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Supporting Information

NH-Type of chiral Ni(II) complexes of glycine Schiff base: design, structural evaluation, reactivity and synthetic applications

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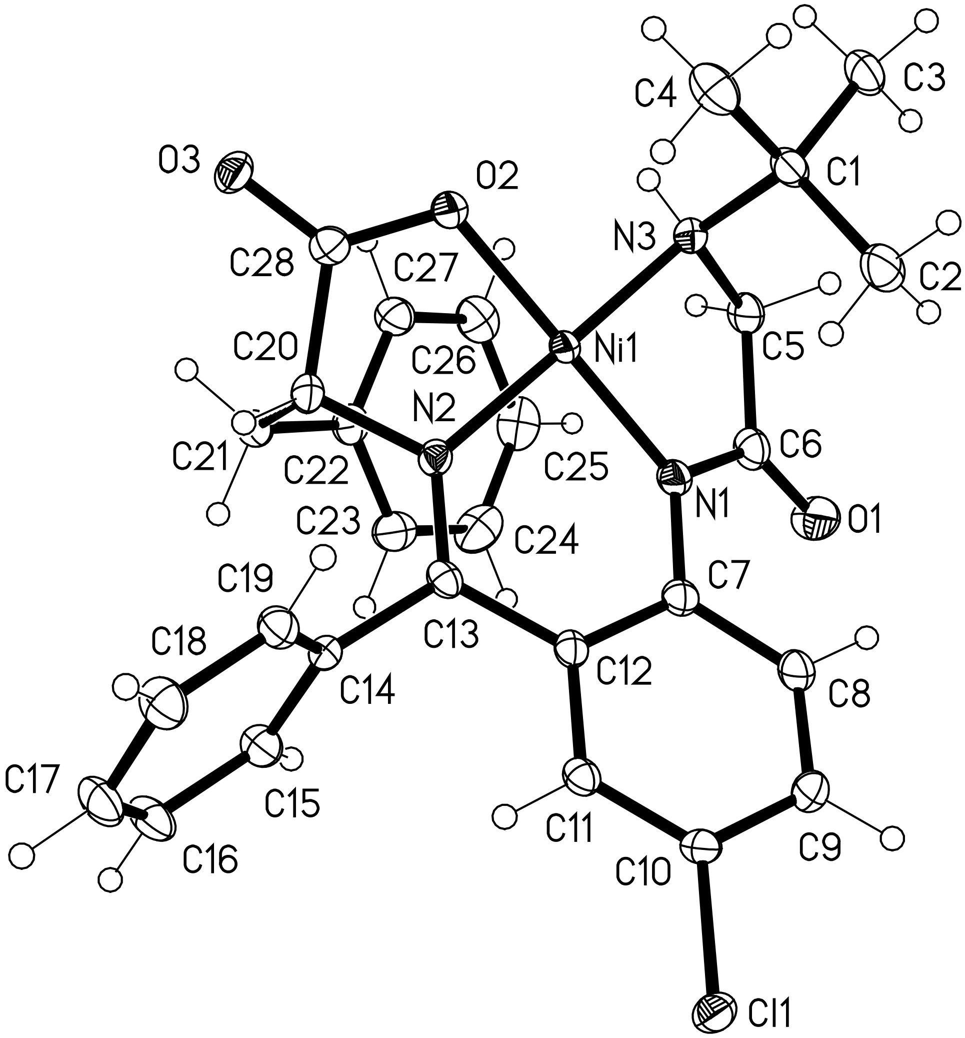
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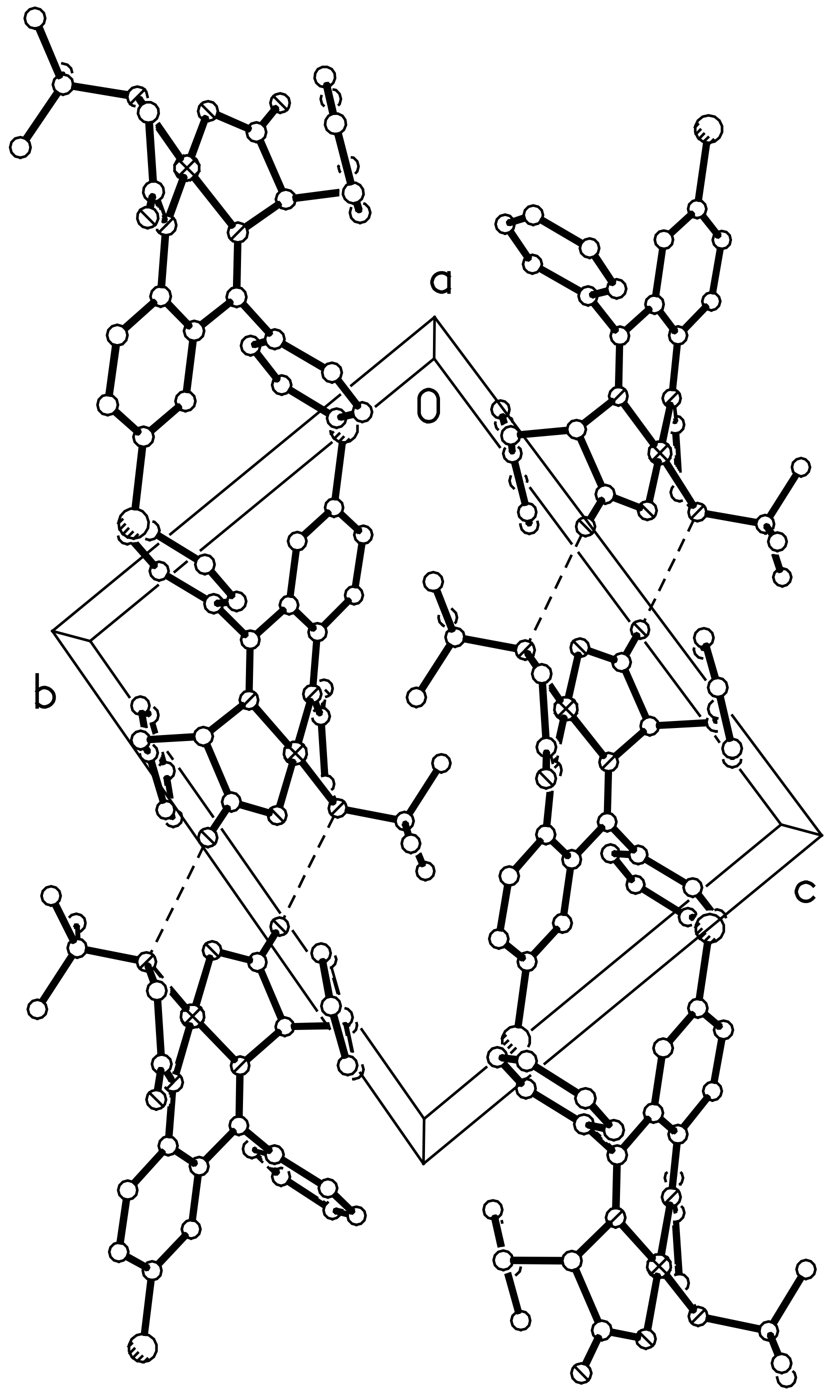
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Comment

The displacement ellipsoids were drawn at the 50% probability level.

Experimental

A red prism-shaped crystal of dimensions 0.47 x 0.15 x 0.13 mm was selected for structural analysis. Intensity data for this compound were collected using an instrument with a Bruker APEX ccd area detector (1) with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The sample was cooled to 100(2) K. Cell parameters were determined from a non-linear least squares fit of 7600 peaks in the range 2.27 < θ < 28.27°. A total of 9916 data were measured in the range 1.98 < θ < 26.00° using ω oscillation frames. The data were corrected for absorption by the semi-empirical method (2) giving minimum and maximum transmission factors of 0.6730 and 0.8903. The data were merged to form a set of 4890 independent data with R(int) = 0.0200 and a coverage of 98.7 %.

The triclinic space group $P\bar{1}$ was determined by statistical tests and verified by subsequent refinement. The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 (3). Hydrogen atom positions were initially determined by geometry and refined by a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom displacement parameters were set to 1.2 (1.5 for methyl) times the displacement parameters of the bonded atoms. A total of 331 parameters were refined against 4890 data to give wR(F^2) = 0.0813 and S = 1.006 for weights of $w = 1/[\sigma^2 (F^2) + (0.0420\ P)^2 + 0.9000\ P]$, where $P = [F_o^2 + 2F_c^2] / 3$. The final R(F) was 0.0321 for the 4458 observed, [$F > 4\sigma(F)$], data. The largest shift/s.u. was 0.001 in the final refinement cycle. The final difference map had maxima and minima of 0.392 and -0.230 e/Å^3, respectively.

Acknowledgment

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References

(1) (a) Data Collection: SMART Software Reference Manual (1998). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA. (b) Data Reduction: SAINT Software Reference Manual (1998). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA.

(2) G. M. Sheldrick (2002). SADABS. Program for Empirical Absorption Correction of Area Detector Data. University of Göttingen, Germany.

(3) (a) G. M. Sheldrick (2000). SHELXTL Version 6.10 Reference Manual. Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA. (b) *International Tables for Crystallography, Vol C,* Tables 6.1.1.4, 4.2.6.8, and 4.2.4.2, Kluwer: Boston (1995).

Table 1. Crystal data and structure refinement for 06087.

Empirical formula	$C_{28} H_{28} Cl N_3 Ni O_3$	
Formula weight	548.69	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 9.263(2) Å	α= 86.448(7)°
	b = 10.294(2) Å	β= 75.238(7)°
	c = 13.643(3) Å	γ= 89.471(7)°
Volume	1255.5(5) $Å^3$	
Z, Z'	2, 1	
Density (calculated)	1.451 Mg/m^3	
Wavelength	0.71073 Å	
Temperature	100(2) K	
F(000)	572	
Absorption coefficient	0.915 mm^{-1}	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8903 and 0.6730	
Theta range for data collection	1.98 to 26.00°	
Reflections collected	9916	
Independent reflections	4890 [R(int) = 0.0200]	
Data / restraints / parameters	4890 / 0 / 331	
$wR(F^2$ all data)	$wR2$ = 0.0813	
$R(F$ obsd data)	$R1$ = 0.0321	
Goodness-of-fit on F^2	1.006	
Observed data [$I > 2\sigma(I)$]	4458	
Largest and mean shift / s.u.	0.001and 0.000	
Largest diff. peak and hole	0.392 and -0.230 $e/Å^3$	

$wR2 = \{ \Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2] \}^{1/2}$

$R1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$

Table 2. Atomic coordinates and equivalent isotropic displacement parameters for 06087. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Ni(1)	0.56214(3)	0.24272(2)	0.612657(17)	0.01300(8)
Cl(1)	0.42132(5)	0.72839(5)	0.97526(4)	0.01921(11)
O(1)	0.92509(16)	0.36666(16)	0.67183(12)	0.0278(4)
O(2)	0.44743(15)	0.13973(13)	0.55160(10)	0.0170(3)
O(3)	0.24907(15)	0.00521(13)	0.59338(11)	0.0196(3)
N(1)	0.67491(17)	0.34274(16)	0.67261(12)	0.0159(3)
N(2)	0.40168(17)	0.23225(15)	0.72605(12)	0.0140(3)
N(3)	0.73452(18)	0.24374(16)	0.49762(12)	0.0152(3)
C(1)	0.7411(2)	0.34434(19)	0.41029(15)	0.0190(4)
C(2)	0.7531(3)	0.4809(2)	0.44548(17)	0.0273(5)
C(3)	0.8747(2)	0.3160(2)	0.32155(16)	0.0246(5)
C(4)	0.5980(2)	0.3300(2)	0.37652(16)	0.0258(5)
C(5)	0.8639(2)	0.2487(2)	0.54287(15)	0.0185(4)
C(6)	0.8258(2)	0.3273(2)	0.63658(15)	0.0192(4)
C(7)	0.6158(2)	0.42995(19)	0.74632(14)	0.0161(4)
C(8)	0.6983(2)	0.5400(2)	0.75767(15)	0.0208(4)
C(9)	0.6390(2)	0.6323(2)	0.82523(15)	0.0203(4)
C(10)	0.4953(2)	0.61422(18)	0.88689(14)	0.0161(4)
C(11)	0.4103(2)	0.50874(18)	0.87892(14)	0.0150(4)
C(12)	0.4671(2)	0.41570(18)	0.80752(14)	0.0140(4)
C(13)	0.3684(2)	0.30825(18)	0.80047(14)	0.0140(4)
C(14)	0.2224(2)	0.29316(17)	0.87907(14)	0.0144(4)
C(15)	0.2139(2)	0.24061(19)	0.97688(15)	0.0184(4)
C(16)	0.0767(2)	0.2343(2)	1.04830(15)	0.0227(4)
C(17)	-0.0504(2)	0.2801(2)	1.02286(16)	0.0234(4)
C(18)	-0.0424(2)	0.3306(2)	0.92512(16)	0.0224(4)
C(19)	0.0934(2)	0.33698(19)	0.85305(15)	0.0182(4)
C(20)	0.3130(2)	0.11428(18)	0.72536(14)	0.0167(4)
C(21)	0.3656(2)	0.00019(19)	0.78750(15)	0.0196(4)
C(22)	0.5339(2)	-0.01037(18)	0.76791(15)	0.0193(4)
C(23)	0.6037(3)	0.02920(19)	0.84022(16)	0.0231(4)
C(24)	0.7580(3)	0.0259(2)	0.82321(18)	0.0289(5)
C(25)	0.8449(3)	-0.0195(2)	0.73360(18)	0.0276(5)
C(26)	0.7768(2)	-0.0608(2)	0.66181(17)	0.0253(5)
C(27)	0.6225(2)	-0.05533(19)	0.67795(15)	0.0211(4)
C(28)	0.3352(2)	0.08263(18)	0.61454(15)	0.0165(4)

Table 3. Bond lengths [Å] and angles [°] for 06087.

Ni(1)-N(1)	1.8384(16)	C(9)-H(9)	0.9500
Ni(1)-N(2)	1.8506(16)	C(10)-C(11)	1.373(3)
Ni(1)-O(2)	1.8784(13)	C(11)-C(12)	1.410(3)
Ni(1)-N(3)	1.9324(17)	C(11)-H(11)	0.9500
Cl(1)-C(10)	1.745(2)	C(12)-C(13)	1.465(3)
O(1)-C(6)	1.224(2)	C(13)-C(14)	1.498(2)
O(2)-C(28)	1.287(2)	C(14)-C(15)	1.392(3)
O(3)-C(28)	1.230(2)	C(14)-C(19)	1.394(3)
N(1)-C(6)	1.370(2)	C(15)-C(16)	1.390(3)
N(1)-C(7)	1.393(3)	C(15)-H(15)	0.9500
N(2)-C(13)	1.293(3)	C(16)-C(17)	1.381(3)
N(2)-C(20)	1.474(2)	C(16)-H(16)	0.9500
N(3)-C(5)	1.484(2)	C(17)-C(18)	1.385(3)
N(3)-C(1)	1.519(2)	C(17)-H(17)	0.9500
N(3)-H(3N)	0.80(2)	C(18)-C(19)	1.385(3)
C(1)-C(4)	1.521(3)	C(18)-H(18)	0.9500
C(1)-C(2)	1.527(3)	C(19)-H(19)	0.9500
C(1)-C(3)	1.535(3)	C(20)-C(28)	1.528(3)
C(2)-H(2A)	0.9800	C(20)-C(21)	1.553(3)
C(2)-H(2B)	0.9800	C(20)-H(20)	1.0000
C(2)-H(2C)	0.9800	C(21)-C(22)	1.517(3)
C(3)-H(3A)	0.9800	C(21)-H(21A)	0.9900
C(3)-H(3B)	0.9800	C(21)-H(21B)	0.9900
C(3)-H(3C)	0.9800	C(22)-C(23)	1.392(3)
C(4)-H(4A)	0.9800	C(22)-C(27)	1.394(3)
C(4)-H(4B)	0.9800	C(23)-C(24)	1.389(3)
C(4)-H(4C)	0.9800	C(23)-H(23)	0.9500
C(5)-C(6)	1.519(3)	C(24)-C(25)	1.387(3)
C(5)-H(5A)	0.9900	C(24)-H(24)	0.9500
C(5)-H(5B)	0.9900	C(25)-C(26)	1.381(3)
C(7)-C(8)	1.410(3)	C(25)-H(25)	0.9500
C(7)-C(12)	1.421(3)	C(26)-C(27)	1.392(3)
C(8)-C(9)	1.377(3)	C(26)-H(26)	0.9500
C(8)-H(8)	0.9500	C(27)-H(27)	0.9500
C(9)-C(10)	1.388(3)		

N(1)-Ni(1)-N(2)	93.68(7)	C(7)-N(1)-Ni(1)	124.27(13)
N(1)-Ni(1)-O(2)	179.70(7)	C(13)-N(2)-C(20)	121.53(16)
N(2)-Ni(1)-O(2)	86.35(6)	C(13)-N(2)-Ni(1)	128.71(13)
N(1)-Ni(1)-N(3)	86.36(7)	C(20)-N(2)-Ni(1)	109.68(12)
N(2)-Ni(1)-N(3)	176.38(7)	C(5)-N(3)-C(1)	114.02(15)
O(2)-Ni(1)-N(3)	93.59(7)	C(5)-N(3)-Ni(1)	104.40(12)
C(28)-O(2)-Ni(1)	114.18(12)	C(1)-N(3)-Ni(1)	118.10(12)
C(6)-N(1)-C(7)	121.64(16)	C(5)-N(3)-H(3N)	110.4(17)
C(6)-N(1)-Ni(1)	114.08(13)	C(1)-N(3)-H(3N)	105.6(17)

Ni(1)-N(3)-H(3N)	103.9(17)	C(12)-C(11)-H(11)	119.7
N(3)-C(1)-C(4)	107.10(16)	C(11)-C(12)-C(7)	119.04(17)
N(3)-C(1)-C(2)	109.98(16)	C(11)-C(12)-C(13)	117.76(17)
C(4)-C(1)-C(2)	110.51(18)	C(7)-C(12)-C(13)	123.20(17)
N(3)-C(1)-C(3)	109.46(16)	N(2)-C(13)-C(12)	121.42(17)
C(4)-C(1)-C(3)	108.85(17)	N(2)-C(13)-C(14)	120.49(16)
C(2)-C(1)-C(3)	110.86(17)	C(12)-C(13)-C(14)	117.99(16)
C(1)-C(2)-H(2A)	109.5	C(15)-C(14)-C(19)	119.90(18)
C(1)-C(2)-H(2B)	109.5	C(15)-C(14)-C(13)	121.79(17)
H(2A)-C(2)-H(2B)	109.5	C(19)-C(14)-C(13)	118.28(17)
C(1)-C(2)-H(2C)	109.5	C(16)-C(15)-C(14)	119.51(18)
H(2A)-C(2)-H(2C)	109.5	C(16)-C(15)-H(15)	120.2
H(2B)-C(2)-H(2C)	109.5	C(14)-C(15)-H(15)	120.2
C(1)-C(3)-H(3A)	109.5	C(17)-C(16)-C(15)	120.45(18)
C(1)-C(3)-H(3B)	109.5	C(17)-C(16)-H(16)	119.8
H(3A)-C(3)-H(3B)	109.5	C(15)-C(16)-H(16)	119.8
C(1)-C(3)-H(3C)	109.5	C(16)-C(17)-C(18)	120.07(19)
H(3A)-C(3)-H(3C)	109.5	C(16)-C(17)-H(17)	120.0
H(3B)-C(3)-H(3C)	109.5	C(18)-C(17)-H(17)	120.0
C(1)-C(4)-H(4A)	109.5	C(19)-C(18)-C(17)	120.11(19)
C(1)-C(4)-H(4B)	109.5	C(19)-C(18)-H(18)	119.9
H(4A)-C(4)-H(4B)	109.5	C(17)-C(18)-H(18)	119.9
C(1)-C(4)-H(4C)	109.5	C(18)-C(19)-C(14)	119.94(18)
H(4A)-C(4)-H(4C)	109.5	C(18)-C(19)-H(19)	120.0
H(4B)-C(4)-H(4C)	109.5	C(14)-C(19)-H(19)	120.0
N(3)-C(5)-C(6)	110.42(16)	N(2)-C(20)-C(28)	107.32(15)
N(3)-C(5)-H(5A)	109.6	N(2)-C(20)-C(21)	109.91(15)
C(6)-C(5)-H(5A)	109.6	C(28)-C(20)-C(21)	111.06(16)
N(3)-C(5)-H(5B)	109.6	N(2)-C(20)-H(20)	109.5
C(6)-C(5)-H(5B)	109.6	C(28)-C(20)-H(20)	109.5
H(5A)-C(5)-H(5B)	108.1	C(21)-C(20)-H(20)	109.5
O(1)-C(6)-N(1)	127.81(19)	C(22)-C(21)-C(20)	114.06(16)
O(1)-C(6)-C(5)	120.26(18)	C(22)-C(21)-H(21A)	108.7
N(1)-C(6)-C(5)	111.92(16)	C(20)-C(21)-H(21A)	108.7
N(1)-C(7)-C(8)	120.89(17)	C(22)-C(21)-H(21B)	108.7
N(1)-C(7)-C(12)	121.01(17)	C(20)-C(21)-H(21B)	108.7
C(8)-C(7)-C(12)	117.96(18)	H(21A)-C(21)-H(21B)	107.6
C(9)-C(8)-C(7)	122.11(19)	C(23)-C(22)-C(27)	118.4(2)
C(9)-C(8)-H(8)	118.9	C(23)-C(22)-C(21)	119.50(19)
C(7)-C(8)-H(8)	118.9	C(27)-C(22)-C(21)	122.05(18)
C(8)-C(9)-C(10)	119.02(18)	C(24)-C(23)-C(22)	121.2(2)
C(8)-C(9)-H(9)	120.5	C(24)-C(23)-H(23)	119.4
C(10)-C(9)-H(9)	120.5	C(22)-C(23)-H(23)	119.4
C(11)-C(10)-C(9)	121.15(18)	C(25)-C(24)-C(23)	119.9(2)
C(11)-C(10)-Cl(1)	119.55(15)	C(25)-C(24)-H(24)	120.1
C(9)-C(10)-Cl(1)	119.30(15)	C(23)-C(24)-H(24)	120.1
C(10)-C(11)-C(12)	120.64(18)	C(26)-C(25)-C(24)	119.5(2)
C(10)-C(11)-H(11)	119.7	C(26)-C(25)-H(25)	120.2

C(24)-C(25)-H(25)	120.2	C(26)-C(27)-H(27)	119.8
C(25)-C(26)-C(27)	120.6(2)	C(22)-C(27)-H(27)	119.8
C(25)-C(26)-H(26)	119.7	O(3)-C(28)-O(2)	126.06(18)
C(27)-C(26)-H(26)	119.7	O(3)-C(28)-C(20)	118.79(17)
C(26)-C(27)-C(22)	120.3(2)	O(2)-C(28)-C(20)	115.13(16)

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Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for 06087. The anisotropic displacement factor exponent takes the form:
$-2\pi^2[h^2 a^{*2} U_{11} + ... + 2hk a^* b^* U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ni(1)	11(1)	16(1)	11(1)	-1(1)	-1(1)	-3(1)
Cl(1)	18(1)	19(1)	21(1)	-6(1)	-4(1)	-1(1)
O(1)	15(1)	43(1)	27(1)	-9(1)	-6(1)	-4(1)
O(2)	15(1)	21(1)	15(1)	-2(1)	-2(1)	-3(1)
O(3)	15(1)	21(1)	23(1)	-7(1)	-4(1)	-4(1)
N(1)	13(1)	20(1)	14(1)	-1(1)	-2(1)	-3(1)
N(2)	13(1)	16(1)	14(1)	0(1)	-5(1)	-2(1)
N(3)	15(1)	15(1)	15(1)	-2(1)	-2(1)	-2(1)
C(1)	23(1)	18(1)	14(1)	0(1)	1(1)	-1(1)
C(2)	39(1)	16(1)	21(1)	-1(1)	2(1)	-1(1)
C(3)	25(1)	25(1)	18(1)	-1(1)	4(1)	-4(1)
C(4)	25(1)	30(1)	21(1)	5(1)	-4(1)	3(1)
C(5)	11(1)	24(1)	20(1)	-2(1)	-2(1)	-2(1)
C(6)	14(1)	23(1)	20(1)	1(1)	-3(1)	-3(1)
C(7)	17(1)	19(1)	13(1)	1(1)	-5(1)	-3(1)
C(8)	15(1)	28(1)	18(1)	-2(1)	-1(1)	-7(1)
C(9)	21(1)	22(1)	18(1)	-1(1)	-6(1)	-8(1)
C(10)	18(1)	17(1)	13(1)	-3(1)	-4(1)	0(1)
C(11)	14(1)	19(1)	12(1)	1(1)	-4(1)	-1(1)
C(12)	14(1)	17(1)	12(1)	3(1)	-5(1)	-3(1)
C(13)	13(1)	17(1)	12(1)	2(1)	-3(1)	1(1)
C(14)	15(1)	13(1)	14(1)	-3(1)	-2(1)	-3(1)
C(15)	14(1)	25(1)	17(1)	-1(1)	-5(1)	1(1)
C(16)	20(1)	34(1)	13(1)	2(1)	-2(1)	0(1)
C(17)	14(1)	34(1)	19(1)	-1(1)	2(1)	1(1)
C(18)	15(1)	29(1)	22(1)	0(1)	-5(1)	2(1)
C(19)	18(1)	22(1)	15(1)	1(1)	-4(1)	-2(1)
C(20)	14(1)	17(1)	18(1)	-2(1)	-2(1)	-4(1)
C(21)	25(1)	18(1)	15(1)	0(1)	-2(1)	-5(1)
C(22)	26(1)	13(1)	19(1)	3(1)	-6(1)	-2(1)
C(23)	32(1)	18(1)	20(1)	-1(1)	-9(1)	1(1)
C(24)	35(1)	24(1)	34(1)	1(1)	-21(1)	-1(1)
C(25)	22(1)	24(1)	38(1)	5(1)	-12(1)	2(1)
C(26)	27(1)	21(1)	26(1)	2(1)	-4(1)	4(1)
C(27)	25(1)	20(1)	18(1)	0(1)	-6(1)	-1(1)
C(28)	15(1)	17(1)	18(1)	-4(1)	-3(1)	2(1)

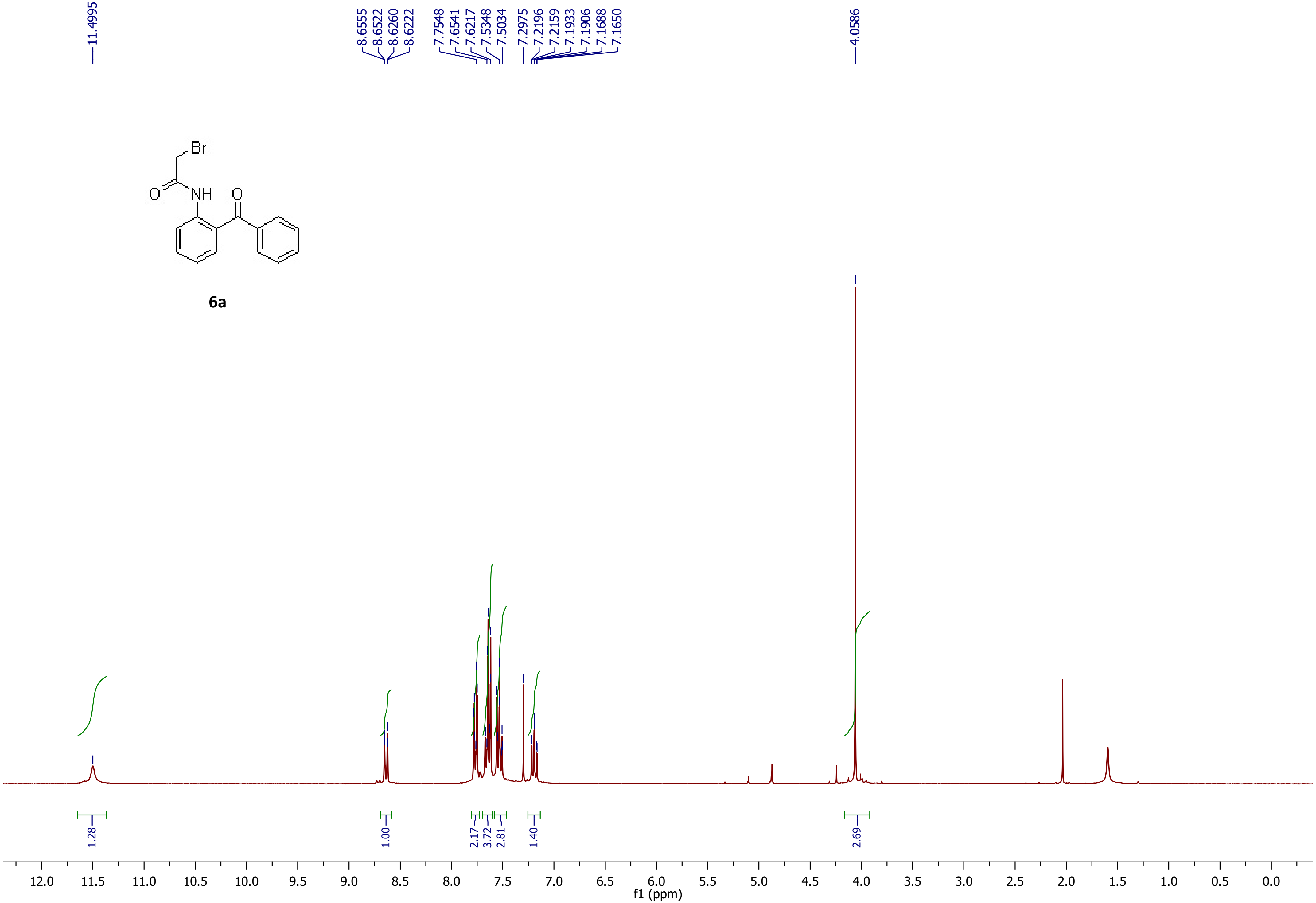
Table 5. Hydrogen coordinates and isotropic displacement parameters for 06087.

	x	y	z	U(eq)
H(3N)	0.730(3)	0.175(2)	0.4746(18)	0.018
H(2A)	0.6691	0.4955	0.5039	0.041
H(2B)	0.7508	0.5454	0.3900	0.041
H(2C)	0.8471	0.4893	0.4651	0.041
H(3A)	0.9676	0.3272	0.3420	0.037
H(3B)	0.8741	0.3764	0.2632	0.037
H(3C)	0.8676	0.2264	0.3027	0.037
H(4A)	0.5899	0.2409	0.3569	0.039
H(4B)	0.5998	0.3914	0.3184	0.039
H(4C)	0.5120	0.3484	0.4326	0.039
H(5A)	0.9506	0.2888	0.4923	0.022
H(5B)	0.8915	0.1591	0.5616	0.022
H(8)	0.7980	0.5508	0.7174	0.025
H(9)	0.6955	0.7073	0.8296	0.024
H(11)	0.3123	0.4983	0.9219	0.018
H(15)	0.3012	0.2093	0.9947	0.022
H(16)	0.0703	0.1982	1.1151	0.027
H(17)	-0.1435	0.2770	1.0725	0.028
H(18)	-0.1302	0.3608	0.9075	0.027
H(19)	0.0987	0.3712	0.7859	0.022
H(20)	0.2050	0.1318	0.7559	0.020
H(21A)	0.3226	0.0114	0.8607	0.024
H(21B)	0.3258	-0.0824	0.7711	0.024
H(23)	0.5448	0.0590	0.9023	0.028
H(24)	0.8040	0.0547	0.8729	0.035
H(25)	0.9505	-0.0222	0.7217	0.033
H(26)	0.8360	-0.0932	0.6008	0.030
H(27)	0.5772	-0.0824	0.6274	0.025

Table 6. Torsion angles [°] for 06087.

N(1)-Ni(1)-O(2)-C(28)	80(13)	Cl(1)-C(10)-C(11)-C(12)	179.46(14)
N(2)-Ni(1)-O(2)-C(28)	-15.81(13)	C(10)-C(11)-C(12)-C(7)	2.3(3)
N(3)-Ni(1)-O(2)-C(28)	160.56(13)	C(10)-C(11)-C(12)-C(13)	-177.75(17)
N(2)-Ni(1)-N(1)-C(6)	151.45(14)	N(1)-C(7)-C(12)-C(11)	-177.95(16)
O(2)-Ni(1)-N(1)-C(6)	56(13)	C(8)-C(7)-C(12)-C(11)	-2.2(3)
N(3)-Ni(1)-N(1)-C(6)	-24.92(14)	N(1)-C(7)-C(12)-C(13)	2.1(3)
N(2)-Ni(1)-N(1)-C(7)	-30.21(15)	C(8)-C(7)-C(12)-C(13)	177.88(17)
O(2)-Ni(1)-N(1)-C(7)	-126(13)	C(20)-N(2)-C(13)-C(12)	172.01(16)
N(3)-Ni(1)-N(1)-C(7)	153.42(15)	Ni(1)-N(2)-C(13)-C(12)	-4.3(3)
N(1)-Ni(1)-N(2)-C(13)	21.38(17)	C(20)-N(2)-C(13)-C(14)	-11.6(3)
O(2)-Ni(1)-N(2)-C(13)	-158.92(17)	Ni(1)-N(2)-C(13)-C(14)	172.16(13)
N(3)-Ni(1)-N(2)-C(13)	111.9(11)	C(11)-C(12)-C(13)-N(2)	167.87(17)
N(1)-Ni(1)-N(2)-C(20)	-155.26(12)	C(7)-C(12)-C(13)-N(2)	-12.2(3)
O(2)-Ni(1)-N(2)-C(20)	24.44(12)	C(11)-C(12)-C(13)-C(14)	-8.6(2)
N(3)-Ni(1)-N(2)-C(20)	-64.8(11)	C(7)-C(12)-C(13)-C(14)	171.28(16)
N(1)-Ni(1)-N(3)-C(5)	32.10(12)	N(2)-C(13)-C(14)-C(15)	106.8(2)
N(2)-Ni(1)-N(3)-C(5)	-58.6(11)	C(12)-C(13)-C(14)-C(15)	-76.6(2)
O(2)-Ni(1)-N(3)-C(5)	-147.60(12)	N(2)-C(13)-C(14)-C(19)	-75.1(2)
N(1)-Ni(1)-N(3)-C(1)	-95.74(14)	C(12)-C(13)-C(14)-C(19)	101.5(2)
N(2)-Ni(1)-N(3)-C(1)	173.5(10)	C(19)-C(14)-C(15)-C(16)	-1.0(3)
O(2)-Ni(1)-N(3)-C(1)	84.56(14)	C(13)-C(14)-C(15)-C(16)	177.04(18)
C(5)-N(3)-C(1)-C(4)	-177.40(16)	C(14)-C(15)-C(16)-C(17)	-0.2(3)
Ni(1)-N(3)-C(1)-C(4)	-54.27(19)	C(15)-C(16)-C(17)-C(18)	1.2(3)
C(5)-N(3)-C(1)-C(2)	-57.3(2)	C(16)-C(17)-C(18)-C(19)	-0.9(3)
Ni(1)-N(3)-C(1)-C(2)	65.85(19)	C(17)-C(18)-C(19)-C(14)	-0.3(3)
C(5)-N(3)-C(1)-C(3)	64.7(2)	C(15)-C(14)-C(19)-C(18)	1.3(3)
Ni(1)-N(3)-C(1)-C(3)	-172.12(13)	C(13)-C(14)-C(19)-C(18)	-176.83(18)
C(1)-N(3)-C(5)-C(6)	96.75(19)	C(13)-N(2)-C(20)-C(28)	155.64(17)
Ni(1)-N(3)-C(5)-C(6)	-33.55(17)	Ni(1)-N(2)-C(20)-C(28)	-27.44(17)
C(7)-N(1)-C(6)-O(1)	12.8(3)	C(13)-N(2)-C(20)-C(21)	-83.5(2)
Ni(1)-N(1)-C(6)-O(1)	-168.81(18)	Ni(1)-N(2)-C(20)-C(21)	93.44(15)
C(7)-N(1)-C(6)-C(5)	-168.48(16)	N(2)-C(20)-C(21)-C(22)	-43.1(2)
Ni(1)-N(1)-C(6)-C(5)	9.9(2)	C(28)-C(20)-C(21)-C(22)	75.5(2)
N(3)-C(5)-C(6)-O(1)	-164.11(18)	C(20)-C(21)-C(22)-C(23)	105.0(2)
N(3)-C(5)-C(6)-N(1)	17.1(2)	C(20)-C(21)-C(22)-C(27)	-73.1(2)
C(6)-N(1)-C(7)-C(8)	26.0(3)	C(27)-C(22)-C(23)-C(24)	0.8(3)
Ni(1)-N(1)-C(7)-C(8)	-152.26(15)	C(21)-C(22)-C(23)-C(24)	-177.38(18)
C(6)-N(1)-C(7)-C(12)	-158.41(18)	C(22)-C(23)-C(24)-C(25)	-1.1(3)
Ni(1)-N(1)-C(7)-C(12)	23.4(2)	C(23)-C(24)-C(25)-C(26)	0.2(3)
N(1)-C(7)-C(8)-C(9)	175.61(18)	C(24)-C(25)-C(26)-C(27)	1.0(3)
C(12)-C(7)-C(8)-C(9)	-0.2(3)	C(25)-C(26)-C(27)-C(22)	-1.3(3)
C(7)-C(8)-C(9)-C(10)	2.4(3)	C(23)-C(22)-C(27)-C(26)	0.3(3)
C(8)-C(9)-C(10)-C(11)	-2.3(3)	C(21)-C(22)-C(27)-C(26)	178.51(18)
C(8)-C(9)-C(10)-Cl(1)	178.17(15)	Ni(1)-O(2)-C(28)-O(3)	-175.89(16)
C(9)-C(10)-C(11)-C(12)	0.0(3)	Ni(1)-O(2)-C(28)-C(20)	2.6(2)

N(2)-C(20)-C(28)-O(3)	-165.04(17)
C(21)-C(20)-C(28)-O(3)	74.8(2)
N(2)-C(20)-C(28)-O(2)	16.3(2)
C(21)-C(20)-C(28)-O(2)	-103.83(19)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O
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O
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1.00
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1.40
2.69
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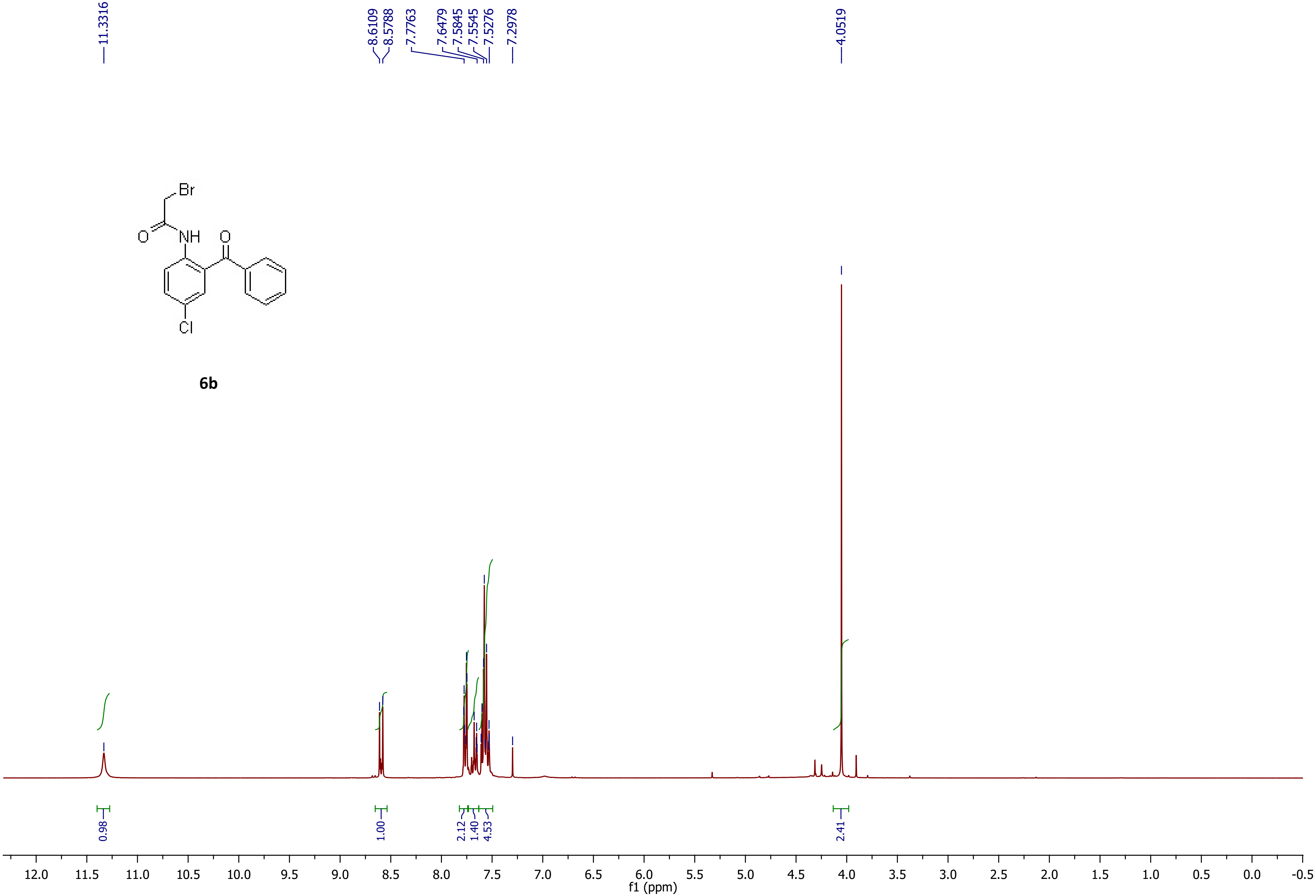
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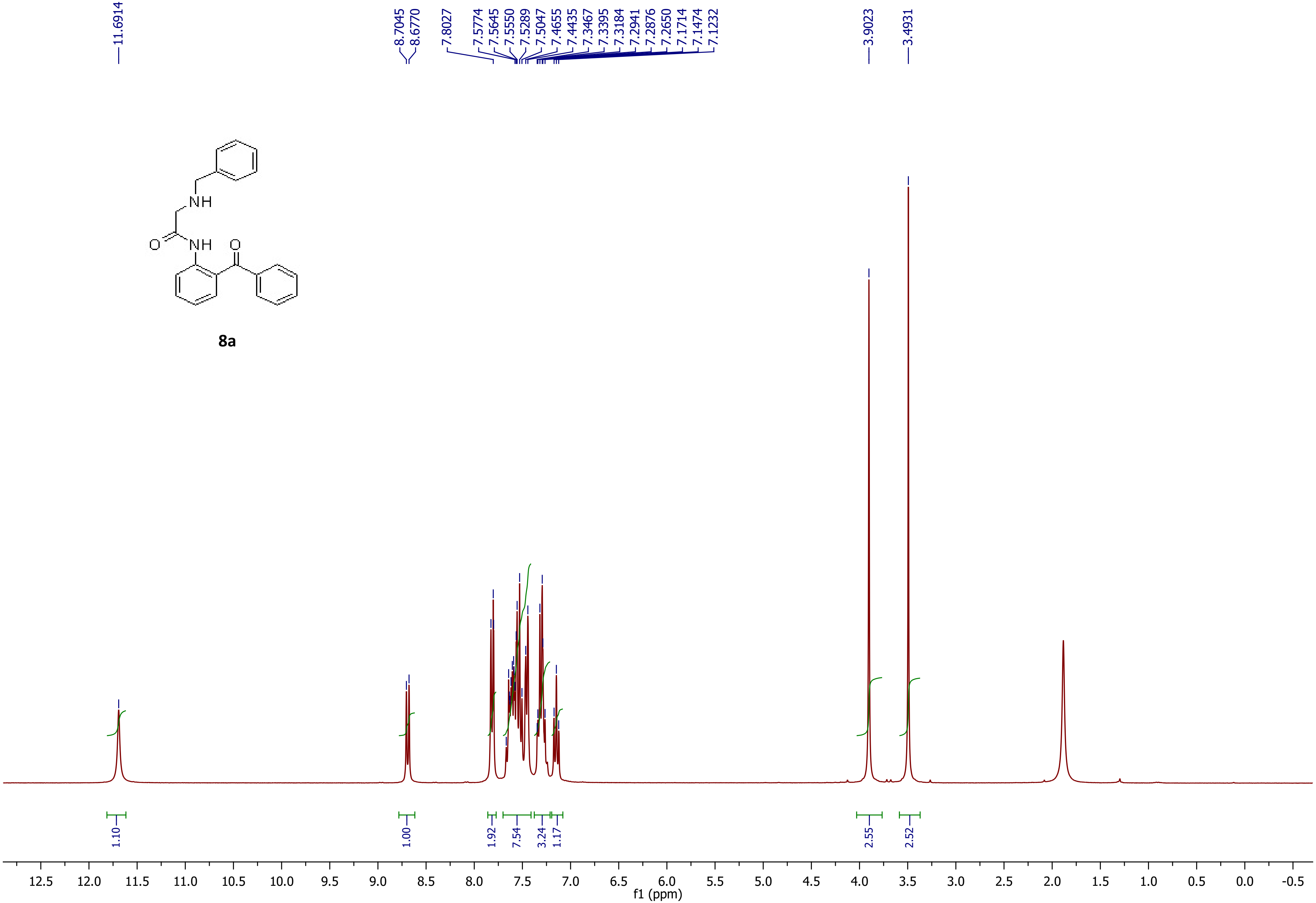
f1 (ppm)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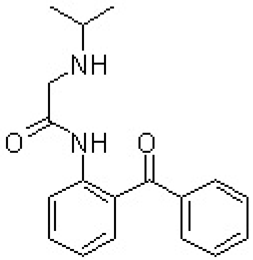
f1 (ppm)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NH
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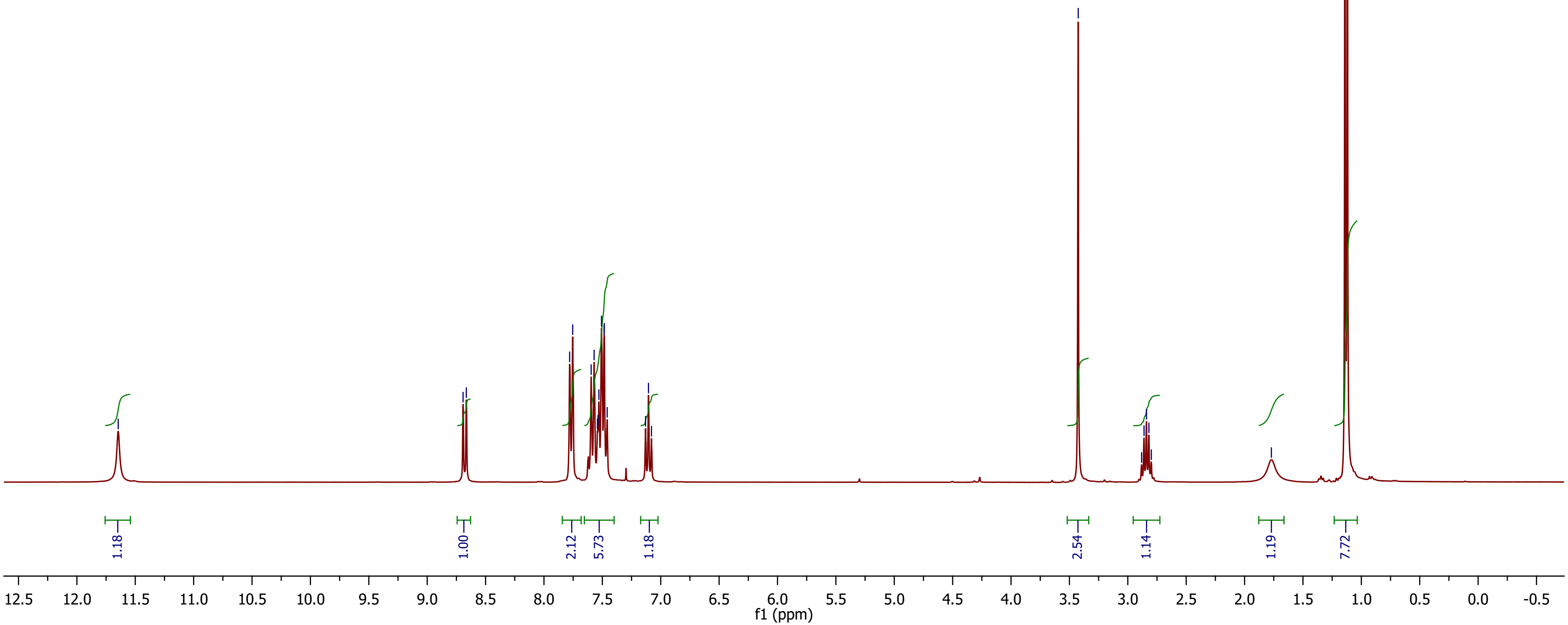
8b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7.72
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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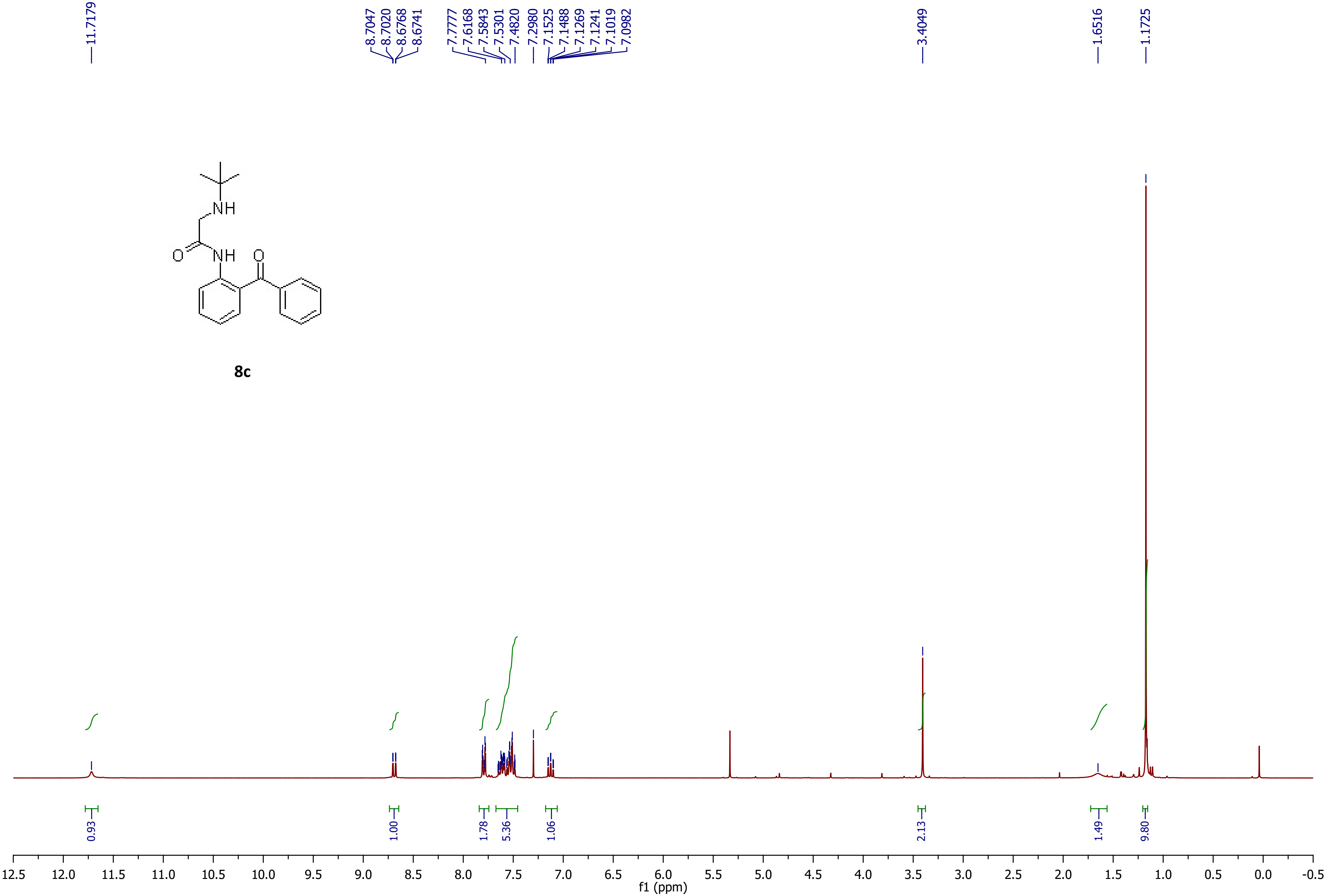
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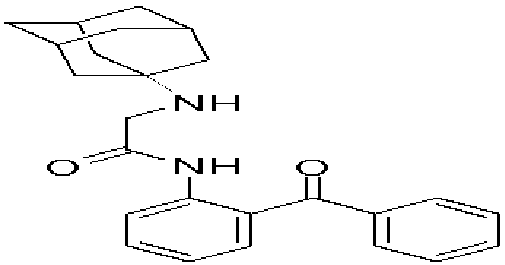
f1 (ppm)



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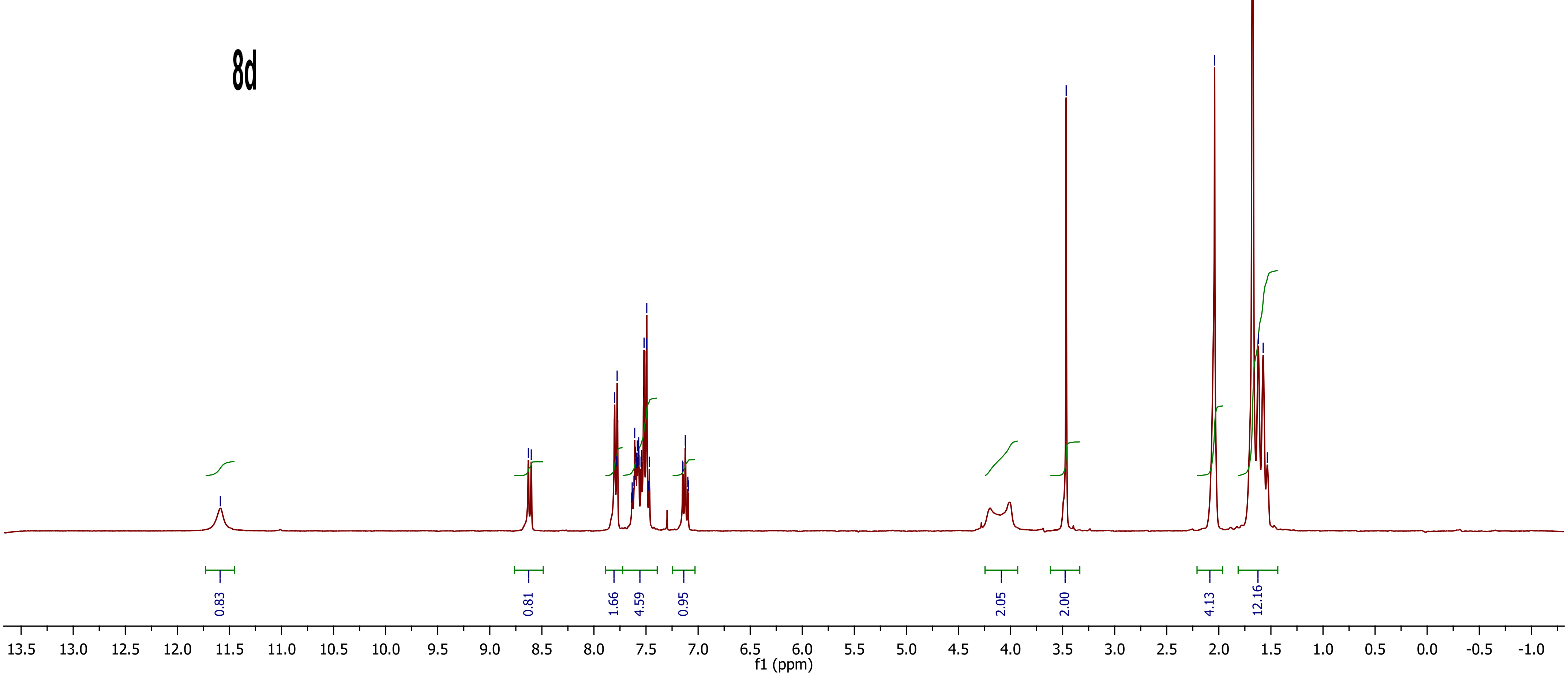
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1.5747
1.5345
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2.00
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11.0
10.5
10.0
9.5
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6.5
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5.0
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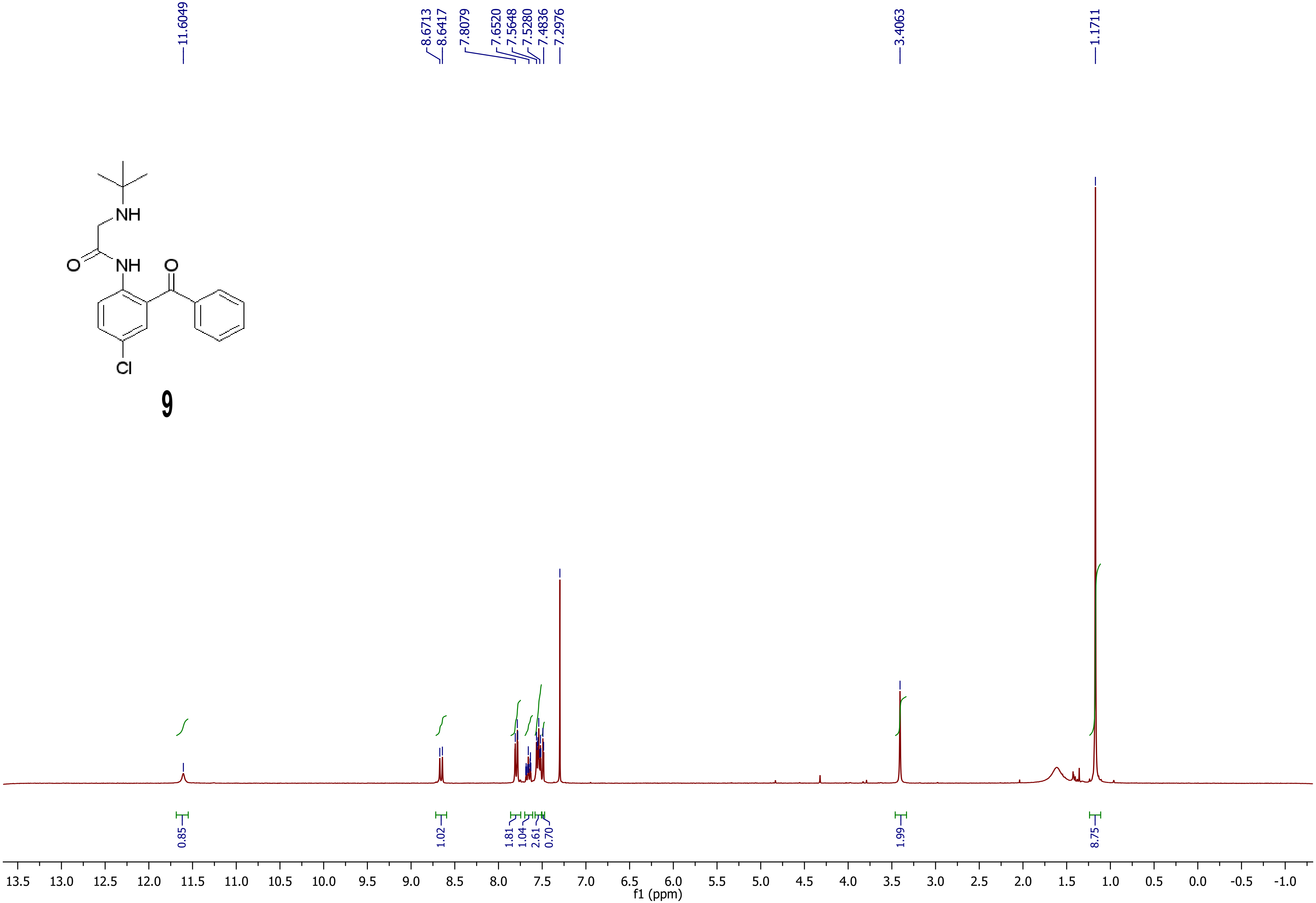
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1.02
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1.04
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6.5
6.0
5.5
5.0
4.5
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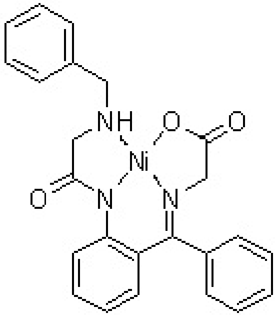

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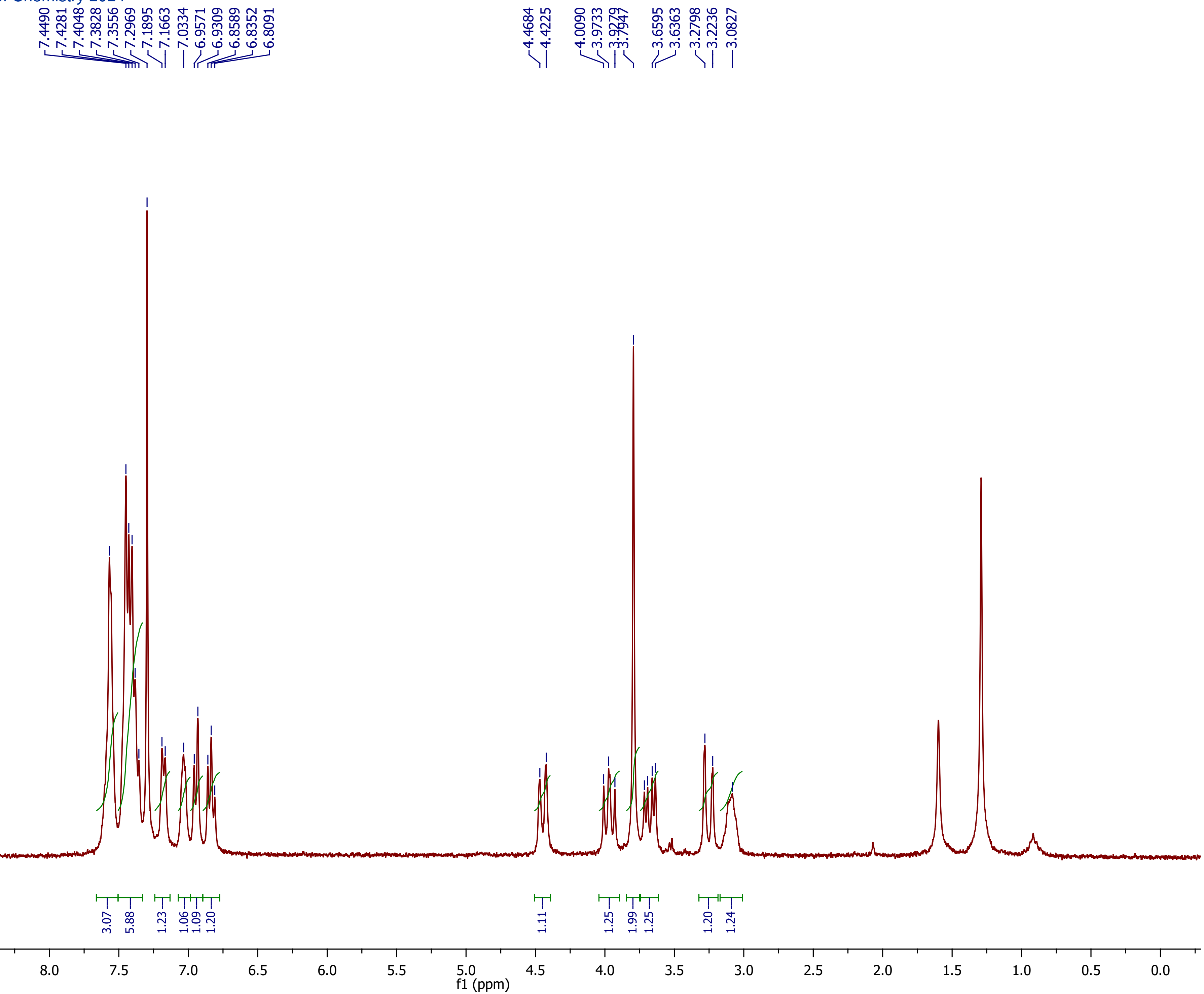
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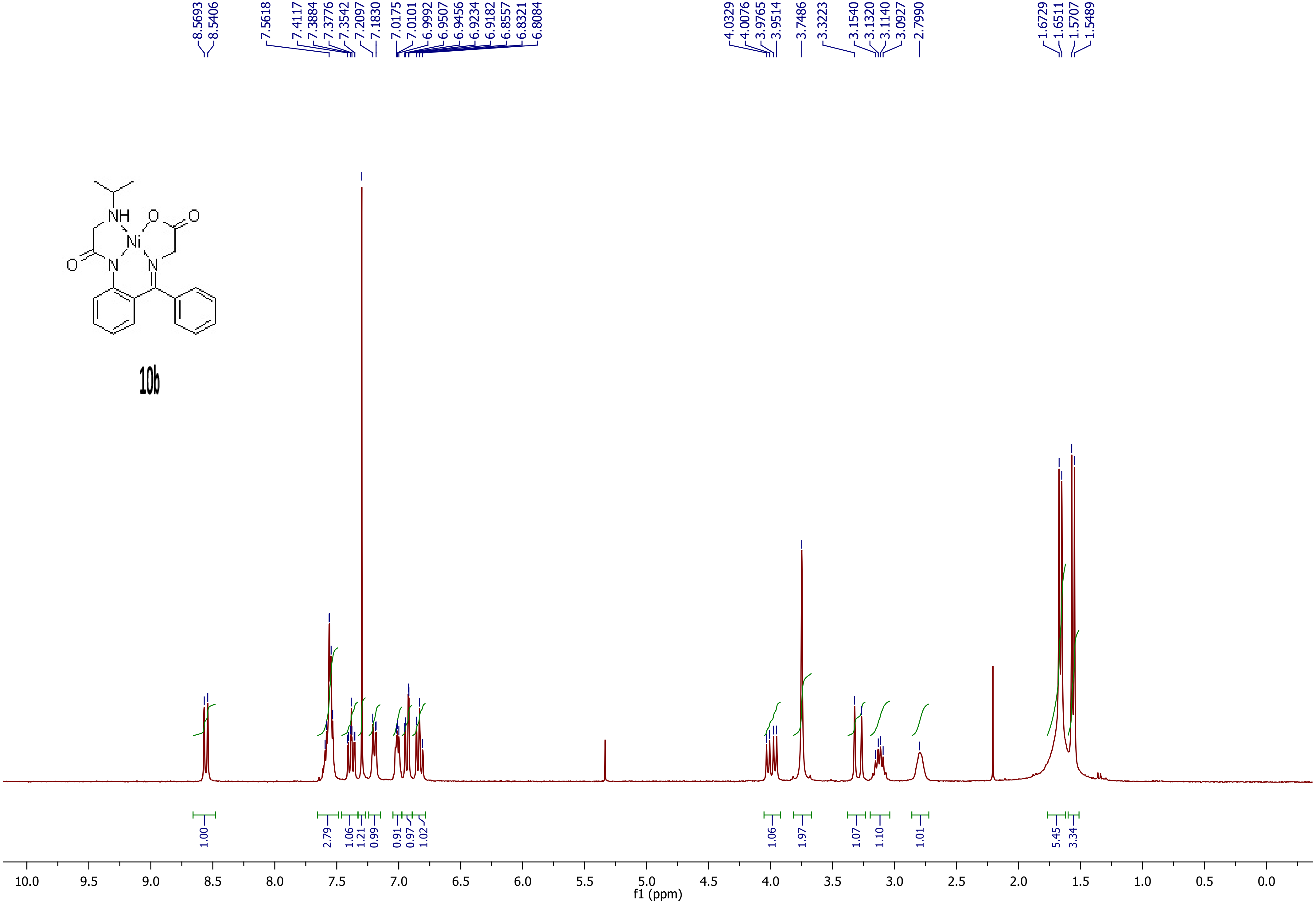

10b

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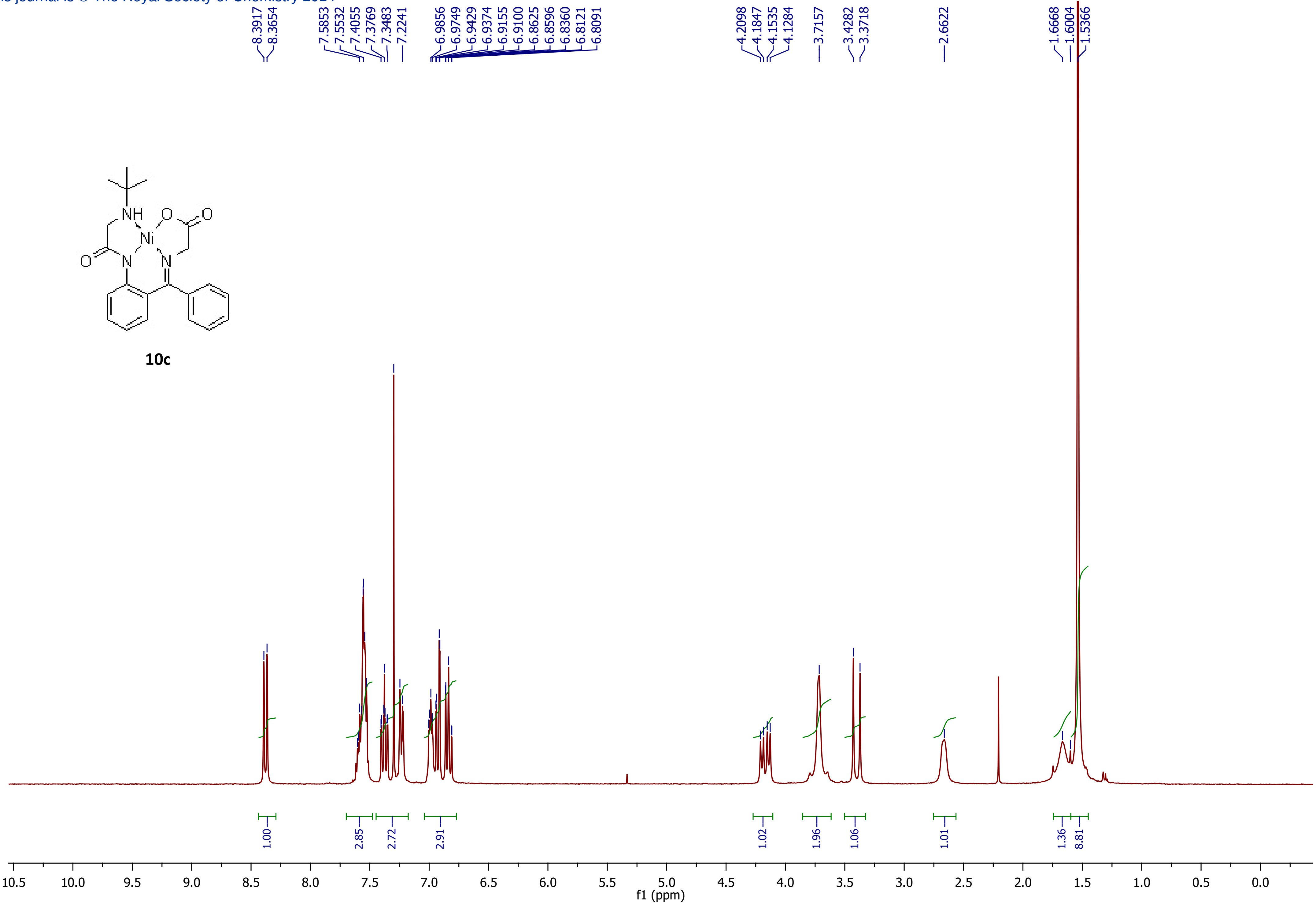
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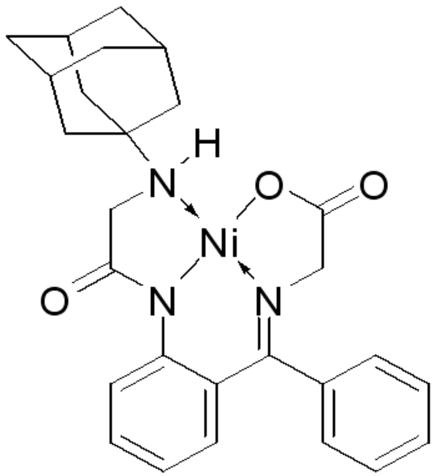
f1 (ppm)

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry



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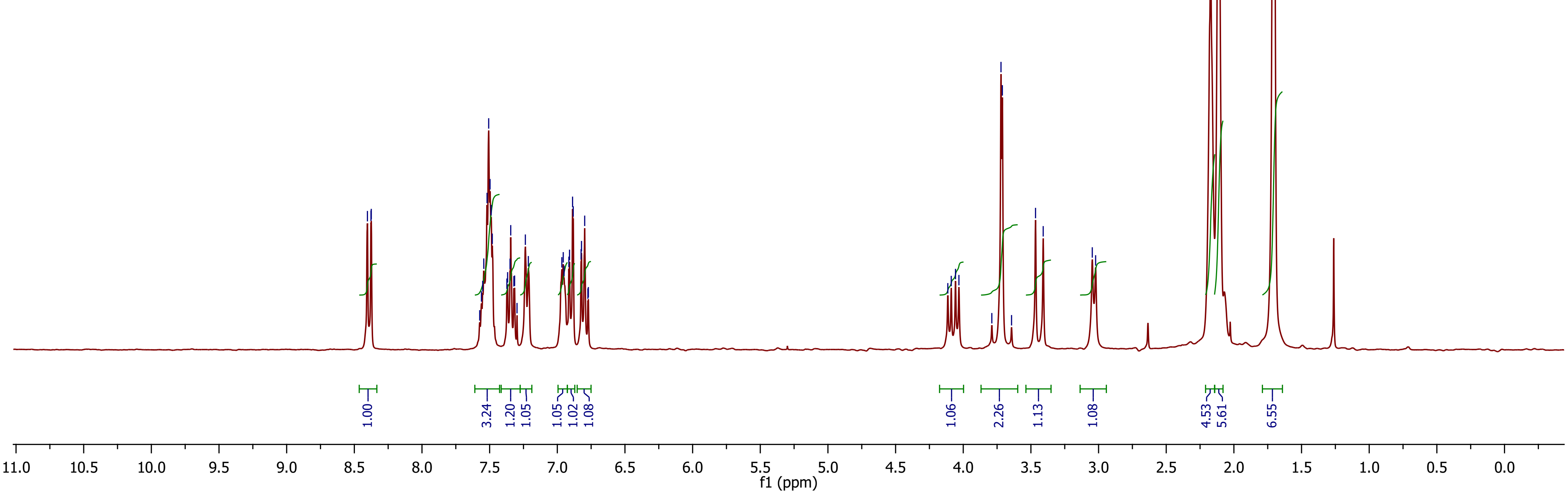

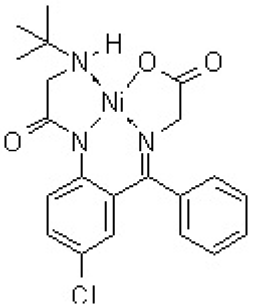

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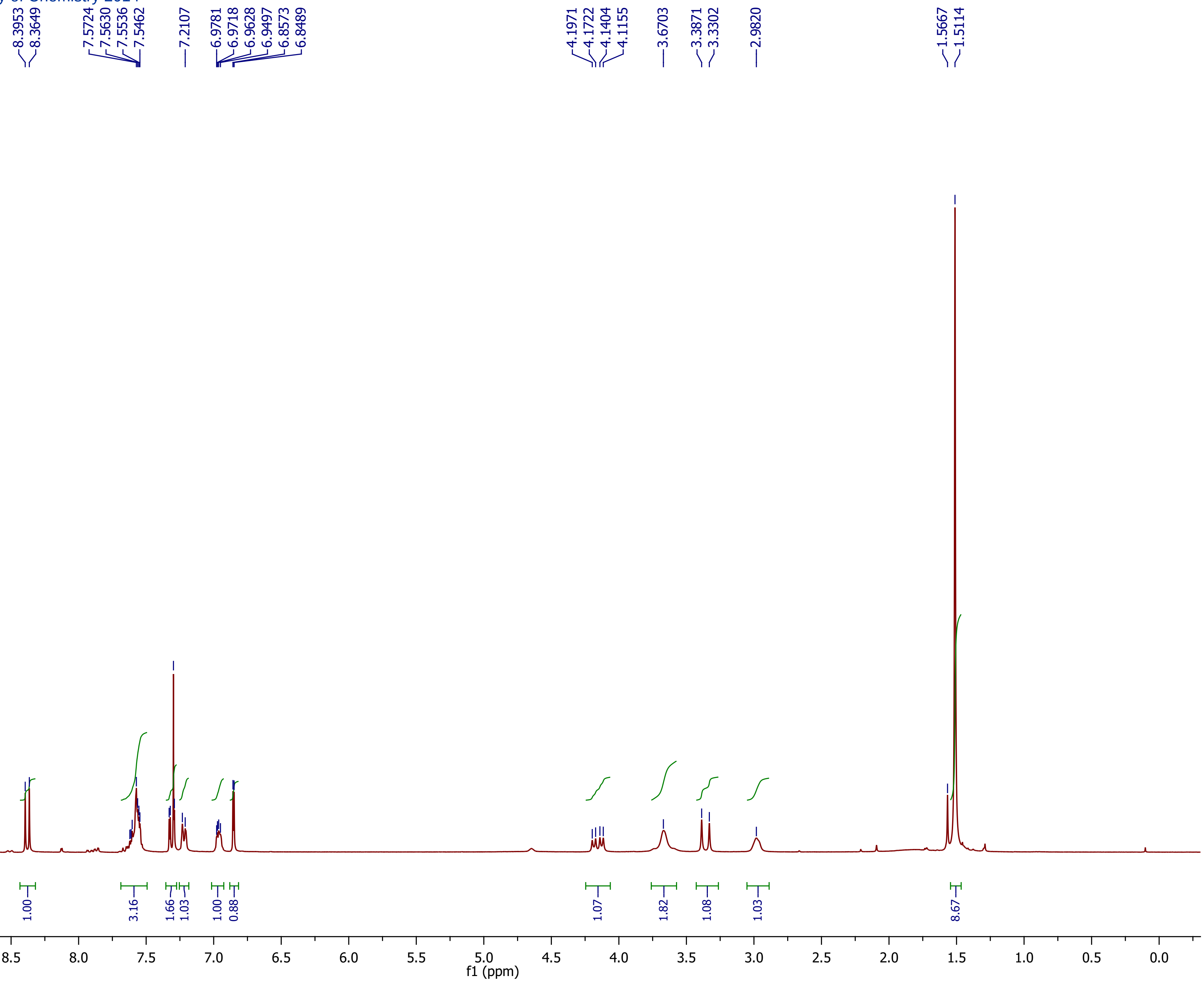
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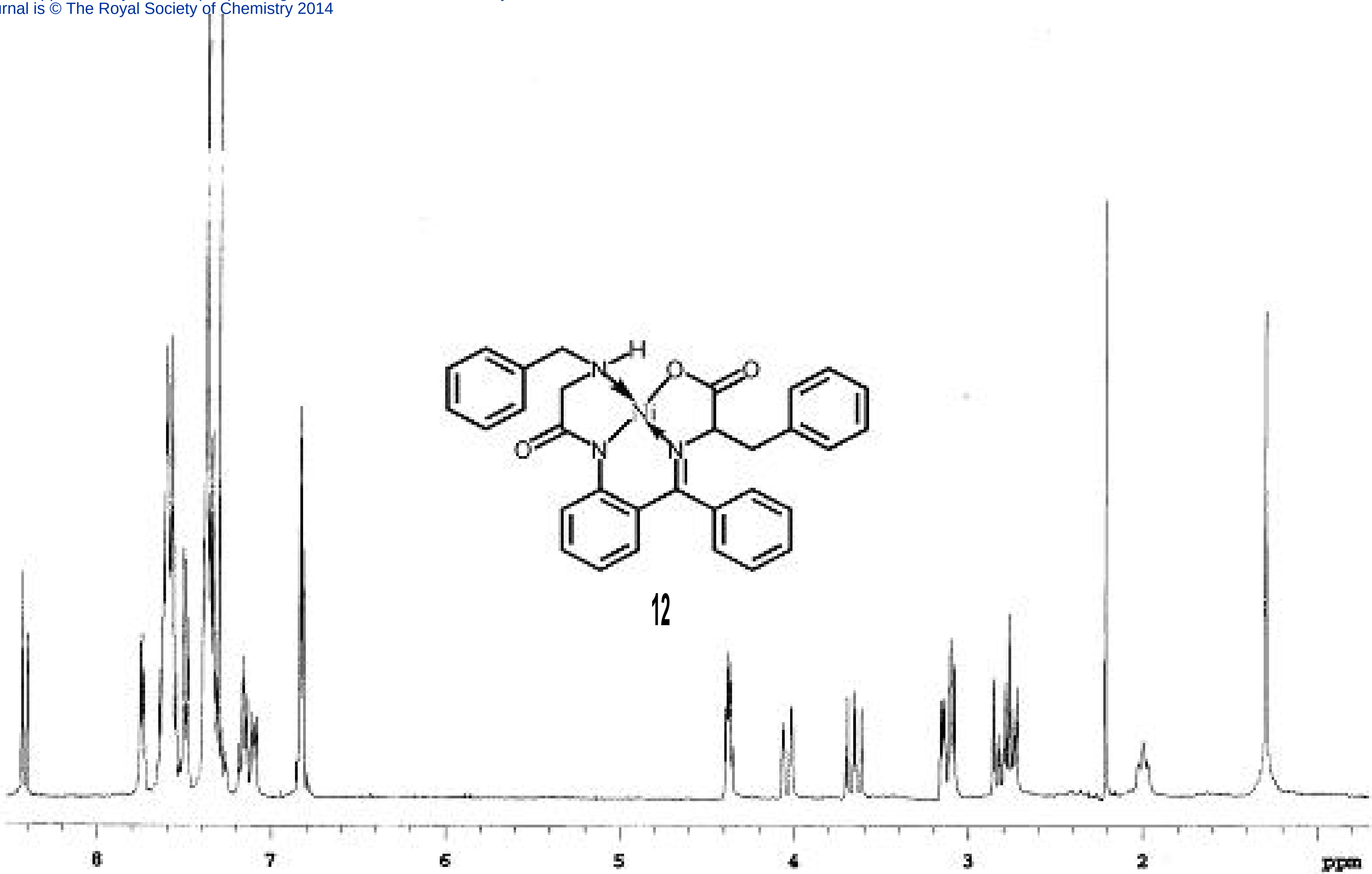
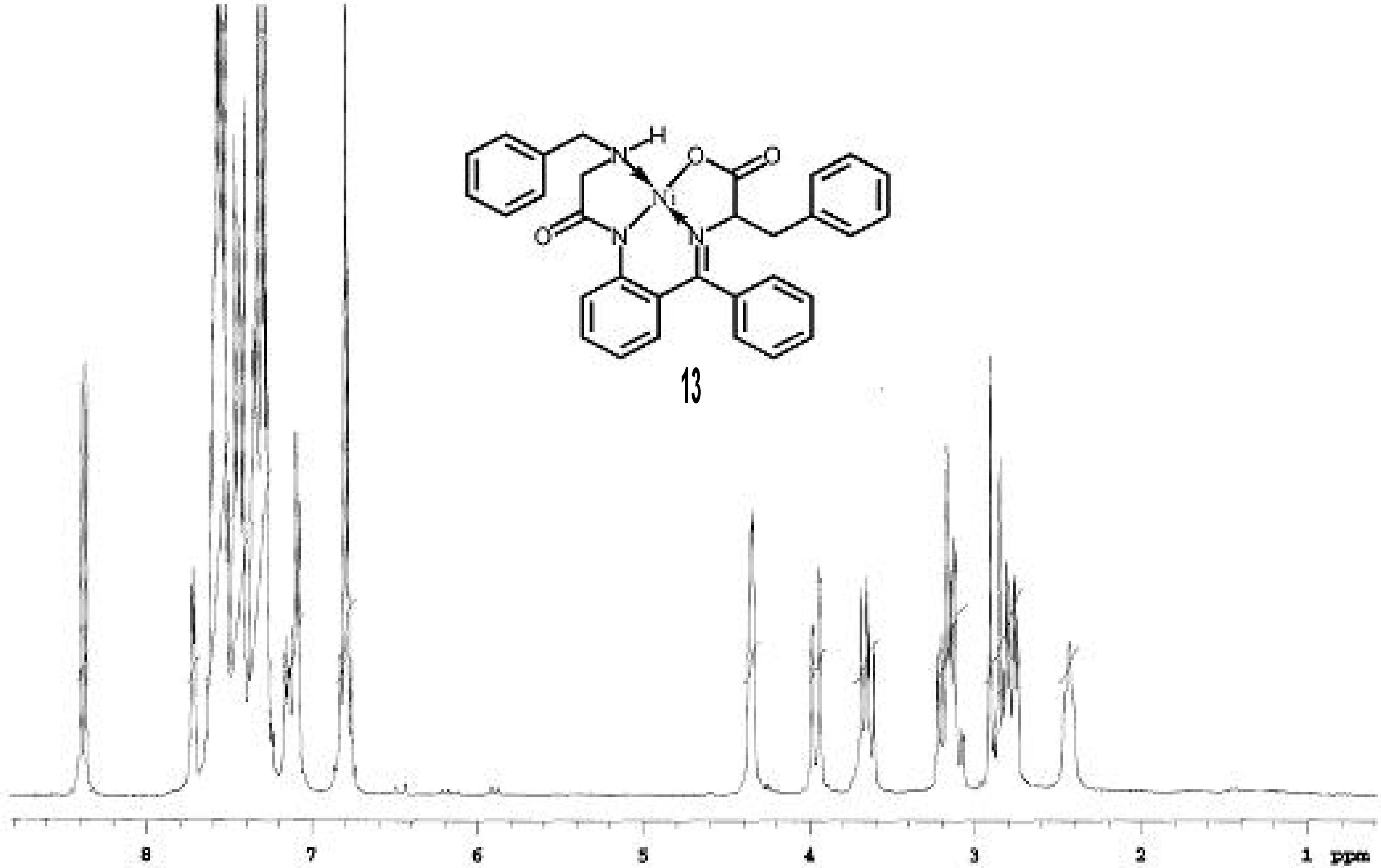
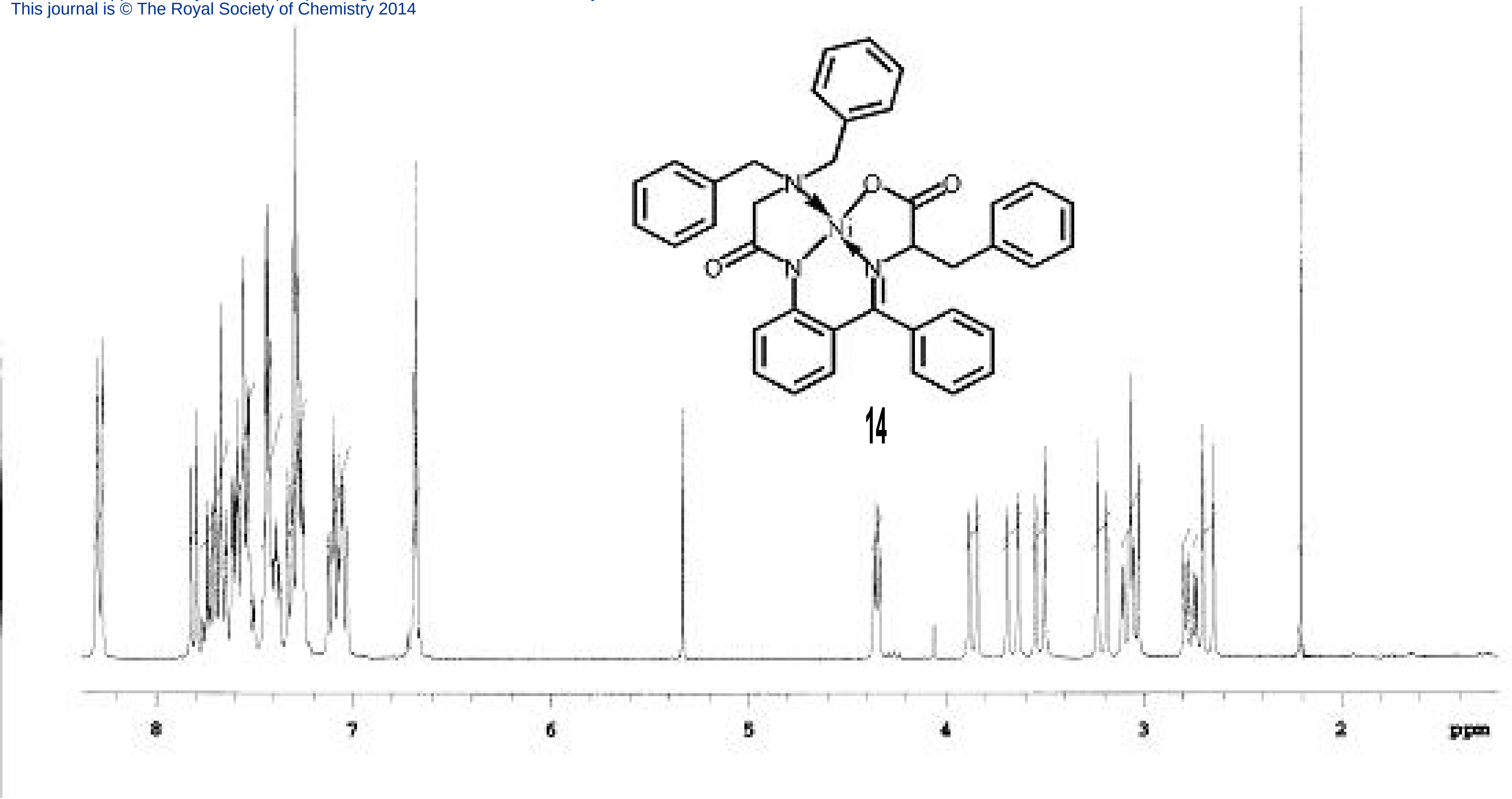
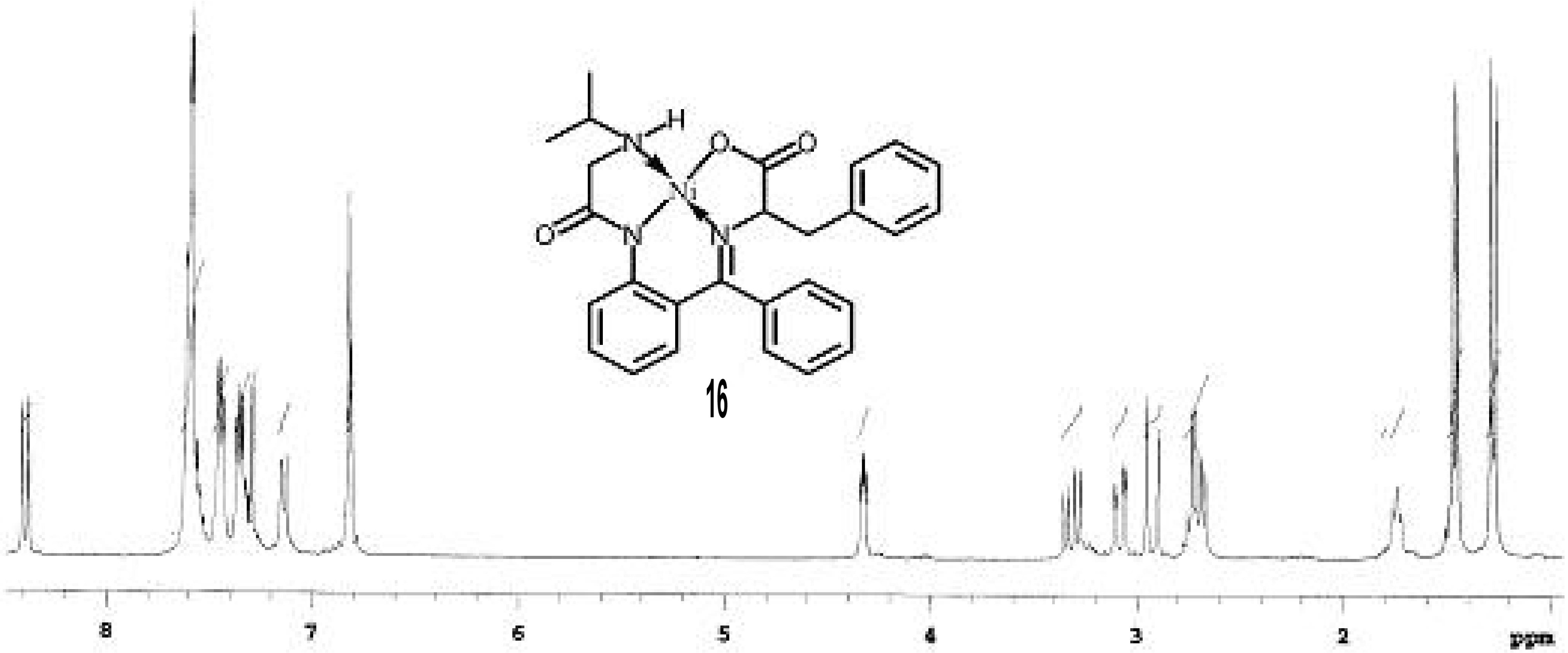
6.9781
6.9718
6.9628
6.9497
6.8573
6.8489
4.1971
4.1722
4.1404
4.1155
3.6703
3.3871
3.3302
2.9820
1.5667
1.5114
1.00
3.16
1.66
1.03
1.00
0.88
1.07
1.82
1.08
1.03
8.67
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

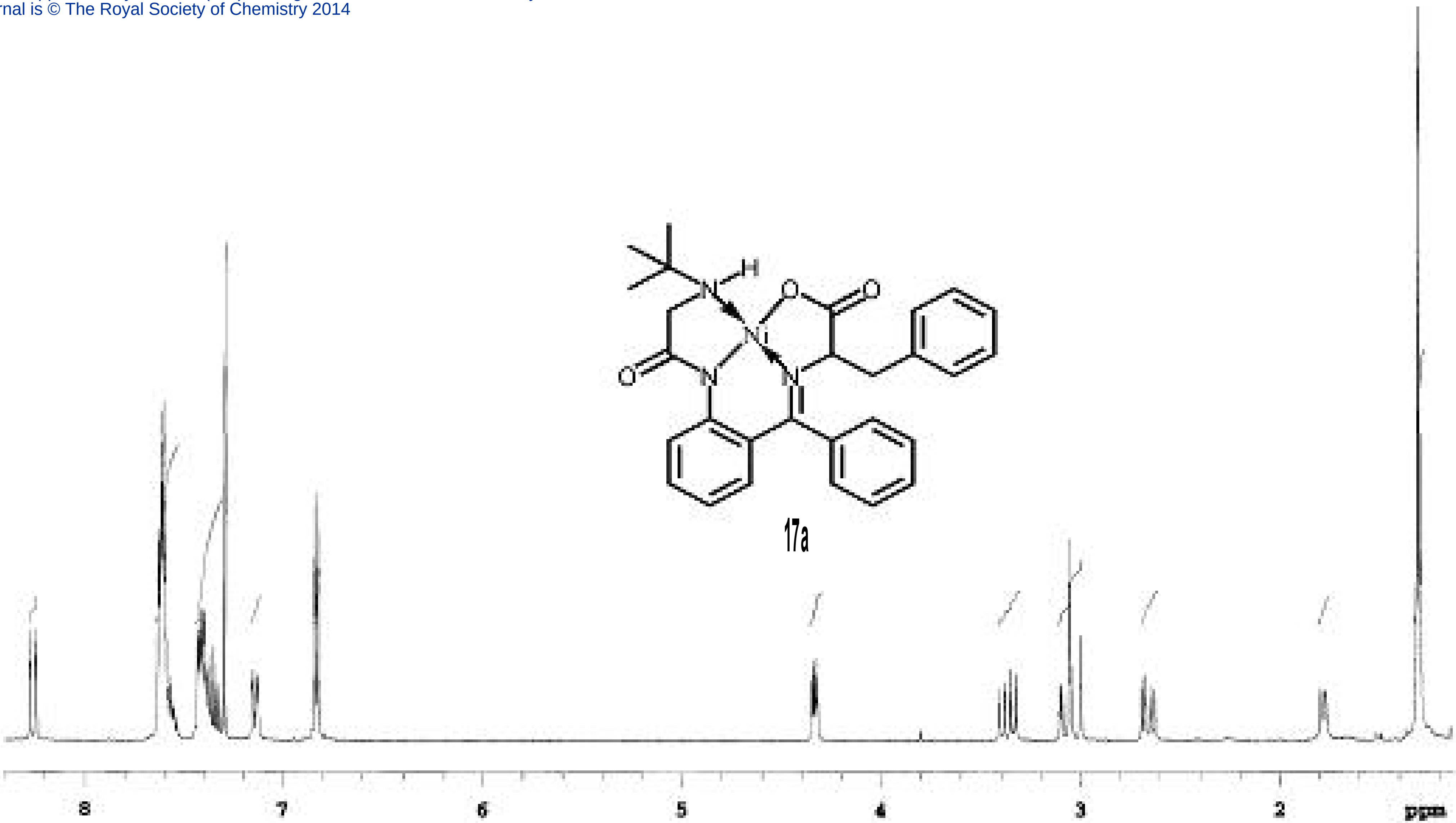

12
8
7
6
5
4
3
2
ppm

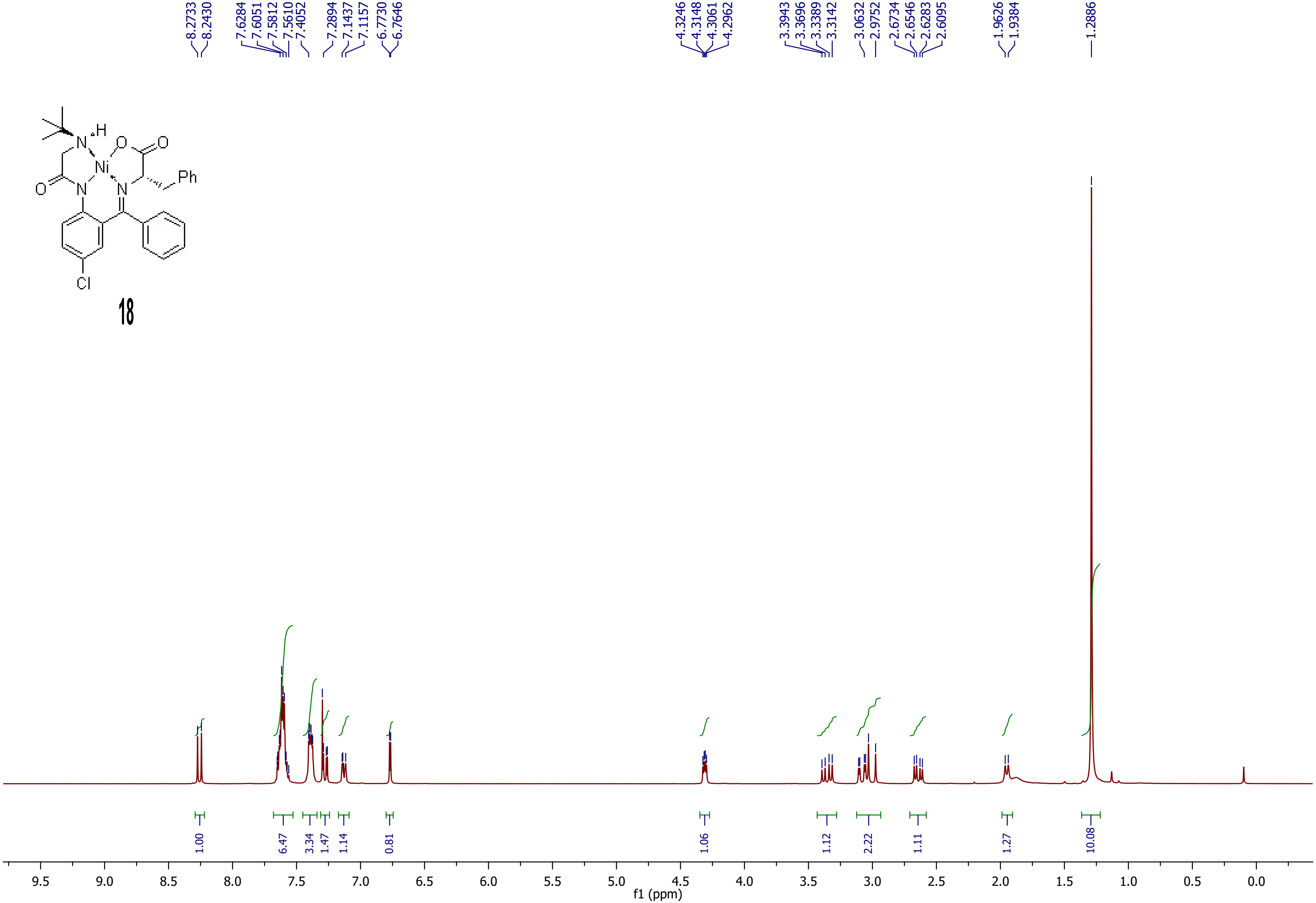

13
8
7
6
5
4
3
2
1
ppm

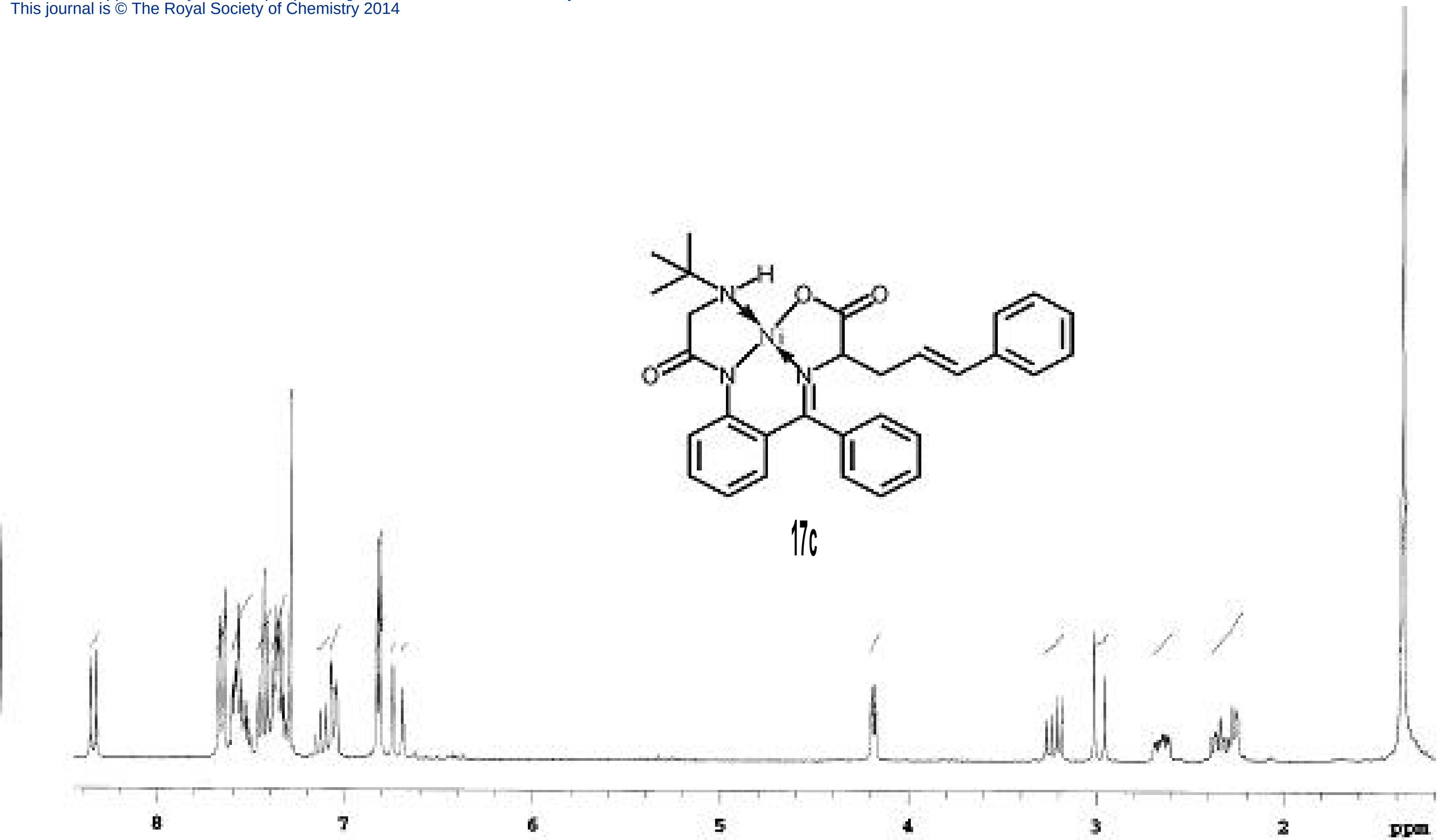

14
8
7
6
5
4
3
2
ppm


O
N
H
Ni
N
O
O
N
16
8
7
6
5
4
3
2
ppm

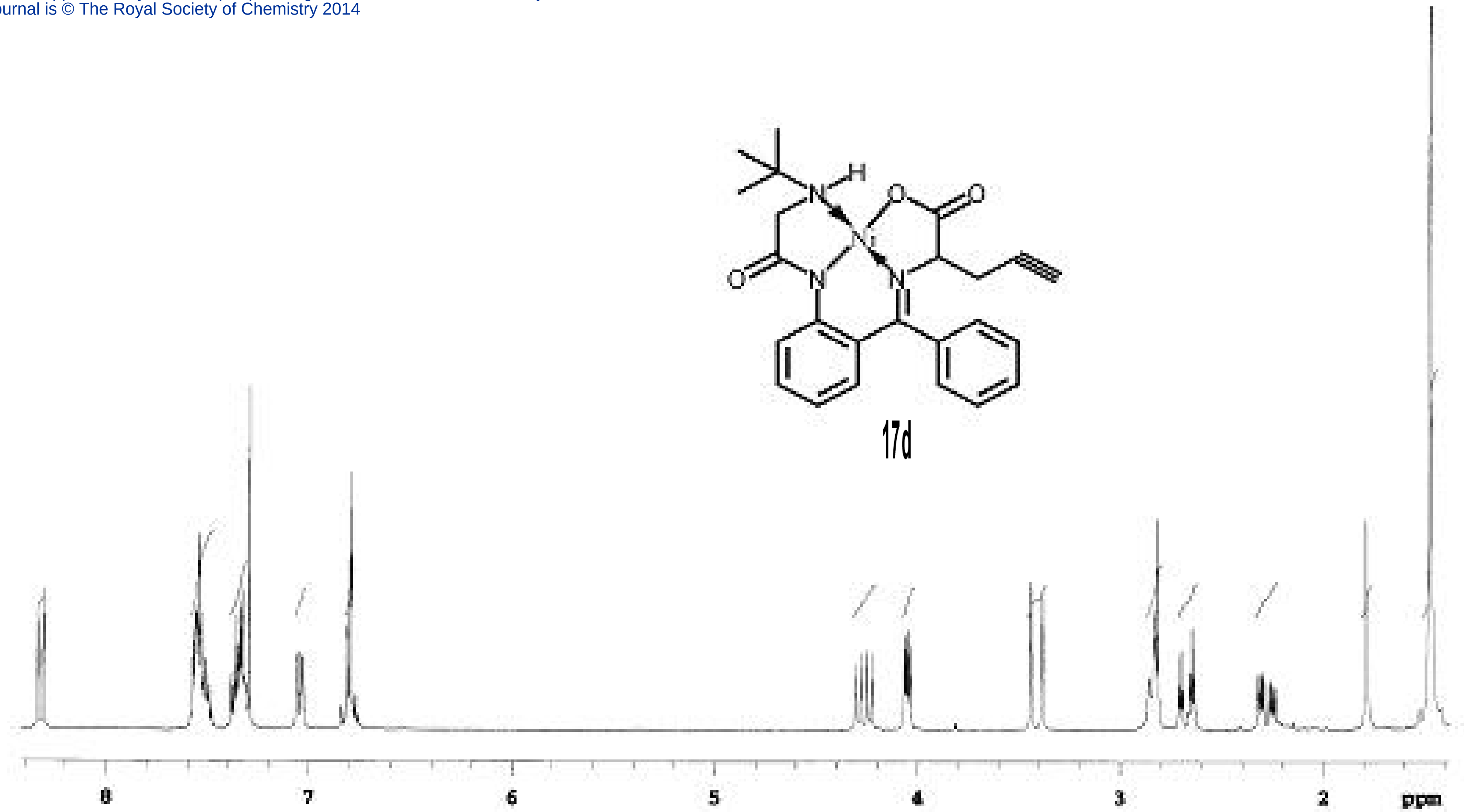
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

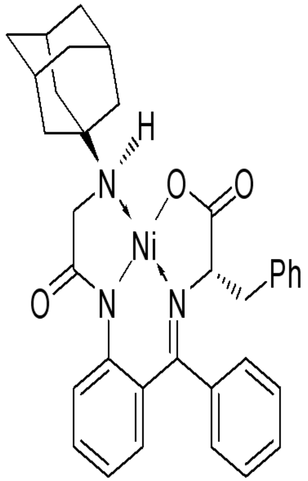



N
H
O
O
Ni
O
N
N
Ph
Cl
18
8.2733
8.2430
7.6284
7.6051
7.5812
7.5610
7.4052
7.2894
7.1437
7.1157
6.7730
6.7646
4.3246
4.3148
4.3061
4.2962
3.3943
3.3696
3.3389
3.3142
3.0632
2.9752
2.6734
2.6546
2.6283
2.6095
1.9626
1.9384
1.2886
1.00
6.47
3.34
1.47
1.14
0.81
1.06
1.12
2.22
1.11
1.27
10.08
9.5
9.0
8.5
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
f1 (ppm)

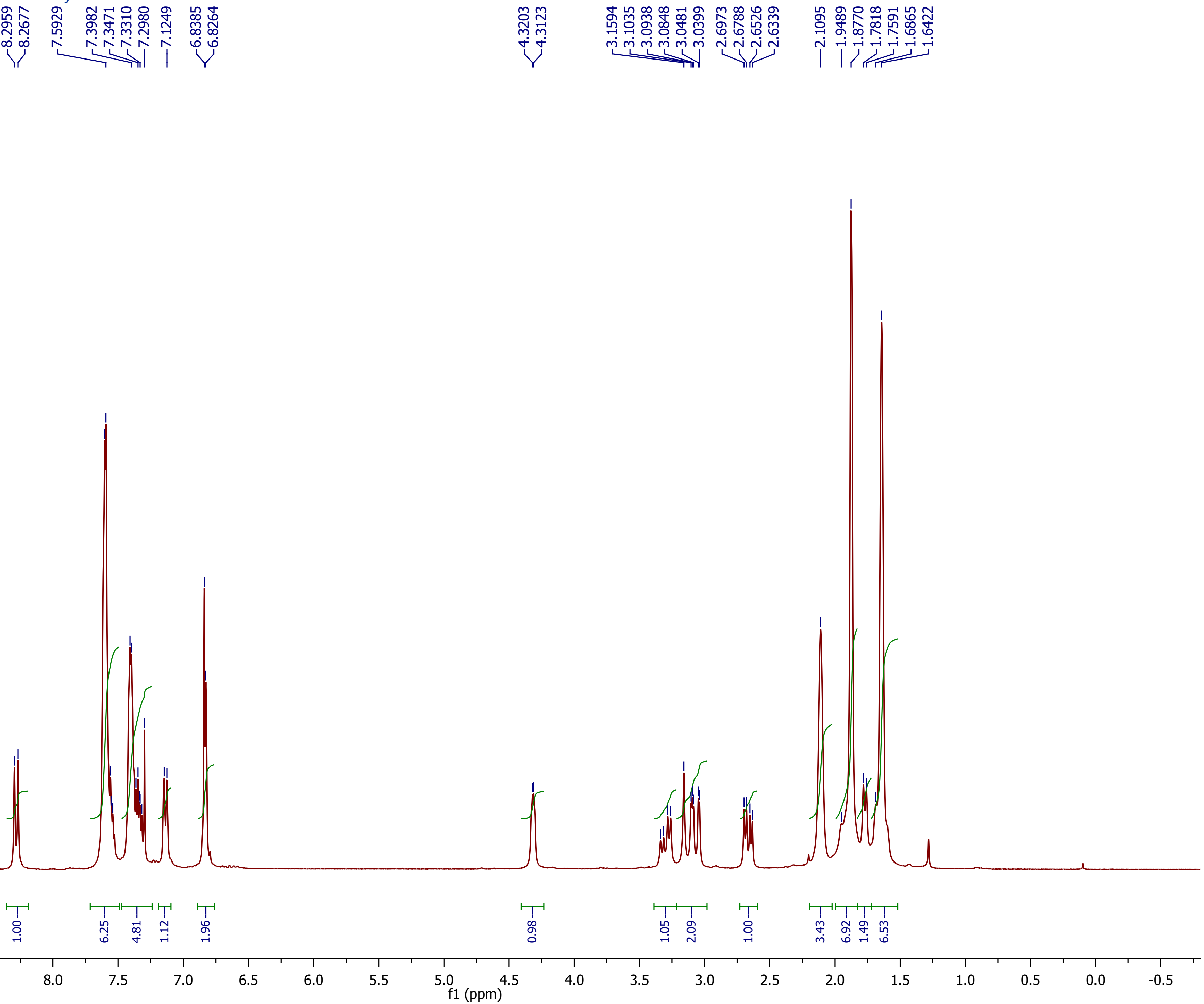

17c
8
7
6
5
4
3
2
ppm

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

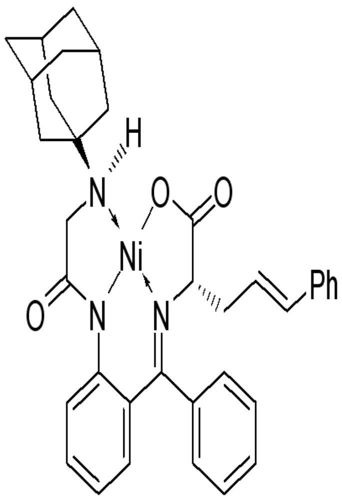




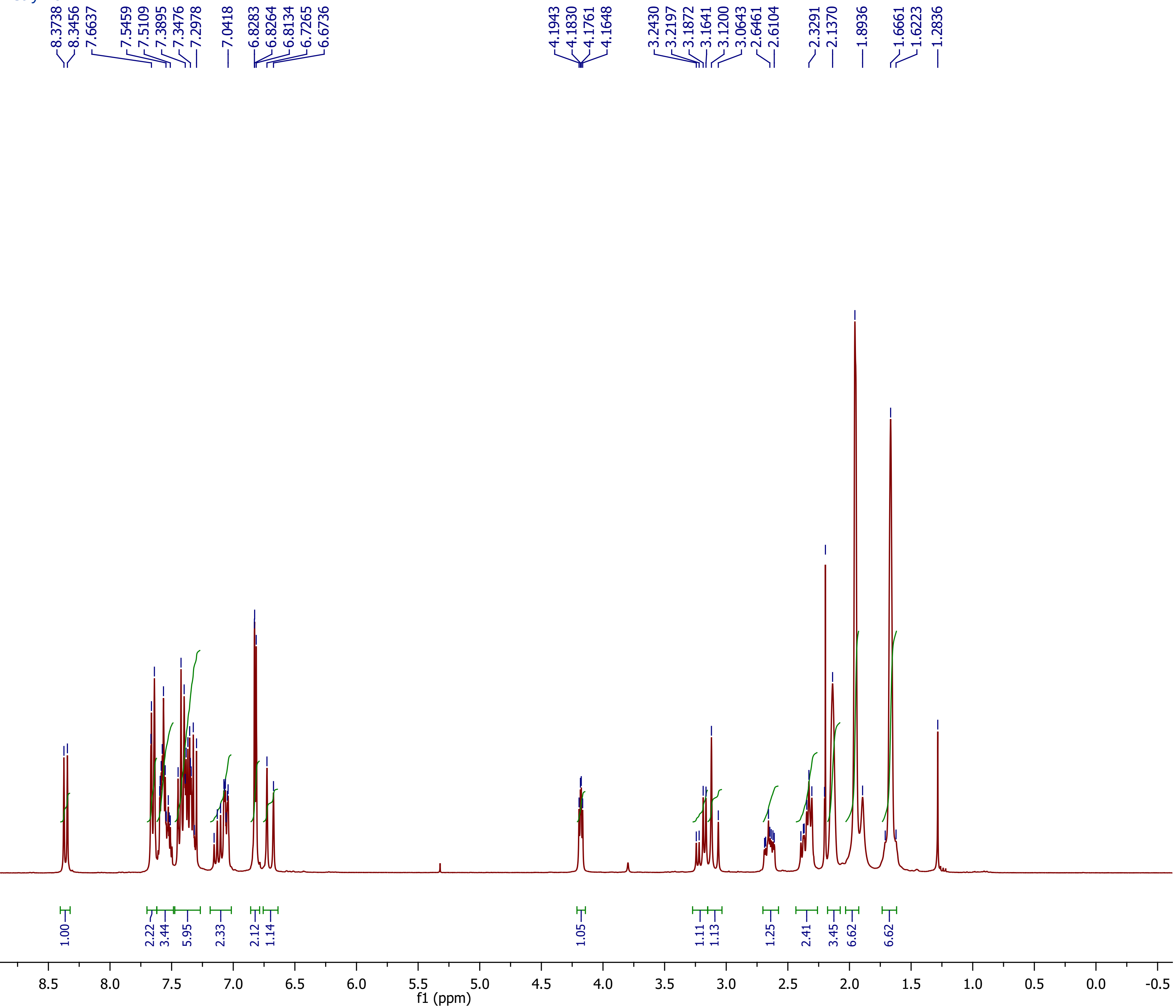
19a


8.2959
8.2677
7.5929
7.3982
7.3471
7.3310
7.2980
7.1249
6.8385
6.8264
4.3203
4.3123
3.1594
3.1035
3.0938
3.0848
3.0481
3.0399
2.6973
2.6788
2.6526
2.6339
2.1095
1.9489
1.8770
1.7818
1.7591
1.6865
1.6422
1.00
6.25
4.81
1.12
1.96
0.98
1.05
2.09
1.00
3.43
6.92
1.49
6.53
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 -0.5
f1 (ppm)

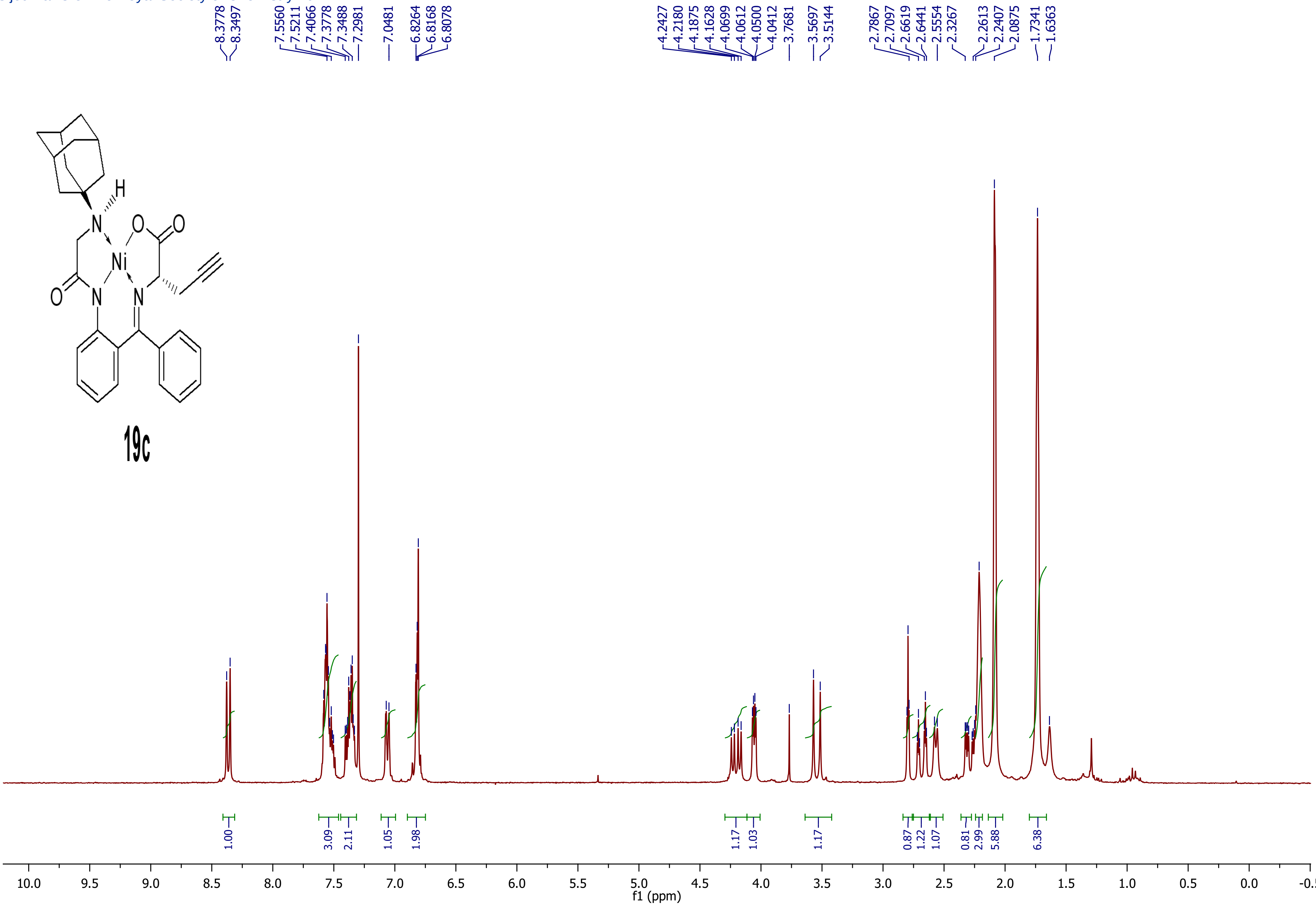
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry



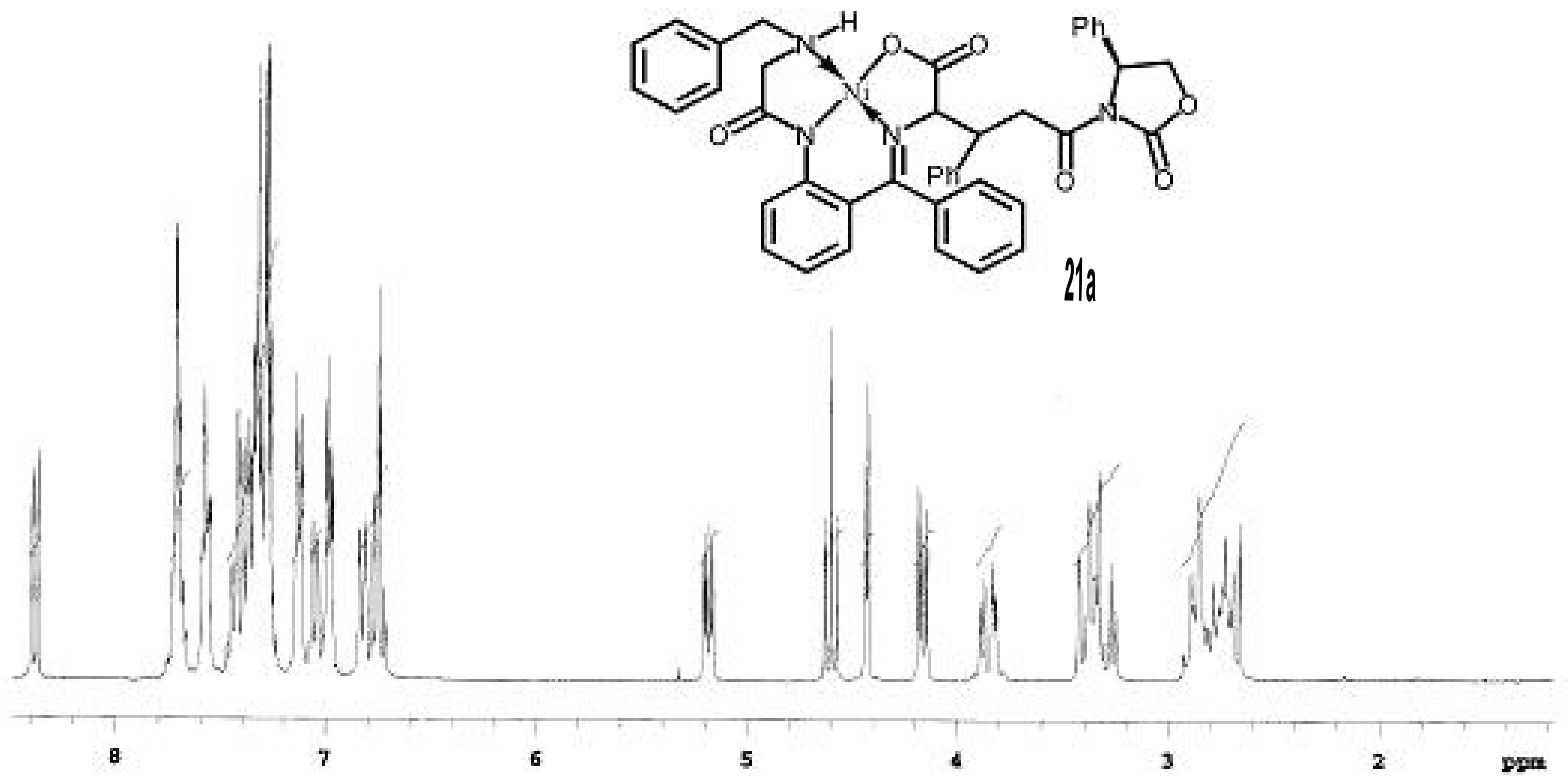
19b

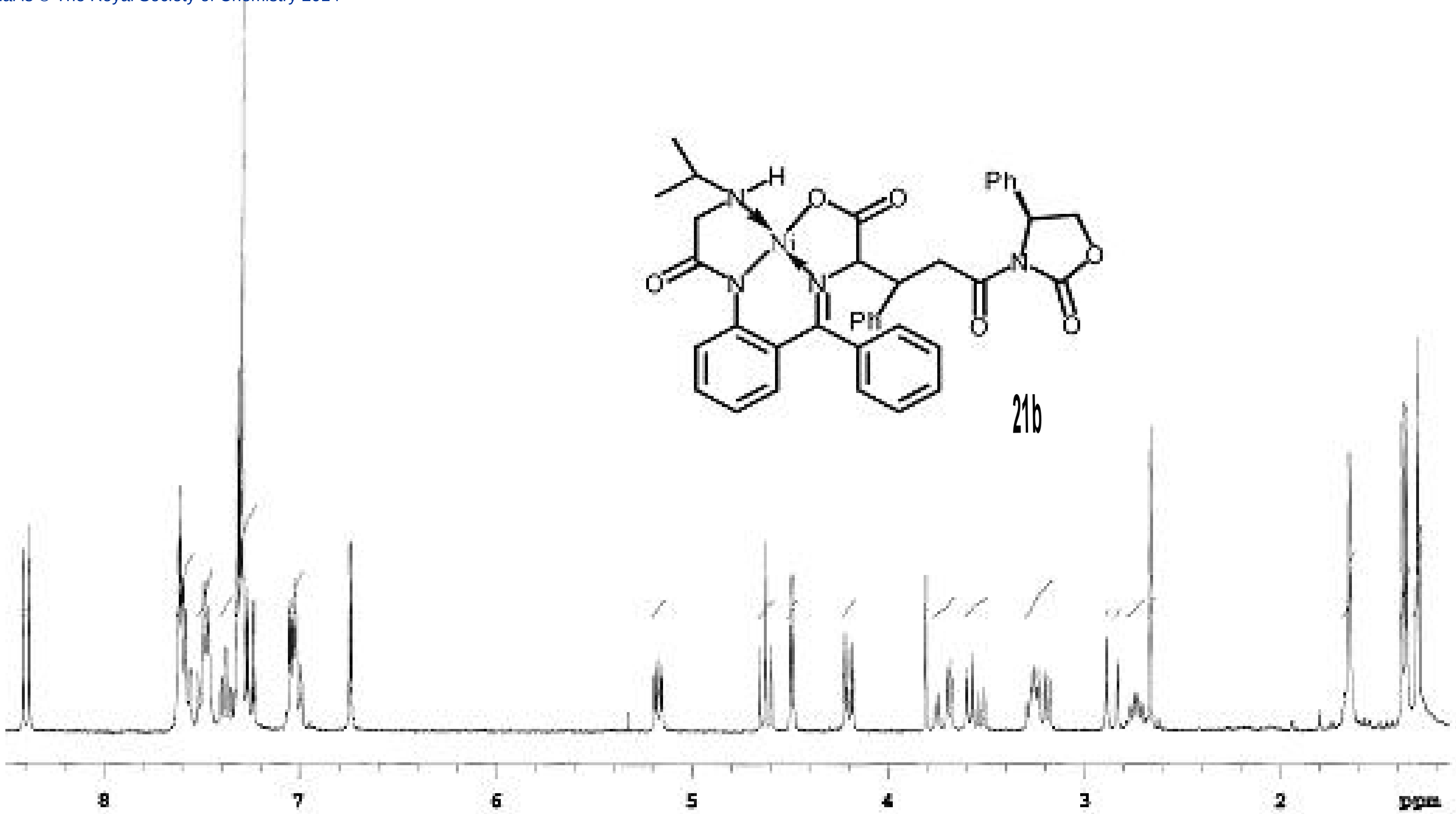


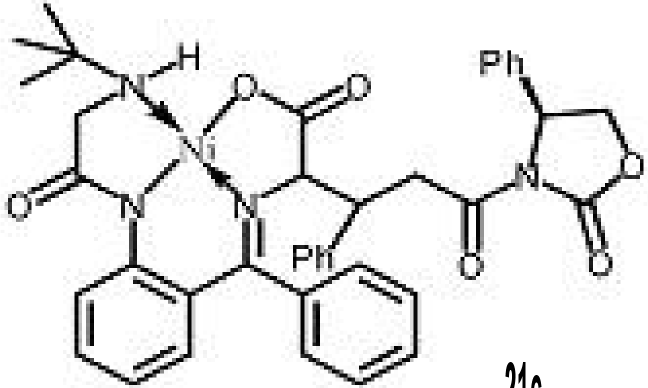
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry



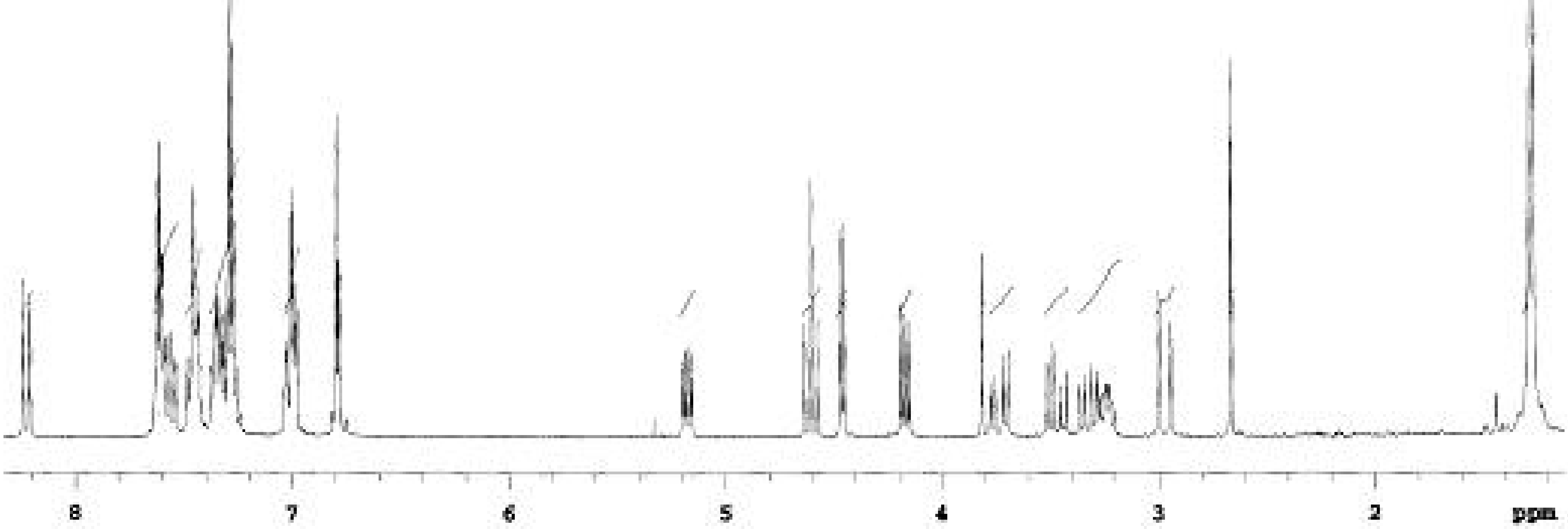
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry



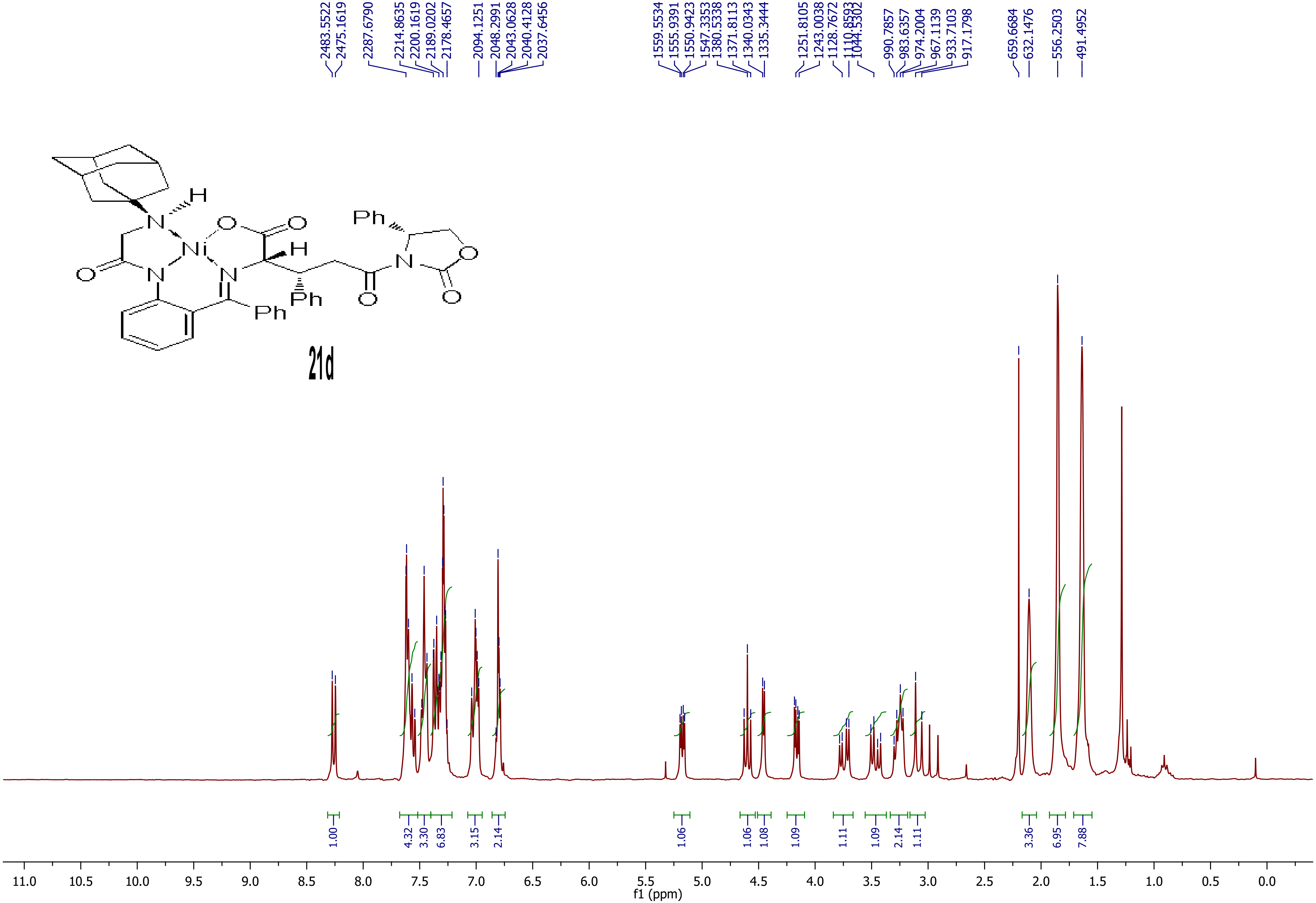

H
O
O
Ph
Ni
N
N
N
N
O
O
O
O
Ph
21b
8
7
6
5
4
3
2
ppm

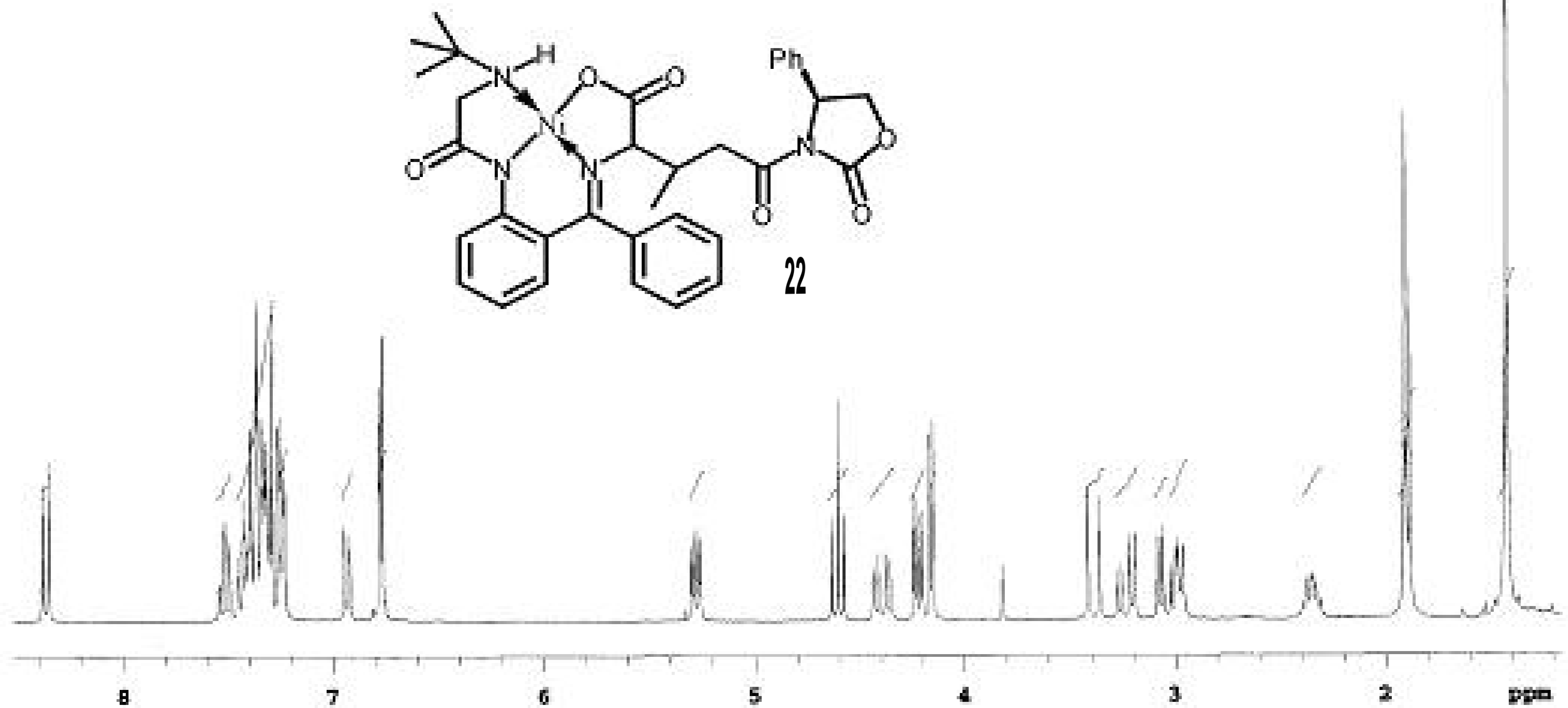


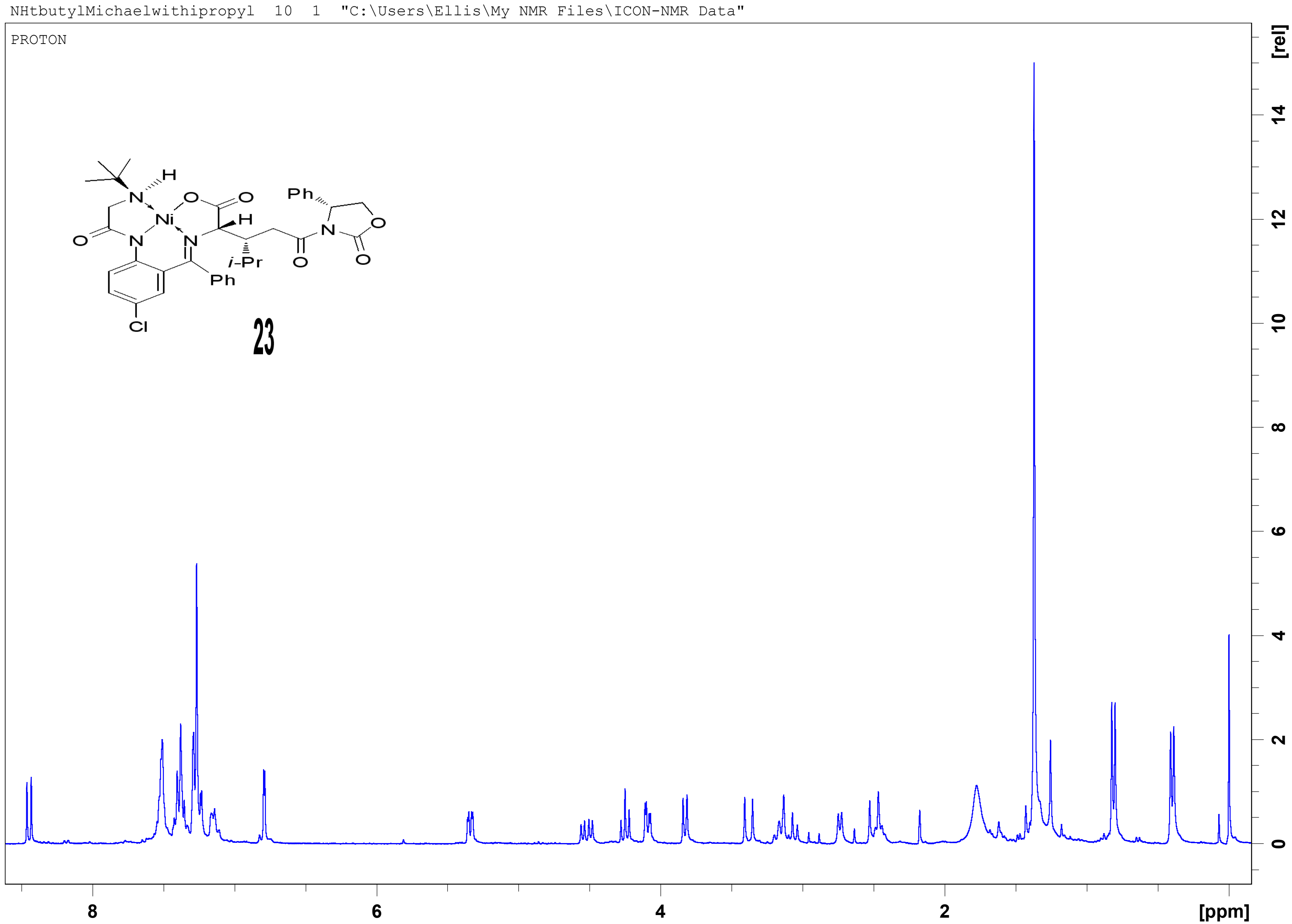
21c

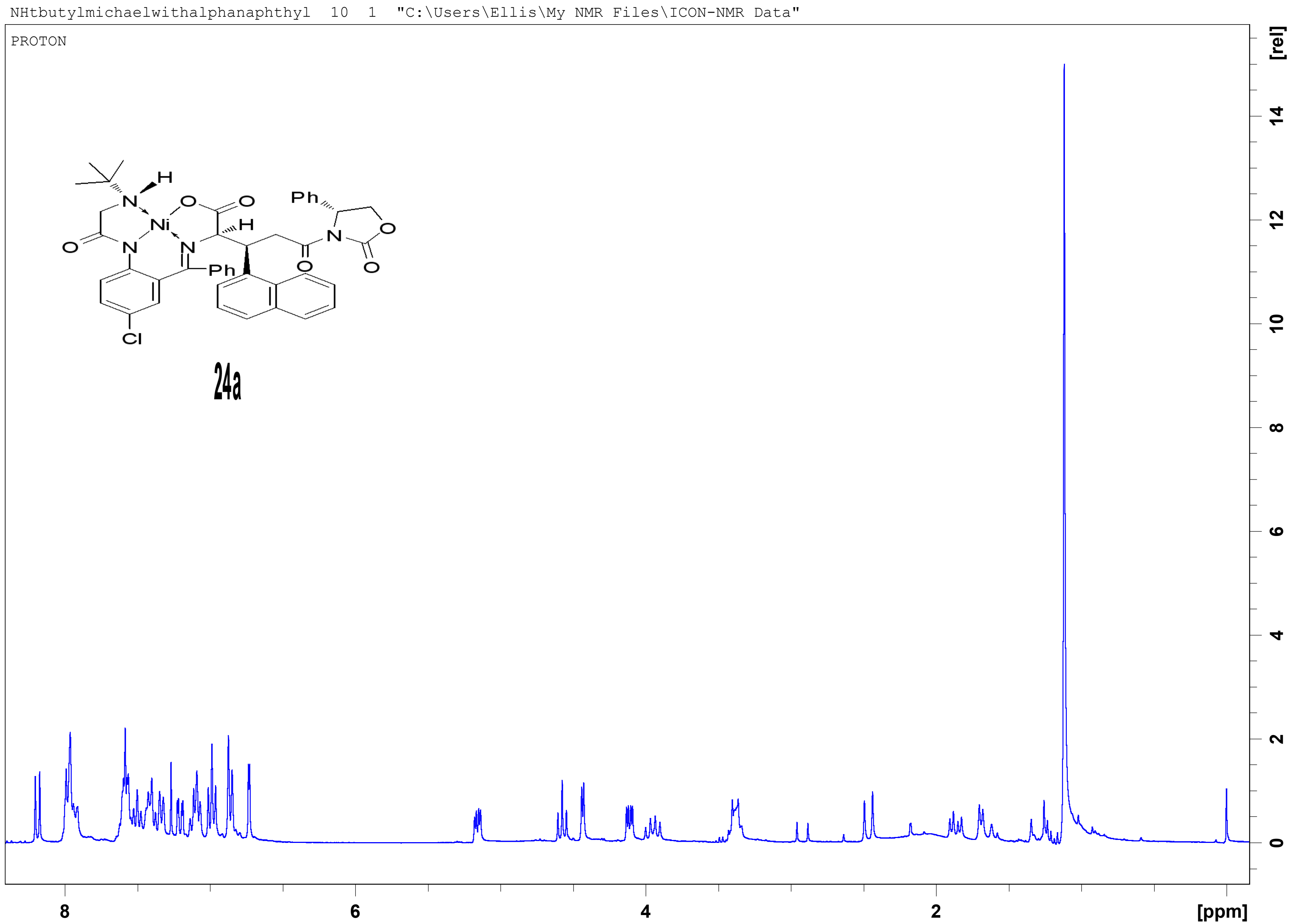


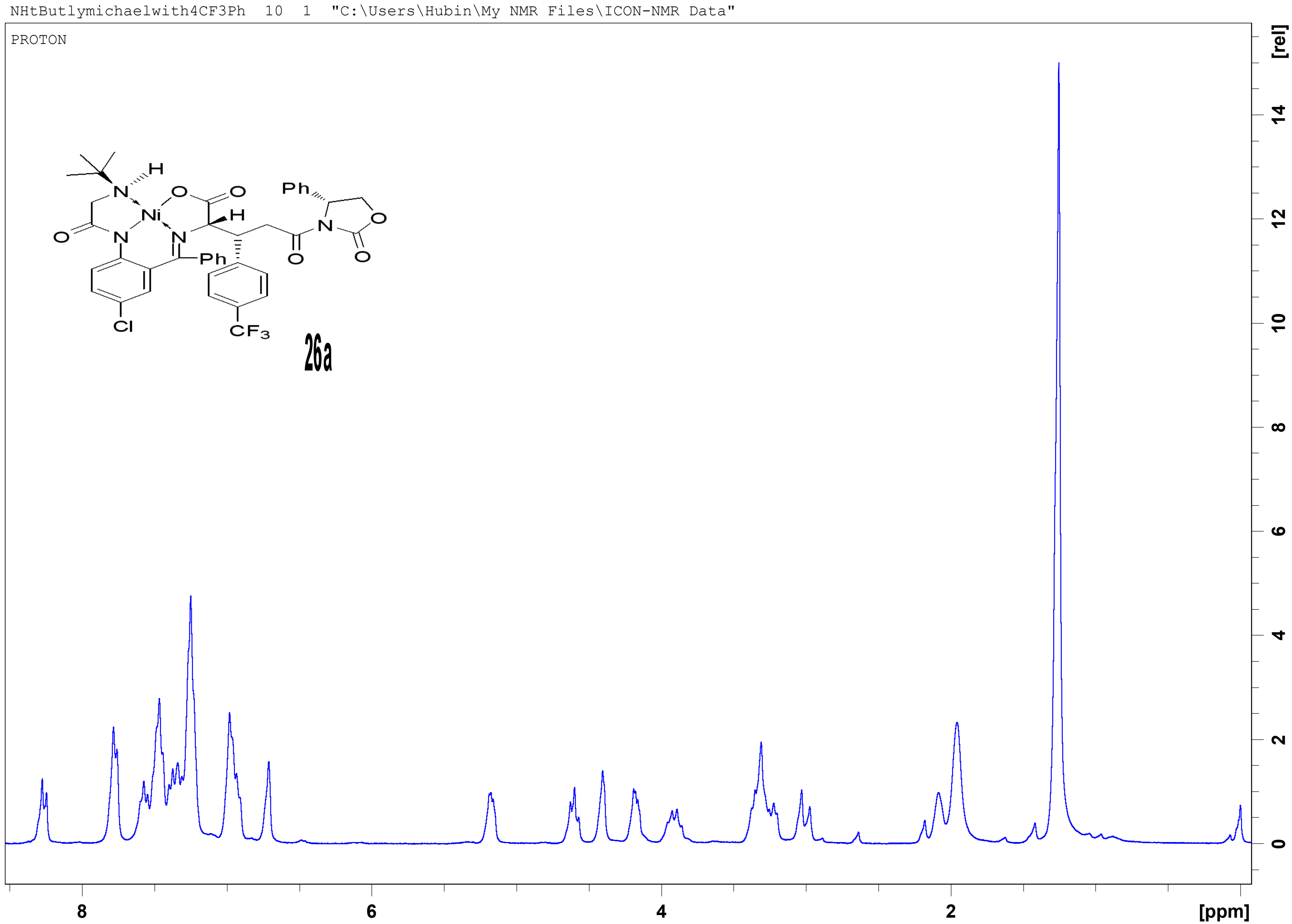
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry



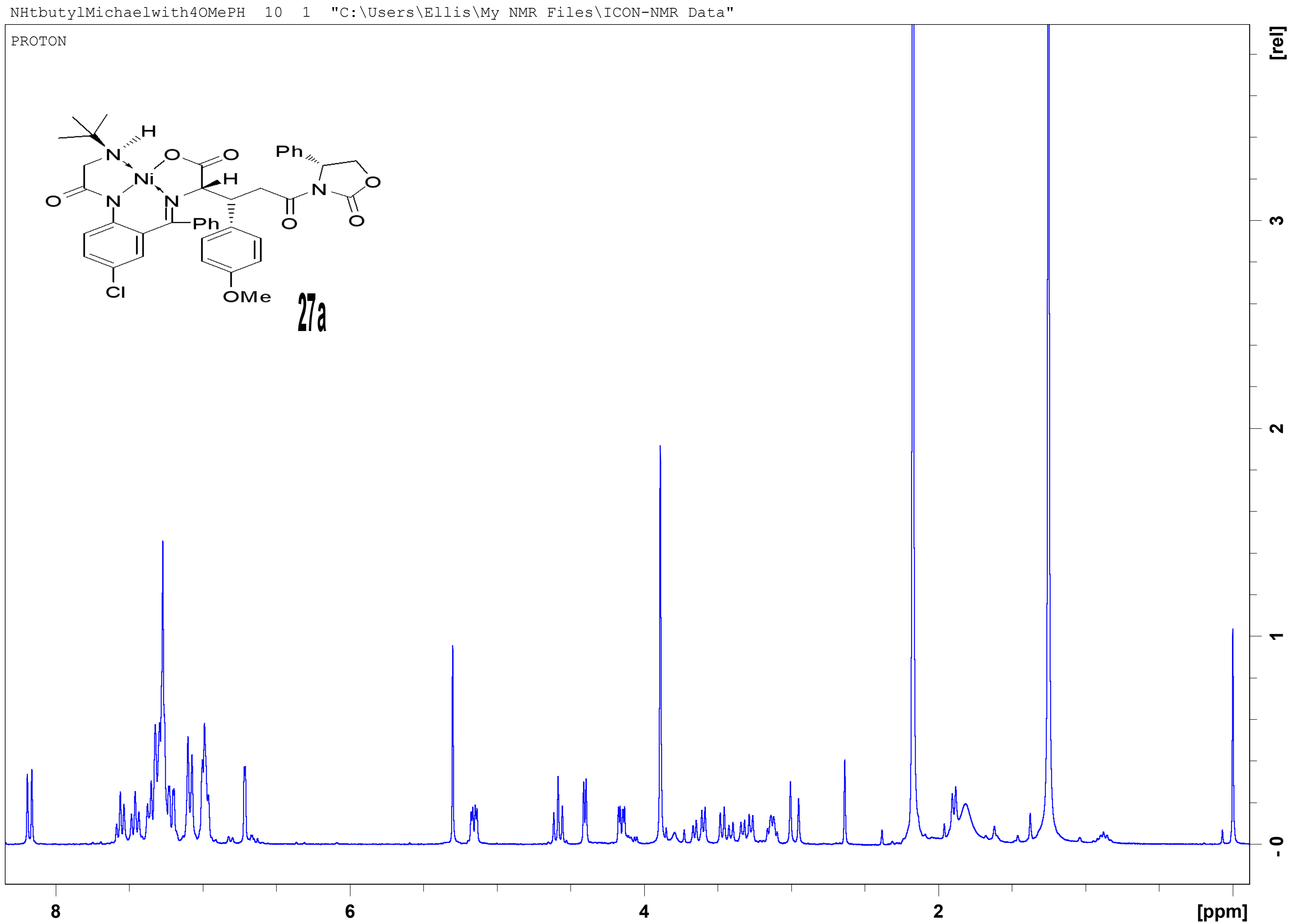

Ph
22
8
7
6
5
4
3
2
ppm

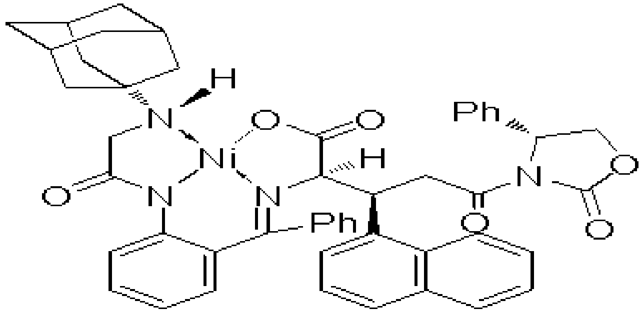

NHtbutylMichaelwithipropyl 10 1 "C:\Users\Ellis\My NMR Files\ICON-NMR Data"
PROTON
23
[rel]
[ppm]
8
6
4
2
0
2
4
6
8
10
12
14


NHtbutylmichaelwithalphanaphthyl 10 1 "C:\Users\Ellis\My NMR Files\ICON-NMR Data"
PROTON
24a
[rel]
[ppm]

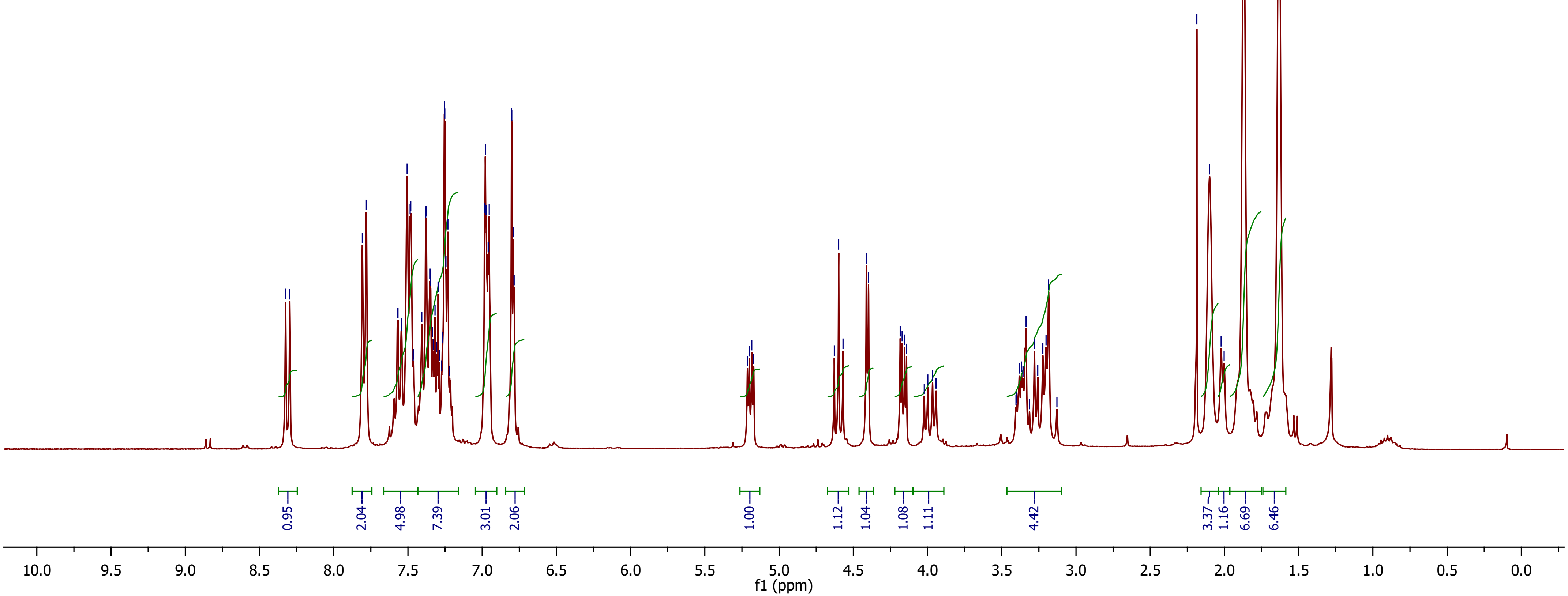

NHtButlymichaelwith4CF3Ph 10 1 "C:\Users\Hubin\My NMR Files\ICON-NMR Data"
PROTON
26a
[rel]
[ppm]

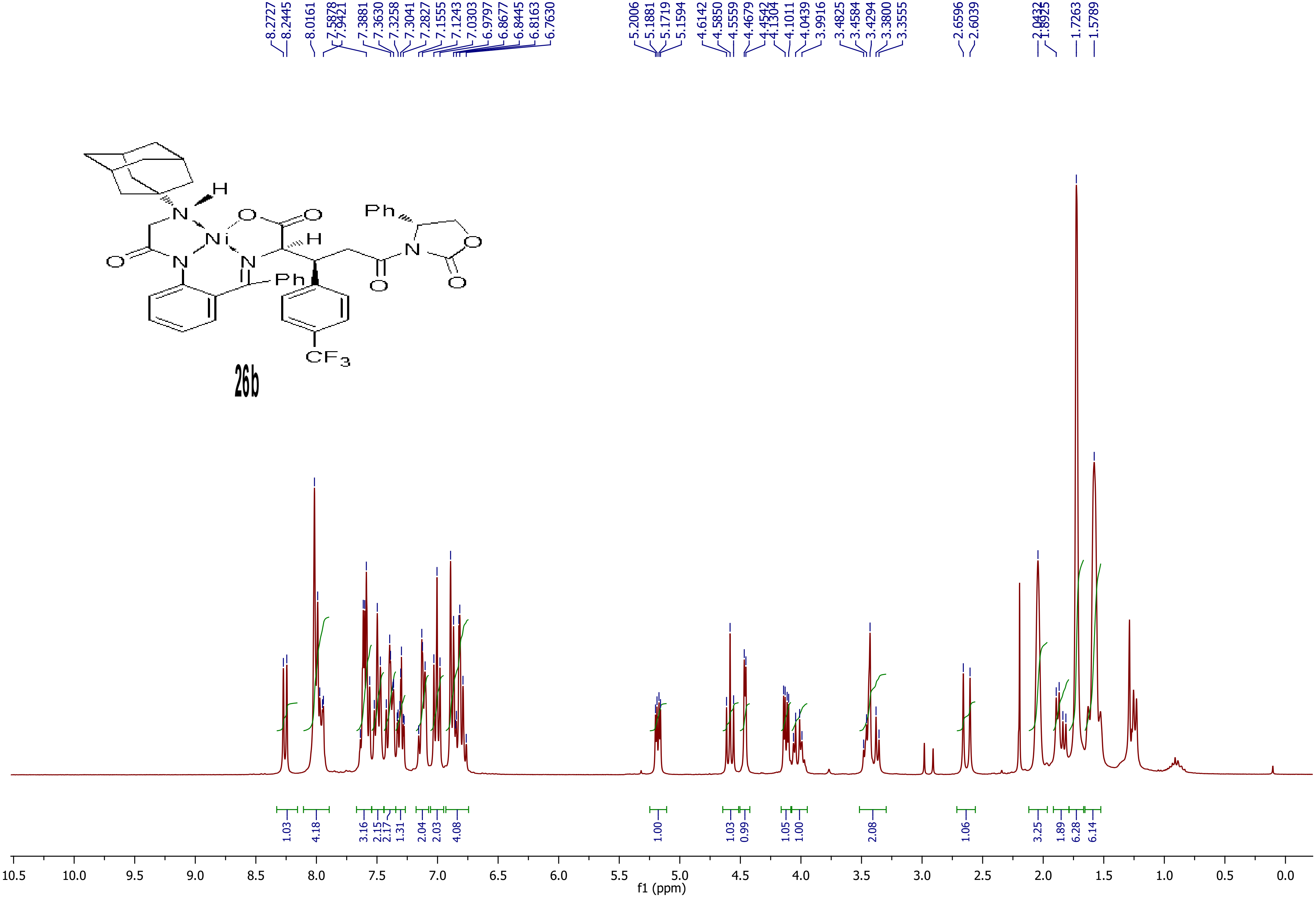
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry




H
N
Ni
O
O
H
Ph
N
O
O
N
N
Ph
O
O

24b


8.3241
8.2961
7.8073
7.7804
7.4612
7.3071
7.2190
6.9527
6.8019
6.8005
6.7905
6.7849
5.2130
5.2001
5.1841
5.1713
4.6280
4.5986
4.5693
4.3976
4.1843
4.0223
3.9983
3.9664
3.3131
3.2798
3.2575
3.2233
3.2018
3.1841
3.1285
2.1858
2.1001
2.0222
2.0022
1.8702
1.6324
0.95
2.04
4.98
7.39
3.01
2.06
1.00
1.12
1.04
1.08
1.11
4.42
3.37
1.16
6.69
6.46
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
f1 (ppm)


26b
CF3
f1 (ppm)

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry