

Supporting Information

Exceptionally Efficient Catalytic Hydrodechlorination of Persistent Organic Pollutants: Application of New Sterically Shielded Palladium Carbene Complexes

Vagiz Sh. Saberov,[†] Daniel A. Evans,[‡] Nikolai I. Korotkikh,^{*†} Alan H. Cowley,[‡]
Tatyana M. Pekhtereva,[†] Anatolii F. Popov,[†] and Oles P. Shvaika[†]

[†]The L.M. Litvinenko Institute of Physical Organic & Coal Chemistry, Ukrainian Academy of Sciences, 70, R. Luxemburg, Donetsk, 83114, Ukraine

[‡]Department of Chemistry, The University of Texas at Austin, 1 University Station A5300, Austin, Texas 78712-0165

Fax: (512)471-6822; Tel: (512)471-7484;

E-mail: nkorotkikh@ua.fm

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[1,3-Bis(2,6-dibenzhydryl-4-ethylphenyl)imidazol-2-ylidene]palladium chloride (**11a**) (CDCl_3).

[1,3-Bis(2,6-dibenzhydryl-4-ethylphenyl)imidazol-2-ylidene]palladium iodide (**11b**) (CDCl_3).

1. General.

^1H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance II 400 spectrometer (400 MHz for ^1H NMR spectra and 100 MHz for ^{13}C NMR spectra) in DMSO-d_6 , C_6D_6 , or CDCl_3 solution. The ^1H NMR and ^{13}C NMR chemical shifts are reported relative to tetramethylsilane (TMS) (solution). The X-ray diffraction data were collected on either a Rigaku AFC12 Saturn 724+ CCD diffractometer equipped with a Rigaku XStream low temperature device that operated at 100 K or on a Rigaku SCX-Mini Diffractometer equipped with a Rigaku XStream low temperature device that operated at 100 K, or on a Nonius-Kappa CCD diffractometer with an Oxford cryostream 600 that operated at 153 K. All instruments used a graphite-monochromated $\text{Mo K}\alpha$ radiation source ($\lambda = 0.71075 \text{ \AA}$). Corrections were applied for Lorentz and polarization effects. The structures were solved by direct methods and refined by full-matrix least-squares cycles on F^2 using the Siemens SHELXTL PLUS 5.0 (PC) software package ¹ and PLATON ². All non-hydrogen atoms were refined anisotropically. The hydrogen atoms were placed in fixed, calculated positions using a riding model.

Thin-layer chromatography was performed on silica gel with either pure chloroform or a 10:1 mixture of chloroform and methanol as the eluent, followed by development with iodine. Elemental analyses were carried out at the Analytical Laboratory of the Litvinenko Institute of Physical Organic and Coal Chemistry.

2. Syntheses of compounds 6a, 7a, 8a, 9a, 10b, 11a,b.

2,6-Dibenzhydryl-4-ethylaniline (6a) was prepared in a similar fashion to the literature procedure. ³ A solution of zinc chloride (2.71 g, 20.4 mmol) in hydrochloric acid (36% HCl, 3.40 mL, 40.7 mmol) was added dropwise to a mixture of 4-ethylaniline (4.92 g, 40.7 mmol) and benzhydrol (15.00 g, 81.4 mmol) at 160 °C. The mixture was refluxed for 3 h until it solidified. Once cooled, the product was dissolved in dichloromethane (100 mL) and washed with saturated aqueous solution of ammonium chloride and water. The organic layer was dried with magnesium sulfate and the dichloromethane solvent was evaporated. The solid that had formed was washed with ethyl acetate (50 mL) until the filtrate became colorless; the solid product was subsequently dried. Yield 13.50 g (73%). mp 182 °C (acetonitrile). Found: C, 90.1; H, 6.8; N, 3.1. Calc. for $\text{C}_{34}\text{H}_{31}\text{N}$: C, 90.0; H, 6.9; N, 3.1%. ^1H

NMR (400 MHz, CDCl₃, Me₄Si): 0.92 (t, 3H, *J* 7.6 Hz, CH₃C), 2.29 (qr, 2H, *J* 7.6 Hz, CH₂C), 3.27 (s, 2H, NH₂), 5.45 (s, 2H, CHPh), 6.42 (s, 2H, C^{3,5}H), 7.08 (m, 8H, *J* 6.8 Hz) (Ar), 7.18 (d, 4H), 7.24 (m, 8H). ¹³C NMR (100 MHz, CDCl₃, Me₄Si): 15.8 (CH₃C), 28.1 (CH₂C), 52.4 (CHPh), 126.5 (C^{3,5}, Ar), 127.8 (C⁴, Ph), 128.4 (C^{3,5}, Ph), 129.1 (*ipso*-C^{2,6}), 129.4 (C⁴, Ar), 133.1 (C¹, Ar), 139.8 (C^{2,6}, Ar), 142.7 (*ipso*-CPh).

N,N'-Bis(2,6-dibenzhydryl-4-ethylphenyl)-1,4-diazabutadiene (7a) was prepared in a similar fashion to the literature procedure.³ Two drops of formic acid (0.06 mL) and a solution of 40% aqueous glyoxal (1.2 mL, 10 mmol) was added dropwise to a suspension of 2,6-dibenzhydryl-4-ethylaniline (9.06 g, 20 mmol) in *n*-propanol (60 mL) and refluxed for 3 h. Once cooled, the precipitate that had formed was filtered off, washed with *n*-propanol (25 mL) and subsequently dried. Yield 7.65 g (82%), mp > 300 °C (chloroform). Found: C, 90.4; H, 6.5; N, 3.1. Calc. for C₇₀H₆₀N₂: C, 90.5; H, 6.5; N, 3.0%. ¹H NMR (400 MHz, CDCl₃, Me₄Si): 1.05 (t, 6H, *J* 7.2 Hz, CH₃C), 2.45 (qr, 2H) (*J* 7.2 Hz, CH₂C), 5.29 (s, 4H, CHPh), 6.73 (s, 4H, C^{3,5}H), 7.03 (m, 16H, Ar), 7.24 (m, 26H, Ar+CHN). ¹³C NMR (100 MHz, CDCl₃, δ, ppm): 15.6 (CH₃C), 28.5 (CH₂C), 51.2 (CHPh), 126.4 (C⁴, Ph), 128.0 (C^{3,5}, Ar), 128.3 (C^{3,5}, Ph), 129.6 (C^{2,6}, Ph), 132.0 (C^{2,6}, Ar), 140.0 (C¹, Ar), 143.9 (*ipso*-C-Ph), 146.9 (C⁴, Ar), 163.9 (CHN).

1,3-Bis(2,6-dibenzhydryl-4-ethylphenyl)imidazolium chloride (8a) was prepared in a similar fashion to the literature procedure.³ A solution of paraformaldehyde (0.32 g, 10.66 mmol, 1.3 eq), zinc chloride (1.45 g, 10.66 mmol, 1.3 eq.), and 36% hydrochloric acid (1.5 mL, 10.66 mmol, 1.3 eq.) was added to a solution of the diimine **7a** (7.64 g, 8.2 mmol) in chloroform solution (60 mL) and stirred for 4 h. Hydrochloric acid (2M HCl, 50 mL) was added to the reaction mixture. The organic layer was washed with water, dried with magnesium sulfate, and solvent was subsequently evaporated. The solid product was refluxed with diethyl ether (50 mL), filtered off, and dried. Yield 6.00 g (75 %). mp > 300°C (benzene). Found: C, 87.1; H, 6.3; Cl, 3.7; N, 2.9. Calc. for C₇₁H₆₁ClN₂: C, 87.2; H, 6.3; Cl, 3.6; N, 2.9 %. ¹H NMR (400 MHz, CDCl₃, Me₄Si): 0.99 (t, 6H, *J* 7.6 Hz, CH₃C), 2.45 (qr, 4H, *J* 7.6 Hz, CH₂C), 5.29 (s, 4H, CHPh), 5.52 (s, 2H, C^{4,5}H-im), 6.78 (m, 12H, C^{3,5}H), 7.12 (m, 16H), 7.23 (m, 16H) (Ar), 12.83 (s, 1H, CHN). ¹³C NMR (100 MHz, CDCl₃, Me₄Si): 14.9 (CH₃C), 28.8 (CH₂C), 51.5 (CHPh), 123.5 (C^{4,5}Im), 126.9, 127.1, 128.71, 128.74, 127.9, 129.3, 129.7, 130.2 (CH, Ar), 142.7 (C²HN), 130.3, 140.8, 142.1, 142.6 (*ipso*-C, Ar, Ph), 147.5 (*ipso*-C^{2,6}, Ar).

1,3-Bis(2,6-dibenzhydryl-4-ethylphenyl)imidazol-2-ylidene (a cyclohexane solvate) (9a). A suspension of sodium hydride in paraffin oil (37% NaH, 0.04 g, 0.6 mmol, 1.5 eq.) was added to a solution of the salt **8a** (0.39 g, 0.4 mmol) in anhydrous THF (7 mL) and stirred for 24 h. The solution was subsequently filtered and the solvent was evaporated resulting in the formation of the solid product. Yield 0.30 g (79%). mp 182–183°C (cyclohexane). Found: C, 90.3; H, 7.1; N, 2.6 %. Calc. for C₇₇H₇₂N₂: C, 90.2; H, 7.1; N, 2.7 %. ¹H NMR (400 MHz, C₆D₆, Me₄Si): 0.87 (m, 6H, CH₃C), 1.42

(s, 12H, CyH), 2.22 (m, 4H, CH₂C), 5.81 (s, 2H, C^{4,5}HN), 6.04 (s, 4H, CHPh), 6.99–7.17 (m, 36H), 7.38 (m, 8H) (Ar). ¹³C NMR (100 MHz, CDCl₃, Me₄Si): 15.4 (CH₃C), 27.2 (CyH, CH₂C), 28.9 (CH₂C), 51.5 (CHPh), 122.5 (C^{4,5}, Im), 126.5 (C^{3,5}, Ar), 128.5, 128.6 (C^{3,5}, Ph), 128.8 (C⁴, Ph), 129.9, 130.2 (C^{2,6}, Ph), 138.3 (*ipso*-C¹, Ar), 141.9, 143.9 (C¹, Ph), 144.5 (*ipso*-C⁴, Ar), 145.0 (*ipso*-C^{2,6}, Ar), 218.6 (C²N).

[1,3-Bis(2,6-diisopropylphenyl)imidazol-2-ylidene]palladium iodide (10b). A solution of 1,3-(2,6-diisopropylphenyl)imidazolium perchlorate (0.98 g, 2.0 mmol), prepared according to the literature procedure,⁴ and potassium *tert*-butoxide (0.24 g, 2.1 mmol) in anhydrous THF (15 mL) was stirred for 2 h. Palladium iodide 0.72 g (2 mmol) was added to the reaction mixture which was stirred at room temperature for 6 h. The solvent was then evaporated resulting in the formation of the product which was then subsequently stirred in petroleum ether. The precipitate that was formed was dissolved in a mixture of cyclohexane and diethyl ether (1:1) and filtered through a thin layer of silica gel (70/230 μm). The solvent was then evaporated and the residue was stirred with petroleum ether. The resulting precipitate that had formed was filtered off and dried. Yield 1.3 g (87 %), mp 288 – 289 °C (cyclohexane). Found: C, 43.2; H, 4.8; I, 34.0; N, 3.7%. Calc. for C₅₄H₇₂I₄N₄Pd₂: C, 43.3; H, 4.9; I, 33.9; N, 3.7%. ¹H NMR (400 MHz, DMSO-d₆, Me₄Si): 0.93 (d, 12H, *J* 6.8 Hz), 1.07 (d, 12H, *J* 6.8 Hz), 1.24 (d, 12H, *J* 6.8 Hz), 1.46 (d, 12H, *J* 6.8 Hz) (CH₃C, *i*-Pr), 2.75 (*t*, 4H, *J* 6.8 Hz), 3.29 (*t*, 4H, *J* 6.8 Hz) (CHC, *i*-Pr), 7.09 (s, 2H, C^{4,5}N), 7.25 (d, 4H, *J* 7.6 Hz), 7.33 (d, 4H, *J* 7.6 Hz), 7.51 (dd, 4H, *J* 7.6 Hz) (Ar). ¹³C NMR (100 MHz, DMSO-d₆, Me₄Si): 23.9, 24.0 (CHC, *i*-Pr), 26.5, 29.0 (CH₃C, *i*-Pr), 124.2, 124.7 (C^{4,5}, Im), 125.4, 130.3 (Ar), 135.3 (*ipso*-C¹, Ar), 145.9, 146.3 (*ipso*-C^{2,6}, Ar), 165.4 (C²-Pd).

[1,3-Bis(2,6-dibenzhydryl-4-ethylphenyl)imidazol-2-ylidene]palladium chloride (11a). A suspension of sodium hydride (37% NaH, 0.05 g, 0.75 mmol, 1.5 eq.) in paraffin oil was added to a solution of the salt **8b** (0.49 g, 0.5 mmol) in anhydrous THF solution (8 mL) and stirred for 24 h. Next, Pd(PhCN)₂Cl₂ (0.19 g, 0.5 mmol) was added and after 5 h of stirring, the solution was filtered and the solvent was evaporated to dryness resulting in formation of the solid product. Yield 0.43 g (77%), mp 251–252°C (acetonitrile). Found: C, 76.3; H, 5.2; Cl, 6.4; N, 2.6. Calc. for C₇₁H₆₀Cl₂N₂Pd: C, 76.2; H, 5.4; Cl, 6.3; N, 2.5%. ¹H NMR (400 MHz, CDCl₃, Me₄Si): 0.66 (*t*, 3H, *J* 7.6 Hz, CH₃C), 0.97 (*tt*, 3H, *J* 7.2 Hz, CH₃C), 2.01 (*qr*, 2H, *J* 7.6 Hz, CH₂C), 2.32 (*qrqr*, 2H, *J* 7.2 Hz, CH₂C), 4.14 (s, 1H, CHPh), 4.32 (s, 1H, CHPh), 5.42 (s, 1H, CHPh), 6.11 (s, 2H, C^{4,5}HN), 6.32 (s, 1H, CHPh), 6.49 (s, 2H, Ar), 6.76 (m, 7H, Ar), 6.92 (m, 4H, Ar), 7.01–7.06 (m, 7H, Ar), 7.16–7.29 (m, 20H, Ar), 7.58 (m, 4H, Ar). ¹³C NMR (100 MHz, CDCl₃, Me₄Si): 14.0, 15.0, 15.6 (CH₃C), 28.1, 28.5 (CH₂C), 51.3, 51.7 (CHPh), 126.0, 126.1 (C^{4,5}, Im), 123.2, 125.8, 127.1, 127.4, 127.6, 127.9, 128.1, 128.4, 128.6,

128.9, 129.5, 129.8, 130.1, 131.2 (CH, Ar), 133.7, 133.8, 134.8, 136.1, 138.6, 140.8, 141.1, 141.5, 142.7 (*ipso*-C, Ar), 143.2, 144.7 (*ipso*-C4, Ar), 143.4, 145.7 (*ipso*-C, Ph), 156.4 (C²-Pd).

[1,3-Bis(2,6-dibenzhydryl-4-ethylphenyl)imidazol-2-ylidene]palladium iodide (11b). A solution of potassium bis(trimethylsilyl)amide in toluene (0.55 mmol) was added to a solution of the salt **8b** (0.49 g, 0.5 mmol) in anhydrous THF solution (8 mL) and stirred for 2 h. Next, palladium iodide (0.18 g, 0.5 mmol) was added and the resulting solution was stirred for 22 h, following which the solution was filtered and the solvent was evaporated. The residue was dissolved in dichloromethane solution and filtered through a thin layer of silica gel. The dichloromethane solution was then evaporated to dryness. Yield 0.50 g (77%), mp > 300 °C (acetonitrile). Found: C, 65.6; H, 4.7; I, 19.4; N, 2.2%. Calc. for C₇₁H₆₀I₂N₂Pd: C, 65.5; H, 4.7; I, 19.5; N, 2.2%. ¹H NMR (400 MHz, CDCl₃, Me₄Si): 0.96 (s, broad., 6H, CH₃C), 2.44 (s, broad., 4H, CH₂C), 5.06 (s, broad., 4H, CHPh), 5.75, 5.93 (s, broad., 2H, C^{4,5}HN), 6.78, 7.17 (m, 44H, Ar); ¹³C NMR (100 MHz, CDCl₃, Me₄Si): 16.1 (CH₃C), 29.8 (CH₂C), 52.6 (CHPh), 128.3, 128.6 (C^{4,5}, Im), 125.4, 129.3, 130.0, 130.9, 132.0 (CH, Ar), 138.2, 141.4, 142.2, 143.1 (*ipso*-C, Ar), 149.1 (C²Pd).

General procedure for the catalytic reaction. A specified amount of catalyst (see Tables 2 and 3) was added to either a solution of *p*-dichlorobenzene (2 mmol) in isopropanol (10 mL) or to a dispersion of hexachlorobenzene (2 mmol) in isopropanol (10 mL). Following this addition, the appropriate solution or mixture was heated to 60 °C. At this temperature, a base (potassium *tert*-butoxide, sodium *tert*-butoxide, or sodium hydroxide) (5.2 mmol for *p*-dichlorobenzene or 16 mmol for hexachlorobenzene) was added to the reaction mixture under an argon atmosphere and subsequently refluxed for 8 or 24 h. The precipitate that had formed was filtered off, washed with isopropanol and petroleum ether, and subsequently dried. The percent conversion was estimated according to the masses of the potassium or sodium chloride precipitate that had formed during the hydrodechlorination reaction. The TON (turnover number, number of cycles of the catalytic reaction) was calculated as a ratio of moles of substrate (per 1 chlorine atom) to the number of moles of catalyst. The TOF (turnover frequency, productivity of the reaction in h⁻¹) was calculated as a ratio of the TON to the reaction time in hours.

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3. Crystallographic data for 9a, 10b and 11a

Table 1. Crystal data and structure refinement for **9a**.

Identification code	shelxl	
Empirical formula	C ₈₃ H ₇₂ N ₂	
Formula weight	1097.43	
Temperature	133(2) K	
Wavelength	0.71073 E	
Crystal system	Monoclinic	
Space group	P21/c	
Unit cell dimensions	a = 20.0820(11) E	$\alpha = 90^\circ$.
	b = 12.4790(7) E	$\beta = 95.975(3)^\circ$.
	c = 25.4950(12) E	$\gamma = 90^\circ$.
Volume	6354.4(6) E ³	
Z	4	
Density (calculated)	1.147 Mg/m ³	
Absorption coefficient	0.065 mm ⁻¹	
F(000)	2336	
Crystal size	0.37 x 0.24 x 0.22 mm ³	
Theta range for data collection	1.02 to 25.00°.	
Index ranges	-23 ≤ h ≤ 23, -14 ≤ k ≤ 14, -30 ≤ l ≤ 30	
Reflections collected	128546	
Independent reflections	11173 [R(int) = 0.0735]	
Completeness to theta = 25.00°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0 and 0.852	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	11173 / 732 / 832
Goodness-of-fit on F^2	1.039
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0668, wR2 = 0.1550
R indices (all data)	R1 = 0.1128, wR2 = 0.2097
Largest diff. peak and hole	0.633 and -0.324 e.E ⁻³

Table 2. Crystal data and structure refinement for **10b**.

Identification code	shelxl	
Empirical formula	C ₅₄ H ₇₂ I ₄ N ₄ Pd ₂	
Formula weight	1497.56	
Temperature	100(2) K	
Wavelength	0.71073 E	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 16.2590(3) E	$\alpha = 81.490(4)^\circ$.
	b = 17.1190(4) E	$\beta = 75.160(4)^\circ$.
	c = 21.2460(3) E	$\gamma = 89.228(4)^\circ$.
Volume	5651.54(19) E ³	
Z	4	
Density (calculated)	1.760 Mg/m ³	
Absorption coefficient	2.858 mm ⁻¹	
F(000)	2912	
Crystal size	0.21 x 0.18 x 0.04 mm ³	
Theta range for data collection	1.68 to 27.50°.	

Index ranges	-20<=h<=21, -22<=k<=22, -27<=l<=27
Reflections collected	60969
Independent reflections	25848 [R(int) = 0.0408]
Completeness to theta = 27.50°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.7247
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	25848 / 0 / 1185
Goodness-of-fit on F ²	1.090
Final R indices [I>2sigma(I)]	R1 = 0.0400, wR2 = 0.0919
R indices (all data)	R1 = 0.0517, wR2 = 0.1023
Largest diff. peak and hole	1.264 and -1.920 e.E ⁻³

Table 3. Crystal data and structure refinement for **11a**

Identification code	shelxl	
Empirical formula	C81 H71 Cl2 N7 Pd	
Formula weight	1319.75	
Temperature	100(2) K	
Wavelength	0.71073 E	
Crystal system	Monoclinic	
Space group	I2/a	
Unit cell dimensions	a = 20.878(4) E	$\alpha = 90^\circ$.
	b = 13.816(3) E	$\beta = 100.548(13)^\circ$.
	c = 24.980(4) E	$\gamma = 90^\circ$.
Volume	7084(2) E ³	

Z	4
Density (calculated)	1.238 Mg/m ³
Absorption coefficient	0.386 mm ⁻¹
F(000)	2744
Crystal size	0.23 x 0.18 x 0.17 mm ³
Theta range for data collection	1.66 to 27.50°.
Index ranges	-27<=h<=27, -17<=k<=17, -32<=l<=32
Reflections collected	63422
Independent reflections	8121 [R(int) = 0.0543]
Completeness to theta = 27.50°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.9273
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	8121 / 35 / 442
Goodness-of-fit on F ²	1.085
Final R indices [I>2sigma(I)]	R1 = 0.0583, wR2 = 0.1605
R indices (all data)	R1 = 0.0606, wR2 = 0.1630
Largest diff. peak and hole	0.804 and -0.957 e.E ⁻³

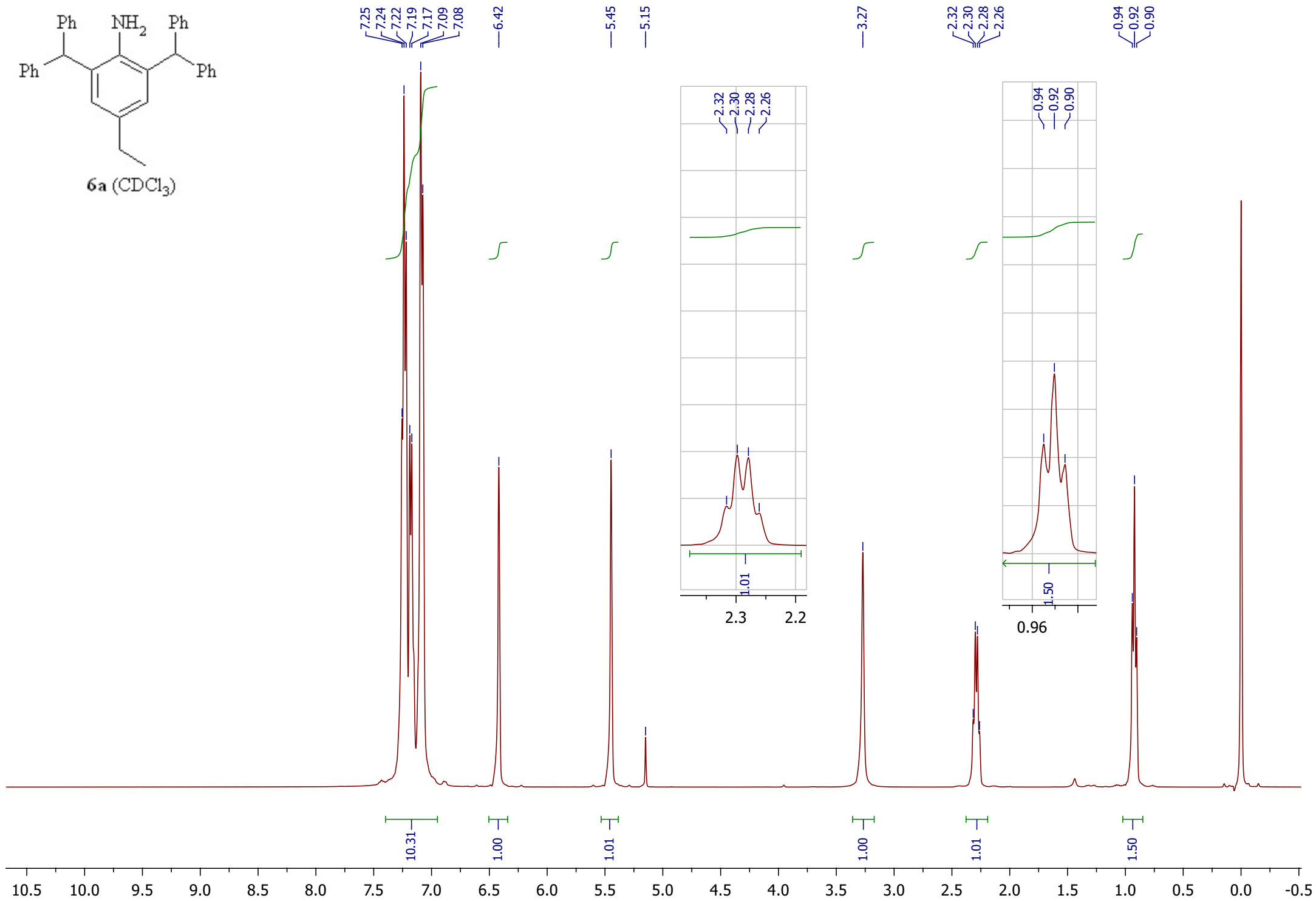
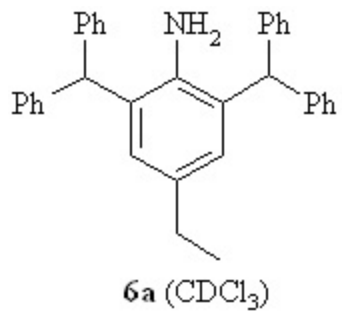
Table 4. Selected bond lengths (Å) and bond angles for complex **10b**

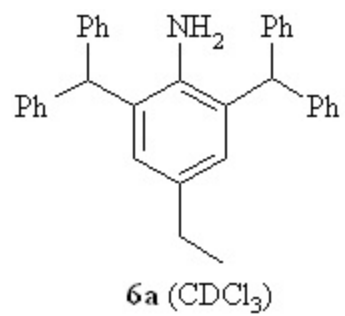
Bond	Length (Å)	Angles	°
C(1)-N(1)	1.347(6)	N(1)-C(1)-N(2)	104.7(4)
C(1)-N(2)	1.352(6)	C(1)-Pd(1)-I(1)	88.87(12)
C(2)-C(3)	1.332 (7)	I(1)-Pd(1)-I(2)	172.883(18)
C(2)-N(1)	1.378 (6)	N(1)-C(2)-C(3)-N(2)	1.4(5)
C(3)-N(2)	1.387(6)	Pd(1)-C(1)-N(1)-C(4)	11.2(6)
N(1)-C(4)	1.454(6)	Pd(1)-C(1)-N(2)-C(16)	-16.8(7)
N(2)-C(16)	1.449(6)	N(1)-C(1)-Pd(1)-I(1)	92.9(4)
C(1)-Pd(1)	1.985(4)	N(2)-C(1)-Pd(1)-I(1)	-78.6(4)

Table 5. Selected bond lengths and bond orders for free carbene **9a** (left) and palladium carbene complex **11a** (right).

Bond 9a	Length (Å) 9a	Bond order 9a	Bond 11a	Length, Å 11a	Bond order 11a
C(1)-N(1)	1.364(4)	1.632	C(1)-N(1)	1.359(3)	1.661
C(2)-C(3)	1.327(5)	2.051	C(2)-C(2a)	1.340(5)	1.985
C(2)-N(1)	1.387(4)	1.500	C(2)-N(1)	1.394 (3)	1.460
C(3)-N(2)	1.381(4)	1.534	-	-	-
C(1)-N(2)	1.368(4)	1.609	C(1)-N(1a)	1.359(3)	1.661
N(1)-C(9)	1.434(4)	1.230	N(1)-C(3)	1.449(3)	1.144
N(2)-C(38)	1.431(4)	1.247	-	-	-
-	-	-	C(1)-Pd(1)	1.961(4)	-
-	-	-	Pd(1)-Cl(1)	2.3002(9)	-

4. ^1H and ^{13}C NMR spectra





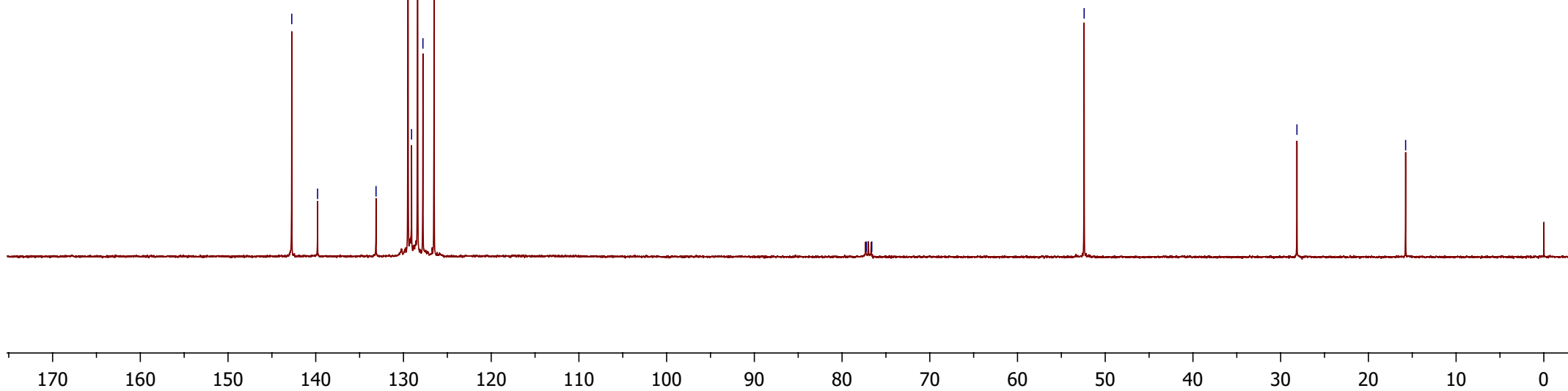
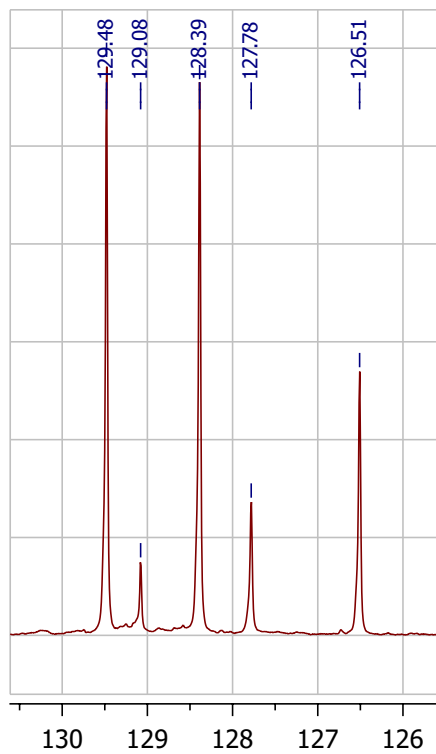
142.73
139.79
133.13
129.48
129.08
128.39
127.78
126.51

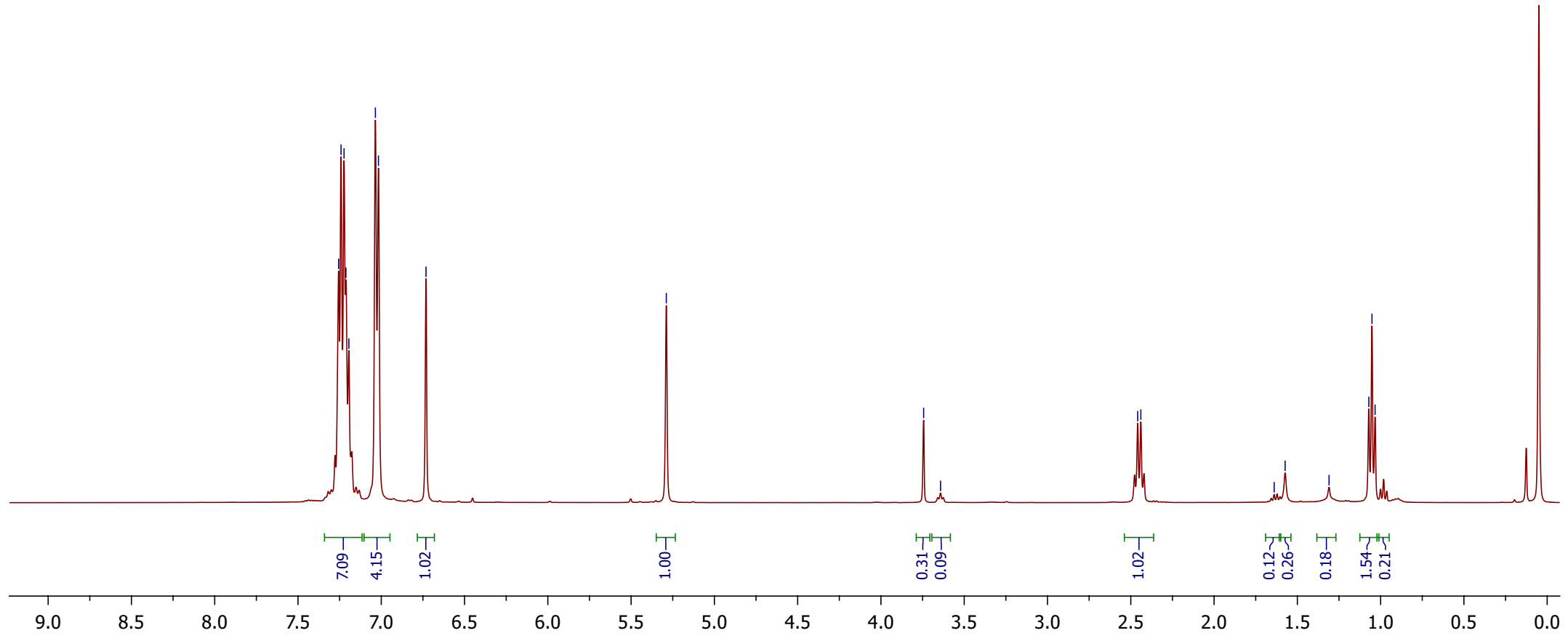
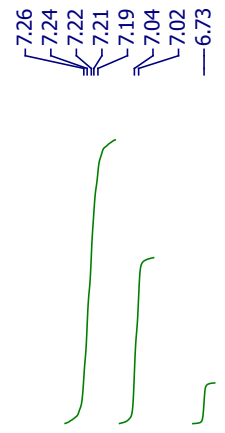
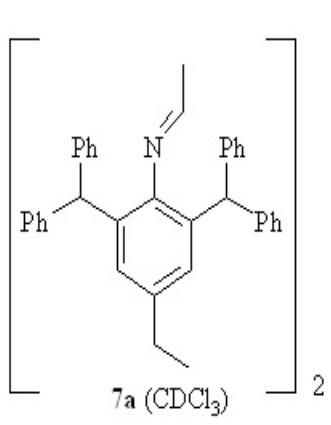
77.22
76.61

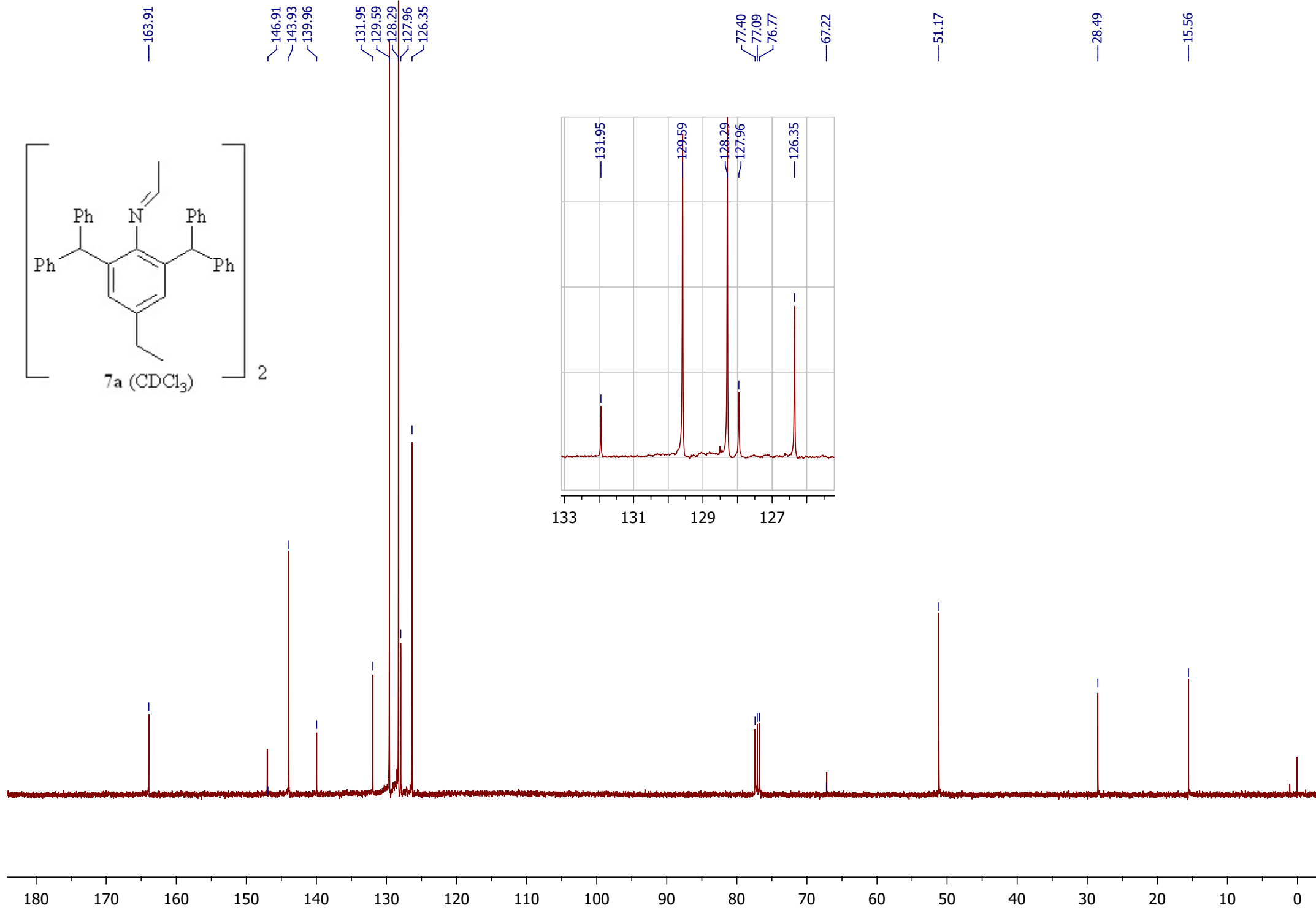
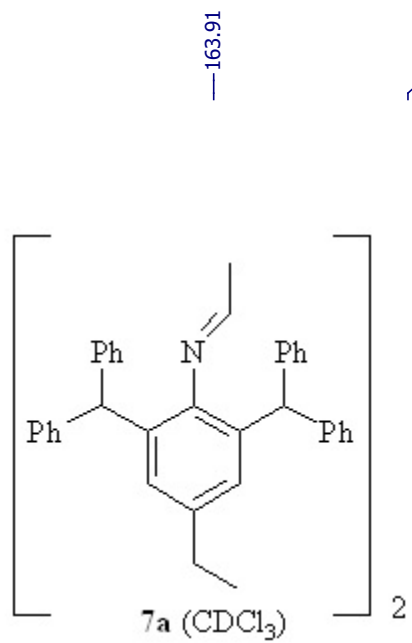
52.40

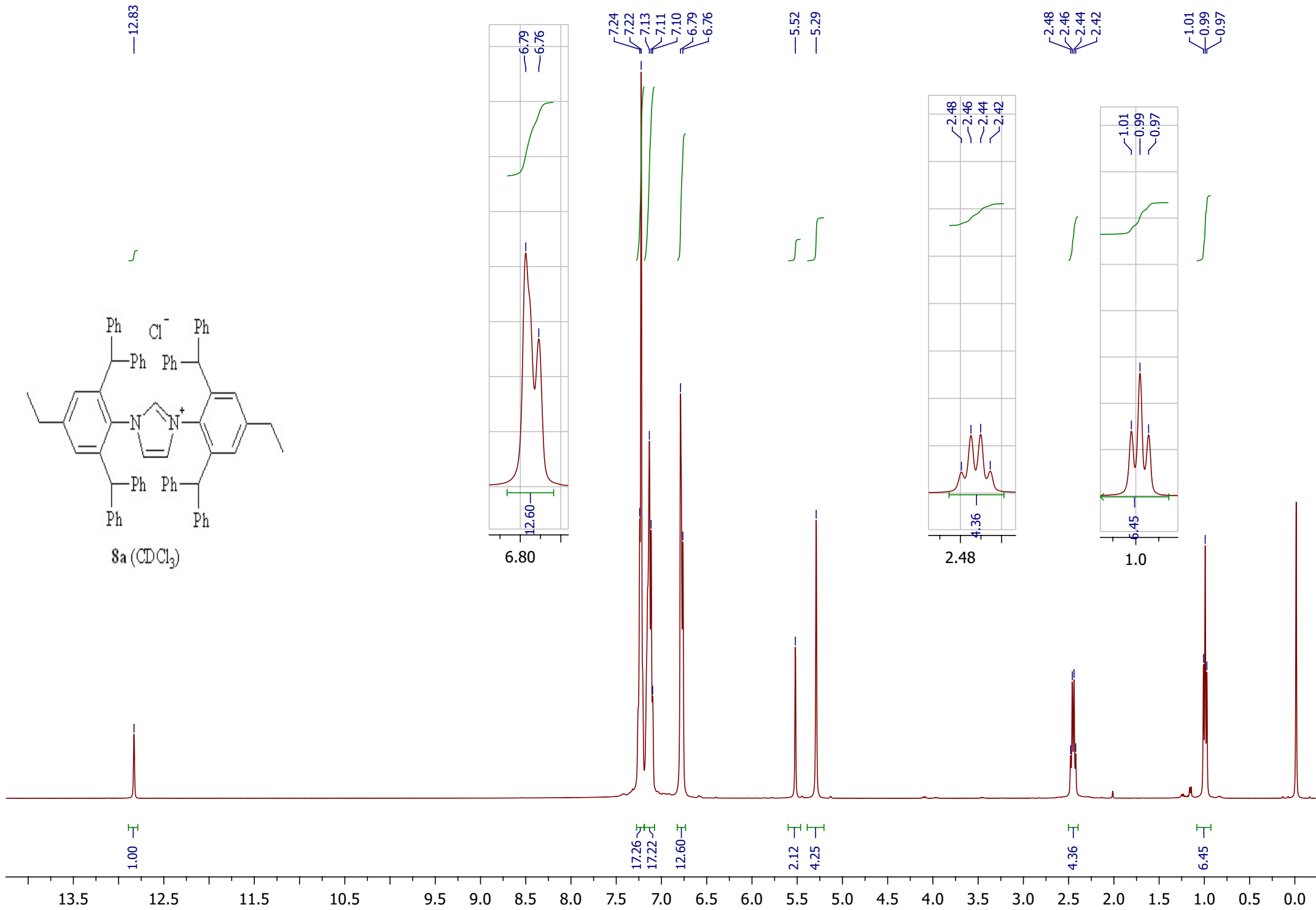
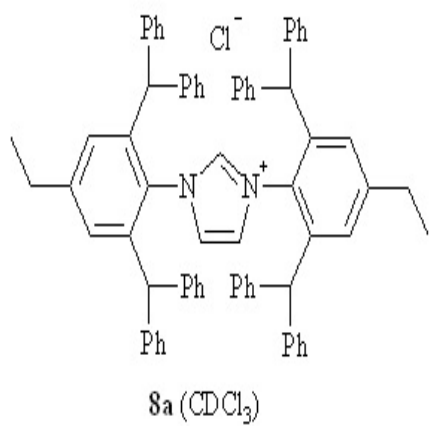
28.14

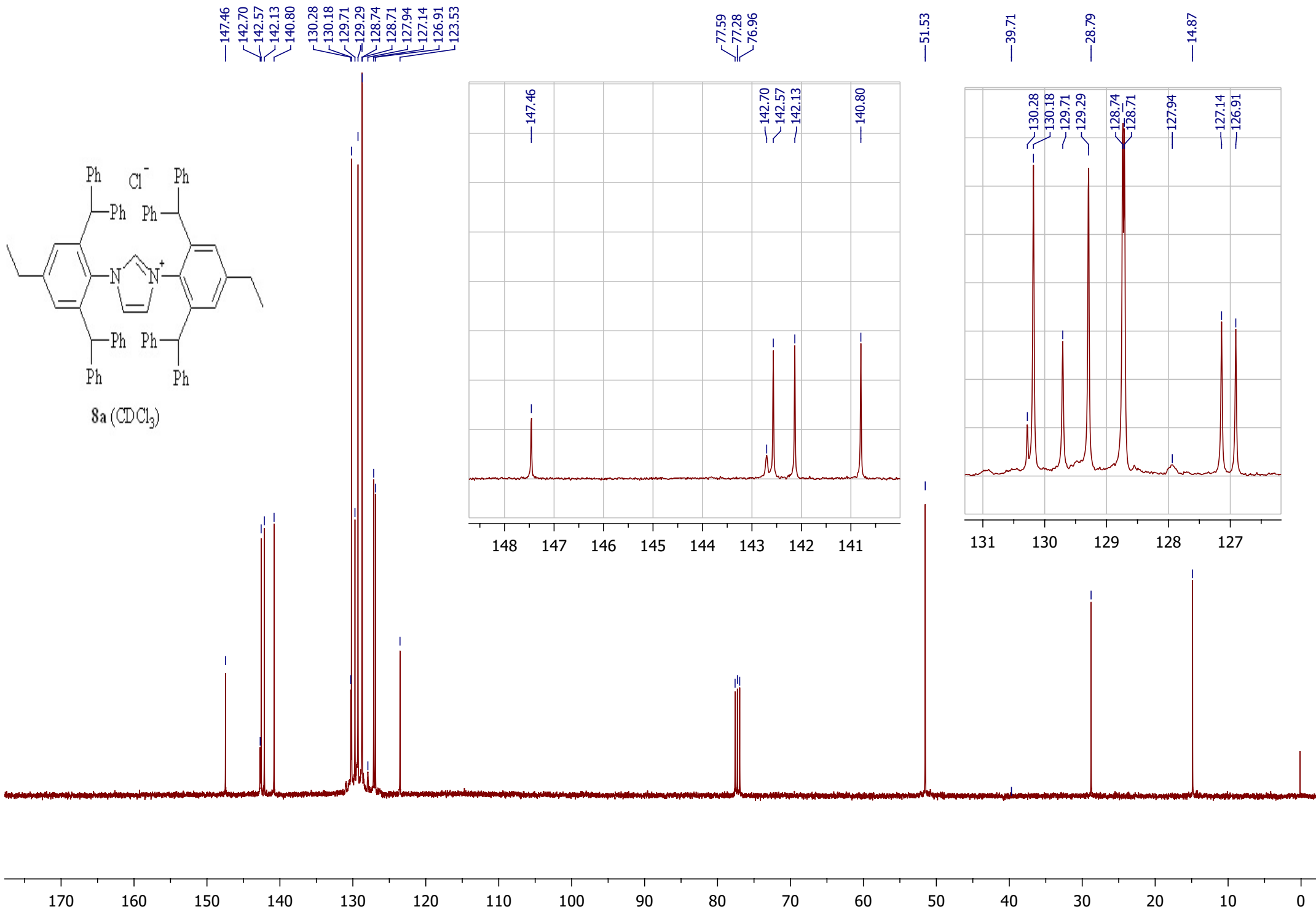
15.75

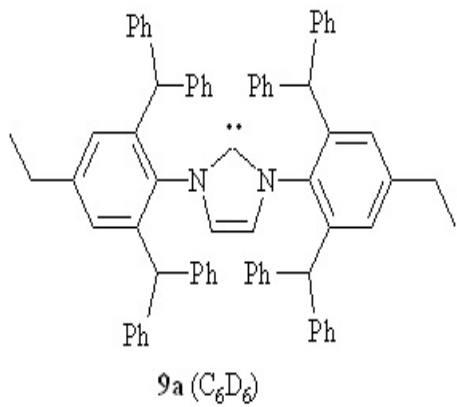












7.39
7.38
7.17
7.11
7.04
6.99

6.04
5.81

2.23
2.21

1.42

0.87



2.04
1.46
2.14
6.15

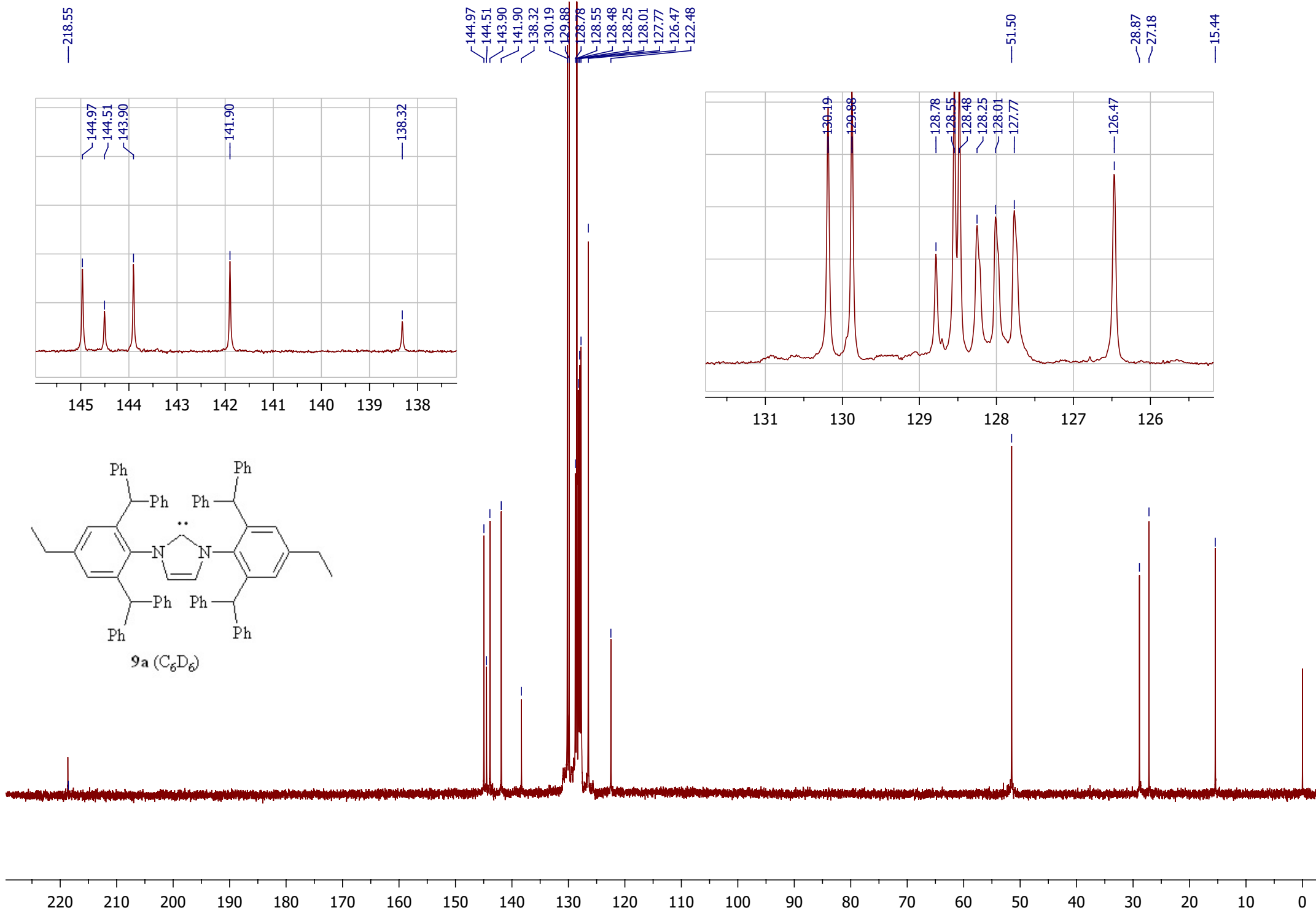
1.00
0.50

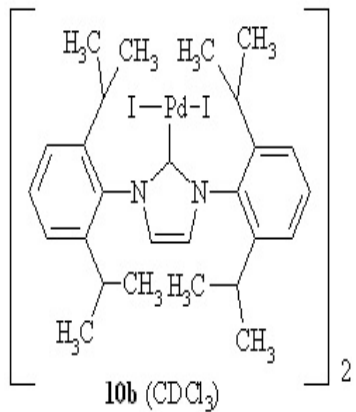
1.01

3.28

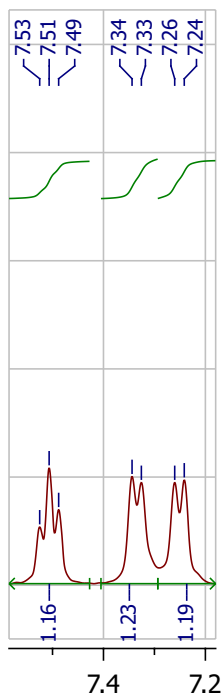
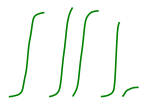
1.55

10.0 9.5 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

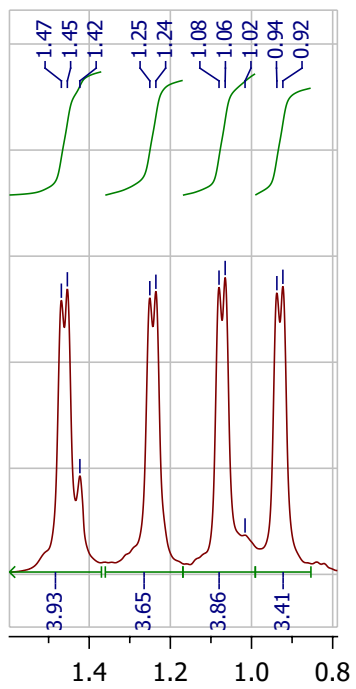




7.53
7.51
7.49
7.34
7.33
7.26
7.24
7.09



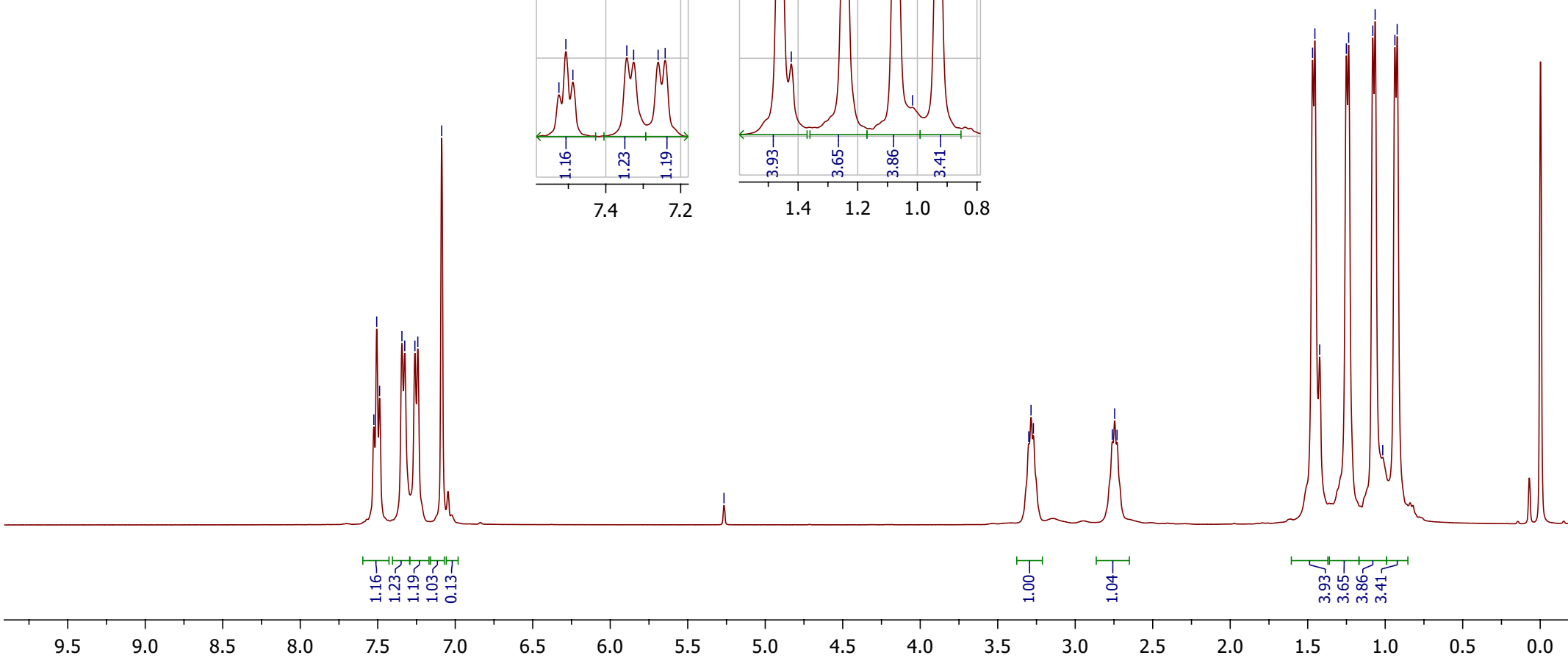
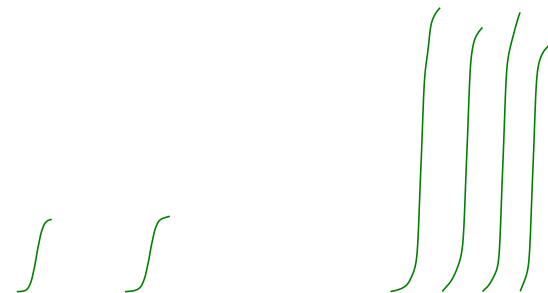
5.27

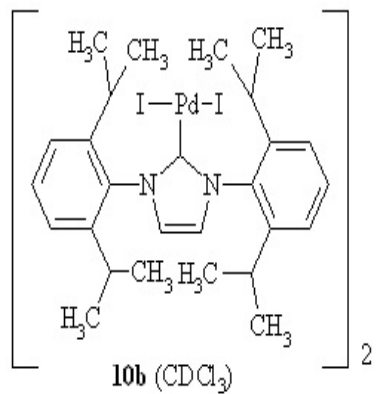


3.30
3.29
3.27

2.76
2.75
2.73

1.47
1.45
1.42
1.25
1.24
1.08
1.06
1.02
0.94
0.92





—165.44

146.33
145.92

—135.34

—130.25

125.38

124.65

124.24

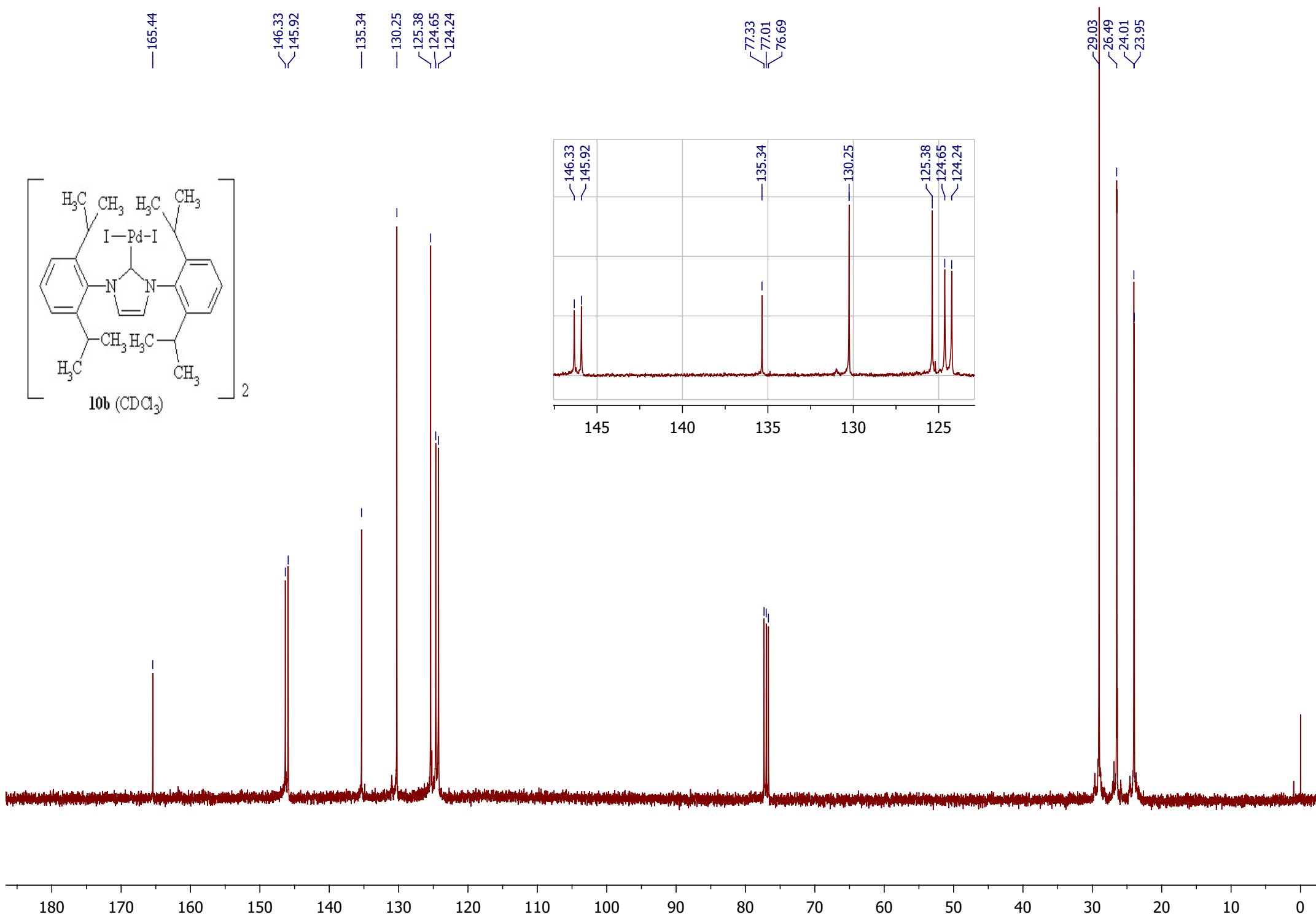
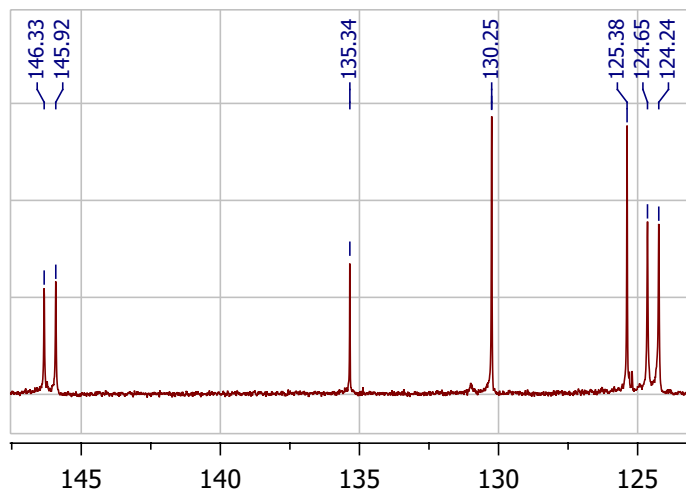
77.33
77.01
76.69

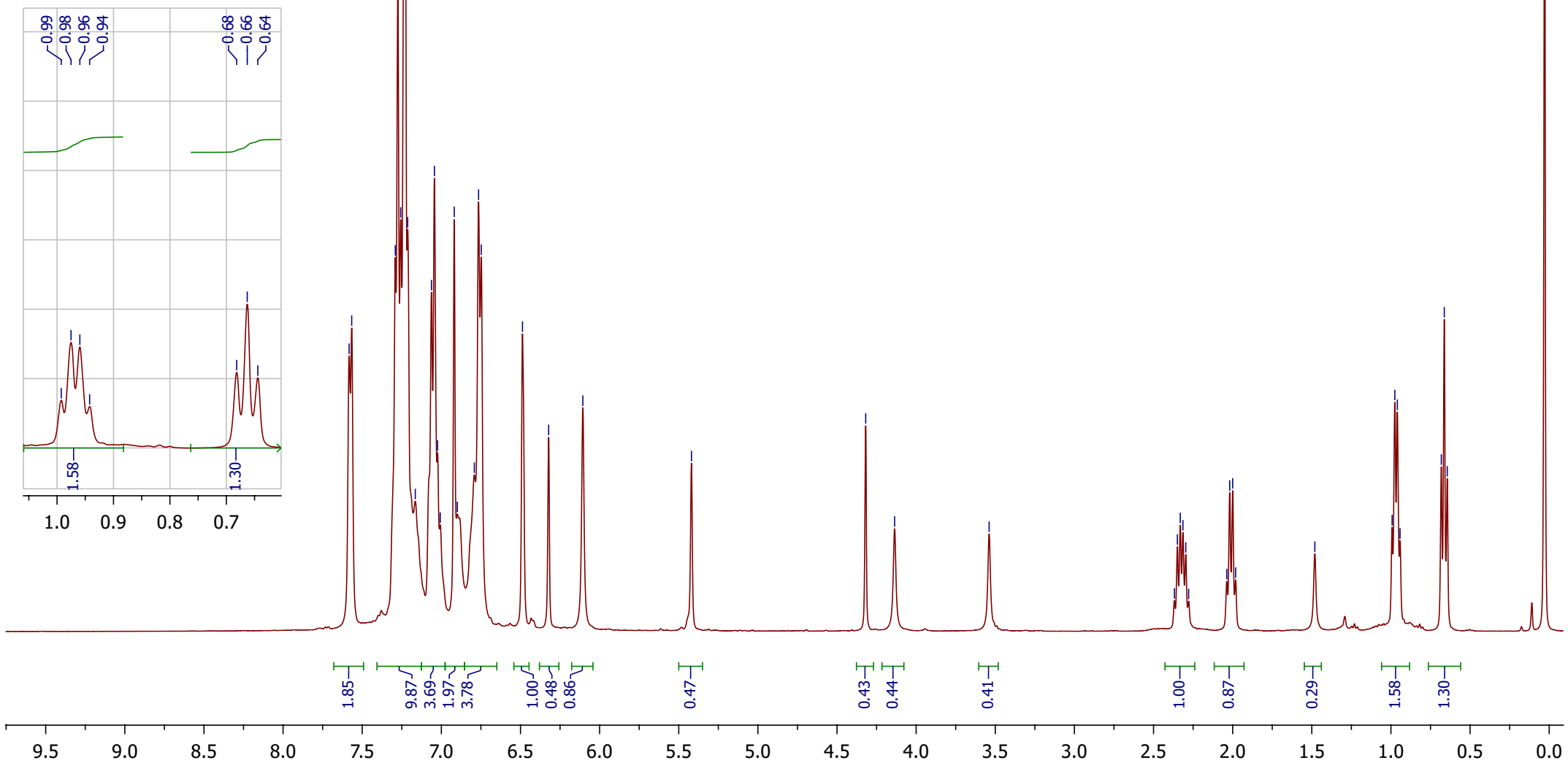
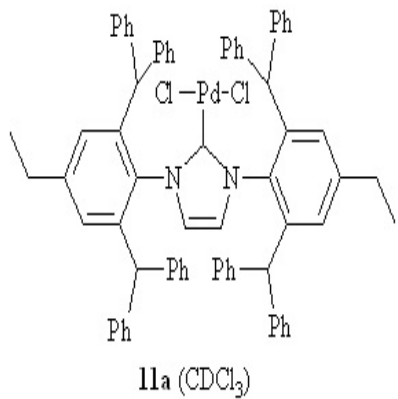
29.03

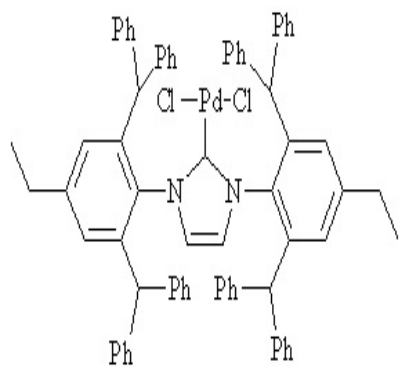
26.49

24.01

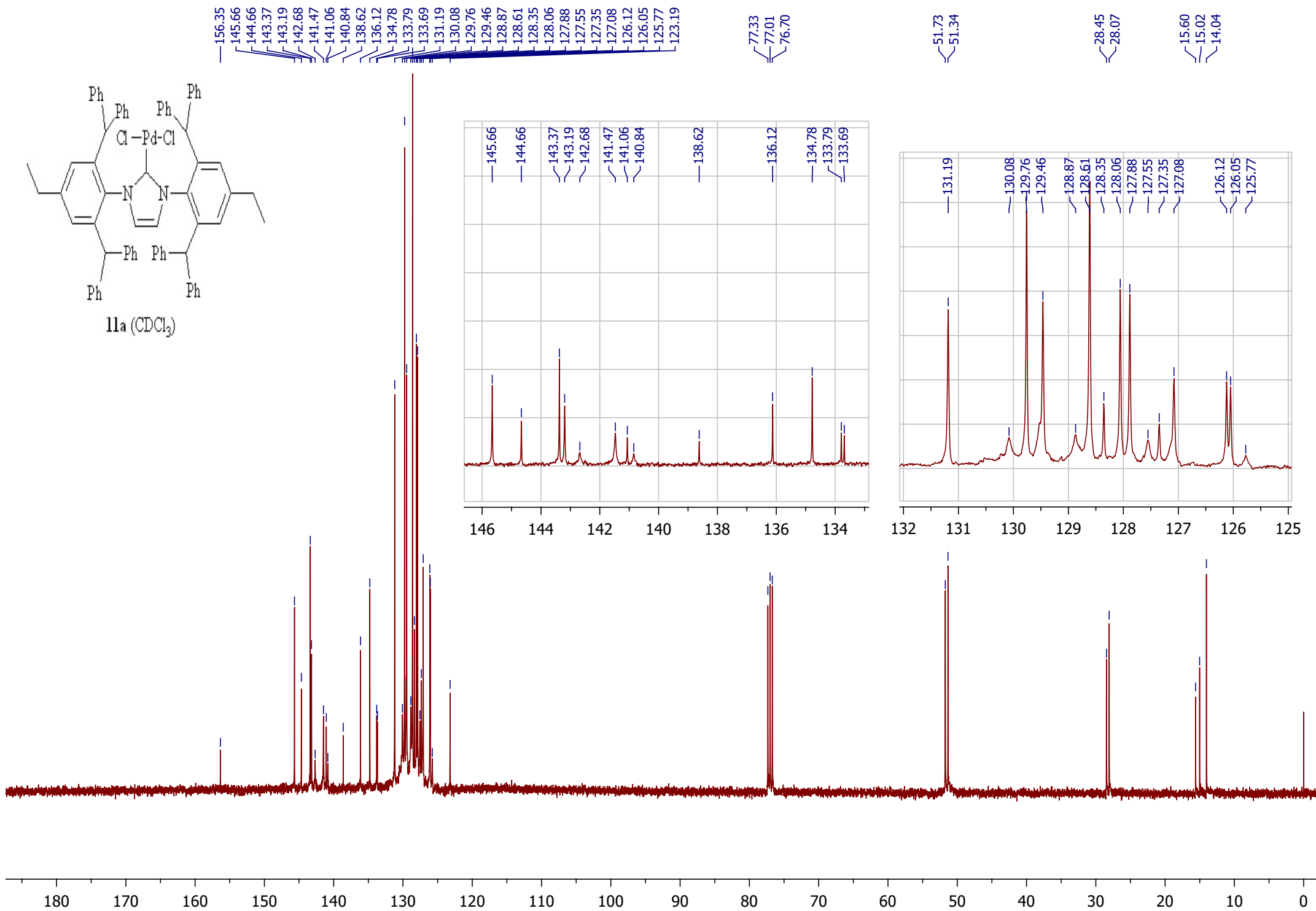
23.95

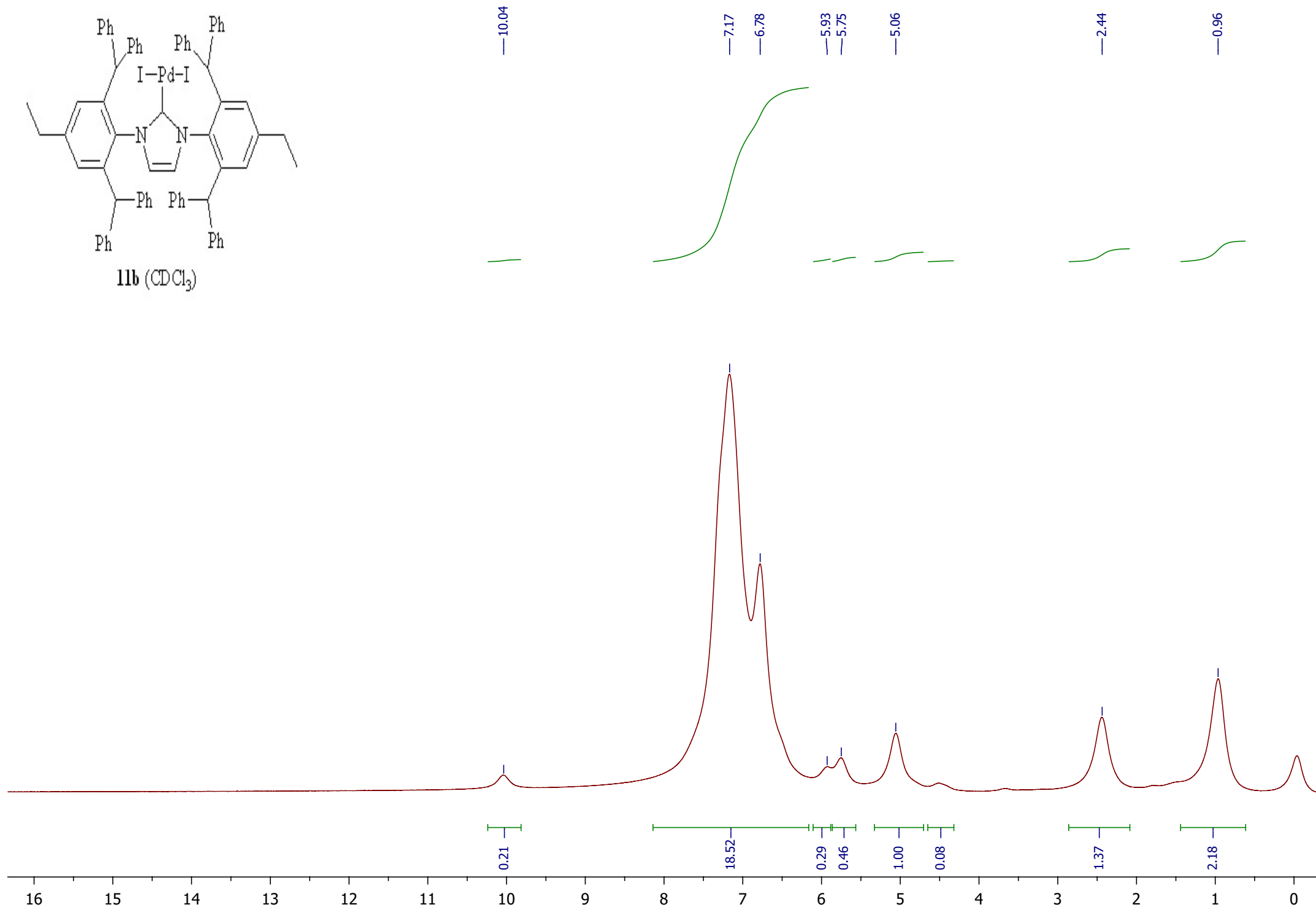
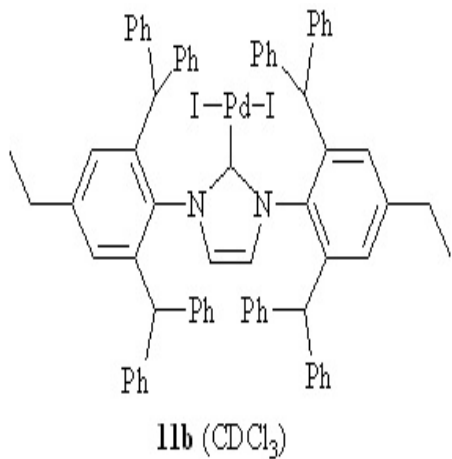


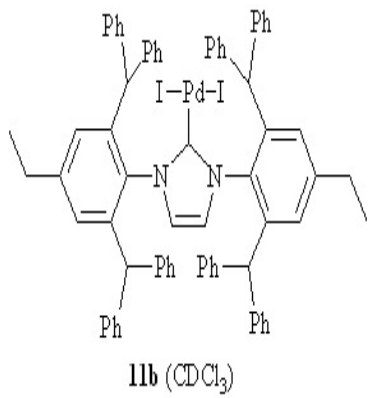




IIa (CDCl₃)







—155.44
—149.09
—143.07
—142.23
—141.39
—138.19
—131.97
—130.85
—129.99
—129.25
—128.57
—128.30
—125.43

—78.07

—52.61

—29.83

—16.06

