Rh-catalyzed annulations of *N*-methoxybenzamides and ketenimines: Synthesis of 3-aminoisoindolinones and 3-diarylmethyleneisoindolinones with strong aggregation induced emission properties

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General considerations

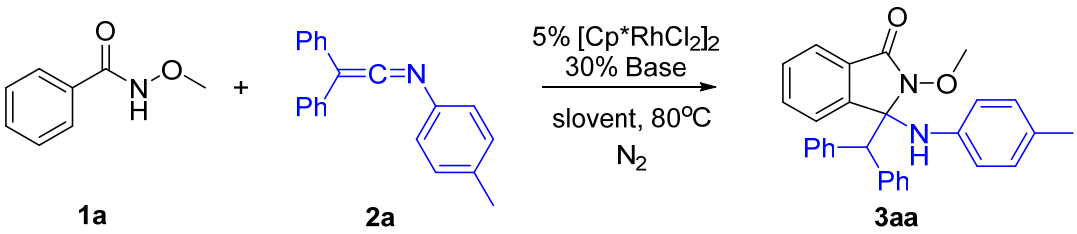
Unless otherwise mentioned, solvents and reagents were purchased from commercial sources and used as received. THF and toluene were distilled from Na before use. MeCN, CH_2Cl_2 and DCE was distilled from CaH_2. Melting points were measured with a micro melting point apparatus. Infrared spectra were obtained with an FTIR spectrometer. NMR spectra were obtained on a Bruker AVANCE DMX500 or 400 spectrometer operating at 500 MHz or 400 MHz for ^{1}H NMR, 126 MHz or 101 MHz for ^{13}C NMR. Unless otherwise noted, all the NMR spectra were recorded at room temperature. Chemical shifts were quoted in parts per million (ppm) referenced to the appropriate solvent peak or 0.0 ppm for tetramethylsilane. Chemical shifts (in ppm) were referenced to tetramethylsilane (δ = 0 ppm) in $CDCl_3$ and d_6-DMSO as an internal standard. ^{13}C NMR spectra were obtained by using the same NMR spectrometers and chemical shifts were reported in ppm referenced to the center line of a triplet at 77.0 ppm of $CDCl_3$ or the center line of a heptet at 39.52 ppm of d_6-DMSO. The following abbreviations are used to describe peak patterns as appropriate: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants J were reported in hertz unit (Hz). High-resolution mass spectra (HRMS) data were obtained with an electron ionization time-of-flight (EI-TOF) mass spectrometer. Flash column chromatography was performed employing 300-400 mesh silica gel. Thin layer chromatography (TLC) was performed on silica gel HSGF254.

The absorption spectra were measured using UV-vis spectrometer (Shimadzu, UV-2450, Japan). FL spectra were recorded on a fluorospectro photometer (Shimadzu, RF-5301PC, Japan) with a xenon lamp excitation source. Particle size analysis was determined at room temperature on a Brookhaven 90 Plus Particle Size Analyzer. TEM micrographs of particle size were obtained using HT7700 transmission electron microscope by copper net as a supporter. SXRD spectra was obtained using Xeuss with SAXS/WAXS system. Distilled water was filtered through a membrane with 0.22μm pore size. Tetrahydrofuran (THF) was distilled from sodium prior to use.

Absorption and emission spectra of these compounds in solutions were measured in THF with a concentration scale of 10^{-5} M. Nanoparticles were formed by adding a certain volume of water into its dilute THF solution with a rotlet stirring sharply for 10 minutes, all the solution concentration was same. The absorption spectra, FL spectra and particle size distribution (DLS) of the resulting solutions were then performed immediately.

Preparation of TEM sample**:** solution of nanoparticles was added to a copper net with carbon membrane. Then the copper net was dried by infrared lamp.

Table S1 Optimization of the [4+1] reaction conditions[a]



1a **2a** **3aa**

Entry	Solvent	Base(equiv.)	T(°C)	Atom-sphere	Cat. (%)	Add-itive	**1a**:**2a**	Yield(%)[b]
1	DCE	CsOAc	80	N_2	5	-	1:2	46
2	CH_3CN	CsOAc	80	N_2	5	-	1:2	trace
3	Toluene	CsOAc	80	N_2	5	-	1:2	28
4	1,4-Dioxane	CsOAc	80	N_2	5	-	1:2	Trace
5	CH_2Cl_2	CsOAc	80	N_2	5	-	1:2	40
6	Acetone	CsOAc	80	N_2	5	-	1:2	35
7	DCE	CsOAc	r.t.	N_2	5	-	1:2	N.D.
8	DCE	CsOAc	50	N_2	5	-	1:2	21
9	DCE	CsOAc	100	N_2	5	-	1:2	45
10	DCE	CsOAc	80	air	5	-	1:2	16
11	DCE	CsOAc	80	O_2	5	-	1:2	Trace
12[c]	DCE	CsOAc	80	N_2	5	-	1:2	40
13	DCE	NaOAc	80	N_2	5	-	1:2	17
14	DCE	KOAc	80	N_2	5	-	1:2	45
15	DCE	$Cl_3CCOONa$	80	N_2	5	-	1:2	55
16	DCE	PivOCs	80	N_2	5	-	1:2	39
17	DCE	$Cl_3CCOOCs$	80	N_2	5	-	1:2	78
18	DCE	Cl_3CCOOK	80	N_2	5	-	1:2	70
19[d]	DCE	$Cl_3CCOOCs$	80	N_2	5	-	1:2	N.D.
20[e]	DCE	$Cl_3CCOOCs$	80	N_2	5	-	1:2	N.D.
21	DCE	None	80	N_2	5	-	1:2	N.D.
22	DCE	$Cl_3CCOOCs$	80	N_2	5	H_2O(1eq.)	1:2	N.D.
23	DCE	$Cl_3CCOOCs$	80	N_2	5	4A MS[f]	1:2	82
24	DCE	$Cl_3CCOOCs$	80	N_2	5	4A MS[f]	1:1.5	81
25	DCE	$Cl_3CCOOCs$	80	N_2	5	4A MS[f]	1:1.2	80
26	DCE	$Cl_3CCOOCs$	80	N_2	5	4A MS[f]	1:1	71
27	DCE	$Cl_3CCOOCs$	80	N_2	5	4A MS[f]	1.2:1	81
28	DCE	$Cl_3CCOOCs$	80	N_2	5	4A MS[f]	1.5:1	82
29	DCE	$Cl_3CCOOCs$	80	N_2	5	4A MS[f]	2:1	84
30	DCE	$Cl_3CCOOCs$	80	N_2	4	4A MS[f]	1.2:1	76
31	DCE	$Cl_3CCOOCs$	80	N_2	2.5	4A MS[f]	1.2:1	70

[a]Reaction condition: **1a** (0.3 mmol), **2a** (0.6 mmol),$[Cp^*RhCl_2]_2$ (0.015 mmol), Base (0.09 mmol), Solvent (2 mL), N_2, 80 °C, 10 h. [b]Isolated yield. [c]CsOAc(1 equiv). [d]2% $[Cp^*IrCl_2]_2$ instead of $[Cp^*RhCl_2]_2$. [e]5% $[RuCl_2(p\text{-cymene})]_2$ instead of $[Cp^*RhCl_2]_2$. [f]4Å MS (60 mg).

Table S2. Photophysical properties of compounds **4a-p** and **5a-d**

	λ_{abs}(nm)				λ_{em}(nm)		Φ_F (%)		
	THF, ($\varepsilon/10^4$)[a]	Nano, ($\varepsilon/10^4$)[b]	Cal.[c]	% Δ[d]	Nano	Solid	Φ_{sol}[e]	Φ_{nano}[e]	Φ_{nano}/Φ_{sol}
4a	373(2.37)	375(1.87)	375	0.7	516	507	0.028	2.572	91.9
4b	373(1.78)	377(1.53)	375	0.5	514	482	0.028	2.571	91.7
4c	372(1.85)	377(1.62)	381	2.3	523	507	0.022	1.773	80.6
4d	376 (1.99)	381(1.62)	372	-1.1	508	510	0.034	2.969	87.3
4e	372(2.09)	379(1.81)	378	1.7	515	498	0.045	4.296	95.5
4f	405(2.61)	407(1.99)	395	-2.6	533	491	0.031	1.352	43.6
4g	375(1.98)	377(1.54)	378	0.8	518	500	0.049	3.324	67.8
4h	394(2.64)	398(2.00)	394	0.0	542	519	0.077	12.571	163.3
4i	412(2.24)	415(1.55)	403	-2.3	535	534	0.053	2.294	43.3
4j	387(2.29)	388(1.86)	381	-1.6	518	501	0.076	7.865	103.5
4k	374(2.09)	378(1.73)	380	1.7	516	525	0.046	1.967	42.8
4l	375(1.95)	379(1.65)	370	-1.5	515	527	0.041	2.102	51.3
4m	362(1.21)	371(0.97)	363	0.3	502	511	0.039	1.374	35.2
4n	357(1.47)	362(1.19)	356	-0.2	492	499	0.031	0.554	18
4o	364(1.89)	370(1.34)	362	-0.5	501	490	0.017	1.291	77.3
4p	362(1.81)	362(1.57)	355	-2.1	492	460	0.025	0.660	26.4
5a	348(1.57)	349（0.10)	346	-0.4	454	449	0.514	4.680	9.1
5b	348(1.60)	349(0.16)	349	0.2	455	458	0.025	0.657	26.3
5c	349(1.73)	354(0.07)	351	0.6	441	451	0.269	8.797	32.7
5d	343(1.17)	348(0.16)	343	0.0	445	446	0.045	1.606	35.7

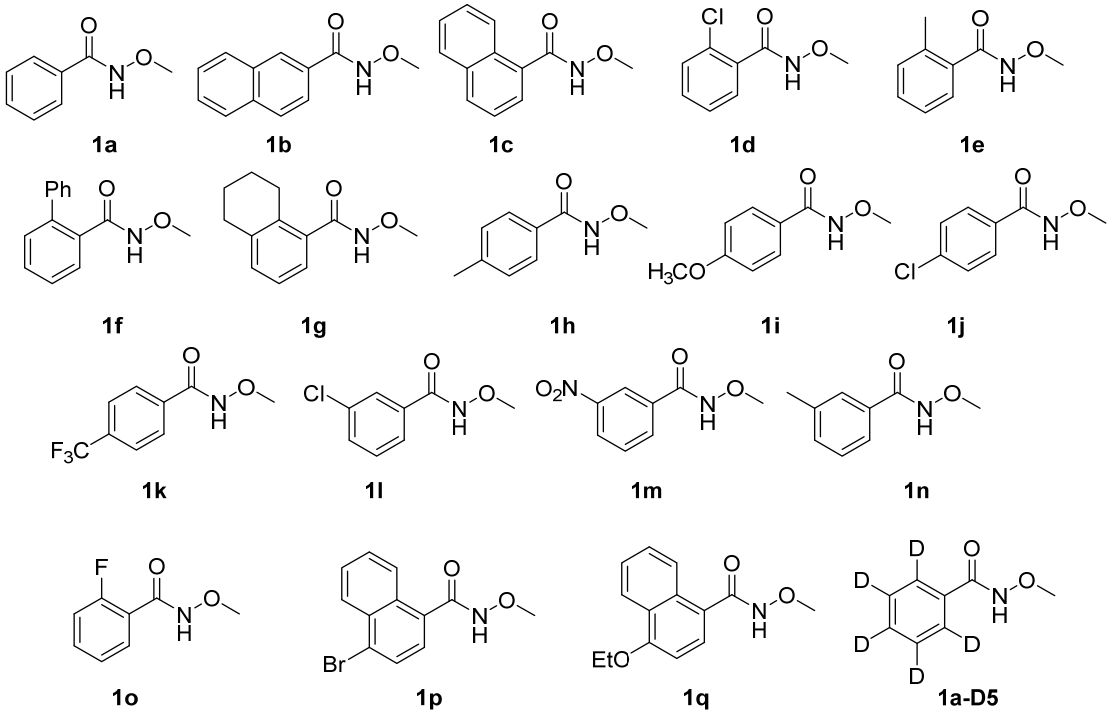
[a]Absorption spectra were measured at a concentration scale of 10^{-5} in THF solution; [b]Nano-suspensions were obtained by adding water into dilute THF solution (water/THF = 95/5); [c]Calculated absorption wavelengths were obtained from HOMO-LUMO gaps using the B3LYP functional and the 6-31G* basis set. [d]% Δ = 100*(Cal.-THF)/Cal. [e]Quantum yields were calculated referring to the standard of Quinine sulfate (Φ =0.54 in 0.1M H_2SO_4).

Preparation of Cesium 2,2,2-trichloroacetate and potassium 2,2,2-trichloroacetate

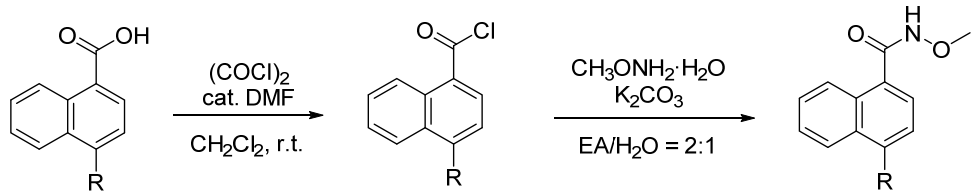
Cesium pivalate was prepared according to the reported procedure.[1]

2,2,2-Trichloroacetic acid (3.24 g, 20 mmol) and cesium carbonate (3.28 g, 10 mmol) or potassium carbonate (1.38 g, 10 mmol) was dissolved in CH_3OH (20 mL). The resulting solution was stirred during 30 min at 50 °C. The solvent was then evaporated under reduced pressure until a white solid appeared. The obtained solid was crushed into a fine powder and dry under high vacuum at 80 °C during 8 h.

General Procedure for the Preparation of N-methoxybenzamides 1



Compounds **1a – 1o, 1a-D5** as well as new compounds **1p** and **1q** were prepared according to the literature procedures.[2]

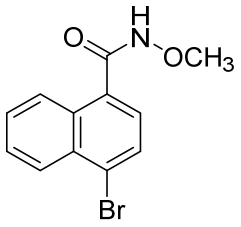


[1] (a) M. A. Campo and R. C. Larock, *Org. Lett.* **2000**, *2*, 3675. (b) T. Piou, A. Bunescu, Q. Wang, L. Neuville and J. Zhu, *Angew. Chem., Int. Ed.* **2013**, *52*, 12385.

[2] (a) N. Guimond, C. Gouliaras and K. Fagnou, *J. Am. Chem. Soc.* **2010**, *132*, 6908. (b) S. Rakshit, C. Grohmann, T. Besset and F. Glorius, *J. Am. Chem. Soc.* **2011**, *133*, 2350. (c) L. E. Fisher, J. M. Caroon, Jahangir, S. R. Stabler, S. Lundberg and J. M. Muchowsk, *J. Org. Chem.* **1993**, *58*, 3643.

4-Substituted-1-naphthoic acid (2 mmol) was dissolved in dry dichloromethane (5 mL), followed by addition of a drop of DMF and oxalyl chloride (0.21 mL, 1.49 g/mL, 2.4 mmol). After 2 hour, the volatile and solvent were evaporated and the resulting acid chloride was obtained and used directly for the subsequent reactions without further purification. To the crude product was then added K_2CO_3 (4 mmol), $MeONH_2$·HCl (2.4 mmol), ethyl acetate (5 mL), and water (2.5 mL) sequentially. The resulting mixture was stirred for 16 h at r.t. and extracted twice with ethyl acetate. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under reduced pressure. The products were obtained as pure solids without need for purification.

4-Bromo-*N*-methoxy-1-naphthamide (1p)



Following the general procedure afforded **1p** (530 mg, 95 %) as a white solid, **m.p.** 140 – 146 °C.

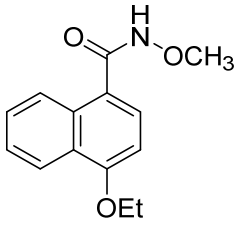
^{1}H NMR (400 MHz, DMSO-d_6) δ 11.80 (s, 1H), 8.22 (td, J = 8.9, 1.5 Hz, 2H), 7.96 (d, J = 7.6 Hz, 1H), 7.82 – 7.68 (m, 2H), 7.52 (d, J = 7.6 Hz, 1H), 3.81 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 164.6 , 131.8 , 131.2 , 131.1 , 129.2 , 128.4 , 128.1 , 126.8 , 126.3 , 125.7 , 124.3 , 63.5 .

IR (ATR): 3140, 2979, 1640, 1563, 1512, 1439, 1296, 1256, 1193, 1072, 1006, 920, 846, 818, 756 cm^{-1};

HRMS (EI) m/z calcd for $C_{12}H_{10}NO_2Br$ [M^+]: 278.9895; found: 278.9899.

4-Ethoxy-*N*-methoxy-1-naphthamide (1q)



Following the general procedure afforded **1q** (451 mg, 92 %) as a white solid, **m.p.** 153 – 154 °C.

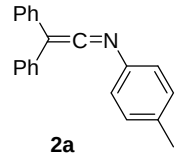
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.54 (s, 1H), 8.33 (dd, J = 8.3, 1.5 Hz, 2H), 7.61 – 7.48 (m, 3H), 6.71 (d, J = 7.9 Hz, 1H), 4.23 (q, J = 7.0 Hz, 2H), 3.95 (s, 3H), 1.57 (t, J = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.3 , 131.6 , 127.9 , 126.9 , 125.8 , 124.9 , 122.4 , 122.3 , 102.8 , 64.8 , 64.0 , 14.7 .

IR (ATR): 3130, 2979, 1638, 1581, 1516, 1383, 1263, 1243, 1155, 1112, 1070, 1030, 954, 902, 839, 758 cm^{-1};

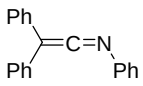
HRMS (EI) m/z calcd for $C_{14}H_{15}NO_3$ [M^+]: 245.1052; found: 245.1053.

General Procedure for the Preparation of Ketenimines 2



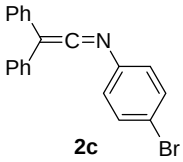
2a

CAS: 5110-45-2



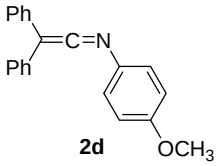
2b

CAS: 14181-84-1



2c

CAS: 29376-76-9



2d

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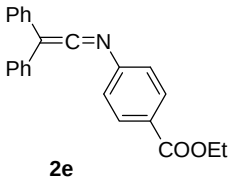
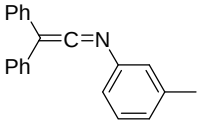


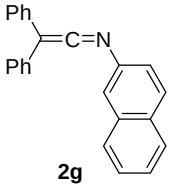
2e

CAS: 1821097-99-7

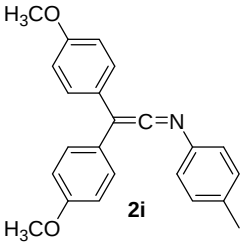


2f

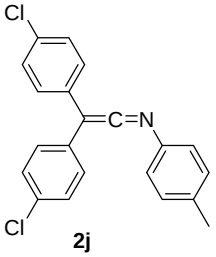
CAS: 17205-61-7



2g

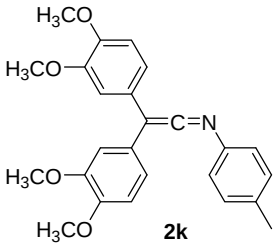


2i

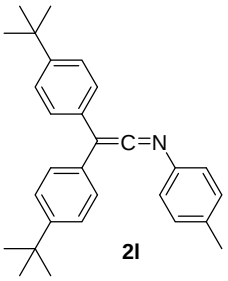


2j

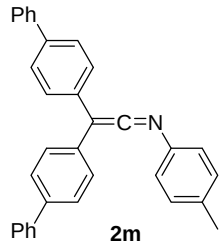
CAS: 22731-54-0



2k

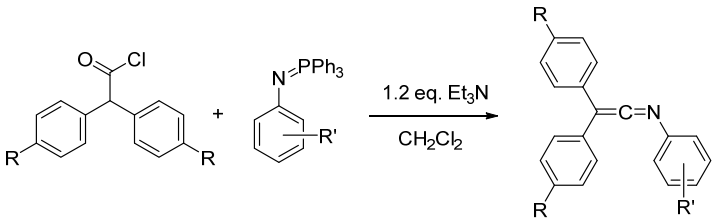


2l



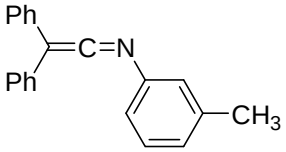
2m

Compounds **2a-2f, 2j** as well as new compounds **2g, 2i,** and **2k-2m** were prepared according to the literature procedures.[3]



To a mixture of corresponding N-(triphenylphosphoranylidene)aniline (2 mmol), Et_3N (2.4 mmol) in CH_2Cl_2 (5 mL) was added corresponding 2,2-diarylacetyl chloride (2 mmol) in CH_2Cl_2 (5 mL) dropwise. The mixture was stirred at room temperature for 15 min and then evaporated on vacuum. The residue was subjected to silica gel column chromatography with petroleum ether as eluent.

2,2-Diphenyl-*N*-(*m*-tolyl)ethen-1-imine (2f)



Following the general procedure afforded **2f** (504 mg, 89 %) as a brown oil.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 – 7.32 (m, 8H), 7.28 – 7.17 (m, 5H), 7.10 (d, *J* = 7.5 Hz, 1H), 2.36 (s, 3H).

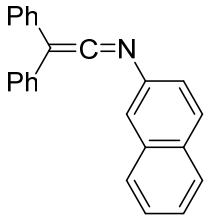
[3] X. Zhou, Z. Jiang, L. Xue, P. Lu and Y. Wang, *Eur. J. Org. Chem.* **2015**, *2015*, 5789.

^{13}C NMR (101 MHz, Chloroform-*d*) δ 190.1 , 139.6 , 134.0 , 129.3 , 128.8 , 128.6 , 127.8 , 126.4 , 124.5 , 121.1 , 77.8 , 21.3 .

IR (film): 3057, 3023, 2001, 1596, 1581, 1493, 1454, 1175, 1123, 1072, 1030, 760, 694, 645 cm^{-1};

HRMS (EI) m/z calcd for $C_{21}H_{17}N$ [M^+]: 283.1361; found: 283.1365.

N-(naphthalen-2-yl)-2,2-diphenylethen-1-imine (2g)



Following the general procedure afforded **2g** (478 mg, 75 %) as a brown solid, **m.p.** 113 – 116 °C.

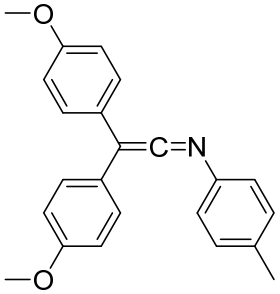
^{1}H NMR (400 MHz, Chloroform-*d*) δ7.86 – 7.81 (m, 4H), 7.56 – 7.47 (m, 3H), 7.42 – 7.33 (m, 8H), 7.26 – 7.22 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.1 , 137.8 , 133.9 , 133.7 , 132.7 , 129.6 , 128.9 , 128.1 , 127.9 , 127.8 , 126.8 , 126.5 , 126.4 , 122.9 , 122.0 , 78.3 .

IR (ATR): 1990, 1594, 1492, 1452, 1286, 1155, 1070, 1027, 815, 746, 693 cm^{-1};

HRMS (EI) m/z calcd for $C_{24}H_{17}N$ [M^+]: 319.1361; found: 319.1359.

2,2-Bis(4-methoxyphenyl)-*N*-(*p*-tolyl)ethen-1-imine (2i)



Following the general procedure afforded **2i** (631 mg, 92 %) as a brown oil.

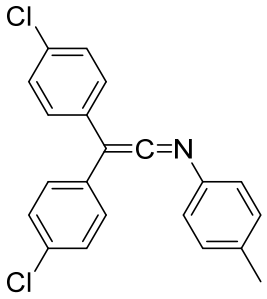
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.21 (m, 6H), 7.17 (d, *J* = 7.8 Hz, 2H), 6.93 – 6.85 (m, 4H), 3.80 (s, 6H), 2.35 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.5 , 158.3 , 138.5 , 137.6 , 130.0 , 128.9 , 126.2 , 123.7 , 114.3 , 77.1 , 55.3 , 21.1 .

IR (film): 2953, 2834, 1990, 1606, 1504, 1246, 1174, 1034, 829 cm^{-1};

HRMS (EI) m/z calcd for $C_{23}H_{21}NO_2$ [M^+]: 343.1572; found: 343.1571.

2,2-Bis(4-chlorophenyl)-*N*-(*p*-tolyl)ethen-1-imine (2j)



Following the general procedure afforded **2j** (498 mg, 71 %) as a brown oil.

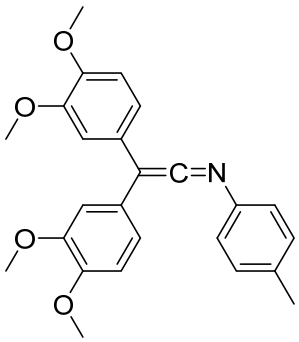
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.28 (m, 4H), 7.28 – 7.17 (m, 8H), 2.37 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 187.9 , 138.4 , 137.0 , 132.4 , 132.1 , 130.3 , 129.1 , 128.8 , 124.0 , 76.3 , 21.2 .

IR (ATR): 2923, 1997, 1582, 1490, 1415, 1280, 1187, 1094, 1014, 911, 824, 751 cm^{-1};

HRMS (EI) m/z calcd for $C_{21}H_{15}NCl_2$ [M^+]: 351.0582; found: 351.0581.

2,2-Bis(3,4-dimethoxyphenyl)-*N*-(*p*-tolyl)ethen-1-imine (2k)



Following the general procedure afforded **2k** (446 mg, 65 %) as a brown oil.

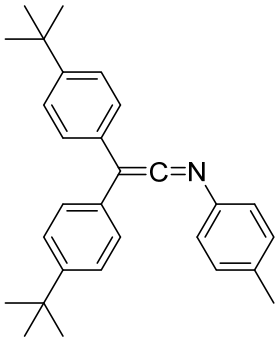
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 (d, *J* = 8.3 Hz, 2H), 7.18 (d, *J* = 8.1 Hz, 2H), 6.92 – 6.83 (m, 6H), 3.87 (s, 6H), 3.81 (s, 6H), 2.35 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.8 , 149.1 , 147.6 , 138.2 , 137.6 , 130.0 , 126.4 , 123.6 , 120.1 , 111.4 , 110.8 , 77.6 , 55.8 , 55.7 , 21.0 .

IR (film): 2953, 2834, 1990, 1606, 1504, 1246, 1174, 1034, 829 cm^{-1};

HRMS (EI) m/z calcd for $C_{23}H_{21}NO_2$ [M^+]: 343.1572; found: 343.1571.

2,2-Bis(4-(*tert*-butyl)phenyl)-*N*-(*p*-tolyl)ethen-1-imine (2l)



Following the general procedure afforded **2l** (561 mg, 71 %) as a yellow solid, **m.p.** 113 – 115 ^{o}C.

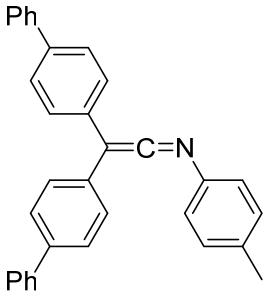
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 – 7.34 (m, 4H), 7.31 – 7.26 (m, 6H), 7.17 (d, *J* = 8.2 Hz, 2H), 2.36 (s, 3H), 1.32 (s, 18H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.4 , 149.3 , 138.2 , 137.7 , 131.1 , 130.0 , 127.4 , 125.7 , 123.9 , 77.6 , 34.5 , 31.3 , 21.1 .

IR (ATR): 2958, 2924, 2865, 1992, 1505, 1462, 1363, 1269, 1189, 1113, 1019, 828, 750, 717 cm^{-1};

HRMS (EI) m/z calcd for $C_{29}H_{33}N$ [M^+]: 395.2613; found: 395.2614

2,2-Di([1,1'-biphenyl]-4-yl)-*N*-(*p*-tolyl)ethen-1-imine (2m)



Following the general procedure afforded **2m** (609 mg, 70 %) as a yellow solid, **m.p.** 119 – 120 ^{o}C.

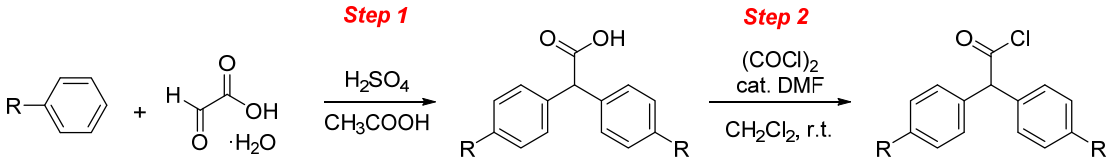
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.63 – 7.58 (m, 8H), 7.48 – 7.41 (m, 8H), 7.37 – 7.30 (m, 4H), 7.21 (d, *J* = 8.2 Hz, 2H), 2.38 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 189.7 , 140.7 , 139.3 , 138.1 , 133.1 , 130.2 , 128.8 , 128.1 , 127.5 , 127.2 , 126.9 , 124.0 , 77.5 , 21.2 .

IR (ATR): 3028, 2923, 1990, 1602, 1485, 1413, 1284, 1186, 909, 837, 821, 764, 730, 696, 664 cm^{-1};

HRMS (EI) m/z calcd for $C_{33}H_{25}N$ [M^+]: 435.1987; found: 435.1978.

General Procedures for the Preparation of 2,2-Di(substitutedphenyl)acetyl chloride



Step 1:

Condition A (R= OCH_3 or 1,2 - dimethoxy):

Following a modified procedure described by Li[4]. A solution of glyoxylic acid (50 mmol) and Substituted benzene (100 mmol) in acetic acid (100 mL) was cooled in an ice bath. Concentrated sulfuric acid (6 mL) was added dropwise under stirring. The solution was then stirred at 40 °C overnight. After most solvent was removed under reduced pressure, the residue was triturated with dichloromethane (200 mL). The solution was washed with aqueous sodium carbonate solution to pH 6, water, and brine and dried over Na_2SO_4. After removed the solvent under reduce pressure, afforded the corresponding acid. The product was used for the next step directly without further purification.

Condition B (R=Cl, t-Bu, Ph):

[4] X. Shao, X. Jiang, X. Zhao, C. Zhao, Y. Chen and Z. Li, *J. Org. Chem.* **2004**, *69*, 899.

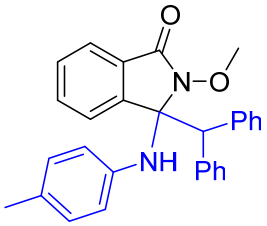
Following a modified procedure described by Marzabadi[5].Substituted benzene (100 mmol) and glyoxylic acid monohydrate (50 mmol) were dissolved in acetic acid (60 mL). The mixture was cooled in an ice/water bath and concentrated sulfuric acid (50 mL) was added dropwise over 0.5 h. The mixture was stirred at 80°C for 12 h and then cooled to room temperature. Water (300 mL) was added and a dark solid was precipitated. The solid was washed with water (50mL) and petroleum ether (3*50mL) and dried in vacuum to give the desired product, which was used in the subsequent step without further purification.

Step 2:

2,2-diarylacetic acid (1.0 equiv) was dissolved in dry dichloromethane (c 0.4 M), followed by addition of a drop of DMF and oxalyl chloride (1.2 equiv). After 2 hour, the volatile and solvent were evaporated and the resulting acid chloride was obtained and used directly for the subsequent reactions without further purification.

Synthetic Procedure and Characterization Data for 3

Typical procedure for the preparation of 3-benzhydryl-2-methoxy-3-(*p*-tolylamino)-isoindolin-1-one (3a)



To an oven-dried Schlenk tube equipped with a magnetic stirring bar were added sequentially **1a** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol), 4A MS (60 mg) and dry DCE (2 mL) under N_2 atmosphere. The reaction vessel was heated to 80 ^oC in oil bath for 10 hours. Upon completion, The reaction mixture was cooled to room temperature and then the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 4:1) to give the product **3a** (107 mg, 82 %) as a white solid; **m.p.** 159 – 161 ^oC.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, J = 7.4 Hz, 1H), 7.54 (td, J = 7.5, 1.1 Hz, 1H), 7.47-7.41 (m, 4.1 Hz, 4H), 7.34 – 7.22 (m, 3H), 7.20 – 7.10 (m, 5H), 6.71 (d, J = 8.3 Hz, 2H), 6.04 (d, J = 8.5 Hz, 2H), 4.92 (s, 1H), 4.51 (s, 1H), 3.82 (s, 3H), 2.09 (s, 3H).

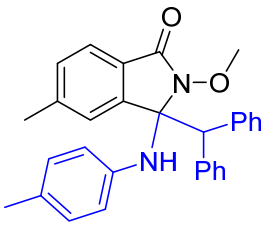
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.5, 141.8, 140.8, 137.7, 136.8, 131.9, 131.1, 130.4, 130.1, 129.4, 129.3, 128.63, 128.60, 128.2, 127.74, 127.73, 123.6, 123.4, 115.2, 82.0, 64.6, 61.1, 20.3.

IR (film): 3418, 2939, 1713, 1615, 1519, 1495, 1451, 1301, 1073, 1002, 908, 809, 716 cm^{-1};

HRMS (EI) m/z calcd for $C_{29}H_{26}N_2O_2$ [M^+]: 434.1994; found:434.2009.

3-Benzhydryl-2-methoxy-5-methyl-3-(*p*-tolylamino)isoindolin-1-one (3b)

[5] Y. Jiang, C. Chen, K. Lu, I. Daniewska, J. De Leon, R. Kong, C. Forray, B. Li, L. G. Hegde, T. D. Wolinsky, D. A. Craig, J. M. Wetzel, K. Andersen and M. R. Marzabadi, *J. Med. Chem.* **2007**, *50*, 3870.



The reaction of **1h** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3b** (108 mg, 80 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 181 – 182 ^oC.

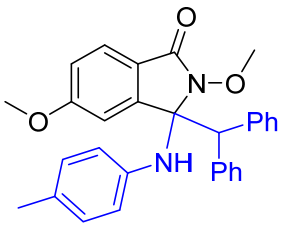
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 6.6 Hz, 2H), 7.30 – 7.22 (m, 5H), 7.22 – 7.14 (m, 5H), 6.72 (d, *J* = 8.3 Hz, 2H), 6.05 (d, *J* = 8.5 Hz, 2H), 4.87 (s, 1H), 4.50 (s, 1H), 3.77 (s, 3H), 2.43 (s, 3H), 2.10 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.8, 142.7, 142.2, 140.9, 137.7, 137.0, 130.5, 130.3, 130.0, 129.4, 128.5, 128.3, 128.2, 127.7, 127.6, 123.9, 123.2, 115.1, 81.6, 64.5, 61.5, 22.2, 20.3.

IR (film): 3418, 2924, 1713, 1620, 1519, 1494, 1451, 1302, 1075, 1008, 908, 808, 729, 707 cm^{-1};

HRMS (EI) m/z calcd for $C_{30}H_{28}N_2O_2$ $[M^+]$: 448.2151; found: 448.2159.

3-Benzhydryl-2,5-dimethoxy-3-(*p*-tolylamino)isoindolin-1-one (3c)



The reaction of **1i** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3c** (113 mg, 81 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 183 – 185 ^oC.

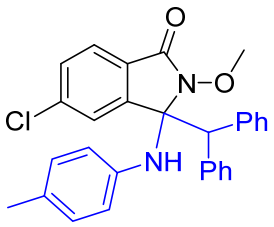
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.4 Hz, 1H), 7.45 (d, *J* = 7.2 Hz, 2H), 7.32 – 7.22 (m, 3H), 7.21-7.15 (m, 5H), 6.96 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.88 (d, *J* = 1.8 Hz, 1H), 6.73 (d, *J* = 8.3 Hz, 2H), 6.08 (d, *J* = 8.3 Hz, 2H), 4.87 (s, 1H), 4.49 (s, 1H), 3.81 (s, 3H), 3.77 (s, 3H), 2.10 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.9, 163.0, 144.2, 140.8, 137.7, 136.8, 130.4, 130.1, 129.4, 128.6, 128.5, 128.2, 127.7, 125.0, 123.4, 115.4, 115.3, 108.9, 81.5, 64.6, 61.3, 55.6, 20.3.

IR (film): 3418, 2939, 1712, 1614, 1519, 1493, 1286, 1250, 1069, 1004, 909, 730, 707 cm^{-1};

HRMS (EI) m/z calcd for $C_{30}H_{28}N_2O_3$ $[M^+]$: 464.2100; found:464.2094.

3-Benzhydryl-5-chloro-2-methoxy-3-(*p*-tolylamino)isoindolin-1-one (3d)



The reaction of **1j** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3d** (83 mg, 59 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 216 – 218 ^oC.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.6 Hz, 1H), 7.42 (d, *J* = 7.6 Hz, 2H), 7.40 (d, *J* = 7.8 Hz, 2H), 7.33 – 7.24 (m, 3H), 7.24 – 7.16 (m, 5H), 6.74 (d, *J* = 8.3 Hz, 2H), 6.05 (d, *J* = 8.4 Hz, 2H), 4.87 (s, 1H), 4.50 (s, 1H), 3.78 (s, 3H), 2.11 (s, 3H).

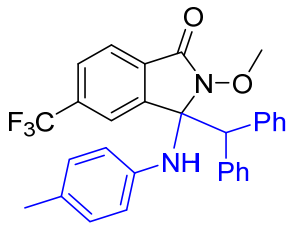
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.6, 144.0, 140.4, 138.5, 137.2, 136.5, 130.4, 130.0, 129.9, 129.5, 129.4, 129.0, 128.7, 128.4, 127.92, 127.90, 124.6, 123.8, 115.2, 81.6, 64.5, 61.4, 20.3.

IR (film): 3418, 2939, 1715, 1614, 1519, 1495, 1424, 1301, 1140, 1090, 909, 732, 707 cm^{-1};

HRMS (EI) m/z calcd for $C_{28}H_{21}N_2OCl$ $[M-CH_4O]^+$: 436.1342; found:436.1339.

MALDI-TOF-MS m/z 469.16 ($[M+H]^+$);

3-Benzhydryl-2-methoxy-3-(*p*-tolylamino)-5-(trifluoromethyl)isoindolin-1-one (3e)



The reaction of **1k** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3e** (92 mg, 61 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 200 – 201 ^oC.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.9 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 1H), 7.66 (s, 1H), 7.36 (d, *J* = 6.6 Hz, 2H), 7.32 – 7.24 (m, 3H), 7.24 – 7.13 (m, 5H), 6.73 (d, *J* = 8.3 Hz, 2H), 6.03 (d, *J* = 8.4 Hz, 2H), 4.89 (s, 1H), 4.55 (s, 1H), 3.79 (s, 3H), 2.10 (s, 3H).

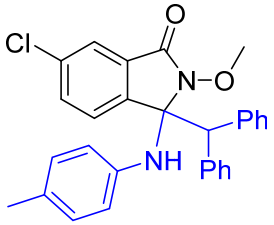
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.0, 143.0, 140.4, 137.0, 136.4, 134.3, 133.7 (q, *J* = 32.8 Hz), 130.3, 129.9, 129.5, 129.3, 128.7, 128.4, 128.0 (d, *J* = 1.2 Hz), 126.5 (q, *J* = 3.8 Hz), 123.8, 123.5 (q, *J* = 271.4 Hz), 120.6 (q, *J* = 3.6 Hz), 115.4, 82.1, 64.5, 61.5, 20.3.

IR (film): 3418, 3030, 2941, 1722, 1715, 1519, 1495, 1324, 1171, 1134, 1089, 1055, 909, 732, 693 cm^{-1};

HRMS (EI) m/z calcd for $C_{29}H_{21}N_2OF_3$ $[M-CH_4O]^+$: 470.1606; found:470.1604.

MALDI-TOF-MS m/z 503.19 ($[M+H]^+$).

3-Benzhydryl-6-chloro-2-methoxy-3-(*p*-tolylamino)isoindolin-1-one (3f)



The reaction of **1l** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3f** (119 mg, 85 %)

after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 186 – 188 ^{o}C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 1.8 Hz, 1H), 7.50 (dd, *J* = 8.1, 2.0 Hz, 1H), 7.42 (d, *J* = 6.7 Hz, 2H), 7.35 – 7.26 (m, 4H), 7.24 – 7.11 (m, 5H), 6.73 (d, *J* = 8.2 Hz, 2H), 6.04 (d, *J* = 8.5 Hz, 2H), 4.89 (s, 1H), 4.49 (s, 1H), 3.81 (s, 3H), 2.11 (s, 3H).

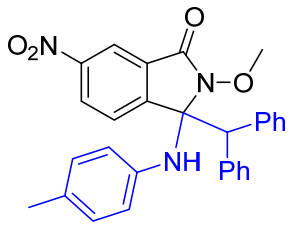
^{13}C NMR (101 MHz, $CDCl_3$) δ 162.1, 140.5, 140.1, 137.4, 136.5, 135.6, 132.9, 132.0, 130.3, 130.0, 129.5, 129.0, 128.8, 128.3, 127.92, 127.91, 125.0, 123.6, 115.3, 82.0, 64.6, 61.0, 20.3.

IR (film): 3418, 2939, 1715, 1614, 1519, 1494, 1452, 1301, 1182, 1088, 909, 808, 733, 706, 631 cm^{-1};

HRMS (EI) m/z calcd for $C_{28}H_{21}N_2OCl$ [M-CH_4O] $^{+}$: 436.1342; found:436.1343.

MALDI-TOF-MS m/z 469.17 ([M+H]$^{+}$);

3-Benzhydryl-2-methoxy-6-nitro-3-(*p*-tolylamino)isoindolin-1-one (3g)



The reaction of **1m** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3g** (14 mg, 10 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a yellow solid; **m.p.** 186 – 188 ^{o}C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.46 (d, *J* = 1.8 Hz, 1H), 8.40 (dd, *J* = 8.3, 2.2 Hz, 1H), 7.61 (d, *J* = 8.3 Hz, 1H), 7.40 (dd, *J* = 7.8, 1.5 Hz, 2H), 7.34 – 7.27 (m, 3H), 7.24 – 7.15 (m, 5H), 6.74 (d, *J* = 8.2 Hz, 2H), 6.03 (d, *J* = 8.5 Hz, 2H), 4.93 (s, 1H), 4.56 (s, 1H), 3.84 (s, 3H), 2.11 (s, 3H).

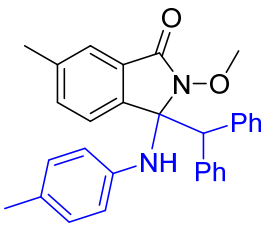
^{13}C NMR (101 MHz, $CDCl_3$) δ 161.1, 148.9, 148.5, 140.2, 136.8, 136.0, 132.8, 130.3, 129.9, 129.6, 129.6, 128.9, 128.5, 128.21, 128.19, 126.7, 124.7, 118.6, 115.4, 82.3, 64.7, 61.2, 20.3.

IR (KBr): 3410, 2939, 1728, 1617, 1530, 1519, 1343, 1309, 1063, 1003, 725, 705 cm^{-1};

HRMS (EI) m/z calcd for $C_{17}H_{24}N_3O_4$ [M-$C_{12}H_1$] $^{+}$: 334.1767; found:334.1723.

MALDI-TOF-MS m/z 480.10 ([M+H]$^{+}$).

3-Benzhydryl-2-methoxy-6-methyl-3-(*p*-tolylamino)isoindolin-1-one (3h)



The reaction of **1n** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3h** (113 mg, 84 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 172 – 174 ^{o}C.

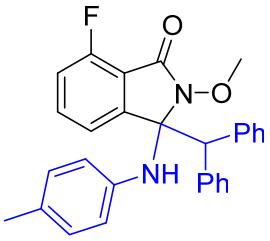
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.41 (m, 3H), 7.36 – 7.22 (m, 5H), 7.20 – 7.09 (m, 5H), 6.71 (d, *J* = 8.3 Hz, 2H), 6.04 (d, *J* = 8.3 Hz, 2H), 4.90 (s, 1H), 4.48 (s, 1H), 3.81 (s, 3H), 2.41 (s, 3H), 2.09 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.8, 140.9, 139.6, 138.7, 137.9, 136.9, 132. 8, 131.2, 130.3, 130.1, 129.3, 128.6, 128.5, 128.1, 127.7, 123.7, 123.5, 115.2, 81.9, 64.5, 61.0, 21.5, 20.3.

IR (film): 3418, 3030, 2939, 1714, 1615, 1519, 1494, 1451, 1301, 1084, 909, 810, 730, 707 cm^{-1};

HRMS (EI) m/z calcd for $C_{30}H_{28}N_2O_2$ [M^+]: 448.2151; found:448.2152.

3-Benzhydryl-7-fluoro-2-methoxy-3-(*p*-tolylamino)isoindolin-1-one(3i)



The reaction of **1o** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3i** (39 mg, 29 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 198-199^{o}C.

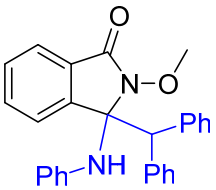
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (td, *J* = 8.0, 4.7 Hz, 1H), 7.44 – 7.39 (m, 2H), 7.32 – 7.21 (m, 9H), 7.09 (t, *J* = 8.6 Hz, 1H), 6.74 (d, *J* = 8.2 Hz, 2H), 6.10 – 6.03 (m, 2H), 4.89 (s, 1H), 4.50 (s, 1H), 3.80 (s, 3H), 2.11 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 160.51 (d, *J* = 1.0 Hz), 158.38 (d, *J* = 262.3 Hz), 144.75 , 140.50 , 137.26 , 136.45 , 133.82 (d, *J* = 7.6 Hz), 130.35 , 129.97 , 129.39 , 128.88 , 128.66 , 128.32 , 127.92 (d, *J* = 6.7 Hz), 119.54 (d, *J* = 3.9 Hz), 118.11 (d, *J* = 13.0 Hz), 116.92 (d, *J* = 19.5 Hz), 115.22 , 81.55 (d, *J* = 1.6 Hz), 64.54 , 61.30 , 20.26 .

IR (film): 3416, 3030, 2939, 1721, 1714, 1625, 1519, 1495, 1492, 1452, 1301, 1252, 1181, 1003, 884, 809, 729, 707 cm^{-1};

HRMS (EI) m/z calcd for $C_{29}H_{25}N_2O_2F$ [M^+]: 452.1900; found:452.1906.

3-Benzhydryl-2-methoxy-3-(phenylamino)isoindolin-1-one (3j)



The reaction of **1a** (0.36 mmol), **2b** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3j** (97 mg, 77 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 162 – 164 ^{o}C.

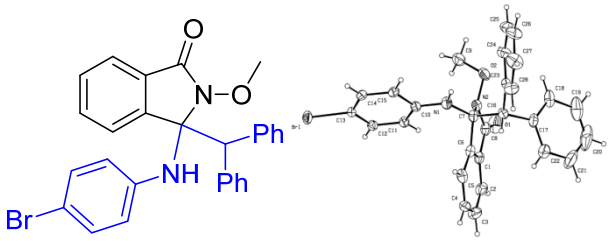
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.4 Hz, 1H), 7.55 (td, *J* = 7.5, 1.0 Hz, 1H), 7.50 – 7.39 (m, 4H), 7.33 – 7.23 (m, 3H), 7.22 – 7.09 (m, 5H), 6.95 – 6.86 (m, 2H), 6.64 (t, *J* = 7.3 Hz, 1H), 6.13 (d, *J* = 7.8 Hz, 2H), 4.93 (s, 1H), 4.61 (s, 1H), 3.81 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.5, 143.2, 141.7, 137.6, 136.7, 132.0, 131.1, 130.4, 130.0, 129.5, 128.8, 128.7, 128.2, 127.8, 123.5, 123.4, 119.4, 115.1, 81.7, 64.6, 61.1.

IR (film): 3416, 2938, 1712, 1602, 1497, 1466, 1312, 1072, 1003, 908, 750, 718, 706 cm^{-1};

HRMS (EI) m/z calcd for $C_{28}H_{24}N_2O_2$ [M^+]: 420.1838; found: 420.1843.

3-Benzhydryl-3-((4-bromophenyl)amino)-2-methoxyisoindolin-1-one (3k)



The reaction of **1a** (0.36 mmol), **2c** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3k** (105 mg, 70 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 192 – 195 ^oC.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, J = 7.4 Hz, 1H), 7.56 (td, J = 7.5, 1.0 Hz, 1H), 7.48 (td, J = 7.5, 0.7 Hz, 1H), 7.43 (d, J = 7.1 Hz, 2H), 7.40 (d, J = 7.6 Hz, 1H), 7.33 – 7.25 (m, 3H), 7.21 – 7.08 (m, 5H), 7.00 (d, J = 8.9 Hz, 2H), 6.01 (d, J = 9.0 Hz, 2H), 4.92 (s, 1H), 4.64 (s, 1H), 3.82 (s, 3H).

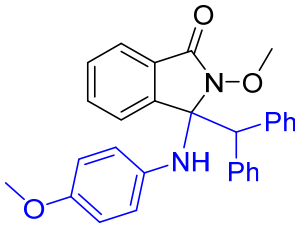
^{13}C NMR (126 MHz, $CDCl_3$) δ 163.5, 142.2, 141.2, 137.4, 136.4, 132.1, 131.6, 131.1, 130.3, 130.0, 129.7, 128.8, 128.2, 127.93, 127.90, 123.6, 123.5, 116.7, 111.5, 81.6, 64.6, 60.9.

IR (film): 3417, 2938, 1712, 1512, 1466, 1241, 1073, 1036, 1002, 908, 822, 732 cm^{-1};

HRMS (EI) m/z calcd for $C_{27}H_{19}N_2OBr$ [M-CH_4O] $^+$: 466.0681; found: 466.0680.

MALDI-TOF-MS m/z 499.10 ([M+H]$^+$).

3-Benzhydryl-2-methoxy-3-((4-methoxyphenyl)amino)isoindolin-1-one (3l)



The reaction of **1a** (0.36 mmol), **2d** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3l** (120 mg, 89 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 142 – 144 ^oC.

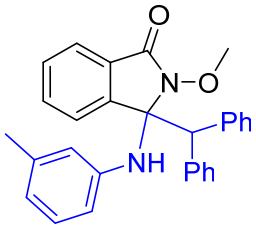
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, J = 7.4 Hz, 1H), 7.57 – 7.50 (m, 1H), 7.47 – 7.37 (m, 4H), 7.32 – 7.21 (m, 3H), 7.20 – 7.08 (m, 5H), 6.48 (d, J = 9.0 Hz, 2H), 6.11 (d, J = 9.0 Hz, 2H), 4.91 (s, 1H), 4.40 (s, 1H), 3.81 (s, 3H), 3.60 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.6, 153.2, 141.9, 137.8, 137.0, 131.9, 131.1, 130.3, 130.1, 129.4, 128.6, 128.2, 127.71, 127.69, 123.7, 123.3, 116.9, 114.2, 82.4, 64.4, 61.1, 55.3.

IR (film): 3417, 2938, 1714, 1593, 1494, 1310, 1182, 1071, 1003, 908, 727 cm^{-1};

HRMS (EI) m/z calcd for $C_{29}H_{26}N_2O_3$ [M^+]: 450.1943; found: 450.1940.

3-Benzhydryl-2-methoxy-3-(*m*-tolylamino)isoindolin-1-one (3m)



The reaction of **1a** (0.36 mmol), **2f** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3m** (60 mg, 46 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a white solid; **m.p.** 185 – 187 ^oC.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.66 (d, *J* = 7.4 Hz, 1H), 7.55 (td, *J* = 7.5, 1.2 Hz, 1H), 7.47 (dd, *J* = 7.4, 1.1 Hz, 1H), 7.45 – 7.41 (m, 3H), 7.33 – 7.25 (m, 3H), 7.21 – 7.11 (m, 5H), 6.76 (t, *J* = 7.8 Hz, 1H), 6.46 (d, *J* = 7.5 Hz, 1H), 6.00 (s, 1H), 5.86 (dd, *J* = 8.1, 2.1 Hz, 1H), 4.92 (s, 1H), 4.56 (s, 1H), 3.82 (s, 3H), 2.05 (s, 3H).

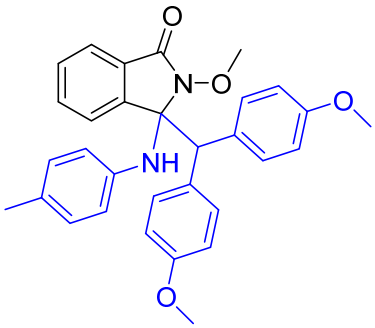
^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.50 , 143.17 , 141.78 , 138.62 , 137.64 , 136.72 , 131.94 , 130.37 , 130.04 , 129.42 , 128.68 , 128.64 , 128.20 , 127.79 , 127.77 , 123.56 , 123.36 , 120.24 , 116.01 , 111.97 , 81.75 , 64.61 , 61.15 , 21.38 .

IR (film): 3417, 3030, 2938, 1720, 1714, 1607, 1519, 1493, 1467, 1451, 1314, 1176, 1134, 1074, 1002, 907, 772, 706 cm^{-1};

HRMS (EI) m/z calcd for $C_{28}H_{22}N_2O$ $[M\text{-}CH_4O]^+$: 402.1732; found: 402.1731.

MALDI-TOF-MS m/z 435.21 ($[M+H]^+$);

3-(Bis(4-methoxyphenyl)methyl)-2-methoxy-3-(*p*-tolylamino)isoindolin-1-one (3n)



The reaction of **1a** (0.36 mmol), **2i** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3n** (55 mg, 37 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a pale yellow solid; **m.p.** 103 – 105 ^oC.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.3 Hz, 1H), 7.53 (td, *J* = 7.5, 1.3 Hz, 1H), 7.44 (td, *J* = 7.5, 1.1 Hz, 1H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.31 (d, *J* = 8.5 Hz, 2H), 7.05 (d, *J* = 7.7 Hz, 2H), 6.80 (d, *J* = 8.7 Hz, 2H), 6.71 (d, *J* = 8.4 Hz, 2H), 6.68 (d, *J* = 8.9 Hz, 2H), 6.05 (d, *J* = 8.5 Hz, 2H), 4.81 (s, 1H), 4.51 (s, 1H), 3.82 (s, 3H), 3.75 (s, 3H), 3.71 (s, 3H), 2.09 (s, 3H).

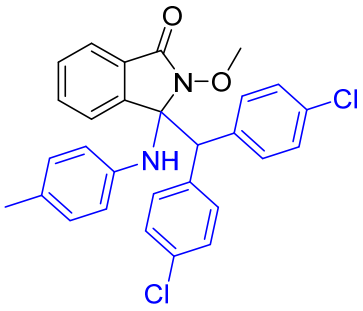
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.5 , 158.84 , 158.79 , 141.95 , 140.85 , 131.8 , 131.3 , 131.0 , 130.9 , 129.9 , 129.3 , 129.0 , 128.4 , 123.3 , 115.1 , 113.9 , 113.4 , 82.0 , 64.5 , 59.5 , 55.1 , 55.0 , 20.2 .

IR (film): 3412, 2999, 2936, 2838, 1722, 1713, 1609, 1514, 1464, 1303, 1250, 1180, 1034, 908, 813, 736 cm^{-1};

HRMS (EI) m/z calcd for $C_{30}H_{26}N_2O_3$ $[M-CH_4O]^+$: 462.1943; found: 462.1947.

MALDI-TOF-MS m/z 495.23 ($[M+H]^+$);

3-(Bis(4-chlorophenyl)methyl)-2-methoxy-3-(*p*-tolylamino)isoindolin-1-one) (3o)



The reaction of **1a** (0.36 mmol), **2j** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3o** (60 mg, 40 %) after chromatography on silica gel (petroleum ether/ethyl acetate 4:1) as a pale yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 7.4 Hz, 1H), 7.56 (td, J = 7.5, 1.3 Hz, 1H), 7.48 (td, J = 7.5, 1.1 Hz, 1H), 7.41 (d, J = 7.5 Hz, 1H), 7.30 (d, J = 8.5 Hz, 2H), 7.25 (d, J = 8.8 Hz, 2H), 7.15 (d, J = 8.5 Hz, 2H), 7.09 (d, J = 8.1 Hz, 2H), 6.73 (d, J = 8.3 Hz, 2H), 6.05 (d, J = 8.5 Hz, 2H), 4.87 (s, 1H), 4.39 (s, 1H), 3.80 (s, 3H), 2.10 (s, 3H).

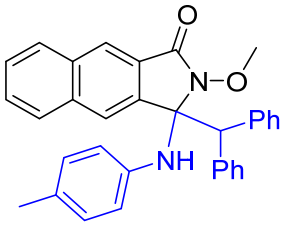
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.4 , 141.5 , 140.4 , 135.8 , 135.0 , 134.0 , 133.9 , 132.2 , 131.6 , 131.2 , 130.8 , 129.7 , 129.4 , 129.1 , 128.8 , 128.5 , 123.7 , 123.3 , 115.4 , 81.6 , 64.6 , 60.1 , 20.3.

IR (film): 3419, 2939, 2920, 1722, 1713, 1616, 1519, 1493, 1467, 1301, 1093, 1073, 1014, 908, 810, 734, 695 cm^{-1};

HRMS (EI) m/z calcd for $C_{28}H_{20}N_2OCl_2$ $[M-CH_4O]^+$: 470.0953; found: 470.0950.

MALDI-TOF-MS m/z 503.13 ($[M+H]^+$);

3-Benzhydryl-2-methoxy-3-(*p*-tolylamino)-2,3-dihydro-1*H*-benzo[*f*]isoindol-1-one(3pa)



The reaction of **1b** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3pa** + **3pb** after chromatography on silica gel (petroleum ether/ethyl acetate 5:1) . **3pa** (45 mg, 31 %) was obtained as a white solid; **m.p.** 193 – 195 °C.

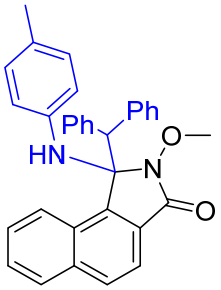
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 8.01 – 7.93 (m, 1H), 7.90 – 7.84 (m, 1H), 7.83 (s, 1H), 7.63 – 7.54 (m, 2H), 7.49 – 7.42 (m, 2H), 7.31 – 7.24 (m, 3H), 7.19 – 7.09 (m, 5H), 6.66 (d, J = 8.3 Hz, 2H), 6.04 (d, J = 8.5 Hz, 2H), 4.98 (s, 1H), 4.63 (s, 1H), 3.82 (s, 3H), 2.05 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.3, 140.6, 137.8, 136.9, 136.8, 134.8, 133.4, 130.3, 130.1, 129.6, 129.4, 128.7, 128.6, 128.5, 128.3, 128.2, 127.9, 127.76, 127.75, 127.0, 123.9, 123.1, 115.3, 81.9, 64.5, 61.5, 20.2

IR (film): 3417, 2925, 1712, 1615, 1519, 1452, 1347, 1302, 1067, 909, 733, 707, 646 cm^{-1};

HRMS (EI) m/z calcd for $C_{33}H_{28}N_2O_2$ [M^+]: 484.2151; found:484.2151.

1-Benzhydryl-2-methoxy-1-(*p*-tolylamino)-1,2-dihydro-3H-benzo[*e*]isoindol-3-one(3pb)



The reaction of **1b** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **3pa** + **3pb** after chromatography on silica gel (petroleum ether/ethyl acetate 5:1) . **3pb** (48 mg, 33 %) was obtained as a white solid; **m.p.** 111 – 112 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (d, *J* = 7.7 Hz, 1H), 7.98 – 7.93 (m, 1H), 7.89 (d, *J* = 8.3 Hz, 1H), 7.80 (d, *J* = 7.3 Hz, 2H), 7.64 – 7.52 (m, 3H), 7.43 (t, *J* = 7.3 Hz, 2H), 7.40 – 7.33 (m, 1H), 7.04 (t, *J* = 7.3 Hz, 1H), 6.96 (t, *J* = 7.5 Hz, 2H), 6.79 (d, *J* = 7.4 Hz, 2H), 6.64 (d, *J* = 8.3 Hz, 2H), 6.08 (d, *J* = 8.5 Hz, 2H), 5.08 (s, 1H), 4.79 (s, 1H), 3.78 (s, 3H), 2.03 (s, 3H).

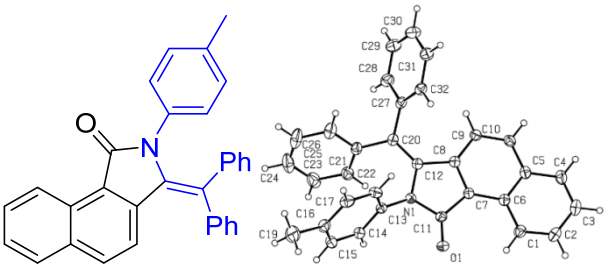
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.6, 141.0, 139.4, 137.1, 136.3, 135.6, 131.5, 130.5, 129.6, 129.5, 129.4, 128.9, 128.8, 128.4, 128.0, 127.9, 127.67, 127.66, 127.5, 127.1, 123.5, 118.9, 115.1, 81.9, 64.5, 64.1, 20.2.

IR (film): 3413, 2925, 1714, 1615, 1519, 1495, 1452, 1301, 1034, 909, 807, 760, 732, 706 cm^{-1};

HRMS (EI) m/z calcd for $C_{33}H_{28}N_2O_2$ [M^+]: 484.2151; found:484.2157.

Synthetic procedure and Characterization data for 4

Typical procedure for the preparation of 3-(Diphenylmethylene)-2-(p-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (4a)



To an oven-dried Schlenk tube equipped with a magnetic stirring bar were added sequentially **1c** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol), 4A MS (60 mg) and dry DCE (2 mL) under N_2 atmosphere. The reaction vessel was heated to 80°C in oil bath for 10 hours. Upon completion, the reaction mixture was cooled to room temperature and then the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 20:1) to give the product **4a** (97 mg, 74 %) as a yellow solid; **m.p.** 146 – 148 °C.

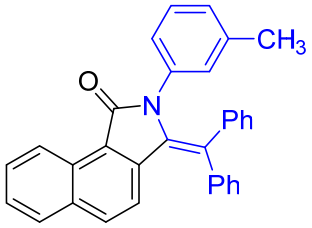
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.23 (d, *J* = 8.3 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.68 – 7.61 (m, 2H), 7.56 – 7.51 (m, 1H), 7.50 – 7.39 (m, 5H), 7.00 (d, *J* = 8.3 Hz, 2H), 6.95 – 6.86 (m, 5H), 6.84 (d, *J* = 8.1 Hz, 2H), 6.54 (d, *J* = 8.8 Hz, 1H), 2.17 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 141.6, 139.9, 138.1, 135.8, 134.8, 134.1, 133.3, 132.0, 131.6, 131.1, 129.0, 128.9, 128.8, 128.7, 128.5, 128.3, 127.8, 127.6, 127.2, 127.1, 126.9, 124.5, 122.7, 120.8, 20.9.

IR (film): 3056, 1698, 1512, 1443, 1359, 1205, 1127, 1096, 825, 756, 701 cm^{-1};

HRMS (EI) m/z calcd for $C_{32}H_{23}NO$ [M^+]: 437.1780; found:437.1782.

3-(Diphenylmethylene)-2-(m-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (4b)



The reaction of **1c** (0.36 mmol), **2f** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4b** (97 mg, 74 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 149 – 150 °C.

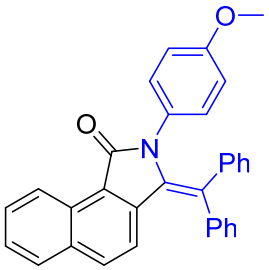
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.23 (d, *J* = 8.5 Hz, 1H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.68 – 7.60 (m, 2H), 7.57 – 7.37 (m, 6H), 7.03 – 6.83 (m, 8H), 6.75 (d, *J* = 7.6 Hz, 1H), 6.55 (d, *J* = 8.8 Hz, 1H), 2.13 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1 , 141.6 , 140.0 , 138.0 , 137.5 , 136.4 , 134.6 , 133.3 , 132.1 , 131.5 , 130.8 , 129.0 , 128.9 , 128.8 , 128.7 , 128.6 , 128.3 , 127.81 , 127.75 , 127.2 , 127.0 , 126.85 , 126.84 , 124.9 , 124.5 , 122.6 , 120.8 , 21.0 .

IR (film): 3054, 1694, 1587, 1518, 1489, 1442, 1360, 1205, 1125, 909, 825, 755, 732, 702, 646 cm^{-1};

HRMS (EI) m/z calcd for $C_{32}H_{23}NO$ [M^+]: 437.1780; found:437.1787.

3-(Diphenylmethylene)-2-(4-methoxyphenyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (4c)



The reaction of **1c** (0.36 mmol), **2d** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4c** (63 mg, 46 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 136 – 138 °C.

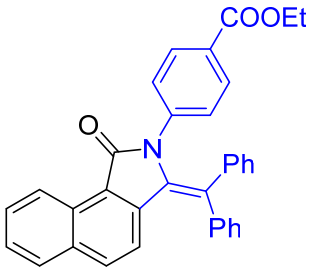
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.22 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.69 – 7.60 (m, 2H), 7.57 – 7.37 (m, 6H), 7.07 – 7.00 (m, 2H), 6.99 – 6.85 (m, 5H), 6.62 – 6.56 (m, 2H), 6.54 (d, *J* = 8.8 Hz, 1H), 3.69 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3 , 157.6 , 141.6 , 139.9 , 138.0 , 134.9 , 133.3 , 132.0 , 131.5 , 131.0 , 129.7 , 129.0 , 128.89 , 128.86 , 128.7 , 128.3 , 127.8 , 127.2 , 126.9 , 124.5 , 122.6 , 120.8 , 113.4 , 55.4 .

IR (film): 3045, 1694, 1510, 1442, 1293, 1246, 1207, 1129, 1031, 910, 826, 756, 732, 702, 622 cm^{-1};

HRMS (EI) m/z calcd for $C_{32}H_{23}NO_2$ [M^+]: 453.1729; found:453.1732.

Ethyl 4-(3-(diphenylmethylene)-1-oxo-1,3-dihydro-2*H*-benzo[*e*]isoindol-2-yl)benzoate (4d)



The reaction of **1c** (0.36 mmol), **2e** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4d** (94 mg, 63 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 91 – 93 ^oC.

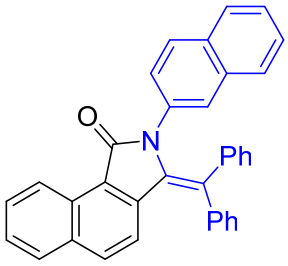
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.20 (d, *J* = 8.5 Hz, 1H), 7.81 (d, *J* = 8.1 Hz, 1H), 7.78 – 7.72 (m, 2H), 7.68 – 7.64 (m, 2H), 7.58 – 7.54 (m, 1H), 7.54 – 7.41 (m, 5H), 7.27 – 7.21 (m, 2H), 6.93 – 6.89 (m, 5H), 6.56 (d, *J* = 8.8 Hz, 1H), 4.32 (q, *J* = 7.1 Hz, 2H), 1.37 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8 , 166.1 , 141.1 , 140.9 , 139.8 , 138.3 , 134.0 , 133.4 , 132.6 , 131.5 , 131.0 , 129.5 , 129.3 , 129.0 , 128.5 , 127.9 , 127.7 , 127.6 , 127.5 , 127.2 , 127.0 , 124.4 , 122.3 , 120.6 , 60.9 , 14.3 .

IR (film): 3056, 2981, 1703, 1604, 1517, 1443, 1358, 1273, 1192, 1106, 1019, 911, 825, 748, 701 cm^{-1};

HRMS (EI) m/z calcd for $C_{34}H_{25}NO_3$ [M^+]: 495.1834; found:495.1833.

3-(Diphenylmethylene)-2-(naphthalen-2-yl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (4e)



The reaction of **1c** (0.36 mmol), **2g** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4e** (95 mg, 67 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 211 – 212 ^oC.

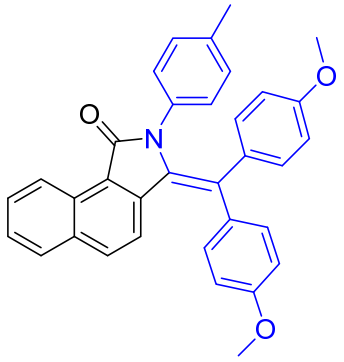
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.25 (d, *J* = 8.4 Hz, 1H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.70 – 7.62 (m, 4H), 7.60 (d, *J* = 2.0 Hz, 1H), 7.54 (ddd, *J* = 8.1, 6.8, 1.2 Hz, 1H), 7.52 – 7.43 (m, 6H), 7.40 – 7.34 (m, 2H), 7.29 – 7.23 (m, 1H), 6.89 (d, *J* = 7.3 Hz, 2H), 6.70 (t, *J* = 7.7 Hz, 2H), 6.63 – 6.56 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2 , 141.4 , 139.8 , 138.1 , 134.7 , 134.2 , 133.4 , 132.7 , 132.3 , 131.6 , 131.5 , 130.8 , 129.2 , 129.0 , 128.9 , 128.8 , 128.3 , 127.8 , 127.7 , 127.6 , 127.3 , 127.1 , 127.0 , 126.9 , 126.7 , 125.8 , 125.7 , 125.6 , 124.5 , 122.6 , 120.8 .

IR (film): 3054, 2924, 1698, 1597, 1517, 1469, 1442, 1367, 1266, 1207, 1117, 825, 754, 701 cm^{-1};

HRMS (EI) m/z calcd for $C_{35}H_{23}NO$ [M^+]: 473.1780; found: 473.1776.

3-(Bis(4-methoxyphenyl)methylene)-2-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (4f)



The reaction of **1c** (0.36 mmol), **2i** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4f** (107 mg, 72 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 181 – 184 °C.

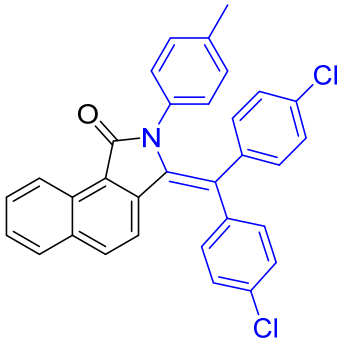
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.23 (d, *J* = 8.3 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.68 – 7.60 (m, 2H), 7.52 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.35 – 7.29 (m, 2H), 7.03 – 6.95 (m, 4H), 6.86 (d, *J* = 8.1 Hz, 2H), 6.83 – 6.77 (m, 2H), 6.64 (d, *J* = 8.8 Hz, 1H), 6.49 – 6.38 (m, 2H), 3.92 (s, 3H), 3.68 (s, 3H), 2.19 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1 , 160.2 , 158.9 , 138.3 , 135.5 , 134.2 , 134.0 , 133.3 , 133.08 , 133.06 , 132.7 , 132.6 , 131.9 , 129.1 , 128.7 , 128.5 , 128.1 , 127.7 , 127.4 , 126.6, 124.5 , 122.2 , 120.8 , 114.2 , 112.7 , 55.4 , 55.2 , 20.9 .

IR (film): 2929, 1693, 1602, 1511, 14621, 1359, 1302, 1249, 1173, 1031, 826, 761, 732 cm^{-1};

HRMS (EI) m/z calcd for $C_{34}H_{27}NO_3$ [M^+]: 497.1991; found: 497.1993.

3-(Bis(4-chlorophenyl)methylene)-2-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (4g)



The reaction of **1c** (0.36 mmol), **2j** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4g** (91 mg, 60 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 199 – 200 °C.

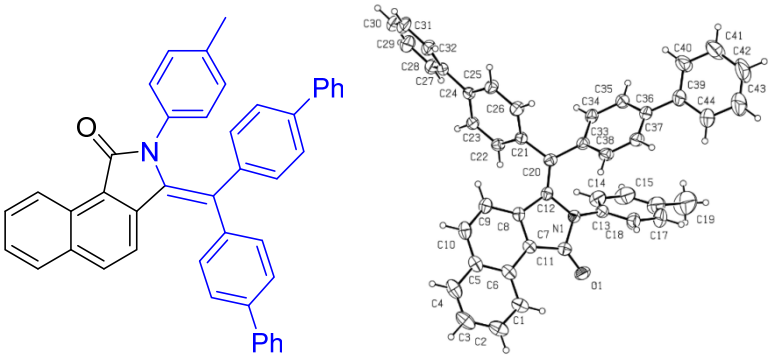
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.25 – 9.15 (m, 1H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.70 (d, *J* = 8.8 Hz, 1H), 7.66 (ddd, *J* = 8.3, 6.9, 1.3 Hz, 1H), 7.55 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.48 – 7.41 (m, 2H), 7.37 – 7.30 (m, 2H), 6.99 – 6.92 (m, 2H), 6.92 – 6.85 (m, 4H), 6.79 – 6.72 (m, 2H), 6.63 (d, *J* = 8.8 Hz, 1H), 2.23 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9 , 139.6 , 138.0 , 137.5 , 136. 5 , 135.5 , 135.0 , 133.7 , 133.4 , 133.3 , 132.9 , 132.3 , 132.1 , 129.3 , 128.9 , 128.8 , 128.5 , 127.9 , 127.6 , 127.4 , 127.1 , 125.3 , 124.5 , 122.8 , 120.5 , 20.9 .

IR (film): 2922, 1703, 1694, 1587, 1514, 1487, 1399, 1361, 1206, 1128, 1090, 1015, 908, 824, 756, 732 cm^{-1};

HRMS (EI) m/z calcd for $C_{32}H_{21}NOCl_2$ [M^+]: 505.1000; found: 505.1000.

3-(Di([1,1'-biphenyl]-4-yl)methylene)-2-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (4h)



The reaction of **1c** (0.36 mmol), **2m** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4h** (164 mg, 93 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 214 – 216 °C.

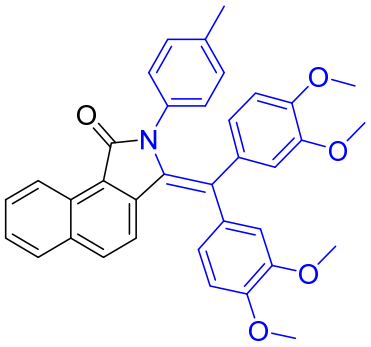
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.25 (d, *J* = 8.1 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.77 – 7.70 (m, 4H), 7.65 (td, *J* = 8.6, 2.3 Hz, 2H), 7.56 – 7.47 (m, 5H), 7.45 – 7.37 (m, 5H), 7.36 – 7.29 (m, 1H), 7.12 (d, *J* = 8.3 Hz, 2H), 7.02 (d, *J* = 8.3 Hz, 2H), 6.96 (d, *J* = 8.3 Hz, 2H), 6.85 (d, *J* = 8.1 Hz, 2H), 6.76 (d, *J* = 8.8 Hz, 1H), 2.10 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1 , 141.4 , 140.8 , 140.4 , 140.2 , 138.8 , 138.0 , 135.9 , 134.9 , 134.0 , 133.3 , 132.2 , 132.1 , 131.5 , 129.0 , 128.9 , 128.7 , 128.6 , 128.3 , 127.83 , 127.80 , 127.74 , 127.70 , 127.4 , 127.3 , 127.0 , 126.92 , 126.87 , 126.0, 124.5 , 122.6 , 120.9 , 20.9 .

IR (film): 3028, 2920, 1694, 1512, 1486, 1361, 1192, 1128, 1095, 825, 766, 740, 619 cm^{-1};

HRMS (EI) m/z calcd for $C_{44}H_{31}NO$ [M^+]: 589.2406; found: 589.2411.

3-(Bis(3,4-dimethoxyphenyl)methylene)-2-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (4i)



The reaction of **1c** (0.36 mmol), **2k** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4i** (117 mg, 70 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 180 – 183 °C.

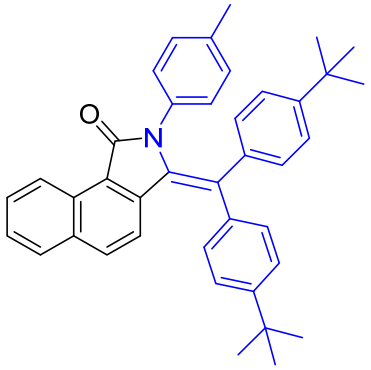
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.23 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.69 – 7.62 (m, 2H), 7.53 (ddd, *J* = 8.1, 7.0, 1.2 Hz, 1H), 7.03 (d, *J* = 7.4 Hz, 3H), 6.95 (d, *J* = 8.2 Hz, 1H), 6.88 (d, *J* = 7.9 Hz, 3H), 6.67 (d, *J* = 8.8 Hz, 1H), 6.46 (s, 3H), 4.00 (s, 3H), 3.81 (s, 3H), 3.76 (s, 3H), 3.69 (s, 3H), 2.19 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.09 , 149.79 , 149.07 , 148.53 , 147.31 , 138.28 , 135.59 , 134.22 , 133.96 , 133.48 , 133.15 , 132.92 , 131.94 , 129.08 , 128.93 , 128.41 , 128.21 , 127.77 , 127.09 , 126.68 , 124.80 , 124.66 , 124.42 , 122.28 , 120.88 , 114.42 , 111.03 , 110.19 , 56.03 , 55.95 , 55.87 , 55.54 , 20.89 .

IR (film): 2930, 1694, 1597, 1512, 1463, 1409, 1325, 1253, 1168, 1139, 1026, 826 cm^{-1};

HRMS (EI) m/z calcd for $C_{36}H_{31}NO_5$ [M^+]: 557.2202; found: 557.2205.

3-(Bis(4-(tert-butyl)phenyl)methylene)-2-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one(4j)



The reaction of **1c** (0.36 mmol), **2l** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4j** (119 mg, 72 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 258 – 261 °C.

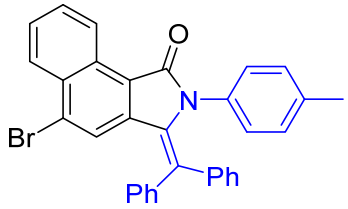
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.23 (d, *J* = 8.5 Hz, 1H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.67 – 7.60 (m, 2H), 7.52 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.48 – 7.42 (m, 2H), 7.37 – 7.31 (m, 2H), 6.99 – 6.93 (m, 2H), 6.92 – 6.87 (m, 2H), 6.84 – 6.77 (m, 4H), 6.57 (d, *J* = 8.8 Hz, 1H), 2.15 (s, 3H), 1.41 (s, 9H), 1.17 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1 , 152.0 , 150.2 , 138.5 , 138.2 , 137.1 , 135.3 , 134.23 , 134.19 , 133.2 , 131.8 , 131.2 , 130.8 , 129.2 , 129.1 , 128.4 , 128.1 , 127.7 , 126.7 , 125.6 , 124.5 , 124.0 , 122.5 , 120.9 , 34.8 , 34.4 , 31.4 , 31.1 , 20.9 .

IR (film): 2961, 2867, 1698, 1513, 1361, 1267, 1213, 1192, 1129, 1111, 1019, 825, 777, 756 cm^{-1};

HRMS (EI) m/z calcd for $C_{40}H_{39}NO$ [M^+]: 549.3032; found: 549.3040.

5-Bromo-3-(diphenylmethylene)-2-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one(4k)



The reaction of **1p** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4k** (76 mg, 49 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 234 – 235 °C.

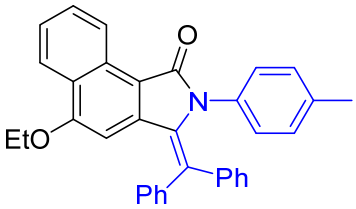
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.27 (d, *J* = 7.8 Hz, 1H), 8.20 (d, *J* = 8.2 Hz, 1H), 7.68 (ddd, *J* = 8.3, 6.9, 1.4 Hz, 1H), 7.62 (ddd, *J* = 8.3, 6.9, 1.5 Hz, 1H), 7.58 – 7.46 (m, 3H), 7.40 (dt, *J* = 6.8, 1.5 Hz, 2H), 7.03 – 6.96 (m, 2H), 6.96 – 6.82 (m, 7H), 6.78 (s, 1H), 2.17 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5 , 141.0 , 139.5 , 138.0 , 136.0 , 133.8 , 133.65 , 131.61 , 131.5 , 131.0 , 129.7 , 129.6 , 129.1 , 128.98 , 128.96 , 128.6 , 128.0 , 127.5 , 127.4 , 127.33 , 127.30 , 127.2 , 125.3 , 124.9 , 122.1 , 20.9 .

IR (film): 3056, 2924, 2854, 1703, 1698, 1513, 1443, 1367, 1208, 1129, 910, 764, 733, 701 cm^{-1};

HRMS (EI) m/z calcd for $C_{32}H_{22}NOBr$ [M^+]: 515.0885; found:515.0884.

3-(Diphenylmethylene)-5-ethoxy-2-(*p*-tolyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one(4l)



The reaction of **1q** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4l** (66 mg, 46 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 262 – 263 °C.

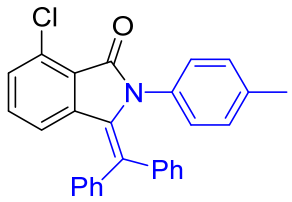
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.14 (d, *J* = 8.3 Hz, 1H), 8.20 (d, *J* = 8.3 Hz, 1H), 7.63 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 1H), 7.53 – 7.41 (m, 6H), 7.03 – 6.98 (m, 2H), 6.95 – 6.87 (m, 5H), 6.83 (d, *J* = 8.0 Hz, 2H), 5.86 (s, 1H), 3.64 (q, *J* = 7.0 Hz, 2H), 2.16 (s, 3H), 1.34 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2 , 157.6 , 141.8 , 139.8 , 139.7 , 135.5 , 135.2 , 134.3 , 131.6 , 130.9 , 129.8 , 128.9 , 128.6 , 128.45 , 128.39 , 127.8 , 127.4 , 127.1 , 126.9 , 126.1 , 125.8 , 124.3 , 122.2 , 115.5 , 99.8 , 63.2 , 20.9 , 14.6 .

IR (ATR): 3058, 1697, 1579, 1512, 1360, 1280, 1209, 1176, 1136, 1097, 1025, 827, 772, 755, 698, 623 cm^{-1};

HRMS (EI) m/z calcd for $C_{34}H_{27}NO_2$ [M^+]: 481.2042; found: 481.2043.

7-Chloro-3-(diphenylmethylene)-2-(*p*-tolyl)isoindolin-1-one(4m)



The reaction of **1d** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4m** (77 mg, 61 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 156 – 158 °C.

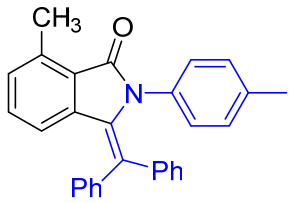
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 – 7.40 (m, 3H), 7.35 (dd, *J* = 7.8, 1.5 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.12 (t, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 8.2 Hz, 2H), 6.92 – 6.84 (m, 3H), 6.81 (d, *J* = 8.2 Hz, 4H), 6.37 (d, *J* = 8.0 Hz, 1H), 2.15 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.8, 141.4, 140.5, 139.6, 136.0, 133.6, 132.7, 132.2, 131. 6, 131.0, 130.7, 130.1, 129.0, 128.7, 128.5, 128.0, 127.4, 127.2, 127.0, 124.8, 122.1, 20.9.

IR (film): 3056, 2923, 1714, 1596, 1512, 1463, 1360, 1330, 1261, 1229, 1099, 803, 756, 695, 669, 648 cm^{-1};

HRMS (EI) m/z calcd for $C_{28}H_{20}NOCl$ [M^+]: 421.1233; found:421.1230.

3-(Diphenylmethylene)-7-methyl-2-(*p*-tolyl)isoindolin-1-one(4n)



The reaction of **1e** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4n** (54 mg, 45 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 175 – 176 °C.

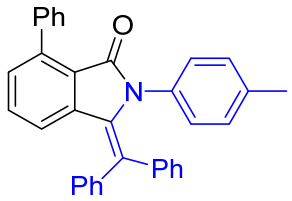
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.39 (m, 3H), 7.38 – 7.33 (m, 2H), 7.14 (d, *J* = 7.4 Hz, 1H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.94 (d, *J* = 8.3 Hz, 2H), 6.90 – 6.84 (m, 3H), 6.84 – 6.77 (m, 4H), 6.31 (d, *J* = 7.7 Hz, 1H), 2.74 (s, 3H), 2.15 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.0, 141.8, 140.0, 138.7, 137.7, 135.7, 134.03, 133.99, 131.3, 131.1, 130.65, 130.63, 128.9, 128.4, 128.3, 127.4, 127.1, 126.6, 126.4, 126.1, 121.2, 20.9, 17.8.

IR (film): 3055, 2923, 1704, 1601, 1513, 1444, 1361, 1332, 1219, 1115, 696, 668, 652 cm^{-1};

HRMS (EI) m/z calcd for $C_{29}H_{23}NO$ [M^+]: 401.1780; found:401.1784.

3-(diphenylmethylene)-7-phenyl-2-(*p*-tolyl)isoindolin-1-one (4o)



The reaction of **1f** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4n** (33 mg, 24 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a yellow solid; **m.p.** 191 – 193 °C.

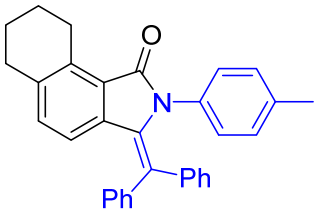
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 – 7.53 (m, 2H), 7.49 – 7.35 (m, 8H), 7.32 (dd, *J* = 7.5, 1.1 Hz, 1H), 7.25 (t, *J* = 3.8 Hz, 1H), 6.94 – 6.90 (m, 2H), 6.90 – 6.81 (m, 5H), 6.76 (d, *J* = 8.1 Hz, 2H), 6.52 (dd, *J* = 7.8, 1.2 Hz, 1H), 2.12 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 167.4 , 141.9 , 140.9 , 140.0 , 139.2 , 137.5 , 135.7 , 133.9 , 133.7 , 131.3 , 131.2 , 130.8 , 130.7 , 129.8 , 129.0 , 128.5 , 128.4 , 127.7 , 127.53 , 127.48 , 127.1 , 126.78 , 126.76 , 124.7 , 122.7 , 20.8.

IR (film): 3057, 3029, 2924, 1711, 1597, 1513, 1474, 1444, 1360, 1259, 1223, 1118, 759, 697 cm^{-1};

HRMS (EI) m/z calcd for $C_{29}H_{23}NO$ [M^+]: 463.1936; found: 463.1931.

3-(Diphenylmethylene)-2-(p-tolyl)-2,3,6,7,8,9-hexahydro-1*H*-benzo[*e*]isoindol-1-one(4p)



The reaction of **1g** (0.36 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $Cl_3CCOOCs$ (0.09 mmol) and 4A MS (60 mg) in DCE (2 mL) following the typical procedure afforded **4n** (61 mg, 46 %) after chromatography on silica gel (petroleum ether/ethyl acetate 20:1) as a pale yellow solid; **m.p.** 157 – 158 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.39 (m, 3H), 7.38 – 7.33 (m, 2H), 6.96 – 6.90 (m, 3H), 6.89 – 6.83 (m, 3H), 6.83 – 6.77 (m, 4H), 6.19 (d, *J* = 8.2 Hz, 1H), 3.32 (t, *J* = 5.6 Hz, 2H), 2.76 (t, *J* = 5.5 Hz, 2H), 2.15 (s, 3H), 1.86 – 1.74 (m, 4H).

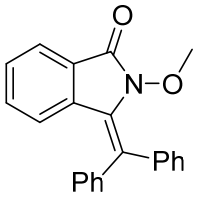
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 141.9, 140.1, 138.5, 136.7, 136.6, 135.6, 134.14, 134.10, 132.7, 131.1, 130.7, 128.8, 128.4, 128.2, 127.4, 127.1, 126.5, 125.7, 125.5, 120.6, 29.6, 25.3, 22.6, 22.5, 20.9.

IR (film): 3028, 2927, 2857, 1704, 1698, 1513, 1486, 1444, 1360, 1203, 1135, 1100, 909, 756, 697, 652 cm^{-1};

HRMS (EI) m/z calcd for $C_{32}H_{27}NO$ [M^+]: 441.2093; found:441.2096.

Synthetic procedure and Characterization data for 5 and 6

Typical procedure for the preparation of 3-(Diphenylmethylene)-2-methoxyisoindolin-1-one (5a):



To a 25 ml pressure tube charged with a magnetic stir bar was added **3a** (0.1 mmol), $BF_3 \cdot OEt_2$ (0.3 mmol), and 1.5 mL acetone as solvent. The reaction vessel was sealed and heated to 80 ^oC in an oil bath for a period of 3 hours. Then the solvent was removed under reduce pressure, and the residue was subject to silica gel column chromatography with ethyl acetate/petroleum ether = 10:1 (v:v) as eluent. The product **5a** was obtained as a white solid (27 mg, 83%), **m.p.** 170 – 171 ^oC.

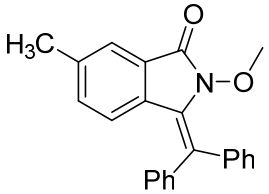
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.6 Hz, 1H), 7.46 – 7.20 (m, 12H), 6.46 (d, *J* = 8.0 Hz, 1H), 3.44 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.3 , 140.5 , 139.5 , 134.0 , 131.9 , 130.6 , 130.3 , 128.9 , 128.7 , 128.5 , 128.3 , 127.7 , 127.3 , 127.0 , 126.1 , 123.6 , 123.1 , 62.6 .

IR (ATR): 3566, 1713, 1442, 1305, 1267, 1189, 1114, 1032, 997, 951, 911, 756, 698, 638 cm^{-1};

HRMS (EI) m/z calcd for $C_{22}H_{17}NO_2$ [M^+]: 327.1259; found:327.1263.

Preparation of 3-(Diphenylmethylene)-2-methoxy-6-methylisoindolin-1-one (5b):



The reaction of **3h** (0.1 mmol), $BF_3 \cdot OEt_2$ (0.3 mmol), and acetone (1.5mL) following the typical procedure afforded **5b** (26 mg, 76 %) after chromatography on silica gel (petroleum ether/ethyl acetate 10:1) as a white solid, **m.p.** 166 – 168 ^oC.

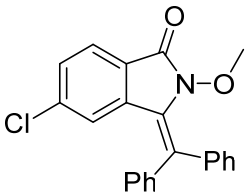
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.44 – 7.38 (m, 3H), 7.37 – 7.27 (m, 7H), 7.08 – 7.02 (m, 1H), 6.33 (d, *J* = 8.1 Hz, 1H), 3.43 (s, 3H), 2.38 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.6 , 140.7 , 139.5 , 139.1 , 133.0 , 131.5 , 130.6 , 130.4 , 128.9 , 128.5 , 128.4 , 127.6 , 127.3 , 127.1 , 125.2 , 123.5 , 123.3 , 62.6 , 21.4 .

IR (ATR): 3055, 1710, 1488, 1443, 1320, 1264, 1194, 1128, 1017, 827, 753, 699, 651, 631 cm^{-1};

HRMS (EI) m/z calcd for $C_{23}H_{19}NO_2$ [M^+]: 341.1416; found: 341.1414.

Preparation of 5-Chloro-3-(diphenylmethylene)-2-methoxyisoindolin-1-one (5c):



The reaction of **3d** (0.1 mmol), $BF_3 \cdot OEt_2$ (0.3 mmol), and acetone (1.5mL) following the typical procedure afforded **5c** (31 mg, 86 %) after chromatography on silica gel (petroleum ether/ethyl acetate 10:1) as a white solid, **m.p.** 215 – 217 ^oC.

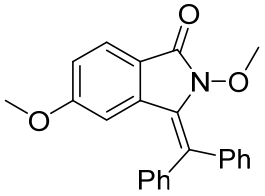
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.1 Hz, 1H), 7.51 – 7.43 (m, 3H), 7.37 – 7.28 (m, 8H), 6.35 (d, *J* = 1.7 Hz, 1H), 3.44 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 162.5 , 139.9 , 139.0 , 138.2 , 135.4 , 130.5 , 130.3 , 129.1 , 129.03 , 128.98 , 128.0 , 127.5 , 127.4 , 125.3 , 124.3 , 123.9 , 62.8 .

IR (ATR): 3077, 1718, 1607, 1451, 1424, 1317, 1273, 1186, 1121, 1075, 1014, 958, 761, 696 cm^{-1};

HRMS (EI) m/z calcd for $C_{22}H_{16}NO_2Cl$ [M^+]: 361.0870; found: 361.0866.

Preparation of 3-(Diphenylmethylene)-2,5-dimethoxyisoindolin-1-one (5d):



The reaction of **3c** (0.1 mmol), $BF_3{\cdot}OEt_2$ (0.3 mmol), and acetone (1.5mL) following the typical procedure afforded **5d** (29 mg, 81 %) after chromatography on silica gel (petroleum ether/ethyl acetate 10:1) as a white solid, **m.p.** 194 – 196 °C.

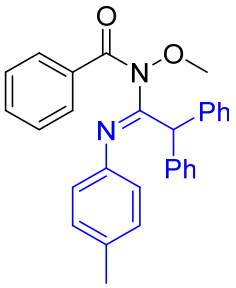
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73 (d, J = 8.4 Hz, 1H), 7.47 – 7.42 (m, 3H), 7.39 – 7.36 (m, 2H), 7.33 – 7.27 (m, 5H), 6.89 (dd, J = 8.4, 2.3 Hz, 1H), 5.88 (d, J = 2.2 Hz, 1H), 3.45 (s, 3H), 3.43 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.8 , 162.8 , 140.6 , 139.4 , 136.3 , 130.6 , 130.3 , 129.0 , 128.8 , 128.5 , 127.7 , 127.4 , 125.8 , 124.6 , 119.8 , 116.6 , 107.6 , 62.5 , 55.0 .

IR (ATR): 3002, 1708, 1617, 1581, 1476, 1440, 1355, 1278, 1214, 1165, 1117, 1009, 962, 757, 697, 642 cm^{-1};

HRMS (EI) m/z calcd for $C_{23}H_{19}NO_3$ [M^+]: 357.1365; found: 357.1368.

Preparation of *N*-(2,2-diphenyl-1-(*p*-tolylimino)ethyl)-*N*-methoxybenzamide (6):



To an oven-dried Schlenk tube equipped with a magnetic stirring bar were added sequentially **1a** (0.45 mmol), **2a** (0.3 mmol), $[Cp^*RhCl_2]_2$ (0.015 mmol), $AgNTf_2$ (0.045 mmol), 4A MS (60 mg) and dry DCE (2 mL) under N_2 atmosphere. The reaction vessel was heated to reflux in oil bath for 10 hours. Upon completion, The reaction mixture was cooled to room temperature and then the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 10:1) to give the product **6** (125 mg, 96 %) as a white solid; **m.p.** 118 – 124 °C.

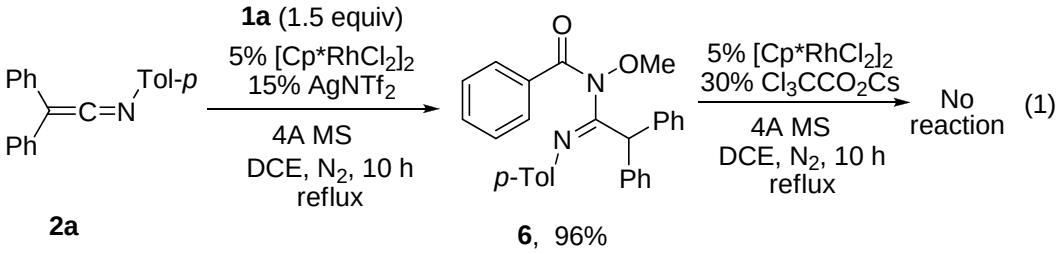
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.25 (m, 8H), 7.24 – 7.20 (m, 3H), 7.12 – 7.06 (m, 4H), 6.92 (d, J = 8.0 Hz, 2H), 6.69 (d, J = 7.9 Hz, 2H), 5.46 (s, 1H), 3.62 (s, 3H), 2.24 (s, 3H).

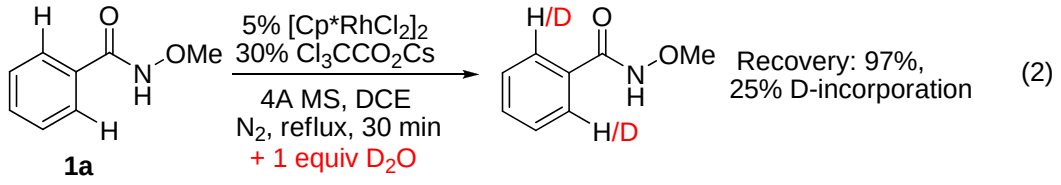
^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.6 , 139.3 , 138.9 , 136.6 , 135.4 , 130.0 , 129.6 , 129.4 , 128.6 , 128.0 , 127.6 , 127.3 , 126.6 , 62.1 , 54.1 , 21.0 .

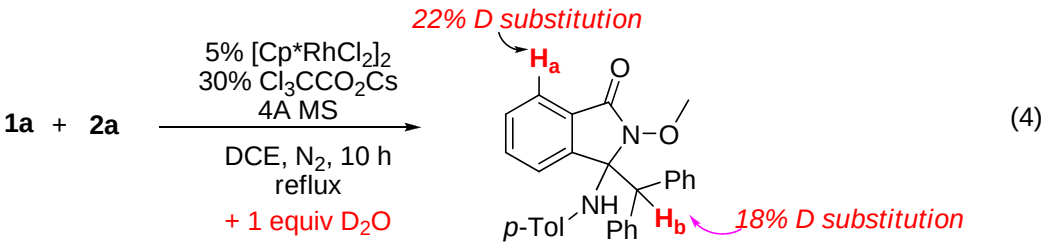
IR (ATR): 3060, 2926, 1661, 1602, 1509, 1451, 1329, 1298, 1180, 1050, 923, 897, 796, 742, 700 cm^{-1};

HRMS (EI) m/z calcd for $C_{29}H_{26}N_2O_2$ [M^+]: 434.1994; found: 434.1996.

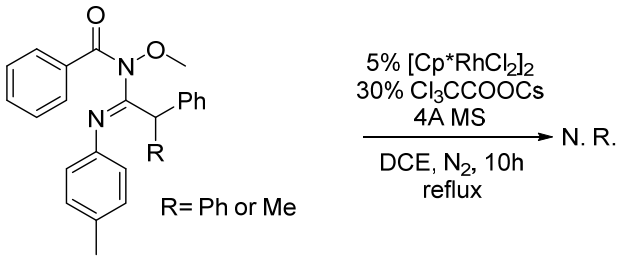
Scheme S1. Mechanistic Experiments and Their Procedures





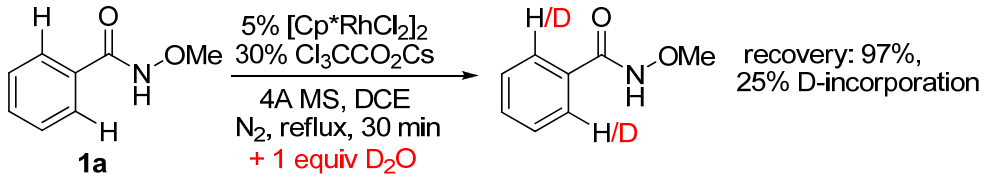


(1) Intermediate exclusion experiment

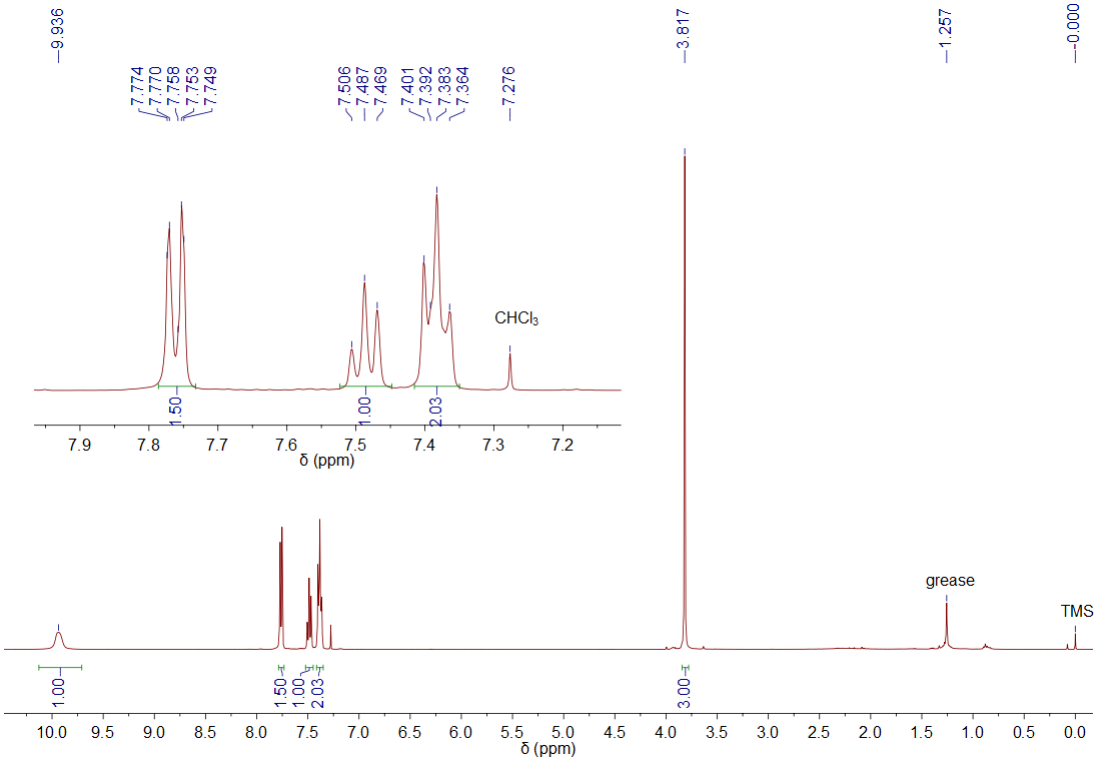


To an oven-dried Schlenk tube equipped with a magnetic stirring bar were added sequentially **6** (0.2 mmol), $[Cp^*RhCl_2]_2$ (0.01 mmol), Cl_3CCO_2Cs (0.06 mmol), 4A MS (40 mg) and dry DCE (1.5 mL) under N_2 atmosphere. The reaction vessel was heated to reflux in oil bath for 10 hours. Upon completion, TLC showed that no [4+1] or [4+2] product was found and the reactant was unreacted.

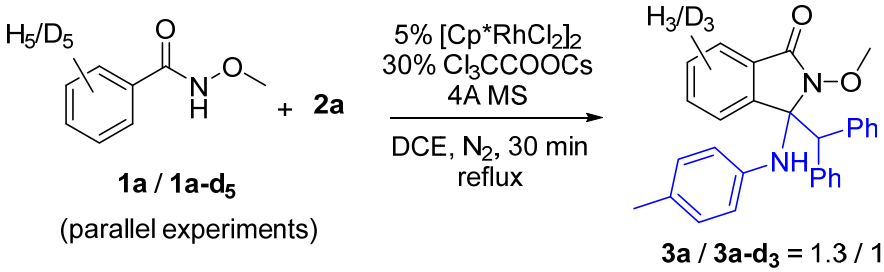
(2) The reversibility of the C–H activation step in absence of ketenimines



To a dried Schlenk tube equipped with a Teflon-coated magnetic stirring bar were added **1a** (31 mg, 0.2 mmol), $[Cp^*RhCl_2]_2$ (6 mg, 0.01 mmol), Cl_3CCO_2Cs (18 mg, 0.06 mmol), 4A MS (40mg), D_2O (0.2 mmol, 4 μL) and DCE (1.5 mL) under N_2 atmosphere. The reaction vessel was stirred at 80°C for 30 minutes. The solvent was removed under vacuum and the residue was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 1:1) to afford **1a** (30 mg, 97%).

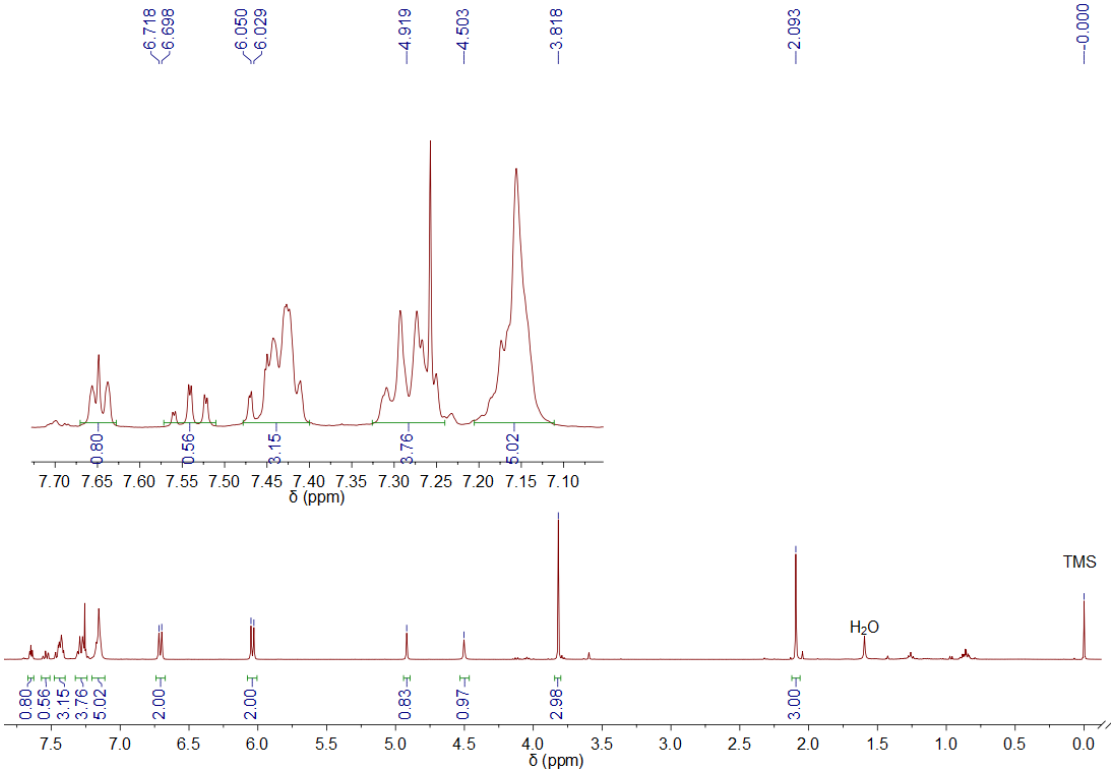


(3) Kinetic isotope effect

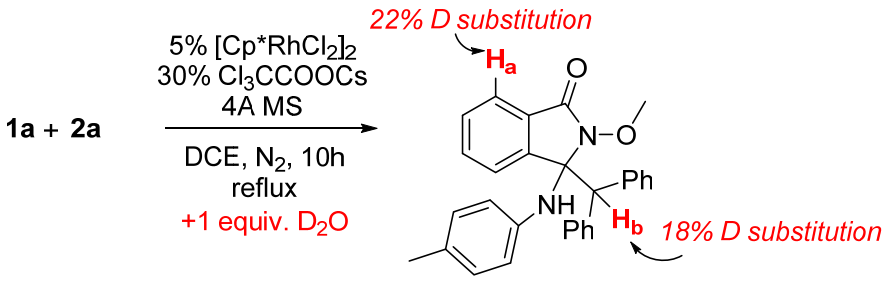


Following general procedure, to a dried Schlenk tube equipped with a Teflon-coated magnetic stirring bar were added **1a** (0.24 mmol), **2a** (0.2 mmol), $[Cp^*RhCl_2]_2$ (0.01 mmol), $Cl_3CCOOCs$ (0.06 mmol), 4A M S(40 mg) and DCE (1.5 mL) under N_2 atmosphere. In another Schlenk tube, **1a-d_5** (0.24 mmol) was used instead of **1a**. The two reactions were allowed to stir at 80 °C for 30 min and then were combined. The solvent was removed under vacuum and the residue was

purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) to afford the product (59 mg, 34% combined yield). The value of K_H/K_D was obtained based on ^{1}HNMR.



(4) [4+1] reaction with the additive of D_2O



To a dried Schlenk tube equipped with a Teflon-coated magnetic stirring bar were added **1a** (0.24 mmol), **2a** (0.2 mmol), ($[Cp^*RhCl_2]_2$ (0.01 mmol), $Cl_3CCOOCs$ (0.06 mmol), 4A MS(40mg), D_2O (0.2 mmol, 4 μL) and DCE (1.5 mL) under N_2 atmosphere. The reaction vessel was stirred at 80°C for 10 hours. The solvent was removed under vacuum and the residue was purified by flash column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 5:1) to afford **1a** (51 mg, 59%).

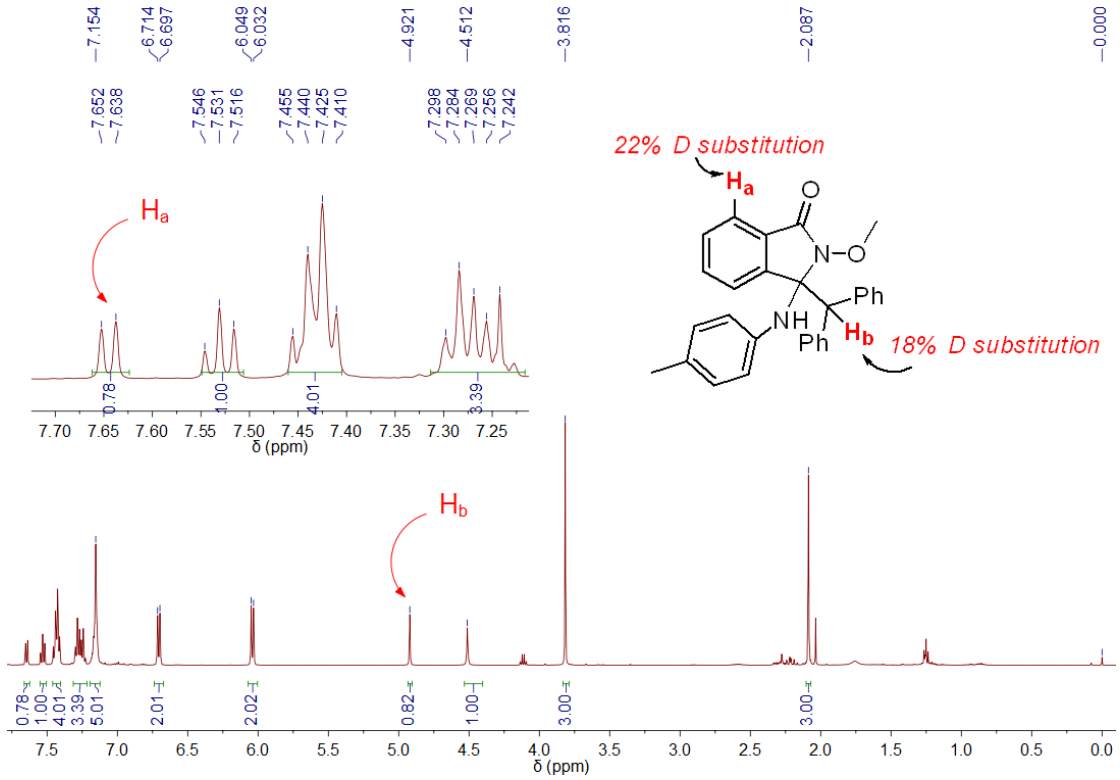
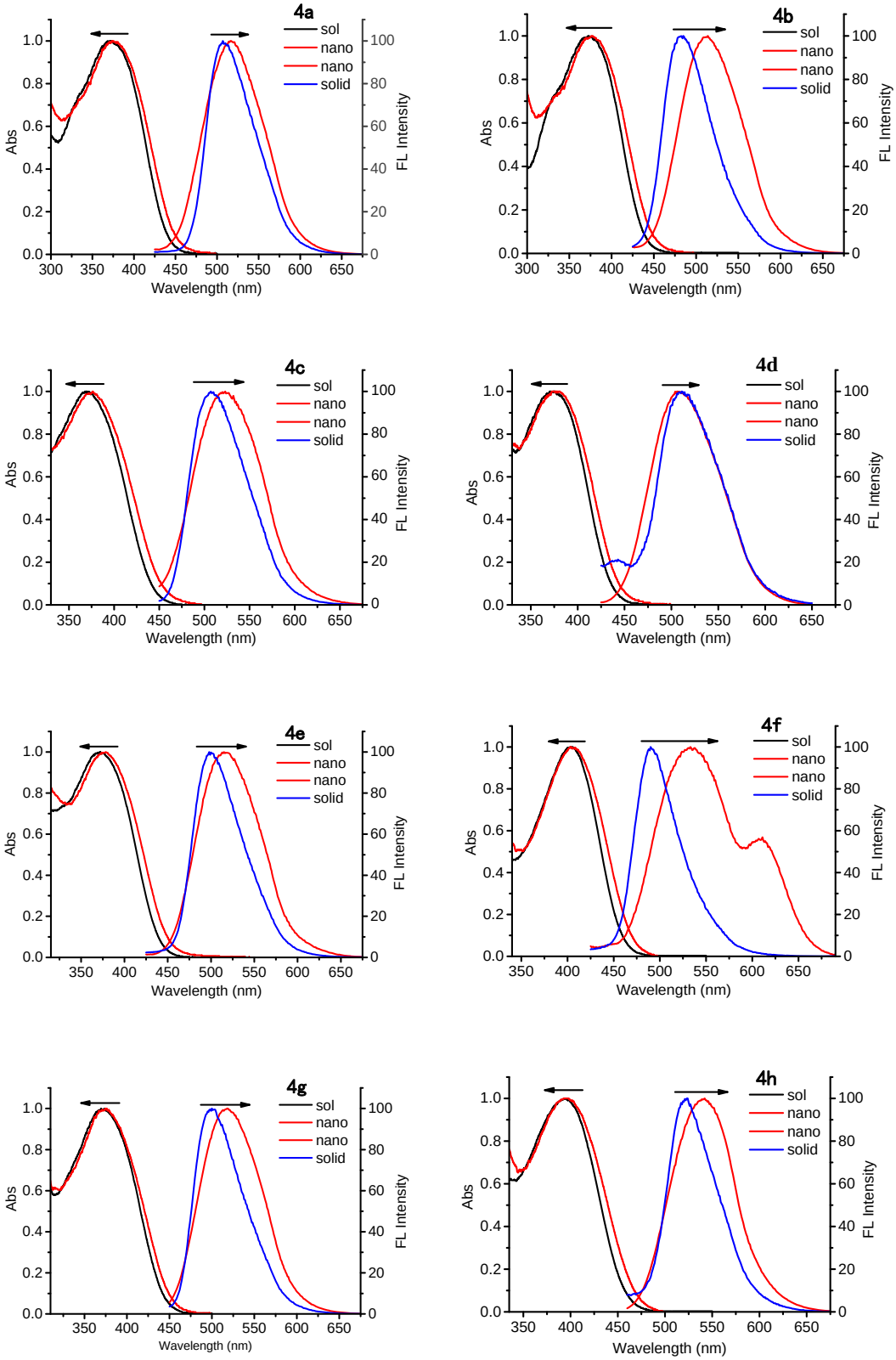
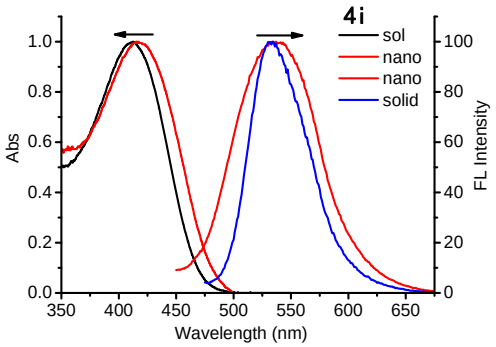
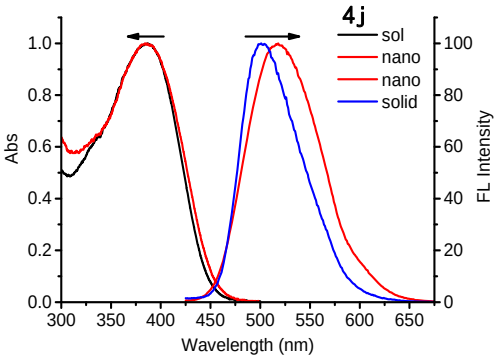

7.154
6.714
6.697
6.049
6.032
4.921
4.512
3.816
2.087
0.000
7.652
7.638
7.546
7.531
7.516
7.455
7.440
7.425
7.410
7.298
7.284
7.269
7.256
7.242
Ha
22% D substitution
Ha
O
N–O
Ph
NH
Ph
Hb
18% D substitution
0.78
1.00
4.01
3.39
7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25
δ (ppm)
Hb
0.78
1.00
4.01
3.39
5.01
2.01
2.02
0.82
1.00
3.00
3.00
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 0.0
δ (ppm)

Figure S3. UV-vis absorption and fluorescence spectra of **4a-5d**




4i
sol
nano
nano
solid
Abs
FL Intensity
Wavelength (nm)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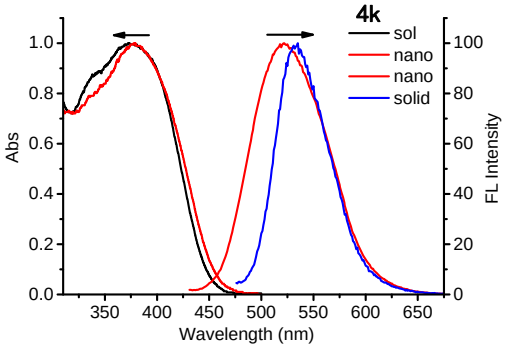
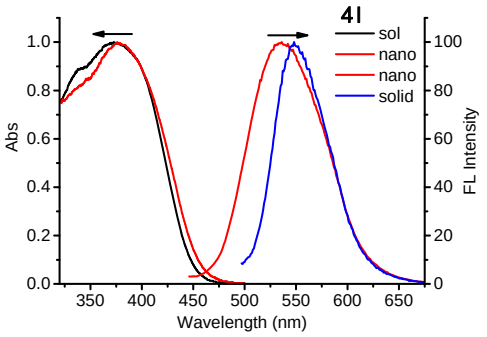
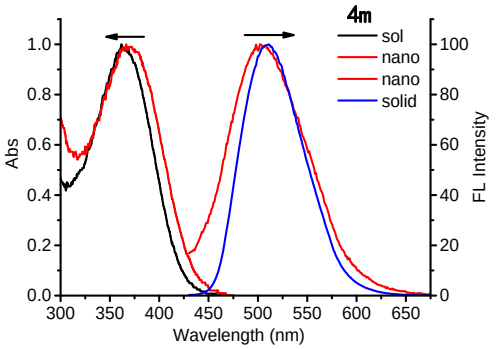
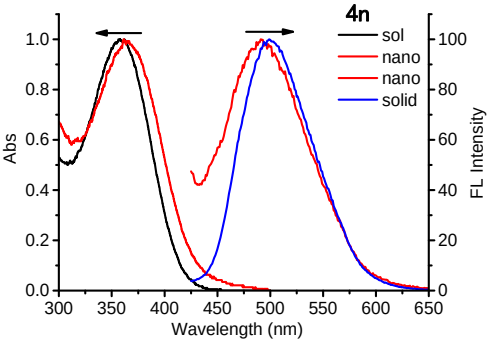
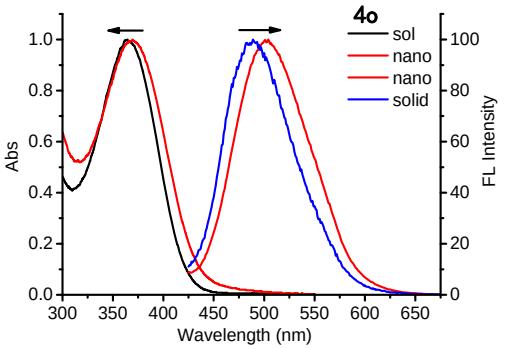
sol
nano
nano
solid
Abs
FL Intensity
Wavelength (nm)

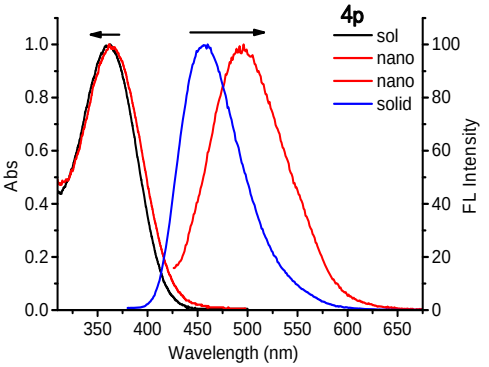

4k
sol
nano
nano
solid
Abs
FL Intensity
Wavelength (nm)

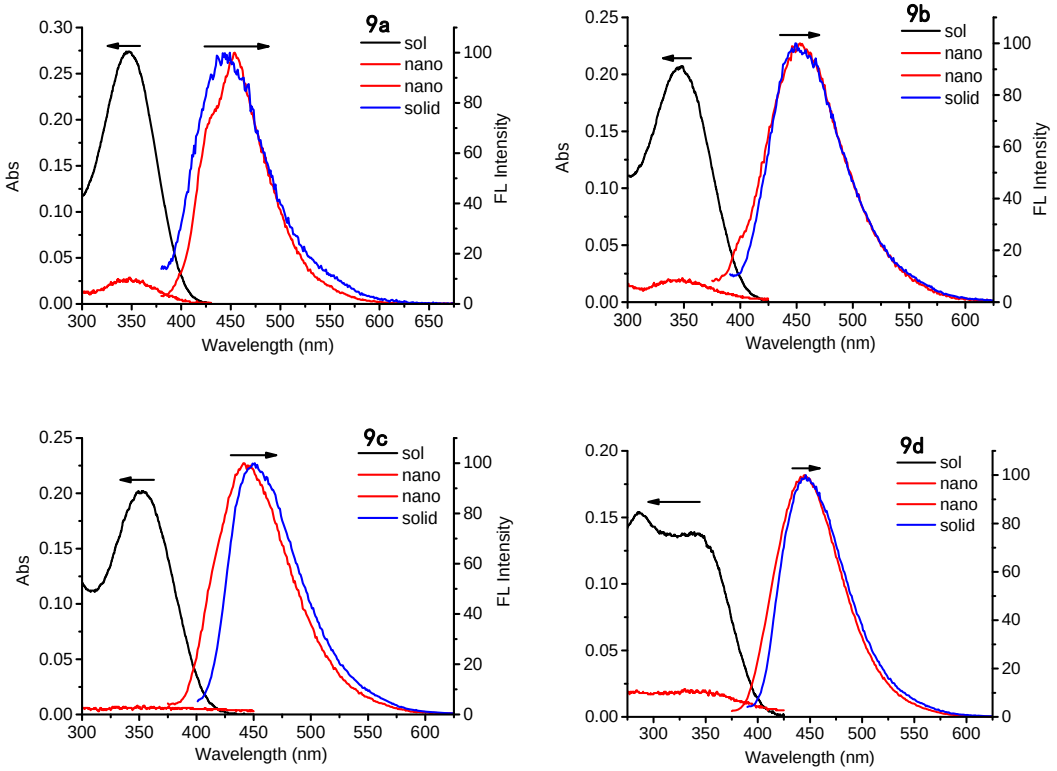

4l
sol
nano
nano
solid
Abs
FL Intensity
Wavelength (nm)


4m
sol
nano
nano
solid
Abs
FL Intensity
Wavelength (nm)


4n
sol
nano
nano
solid
Abs
FL Intensity
Wavelength (nm)


4o
sol
nano
nano
solid
Abs
FL Intensity
Wavelength (nm)


4p
sol
nano
nano
solid
Abs
FL Intensity
Wavelength (nm)



All the absorption and emission spectra were obtained in a concentration scale of 10^{-5} M. Then they were normalized except the absorption spectra of **5a-d**. Back line respects absorption in the THF. Red line respect absorption and emission in THF/water mixtures (5:95), respectively. Blue line stands for emission in solid.

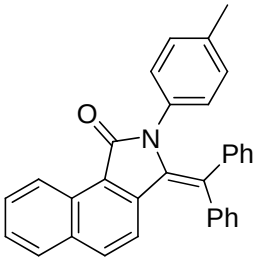
Table S3. Data of DFT Studies

Method / Compound	UV Absorption (nm) [a]	B3LYP/6-31G* LUMO (Hartree)	HOMO (Hartree)	Gap (Hartree)	Gap (nm)	% Δ[b]
4a	373	-0.07686	-0.19821	0.12135	375	0.7
4b	373	-0.08789	-0.20940	0.12151	375	0.5
4c	372	-0.08745	-0.20712	0.11967	381	2.3
4d	376	-0.08215	-0.20465	0.12250	372	-1.1
4e	372	-0.08904	-0.20949	0.12045	378	1.7
4f	405	-0.08240	-0.19788	0.11548	395	-2.6
4g	375	-0.09815	-0.21873	0.12058	378	0.8
4h	394	-0.08900	-0.20455	0.11555	394	0
4i	412	-0.08484	-0.19799	0.11315	403	-2.3
4j	387	-0.08279	-0.20239	0.11960	381	-1.6
4k	374	-0.09337	-0.21318	0.11981	380	1.7
4l	375	-0.08386	-0.20716	0.12330	370	-1.5
4m	362	-0.08704	-0.21248	0.12544	363	0.3
4n	357	-0.07843	-0.20627	0.12784	356	-0.2
4o	364	-0.08053	-0.20635	0.12582	362	-0.5
4p	362	-0.07522	-0.20373	0.12851	355	-2.1
5a	348	-0.08218	-0.21369	0.13151	346	-0.4
5b	348	-0.08051	-0.21113	0.13062	349	0.2
5c	349	-0.08977	-0.21950	0.12973	351	0.6
5d	343	-0.07770	-0.21056	0.13286	343	0

[a] The wavelength of absorption was obtained by UV-vis spectrometer in the THF.

[b] %Δ = 100 x (Gap - Absorption)/Gap

HOMO LUMO



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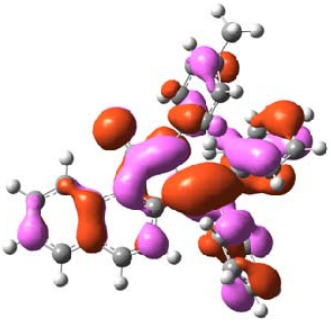
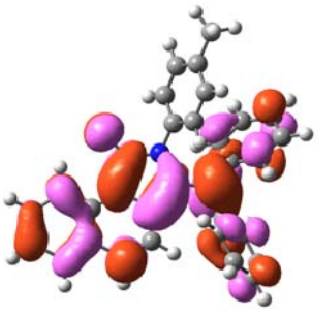
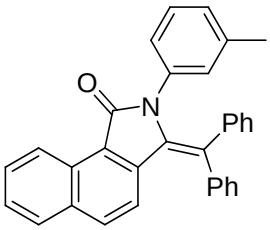
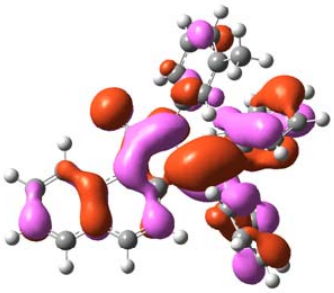


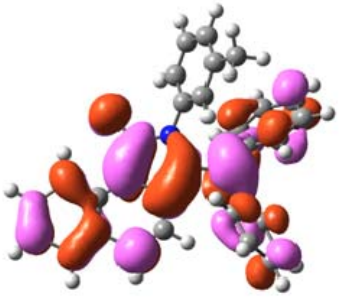


-0.19821 **0.12135** -0.07686

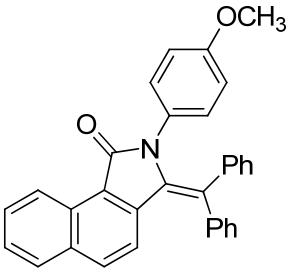


4b





-0.20940 **0.12151** -0.08789



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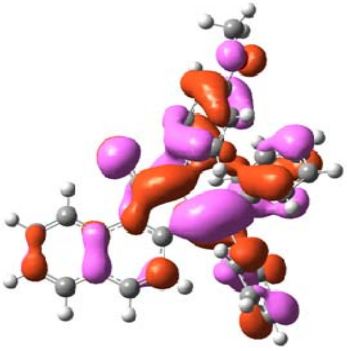
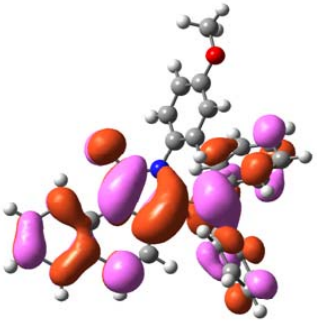
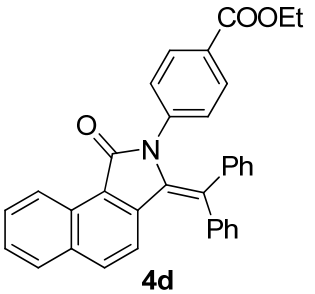




-0.20712 **0.11967** -0.08745



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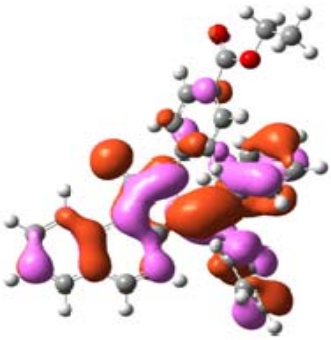
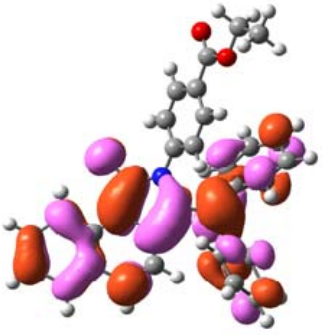
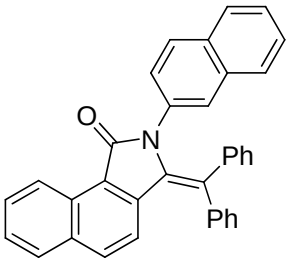




-0.20465 **0.12250** -0.08215

HOMO LUMO



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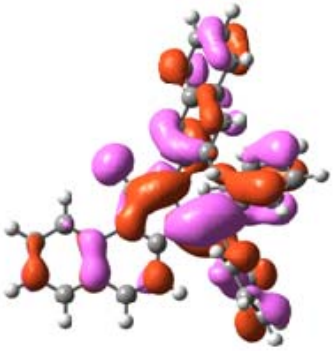
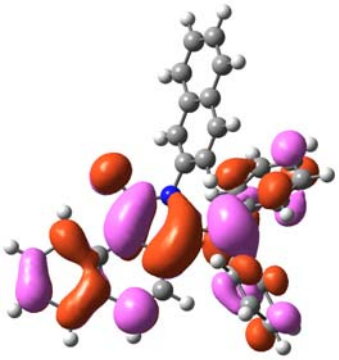
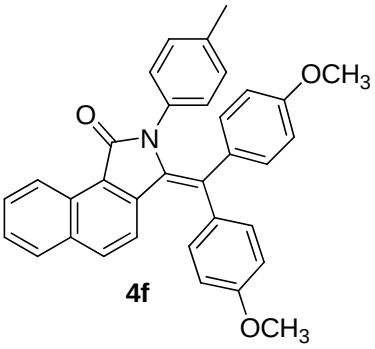




-0.20949 **0.12045** -0.08904



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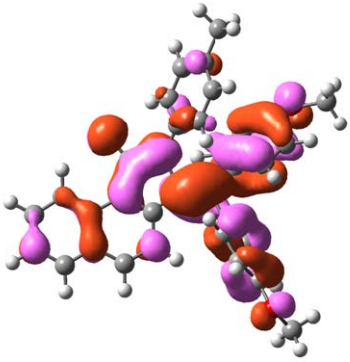
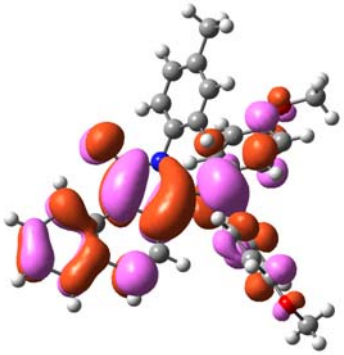
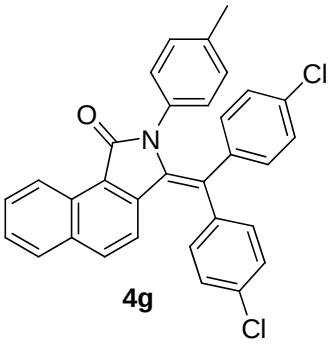




-0.19788 **0.11548** -0.08240



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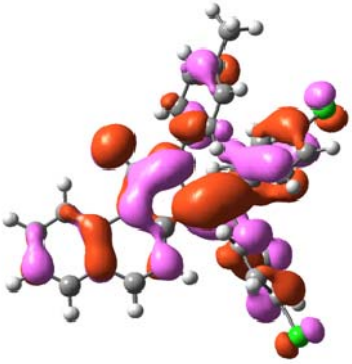
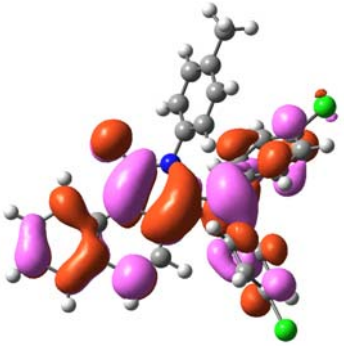
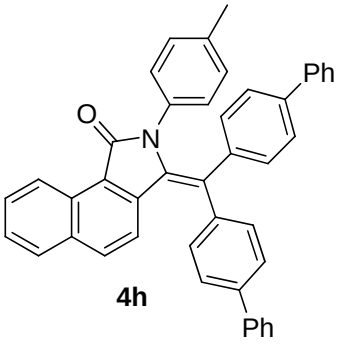




-0.21873 **0.12058** -0.09815



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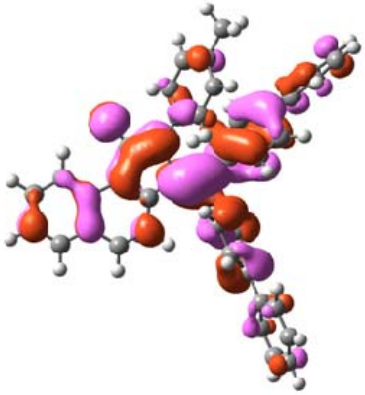
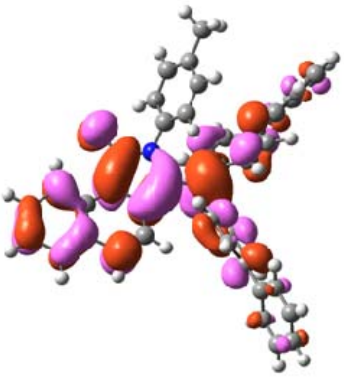
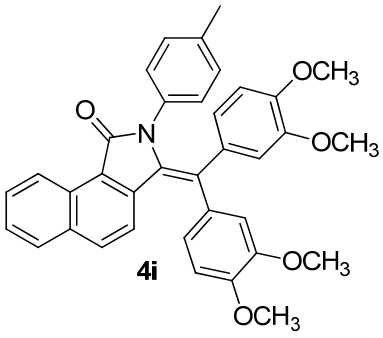




-0.20455 **0.11555** -0.08900

HOMO LUMO



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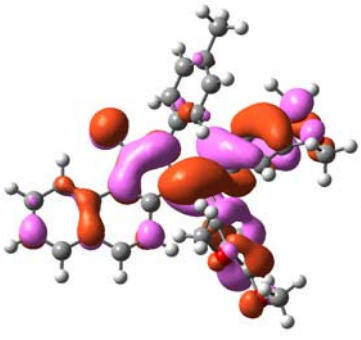

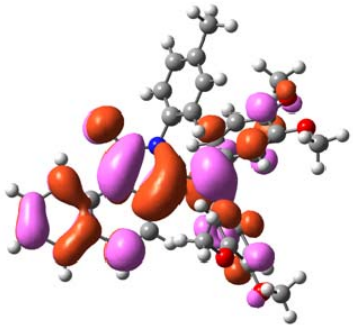

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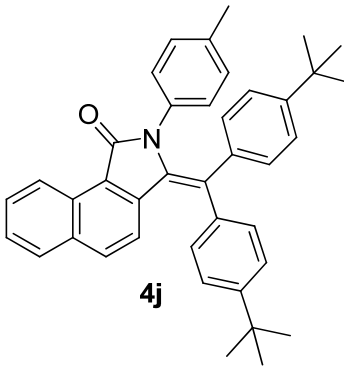

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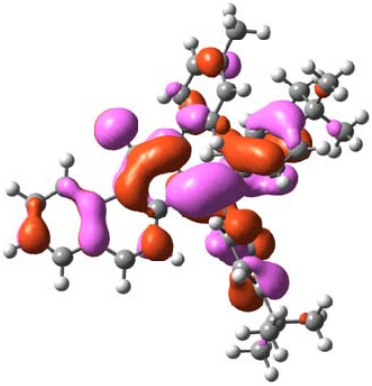

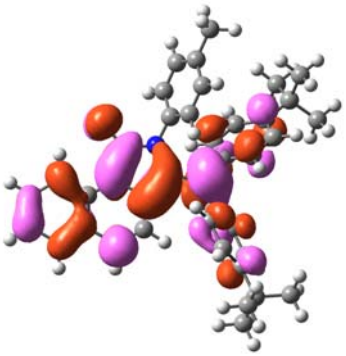

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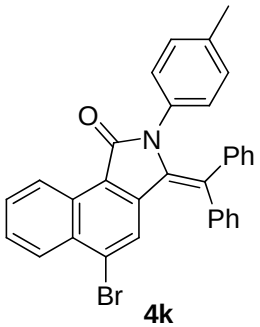

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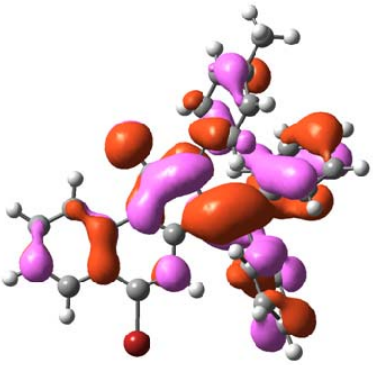

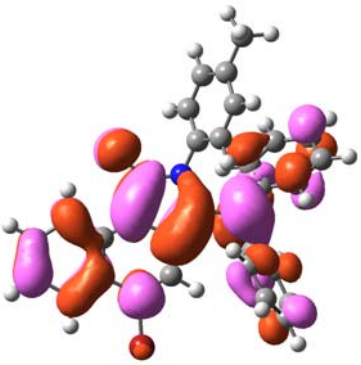

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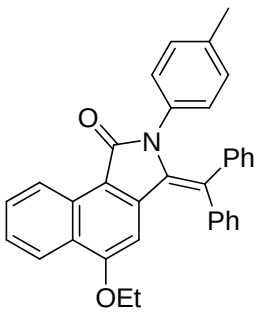

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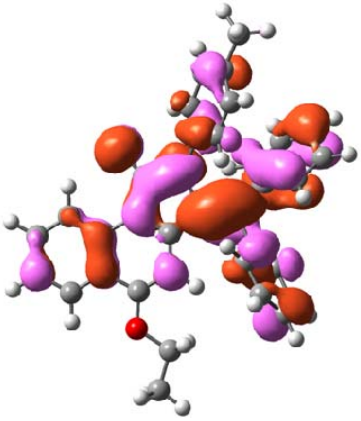
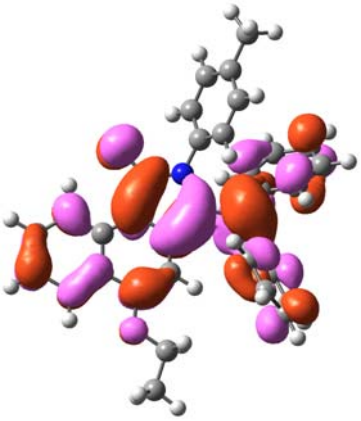
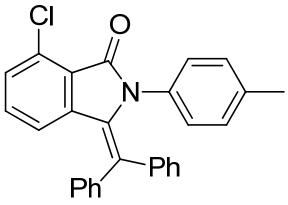




-0.20716 **0.12330** -0.08386

HOMO LUMO



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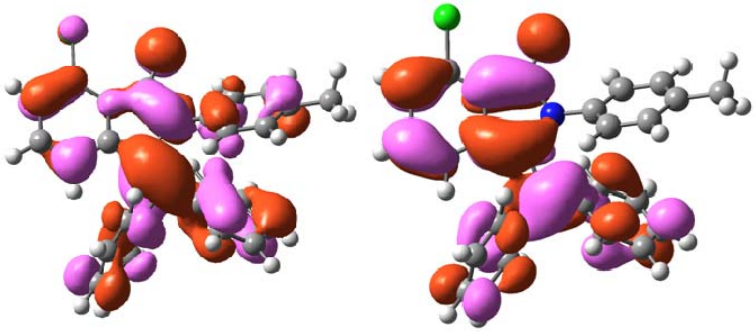

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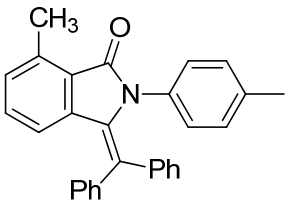

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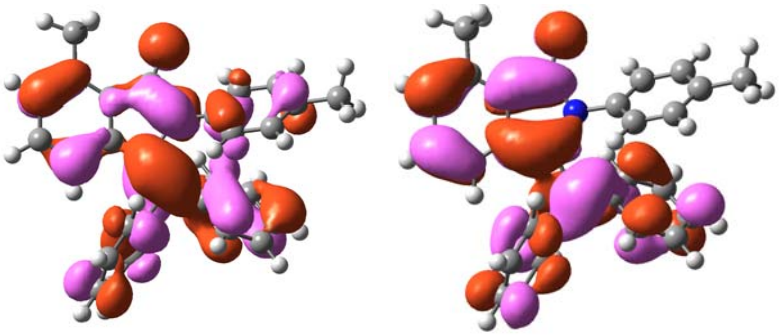

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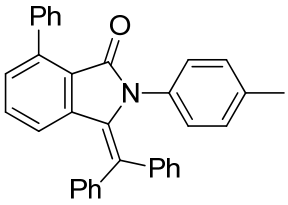

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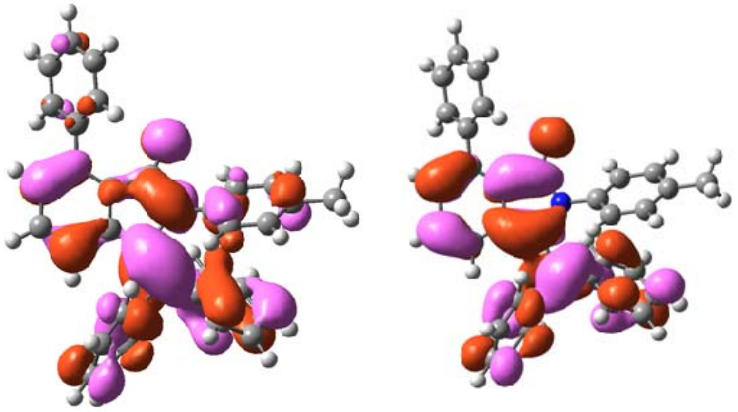

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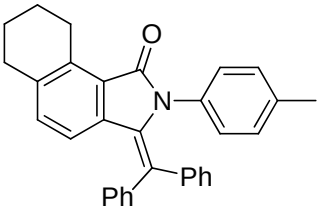

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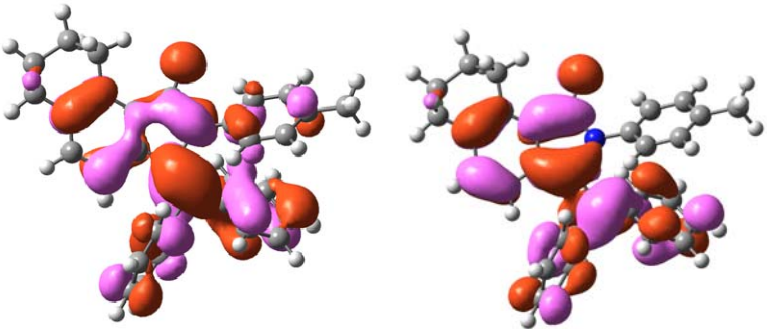
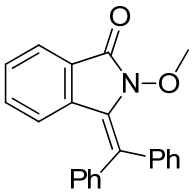
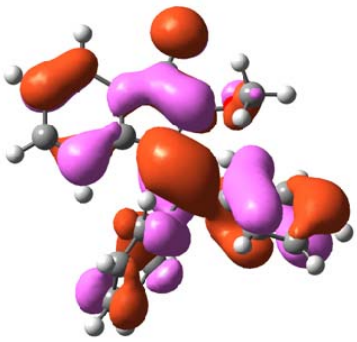


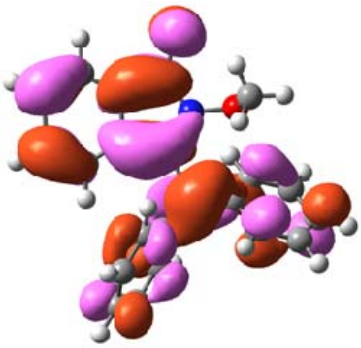
-0.20373 **0.12851** -0.07522

HOMO LUMO

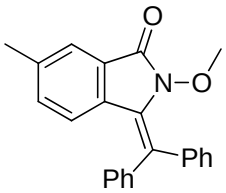


9a

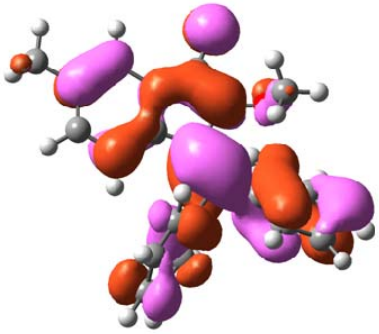


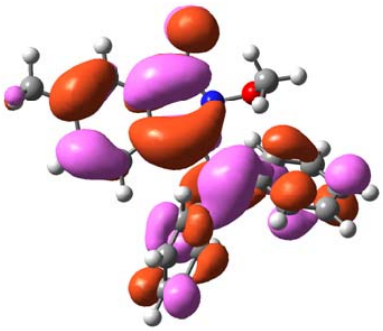


-0.21369 **0.13151** -0.08218

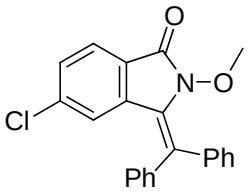


9b





-0.21113 **0.13062** -0.08051



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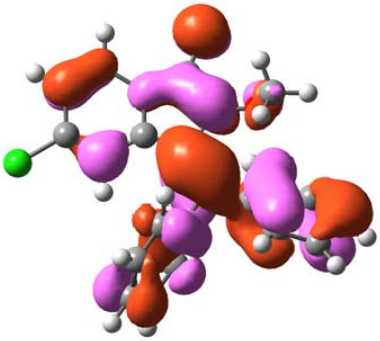
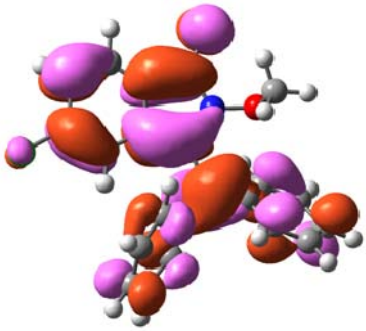
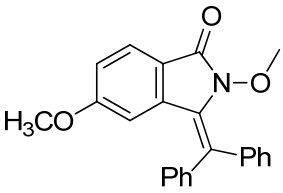
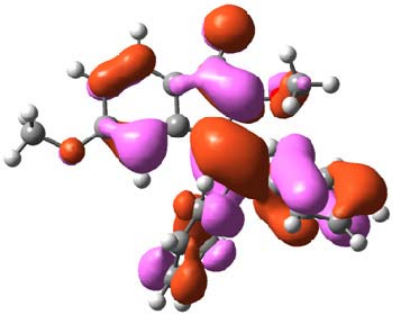


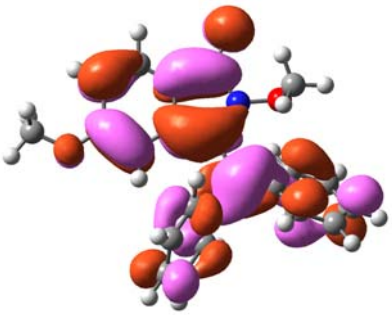


-0.21950 **0.12973** -0.08977



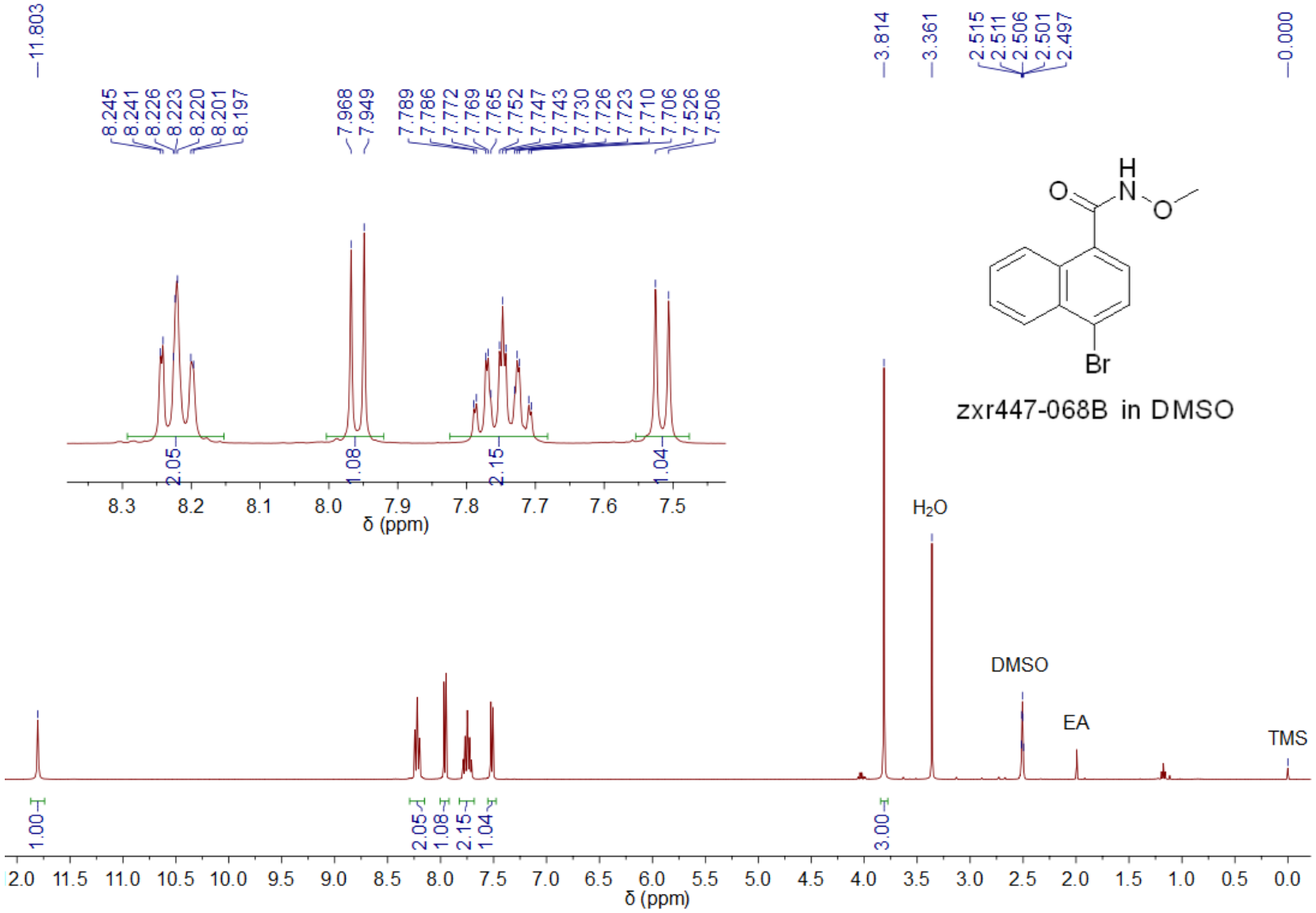
9d



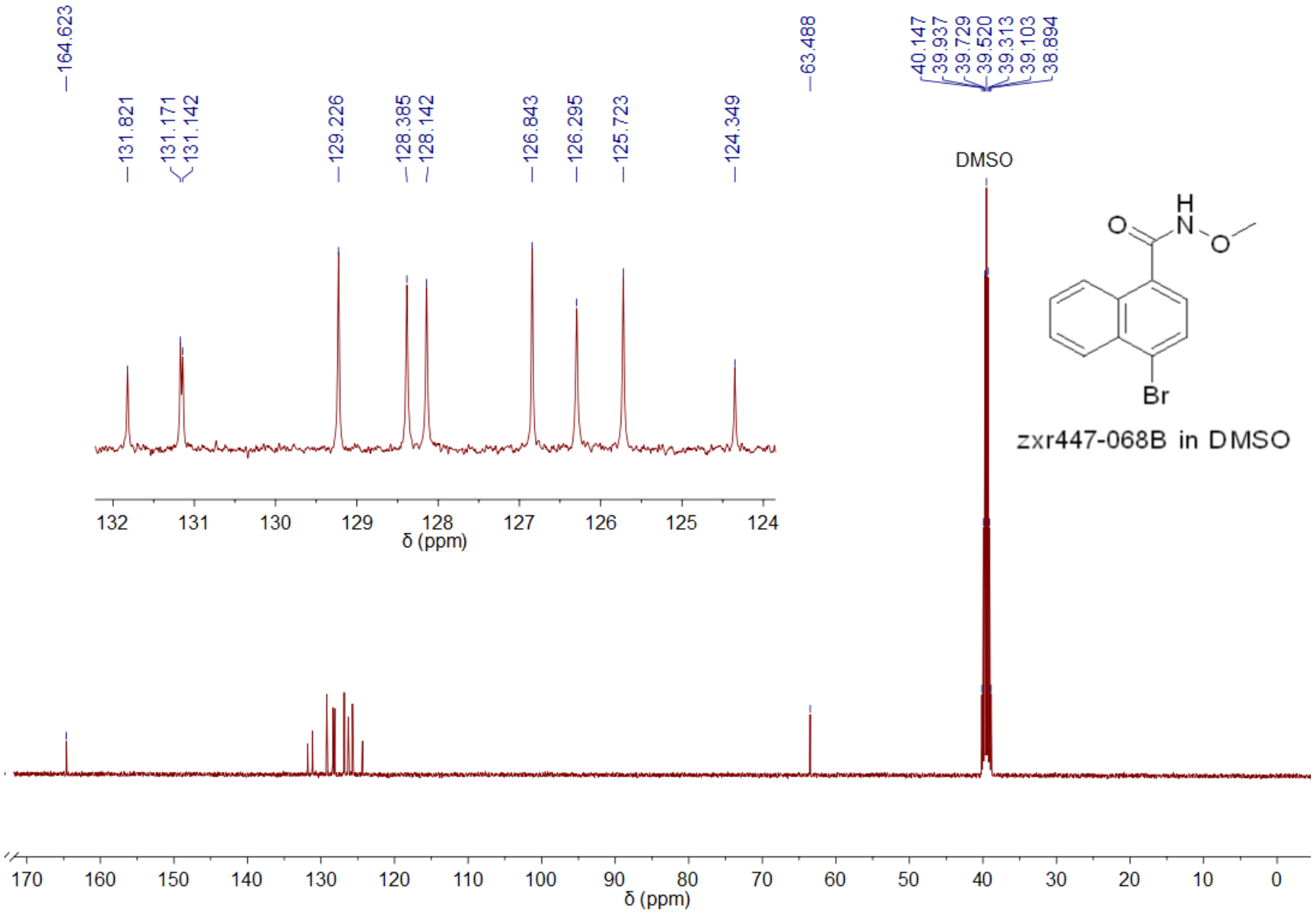


-0.21056 **0.13286** -0.07770

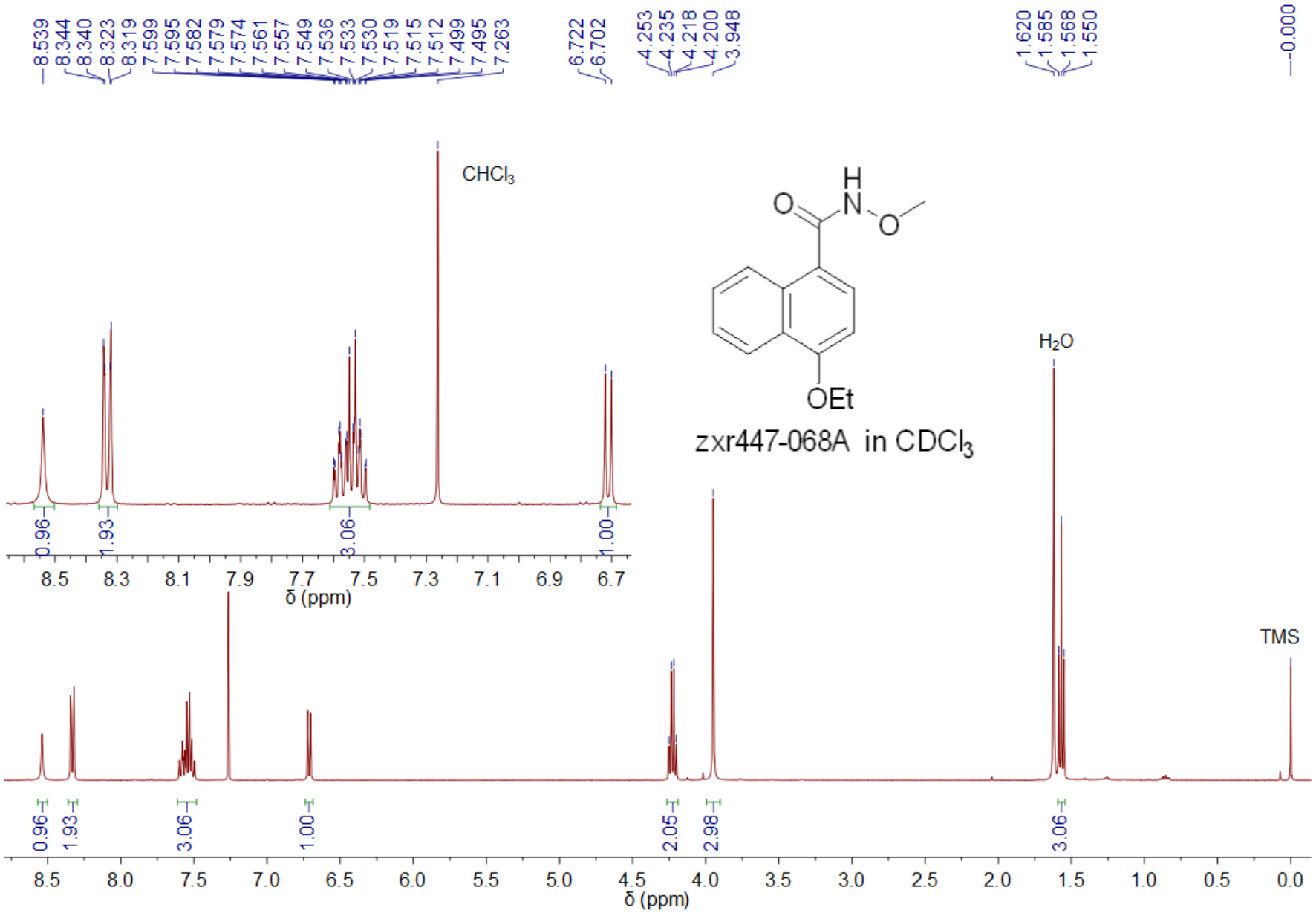
Copies of NMR Spectra



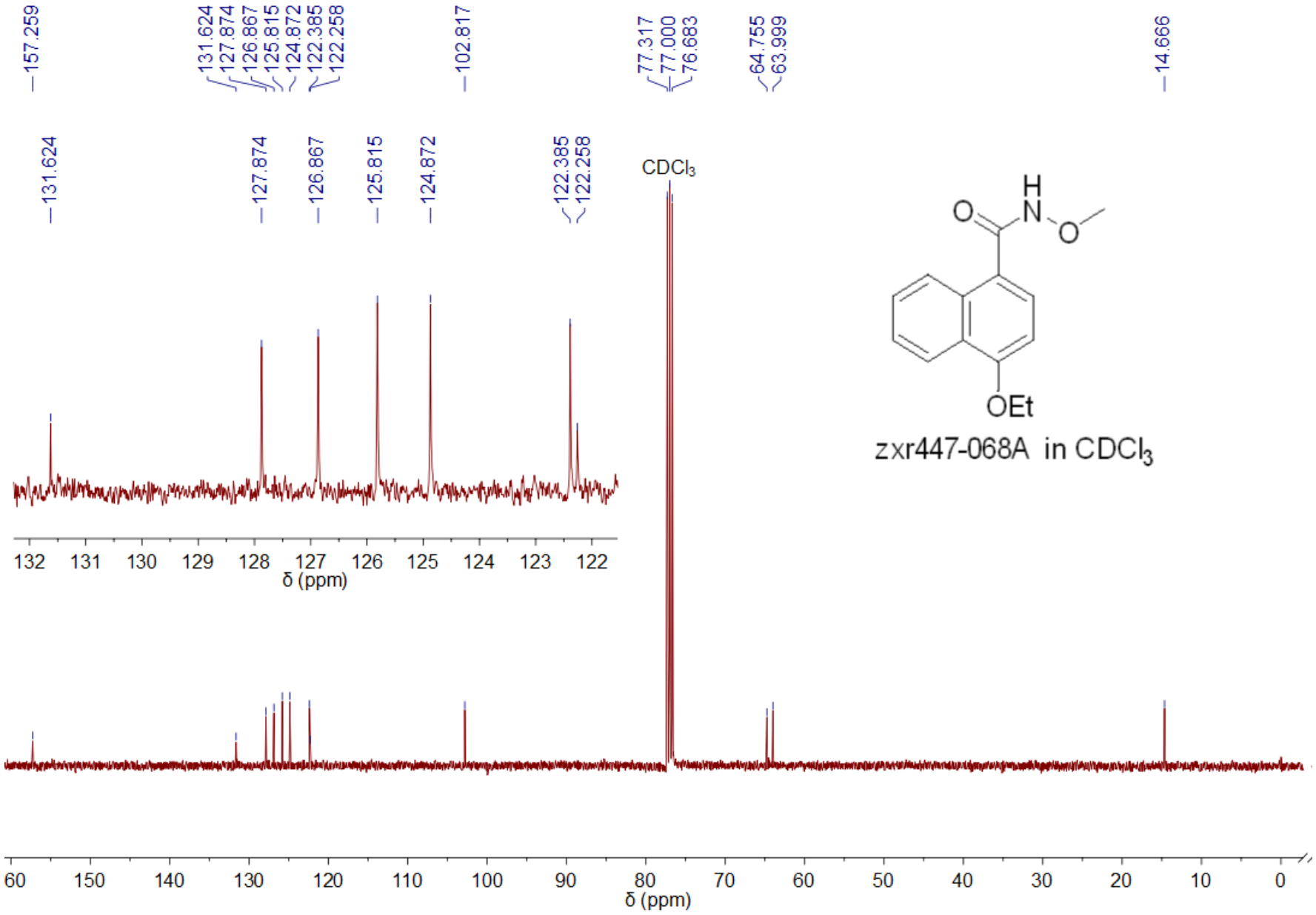
1p - ^{1}H NMR


164.623
131.821
131.171
131.142
129.226
128.385
128.142
126.843
126.295
125.723
124.349
63.488
40.147
39.937
39.729
39.520
39.313
39.103
38.894
DMSO
O
H
N
O
Br
zxr447-068B in DMSO
132
131
130
129
128
127
126
125
124
δ (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

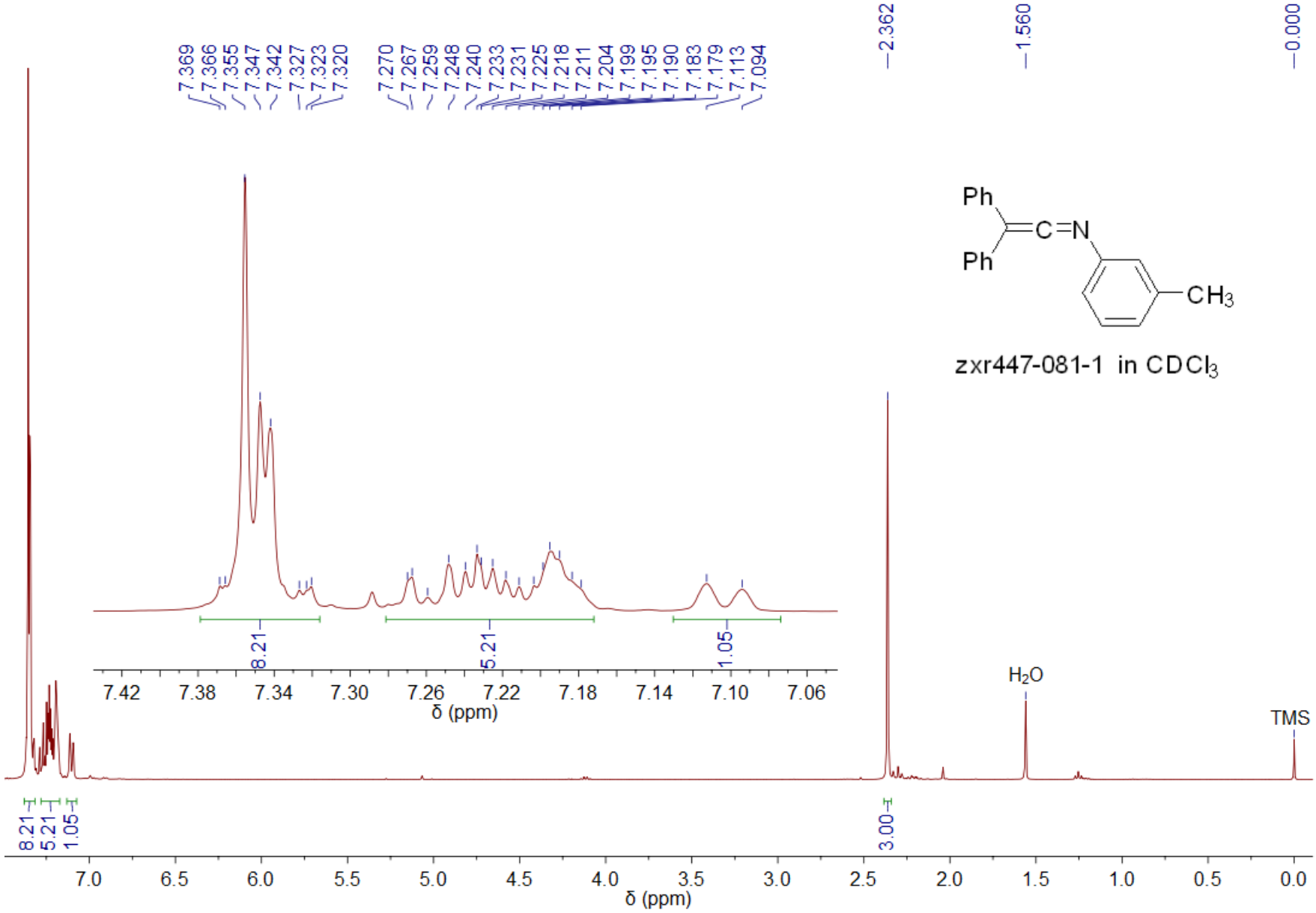
1p - ^{13}C NMR


8.539
8.344
8.340
8.323
8.319
7.599
7.595
7.582
7.579
7.574
7.561
7.557
7.549
7.536
7.533
7.530
7.519
7.515
7.512
7.499
7.495
7.263
6.722
6.702
4.253
4.235
4.218
4.200
3.948
1.620
1.585
1.568
1.550
0.000
CHCl3
H2O
TMS
OEt
zxr447-068A in CDCl3
0.96
1.93
3.06
1.00
2.05
2.98
δ (ppm)

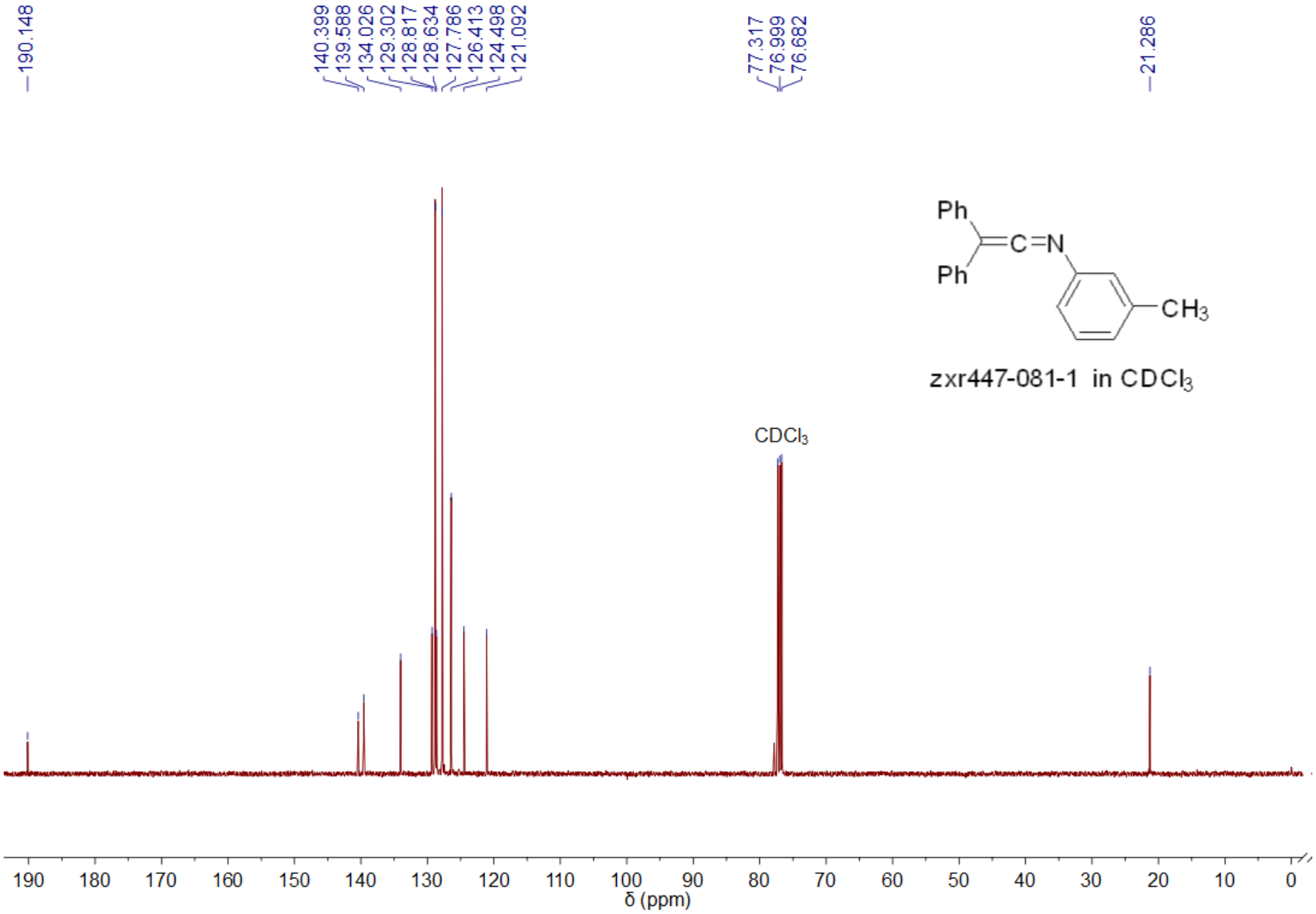
1q - ^{1}H NMR


157.259
131.624
127.874
126.867
125.815
124.872
122.385
122.258
102.817
77.317
77.000
76.683
64.755
63.999
14.666
CDCl3
O
H
N
O
OEt
zxr447-068A in CDCl3
δ (ppm)
δ (ppm)

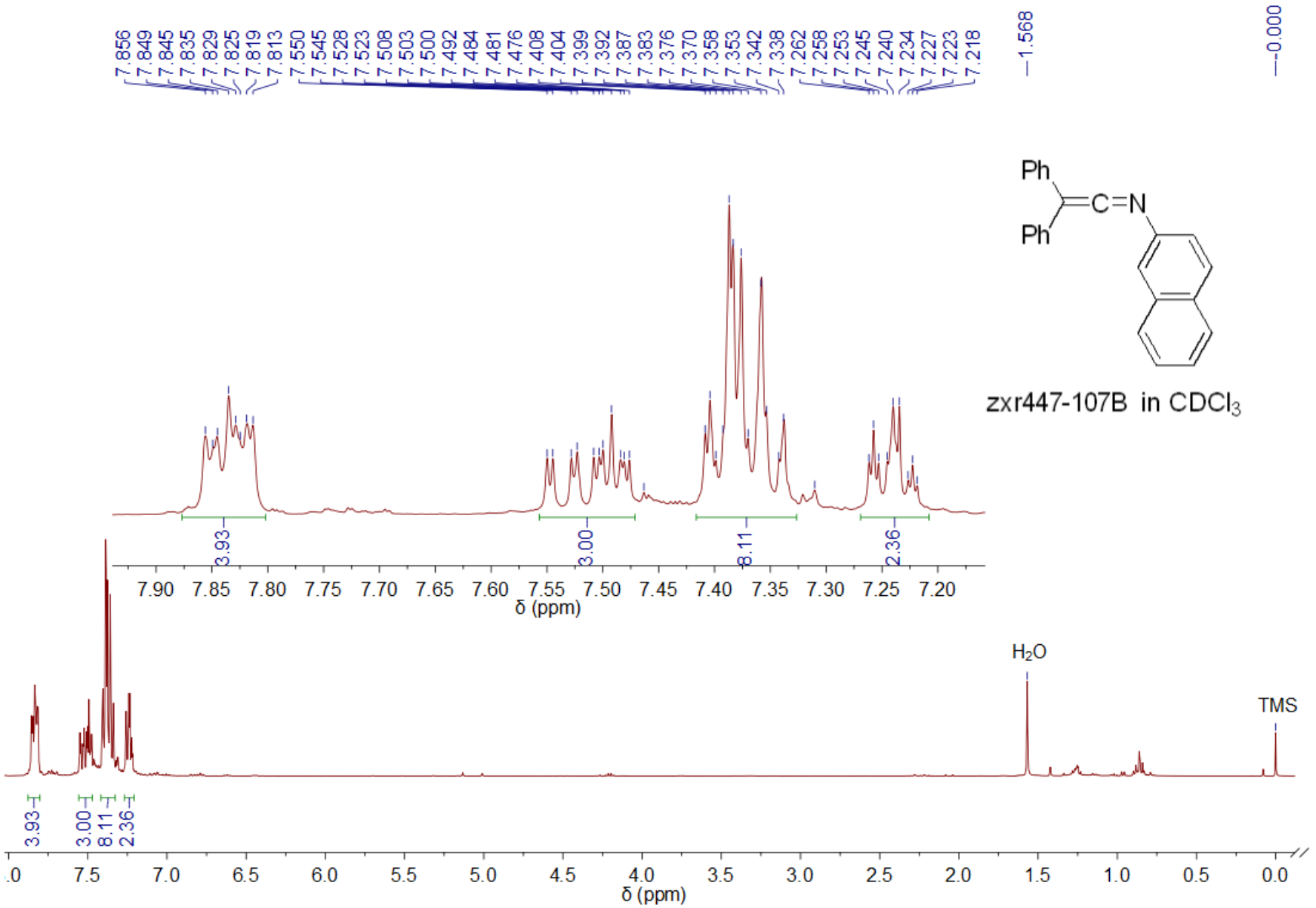
1q - ^{13}C NMR


7.369
7.366
7.355
7.347
7.342
7.327
7.323
7.320
7.270
7.267
7.259
7.248
7.240
7.233
7.231
7.225
7.218
7.211
7.204
7.199
7.195
7.190
7.183
7.179
7.113
7.094
2.362
1.560
0.000
Ph
Ph
C=N
CH3
zxr447-081-1 in CDCl3
8.21
5.21
1.05
3.00
H2O
TMS
δ (ppm)

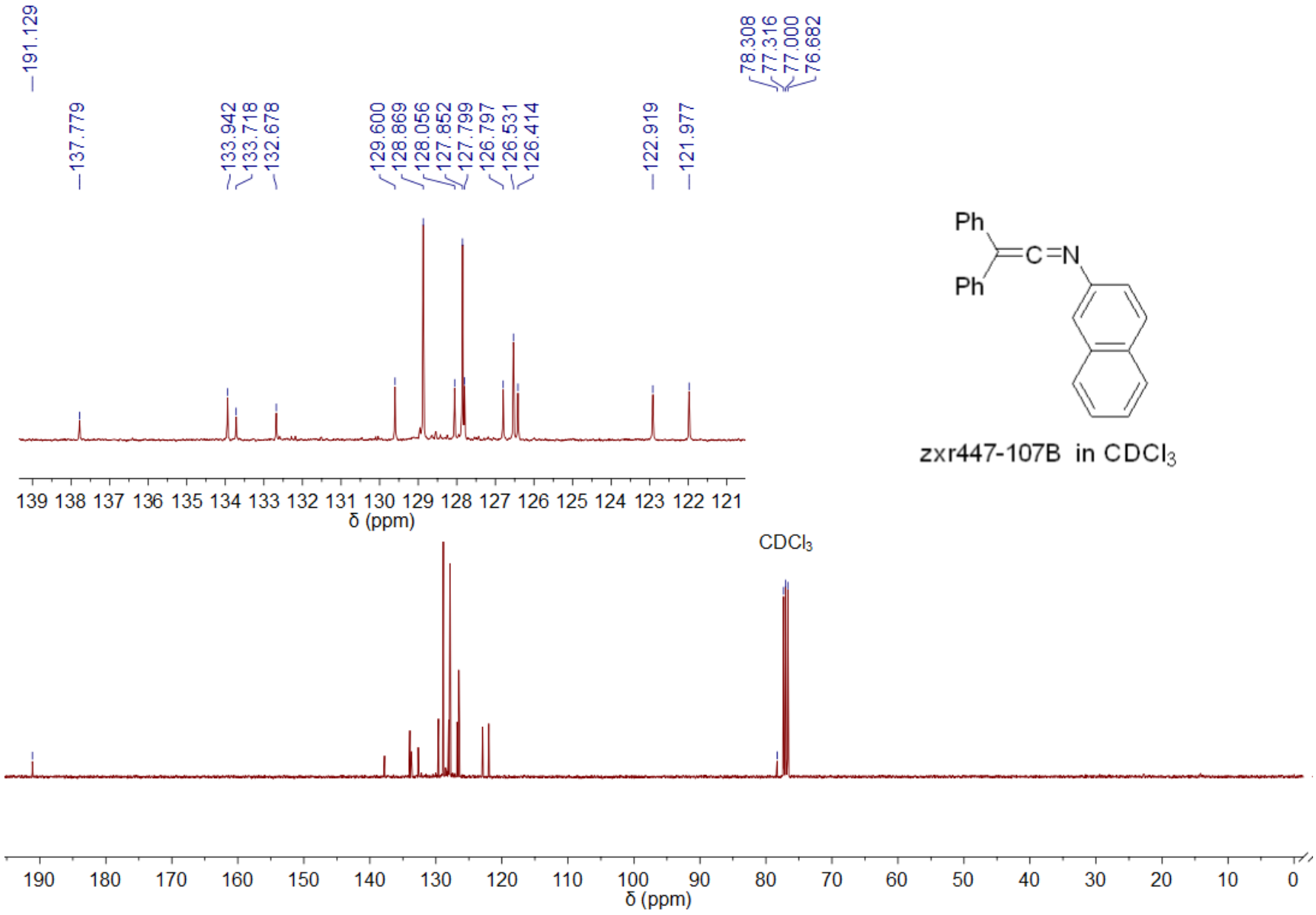
2f - ^{1}H NMR


190.148
140.399
139.588
134.026
129.302
128.817
128.634
127.786
126.413
124.498
121.092
77.317
76.999
76.682
21.286
Ph
Ph
C=N
CH3
zxr447-081-1 in CDCl3
CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

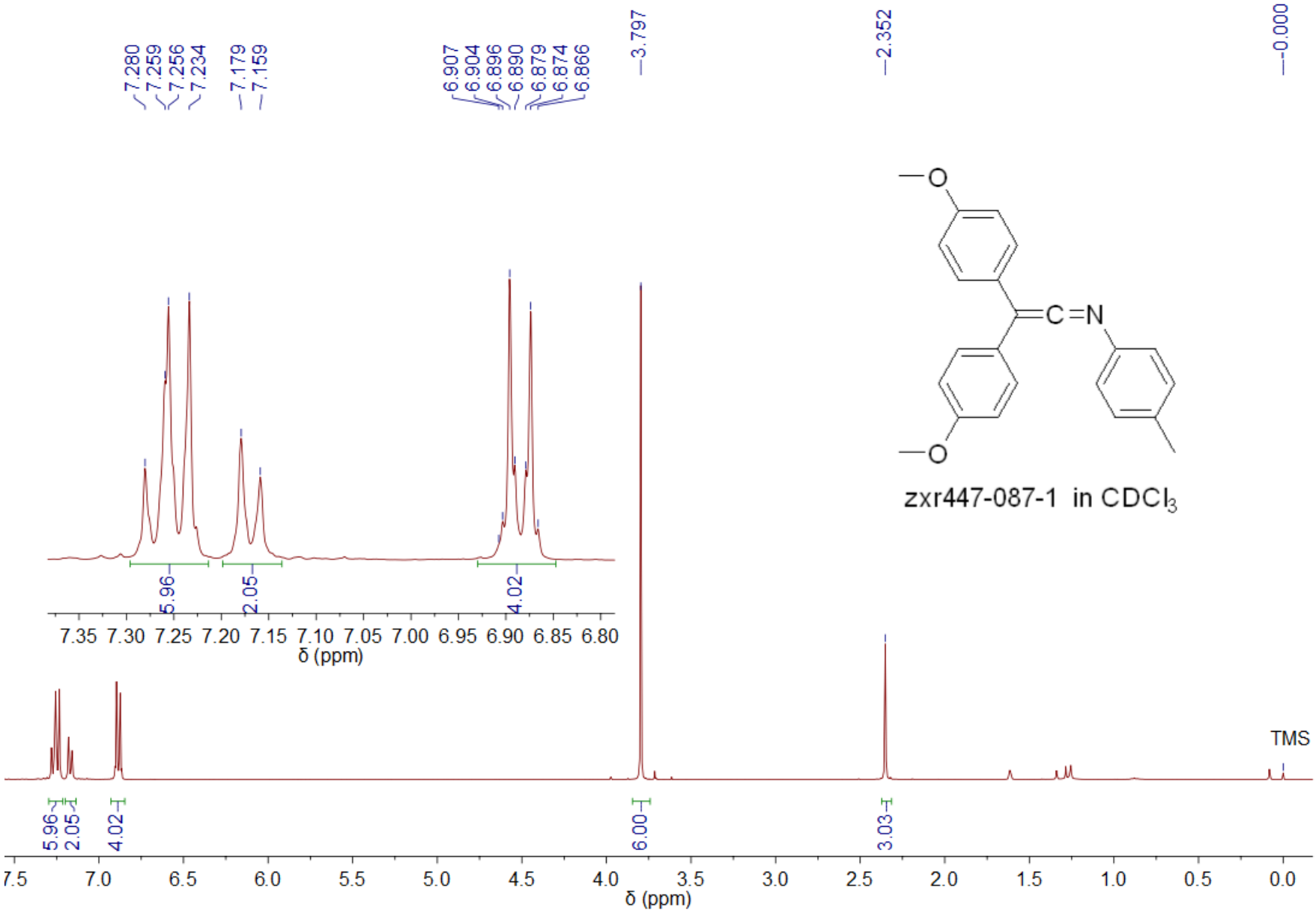
2f - ^{13}C NMR


7.856
7.849
7.845
7.835
7.829
7.825
7.819
7.813
7.550
7.545
7.528
7.523
7.508
7.503
7.500
7.492
7.484
7.481
7.476
7.408
7.404
7.399
7.392
7.387
7.383
7.376
7.370
7.358
7.353
7.342
7.338
7.262
7.258
7.253
7.245
7.240
7.234
7.227
7.223
7.218
1.568
0.000
Ph
Ph
C=N
zxr447-107B in CDCl3
3.93
3.00
8.11
2.36
7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20
δ (ppm)
H2O
TMS
3.93
3.00
8.11
2.36
.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 0.0
δ (ppm)

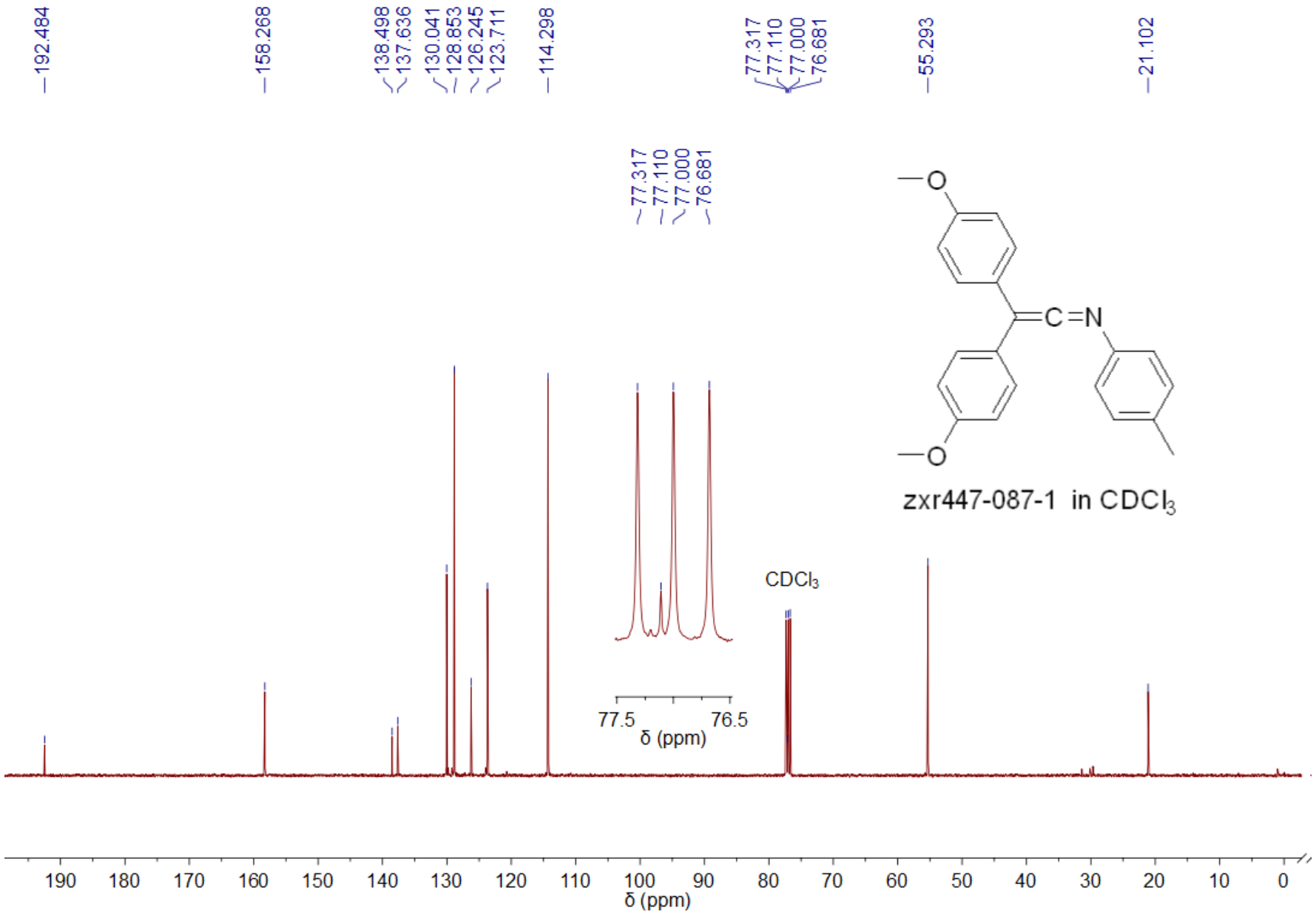
2g - ^{1}H NMR


191.129
137.779
133.942
133.718
132.678
129.600
128.869
128.056
127.852
127.799
126.797
126.531
126.414
122.919
121.977
78.308
77.316
77.000
76.682
Ph
Ph
C
N
zxr447-107B in CDCl3
139 138 137 136 135 134 133 132 131 130 129 128 127 126 125 124 123 122 121
δ (ppm)
CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

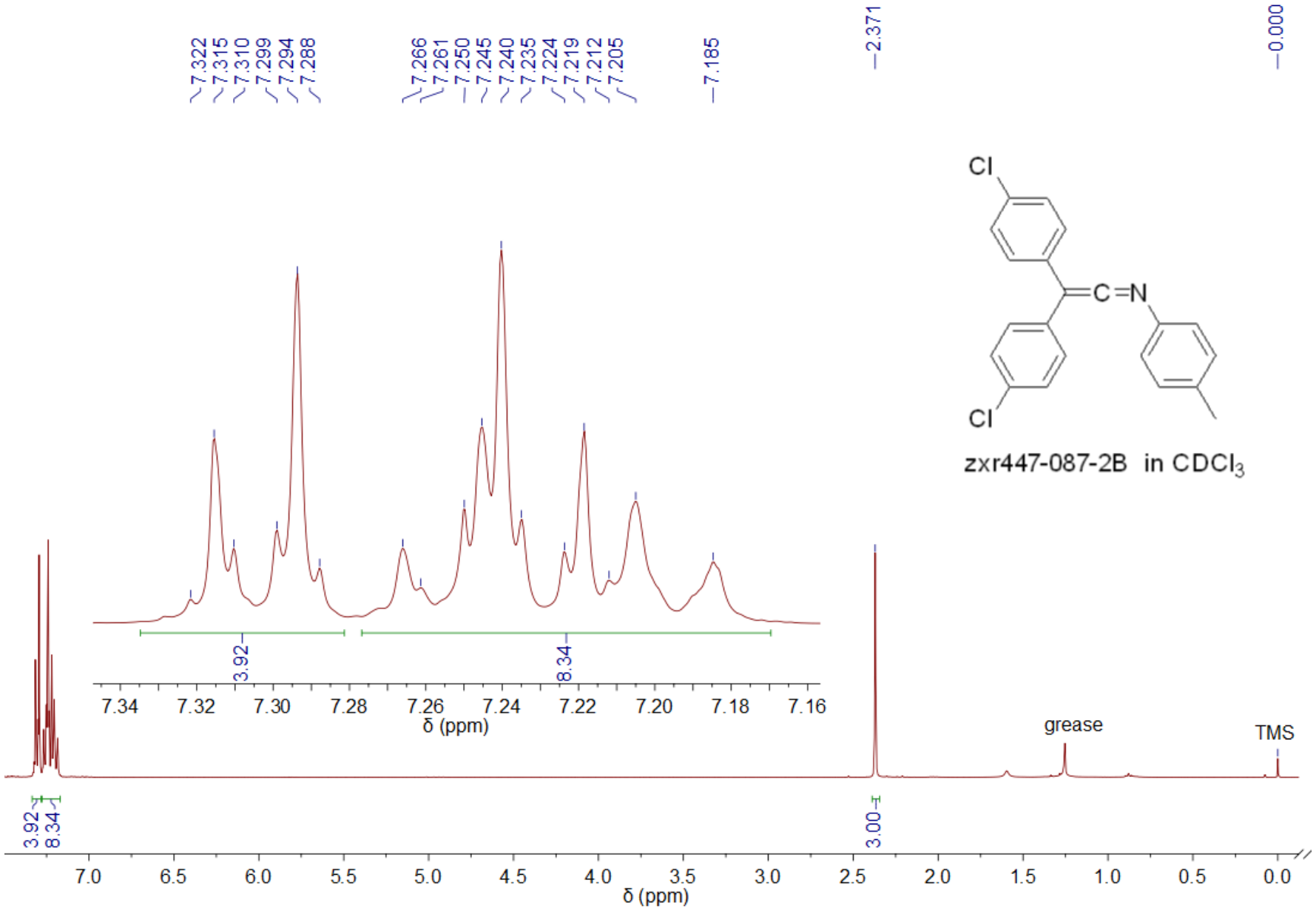
2g - ^{13}C NMR


7.280
7.259
7.256
7.234
7.179
7.159
6.907
6.904
6.896
6.890
6.879
6.874
6.866
3.797
2.352
0.000
O
C=N
O
zxr447-087-1 in CDCl3
5.96
2.05
4.02
7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80
δ (ppm)
TMS
5.96
2.05
4.02
6.00
3.03
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 0.0
δ (ppm)

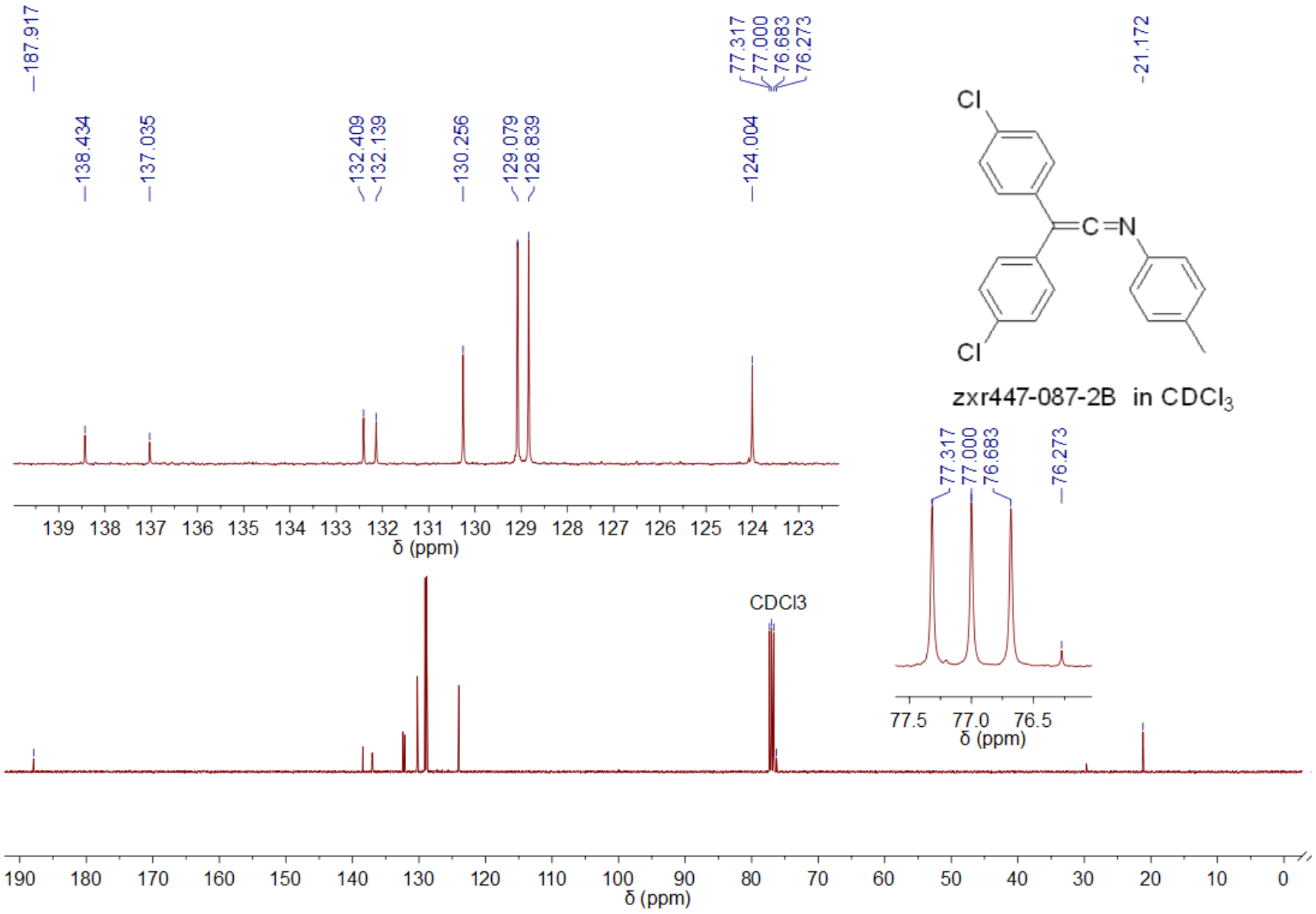
2i - ^{1}H NMR


192.484
158.268
138.498
137.636
130.041
128.853
126.245
123.711
114.298
77.317
77.110
77.000
76.681
55.293
21.102
77.317
77.110
77.000
76.681
O
C=N
O
zxr447-087-1 in CDCl3
CDCl3
77.5
76.5
δ (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

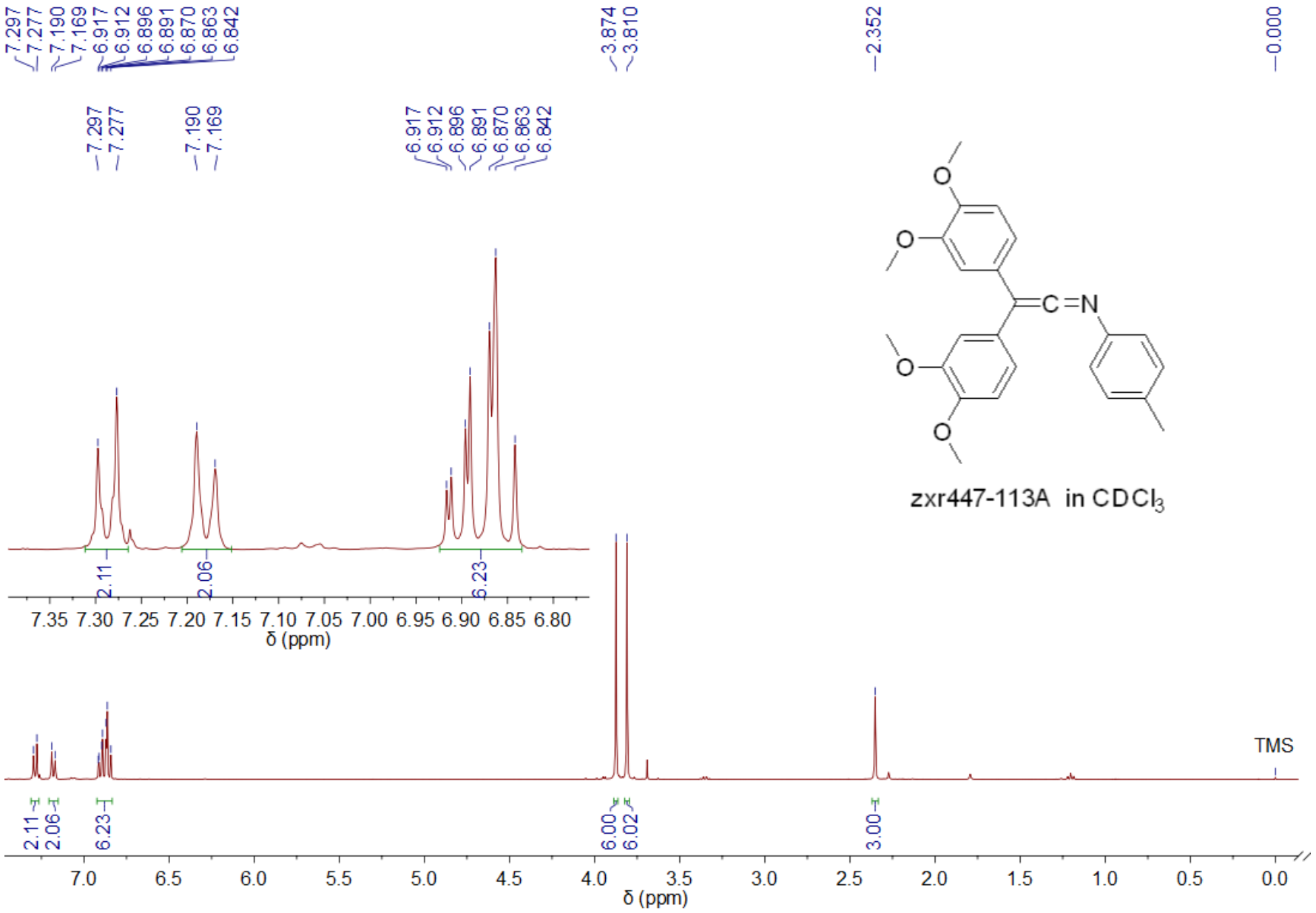
2i - ^{13}C NMR


7.322
7.315
7.310
7.299
7.294
7.288
7.266
7.261
7.250
7.245
7.240
7.235
7.224
7.219
7.212
7.205
7.185
2.371
0.000
Cl
C=N
Cl
zxr447-087-2B in CDCl3
3.92
8.34
7.34 7.32 7.30 7.28 7.26 7.24 7.22 7.20 7.18 7.16
δ (ppm)
grease
TMS
3.92
8.34
3.00
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 0.0
δ (ppm)

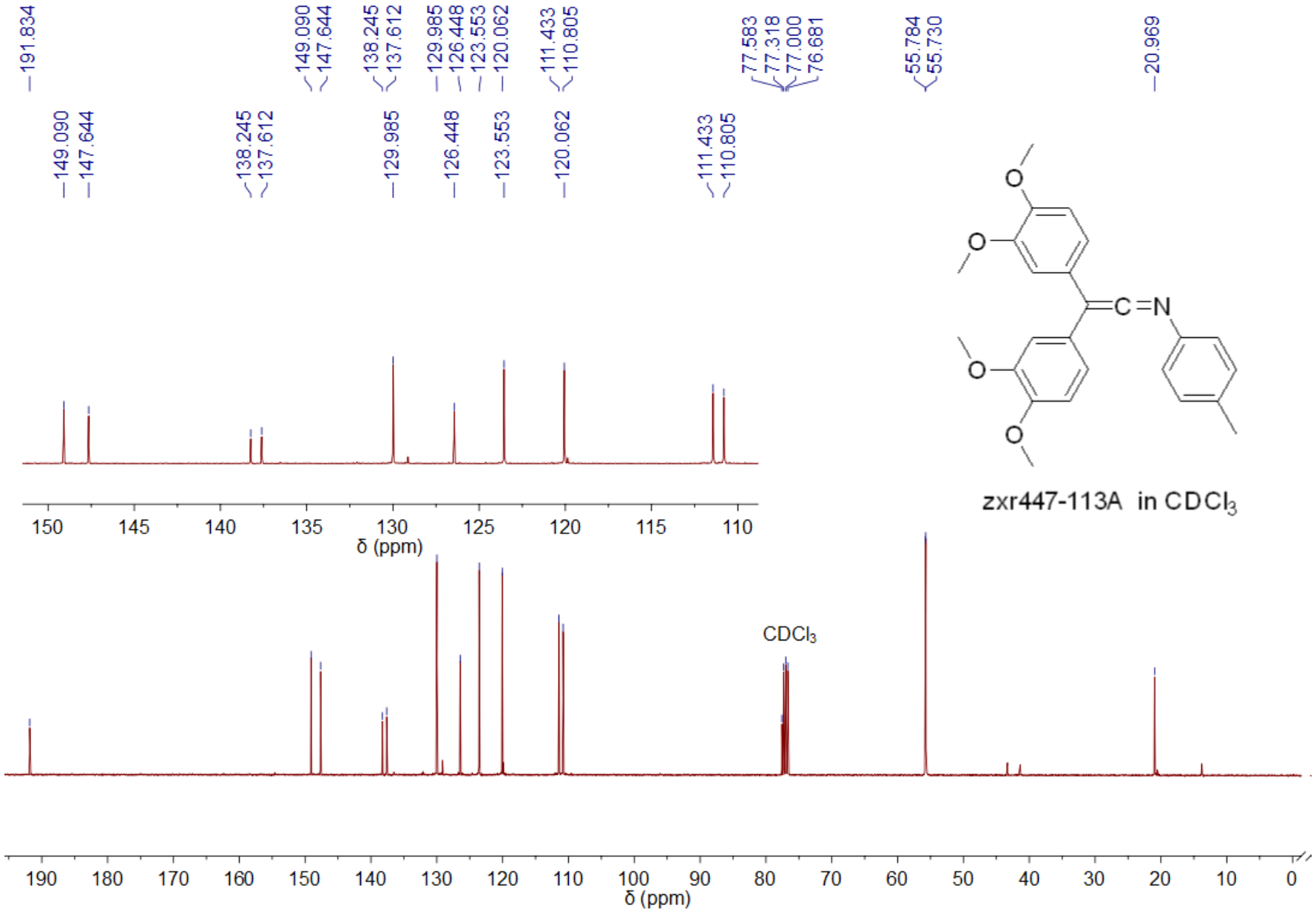
2j - ^{1}H NMR


187.917
138.434
137.035
132.409
132.139
130.256
129.079
128.839
124.004
77.317
77.000
76.683
76.273
21.172
Cl
C=N
Cl
zxr447-087-2B in CDCl3
δ (ppm)
CDCl3
δ (ppm)

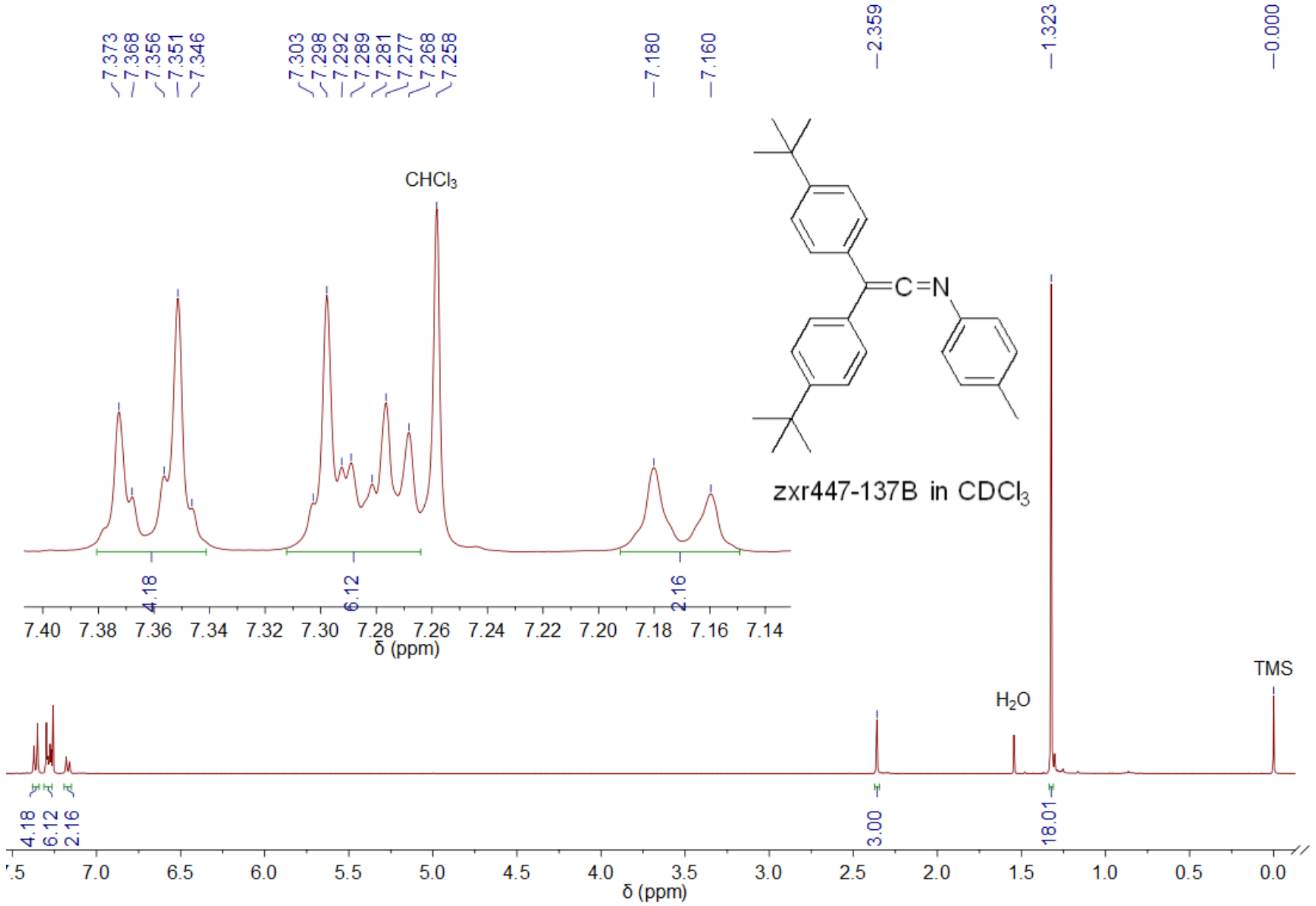
2j - ^{13}C NMR


7.297
7.277
7.190
7.169
6.917
6.912
6.896
6.891
6.870
6.863
6.842
3.874
3.810
2.352
0.000
zxr447-113A in CDCl3
TMS
2.11
2.06
6.23
6.00
6.02
3.00
δ (ppm)

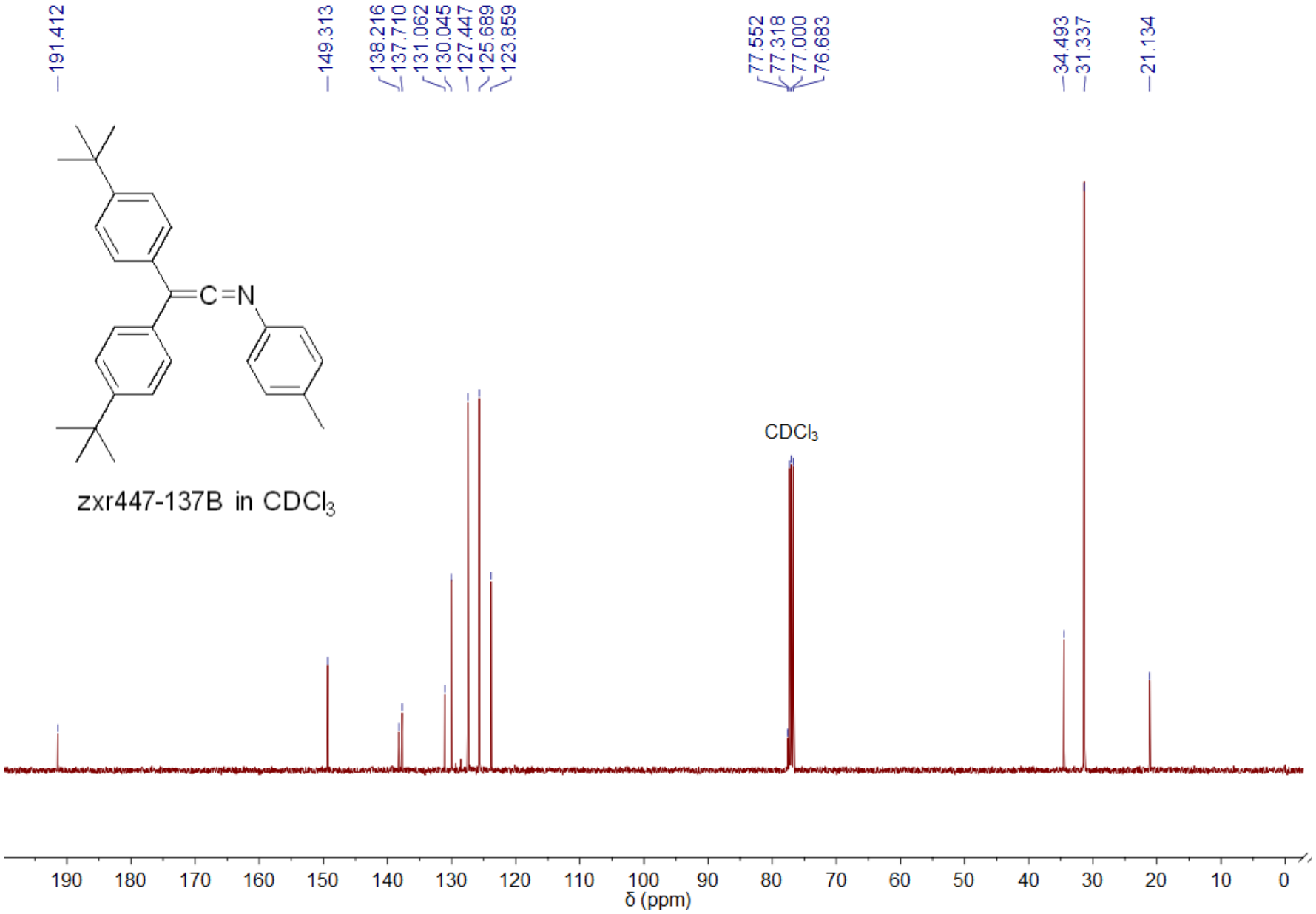
2k - ^{1}H NMR


191.834
149.090
147.644
138.245
137.612
129.985
126.448
123.553
120.062
111.433
110.805
77.583
77.318
77.000
76.681
55.784
55.730
20.969
O
O
C=N
O
O
zxr447-113A in CDCl3
δ (ppm)
CDCl3
δ (ppm)

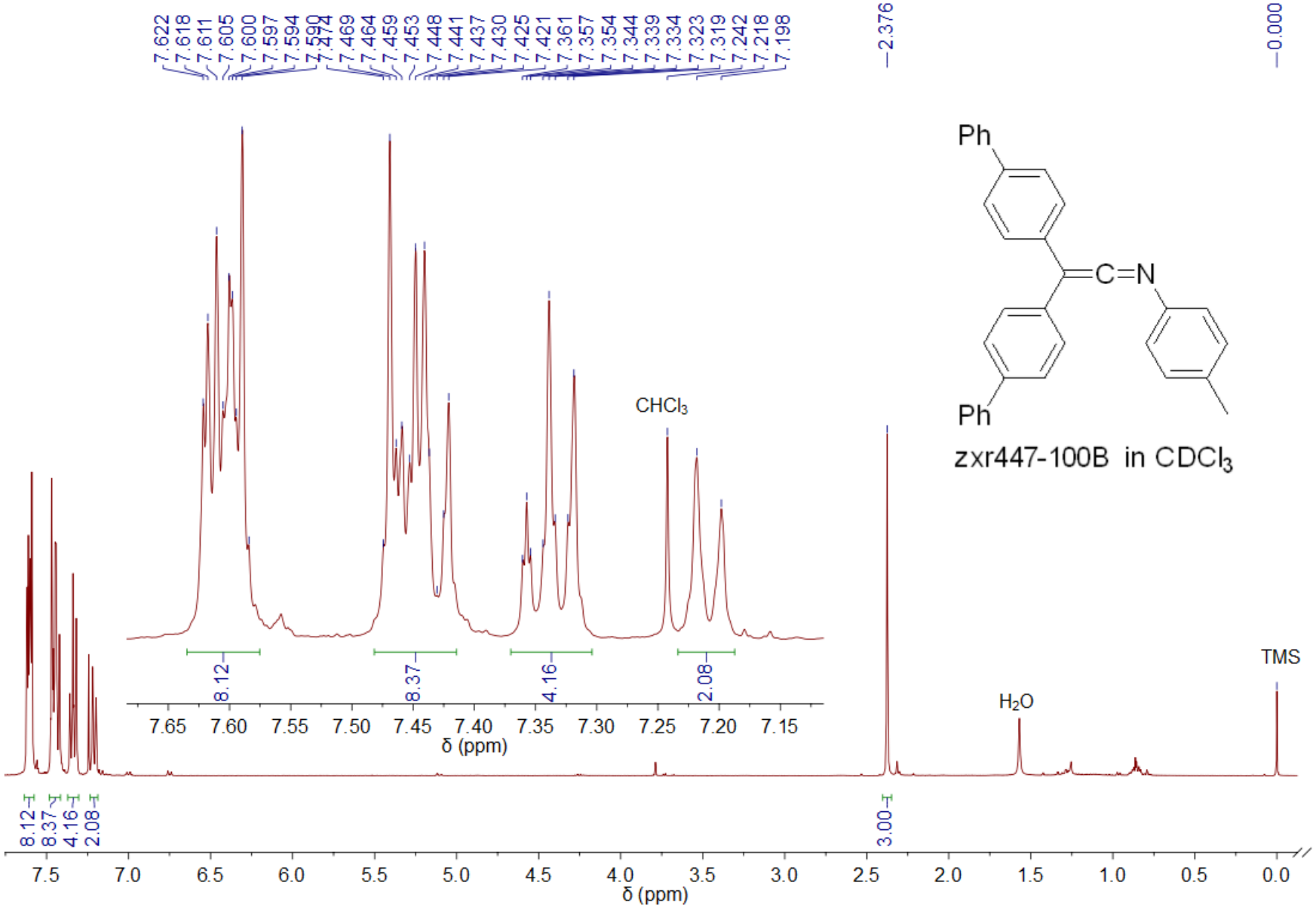
2k - ^{13}C NMR


7.373
7.368
7.356
7.351
7.346
7.303
7.298
7.292
7.289
7.281
7.277
7.268
7.258
7.180
7.160
2.359
1.323
0.000
CHCl3
C=N
zxr447-137B in CDCl3
4.18
6.12
2.16
δ (ppm)
H2O
TMS
3.00
18.01

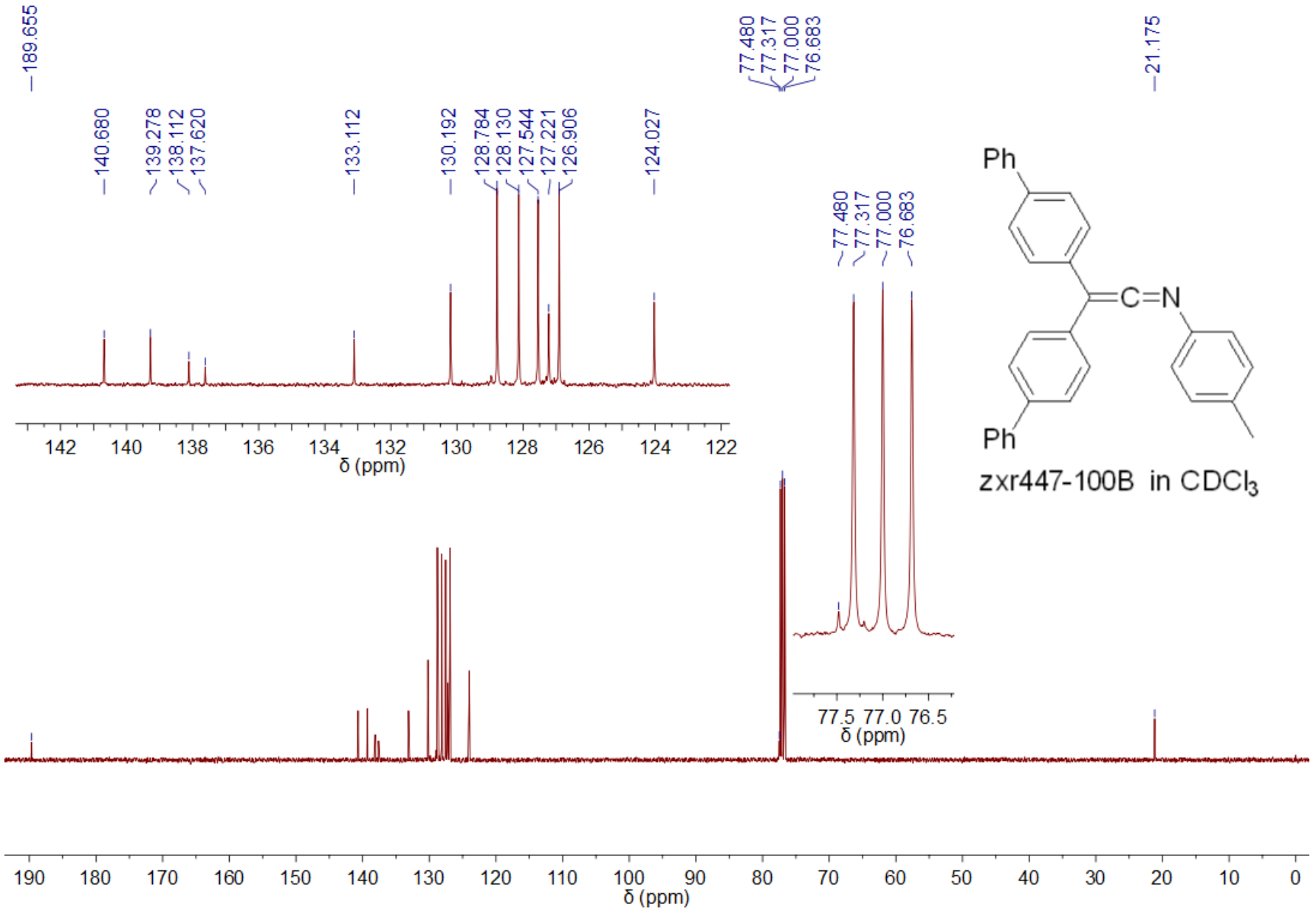
2l - ^{1}H NMR


191.412
149.313
138.216
137.710
131.062
130.045
127.447
125.689
123.859
77.552
77.318
77.000
76.683
34.493
31.337
21.134
C=N
CDCl3
zxr447-137B in CDCl3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

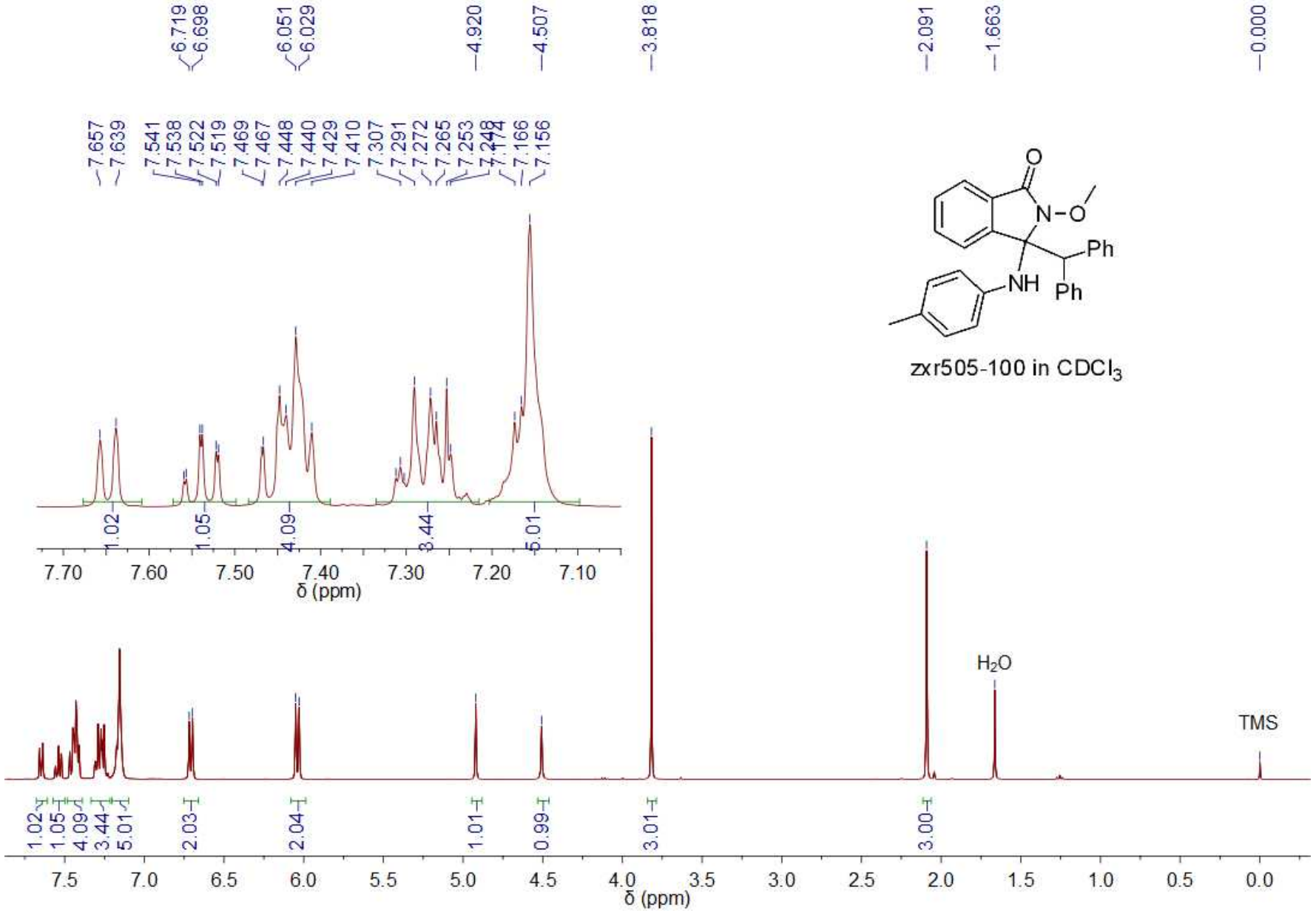
2l - ^{13}C NMR


7.622
7.618
7.611
7.605
7.600
7.597
7.594
7.590
7.474
7.469
7.464
7.459
7.453
7.448
7.441
7.437
7.430
7.425
7.421
7.361
7.357
7.354
7.344
7.339
7.334
7.323
7.319
7.242
7.218
7.198
2.376
0.000
Ph
C=N
Ph
zxr447-100B in CDCl3
CHCl3
8.12
8.37
4.16
2.08
7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15
δ (ppm)
H2O
TMS
8.12
8.37
4.16
2.08
3.00
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 0.0
δ (ppm)

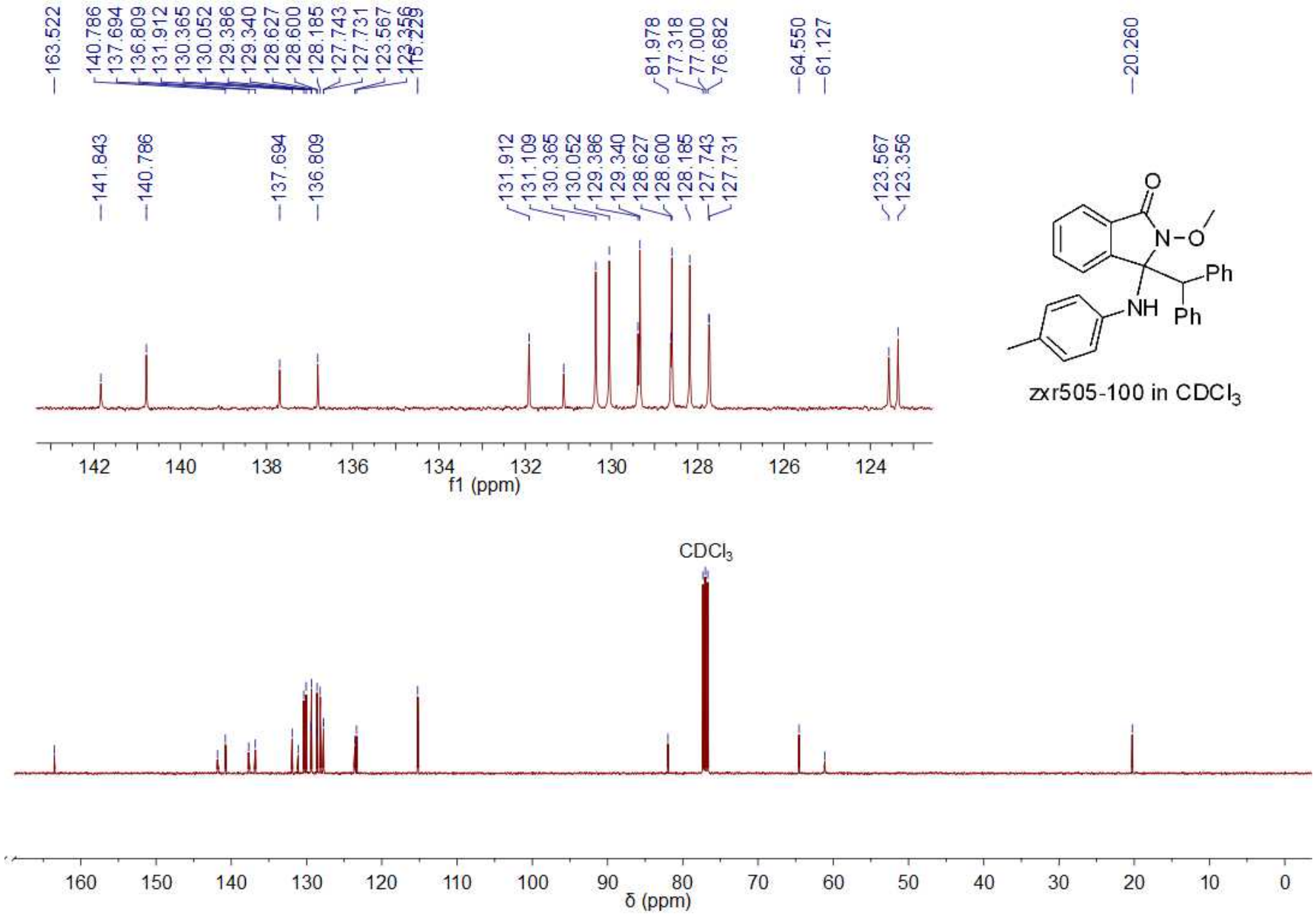
2n - ^{1}H NMR


189.655
140.680
139.278
138.112
137.620
133.112
130.192
128.784
128.130
127.544
127.221
126.906
124.027
77.480
77.317
77.000
76.683
21.175
δ (ppm)
Ph
C=N
Ph
zxr447-100B in CDCl3

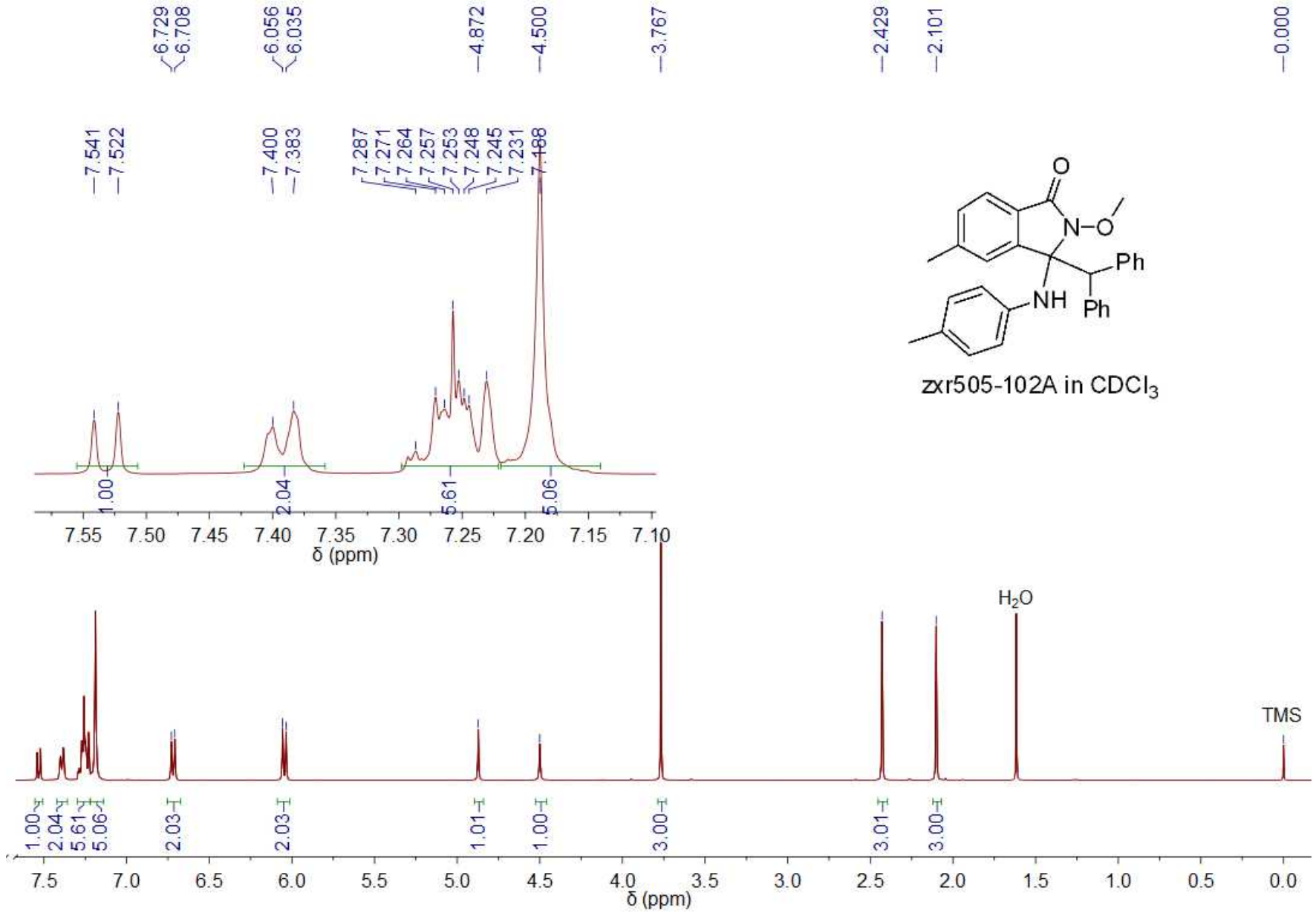
2n - ^{13}C NMR


6.719
6.698
6.051
6.029
4.920
4.507
3.818
2.091
1.663
0.000
7.657
7.639
7.541
7.538
7.522
7.519
7.469
7.467
7.448
7.440
7.429
7.410
7.307
7.291
7.272
7.265
7.253
7.248
7.174
7.166
7.156
O
N–O
Ph
NH
Ph
zxr505-100 in CDCl3
1.02
1.05
4.09
3.44
5.01
7.70
7.60
7.50
7.40
7.30
7.20
7.10
δ (ppm)
H2O
TMS
1.02
1.05
4.09
3.44
5.01
2.03
2.04
1.01
0.99
3.01
3.00
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
0.0
δ (ppm)

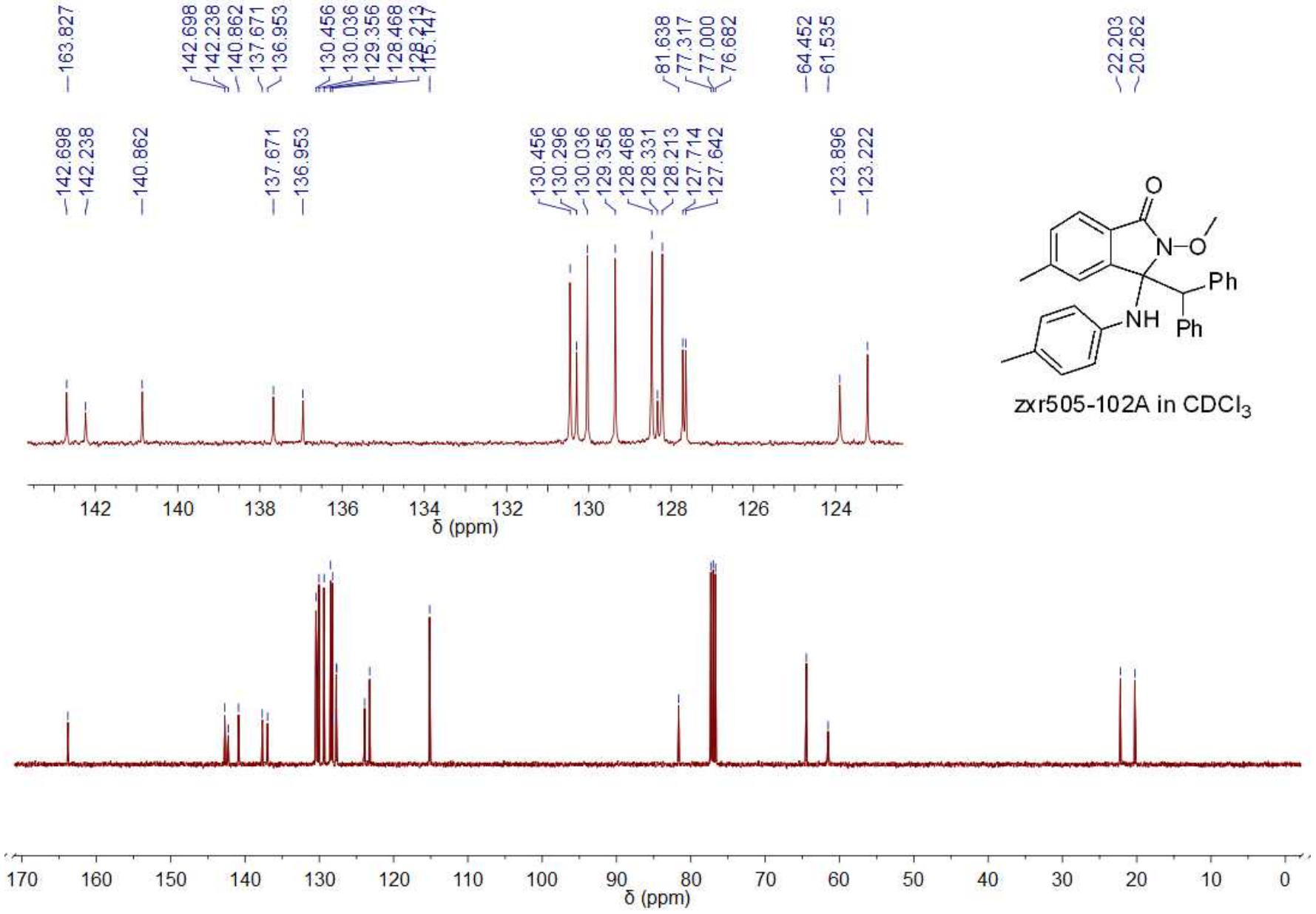
3a - ^{1}H NMR


163.522
140.786
137.694
136.809
131.912
130.365
130.052
129.386
129.340
128.627
128.600
128.185
127.743
127.731
123.567
123.356
81.978
77.318
77.000
76.682
64.550
61.127
20.260
141.843
140.786
137.694
136.809
131.912
131.109
130.365
130.052
129.386
129.340
128.627
128.600
128.185
127.743
127.731
123.567
123.356
O
N–O
Ph
NH
Ph
zxr505-100 in CDCl3
142
140
138
136
134
132
130
128
126
124
f1 (ppm)
CDCl3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

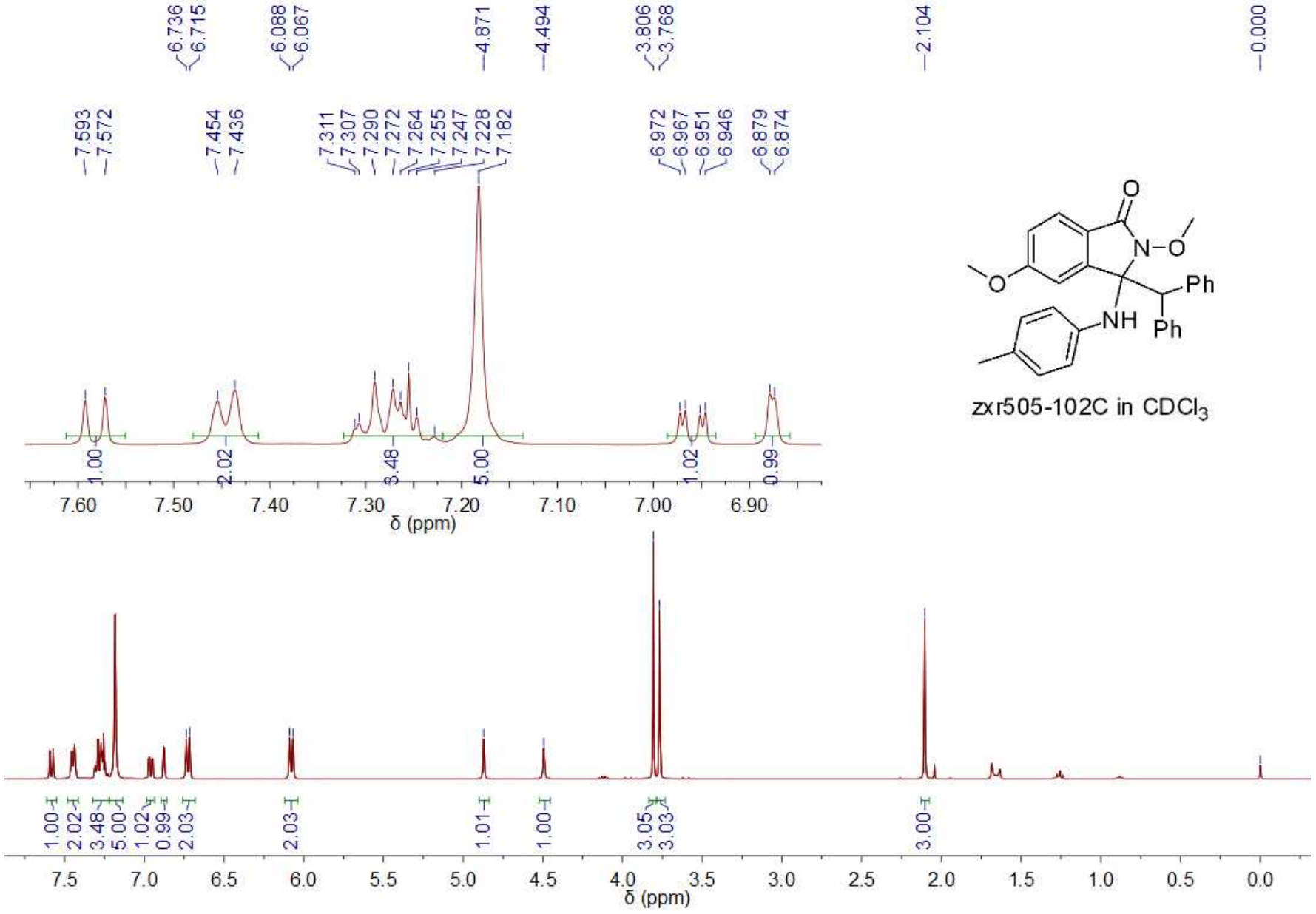
3a – ^{13}C NMR


6.729
6.708
6.056
6.035
4.872
4.500
3.767
2.429
2.101
0.000
7.541
7.522
7.400
7.383
7.287
7.271
7.264
7.257
7.253
7.248
7.245
7.231
7.188
O
N–O
Ph
NH
Ph
zxr505-102A in CDCl3
1.00
2.04
5.61
5.06
δ (ppm)
H2O
TMS
2.03
1.01
3.00
3.01

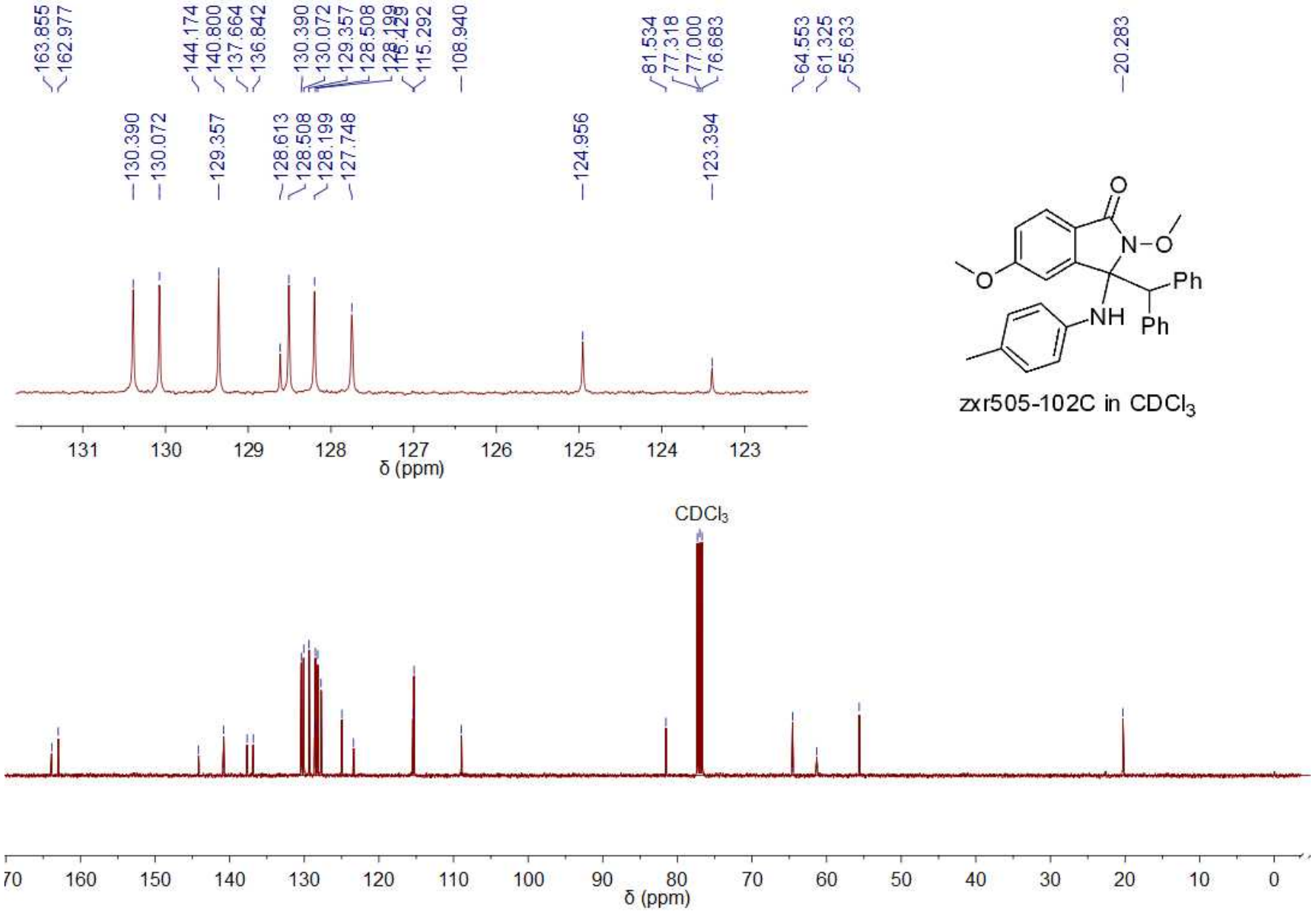
3b - ^{1}H NMR


163.827
142.698
142.238
140.862
137.671
136.953
130.456
130.036
129.356
128.468
128.213
115.147
81.638
77.317
77.000
76.682
64.452
61.535
22.203
20.262
142.698
142.238
140.862
137.671
136.953
130.456
130.296
130.036
129.356
128.468
128.331
128.213
127.714
127.642
123.896
123.222
O
N–O
Ph
NH
Ph
zxr505-102A in CDCl3
δ (ppm)
δ (ppm)

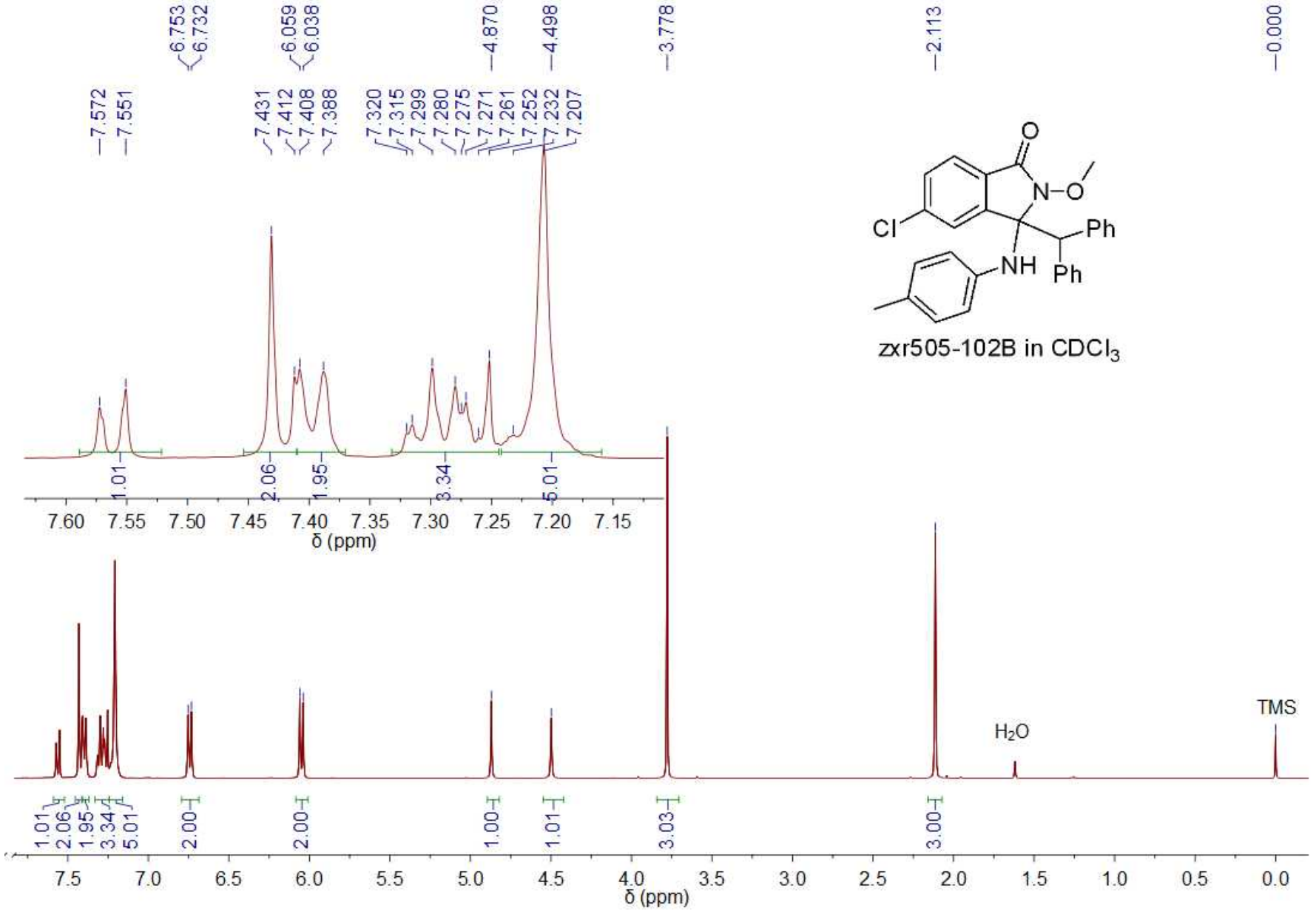
3b – ^{13}C NMR


6.736
6.715
6.088
6.067
4.871
4.494
3.806
3.768
2.104
0.000
7.593
7.572
7.454
7.436
7.311
7.307
7.290
7.272
7.264
7.255
7.247
7.228
7.182
6.972
6.967
6.951
6.946
6.879
6.874
1.00
2.02
3.48
5.00
1.02
0.99
7.60
7.50
7.40
7.30
7.20
7.10
7.00
6.90
δ (ppm)
1.00
2.02
3.48
5.00
1.02
0.99
2.03
2.03
1.01
1.00
3.05
3.03
3.00
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
0.0
δ (ppm)
O
N–O
O
Ph
NH
Ph
zxr505-102C in CDCl3

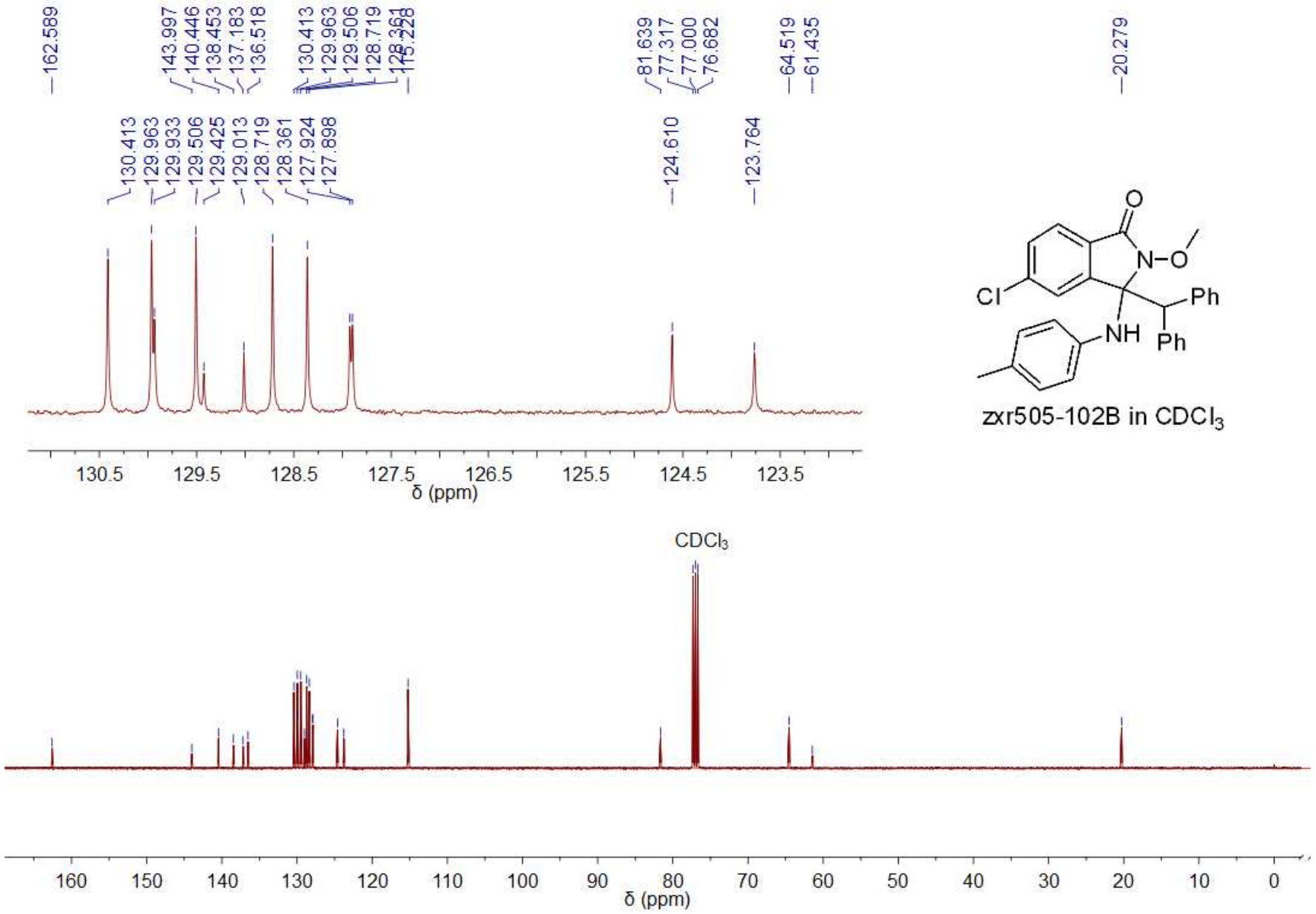
3c - ^{1}H NMR


163.855
162.977
144.174
140.800
137.664
136.842
130.390
130.072
129.357
128.508
115.292
108.940
81.534
77.318
77.000
76.683
64.553
61.325
55.633
20.283
130.390
130.072
129.357
128.613
128.508
128.199
127.748
124.956
123.394
O
N–O
O
Ph
NH
Ph
zxr505-102C in CDCl3
131
130
129
128
127
126
125
124
123
δ (ppm)
CDCl3
70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

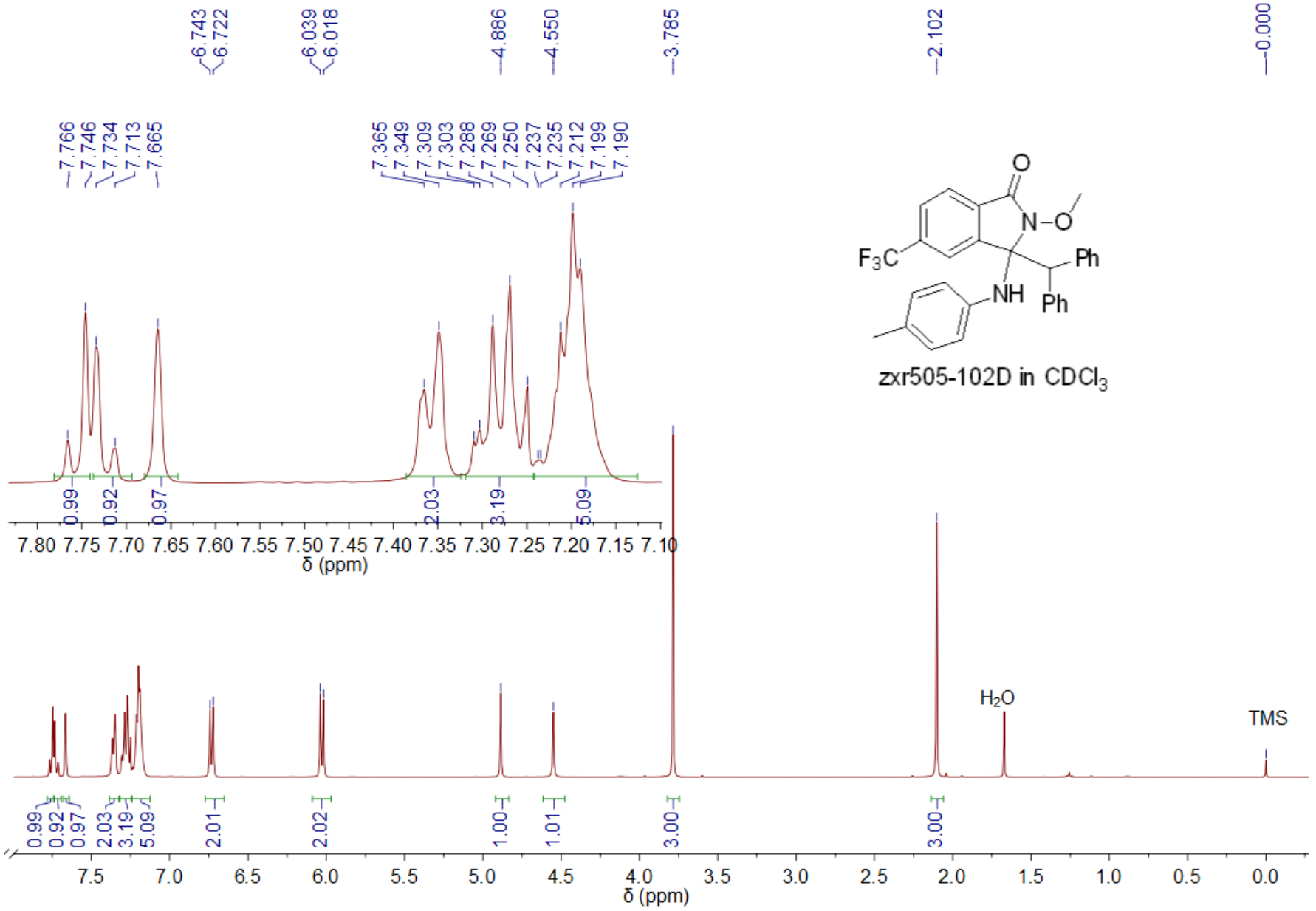
3c – ^{13}C NMR


6.753
6.732
6.059
6.038
4.870
4.498
3.778
2.113
0.000
7.572
7.551
7.431
7.412
7.408
7.388
7.320
7.315
7.299
7.280
7.275
7.271
7.261
7.252
7.232
7.207
O
N–O
Cl
Ph
NH
Ph
zxr505-102B in CDCl3
1.01
2.06
1.95
3.34
5.01
δ (ppm)
TMS
H2O
2.00
1.00
3.03
3.00

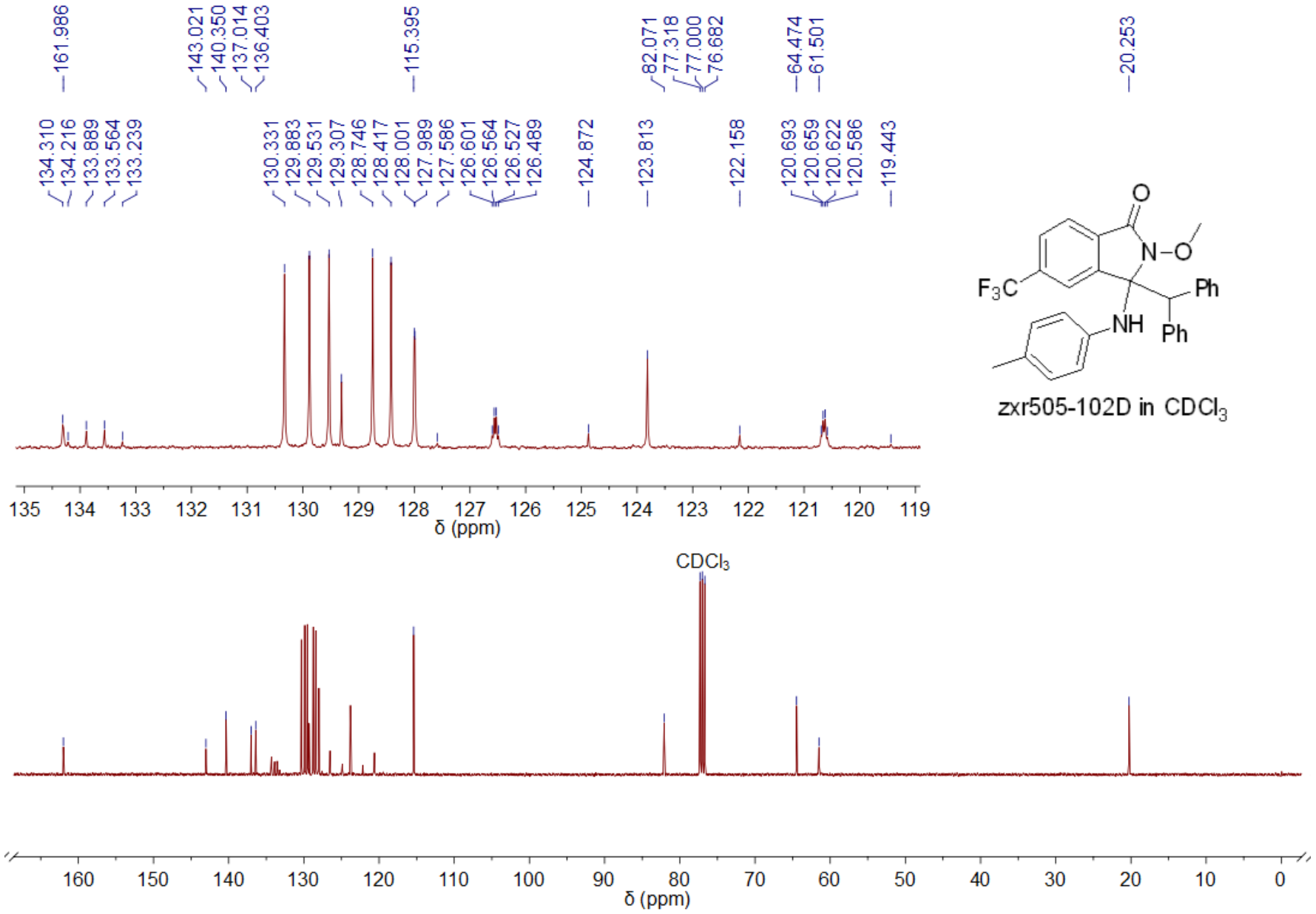
3d - ^{1}H NMR


162.589
143.997
140.446
138.453
137.183
136.518
130.413
129.963
129.506
128.719
81.639
77.317
77.000
76.682
64.519
61.435
20.279
130.413
129.963
129.933
129.506
129.425
129.013
128.719
128.361
127.924
127.898
124.610
123.764
O
N–O
Cl
Ph
NH
Ph
zxr505-102B in CDCl3
130.5
129.5
128.5
127.5
126.5
125.5
124.5
123.5
δ (ppm)
CDCl3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

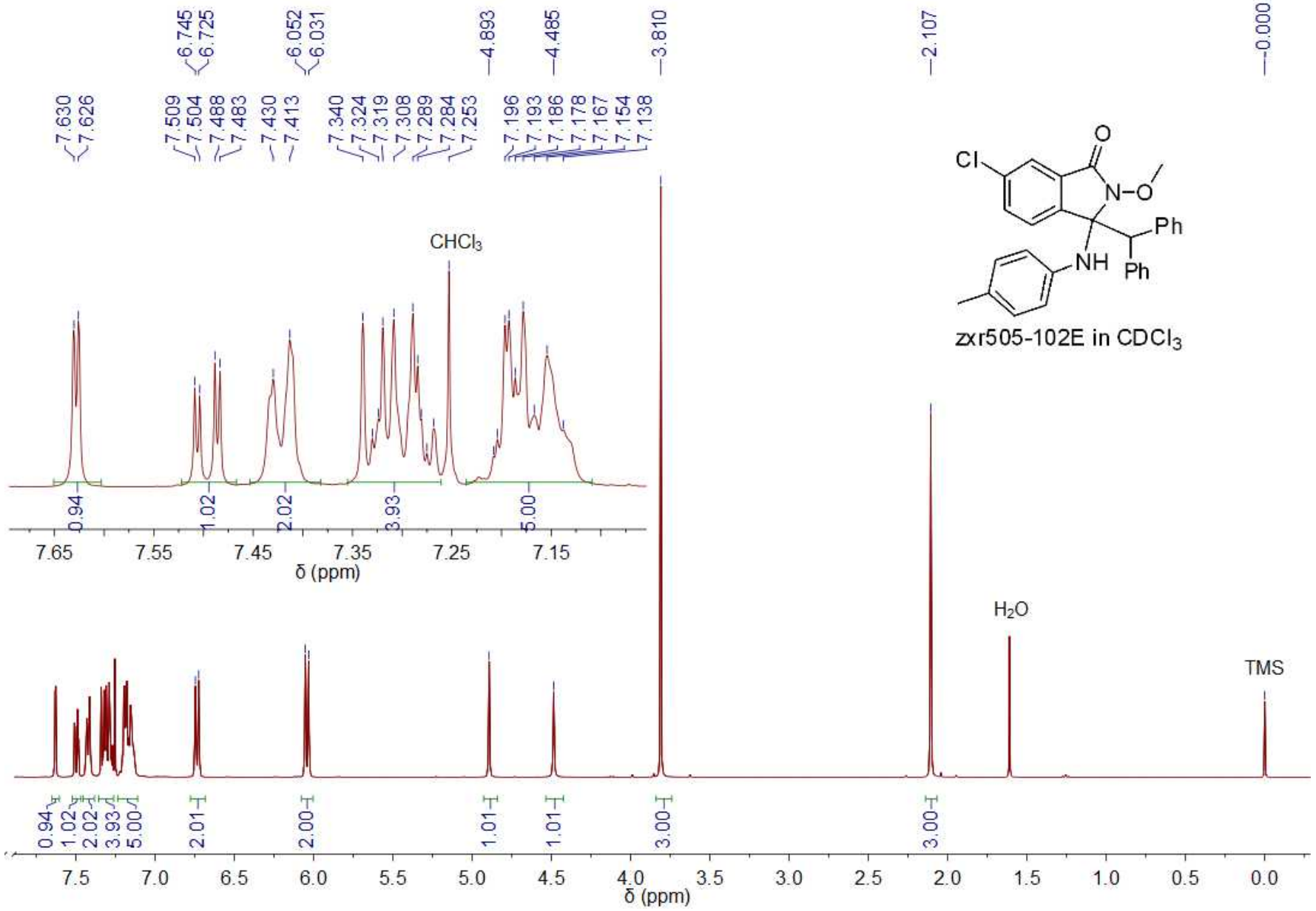
3d – ^{13}C NMR


6.743
6.722
6.039
6.018
4.886
4.550
3.785
2.102
0.000
7.766
7.746
7.734
7.713
7.665
7.365
7.349
7.309
7.303
7.288
7.269
7.250
7.237
7.235
7.212
7.199
7.190
O
N–O
F3C
Ph
NH
Ph
zxr505-102D in CDCl3
0.99
0.92
0.97
2.03
3.19
5.09
2.01
2.02
1.00
1.01
3.00
3.00
H2O
TMS
7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10
δ (ppm)
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 0.0
δ (ppm)

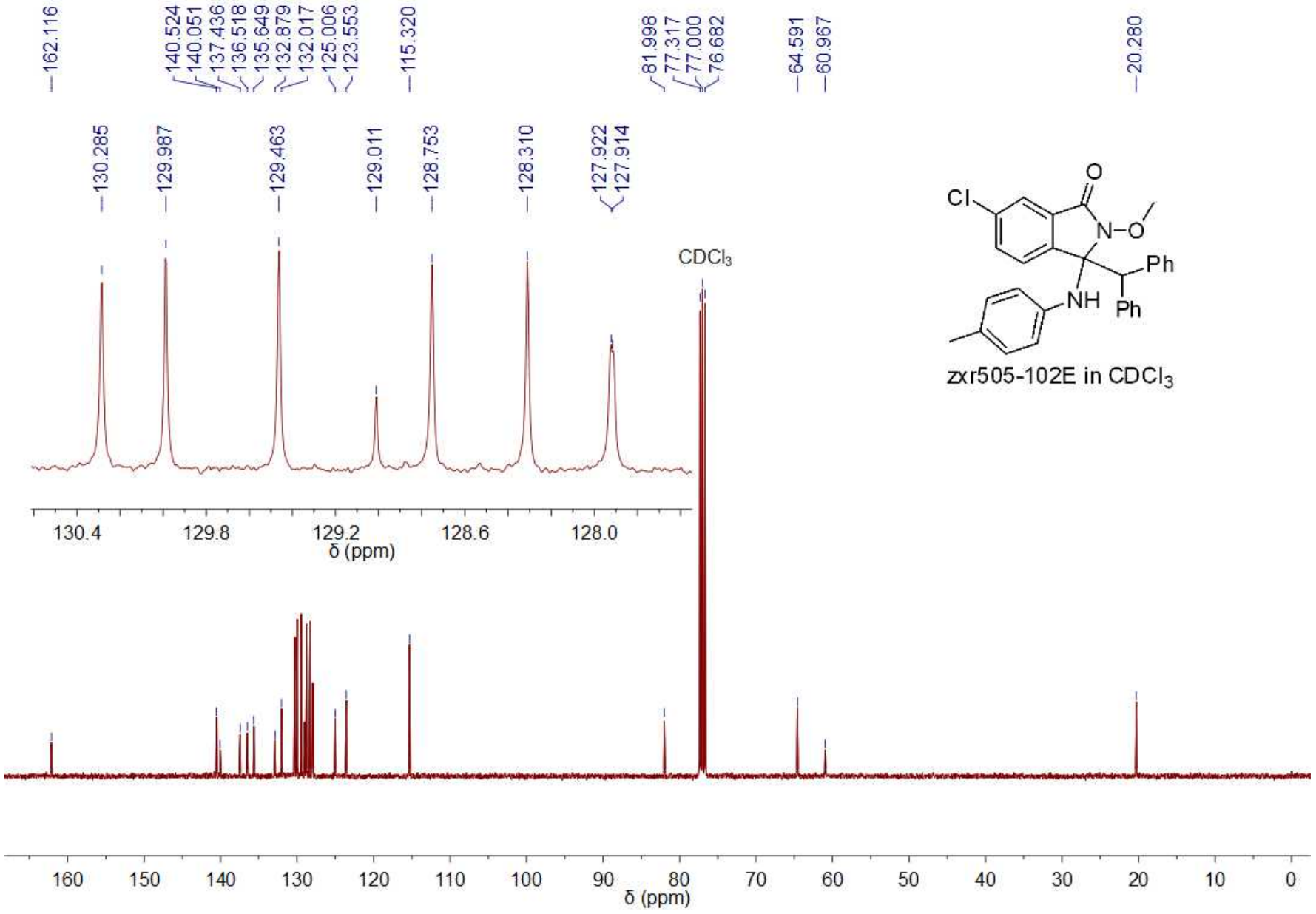
3e - ^{1}H NMR


161.986
143.021
140.350
137.014
136.403
115.395
82.071
77.318
77.000
76.682
64.474
61.501
20.253
134.310
134.216
133.889
133.564
133.239
130.331
129.883
129.531
129.307
128.746
128.417
128.001
127.989
127.586
126.601
126.564
126.527
126.489
124.872
123.813
122.158
120.693
120.659
120.622
120.586
119.443
O
N–O
F3C
Ph
NH
Ph
zxr505-102D in CDCl3
135 134 133 132 131 130 129 128 127 126 125 124 123 122 121 120 119
δ (ppm)
CDCl3
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

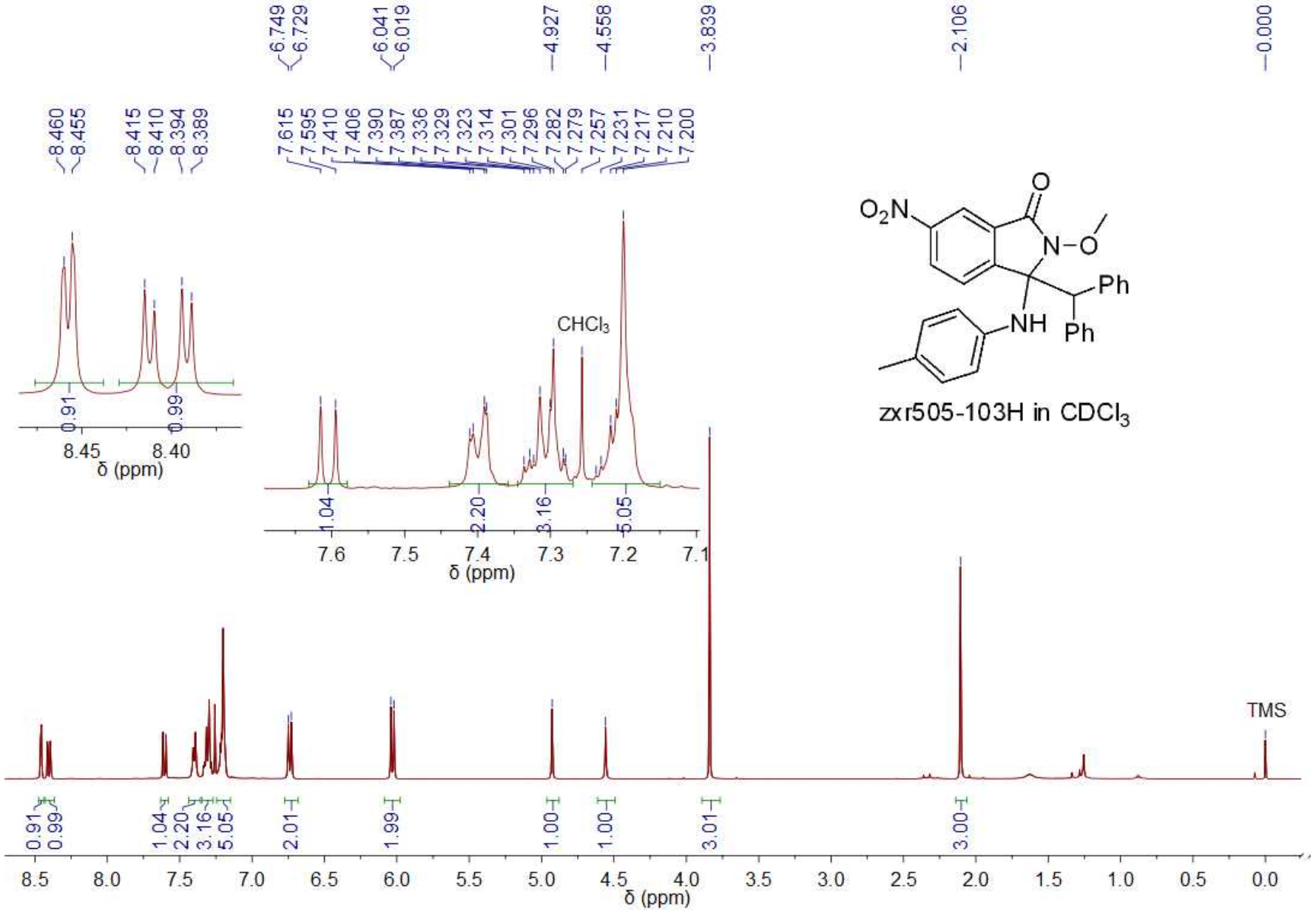
3e – ^{13}C NMR


7.630
7.626
7.509
7.504
7.488
7.483
7.430
7.413
7.340
7.324
7.319
7.308
7.289
7.284
7.253
7.196
7.193
7.186
7.178
7.167
7.154
7.138
6.745
6.725
6.052
6.031
4.893
4.485
3.810
2.107
0.000
CHCl3
0.94
1.02
2.02
3.93
5.00
δ (ppm)
H2O
TMS
2.01
2.00
1.01
1.01
3.00
3.00
zxr505-102E in CDCl3

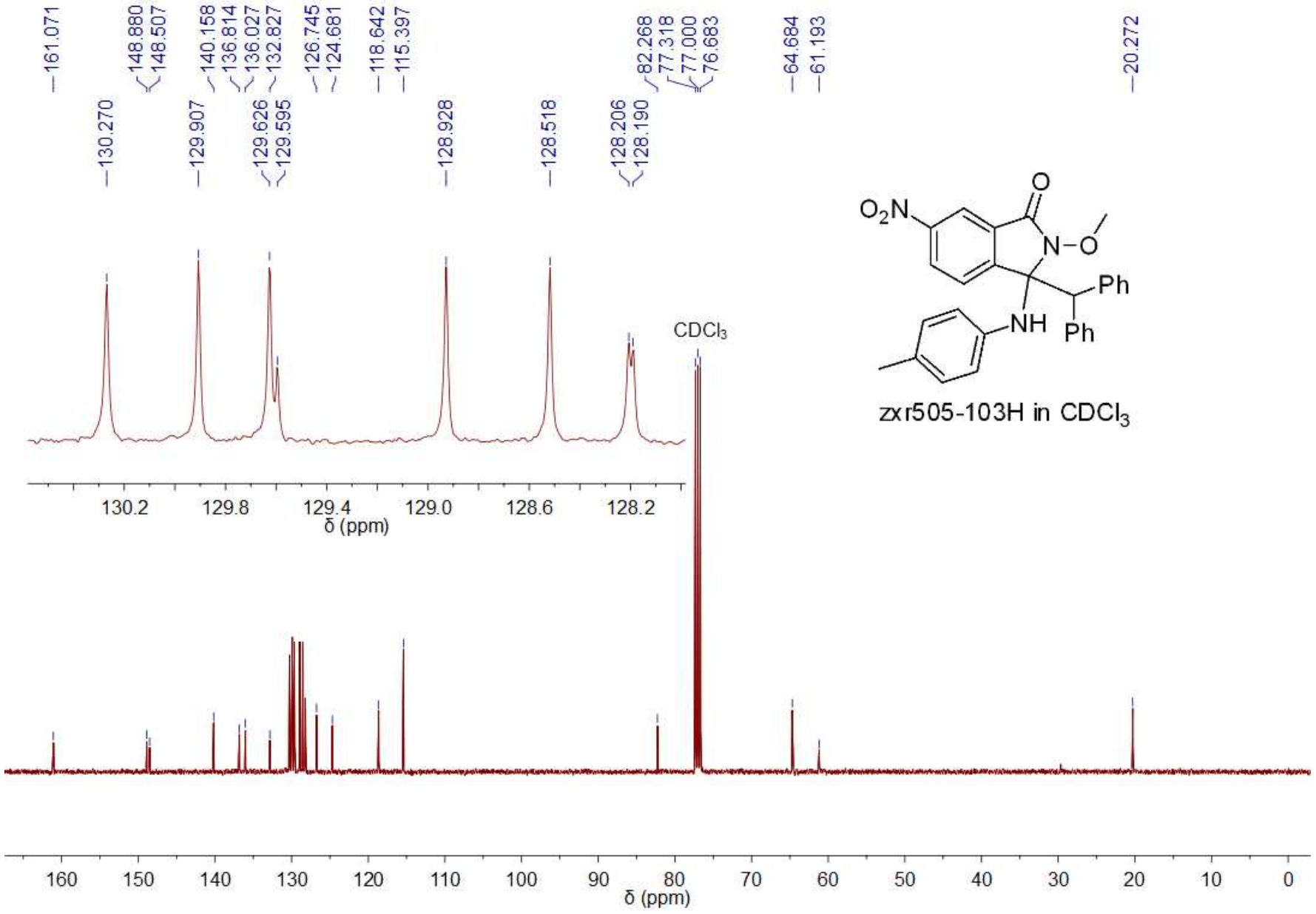
3f - ^{1}H NMR


162.116
140.524
140.051
137.436
136.518
135.649
132.879
132.017
125.006
123.553
115.320
81.998
77.317
77.000
76.682
64.591
60.967
20.280
130.285
129.987
129.463
129.011
128.753
128.310
127.922
127.914
CDCl3
130.4
129.8
129.2
128.6
128.0
δ (ppm)
Cl
O
N–O
Ph
NH
Ph
zxr505-102E in CDCl3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

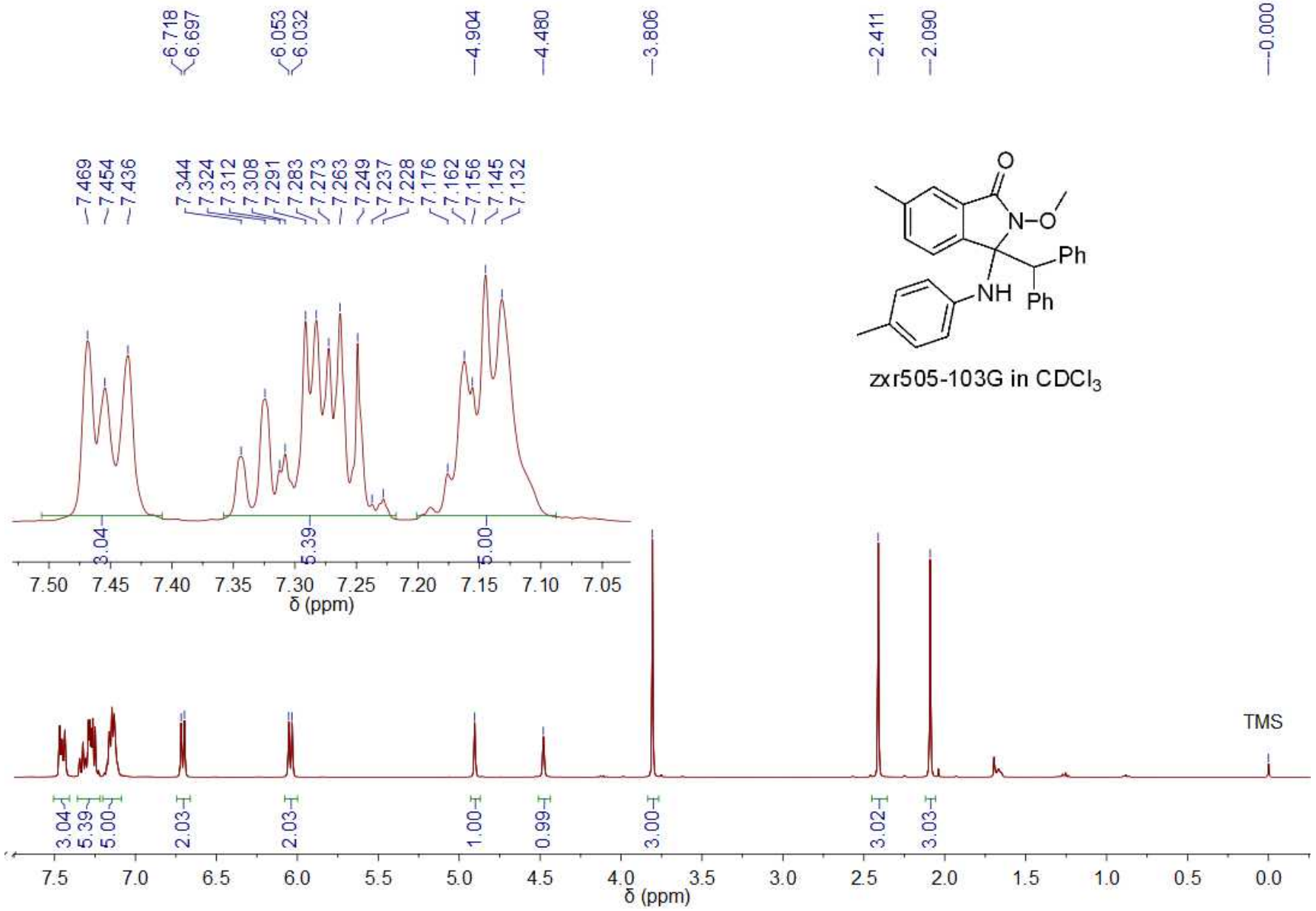
3f – ^{13}C NMR


6.749
6.729
6.041
6.019
4.927
4.558
3.839
2.106
0.000
8.460
8.455
8.415
8.410
8.394
8.389
7.615
7.595
7.410
7.406
7.390
7.387
7.336
7.329
7.323
7.314
7.301
7.296
7.282
7.279
7.257
7.231
7.217
7.210
7.200
CHCl3
O2N
O
N–O
Ph
NH
Ph
zxr505-103H in CDCl3
0.91
0.99
8.45
8.40
δ (ppm)
1.04
2.20
3.16
5.05
7.6
7.5
7.4
7.3
7.2
7.1
δ (ppm)
TMS
0.91
0.99
1.04
2.20
3.16
5.05
2.01
1.99
1.00
1.00
3.01
3.00
8.5
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
0.0
δ (ppm)

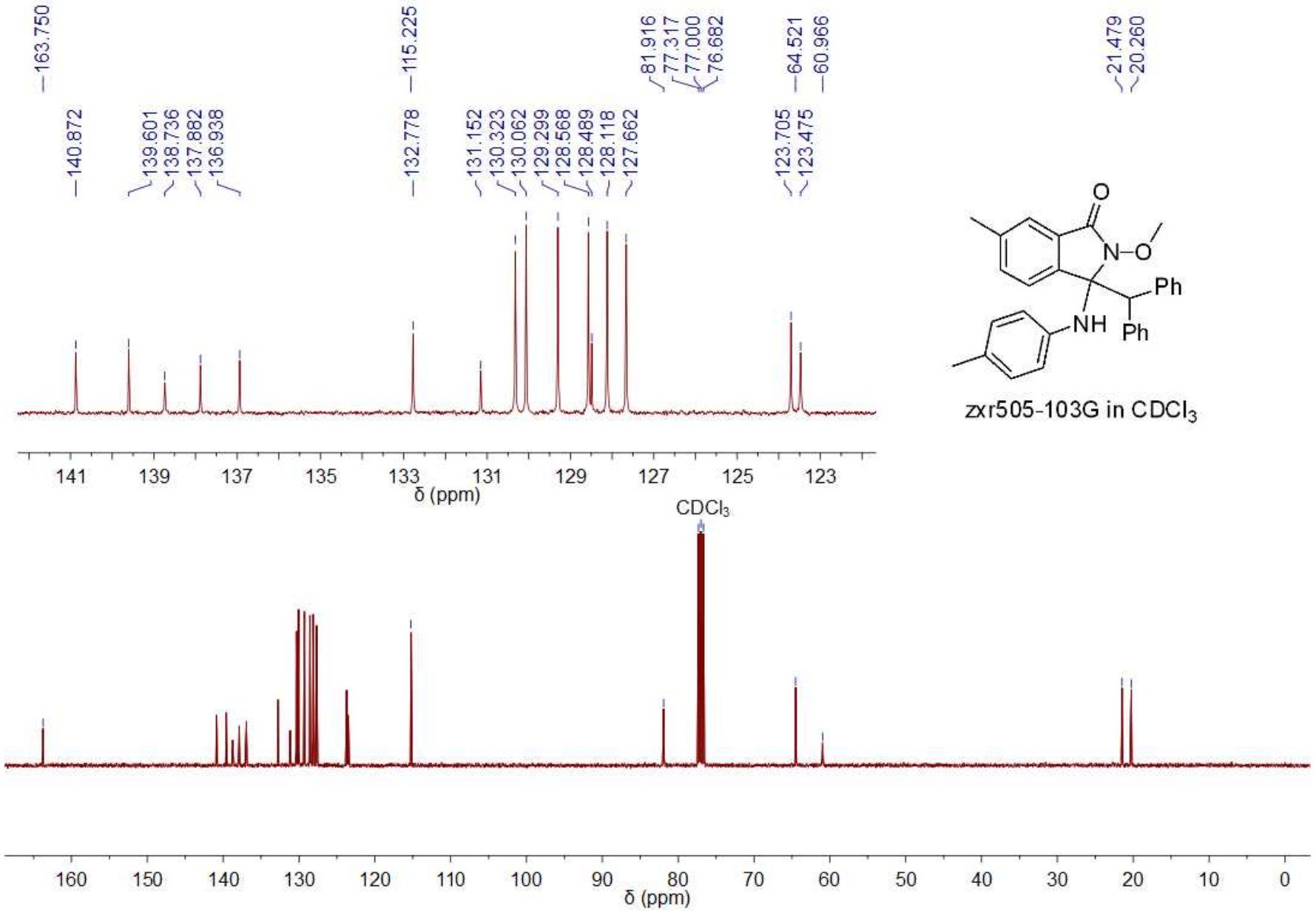
3g - ^{1}H NMR


161.071
148.880
148.507
140.158
136.814
136.027
132.827
126.745
124.681
118.642
115.397
130.270
129.907
129.626
129.595
128.928
128.518
128.206
128.190
82.268
77.318
77.000
76.683
64.684
61.193
20.272
CDCl3
O2N
O
N–O
Ph
NH
Ph
zxr505-103H in CDCl3
130.2
129.8
129.4
129.0
128.6
128.2
δ (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

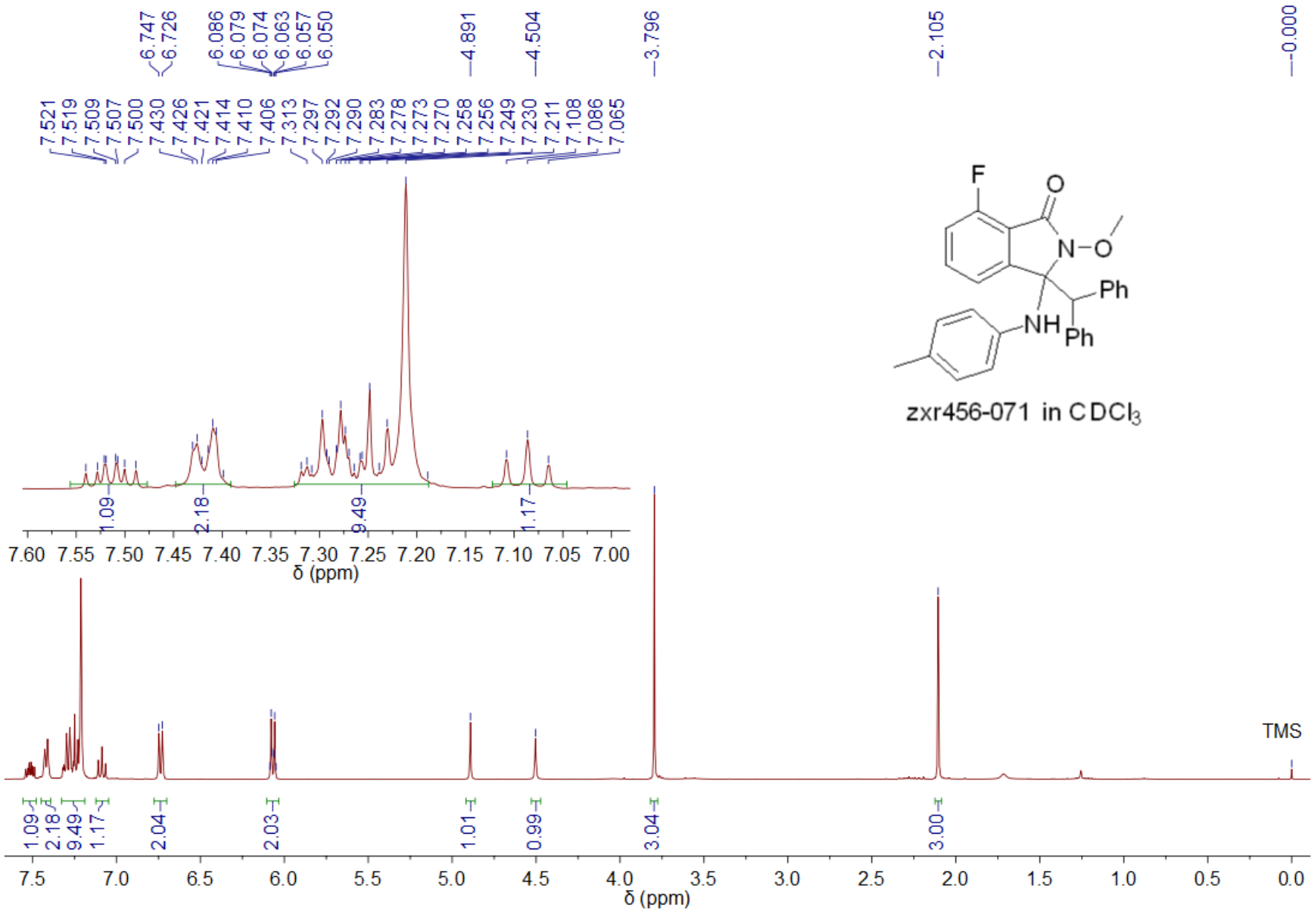
3g – ^{13}C NMR


6.718
6.697
6.053
6.032
4.904
4.480
3.806
2.411
2.090
0.000
7.469
7.454
7.436
7.344
7.324
7.312
7.308
7.291
7.283
7.273
7.263
7.249
7.237
7.228
7.176
7.162
7.156
7.145
7.132
O
N–O
Ph
NH
Ph
zxr505-103G in CDCl3
3.04
5.39
5.00
7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05
δ (ppm)
TMS
3.04
5.39
5.00
2.03
2.03
1.00
0.99
3.00
3.02
3.03
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 0.0
δ (ppm)

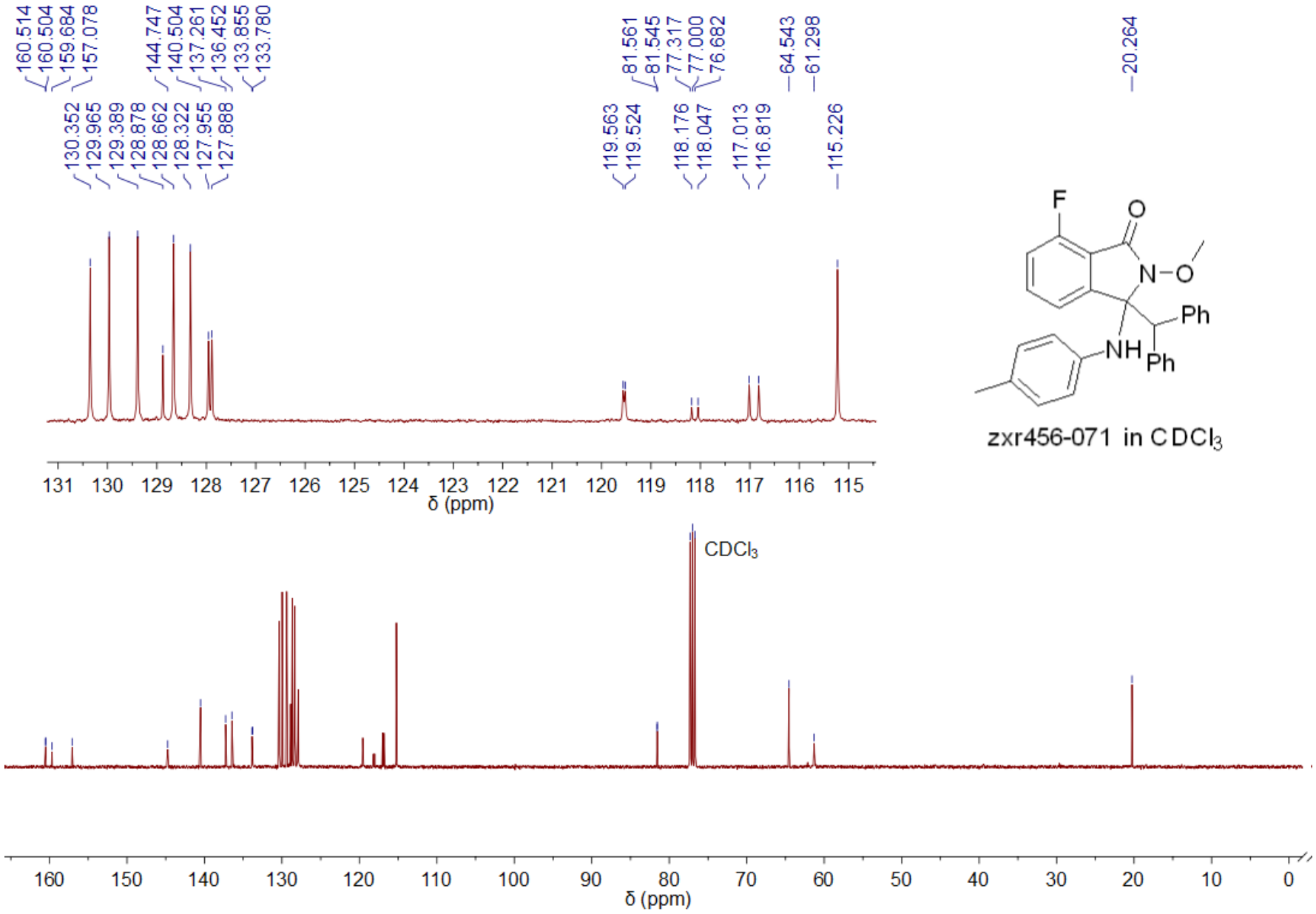
3h - ^{1}H NMR


163.750
140.872
139.601
138.736
137.882
136.938
115.225
132.778
131.152
130.323
130.062
129.299
128.568
128.489
128.118
127.662
81.916
77.317
77.000
76.682
64.521
123.705
123.475
60.966
21.479
20.260
O
N–O
Ph
NH
Ph
zxr505-103G in CDCl3
141
139
137
135
133
131
129
127
125
123
δ (ppm)
CDCl3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

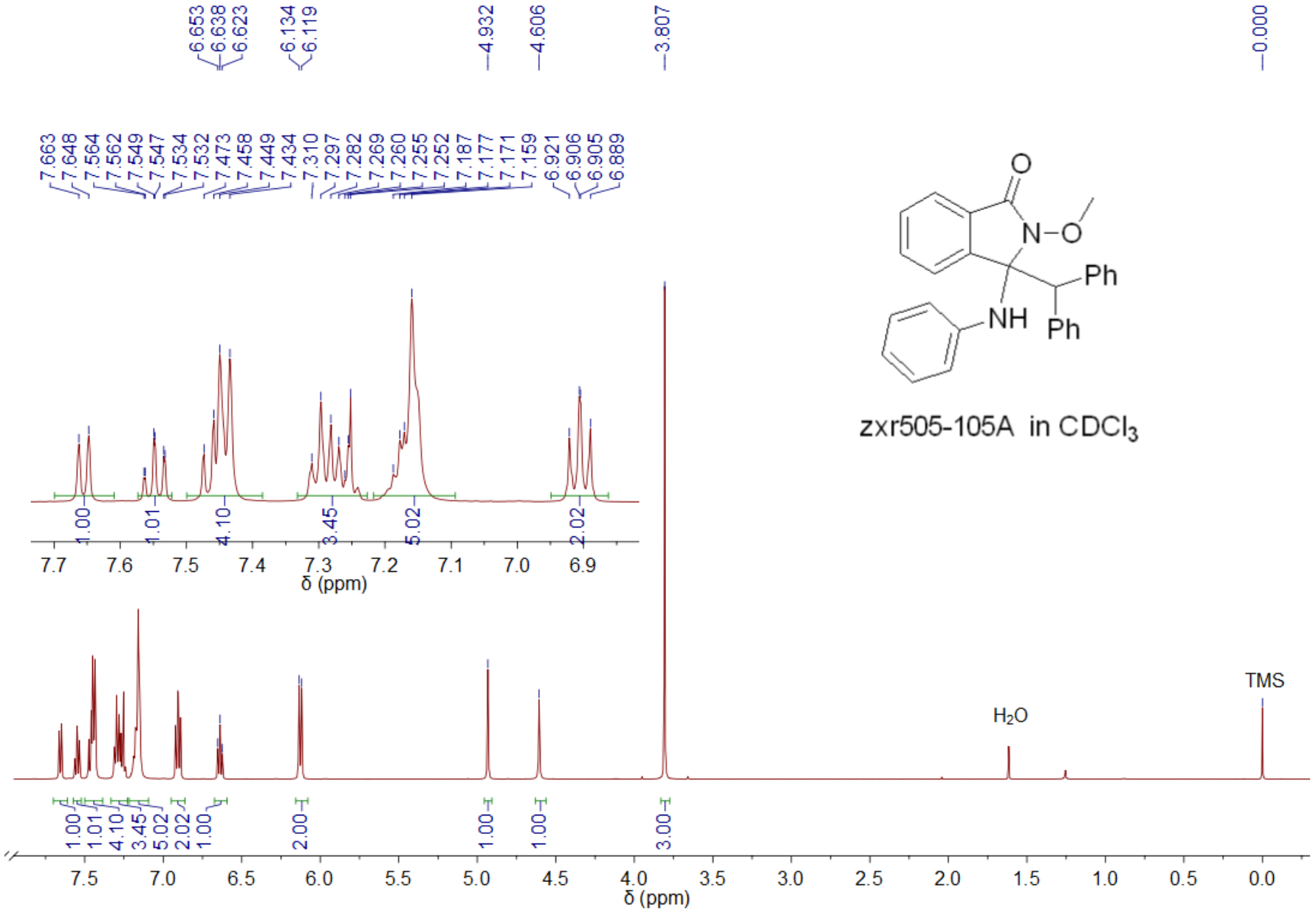
3h – ^{13}C NMR


6.747
6.726
6.086
6.079
6.074
6.063
6.057
6.050
4.891
4.504
3.796
2.105
0.000
7.521
7.519
7.509
7.507
7.500
7.430
7.426
7.421
7.414
7.410
7.406
7.313
7.297
7.292
7.290
7.283
7.278
7.273
7.270
7.258
7.256
7.249
7.230
7.211
7.108
7.086
7.065
F
O
N–O
Ph
NH
Ph
zxr456-071 in CDCl3
1.09
2.18
9.49
1.17
7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00
δ (ppm)
TMS
1.09
2.18
9.49
1.17
2.04
2.03
1.01
0.99
3.04
3.00
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 0.0
δ (ppm)

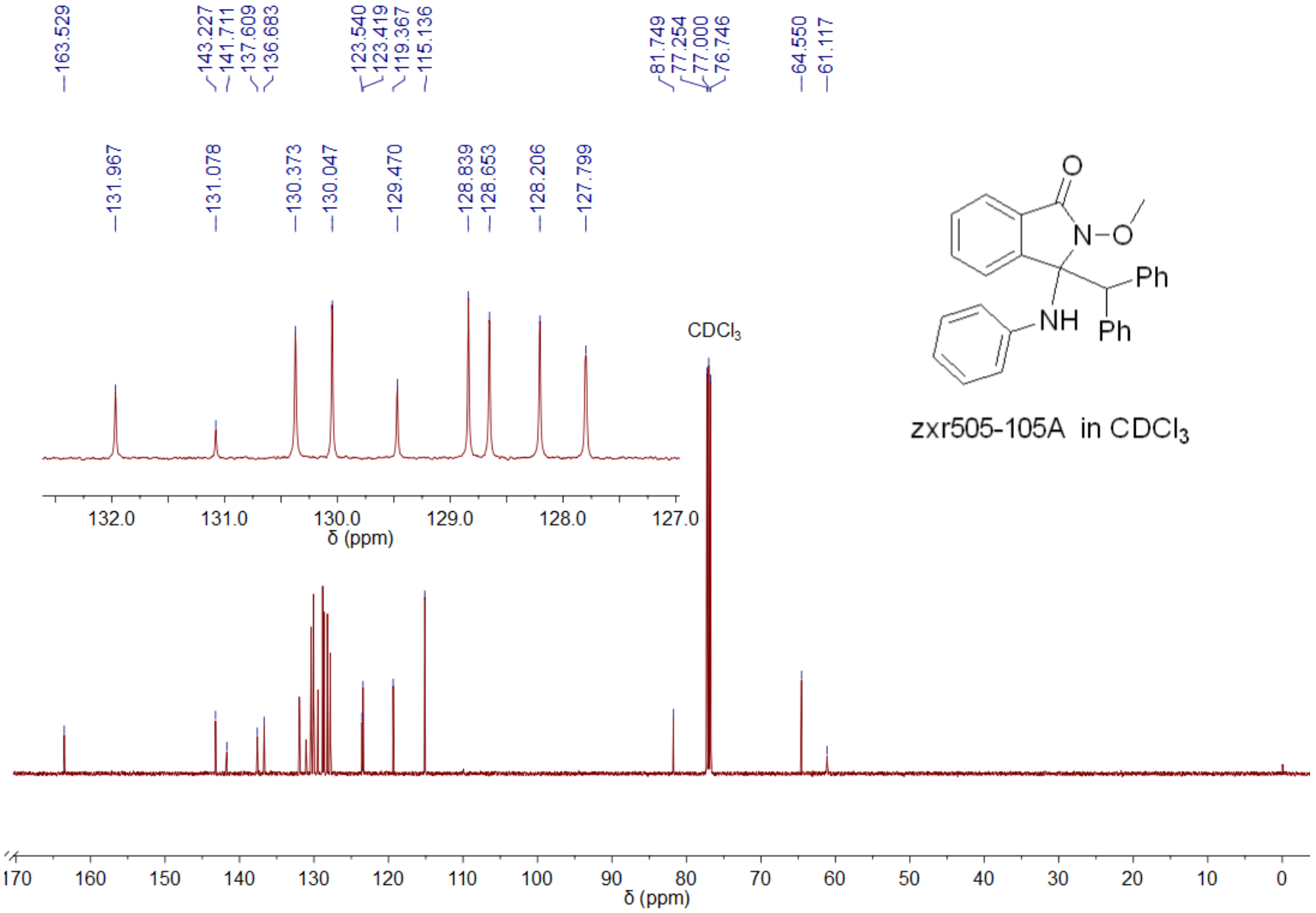
3i - ^{1}H NMR


160.514
160.504
159.684
157.078
144.747
140.504
137.261
136.452
133.855
133.780
130.352
129.965
129.389
128.878
128.662
128.322
127.955
127.888
119.563
119.524
118.176
118.047
117.013
116.819
81.561
81.545
77.317
77.000
76.682
64.543
61.298
115.226
20.264
F
O
N
O
Ph
NH
Ph
zxr456-071 in CDCl3
131 130 129 128 127 126 125 124 123 122 121 120 119 118 117 116 115
δ (ppm)
CDCl3
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

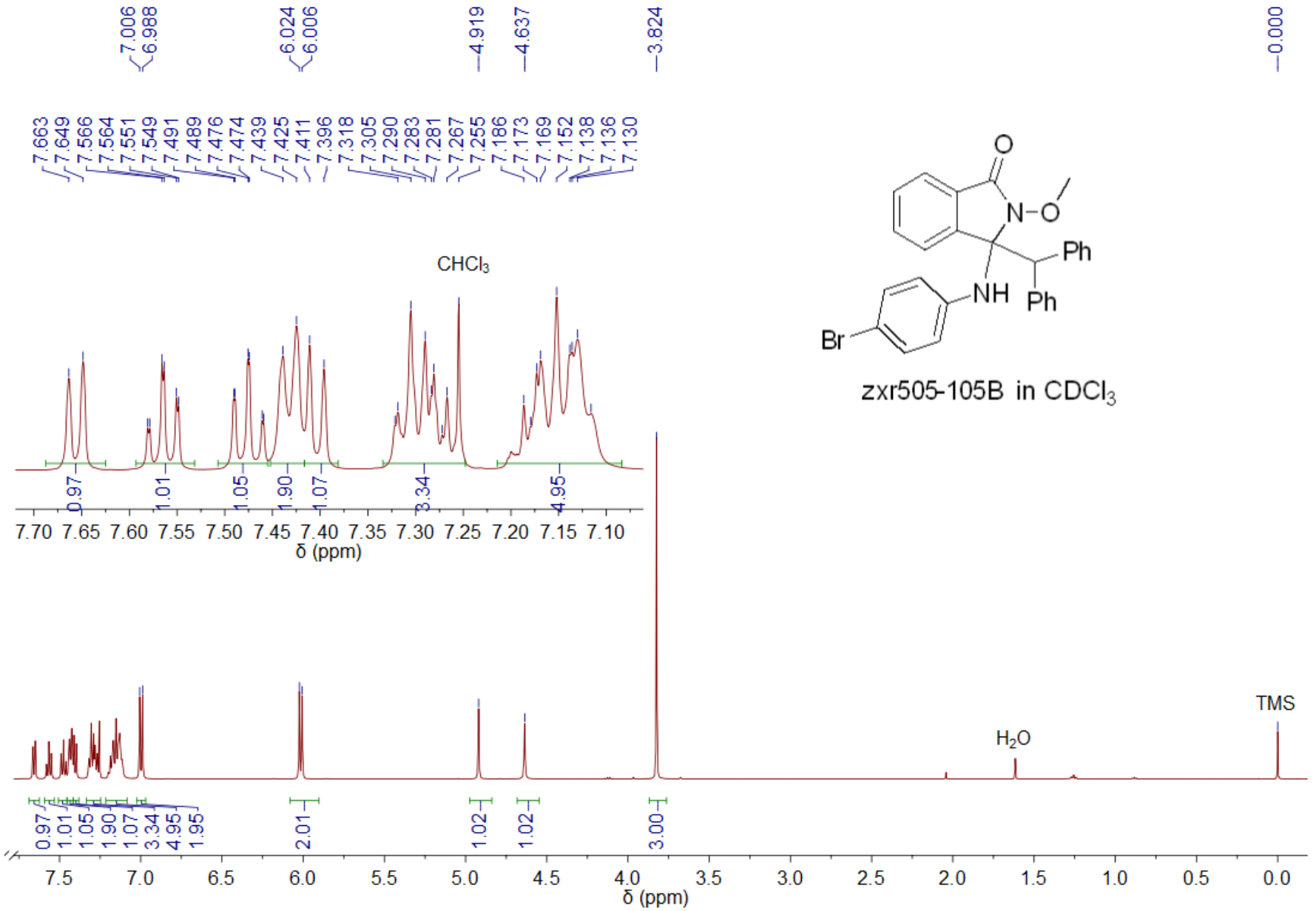
3i – ^{13}C NMR


6.653
6.638
6.623
6.134
6.119
4.932
4.606
3.807
0.000
7.663
7.648
7.564
7.562
7.549
7.547
7.534
7.532
7.473
7.458
7.449
7.434
7.310
7.297
7.282
7.269
7.260
7.255
7.252
7.187
7.177
7.171
7.159
6.921
6.906
6.905
6.889
O
N–O
Ph
NH
Ph
zxr505-105A in CDCl3
1.00
1.01
4.10
3.45
5.02
2.02
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
δ (ppm)
TMS
H2O
1.00
1.01
4.10
3.45
5.02
2.02
1.00
2.00
1.00
1.00
3.00
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
0.0
δ (ppm)

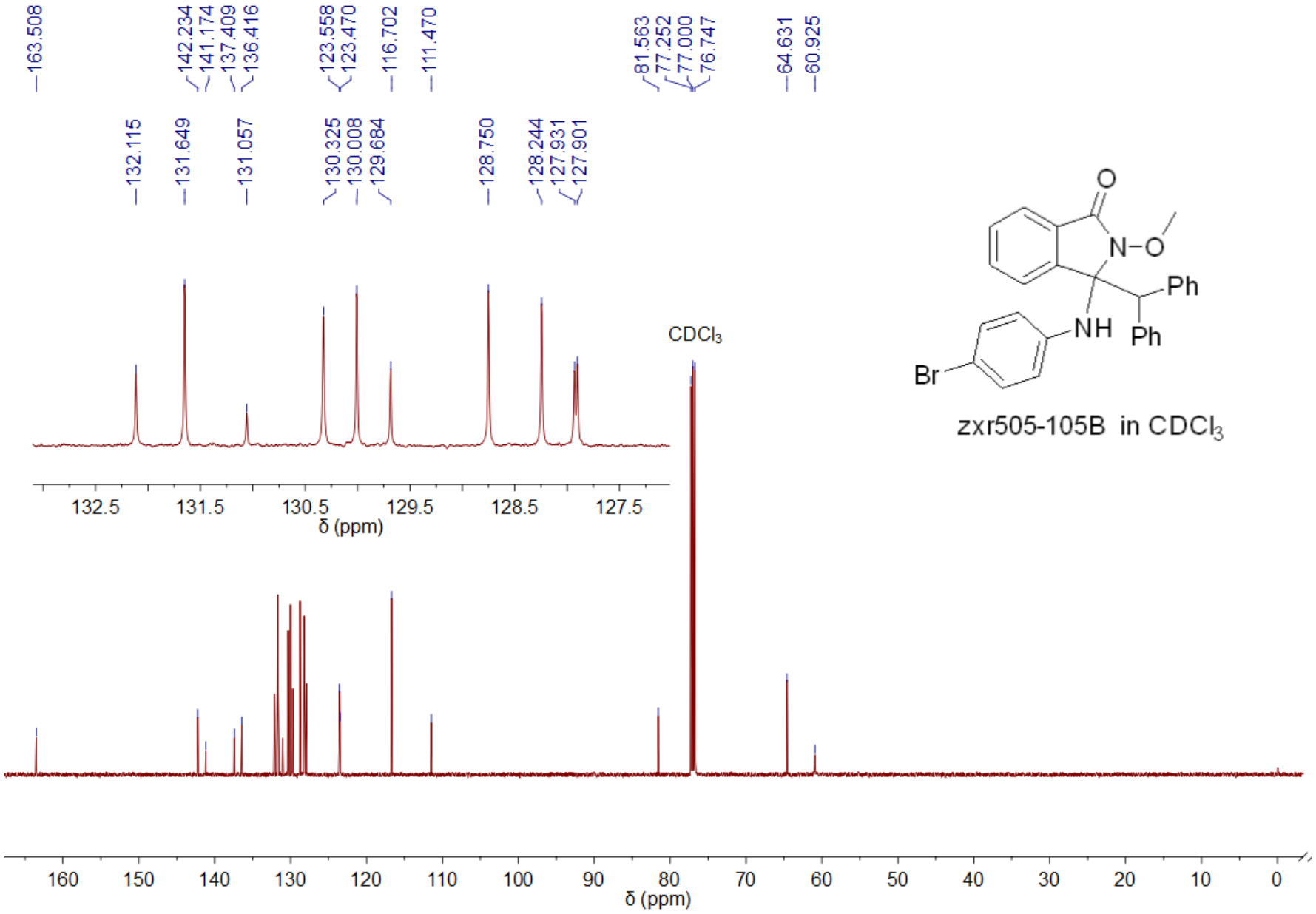
3j - ^{1}H NMR


163.529
143.227
141.711
137.609
136.683
123.540
123.419
119.367
115.136
81.749
77.254
77.000
76.746
64.550
61.117
131.967
131.078
130.373
130.047
129.470
128.839
128.653
128.206
127.799
CDCl3
O
N–O
Ph
NH
Ph
zxr505-105A in CDCl3
132.0
131.0
130.0
129.0
128.0
127.0
δ (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

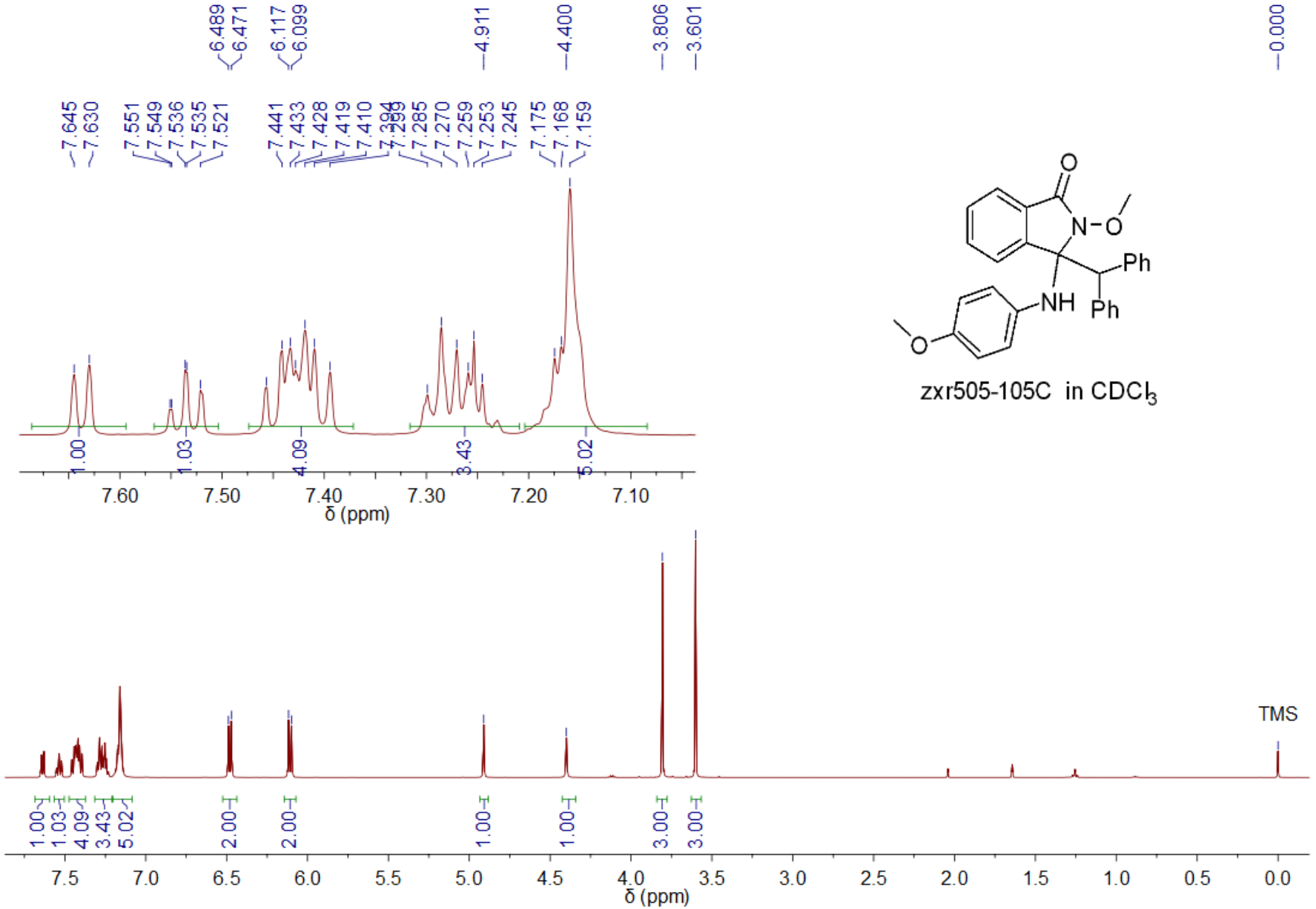
3j – ^{13}C NMR


7.006
6.988
6.024
6.006
4.919
4.637
3.824
0.000
7.663
7.649
7.566
7.564
7.551
7.549
7.491
7.489
7.476
7.474
7.439
7.425
7.411
7.396
7.318
7.305
7.290
7.283
7.281
7.267
7.255
7.186
7.173
7.169
7.152
7.138
7.136
7.130
CHCl3
O
N–O
Ph
NH
Ph
Br
zxr505-105B in CDCl3
0.97
1.01
1.05
1.90
1.07
3.34
4.95
7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10
δ (ppm)
TMS
H2O
0.97
1.01
1.05
1.90
1.07
3.34
4.95
1.95
2.01
1.02
1.02
3.00
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 0.0
δ (ppm)

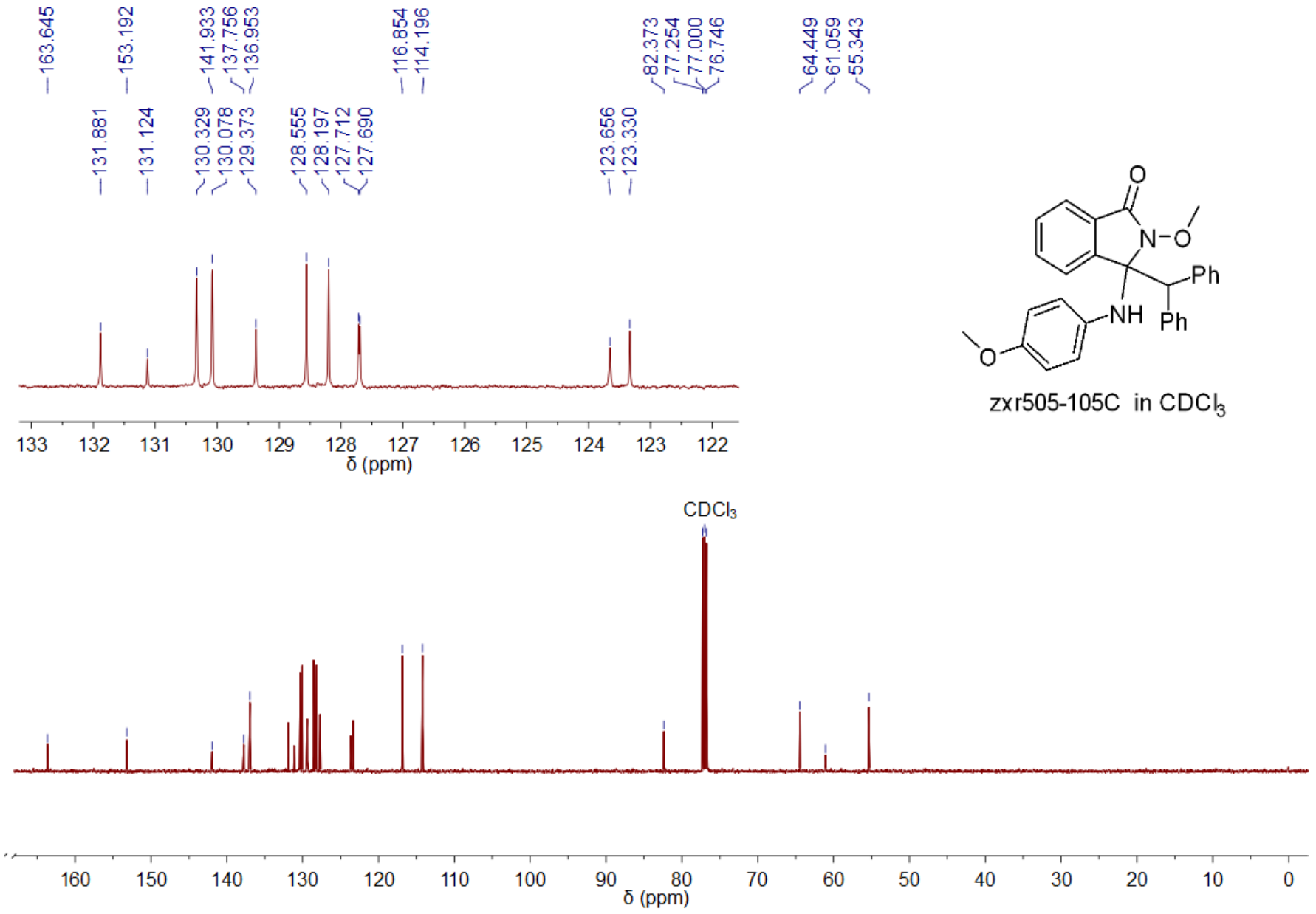
3k - ^{1}H NMR


163.508
142.234
141.174
137.409
136.416
123.558
123.470
116.702
111.470
81.563
77.252
77.000
76.747
64.631
60.925
132.115
131.649
131.057
130.325
130.008
129.684
128.750
128.244
127.931
127.901
CDCl3
O
N–O
Ph
NH
Ph
Br
zxr505-105B in CDCl3
δ (ppm)
δ (ppm)

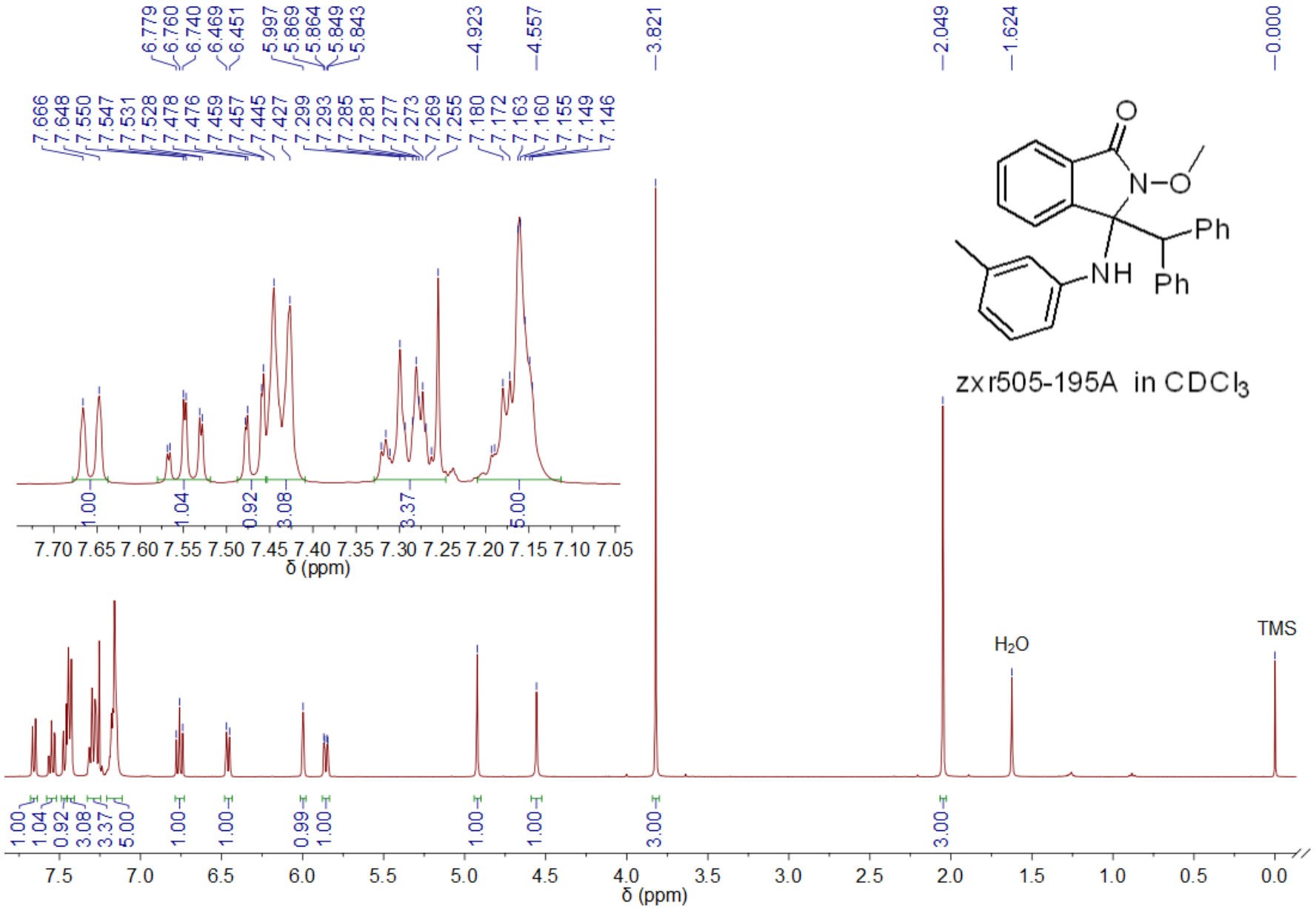
3k – ^{13}C NMR


6.489
6.471
6.117
6.099
4.911
4.400
3.806
3.601
0.000
7.645
7.630
7.551
7.549
7.536
7.535
7.521
7.441
7.433
7.428
7.419
7.410
7.285
7.270
7.259
7.253
7.245
7.175
7.168
7.159
1.00
1.03
4.09
3.43
5.02
7.60
7.50
7.40
7.30
7.20
7.10
δ (ppm)
O
N-O
Ph
NH
Ph
O
zxr505-105C in CDCl3
TMS
1.00
1.03
4.09
3.43
5.02
2.00
2.00
1.00
1.00
3.00
3.00
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
0.0
δ (ppm)

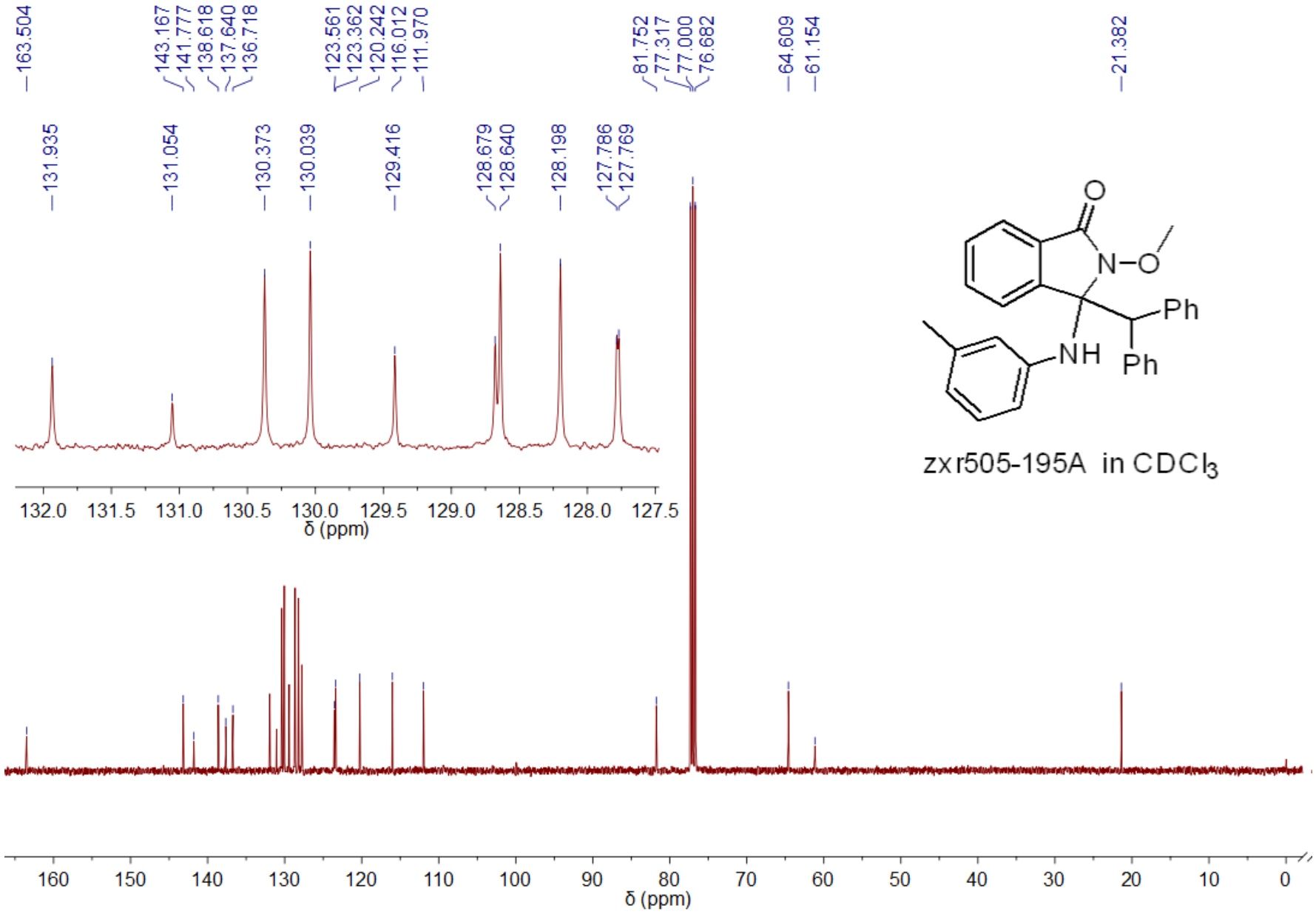
3l - ^{1}H NMR


163.645
153.192
141.933
137.756
136.953
116.854
114.196
82.373
77.254
77.000
76.746
64.449
61.059
55.343
131.881
131.124
130.329
130.078
129.373
128.555
128.197
127.712
127.690
123.656
123.330
O
N–O
Ph
NH
Ph
O
zxr505-105C in CDCl3
133 132 131 130 129 128 127 126 125 124 123 122
δ (ppm)
CDCl3
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

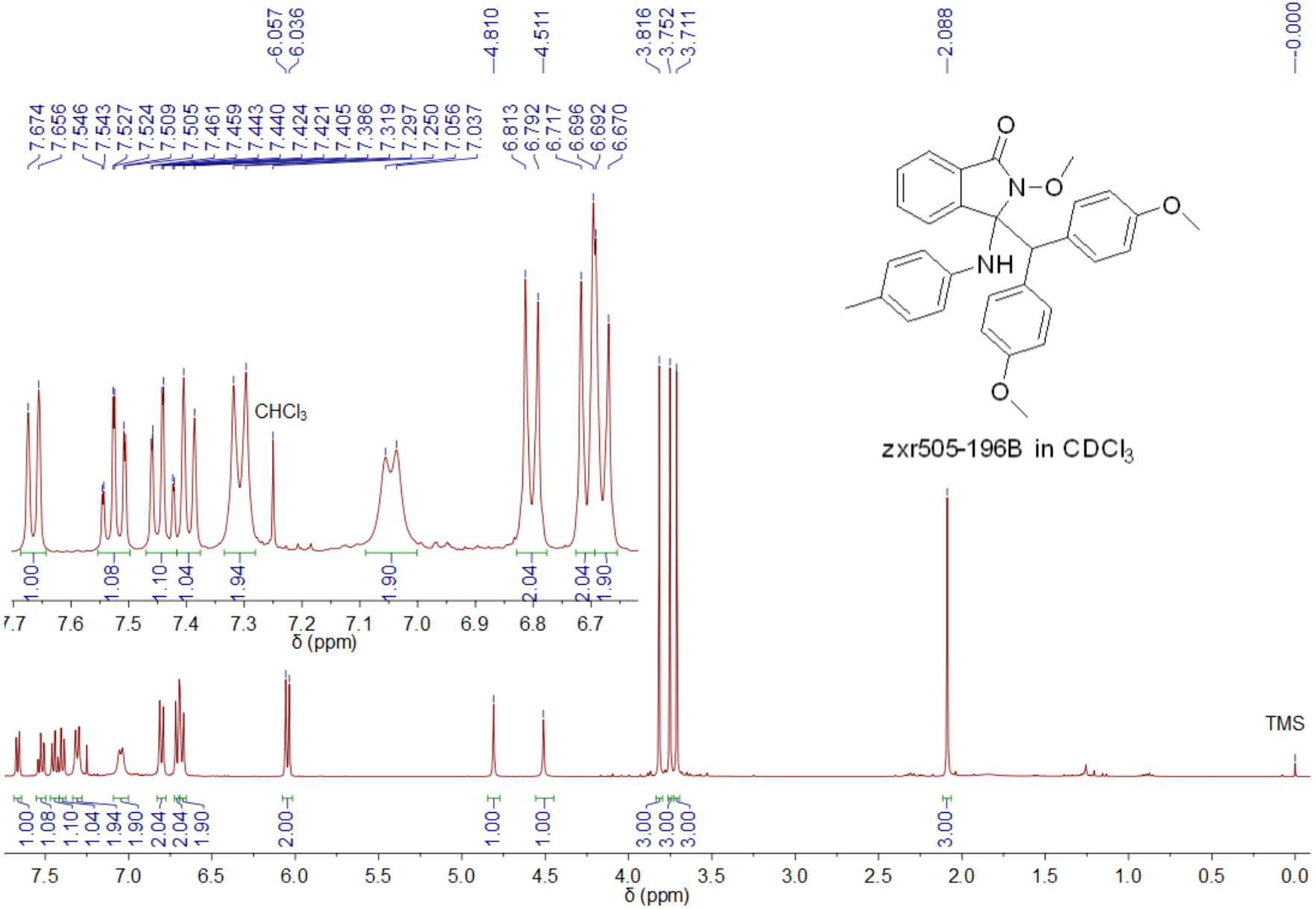
3l – ^{13}C NMR


6.779
6.760
6.740
6.469
6.451
5.997
5.869
5.864
5.849
5.843
4.923
4.557
3.821
2.049
1.624
0.000
7.666
7.648
7.550
7.547
7.531
7.528
7.478
7.476
7.459
7.457
7.445
7.427
7.299
7.293
7.285
7.281
7.277
7.273
7.269
7.255
7.180
7.172
7.163
7.160
7.155
7.149
7.146
O
N–O
Ph
NH
Ph
zxr505-195A in CDCl3
1.00
1.04
0.92
3.08
3.37
5.00
7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05
δ (ppm)
H2O
TMS
1.00
1.04
0.92
3.08
3.37
5.00
1.00
1.00
0.99
1.00
1.00
1.00
3.00
3.00
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 0.0
δ (ppm)

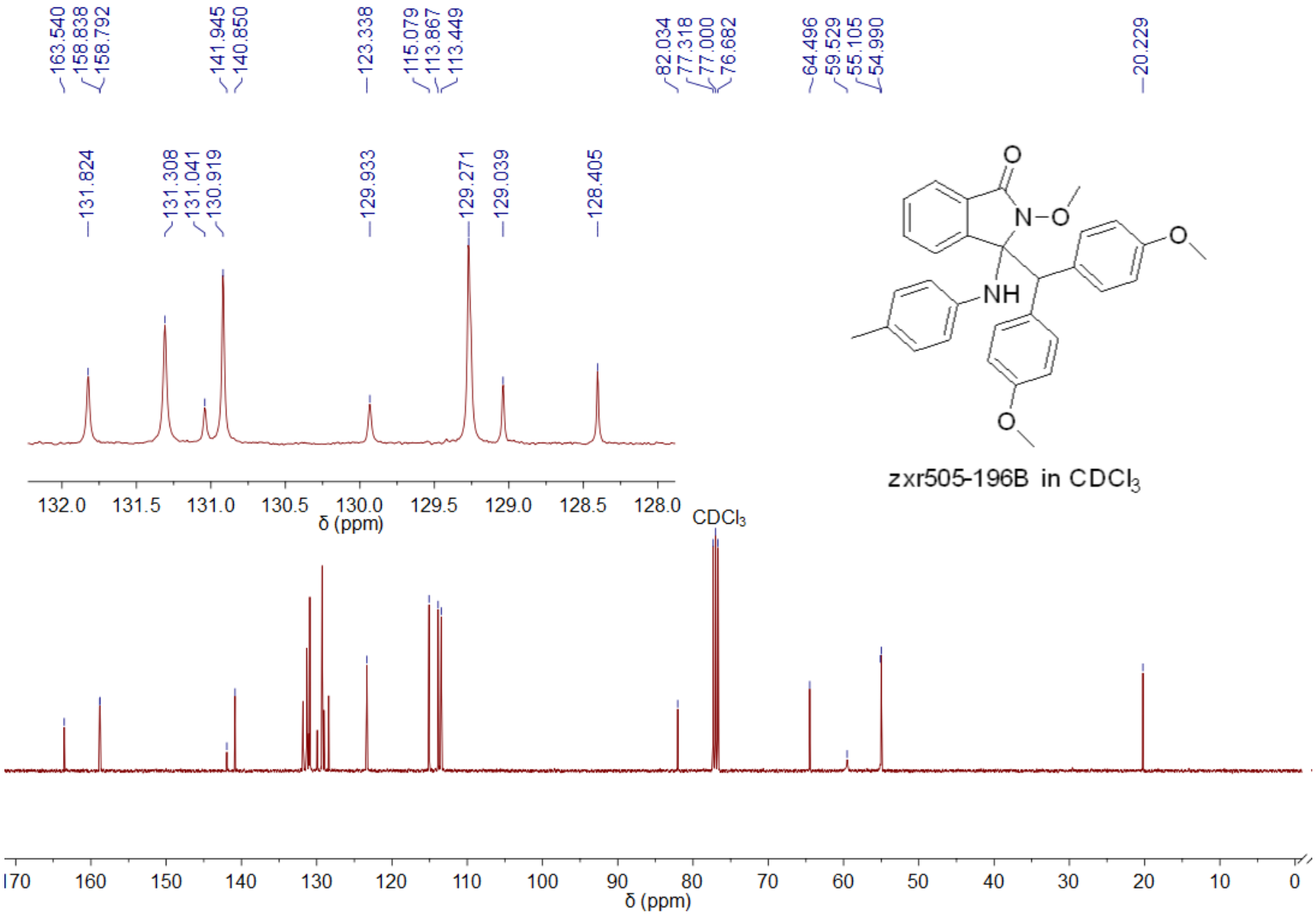
3m - ^{1}H NMR


163.504
143.167
141.777
138.618
137.640
136.718
123.561
123.362
120.242
116.012
111.970
81.752
77.317
77.000
76.682
64.609
61.154
21.382
131.935
131.054
130.373
130.039
129.416
128.679
128.640
128.198
127.786
127.769
O
N–O
Ph
Ph
NH
zxr505-195A in CDCl3
δ (ppm)
δ (ppm)

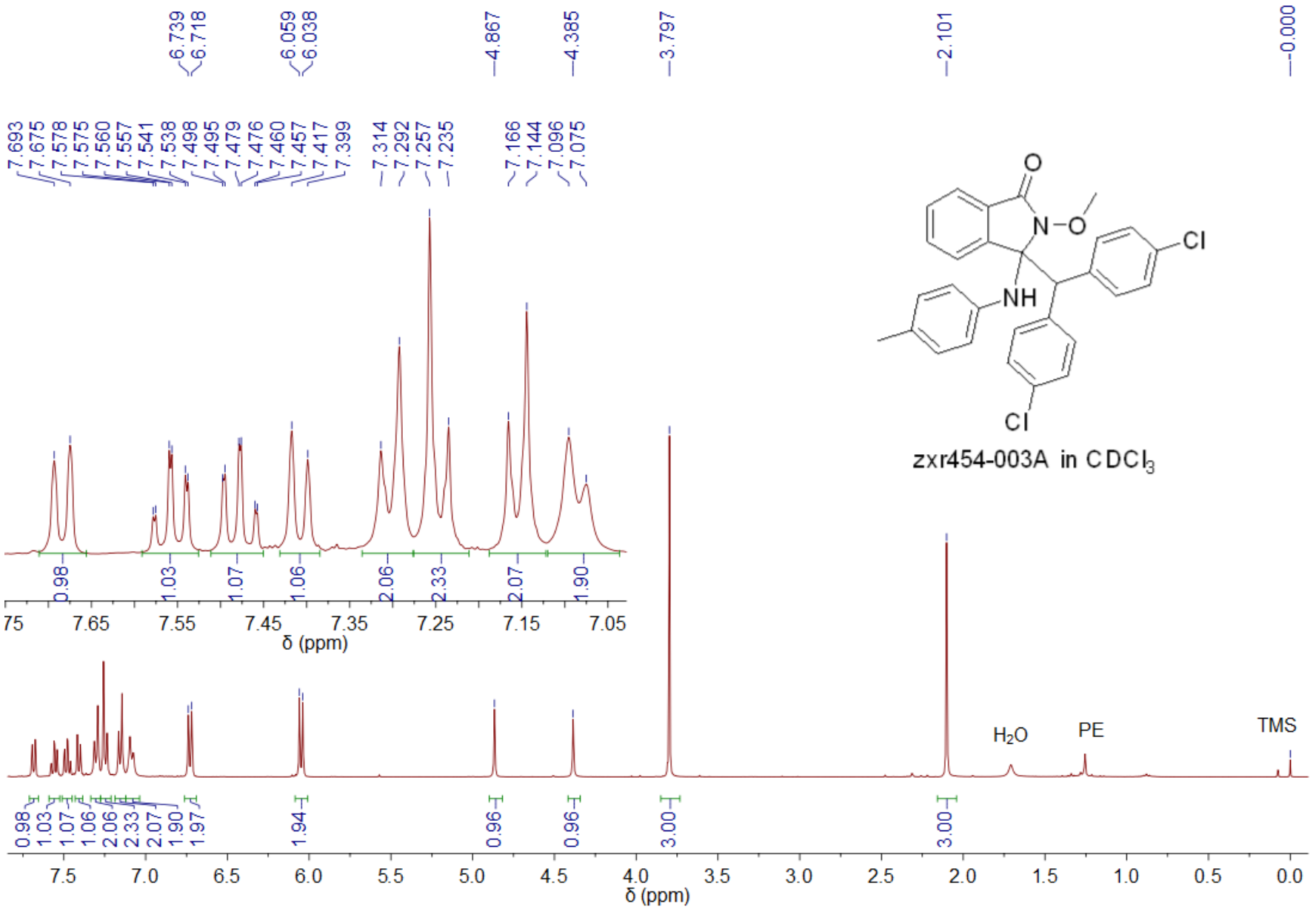
3m – ^{13}C NMR


6.057
6.036
4.810
4.511
3.816
3.752
3.711
2.088
0.000
7.674
7.656
7.546
7.543
7.527
7.524
7.509
7.505
7.461
7.459
7.443
7.440
7.424
7.421
7.405
7.386
7.319
7.297
7.250
7.056
7.037
6.813
6.792
6.717
6.696
6.692
6.670
CHCl3
zxr505-196B in CDCl3
TMS
δ (ppm)
δ (ppm)

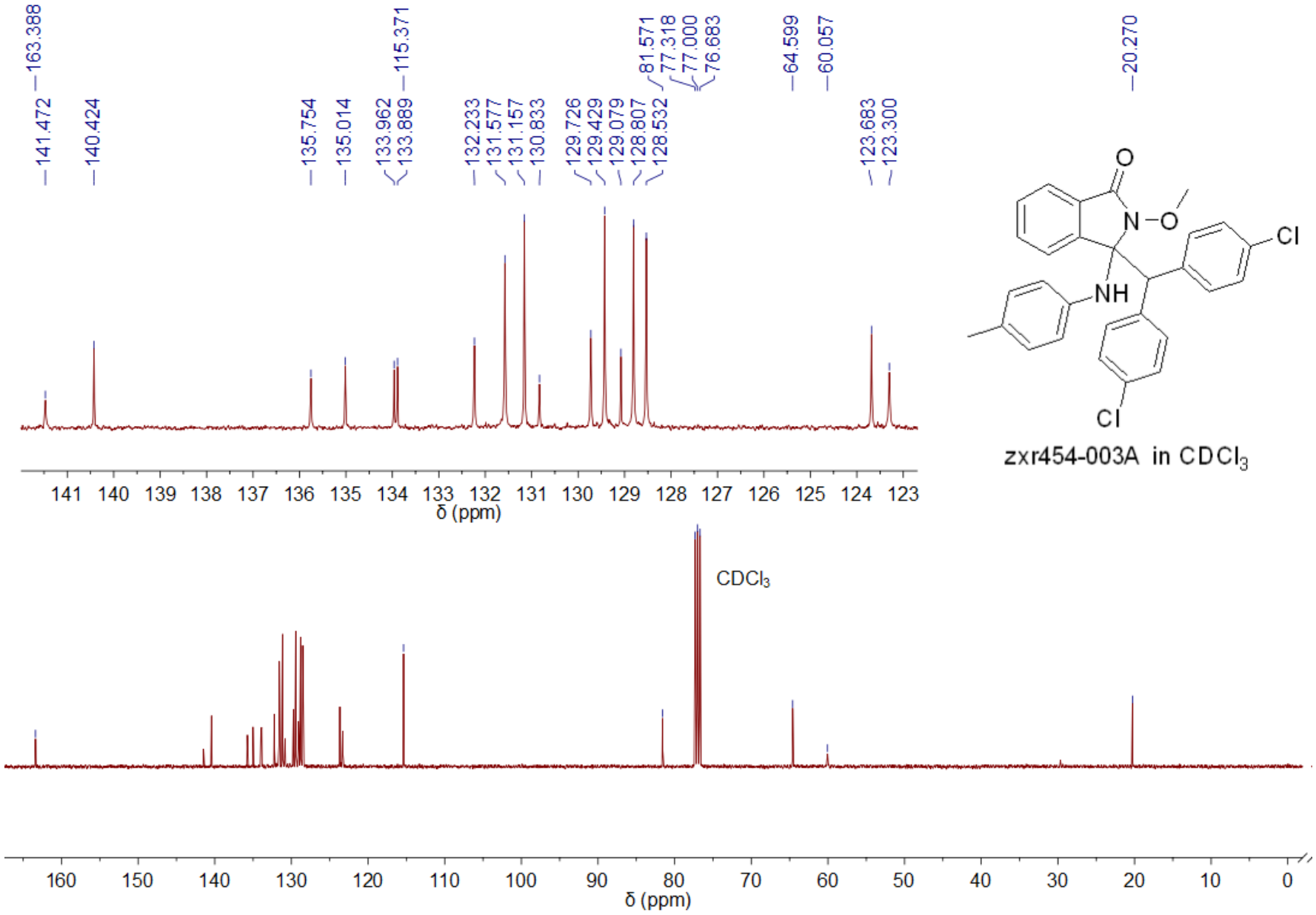
3n - ^{1}H NMR


163.540
158.838
158.792
141.945
140.850
123.338
115.079
113.867
113.449
82.034
77.318
77.000
76.682
64.496
59.529
55.105
54.990
20.229
131.824
131.308
131.041
130.919
129.933
129.271
129.039
128.405
O
N–O
O
NH
O
zxr505-196B in CDCl3
CDCl3
δ (ppm)
δ (ppm)

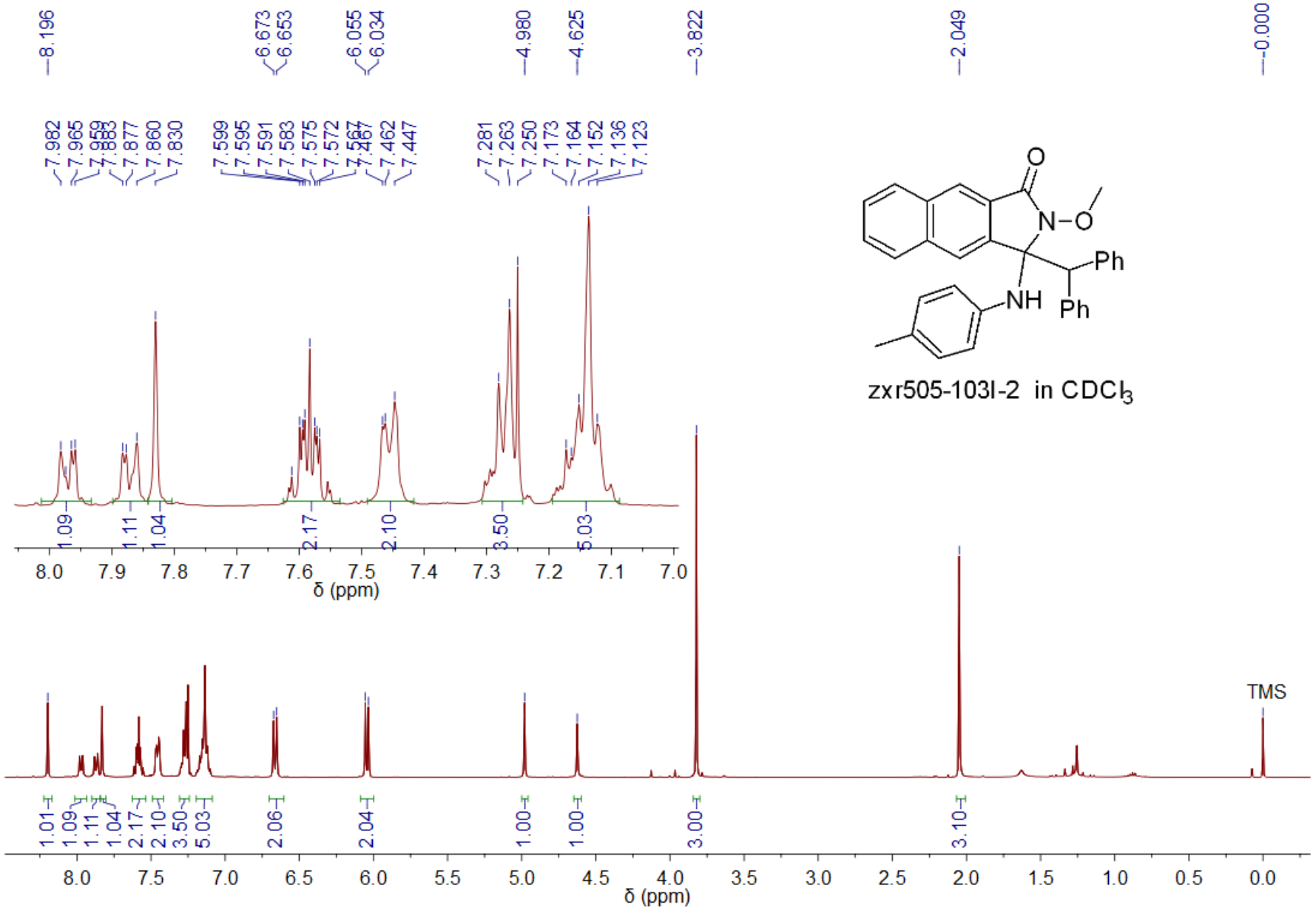
3n – ^{13}C NMR


6.739
6.718
6.059
6.038
4.867
4.385
3.797
2.101
0.000
7.693
7.675
7.578
7.575
7.560
7.557
7.541
7.538
7.498
7.495
7.479
7.476
7.460
7.457
7.417
7.399
7.314
7.292
7.257
7.235
7.166
7.144
7.096
7.075
O
N–O
Cl
NH
Cl
zxr454-003A in CDCl3
0.98
1.03
1.07
1.06
2.06
2.33
2.07
1.90
δ (ppm)
H2O
PE
TMS
0.98
1.03
1.07
1.06
2.06
2.33
2.07
1.90
1.97
1.94
0.96
0.96
3.00
3.00
δ (ppm)

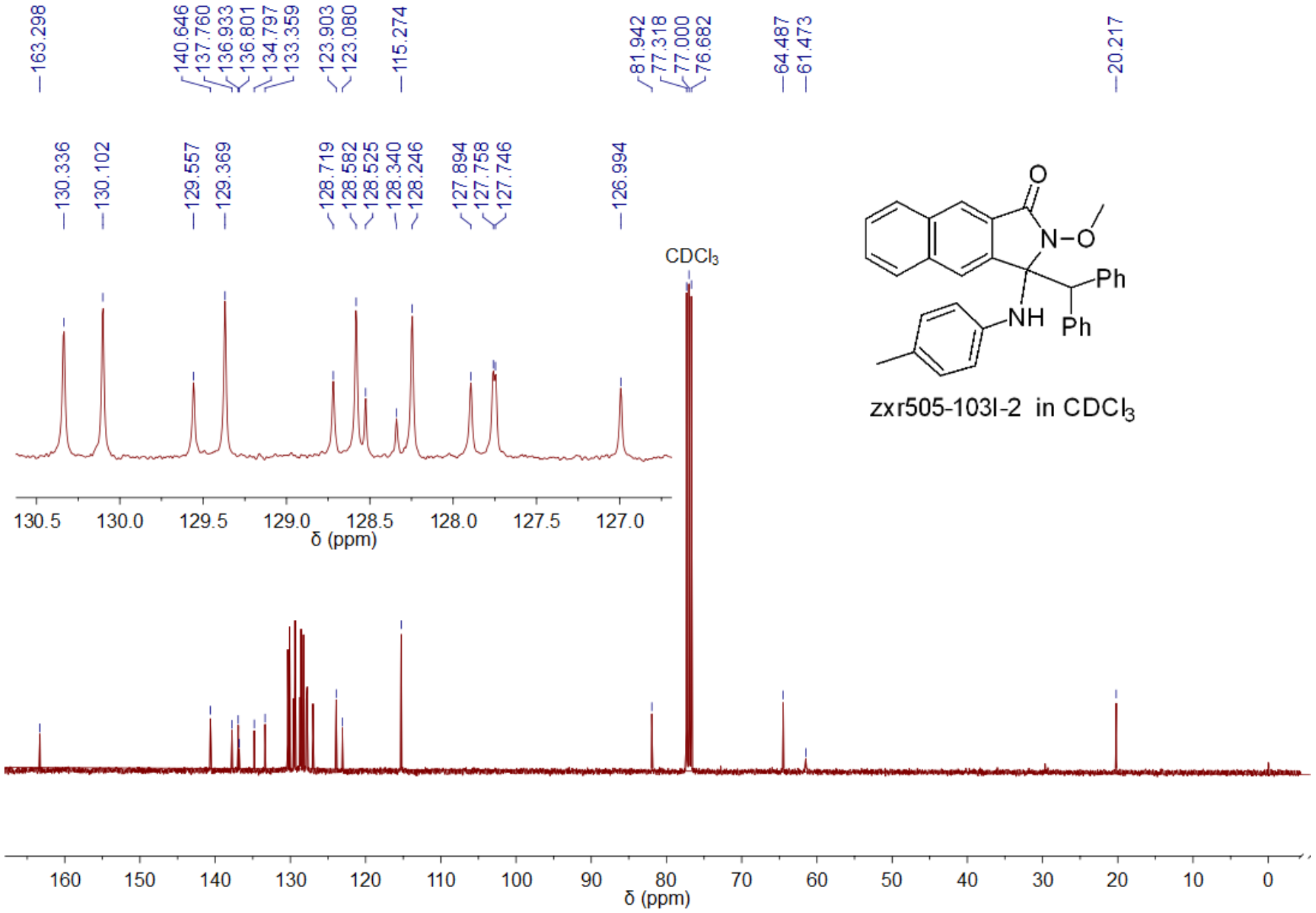
3o - ^{1}H NMR


163.388
141.472
140.424
135.754
135.014
133.962
133.889
115.371
132.233
131.577
131.157
130.833
129.726
129.429
129.079
128.807
128.532
81.571
77.318
77.000
76.683
64.599
60.057
123.683
123.300
20.270
O
N
O
Cl
NH
Cl
zxr454-003A in CDCl3
141 140 139 138 137 136 135 134 133 132 131 130 129 128 127 126 125 124 123
δ (ppm)
CDCl3
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

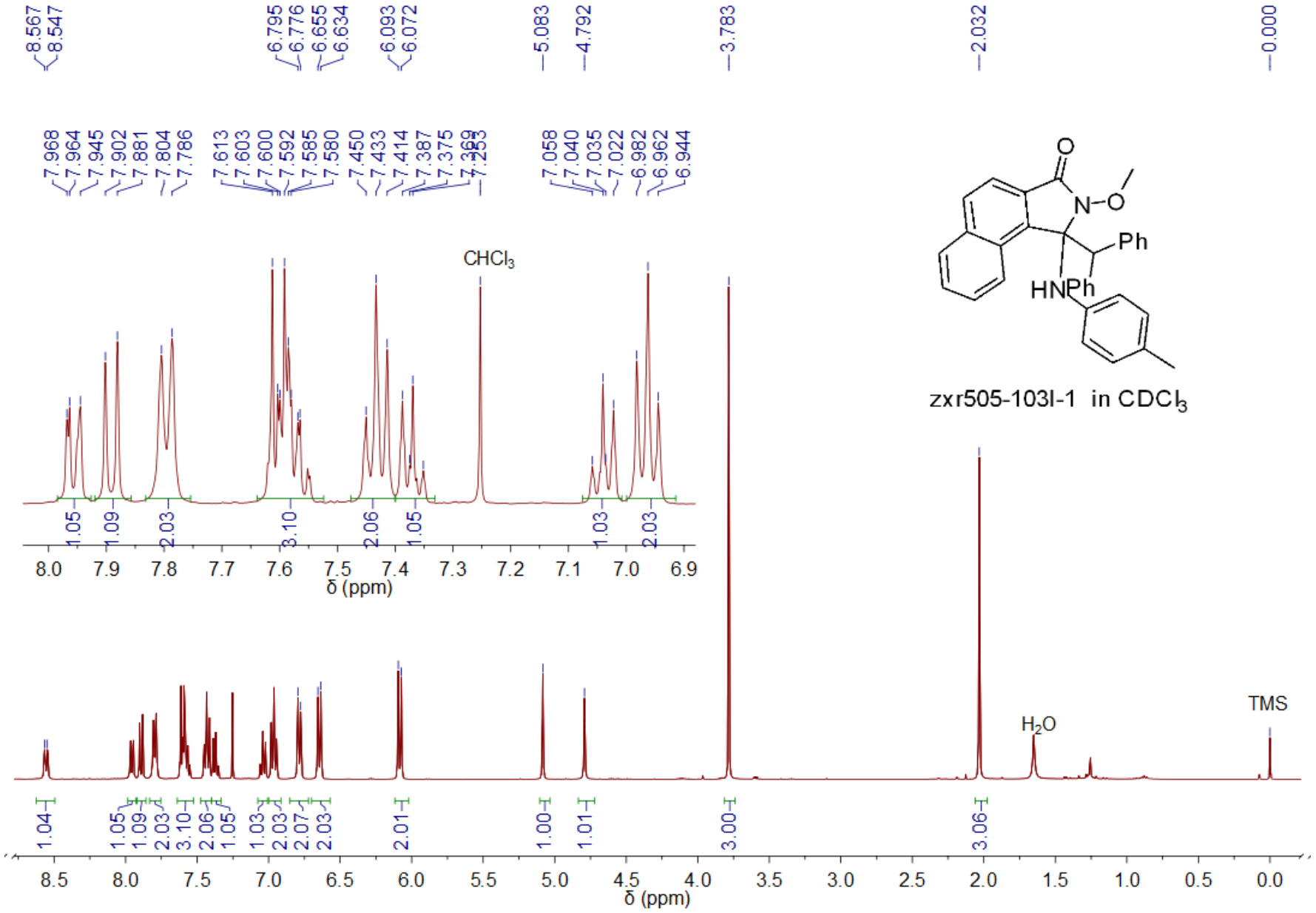
3o – ^{13}C NMR


8.196
6.673
6.653
6.055
6.034
4.980
4.625
3.822
2.049
0.000
7.982
7.965
7.959
7.883
7.877
7.860
7.830
7.599
7.595
7.591
7.583
7.575
7.572
7.567
7.462
7.447
7.281
7.263
7.250
7.173
7.164
7.152
7.136
7.123
O
N–O
Ph
NH
Ph
zxr505-103l-2 in CDCl3
1.09
1.11
1.04
2.17
2.10
3.50
5.03
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
δ (ppm)
TMS
1.01
1.09
1.11
1.04
2.17
2.10
3.50
5.03
2.06
2.04
1.00
1.00
3.00
3.10
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
0.0
δ (ppm)

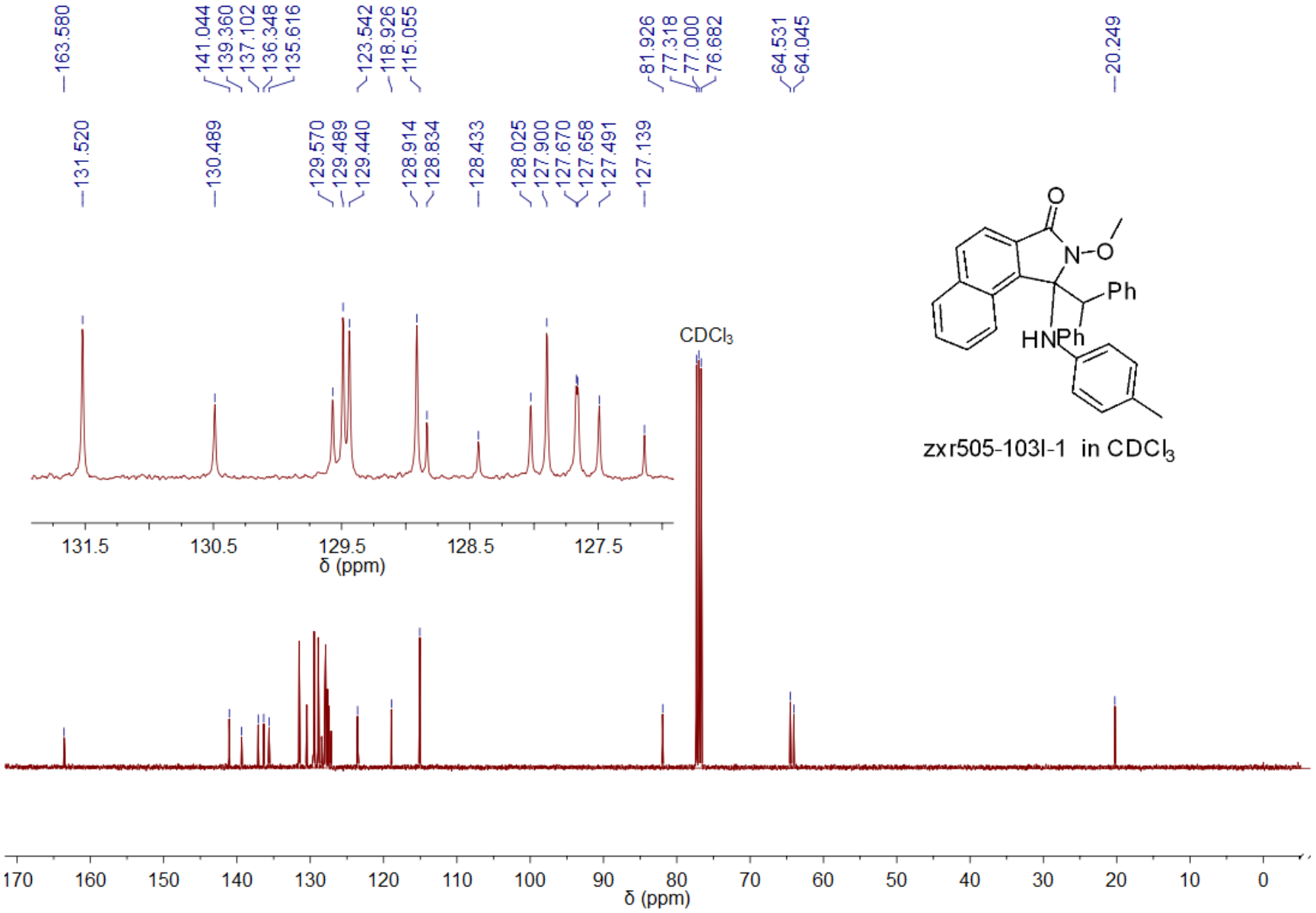
3pa - ^{1}H NMR


163.298
140.646
137.760
136.933
136.801
134.797
133.359
123.903
123.080
115.274
81.942
77.318
77.000
76.682
64.487
61.473
20.217
130.336
130.102
129.557
129.369
128.719
128.582
128.525
128.340
128.246
127.894
127.758
127.746
126.994
CDCl3
O
N–O
Ph
NH
Ph
zxr505-103l-2 in CDCl3
130.5
130.0
129.5
129.0
128.5
128.0
127.5
127.0
δ (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

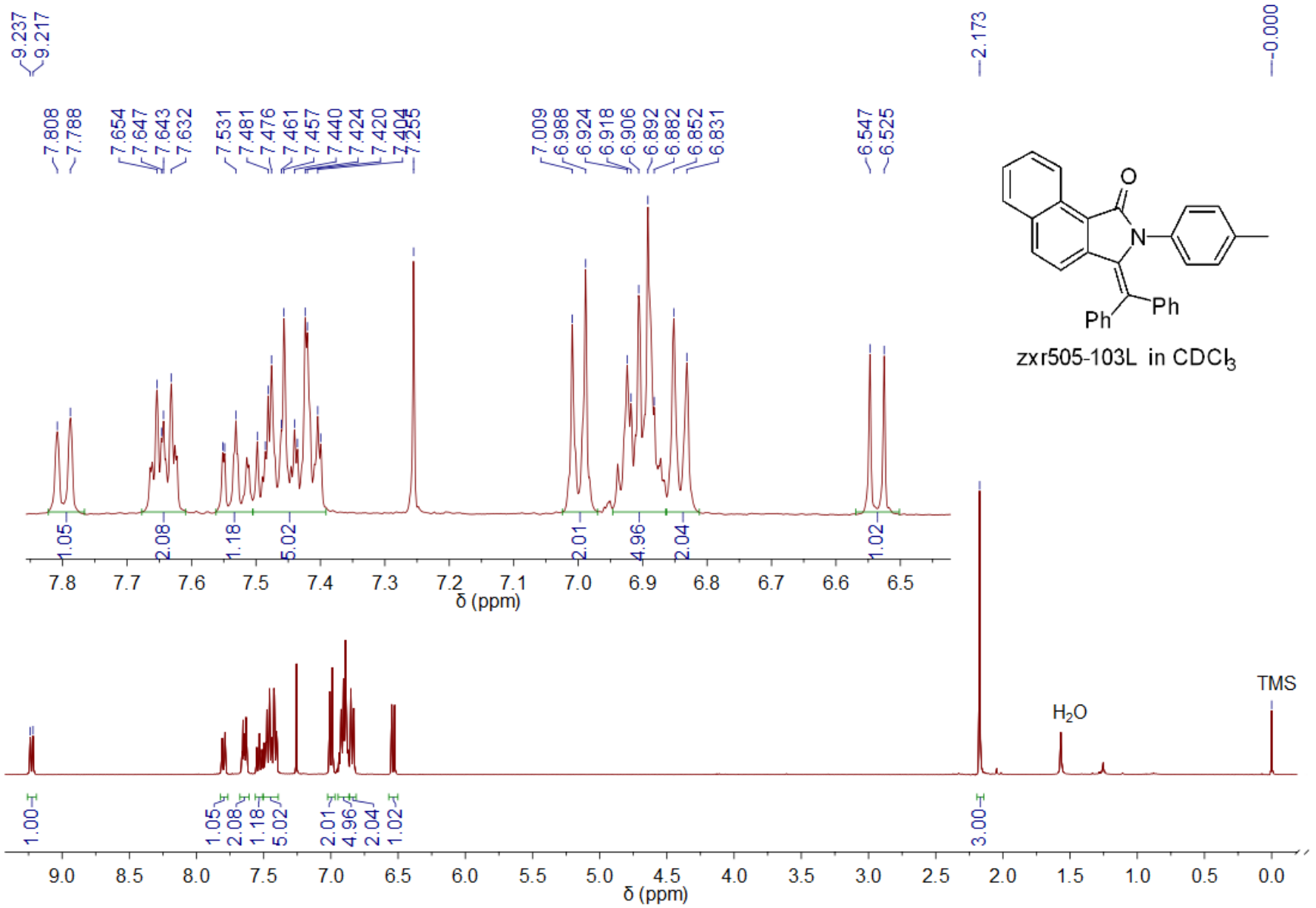
3pa – ^{13}C NMR


8.567
8.547
6.795
6.776
6.655
6.634
6.093
6.072
5.083
4.792
3.783
2.032
0.000
7.968
7.964
7.945
7.902
7.881
7.804
7.786
7.613
7.603
7.600
7.592
7.585
7.580
7.450
7.433
7.414
7.387
7.375
7.058
7.040
7.035
7.022
6.982
6.962
6.944
CHCl3
O
N
O
Ph
HNPh
zxr505-103l-1 in CDCl3
H2O
TMS
δ (ppm)
δ (ppm)

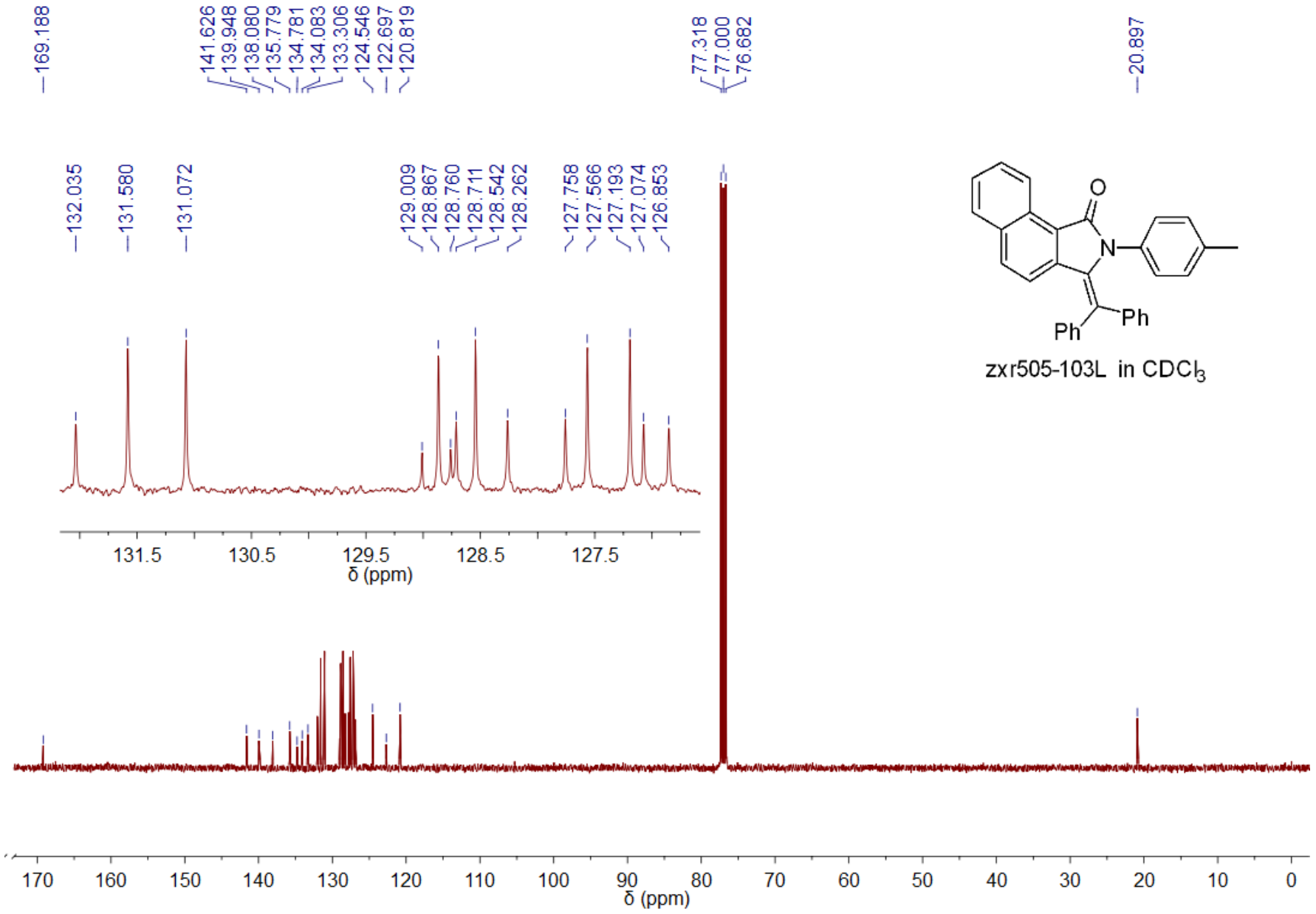
3pb - ^{1}H NMR


163.580
141.044
139.360
137.102
136.348
135.616
123.542
118.926
115.055
81.926
77.318
77.000
76.682
64.531
64.045
20.249
131.520
130.489
129.570
129.489
129.440
128.914
128.834
128.433
128.025
127.900
127.670
127.658
127.491
127.139
CDCl3
O
N-O
Ph
HNPh
zxr505-103l-1 in CDCl3
131.5
130.5
129.5
128.5
127.5
δ (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

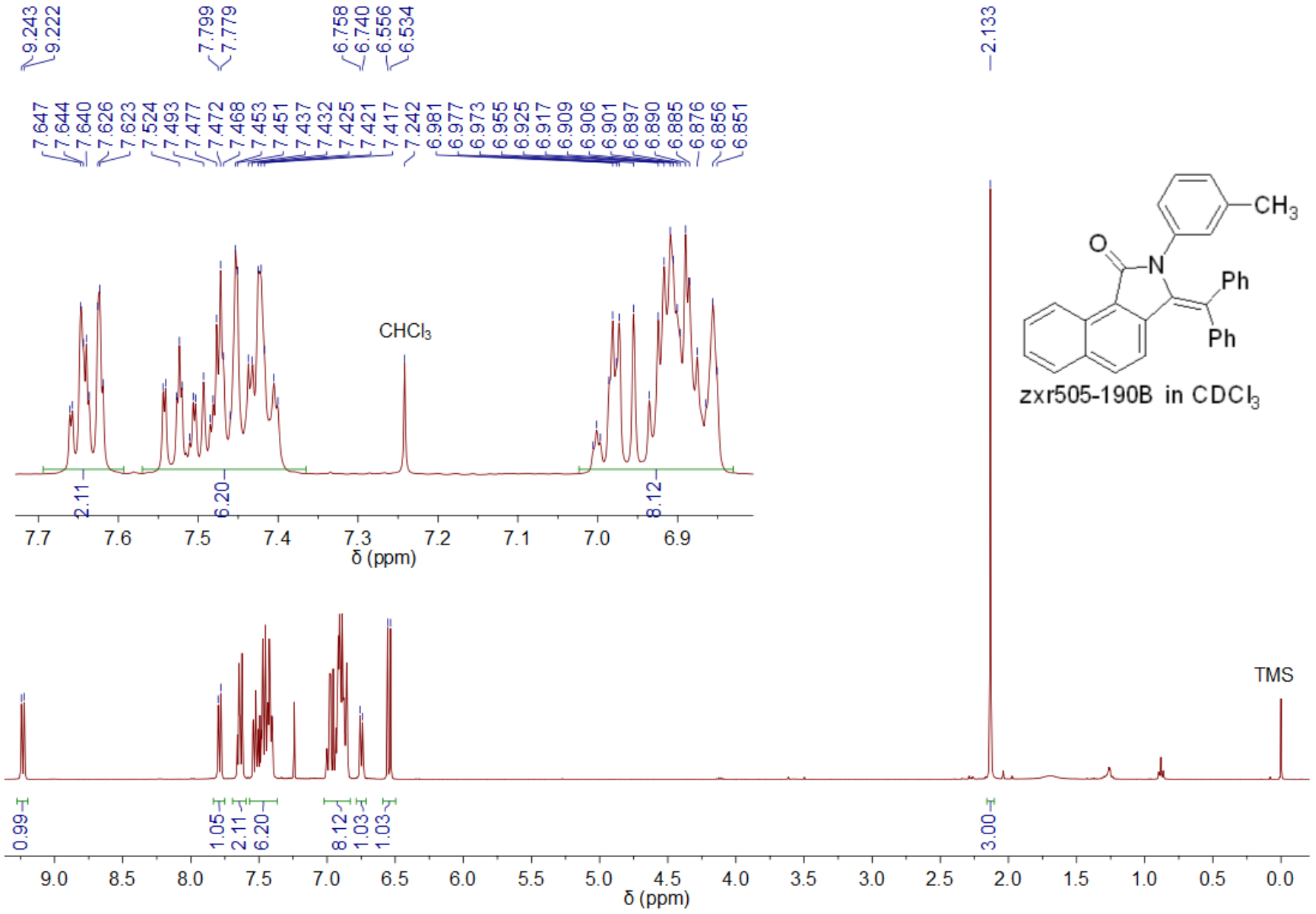
3pb – ^{13}C NMR


9.237
9.217
2.173
0.000
7.808
7.788
7.654
7.647
7.643
7.632
7.531
7.481
7.476
7.461
7.457
7.440
7.424
7.420
7.404
7.255
7.009
6.988
6.924
6.918
6.906
6.892
6.882
6.852
6.831
6.547
6.525
O
N
Ph
Ph
zxr505-103L in CDCl3
1.05
2.08
1.18
5.02
2.01
4.96
2.04
1.02
δ (ppm)
TMS
H2O
1.00
3.00
δ (ppm)

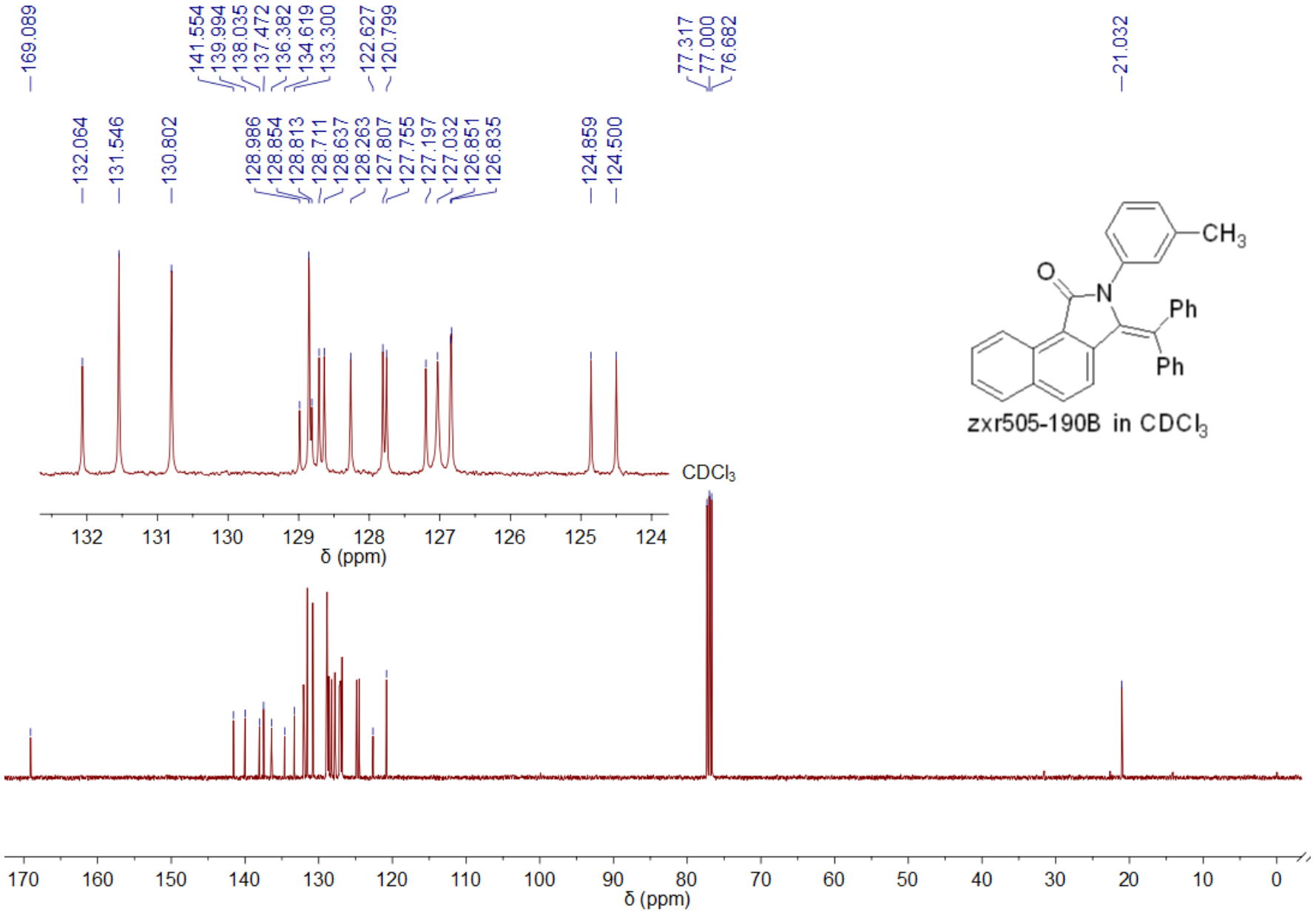
4a - ^{1}H NMR


169.188
141.626
139.948
138.080
135.779
134.781
134.083
133.306
124.546
122.697
120.819
77.318
77.000
76.682
20.897
132.035
131.580
131.072
129.009
128.867
128.760
128.711
128.542
128.262
127.758
127.566
127.193
127.074
126.853
O
N
Ph
Ph
zxr505-103L in CDCl3
131.5
130.5
129.5
128.5
127.5
δ (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

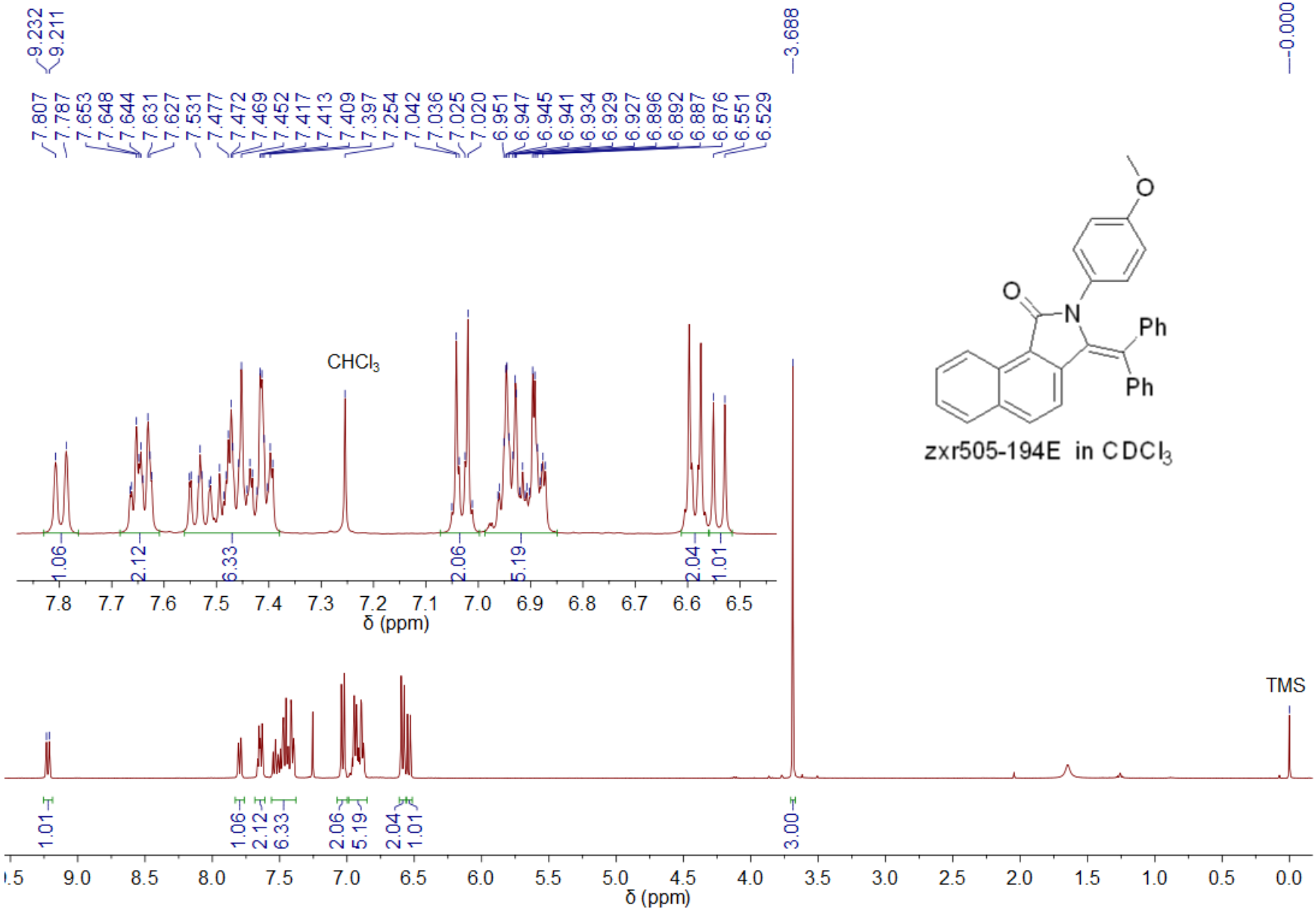
4a – ^{13}C NMR


9.243
9.222
7.799
7.779
6.758
6.740
6.556
6.534
2.133
7.647
7.644
7.640
7.626
7.623
7.524
7.493
7.477
7.472
7.468
7.453
7.451
7.437
7.432
7.425
7.421
7.417
7.242
6.981
6.977
6.973
6.955
6.925
6.917
6.909
6.906
6.901
6.897
6.890
6.885
6.876
6.856
6.851
CHCl3
2.11
6.20
8.12
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
δ (ppm)
CH3
O
N
Ph
Ph
zxr505-190B in CDCl3
TMS
0.99
1.05
2.11
6.20
8.12
1.03
1.03
3.00
9.0
8.5
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
0.0
δ (ppm)

4b - ^{1}H NMR


169.089
141.554
139.994
138.035
137.472
136.382
134.619
133.300
122.627
120.799
77.317
77.000
76.682
21.032
132.064
131.546
130.802
128.986
128.854
128.813
128.711
128.637
128.263
127.807
127.755
127.197
127.032
126.851
126.835
124.859
124.500
CH3
O
N
Ph
Ph
zxr505-190B in CDCl3
CDCl3
132
131
130
129
128
127
126
125
124
δ (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

4b – ^{13}C NMR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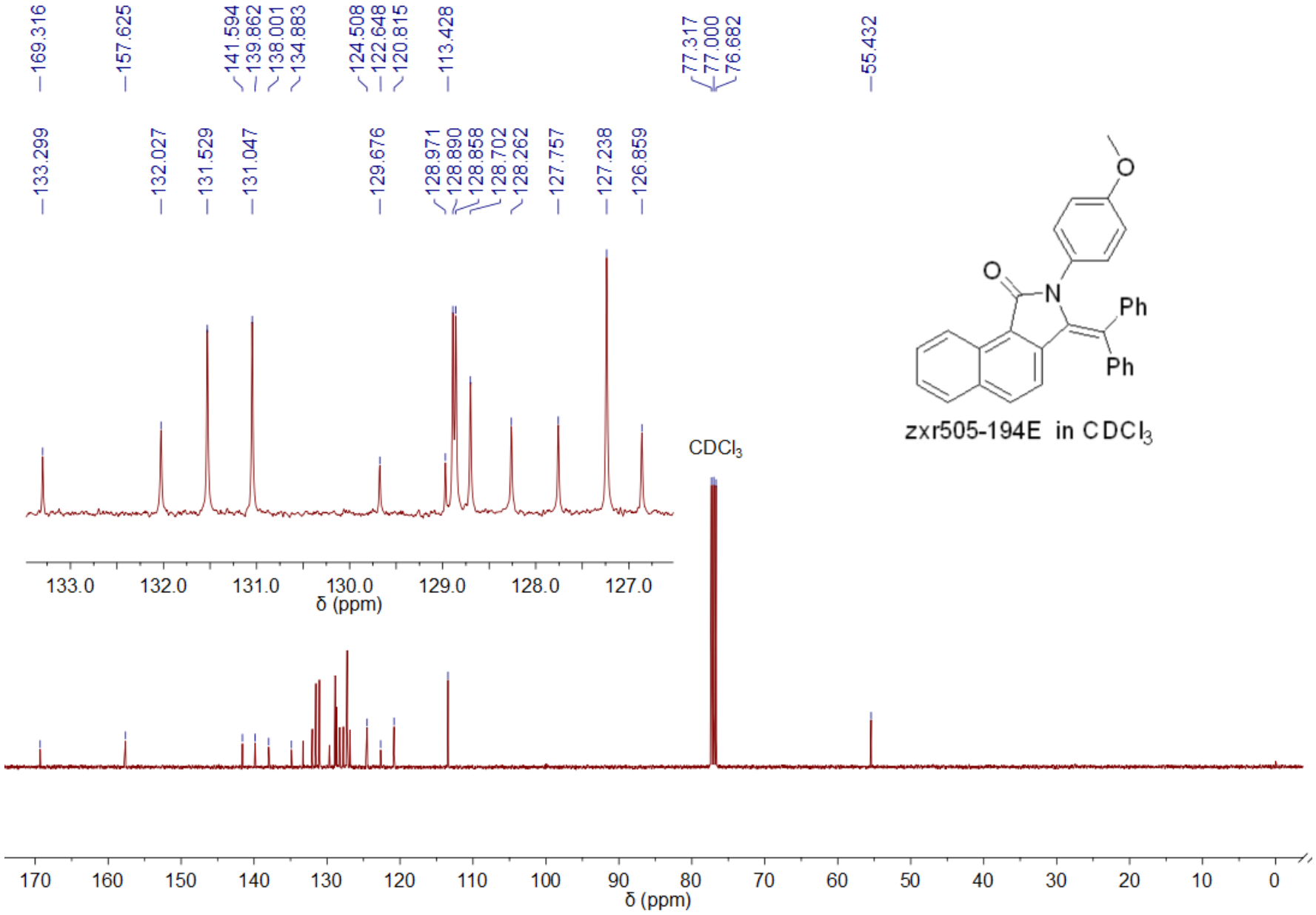
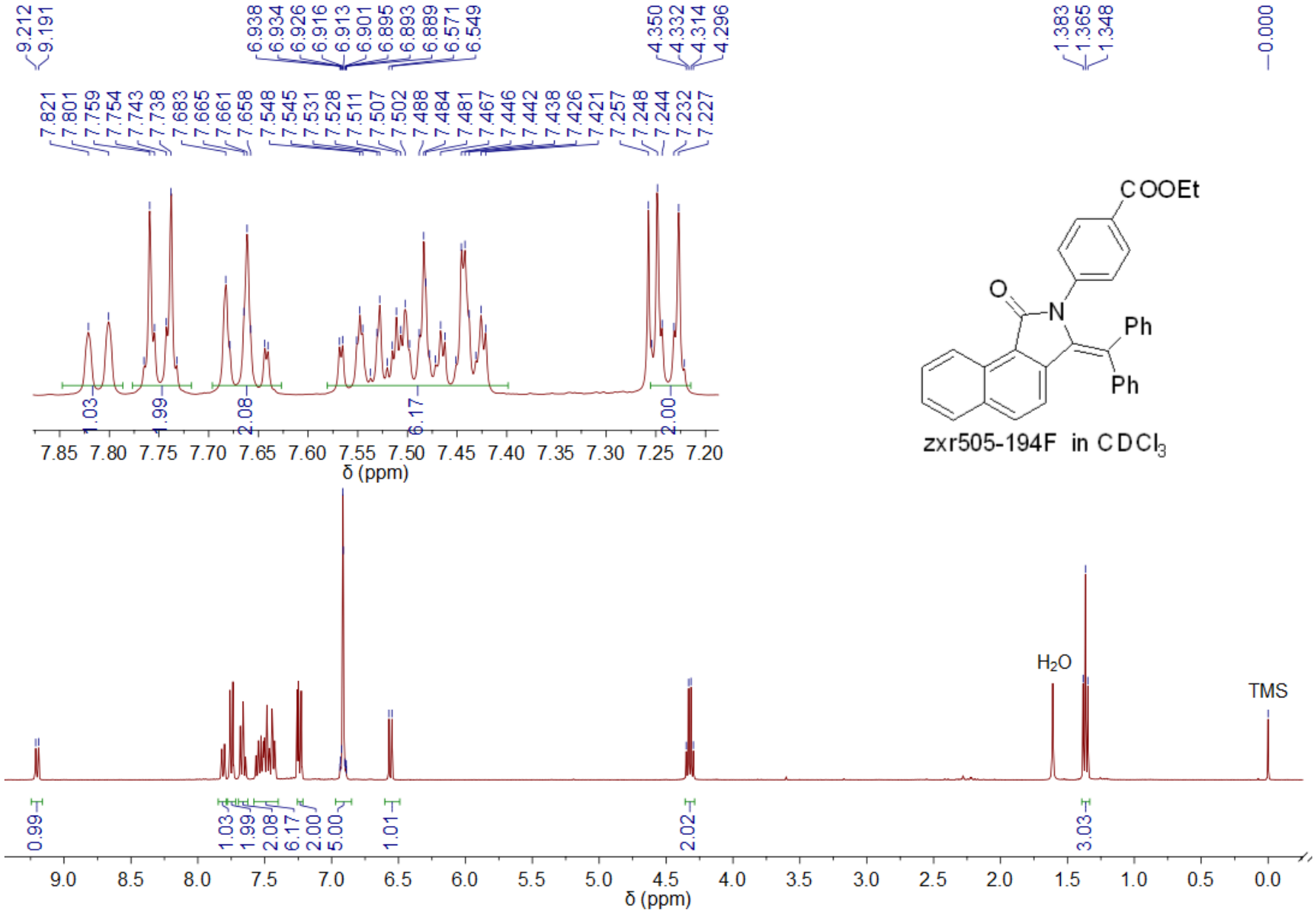
9.211
7.807
7.787
7.653
7.648
7.644
7.631
7.627
7.531
7.477
7.472
7.469
7.452
7.417
7.413
7.409
7.397
7.254
7.042
7.036
7.025
7.020
6.951
6.947
6.945
6.941
6.934
6.929
6.927
6.896
6.892
6.887
6.876
6.551
6.529
3.688
0.000
CHCl3
O
O
N
Ph
Ph
zxr505-194E in CDCl3
1.06
2.12
6.33
2.06
5.19
2.04
1.01
δ (ppm)
TMS
3.00
δ (ppm)

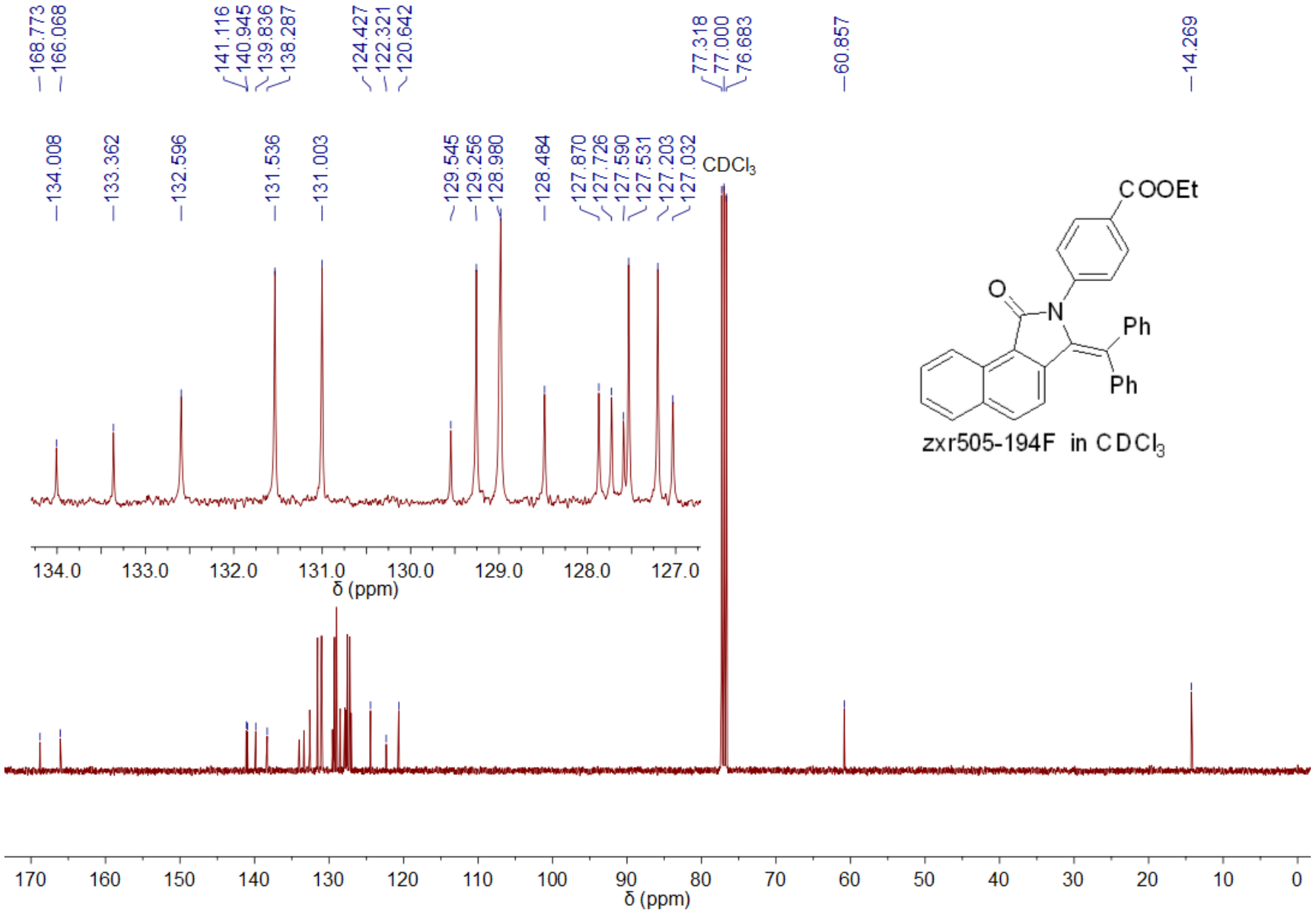
4c - ^{1}H NMR


169.316
157.625
141.594
139.862
138.001
134.883
124.508
122.648
120.815
113.428
77.317
77.000
76.682
55.432
133.299
132.027
131.529
131.047
129.676
128.971
128.890
128.858
128.702
128.262
127.757
127.238
126.859
CDCl3
O
N
Ph
Ph
O
zxr505-194E in CDCl3
133.0
132.0
131.0
130.0
129.0
128.0
127.0
δ (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

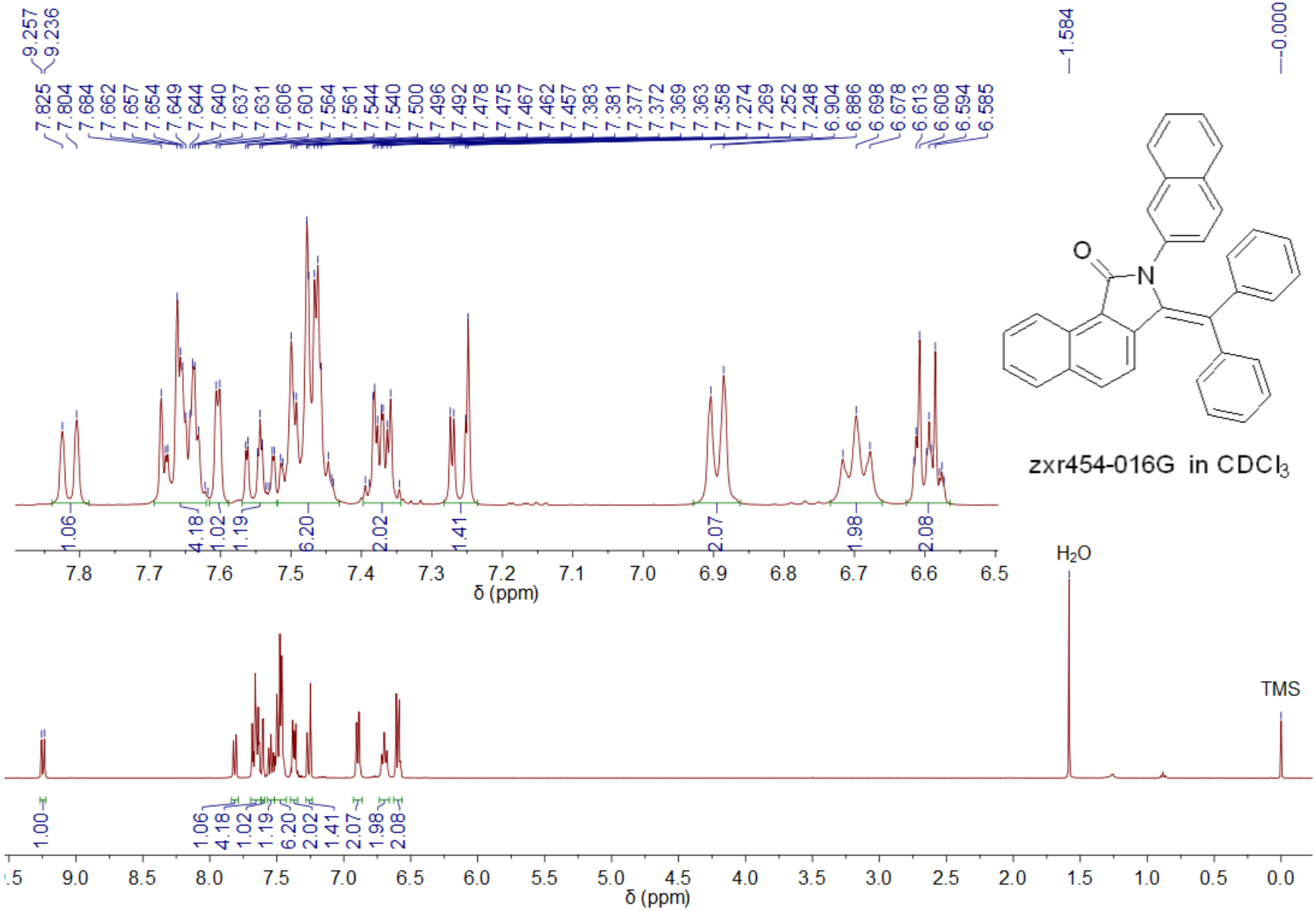
4c – ^{13}C NMR


9.212
9.191
6.938
6.934
6.926
6.916
6.913
6.901
6.895
6.893
6.889
6.571
6.549
4.350
4.332
4.314
4.296
1.383
1.365
1.348
0.000
7.821
7.801
7.759
7.754
7.743
7.738
7.683
7.665
7.661
7.658
7.548
7.545
7.531
7.528
7.511
7.507
7.502
7.488
7.484
7.481
7.467
7.446
7.442
7.438
7.426
7.421
7.257
7.248
7.244
7.232
7.227
1.03
1.99
2.08
6.17
2.00
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 7.20
δ (ppm)
COOEt
O
N
Ph
Ph
zxr505-194F in CDCl3
H2O
TMS
0.99
1.03
1.99
2.08
6.17
2.00
5.00
1.01
2.02
3.03
9.0 8.5 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 0.0
δ (ppm)

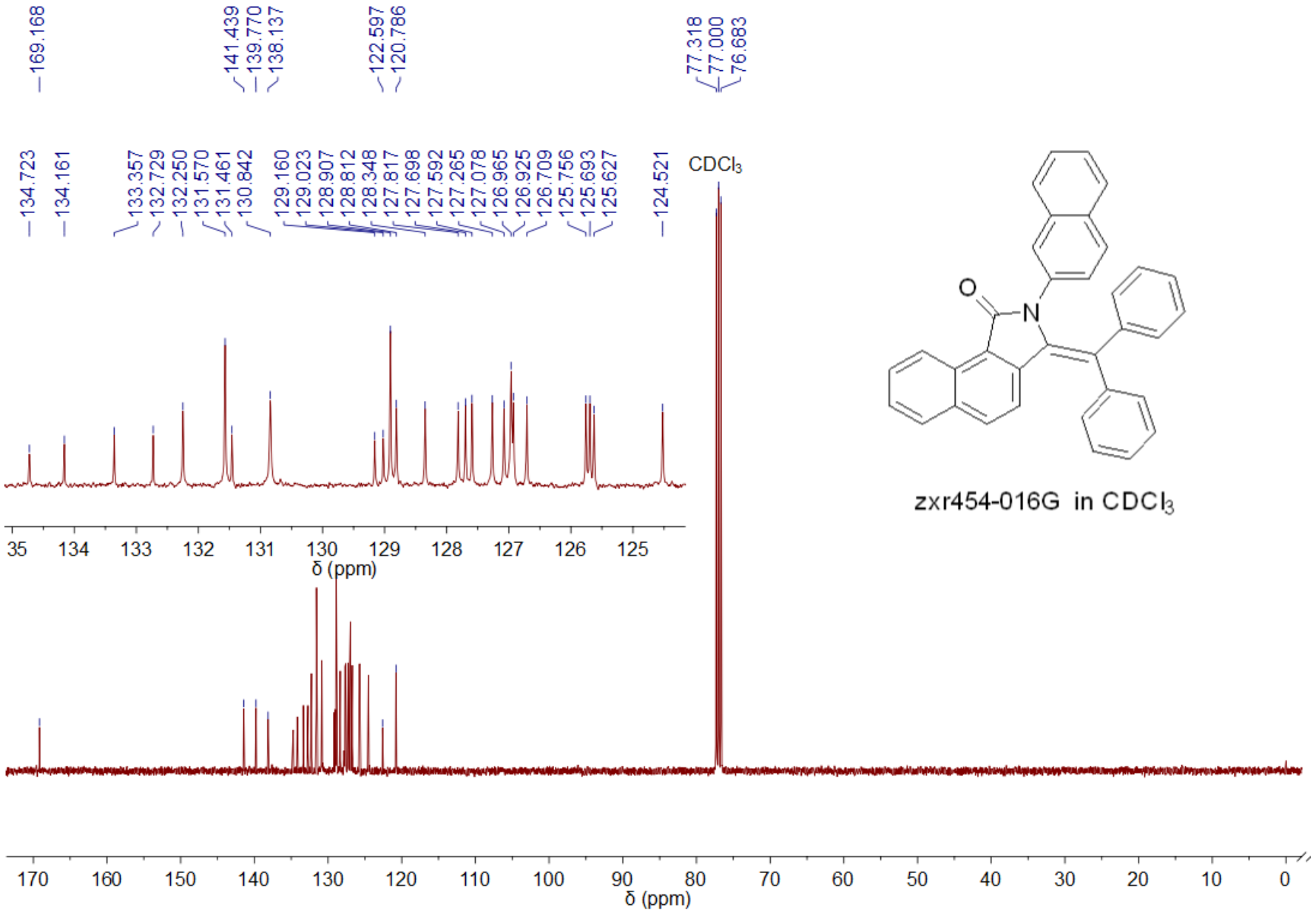
4d - ^{1}H NMR


168.773
166.068
141.116
140.945
139.836
138.287
124.427
122.321
120.642
77.318
77.000
76.683
60.857
14.269
134.008
133.362
132.596
131.536
131.003
129.545
129.256
128.980
128.484
127.870
127.726
127.590
127.531
127.203
127.032
CDCl3
COOEt
O
N
Ph
Ph
zxr505-194F in CDCl3
134.0
133.0
132.0
131.0
130.0
129.0
128.0
127.0
δ (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

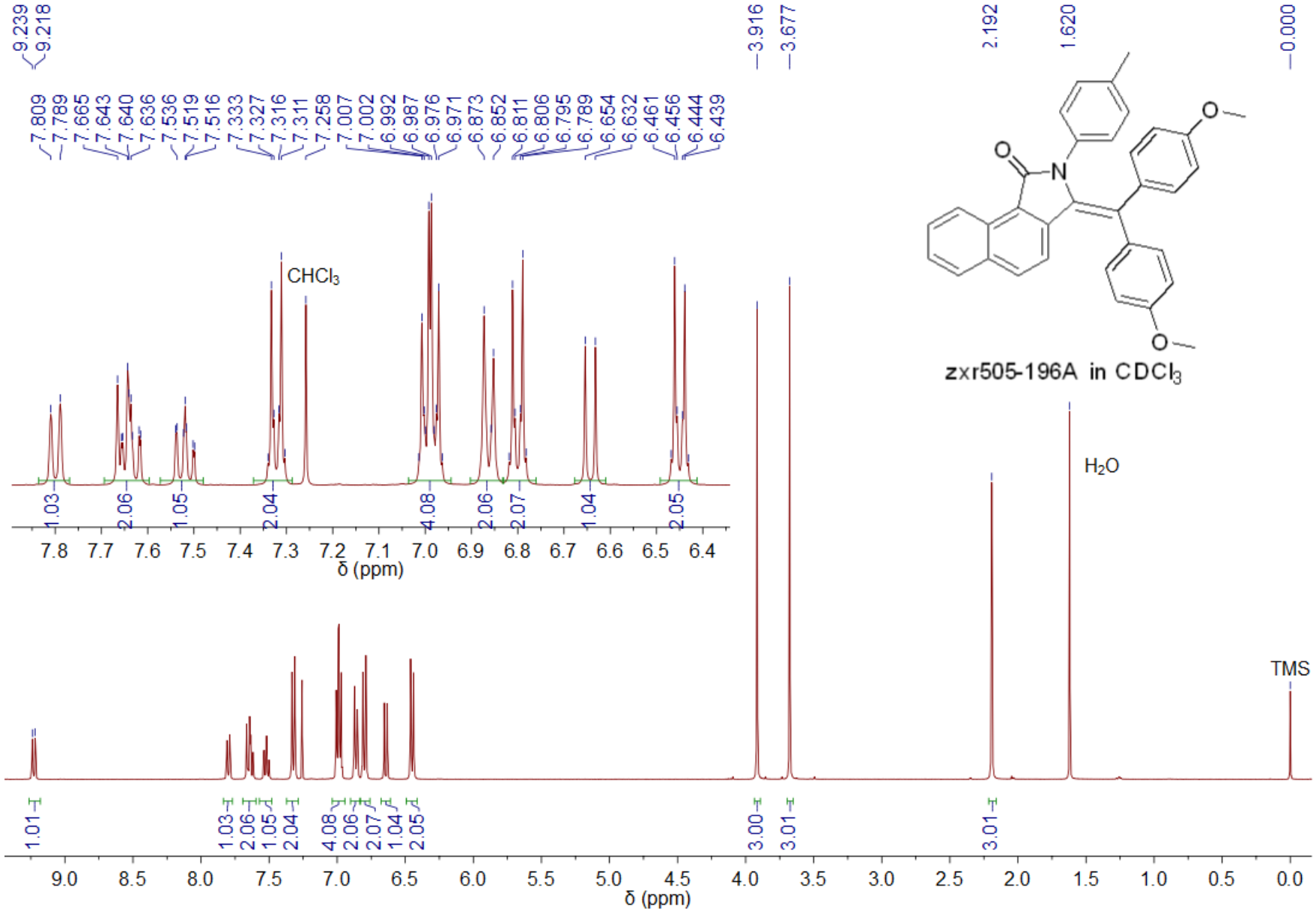
4d – ^{13}C NMR


9.257
9.236
7.825
7.804
7.684
7.662
7.657
7.654
7.649
7.644
7.640
7.637
7.631
7.606
7.601
7.564
7.561
7.544
7.540
7.500
7.496
7.492
7.478
7.475
7.467
7.462
7.457
7.383
7.381
7.377
7.372
7.369
7.363
7.358
7.274
7.269
7.252
7.248
6.904
6.886
6.698
6.678
6.613
6.608
6.594
6.585
1.584
0.000
O
N
zxr454-016G in CDCl3
1.06
4.18
1.02
1.19
6.20
2.02
1.41
2.07
1.98
2.08
δ (ppm)
H2O
TMS
1.00
δ (ppm)

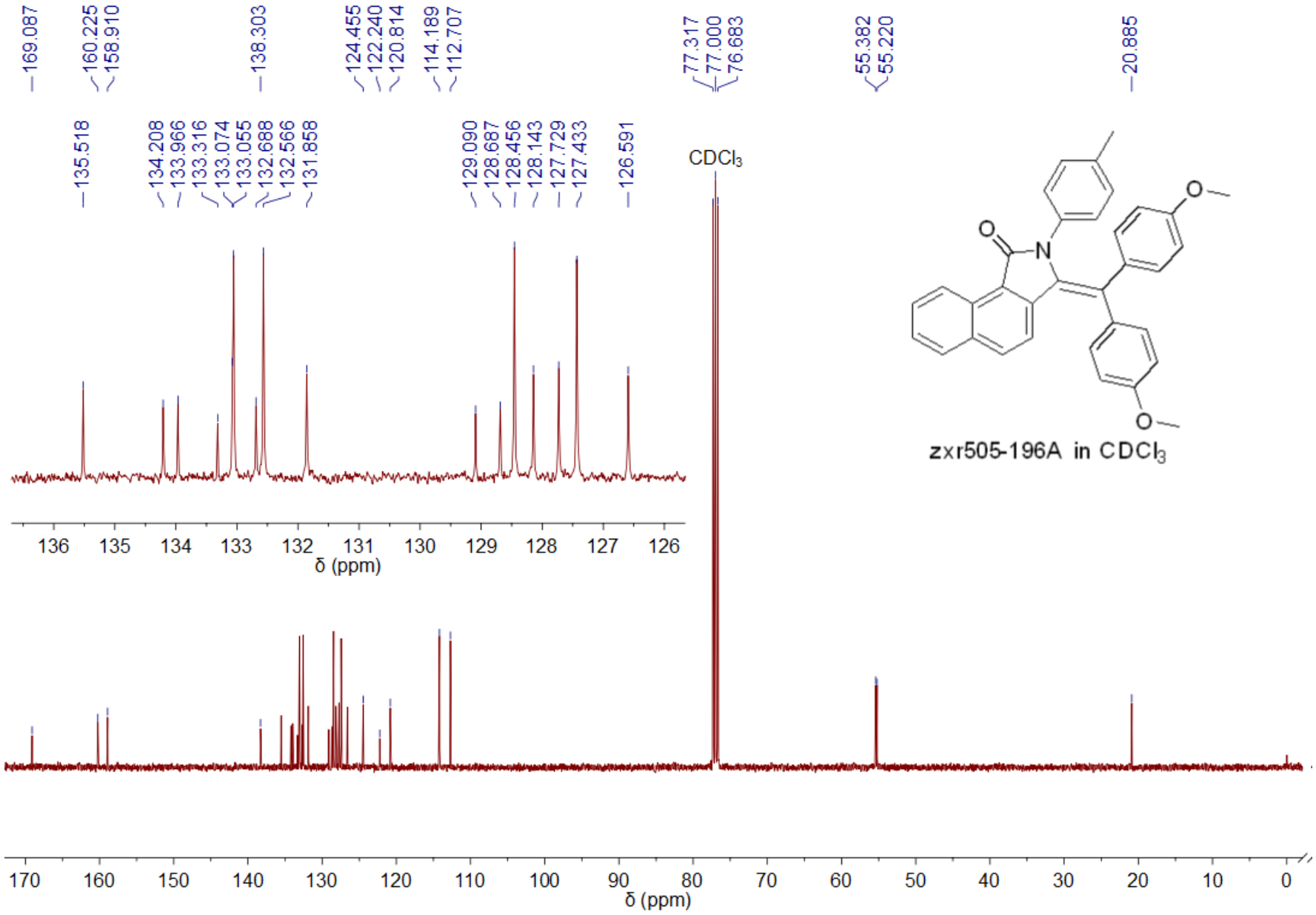
4e - ^{1}H NMR


169.168
141.439
139.770
138.137
122.597
120.786
77.318
77.000
76.683
134.723
134.161
133.357
132.729
132.250
131.570
131.461
130.842
129.160
129.023
128.907
128.812
128.348
127.817
127.698
127.592
127.265
127.078
126.965
126.925
126.709
125.756
125.693
125.627
124.521
CDCl3
O
N
zxr454-016G in CDCl3
35 134 133 132 131 130 129 128 127 126 125
δ (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

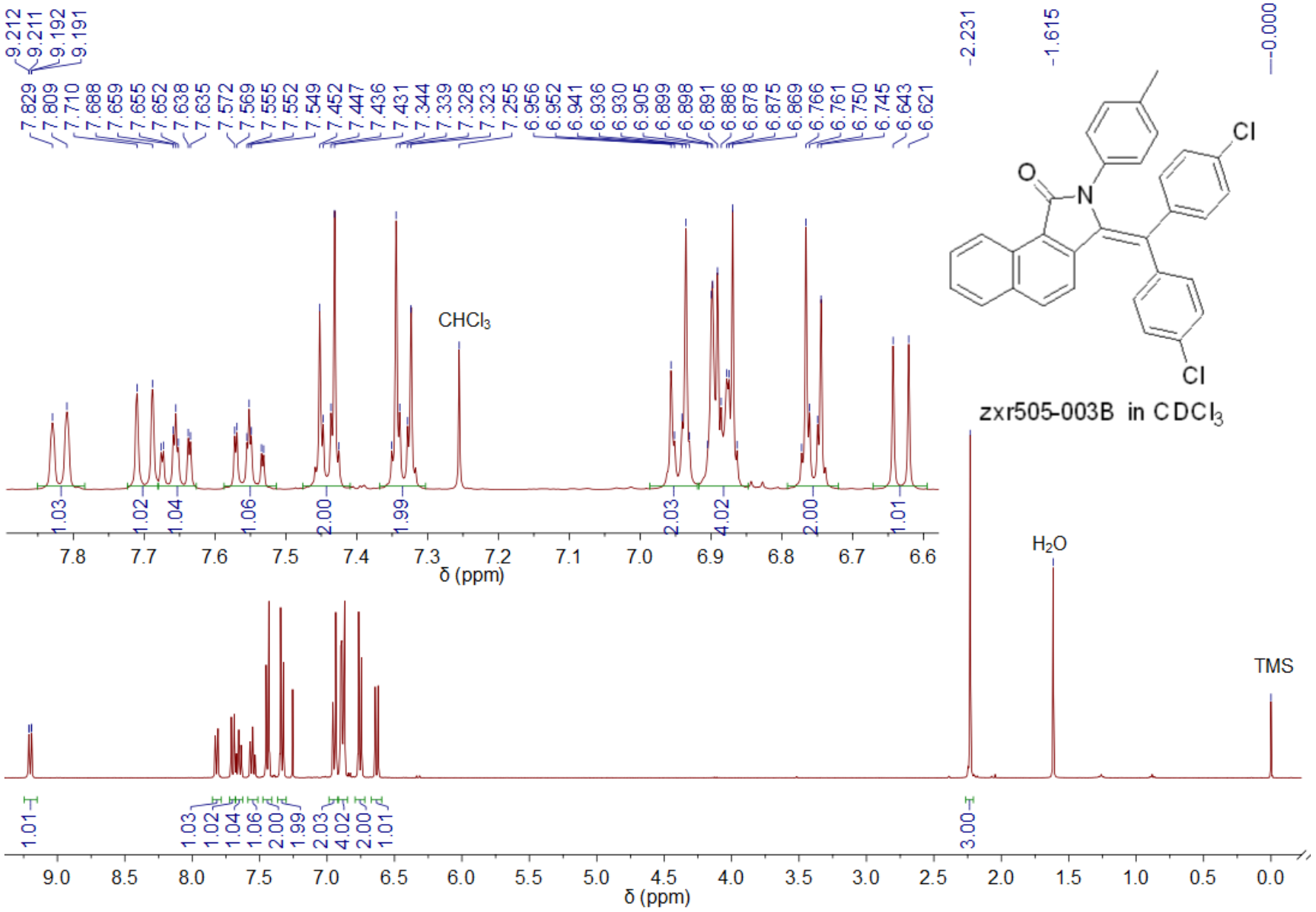
4e – ^{13}C NMR


9.239
9.218
7.809
7.789
7.665
7.643
7.640
7.636
7.536
7.519
7.516
7.333
7.327
7.316
7.311
7.258
7.007
7.002
6.992
6.987
6.976
6.971
6.873
6.852
6.811
6.806
6.795
6.789
6.654
6.632
6.461
6.456
6.444
6.439
3.916
3.677
2.192
1.620
0.000
CHCl3
H2O
TMS
zxr505-196A in CDCl3
δ (ppm)
δ (ppm)

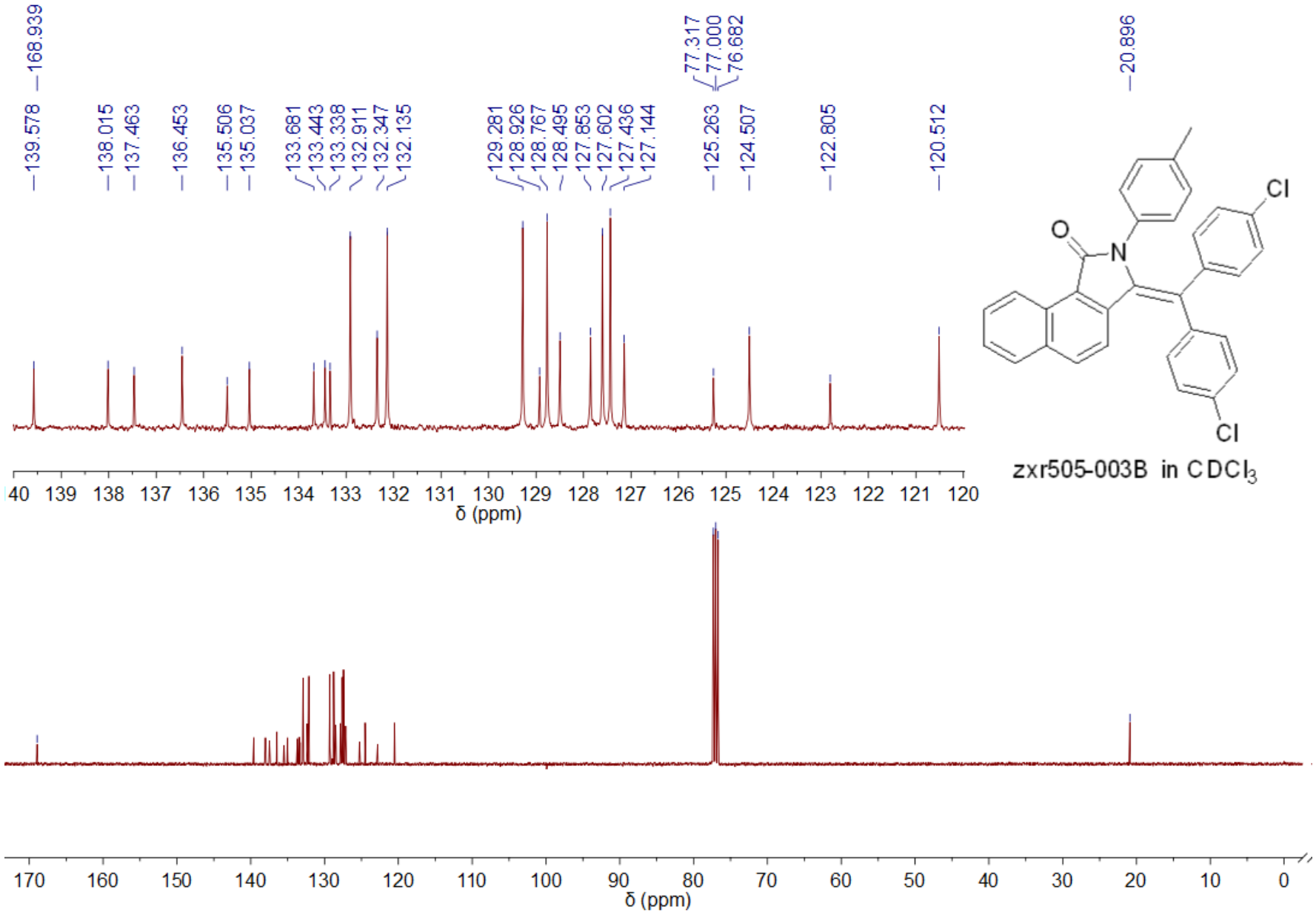
4f - ^{1}H NMR


169.087
160.225
158.910
138.303
124.455
122.240
120.814
114.189
112.707
77.317
77.000
76.683
55.382
55.220
20.885
135.518
134.208
133.966
133.316
133.074
133.055
132.688
132.566
131.858
129.090
128.687
128.456
128.143
127.729
127.433
126.591
CDCl3
O
N
O
O
zxr505-196A in CDCl3
136 135 134 133 132 131 130 129 128 127 126
δ (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

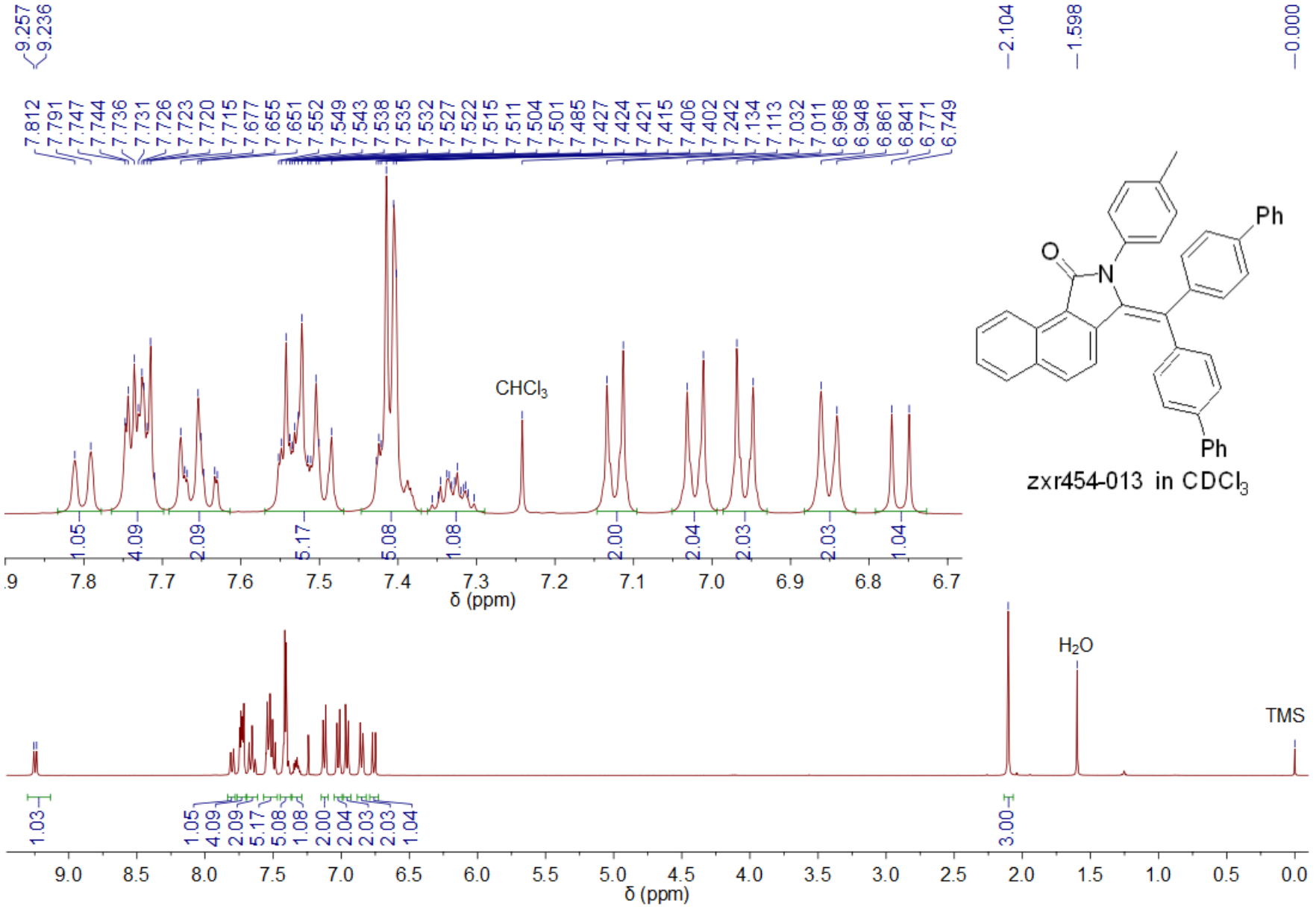
4f – ^{13}C NMR


9.212
9.211
9.192
9.191
7.829
7.809
7.710
7.688
7.659
7.655
7.652
7.638
7.635
7.572
7.569
7.555
7.552
7.549
7.452
7.447
7.436
7.431
7.344
7.339
7.328
7.323
7.255
6.956
6.952
6.941
6.936
6.930
6.905
6.899
6.898
6.891
6.886
6.878
6.875
6.869
6.766
6.761
6.750
6.745
6.643
6.621
-2.231
-1.615
-0.000
CHCl3
H2O
TMS
Cl
Cl
O
N
zxr505-003B in CDCl3
δ (ppm)
δ (ppm)

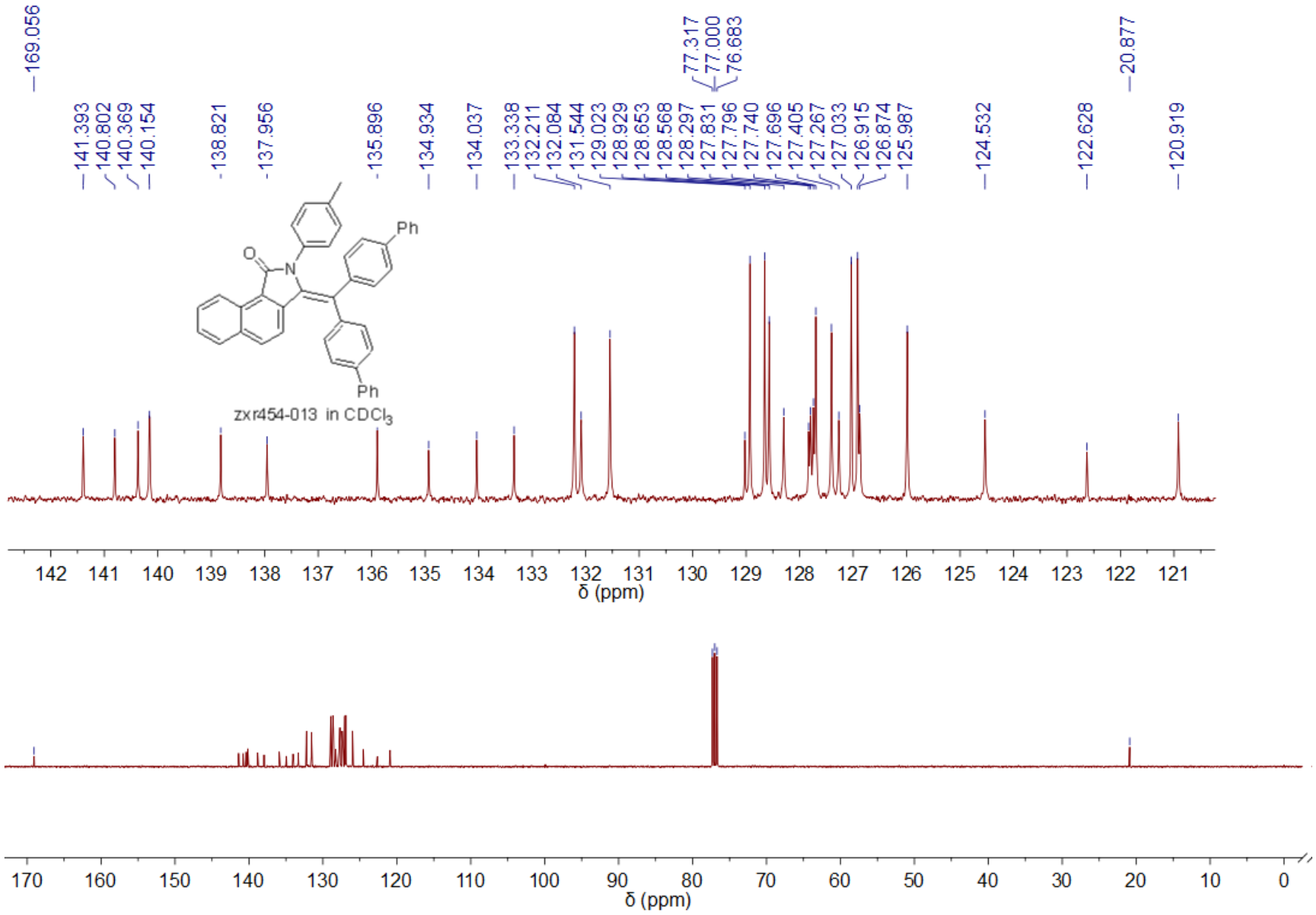
4g - ^{1}H NMR


168.939
139.578
138.015
137.463
136.453
135.506
135.037
133.681
133.443
133.338
132.911
132.347
132.135
129.281
128.926
128.767
128.495
127.853
127.602
127.436
127.144
77.317
77.000
76.682
125.263
124.507
122.805
120.512
20.896
O
N
Cl
Cl
zxr505-003B in CDCl3
δ (ppm)
δ (ppm)

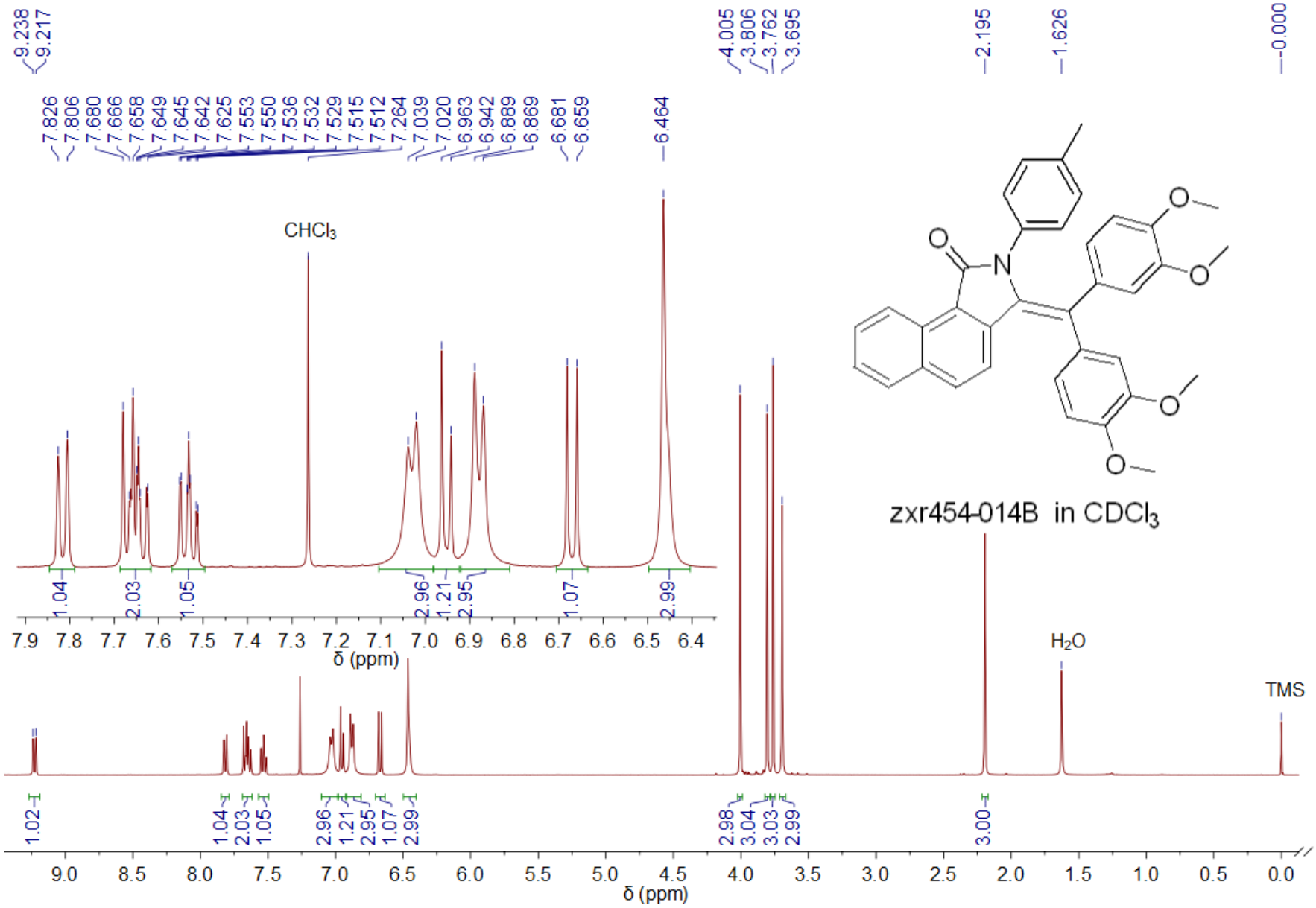
4g – ^{13}C NMR


9.257
9.236
2.104
1.598
0.000
7.812
7.791
7.747
7.744
7.736
7.731
7.726
7.723
7.720
7.715
7.677
7.655
7.651
7.552
7.549
7.543
7.538
7.535
7.532
7.527
7.522
7.515
7.511
7.504
7.501
7.485
7.427
7.424
7.421
7.415
7.406
7.402
7.242
7.134
7.113
7.032
7.011
6.968
6.948
6.861
6.841
6.771
6.749
CHCl3
Ph
O
N
Ph
zxr454-013 in CDCl3
1.05
4.09
2.09
5.17
5.08
1.08
2.00
2.04
2.03
2.03
1.04
δ (ppm)
H2O
TMS
1.03
3.00
δ (ppm)

4h - ^{1}H NMR


169.056
141.393
140.802
140.369
140.154
138.821
137.956
135.896
134.934
134.037
133.338
132.211
132.084
131.544
129.023
128.929
128.653
128.568
128.297
127.831
127.796
127.740
127.696
127.405
127.267
127.033
126.915
126.874
125.987
124.532
122.628
77.317
77.000
76.683
20.877
120.919
Ph
Ph
O
N
zxr454-013 in CDCl3
δ (ppm)
δ (ppm)

4h – ^{13}C NMR


9.238
9.217
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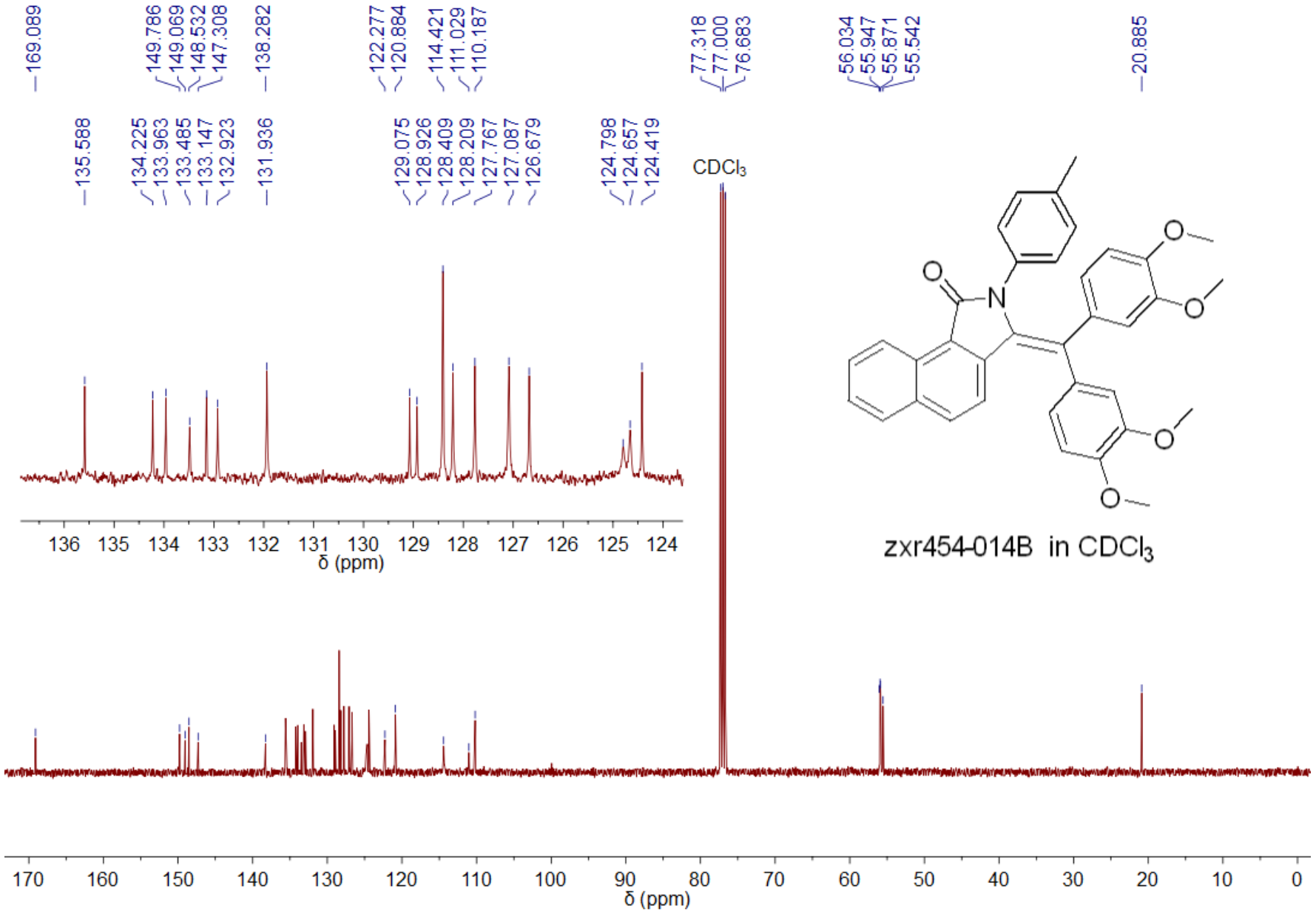
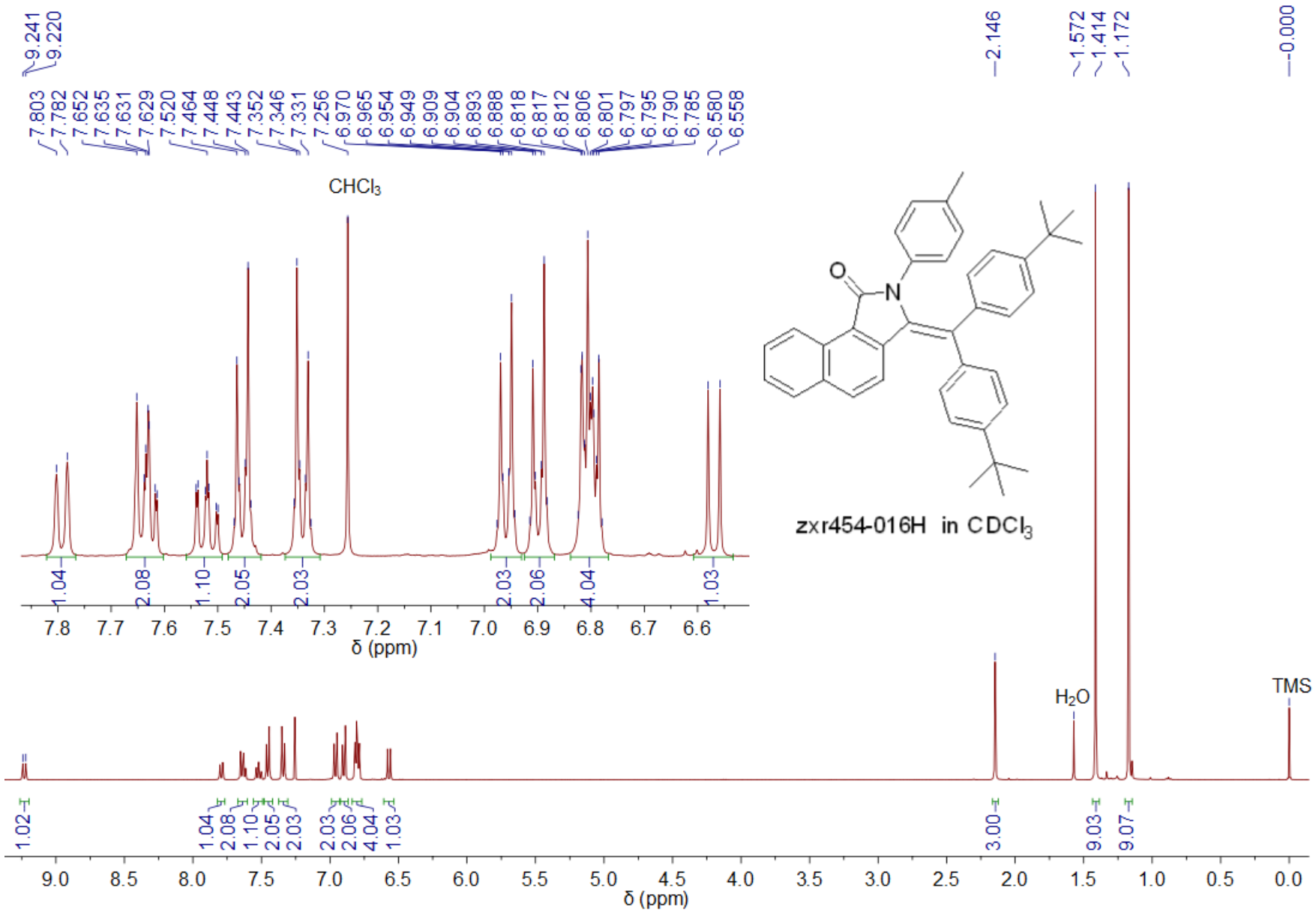
7.806
7.680
7.666
7.658
7.649
7.645
7.642
7.625
7.553
7.550
7.536
7.532
7.529
7.515
7.512
7.264
7.039
7.020
6.963
6.942
6.889
6.869
6.681
6.659
6.464
4.005
3.806
3.762
3.695
2.195
1.626
0.000
CHCl3
zxr454-014B in CDCl3
H2O
TMS
δ (ppm)
δ (ppm)

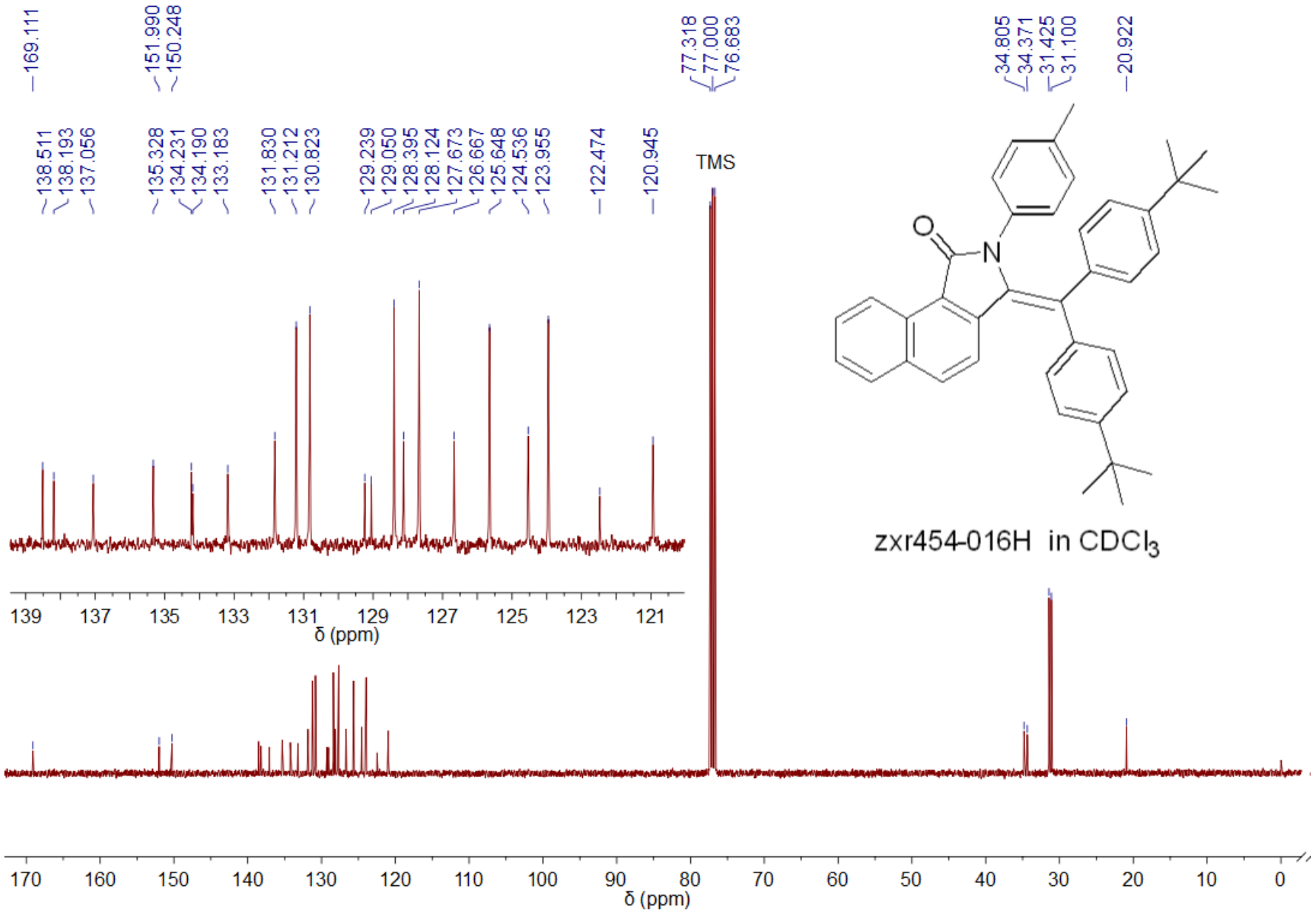
4i - ^{1}H NMR


169.089
149.786
149.069
148.532
147.308
138.282
122.277
120.884
114.421
111.029
110.187
77.318
77.000
76.683
56.034
55.947
55.871
55.542
20.885
135.588
134.225
133.963
133.485
133.147
132.923
131.936
129.075
128.926
128.409
128.209
127.767
127.087
126.679
124.798
124.657
124.419
CDCl3
136 135 134 133 132 131 130 129 128 127 126 125 124
δ (ppm)
zxr454-014B in CDCl3
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

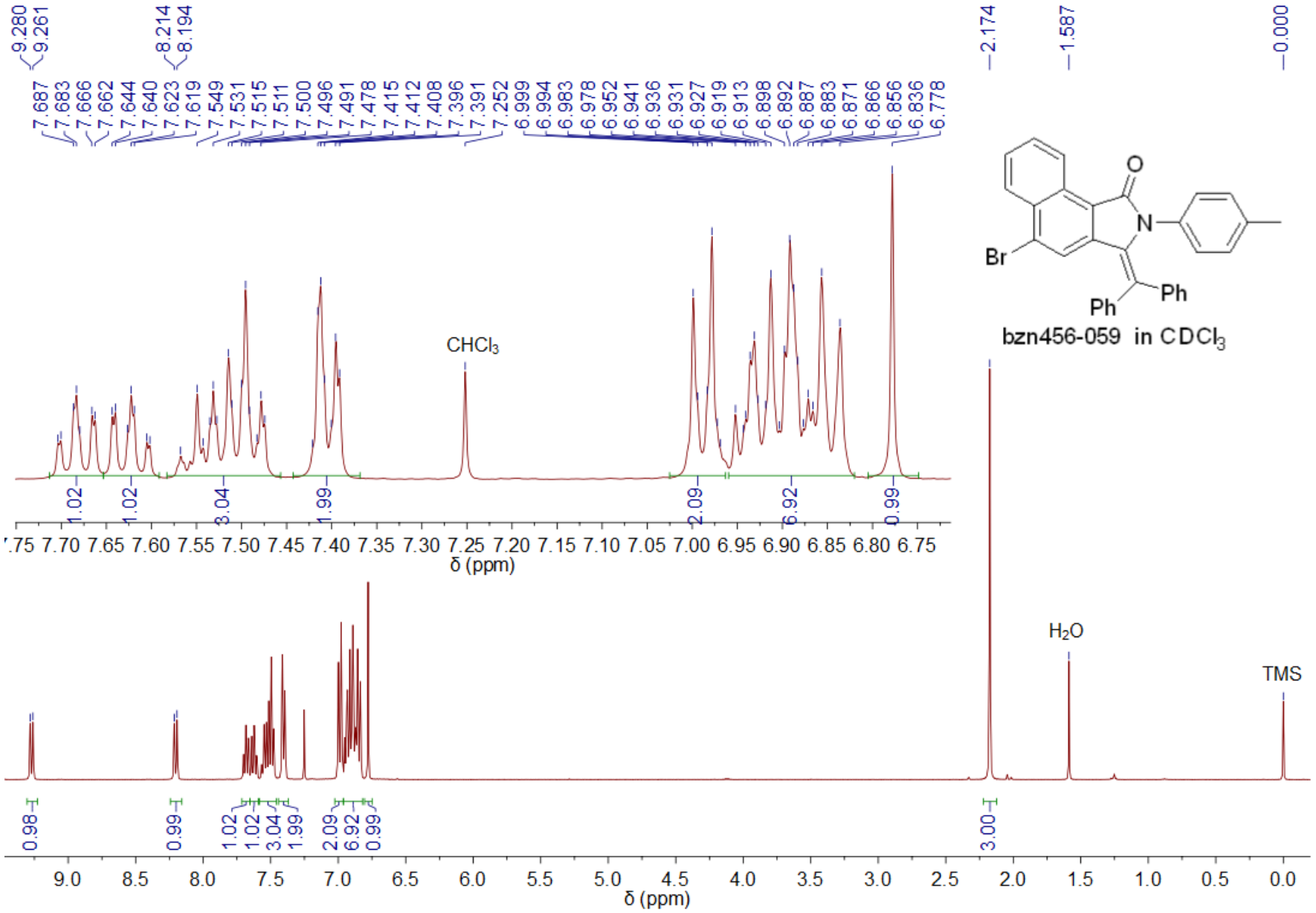
4i – ^{13}C NMR


9.241
9.220
7.803
7.782
7.652
7.635
7.631
7.629
7.520
7.464
7.448
7.443
7.352
7.346
7.331
7.256
6.970
6.965
6.954
6.949
6.909
6.904
6.893
6.888
6.818
6.817
6.812
6.806
6.801
6.797
6.795
6.790
6.785
6.580
6.558
2.146
1.572
1.414
1.172
0.000
CHCl3
O
N
zxr454-016H in CDCl3
1.04
2.08
1.10
2.05
2.03
2.03
2.06
4.04
1.03
7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6
δ (ppm)
H2O
TMS
1.02
1.04
2.08
1.10
2.05
2.03
2.03
2.06
4.04
1.03
3.00
9.03
9.07
9.0 8.5 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 0.0
δ (ppm)

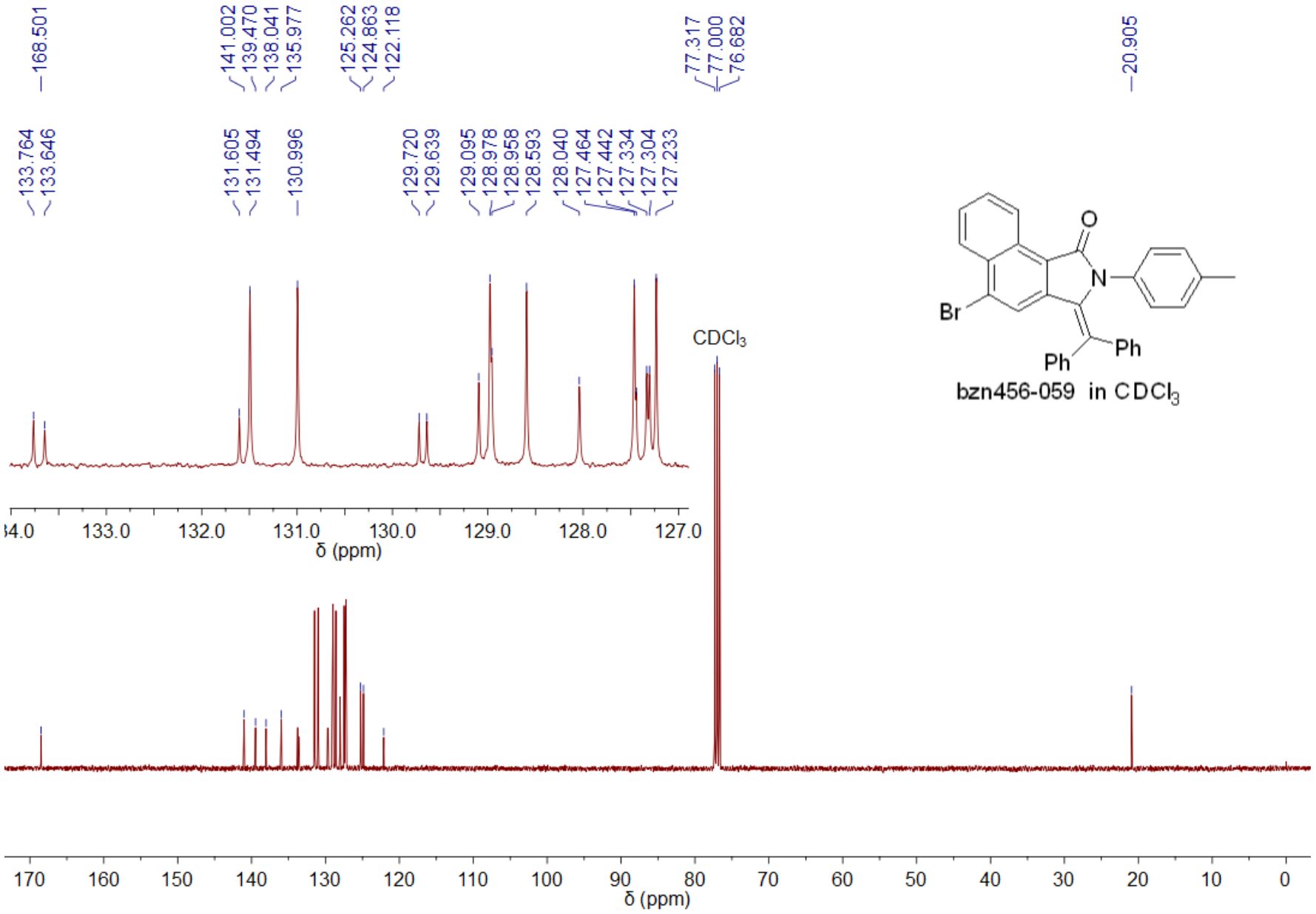
4j - ^{1}H NMR


169.111
151.990
150.248
77.318
77.000
76.683
34.805
34.371
31.425
31.100
20.922
138.511
138.193
137.056
135.328
134.231
134.190
133.183
131.830
131.212
130.823
129.239
129.050
128.395
128.124
127.673
126.667
125.648
124.536
123.955
122.474
120.945
TMS
O
N
zxr454-016H in CDCl3
δ (ppm)
δ (ppm)

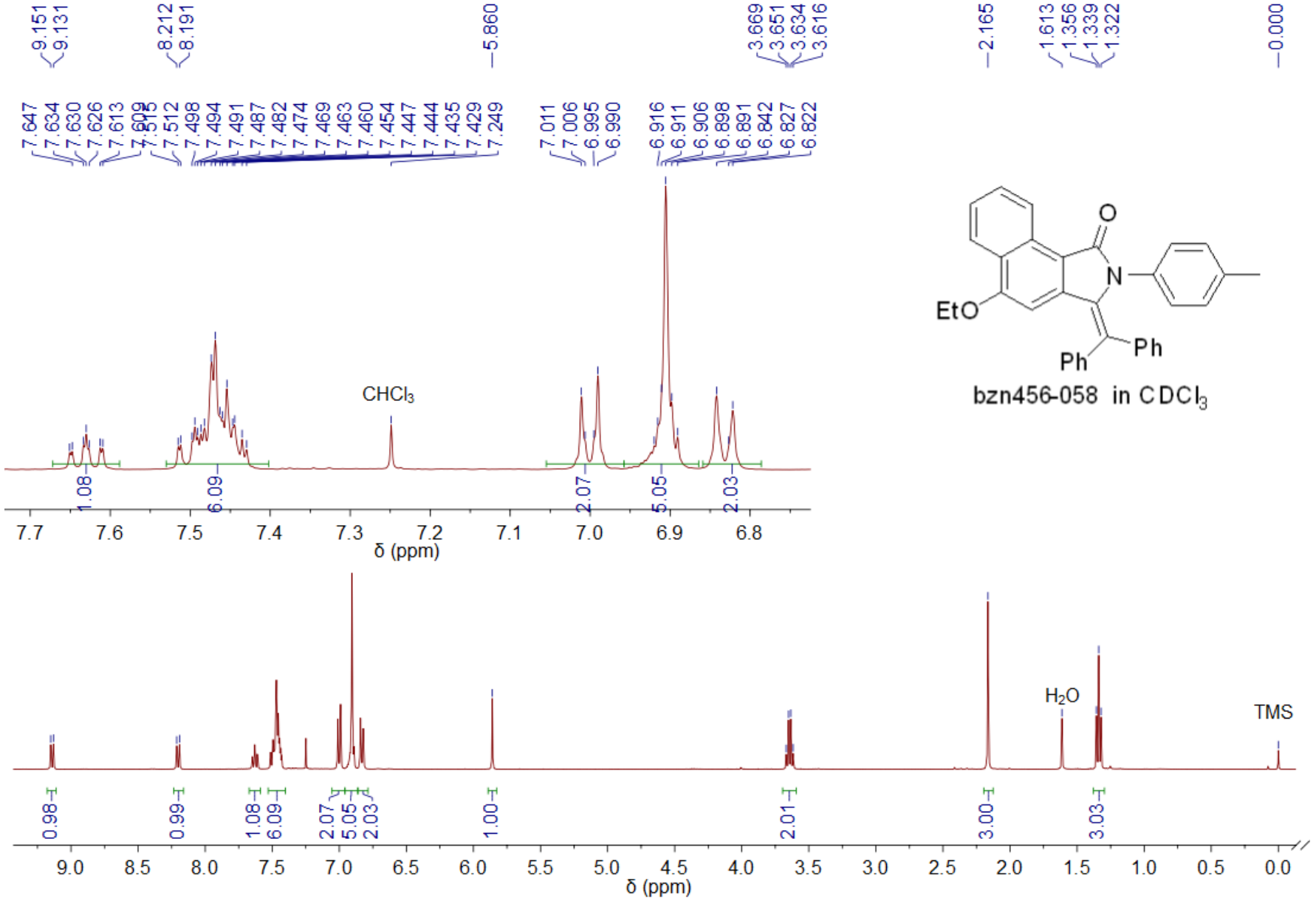
4j – ^{13}C NMR


9.280
9.261
8.214
8.194
7.687
7.683
7.666
7.662
7.644
7.640
7.623
7.619
7.549
7.531
7.515
7.511
7.500
7.496
7.491
7.478
7.415
7.412
7.408
7.396
7.391
7.252
6.999
6.994
6.983
6.978
6.952
6.941
6.936
6.931
6.927
6.919
6.913
6.898
6.892
6.887
6.883
6.871
6.866
6.856
6.836
6.778
2.174
1.587
0.000
CHCl3
H2O
TMS
Br
O
N
Ph
Ph
bzn456-059 in CDCl3
δ (ppm)
δ (ppm)

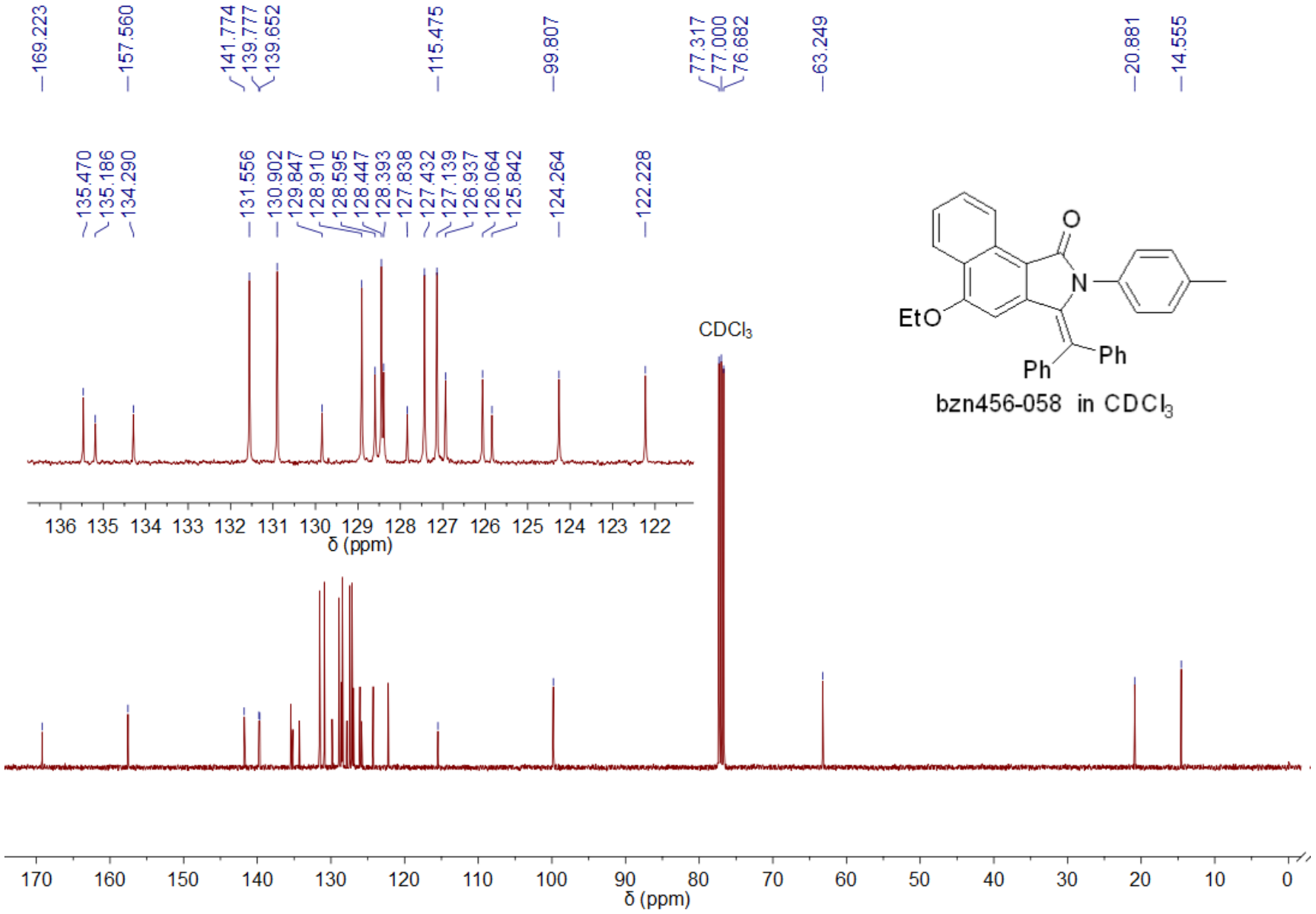
4k - ^{1}H NMR


168.501
141.002
139.470
138.041
135.977
125.262
124.863
122.118
77.317
77.000
76.682
20.905
133.764
133.646
131.605
131.494
130.996
129.720
129.639
129.095
128.978
128.958
128.593
128.040
127.464
127.442
127.334
127.304
127.233
CDCl3
O
N
Br
Ph
Ph
bzn456-059 in CDCl3
δ (ppm)
δ (ppm)

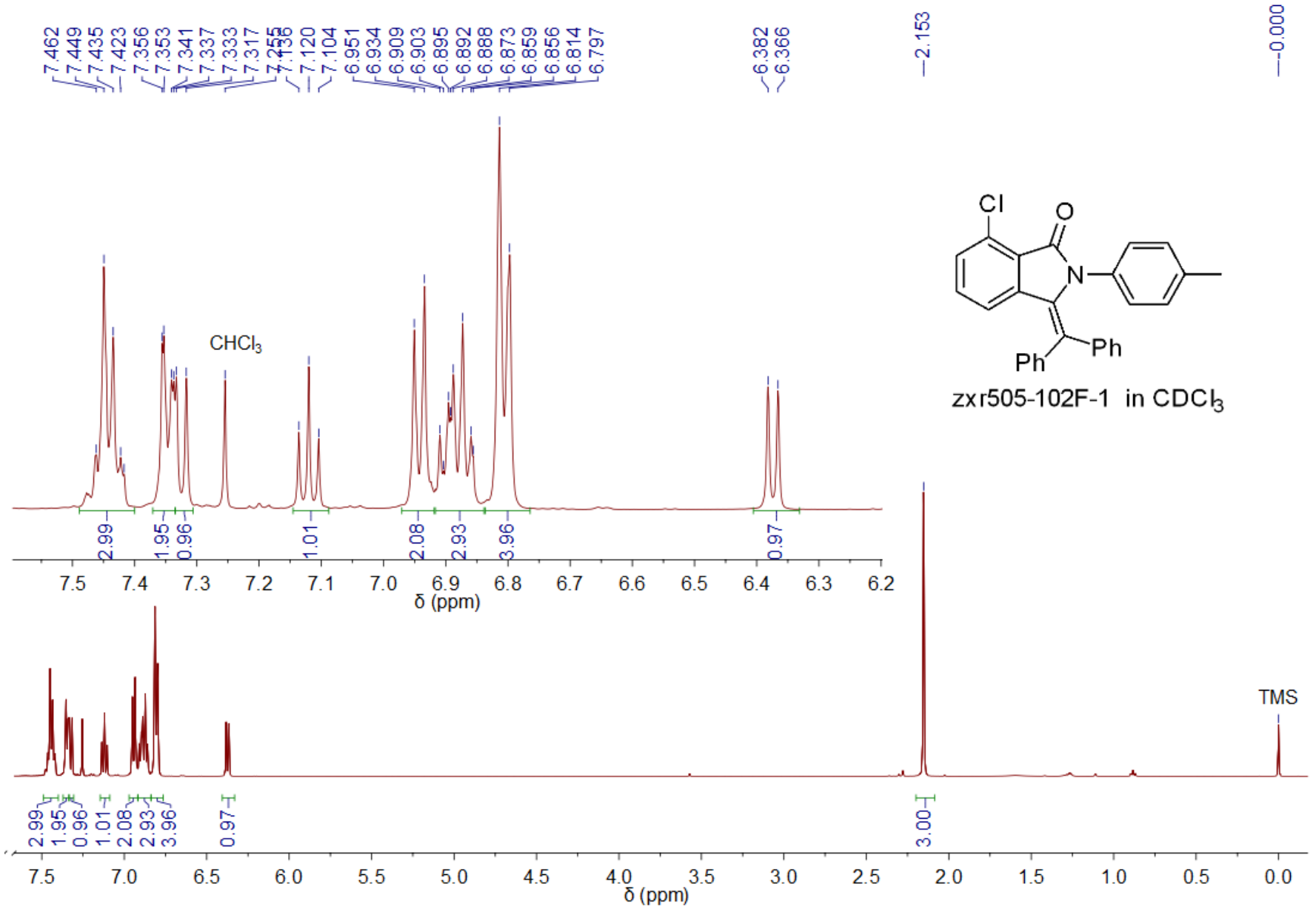
4k – ^{13}C NMR


9.151
9.131
8.212
8.191
5.860
3.669
3.651
3.634
3.616
2.165
1.613
1.356
1.339
1.322
0.000
7.647
7.634
7.630
7.626
7.613
7.512
7.498
7.494
7.491
7.487
7.482
7.474
7.469
7.463
7.460
7.454
7.447
7.444
7.435
7.429
7.249
7.011
7.006
6.995
6.990
6.916
6.911
6.906
6.898
6.891
6.842
6.827
6.822
CHCl3
1.08
6.09
2.07
5.05
2.03
δ (ppm)
EtO
O
N
Ph
Ph
bzn456-058 in CDCl3
H2O
TMS
0.98
0.99
1.08
6.09
2.07
5.05
2.03
1.00
2.01
3.00
3.03
δ (ppm)

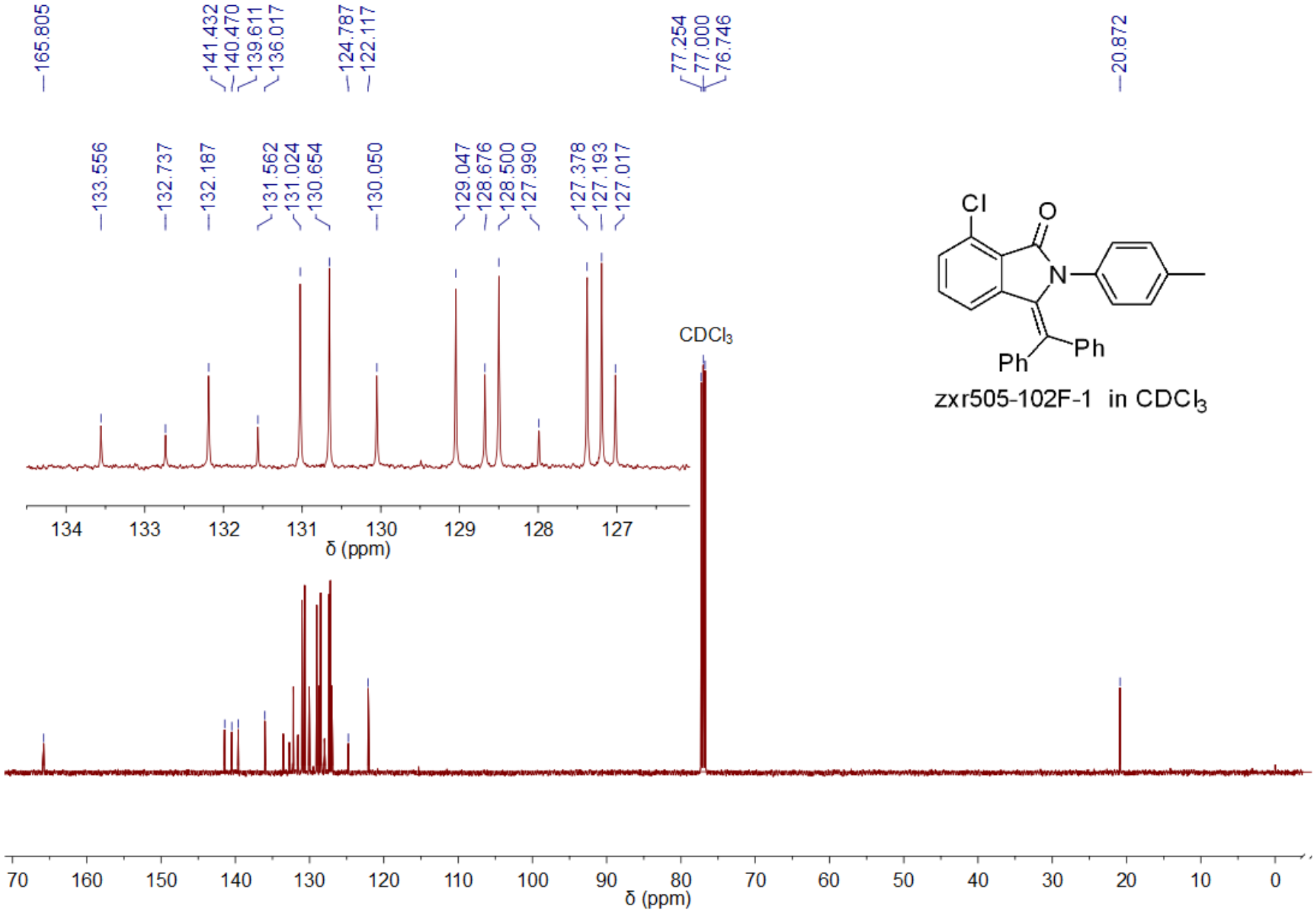
4l - ^{1}H NMR


169.223
157.560
141.774
139.777
139.652
115.475
99.807
77.317
77.000
76.682
63.249
20.881
14.555
135.470
135.186
134.290
131.556
130.902
129.847
128.910
128.595
128.447
128.393
127.838
127.432
127.139
126.937
126.064
125.842
124.264
122.228
CDCl3
EtO
O
N
Ph
Ph
bzn456-058 in CDCl3
136 135 134 133 132 131 130 129 128 127 126 125 124 123 122
δ (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

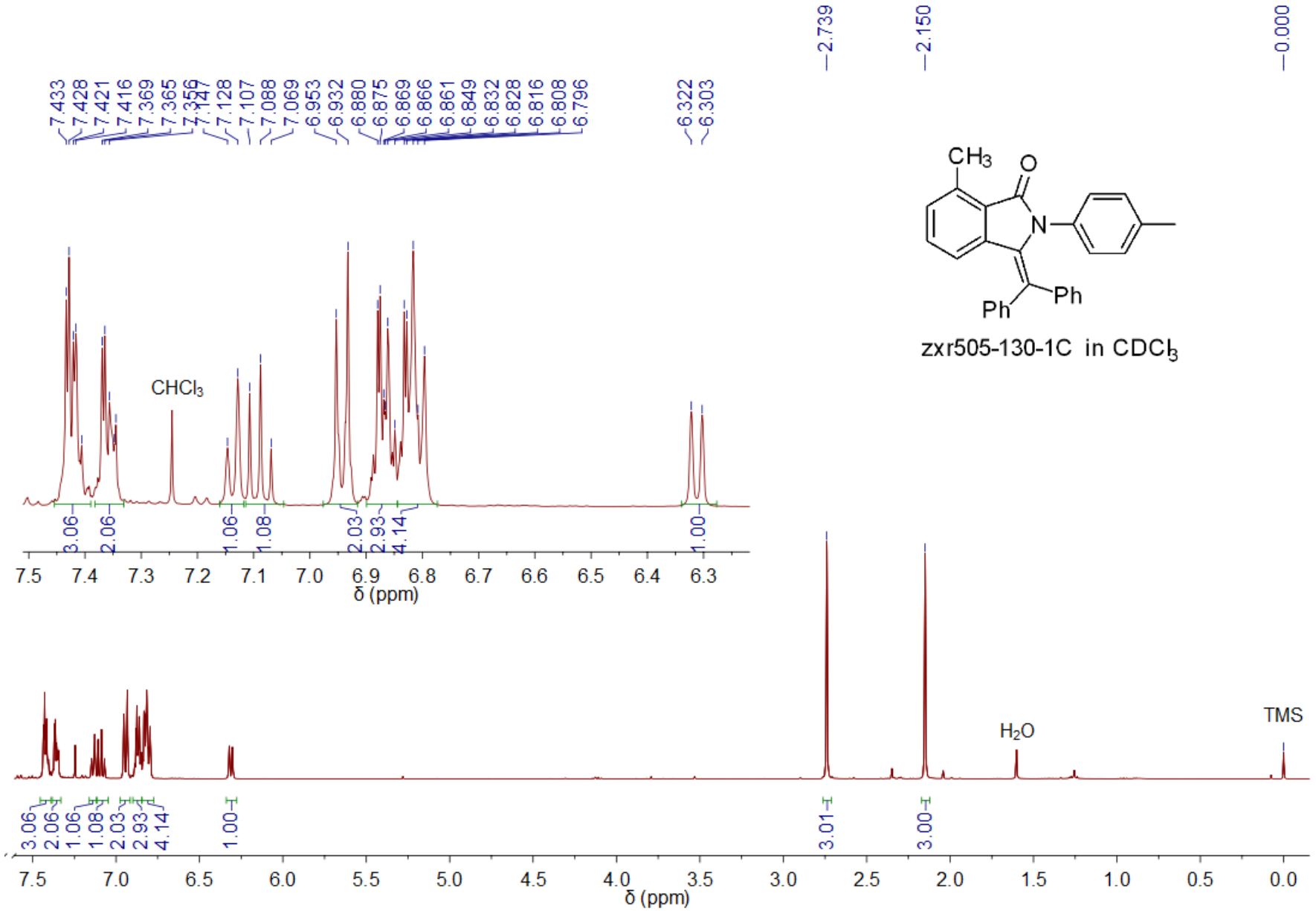
4l – ^{13}C NMR


7.462
7.449
7.435
7.423
7.356
7.353
7.341
7.337
7.333
7.317
7.255
7.138
7.120
7.104
6.951
6.934
6.909
6.903
6.895
6.892
6.888
6.873
6.859
6.856
6.814
6.797
6.382
6.366
2.153
0.000
CHCl3
Cl
O
N
Ph
Ph
zxr505-102F-1 in CDCl3
2.99
1.95
0.96
1.01
2.08
2.93
3.96
0.97
3.00
TMS
δ (ppm)

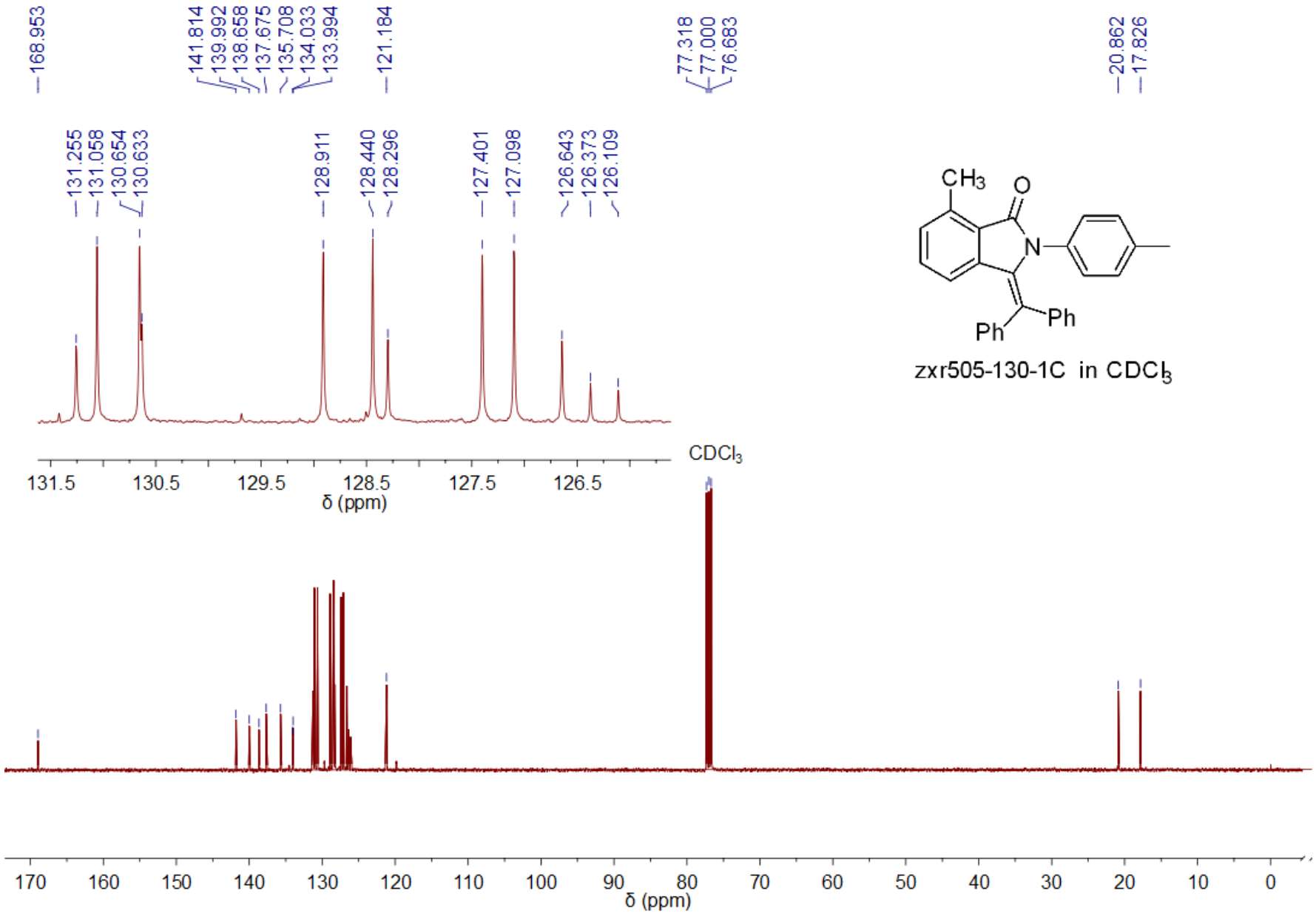
4m - ^{1}H NMR


165.805
141.432
140.470
139.611
136.017
124.787
122.117
77.254
77.000
76.746
20.872
133.556
132.737
132.187
131.562
131.024
130.654
130.050
129.047
128.676
128.500
127.990
127.378
127.193
127.017
CDCl3
Cl
O
N
Ph
Ph
zxr505-102F-1 in CDCl3
134
133
132
131
130
129
128
127
δ (ppm)
70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

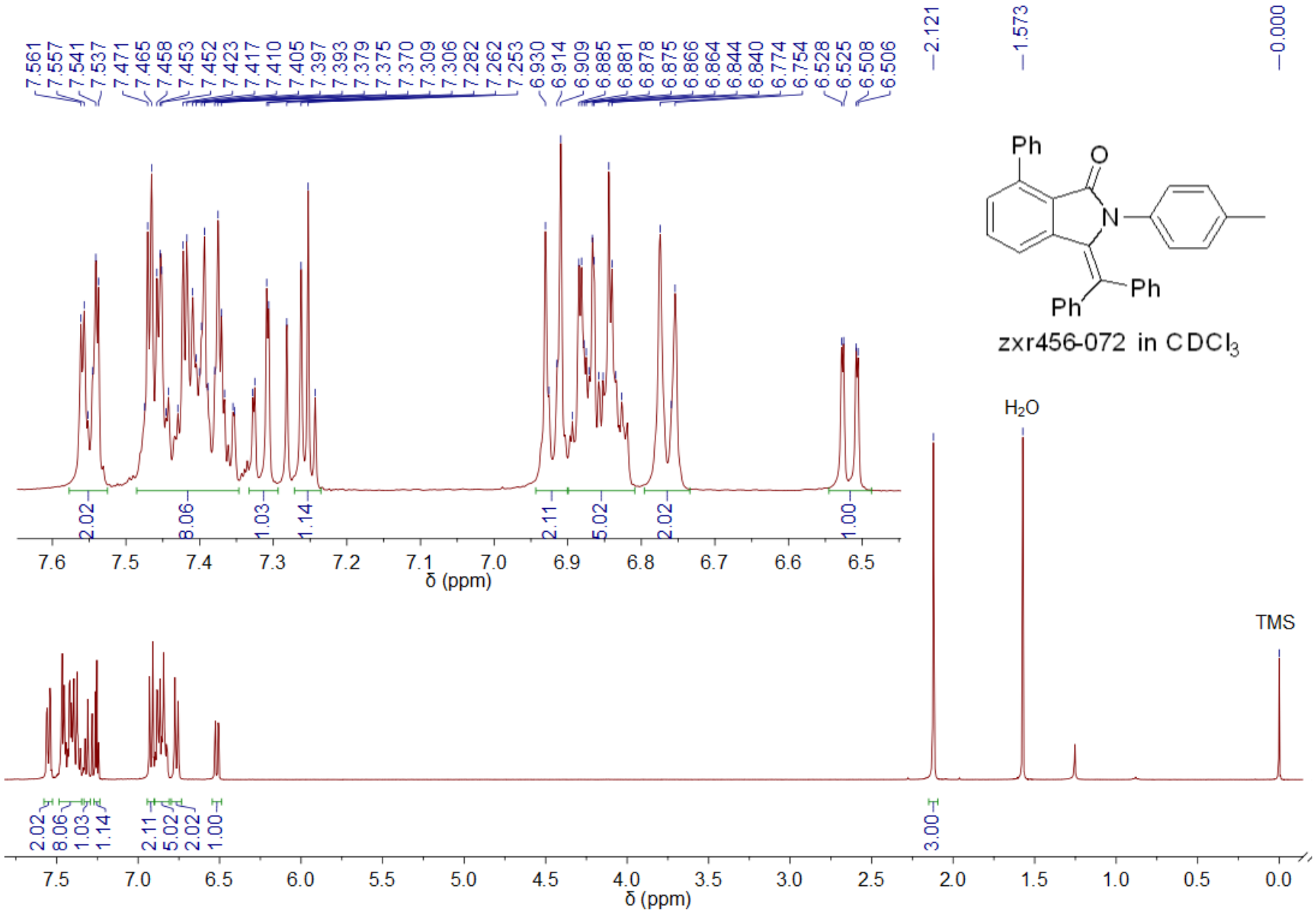
4m – ^{13}C NMR


7.433
7.428
7.421
7.416
7.369
7.365
7.356
7.147
7.128
7.107
7.088
7.069
6.953
6.932
6.880
6.875
6.869
6.866
6.861
6.849
6.832
6.828
6.816
6.808
6.796
6.322
6.303
2.739
2.150
0.000
CH3
O
N
Ph
Ph
zxr505-130-1C in CDCl3
CHCl3
3.06
2.06
1.06
1.08
2.03
2.93
4.14
1.00
δ (ppm)
H2O
TMS
3.01
3.00

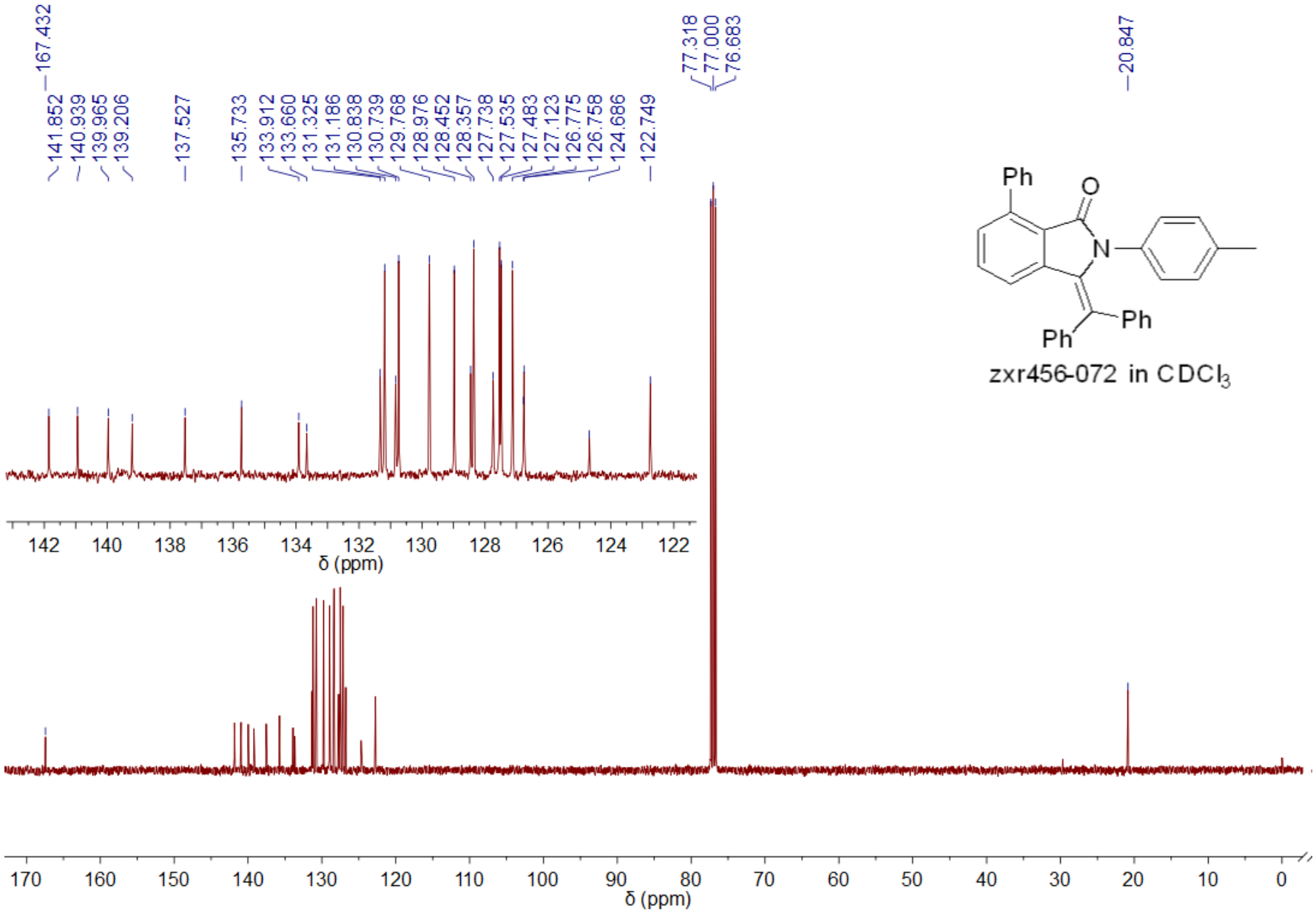
4n - ^{1}H NMR


168.953
141.814
139.992
138.658
137.675
135.708
134.033
133.994
121.184
77.318
77.000
76.683
20.862
17.826
131.255
131.058
130.654
130.633
128.911
128.440
128.296
127.401
127.098
126.643
126.373
126.109
CH3
O
N
Ph
Ph
zxr505-130-1C in CDCl3
CDCl3
131.5
130.5
129.5
128.5
127.5
126.5
δ (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

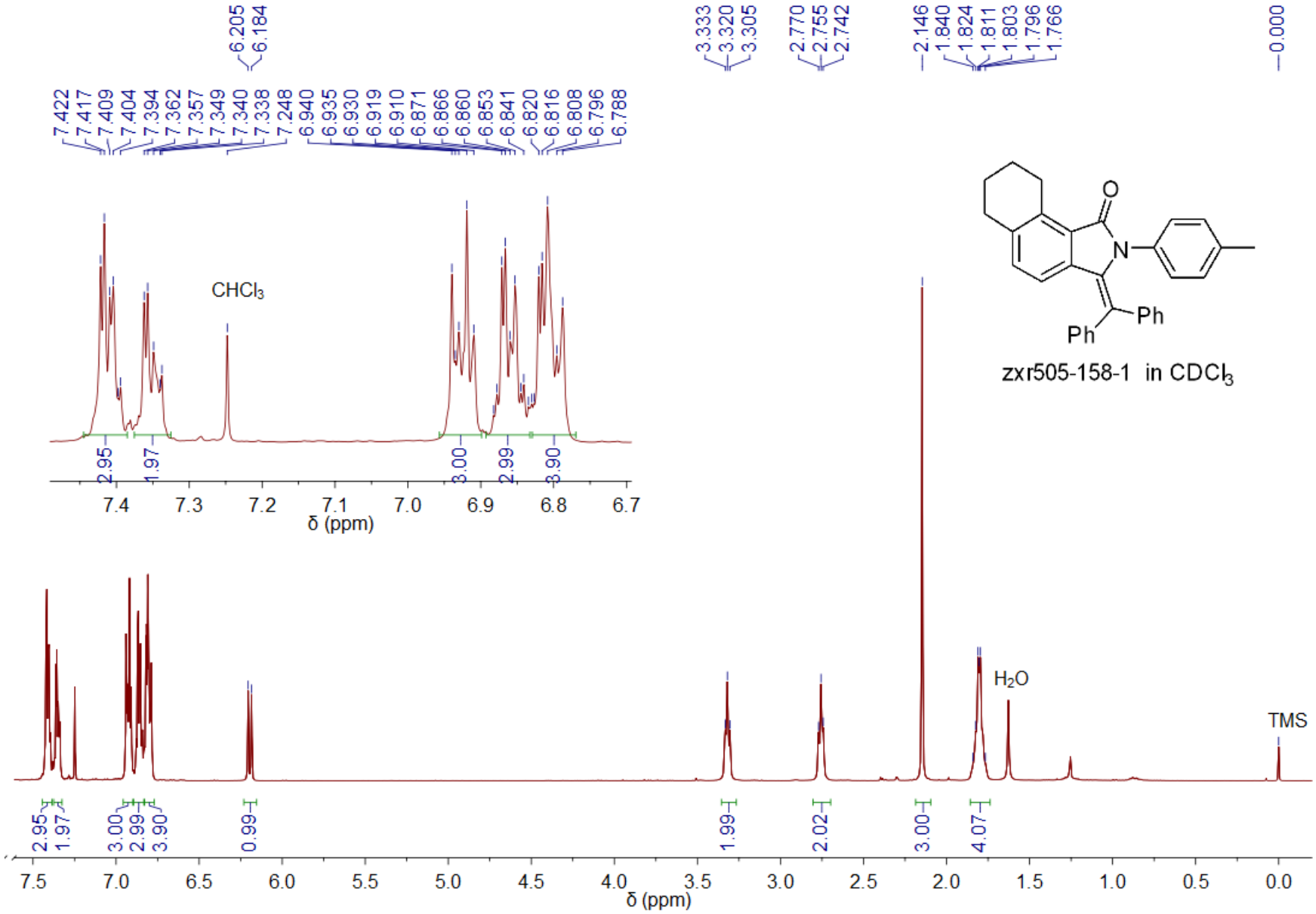
4n – ^{13}C NMR


7.561
7.557
7.541
7.537
7.471
7.465
7.458
7.453
7.452
7.423
7.417
7.410
7.405
7.397
7.393
7.379
7.375
7.370
7.309
7.306
7.282
7.262
7.253
6.930
6.914
6.909
6.885
6.881
6.878
6.875
6.866
6.864
6.844
6.840
6.774
6.754
6.528
6.525
6.508
6.506
2.121
1.573
0.000
Ph
O
N
Ph
Ph
zxr456-072 in CDCl3
H2O
TMS
2.02
8.06
1.03
1.14
2.11
5.02
2.02
1.00
3.00
δ (ppm)

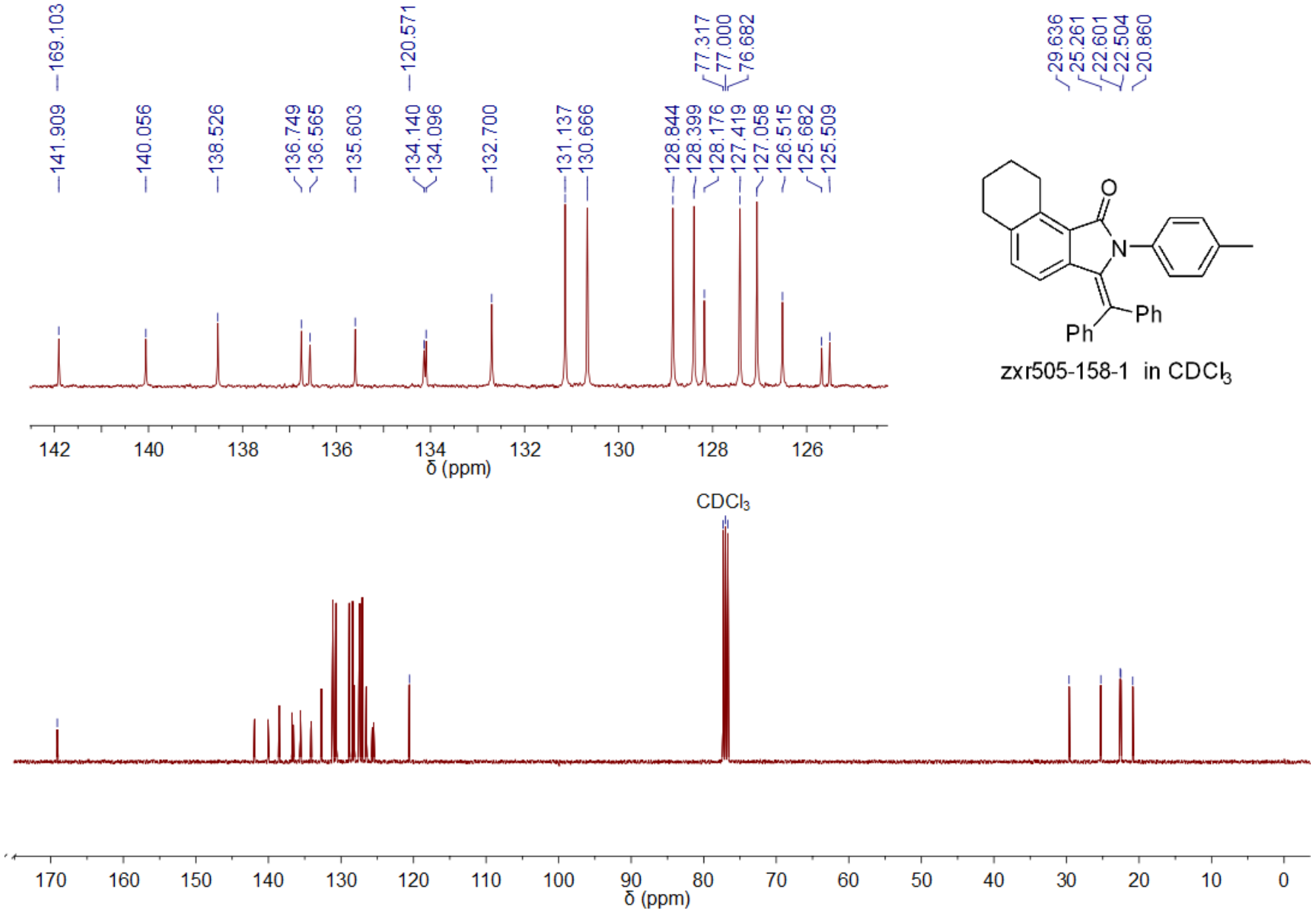
4o - ^{1}H NMR


167.432
141.852
140.939
139.965
139.206
137.527
135.733
133.912
133.660
131.325
131.186
130.838
130.739
129.768
128.976
128.452
128.357
127.738
127.535
127.483
127.123
126.775
126.758
124.686
122.749
77.318
77.000
76.683
20.847
Ph
O
N
Ph
Ph
zxr456-072 in CDCl3
142 140 138 136 134 132 130 128 126 124 122
δ (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

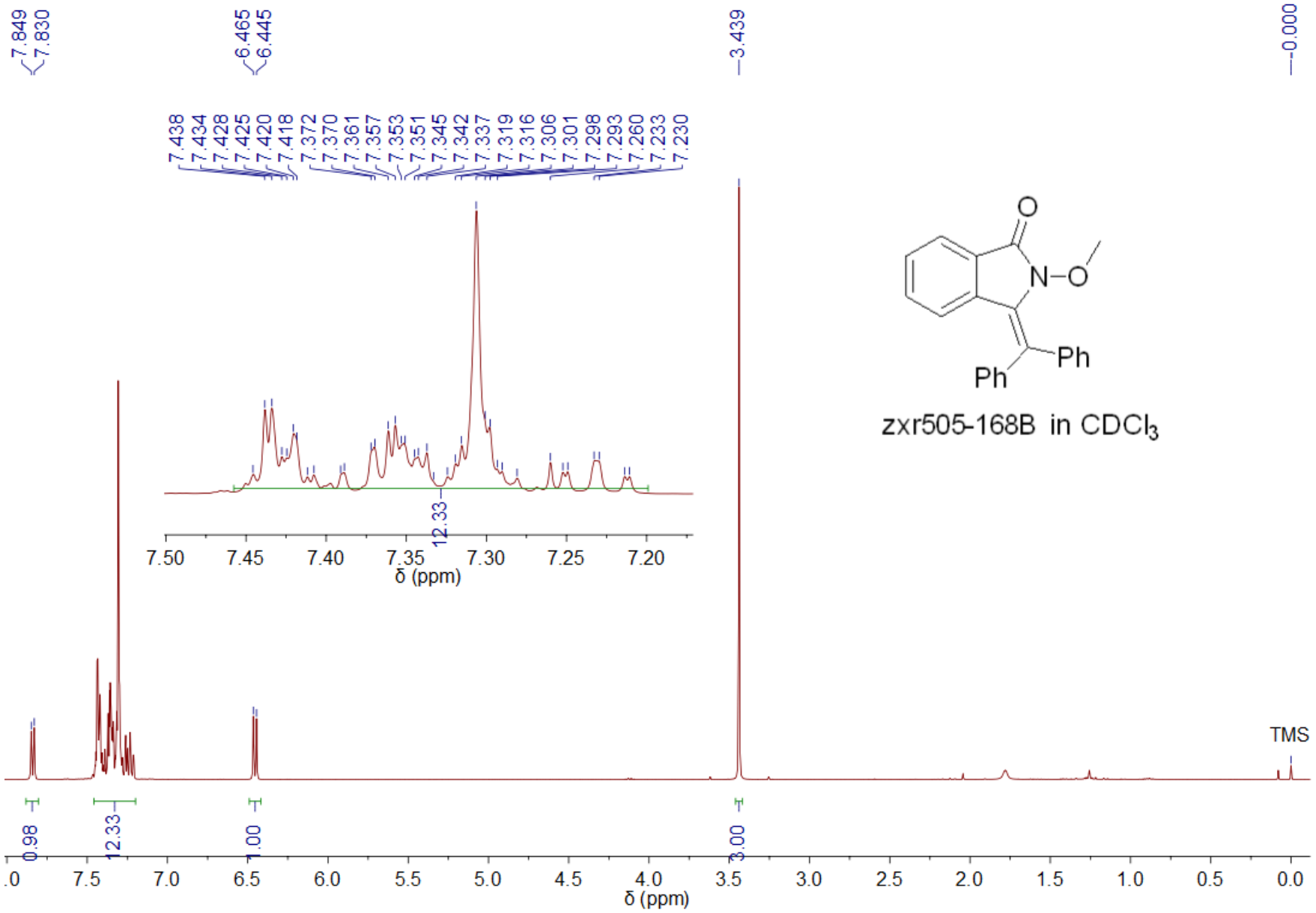
4o – ^{13}C NMR


6.205
6.184
3.333
3.320
3.305
2.770
2.755
2.742
2.146
1.840
1.824
1.811
1.803
1.796
1.766
0.000
7.422
7.417
7.409
7.404
7.394
7.362
7.357
7.349
7.340
7.338
7.248
6.940
6.935
6.930
6.919
6.910
6.871
6.866
6.860
6.853
6.841
6.820
6.816
6.808
6.796
6.788
CHCl3
2.95
1.97
3.00
2.99
3.90
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
δ (ppm)
O
N
Ph
Ph
zxr505-158-1 in CDCl3
H2O
TMS
2.95
1.97
3.00
2.99
3.90
0.99
1.99
2.02
3.00
4.07
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
0.0
δ (ppm)

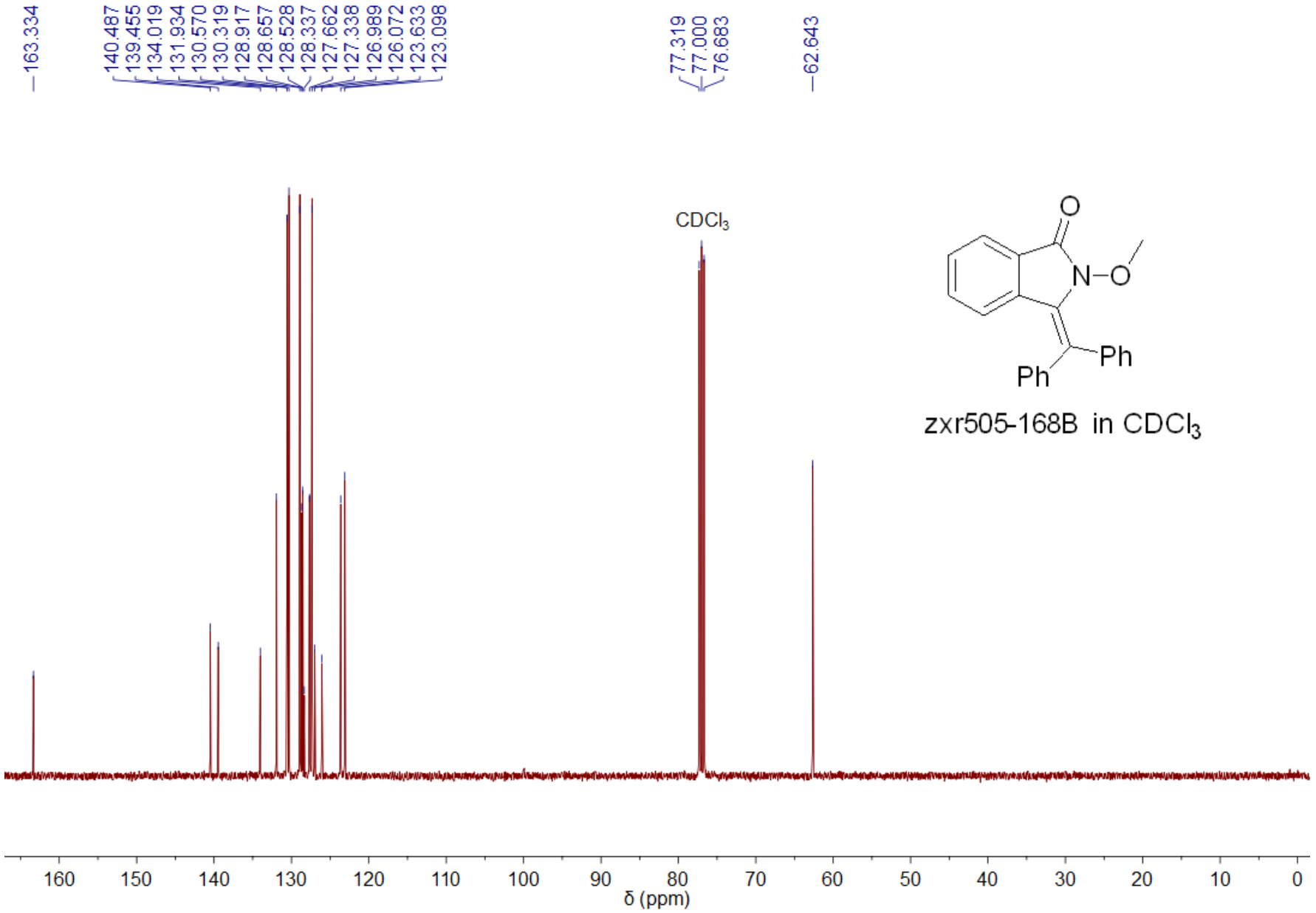
4p - ^{1}H NMR


169.103
141.909
140.056
138.526
136.749
136.565
135.603
120.571
134.140
134.096
132.700
131.137
130.666
128.844
128.399
128.176
127.419
127.058
126.515
125.682
125.509
77.317
77.000
76.682
29.636
25.261
22.601
22.504
20.860
O
N
Ph
Ph
zxr505-158-1 in CDCl3
δ (ppm)
CDCl3
δ (ppm)

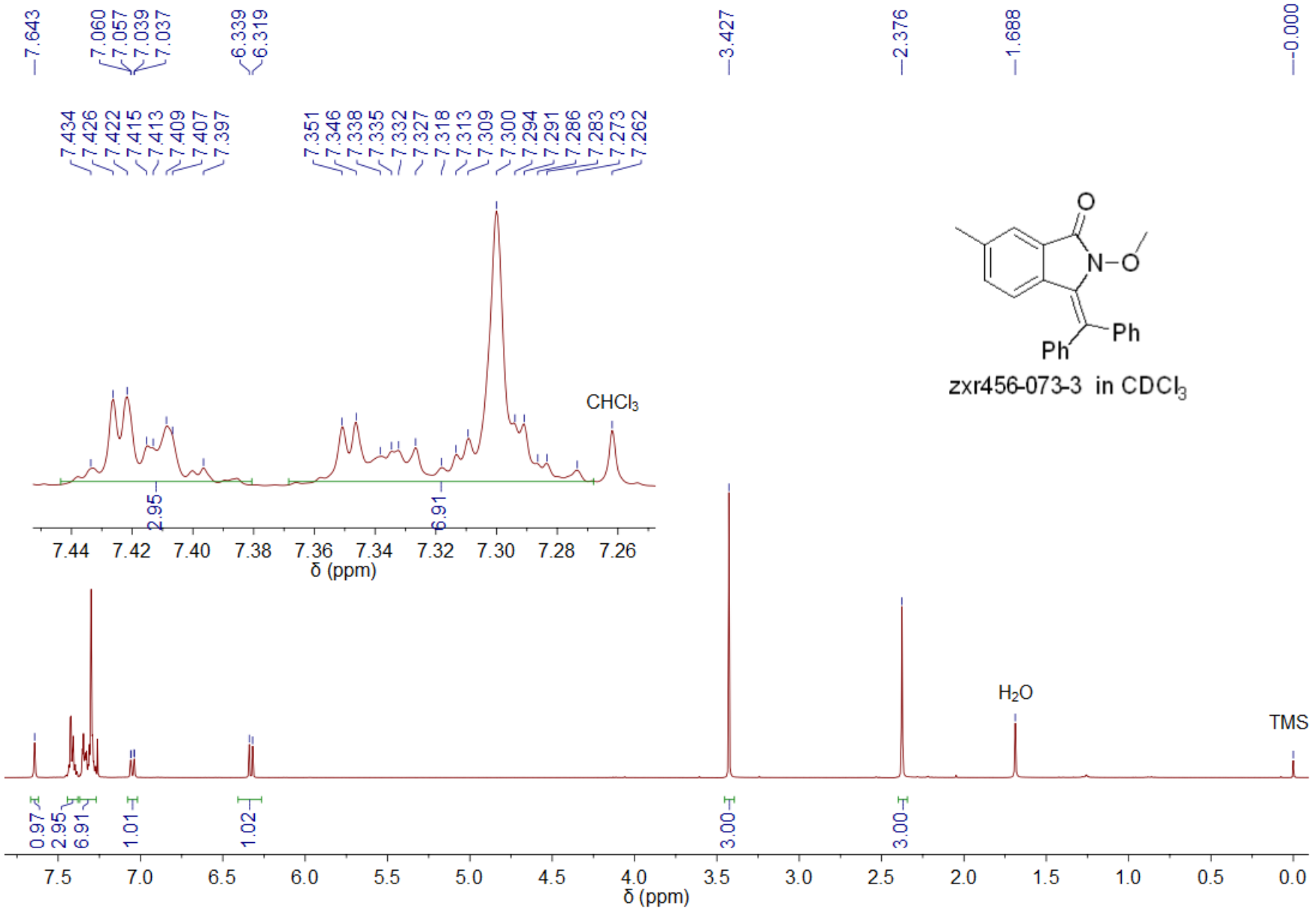
4p – ^{13}C NMR


7.849
7.830
6.465
6.445
3.439
0.000
7.438
7.434
7.428
7.425
7.420
7.418
7.372
7.370
7.361
7.357
7.353
7.351
7.345
7.342
7.337
7.319
7.316
7.306
7.301
7.298
7.293
7.260
7.233
7.230
12.33
7.50
7.45
7.40
7.35
7.30
7.25
7.20
δ (ppm)
O
N–O
Ph
Ph
zxr505-168B in CDCl3
TMS
0.98
12.33
1.00
3.00
.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
0.0
δ (ppm)

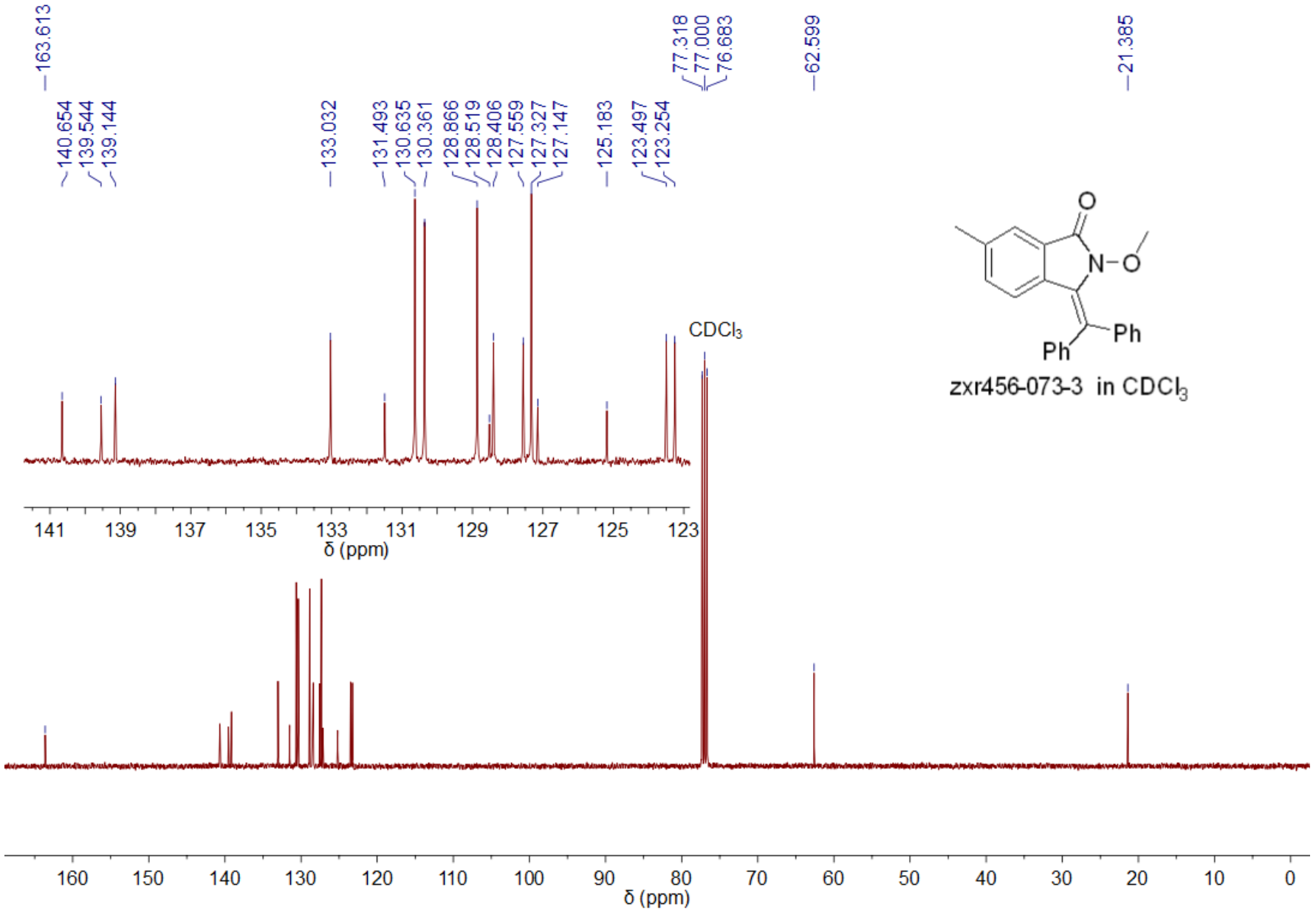
5a –^{1}H NMR


163.334
140.487
139.455
134.019
131.934
130.570
130.319
128.917
128.657
128.528
128.337
127.662
127.338
126.989
126.072
123.633
123.098
77.319
77.000
76.683
62.643
CDCl3
O
N–O
Ph
Ph
zxr505-168B in CDCl3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

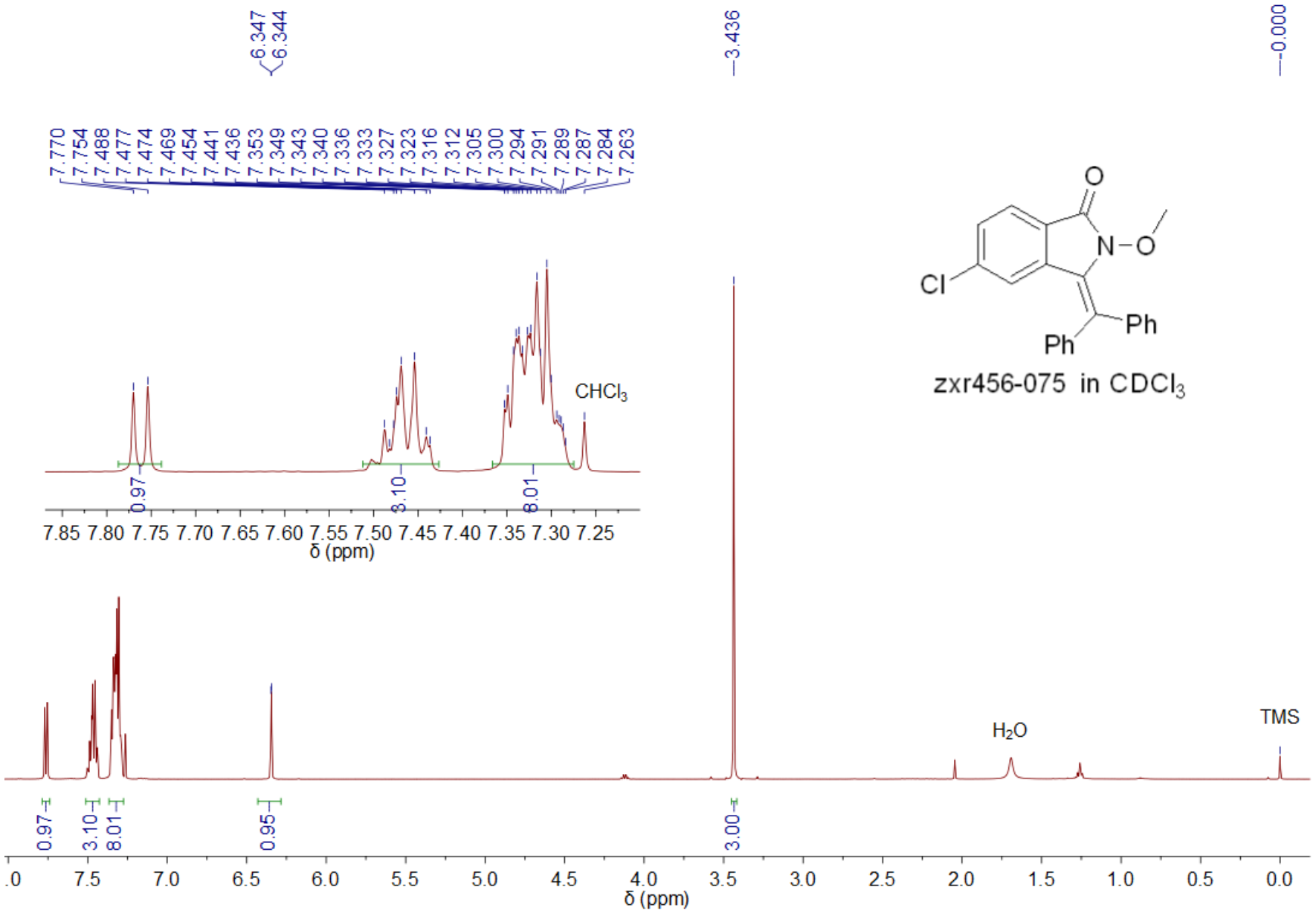
5a – ^{13}C NMR


7.643
7.060
7.057
7.039
7.037
6.339
6.319
3.427
2.376
1.688
0.000
7.434
7.426
7.422
7.415
7.413
7.409
7.407
7.397
7.351
7.346
7.338
7.335
7.332
7.327
7.318
7.313
7.309
7.300
7.294
7.291
7.286
7.283
7.273
7.262
CHCl3
2.95
6.91
7.44 7.42 7.40 7.38 7.36 7.34 7.32 7.30 7.28 7.26
δ (ppm)
O
N–O
Ph
Ph
zxr456-073-3 in CDCl3
H2O
TMS
0.97
2.95
6.91
1.01
1.02
3.00
3.00
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 0.0
δ (ppm)

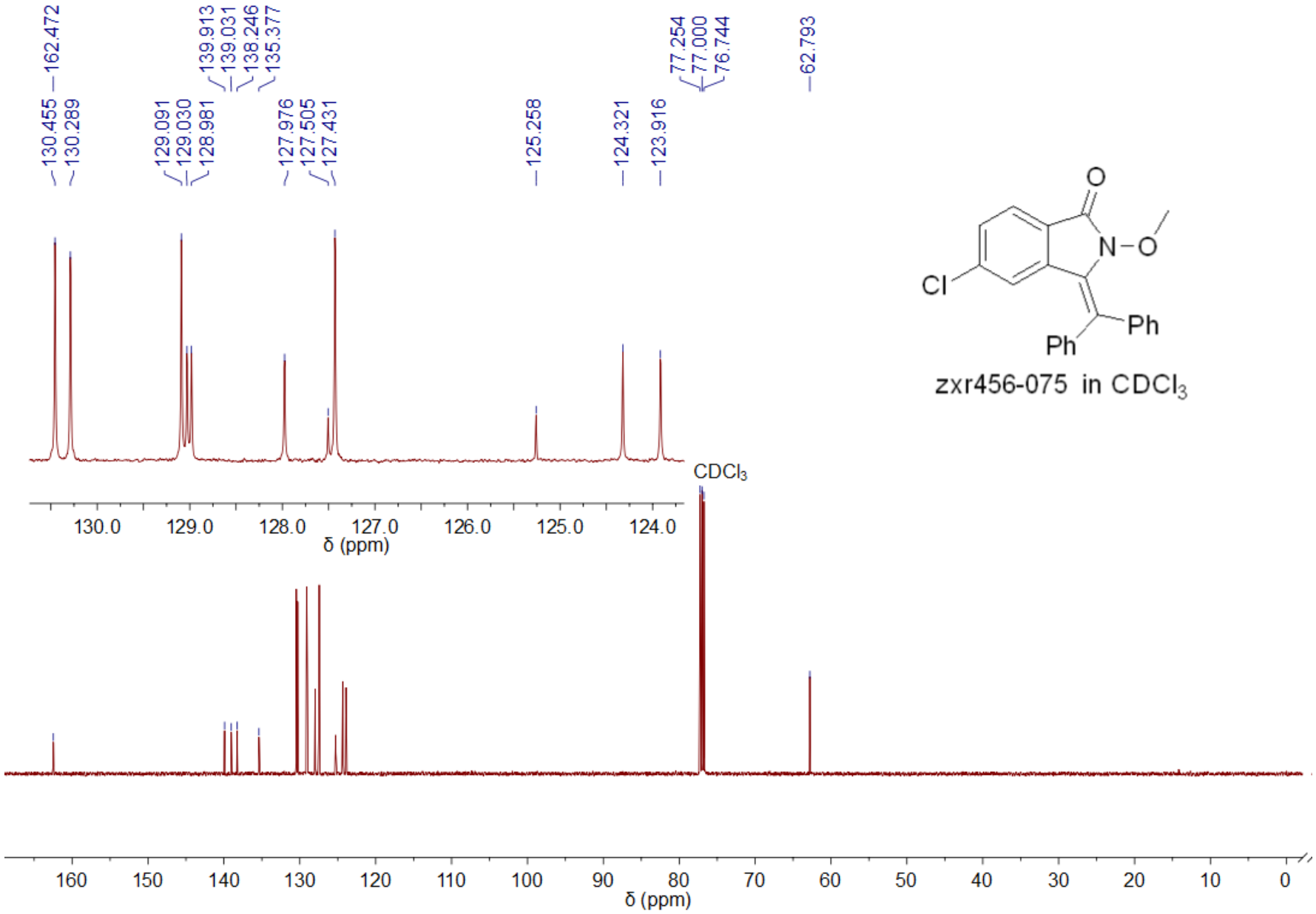
5b – ^{1}H NMR


163.613
140.654
139.544
139.144
133.032
131.493
130.635
130.361
128.866
128.519
128.406
127.559
127.327
127.147
125.183
123.497
123.254
77.318
77.000
76.683
62.599
21.385
CDCl3
O
N
O
Ph
Ph
zxr456-073-3 in CDCl3
δ (ppm)
δ (ppm)

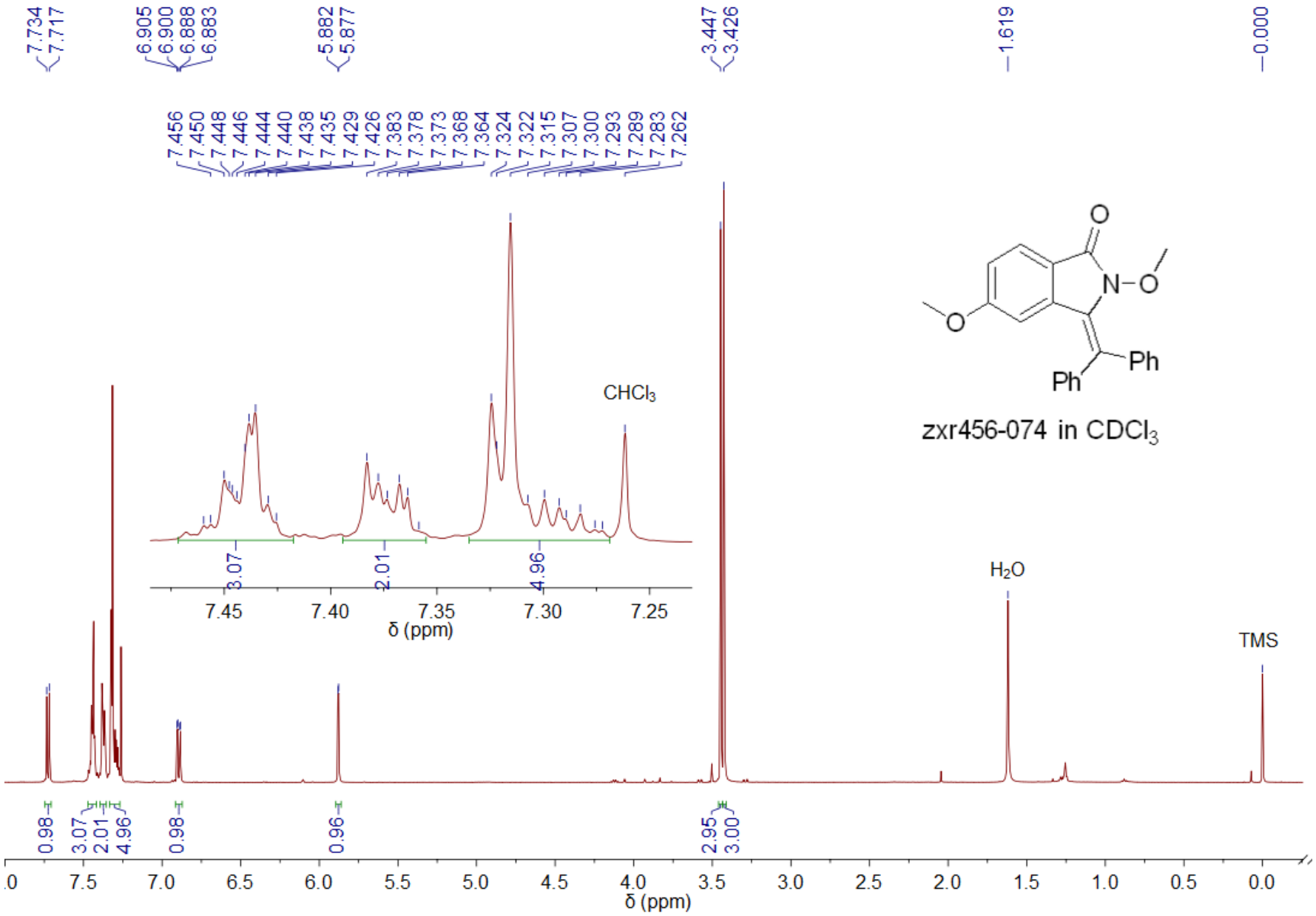
5b – ^{13}C NMR


6.347
6.344
3.436
0.000
7.770
7.754
7.488
7.477
7.474
7.469
7.454
7.441
7.436
7.353
7.349
7.343
7.340
7.336
7.333
7.327
7.323
7.316
7.312
7.305
7.300
7.294
7.291
7.289
7.287
7.284
7.263
CHCl3
0.97
3.10
8.01
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25
δ (ppm)
O
N–O
Cl
Ph
Ph
zxr456-075 in CDCl3
H2O
TMS
0.97
3.10
8.01
0.95
3.00
.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 0.0
δ (ppm)

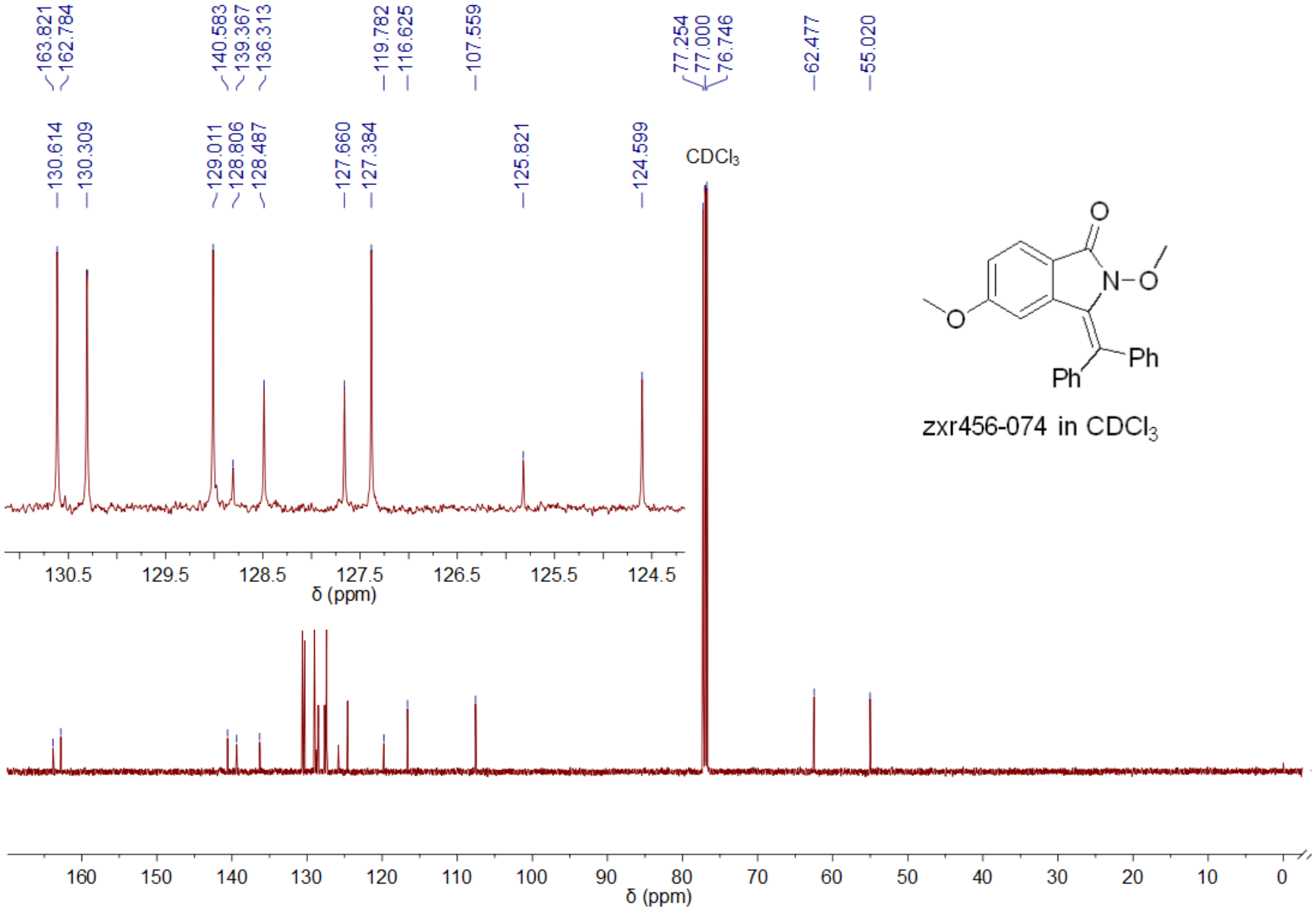
5c –^{1}H NMR


162.472
130.455
130.289
139.913
139.031
138.246
135.377
129.091
129.030
128.981
127.976
127.505
127.431
125.258
124.321
123.916
77.254
77.000
76.744
62.793
CDCl3
130.0
129.0
128.0
127.0
126.0
125.0
124.0
δ (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)
O
N
O
Cl
Ph
Ph
zxr456-075 in CDCl3

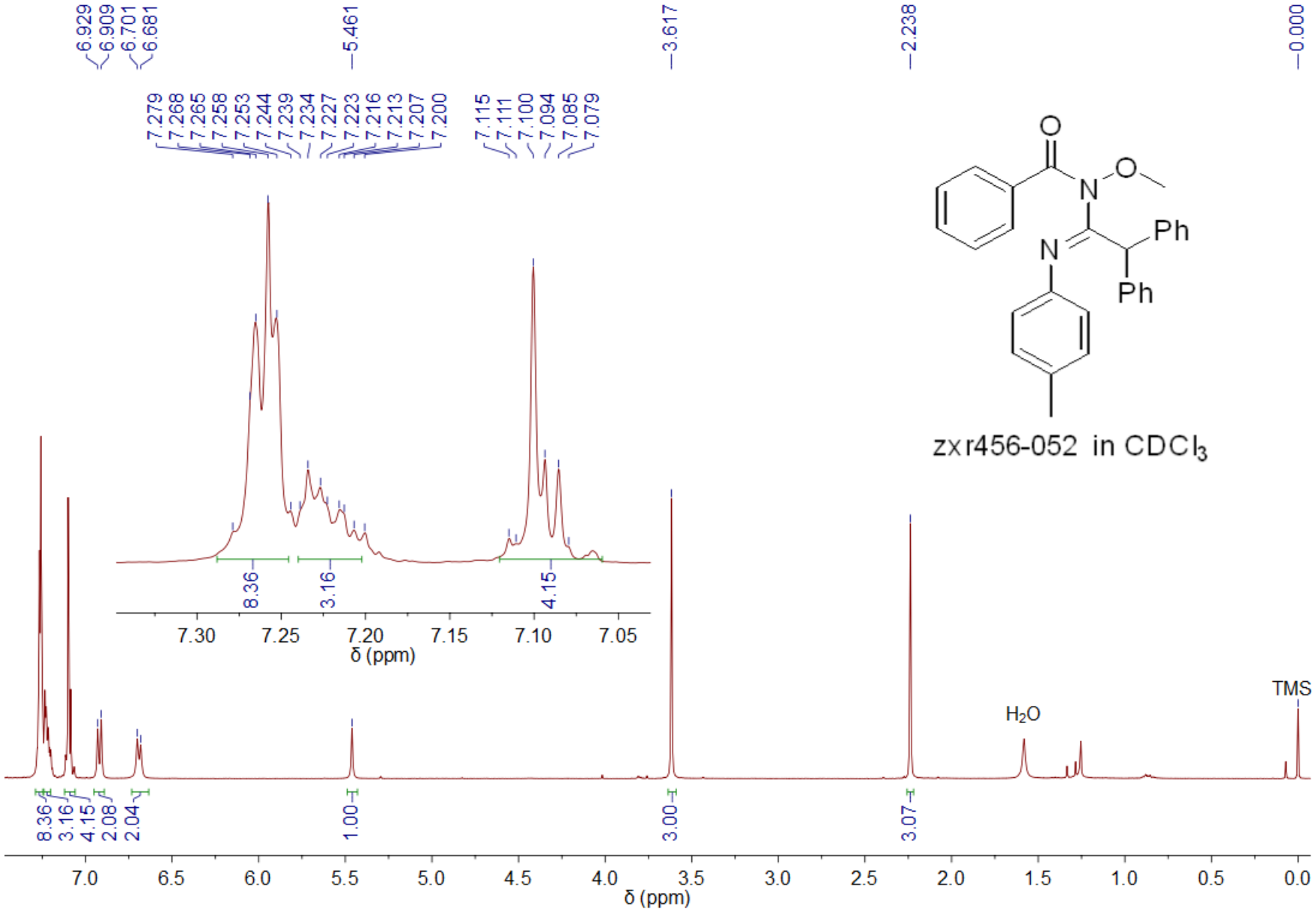
5c – ^{13}C NMR


7.734
7.717
6.905
6.900
6.888
6.883
5.882
5.877
3.447
3.426
1.619
0.000
7.456
7.450
7.448
7.446
7.444
7.440
7.438
7.435
7.429
7.426
7.383
7.378
7.373
7.368
7.364
7.324
7.322
7.315
7.307
7.300
7.293
7.289
7.283
7.262
CHCl3
3.07
2.01
4.96
7.45
7.40
7.35
7.30
7.25
δ (ppm)
O
N–O
O
Ph
Ph
zxr456-074 in CDCl3
H2O
TMS
0.98
3.07
2.01
4.96
0.98
0.96
2.95
3.00
.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
0.0
δ (ppm)

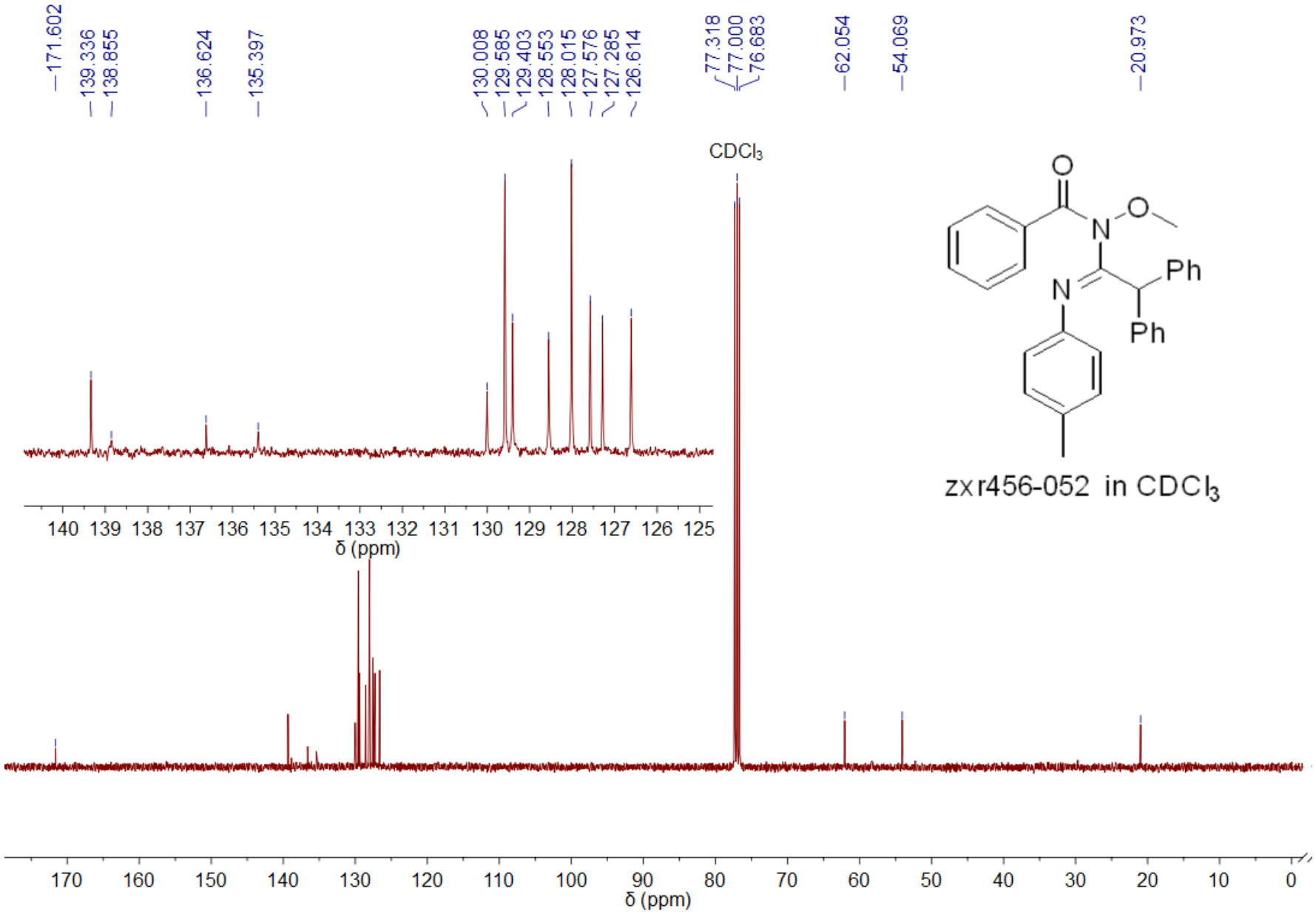
5d – ^{1}H NMR


163.821
162.784
140.583
139.367
136.313
119.782
116.625
107.559
77.254
77.000
76.746
62.477
55.020
130.614
130.309
129.011
128.806
128.487
127.660
127.384
125.821
124.599
CDCl3
O
N–O
O
Ph
Ph
zxr456-074 in CDCl3
130.5
129.5
128.5
127.5
126.5
125.5
124.5
δ (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

5d – ^{13}C NMR


6.929
6.909
6.701
6.681
5.461
3.617
2.238
0.000
7.279
7.268
7.265
7.258
7.253
7.244
7.239
7.234
7.227
7.223
7.216
7.213
7.207
7.200
7.115
7.111
7.100
7.094
7.085
7.079
O
N
O
Ph
N
Ph
zxr456-052 in CDCl3
8.36
3.16
4.15
7.30
7.25
7.20
7.15
7.10
7.05
δ (ppm)
TMS
H2O
8.36
3.16
4.15
2.08
2.04
1.00
3.00
3.07
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
0.0
δ (ppm)

6 –^{1}H NMR


171.602
139.336
138.855
136.624
135.397
130.008
129.585
129.403
128.553
128.015
127.576
127.285
126.614
77.318
77.000
76.683
62.054
54.069
20.973
CDCl3
O
N
O
Ph
N
Ph
zxr456-052 in CDCl3
140 139 138 137 136 135 134 133 132 131 130 129 128 127 126 125
δ (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

6 – ^{13}C NMR