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Hydrogen bonding in acetylene containing dichlorodiazaalkadienes

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Experimental details. All reagent grade chemicals were used as purchased unless otherwise noted. Analytical TLC: aluminium-backed plates precoated (0.25 mm) with Silica Gel 60 F254 (Merck). Compounds were visualized by exposure to UV light or by dipping the plates in $KMnO_4$ stain followed by heating. Column chromatography was performed using Macherey-Nagel Silica gel 60 (70–230 mesh). All mixed eluents are reported as v/v solutions. Solvents were purified by standard methods. DMSO was distilled over CaH_2. Tetrachloromethane was distilled over P_2O_5. ^1H and ^{13}C NMR spectra were acquired at 400.1 and 100.6 MHz, respectively, on an AVANCE 400 MHz spectrometer in $CDCl_3$ (unless otherwise stated). HRMS (ESI-MS) spectra were measured on MicroTof. IR spectra of SOLID samples were recorded on a Fourier transform spectrometer Nicolet iS5 (Thermo Scientific) using an accessory for attenuated total reflection (ATR) with a diamond optical element, an angle of incidence of 45°, the resolution 4 cm^{-1}, the number of scans was 32. IR spectra of CCl_4 solutions were recorded in a cuvette with a thickness of 0.01-0.06 cm with KBr windows. The solution concentrations were within the range of 0.01–0.04 mol dm^{-3}. Single-crystal X-ray studies for **5** and **14** were collected on a three-circle diffractometer Bruker D8 QUEST using a PHOTON III CCD detector (λ(MoK$_\alpha$)-radiation, graphite monochromator and corrected for absorption using the SADABS program[S1]. The data were indexed and integrated using the SAINT program[S2]. The structures were determined by direct methods and refined by full-matrix least squares technique with anisotropic displacement parameters for non-hydrogen atoms. The hydrogen atoms were placed in calculated positions and refined within riding model with fixed isotropic displacement parameters. All calculations were carried out using the SHELXTL[S3] program suite. Quantum chemical calculations were performed by the density functional method (DFT) of the Perdew-Burke-Ernzerhof (PBE) generalized gradient approximation (GGA) exchange-correlation energy functional[S4] using full-electron double set size extended split bases[S5]. All calculations were performed in the Joint Supercomputer Center (JSCC, Moscow) using the program PRIRODA04[S6].

(*E*)-2-Aryl-1-{2,2-dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}diazenes (general procedure). A 20 mL screw cap vial was charged with DMSO (10 mL), the corresponding *o*-propargyloxy- benzaldehyde[S7] (1 mmol, 1 eq.) and hydrazine (1 eq.). After 2 h stirring, TMEDA (2.5 eq.), CuCl (0.01 eq.) were added. Tetrachloromethane (10 eq.) was then added within 5 min under cooling with a water bath. The reaction was carried out at room temperature for 3 h (TLC control for consumption of the corresponding hydrazone). The mixture was poured into water (200 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The extract was washed with water (3 × 50 mL), brine (1 × 30 mL), dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel using hexane – CH_2Cl_2 mixture (3:1) as the eluent.

(*E*)-1-{2,2-Dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-phenyldiazene (1). Yield 248 mg (75%), orange-red solid, m.p. 85 °C. IR (ν, cm^{-1}): 1573, 1584, 1604, 3261. ^{1}H-NMR: δ 2.45 (t, 1H, J = 2.4 Hz), 4.63 (d, 2H, J = 2.4 Hz), 7.08-7.15 (m, 3H), 7.42-7.46 (m, 4H), 7.76-7.81 (m, 2H). ^{13}C-NMR: δ 56.1, 75.5, 78.5, 112.7, 121.3, 122.9, 123.2, 128.9, 130.3, 131.3, 131.3, 136.0, 149.9, 153.0, 154.9. ESI-HRMS (m/z): calcd. for ($C_{17}H_{13}Cl_2N_2O$) [M+H$^+$] 331.0400, found 331.0400.

(*E*)-1-{2,2-Dichloro-1-[3-methoxy-2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-phenyldiazene (2) Yield 217 mg (60%), orange-red oil. IR (ν, cm^{-1}): 1582, 1603, 3297. ^{1}H-NMR: δ 2.33 (t, 1H, J = 2.5 Hz), 3.91 (s, 3H), 4.60 (s, 2H), 6.73 (dd, 1H, J = 7.7, 1.4 Hz), 7.01 (dd, 1H, J = 8.3, 1.3 Hz), 7.15 (t, 1H, J = 8.0 Hz), 7.42-7.45 (m, 3H), 7.76-7.80 (m, 2H). ^{13}C-NMR: δ 55.7, 60.2, 74.9, 79.3, 113.2, 122.5, 123.2, 124.4, 128.0, 128.9, 131.3, 136.3, 144.7, 149.9, 152.4, 152.9. ESI-HRMS (m/z): calcd. for ($C_{187}H_{15}Cl_2N_2O_2$) [M + H$^+$] 361.0506, found 361.0505.

(*E*)-1-{2,2-Dichloro-1-[3-nitro-2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-phenyldiazene (3). Yield 196 mg (52%), orange oil. IR (ν, cm^{-1}): 1533, 1574, 1602, 3292. ^{1}H-NMR: δ 2.49 (t, 1H, J = 2.5 Hz), 4.55 (d, 2H, J = 9.5 Hz), 7.32-7.37 (m, 2H), 7.43-7.49 (m, 3H), 7.75-7.79 (m, 2H), 7.98 (dd, 1H, J = 7.0, 2.9 Hz). ^{13}C-NMR: δ 62.6, 76.8, 77.4, 110.5, 123.3, 124.6, 126.3, 129.1, 130.9, 132.0, 133.3, 136.3, 137.6, 144.3, 148.2, 149.5, 152.5. ESI-HRMS (m/z): calcd. for ($C_{17}H_{12}Cl_2N_3O_3$) [M + H$^+$] 376.0251, found 376.0275.

(*E*)-1-{2,2-Dichloro-1-[5-nitro-2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-phenyldiazene (4). Yield 290 mg (77%), orange-red solid, m.p. 105 °C. IR (ν, cm^{-1}): 1568, 1585, 1612, 3290. ^{1}H-NMR: δ 2.53 (t, 1H, J = 2.4 Hz), 4.74 (d, 2H, J = 2.4 Hz), 7.20 (d, 1H, J = 9.2 Hz), 7.41-7.48 (m, 3H), 7.72-7.77 (m, 2H), 8.06 (d, 1H, J = 2.8 Hz), 8.35 (dd, 1H, J = 9.2, 2.8 Hz). ^{13}C-NMR: δ 56.7, 77.0, 77.5, 112.3, 123.4, 123.9, 126.6, 127.6, 129.2, 131.9, 137.2, 141.9, 148.1, 152.8, 159.7. ESI-HRMS (m/z): calcd. for ($C_{17}H_{12}Cl_2N_3O_3$) [M + H$^+$] 376.0251, found 376.0260.

(*E*)-1-{1-[5-Bromo-2-(prop-2-yn-1-yloxy)phenyl]-2,2-dichlorovinyl}-2-phenyldiazene (5). Yield 271 mg (66%), orange-red solid, m.p. 107 °C. IR (ν, cm^{-1}): 1564, 1582, 1598, 3310. ^{1}H-NMR: δ 2.46 (t, 1H, J = 2.4 Hz), 4.60 (d, 2H, J = 2.4 Hz), 7.01 (d, 1H, J = 8.8 Hz), 7.26 (d, 1H, J = 2.5 Hz), 7.44-7.47 (m, 3H), 7.52 (dd, 1H, J=8.9, 2.5 Hz), 7.76–7.80 (m, 2H). ^{13}C-NMR: δ 56.2, 75.9, 77.9, 113.4, 114.4, 123.2, 124.9, 128.9, 131.5, 133.0, 133.8, 136.5, 148.6, 152.8, 154.0. ESI-HRMS (m/z): calcd. for ($C_{17}H_{12}BrCl_2N_2O$) [M+H$^+$] 408.9505, found 408.9520.

(*E*)-1-{2,2-Dichloro-1-[3,5-di-*tert*-butyl-2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-phenyldiazene (6). Yield 230 mg (52%), orange-red oil. IR (ν, cm^{-1}): 1562, 1599, 3307. ^{1}H-NMR: δ 1.31 (s, 9H), 1.45 (s, 9H), 2.50 (t, 1H, J=2.5 Hz), 4.19-4.38 (m, 2H), 6.87 (d, 1H, J=2.5 Hz), 7.41 (d, 1H, J=2.5 Hz), 7.45-7.47 (m, 3H), 7.79-7.81 (m, 2H). ^{13}C-NMR: δ 30.3, 31.0, 34.2, 34.8, 60.6, 74.6, 78.7, 122.9, 124.5, 125.0, 126.3, 128.6, 131.1, 136.4, 141.4, 145.1, 150.7, 152.6, 154.0. ESI-HRMS (m/z): calcd. for ($C_{25}H_{29}Cl_2N_2O$) [M + H$^+$] 443.1652, found 443.1649.

(*E*)-1-{2,2-Dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-(4-fluorophenyl)diazene (7). Yield 175 mg (50%), orange-red solid, m.p. 60 °C. IR (ν, cm^{-1}): 1574, 1592, 1603, 3301. ^{1}H-NMR: δ 2.44 (t, 1H, J = 2.4 Hz), 4.63 (d, 2H, J = 2.4 Hz), 7.06–7.13 (m, 5H), 7.41–7.45 (m, 1H), 7.76–7.82 (m, 2H). ^{13}C-NMR: δ 56.1, 75.5, 78.4, 112.7, 115.8, 116.0, 121.3, 122.8, 125.2, 125.3, 130.4, 131.3, 149.5, 149.7, 154.9, 163.3, 165.8. ESI-HRMS (m/z): calcd. for ($C_{17}H_{12}Cl_2FN_2O$) [M+H$^+$] 349.0305, found 349.0307.

(*E*)-1-(2-Chlorophenyl)-2-{2,2-dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}diazene (8). Yield 201 mg (55%), orange-red oil. IR (ν, cm^{-1}): 1581, 1602, 3297. ^{1}H-NMR: δ 2.44 (t, 1H, J = 2.4 Hz), 4.63 (d, 2H, J = 2.3 Hz), 7.05–7.15 (m, 3H), 7.30–7.38 (m, 2H), 7.39-7.45 (m, 2H), 7.65 (dd, 1H, J = 7.8, 2.0 Hz). ^{13}C-NMR: δ 56.4, 75.5, 78.6, 112.6, 117.7, 121.3, 122.5, 127.1, 130.5, 130.6, 131.2, 131.9, 135.7, 137.3, 149.1, 150.6, 155.2. ESI-HRMS (m/z): calcd. for ($C_{17}H_{12}Cl_3N_2O$) [M + H^+] 365.0010, found 365.0005.

(*E*)-1-{2,2-Dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-(2,4-dichlorophenyl)diazene (9). Yield 244 mg (61%), orange-red solid, m.p. 104 °C. IR (ν, cm^{-1}): 1575, 1599, 3300. ^{1}H-NMR: δ 2.44 (t, 1H, J = 2.4 Hz), 4.62 (d, 2H, J = 2.3 Hz), 7.05-7.15 (m, 3H), 7.27-7.29 (m, 1H), 7.40--7.44 (m, 1H), 7.47 (d, 1H, J = 2.1 Hz), 7.63 (d, 1H, J = 8.7 Hz). ^{13}C-NMR: δ 56.3, 75.5, 78.4, 112.5, 118.5, 121.3, 122.3, 127.6, 130.3, 130.6, 131.2, 136.5, 137.4, 137.9, 147.6, 150.8, 155.1. ESI-HRMS (m/z): calcd. for ($C_{17}H_{11}Cl_4N_2O$) [M + H^+] 398.9620, found 398.9622.

(E)-1-(2,2-Dichloro-1-(2-(prop-2-yn-1-yloxy)phenyl)vinyl)-2-(2-methoxyphenyl)diazene (10). Yield 235 mg (65%), orange-red solid, m.p. 102 °C. IR (ν, cm^{-1}): 1570, 1585, 1594, 1607, 3235. ^{1}H-NMR: δ 2.44 (t, 1H, J = 2.4 Hz), 3.73 (s, 3H), 4.61 (d, 2H, J = 2.5 Hz), 6.95–7.00 (m, 2H), 7.07 (td, 1H, J = 7.4, 0.9 Hz), 7.14–7.16 (m, 2H), 7.35–7.42 (m, 2H), 7.62 (dd, 1H, J = 7.9, 1.6 Hz). ^{13}C-NMR: δ 56.6, 57.2, 75.3, 78.7, 113.1, 114.3, 117.3, 120.9, 121.5, 123.1, 130.3, 131.3, 132.7, 135.2, 143.0, 150.3, 155.3, 157.3. ESI-HRMS (m/z): calcd. for ($C_{18}H_{15}Cl_2N_2O_2$) [M + H^+] 361.0505, found 361.0519.

(*E*)-1-{2,2-Dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-(4-methoxyphenyl)diazene (11). Yield 101 mg (28%), orange-red solid, m.p. 69 °C. IR (ν, cm^{-1}): 1569, 1581, 1601, 3286. ^{1}H-NMR: δ 2.43 (t, 1H, J = 2.4 Hz), 3.86 (s, 3H), 4.62 (d, 2H, J = 2.4 Hz), 6.92 (dt, 2H, J = 9.8, 2.6 Hz), 7.07 (td, 1H, J = 7.3, 0.9 Hz), 7.10–7.14 (m, 2H), 7.39–7.44 (m, 1H), 7.77 (dt, 2H, J = 9.7, 2.6 Hz). ^{13}C-NMR: δ 55.5, 56.2, 75.4, 78.6, 112.8, 114.0, 121.3, 123.3, 125.2, 130.2, 131.4, 133.9, 147.4, 149.7, 155.0, 162.3. ESI-HRMS (m/z): calcd. for ($C_{18}H_{15}Cl_2N_2O_2$) [M + H^+] 361.0505, found 361.0512.

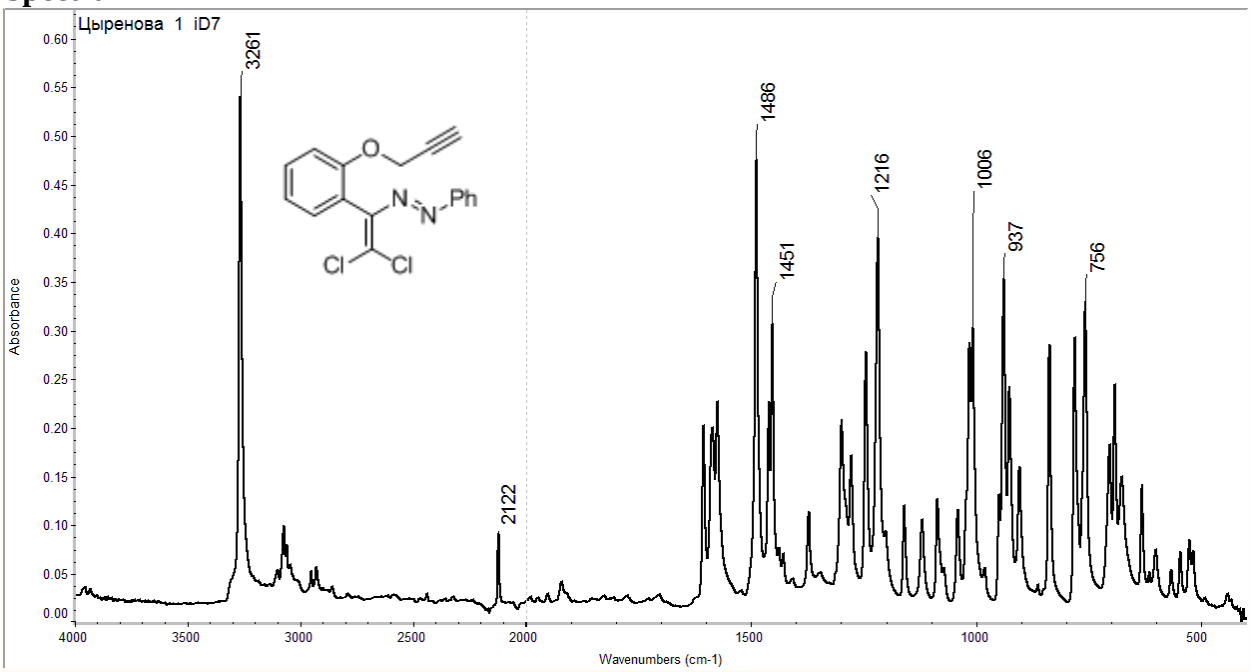
(*E*)-1-{2,2-Dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-(2,4-dimethylphenyl)diazene (12). Yield 320 mg (89%), orange-red oil. IR (ν, cm^{-1}): 1575, 1584, 1606, 3298. ^{1}H-NMR: δ 2.24 (s, 3H), 2.35 (s, 3H), 2.44 (t, 1H, J = 2.4 Hz), 4.62 (d, 2H, J = 2.4 Hz), 7.03–7.13 (m, 5H), 7.39–7.43 (m, 1H), 7.57–7.60 (m, 1H). ^{13}C- NMR: δ 17.0, 21.4, 56.1, 75.5, 78.5, 112.4, 115.4, 121.1, 123.6, 127.1, 130.1, 131.2, 131.7, 134.2, 139.0, 141.9, 148.9, 150.4, 154.9. ESI-HRMS (m/z): calcd. for ($C_{19}H_{17}Cl_2N_2O$) [M + H^+] 359.0712, found 359.0715.

(*E*)-1-{2,2-Dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}-2-(2,6-dimethylphenyl)diazene (13). Yield 320 mg (89%), orange-red oil. IR (ν, cm^{-1}): 1584, 1605, 3300. ^{1}H-NMR: δ 2.29 (s, 6H), 2.47 (t, 1H, J = 2.4 Hz), 4.65 (s, 2H), 7.09–7.18 (m, 6H), 7.42–7.46 (m, 1H). ^{13}C-NMR: δ 19.4, 56.0, 75.6, 78.3, 112.2, 121.2, 123.1, 129.0, 129.1, 130.2, 131.1, 135.5, 150.5, 150.6, 154.7. ESI-HRMS (m/z): calcd. for ($C_{19}H_{17}Cl_2N_2O$) [M + H^+] 359.0712, found 359.0718.

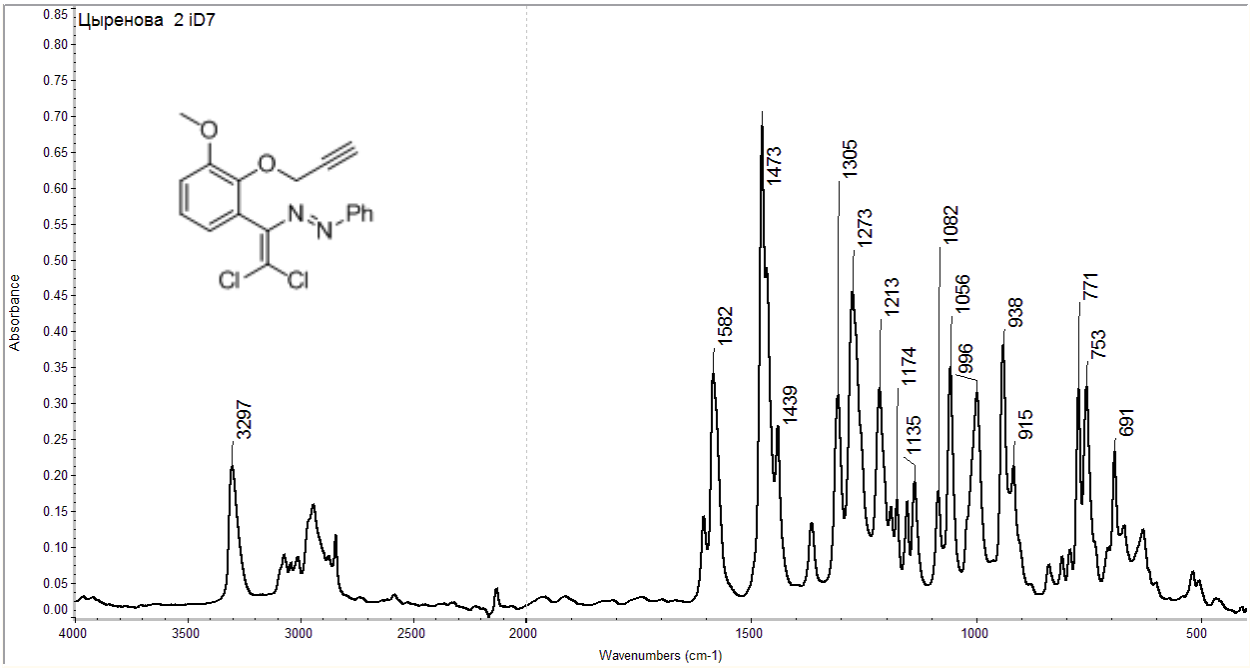
(*E*)-4-({2,2-Dichloro-1-[2-(prop-2-yn-1-yloxy)phenyl]vinyl}diazenyl)benzonitrile (14). Yield 146 mg (41%), orange-red solid, m.p. 154 °C. IR (ν, cm^{-1}): 1578, 1608, 3303. ^{1}H-NMR: δ 2.44 (t, 1H, J = 2.4 Hz), 4.63 (d, 2H, J = 2.3 Hz), 7.07–7.13 (m, 3H), 7.42–7.47 (m, 1H), 7.71–7.73 (m, 2H), 7.82–7.84 (m, 2H). ^{13}C-NMR: δ 55.9, 75.6, 78.2, 112.5, 114.0, 118.4, 121.4, 122.0, 123.6, 130.6, 131.2, 133.0, 139.3, 150.3, 154.7, 154.8. ESI-HRMS (m/z): calcd. for ($C_{18}H_{12}Cl_2N_3O$) [M + H^+] 356.0352, found 356.0347.

(*E*)-1-{2,2-Dichloro-1-[2-(prop-2-yn-1-yloxy)naphthalen-1-yl]vinyl}-2-phenyldiazene (15). Yield 175 mg (46%), orange-red oil. IR (ν, cm^{-1}): 1567, 1587, 1595, 3295. ^{1}H-NMR: δ 2.45 (t, 1H, J = 2.4 Hz), 4.75 (d, 2H, J = 2.4 Hz), 7.36-7.44 (m, 6H), 7.47 (d, 1H, J = 9.1 Hz), 7.70-7.73 (m, 2H), 7.85 (d, 1H, J=7.7 Hz), 7.96 (d, 1H, J = 9.1 Hz). ^{13}C-NMR: δ 57.0, 75.6, 78.8, 114.6, 117.9, 123.2, 124.1, 124.2, 127.2, 128.2, 128.8, 129.2, 131.0, 131.3, 132.1 136.9, 148.2, 152.7, 153.0. ESI-HRMS (m/z): calcd. for ($C_{21}H_{15}Cl_2N_2O$) [M + H^+] 381.0556, found 381.0564.

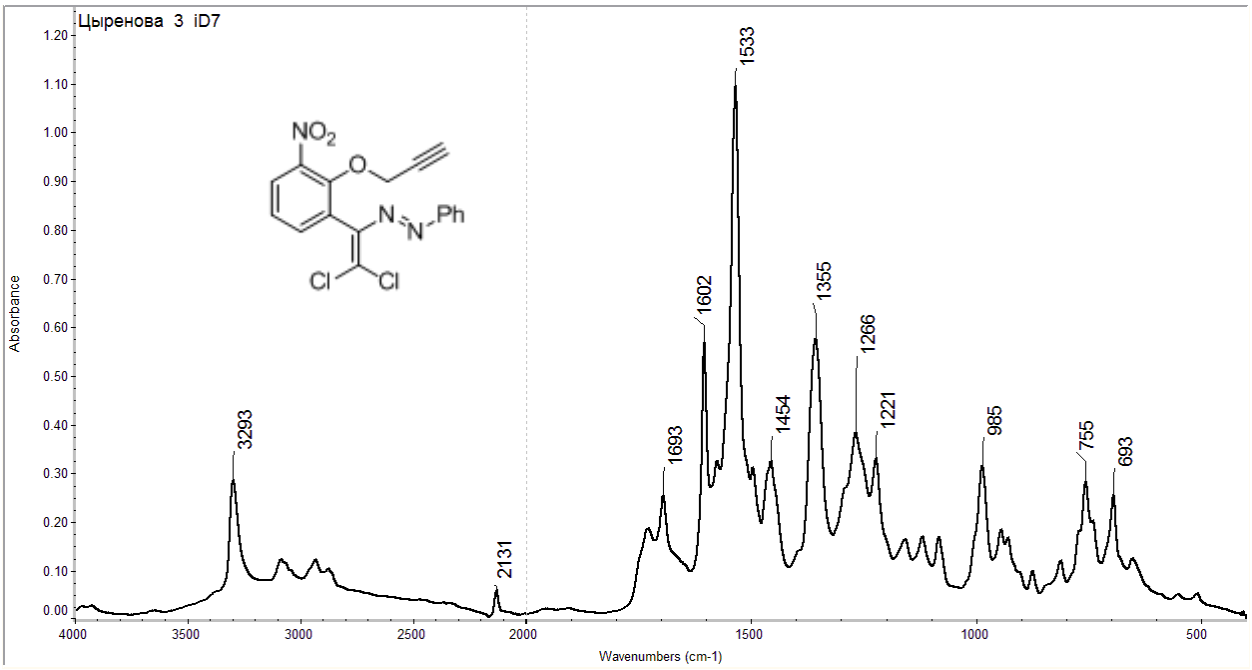
IR Spectra



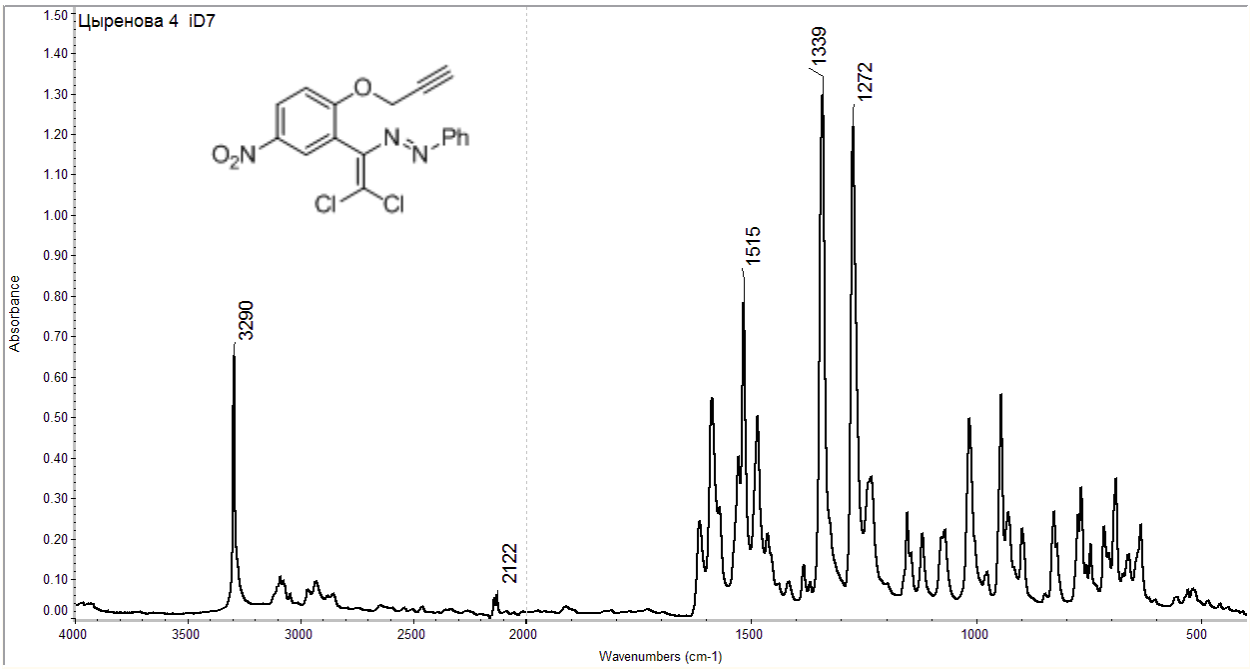
IR spectrum for **1** in the solid state.



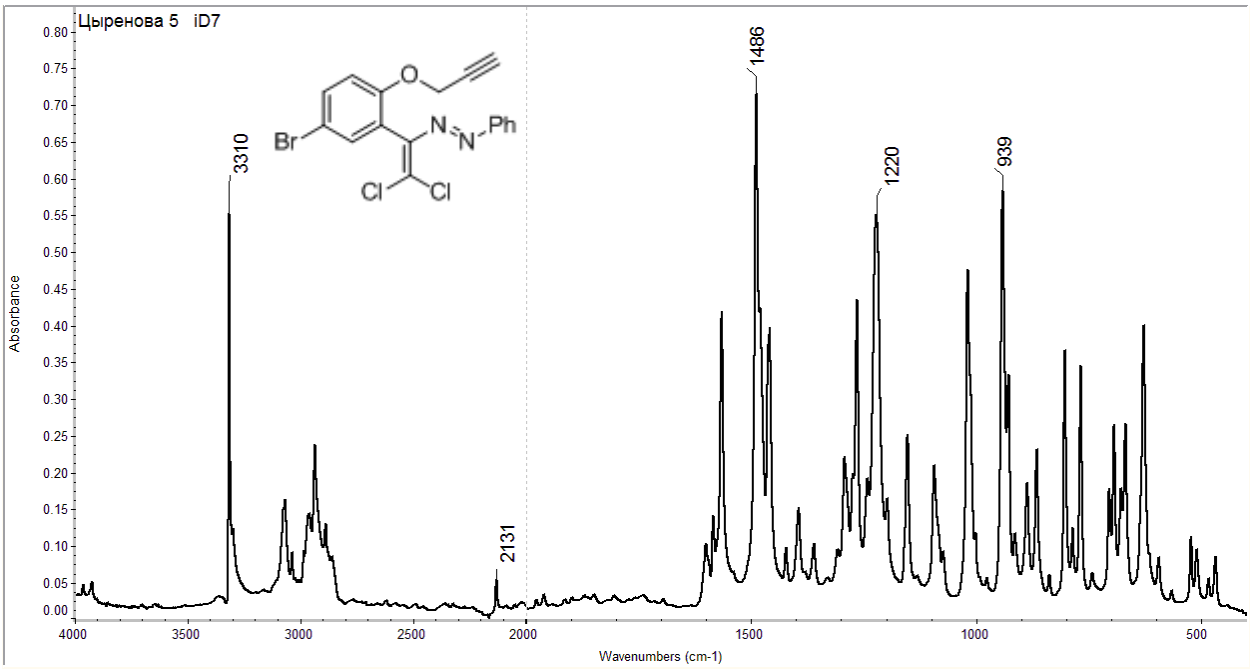
IR spectrum for **2** in the solid state.



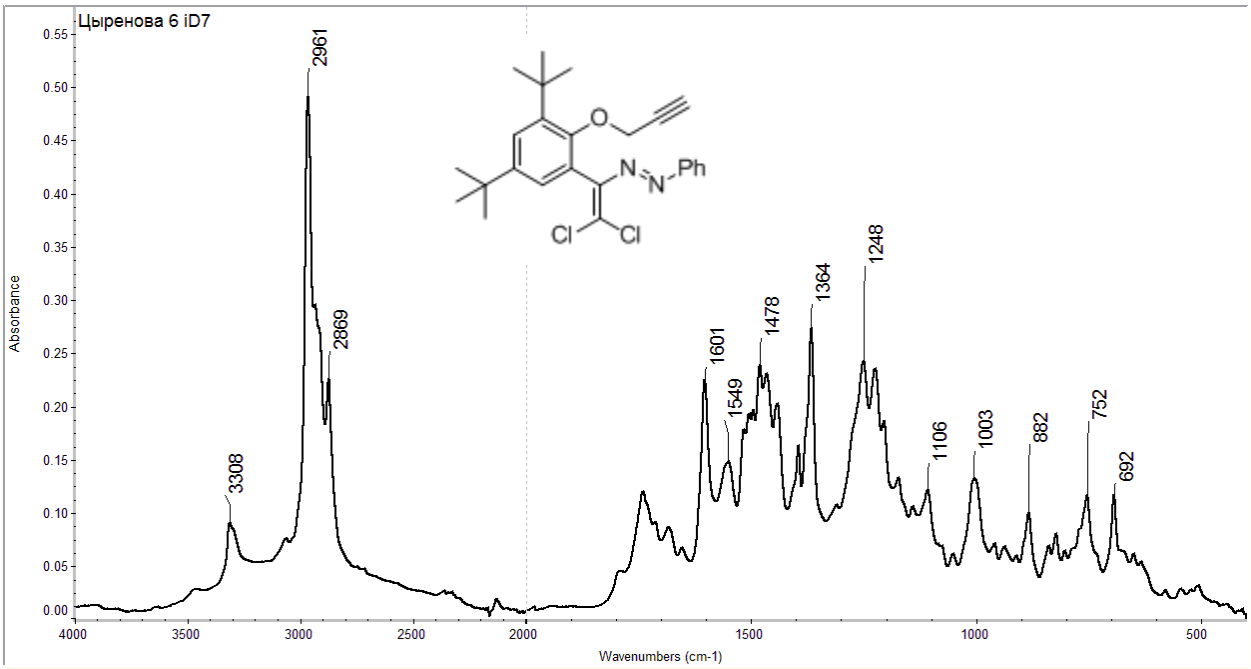
IR spectrum for **3** in the solid state.



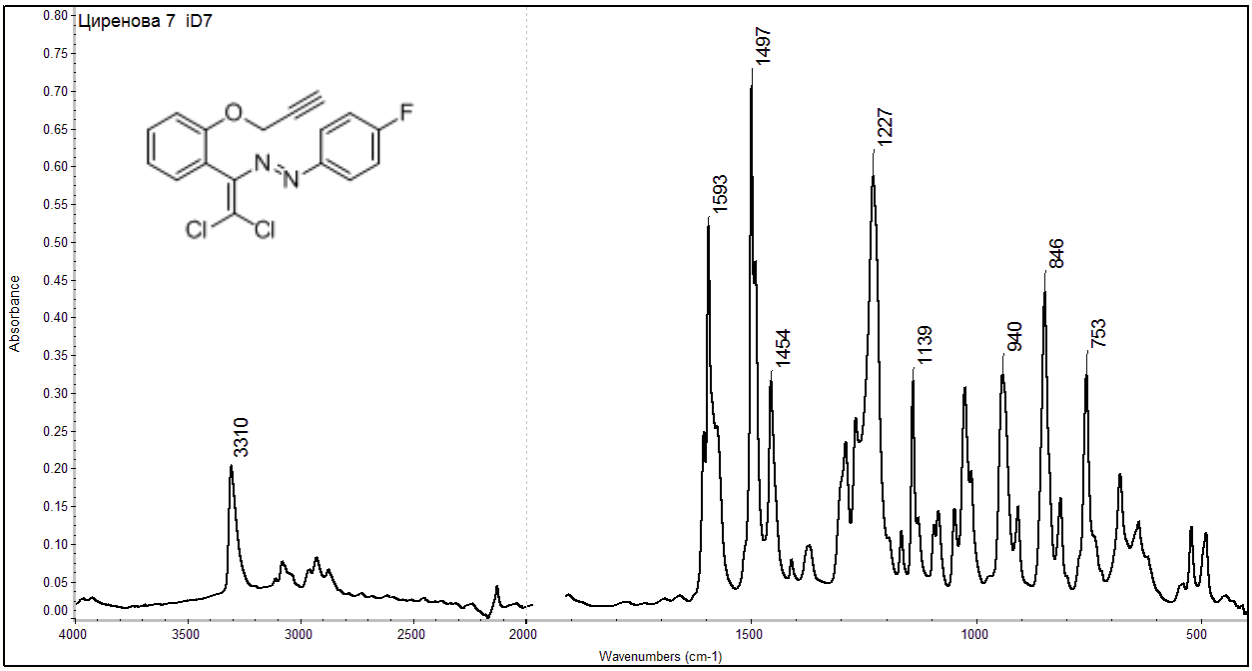
IR spectrum for **4** in the solid state.



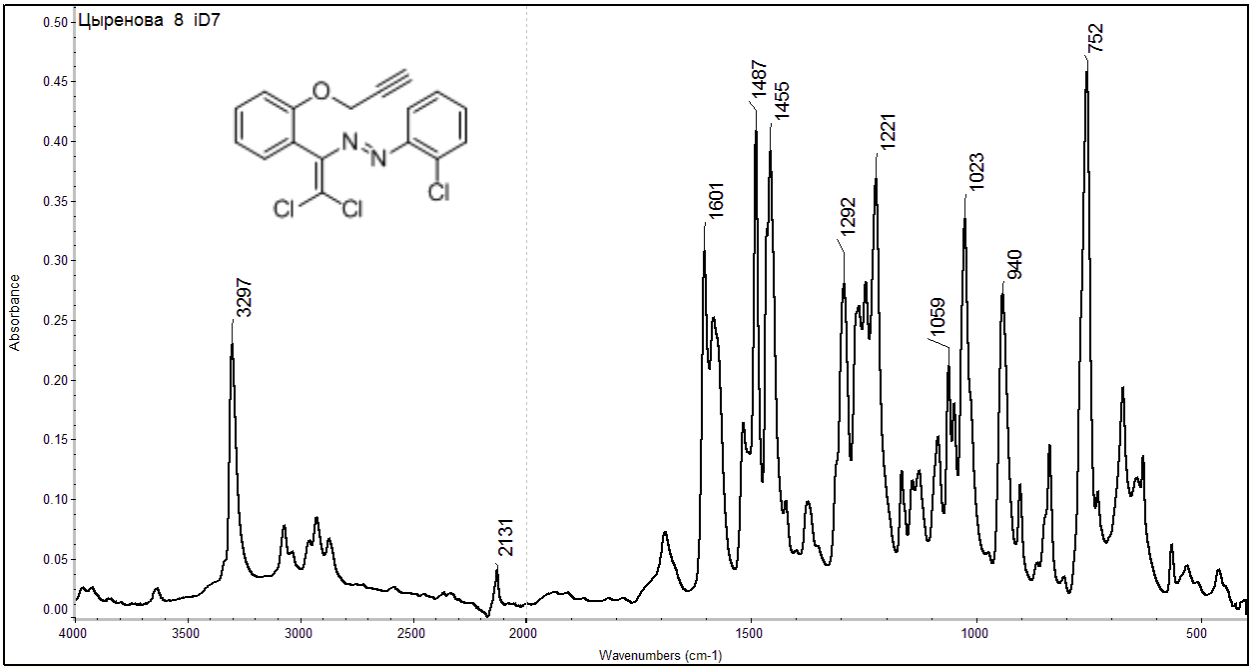
IR spectrum for **5** in the solid state.



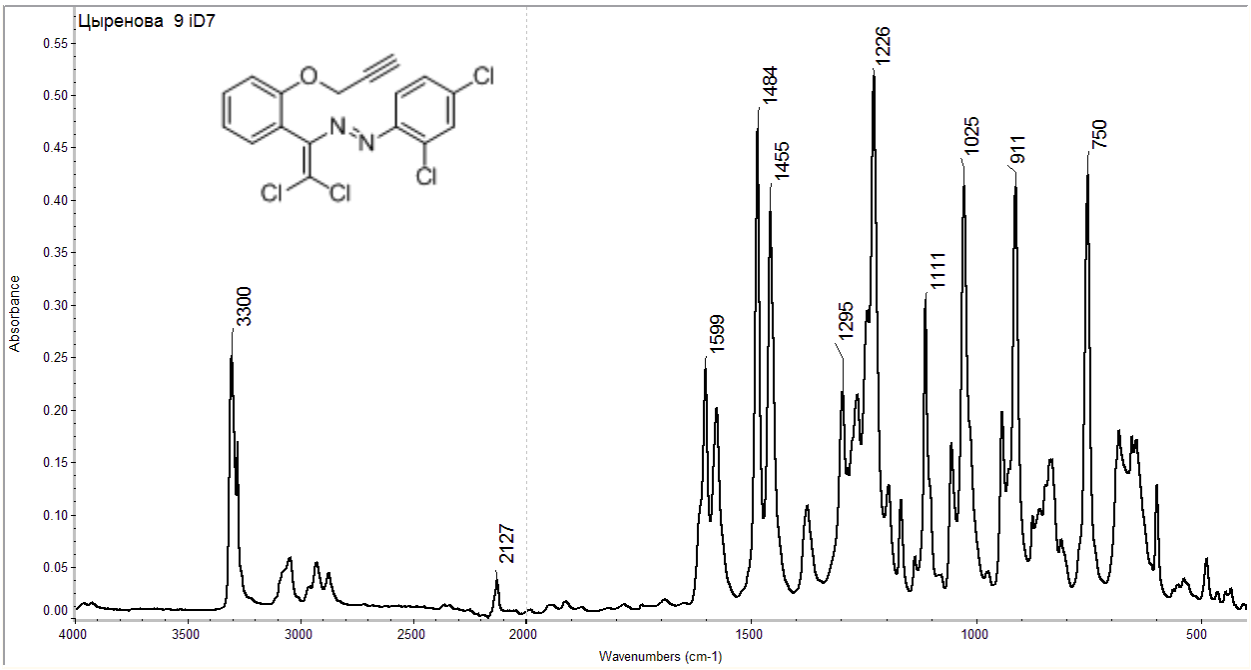
IR spectrum for **6** in the solid state.



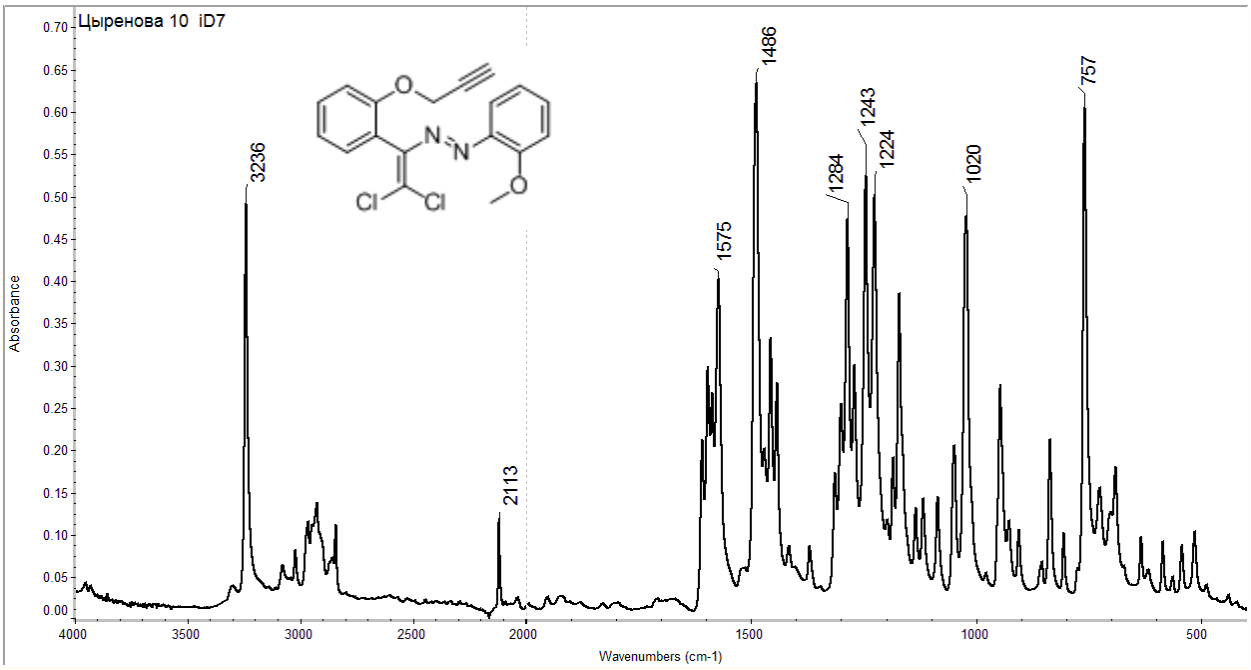
IR spectrum for **7** in the solid state.



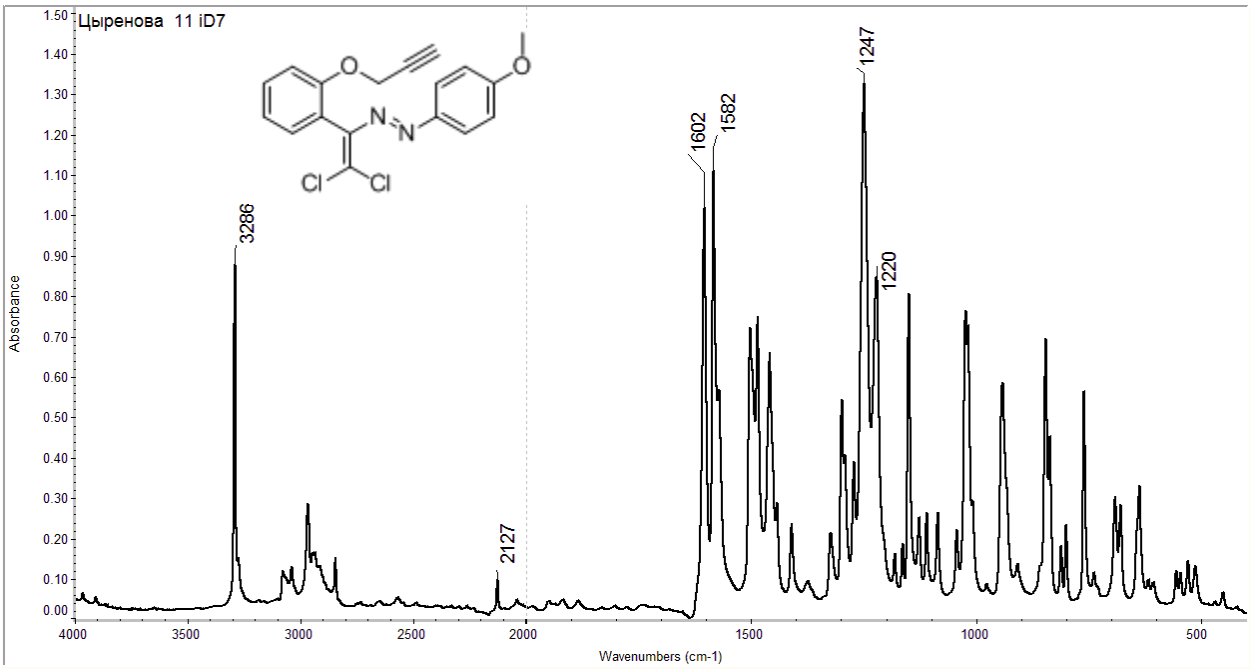
IR spectrum for **8** in the solid state.



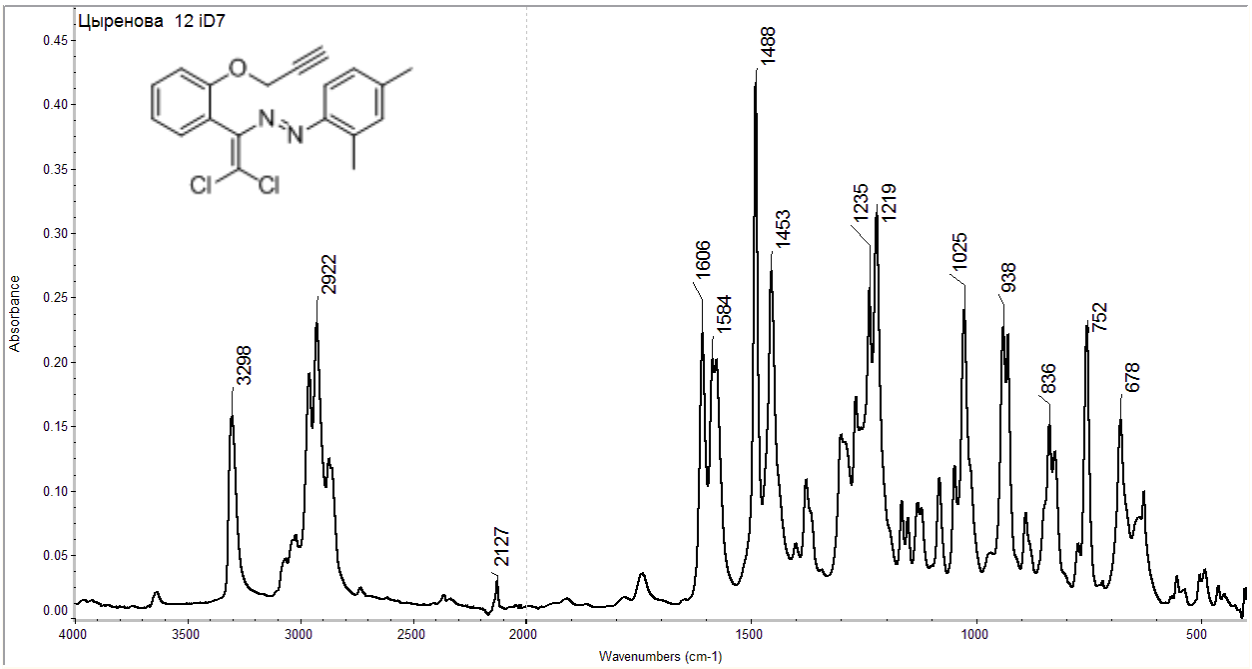
IR spectrum for **9** in the solid state.



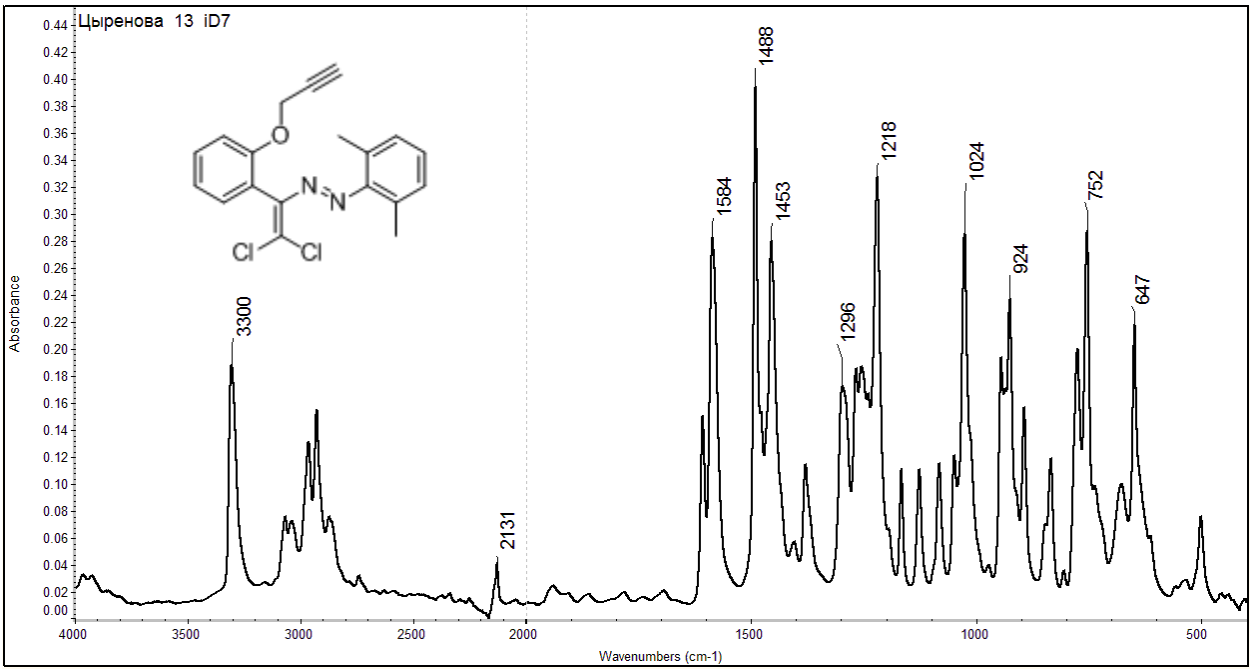
IR spectrum for **10** in the solid state.



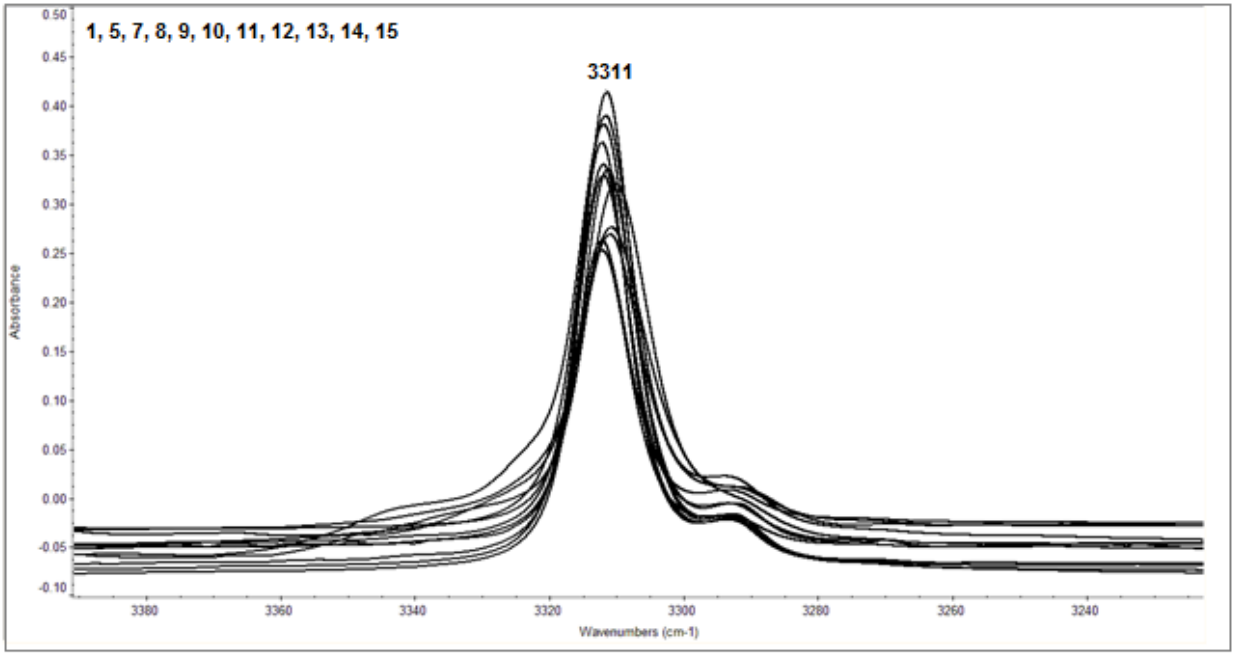
IR spectrum for **11** in the solid state.



IR spectrum for **12** in the solid state.



IR spectrum for **13** in the solid state.



IR spectra in the absorption region of CH valence vibrations in a CCl_4 solution of compounds **1**, **5**, **7**, **8**, **9**, **10**, **11**, **12**, **13**, **14**, **15**.

Crystallographic Data for 5

Table S1. Crystal data and structure refinement for ***5***.

Identification code	**5**	
Empirical formula	C17 H11 Br Cl2 N2 O	
Formula weight	410.08	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 8.8134(2) Å	α = 68.9550(10)°.
	b = 9.7628(3) Å	β = 69.5630(10)°.
	c = 11.2561(3) Å	γ = 71.3990(11)°.
Volume	826.01(4) Å^3	
Z	2	
Density (calculated)	1.649 Mg/m^3	
Absorption coefficient	2.816 mm^{-1}	
F(000)	408	
Crystal size	0.21 x 0.15 x 0.13 mm^3	
Theta range for data collection	2.292 to 32.594°.	
Index ranges	-13<=h<=13, -14<=k<=14, -17<=l<=17	
Reflections collected	18950	
Independent reflections	5968 [R(int) = 0.0195]	
Completeness to theta = 25.242°	98.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.681 and 0.559	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5968 / 0 / 208	
Goodness-of-fit on F^2	1.031	
Final R indices [for 5309 rflns with I>2σ(I)]	R1 = 0.0257, wR2 = 0.0682	
R indices (all data)	R1 = 0.0304, wR2 = 0.0711	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.880 and -0.250 e. Å^{-3}	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **5**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
Br(1)	1933(1)	2413(1)	3612(1)	32(1)
Cl(1)	8562(1)	6005(1)	3260(1)	32(1)
Cl(2)	8304(1)	4661(1)	1464(1)	35(1)
O(1)	9071(1)	959(1)	3708(1)	29(1)
N(1)	6936(1)	3733(1)	5351(1)	24(1)
N(2)	6217(1)	2772(1)	6294(1)	25(1)
C(1)	7100(1)	3652(1)	4089(1)	23(1)
C(2)	7879(1)	4655(1)	3065(1)	26(1)
C(3)	6448(1)	2562(1)	3869(1)	23(1)
C(4)	7492(1)	1222(1)	3617(1)	24(1)
C(5)	6880(1)	253(1)	3337(1)	27(1)
C(6)	5216(2)	599(1)	3346(1)	27(1)
C(7)	4193(1)	1923(1)	3612(1)	26(1)
C(8)	4785(1)	2905(1)	3881(1)	25(1)
C(9)	10323(2)	-145(1)	3125(1)	32(1)
C(10)	10672(2)	356(2)	1688(2)	39(1)
C(11)	10922(2)	811(3)	523(2)	56(1)
C(12)	6156(2)	2850(1)	7550(1)	26(1)
C(13)	7164(2)	3588(1)	7718(1)	30(1)
C(14)	7021(2)	3617(2)	8975(1)	37(1)
C(15)	5888(2)	2919(2)	10059(1)	39(1)
C(16)	4915(2)	2163(2)	9894(1)	37(1)
C(17)	5054(2)	2117(1)	8639(1)	31(1)

Table S3. Bond lengths [Å] and angles [°] for **5**

Br(1)-C(7)	1.8946(11)	C(8)-H(8)	0.9500
Cl(1)-C(2)	1.7180(11)	C(9)-C(10)	1.460(2)
Cl(2)-C(2)	1.7052(11)	C(9)-H(9A)	0.9900
O(1)-C(4)	1.3656(14)	C(9)-H(9B)	0.9900
O(1)-C(9)	1.4326(14)	C(10)-C(11)	1.186(3)
N(1)-N(2)	1.2658(13)	C(11)-H(11)	0.9500
N(1)-C(1)	1.4047(14)	C(12)-C(17)	1.3932(17)
N(2)-C(12)	1.4238(15)	C(12)-C(13)	1.4021(16)
C(1)-C(2)	1.3508(15)	C(13)-C(14)	1.3858(17)
C(1)-C(3)	1.4855(14)	C(13)-H(13)	0.9500
C(3)-C(8)	1.3916(15)	C(14)-C(15)	1.391(2)
C(3)-C(4)	1.3976(15)	C(14)-H(14)	0.9500
C(4)-C(5)	1.3938(15)	C(15)-C(16)	1.389(2)
C(5)-C(6)	1.3934(16)	C(15)-H(15)	0.9500
C(5)-H(5)	0.9500	C(16)-C(17)	1.3902(18)
C(6)-C(7)	1.3858(16)	C(16)-H(16)	0.9500
C(6)-H(6)	0.9500	C(17)-H(17)	0.9500
C(7)-C(8)	1.3856(15)		

C(4)-O(1)-C(9)	117.98(9)	C(4)-C(5)-H(5)	120.0
N(2)-N(1)-C(1)	114.54(9)	C(7)-C(6)-C(5)	119.25(10)
N(1)-N(2)-C(12)	112.37(9)	C(7)-C(6)-H(6)	120.4
C(2)-C(1)-N(1)	115.68(10)	C(5)-C(6)-H(6)	120.4
C(2)-C(1)-C(3)	121.19(10)	C(8)-C(7)-C(6)	121.48(10)
N(1)-C(1)-C(3)	123.13(9)	C(8)-C(7)-Br(1)	118.89(8)
C(1)-C(2)-Cl(2)	122.55(9)	C(6)-C(7)-Br(1)	119.63(8)
C(1)-C(2)-Cl(1)	123.14(9)	C(7)-C(8)-C(3)	119.24(10)
Cl(2)-C(2)-Cl(1)	114.29(6)	C(7)-C(8)-H(8)	120.4
C(8)-C(3)-C(4)	120.03(10)	C(3)-C(8)-H(8)	120.4
C(8)-C(3)-C(1)	119.16(9)	O(1)-C(9)-C(10)	111.22(11)
C(4)-C(3)-C(1)	120.79(10)	O(1)-C(9)-H(9A)	109.4
O(1)-C(4)-C(5)	125.06(10)	C(10)-C(9)-H(9A)	109.4
O(1)-C(4)-C(3)	114.95(9)	O(1)-C(9)-H(9B)	109.4
C(5)-C(4)-C(3)	119.97(10)	C(10)-C(9)-H(9B)	109.4
C(6)-C(5)-C(4)	120.01(10)	H(9A)-C(9)-H(9B)	108.0
C(6)-C(5)-H(5)	120.0	C(11)-C(10)-C(9)	177.71(18)
C(10)-C(11)-H(11)	180.0	C(16)-C(15)-C(14)	120.29(12)
C(17)-C(12)-C(13)	120.38(11)	C(16)-C(15)-H(15)	119.9
C(17)-C(12)-N(2)	116.58(10)	C(14)-C(15)-H(15)	119.9
C(13)-C(12)-N(2)	123.02(10)	C(15)-C(16)-C(17)	120.04(13)
C(14)-C(13)-C(12)	119.35(12)	C(15)-C(16)-H(16)	120.0
C(14)-C(13)-H(13)	120.3	C(17)-C(16)-H(16)	120.0
C(12)-C(13)-H(13)	120.3	C(16)-C(17)-C(12)	119.63(12)
C(13)-C(14)-C(15)	120.27(13)	C(16)-C(17)-H(17)	120.2
C(13)-C(14)-H(14)	119.9	C(12)-C(17)-H(17)	120.2
C(15)-C(14)-H(14)	119.9		

Table S4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	25(1)	34(1)	42(1)	-12(1)	-12(1)	-8(1)
Cl(1)	33(1)	29(1)	36(1)	-11(1)	-5(1)	-13(1)
Cl(2)	44(1)	36(1)	25(1)	-9(1)	-4(1)	-14(1)
O(1)	23(1)	29(1)	41(1)	-18(1)	-11(1)	-1(1)
N(1)	26(1)	24(1)	26(1)	-9(1)	-7(1)	-6(1)
N(2)	25(1)	26(1)	26(1)	-9(1)	-6(1)	-6(1)
C(1)	22(1)	23(1)	26(1)	-9(1)	-7(1)	-5(1)
C(2)	26(1)	26(1)	26(1)	-9(1)	-5(1)	-7(1)
C(3)	24(1)	24(1)	23(1)	-8(1)	-6(1)	-6(1)
C(4)	24(1)	25(1)	27(1)	-10(1)	-8(1)	-5(1)
C(5)	28(1)	25(1)	30(1)	-11(1)	-8(1)	-5(1)
C(6)	29(1)	27(1)	28(1)	-9(1)	-8(1)	-10(1)
C(7)	24(1)	27(1)	28(1)	-8(1)	-8(1)	-8(1)
C(8)	24(1)	26(1)	27(1)	-9(1)	-7(1)	-6(1)
C(9)	25(1)	30(1)	44(1)	-17(1)	-9(1)	-1(1)
C(10)	29(1)	44(1)	46(1)	-21(1)	-4(1)	-8(1)
C(11)	46(1)	77(1)	44(1)	-20(1)	-2(1)	-20(1)
C(12)	28(1)	25(1)	25(1)	-8(1)	-6(1)	-6(1)
C(13)	35(1)	31(1)	27(1)	-8(1)	-10(1)	-11(1)
C(14)	51(1)	38(1)	30(1)	-10(1)	-14(1)	-16(1)
C(15)	54(1)	39(1)	27(1)	-10(1)	-11(1)	-12(1)
C(16)	44(1)	38(1)	28(1)	-8(1)	-4(1)	-12(1)
C(17)	34(1)	31(1)	28(1)	-9(1)	-5(1)	-10(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **5**.

Atom	x	y	z	U(iso)
H(5)	7598	-642	3140	32
H(6)	4788	-66	3171	32
H(8)	4064	3803	4071	30
H(9A)	9946	-1100	3463	38
H(9B)	11353	-336	3383	38
H(11)	11122	1175	-410	67
H(13)	7937	4064	6977	36
H(14)	7699	4114	9097	45
H(15)	5781	2960	10916	47
H(16)	4153	1678	10639	45
H(17)	4402	1588	8526	37

Table S6. Torsion angles [°] for **5**.

C(1)-N(1)-N(2)-C(12)	177.03(9)
N(2)-N(1)-C(1)-C(2)	-179.32(10)
N(2)-N(1)-C(1)-C(3)	1.59(15)
N(1)-C(1)-C(2)-Cl(2)	176.07(8)
C(3)-C(1)-C(2)-Cl(2)	-4.83(16)
N(1)-C(1)-C(2)-Cl(1)	-2.59(15)
C(3)-C(1)-C(2)-Cl(1)	176.52(8)
C(2)-C(1)-C(3)-C(8)	-95.89(13)
N(1)-C(1)-C(3)-C(8)	83.15(13)
C(2)-C(1)-C(3)-C(4)	82.43(14)
N(1)-C(1)-C(3)-C(4)	-98.52(13)
C(9)-O(1)-C(4)-C(5)	18.55(17)
C(9)-O(1)-C(4)-C(3)	-163.25(11)
C(8)-C(3)-C(4)-O(1)	-176.26(10)
C(1)-C(3)-C(4)-O(1)	5.43(15)
C(8)-C(3)-C(4)-C(5)	2.04(17)
C(1)-C(3)-C(4)-C(5)	-176.27(10)
O(1)-C(4)-C(5)-C(6)	176.22(11)
C(3)-C(4)-C(5)-C(6)	-1.90(17)
C(4)-C(5)-C(6)-C(7)	1.14(17)
C(5)-C(6)-C(7)-C(8)	-0.53(18)
C(5)-C(6)-C(7)-Br(1)	179.45(9)
C(6)-C(7)-C(8)-C(3)	0.68(17)
Br(1)-C(7)-C(8)-C(3)	-179.31(8)
C(4)-C(3)-C(8)-C(7)	-1.42(16)
C(1)-C(3)-C(8)-C(7)	176.91(10)
C(4)-O(1)-C(9)-C(10)	67.94(14)
N(1)-N(2)-C(12)-C(17)	164.65(11)
N(1)-N(2)-C(12)-C(13)	-16.77(16)
C(17)-C(12)-C(13)-C(14)	-1.75(19)
N(2)-C(12)-C(13)-C(14)	179.72(12)
C(12)-C(13)-C(14)-C(15)	0.0(2)
C(13)-C(14)-C(15)-C(16)	1.2(2)
C(14)-C(15)-C(16)-C(17)	-0.8(2)
C(15)-C(16)-C(17)-C(12)	-1.0(2)
C(13)-C(12)-C(17)-C(16)	2.22(19)
N(2)-C(12)-C(17)-C(16)	-179.16(12)

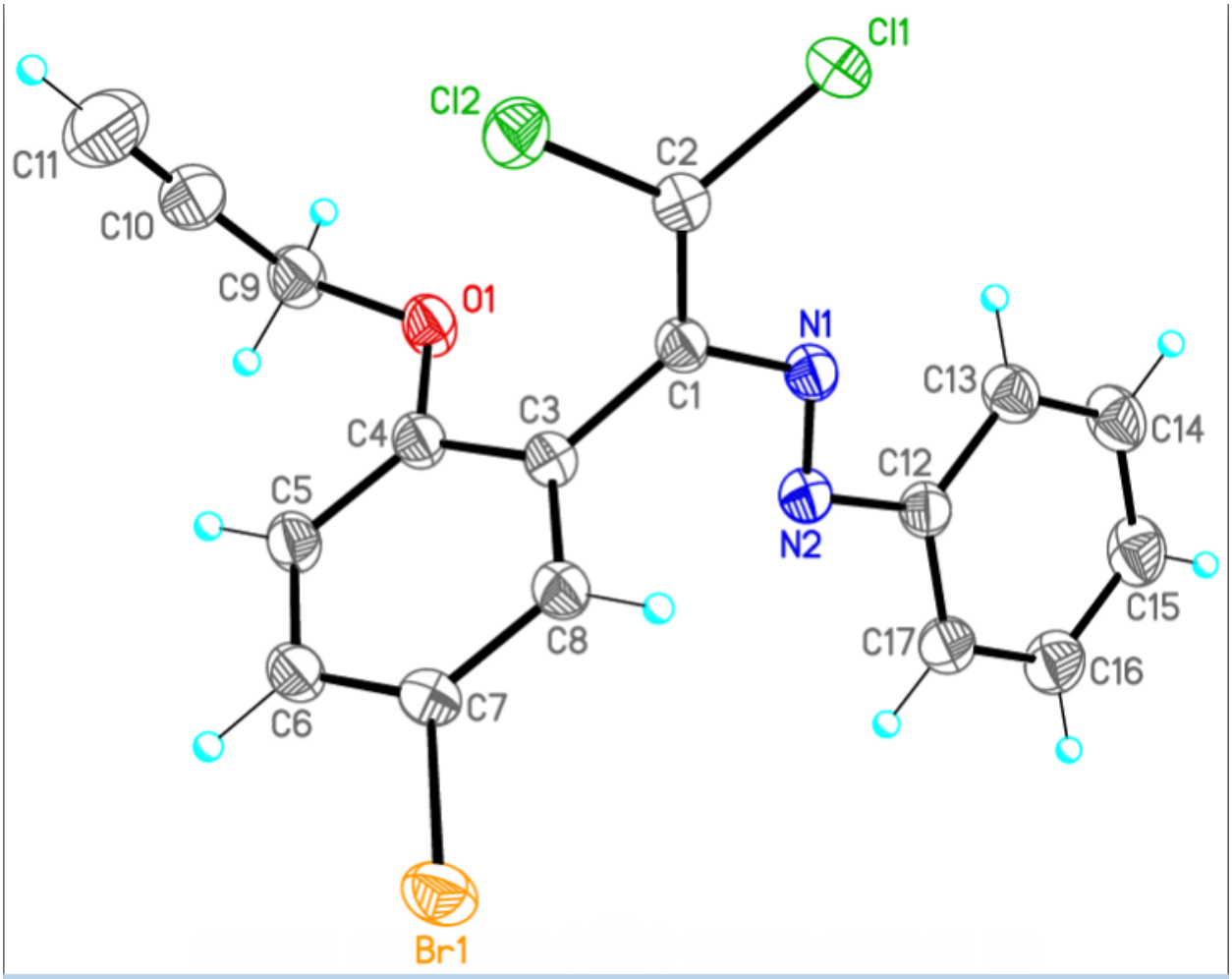


Figure S1. Structure **5** from X-ray study.

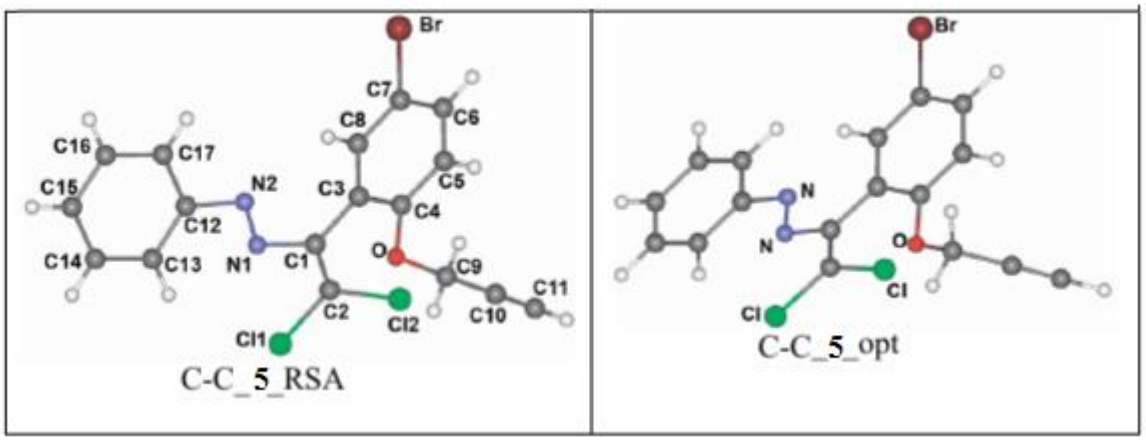


Figure S2. Structure **5** from X-ray study and from optimusation GGA PBE.

Comparison of Crystallographic and Computational Data for 5

Table S7. Comparison of Crystallographic and Computational Data for **5.**

	C-C_**5**_RSA	C-C_**5**_opt
C1-C2	1.351	1.366
C1-C3	1.485	1.490
C1-N1	1.266	1.394
C2-Cl1	1.718	1.732
C2-Cl2	1.705	1.728
C3-C4	1.398	1.412
C3-C8	1.392	1.396
C4-C5	1.394	1.399
C4-O	1.366	1.366
C5-C6	1.393	1.397
C6-C7	1.386	1.392
C7-C8	1.386	1.394
C9-O	1.433	1.436
C9-C10	1.460	1.460
C10-C11	1.186	1.212
C7-Br	1.895	1.924
N1-N2	1.266	1.278
N2-C12	1.424	1.410
C12-C13	1.402	1.409
C12-C17	1.393	1.405
C13-C14	1.386	1.390
C14-C15	1.391	1.403
C15-C16	1.389	1.398
C16-C17	1.390	1.394

Crystallographic Data for 14

Table S8. Crystal data and structure refinement for **14**.

Identification code	**14**	
Empirical formula	C18 H11 Cl2 N3 O	
Formula weight	356.20	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 9.3027(3) Å	α = 79.0640(10)°.
	b = 9.5857(3) Å	β = 81.9050(11)°.
	c = 10.0639(3) Å	γ = 72.6290(10)°.
Volume	837.54(5) $Å^3$	
Z	2	
Density (calculated)	1.412 Mg/m^3	
Absorption coefficient	0.397 mm^{-1}	
F(000)	364	
Crystal size	0.15 x 0.13 x 0.11 mm^3	
Theta range for data collection	2.303 to 32.566°.	
Index ranges	-14<=h<=14, -14<=k<=14, -15<=l<=15	
Reflections collected	15137	
Independent reflections	6045 [R(int) = 0.0411]	
Completeness to theta = 25.242°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.949 and 0.933	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6045 / 0 / 217	
Goodness-of-fit on F^2	1.032	
Final R indices [for 4373 rflns with I>2σ(I)]	R1 = 0.0428, wR2 = 0.0860	
R indices (all data)	R1 = 0.0719, wR2 = 0.1006	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.389 and -0.334 e. $Å^{-3}$	

Table S9. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **14** U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
Cl(1)	6465(1)	1379(1)	4955(1)	28(1)
Cl(2)	5530(1)	77(1)	7624(1)	29(1)
O(1)	3645(1)	3981(1)	8319(1)	20(1)
N(1)	6942(1)	3633(1)	6287(1)	18(1)
N(2)	7297(1)	4519(1)	6876(1)	18(1)
N(3)	9994(2)	9856(2)	2850(1)	26(1)
C(1)	6404(2)	2552(2)	7206(1)	17(1)
C(2)	6161(2)	1478(2)	6658(1)	20(1)
C(3)	6136(2)	2607(2)	8689(1)	17(1)
C(4)	4716(2)	3362(2)	9233(1)	17(1)
C(5)	4463(2)	3488(2)	10605(1)	20(1)
C(6)	5633(2)	2843(2)	11432(1)	22(1)
C(7)	7040(2)	2070(2)	10918(2)	24(1)
C(8)	7291(2)	1960(2)	9540(2)	22(1)
C(9)	2125(2)	4587(2)	8861(2)	22(1)
C(10)	1476(2)	3433(2)	9664(2)	23(1)
C(11)	1032(2)	2456(2)	10322(2)	29(1)
C(12)	7820(2)	5632(2)	5956(1)	17(1)
C(13)	7436(2)	6110(2)	4626(1)	21(1)
C(14)	7993(2)	7216(2)	3828(2)	22(1)
C(15)	8938(2)	7815(2)	4362(1)	19(1)
C(16)	9308(2)	7347(2)	5702(2)	23(1)
C(17)	8722(2)	6266(2)	6501(2)	22(1)
C(18)	9526(2)	8953(2)	3525(2)	21(1)

Table S10. Bond lengths [Å] and angles [°] for **14**

Cl(1)-C(2)	1.7136(14)	C(7)-H(7)	0.9500
Cl(2)-C(2)	1.7052(15)	C(8)-H(8)	0.9500
O(1)-C(4)	1.3741(17)	C(9)-C(10)	1.470(2)
O(1)-C(9)	1.4303(17)	C(9)-H(9A)	0.9900
N(1)-N(2)	1.2623(16)	C(9)-H(9B)	0.9900
N(1)-C(1)	1.4125(18)	C(10)-C(11)	1.185(2)
N(2)-C(12)	1.4286(17)	C(11)-H(11)	0.9500
N(3)-C(18)	1.1486(19)	C(12)-C(13)	1.3905(19)
C(1)-C(2)	1.3430(19)	C(12)-C(17)	1.391(2)
C(1)-C(3)	1.4865(19)	C(13)-C(14)	1.387(2)
C(3)-C(8)	1.390(2)	C(13)-H(13)	0.9500
C(3)-C(4)	1.3948(19)	C(14)-C(15)	1.394(2)
C(4)-C(5)	1.3901(19)	C(14)-H(14)	0.9500
C(5)-C(6)	1.384(2)	C(15)-C(16)	1.395(2)
C(5)-H(5)	0.9500	C(15)-C(18)	1.441(2)
C(6)-C(7)	1.380(2)	C(16)-C(17)	1.384(2)
C(6)-H(6)	0.9500	C(16)-H(16)	0.9500
C(7)-C(8)	1.392(2)	C(17)-H(17)	0.9500
C(4)-O(1)-C(9)	117.09(11)	O(1)-C(9)-C(10)	111.83(12)
N(2)-N(1)-C(1)	112.87(11)	O(1)-C(9)-H(9A)	109.3
N(1)-N(2)-C(12)	113.40(11)	C(10)-C(9)-H(9A)	109.3
C(2)-C(1)-N(1)	116.00(12)	O(1)-C(9)-H(9B)	109.3
C(2)-C(1)-C(3)	122.50(12)	C(10)-C(9)-H(9B)	109.3
N(1)-C(1)-C(3)	121.50(12)	H(9A)-C(9)-H(9B)	107.9
C(1)-C(2)-Cl(2)	122.08(11)	C(11)-C(10)-C(9)	176.35(17)
C(1)-C(2)-Cl(1)	123.65(11)	C(10)-C(11)-H(11)	180.0
Cl(2)-C(2)-Cl(1)	114.27(8)	C(13)-C(12)-C(17)	121.05(13)
C(8)-C(3)-C(4)	119.21(13)	C(13)-C(12)-N(2)	123.96(13)
C(8)-C(3)-C(1)	121.17(13)	C(17)-C(12)-N(2)	114.95(12)
C(4)-C(3)-C(1)	119.58(12)	C(14)-C(13)-C(12)	119.07(14)
O(1)-C(4)-C(5)	124.09(13)	C(14)-C(13)-H(13)	120.5
O(1)-C(4)-C(3)	115.44(12)	C(12)-C(13)-H(13)	120.5
C(5)-C(4)-C(3)	120.45(13)	C(13)-C(14)-C(15)	119.75(13)
C(6)-C(5)-C(4)	119.33(14)	C(13)-C(14)-H(14)	120.1
C(6)-C(5)-H(5)	120.3	C(15)-C(14)-H(14)	120.1
C(4)-C(5)-H(5)	120.3	C(14)-C(15)-C(16)	121.12(13)
C(7)-C(6)-C(5)	121.11(13)	C(14)-C(15)-C(18)	119.55(13)
C(7)-C(6)-H(6)	119.4	C(16)-C(15)-C(18)	119.32(13)
C(5)-C(6)-H(6)	119.4	C(17)-C(16)-C(15)	118.80(14)
C(6)-C(7)-C(8)	119.32(14)	C(17)-C(16)-H(16)	120.6
C(6)-C(7)-H(7)	120.3	C(15)-C(16)-H(16)	120.6
C(8)-C(7)-H(7)	120.3	C(16)-C(17)-C(12)	120.15(13)
C(3)-C(8)-C(7)	120.56(14)	C(16)-C(17)-H(17)	119.9
C(3)-C(8)-H(8)	119.7	C(12)-C(17)-H(17)	119.9
C(7)-C(8)-H(8)	119.7	N(3)-C(18)-C(15)	179.54(17)

Table S11. Anisotropic displacement parameters ($Å^2$ x 10^3) for **14**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	41(1)	26(1)	18(1)	-8(1)	0(1)	-10(1)
Cl(2)	44(1)	25(1)	25(1)	-2(1)	-2(1)	-20(1)
O(1)	17(1)	27(1)	16(1)	-3(1)	-1(1)	-5(1)
N(1)	19(1)	17(1)	17(1)	-3(1)	1(1)	-6(1)
N(2)	19(1)	20(1)	16(1)	-3(1)	0(1)	-7(1)
N(3)	25(1)	24(1)	27(1)	0(1)	-1(1)	-8(1)
C(1)	17(1)	18(1)	16(1)	-3(1)	0(1)	-4(1)
C(2)	24(1)	20(1)	17(1)	-3(1)	0(1)	-7(1)
C(3)	20(1)	17(1)	15(1)	-1(1)	0(1)	-9(1)
C(4)	18(1)	18(1)	16(1)	-1(1)	-1(1)	-8(1)
C(5)	23(1)	24(1)	16(1)	-5(1)	2(1)	-12(1)
C(6)	30(1)	27(1)	14(1)	-1(1)	-2(1)	-17(1)
C(7)	27(1)	28(1)	19(1)	4(1)	-7(1)	-12(1)
C(8)	21(1)	24(1)	20(1)	0(1)	-2(1)	-6(1)
C(9)	18(1)	24(1)	23(1)	-3(1)	0(1)	-4(1)
C(10)	20(1)	27(1)	24(1)	-7(1)	-2(1)	-6(1)
C(11)	25(1)	31(1)	32(1)	-7(1)	1(1)	-12(1)
C(12)	18(1)	17(1)	16(1)	-2(1)	2(1)	-6(1)
C(13)	26(1)	22(1)	18(1)	-3(1)	-2(1)	-10(1)
C(14)	28(1)	21(1)	16(1)	0(1)	-2(1)	-9(1)
C(15)	20(1)	16(1)	21(1)	-3(1)	2(1)	-5(1)
C(16)	26(1)	25(1)	21(1)	-2(1)	-3(1)	-12(1)
C(17)	26(1)	24(1)	17(1)	0(1)	-3(1)	-11(1)
C(18)	20(1)	20(1)	21(1)	-3(1)	1(1)	-4(1)

Table S12. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for **14**.

Atom	x	y	z	U(iso)
H(5)	3497	4010	10972	24
H(6)	5464	2934	12369	27
H(7)	7829	1619	11498	29
H(8)	8259	1437	9179	26
H(9A)	1495	5112	8105	26
H(9B)	2110	5316	9443	26
H(11)	676	1673	10850	35
H(13)	6802	5685	4269	25
H(14)	7731	7564	2921	26
H(16)	9950	7763	6059	28
H(17)	8936	5956	7424	26

Table S13. Torsion angles [°] for **14.**

C(1)-N(1)-N(2)-C(12)	-179.09(11)
N(2)-N(1)-C(1)-C(2)	-173.04(13)
N(2)-N(1)-C(1)-C(3)	6.66(19)
N(1)-C(1)-C(2)-Cl(2)	179.39(10)
C(3)-C(1)-C(2)-Cl(2)	-0.3(2)
N(1)-C(1)-C(2)-Cl(1)	-0.1(2)
C(3)-C(1)-C(2)-Cl(1)	-179.76(11)
C(2)-C(1)-C(3)-C(8)	92.47(18)
N(1)-C(1)-C(3)-C(8)	-87.21(17)
C(2)-C(1)-C(3)-C(4)	-89.59(18)
N(1)-C(1)-C(3)-C(4)	90.73(17)
C(9)-O(1)-C(4)-C(5)	-10.19(19)
C(9)-O(1)-C(4)-C(3)	171.65(12)
C(8)-C(3)-C(4)-O(1)	179.35(12)
C(1)-C(3)-C(4)-O(1)	1.37(18)
C(8)-C(3)-C(4)-C(5)	1.1(2)
C(1)-C(3)-C(4)-C(5)	-176.86(12)
O(1)-C(4)-C(5)-C(6)	-178.72(13)
C(3)-C(4)-C(5)-C(6)	-0.6(2)
C(4)-C(5)-C(6)-C(7)	-0.5(2)
C(5)-C(6)-C(7)-C(8)	1.1(2)
C(4)-C(3)-C(8)-C(7)	-0.5(2)
C(1)-C(3)-C(8)-C(7)	177.43(13)
C(6)-C(7)-C(8)-C(3)	-0.6(2)
C(4)-O(1)-C(9)-C(10)	-66.36(16)
N(1)-N(2)-C(12)-C(13)	24.8(2)
N(1)-N(2)-C(12)-C(17)	-157.35(13)
C(17)-C(12)-C(13)-C(14)	1.4(2)
N(2)-C(12)-C(13)-C(14)	179.04(13)
C(12)-C(13)-C(14)-C(15)	0.8(2)
C(13)-C(14)-C(15)-C(16)	-1.6(2)
C(13)-C(14)-C(15)-C(18)	179.38(14)
C(14)-C(15)-C(16)-C(17)	0.2(2)
C(18)-C(15)-C(16)-C(17)	179.23(14)
C(15)-C(16)-C(17)-C(12)	1.9(2)
C(13)-C(12)-C(17)-C(16)	-2.8(2)
N(2)-C(12)-C(17)-C(16)	179.34(13)

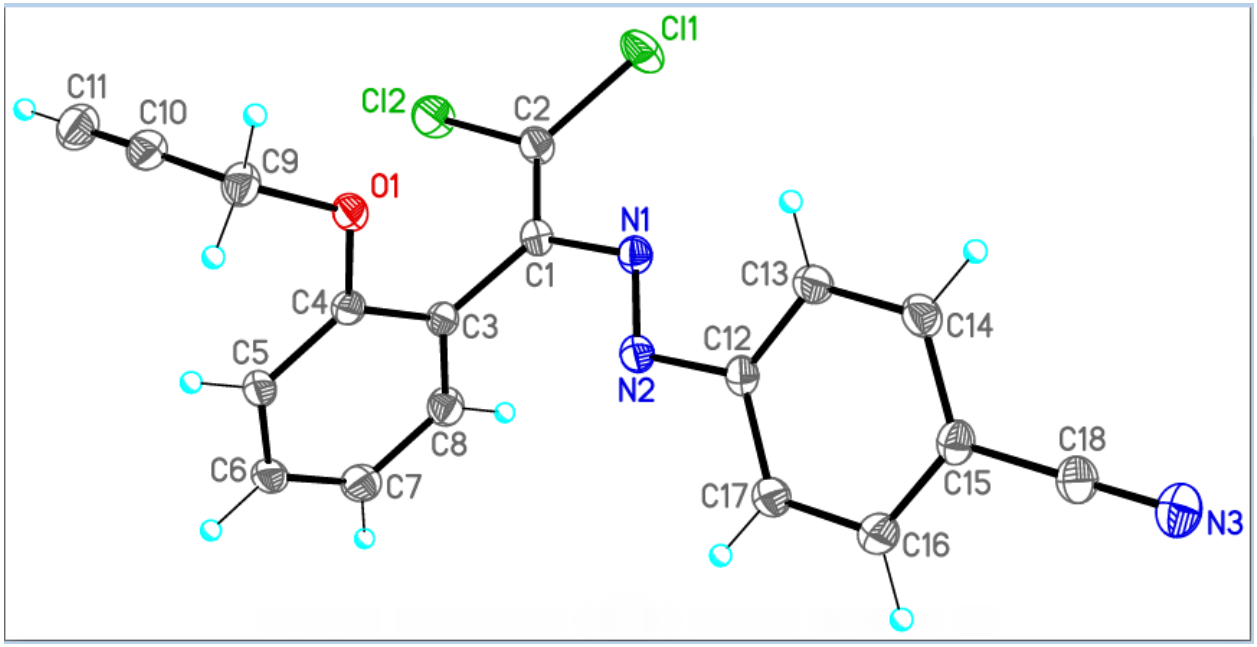


Figure S3. Structure **14** from X-ray study.

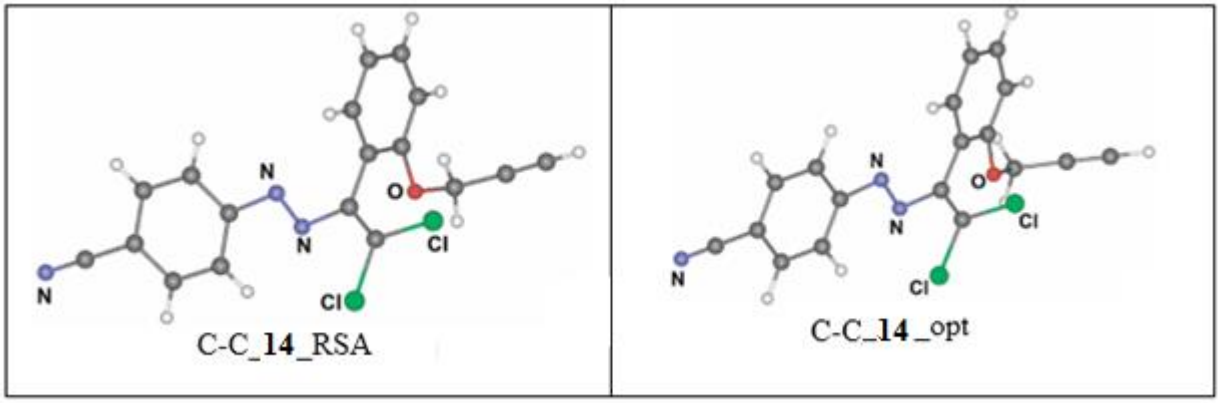


Figure S4. Structure **14** from X-ray study and from .optimusation GGA PBE.

Comparison of Crystallographic and Computational Data for 14

Table S14. Comparison of Crystallographic and Computational Data for **14.**

	C-C_**14**_RSA	C-C_**14**_opt
C1-C2	1.343	1.369
C1-C3	1.486	1.488
C1-N1	1.412	1.391
C2-Cl1	1.714	1.731
C2-Cl2	1.705	1.726
C3-C4	1.395	1.412
C3-C8	1.390	1.397
C4-C5	1.390	1.400
C4-O	1.374	1.368
C5-C6	1.384	1.398
C6-C7	1.380	1.394
C7-C8	1.392	1.396
C9-O	1.430	1.436
C9-C10	1.470	1.460
C10-C11	1.185	1.212
N1-N2	1.262	1.279
N2-C12	1.429	1.409
C12-C13	1.391	1.410
C12-C17	1.391	1.406
C13-C14	1.387	1.385
C14-C15	1.394	1.412
C15-C16	1.395	1.407
C16-C17	1.384	1.389
C15-C18	1.441	1.427
C18-N	1.149	1.166

References

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NMR Spectra

22.04.2019 21:59:35

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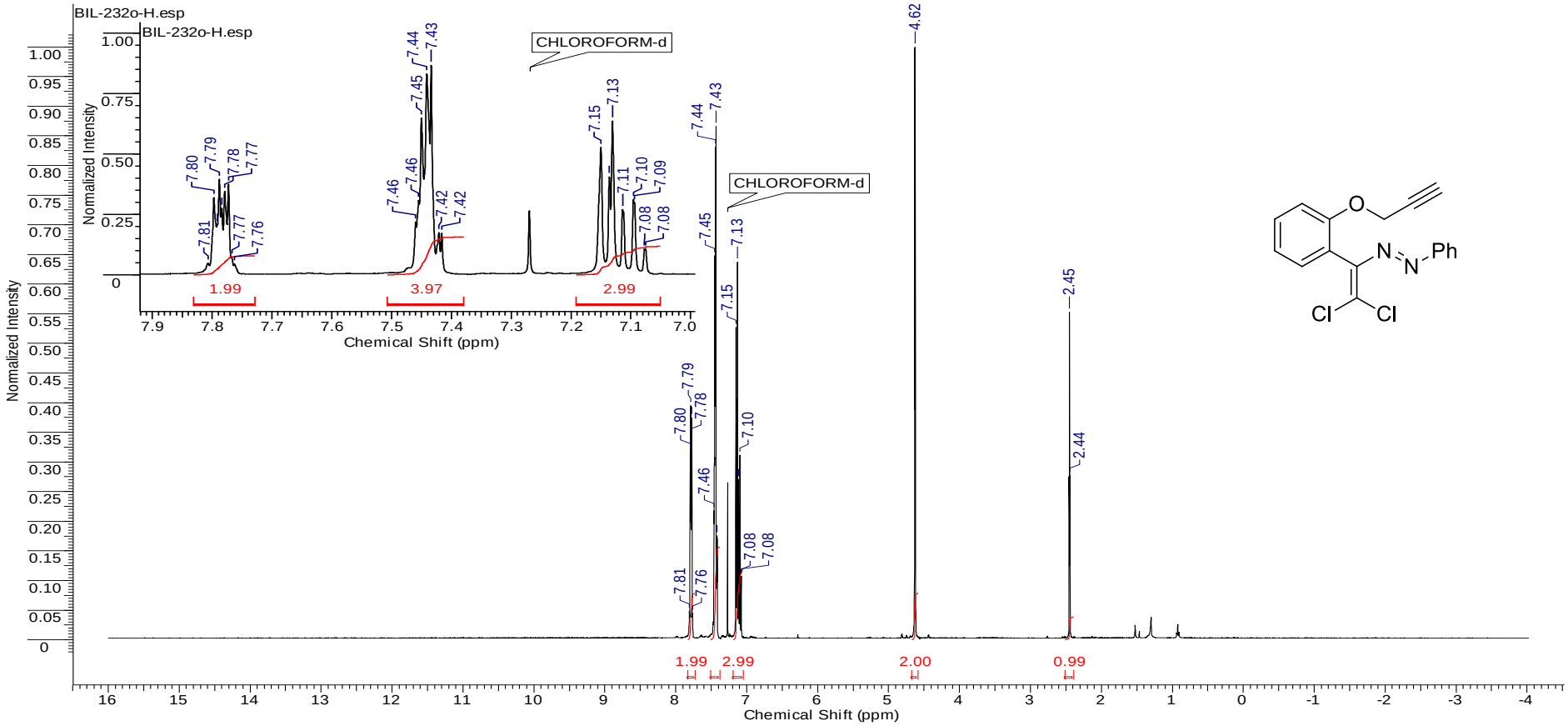
Owner	root	***Points Count***	131072	***Pulse Sequence***	zg30	***Receiver Gain***	114.00
SW(cyclical) (Hz)	8012.82	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2395.8254
Sweep Width (Hz)	8012.76	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **1** (400.1 MHz, $CDCl_3$)

22.04.2019 21:54:14

Acquisition Time (sec)	0.6783	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	01 Feb 2019 13:05:04
Date Stamp	01 Feb 2019 13:05:04						
File Name						***Frequency (MHz)***	100.61
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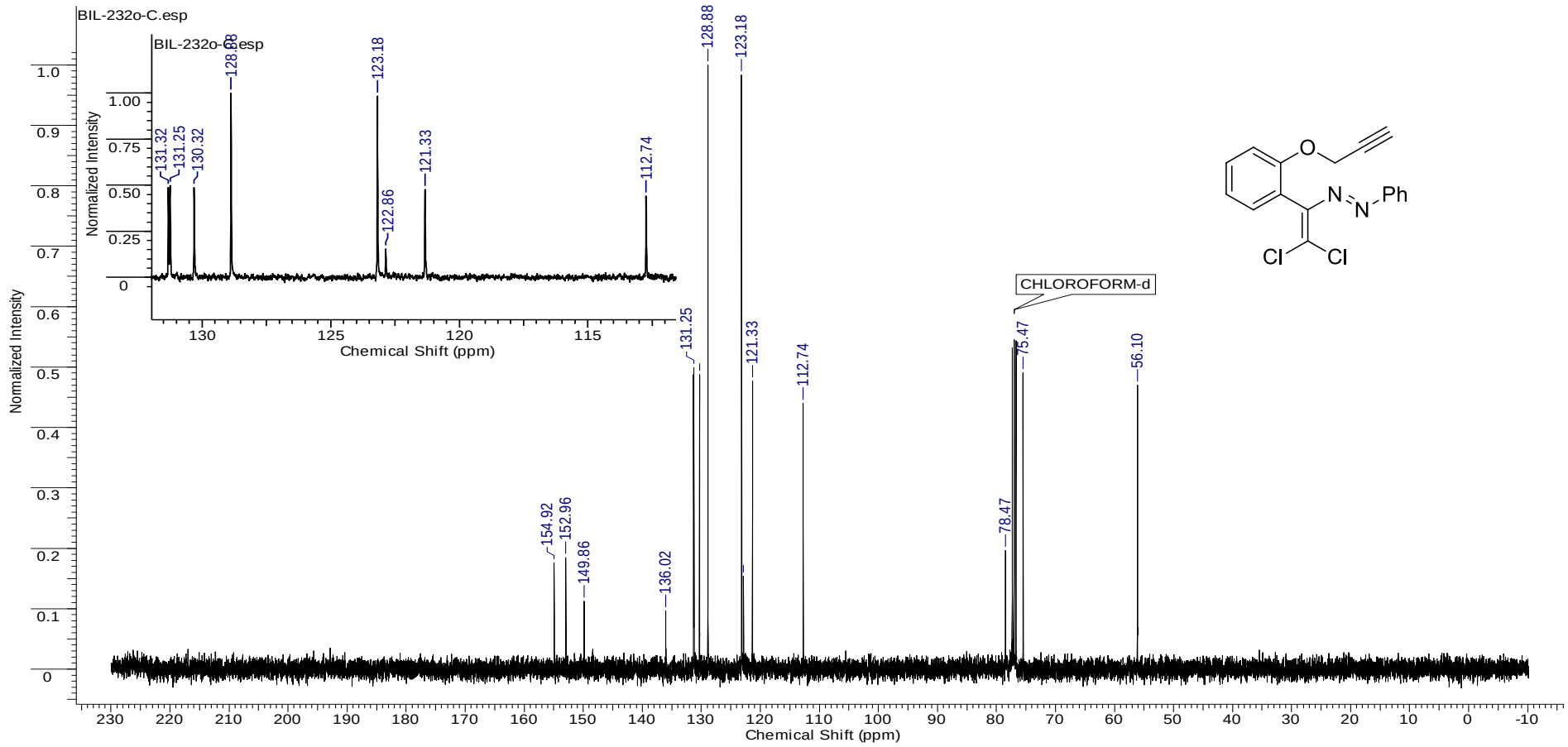
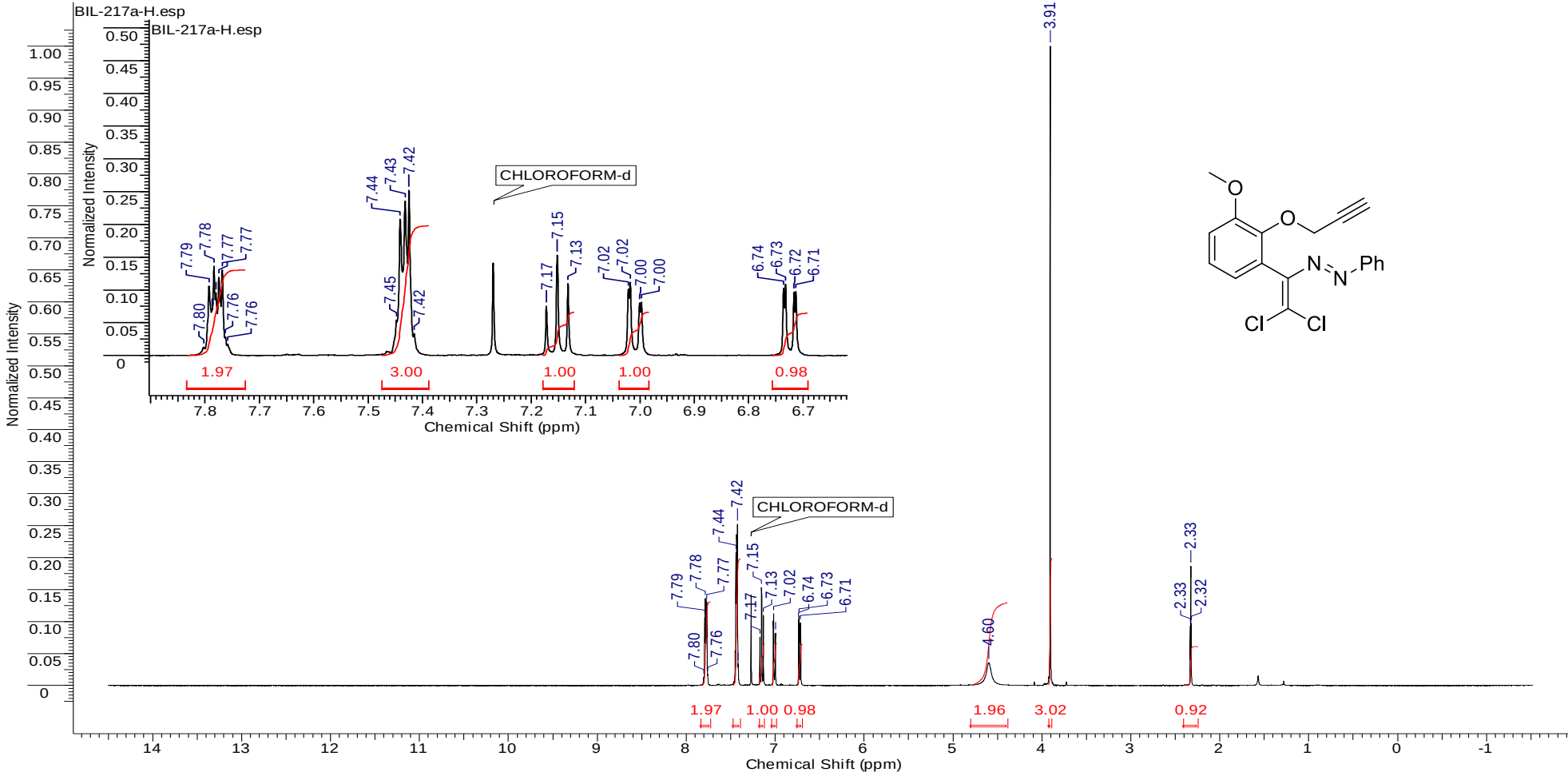
Owner	root	***Points Count***	131072	***Pulse Sequence***	zgpg30	***Receiver Gain***	13004.00
SW(cyclical) (Hz)	24154.59	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11059.6152	***Sweep Width (Hz)***	24154.41
Temperature (degree C)	27.000						



^{13}C NMR spectrum of **1** (100.6 MHz, $CDCl_3$)

22.04.2019 22:32:15

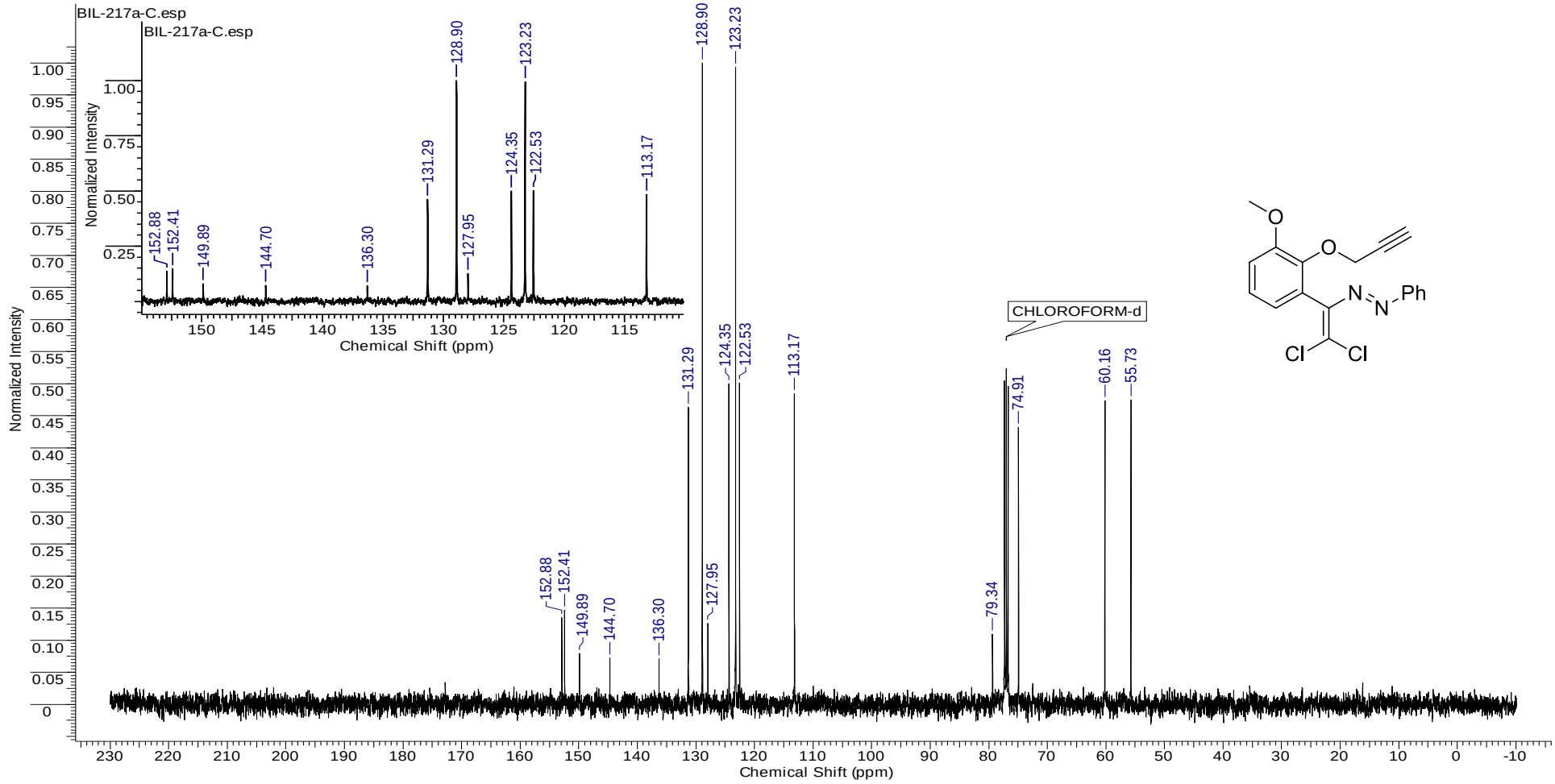
Acquisition Time (sec)	2.5559	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	08 Jun 2018 12:18:08
Date Stamp	08 Jun 2018 12:18:08			***File Name***		***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	4	***Origin***	spect	***Original Points Count***	16384	***Owner***	root
Points Count	65536	***Pulse Sequence***	zg30	***Receiver Gain***	161.30	***SW(cyclical) (Hz)***	6410.26	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2595.7979	***Sweep Width (Hz)***	6410.16	***Temperature (degree C)***	27.000				



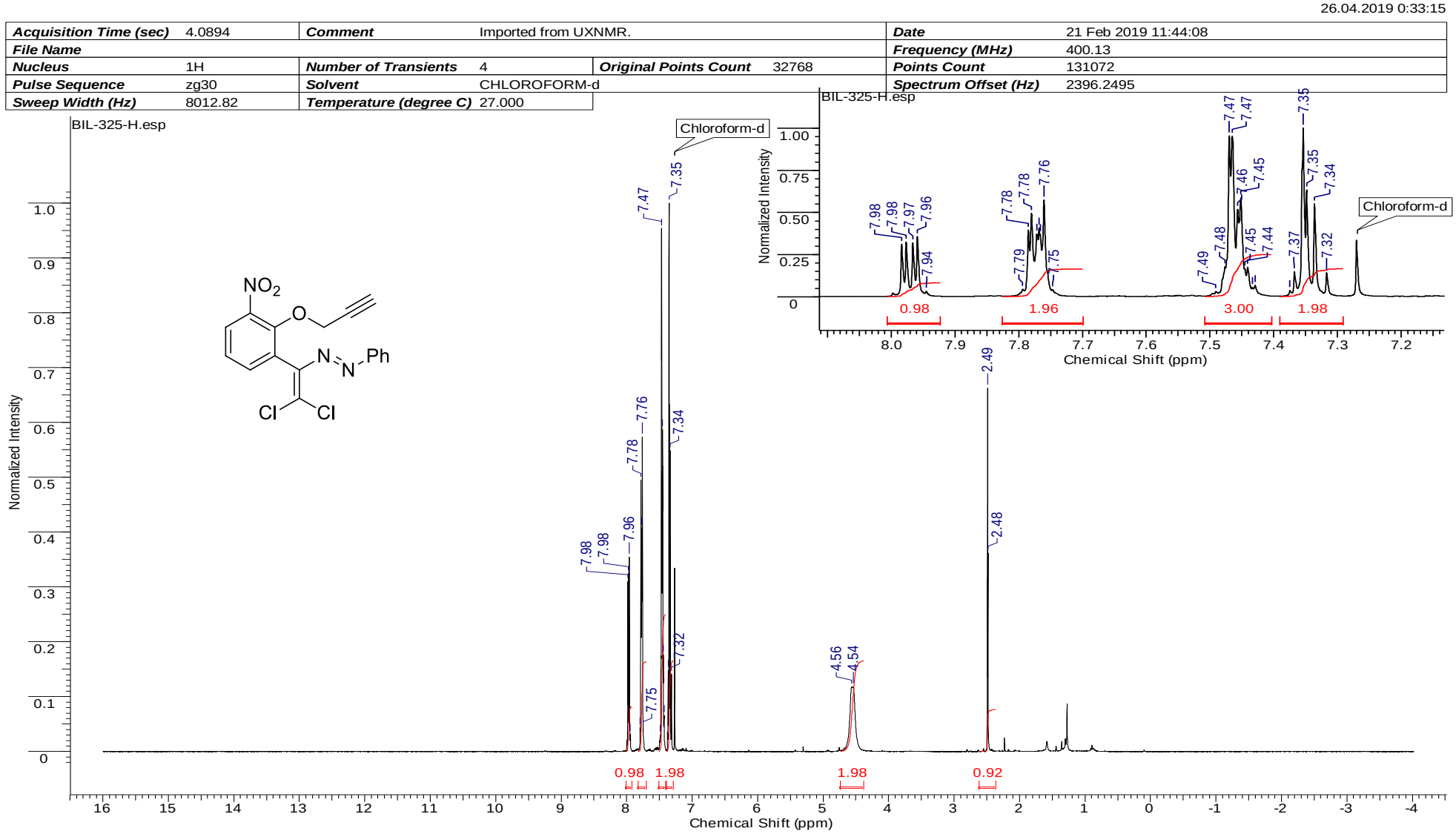
^{1}H NMR spectrum of **2** (400.1 MHz, $CDCl_3$)

22.04.2019 22:33:14

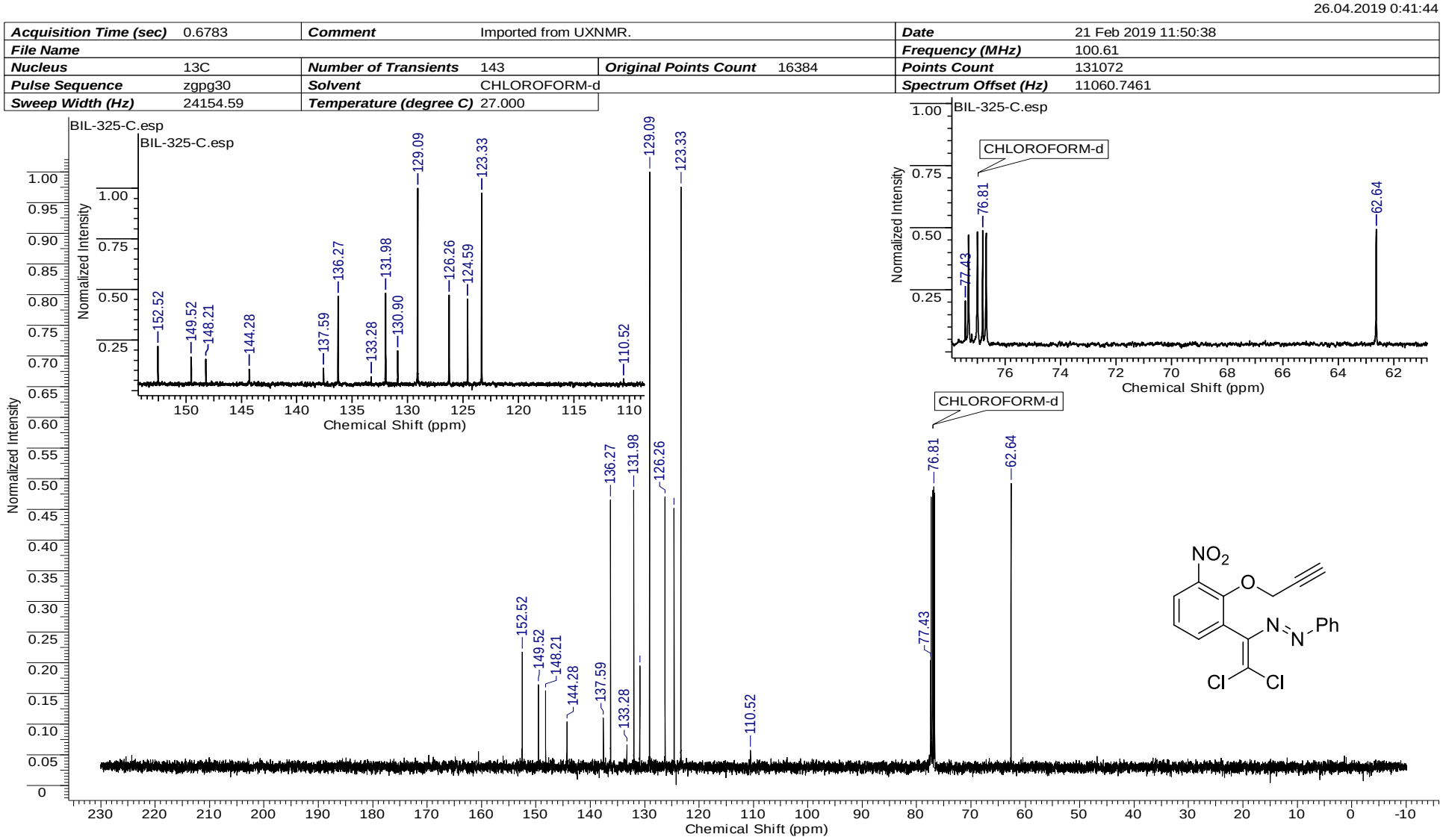
Acquisition Time (sec)	0.4999	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	07 Jun 2018 09:46:40		
Date Stamp	07 Jun 2018 09:46:40			***File Name***				***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	262	***Origin***	spect	***Original Points Count***	12076	***Owner***	root
Points Count	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	13004.00	***SW(cyclical) (Hz)***	24154.59		
Solvent	CHLOROFORM-d			***Spectrum Offset (Hz)***	11061.1816	***Sweep Width (Hz)***	24154.22	***Temperature (degree C)***	27.000



^{13}C NMR spectrum of **2** (100.6 MHz, $CDCl_3$)



^{1}H NMR spectrum of **3** (400.1 MHz, $CDCl_3$)



^{13}C NMR spectrum of **3** (100.6 MHz, $CDCl_3$)

Acquisition Time (sec)	4.0894	***Comment***	5 mm Dual 13C/1H Z3756/0200			***Date***	19 Feb 2019 11:46:08
Date Stamp	19 Feb 2019 11:46:08						
File Name						***Frequency (MHz)***	400.13
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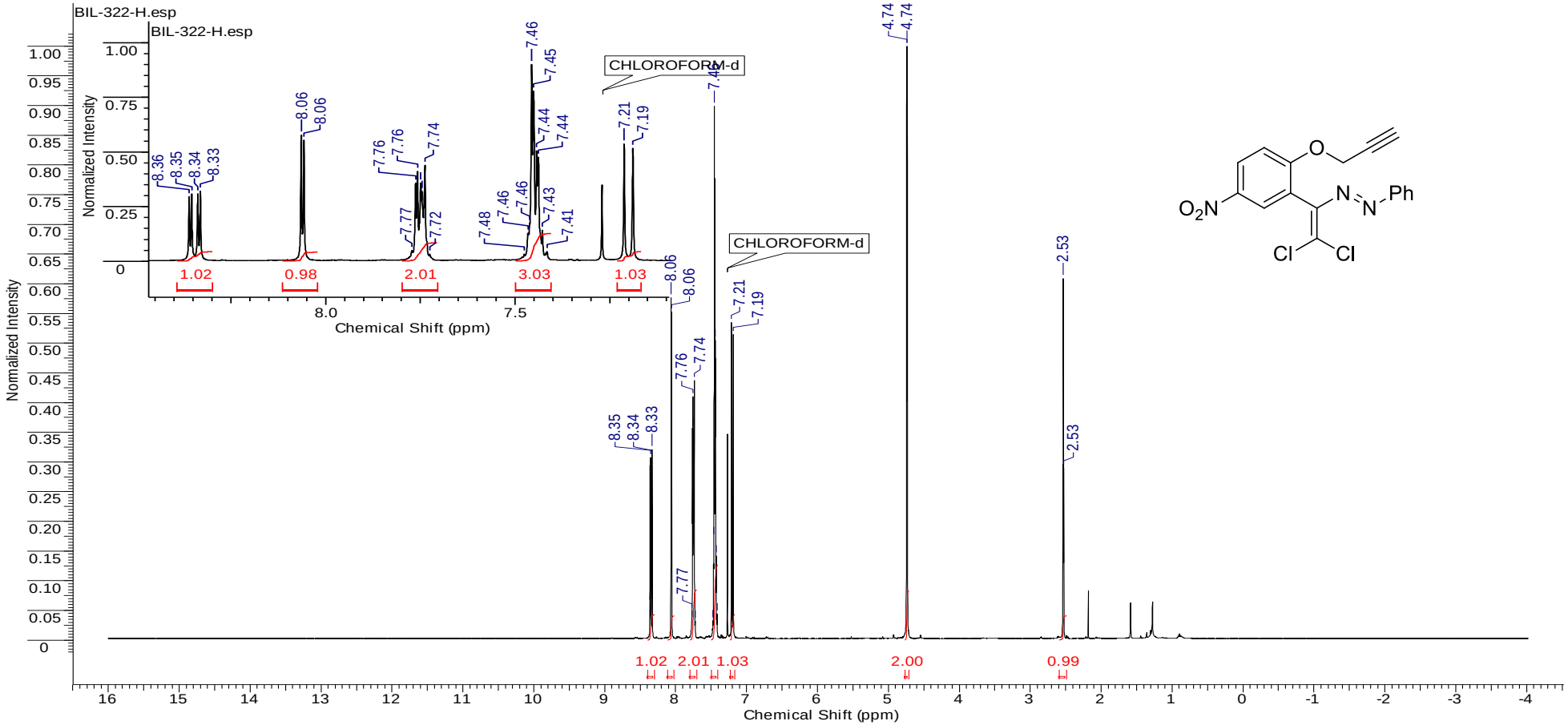
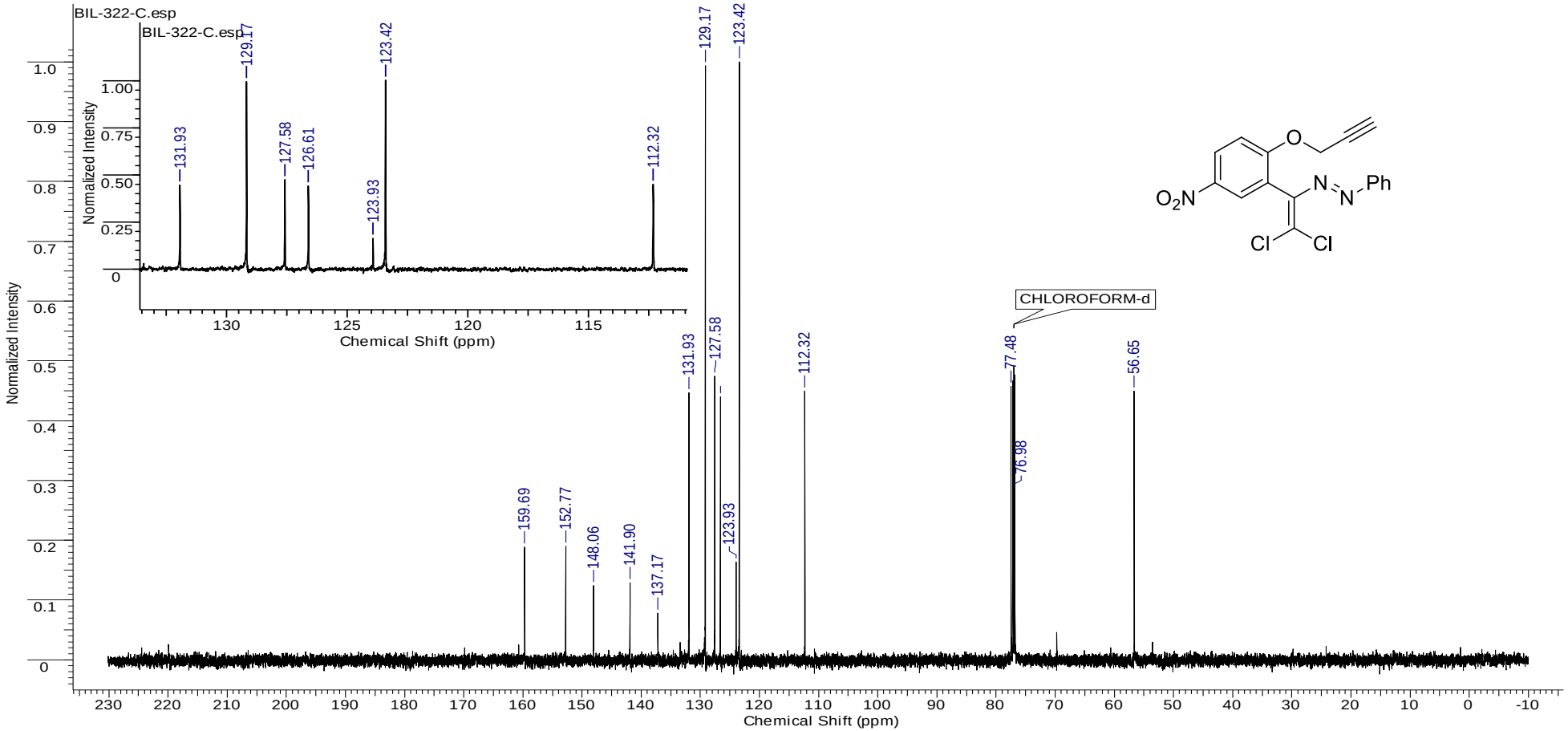
Owner	root	***Points Count***	131072	***Pulse Sequence***	zg30	***Receiver Gain***	114.00
SW(cyclical) (Hz)	8012.82	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2396.6816
Sweep Width (Hz)	8012.76	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of 4 (400.1 MHz, $CDCl_3$)

23.04.2019 0:20:59

Acquisition Time (sec)	0.6783	***Comment***	5 mm Dual 13C/1H Z3756/0200			***Date***	19 Feb 2019 11:46:08
Date Stamp	19 Feb 2019 11:46:08						
File Name						***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	97	***Origin***	spect	***Original Points Count***	16384
Owner	root	***Points Count***	131072	***Pulse Sequence***	zgpg30	***Receiver Gain***	14596.50
SW(cyclical) (Hz)	24154.59	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11077.1221	***Sweep Width (Hz)***	24154.41
Temperature (degree C)	27.000						



^{13}C NMR spectrum of **4** (100.6 MHz, $CDCl_3$)

22.04.2019 23:41:47

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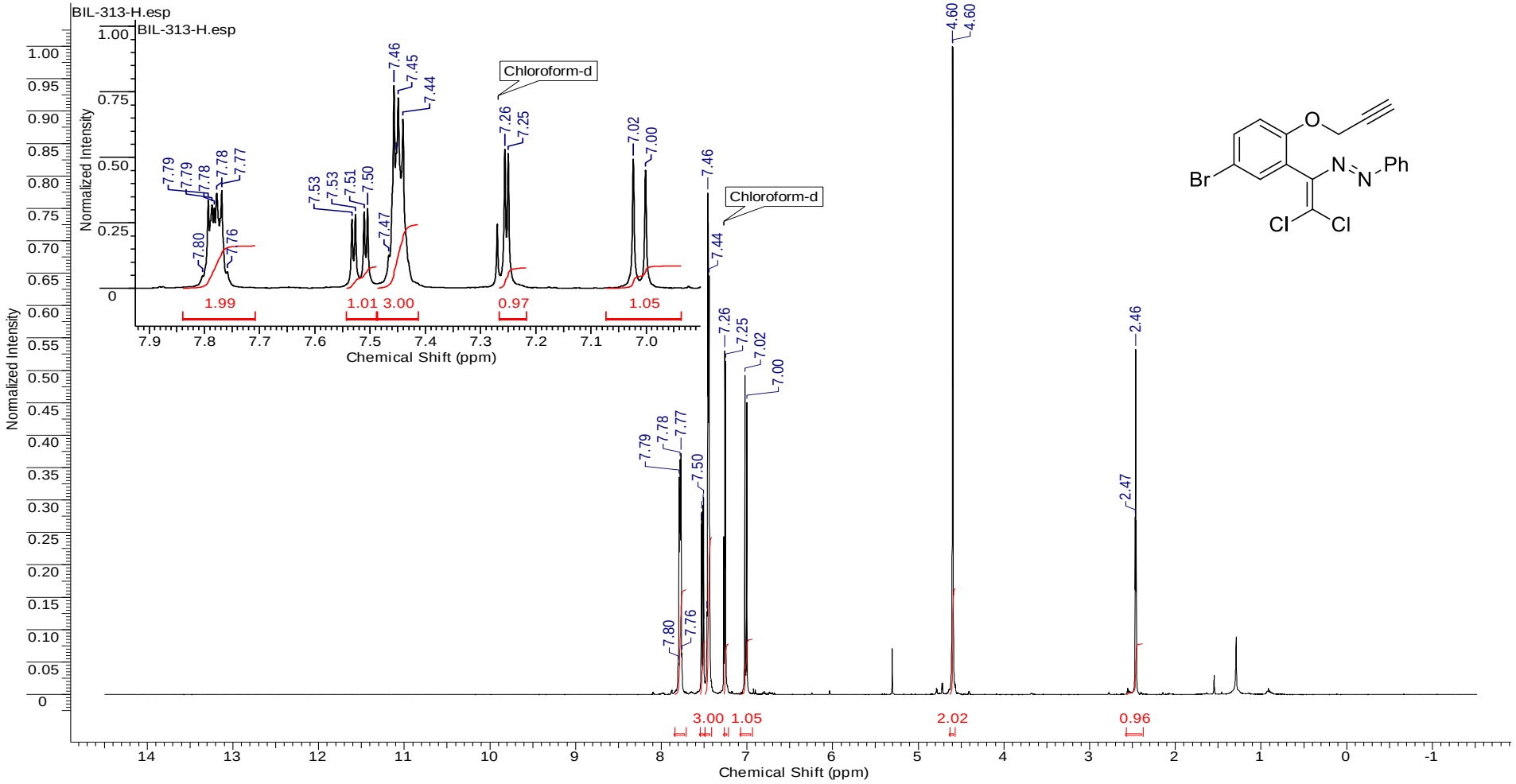
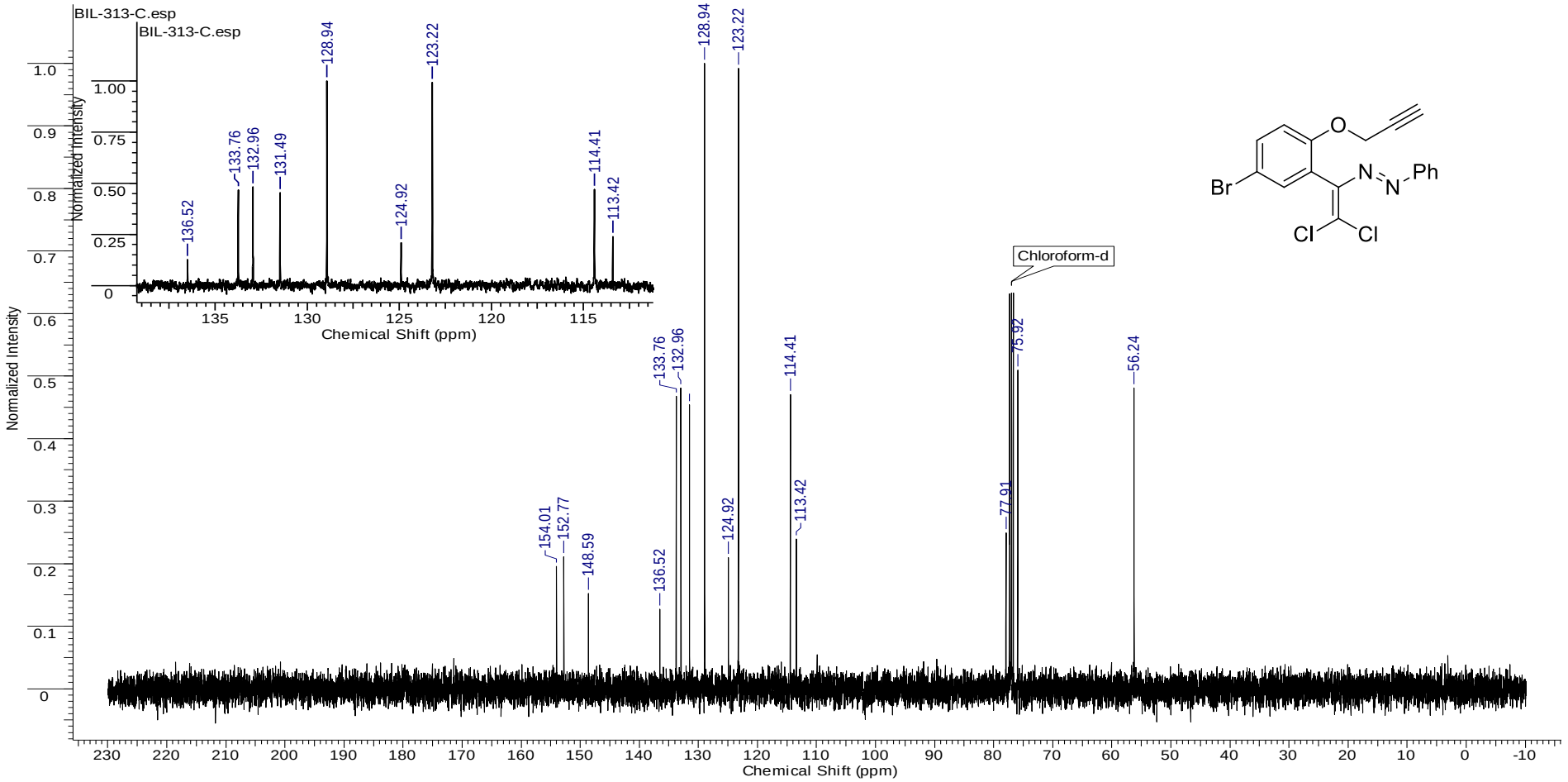
File Name					***Frequency (MHz)***	400.13	***Nucleus*** 1H
Number of Transients	4	***Original Points Count***	16384	***Points Count*** 65536	***Pulse Sequence***	zg30	***Solvent*** DMSO-d6
Spectrum Offset (Hz)	2595.9883	***Sweep Width (Hz)***	6410.26	***Temperature (degree C)*** 27.000			



^{1}H NMR spectrum of 5 (400.1 MHz, $CDCl_3$)

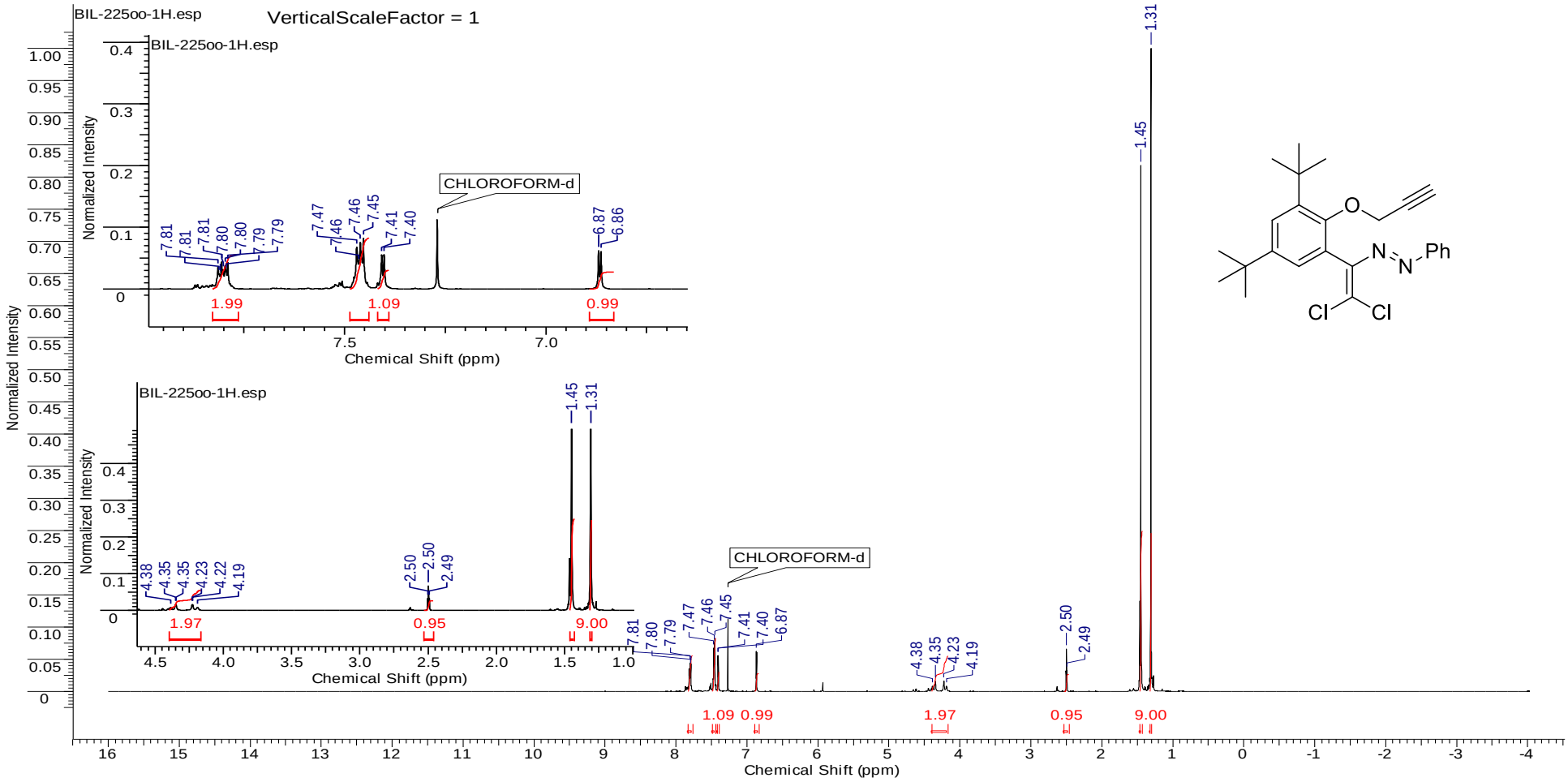
22.04.2019 23:42:33

Acquisition Time (sec)	0.6783	***Comment***	Imported from UXNMR.			***Date***	28 Jan 2019 15:40:56
File Name						***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	23	***Original Points Count***	16384	***Points Count***	131072
Pulse Sequence	zgpg30	***Solvent***	CHLOROFORM-D	***Spectrum Offset (Hz)***	11058.7188	***Sweep Width (Hz)***	24154.59
Temperature (degree C)	27.000						



^{13}C NMR spectrum of **5** (100.6 MHz, $CDCl_3$)

Acquisition Time (sec)	4.0894	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	30 Apr 2019 12:39:28		
Date Stamp	30 Apr 2019 12:39:28			***File Name***					
Frequency (MHz)	400.13	***Nucleus***	1H	***Number of Transients***	4	***Origin***	spect	***Original Points Count***	32768
Owner	root	***Points Count***	131072	***Pulse Sequence***	zg30	***Receiver Gain***	128.00	***SW(cyclical) (Hz)***	8012.82
Solvent	CHLOROFORM-d			***Spectrum Offset (Hz)***	2395.7644	***Sweep Width (Hz)***	8012.76	***Temperature (degree C)***	27.000



^{1}H NMR spectrum of **6** (400.1 MHz, $CDCl_3$)

24.06.2019 20:26:57

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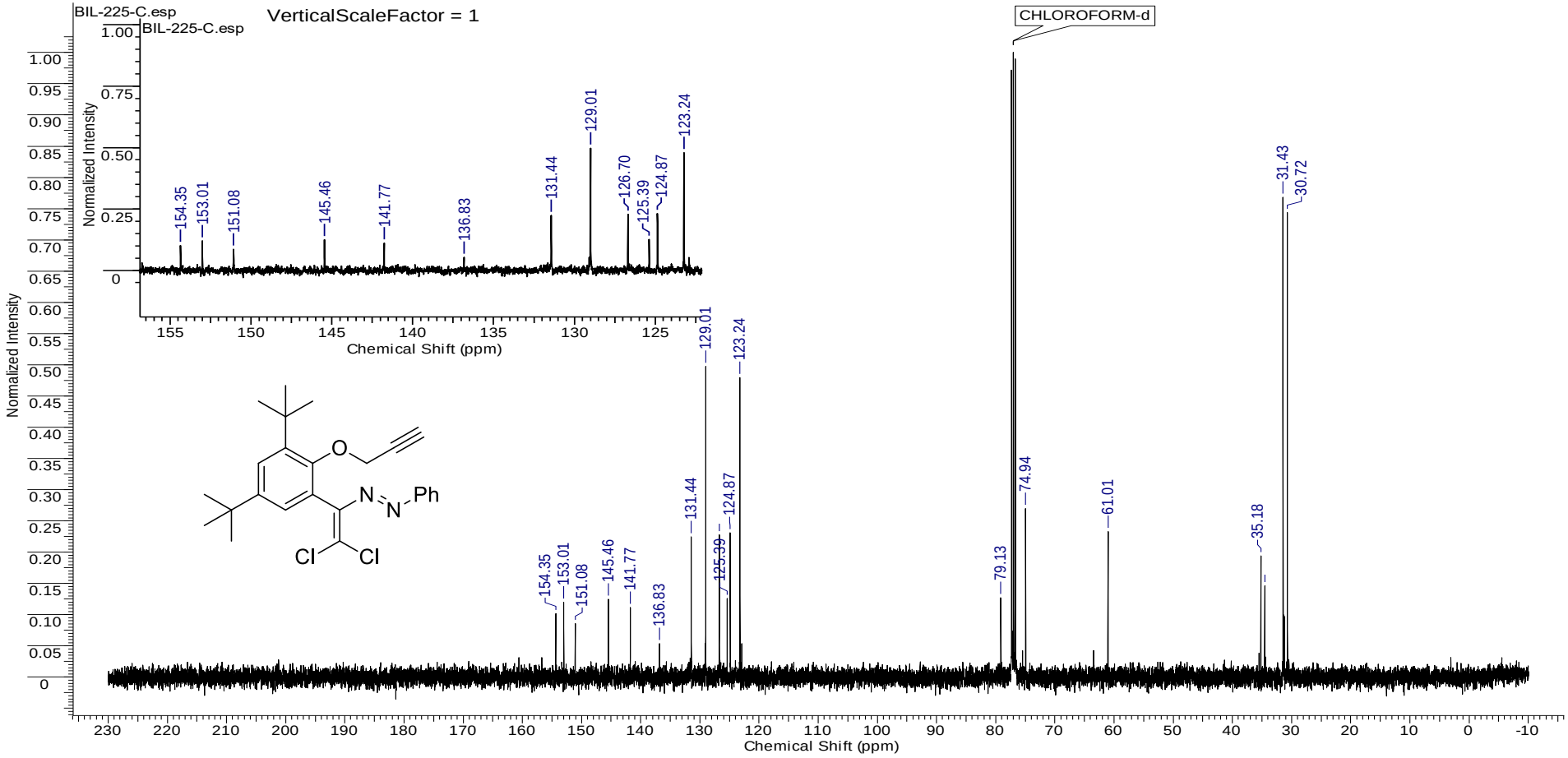
Frequency (MHz)	100.61	***Nucleus***	13C	***Number of Transients***	145	***Origin***	spect
Original Points Count	16384	***Owner***	root	***Points Count***	131072	***Pulse Sequence***	zgpg30
Receiver Gain	13004.00	***SW(cyclical) (Hz)***	24154.59	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	11063.6699	***Sweep Width (Hz)***	24154.41	***Temperature (degree C)***	27.000		



^{13}C NMR spectrum of **6** (100.6 MHz, $CDCl_3$)

20.11.2019 11:04:41

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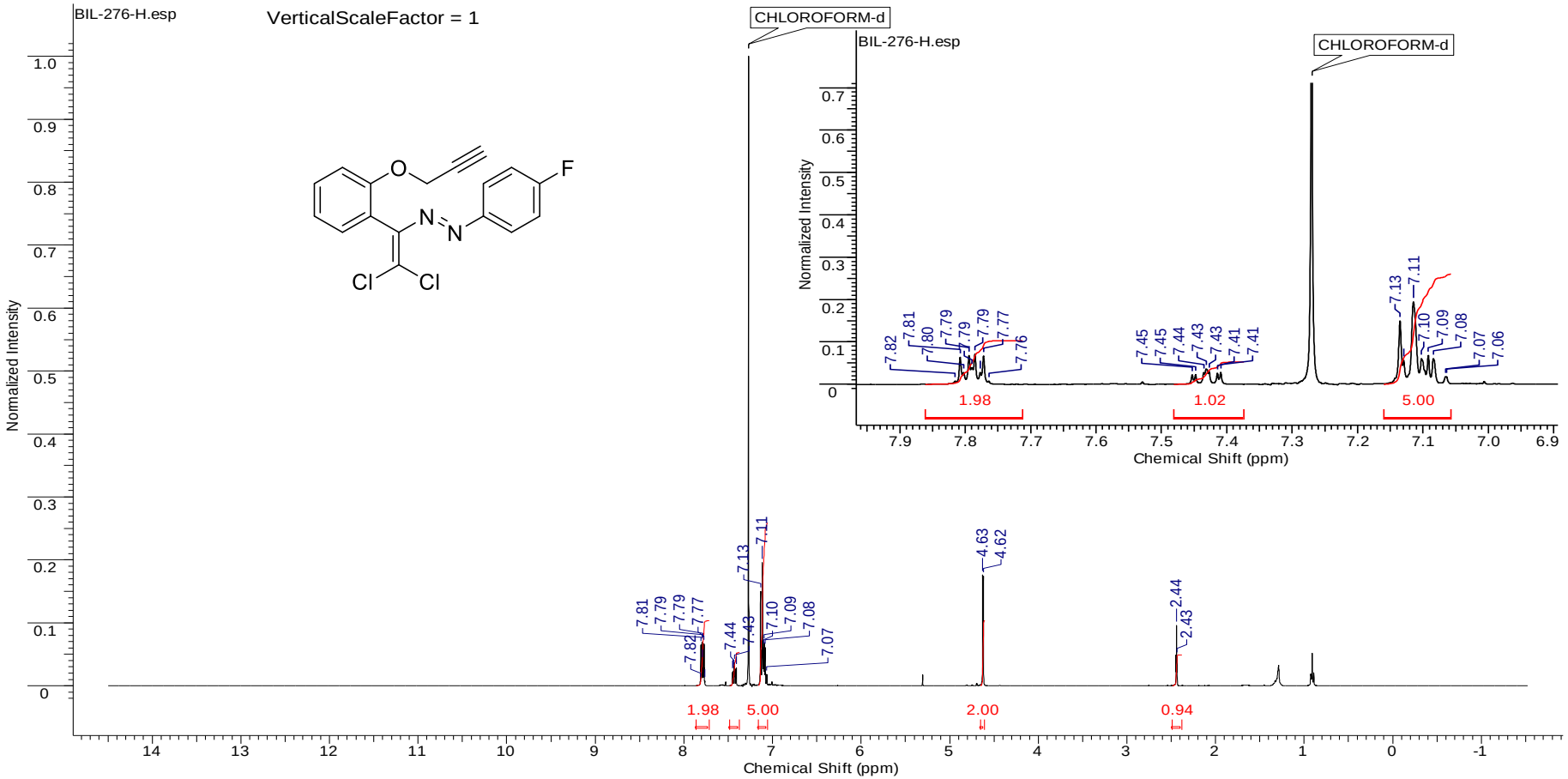
Date Stamp	26 Oct 2018 12:39:28						
File Name						***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	5	***Origin***	spect	***Original Points Count***	16384
Owner	root	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	114.00
SW(cyclical) (Hz)	6410.26	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2595.7979
Sweep Width (Hz)	6410.16	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **7** (400.1 MHz, $CDCl_3$)

12.05.2019 15:55:13

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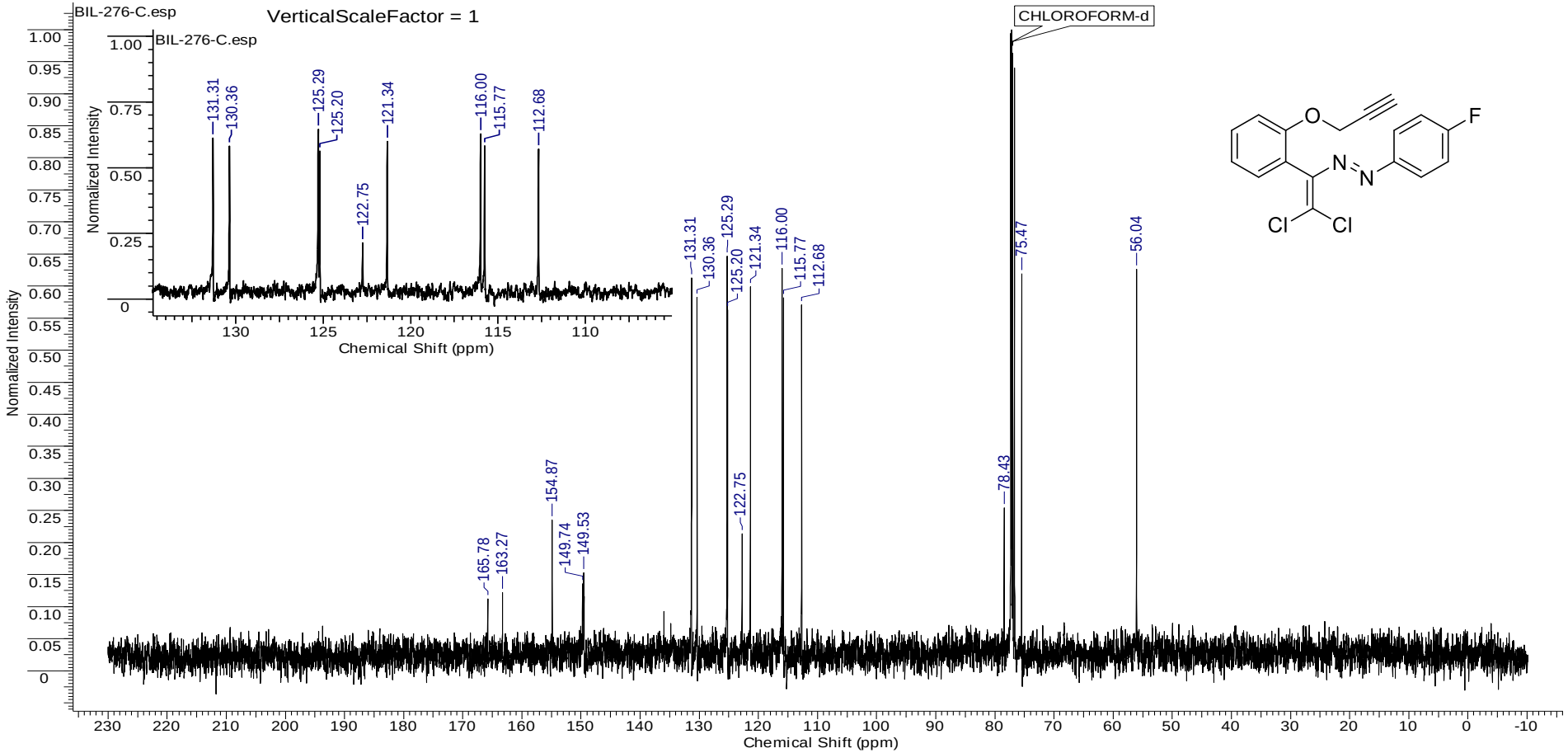
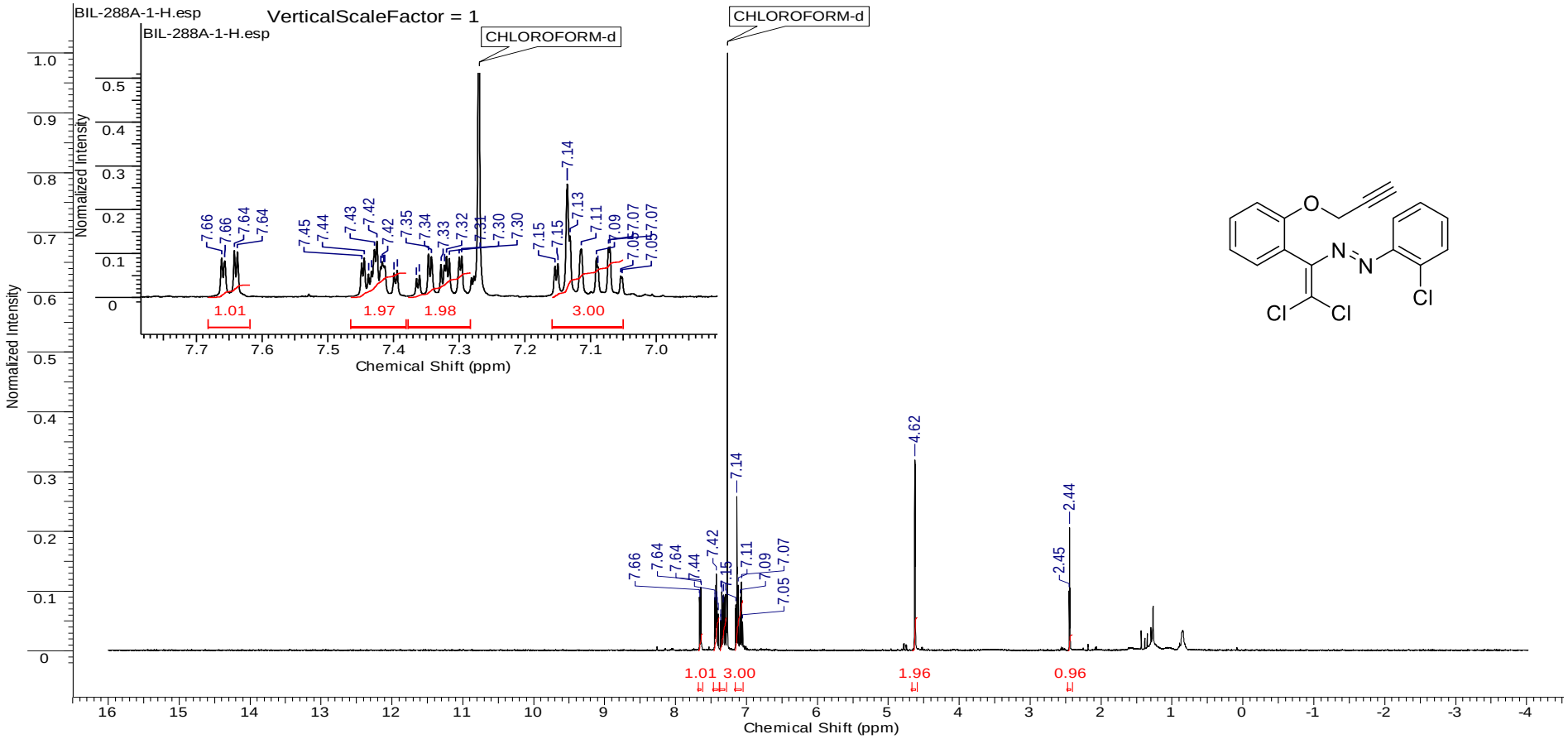
Date Stamp	26 Oct 2018 12:41:36						
File Name						***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	116	***Origin***	spect	***Original Points Count***	12076
Owner	root	***Points Count***	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	8192.00
SW(cyclical) (Hz)	24154.59	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11061.9189	***Sweep Width (Hz)***	24154.22
Temperature (degree C)	27.000						



^{13}C NMR spectrum of **7** (100.6 MHz, $CDCl_3$)

14.05.2019 18:37:09

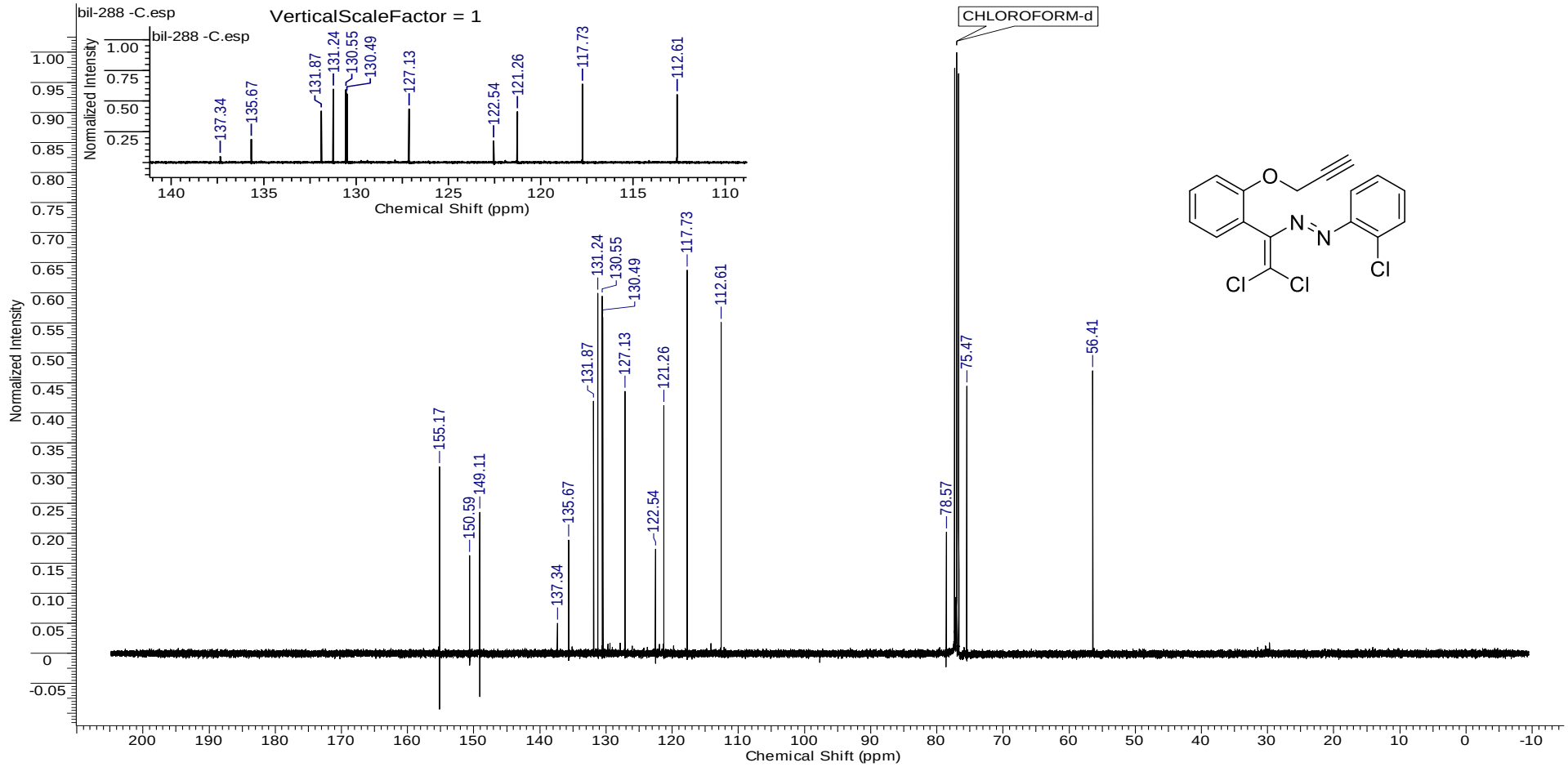
Acquisition Time (sec)	4.0894	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	07 Feb 2019 08:14:56
Date Stamp	07 Feb 2019 08:14:56						
File Name						***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	4	***Origin***	spect	***Original Points Count***	32768
Owner	root	***Points Count***	131072	***Pulse Sequence***	zg30	***Receiver Gain***	362.00
SW(cyclical) (Hz)	8012.82	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2395.7031
Sweep Width (Hz)	8012.76	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **8** (400.1 MHz, $CDCl_3$)

14.05.2019 18:35:53

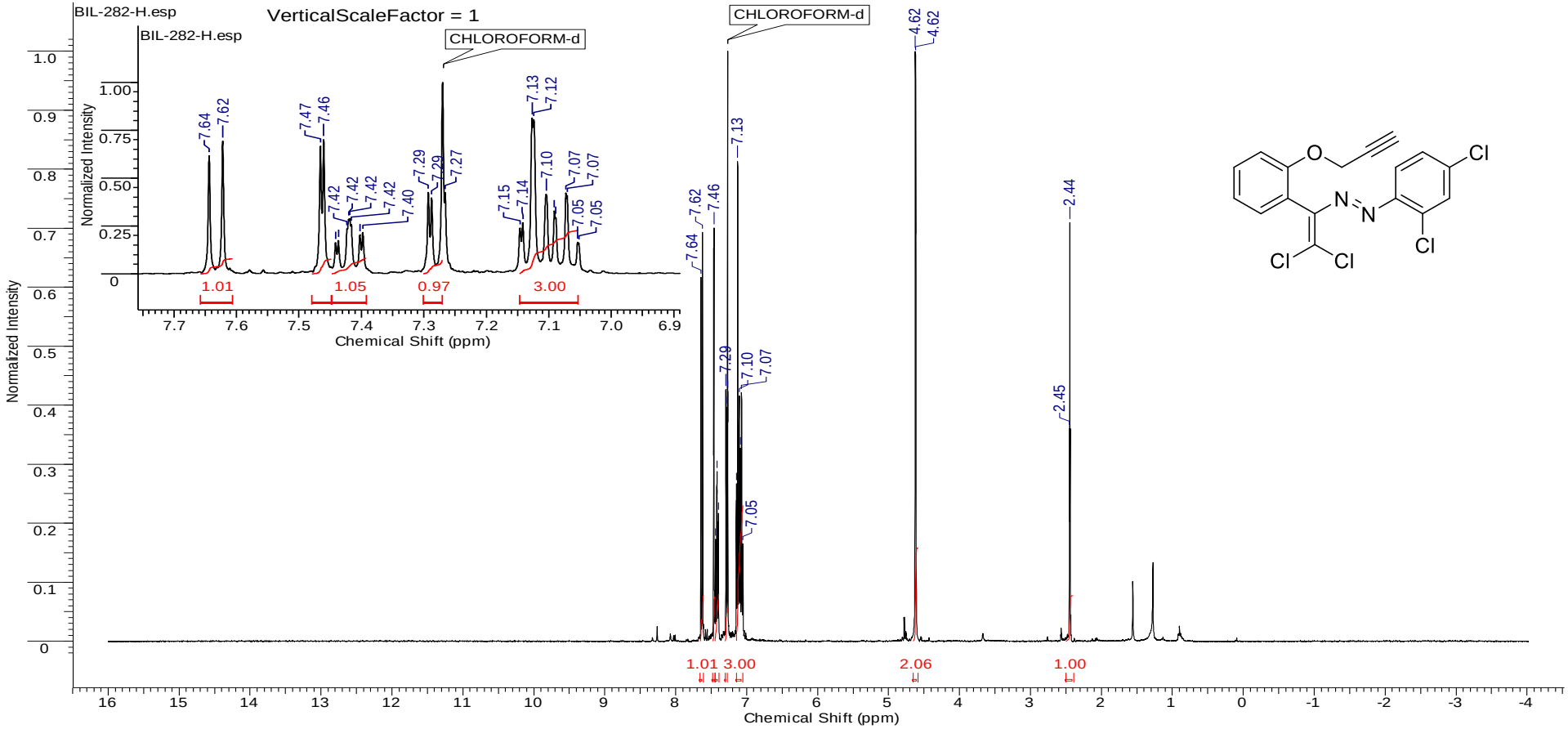
Acquisition Time (sec)	2.0000	***Date***	Feb 11 2019	***Date Stamp***	Feb 11 2019		
File Name							
Frequency (MHz)	100.58	***Nucleus***	13C	***Number of Transients***	18000	***Original Points Count***	43103
Points Count	65536	***Pulse Sequence***	s2pul	***Receiver Gain***	54.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	9822.5713	***Sweep Width (Hz)***	21551.72	***Temperature (degree C)***	24.000		



^{13}C NMR spectrum of **8** (100.6 MHz, $CDCl_3$)

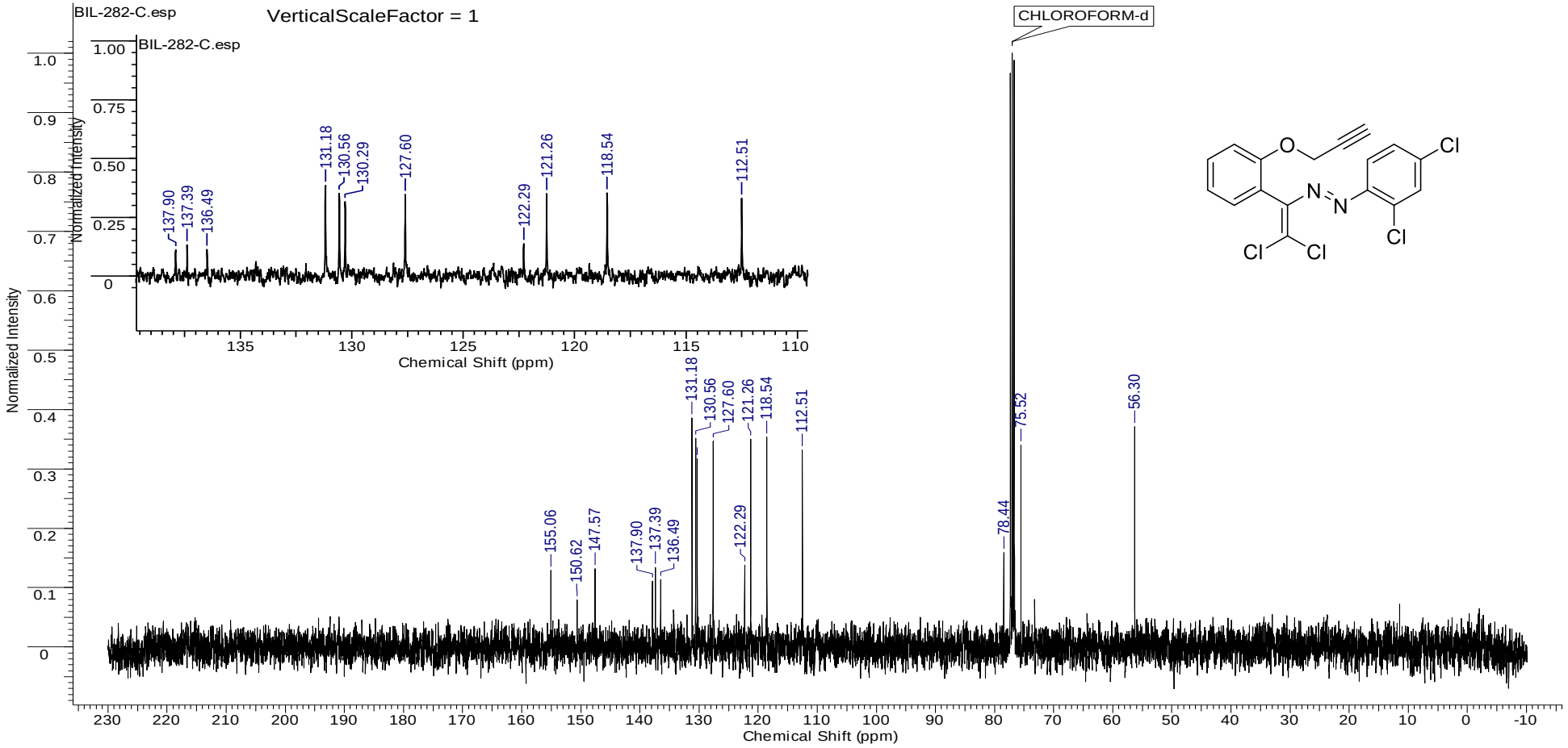
14.05.2019 12:27:41

Acquisition Time (sec)	4.0894	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	31 Oct 2018 14:30:24
Date Stamp	31 Oct 2018 14:30:24						
File Name						***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	5	***Origin***	spect	***Original Points Count***	32768
Owner	root	***Points Count***	131072	***Pulse Sequence***	zg30	***Receiver Gain***	228.10
SW(cyclical) (Hz)	8012.82	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2395.6421
Sweep Width (Hz)	8012.76	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **9** (400.1 MHz, $CDCl_3$)

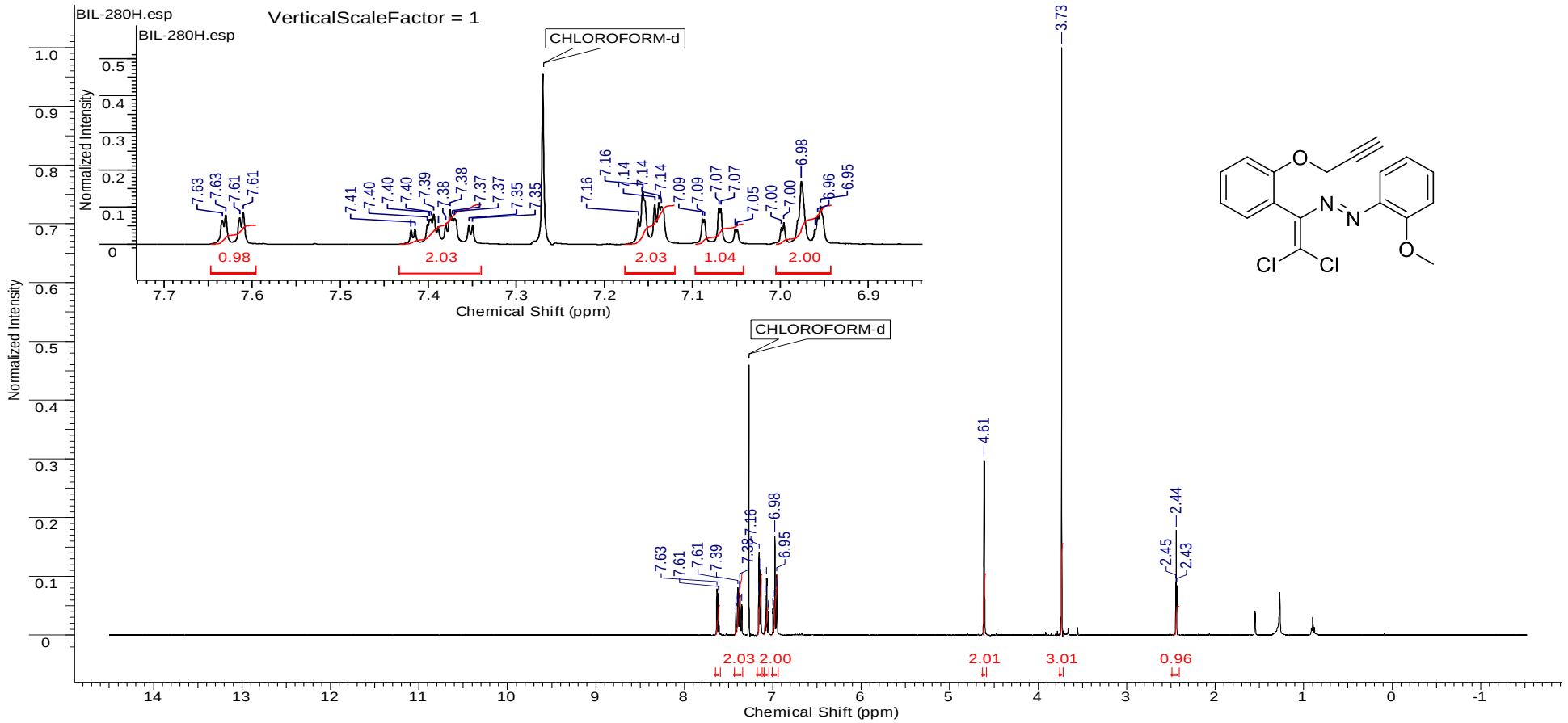
Acquisition Time (sec)	0.4999	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	31 Oct 2018 14:32:32
Date Stamp	31 Oct 2018 14:32:32						
File Name						***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	92	***Origin***	spect	***Original Points Count***	12076
Owner	root	***Points Count***	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	8192.00
SW(cyclical) (Hz)	24154.59	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11062.6563	***Sweep Width (Hz)***	24154.22
Temperature (degree C)	27.000						



^{13}C NMR spectrum of **9** (100.6 MHz, $CDCl_3$)

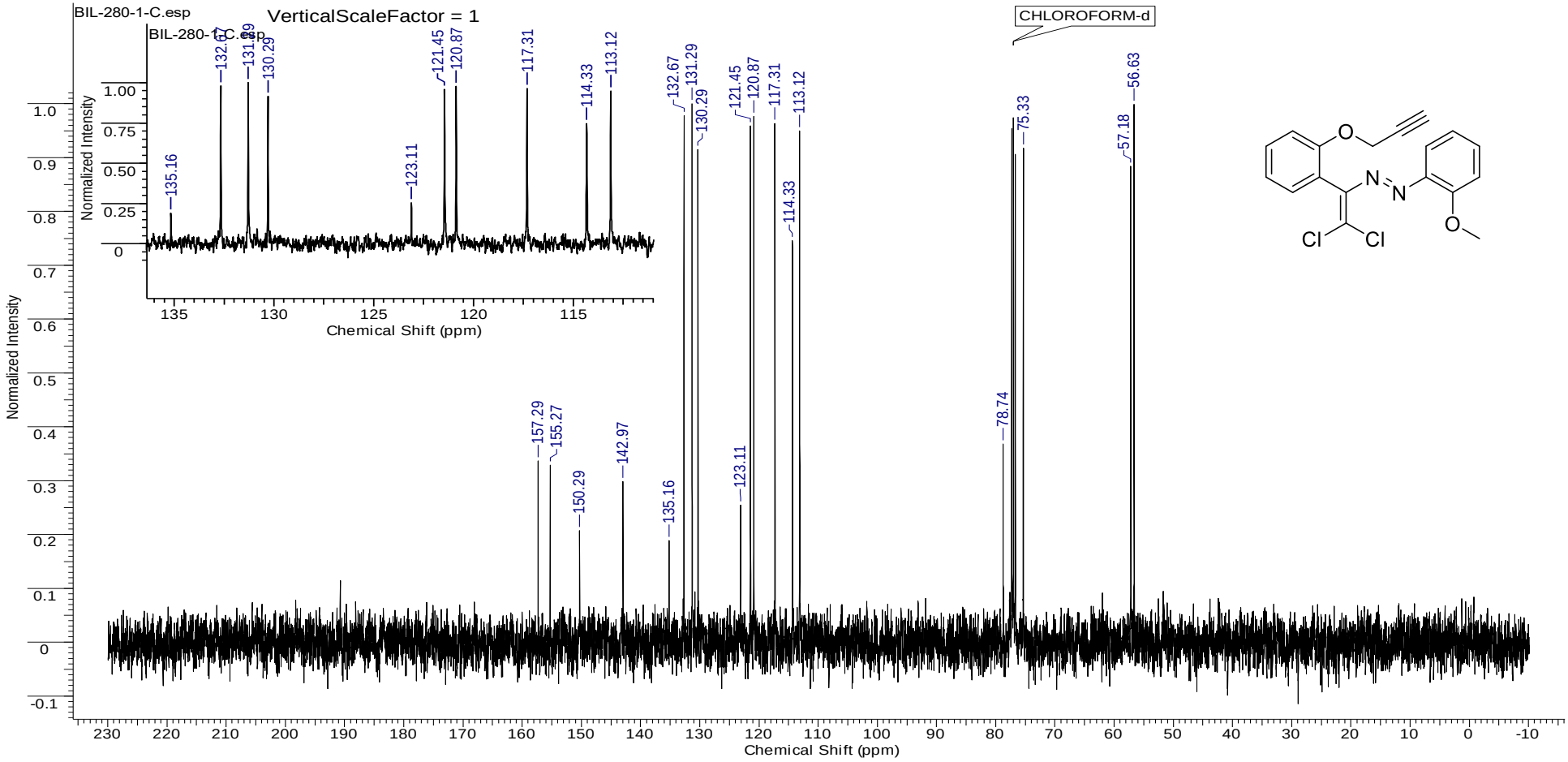
14.05.2019 18:28:59

Acquisition Time (sec)	2.5559	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	31 Oct 2018 14:36:48
Date Stamp	31 Oct 2018 14:36:48						
File Name						***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	5	***Origin***	spect	***Original Points Count***	16384
Owner	root	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	362.00
SW(cyclical) (Hz)	6410.26	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2595.7000
Sweep Width (Hz)	6410.16	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **10** (400.1 MHz, $CDCl_3$)

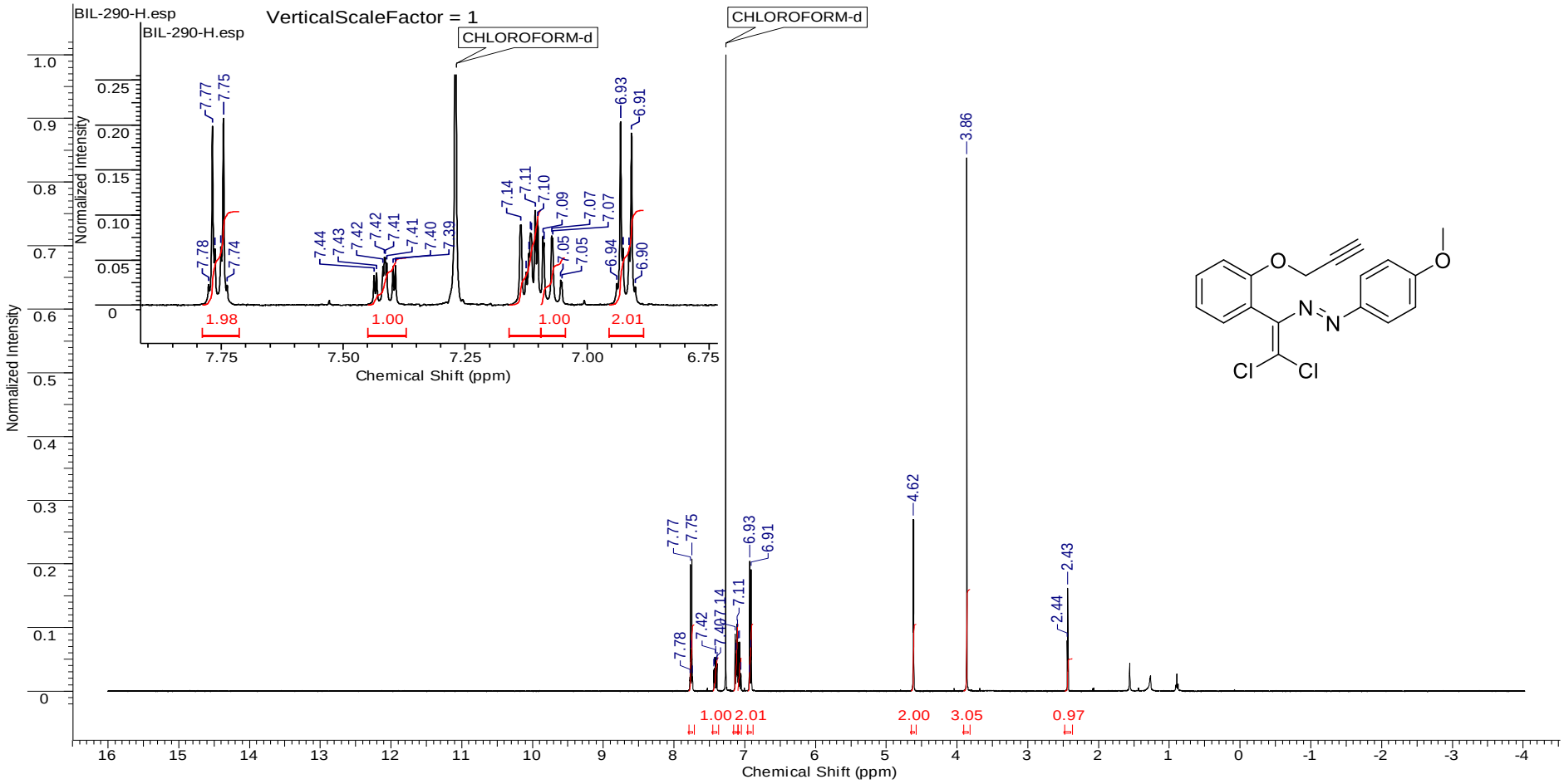
Acquisition Time (sec)	0.4999	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	14 Nov 2018 14:17:36
Date Stamp	14 Nov 2018 14:17:36			***File Name***			
Frequency (MHz)	100.61	***Nucleus***	13C	***Number of Transients***	129	***Origin***	spect
Original Points Count	12076	***Owner***	root	***Points Count***	65536	***Pulse Sequence***	zgpg30
Receiver Gain	8192.00	***SW(cyclical) (Hz)***	24154.59	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	11056.0225	***Sweep Width (Hz)***	24154.22	***Temperature (degree C)***	27.000		



^{13}C NMR spectrum of **10** (100.6 MHz, $CDCl_3$)

14.05.2019 19:31:46

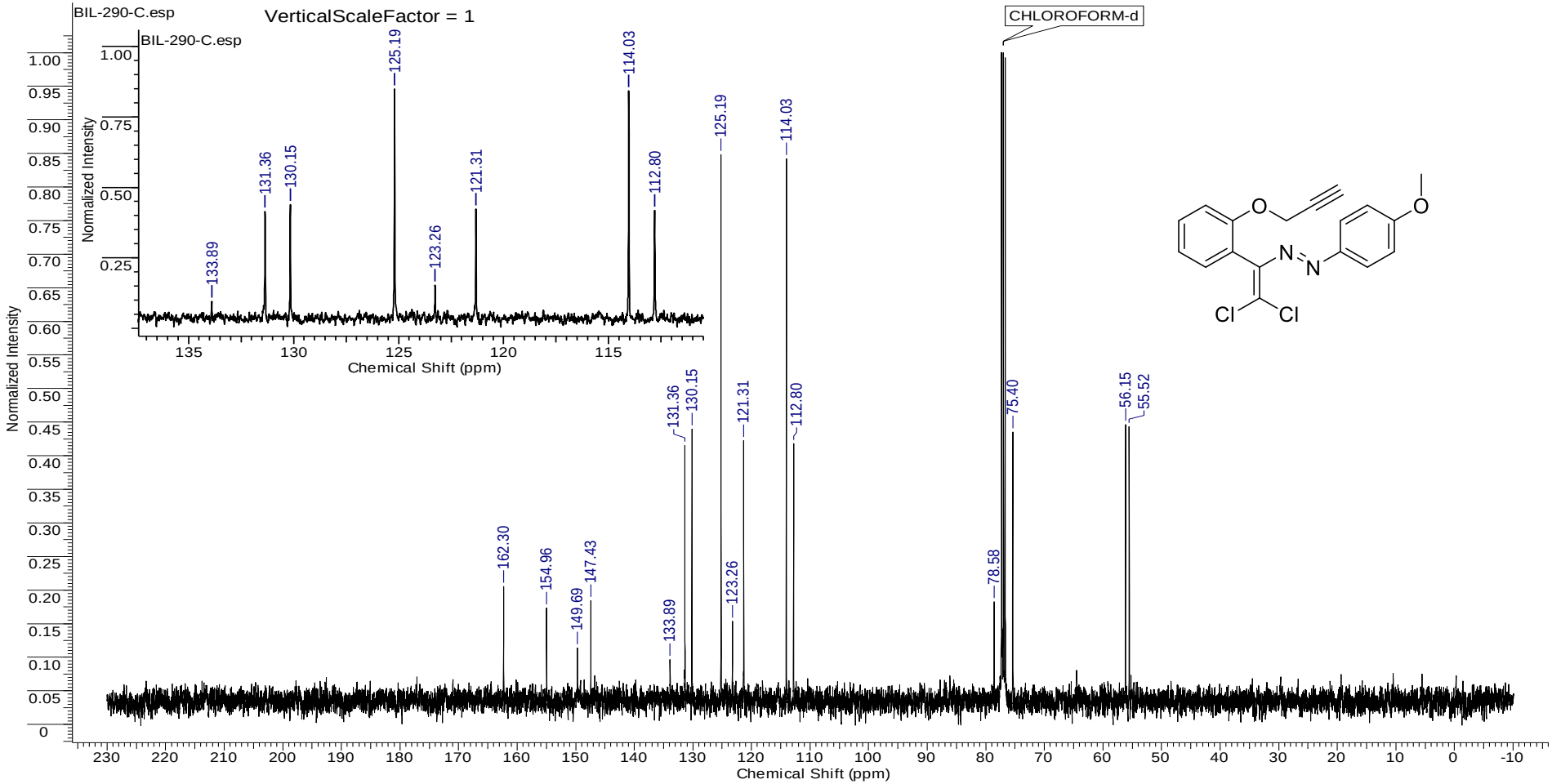
Acquisition Time (sec)	4.0894	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	07 Dec 2018 12:26:40
Date Stamp	07 Dec 2018 12:26:40			***File Name***		***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	4	***Origin***	spect	***Original Points Count***	32768	***Owner***	root
Points Count	131072	***Pulse Sequence***	zg30	***Receiver Gain***	724.10	***SW(cyclical) (Hz)***	8012.82	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2395.7031	***Sweep Width (Hz)***	8012.76	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **11** (400.1 MHz, $CDCl_3$)

14.05.2019 19:33:00

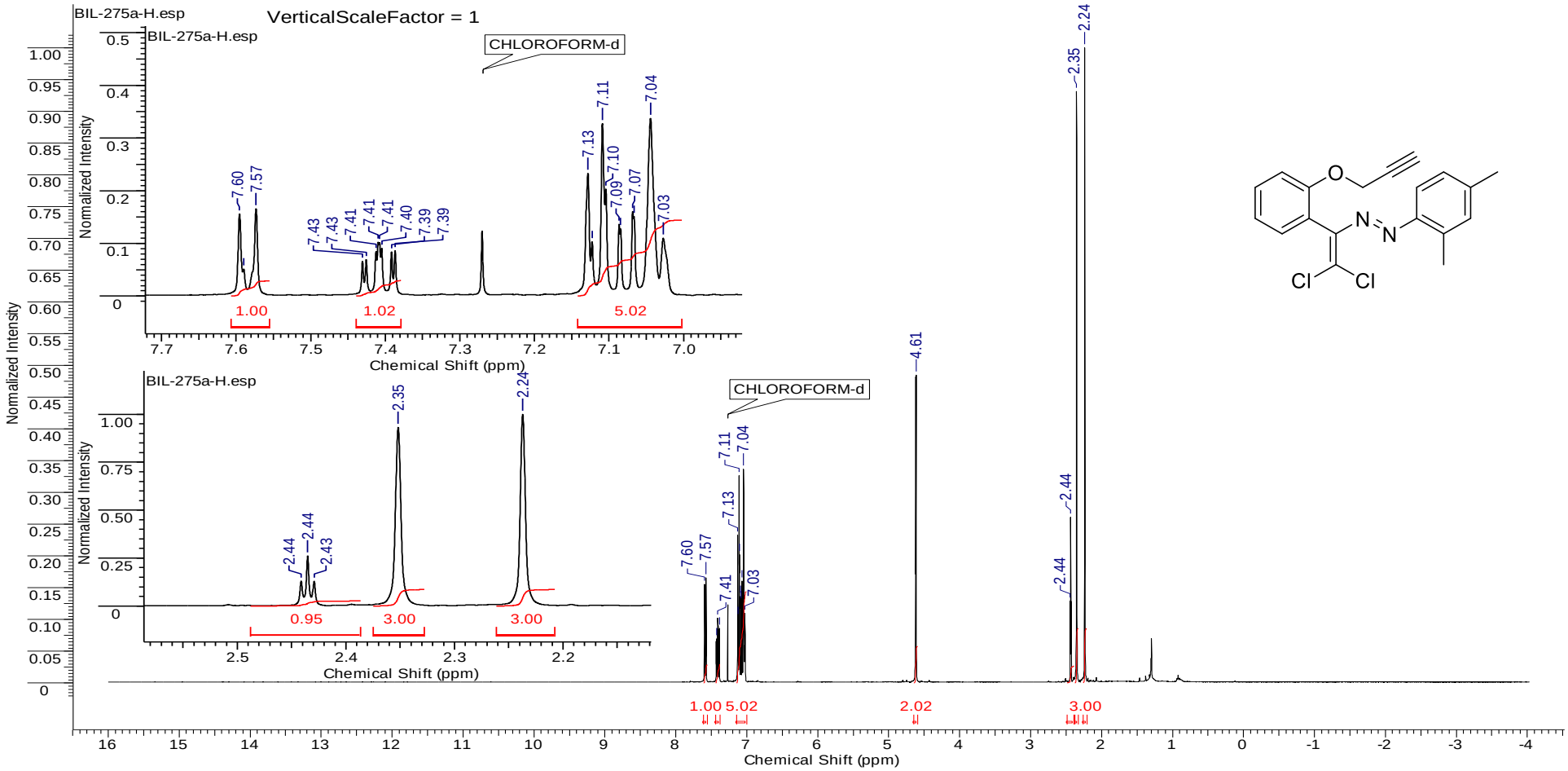
Acquisition Time (sec)	0.4999	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	20 Nov 2018 11:37:36		
Date Stamp	20 Nov 2018 11:37:36			***File Name***				***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	195	***Origin***	spect	***Original Points Count***	12076	***Owner***	root
Points Count	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	8192.00	***SW(cyclical) (Hz)***	24154.59		
Solvent	CHLOROFORM-d			***Spectrum Offset (Hz)***	11062.2871	***Sweep Width (Hz)***	24154.22	***Temperature (degree C)***	27.000



^{13}C NMR spectrum of **11** (100.6 MHz, $CDCl_3$)

15.05.2019 12:13:45

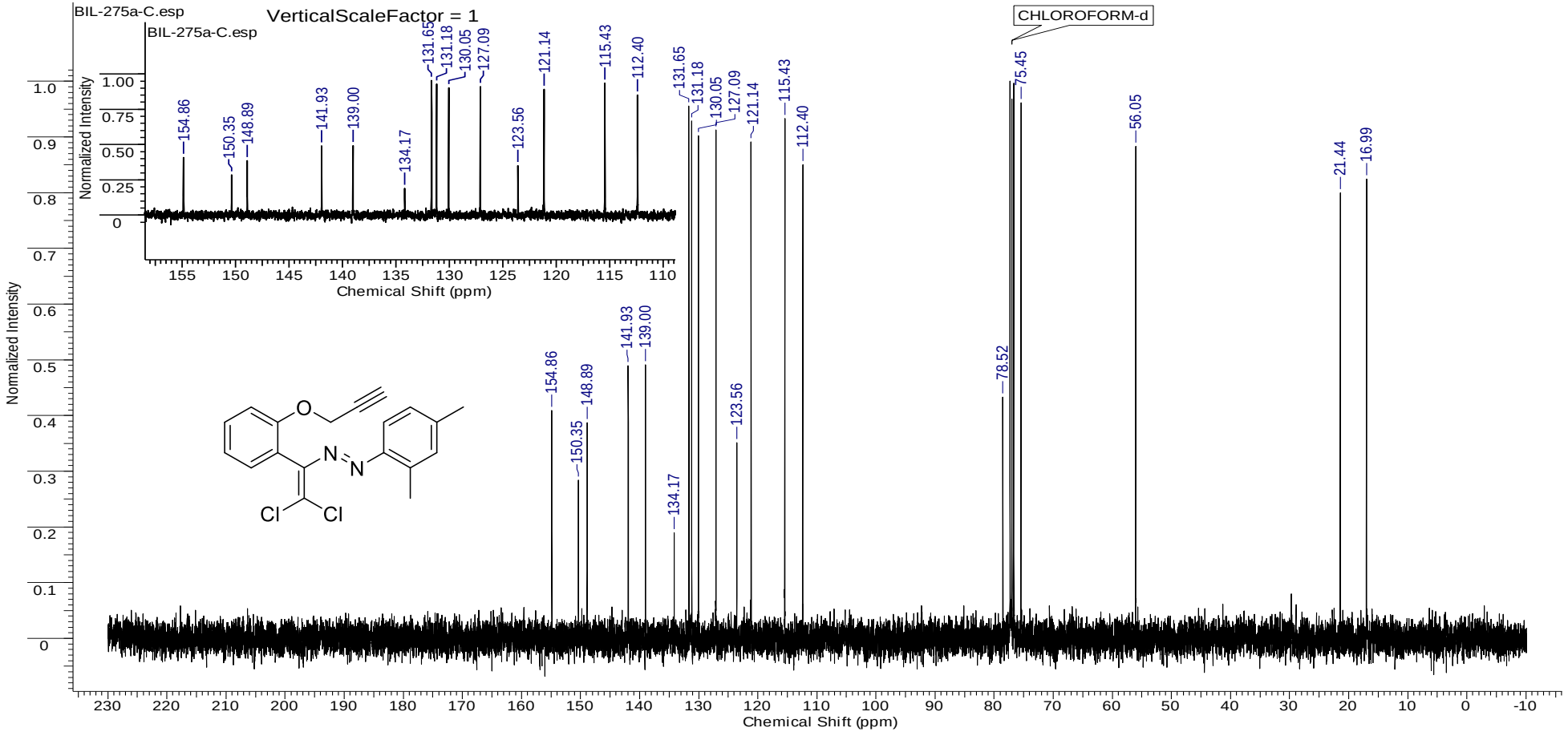
Acquisition Time (sec)	4.0894	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	05 Feb 2019 11:33:20		
Date Stamp	05 Feb 2019 11:33:20			***File Name***				***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	5	***Origin***	spect	***Original Points Count***	32768	***Owner***	root
Points Count	131072	***Pulse Sequence***	zg30	***Receiver Gain***	57.00	***SW(cyclical) (Hz)***	8012.82	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2395.8254	***Sweep Width (Hz)***	8012.76	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **12** (400.1 MHz, $CDCl_3$)

15.05.2019 12:15:05

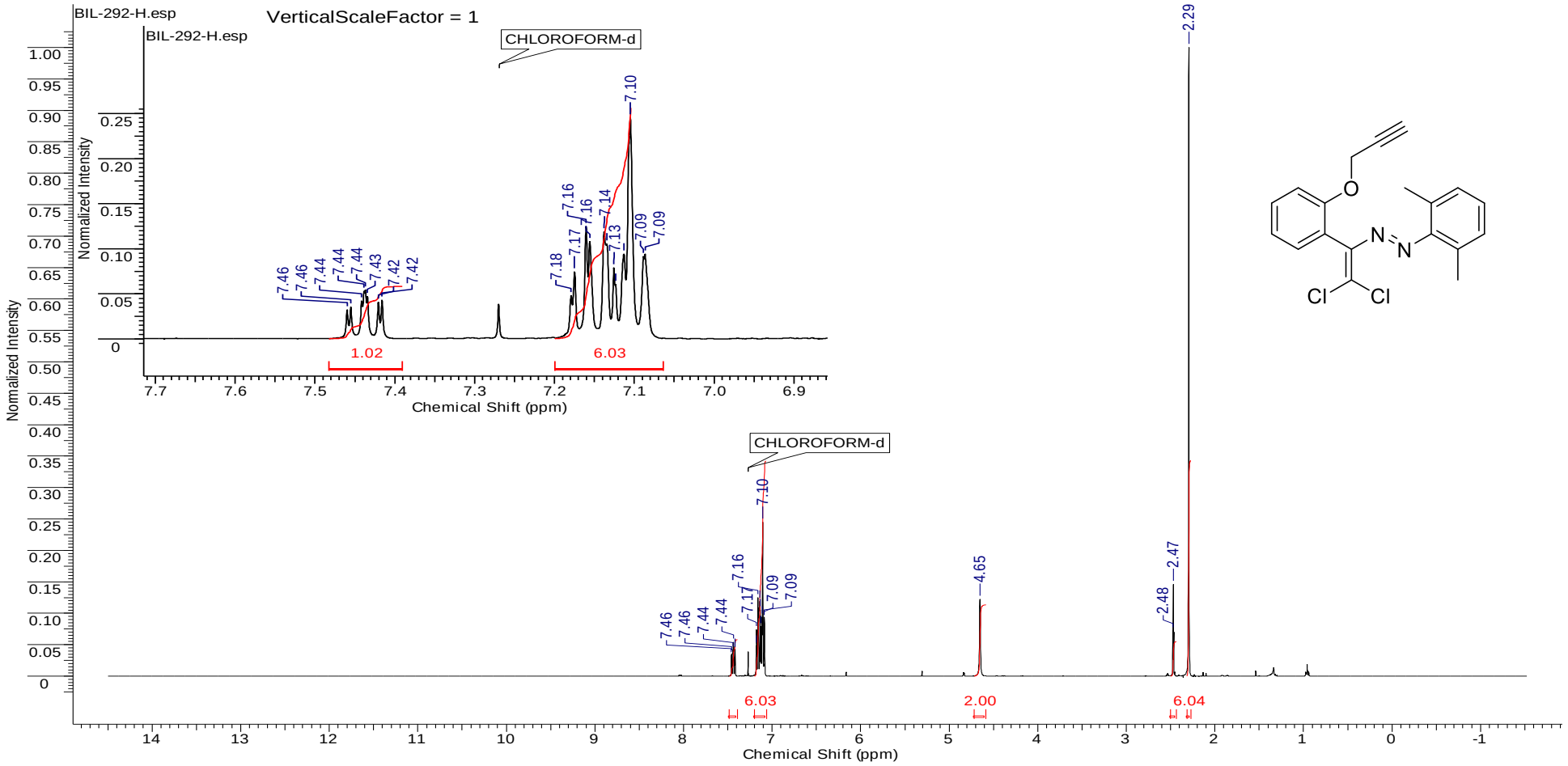
Acquisition Time (sec)	0.6783	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	05 Feb 2019 11:35:28
Date Stamp	05 Feb 2019 11:35:28						
File Name						***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	73	***Origin***	spect	***Original Points Count***	16384
Owner	root	***Points Count***	131072	***Pulse Sequence***	zgpg30	***Receiver Gain***	13004.00
SW(cyclical) (Hz)	24154.59	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11060.7207	***Sweep Width (Hz)***	24154.41
Temperature (degree C)	27.000						



^{13}C NMR spectrum of **12** (100.6 MHz, $CDCl_3$)

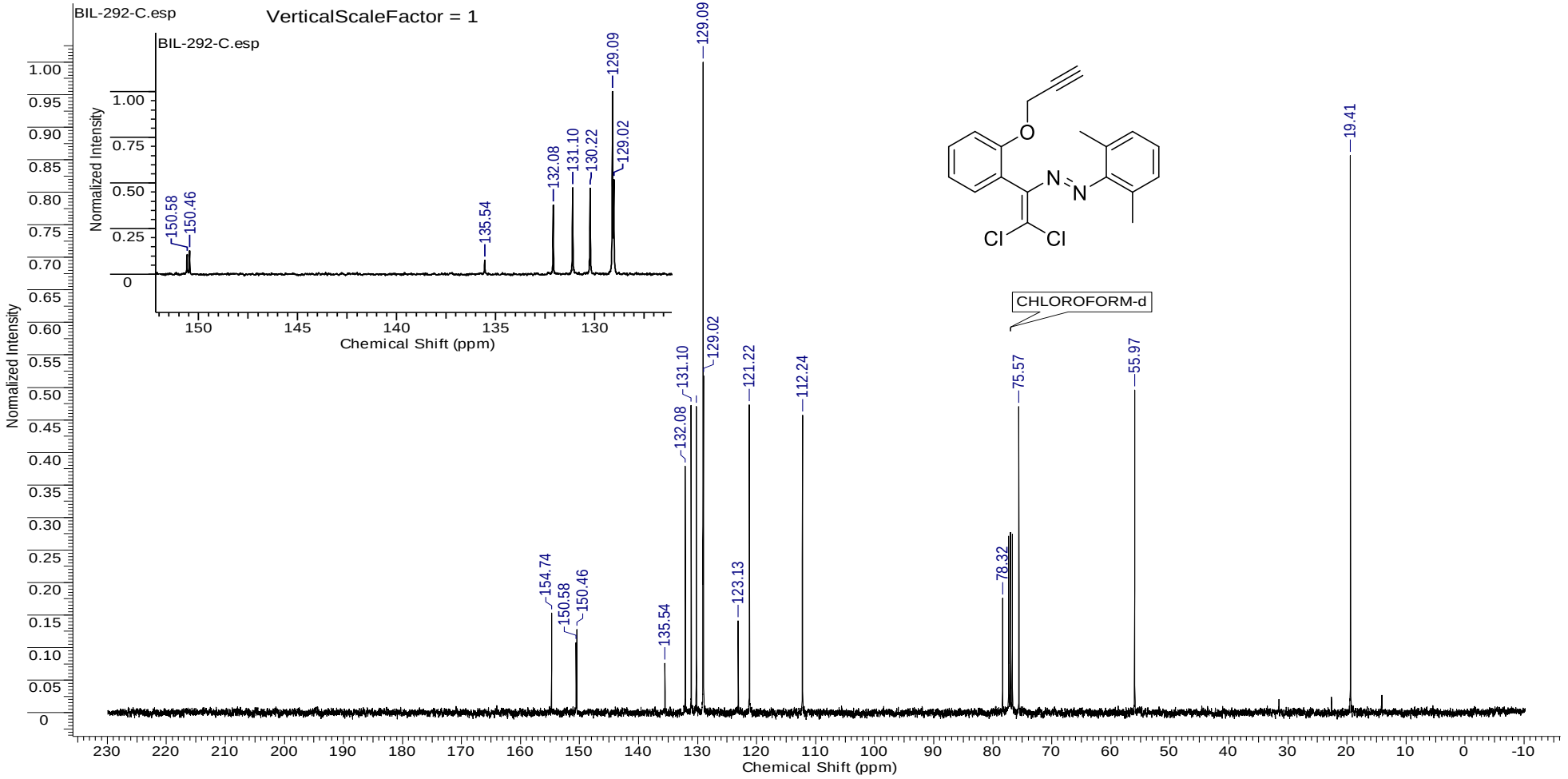
14.05.2019 20:17:46

Acquisition Time (sec)	2.5559	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	21 Nov 2018 14:30:24
Date Stamp	21 Nov 2018 14:30:24			***File Name***		***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	5	***Origin***	spect	***Original Points Count***	16384	***Owner***	root
Points Count	65536	***Pulse Sequence***	zg30	***Receiver Gain***	64.00	***SW(cyclical) (Hz)***	6410.26	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2595.9934	***Sweep Width (Hz)***	6410.16	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **13** (400.1 MHz, $CDCl_3$)

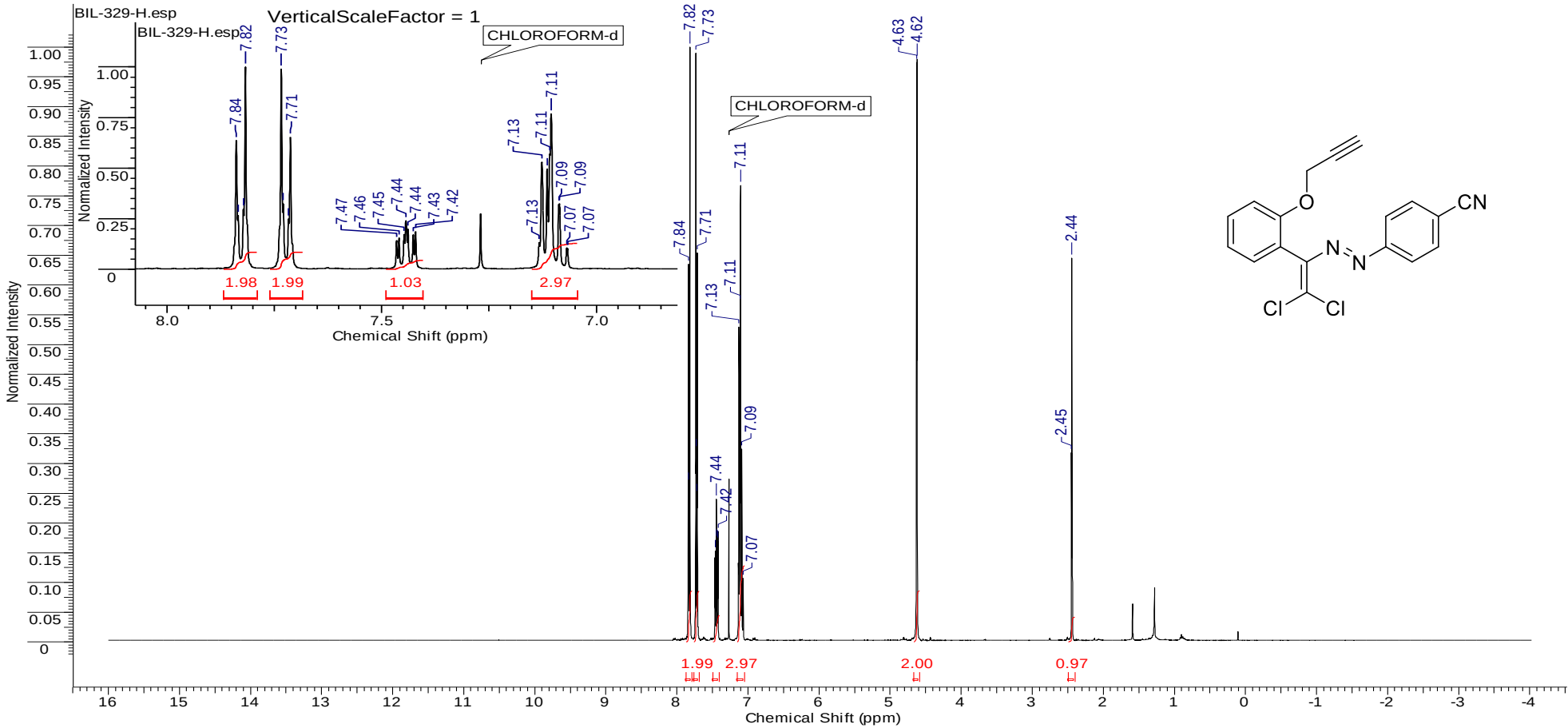
Acquisition Time (sec)	0.4999	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	22 Nov 2018 08:10:40		
Date Stamp	22 Nov 2018 08:10:40			***File Name***				***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	154	***Origin***	spect	***Original Points Count***	12076	***Owner***	root
Points Count	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	8192.00	***SW(cyclical) (Hz)***	24154.59		
Solvent	CHLOROFORM-d			***Spectrum Offset (Hz)***	11053.8105	***Sweep Width (Hz)***	24154.22	***Temperature (degree C)***	27.000



^{13}C NMR spectrum of **13** (100.6 MHz, $CDCl_3$)

14.05.2019 20:43:48

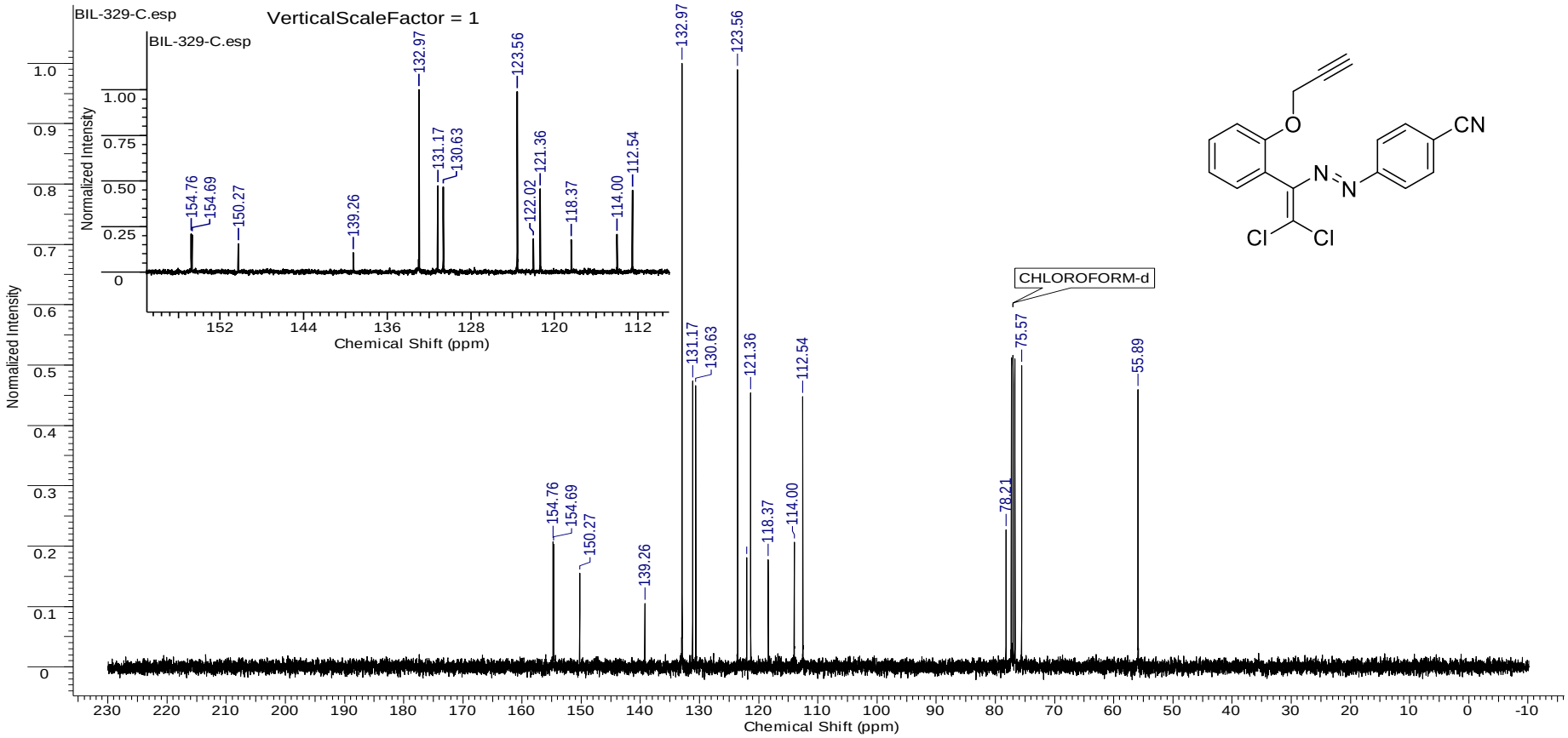
Acquisition Time (sec)	4.0894	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	05 Mar 2019 11:33:20
Date Stamp	05 Mar 2019 11:33:20						
File Name						***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	4	***Origin***	spect	***Original Points Count***	32768
Owner	root	***Points Count***	131072	***Pulse Sequence***	zg30	***Receiver Gain***	90.50
SW(cyclical) (Hz)	8012.82	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2395.8254
Sweep Width (Hz)	8012.76	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **14** (400.1 MHz, $CDCl_3$)

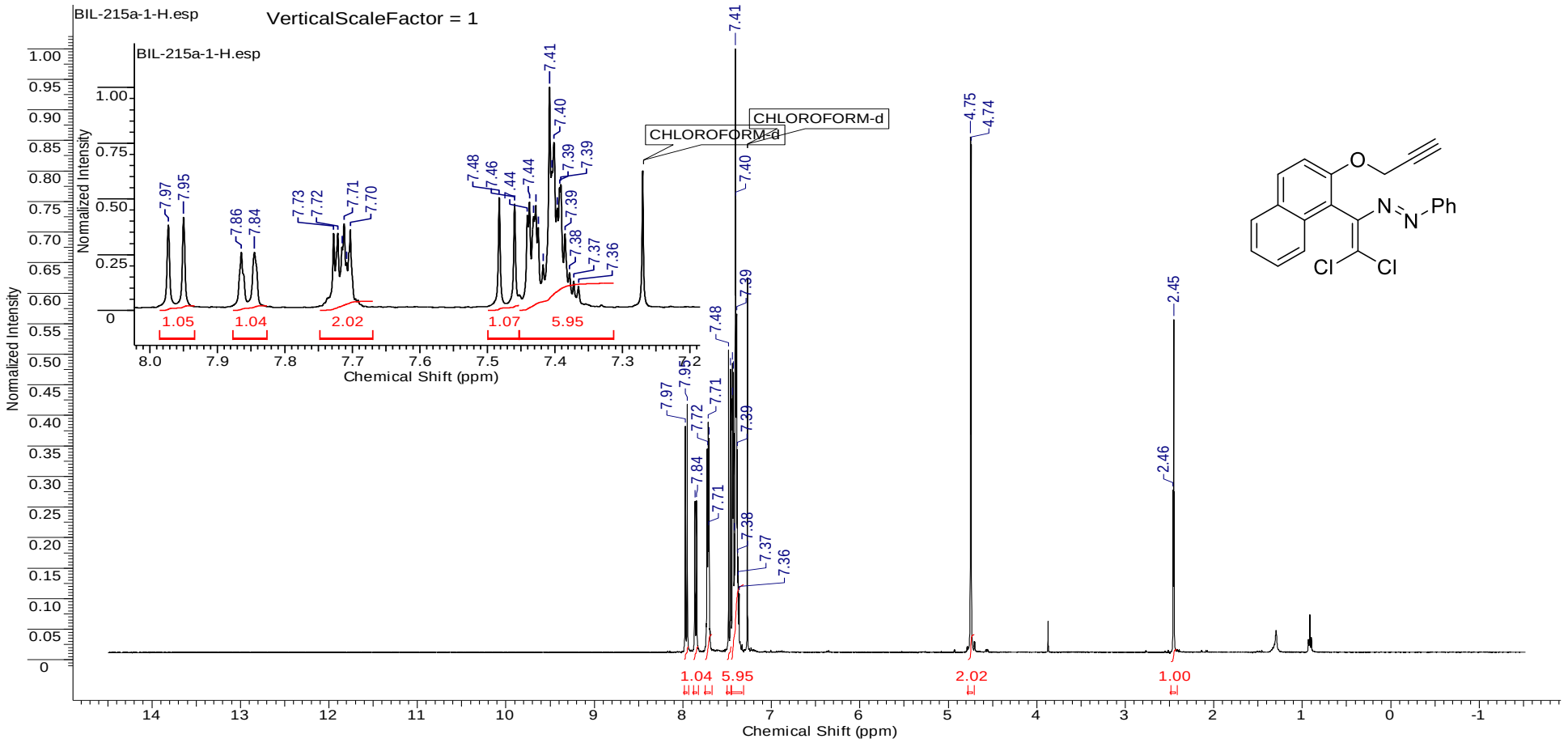
14.05.2019 20:45:16

Acquisition Time (sec)	0.6783	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	05 Mar 2019 11:35:28
Date Stamp	05 Mar 2019 11:35:28						
File Name						***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	65	***Origin***	spect	***Original Points Count***	16384
Owner	root	***Points Count***	131072	***Pulse Sequence***	zgpg30	***Receiver Gain***	16384.00
SW(cyclical) (Hz)	24154.59	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11057.0352	***Sweep Width (Hz)***	24154.41
Temperature (degree C)	27.000						



^{13}C NMR spectrum of **14** (100.6 MHz, $CDCl_3$)

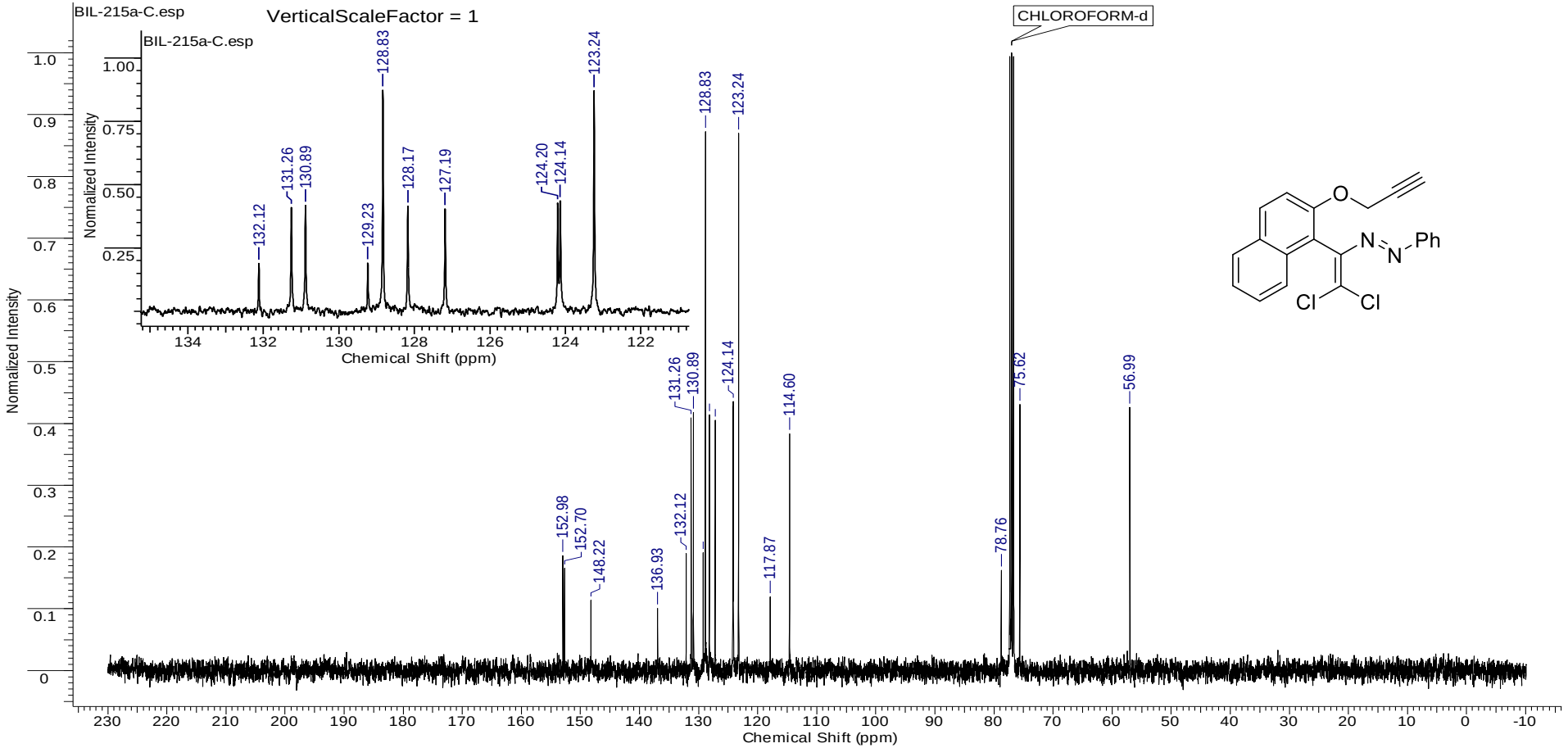
Acquisition Time (sec)	2.5559	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	22 Oct 2018 13:02:56
Date Stamp	22 Oct 2018 13:02:56						
File Name						***Frequency (MHz)***	400.13
Nucleus	1H	***Number of Transients***	4	***Origin***	spect	***Original Points Count***	16384
Owner	root	***Points Count***	65536	***Pulse Sequence***	zg30	***Receiver Gain***	181.00
SW(cyclical) (Hz)	6410.26	***Solvent***	CHLOROFORM-d			***Spectrum Offset (Hz)***	2595.7979
Sweep Width (Hz)	6410.16	***Temperature (degree C)***	27.000				



^{1}H NMR spectrum of **15** (400.1 MHz, $CDCl_3$)

12.05.2019 15:05:34

Acquisition Time (sec)	0.4999	***Comment***	5 mm BBO BB-1H/D Z3918/0123			***Date***	24 Oct 2018 15:00:16
Date Stamp	24 Oct 2018 15:00:16						
File Name						***Frequency (MHz)***	100.61
Nucleus	13C	***Number of Transients***	320	***Origin***	spect	***Original Points Count***	12076
Owner	root	***Points Count***	65536	***Pulse Sequence***	zgpg30	***Receiver Gain***	13004.00
SW(cyclical) (Hz)	24154.59	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	11061.5498	***Sweep Width (Hz)***	24154.22
Temperature (degree C)	27.000						



^{13}C NMR spectrum of **15** (100.6 MHz, $CDCl_3$)