.07, 146.04, 145.79, 145.74, 145.71, 145.42, 145.22, 145.03, 144.95, 144.75, 144.61, 144.57, 144.24, 143.90, 142.81, 142.66 (4C), 142.30 (4C), 142.16, 142.11, 141.92, 141.52, 140.13, 140.11, 136.83, 136.46, 136.13 (1C), 130.22, 128.54, 128.01 (1C), 127.20 (1C), 121.45, 115.23, 103.80 (1C, sp^3-*C* of C_{60}), 79.01 (1C, sp^3-*C* of C_{60}), 69.94 (1C). FT-IR ν/cm^{-1} (KBr) 2925, 2864, 1604, 1509, 1453, 1425, 1378, 1311, 1245, 1175, 1105, 1000, 974, 900, 866, 827, 770, 748, 728, 691, 651, 565, 527. λ_{max}/nm ($CHCl_3$) 256, 316, 424, 678. MALDI-TOF MS m/z calcd for $C_{74}H_{12}NO_2$ $[M+H]^+$ 946.0863, found 946.0853.



2h

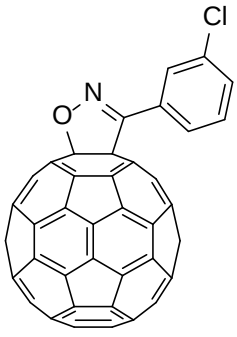
Spectral data of **2h**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.08 (d, J = 8.4 Hz, 2H), 7.31 (d, J = 8.4, 2H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 152.97 (1C), 147.67 (1C), 147.17 (1C), 146.30, 146.18, 146.15, 146.08, 145.90, 145.84, 145.71, 145.53, 145.30, 145.14, 145.05, 144.66, 144.60, 144.40, 144.32, 143.98, 142.90, 142.76 (4C), 142.62 (1C), 142.38 (4C), 142.19, 141.96, 141.60, 140.23, 140.20, 136.96, 136.60, 128.98, 125.95, 125.09 (1C), 104.09 (1C, sp^3-*C* of C_{60}), 78.96 (1C, sp^3-*C* of C_{60}), 15.12 (1C). FT-IR

ν/cm^{-1} (KBr) 2913, 1592, 1493, 1431, 1399, 1307, 1188, 1097, 1014, 981, 907, 865, 808, 770, 725, 607, 565, 527. λ_{max}/nm ($CHCl_3$) 255, 317, 425, 679. MALDI-TOF MS m/z calcd for $C_{68}H_8NOS$ $[M+H]^+$ 886.0321, found 886.0325.



2i

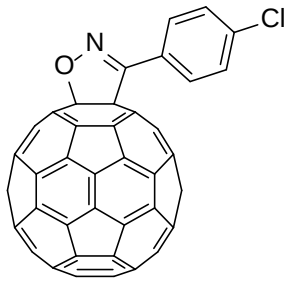
Spectral data of **2i**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 7.69 (dd, J = 7.6, 2.0 Hz, 1H), 7.58 (dd, J = 8.0, 1.2 Hz, 1H), 7.50−7.39 (m, 2H). ^{13}C NMR (150 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 150.98 (1C), 147.35 (1C), 146.80 (1C), 145.92, 145.90, 145.83, 145.57, 145.46, 145.19, 144.98, 144.93, 144.76, 144.71, 144.42, 143.93, 143.75, 143.67, 143.56, 142.58, 142.40, 142.36, 142.05, 141.99, 141.90, 141.82, 141.75, 141.32, 140.19, 139.77, 136.51, 136.34, 134.78 (1C), 131.23 (1C), 130.98 (1C), 130.25 (1C), 127.30 (1C), 126.47 (1C), 102.77 (1C, sp^3-*C* of C_{60}), 80.62 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 2921, 1621, 1587, 1512, 1474, 1429, 1302, 1186, 1108, 1080, 1036, 971, 897, 854, 772, 758, 749, 725, 693, 648, 603, 572, 527. λ_{max}/nm ($CHCl_3$) 255, 316, 425, 682. MALDI-TOF MS m/z calcd for $C_{67}H_5ClNO$ $[M+H]^+$ 874.0054, found 874.0049.



2j

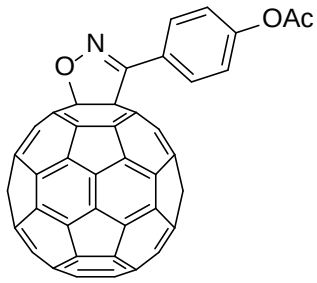
Spectral data of **2j**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.16 (t, J = 2.0 Hz, 1H), 8.07 (dt, J = 7.2, 2.0 Hz, 1H), 7.53−7.44 (m, 2H). ^{13}C NMR (100 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 151.97 (1C), 147.52 (1C),

147.03 (1C), 146.17, 146.03 (4C), 145.76, 145.71, 145.45, 145.33, 145.11, 145.01, 144.91, 144.44, 144.16, 143.98, 143.91, 143.82, 142.76, 142.63 (4C), 142.24 (4C), 142.04 (4C), 141.82, 141.51, 140.20, 140.10, 136.92, 136.39, 135.23 (1C), 130.62 (1C), 130.53(1C), 130.04 (1C), 128.81 (1C), 126.44 (1C), 104.27 (1C, sp^3-*C* of C_{60}), 78.48 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 2920, 1592, 1562, 1426, 1302, 1267, 1180, 1168, 1098, 972, 881, 866, 786, 764, 748, 723, 695, 685, 652, 604, 573, 554, 527. λ_{max}/nm ($CHCl_3$) 255, 317, 425, 679. MALDI-TOF MS m/z calcd for $C_{67}H_5ClNO$ $[M+H]^+$ 874.0054, found 874.0052.



2k

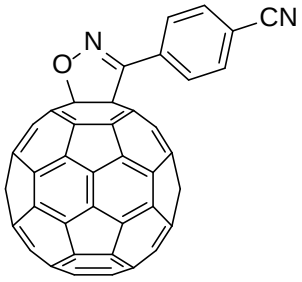
Spectral data of **2k**:[1] ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.13 (d, J = 8.4 Hz, 2H), 7.49 (d, J = 8.4 Hz, 2H).



2l

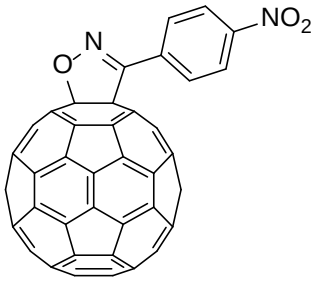
Spectral data of **2l**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.20 (d, J = 8.8 Hz, 2H), 7.25 (d, J = 8.8 Hz, 2H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3/CS_2$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 168.55 (1C), 152.80 (1C), 152.40 (1C), 147.72 (1C), 147.21 (1C), 146.34, 146.22, 146.19, 145.94, 145.89, 145.65, 145.60, 145.32, 145.18, 145.09, 144.66, 144.41, 144.33, 144.24, 144.02, 142.93, 142.79 (4C), 142.40 (4C), 142.23 (4C), 141.98, 141.66, 140.34, 140.23, 137.03, 136.65, 130.00, 126.48 (1C), 122.27, 104.29 (1C, sp^3-*C* of C_{60}), 78.88 (1C, sp^3-*C* of C_{60}), 21.07 (1C). FT-IR ν/cm^{-1} (KBr) 2948, 1765, 1595, 1505, 1429, 1362, 1309, 1188, 1166, 1015, 981,

909, 866, 845, 770, 680, 565, 527. λ_{max}/nm ($CHCl_3$) 255, 316, 424, 678. MALDI-TOF MS m/z calcd for $C_{69}H_8NO_3$ $[M+H]^+$ 898.0499, found 898.0489.



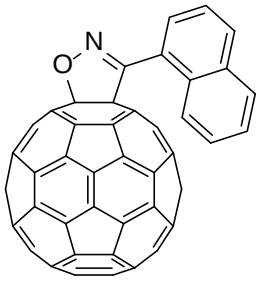
2m

Spectral data of **2m**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.37 (d, J = 8.8 Hz, 2H), 7.80 (d, J = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3/CS_2$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 152.17 (1C), 147.68 (1C), 147.20 (1C), 146.33, 146.18 (4C), 145.93, 145.87, 145.59, 145.19, 145.17 (4C), 145.05, 144.44, 144.29, 143.89, 143.64, 143.60, 142.92, 142.80, 142.78, 142.36 (4C), 142.12(4C), 141.84, 141.61, 140.33, 140.27, 137.16, 136.51, 133.33 (1C), 132.61, 129.20, 117.64 (1C), 114.42 (1C), 104.92 (1C, sp^3-*C* of C_{60}), 78.13 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 2227, 1582, 1510, 1425, 1303, 1181, 1106, 982, 909, 864, 843, 830, 768, 610, 586, 566, 552, 526. λ_{max}/nm ($CHCl_3$) 255, 315, 424, 678. MALDI-TOF MS m/z calcd for $C_{68}H_5N_2O$ $[M+H]^+$ 865.0396, found 865.0391.



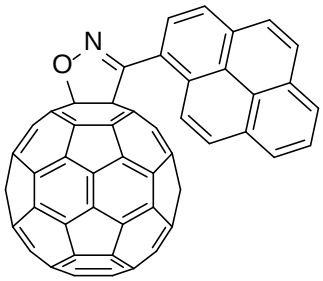
2n

Spectral data of **2n**:[1] ^{1}H NMR (400 MHz, DMSO-d_6/CS_2) δ 8.44 (d, J = 9.2 Hz, 2H), 8.37 (d, J = 9.2 Hz, 2H).



2o

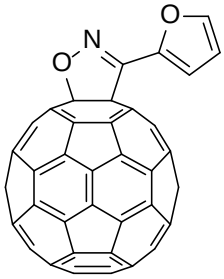
Spectral data of **2o**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.54 (d, J = 8.8 Hz, 1H), 8.00 (d, J = 8.2 Hz, 1H), 7.93−7.89 (m, 2H), 8.54 (dt, J = 6.8, 1.2 Hz, 1H), 7.66−7.56 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3/CS_2$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 152.30 (1C), 147.59 (1C), 147.03 (1C), 146.14 (4C), 146.08, 145.80 145.71, 145.45, 145.39, 145.15, 145.00, 144.94, 144.64, 144.32, 144.13, 143.93, 143.87, 142.78, 142.64, 142.58, 142.27, 142.25, 142.07, 142.04, 141.98, 141.52, 140.40, 140.05, 136.54, 136.42, 133.81 (1C), 132.09 (1C), 130.69 (1C), 128.46 (1C), 127.70 (1C), 127.51 (1C), 126.72 (1C), 125.39 (1C), 125.33 (1C), 124.73 (1C), 102.94 (1C, sp^3-*C* of C_{60}), 81.47 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 2911, 1506, 1427, 1295, 1250, 1188, 1124, 1005, 972, 928, 896, 860, 796, 773, 657, 603, 573, 554, 526. λ_{max}/nm ($CHCl_3$) 256, 317, 424, 680. MALDI-TOF MS m/z calcd for $C_{71}H_8NO$ $[M+H]^+$ 890.0600, found 890.0607.



2p

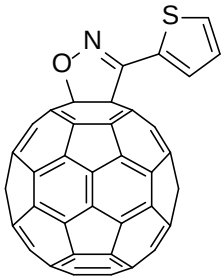
Spectral data of **2p**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.78 (d, J = 9.2 Hz, 1H), 8.38 (d, J = 7.6 Hz, 1H), 8.29−8.23 (m, 4H), 8.16 (d, J = 8.8 Hz, 1H), 8.09 (d, J = 8.8 Hz, 1H), 8.06 (t, J = 8.0 Hz, 1H). ^{13}C NMR (150 MHz, $CS_2/CDCl_3$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 152.41 (1C), 147.38 (1C), 146.82 (1C), 145.97, 145.95, 145.89, 145.61, 145.50, 145.28, 145.22, 144.98, 144.81, 144.74, 144.53, 144.21, 143.97, 143.91, 143.68, 142.60, 142.46, 142.38, 142.11, 142.09,

141.93, 141.92, 141.77, 141.32, 140.20, 139.90, 136.39, 136.32, 132.32 (1C), 130.86 (1C), 130.56 (1C), 130.51 (1C), 129.10 (1C), 128.92 (1C), 127.30 (1C), 127.02 (1C), 126.42 (1C), 126.40 (1C), 125.91 (1C), 128.83 (1C), 124.79 (1C), 124.31 (1C), 124.22 (1C), 121.88 (1C), 102.83 (1C, sp^3-*C* of C_{60}), 81.69 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 2926, 1596, 1512, 1430, 1329, 1268, 1179, 1125, 1074, 1025, 972, 900, 865, 846, 820, 769, 716, 696, 554, 526. λ_{max}/nm ($CHCl_3$) 255, 335, 424, 680. MALDI-TOF MS m/z calcd for $C_{77}H_{10}NO$ $[M+H]^+$ 864.0757, found 864.0758.



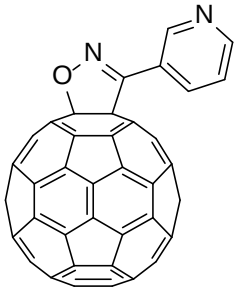
2q

Spectral data of **2q**:[3] ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 7.63 (d, J = 1.6 Hz, 1H), 7.36 (d, J = 3.6 Hz, 1H), 6.63 (dd, J = 3.6, 1.6 Hz, 1H).



2r

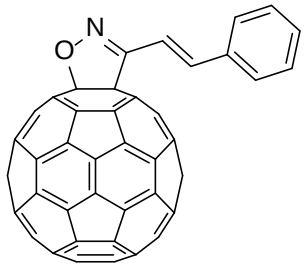
Spectral data of **2r**: [3] ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 8.03 (d, J = 3.6 Hz, 1H), 7.54 (dd, J = 5.2, 0.8 Hz, 1H), 6.63 (dd, J = 5.2, 3.6 Hz, 1H).



2s

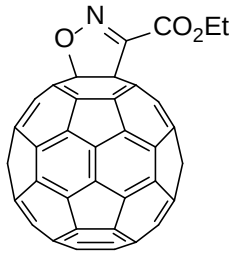
Spectral data of **2s**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 9.44 (d, J = 2.4 Hz, 1H), 8.76

(dd, J = 4.8, 1.2 Hz, 1H), 8.49 (dt, J = 8.0, 2.0 Hz, 1H), 7.47 (dd, J = 8.0, 4.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3/CS_2$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 151.23 (1C), 150.99 (1C), 148.96 (1C), 147.48 (1C), 146.98 (1C), 146.12, 145.99 (4C), 145.72, 145.66, 145.42, 145.13, 145.01, 144.96, 144.84, 144.33, 144.09, 143.74, 143.59 (4C), 142.71, 142.58, 142.57, 142.17 (4C), 141.96 (4C), 141.72, 141.45, 140.21, 140.04, 136.99, 136.37, 135.37 (1C), 125.19 (1C), 123.45 (1C), 104.28 (1C, sp^3-*C* of C_{60}), 78.30 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 2924, 1710, 1583, 1428, 1411, 1304, 1187, 1107, 1063, 1020, 982, 906, 865, 805, 770, 703, 656, 604, 571, 554, 527. λ_{max}/nm ($CHCl_3$) 255, 317, 424, 678. MALDI-TOF MS m/z calcd for $C_{66}H_5N_2O$ $[M+H]^+$ 841.0396, found 841.0393.



2t

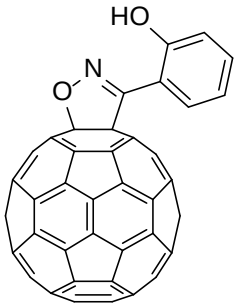
Spectral data of **2t**:[4] ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 7.96 (d, J = 16.4 Hz, 1H), 7.53−7.51 (m, 2H), 7.37−7.29 (m, 3H), 7.25−7.23 (m, 1H).



2u

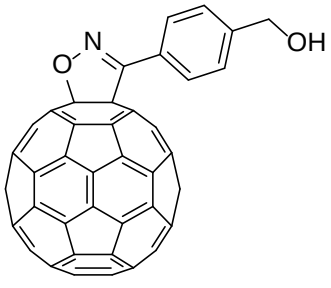
Spectral data of **2u**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 4.55 (q, J = 6.8 Hz, 2H), 1.51 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3/CS_2$ with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 159.40 (1C), 147.59 (1C), 146.99 (1C), 146.72, 146.17, 146.15 (3C), 146.14, 145.78 (4C), 145.51, 145.09, 144.99, 144.97, 144.35, 143.99, 143.97, 143.42, 142.68 (4C), 142.63, 142.36, 142.28 (4C), 142.19, 141.97, 141.64, 141.58, 140.07, 140.01, 136.77, 136.29, 105.88 (1C, sp^3-*C* of C_{60}), 75.34 (1C, sp^3-*C* of C_{60}), 62.76 (1C), 14.21 (1C). FT-IR ν/cm^{-1} (KBr) 2980, 2932, 1744, 1720,

1591, 1430, 1370, 1328, 1292, 1174, 1141, 1097, 1065, 1016, 981, 909, 877, 821, 807, 769, 747, 725, 670, 606, 564, 554, 526. λ_{max}/nm ($CHCl_3$) 254, 316, 424, 676. MALDI-TOF MS m/z calcd for $C_{64}H_6NO_3$ $[M+H]^+$ 836.0342, found 836.0338.



2v

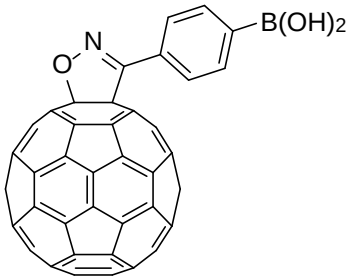
Spectral data of **2v**: ^{1}H NMR (400 MHz, $CDCl_3/CS_2$) δ 10.22 (s, 1H), 8.60 (dd, J = 8.4, 1.2 Hz, 1H), 7.41−7.36 (m, 1H), 7.18 (dd, J = 8.4, 1.2 Hz, 1H), 6.95−6.91 (m, 1H). ^{13}C NMR (100 MHz, CD_3OD/CS_2 with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 158.23 (1C), 152.32 (1C), 147.86 (1C), 147.40 (1C), 146.55, 146.44, 146.37, 146.11, 146.08, 145.99, 145.85, 145.48, 145.36, 145.25, 144.80, 144.73, 144.59 (4C), 144.20, 143.13, 143.02 (4C), 142.64, 142.61, 142.51, 142.47, 142.10, 141.75, 140.51, 140.17, 137.33, 136.78, 132.35 (1C), 128.47 (1C), 119.78 (1C), 118.36 (1C), 114.23 (1C), 102.85 (1C, sp^3-*C* of C_{60}), 78.96 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 3156, 2920, 1711, 1614, 1585, 1510, 1489, 1456, 1425, 1301, 1251, 1221, 1157, 993, 872, 822, 769, 745, 680, 666, 652, 604, 573, 554, 526. λ_{max}/nm ($CHCl_3$) 255, 319, 424, 676. MALDI-TOF MS m/z calcd for $C_{67}H_6NO_2$ $[M+H]^+$ 856.0393, found 856.0391.



2w

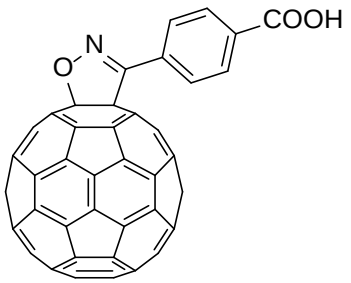
Spectral data of **2w**: ^{1}H NMR (400 MHz, CD_3OD/CS_2) δ 8.04 (d, J = 8.4 Hz, 2H), 7.41 (d, J = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, CD_3OD/CS_2 with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 152.35 (1C), 147.35 (1C), 146.85 (1C),

146.01, 145.88 (4C), 145.61, 145.57, 145.54, 145.27, 145.05, 144.84, 144.75, 144.50, 144.43, 144.27, 144.06, 143.74, 142.64, 142.49 (4C), 142.13 (5C), 141.99 (4C), 141.80, 141.37, 139.95 (4C), 136.65, 136.26, 128.52, 127.37 (1C), 126.87, 103.69 (1C, sp^3-*C* of C_{60}), 78.82 (1C, sp^3-*C* of C_{60}), 63.62 (1C). FT-IR ν/cm^{-1} (KBr) 3588, 2920, 1702, 1588, 1510, 1428, 1411, 1301, 1182, 1106, 1045, 1015, 981, 865, 769, 750, 727, 602, 566, 554, 527. λ_{max}/nm ($CHCl_3$) 256, 315, 425, 675. MALDI-TOF MS m/z calcd for $C_{68}H_8NO_2$ $[M+H]^+$ 870.0550, found 856.0543.



2x

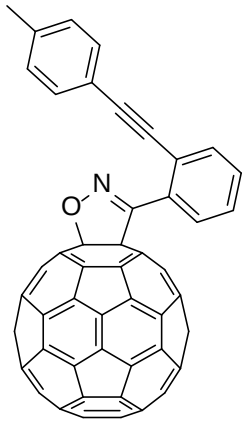
Spectral data of **2x**: ^{1}H NMR (400 MHz, CD_3OD/CS_2) δ 8.09 (d, J = 8.0 Hz, 2H), 7.71 (d, J = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, CD_3OD/CS_2 with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 152.35 (1C), 147.44 (1C), 146.94 (1C), 146.10, 145.96 (4C), 145.68 (4C), 145.64 (4C), 145.35, 145.13, 144.93, 144.84, 144.56, 144.50, 144.32, 144.13, 143.81, 142.72, 142.57 (4C), 142.20 (4C), 142.08, 142.03, 141.87, 141.46, 140.04 (4C), 136.76, 136.34, 134.14 (1C), 129.89 (1C), 127.66, 103.97 (1C, sp^3-*C* of C_{60}), 78.74 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 3455, 2950, 2920, 1607, 1511, 1400, 1341, 1302, 1108, 1017, 981, 864, 829, 769, 747, 724, 659, 565, 554, 527. λ_{max}/nm ($CHCl_3$) 255, 316, 424, 680. MALDI-TOF MS m/z calcd for $C_{67}H_7BNO_3$ $[M+H]^+$ 884.0514, found 884.0511.



2y

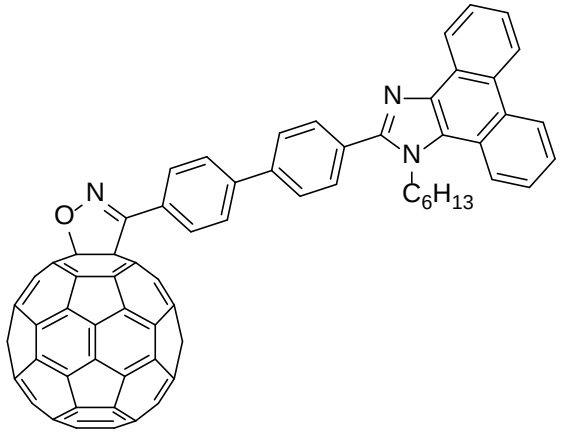
Spectral data of **2y**: ^{1}H NMR (400 MHz, DMSO-d_6/CS_2) δ 12.80 (s, 1H), 8.19 (d, J =

7.6 Hz, 2H), 8.07 (d, J = 7.6 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6/CS_2 with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 166.56 (1C), 152.56 (1C), 147.85 (1C), 147.33 (1C), 146.49, 146.38, 146.34, 146.08, 146.05, 145.90, 145.79, 145.50, 145.34, 145.24, 144.81, 144.65, 144.50, 144.45, 144.21, 143.11, 142.97 (4C), 142.58 (4C), 142.44 (4C), 142.23, 141.88, 140.53, 140.43, 137.22, 136.79, 133.72 (1C), 132.65 (1C), 130.57, 128.79, 104.54 (1C, sp^3-*C* of C_{60}), 78.99 (1C, sp^3-*C* of C_{60}). FT-IR ν/cm^{-1} (KBr) 2961, 2926, 2854, 1729, 1690, 1604, 1454, 1420, 1289, 1262, 1224, 1075, 971, 861, 803, 708, 668, 605, 553, 525. λ_{max}/nm ($CHCl_3$) 255, 314, 425, 682. MALDI-TOF MS m/z calcd for $C_{68}H_6NO_3$ $[M+H]^+$ 884.0342, found 884.0338.



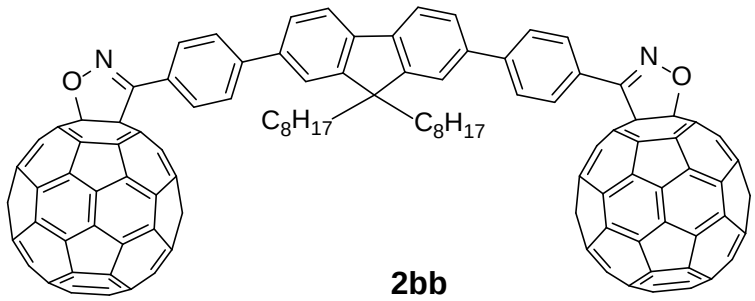
2z

Spectral data of **2z**: ^{1}H NMR (400 MHz, $CDCl_3$/CS_2) δ 7.73−7.69 (m, 2H), 7.69−7.44 (m, 4H), 7.15 (d, J = 8.0 Hz, 2H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$/CS_2 with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 153.06 (1C), 147.51(1C), 146.90 (1C), 146.00 (4C), 145.97, 145.68, 145.58, 145.27, 145.24, 145.06, 144.86, 144.85, 144.58, 144.16, 143.99, 143.82 (4C), 142.65, 142.50, 142.47, 142.14, 142.10, 141.94, 141.92, 141.84, 141.46, 140.31, 139.83, 138.71 (1C), 136.61, 136.43, 132.57 (1C), 131.81, 130.10 (1C), 130.05 (1C), 129.49 (1C), 129.03, 127.85 (1C), 124.71 (1C), 119.66 (1C), 102.85 (1C, sp^3-*C* of C_{60}), 95.07 (1C), 87.19 (1C), 81.13 (1C, sp^3-*C* of C_{60}), 21.57 (1C). FT-IR ν/cm^{-1} (KBr) 2913, 2214, 1612, 1589, 1509, 1300, 1180, 1111, 1029, 971, 897, 859, 849, 813, 771, 759, 677, 648, 604, 573, 554, 526. λ_{max}/nm ($CHCl_3$) 255, 319, 424, 675. MALDI-TOF MS m/z calcd for $C_{76}H_{12}NO$ $[M+H]^+$ 954.0913, found 954.0904.



2aa

Spectral data of **2aa**: ^{1}H NMR (400 MHz, DMSO-d_6/CS_2) δ 8.80 (d, J = 8.0 Hz, 1H), 8.64 (d, J = 8.0 Hz, 2H), 8.36 (d, J = 8.0 Hz, 2H), 8.27 (d, J = 8.0 Hz, 1H), 7.93−7.89 (m, 6H), 7.69−7.60 (m, 4H), 4.73 (t, 2H), 2.01 (bs, 2H), 1.30 (bs, 6H), 0.91 (bs, 3H). ^{13}C NMR (100 MHz, DMSO-d_6/CS_2 with $Cr(acac)_3$ as relaxation reagent, all 2C unless indicated) δ 152.51 (1C), 151.65 (1C), 147.85 (1C), 147.35 (1C), 146.52, 146.38, 146.35, 146.10, 146.05 (4C), 145.79, 145.53, 145.35, 145.26, 144.94, 144.88, 144.75, 144.54, 144.23, 143.14, 143.00 (4C), 142.61 (4C), 142.51, 142.44, 142.28, 141.88, 140.51, 140.46, 138.68 (1C), 137.23, 136.78, 131.24 (1C), 130.93, 129.74, 129.25 (1C), 128.64 (1C), 128.17 (1C), 128.00, 127.86 (1C), 127.55, 127.41 (1C), 127.03 (1C), 126.52 (1C), 125.75, 125.01 (1C), 124.84 (1C), 123.74 (1C), 123.28, 121.06, 104.40 (1C, sp^3-*C* of C_{60}), 79.20 (1C, sp^3-*C* of C_{60}), 47.36 (1C), 31.82 (1C), 30.88 (1C), 26.66 (1C), 23.33 (1C), 14.67 (1C). FT-IR ν/cm^{-1} (KBr) 2923, 1608, 1471, 1449, 1424, 1359, 1305, 1185, 1156, 1090, 1005, 982, 865, 824, 751, 723, 606, 572, 527. λ_{max}/nm ($CHCl_3$) 258, 320, 362, 424, 688. MALDI-TOF MS m/z calcd for $C_{94}H_{30}N_3O$ $[M+H]^+$ 1216.2383, found 1216.2371.



2bb

Spectral data of **2bb**: ^{1}H NMR (400 MHz, $CDCl_3$/CS_2) δ 8.28 (d, J = 8.8 Hz, 4H),

7.79 (d, J = 8.8 Hz, 4H), 7.75 (d, J = 8.8 Hz, 2H), 7.60 (d, J = 8.8 Hz, 2H), 7.56 (s, 2H), 2.04−2.00 (m, 4H), 1.15−1.03 (m, 20H), 0.76 (t, J = 6.8 Hz, 6H), 0.67 (bs, 4H). ^{13}C NMR (100 MHz, $CDCl_3/CS_2$ with $Cr(acac)_3$ as relaxation reagent, all 4C unless indicated) δ 152.79 (2C), 151.63 (2C), 147.54 (2C), 147.06 (2C), 146.20, 146.06 (8C), 145.79, 145.73, 145.69, 145.43, 145.20, 145.02, 144.94, 144.57 (8C), 144.33, 144.22, 143.88, 143.50 (2C), 142.80, 142.66 (8C), 142.28 (8C), 142.15, 142.09, 141.89, 141.52, 140.36 (2C), 140.13 (8C), 138.80 (2C), 136.89, 136.48, 129.14, 127.58 (2C), 127.49, 126.12 (2C), 121.24 (2C), 120.31 (2C), 104.63 (2C, sp^3-*C* of C_{60}), 78.92 (2C, sp^3-*C* of C_{60}), 55.09 (1C), 40.44 (2C), 31.82 (2C), 30.15 (2C), 29.34 (2C), 29.31 (2C), 23.94 (2C), 22.77 (2C), 14.17 (2C). FT-IR ν/cm^{-1} (KBr) 2919, 2848, 1710, 1602, 1463, 1429, 1300, 1181, 1104, 981, 863, 812, 769, 647, 604, 571, 553, 526. λ_{max}/nm ($CHCl_3$) 255, 339, 422, 682. MALDI-TOF MS m/z calcd for $C_{163}H_{49}N_2O_2$ $[M+H]^+$ 2065.3789, found 2065. 3781.

References

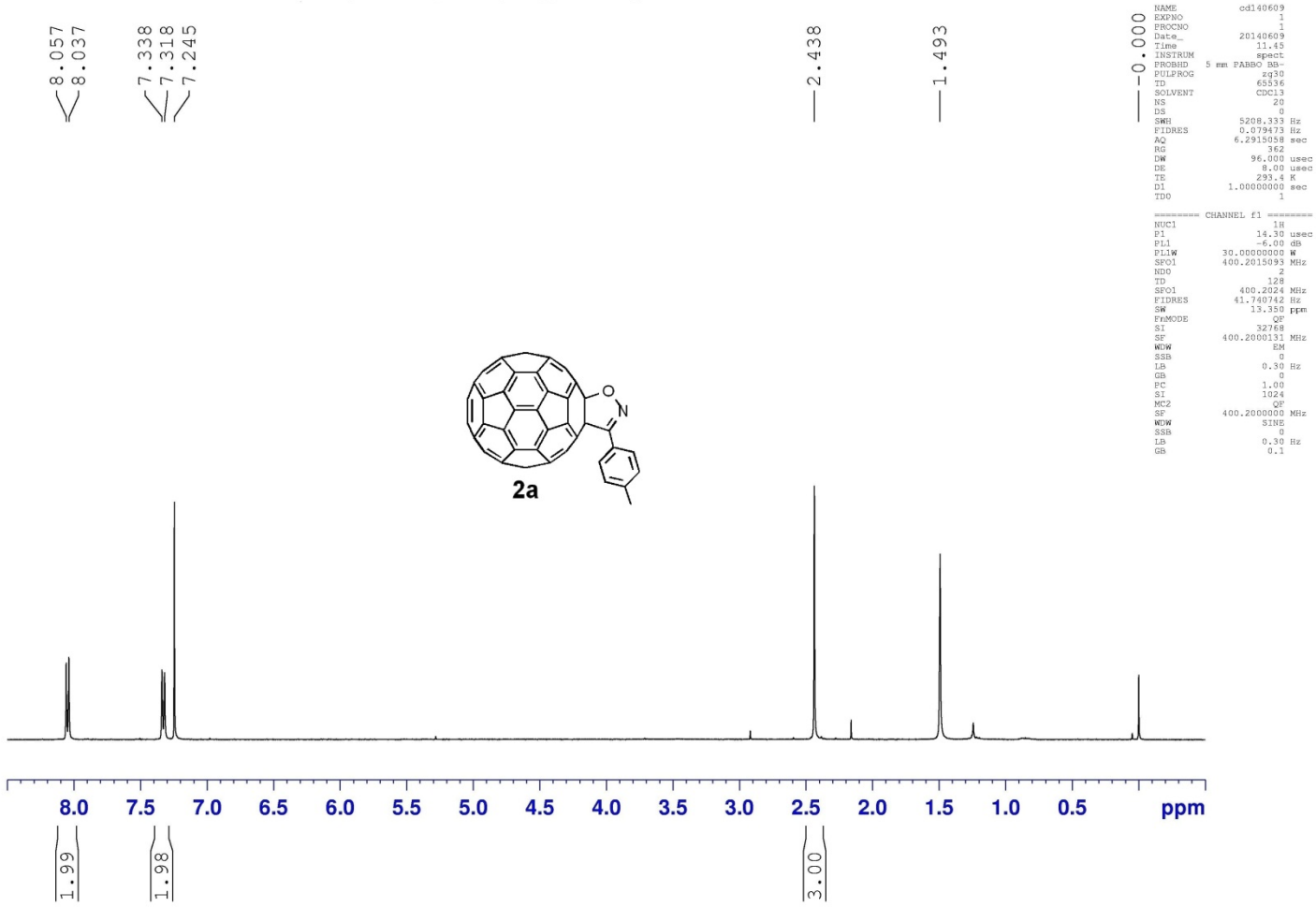
[1] H.-T. Yang, X.-J. Ruan, C.-B. Miao and X.-Q. Sun, *Tetrahedron Lett.* 2010, **51**, 6056.

[2] M. S. Meier, *Tetrahedron* 1996, 5**2**, 5043.

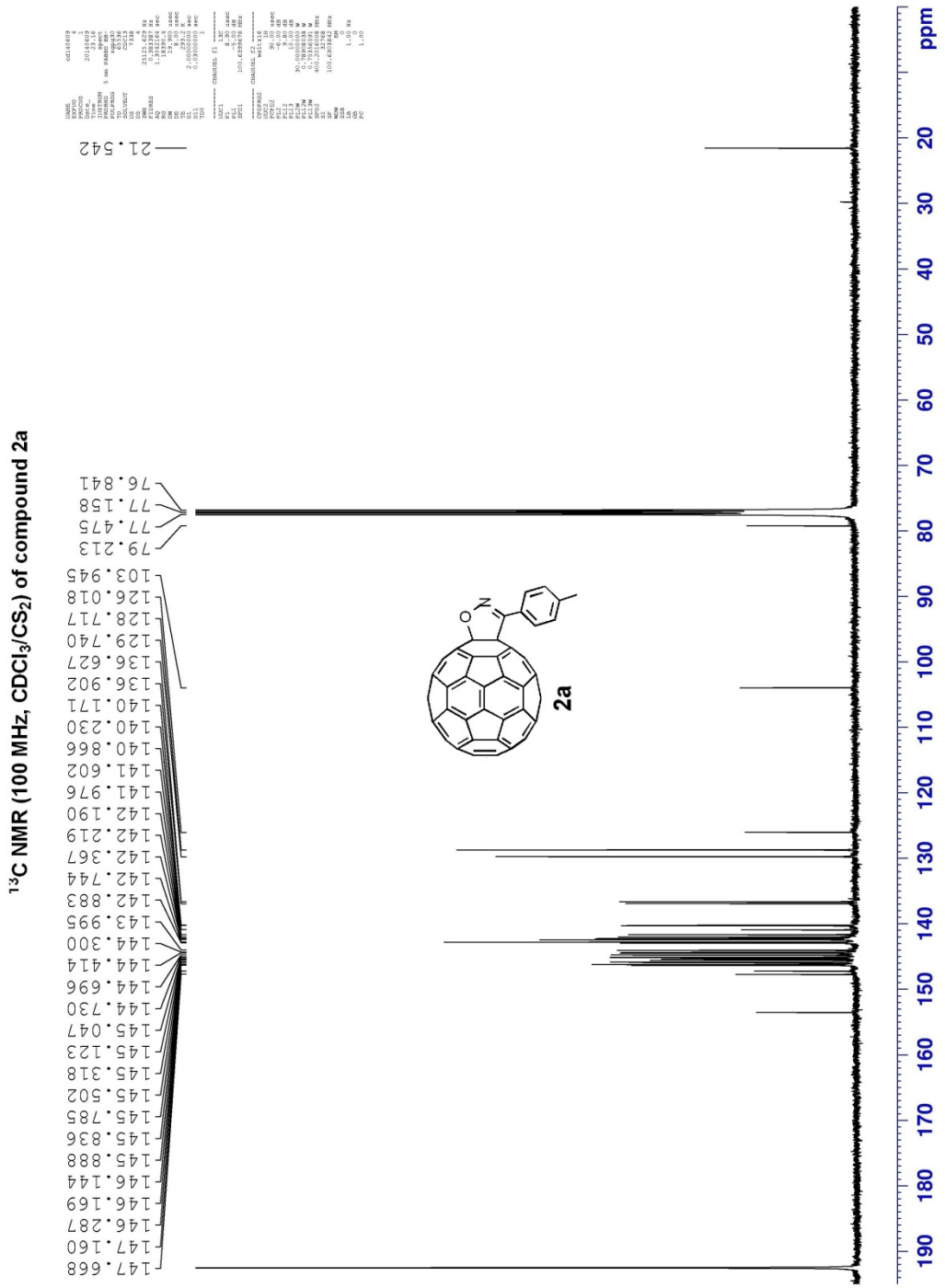
[3] F. Langa, P. de la Cruz, E. Espíldora, A. González-Cortés, A. de la Hoz and V. López-Arza, *J. Org. Chem.* 2000, **65**, 8675.

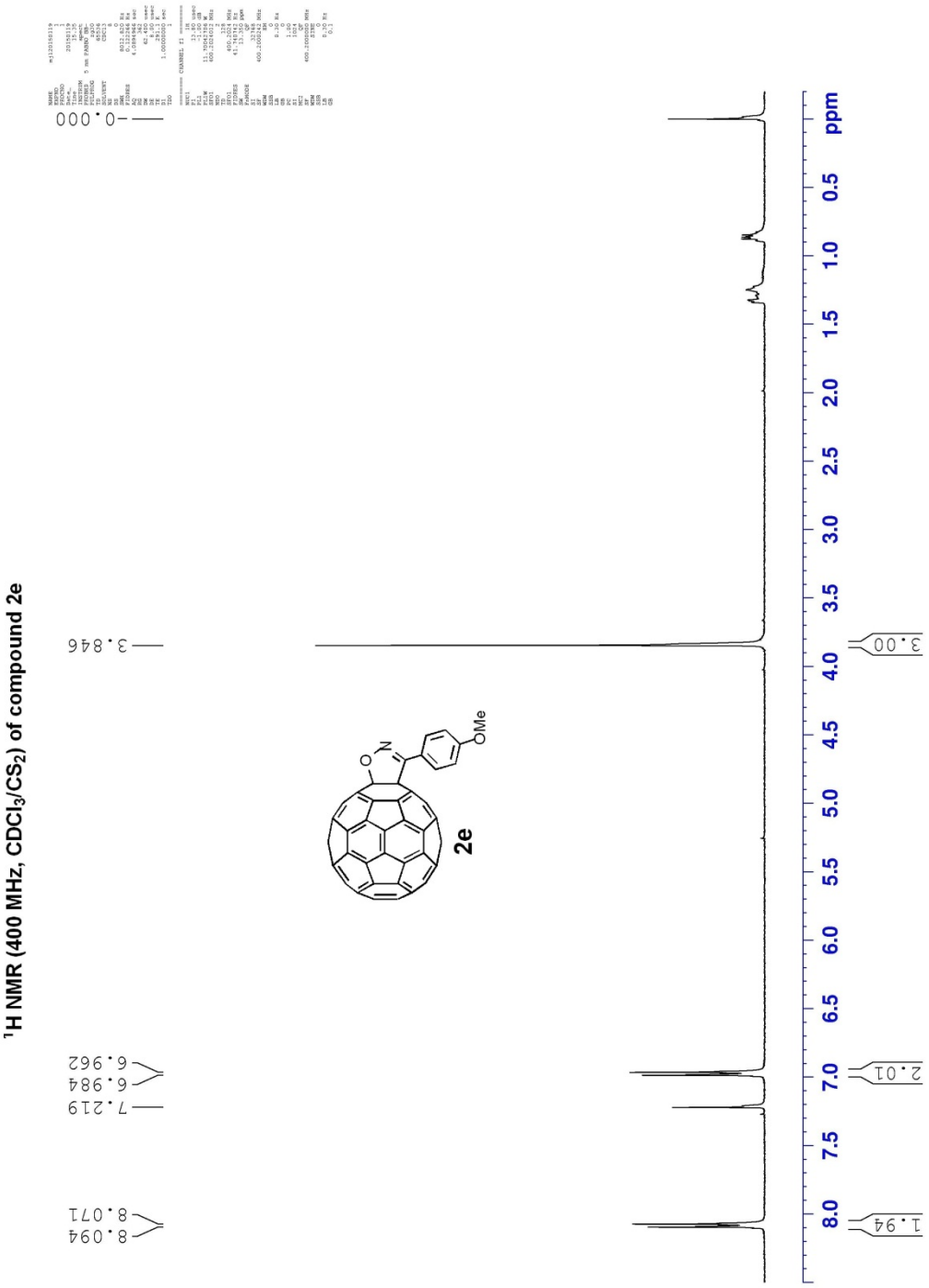
[4] H. Irngartinger, P. W. Fettel, T. Escher, P. Tiniefeld, S. Nord and M. Sauer, *Eur. J. Org. Chem*. 2000, 455.

^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2a

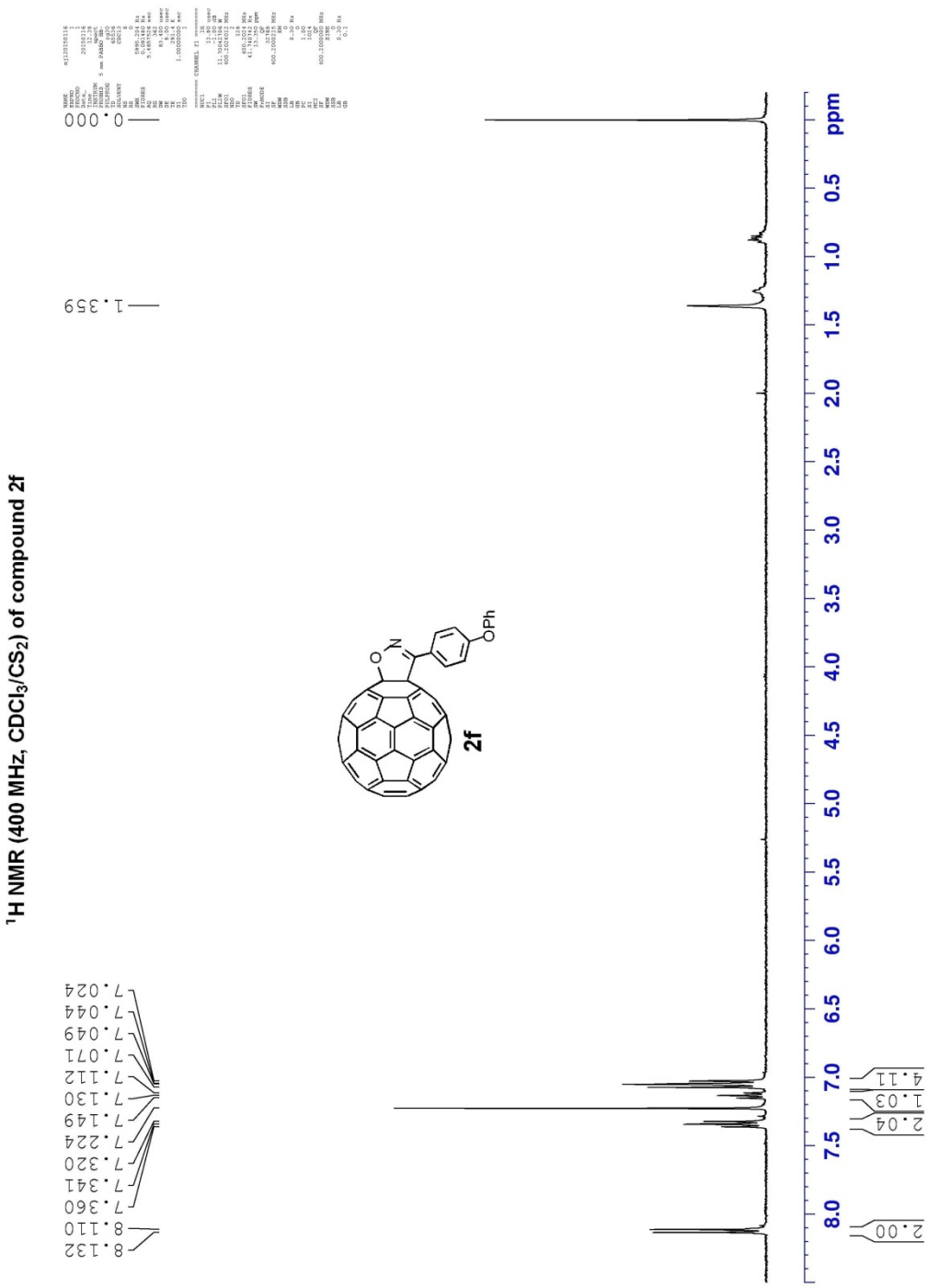


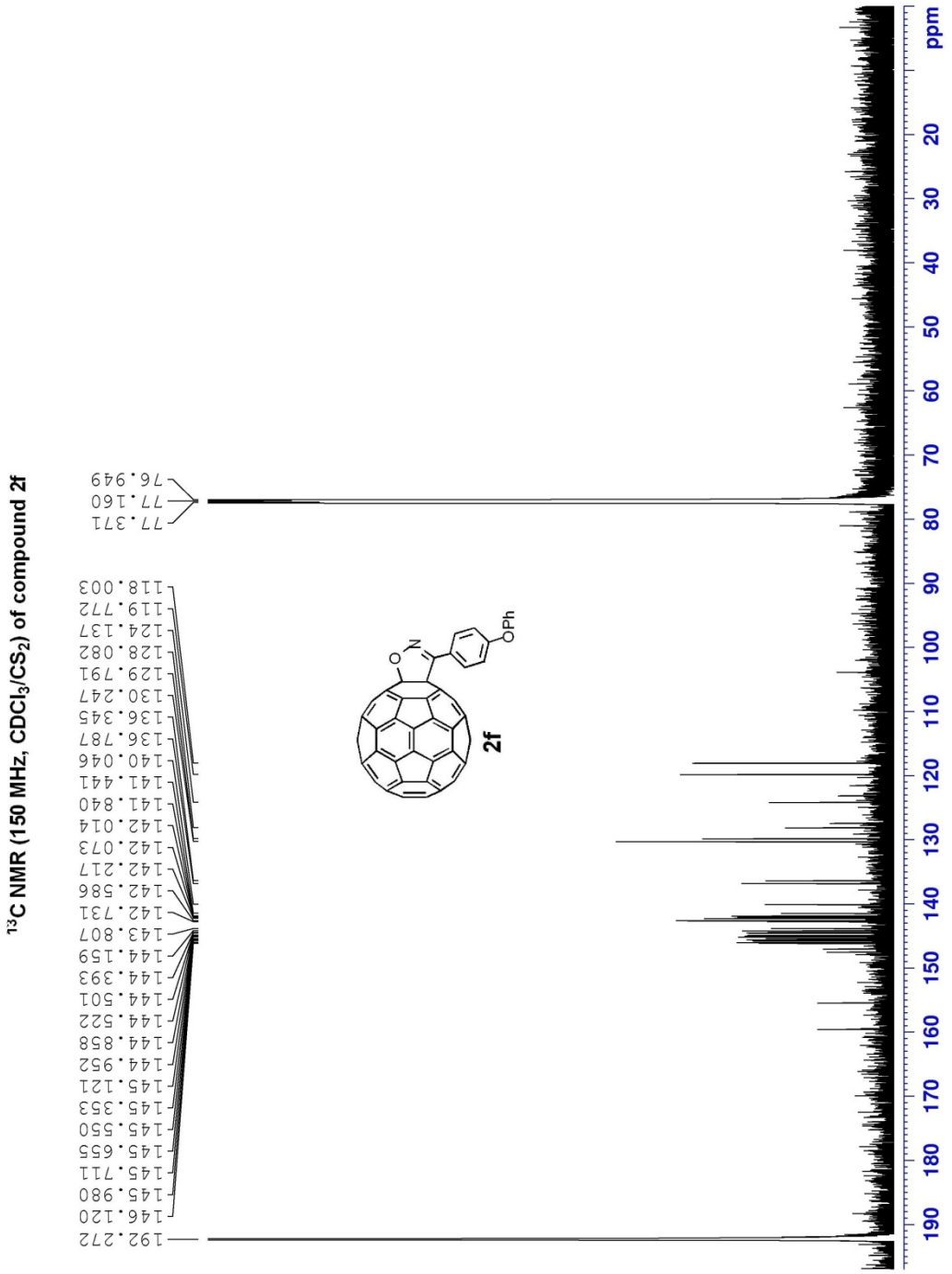
NAME	cd140609
EXPNO	1
PROCNO	1
Date_	20140609
Time	11.45
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	20
DS	0
SWH	5208.333 Hz
FIDRES	0.079473 Hz
AQ	6.2915058 sec
RG	362
DW	96.000 usec
DE	8.00 usec
TE	293.4 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.30 usec
PL1	-6.00 dB
PL1W	30.00000000 W
SFO1	400.2015093 MHz
NDO	2
TD	128
SFO1	400.2024 MHz
FIDRES	41.740742 Hz
SW	13.350 ppm
FnMODE	QF
SI	32768
SF	400.2000131 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00
SI	1024
MC2	QF
SF	400.2000000 MHz
WDW	SINE
SSB	0
LB	0.30 Hz
GB	0.1


^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2a
2a
21.542
76.841
77.158
77.475
79.213
103.945
126.018
128.717
129.740
136.627
136.902
140.171
140.230
140.866
141.602
141.976
142.190
142.219
142.367
142.744
142.883
143.995
144.300
144.414
144.696
144.730
145.047
145.123
145.318
145.502
145.785
145.836
145.888
146.144
146.169
146.287
147.160
147.668
ppm

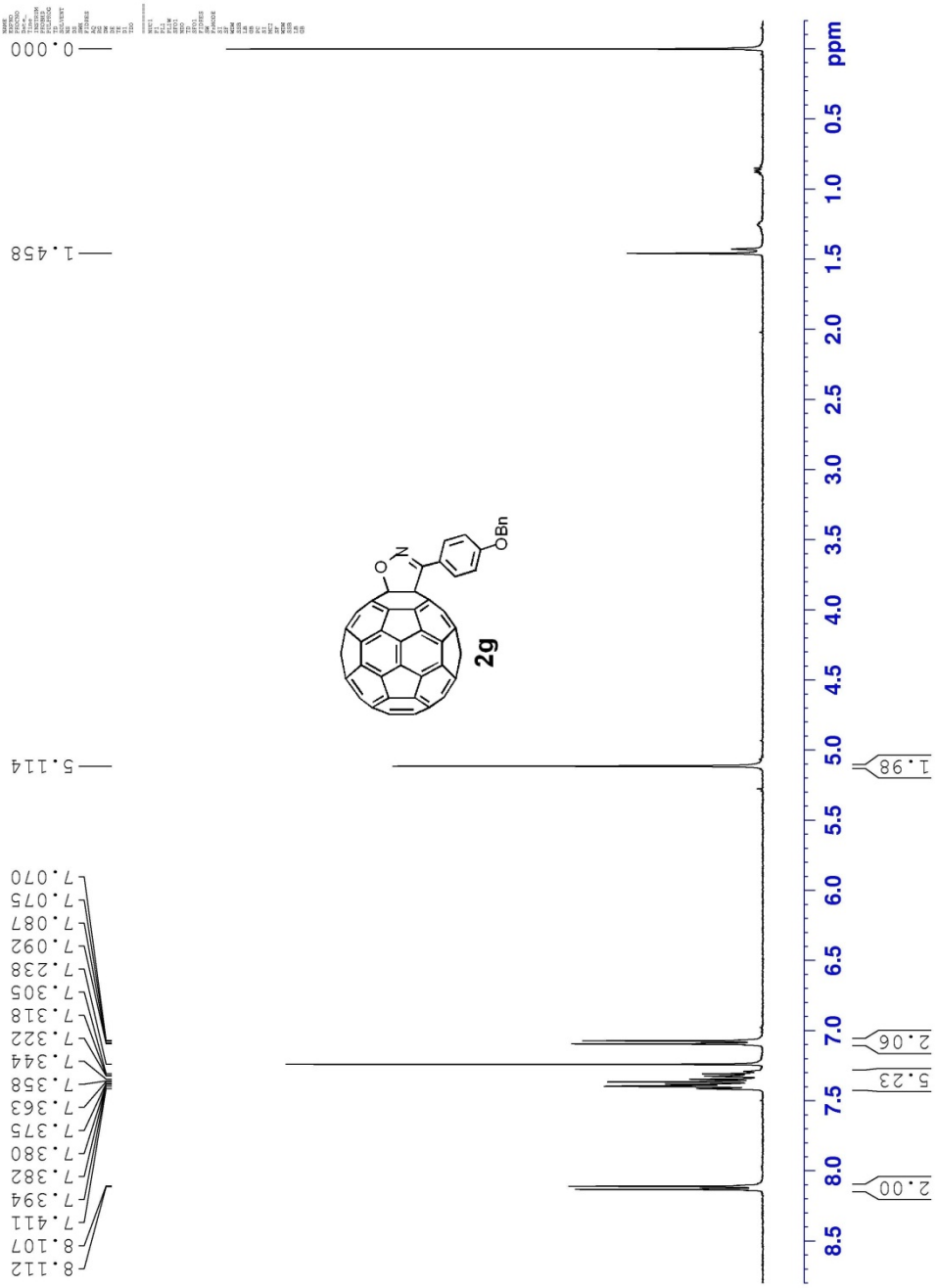


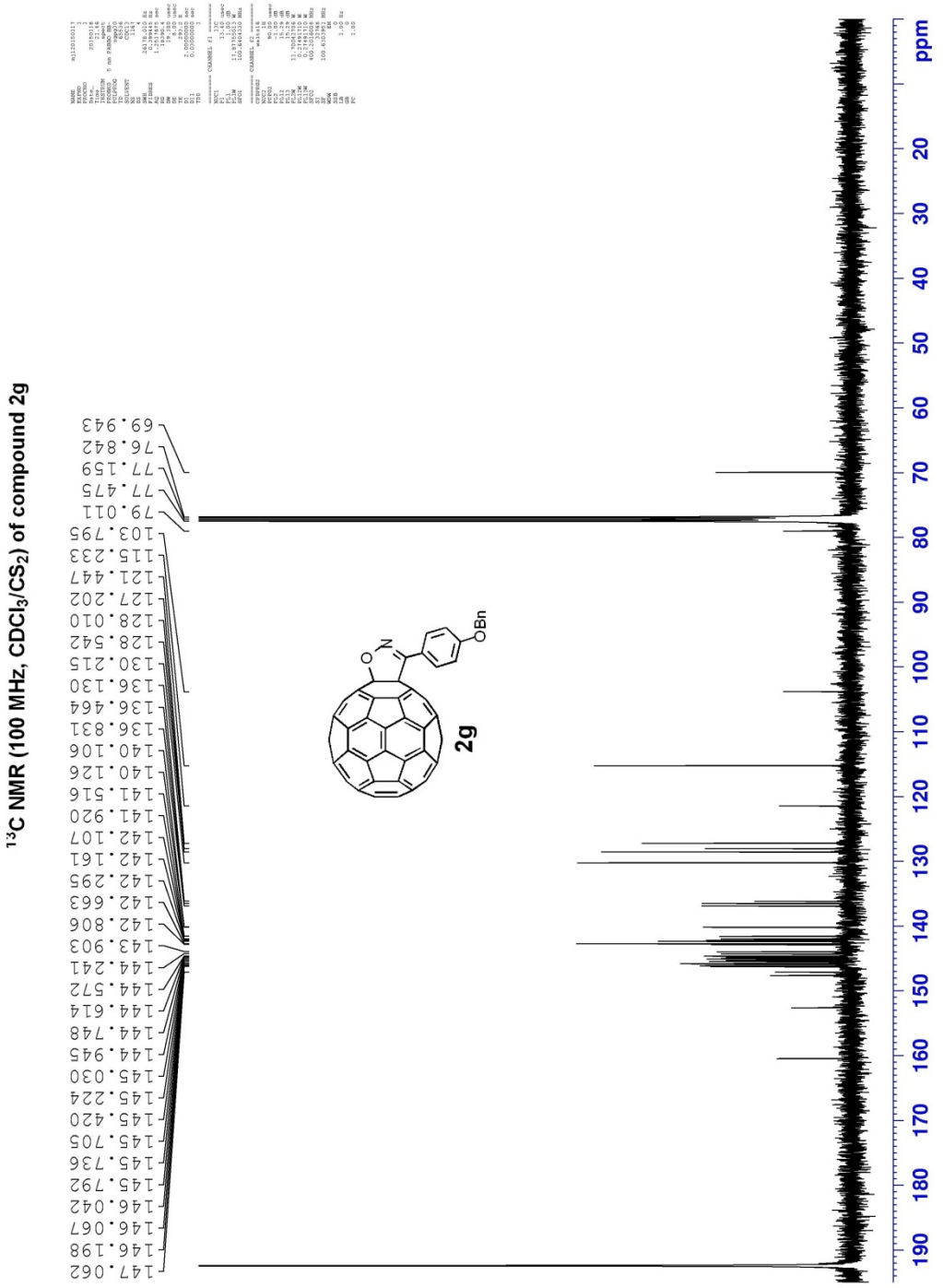
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2e

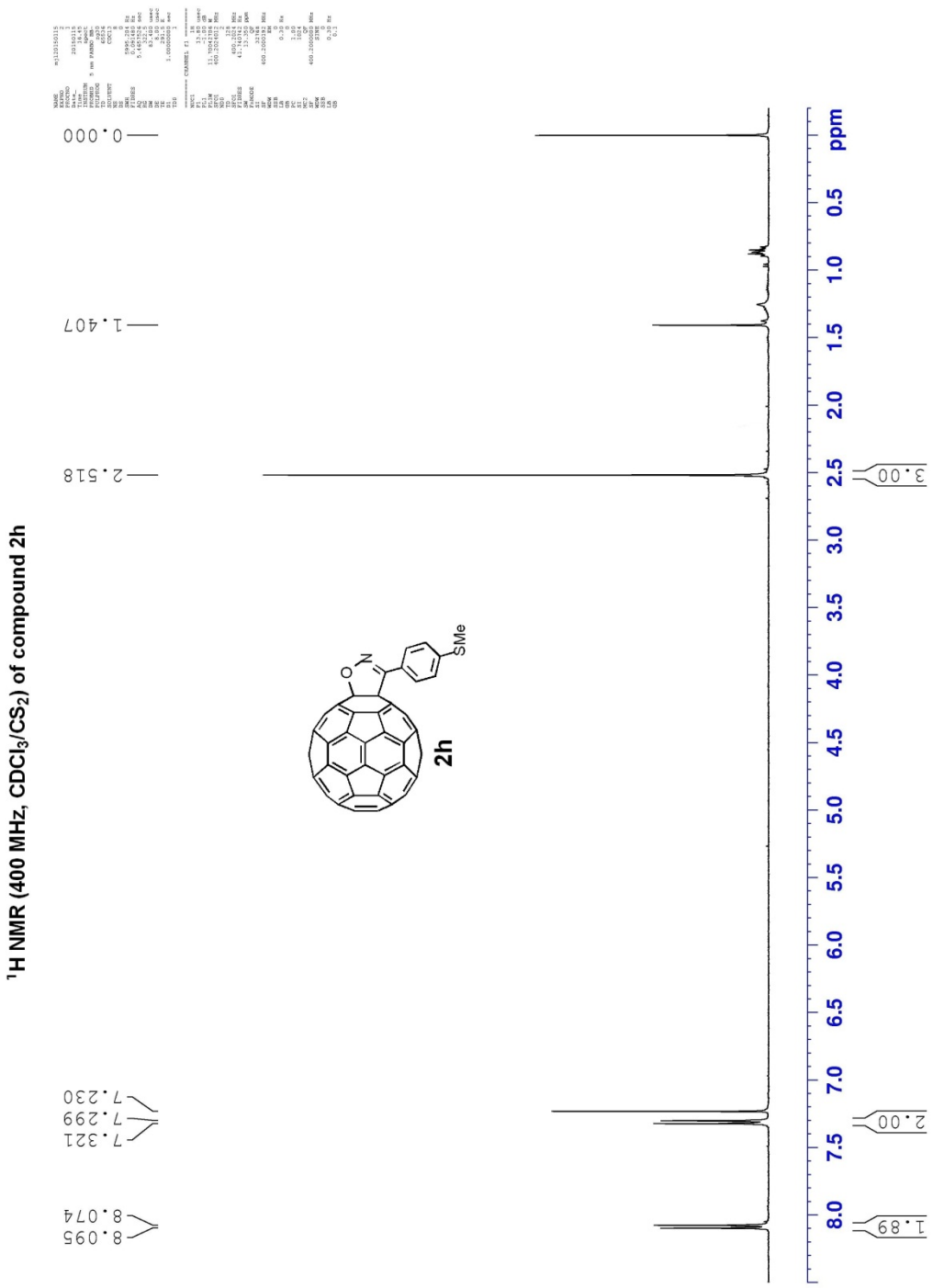

¹H NMR (400 MHz, CDCl3/CS2) of compound 2f
2f
Ph
O
N
ppm
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
2.00
4.11
1.03
2.04
8.132
8.110
7.360
7.341
7.320
7.224
7.149
7.130
7.112
7.071
7.049
7.044
7.024
1.359
0.000


^{13}C NMR (150 MHz, $CDCl_3/CS_2$) of compound 2f
192.272
146.120
145.980
145.711
145.655
145.550
145.353
145.121
144.952
144.858
144.522
144.501
144.393
144.159
143.807
142.731
142.586
142.217
142.073
142.014
141.840
141.441
140.046
136.787
136.345
130.247
129.791
128.082
124.137
119.772
118.003
77.371
77.160
76.949
OPh
2f
ppm

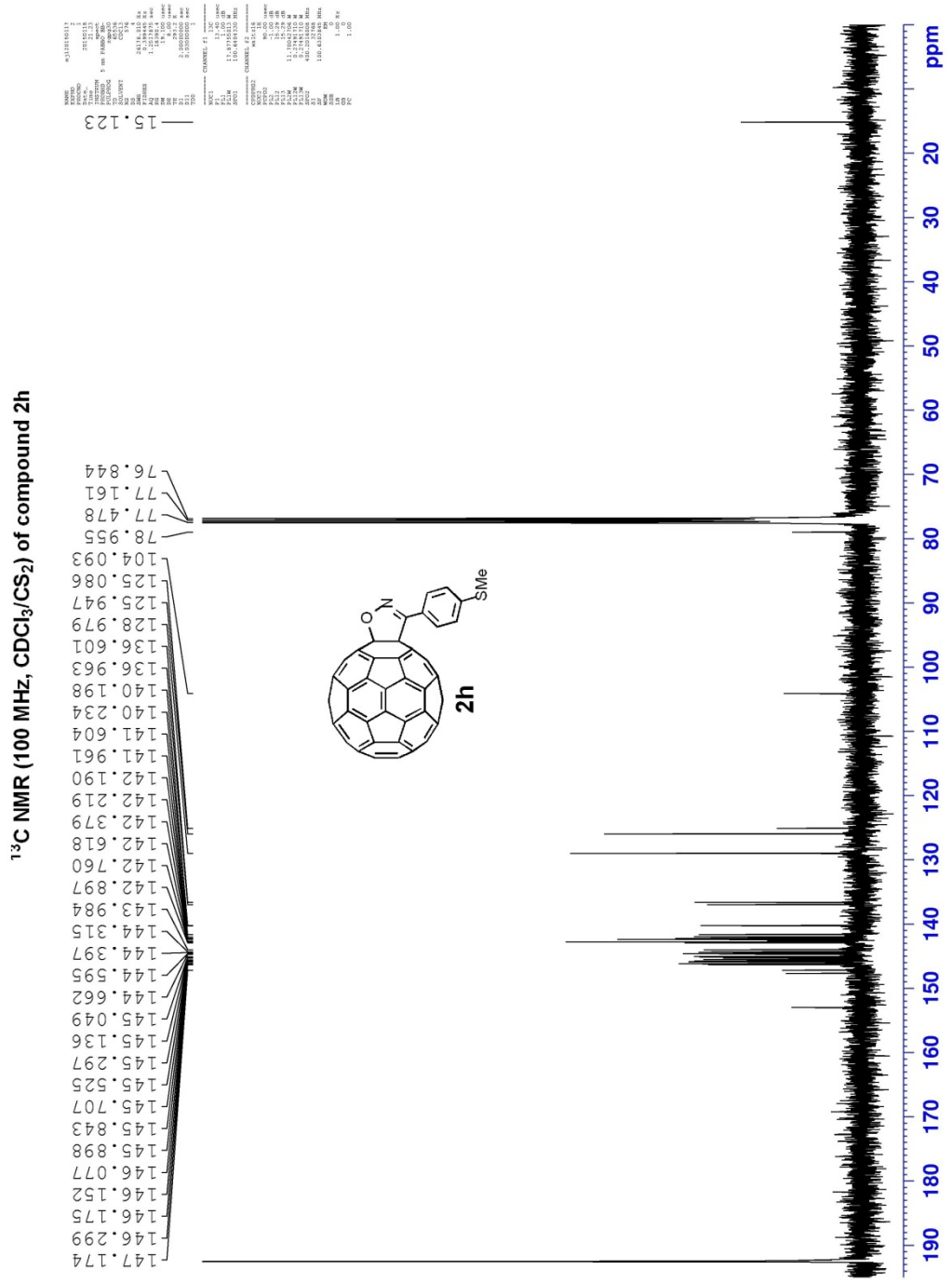
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2g



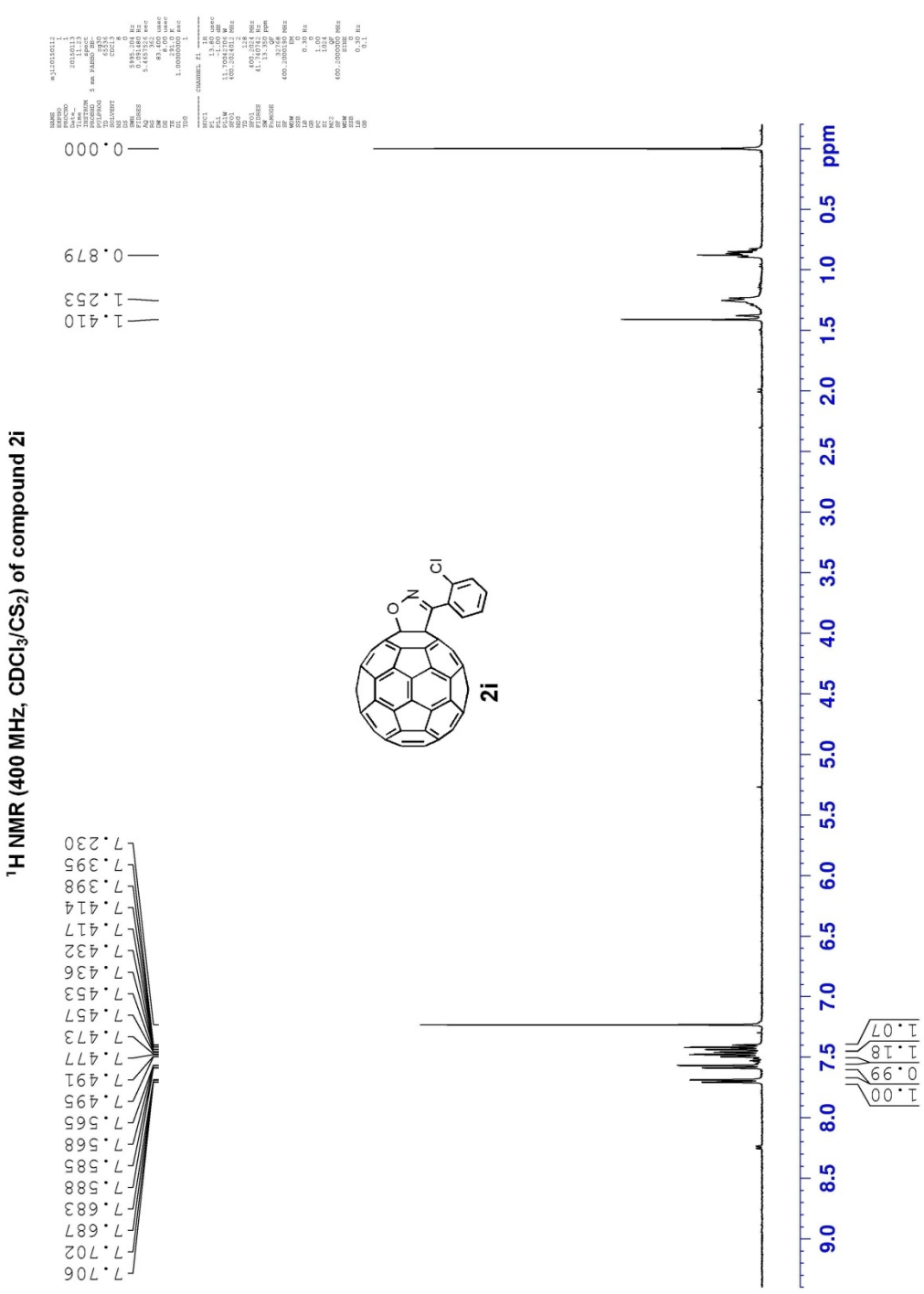

^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2g
147.062
146.198
146.067
146.042
145.792
145.736
145.705
145.420
145.224
145.030
144.945
144.748
144.614
144.572
144.241
143.903
142.806
142.663
142.295
142.161
142.107
141.920
141.516
140.126
140.106
136.831
136.464
136.130
130.215
128.542
128.010
127.202
121.447
115.233
103.795
79.011
77.475
77.159
76.842
69.943
2g
ppm

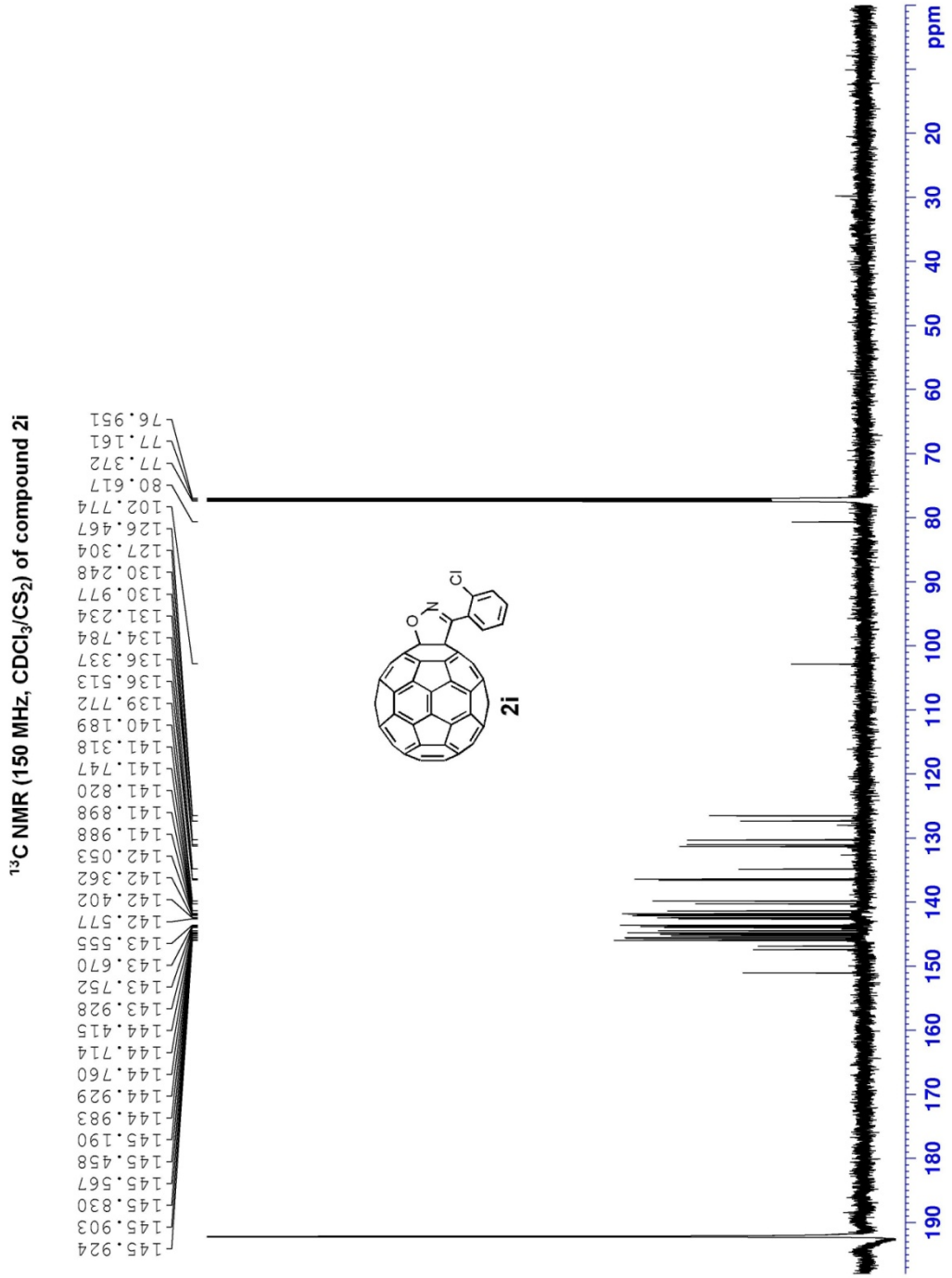


^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2h

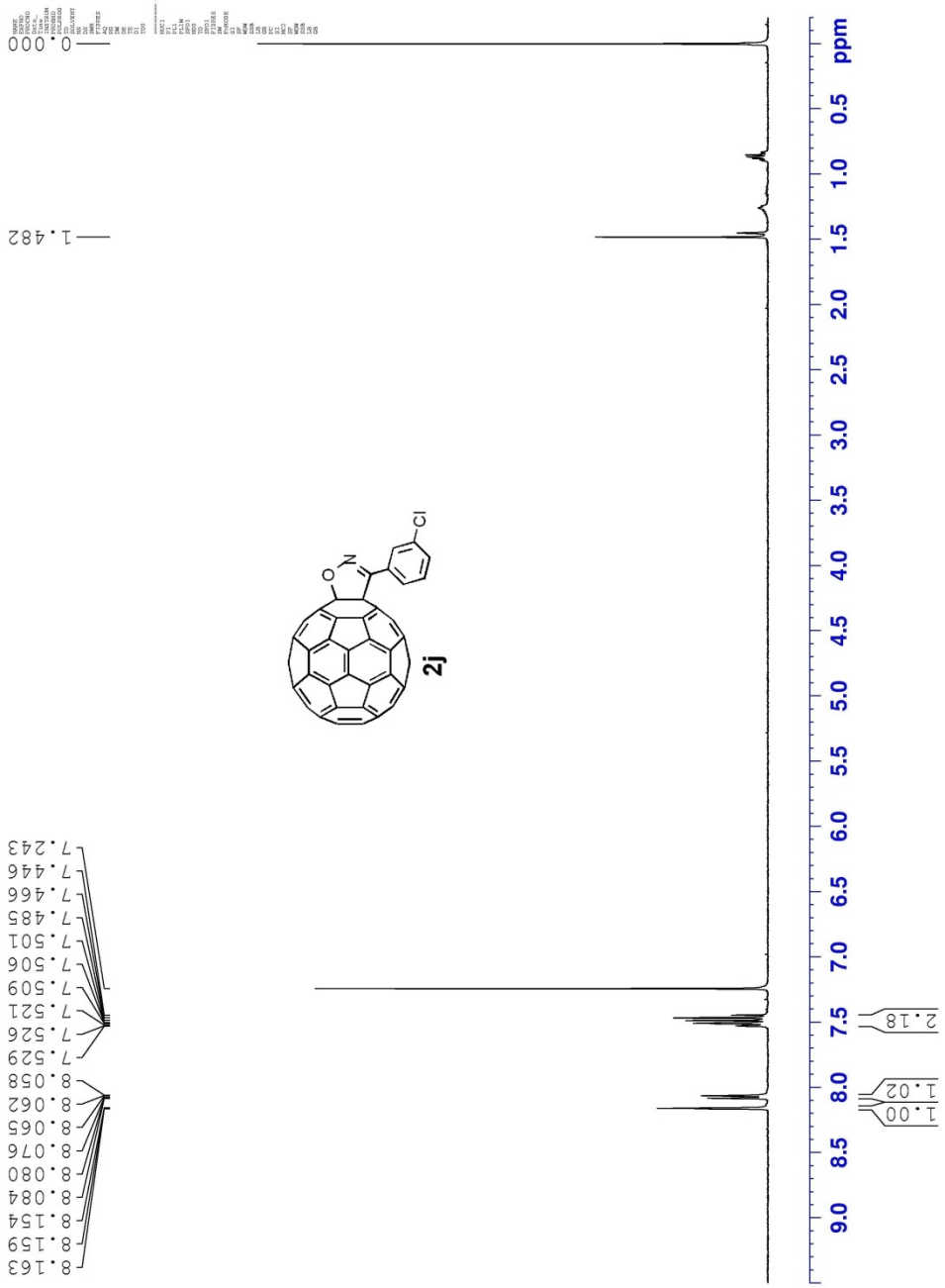

^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2h
15.123
76.844
77.161
77.478
78.955
104.093
125.086
125.947
128.979
136.601
136.963
140.198
140.234
141.604
141.961
142.190
142.219
142.379
142.618
142.760
142.897
143.984
144.315
144.397
144.595
144.662
145.049
145.136
145.297
145.525
145.707
145.843
145.898
146.077
146.152
146.175
146.299
147.174
SMe
2h
ppm

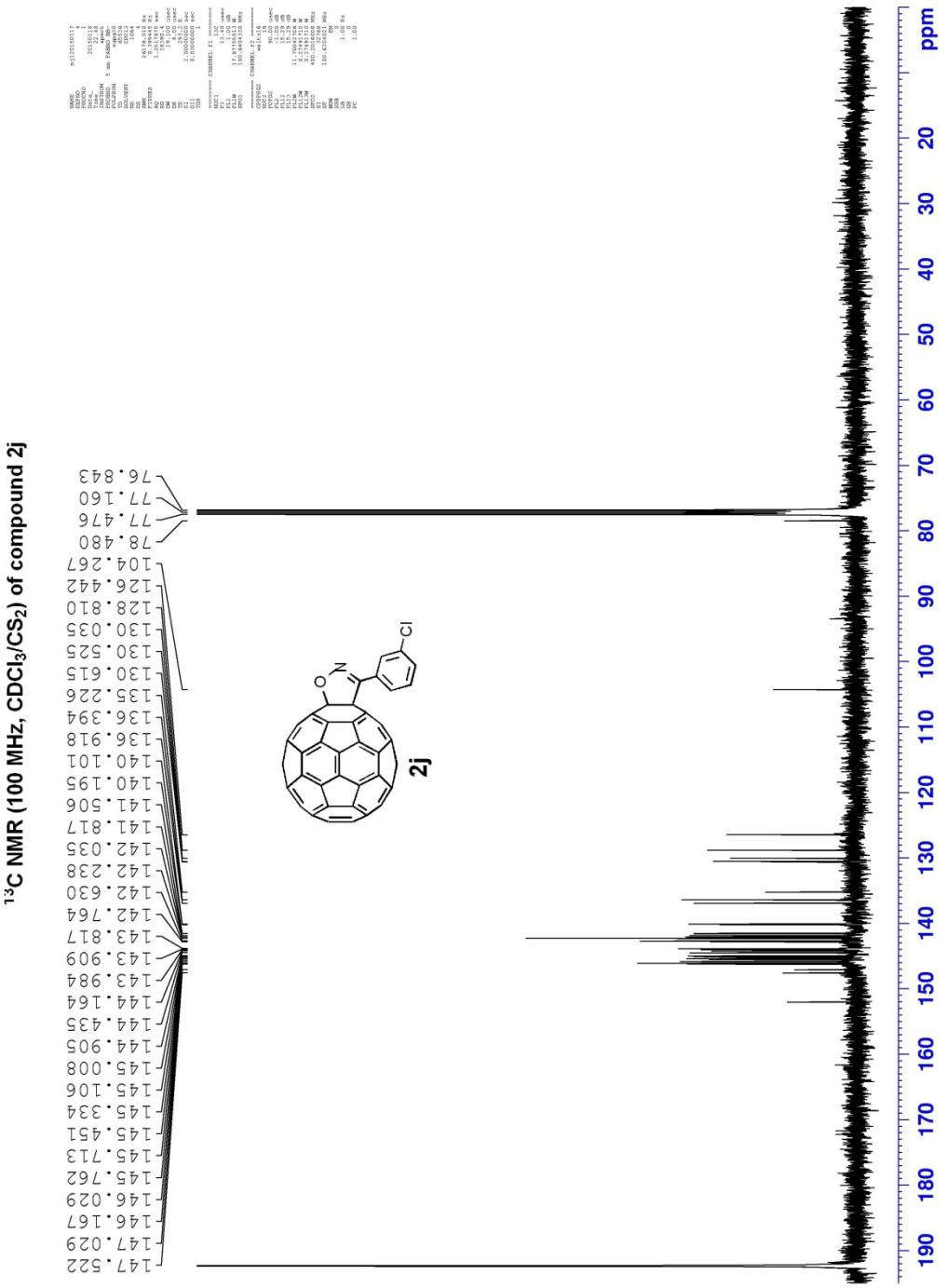
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2i



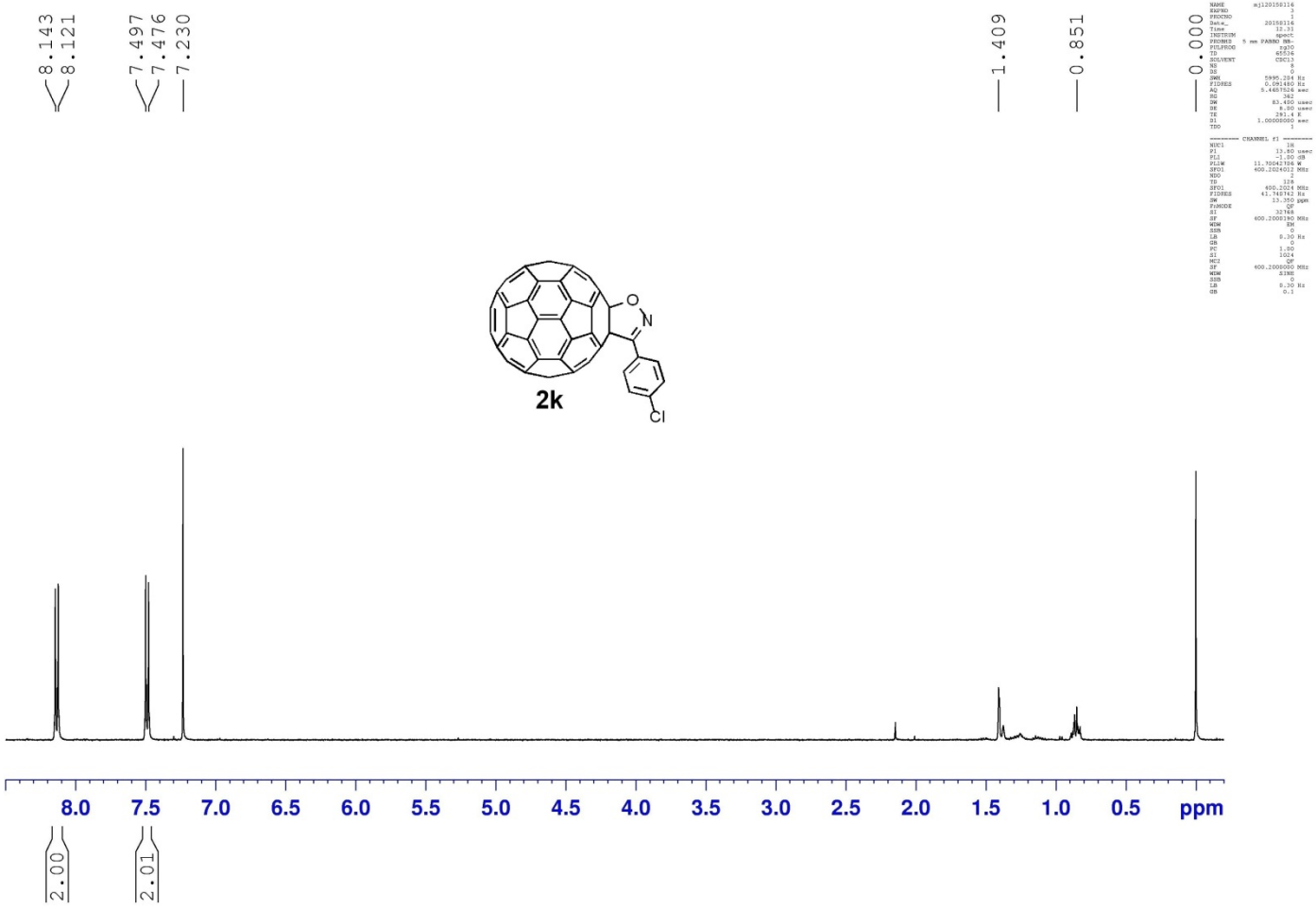

^{13}C NMR (150 MHz, $CDCl_3/CS_2$) of compound 2i
145.924
145.903
145.830
145.567
145.458
145.190
144.983
144.929
144.760
144.714
144.415
143.928
143.752
143.670
143.555
142.577
142.402
142.362
142.053
141.988
141.898
141.820
141.747
141.318
140.189
139.772
136.513
136.337
134.784
131.234
130.977
130.248
127.304
126.467
102.774
80.617
77.372
77.161
76.951
Cl
N
O
2i
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2j

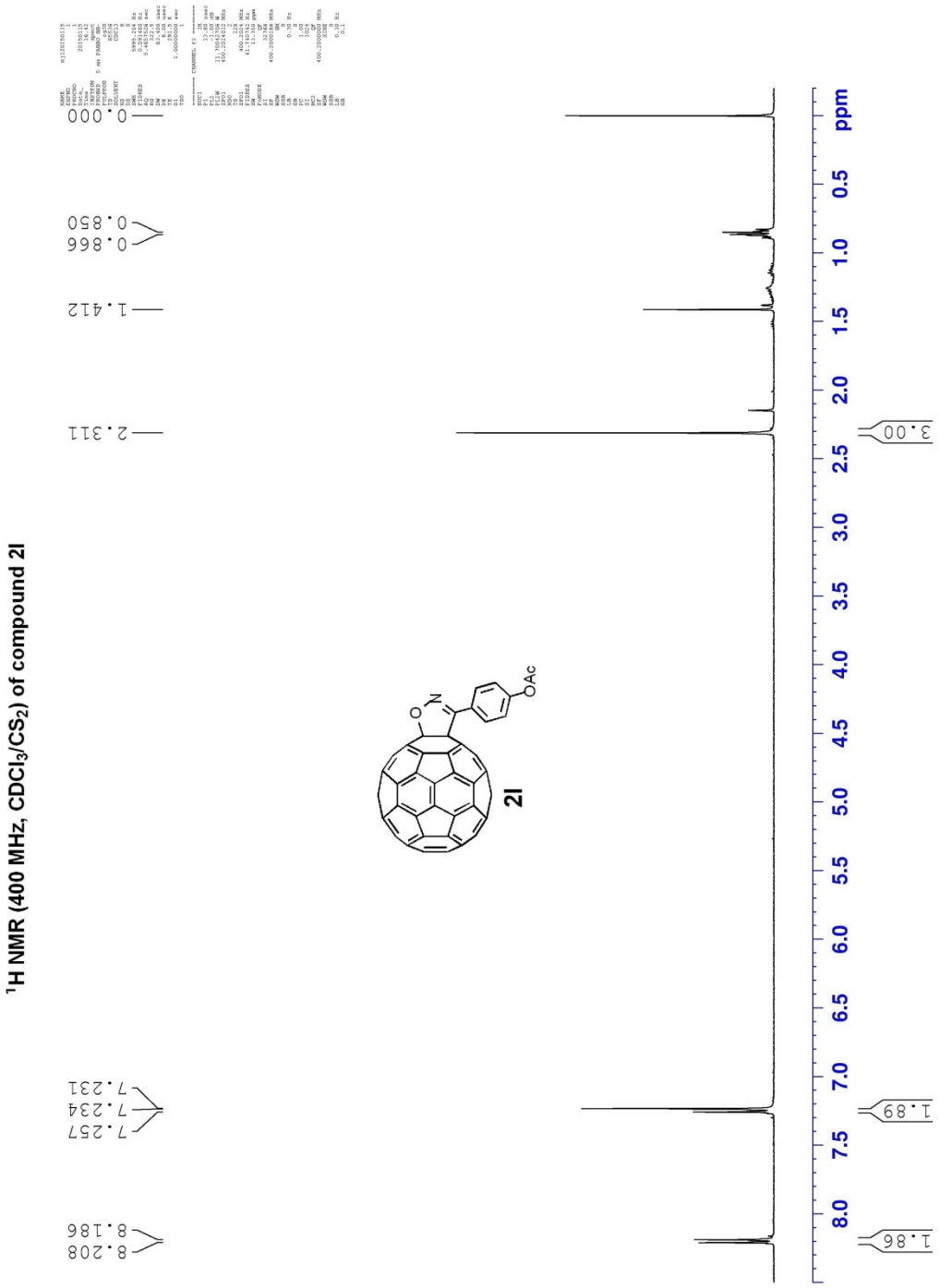



^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2j
147.522
147.029
146.167
146.029
145.762
145.713
145.451
145.334
145.106
145.008
144.905
144.435
144.164
143.984
143.909
143.817
142.764
142.630
142.238
142.035
141.817
141.506
140.195
140.101
136.918
136.394
135.226
130.615
130.525
130.035
128.810
126.442
104.267
78.480
77.476
77.160
76.843
2j
Cl
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

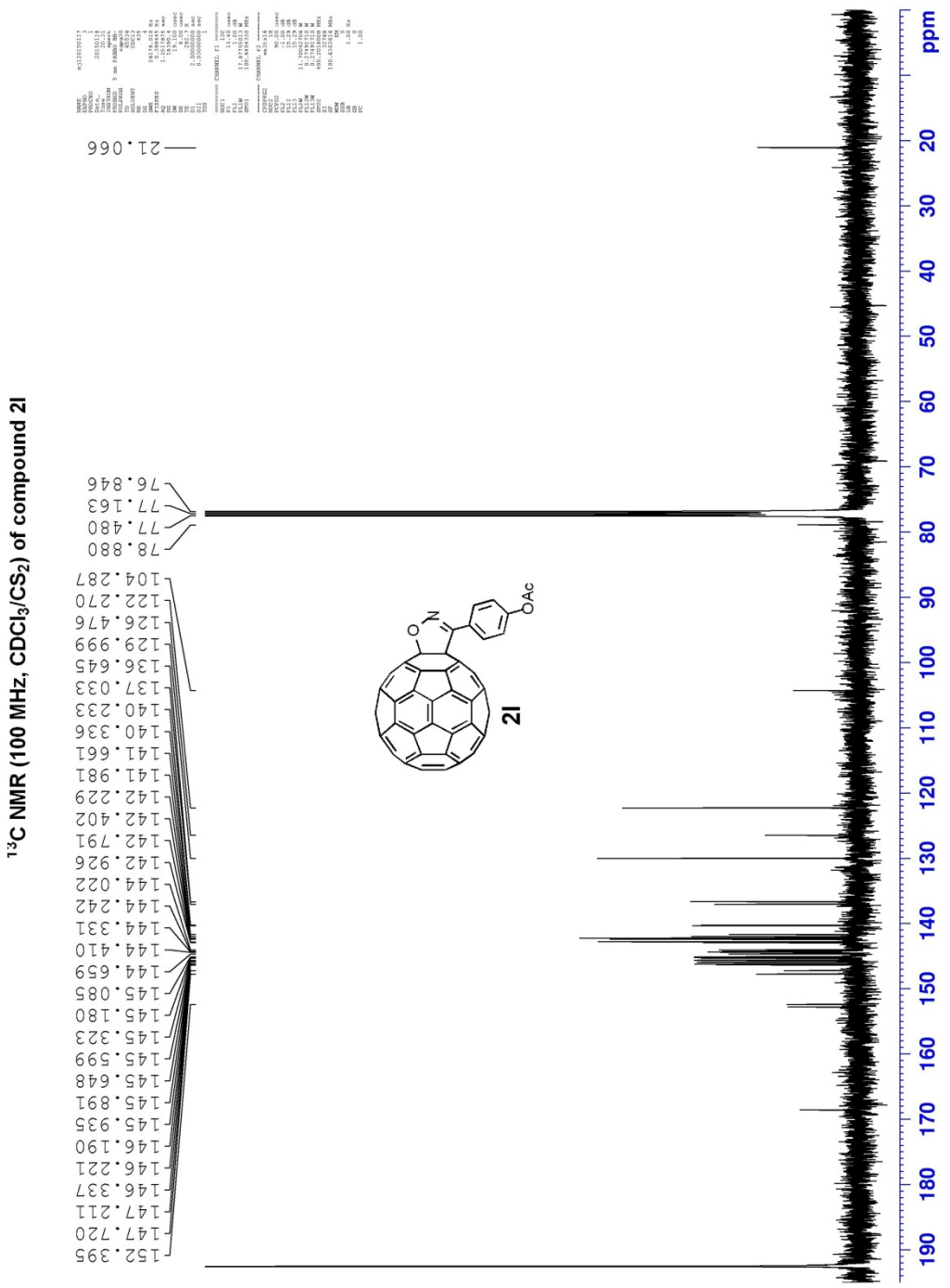
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2k



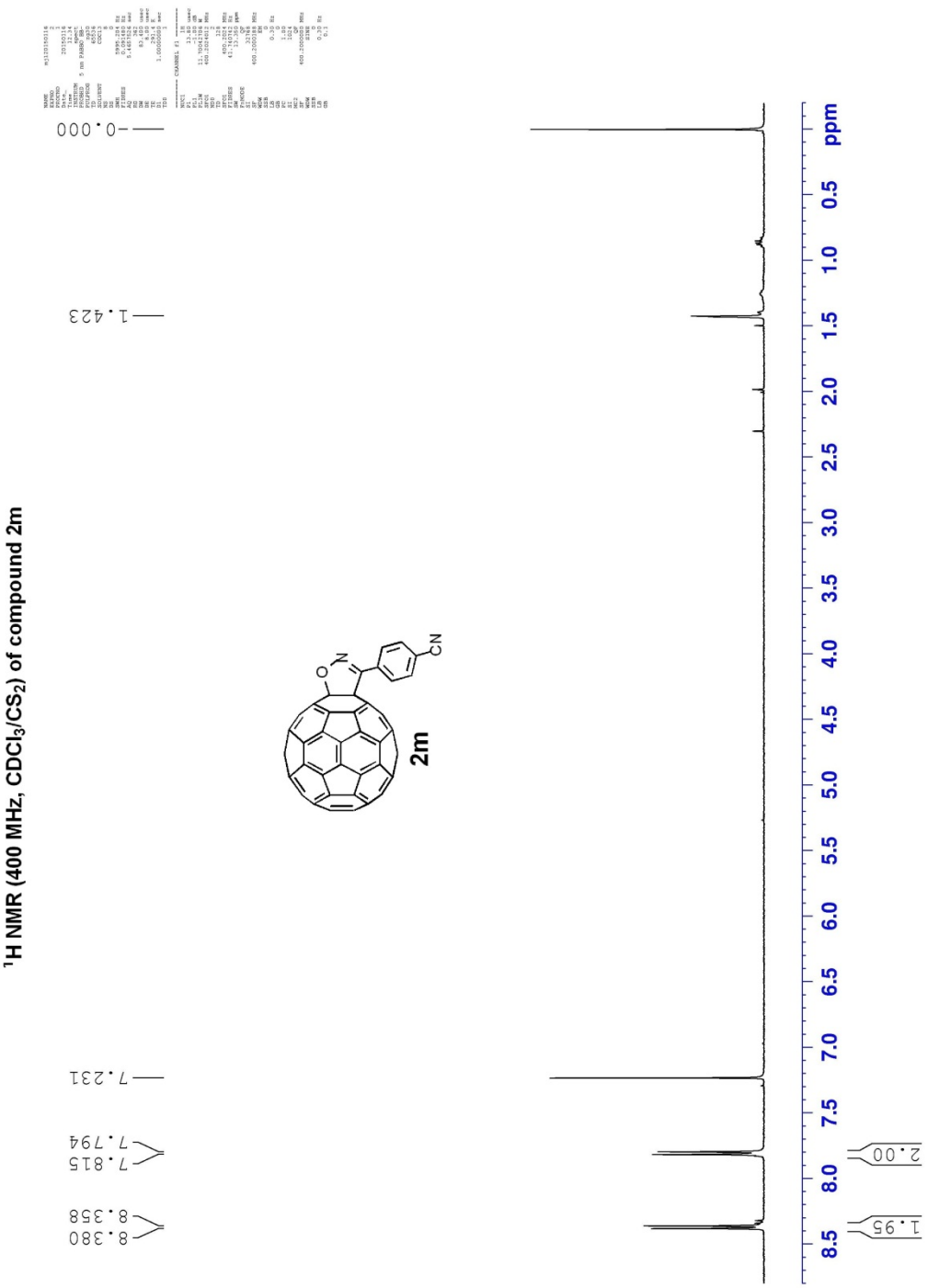
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2l



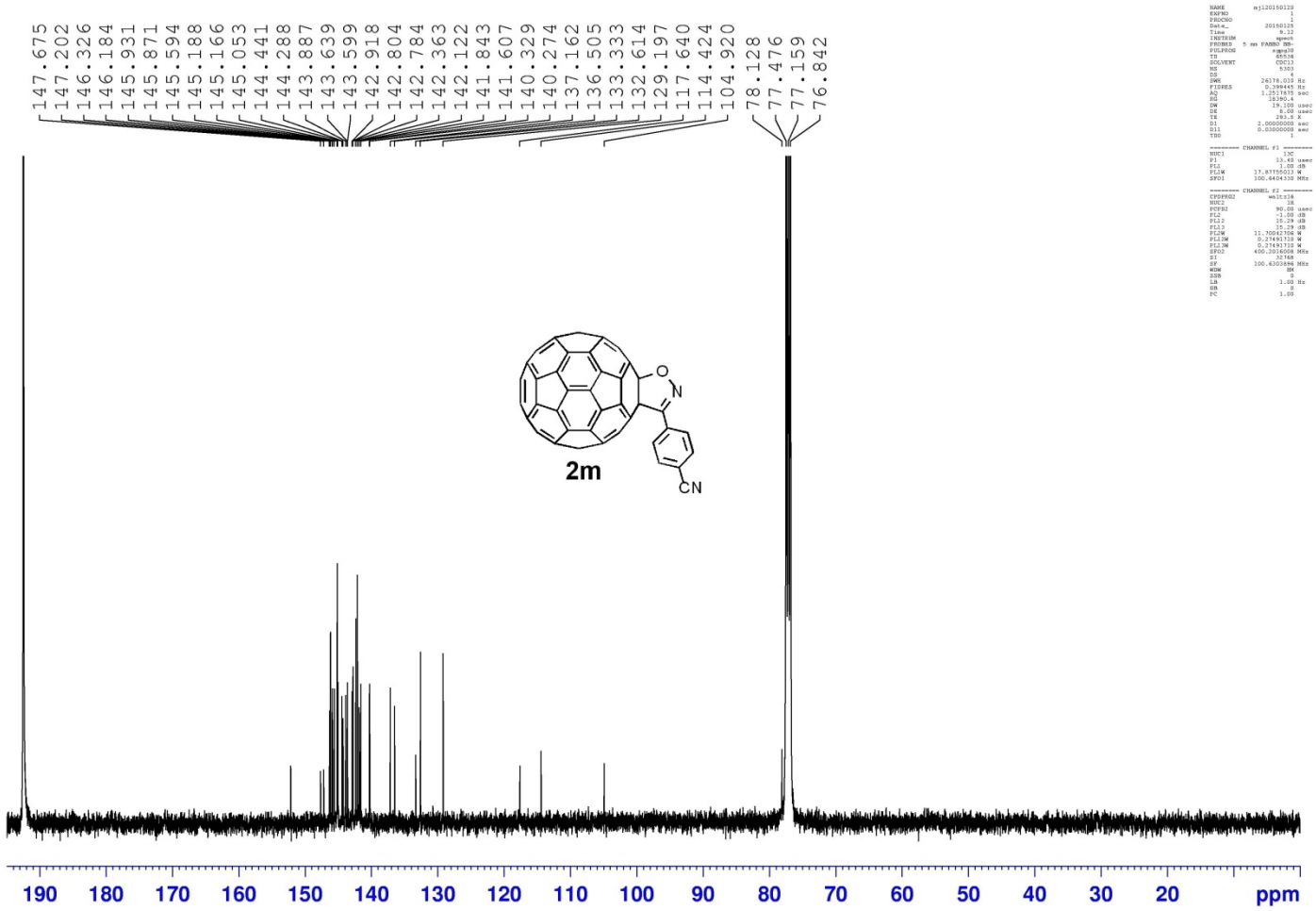
2l


13C NMR (100 MHz, CDCl3/CS2) of compound 2l
21.066
76.846
77.163
77.480
78.880
104.287
122.270
126.476
129.999
136.645
137.033
140.233
140.336
141.661
141.981
142.229
142.402
142.791
142.926
144.022
144.242
144.331
144.410
144.659
145.085
145.180
145.323
145.599
145.648
145.891
145.935
146.190
146.221
146.337
147.211
147.720
152.395
2l
OAc
N
O
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

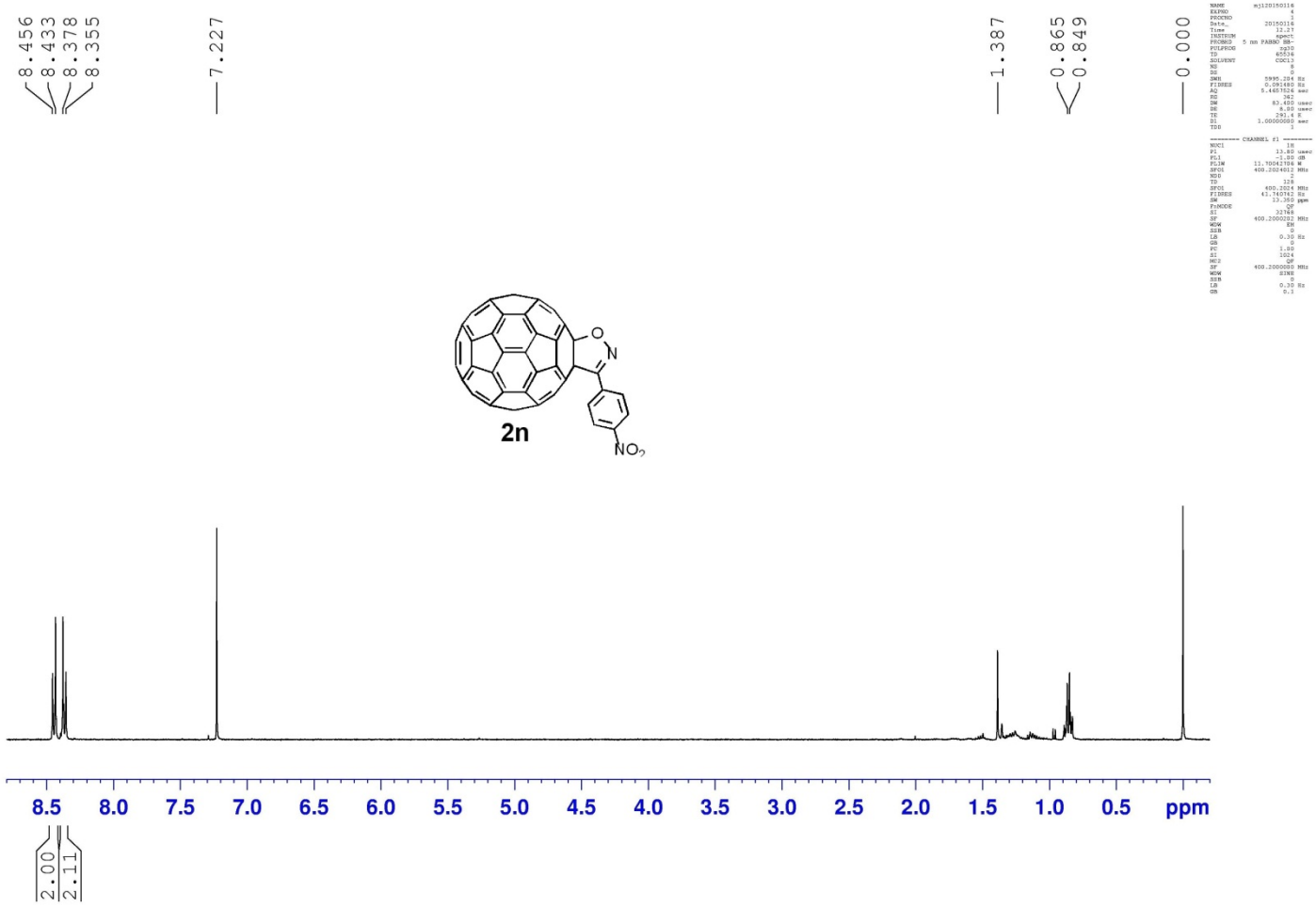
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2m



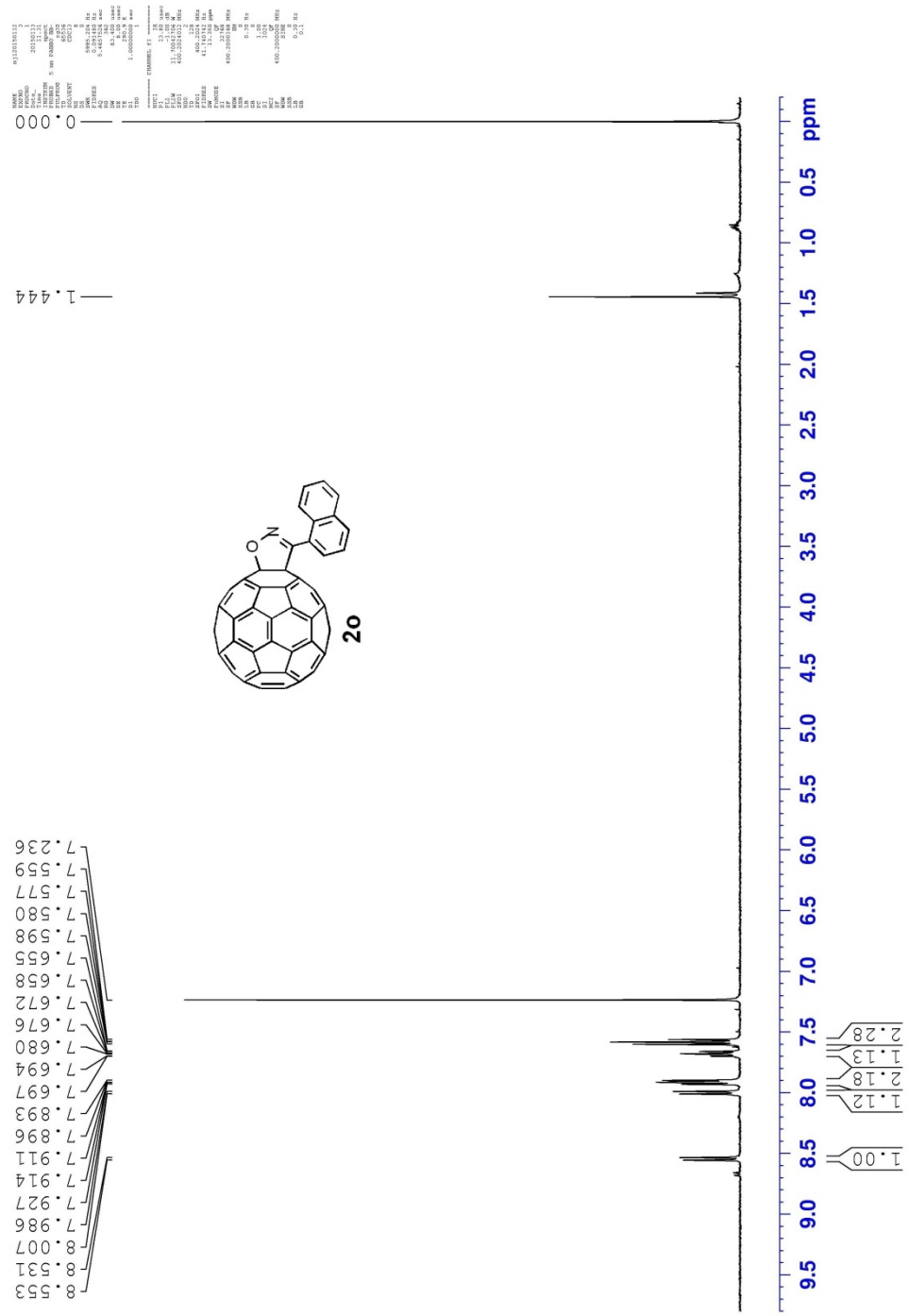
^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2m

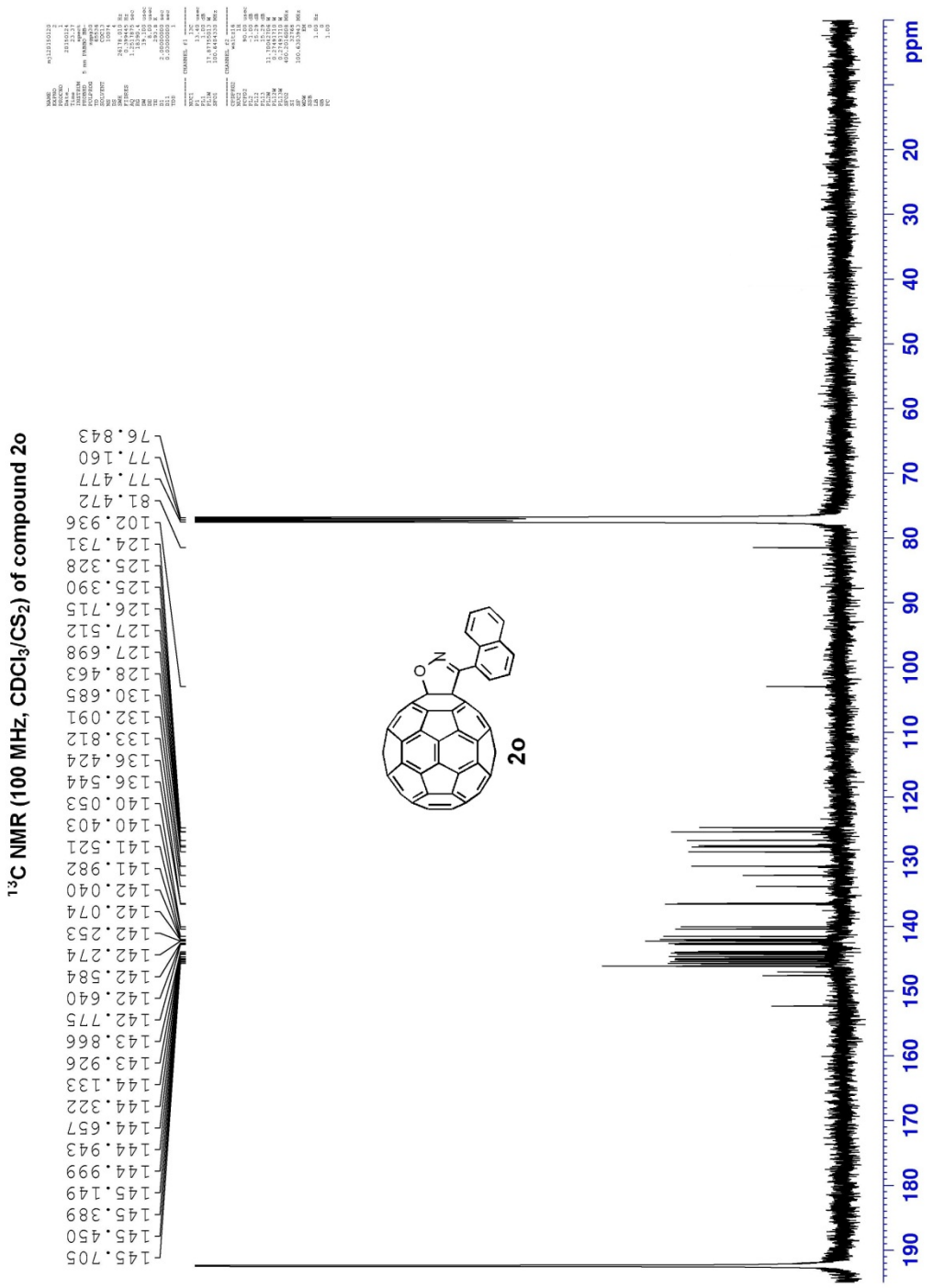


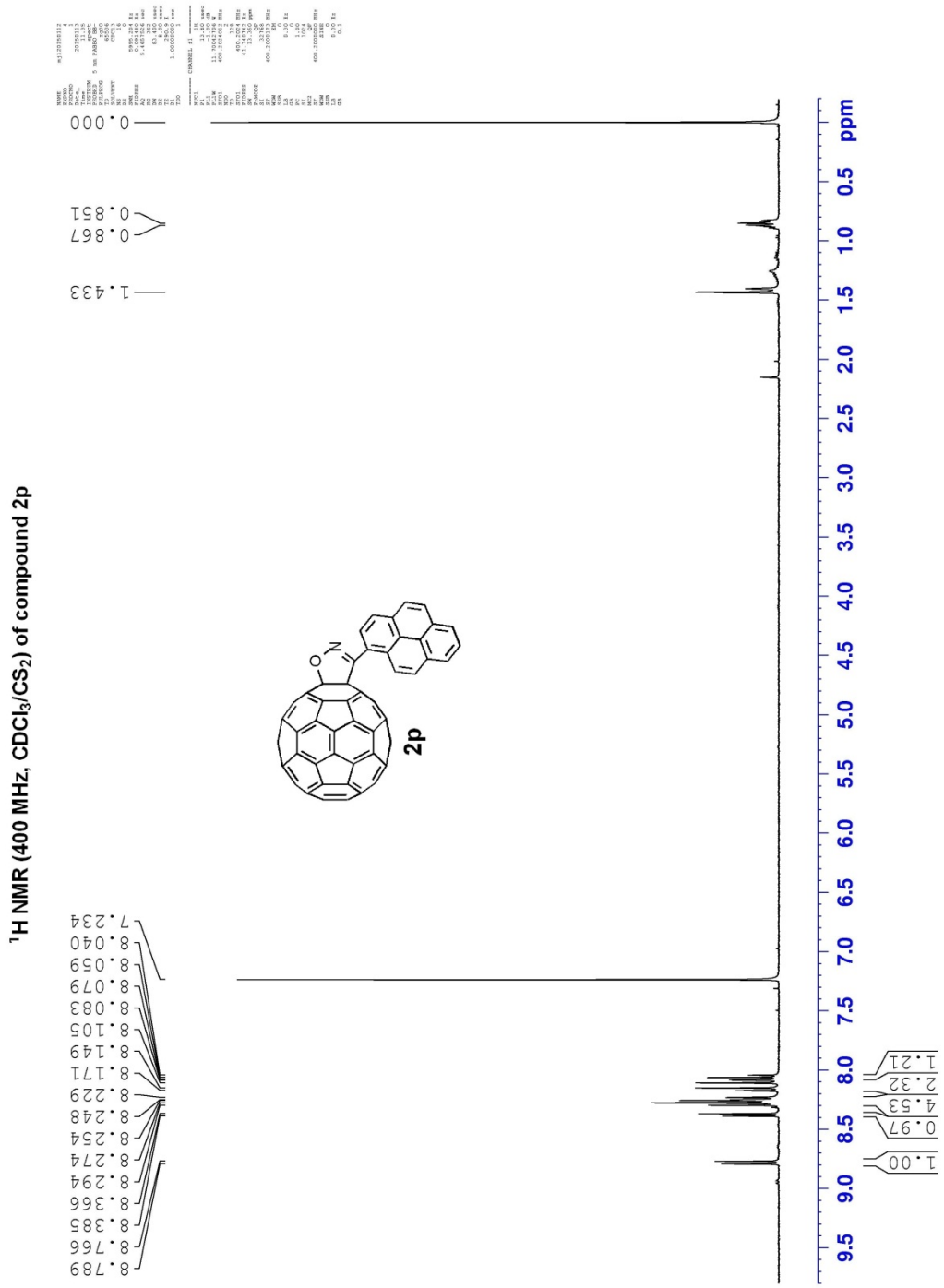
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2n

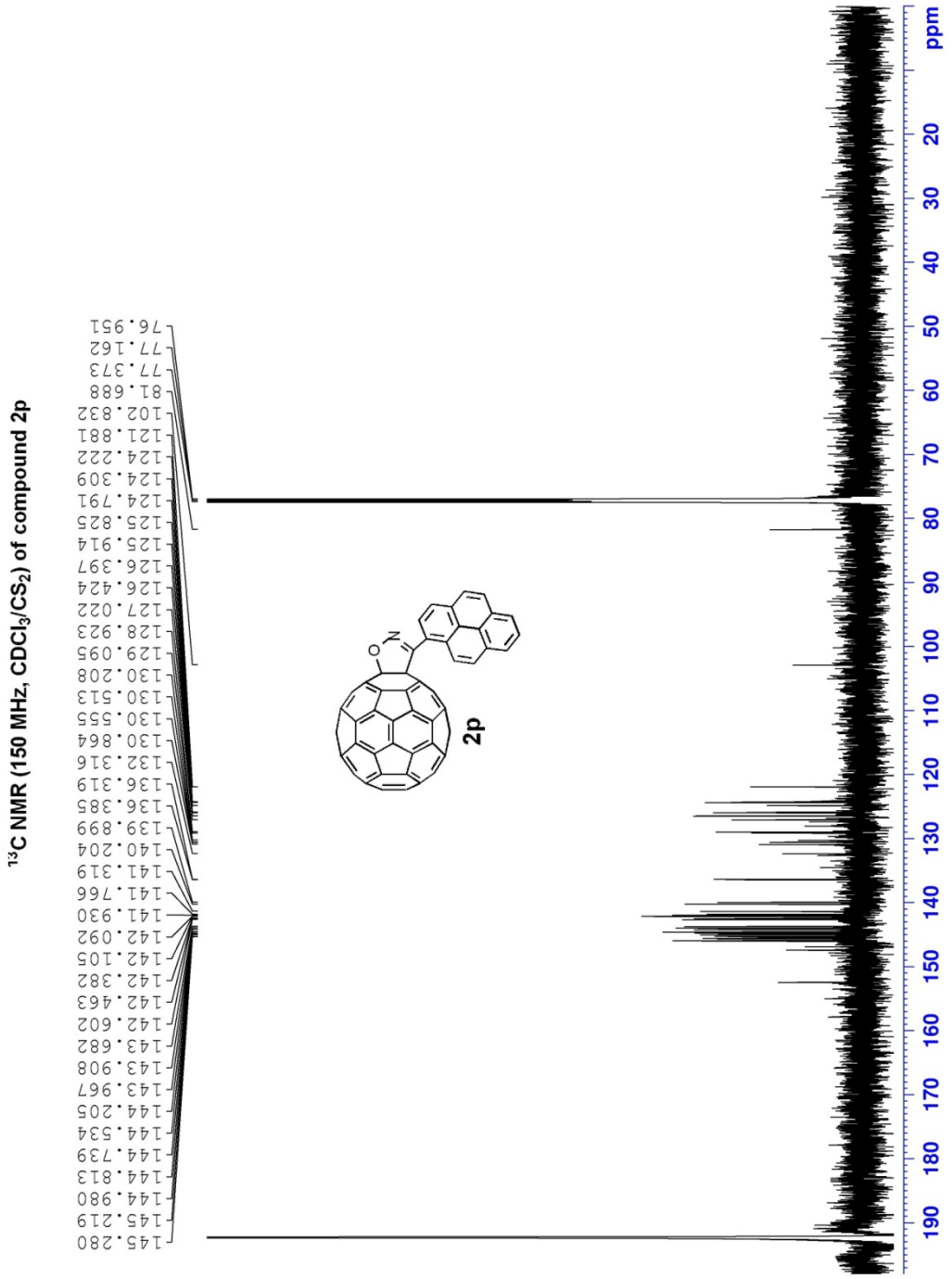


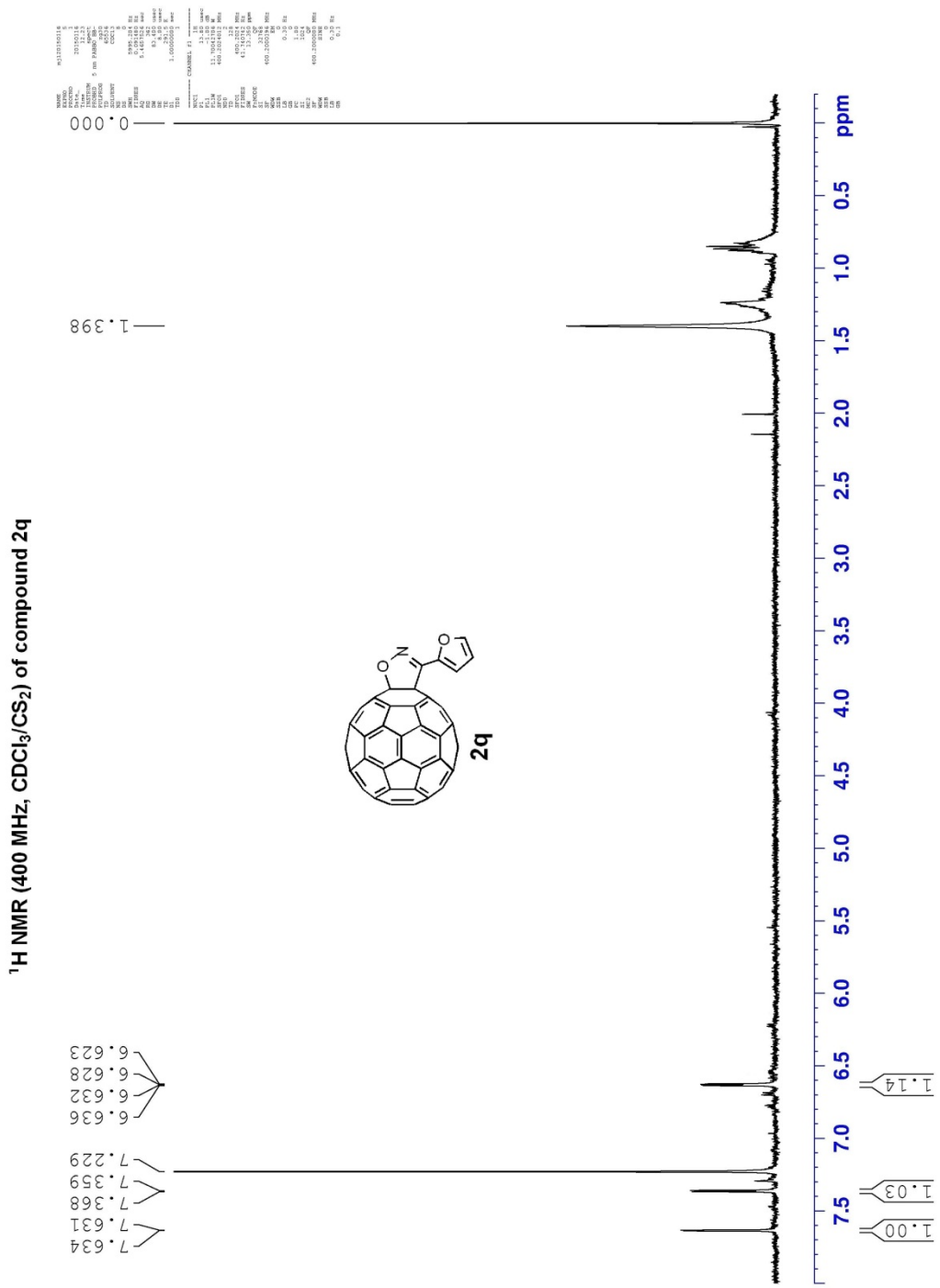
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2o



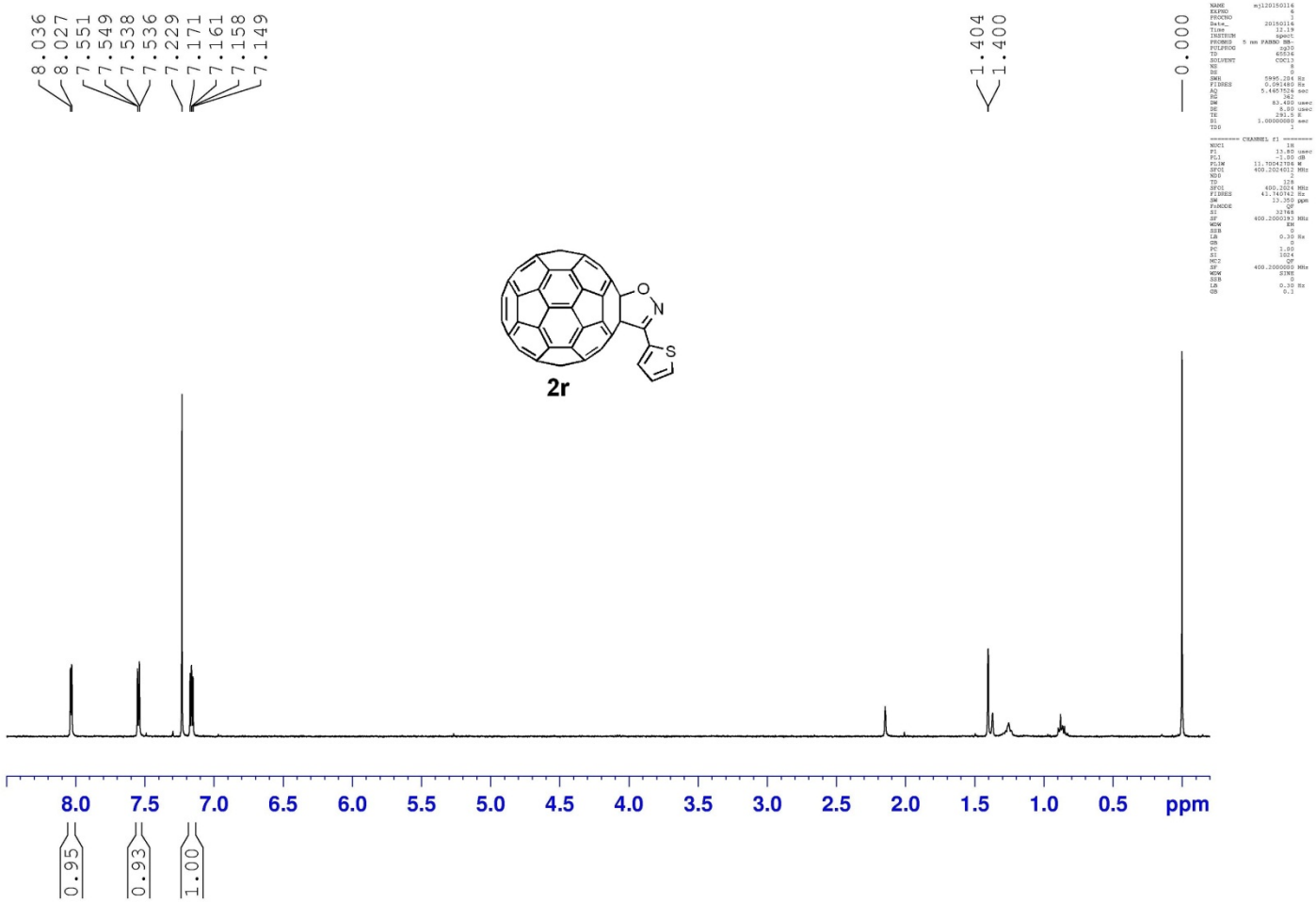

^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2o
145.705
145.450
145.389
145.149
144.999
144.943
144.657
144.322
144.133
143.926
143.866
142.775
142.640
142.584
142.274
142.253
142.074
142.040
141.982
141.521
140.403
140.053
136.544
136.424
133.812
132.091
130.685
128.463
127.698
127.512
126.715
125.390
125.328
124.731
102.936
81.472
77.477
77.160
76.843
2o
ppm


1H NMR (400 MHz, CDCl3/CS2) of compound 2p
2p
ppm

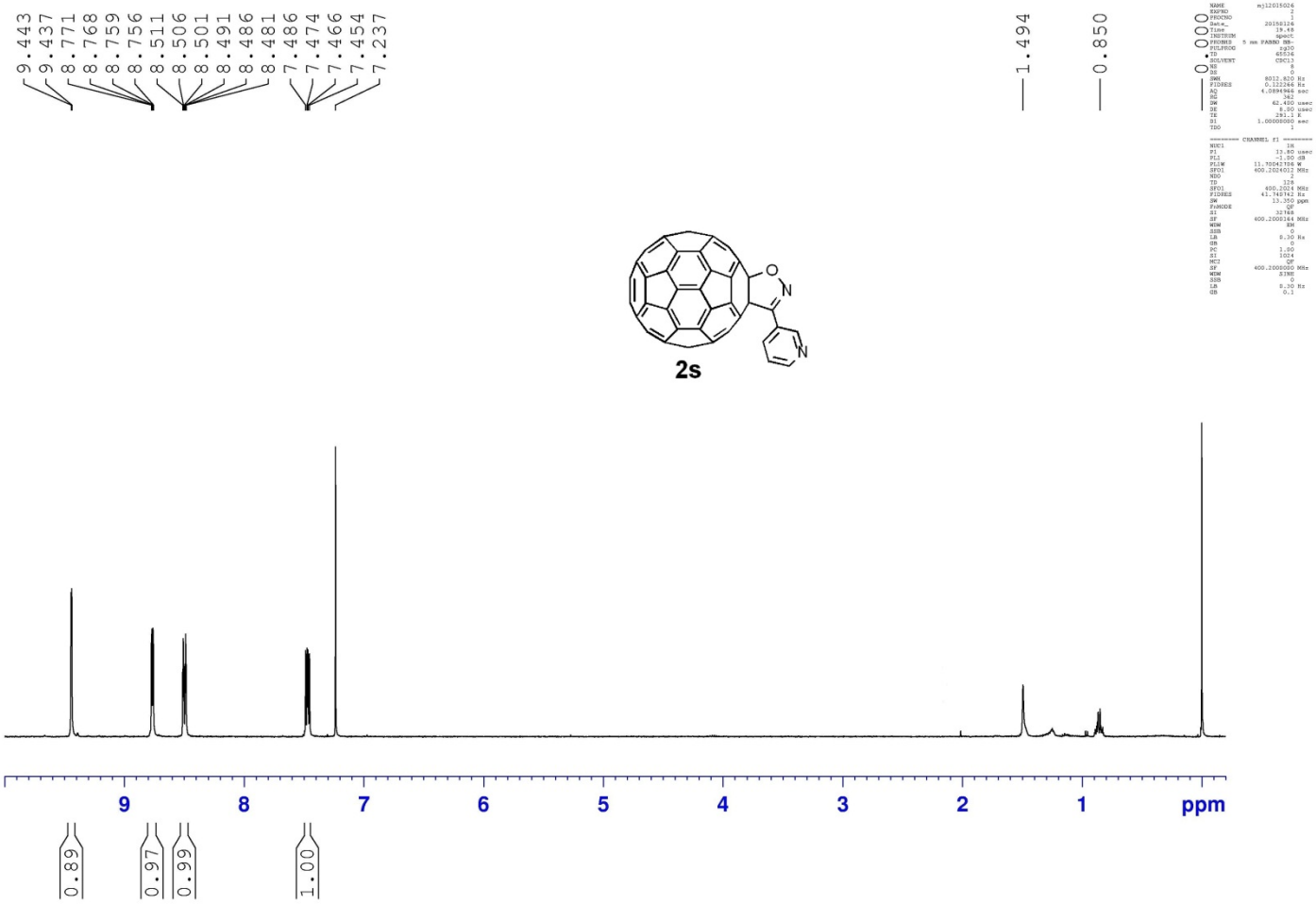

^{13}C NMR (150 MHz, $CDCl_3/CS_2$) of compound 2p
145.280
145.219
144.980
144.813
144.739
144.534
144.205
143.967
143.908
143.682
142.602
142.463
142.382
142.105
142.092
141.930
141.766
141.319
140.204
139.899
136.385
136.319
132.316
130.864
130.555
130.513
130.208
129.095
128.923
127.022
126.424
126.397
125.914
125.825
124.791
124.309
124.222
121.881
102.832
81.688
77.373
77.162
76.951
2p
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20


1H NMR (400 MHz, CDCl3/CS2) of compound 2q
2q
7.634
7.631
7.368
7.359
7.229
6.636
6.632
6.628
6.623
1.398
0.000
1.00
1.03
1.14
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

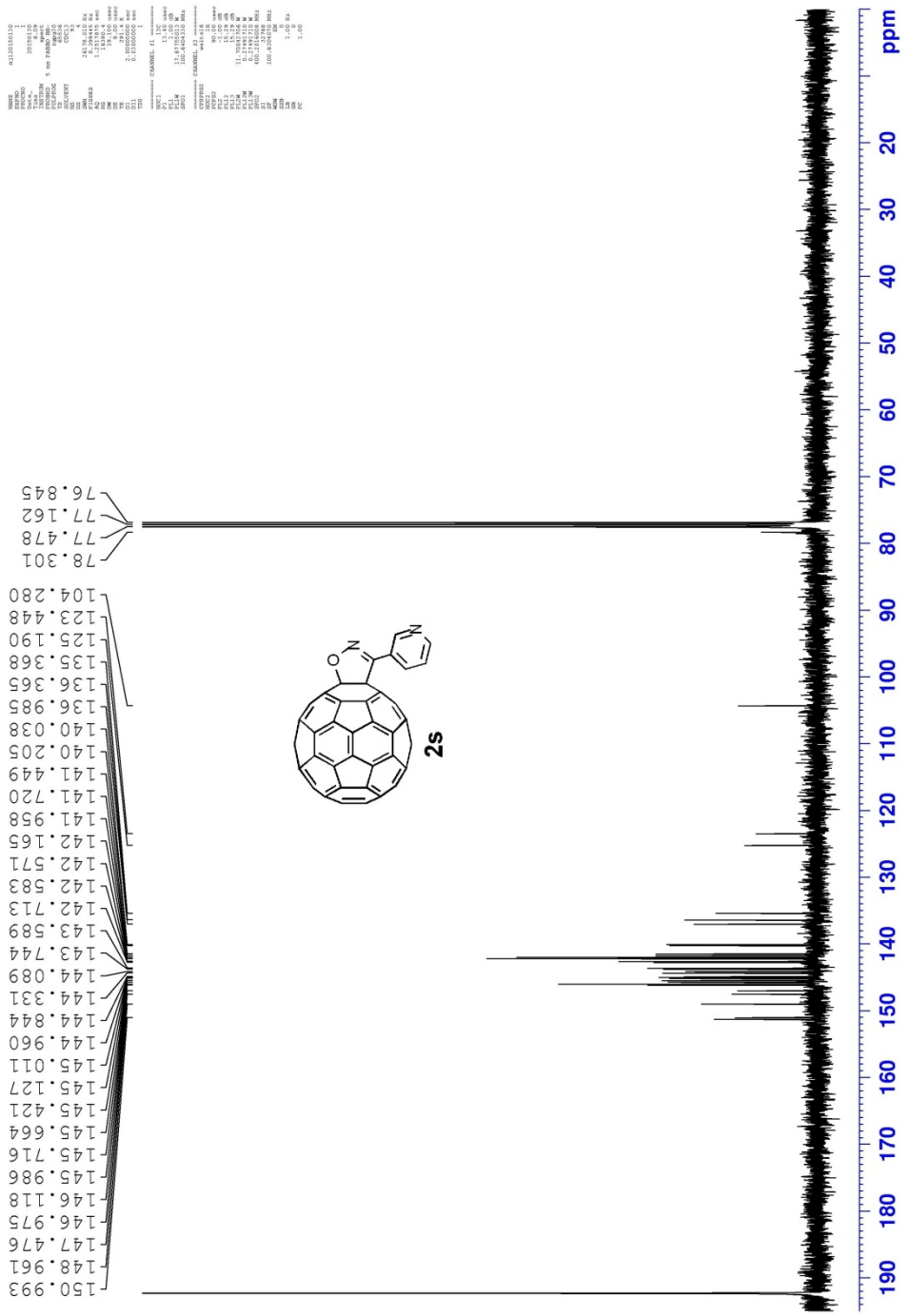
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2r



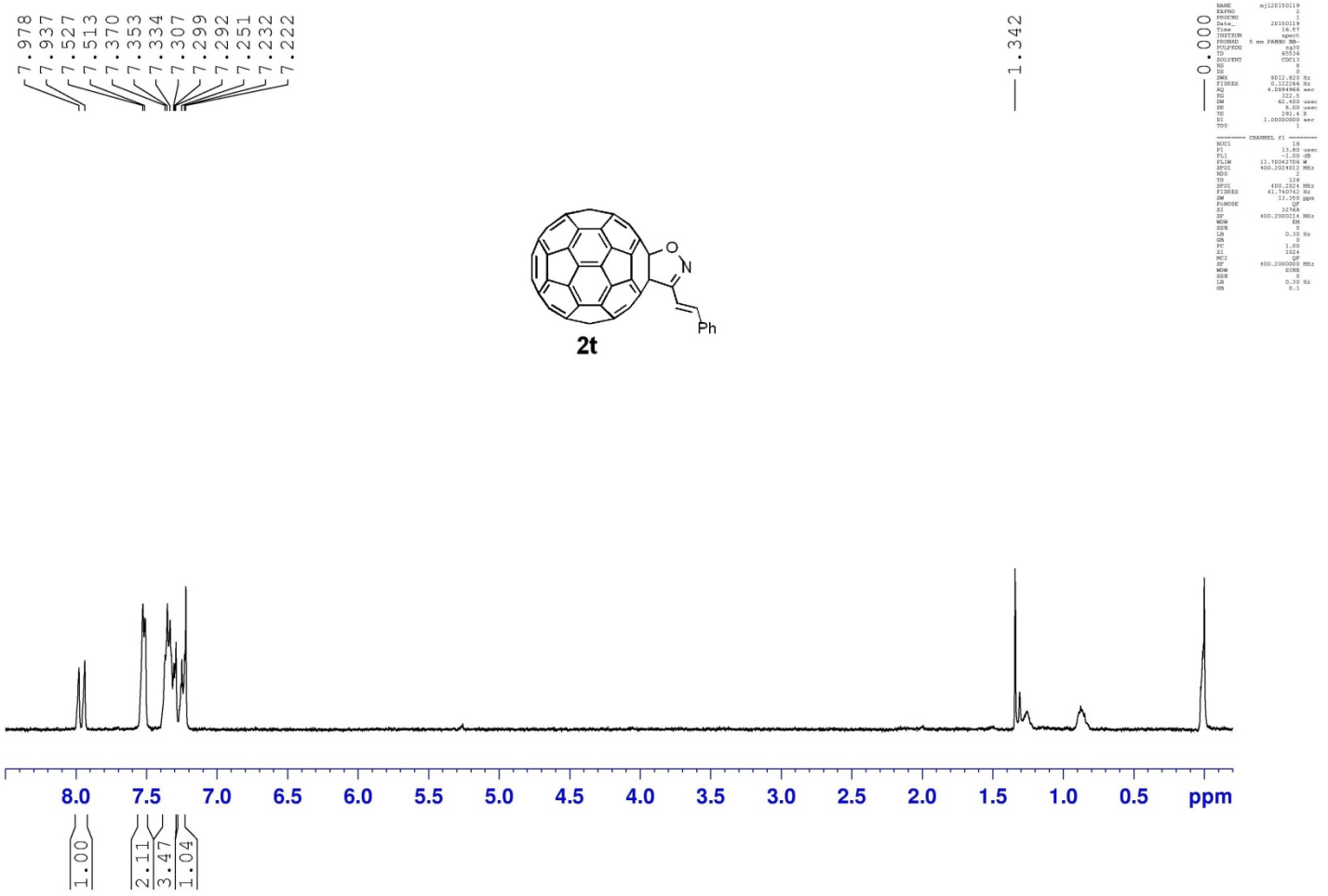
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2s



^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2s

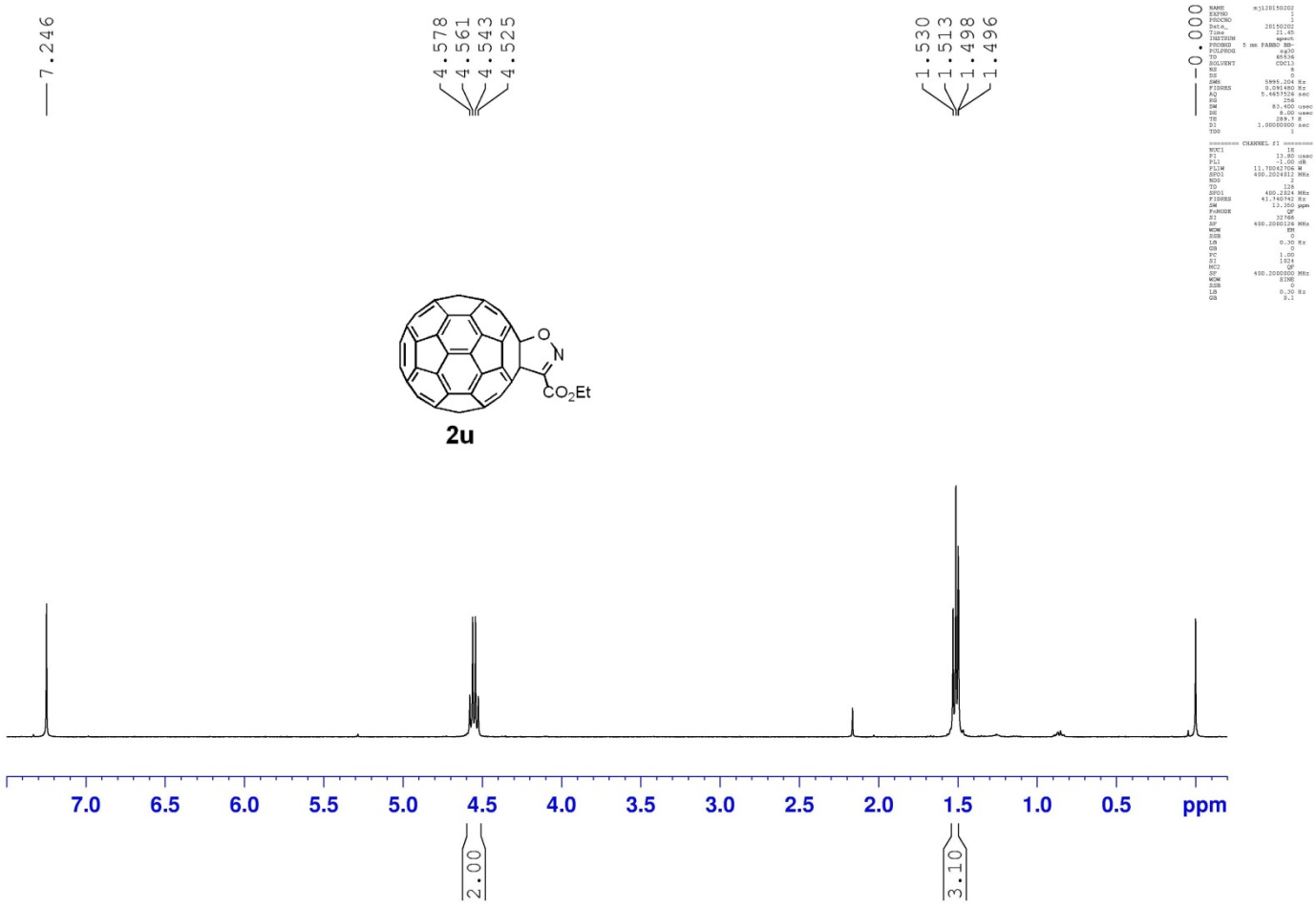


^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2t

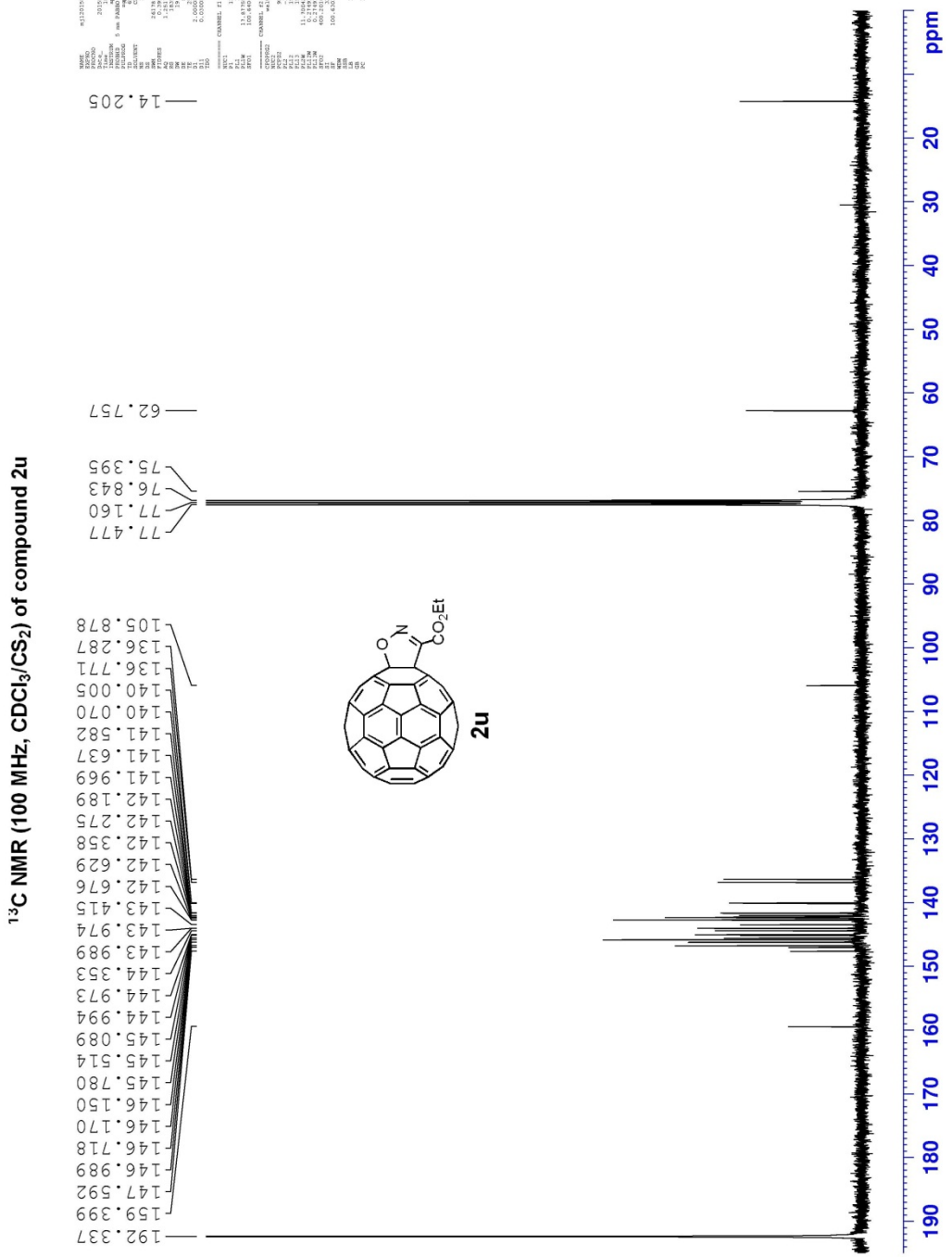


2t

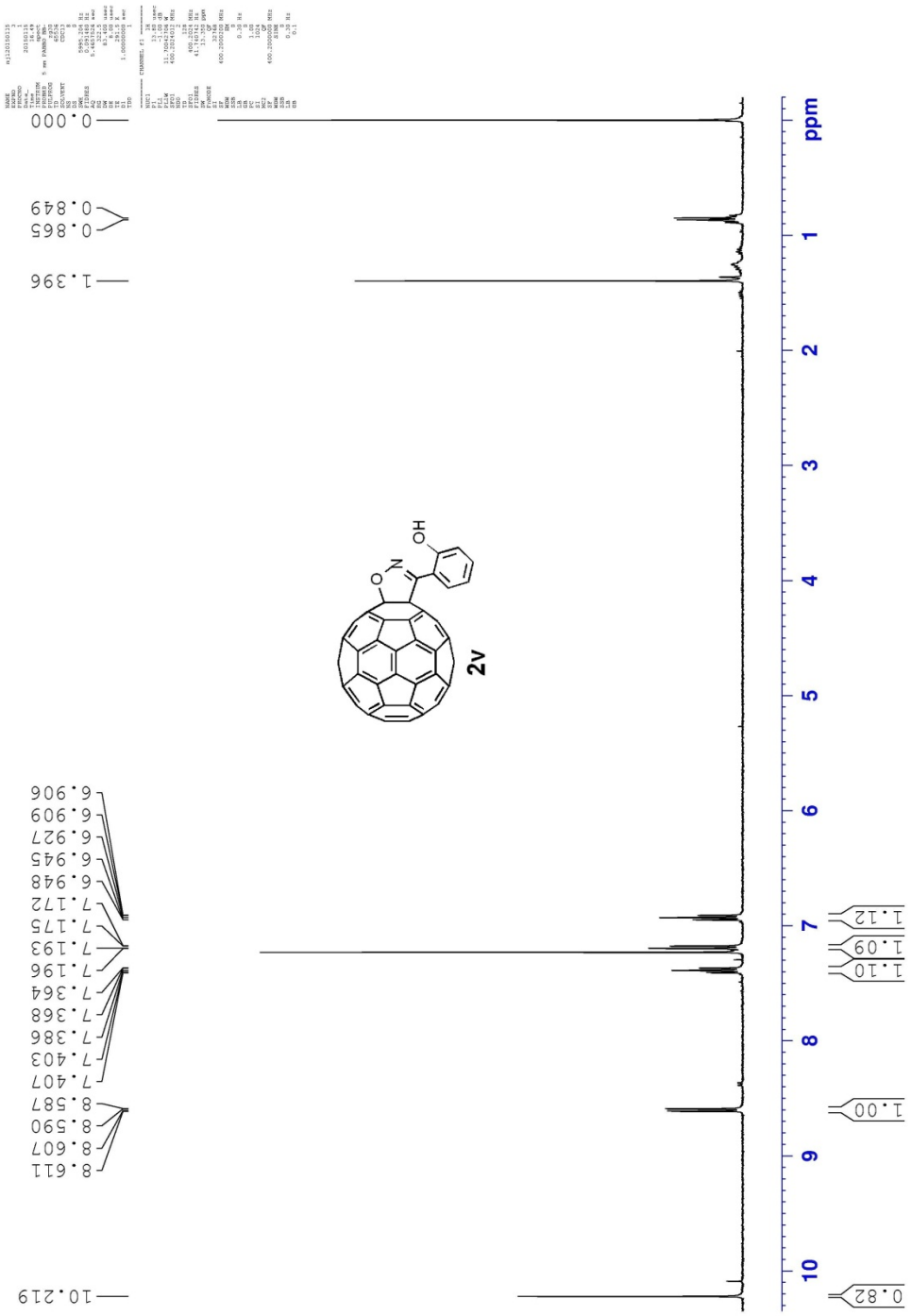
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2u

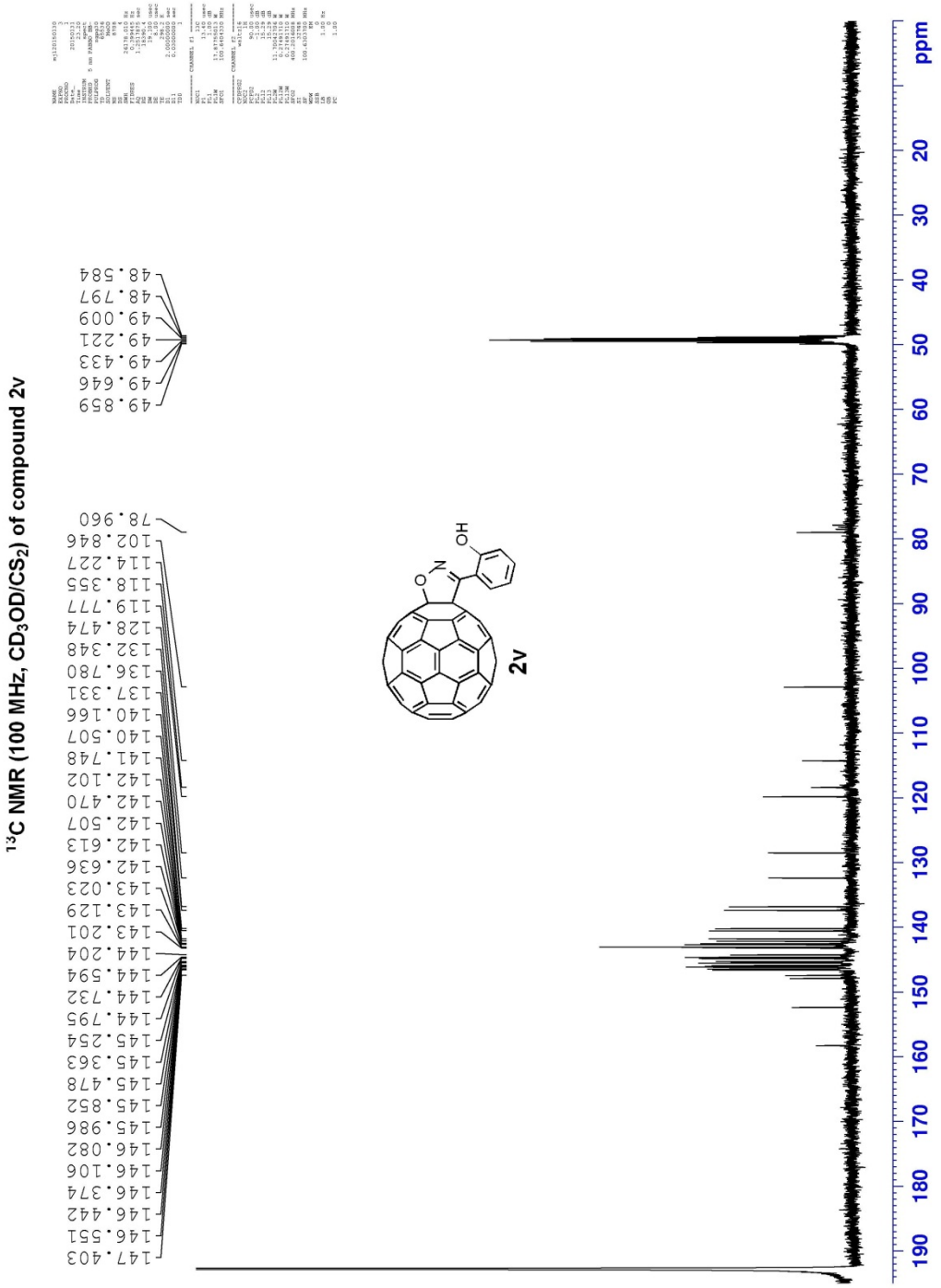


2u

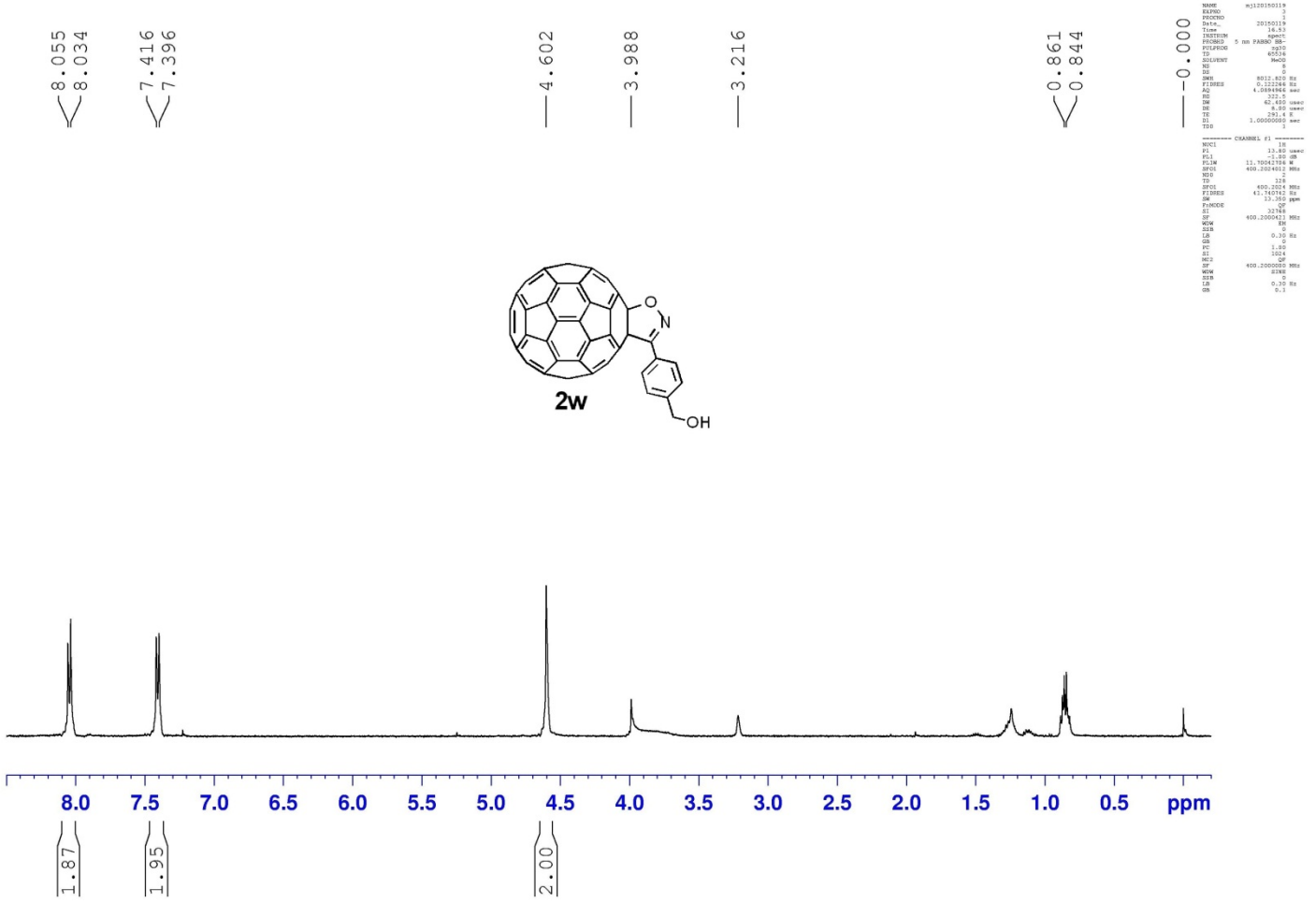

^{13}C NMR (100 MHz, CDCl$_3$/CS$_2$) of compound 2u
192.337
159.399
147.592
146.989
146.718
146.170
146.150
145.780
145.514
145.089
144.994
144.973
144.353
143.989
143.974
143.415
142.676
142.629
142.358
142.275
142.189
141.969
141.637
141.582
140.070
140.005
136.771
136.287
105.878
77.477
77.160
76.843
75.395
62.757
14.205
2u
ppm

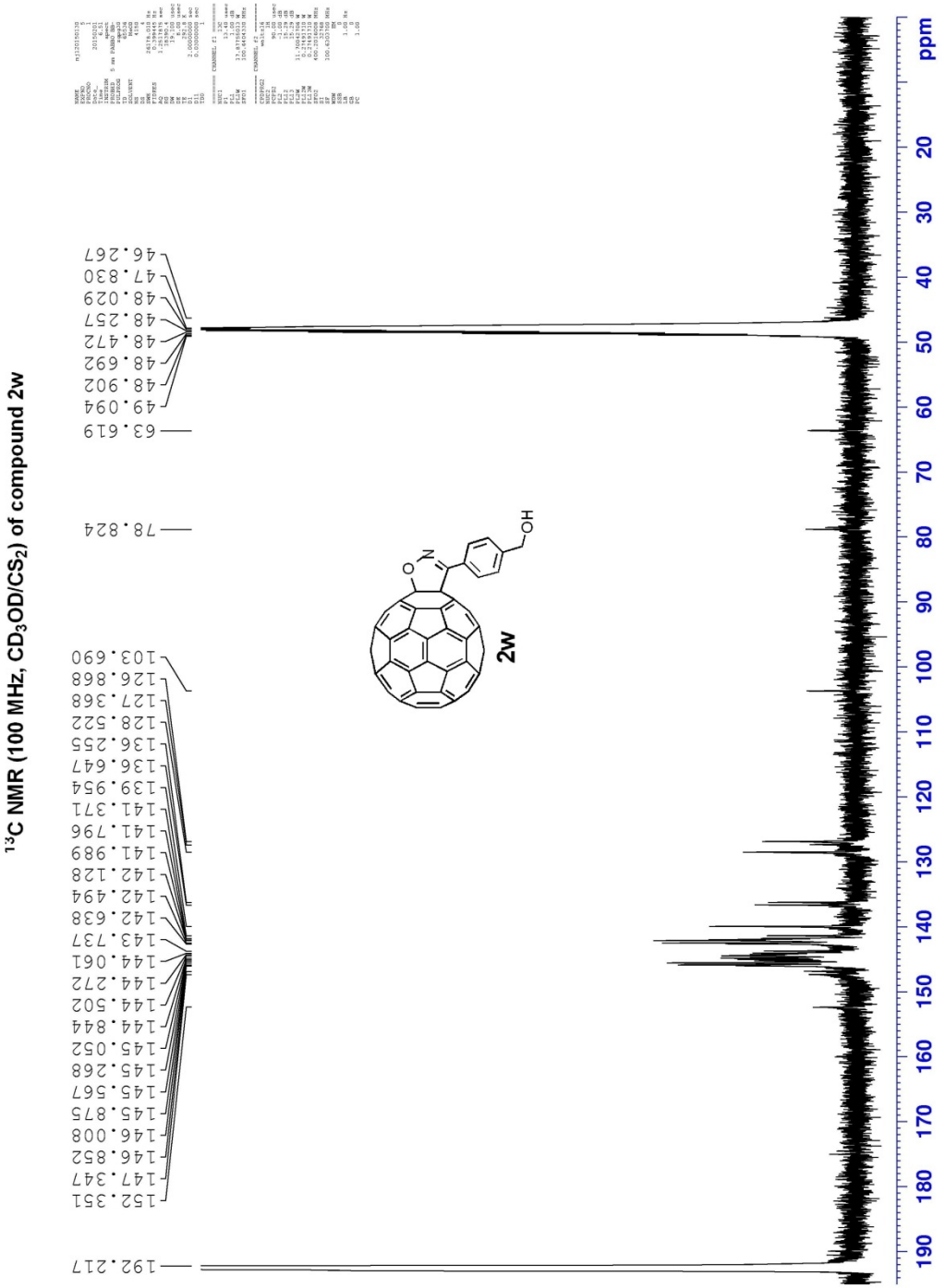
^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2v




^{13}C NMR (100 MHz, CD_3OD/CS_2) of compound 2v
147.403
146.551
146.442
146.374
146.106
146.082
145.986
145.852
145.478
145.363
145.254
144.795
144.732
144.594
144.204
143.201
143.129
143.023
142.636
142.613
142.507
142.470
142.102
141.748
140.507
140.166
137.331
136.780
132.348
128.474
119.777
118.355
114.227
102.846
78.960
49.859
49.646
49.433
49.221
49.009
48.797
48.584
OH
N
O
2v
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

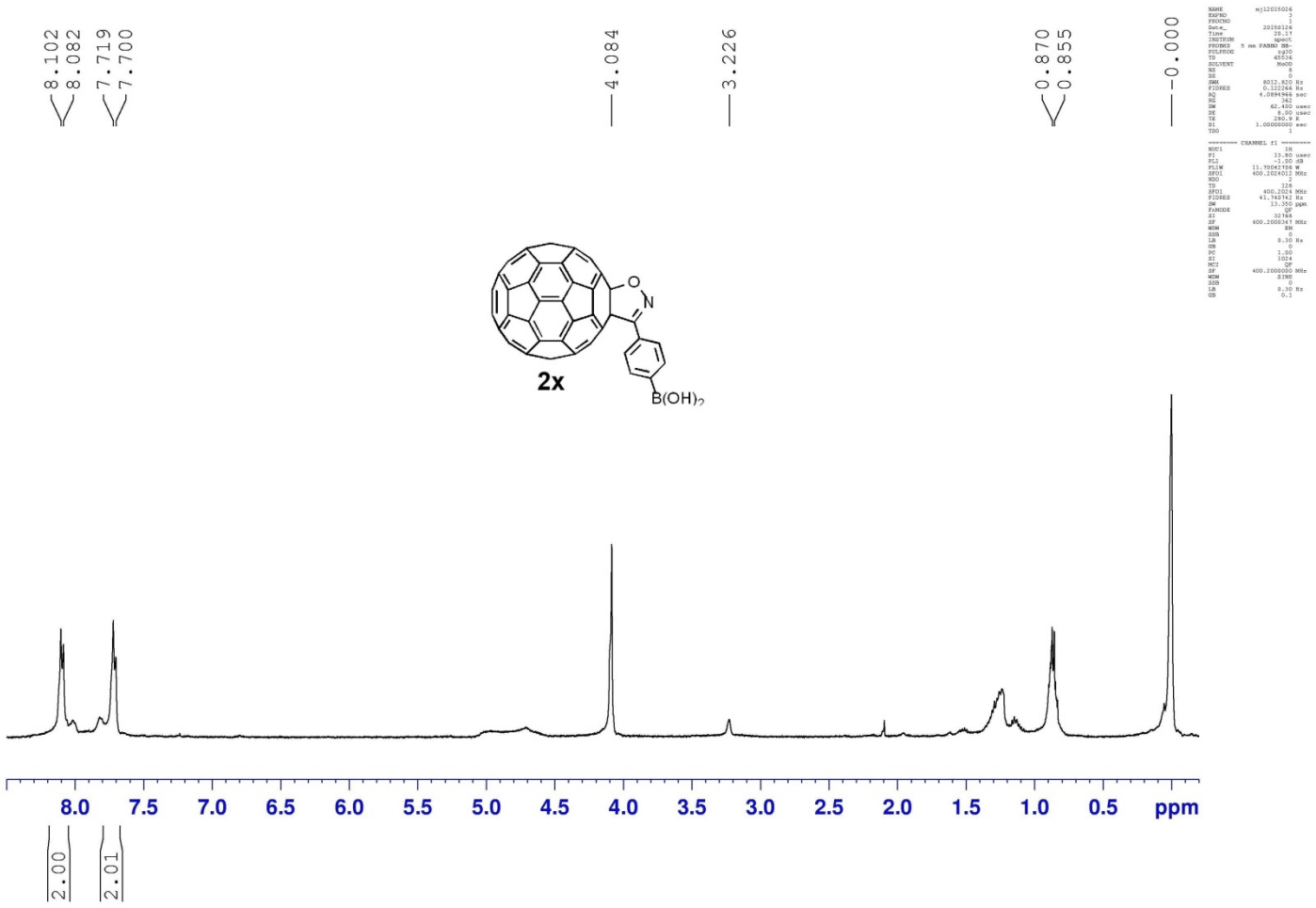
^{1}H NMR (400 MHz, CD_3OD/CS_2) of compound 2w



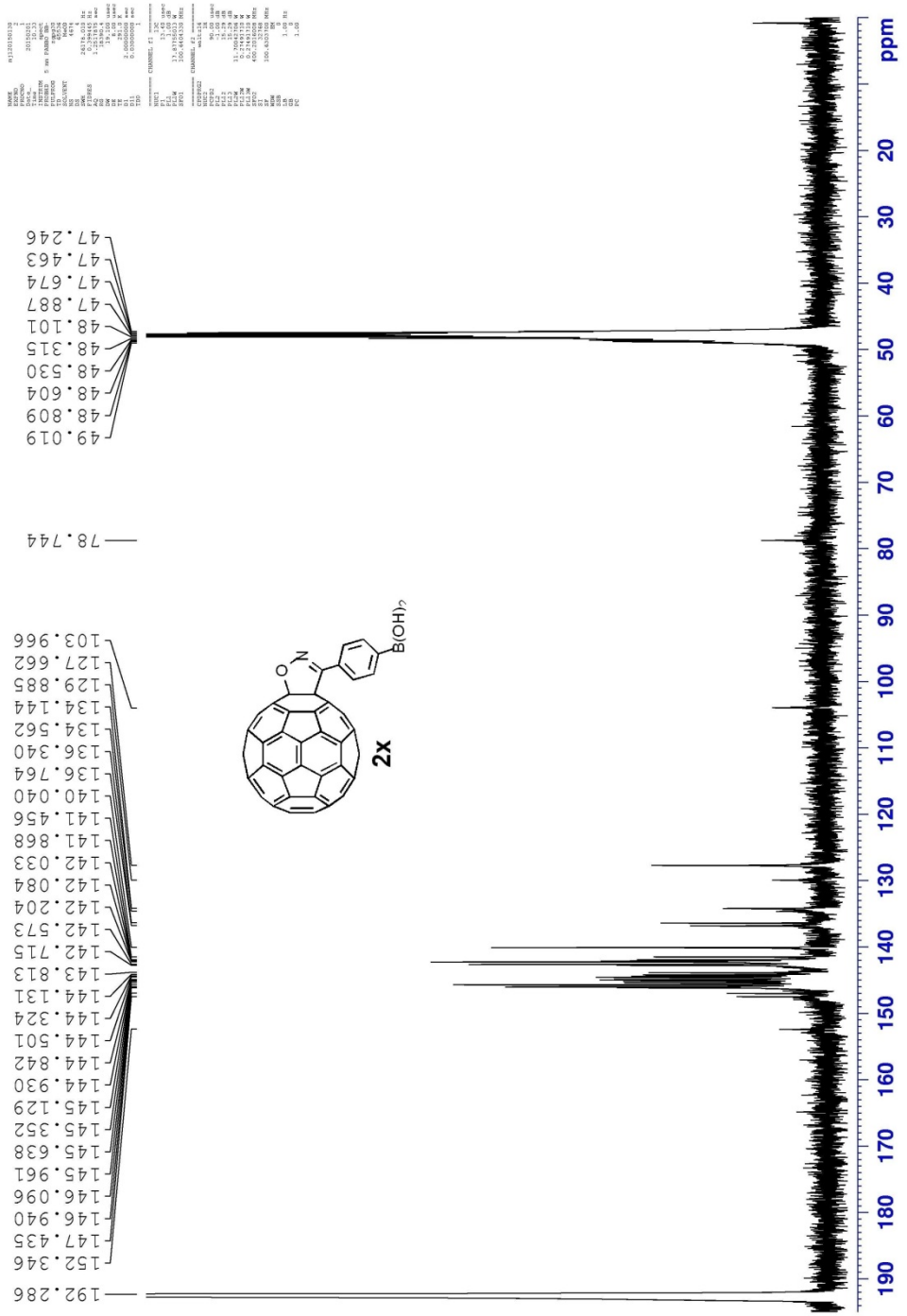


^{13}C NMR (100 MHz, CD_3OD/CS_2) of compound 2w

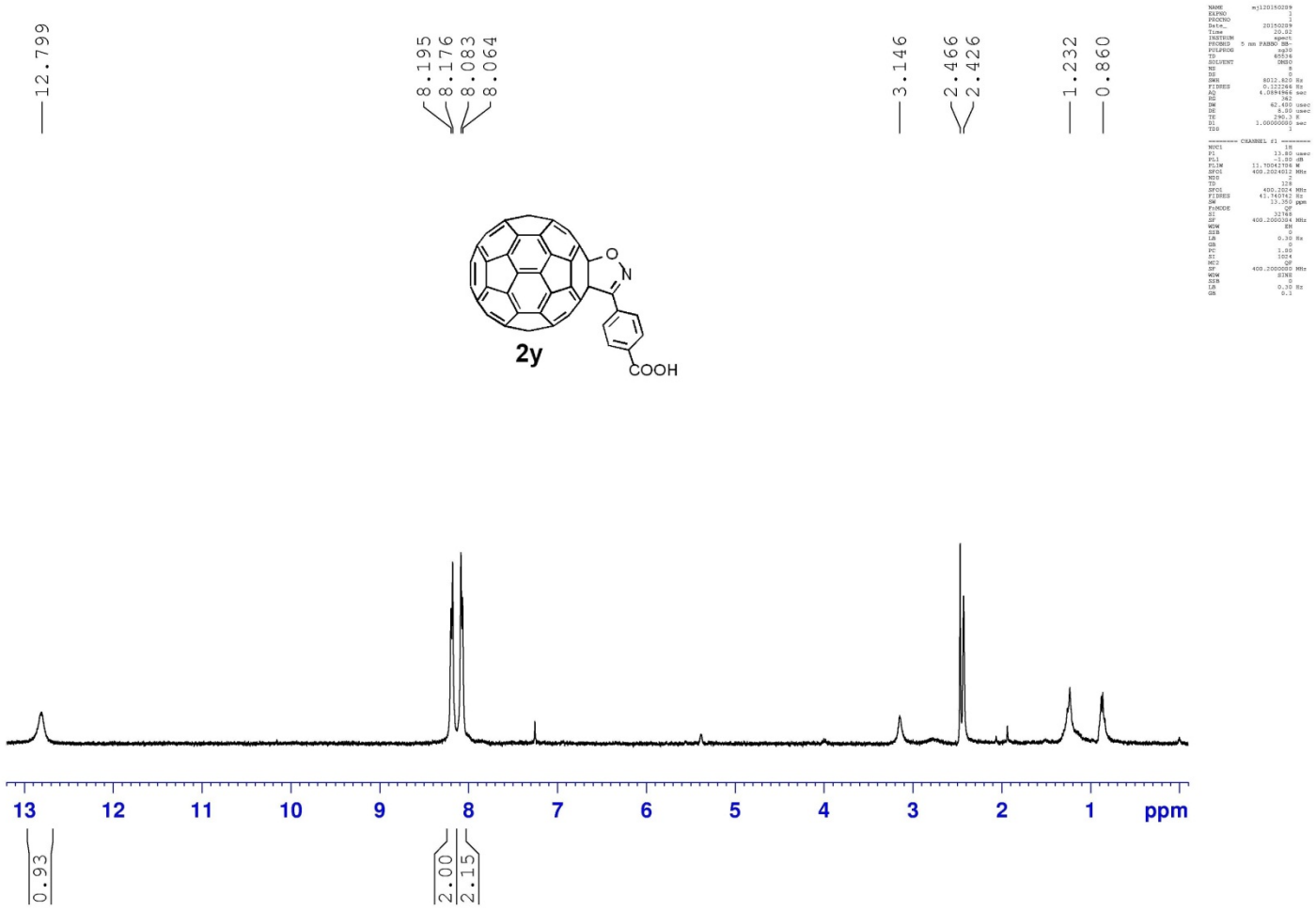
^{1}H NMR (400 MHz, CD_3OD/CS_2) of compound 2x

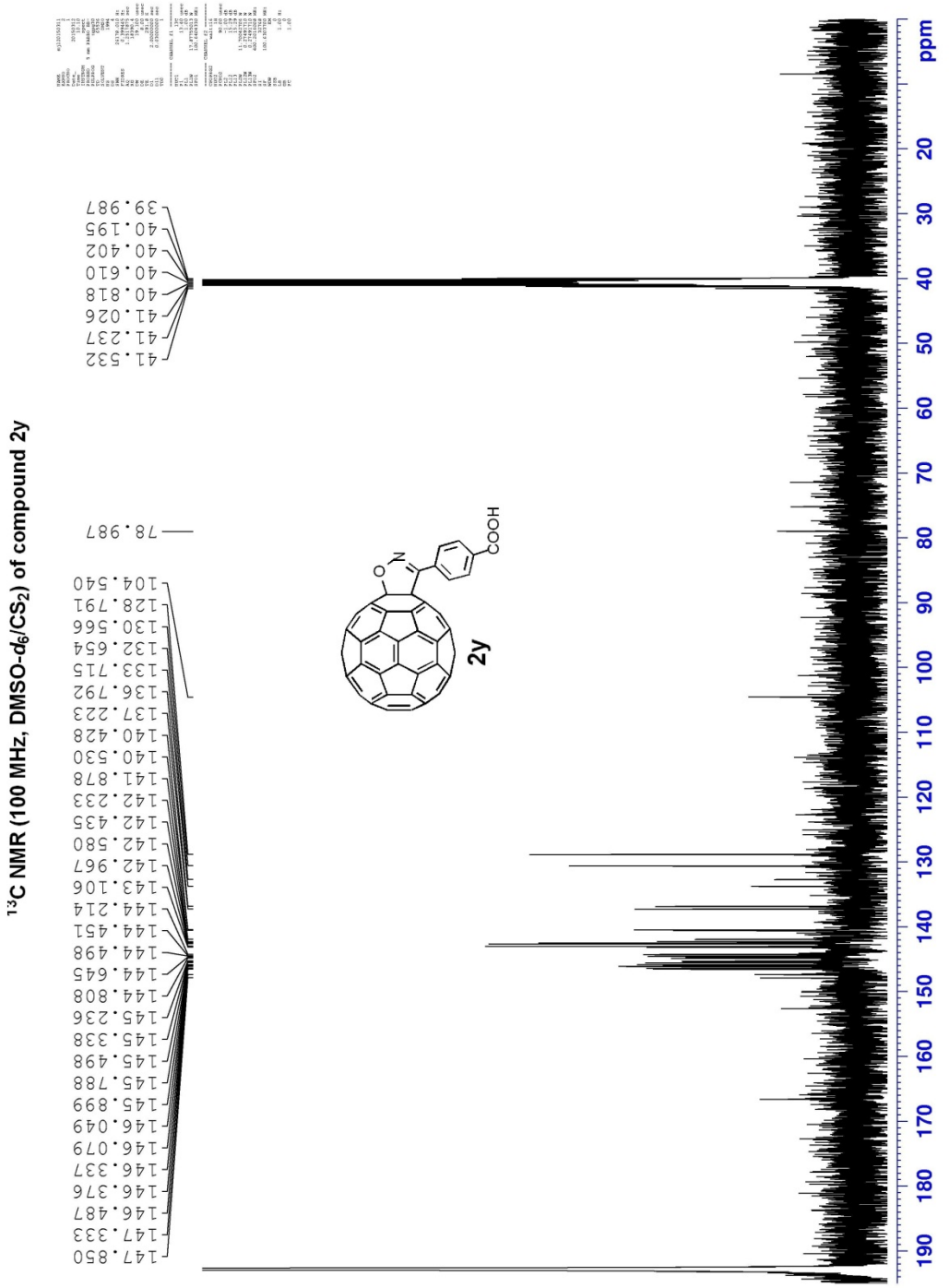


^{13}C NMR (100 MHz, CD_3OD/CS_2) of compound 2x

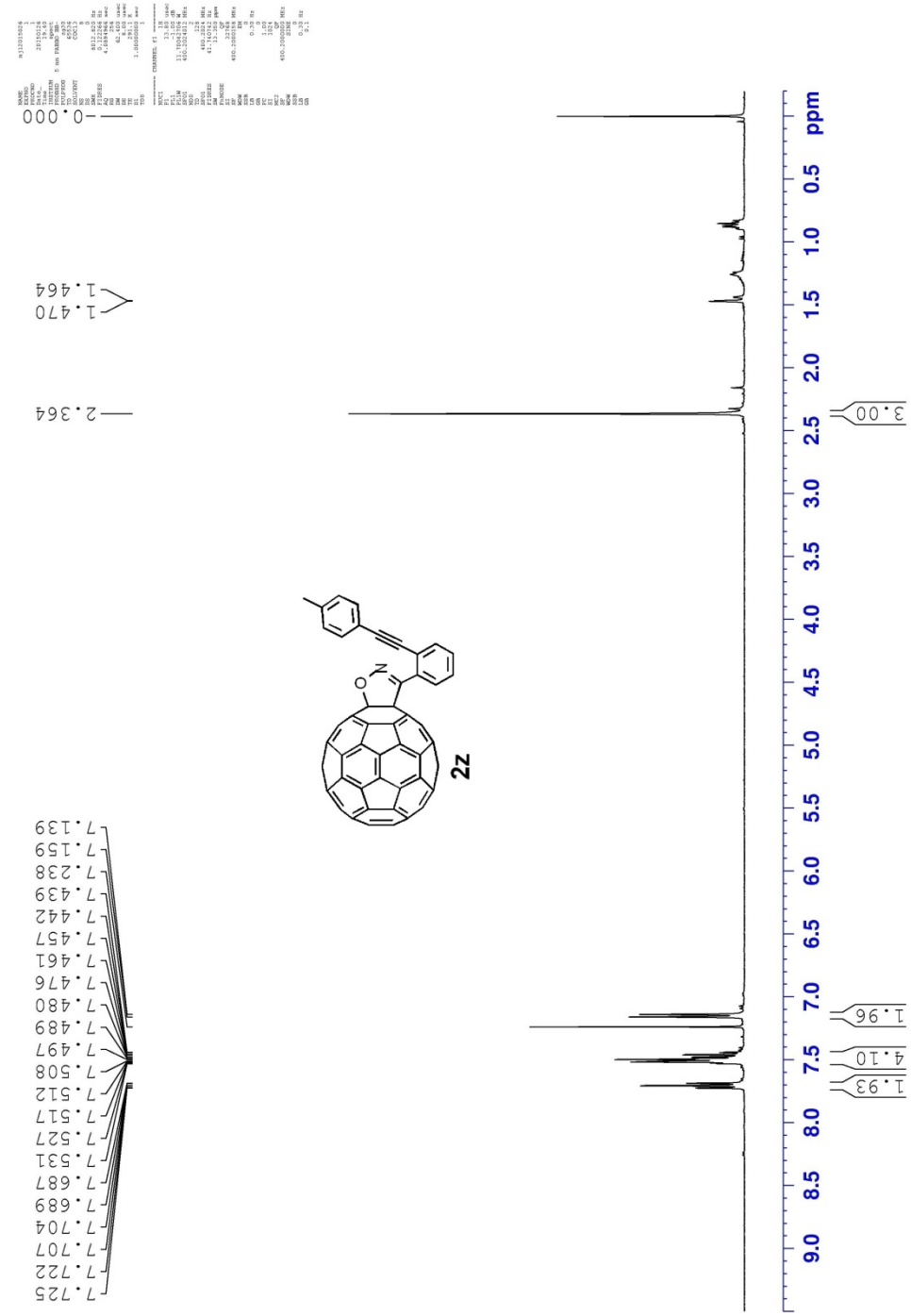


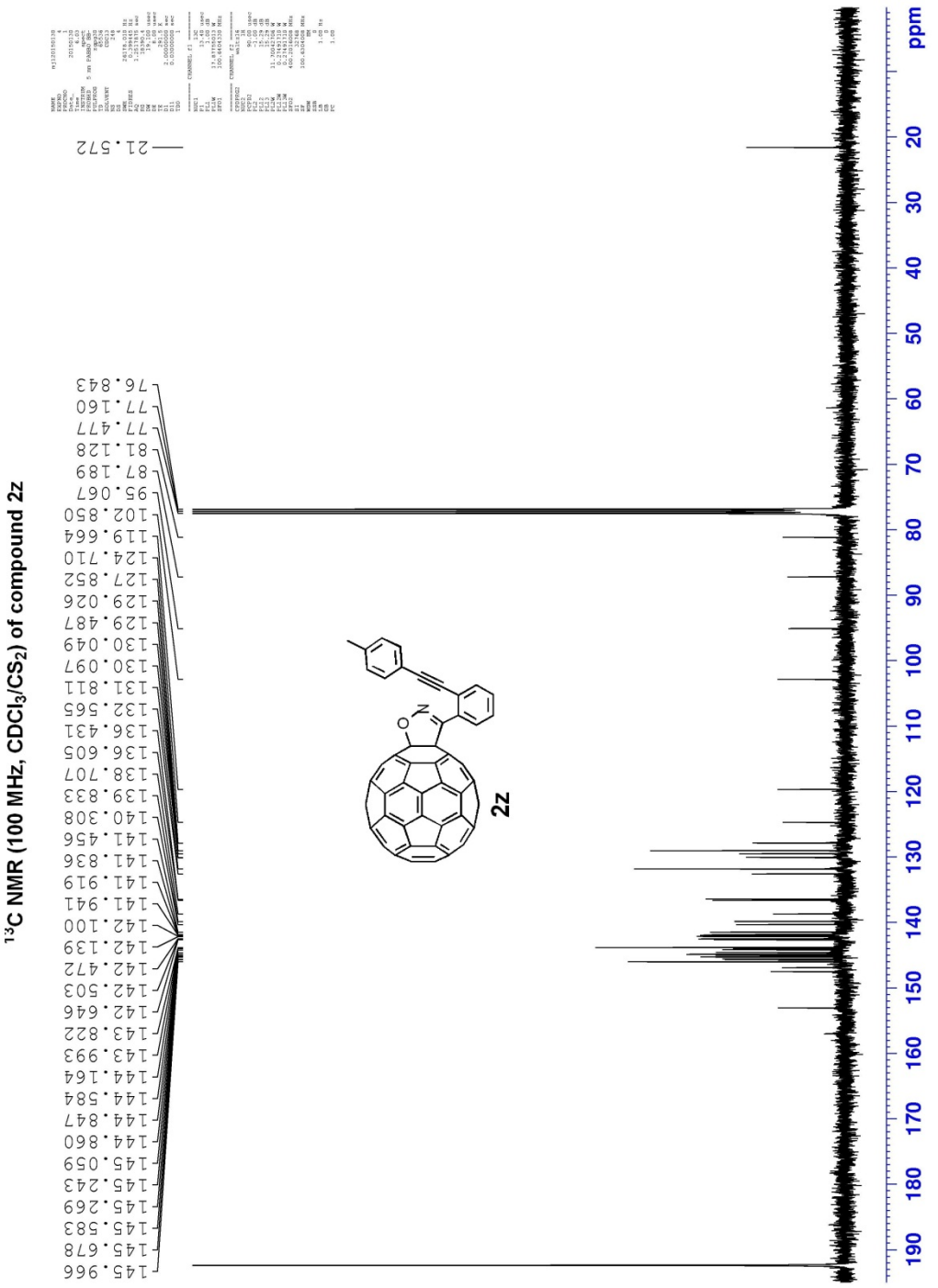
^{1}H NMR (400 MHz, DMSO-d_6/CS_2) of compound 2y



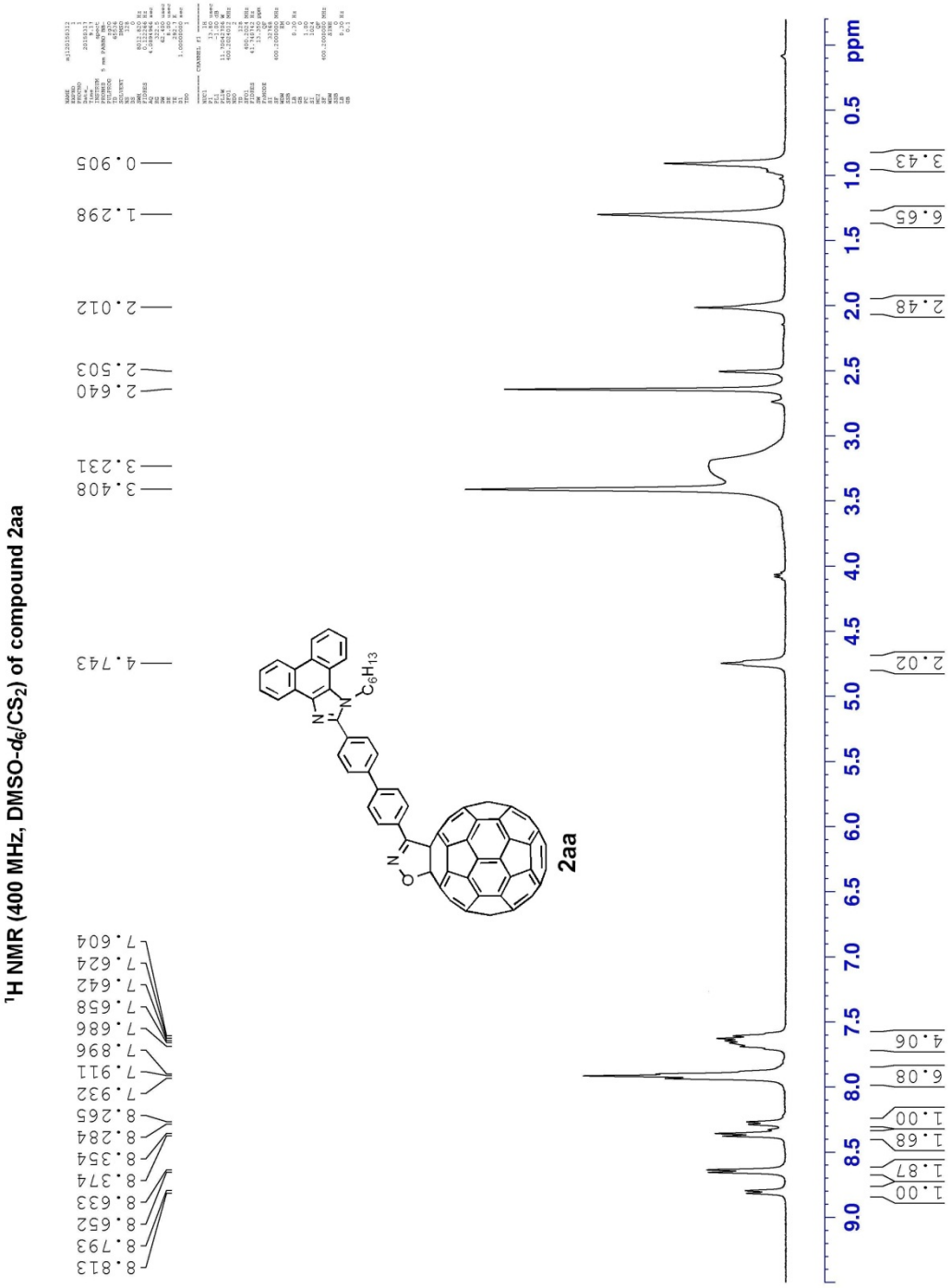

^{13}C NMR (100 MHz, DMSO-d_6/CS_2) of compound 2y
2y
COOH
N
O
ppm

^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2z

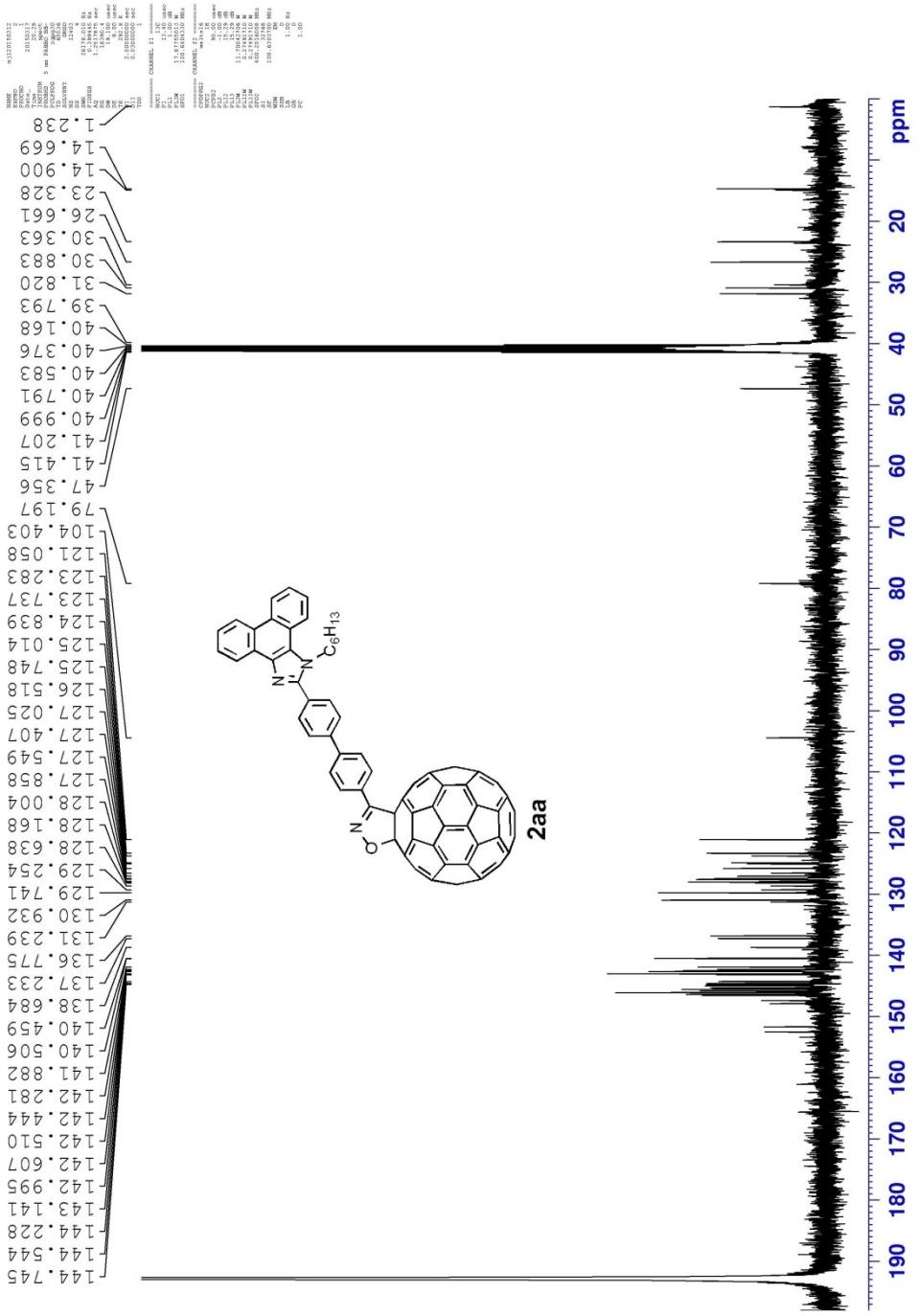



^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2z
2z
21.572
145.966
145.678
145.583
145.269
145.243
145.059
144.860
144.847
144.584
144.164
143.993
143.822
142.646
142.503
142.472
142.139
142.100
141.941
141.919
141.836
141.456
140.308
139.833
138.707
136.605
136.431
132.565
131.811
130.097
130.049
129.487
129.026
127.852
124.710
119.664
102.850
95.067
87.189
81.128
77.477
77.160
76.843
ppm

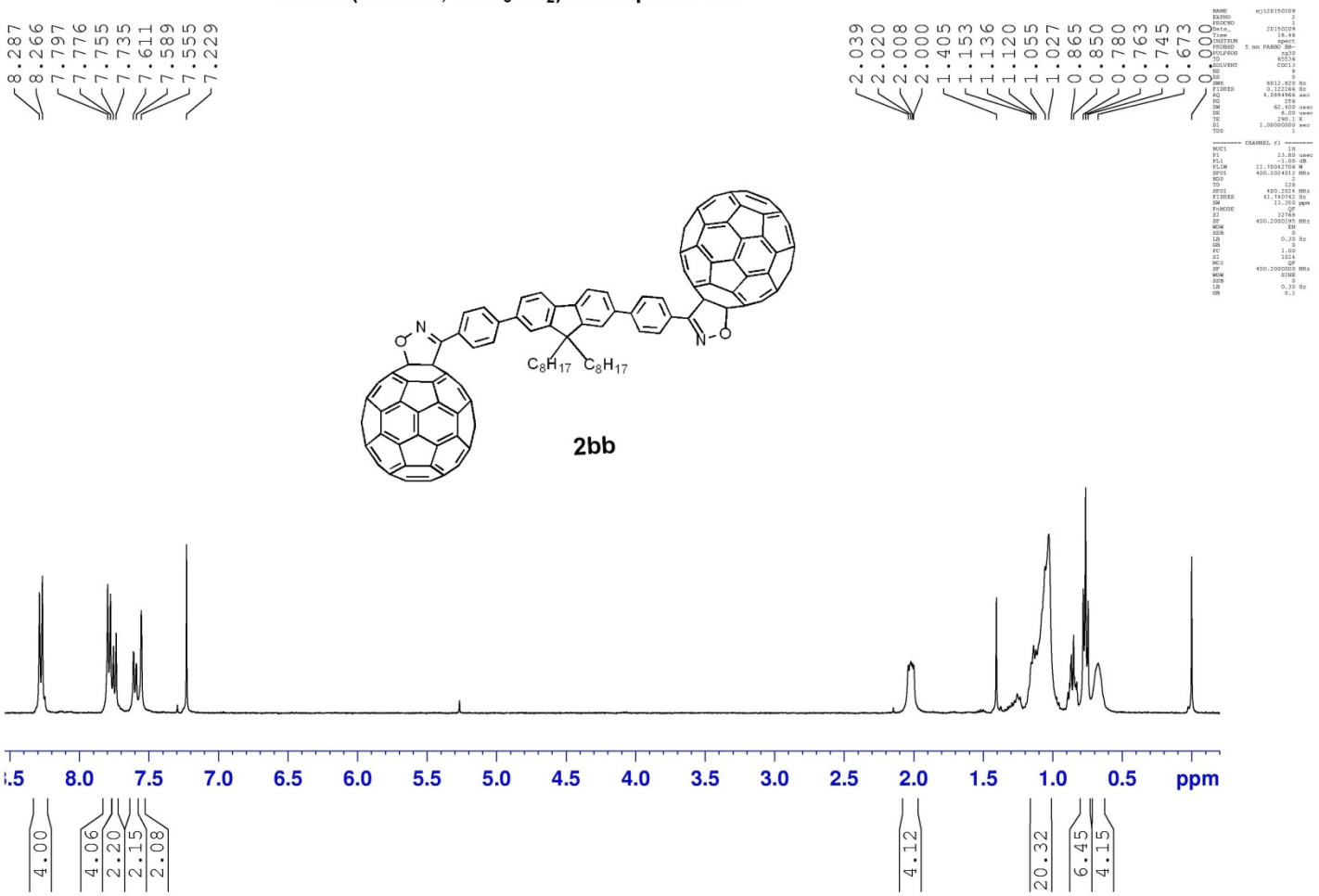
^{1}H NMR (400 MHz, DMSO-d_6/CS_2) of compound 2aa

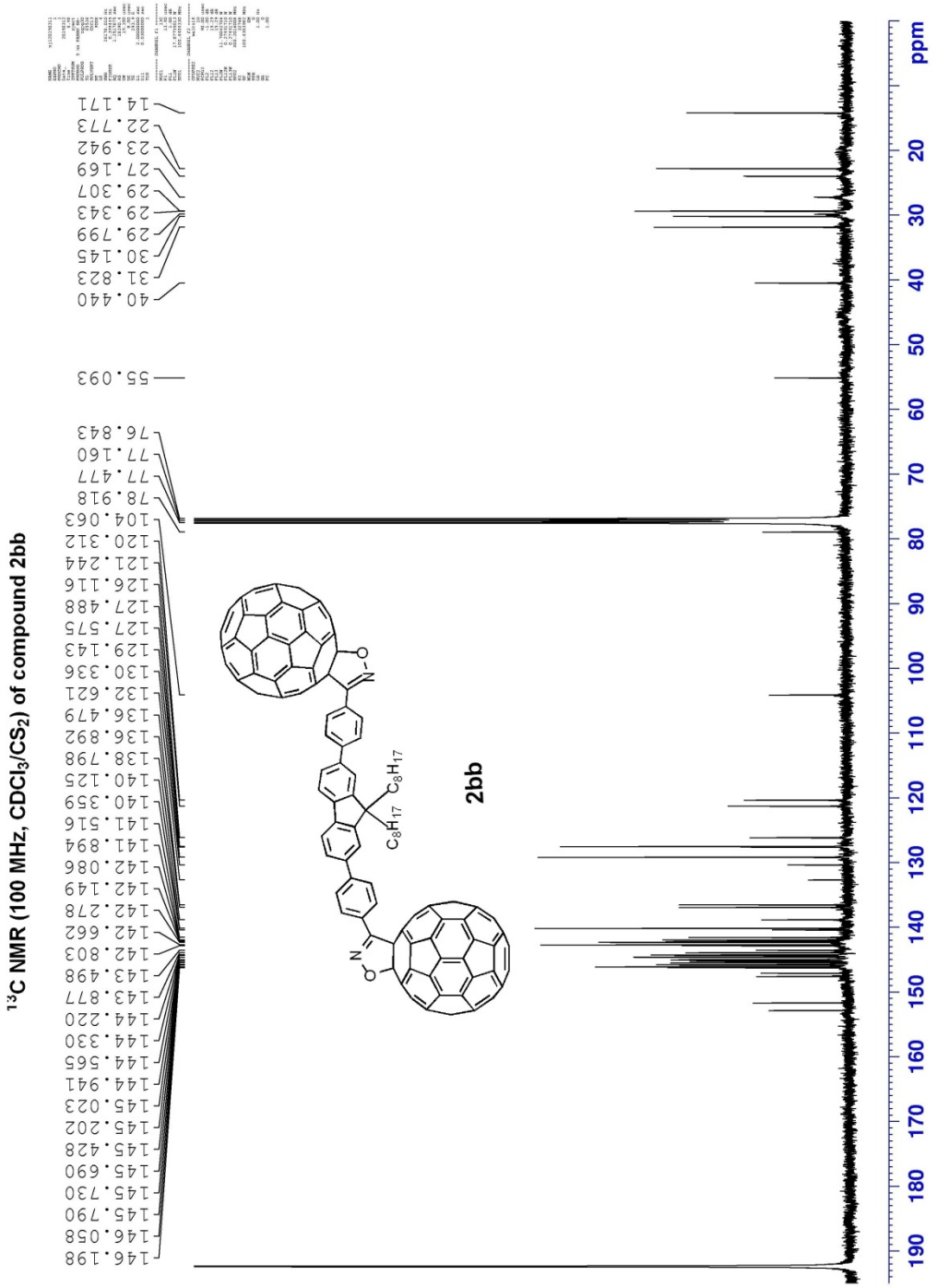


^{13}C NMR (100 MHz, DMSO-d_6/CS_2) of compound 2aa



^{1}H NMR (400 MHz, $CDCl_3/CS_2$) of compound 2bb




^{13}C NMR (100 MHz, $CDCl_3/CS_2$) of compound 2bb
2bb
C_8H_{17}
C_8H_{17}
14.171
22.773
23.942
27.169
29.307
29.343
29.799
30.145
31.823
40.440
55.093
76.843
77.160
77.477
78.918
104.063
120.312
121.244
126.116
127.488
127.575
129.143
130.336
132.621
136.479
136.892
138.798
140.125
140.359
141.516
141.894
142.086
142.149
142.278
142.662
142.803
143.498
143.877
144.220
144.330
144.565
144.941
145.023
145.202
145.428
145.690
145.730
145.790
146.058
146.198
ppm