Electronic Supplementary Information (ESI)

Oxidant Controlled Regio- and Stereodivergent Azidohydroxylation of Alkenes via I_2 Catalysis

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General Description: Solvents were purified and dried by standard procedures before use; petroleum ether of boiling range 60–80 °C was used. Melting points are uncorrected and recorded on a Buchi B-542 instrument. ^{1}H NMR and ^{13}C NMR spectra were recorded on Brucker AC-200 spectrometer unless mentioned otherwise. Deuterated solvent $CDCl_3$+ CCl_4 (70:30) were used as internal standard and singlet at 96.1 ppm in ^{13}C NMR corresponds to carbon of CCl_4. Elemental analysis was carried out on a Carlo Erba CHNS-O analyzer. HRMS data were recorded on a Waters SYNAPT G2 High Definition Mass Spectrometry System. Purification was done using column chromatography (230-400 mesh). The compounds **1a-m** and TBHP **(**5-6 M solution in decane: <4% water**)** are commercially available and were procured from Sigma Aldrich (used as such without any further purification). The relative configuration of diastereomers was determined by comparison of their ^{1}HNMR spectra with literature data.[4-7]

EXPERIMENTAL SECTION:

General experimental procedure for the preparation of vicinal azido alcohols (2a-n)

To a stirred solution of alkene (1 mmol) in DMSO: DMF (4 mL: 4 mL) at 0 °C was added I_2 (10 mol %) followed by dropwise addition of 5- 6 M TBHP in decane (2 mmol, 0.360 mL). The addition of Et_3N (1 mmol, 0.140 mL) was then done slowly (slow decolorisation of reaction mixture was observed) and finally sodium azide (2 mmol, 130 mg) was added pinchwise. The reaction mixture was then allowed to stir at room temperature for 8 hours (monitored by TLC). After completion, the reaction mixture was then cooled to 0 °C and excess sodium azide was quenched with water. Organic layer was diluted with EtOAc. The organic layer was separated and the aqueous layer was extracted with EtOAc (3 x 20 mL). The combined organic extracts were repeatedly washed with saturated brine solution, dried over anhyd. Na_2SO_4 and concentrated under reduced pressure to give crude products which were purified by column

chromatography [silica gel (230-400 mesh)] using petroleum ether: EtOAc (8:2) as an eluent to afford corresponding vicinal azido alcohol (**2a-n)** in 74-90% yield.

Experimental procedure for the preparation of *syn-(1RS,2RS)*-2-azido-1-phenylpropane-1,3-diol (2m):

To a stirred solution of alkene (10 mmol, 1.34 g) in DMSO: DMF (40 mL: 40 mL) at 0 °C was added I_2 (10 mol %, 0.253 g) followed by dropwise addition of 5- 6 M TBHP in decane (20 mmol, 3.60 mL). The addition of Et_3N (10 mmol, 1.3 mL) was then done slowly (slow decolorisation of reaction mixture was observed) and finally sodium azide (20 mmol, 1.28 g) was added pinchwise. The reaction mixture was then allowed to stir at room temperature for 8 hours (monitored by TLC). After completion, the reaction mixture was then cooled to 0 °C and excess sodium azide was quenched with water. Organic layer was diluted with EtOAc. The organic layer was separated and the aqueous layer was extracted with EtOAc (3 x 100 mL). The combined organic extracts were repeatedly washed with saturated brine solution, dried over anhyd. Na_2SO_4 and concentrated under reduced pressure to give crude products which were purified by column chromatography [silica gel (230-400 mesh)] using petroleum ether: EtOAc (8:2) as an eluent to afford corresponding vicinal azido alcohol (**2m)** in 88% (1.7 g) yield.

Synthesis of (±)-Chloramphenicol (9):

To a stirred solution of azidoalcohol (**2m**) (1.5 g, 7.7 mmol) in methanol (40 ml) was added 20% $Pd(OH)_2/C$ (50 mg) carefully at room temperature and a H_2 balloon was kept to provide hydrogen atmosphere. After the completion of the reaction as monitored by TLC, it was filtered over celite and the filtrate was concentrated under reduced pressure to give aminodiol, which

was added methyl dichloroacetate (3 mL) and heated at 90 °C for 1 h. The excess ester was removed under reduced pressure to give the crude product. To a stirred solution of conc. HNO_3: conc. H_2SO_4 (1:1) (5 mL) was added the crude product at -20 °C, the resulting solution was stirred for 1.5 h at 0 °C. After the completion of the reaction as monitored by TLC, it was poured into water and extracted with diethylether (3x50 mL), washed with water, brine and dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give crude product which were purified by column chromatography [silica gel (230-400 mesh)] using petroleum ether: EtOAc (6:4) as an eluent to afford chloramphenicol (**9**) in 71% yield.

General experimental procedure for the preparation of vicinal azido alcohols (3a-n):

To a stirred solution of alkene (1 mmol) in DMSO: DMF (4 ml: 4 ml) at 0 °C was added I_2 (10 mol %) followed by dropwise addition of 50% aqueous H_2O_2 (2 mmol, 0.140 mL). The addition of Et_3N (1 mmol, 0.140 mL) was then done slowly (vigorous decolorisation of reaction mixture was observed) and finally sodium azide (2 mmol, 130 mg) was added pinchwise. The reaction mixture was then allowed to stir at room temperature for 8 hours (monitored by TLC). Organic layer was diluted with EtOAc. The organic layer was separated and the aqueous layer was extracted with EtOAc (3 x 20 mL). The combined organic extracts were repeatedly washed with saturated brine solution, dried over anhyd. Na_2SO_4 and concentrated under reduced pressure to give crude products which were purified by column chromatography [silica gel (230-400 mesh)] using petroleum ether: EtOAc (8:2) as an eluent to afford corresponding vicinal azido alcohol (**3a-n)** in 74-92% yield.

***tert*-butyl *anti*-(1*RS*,2*RS*)-2,3-dihydroxy-1-(4-methoxyphenyl)propyl)carbamate (10):**

To a stirred solution of azidoalcohol **3n** (0.5g, 2.2 mmol) in MeOH (20 mL) was added 20% $Pd(OH)_2/C$ (25 mg) carefully at room temperature and a H_2 balloon was kept to provide hydrogen atmosphere. After the completion of the reaction as monitored by TLC, it was filtered over celite and the filtrate was concentrated under reduced pressure to give aminodiol, which was added $(Boc)_2$ (2.4 m mol, 0.487 g) and Et_3N (4.4 mmol, 0.44 g) and allowed to stir at 25 °C for 2 h. After the completion of the reaction as monitored by TLC, it was poured into water and extracted with diethylether (3x50 mL), washed with water, brine and dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give crude product which were purified by column chromatography [silica gel (230-400 mesh)] using petroleum ether: EtOAc (6:4) as an eluent to afford compound **9** in 76% yield (496 mg).

(±)-Cytoxazone (11):

To a solution of *anti*-3-amino-1,2-diol **9** (0.3 g, 1.0 mmol) in dry THF (10 mL) was added NaH (0.05 g, 60% w/w, 2.0 mmol) at 25 °C, and the mixture was stirred under nitrogen atmosphere for 3 h. The reaction mixture was concentrated and the resulting mixture was extracted with EtOAc (3 x 10 mL), washed with saturated aq. NH_4Cl (5 mL) and brine solution (5 mL). The organic layers were separated, dried over anhyd. Na_2SO_4, and concentrated to give the crude product, which was then purified by column chromatography over silica gel using pet. ether:EtOAc (60:40) as am eluent to give **10** (0.2 g) as a colorless solid.

2-azido-2phenylethan-1-ol (^{18}O-3a): To a stirred solution of styrene (0.5 mmol) in DMSO: ^{18}O-DMF (which was purified and prepared by dimethylaminomethylene)dimethylamm-onium chloride and ^{18}O-H_2O (>97%-18O) heating in 110 °C for 12 hours) (0.5 ml: 0.5 ml) at 0 °C was added I_2 (10 mol %) followed by dropwise addition of 50% aqueous H_2O_2 (1 mmol). The addition of Et_3N (0.5 mmol) was then done slowly (vigorous decolorisation of reaction mixture

was observed) and finally sodium azide (1 mmol) was added pinchwise. The reaction mixture was then allowed to stir at room temperature for 8 hours (monitored by TLC). Organic layer was diluted with EtOAc. The organic layer was separated and the aqueous layer was extracted with EtOAc (3 x 8 mL). The combined organic extracts were repeatedly washed with saturated brine solution, dried over anhyd. Na_2SO_4 and concentrated under reduced pressure to give crude products which were purified by column chromatography [silica gel (230-400 mesh)] using petroleum ether: EtOAc (8:2) as an eluent to afford corresponding vicinal azido alcohol (**^{18}O-3a)** in 78% yield and (**^{18}O-2a)** in 6% yield. The ^{1}HNMR and ^{13}C NMR data was in well agreement as **3a** and **2a** compounds. **HRMS** calcd for $[(C_8H_9N_3O+H)^+]$ 166.0861; found: 166.0860.

2-azido-1-phenylethan-1-ol (2a)[1]

Yield: 90% (146 mg); Colorless viscous liquid; R_f = 0.40 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 1031, 1101, 1247, 2103, 2847, 2933, 3356; **^{1}H NMR** (400 MHz,$CDCl_3$): δ 2.61 (br. s, 1H), 3.41 (dd, *J* = 3.7, 12.4, 1H), 3.47 (dd, *J* = 8.2, 12.4, 1H,), 4.85 (dd, *J* = 8.2, 3.9 Hz, 1H), 7.30 - 7.39 (m, 5H); **^{13}C NMR** (50 MHz, $CDCl_3$) δ 58.1, 73.4, 125.9, 128.3, 128.7, 140.6; **HRMS** calcd for $[(C_8H_9N_3O+Na)^+]$ 186.0638; found: 186.0640.

2-azido-1-(p-tolyl)ethan-1-ol (2b)[1]

Yield: 88% (155 mg); Colorless gum; R_f = 0.40 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 750, 1222, 2095, 2950, 3020, 3412; **^{1}H NMR** (200 MHz,$CDCl_3$) δ 2.34 (s, 3H), 2.63 (br. s., 1H), 3.32 - 3.51 (m, 2H), 4.81 (dd, *J* = 7.6, 4.4 Hz, 1H), 7.17 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 8.2 Hz, 2H); **^{13}C NMR** (50 MHz, $CDCl_3$) δ 21.1, 58.0, 73.2, 125.8, 129.3, 137.6, 138.1; **HRMS** calcd for $[(C_9H_{11}N_3O+Na)^+]$: 200.0794; found: 200.0793.

4-(2-azido-1-hydroxyethyl)phenol (2c)

Yield: 76% (135 mg); Colorless liquid; R_f = 0.40 (Pet ether: EtOAc = 7: 3); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 1247,1607, 2103, 2923, 3356; **^{1}H NMR** (200 MHz,$CDCl_3$) δ 3.41 - 3.46 (m, 2H), 4.32 - 4.36 (m, 1H), 4.81 (dd, *J* = 7.7, 4.5 Hz, 1H), 6.81 (d, 2H, *J* = 8.5 Hz), 7.24 (d, 2H, *J* = 8.4 Hz), 7.97 (s, D_2O exchangeable, 1H); **^{13}C NMR** (50 MHz,$CDCl_3$) δ 58.1, 73.1, 115.6, 127.5, 128.6, 155.8; **HRMS** calcd for [$(C_8H_9N_3O_2+Na)^+$] 202.0587 found 202.0579.

2-azido-1-(2-bromophenyl)ethan-1-ol (2d)

Yield: 82% (196 mg); Colorless liquid; R_f = 0.40 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 761, 1012, 1214, 2103, 2724, 3018; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 2.46 (d, *J* = 3.4 Hz, 1H), 3.35 (dd, *J* = 12.6, 8.2 Hz, 1H), 3.60 (dd, *J* = 12.6, 2.9 Hz, 1H), 5.26 (dt, 3.0 Hz, 1H), 7.19 (t, *J* = 8.5 Hz, 1H), 7.38 (t, *J* = 8.5 Hz, 1H), 7.54 (d, *J* = 8.8 Hz, 1H), 7.64 (d, *J* = 9.1 Hz, 1H); **^{13}C NMR** (125 MHz,$CDCl_3$) δ 56.5, 72.4, 121.7, 127.8, 127.9, 129.7, 132.8, 139.5; **HRMS** calcd for [$(C_8H_8BrN_3O+Na)^+$] 263.9743 found 263.9738.

2-azido-1-(3-nitrophenyl)ethan-1-ol (2e)[8]

Yield: 77% (160 mg): Colorless liquid; R_f = 0.40 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 1211, 1350, 1531, 2104, 3438; **^{1}H NMR** (200 MHz, $CDCl_3$) δ 2.59 (s, 1H), 3.52 - 3.56 (m, 2H), 5.01 (t, *J* = 5.9 Hz, 1H), 7.58 (t, *J* = 7.9 Hz, 1H), 7.75 (d, *J* = 7.8 Hz, 1H), 8.19 - 8.30 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 57.9, 72.3, 121.0, 123.2, 129.6, 132.0, 142.6, 148.4. **HRMS** calcd for [$(C_8H_8N_4O_4+H)^+$] 209.0674 found 209.0673.

1-azido-2-phenylpropan-2-ol (2f)[2]

Yield: 78% (140 mg); Colorless gum; R_f = 0.40 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 761, 1272, 2096, 2828, 2950, 3020, 3422 ; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.61 (s, 3H), 2.36 (s, 1H), 3.45 (d, *J* = 12.3 Hz, 1H), 3.61 (d, *J* = 12.3 Hz, 1H), 7.29 - 7.32 (m, 1H), 7.37 – 7.40 (m, 2H), 7.45 - 7.47 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 27.1, 62.2, 74.5, 124.8, 127.5, 128.5, 144.7; **HRMS** calcd for [$(C_9H_{11}N_3O + Na)^+$] 200.0794; found: 200.0794.

1-azidooctan-2-ol (2g) [3]

Yield: 79% (135 mg); Colorless liquid; R_f = 0.60 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 759, 1261, 2104, 2937, 3404 ; **^{1}H NMR** (200 MHz,$CDCl_3$) δ 0.82 (t, *J* = 6.2 Hz, 3H), 1.23 (br. s., 7H), 1.38 (br. s., 3H), 1.97 (br. s., 1H), 3.18 - 3.34 (m, 2H), 3.67 (br. s., 1H); **^{13}C NMR** (50 MHz,$CDCl_3$) δ 14.1, 22.6, 25.4, 29.2, 31.8, 34.3, 57.2, 70.8; **HRMS** calcd for [$(C_8H_{17}N_3O+Na)^+$] 194.1264; found:194.1263.

1-azido-4-(benzyloxy)-2-methylbutan-2-ol (2h)

Yield: 84% (196 mg); Colorless liquid; R_f = 0.50 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 705, 1082, 1274, 1717, 2106, 2926, 2974, 3412; **^{1}H NMR** (400 MHz,$CDCl_3$) δ 1.35 (s, 3H), 1.82 – 1.99 (m, 2H), 2.59 (d, *J* = 4.9 Hz, 1H), 2.70 (d, *J* = 4.9 Hz, 1H), 3.53- 3.62 (m, 2H), 4.50 (s, 2H), 7.28 - 7.37 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 21.6, 36.6, 53.9, 55.4, 66.6, 73.0, 127.6, 128.4, 138.3; **HRMS** calcd for [$(C_{12}H_{17}N_3O_2+Na)^+$] 258.1213; found: 258.1209.

3-azido-4-(benzyloxy)-2-methylbutan-2-ol (2i)

Yield: 74% (175 mg); Colorless liquid; R_f = 0.50 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 705, 765, 1082, 1274, 1377, 1612, 1717, 2106, 2926, 2974, 3412; **^{1}H NMR** (200 MHz,$CDCl_3$) δ 1.27 (s, 3H), 1.35 (s, 3H), 2.98 (t, *J* = 5.4 Hz, 1H), 3.51 - 3.69 (m, 2H), 4.49 - 4.67 (m, 2H), 7.30 - 7.36 (m, 5H); **^{13}C NMR** (50 MHz,$CDCl_3$) δ 18.9, 24.7, 57.5, 61.9, 68.8, 73.2, 127.8, 128.4, 137.9; **HRMS** calcd for [$(C_{12}H_{17}N_3O_2+Na)^+$] 258.1213; found: 258.1210.

***cis*-(1*RS*,2*SR*)-2-azidocyclohexan-1-ol (2j)**

Yield: 87% (122 mg); Colorless liquid; R_f = 0.40 (Pet ether: EtOAc = 9: 1); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 760, 1259, 2102, 2937, 3403; **^{1}H NMR** (400 MHz,$CDCl_3$) δ 1.28 - 1.34 (m, 3H), 1.37 - 1.56 (m, 1H), 1.83 - 1.90 (m, 1H), 2.00 - 2.16 (m, 2H), 2.34 (d, *J* = 2.3 Hz, 1H), 2.45 - 2.51 (m, 1H), 3.66 (td, *J* = 9.8, 3.9 Hz, 1H), 4.05 (ddd, *J* = 12.4, 9.8, 4.4 Hz, 1H); **^{13}C NMR** (50 MHz, $CDCl_3$) δ 24.4, 28.0, 33.6, 38.6, 43.5, 76.0; **HRMS** calcd for [$(C_6H_{11}N_3O+Na)^+$] 164.0794; found: 164.0794.

***cis*-(1*RS*,2*SR*)-2-azido-2,3-dihydro-1H-inden-1-ol (2k)**[5]

Yield: 87% (152 mg); Colorless liquid; R_f = 0.50 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 770, 1219, 2097, 2916, 3356; **^{1}H NMR** (500 MHz,$CDCl_3$) δ 2.39 (d, *J* = 5.2 Hz, 1H), 3.14 - 3.22 (m, 2H), 4.35 (q, *J* = 5.2 Hz, 1H), 5.16 (s, 1H), 7.28 - 7.34 (m, 3H), 7.45 - 7.47 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 35.2, 65.7, 76.4, 124.7, 125.1, 127.6, 129.0, 139.0, 141.9; **HRMS** calcd for [$(C_9H_9N_3O+Na)^+$] 198.0638; found:198.0640.

***syn*-(1*RS*,2*RS*)-2-azido-1-phenylpropan-1-ol (2l)**[6]

Yield: 88% (155 mg); Colorless gum; R_f = 0.40 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 771, 1229, 1605, 2101, 2926, 3013, 3346; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.58 (d, *J* = 6.1 Hz, 3H), 4.56 - 4.66 (m, 1H), 5.12 (d, *J* = 7.8 Hz, 1H), 7.31 - 7.45 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 18.4, 80.6, 84.8, 126.0, 129.2, 129.8, 135.2; **HRMS** calcd for [$(C_9H_{11}N_3O+Na)^+$] 200.0794; found: 200.0788;

***syn*-(1*RS*,2*RS*)-2-azido-1-phenylpropane-1,3-diol (2m)**[7]

Yield: 80% (115 mg); colorless gum; R_f = 0.30 (Pet ether: EtOAc = 6: 4); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 761, 1219, 1605, 2105, 2893, 2933, 3013, 3416; **^{1}H NMR** (200 MHz, $CDCl_3$) δ 2.64 (br. s., 1H), 2.70 (br. s, 1H), 3.51 - 3.73 (m, 2H), 3.83 (d, *J* = 4.7 Hz, 1H), 4.85 (t, *J* = 6.5 Hz, 1H), 7.34-7.43 (m, 5 H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 62.8, 67.0, 74.0, 126.5, 127.8, 128.9, 136.2; **HRMS** calcd for [$(C_9H_{11}N_3O_2+Na)^+$] 216.0749 found: 216.0736.

***syn*-(1*RS*,2*RS*)-2-azido-1-(4-methoxyphenyl)propane-1,3-diol (2n)**

Yield: 82% (182 mg); colorless gum: R_f = 0.30 (Pet ether: EtOAc = 6: 4); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 754, 1222, 2103, 2822, 2937, 3397; **^{1}H NMR** (200 MHz, $CDCl_3$) δ 3.51 - 3.90 (m, 6H), 4.78 (t, *J* = 5.56 Hz, 1H), 6.88- 6.95 (m, 2H), 7.29- 7.35 (m, 2H). **^{13}C NMR** (100 MHz,) $CDCl_3$ δ 55.3, 62.7, 69.1, 74.4, 114.1, 127.6, 132.2, 159.7; **HRMS** calcd for [$(C_{10}H_{13}N_3O_3+H)^+$] 224.1035 found: 224.1033.

(±)-Chloramphenicol (9)

Yield: 71% (1.8g); colorless gum; R_f = 0.40 (Pet ether: EtOAc = 7: 3); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 850, 1049, 1216, 1348, 1416, 1454, 1523, 1604, 1686, 2929, 3020, 3420; **^{1}H NMR** (400 MHz, acetone- d_6) δ 3.58 - 3.88 (m, 2H), 4.09 - 4.17 (m, 1H), 4.52 (br. s., 3H), 5.25 (s, 1H), 6.10 (s, 1H), 7.60 (d, *J* = 8.5 Hz, 2H), 8.09 (d, *J* = 8.5 Hz, 2H); **^{13}C NMR** (100 MHz, acetone- d_6) δ 55.9, 60.6, 65.7, 69.6, 122.3, 126.3, 146.3, 149.2, 163.4; **HRMS** calcd for $[(C_{11}H_{12}Cl_2N_2O_5+Na)^+]$ 345.0015; found: 345.0009.

2-azido-2-phenylethan-1-ol (3a)[1]

Yield: 82% (150 mg); Colorless liquid; R_f = 0.35 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 1026, 1105, 1227, 2106, 2847, 2933, 3416; **^{1}H NMR** (400 MHz,$CDCl_3$) δ 2.02 (s, 1H), 3.74 (t, *J* = 5.6 Hz, 2H), 4.68 (t, *J* = 6.4 Hz, 1H), 7.33 - 7.41 (m, 5H); **^{13}C NMR** (50 MHz,$CDCl_3$) δ 66.3, 67.7, 127.1, 128.6, 128.8, 136.2 ; **HRMS** calcd for $[(C_8H_9N_3O +Na)^+]$ 186.0638; found: 186.0640.

2-azido-2-(p-tolyl)ethan-1-ol (3b)[1]

Yield: 89% (158 mg); Colorless liquid; R_f = 0.35 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 752, 1232, 2104, 2893, 2950, 3021, 3382; **^{1}H NMR** (200 MHz, $CDCl_3$) δ 2.35 (s, 3H), 3.69 (d, *J* = 6.4 Hz, 2H), 4.60 (t, *J* = 6.4 Hz, 1H), 7.19 (s, 4H); **^{13}C NMR** (50 MHz, $CDCl_3$) δ 21.1, 66.3, 67.6, 127.1, 129.5, 133.2, 138.4; **HRMS** calcd for $[(C_9H_{11}N_3O +Na)^+]$ 200.0794; found: 200.0793;

2-azido-2-(2-bromophenyl)ethan-1-ol (3d)

Yield: 86% (205 mg); Colorless liquid; R_f = 0.35 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 761, 1032, 1255, 2103, 2931, 3367; **^{1}H NMR** (500 MHz,$CDCl_3$) δ 2.24 (br. s., 1H), 3.63 (t, *J* = 9.6 Hz, 1H), 3.87 (d, *J* = 11.0 Hz, 1H), 5.18 (dd, *J* = 8.1, 3.7 Hz, 1H), 7.19 - 7.22 (m, 1H), 7.37 (t, *J* = 7.3 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.59 (d, *J* = 7.9 Hz, 1H); **^{13}C NMR** (125 MHz,$CDCl_3$) δ 65.4, 66.7, 123.1, 128.0, 128.5, 129.9, 133.1, 135.8; **HRMS** calcd for $[(C_8H_8BrN_3O + Na)^+]$ 263.9743; found: 263.9739.

2-azido-2-(3-nitrophenyl)ethan-1-ol (3e)

Yield: 76% (158 mg); Colorless liquid; R_f = 0.35 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 757, 1350, 1530, 1631, 2107, 2925, 3085, 3413; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 2.81 (dd, *J* = 5.4, 2.5 Hz, 1H), 3.24 (dd, *J* = 5.4, 4.0 Hz, 1H), 3.98 (dd, *J* = 3.9, 2.5 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.62 (d, *J* = 7.6 Hz, 1 H), 8.17 - 8.19 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 51.4, 76, 120.7, 123.1, 129.6, 131.4, 140.1; **HRMS** calcd for $[(C_8H_8N_4O_4+H)^+]$ 209.0674 found 209.0670.

2-azido-2-phenylpropan-1-ol (3f)[2]

Yield: 83% (146 mg); Colorless liquid; R_f = 0.35 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} **^{1}H NMR** (200 MHz,$CDCl_3$) δ 1.43 (s, 3H), 2.32 (s, 1H), 3.50 (d, *J* = 11.3 Hz, 1H), 3.67 (d, *J* = 11.3 Hz, 1H), 7.21 - 7.37 (m, 5H); **^{13}C NMR** (50 MHz, $CDCl_3$) δ 26.0, 70.9, 74.8, 125.1, 127.1, 128.4, 145.0; **HRMS** calcd for $[(C_9H_{11}N_3O + Na)^+]$ 200.0794; found: 200.0794.

2-azidooctan-1-ol (3g)[3]

Yield: 83% (142 mg); Colorless liquid; R_f = 0.50 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} **^{1}H NMR** (200 MHz,$CDCl_3$) δ 0.87 - 0.93 (m, 3H), 1.30 (br. s., 8H), 1.50 (d, *J* = 5.2 Hz,

2H), 1.95 (br. s., 1H), 3.41 - 3.60 (m, 2H), 3.62 - 3.76 (m, 1H); **^{13}C NMR** (50 MHz, $CDCl_3$) δ 14.1, 22.6, 26.0, 29.1, 30.6, 31.7, 64.5, 65.2; **HRMS** calcd for [$(C_8H_{17}N_3O+Na)^+$] 194.1264; found:194.1263.

2-azido-4-(benzyloxy)-2-methylbutan-1-ol (3h)

Yield: 74% (172 mg); Colorless liquid; R_f = 0.45 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 744, 1104, 1292, 2102, 2867, 2926, 3460; **^{1}H NMR** (400 MHz,$CDCl_3$) δ 1.25 (s, 3H), 1.64 (s, 1H), 1.74 - 1.81 (m, 1H), 1.92 – 1.99 (m, 1H), 3.24 (s, 2H), 3.58 (s, 1H), 3.70 - 3.76 (m, 2H), 4.55 (s, 2H), 7.31 - 7.40 (m, 5H); **^{13}C NMR** (100 MHz,$CDCl_3$) δ 25.0, 37.5, 60.5, 66.9, 72.8, 73.4, 127.8, 127.9, 128.5, 137.4; **HRMS** calcd for [$(C_{12}H_{17}N_3O_2+Na)^+$] 258.1213; found: 258.1209.

3-azido-1-(benzyloxy)-3-methylbutan-2-ol (3i)

Yield: 78% (185 mg); Colorless liquid; R_f = 0.45 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 771,1077, 1456, 2121, 2924, 3416; **^{1}H NMR** (500 MHz,$CDCl_3$) δ 1.17 (s, 3H), 1.23 (s, 3H), 2.16 (s, 1H), 3.55 - 3.59 (m, 2H), 3.64 (d, *J* = 8.2 Hz, 1H), 4.54 (dd, *J* = 12.2, 4.6 Hz, 2H), 7.31 - 7.38 (m, 5H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 25.2, 26.6, 71.5, 71.8, 73.7, 75.6, 127.8, 128.0, 128.5, 137.5; **HRMS** calcd for [$(C_{12}H_{17}N_3O_2+Na)^+$] 258.1213; found: 258.1210.

***trans*-(1*S*,2*S*)-2-azidocyclohexan-1-ol (3j)**

Yield: 92% (130 mg); Colorless liquid; R_f = 0.35 (Pet ether: EtOAc = 9: 1); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 764, 1285,1455, 2100, 3410; **^{1}H NMR** (400 MHz,$CDCl_3$) δ 1.25 - 1.33 (m, 4H), 1.65 - 1.8 (br. s, 2H), 1.97- 2.04 (m, 2H), 2.76 (br. s., 1H), 3.12 - 3.18 (m, 1H), 3.35 (dt, *J* = 9.6, 4.5 Hz,

1H); **^{13}C NMR** (100 MHz,$CDCl_3$) δ 23.8, 24.1, 29.7, 33.0, 66.9, 73.4; **HRMS** calcd for [$(C_6H_{11}N_3O$ +Na)$^+$] 164.0794; found: 164.0794.

***trans*-(1*RS*,2*RS*)-1-azido-2,3-dihydro-1H-inden-2-ol (3k)**[5]

Yield: 82% (144 mg); Colorless liquid; R_f = 0.40 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 761, 1219, 2098, 2844, 2926, 3366; **^{1}H NMR** (400 MHz,$CDCl_3$) δ 2.86 (dd, *J* = 16.0, 5.9 Hz, 1 H), 3.28 (dd, *J* = 16.0, 6.7 Hz, 1H), 3.44 (br. s, 1H), 4.46 (q, *J* = 5.9 Hz, 1H), 4.65 (d, *J* = 4.9 Hz, 1H), 7.29-7.39 (m, 4H); **^{13}C NMR** (100 MHz,$CDCl_3$) δ 35.2, 65.7, 76.4, 124.7, 125.1, 127.6, 129.0, 139.0, 141.9; **HRMS** calcd for [$(C_9H_9N_3O$+Na)$^+$] 198.0638; found:198.0640.

***anti*-(1*RS*,2*SR*)-1-azido-1-phenylpropan-2-ol (3l)**[6]

Yield: 86% (152 mg); Colorless liquid R_f = 0.35 (Pet ether: EtOAc = 8: 2); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 759, 1269, 2109, 2829, 2950, 3020, 3322; **^{1}H NMR** (200 MHz,$CDCl_3$) δ 1.11 (d, *J* = 6.2 Hz, 3H), 1.63 (br. s., 1H), 3.88 (quin, *J* = 6.1 Hz, 1H), 4.38 (d, *J* = 5.8 Hz, 1H), 7.23 - 7.35 (m, 5H); **^{13}C NMR** (50 MHz,$CDCl_3$) δ 18.6 , 70.6, 71.6, 127.8, 128.6, 128.9, 136.3; **HRMS** calcd for [$(C_9H_{11}N_3O$ +Na)$^+$] 200.0794; found: 200.0794.

***anti*-(2*RS*,3*RS*)-3-azido-3-phenylpropane-1,2-diol (3m)**[7]

Yield: 78% (112 mg); Colorless liquid; R_f = 0.25 (Pet ether: EtOAc = 6: 4); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 777, 1309, 1604, 2107, 2933, 3014, 3389; **^{1}H NMR** (400 MHz,$CDCl_3$) δ 2.68 (br. s., 2 H), 3.61 - 3.71 (m, 2H), 3.80 (td, *J* = 6.4, 3.3 Hz, 1H), 4.59 (d, *J* = 7.1 Hz, 1H), 7.34 - 7.44 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 62.8, 67.1, 74.0, 127.8, 128.8, 129.0, 136.2; **HRMS** calcd for [$(C_9H_{11}N_3O_2$+Na)$^+$] 216.0749 found: 216.0745.

***anti*-(2*RS*,3*RS*)-3-azido-3-(4-methoxyphenyl)propane-1,2-diol (3n)**

Yield: 80% (890 mg); colorless liquid; R_f = 0.25 (Pet ether: EtOAc = 6: 4); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 1035, 1195, 1513, 1616, 2100, 2920, 3050, 3368 (broad); **^{1}H NMR** (200 MHz,$CDCl_3$) δ 2.73 (br. s., 1H), 3.21 - 3.68 (m, 2H), 3.74 - 3.82 (m, 4H), 4.52 (d, *J* = 7.2 Hz, 1 H), 6.92 (d, *J* = 8.7 Hz, 2 H), 7.27 (d, *J* = 8.7 Hz, 2 H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 55.2, 63.0, 66.4, 73.9, 114.3, 128.0, 129.1, 159.8; **HRMS** calcd for $[(C_{10}H_{13}N_3O_3+H)^+]$ 224.1035 found: 224.1036.

***tert*-butyl-a*nti*-(1*RS*,2*RS*)-2,3-dihydroxy-1-(4-methoxyphenyl)propyl)carbamate (10):**

Yield: 76% (496 mg); colorless liquid; R_f = 0.25 (Pet ether: EtOAc = 5: 5); **mp:** 114-116 ºC, (lit.[67] **mp**: 116-118 ºC); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 669, 757, 831, 927, 1035, 1167, 1216, 1368, 1585, 1612, 1701, 2400, 2839, 2981, 3019, 3438, 3682; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 1.34 (br. s., 10H), 3.01 - 3.25 (m, 1H), 3.54 (br. s., 2H), 3.71 (s, 4H), 4.55 (br. s., 1H), 5.29 (br. s., 1H), 6.78 (d, *J* = 5.5 Hz, 2H), 7.15 (d, *J* = 6.7 Hz, 2H): **^{13}C NMR** (126 MHz, $CDCl_3$) δ 28.3, 55.2, 56.1, 63.2, 74.1, 76.7, 77.3, 80.1, 96.1, 114.2, 128.5, 131.1, 156.2, 159.2; **HRMS** calcd for $[(C_{15}H_{24}NO_5+H)^+]$ 298.1654 found: 298.1650.

***anti*-(4*RS*,5*RS*)-5-(hydroxymethyl)-4-(4-methoxyphenyl) oxazolidin-2-one (11)**

Yield: 90% (335 mg); colorless solid; **mp:** 116-118 °C, (lit.[67] **mp**: 119-121 °C); R_f = 0.25 (Pet ether: EtOAc = 7: 3); **IR** ($CHCl_3$, cm^{-1}) ν_{max} 769, 843, 1028, 1248, 1395, 1513, 1610, 1733, 2580, 2924, 3272; **^{1}H NMR** (500 MHz, *DMSO-d*$_6$) δ 3.00 - 3.03 (m, 2H), 3.26 (t, *J* = 3.7 Hz, 2H), 3.77 (s, 3H), 4.69- 4.71 (m, 2H), 4.88 (d, *J* = 8.2 Hz, 1H), 6.89 (dd, *J* = 8.4, 2.0 Hz, 2H), 7.15 (d, *J* = 8.5 Hz, 2H), 7.98 (br. s., 1H): **^{13}C NMR** (126 MHz, *DMSO-d*$_6$) δ 39.0, 39.2, 39.3, 39.7, 39.8,

40.0, 54.8, 56.3, 60.9, 78.4, 78.6, 78.9, 80.0, 95.5, 113.4, 127.8, 129.0, 158.6, 158.9. **HRMS** calcd for $[(C_{11}H_{13}NO_4+H)^+]$ 224.0922; found: 224.0920.

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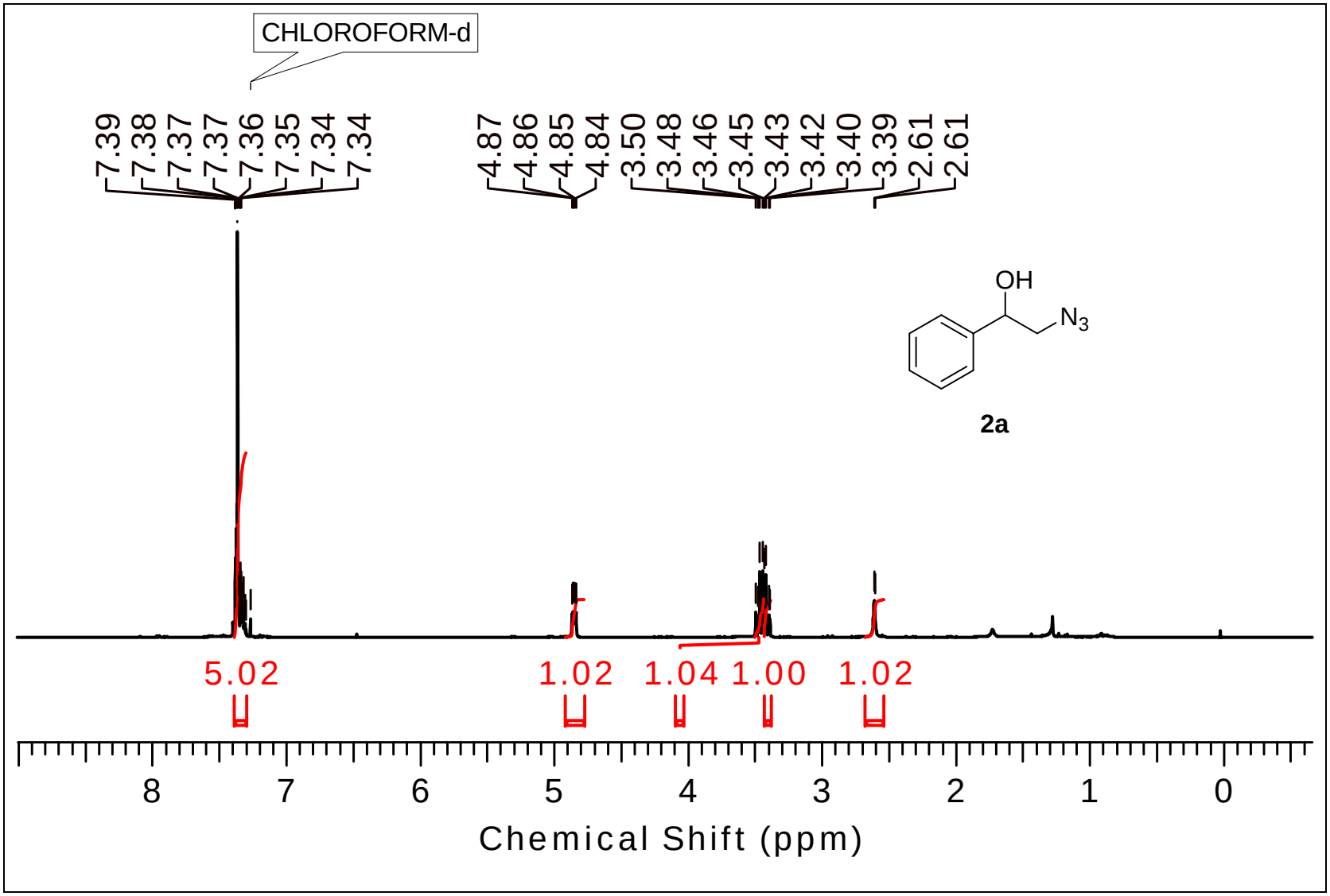
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N3
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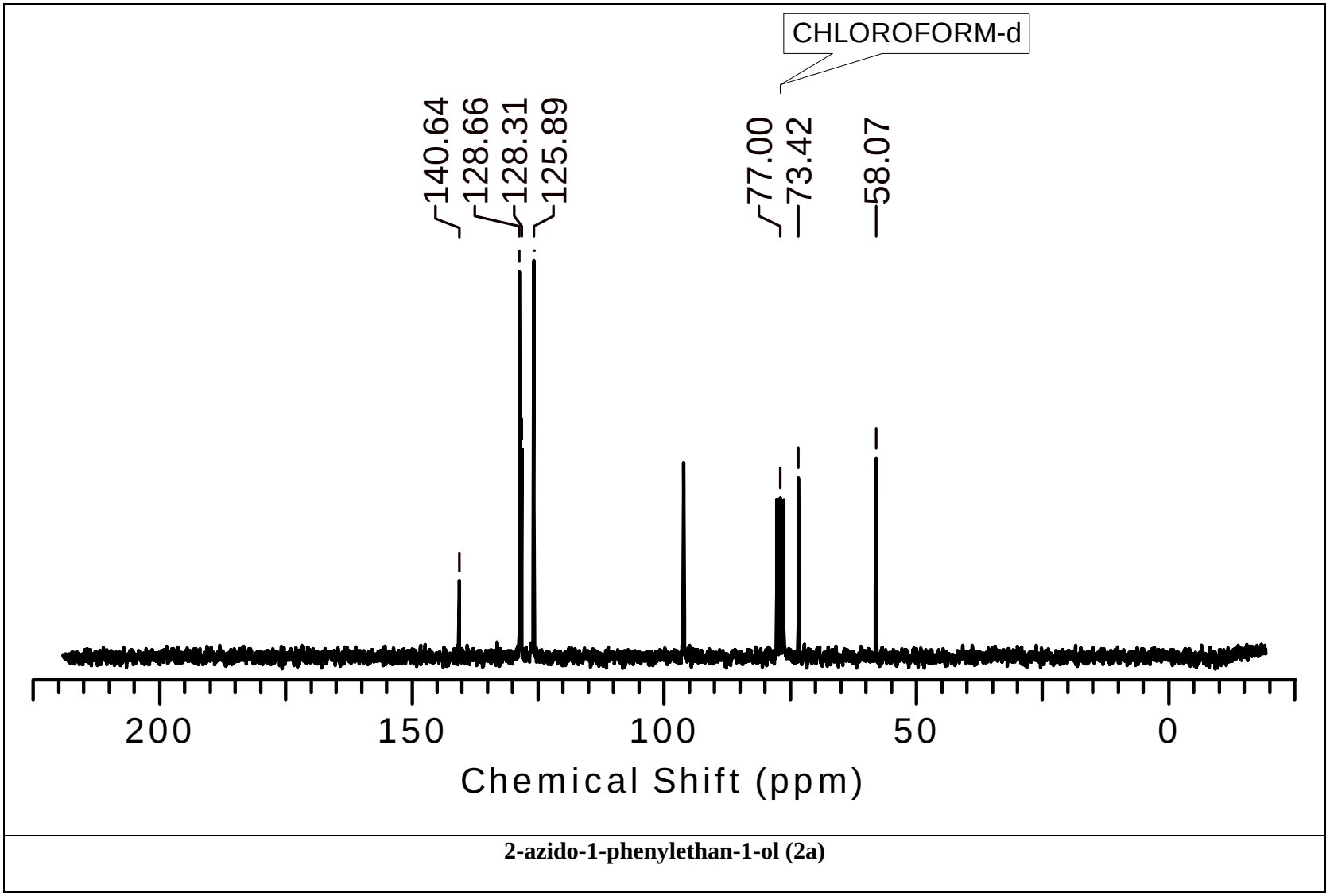
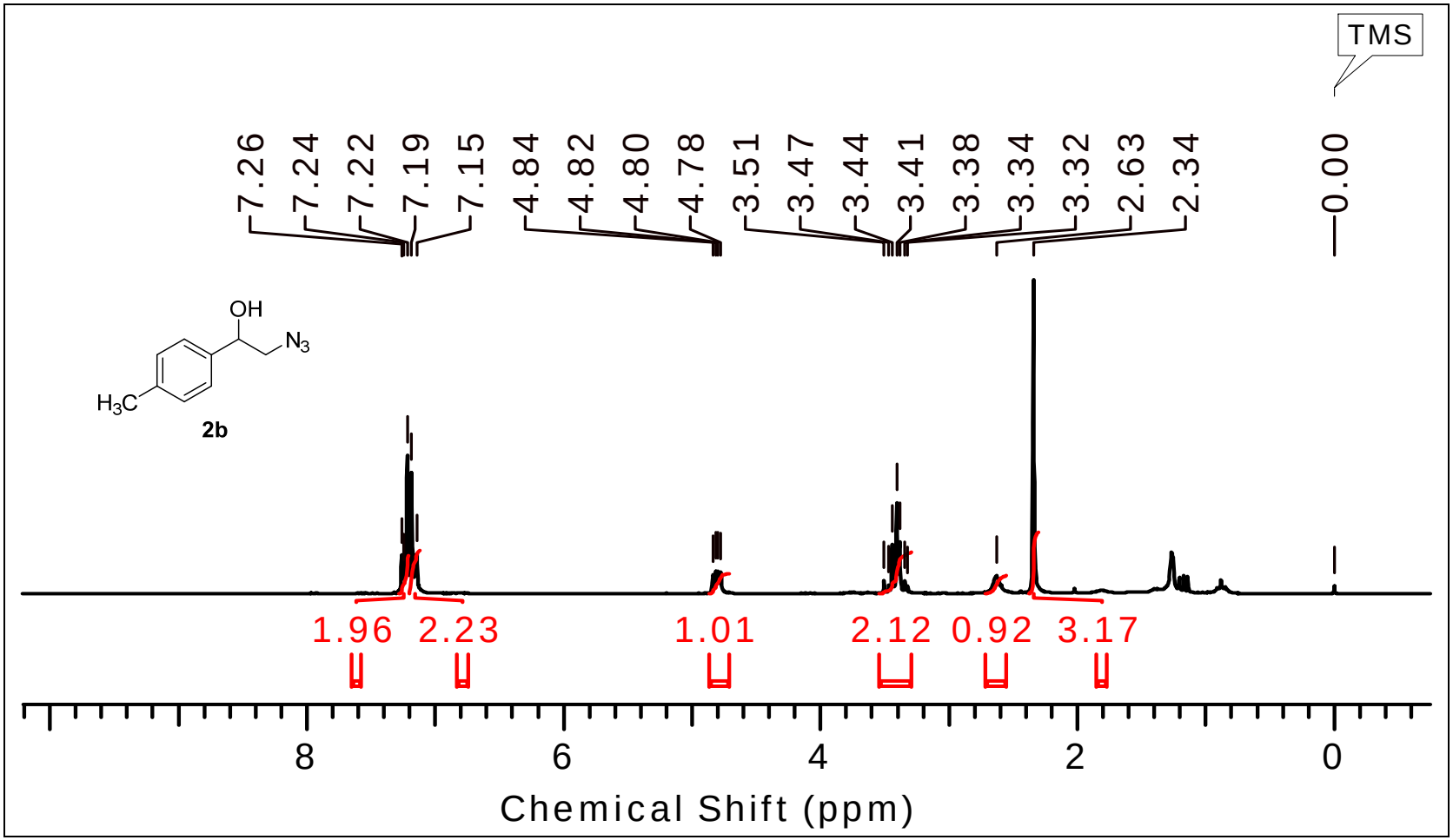


2-azido-1-phenylethan-1-ol (2a)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3.34
3.32
2.63
2.34
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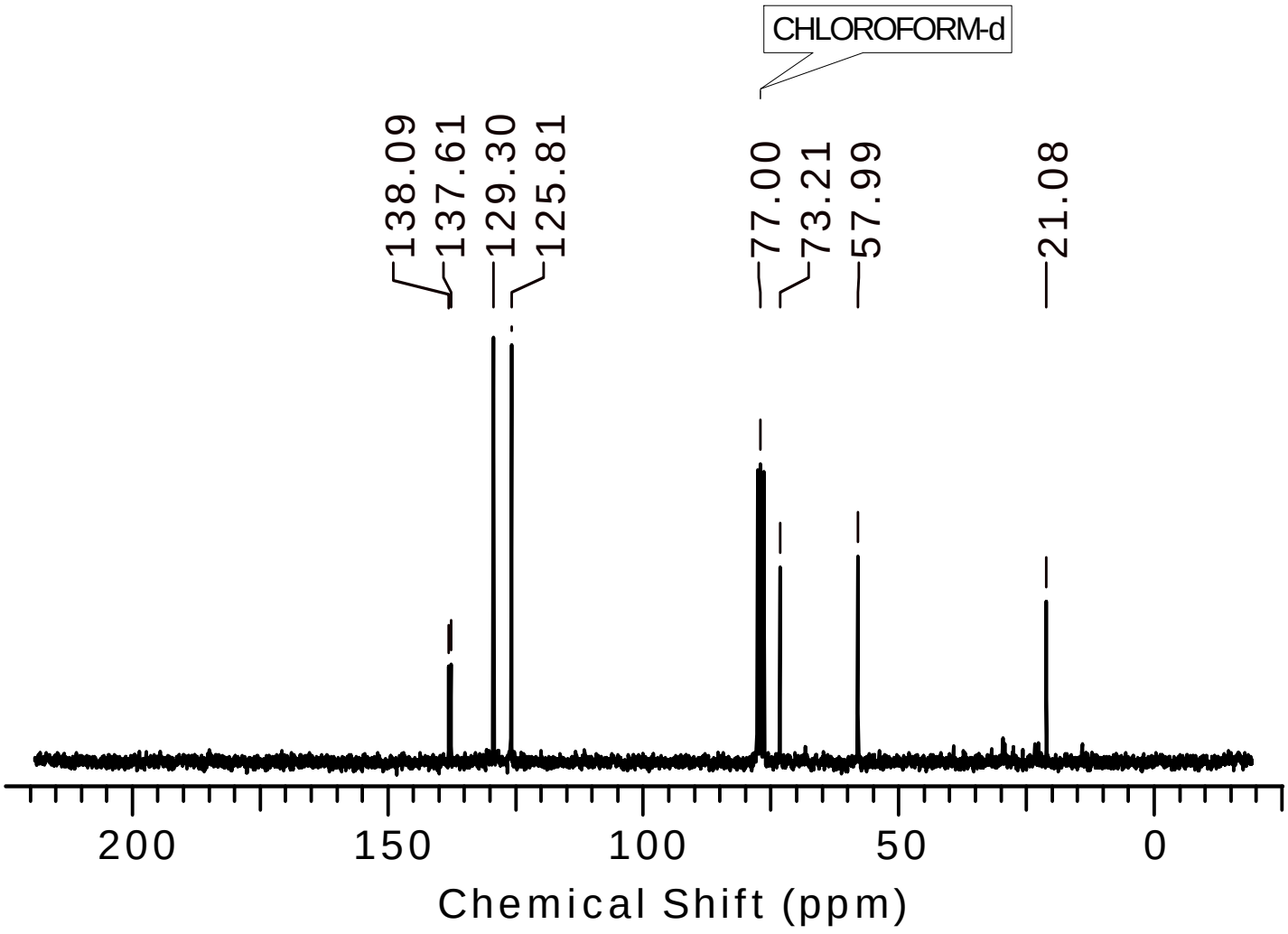
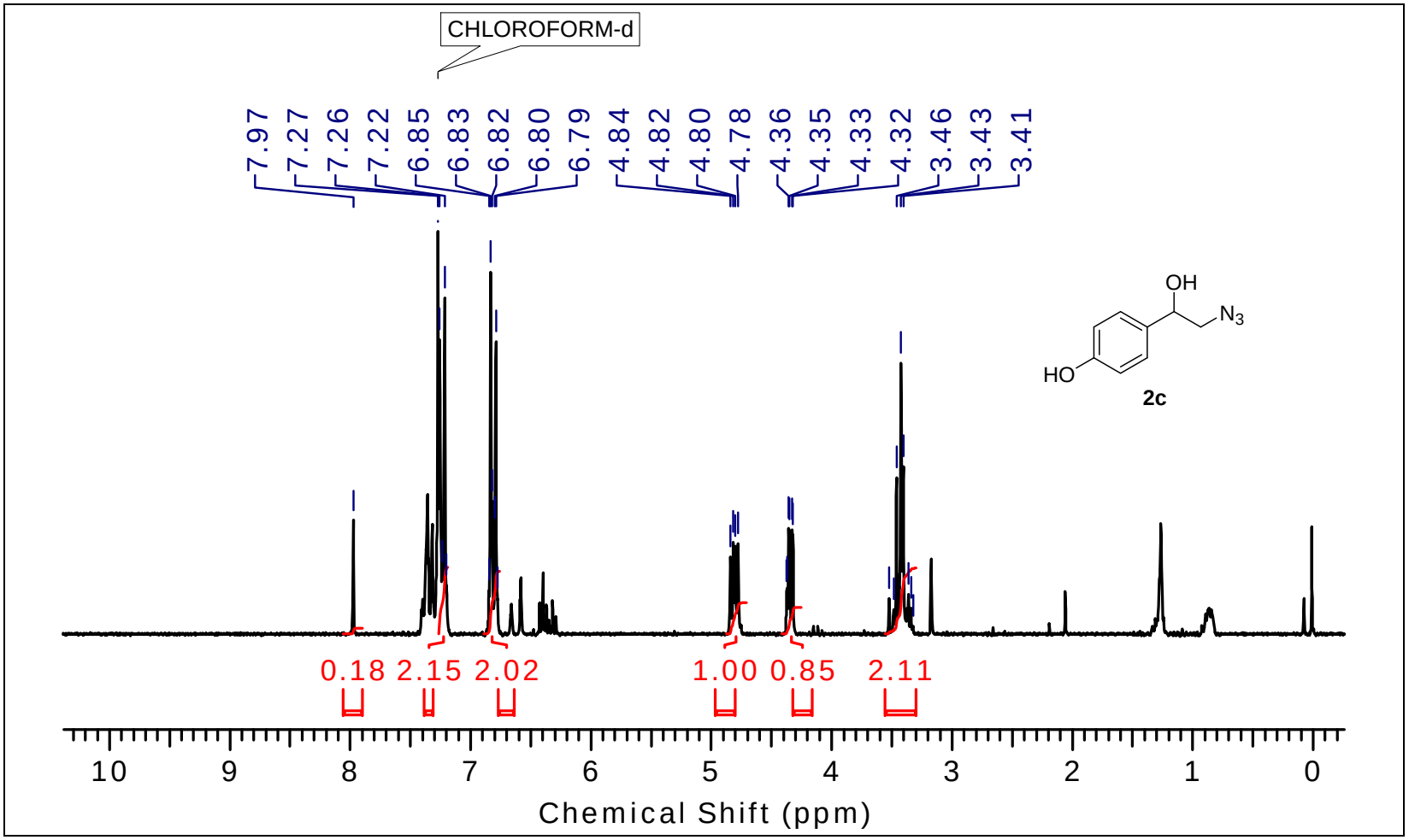
Chemical Shift (ppm)



2-azido-1-(p-tolyl)ethan-1-ol (2b)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4.33
4.32
3.46
3.43
3.41
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N3
HO
2c
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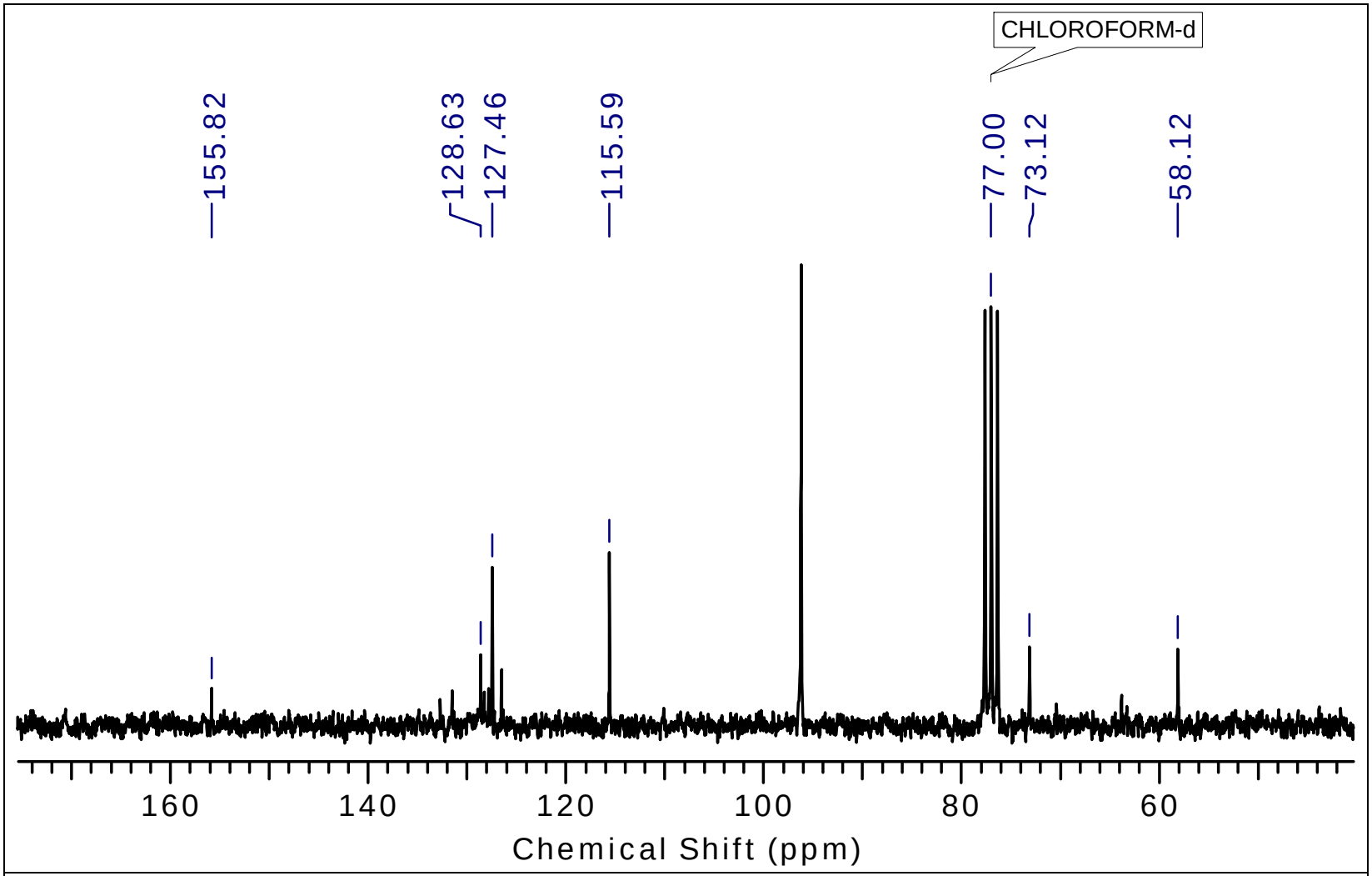
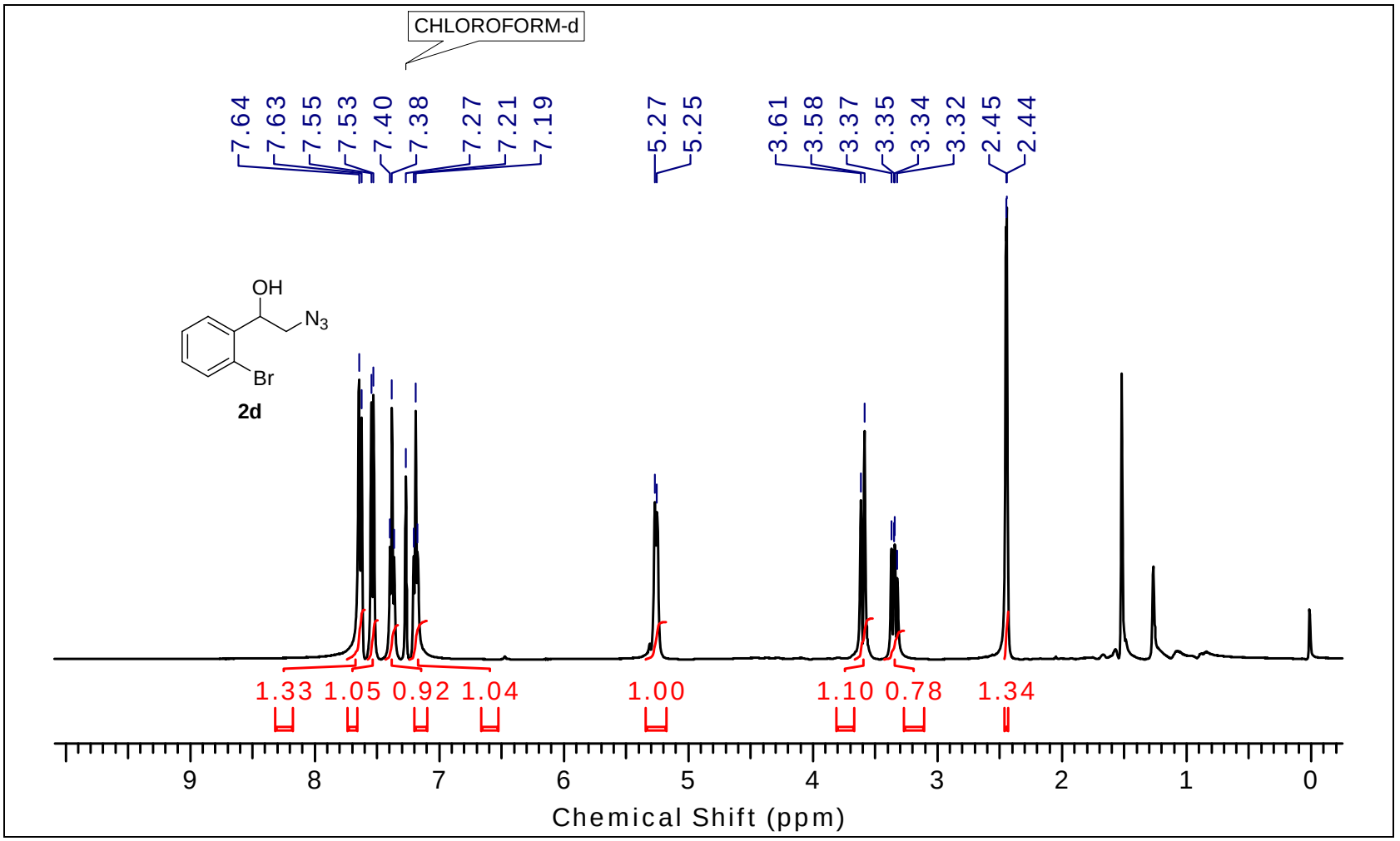
Chemical Shift (ppm)



4-(2-azido-1-hydroxyethyl)phenol (2c)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3.61
3.58
3.37
3.35
3.34
3.32
2.45
2.44
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N3
Br
2d
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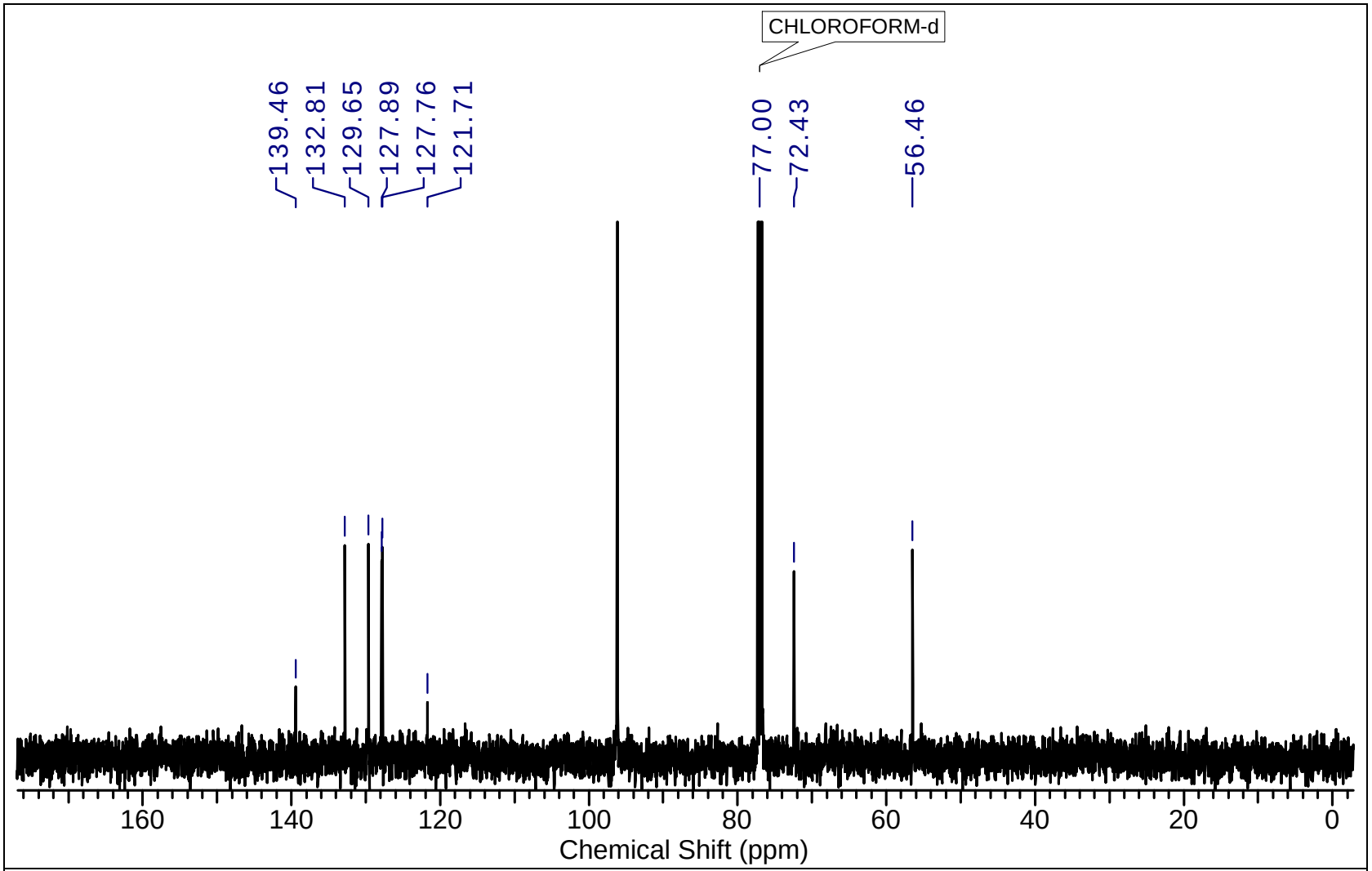
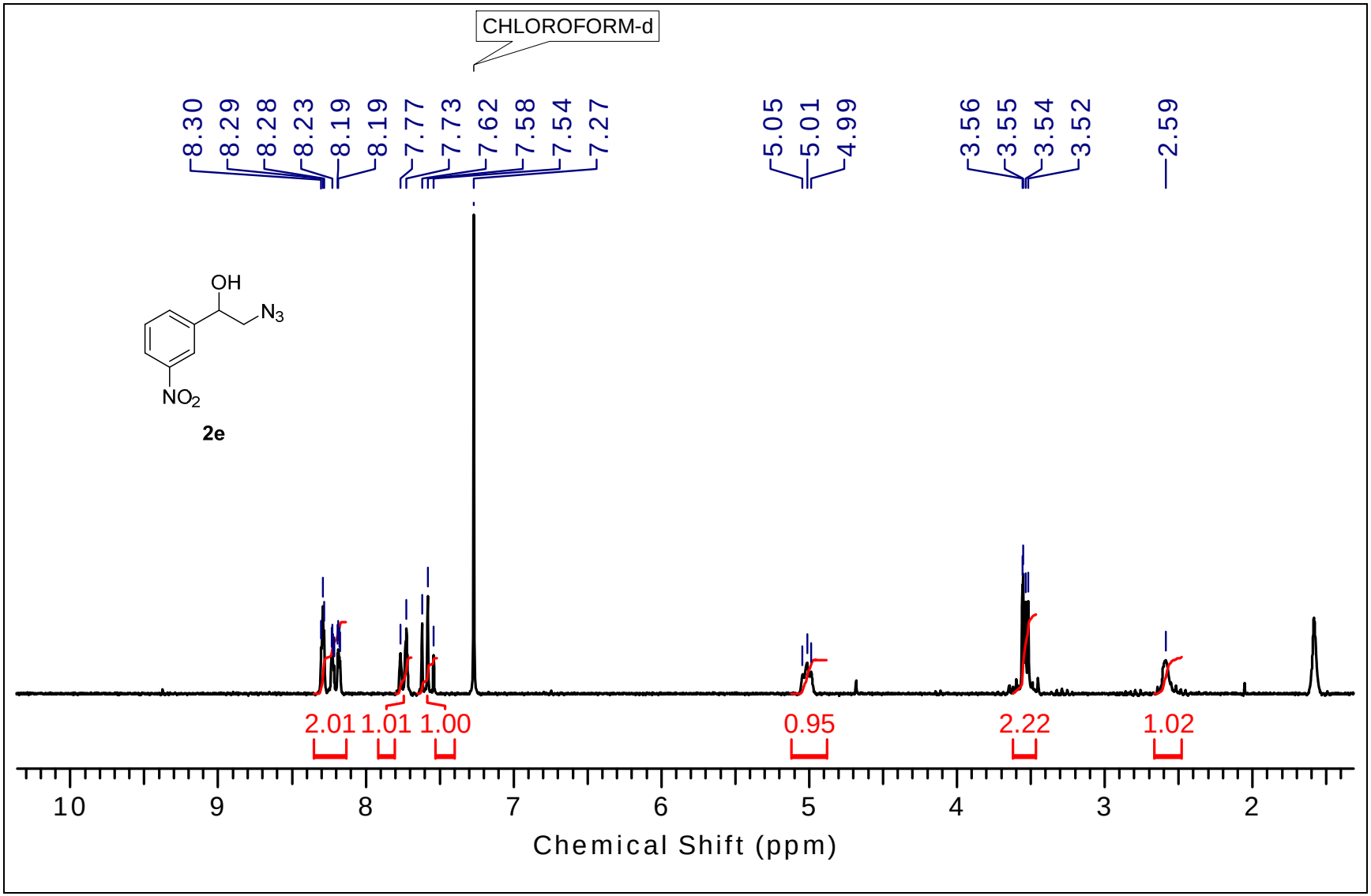
Chemical Shift (ppm)



2-azido-1-(2-bromophenyl)ethan-1-ol (2d)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3.54
3.52
2.59
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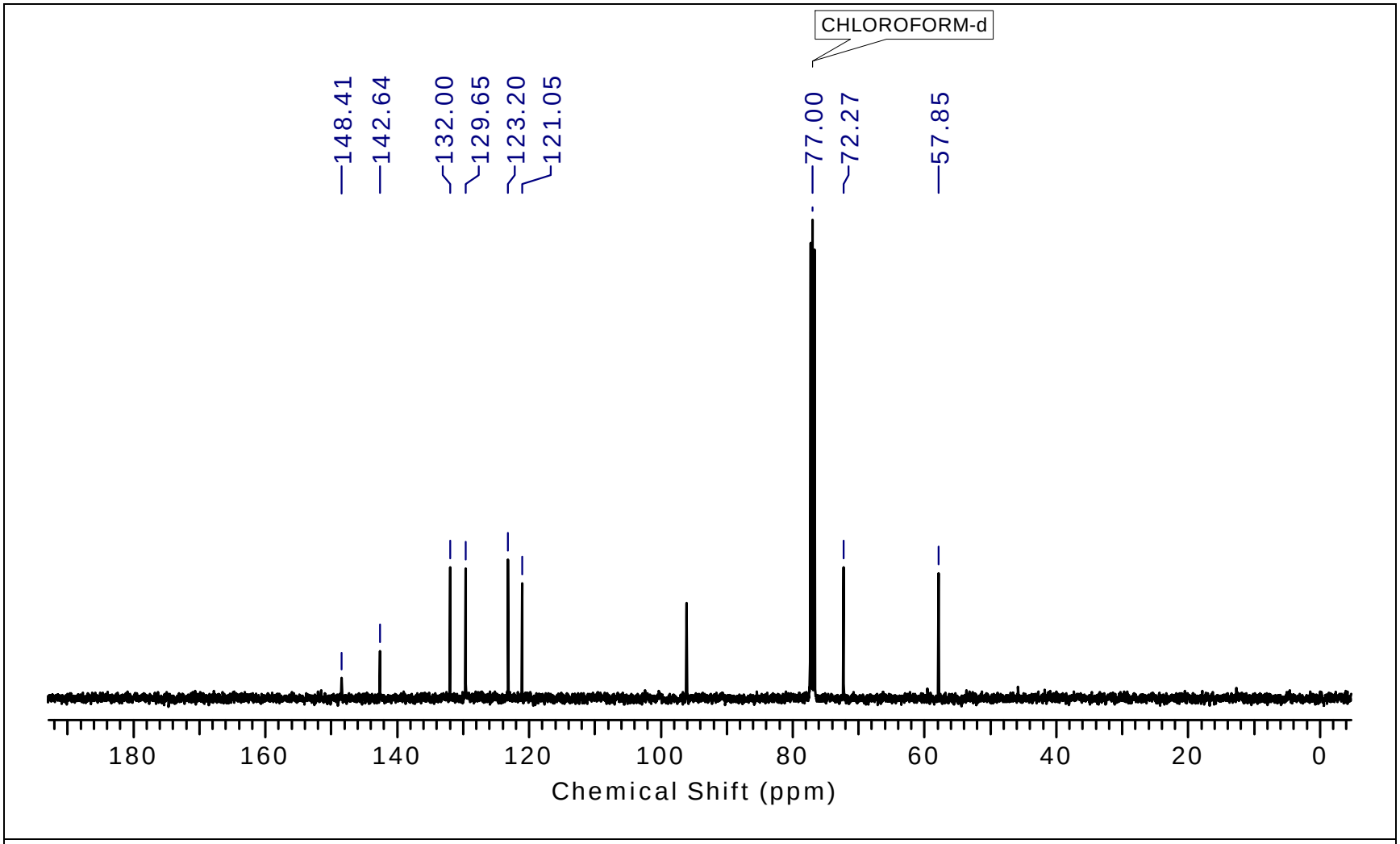
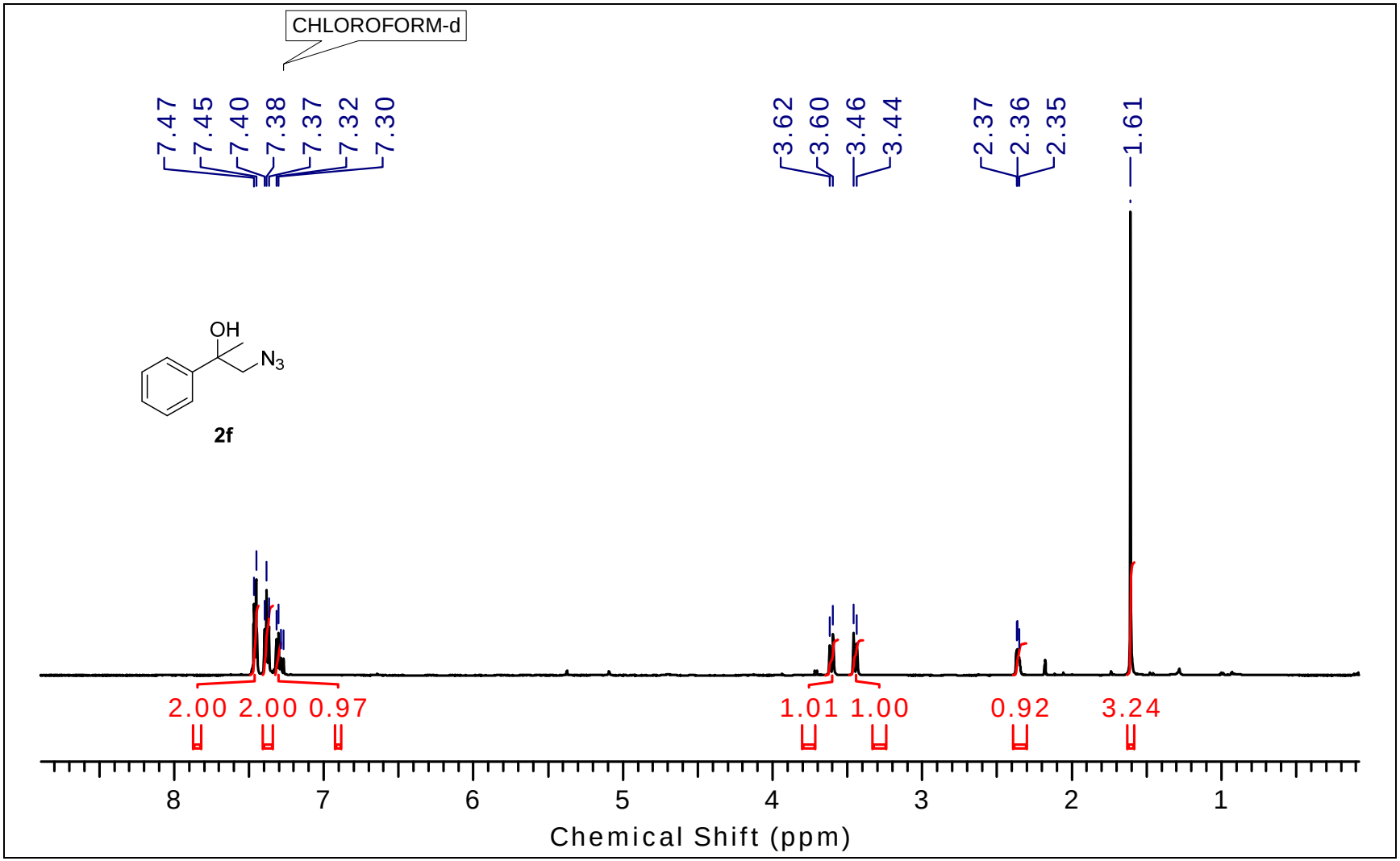


2-azido-1-(3-nitrophenyl)ethan-1-ol (2e)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2.35
1.61
OH
N3
2f
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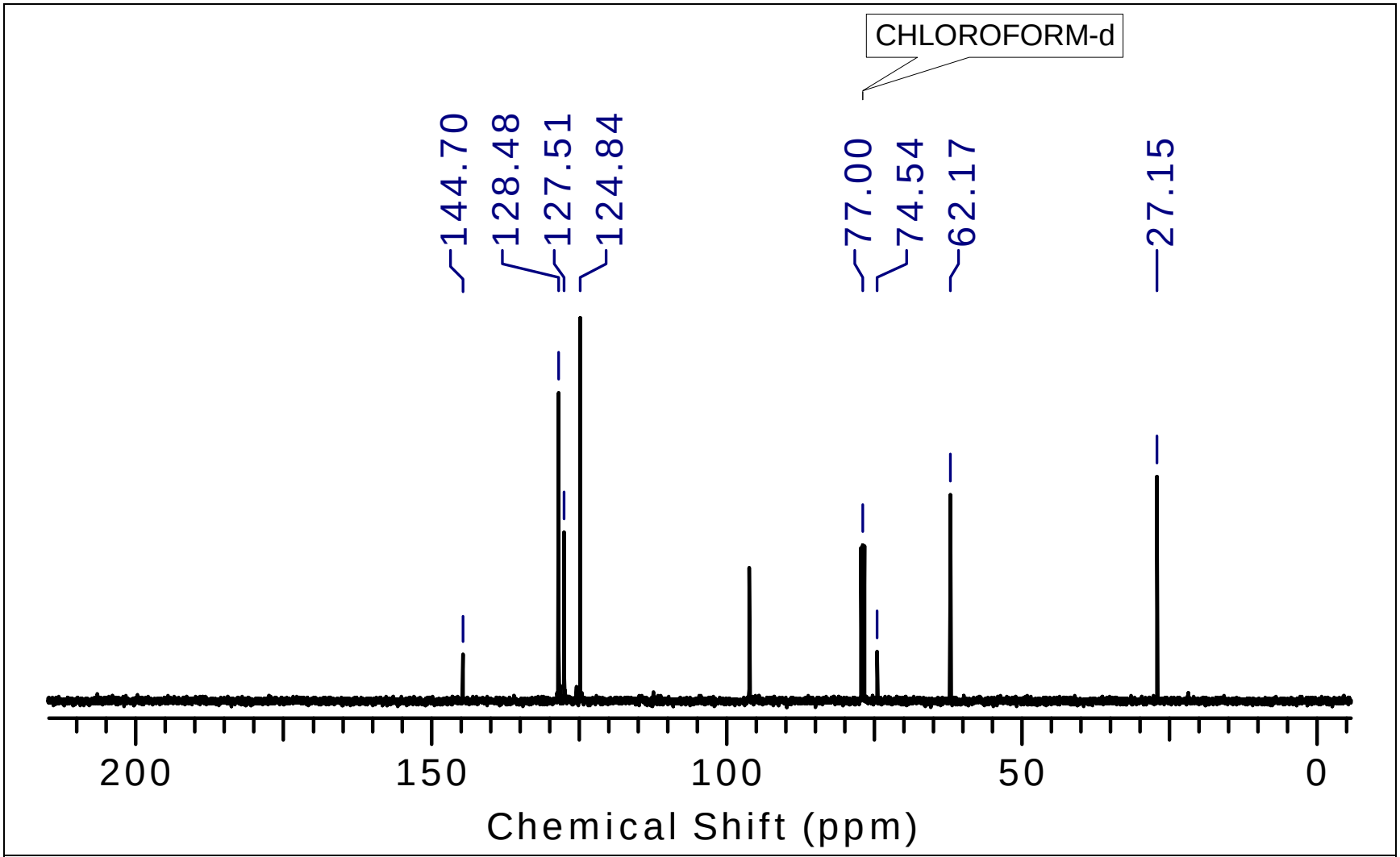
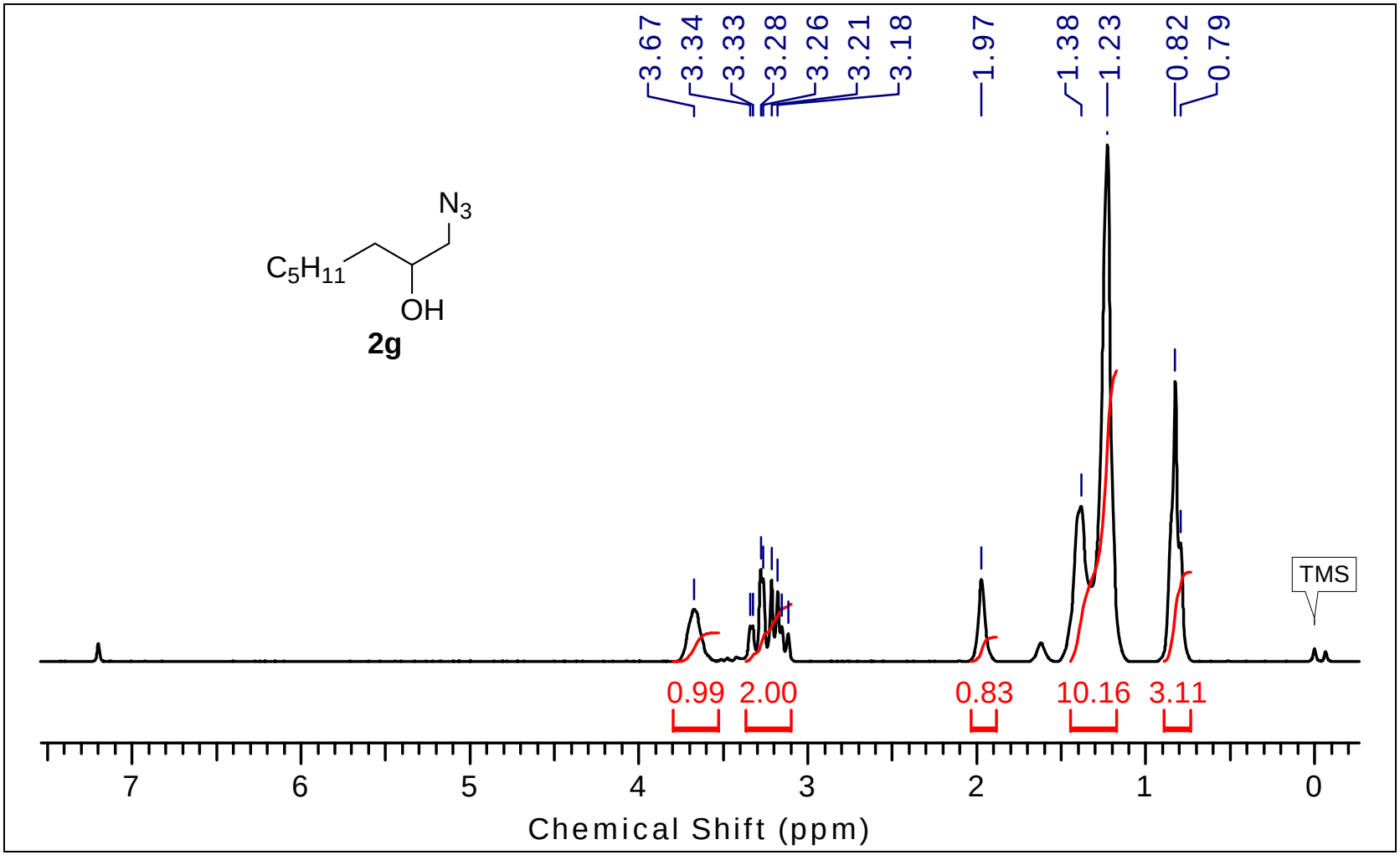


1-azido-2-phenylpropan-2-ol (2f)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OH
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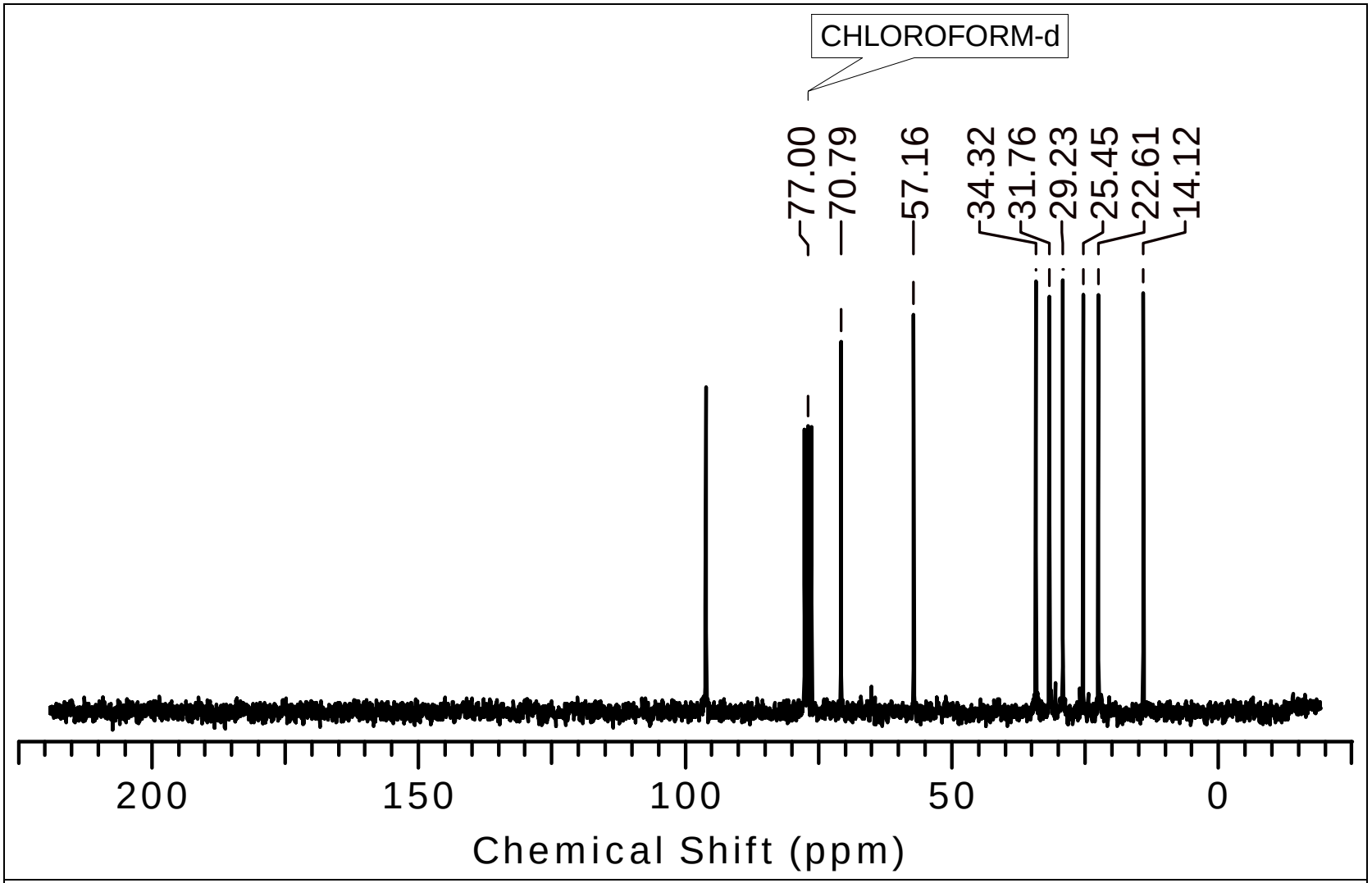
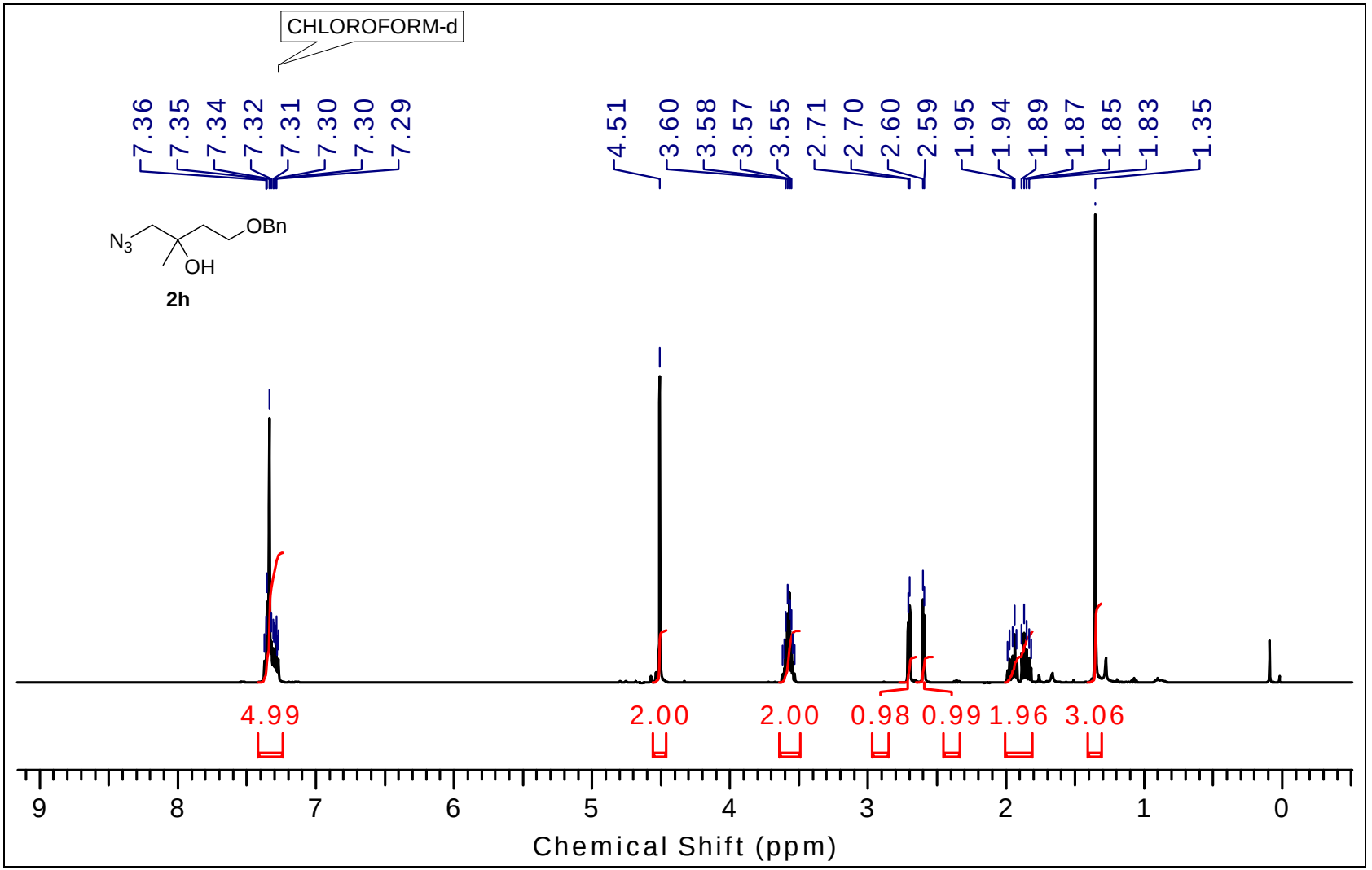


1-azidooctan-2-ol (2g)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1.89
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2h
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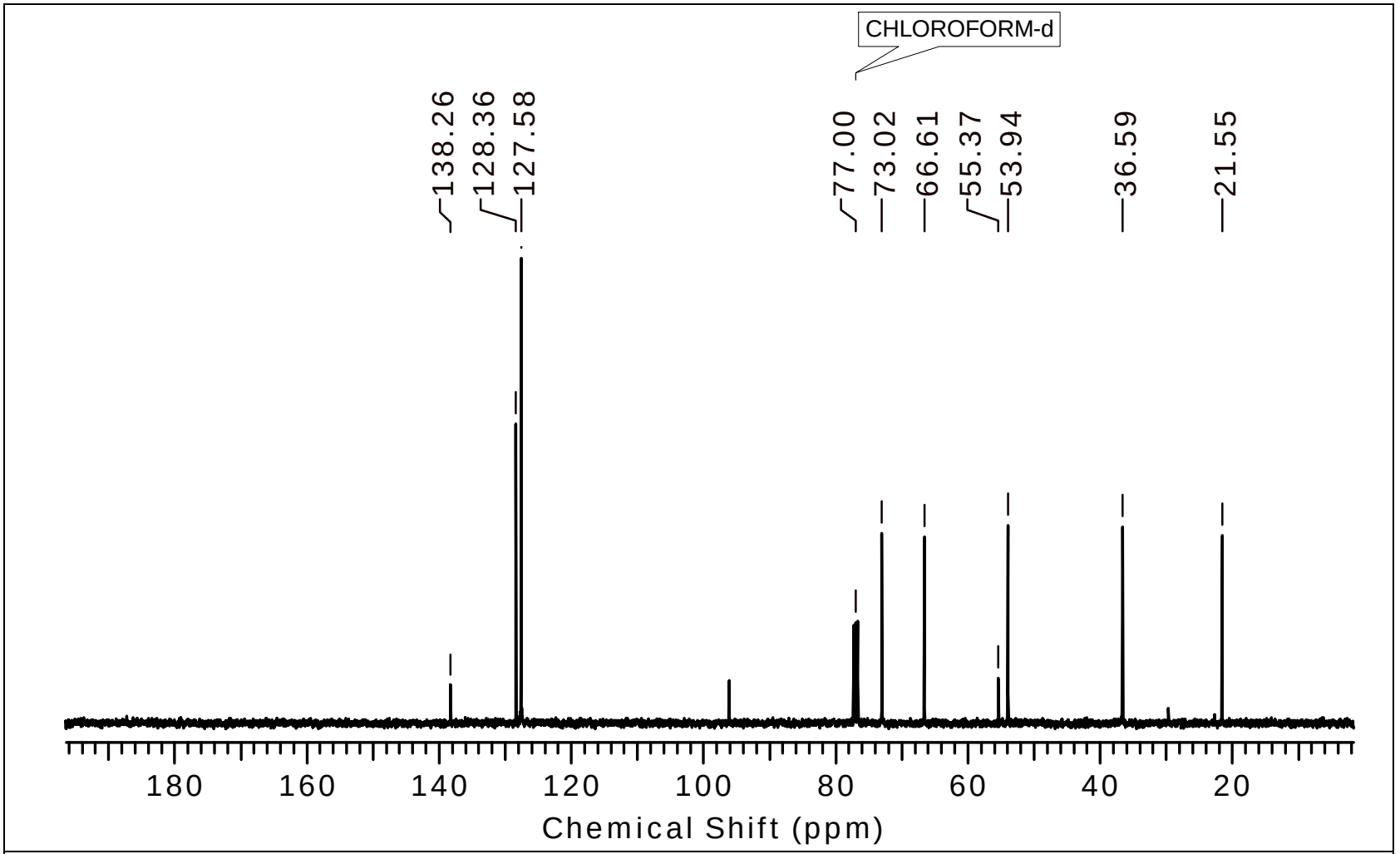
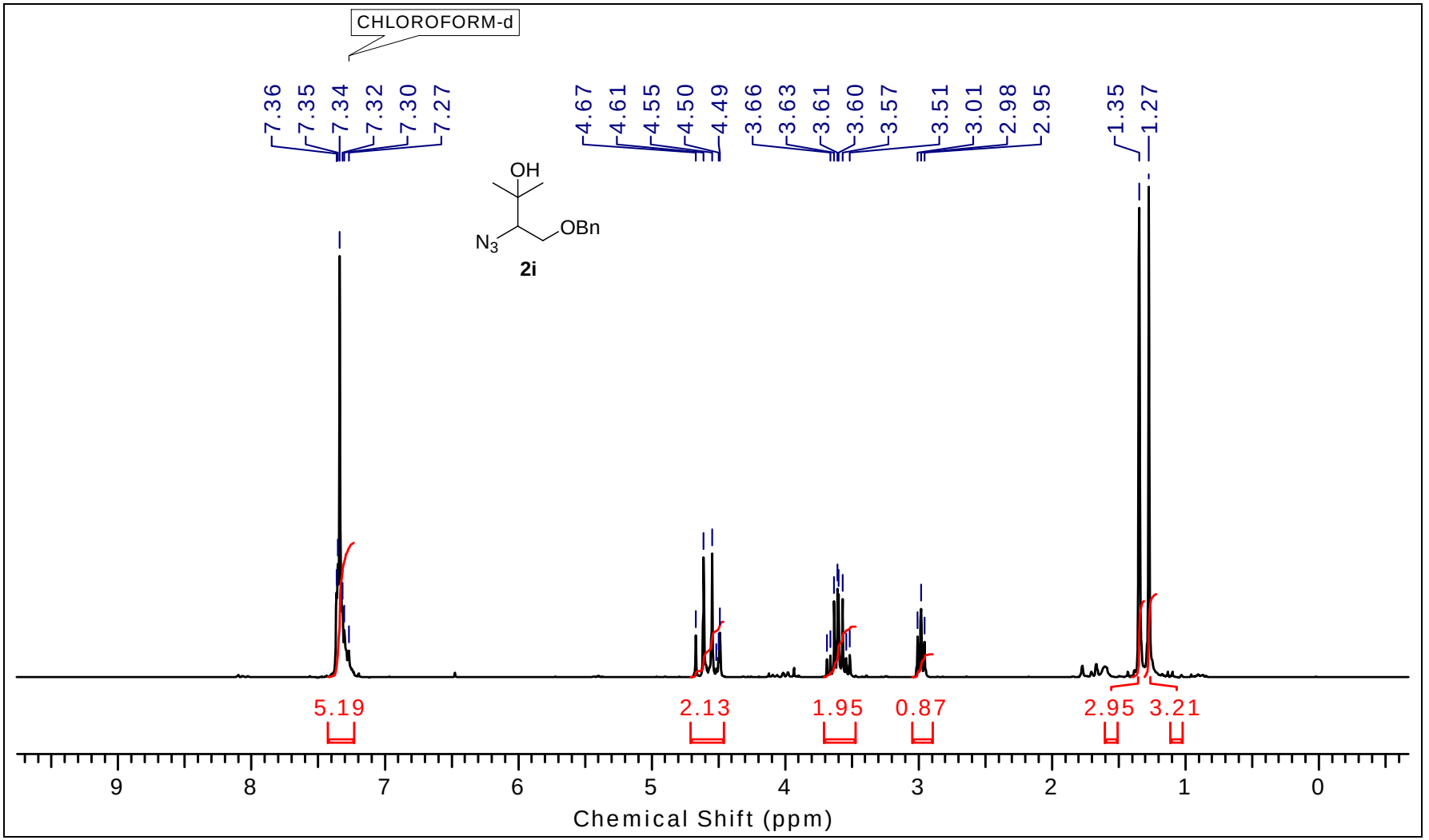


1-azido-4-(benzyloxy)-2-methylbutan-2-ol (2h)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1.27
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OBn
N3
2i
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2.13
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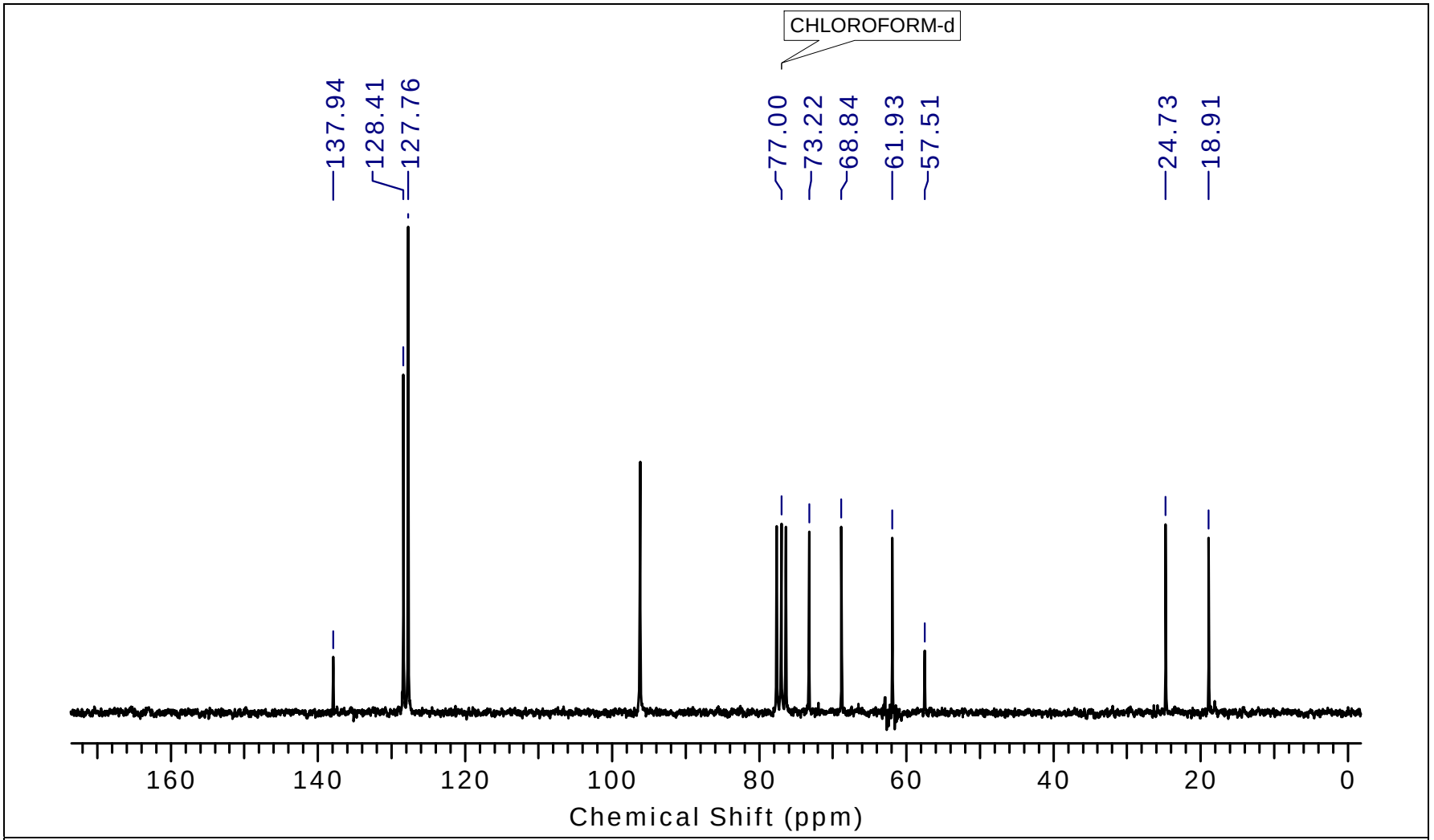
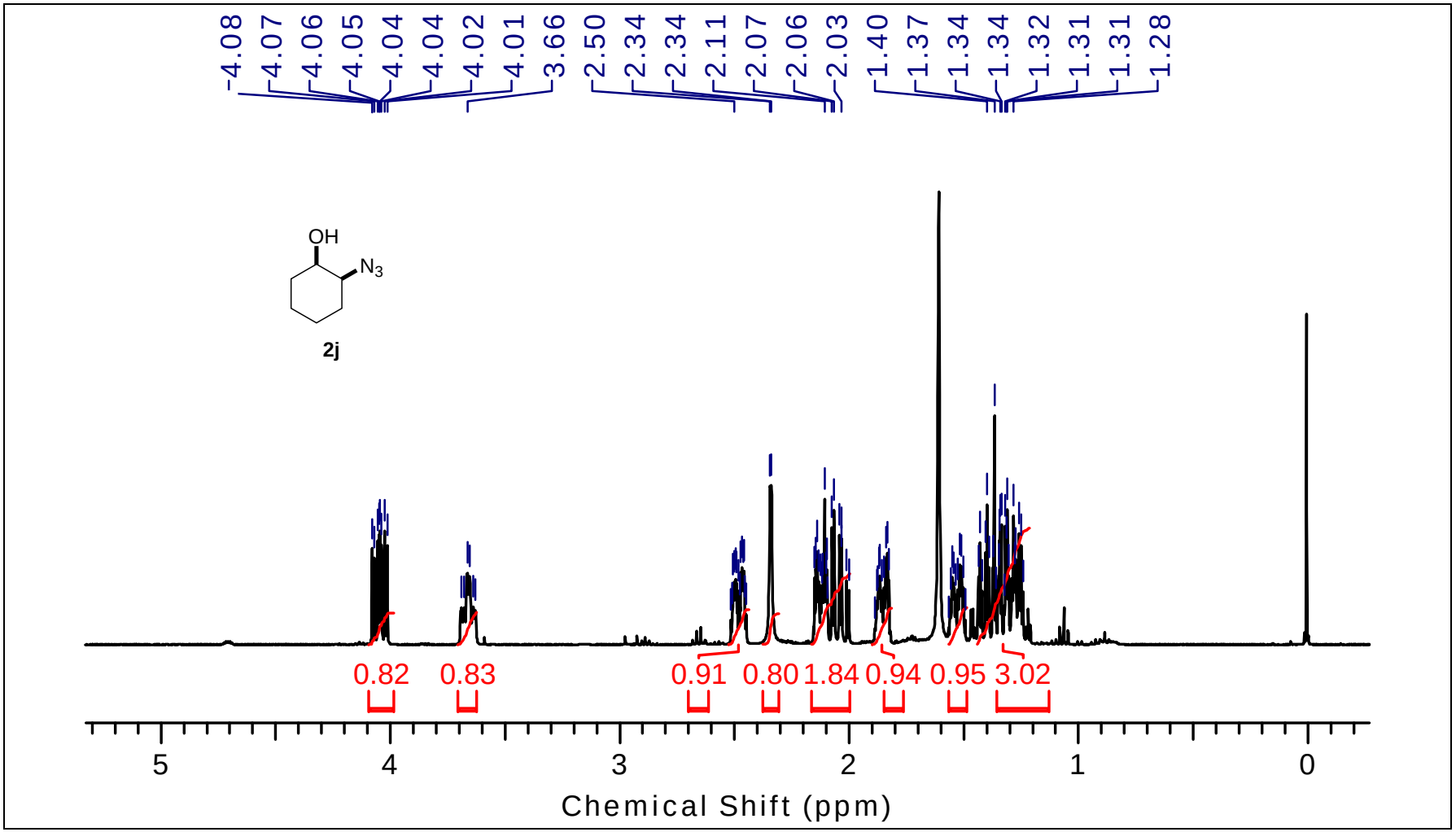
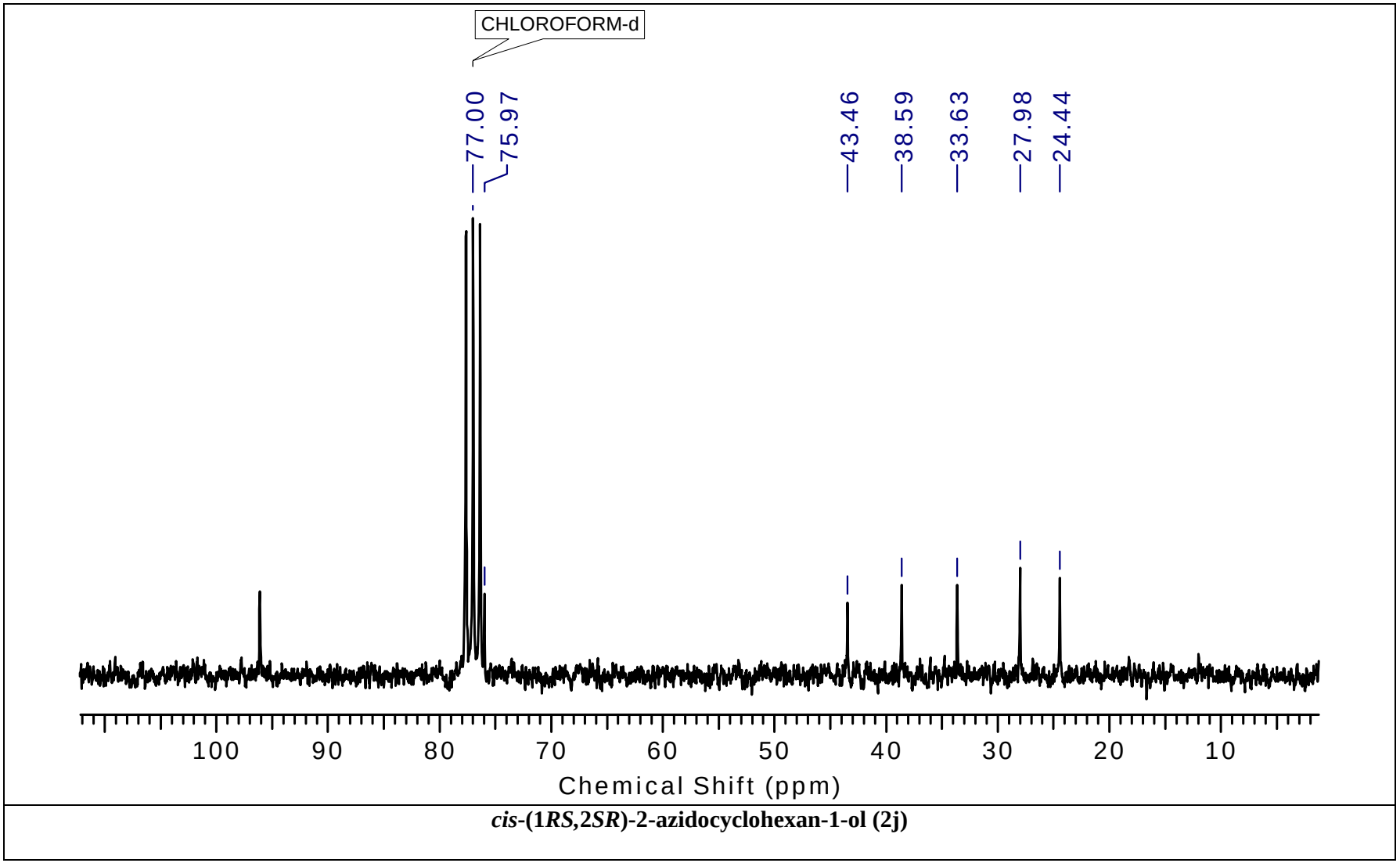
3.21
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

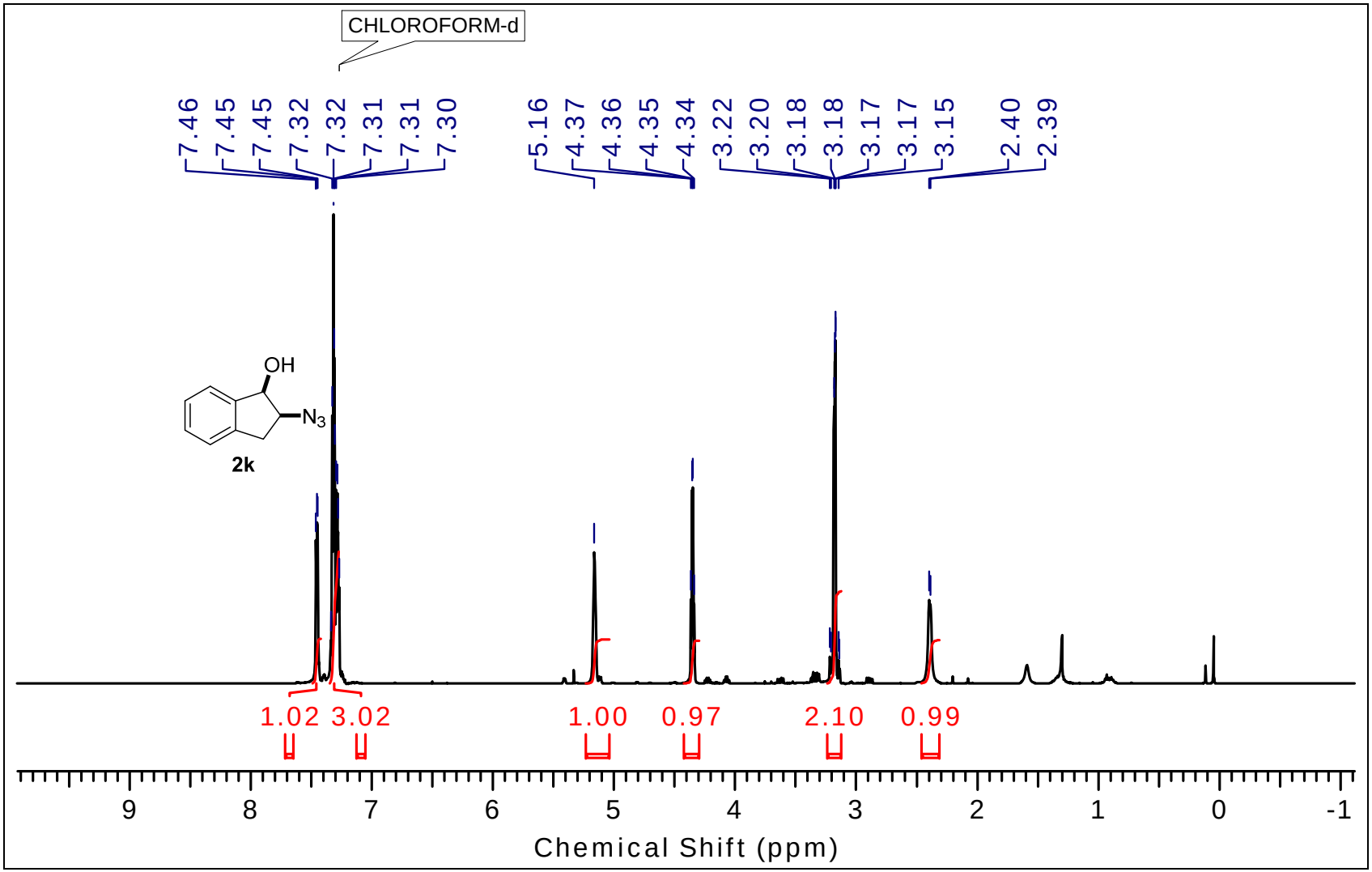


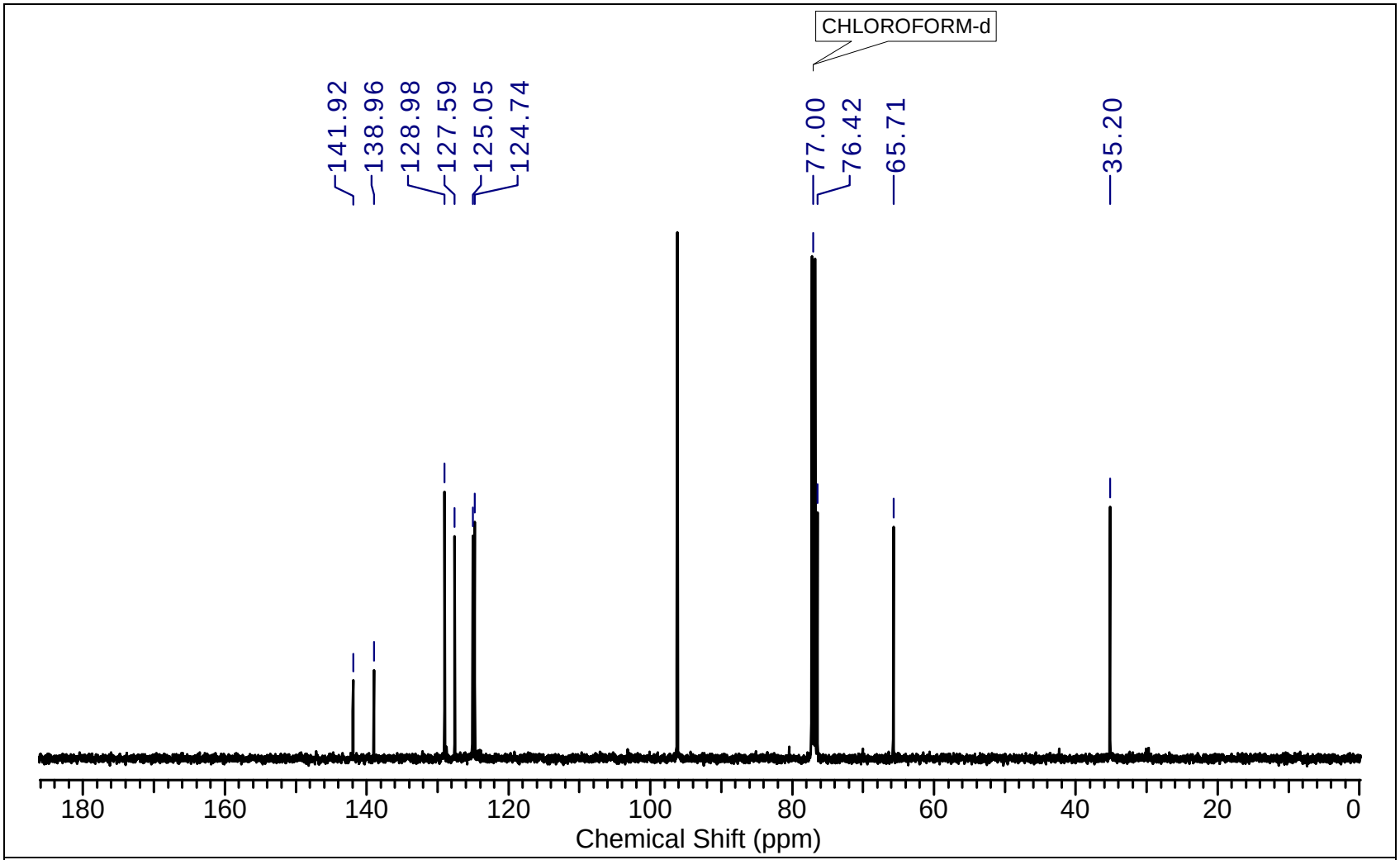
3-azido-4-(benzyloxy)-2-methylbutan-2-ol (2i)


4.08
4.07
4.06
4.05
4.04
4.04
4.02
4.01
3.66
2.50
2.34
2.34
2.11
2.07
2.06
2.03
1.40
1.37
1.34
1.34
1.32
1.31
1.31
1.28
OH
N3
2j
0.82
0.83
0.91
0.80
1.84
0.94
0.95
3.02
5
4
3
2
1
0
Chemical Shift (ppm)

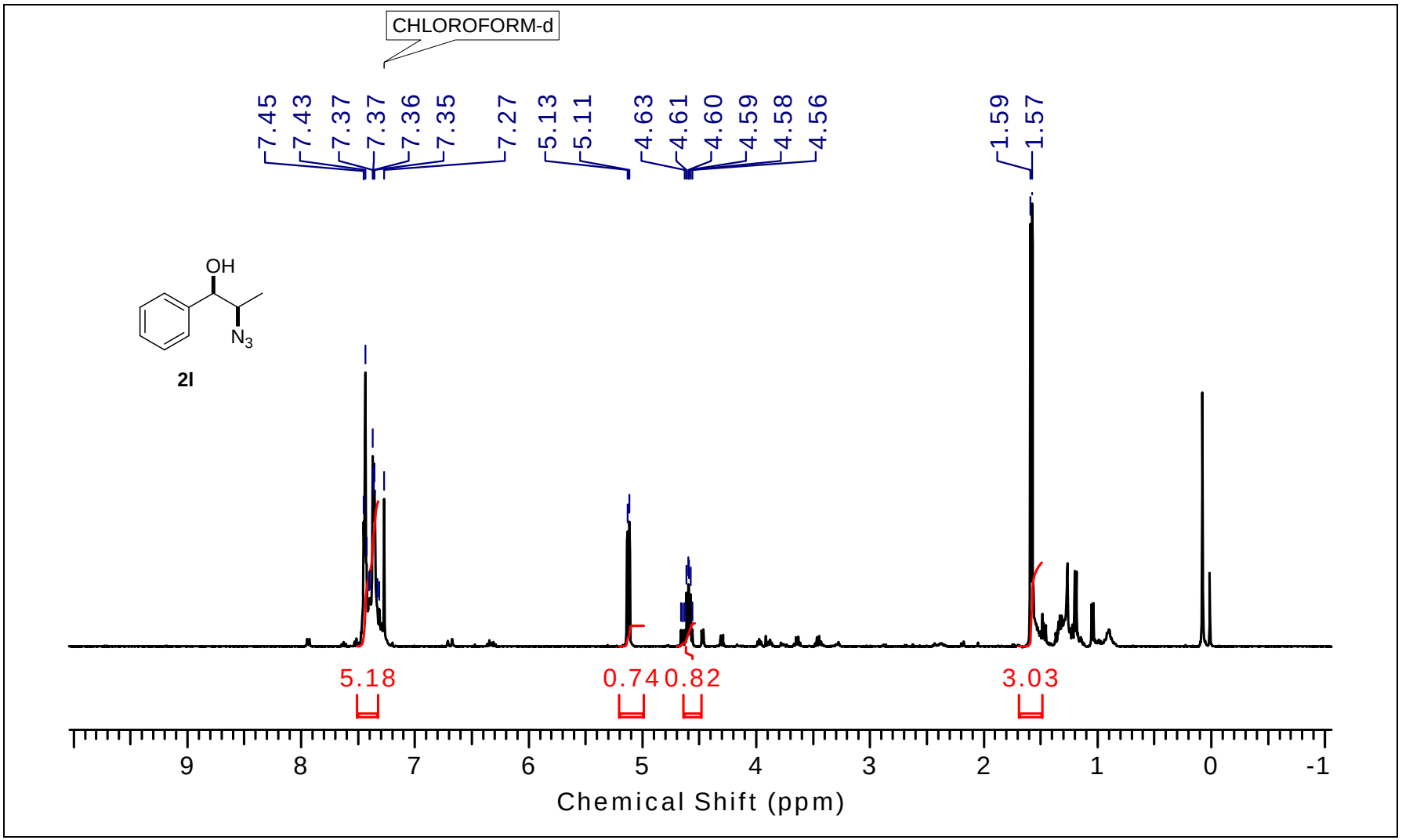


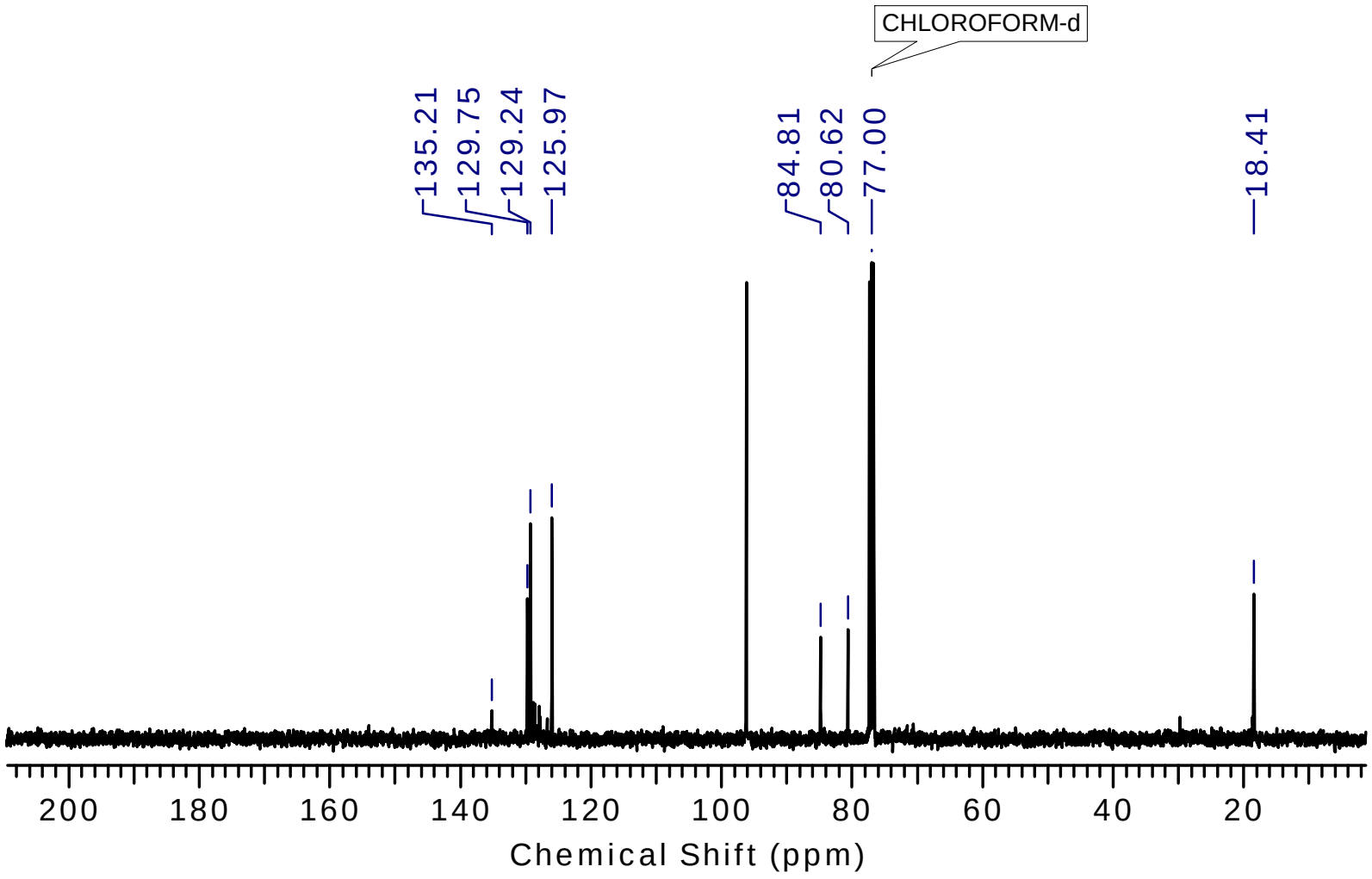
***cis*-(1*RS*,2*SR*)-2-azidocyclohexan-1-ol (2j)**


CHLOROFORM-d
7.46
7.45
7.45
7.32
7.32
7.31
7.31
7.30
5.16
4.37
4.36
4.35
4.34
3.22
3.20
3.18
3.18
3.17
3.17
3.15
2.40
2.39
OH
N3
2k
1.02
3.02
1.00
0.97
2.10
0.99
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

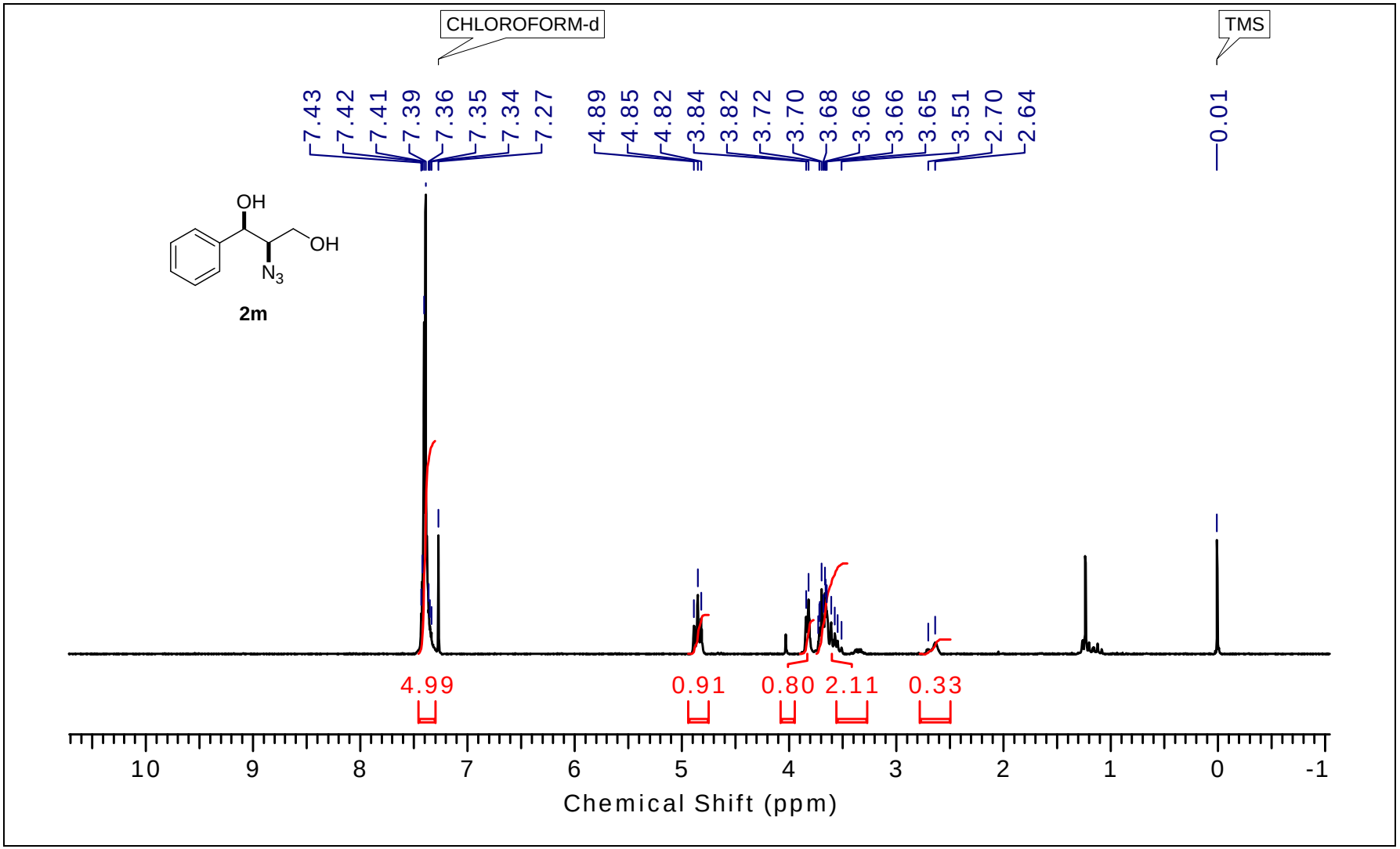


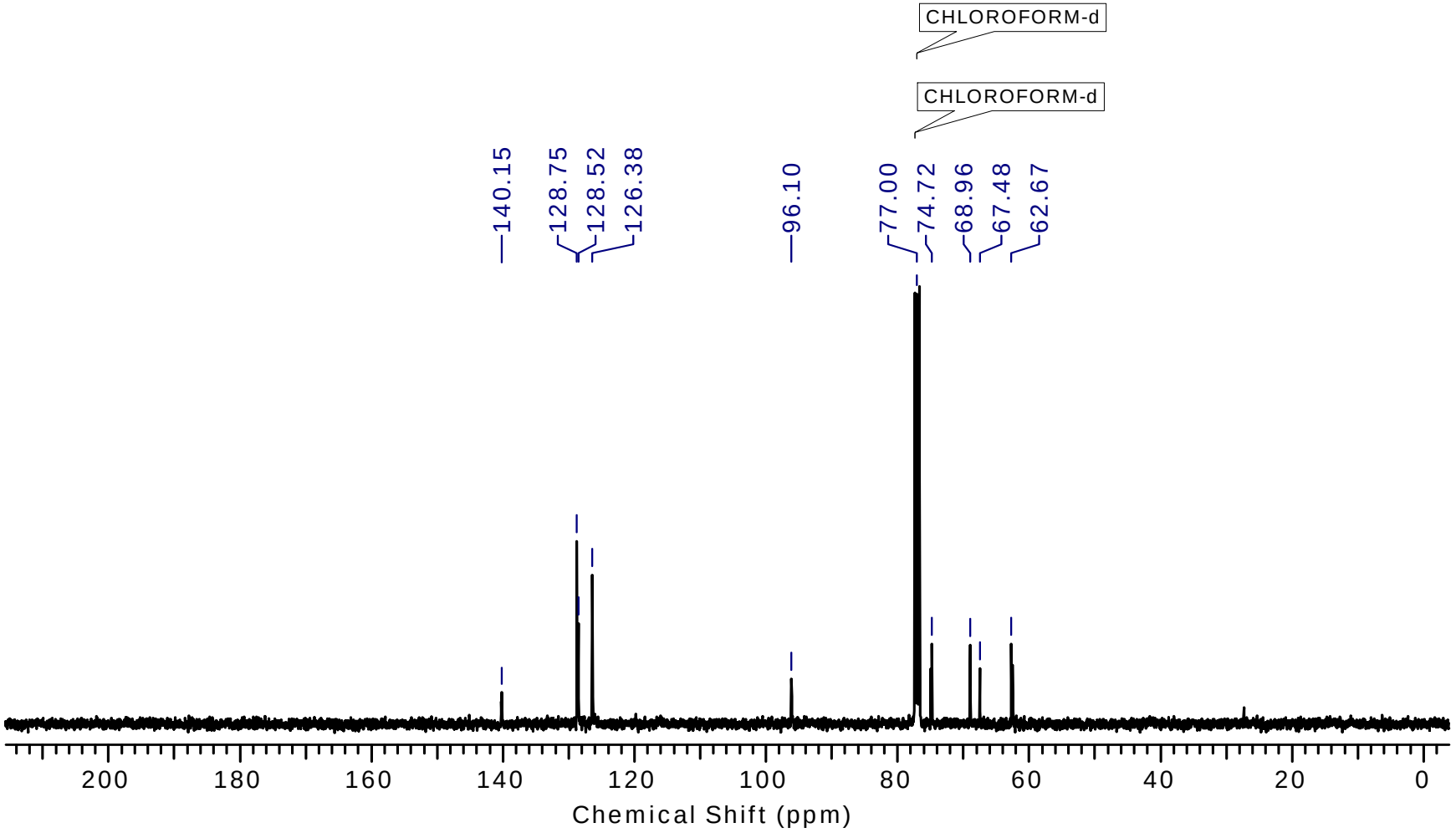
***cis*-(1*RS*,2*SR*)-2-azido-2,3-dihydro-1H-inden-1-ol (2k)**


CHLOROFORM-d
7.45
7.43
7.37
7.37
7.36
7.35
7.27
5.13
5.11
4.63
4.61
4.60
4.59
4.58
4.56
1.59
1.57
OH
N3
2l
5.18
0.74
0.82
3.03
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

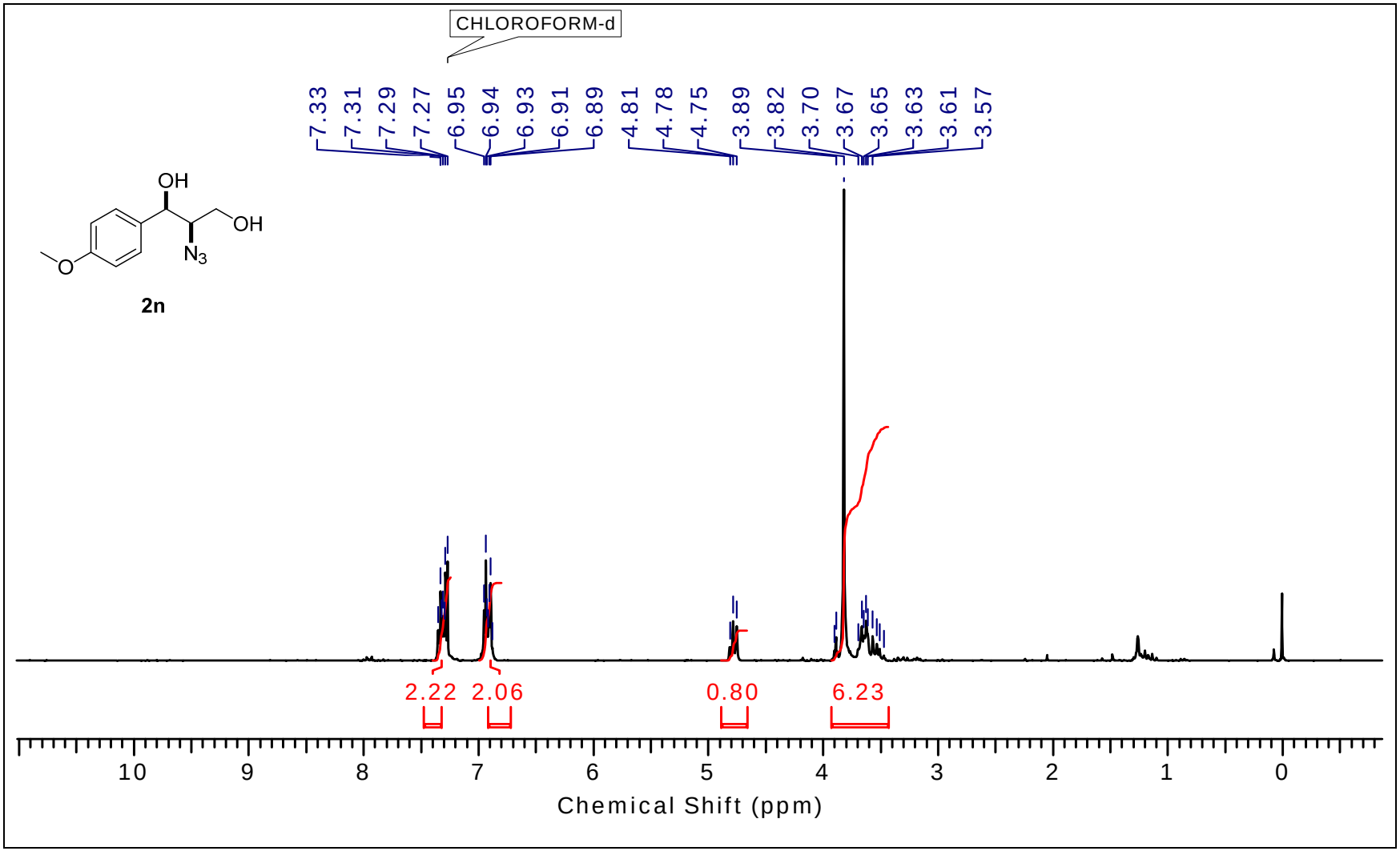


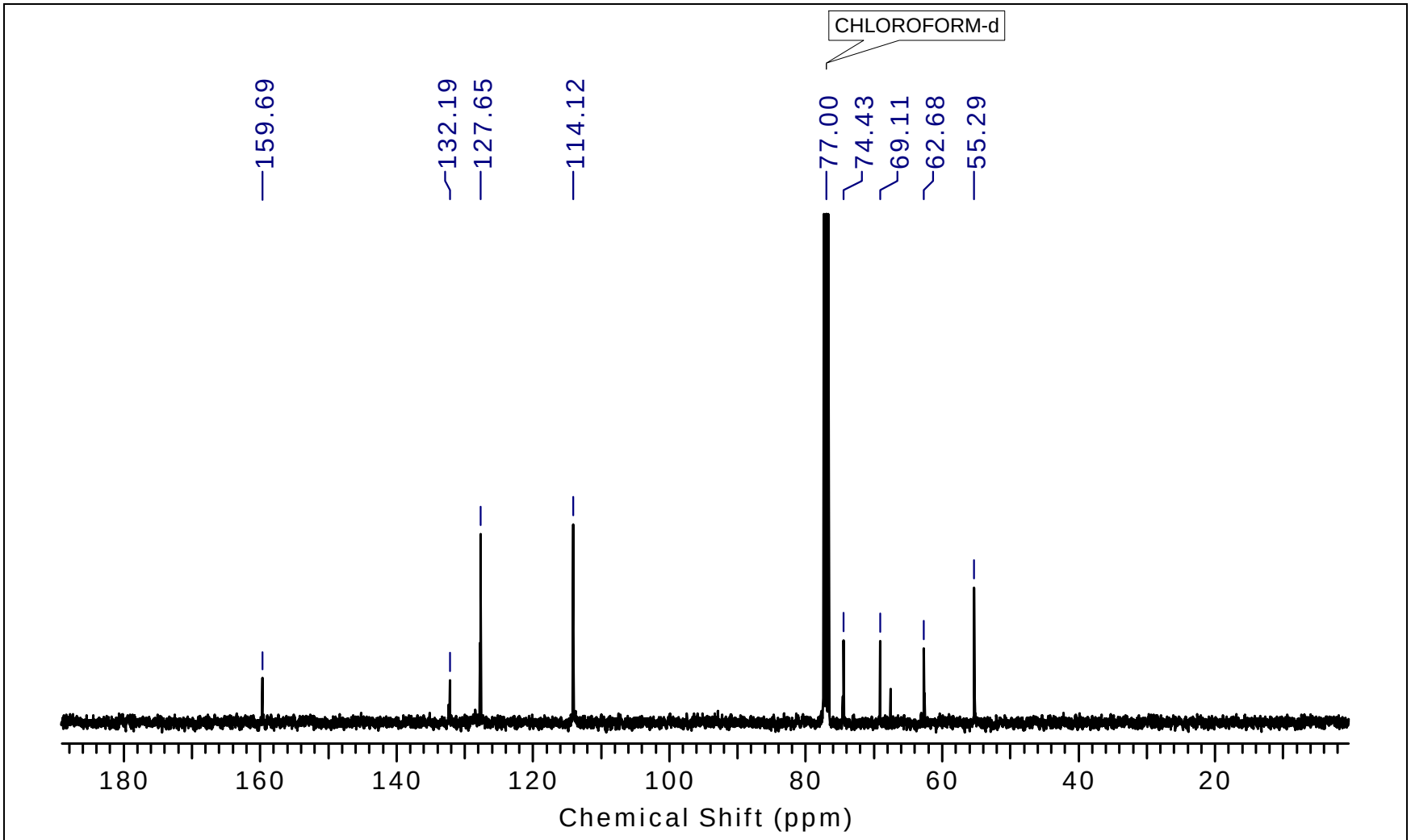
***syn*-(1*RS*,2*RS*)-2-azido-1-phenylpropan-1-ol (2l)**


CHLOROFORM-d
TMS
7.43
7.42
7.41
7.39
7.36
7.35
7.34
7.27
4.89
4.85
4.82
3.84
3.82
3.72
3.70
3.68
3.66
3.66
3.65
3.51
2.70
2.64
0.01
OH
OH
N3
2m
4.99
0.91
0.80
2.11
0.33
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

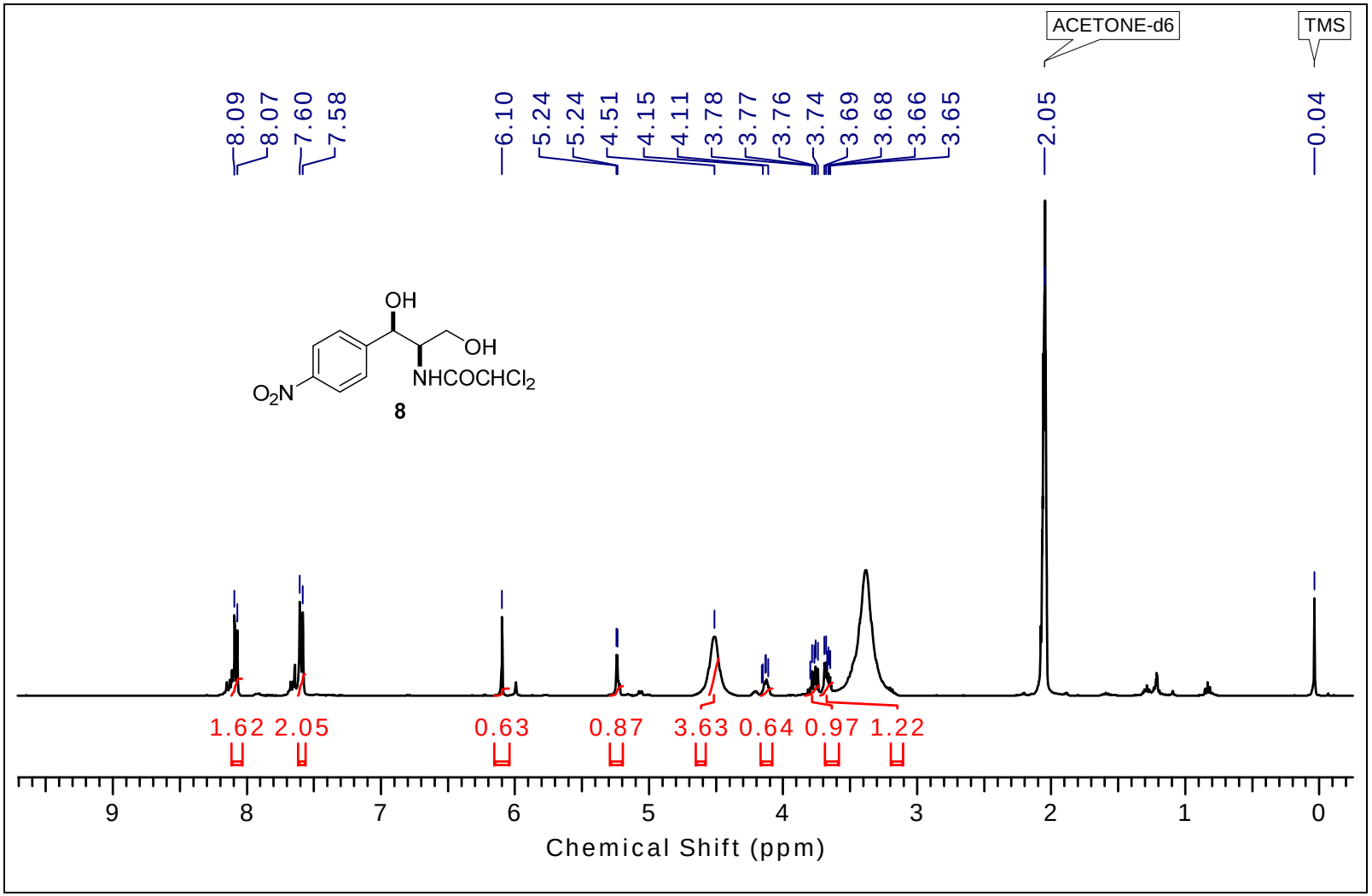


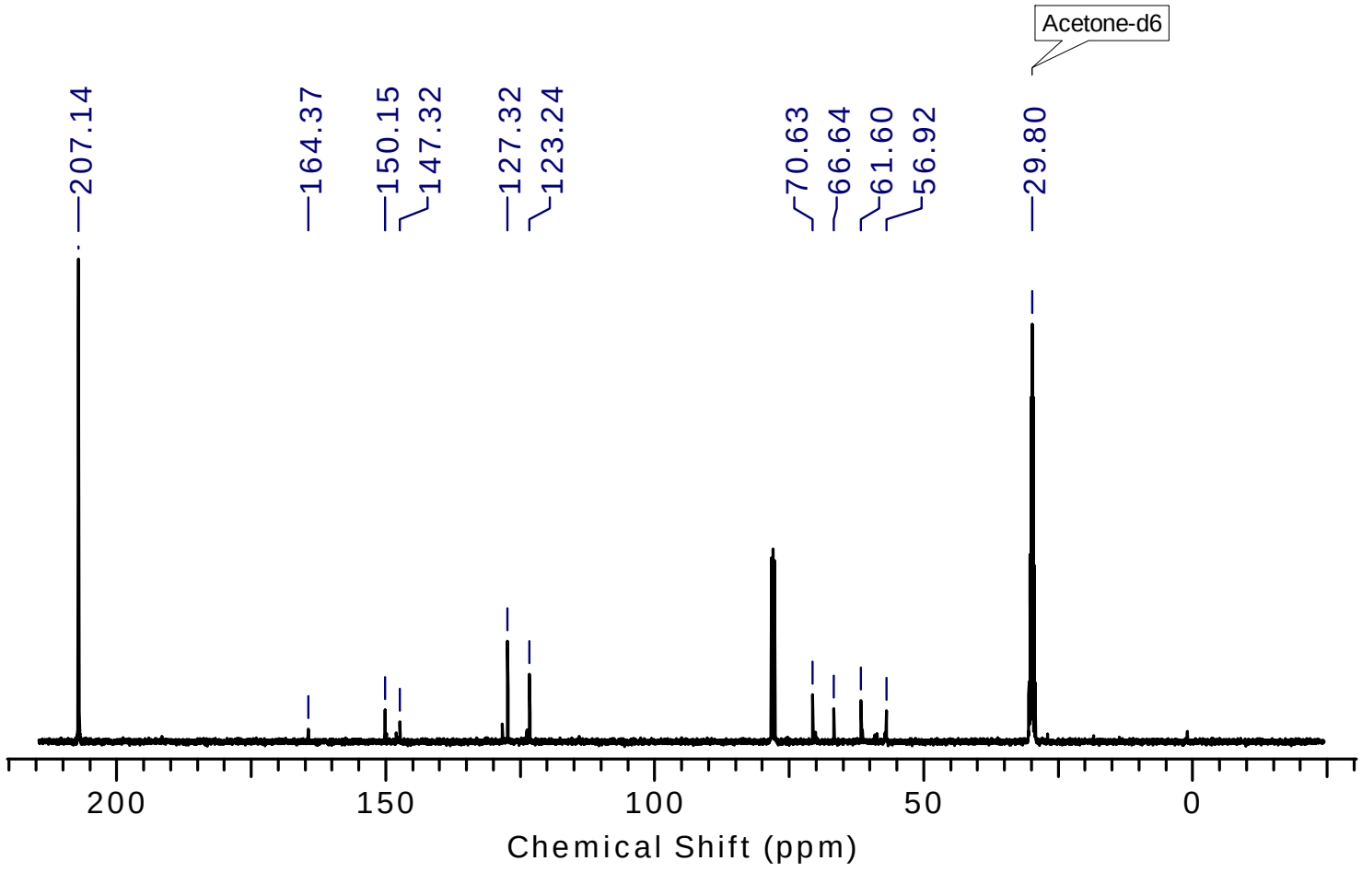
***syn*-(1*RS*,2*RS*)-2-azido-1-phenylpropane-1,3-diol (2m)**


CHLOROFORM-d
7.33
7.31
7.29
7.27
6.95
6.94
6.93
6.91
6.89
4.81
4.78
4.75
3.89
3.82
3.70
3.67
3.65
3.63
3.61
3.57
OH
OH
N3
O
2n
2.22
2.06
0.80
6.23
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

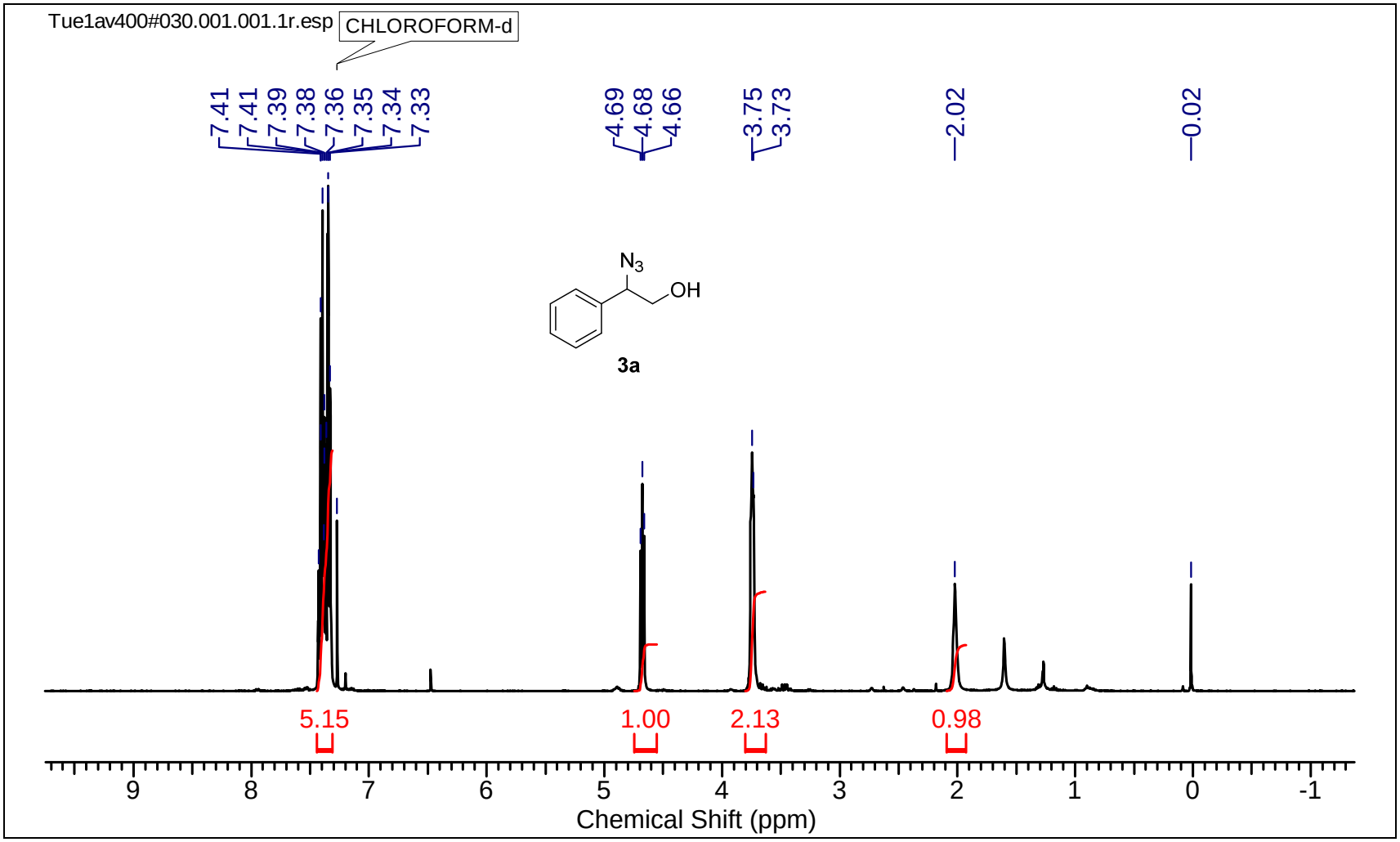


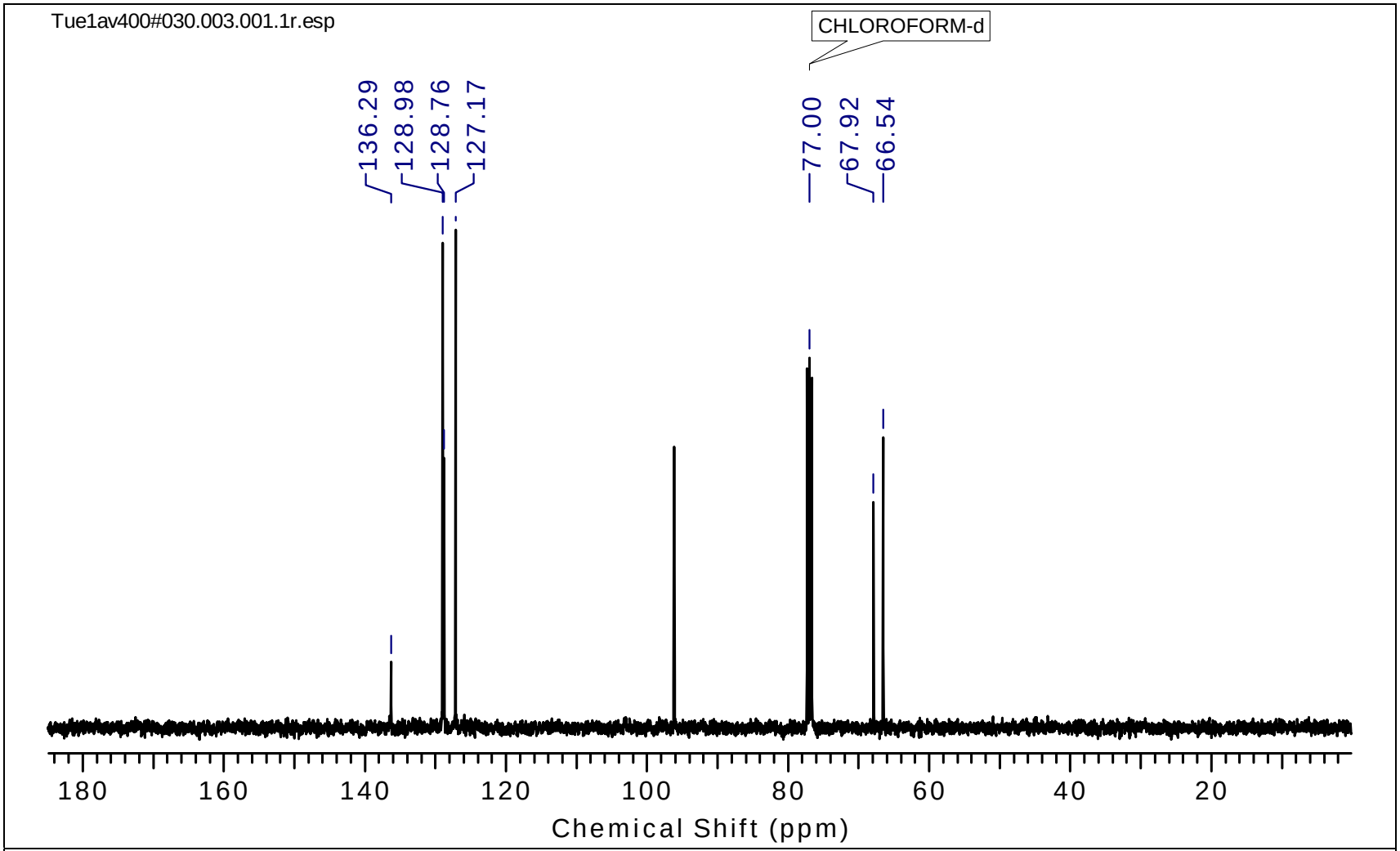
***syn*-(1*RS*,2*RS*)-2-azido-1-(4-methoxyphenyl)propane-1,3-diol (2n)**


ACETONE-d6
TMS
8.09
8.07
7.60
7.58
6.10
5.24
5.24
4.51
4.15
4.11
3.78
3.77
3.76
3.74
3.69
3.68
3.66
3.65
2.05
0.04
OH
OH
NHCOCHCl2
O2N
8
1.62 2.05
0.63
0.87
3.63 0.64 0.97 1.22
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

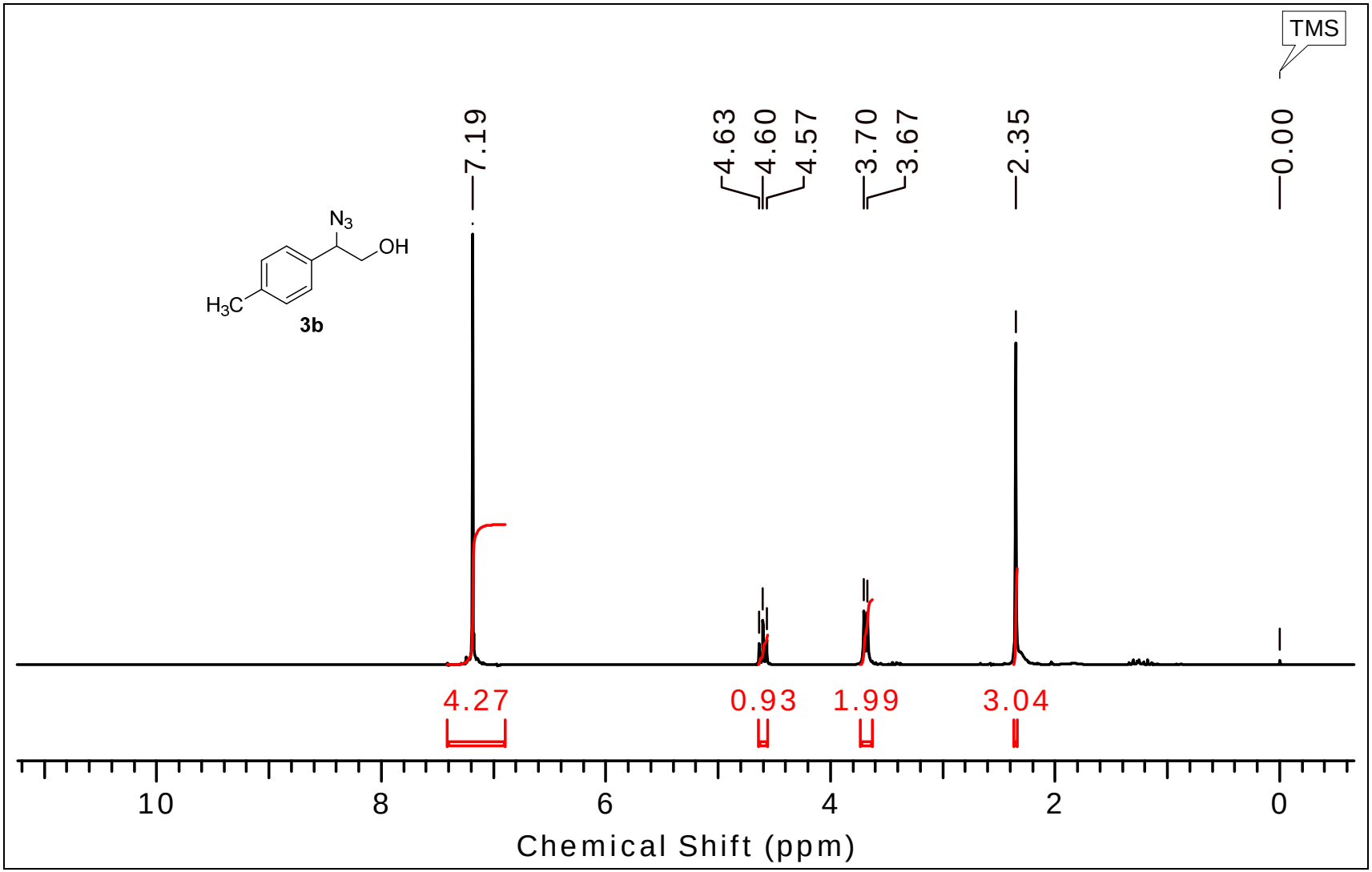


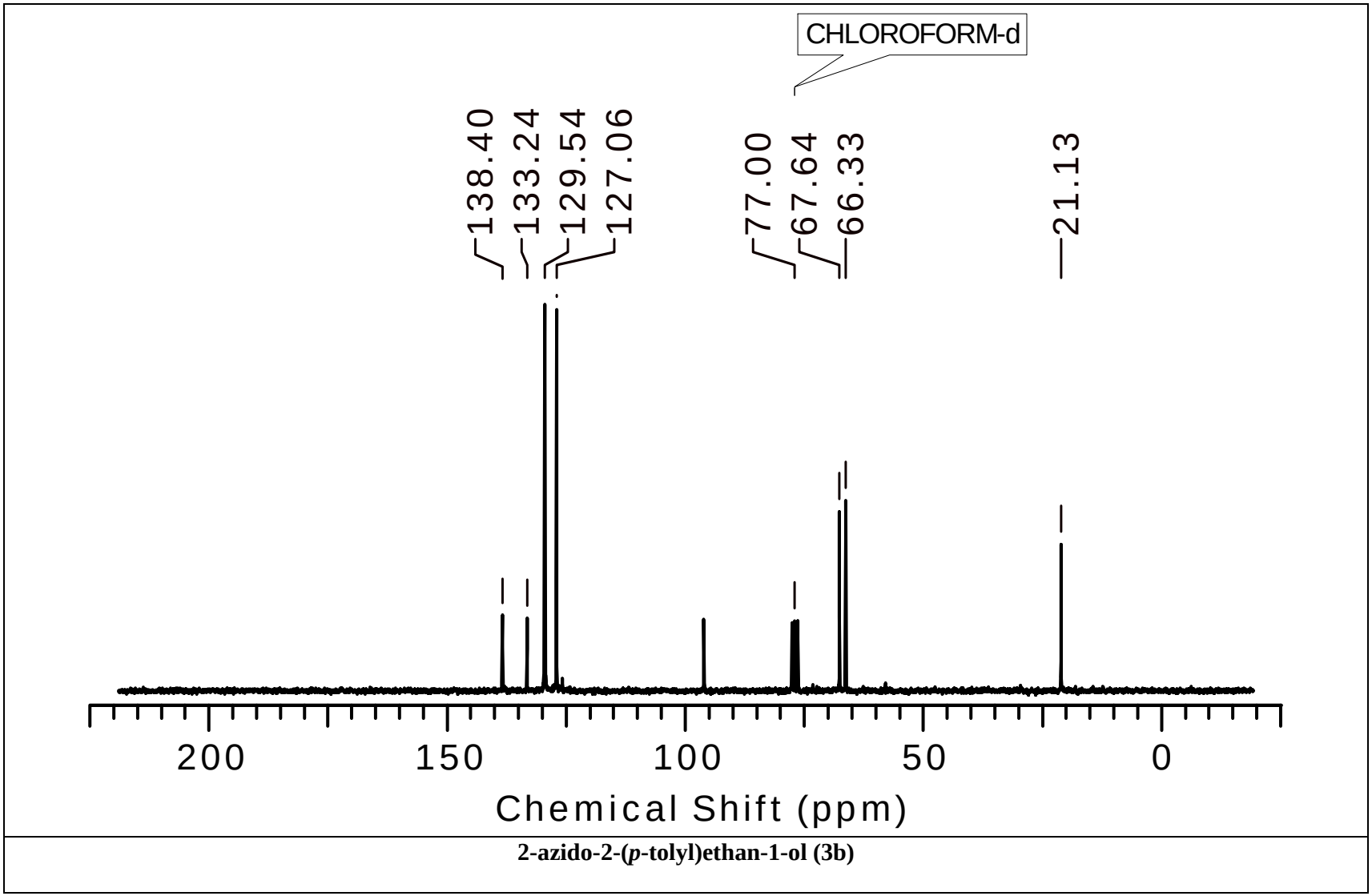
(±)-Chloramphenicol (9)


Tue1av400#030.001.001.1r.esp
CHLOROFORM-d
7.41
7.41
7.39
7.38
7.36
7.35
7.34
7.33
4.69
4.68
4.66
3.75
3.73
2.02
0.02
N3
OH
3a
5.15
1.00
2.13
0.98
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

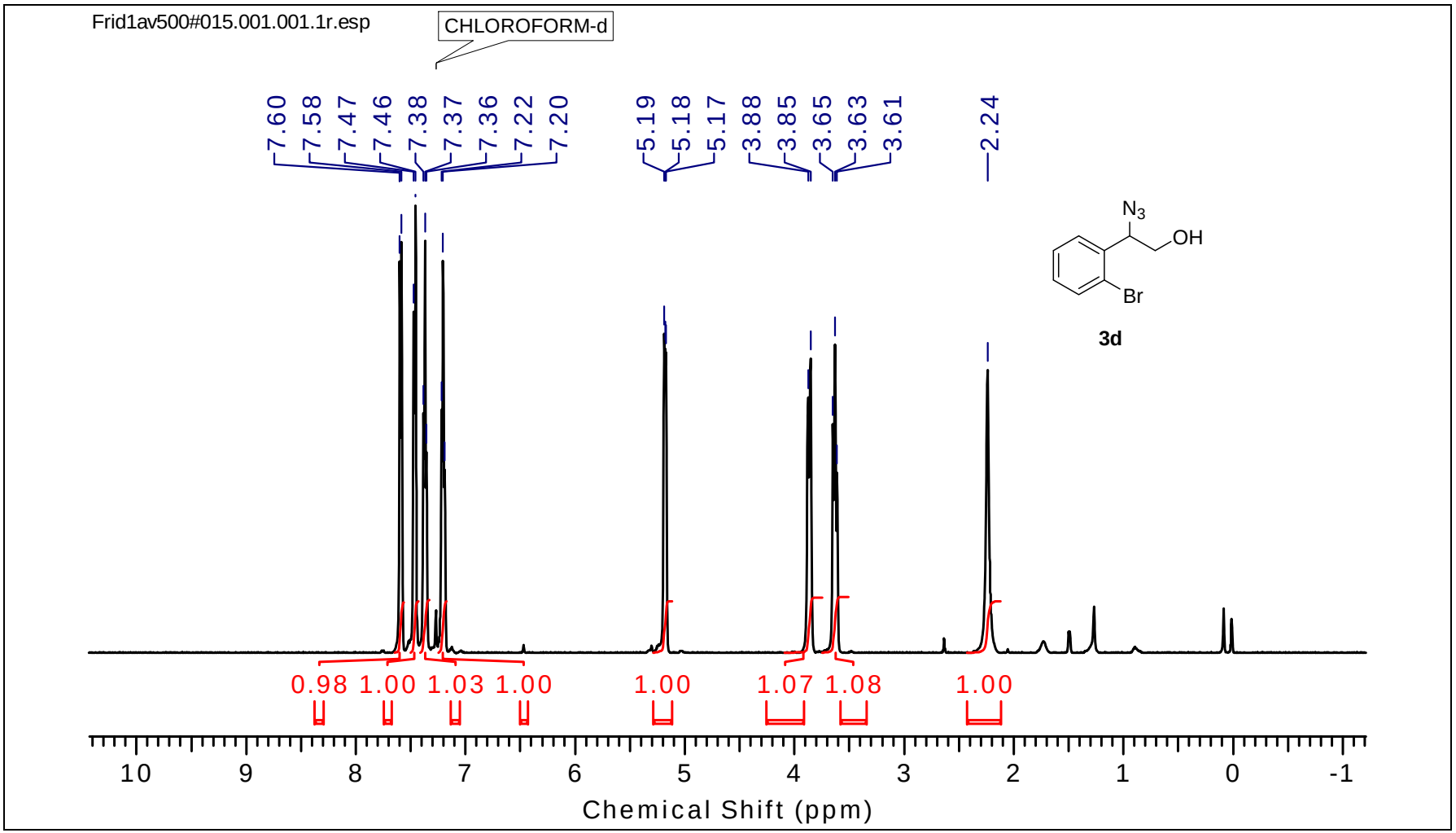


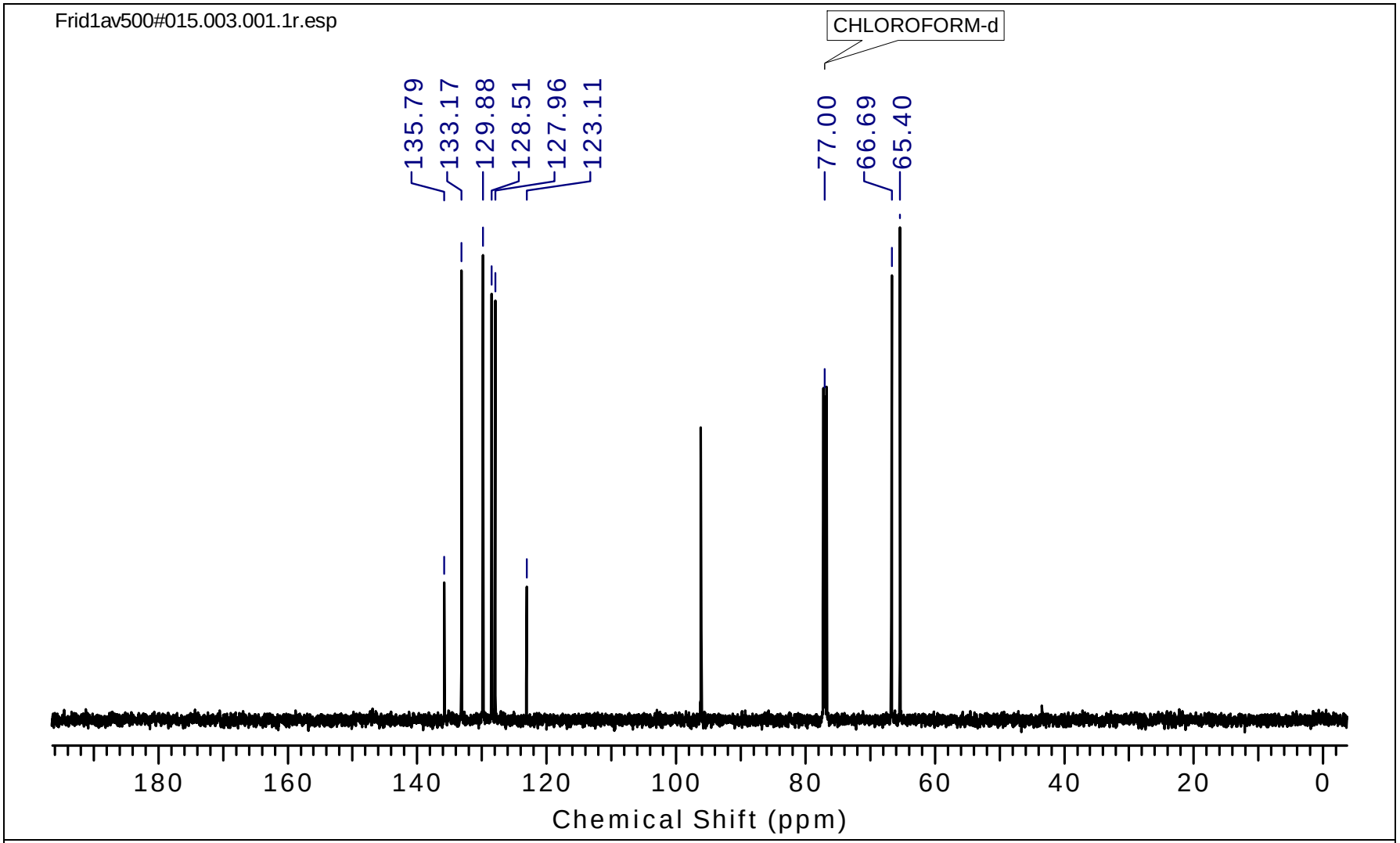
2-azido-2-phenylethan-1-ol (3a)


TMS
7.19
4.63
4.60
4.57
3.70
3.67
2.35
0.00
N3
OH
H3C
3b
4.27
0.93
1.99
3.04
10
8
6
4
2
0
Chemical Shift (ppm)

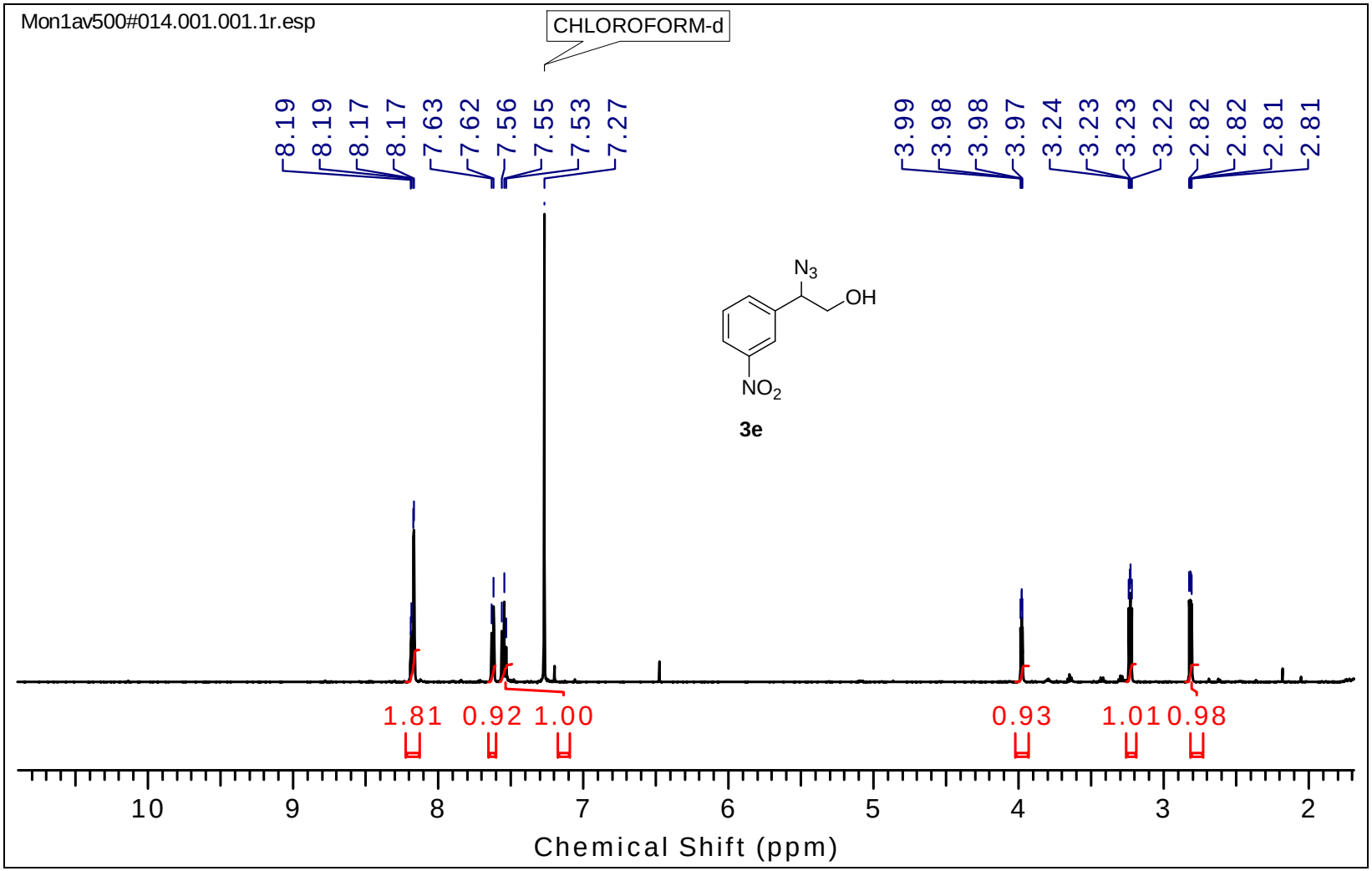


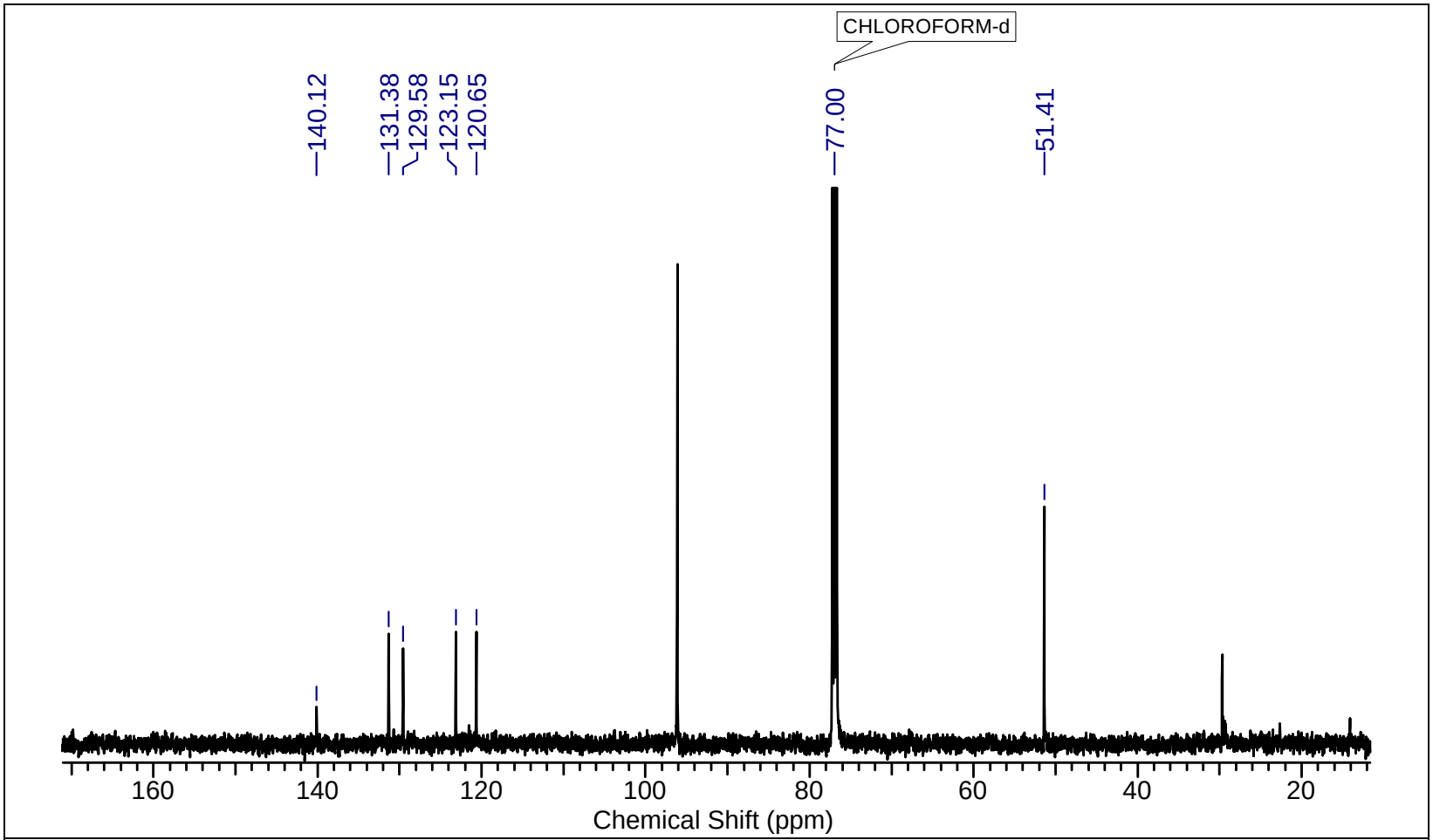
2-azido-2-(*p*-tolyl)ethan-1-ol (3b)


Frid1av500#015.001.001.1r.esp
CHLOROFORM-d
7.60
7.58
7.47
7.46
7.38
7.37
7.36
7.22
7.20
5.19
5.18
5.17
3.88
3.85
3.65
3.63
3.61
2.24
N3
OH
Br
3d
0.98 1.00 1.03 1.00 1.00 1.07 1.08 1.00
10 9 8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

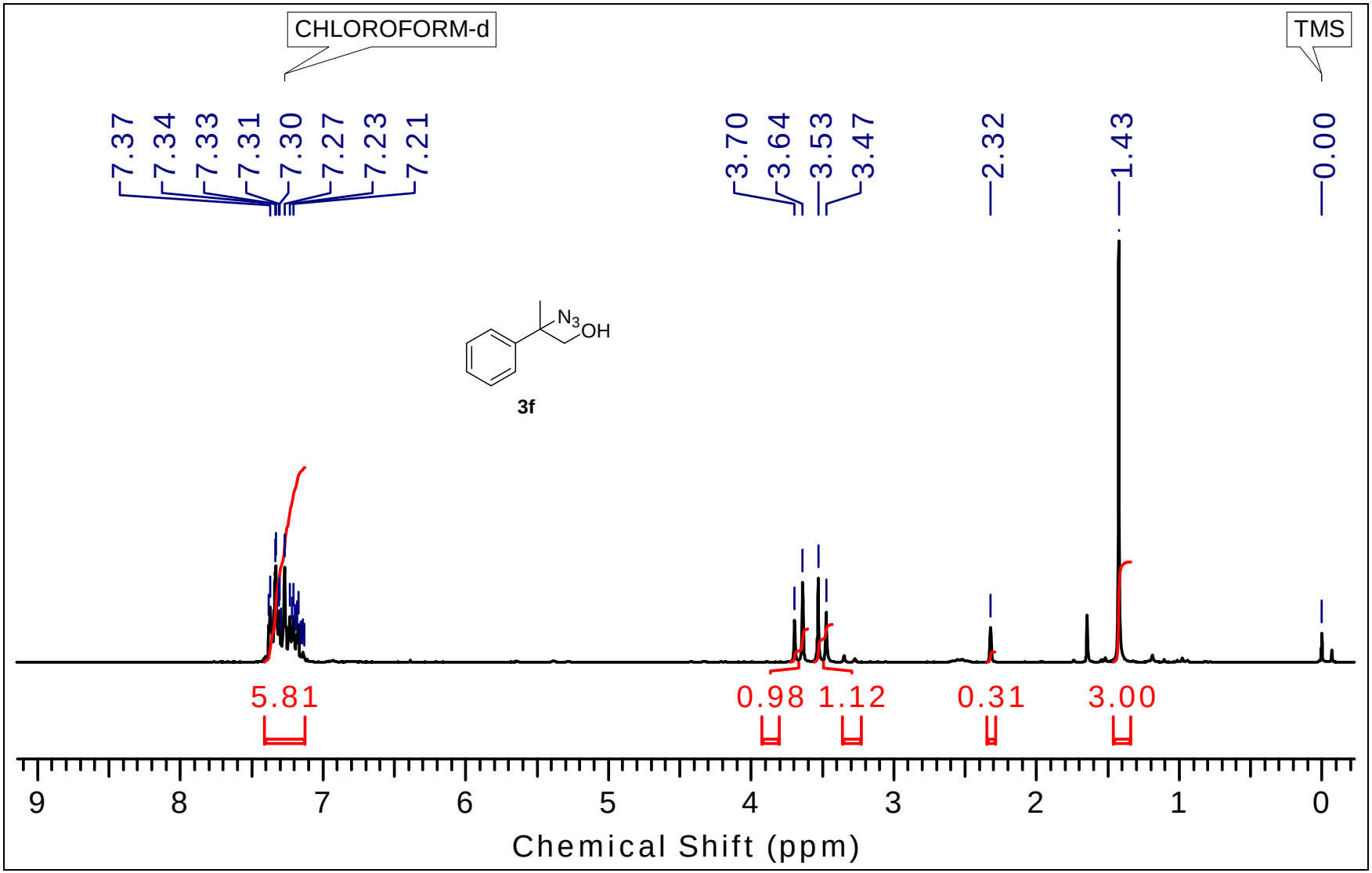


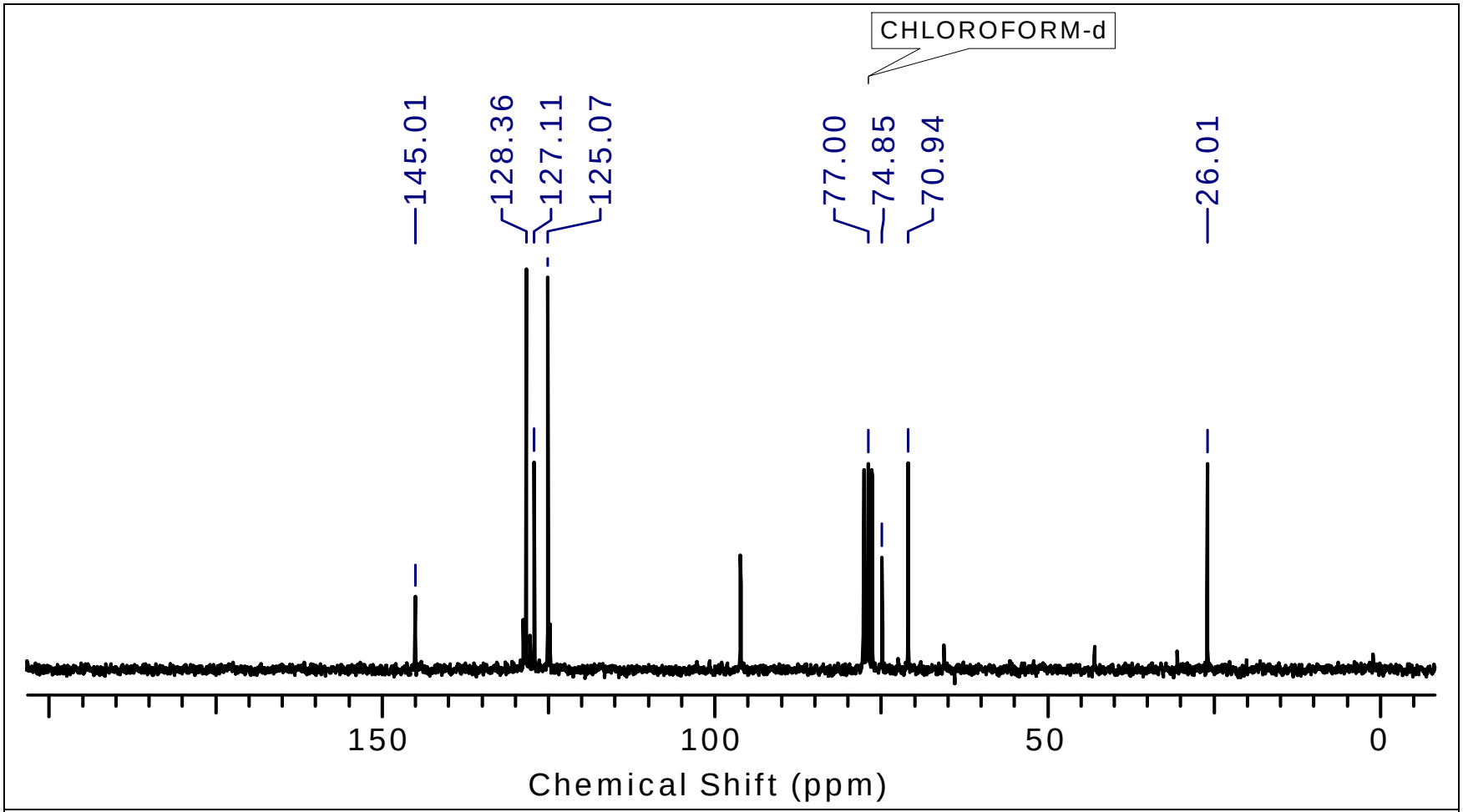
2-azido-2-(2-bromophenyl)ethan-1-ol (3d)


Mon1av500#014.001.001.1r.esp
CHLOROFORM-d
8.19
8.19
8.17
8.17
7.63
7.62
7.56
7.55
7.53
7.27
3.99
3.98
3.98
3.97
3.24
3.23
3.23
3.22
2.82
2.82
2.81
2.81
N3
OH
NO2
3e
1.81
0.92
1.00
0.93
1.01
0.98
10
9
8
7
6
5
4
3
2
Chemical Shift (ppm)

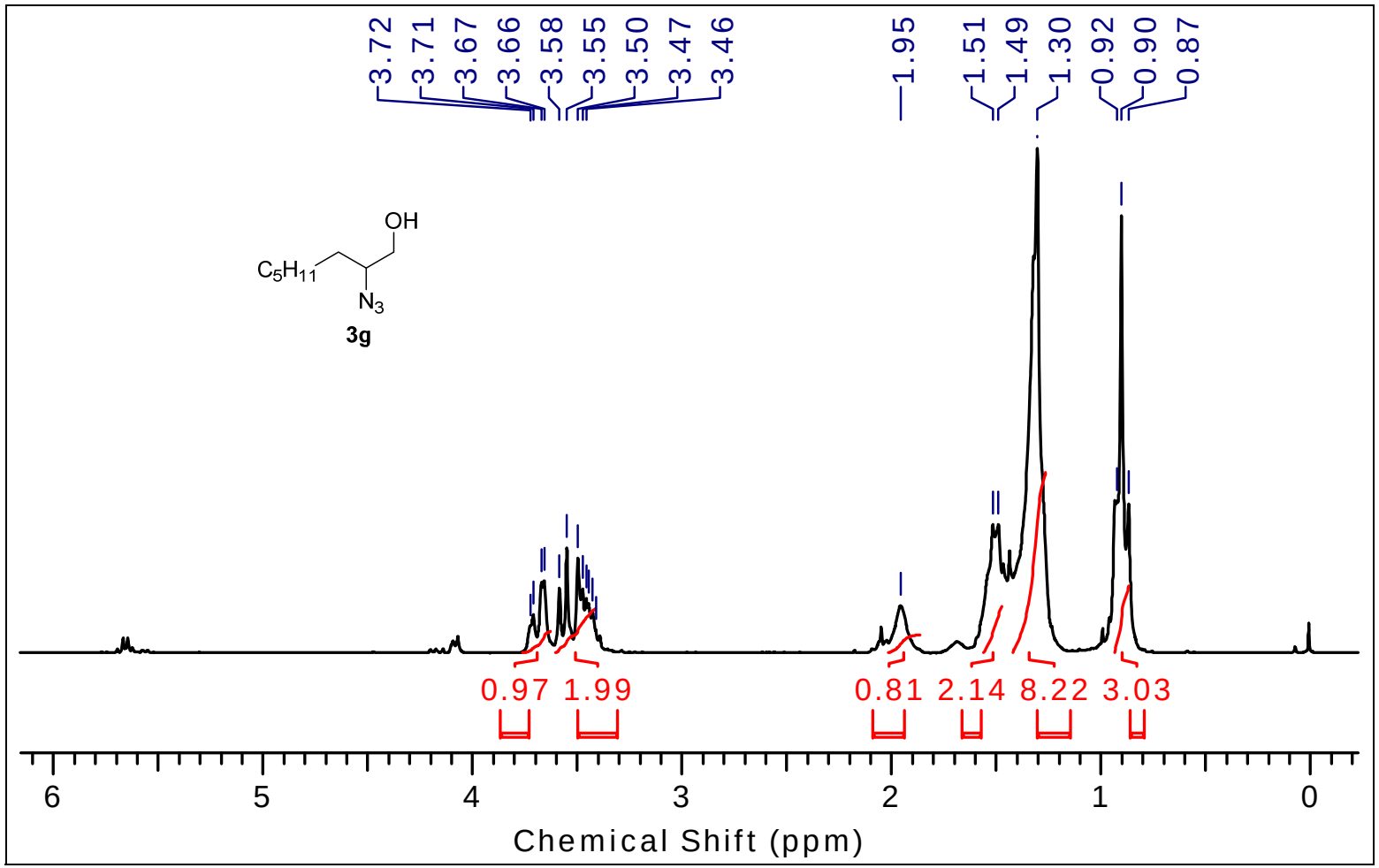


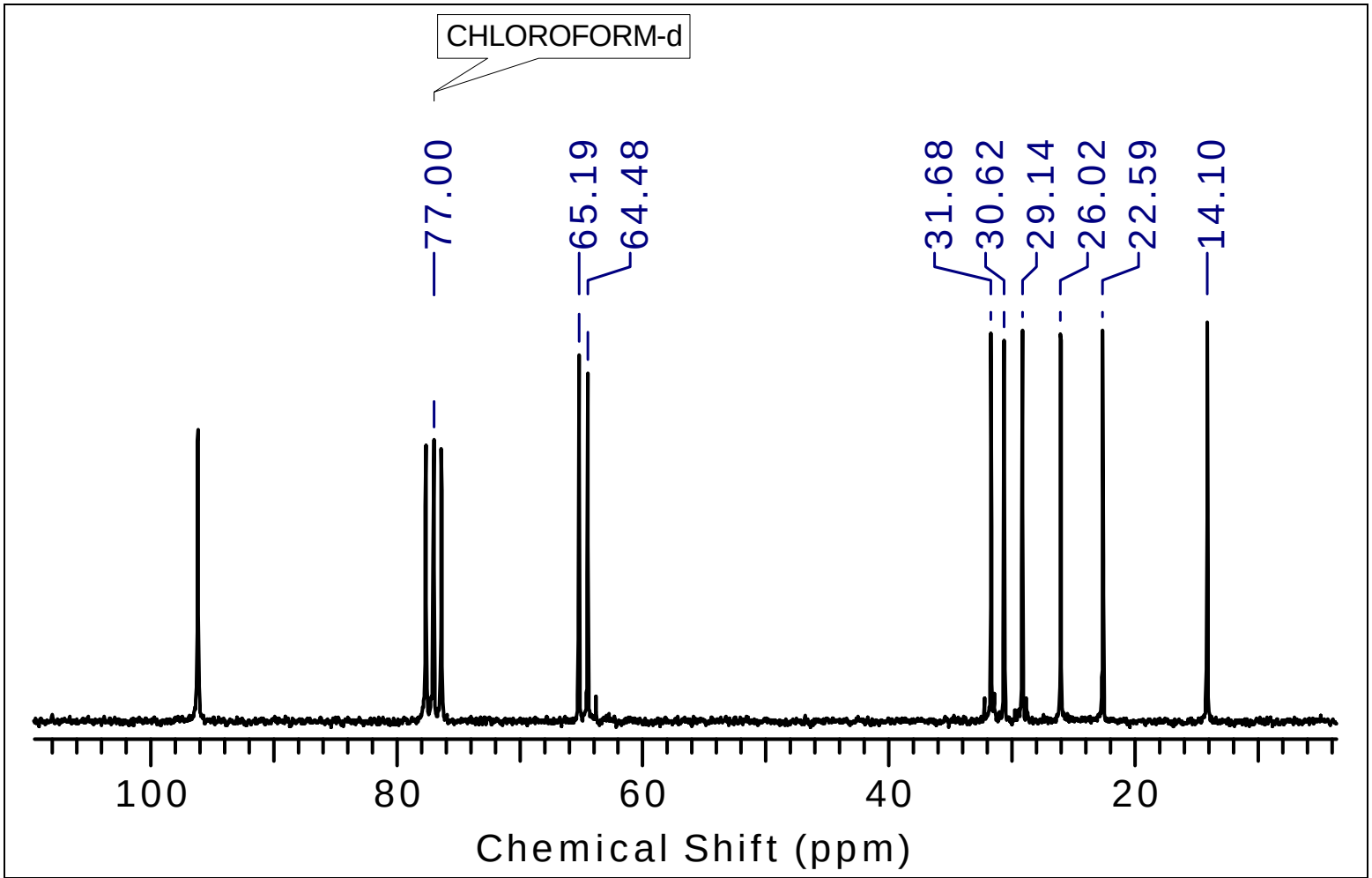
2-azido-2-(3-nitrophenyl)ethan-1-ol (3e)


CHLOROFORM-d
TMS
7.37
7.34
7.33
7.31
7.30
7.27
7.23
7.21
3.70
3.64
3.53
3.47
2.32
1.43
0.00
N3
OH
3f
5.81
0.98
1.12
0.31
3.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

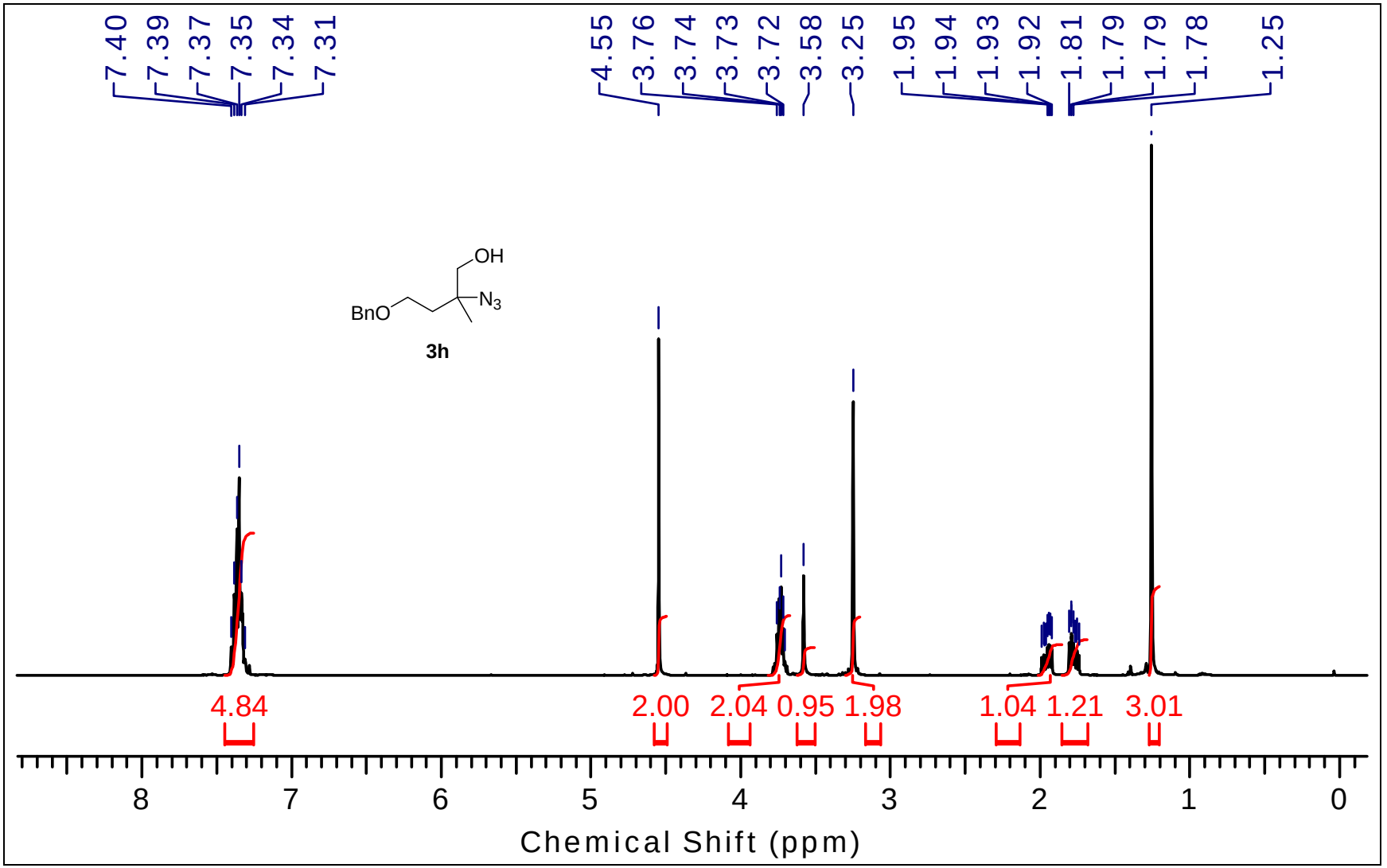


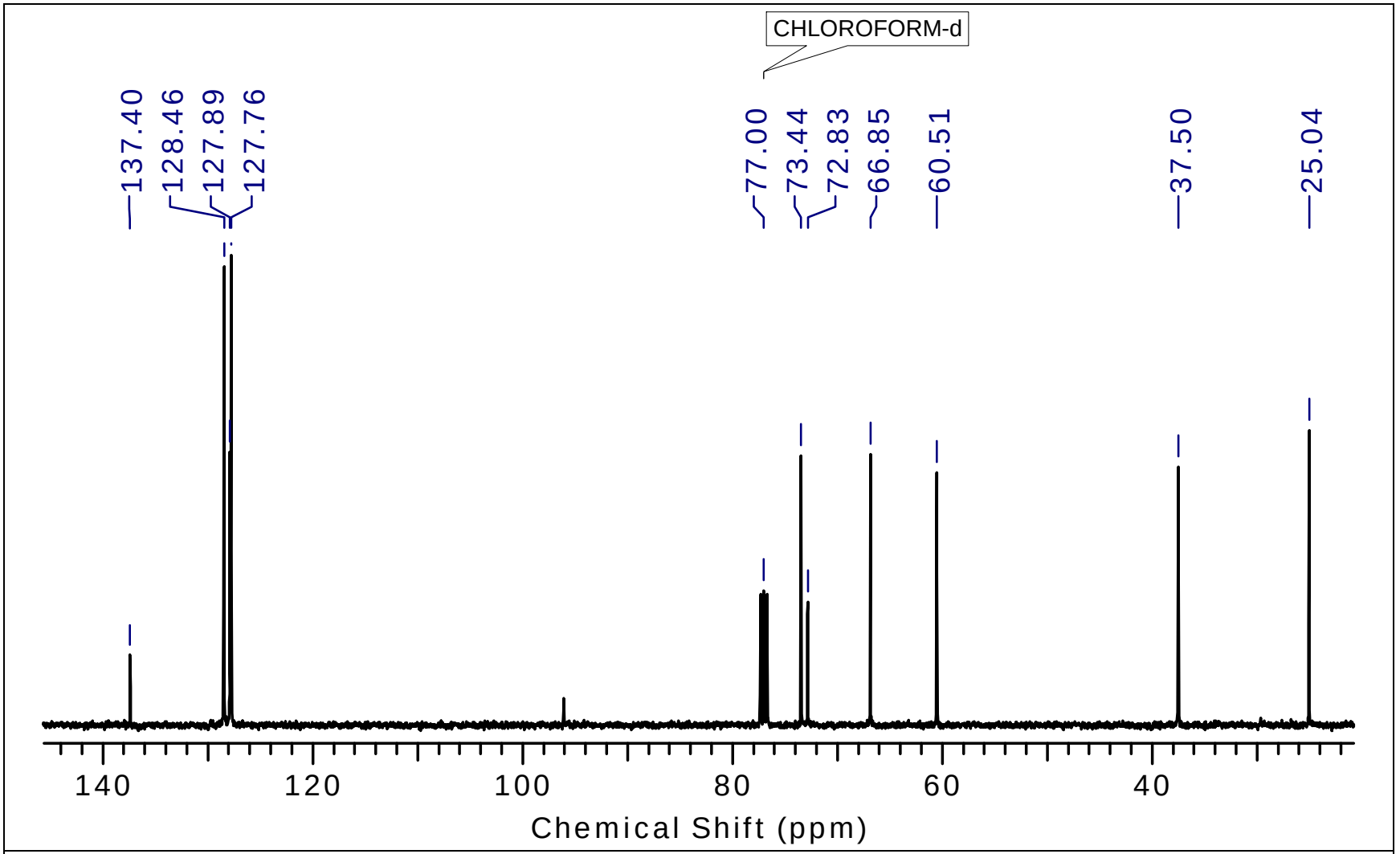
2-azido-2-phenylpropan-1-ol (3f)


3.72
3.71
3.67
3.66
3.58
3.55
3.50
3.47
3.46
1.95
1.51
1.49
1.30
0.92
0.90
0.87
OH
C5H11
N3
3g
0.97 1.99
0.81 2.14 8.22 3.03
6
5
4
3
2
1
0
Chemical Shift (ppm)

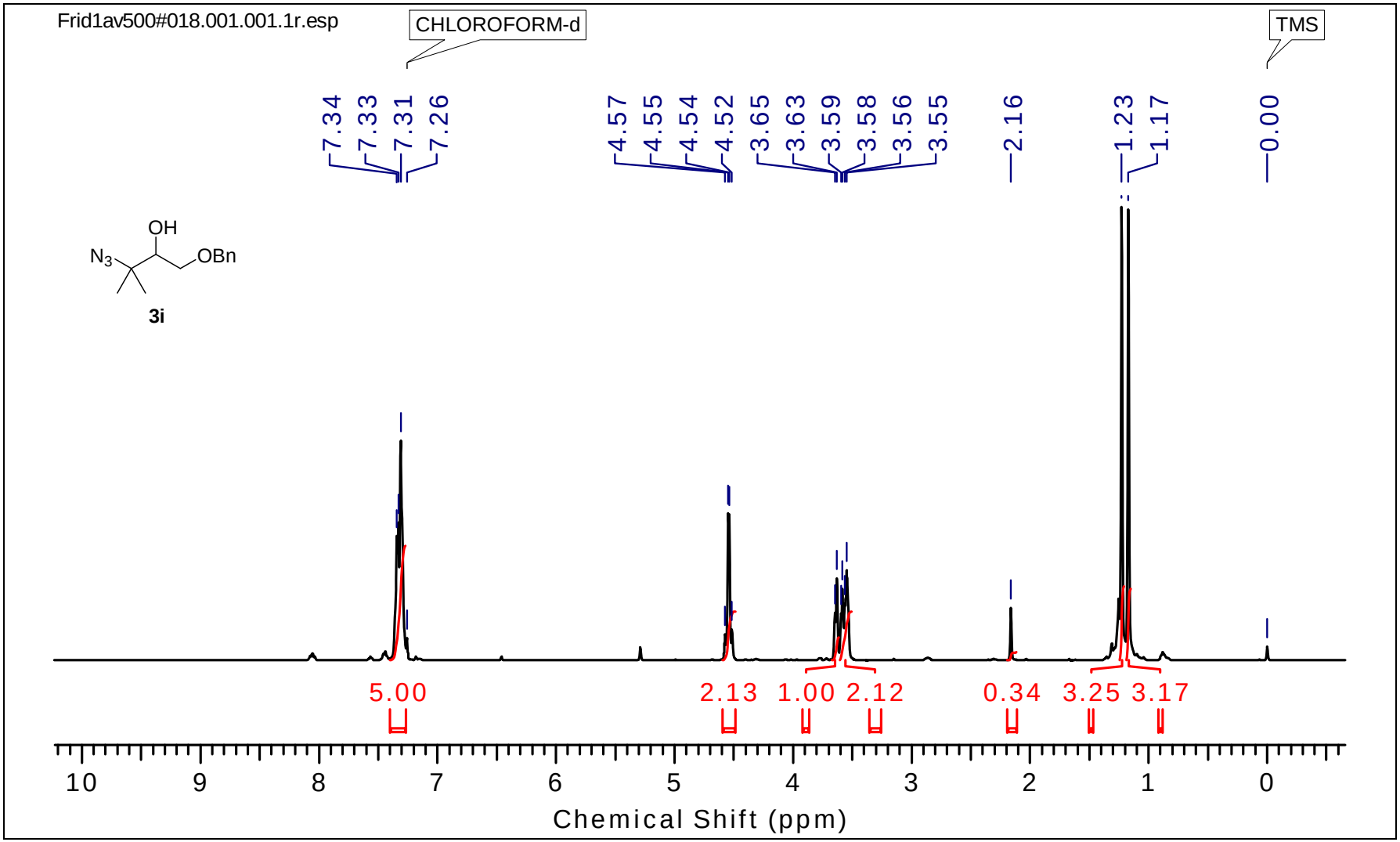


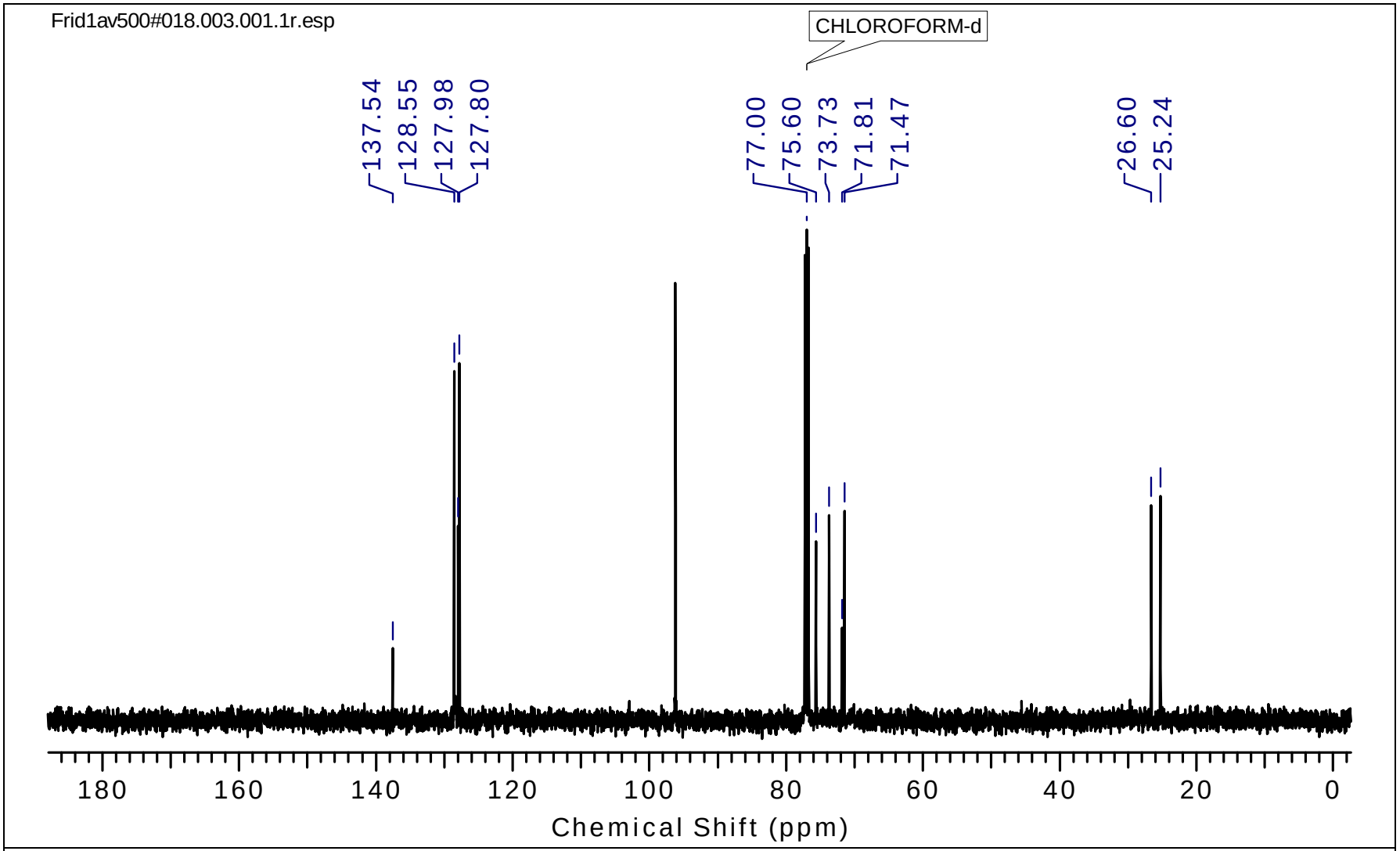
2-azidooctan-1-ol (3g)


7.40
7.39
7.37
7.35
7.34
7.31
4.55
3.76
3.74
3.73
3.72
3.58
3.25
1.95
1.94
1.93
1.92
1.81
1.79
1.79
1.78
1.25
OH
N3
BnO
3h
4.84
2.00
2.04
0.95
1.98
1.04
1.21
3.01
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

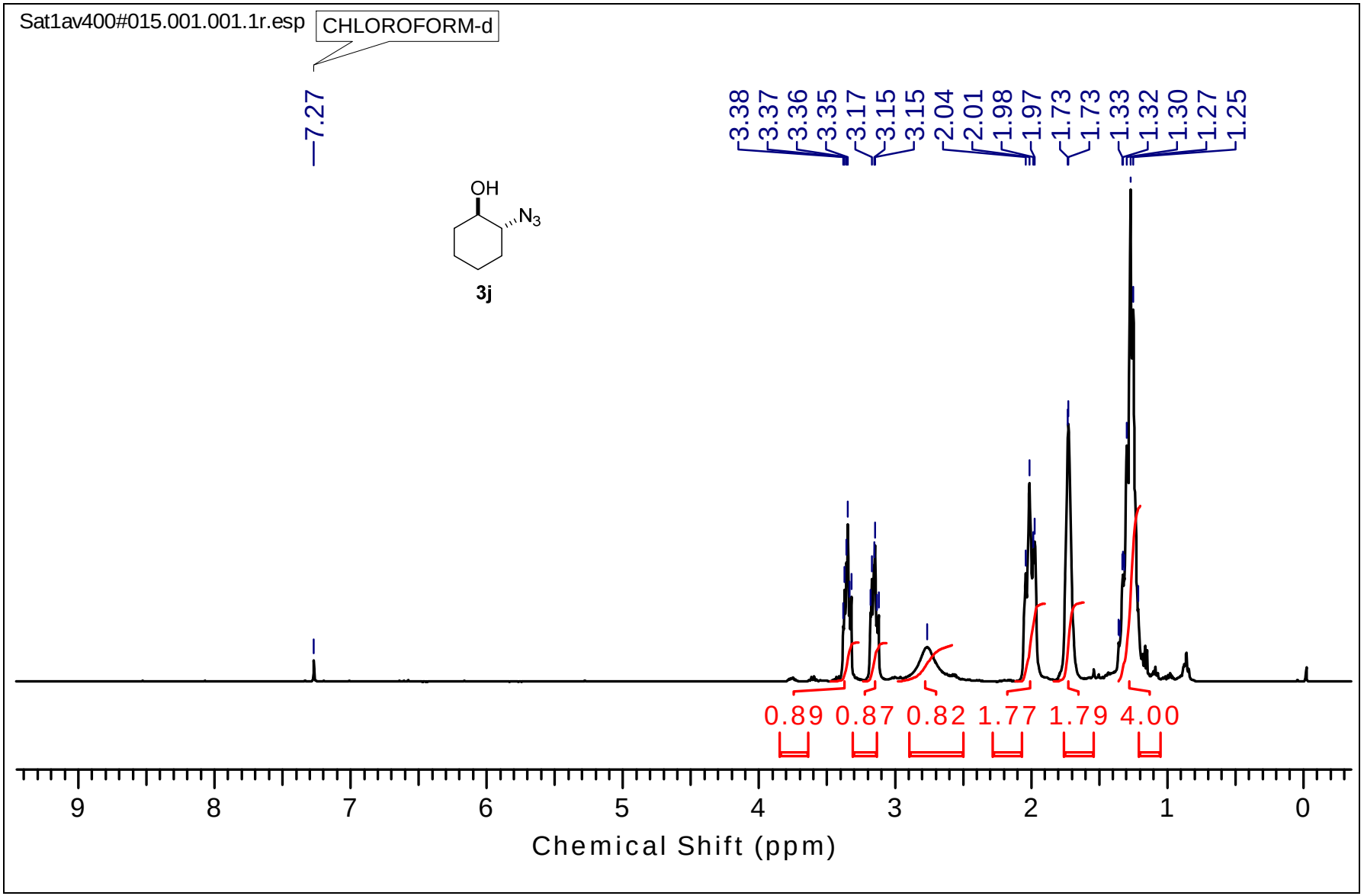


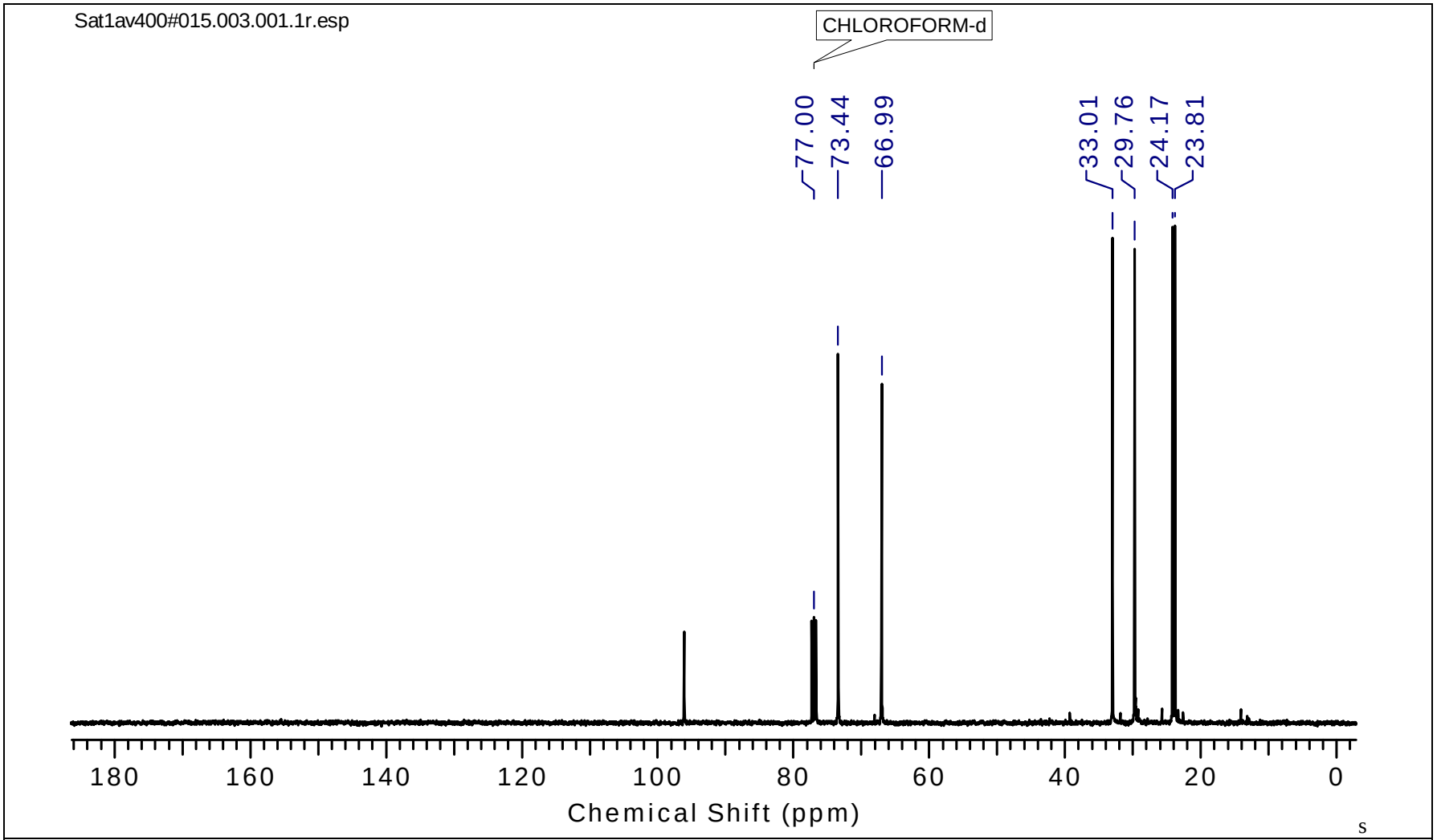
2-azido-4-(benzyloxy)-2-methylbutan-1-ol (3h)


Frid1av500#018.001.001.1r.esp
CHLOROFORM-d
TMS
7.34
7.33
7.31
7.26
4.57
4.55
4.54
4.52
3.65
3.63
3.59
3.58
3.56
3.55
2.16
1.23
1.17
0.00
OH
N3
OBn
3i
5.00
2.13
1.00
2.12
0.34
3.25
3.17
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

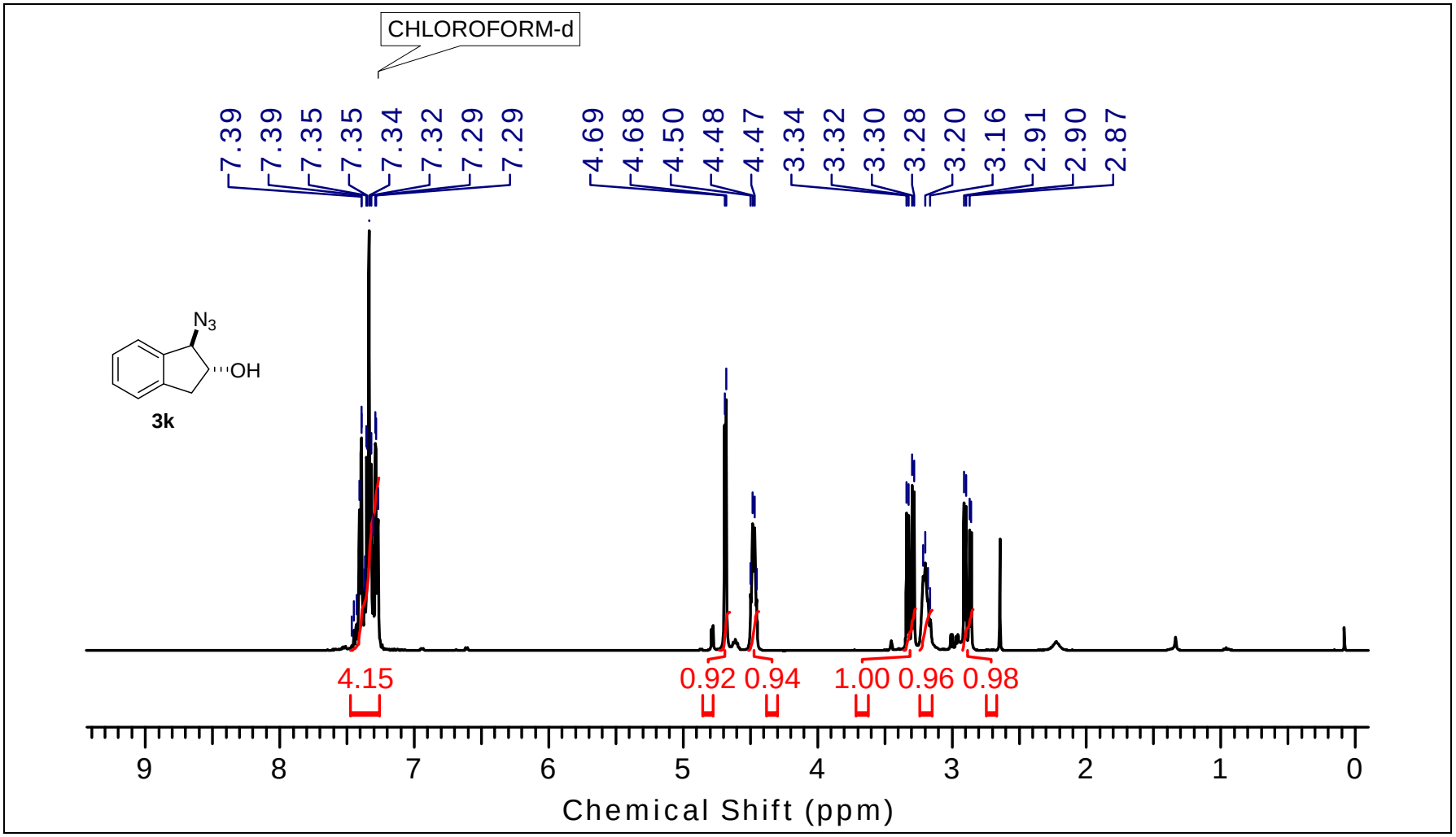


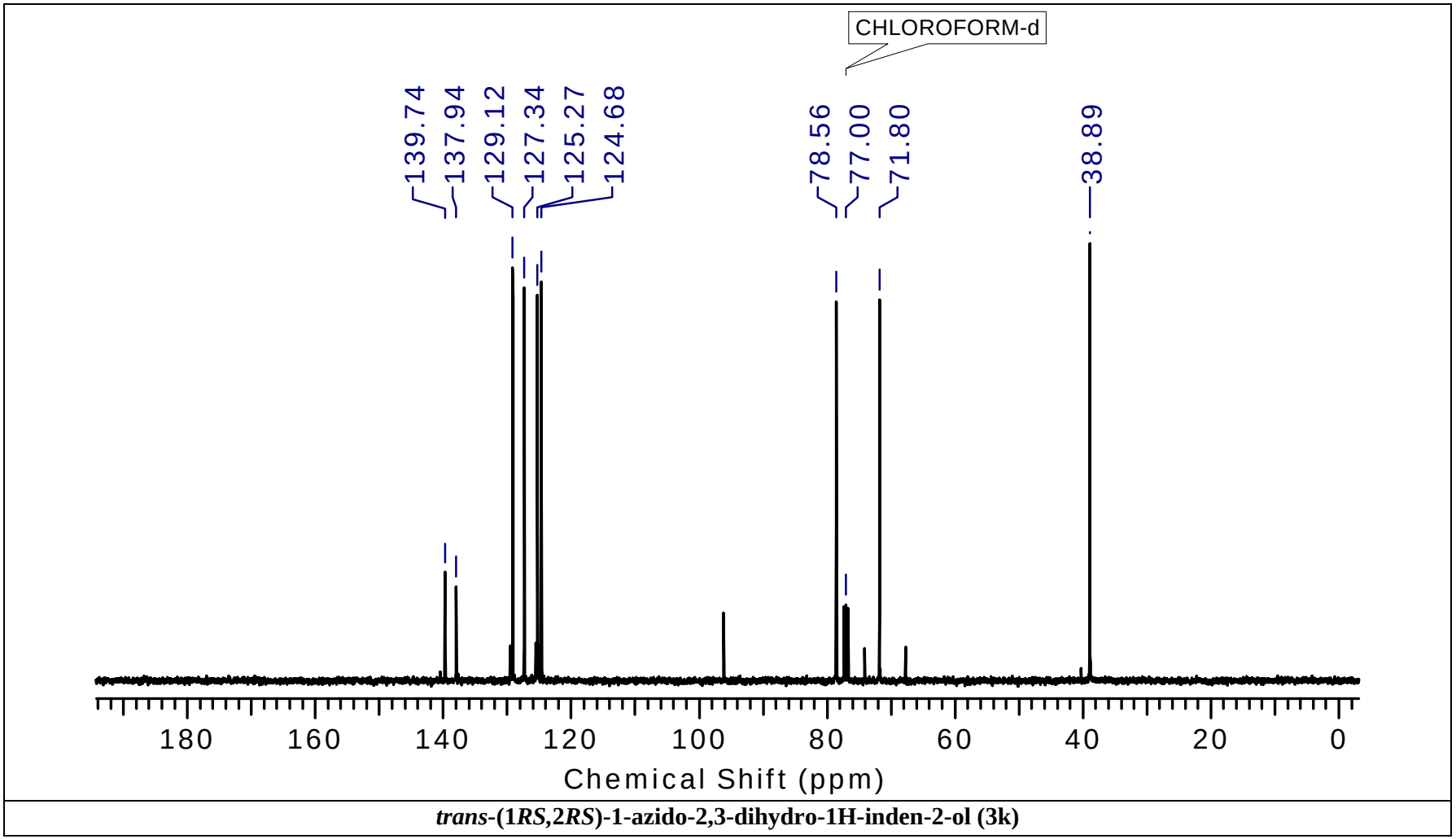
3-azido-1-(benzyloxy)-3-methylbutan-2-ol (3i)


Sat1av400#015.001.001.1r.esp
CHLOROFORM-d
7.27
3.38
3.37
3.36
3.35
3.17
3.15
3.15
2.04
2.01
1.98
1.97
1.73
1.73
1.33
1.32
1.30
1.27
1.25
OH
N3
3j
0.89 0.87 0.82 1.77 1.79 4.00
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

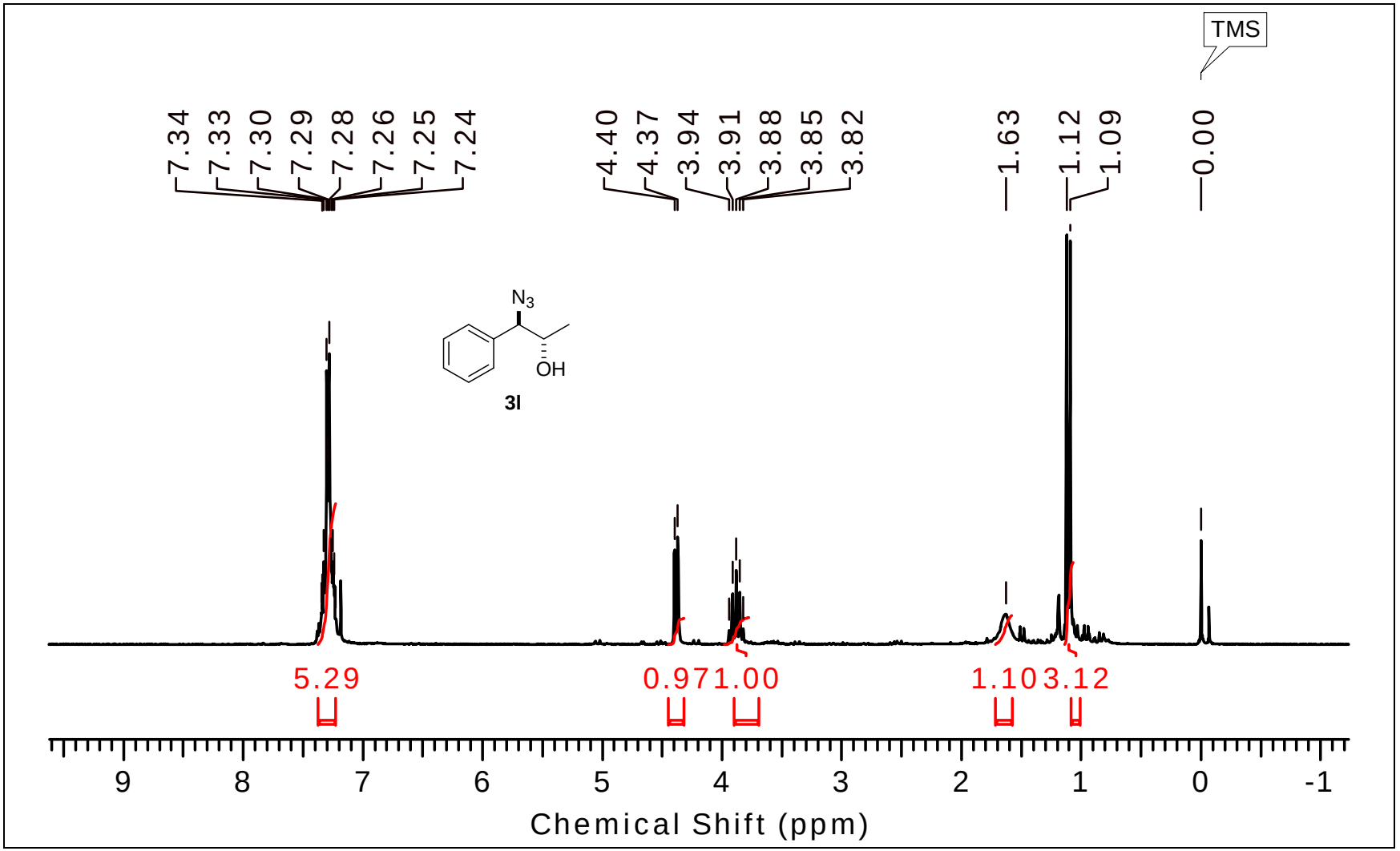


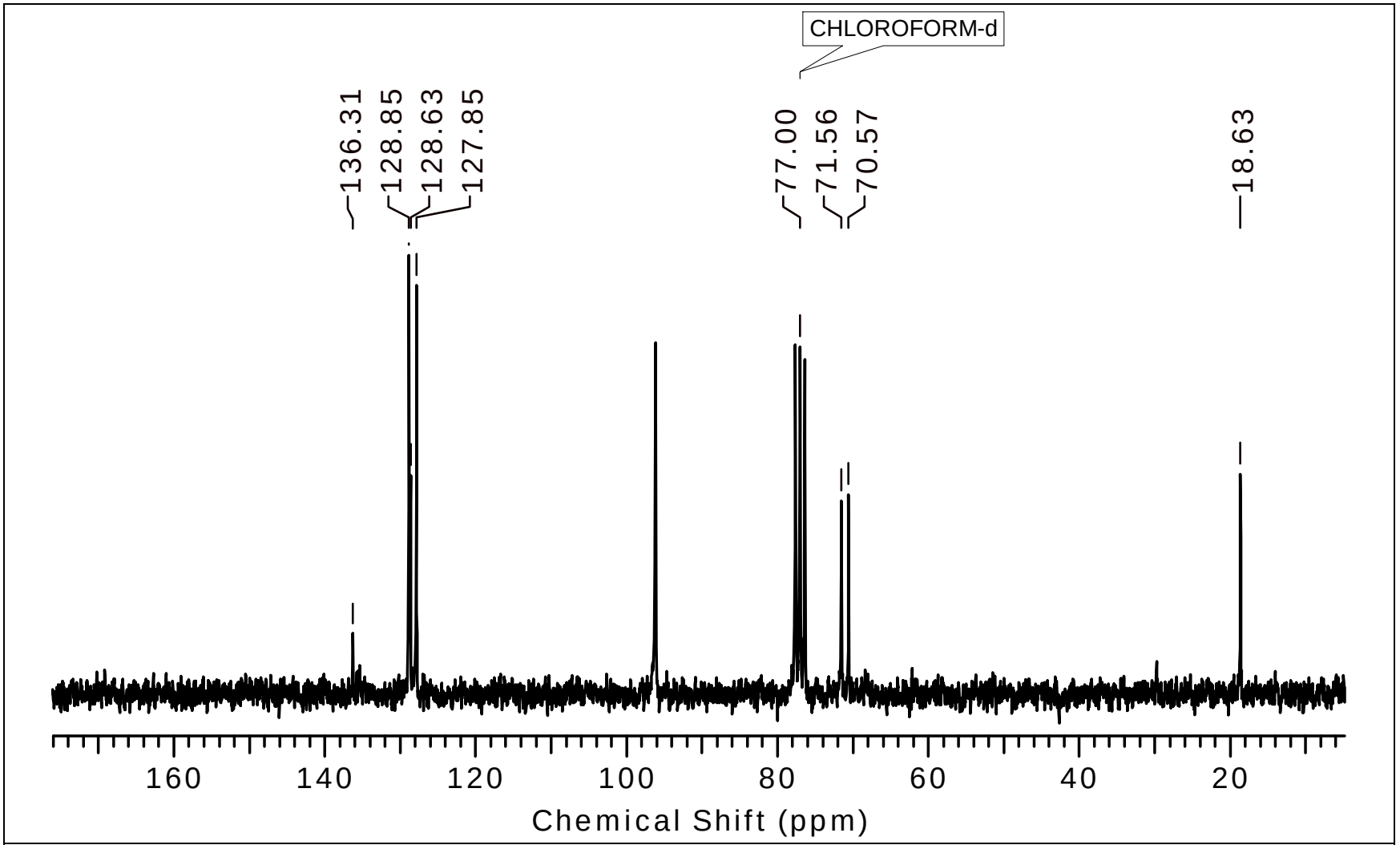
***trans*-(1*SR*,2*SR*)-2-azidocyclohexan-1-ol (3j)**


CHLOROFORM-d
7.39
7.39
7.35
7.35
7.34
7.32
7.29
7.29
4.69
4.68
4.50
4.48
4.47
3.34
3.32
3.30
3.28
3.20
3.16
2.91
2.90
2.87
N3
OH
3k
4.15
0.92
0.94
1.00
0.96
0.98
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

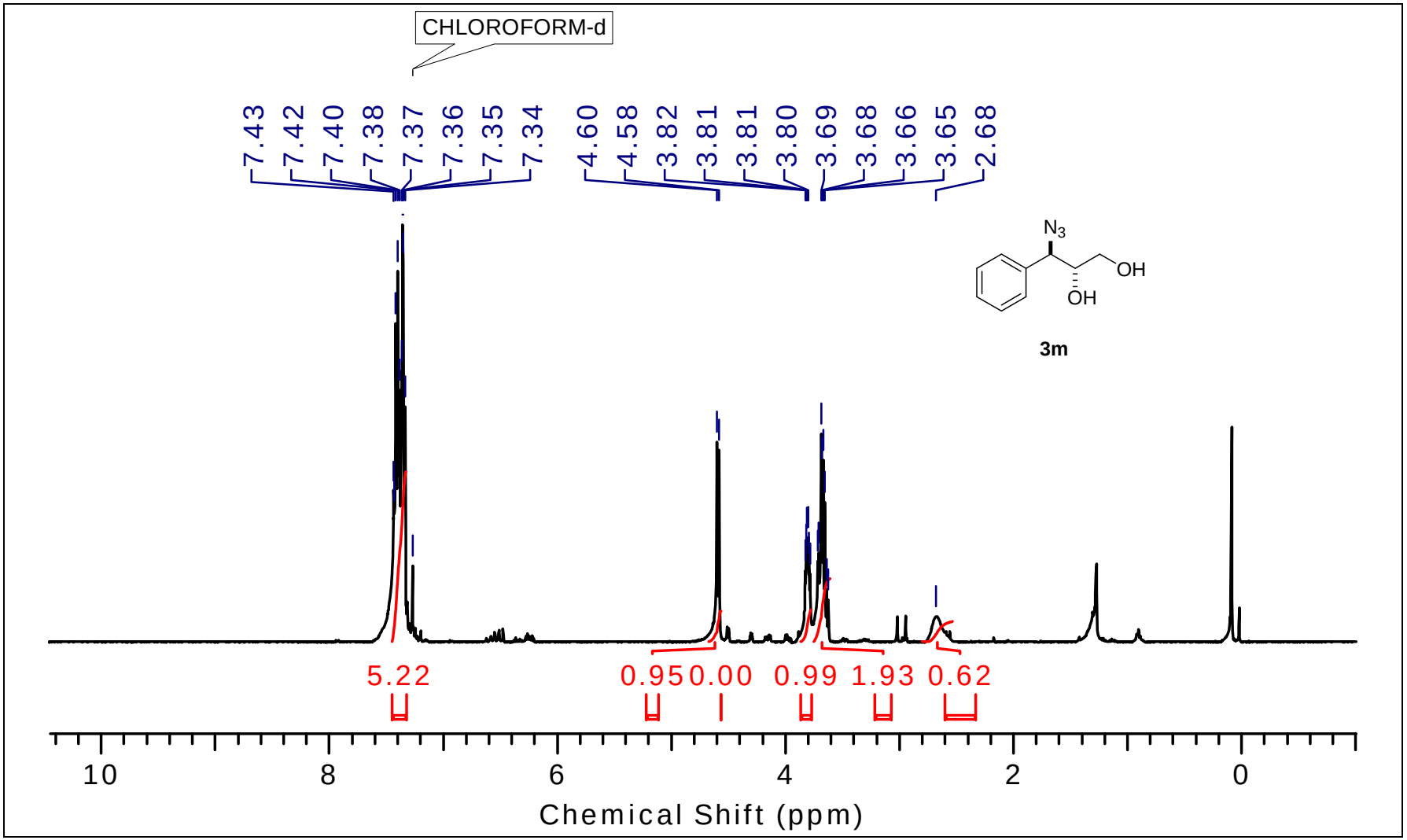


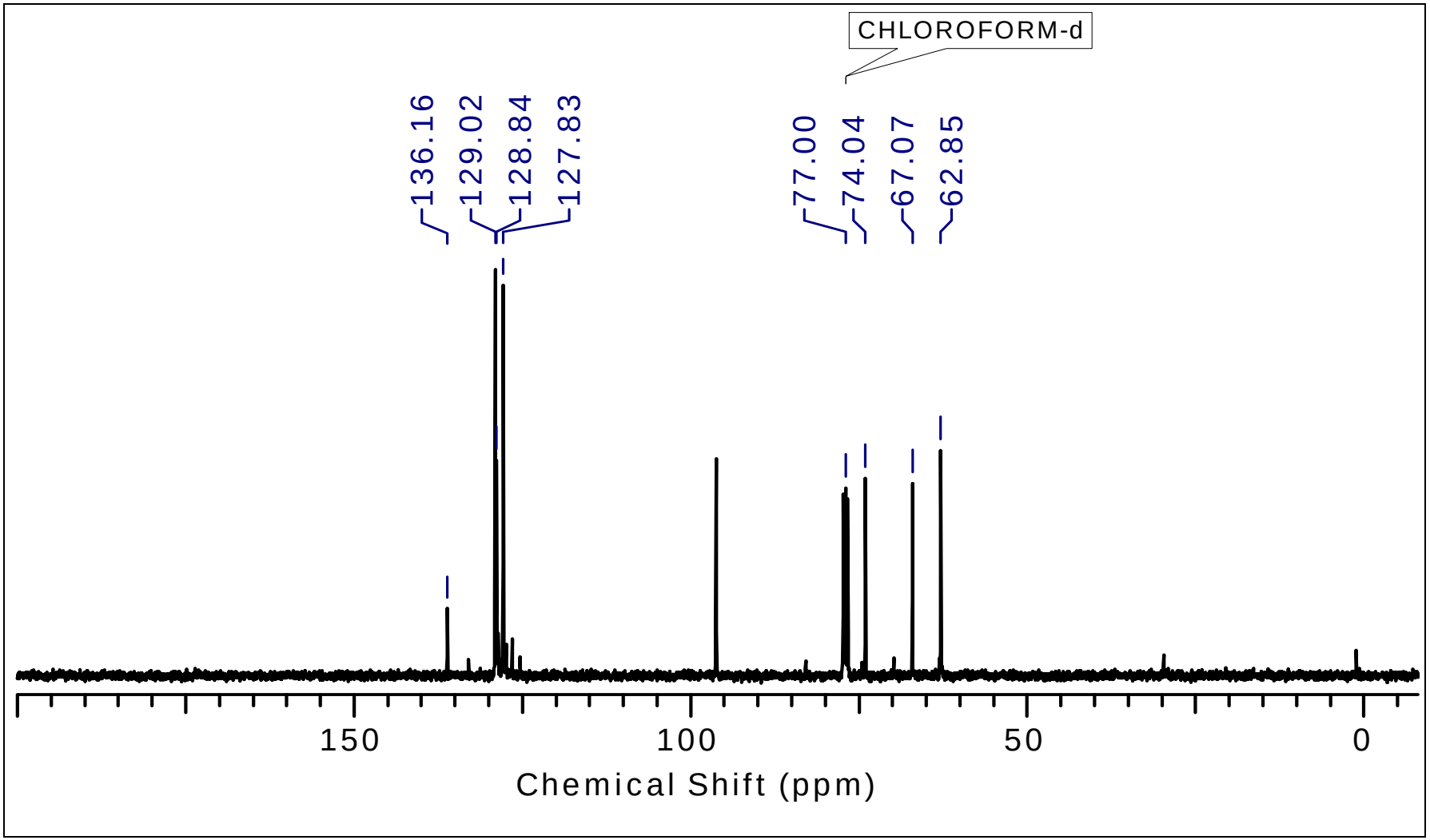
***trans*-(1*RS*,2*RS*)-1-azido-2,3-dihydro-1H-inden-2-ol (3k)**

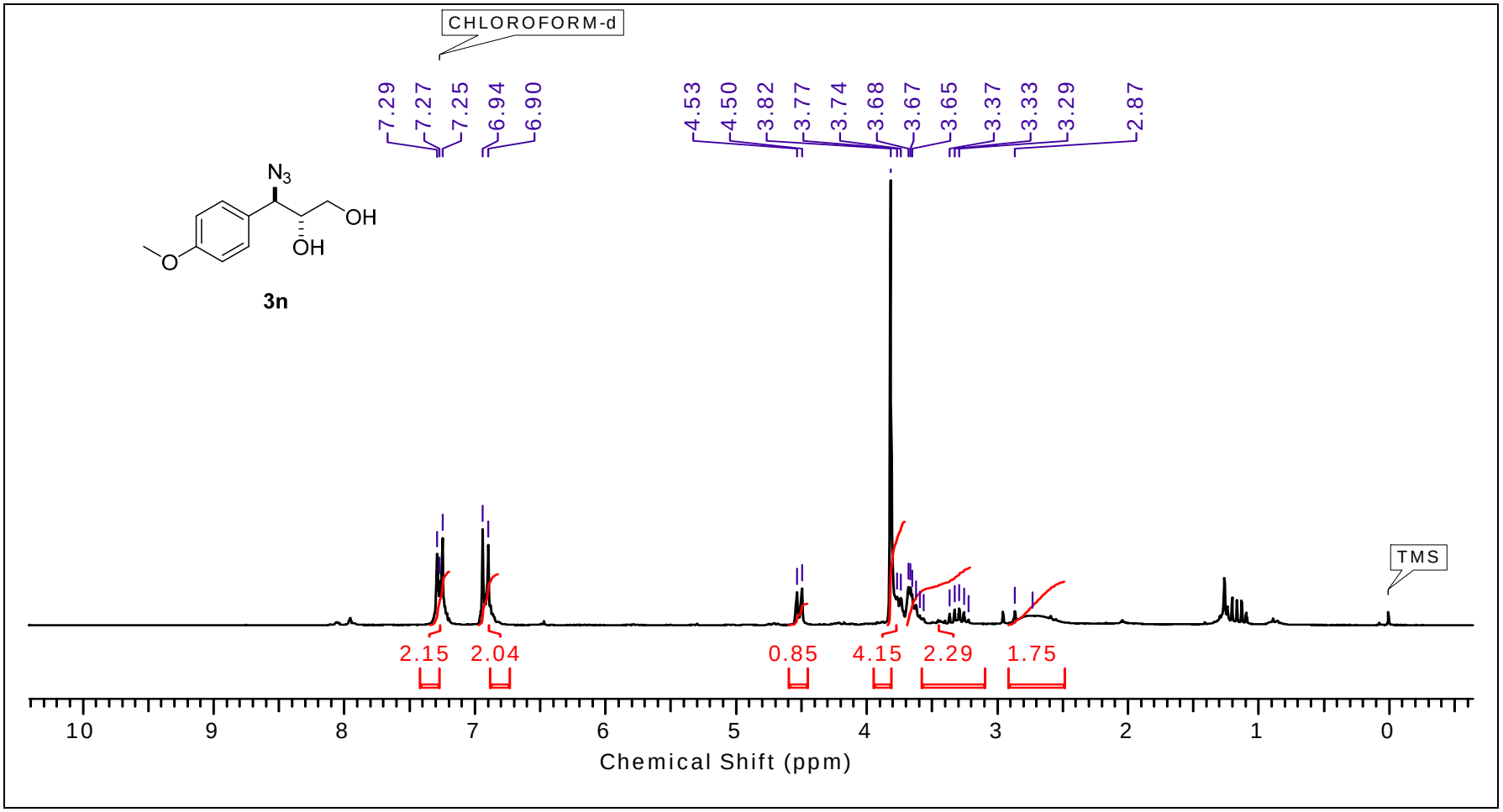

TMS
7.34
7.33
7.30
7.29
7.28
7.26
7.25
7.24
4.40
4.37
3.94
3.91
3.88
3.85
3.82
1.63
1.12
1.09
0.00
N3
OH
3l
5.29
0.97
1.00
1.10
3.12
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

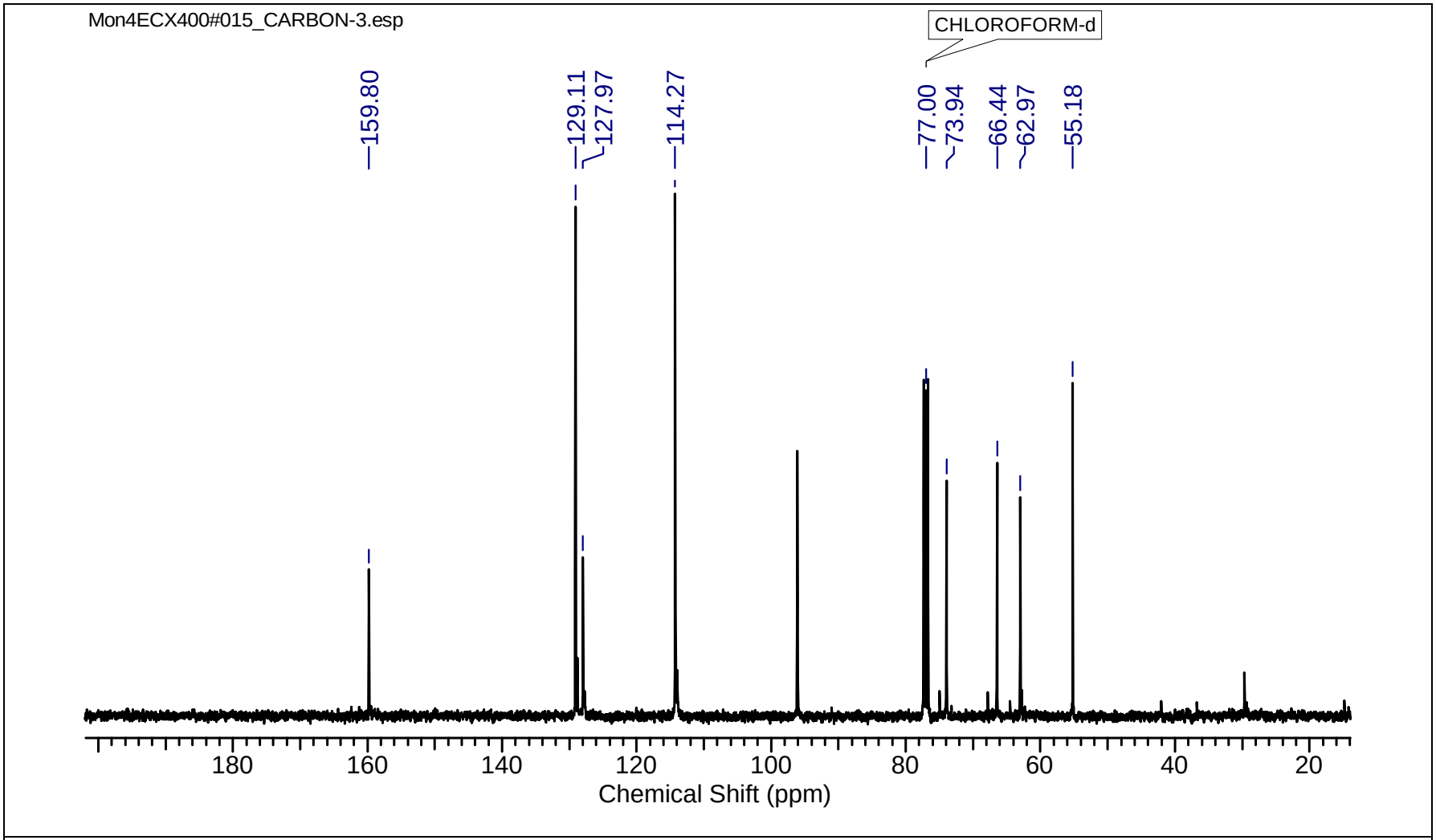


***anti*-(1*RS*,2*SR*)-1-azido-1-phenylpropan-2-ol (3l)**

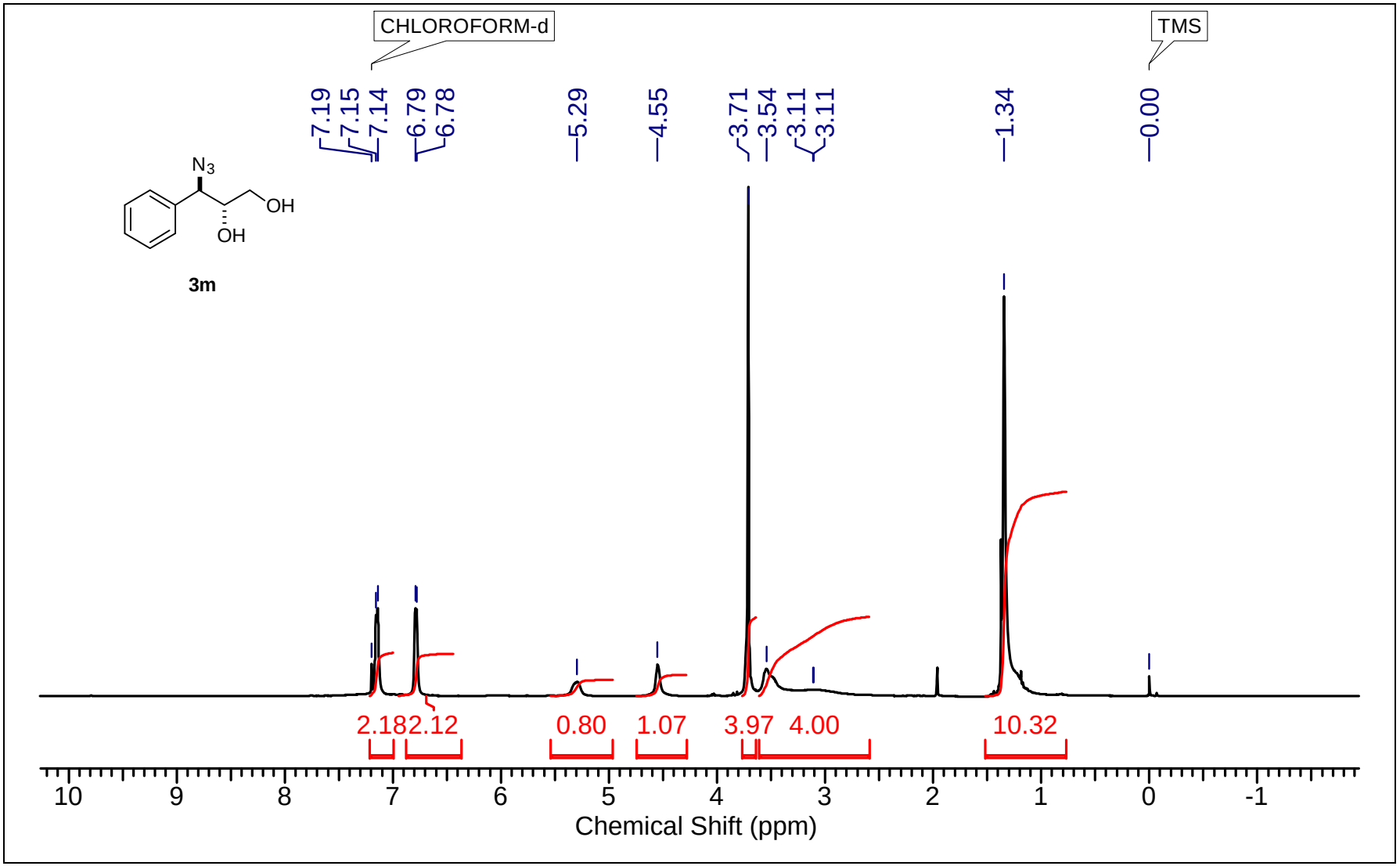

CHLOROFORM-d
7.43
7.42
7.40
7.38
7.37
7.36
7.35
7.34
4.60
4.58
3.82
3.81
3.81
3.80
3.69
3.68
3.66
3.65
2.68
N3
OH
OH
3m
5.22
0.95
0.00
0.99
1.93
0.62
10
8
6
4
2
0
Chemical Shift (ppm)

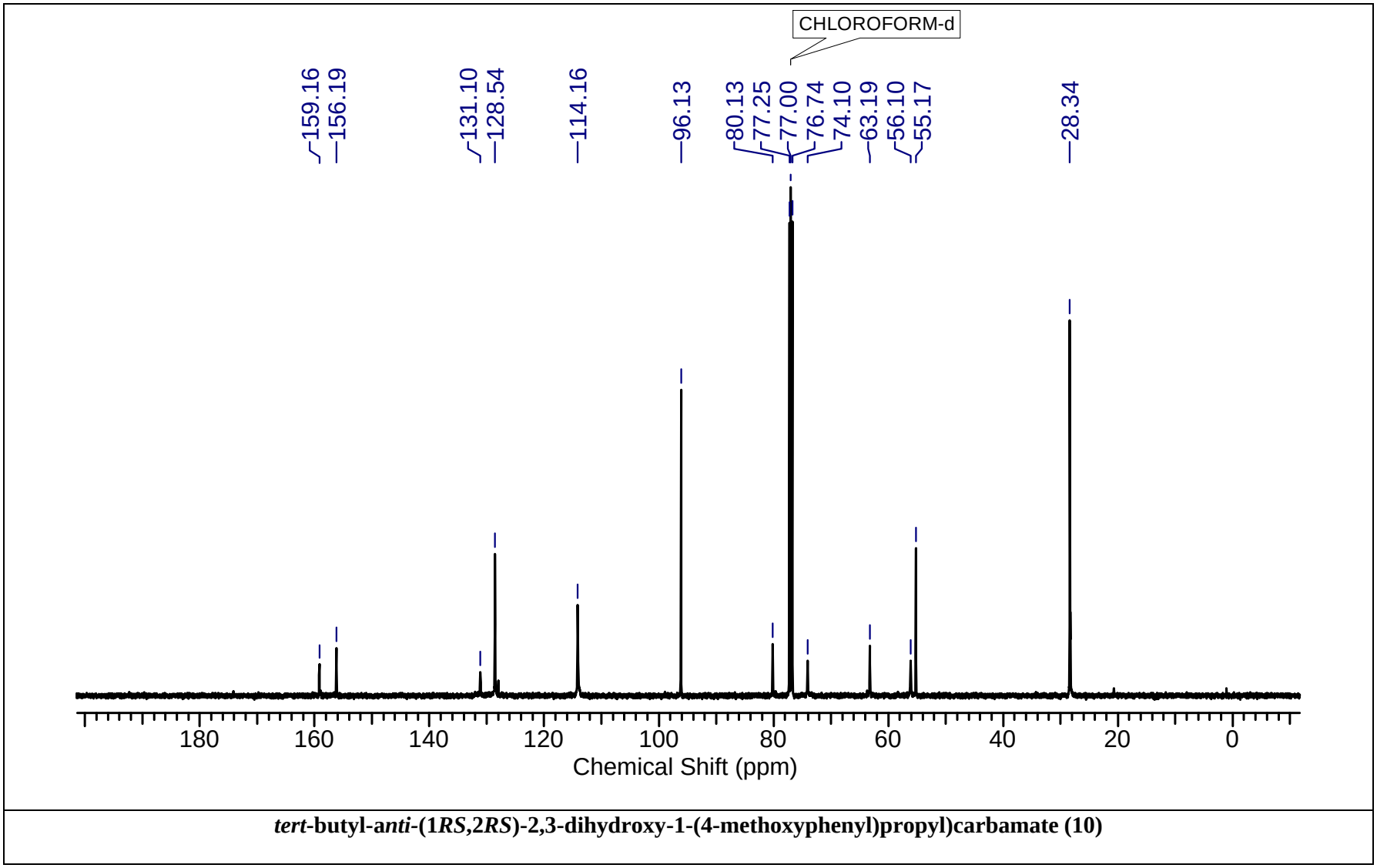

CHLOROFORM-d
136.16
129.02
128.84
127.83
77.00
74.04
67.07
62.85
150
100
50
0
Chemical Shift (ppm)


CHLOROFORM-d
7.29
7.27
7.25
6.94
6.90
4.53
4.50
3.82
3.77
3.74
3.68
3.67
3.65
3.37
3.33
3.29
2.87
N3
OH
OH
O
3n
TMS
2.15
2.04
0.85
4.15
2.29
1.75
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

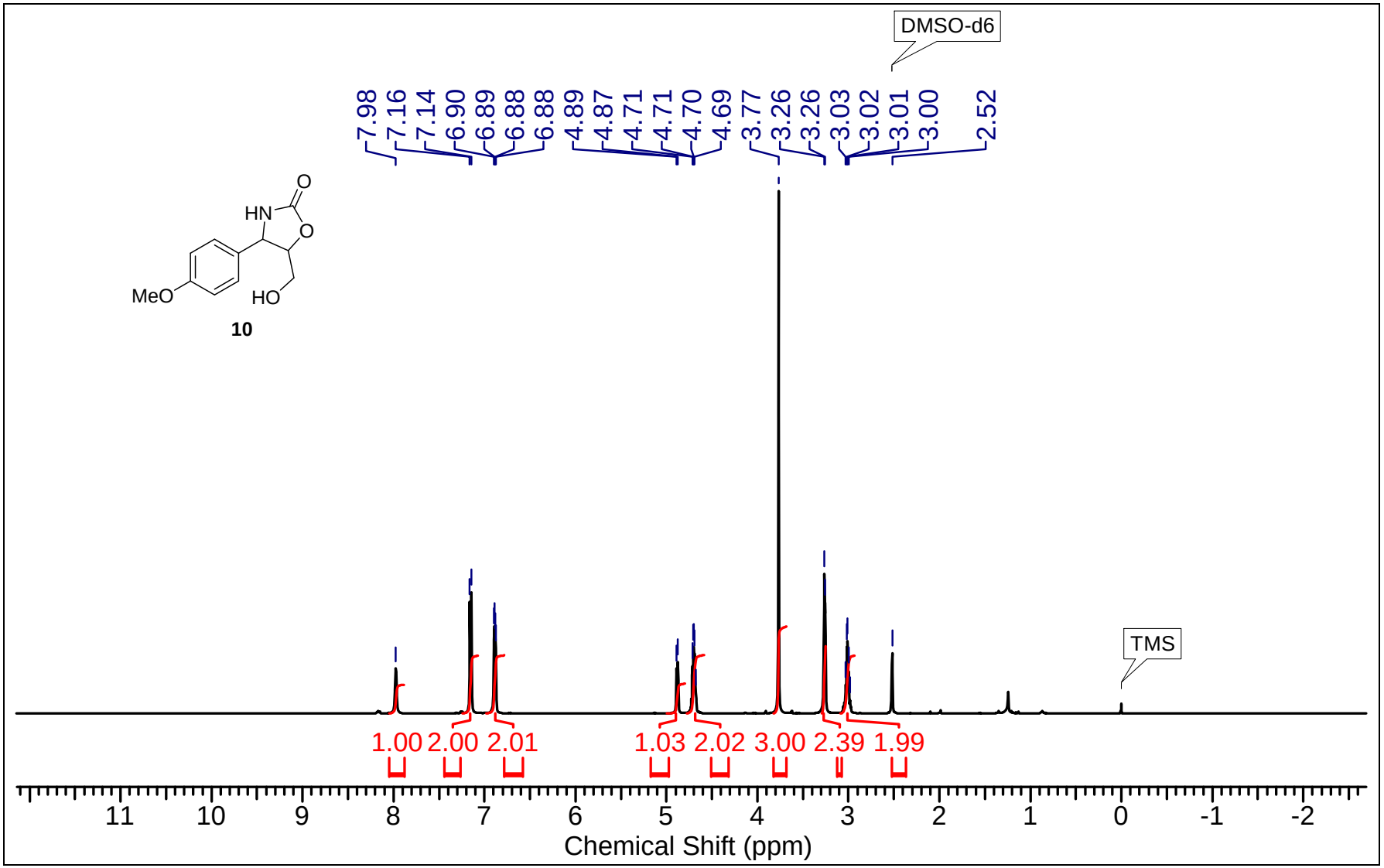


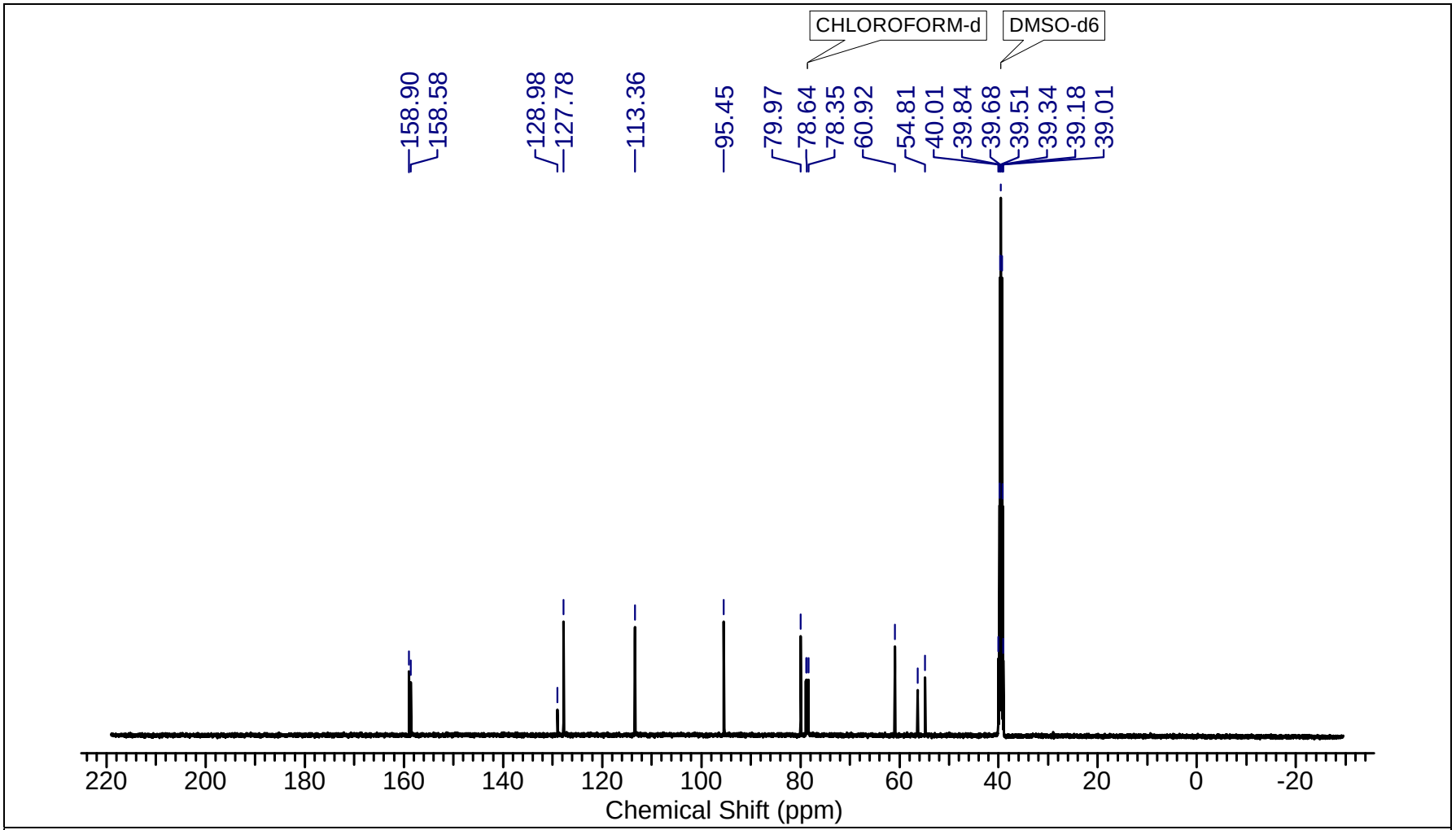
***anti*-(2*RS*,3*RS*)-3-azido-3-(4-methoxyphenyl)propane-1,2-diol (3n)**


CHLOROFORM-d
TMS
7.19
7.15
7.14
6.79
6.78
5.29
4.55
3.71
3.54
3.11
3.11
1.34
0.00
N3
OH
OH
3m
2.18
2.12
0.80
1.07
3.97
4.00
10.32
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

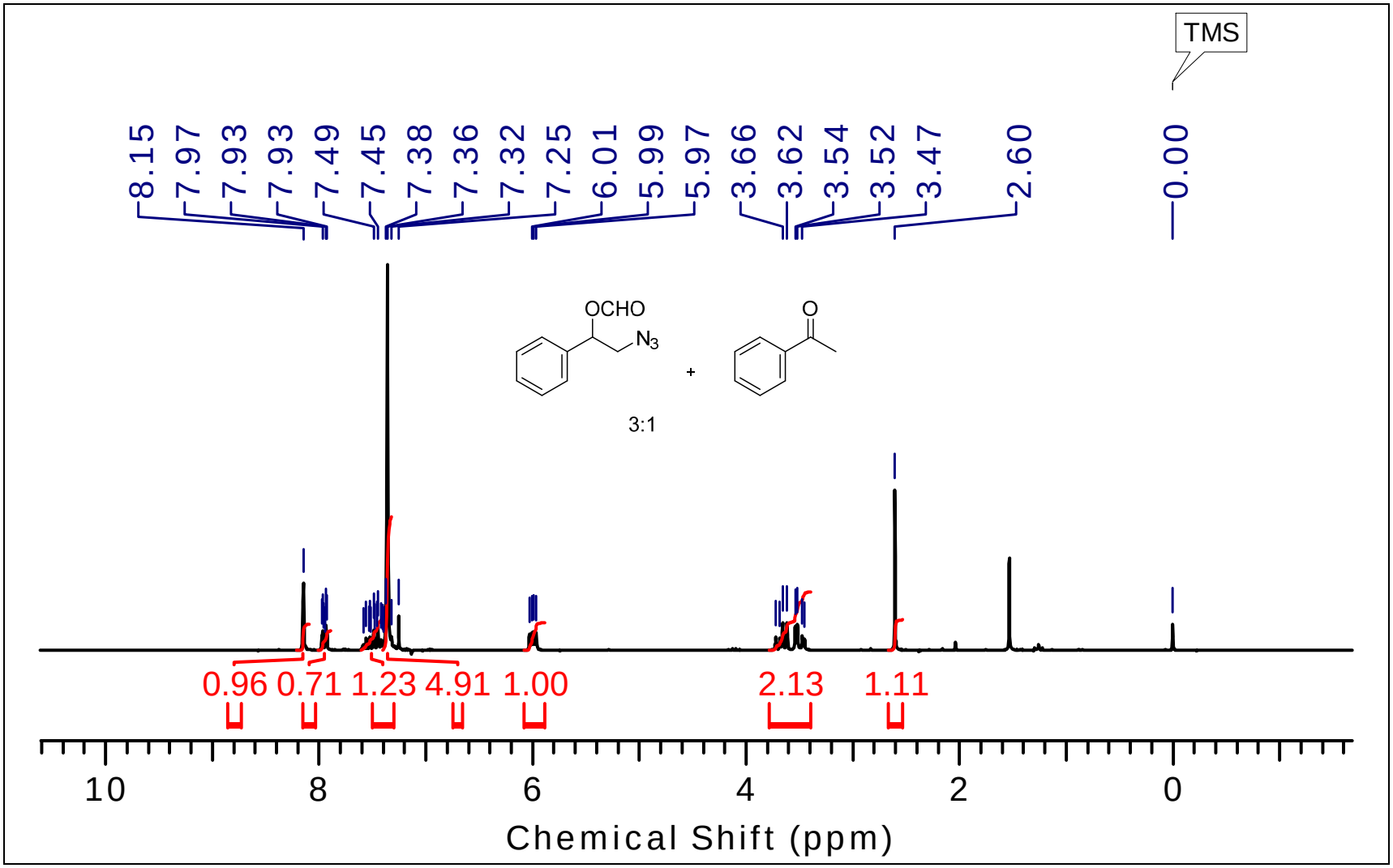


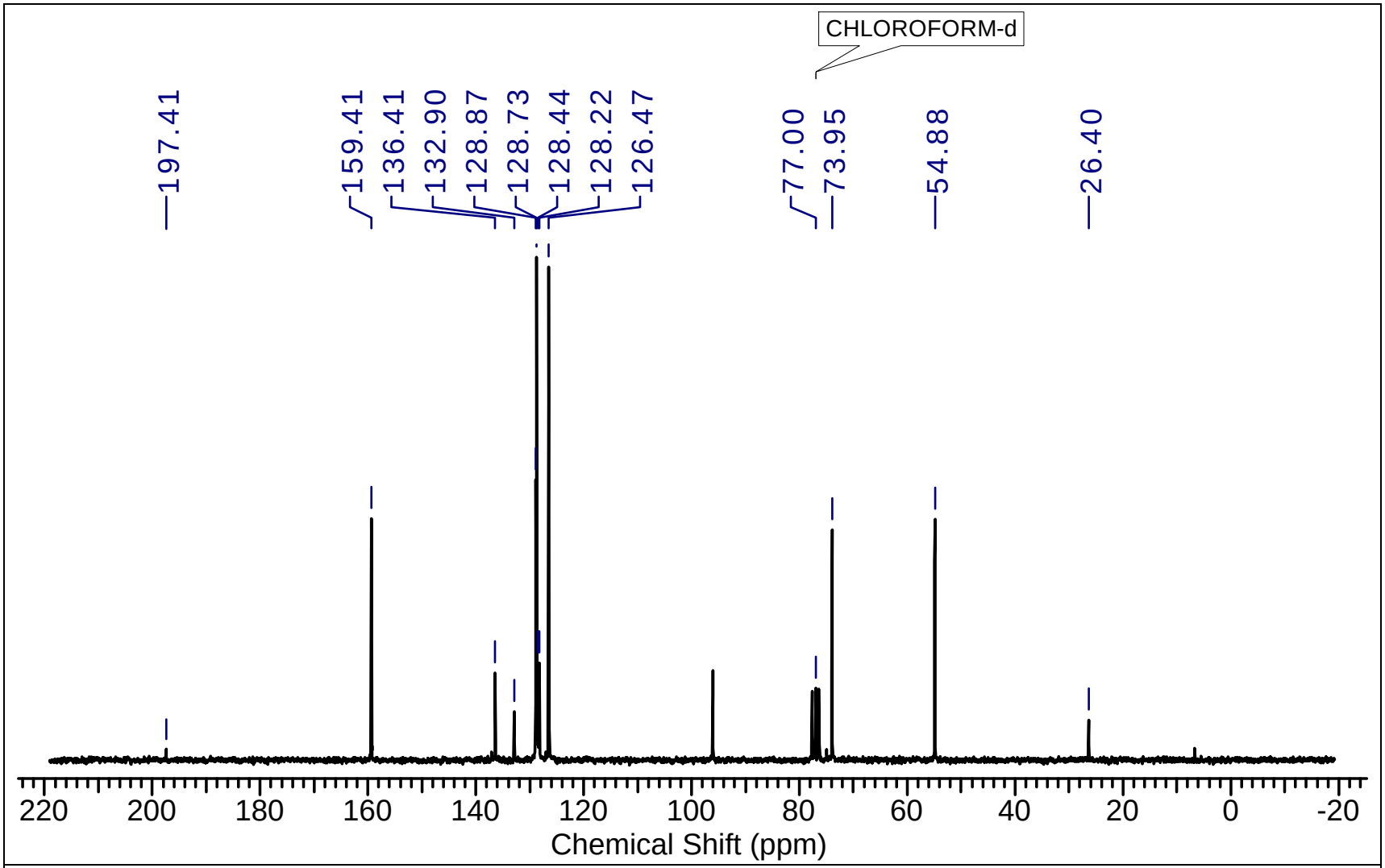
***tert*-butyl-a*nti*-(1*RS*,2*RS*)-2,3-dihydroxy-1-(4-methoxyphenyl)propyl)carbamate (10)**


DMSO-d6
7.98
7.16
7.14
6.90
6.89
6.88
6.88
4.89
4.87
4.71
4.71
4.70
4.69
3.77
3.26
3.26
3.03
3.02
3.01
3.00
2.52
HN
O
O
MeO
HO
10
TMS
1.00
2.00
2.01
1.03
2.02
3.00
2.39
1.99
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
Chemical Shift (ppm)

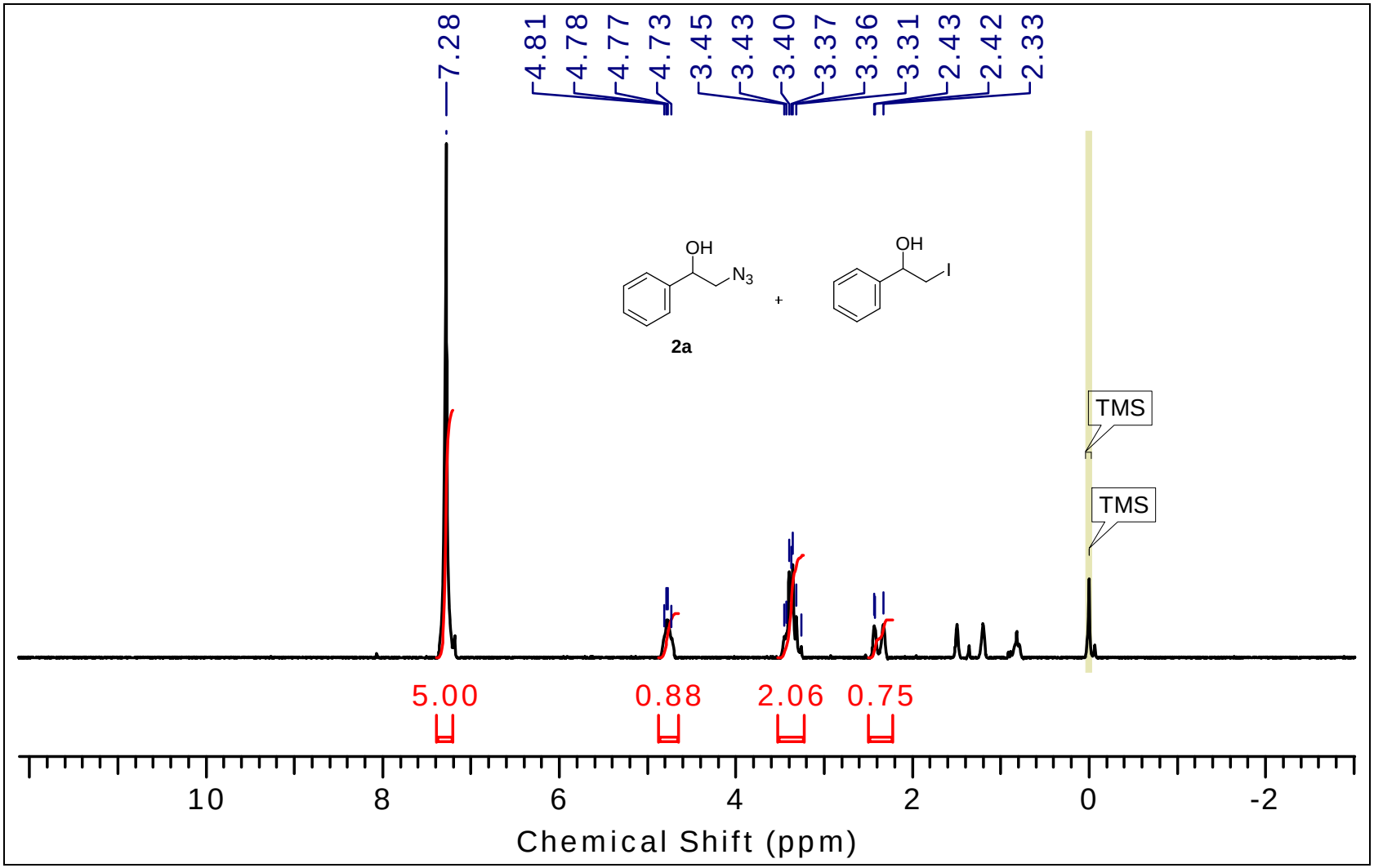


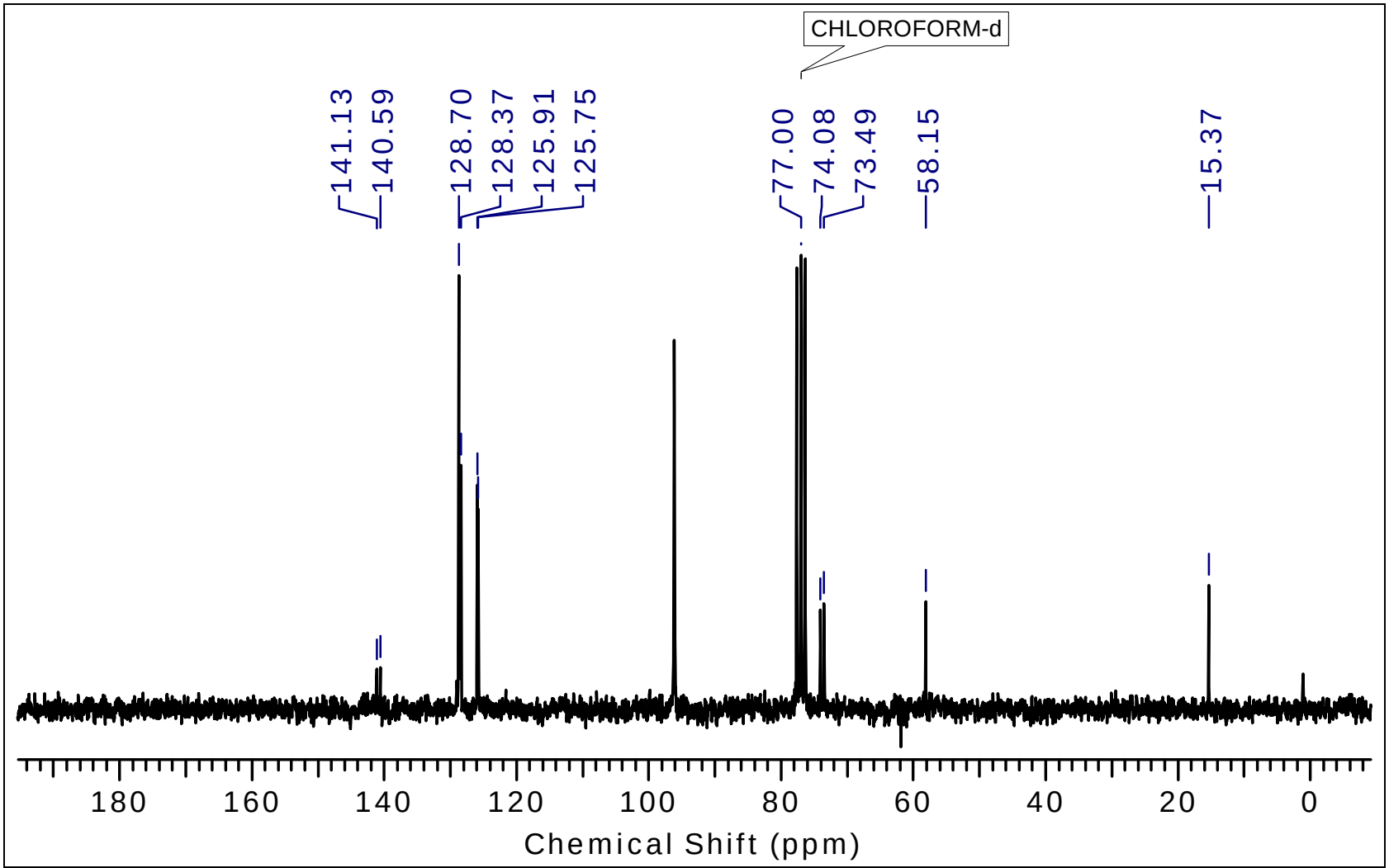
(±)-Cytoxazone (11)


TMS
8.15
7.97
7.93
7.93
7.49
7.45
7.38
7.36
7.32
7.25
6.01
5.99
5.97
3.66
3.62
3.54
3.52
3.47
2.60
0.00
OCHO
N3
+
O
3:1
0.96
0.71
1.23
4.91
1.00
2.13
1.11
10
8
6
4
2
0
Chemical Shift (ppm)

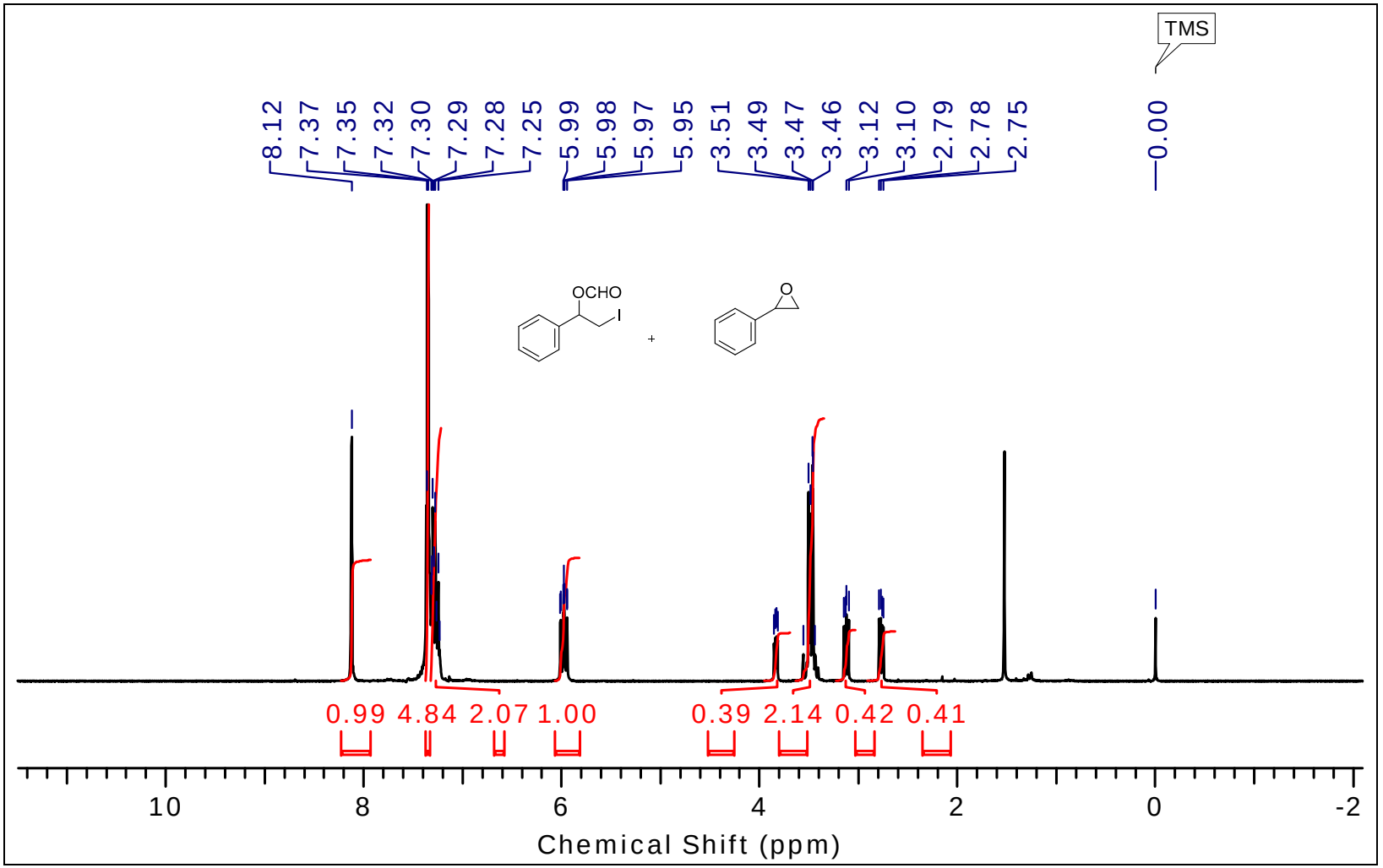


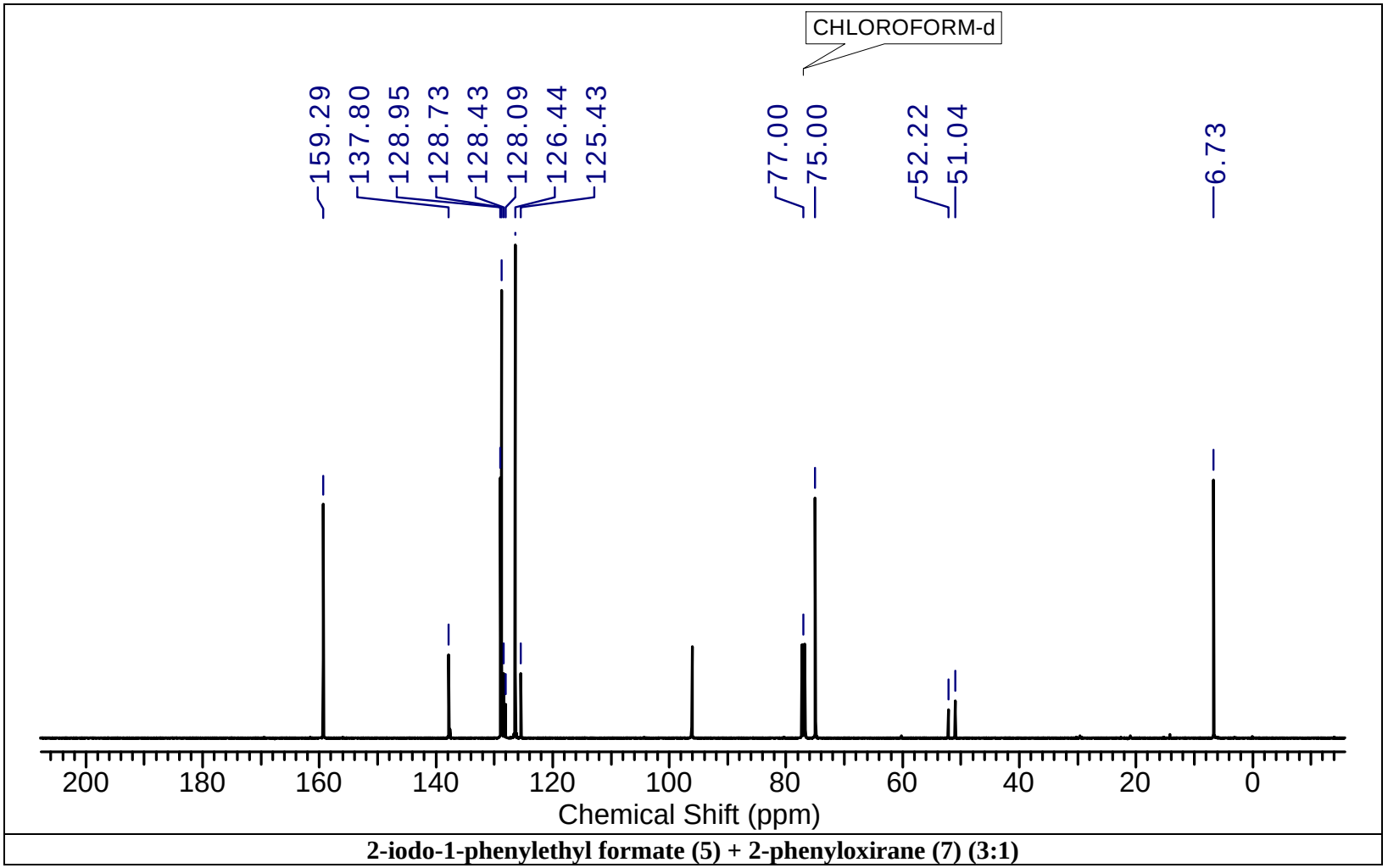
2-azido-1-phenylethyl formate (8) + acetophenone (3:1)


7.28
4.81
4.78
4.77
4.73
3.45
3.43
3.40
3.37
3.36
3.31
2.43
2.42
2.33
OH
N3
+
OH
I
2a
TMS
TMS
5.00
0.88
2.06
0.75
10
8
6
4
2
0
-2
Chemical Shift (ppm)

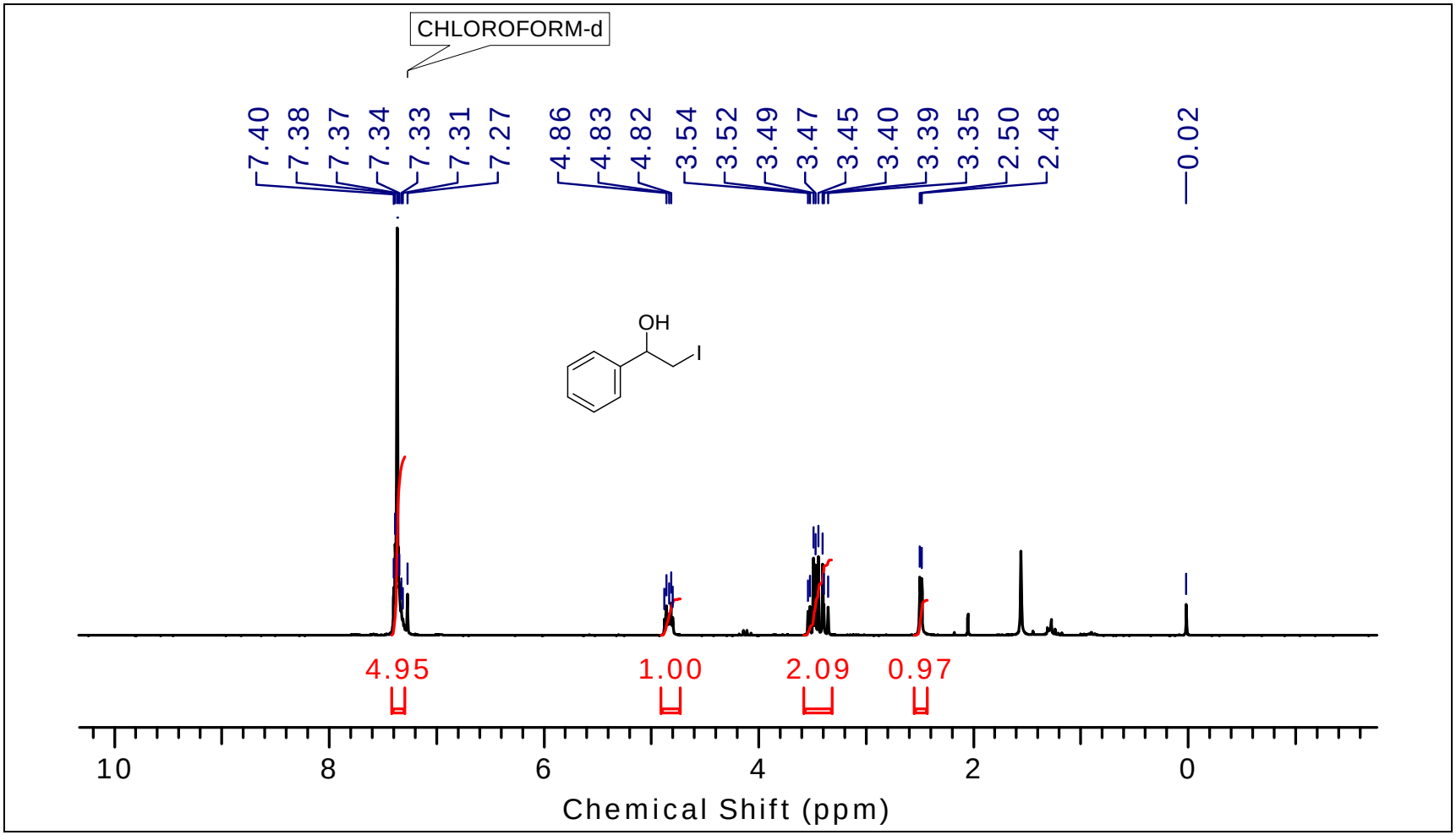


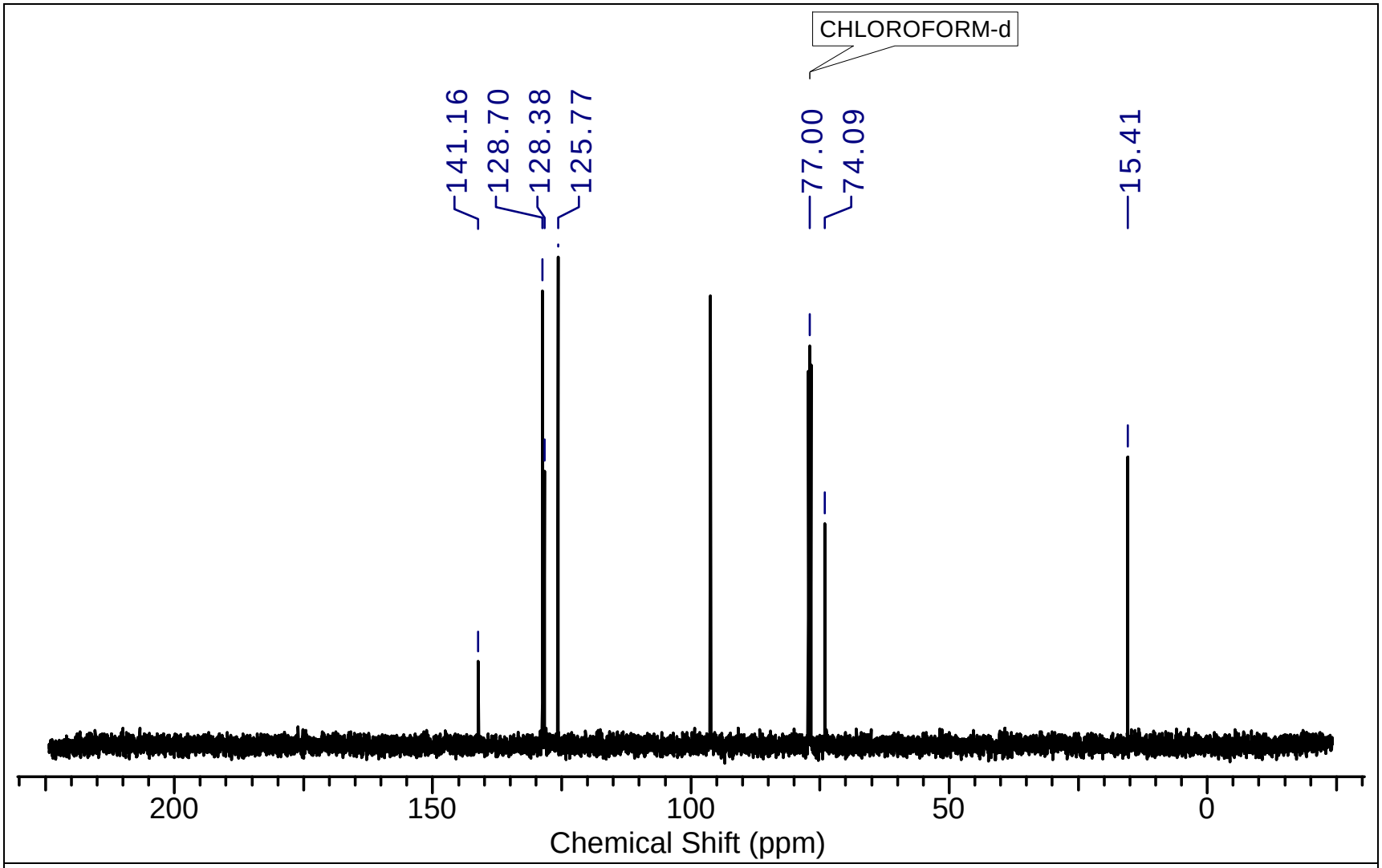
2-azido-1-phenylethan-1-ol(2a) + 2-iodo-1-phenylethan-1-ol (6)


TMS
8.12
7.37
7.35
7.32
7.30
7.29
7.28
7.25
5.99
5.98
5.97
5.95
3.51
3.49
3.47
3.46
3.12
3.10
2.79
2.78
2.75
0.00
OCHO
I
O
0.99
4.84
2.07
1.00
0.39
2.14
0.42
0.41
10
8
6
4
2
0
-2
Chemical Shift (ppm)

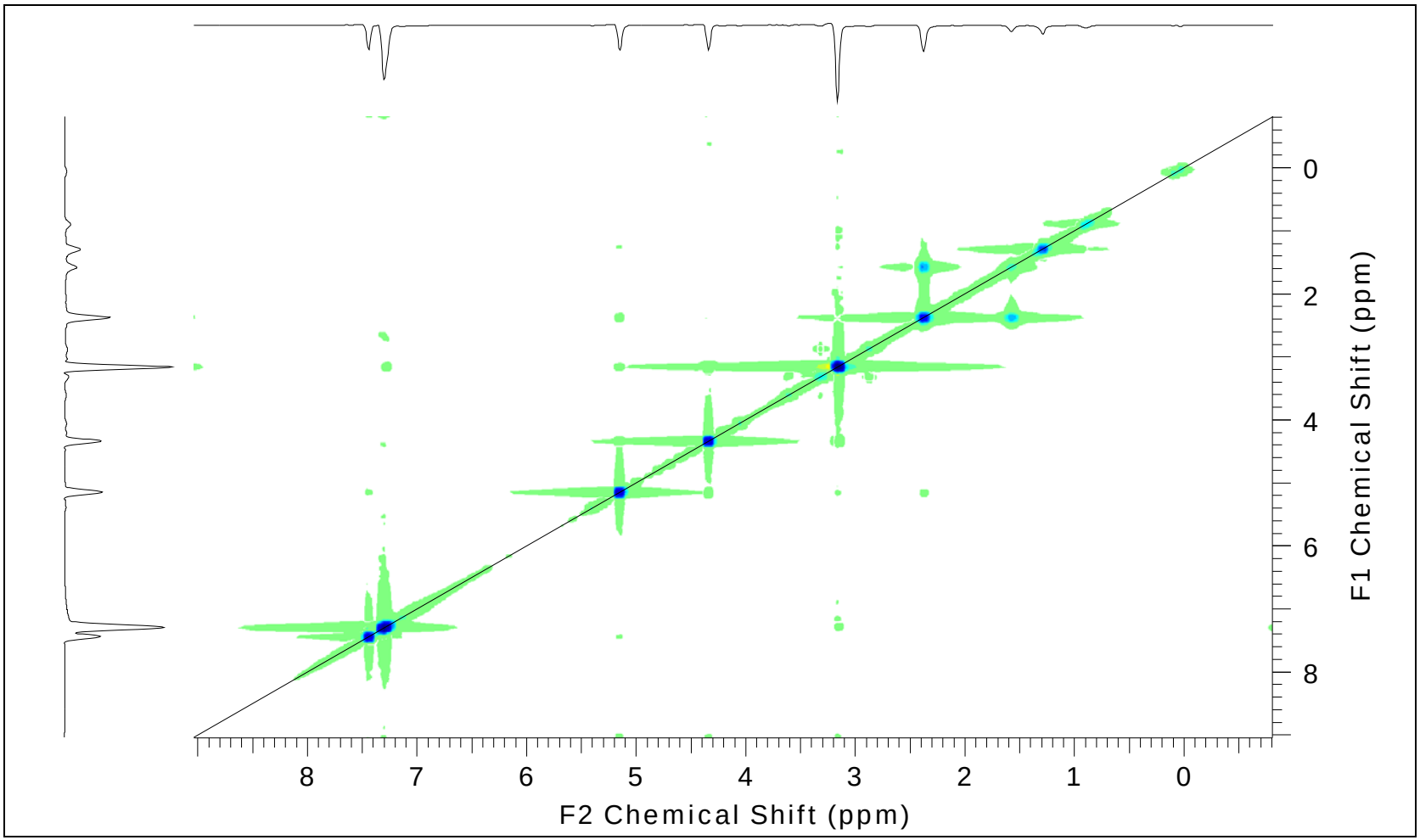


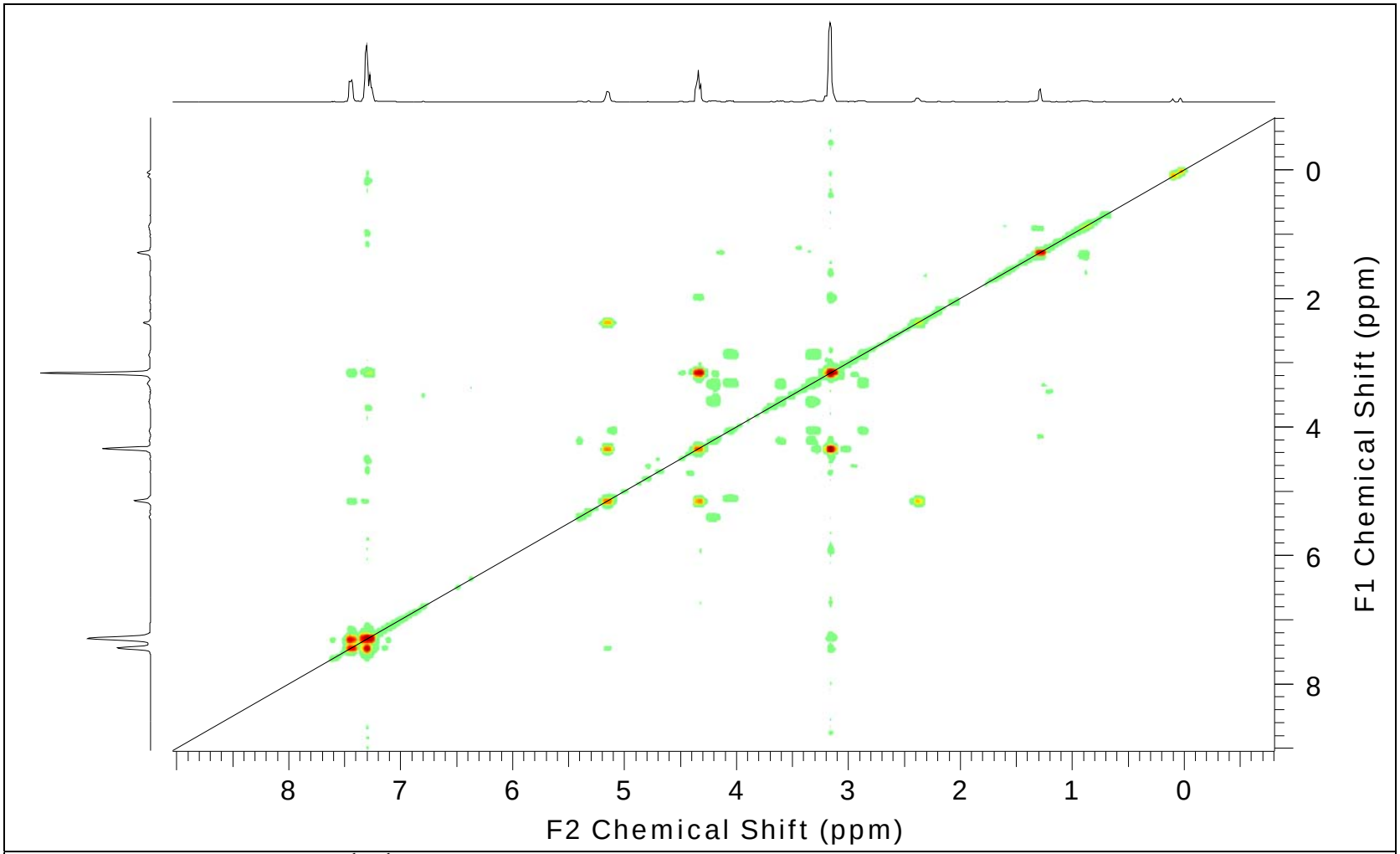
2-iodo-1-phenylethyl formate (5) + 2-phenyloxirane (7) (3:1)


CHLOROFORM-d
7.40
7.38
7.37
7.34
7.33
7.31
7.27
4.86
4.83
4.82
3.54
3.52
3.49
3.47
3.45
3.40
3.39
3.35
2.50
2.48
0.02
OH
I
4.95
1.00
2.09
0.97
10
8
6
4
2
0
Chemical Shift (ppm)

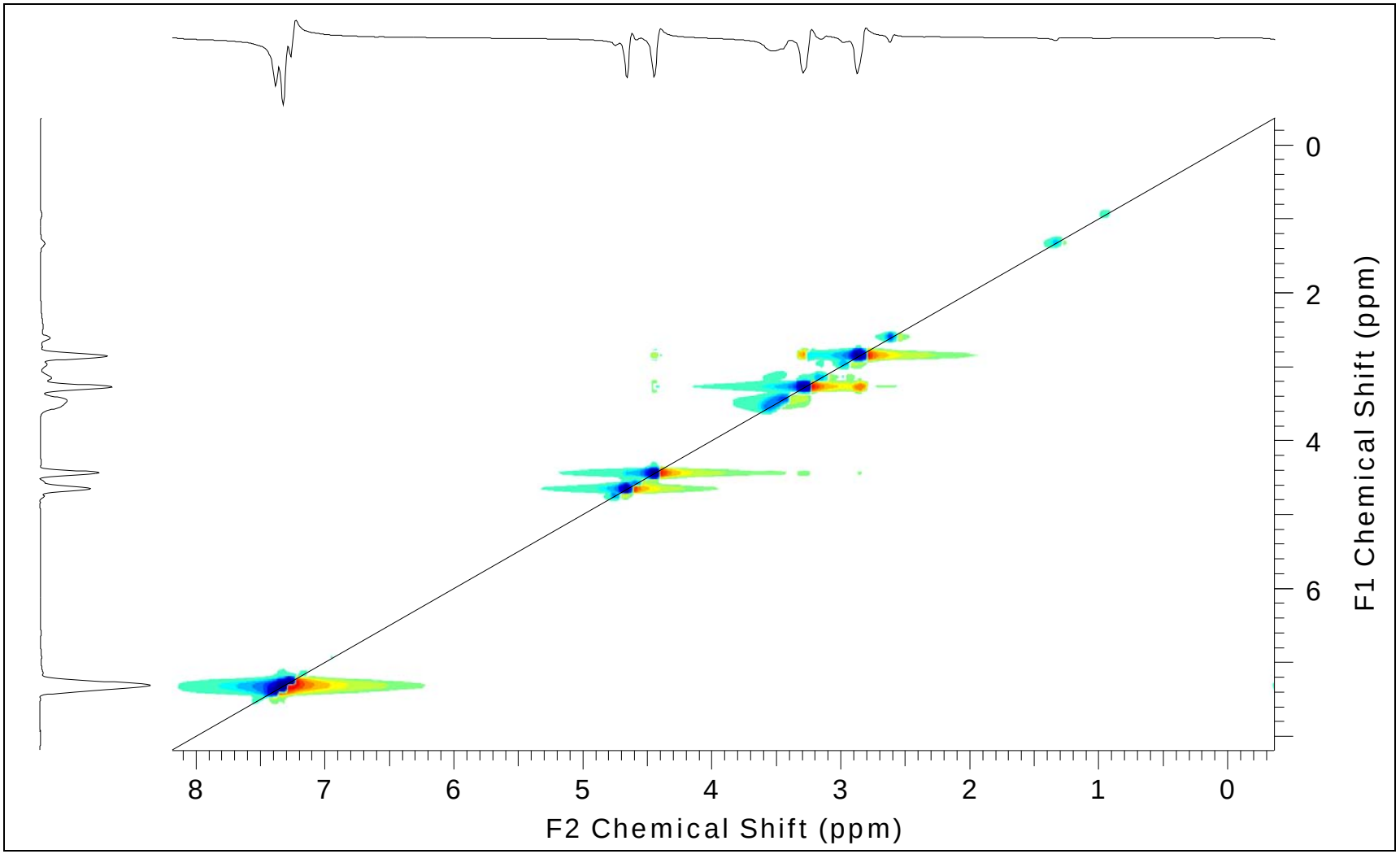


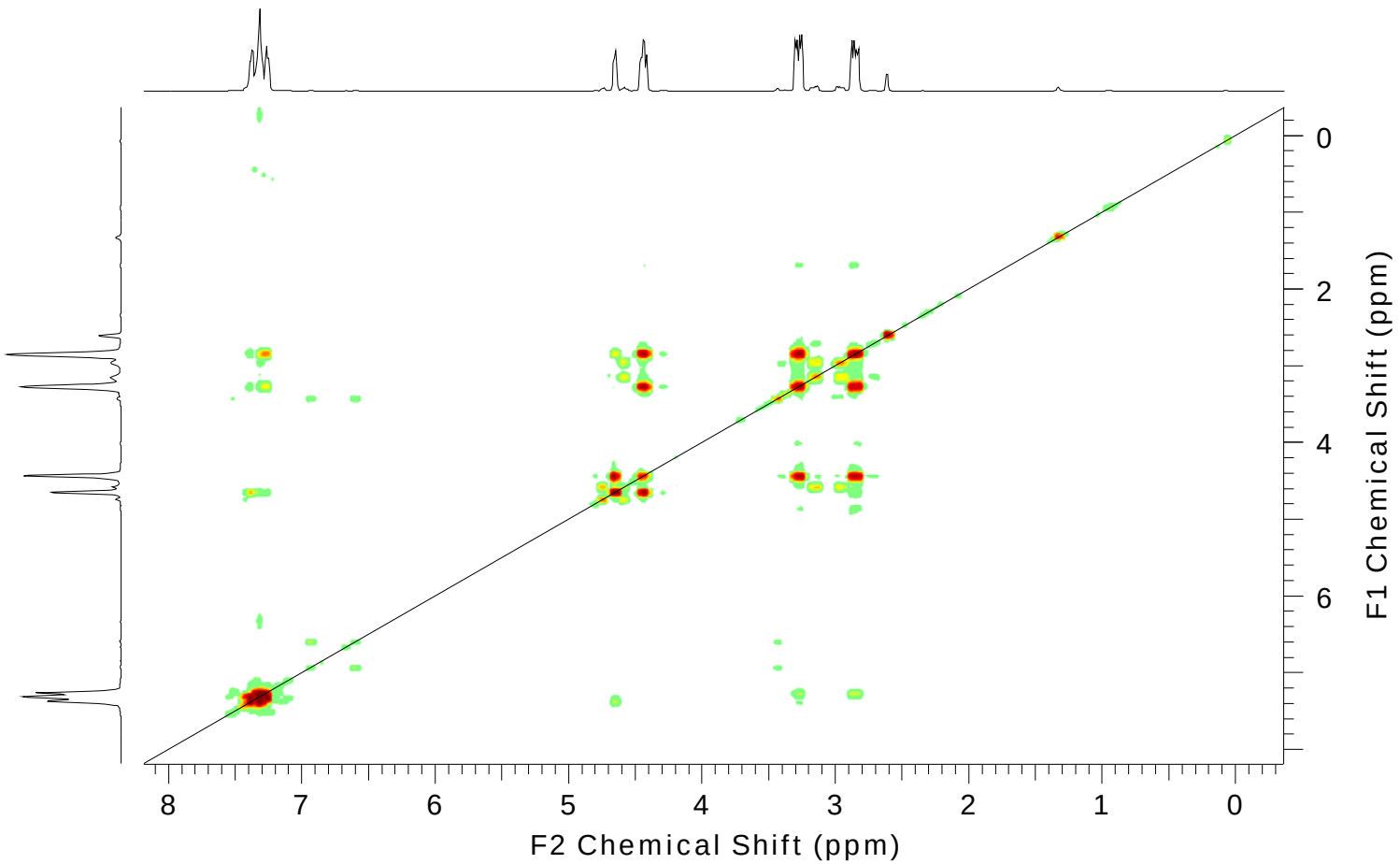
2-iodo-1-phenylethan-1-ol (6)


0
2
4
6
8
F1 Chemical Shift (ppm)
8
7
6
5
4
3
2
1
0
F2 Chemical Shift (ppm)

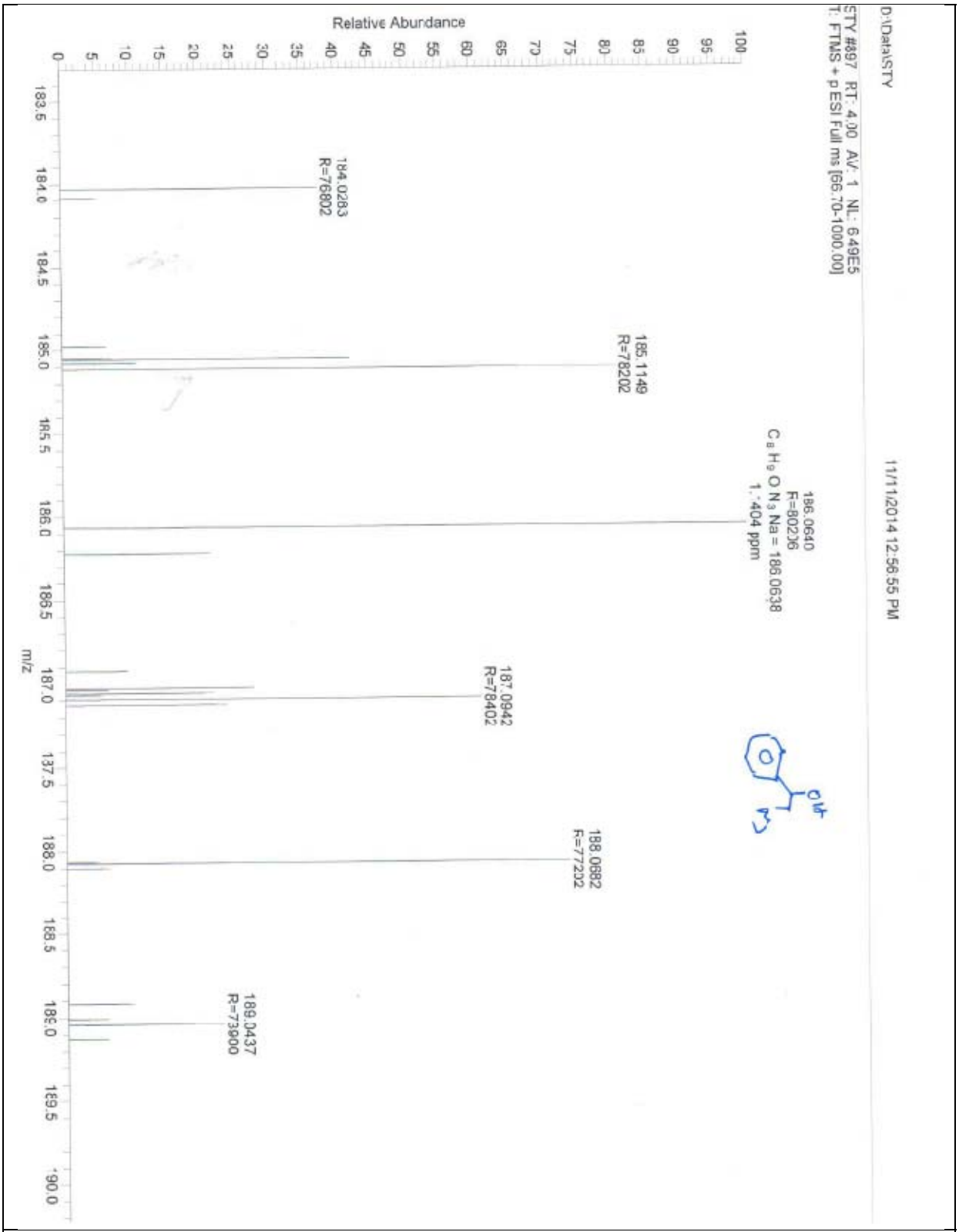


NOESY and ^{1}H ^{1}HCOSY spectra of *Syn*(1*RS*,2*SR*)-2-azido-2,3-dihydro-1H-inden-1-ol (2k)

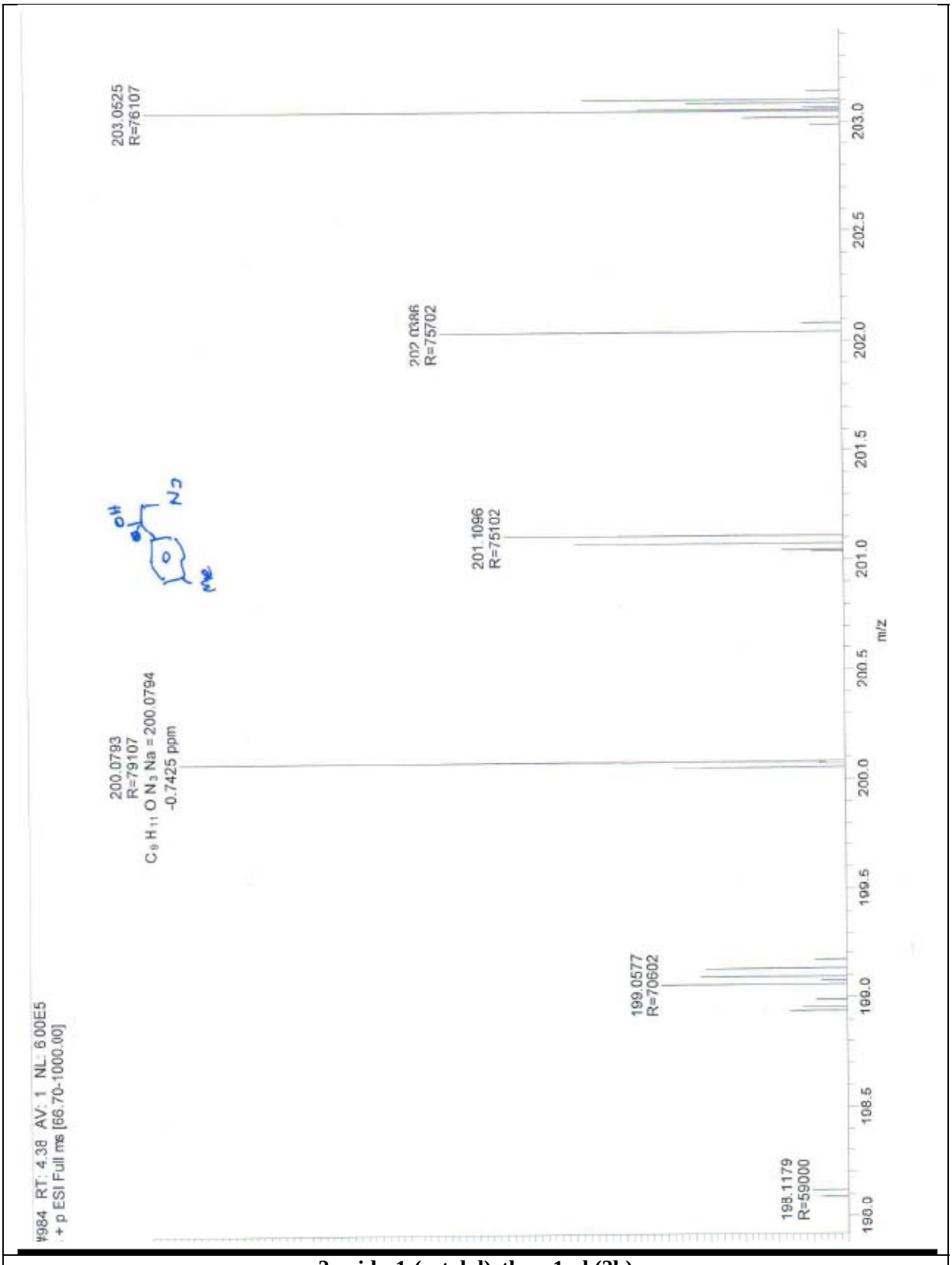

8
7
6
5
4
3
2
1
0
F2 Chemical Shift (ppm)
0
2
4
6
F1 Chemical Shift (ppm)



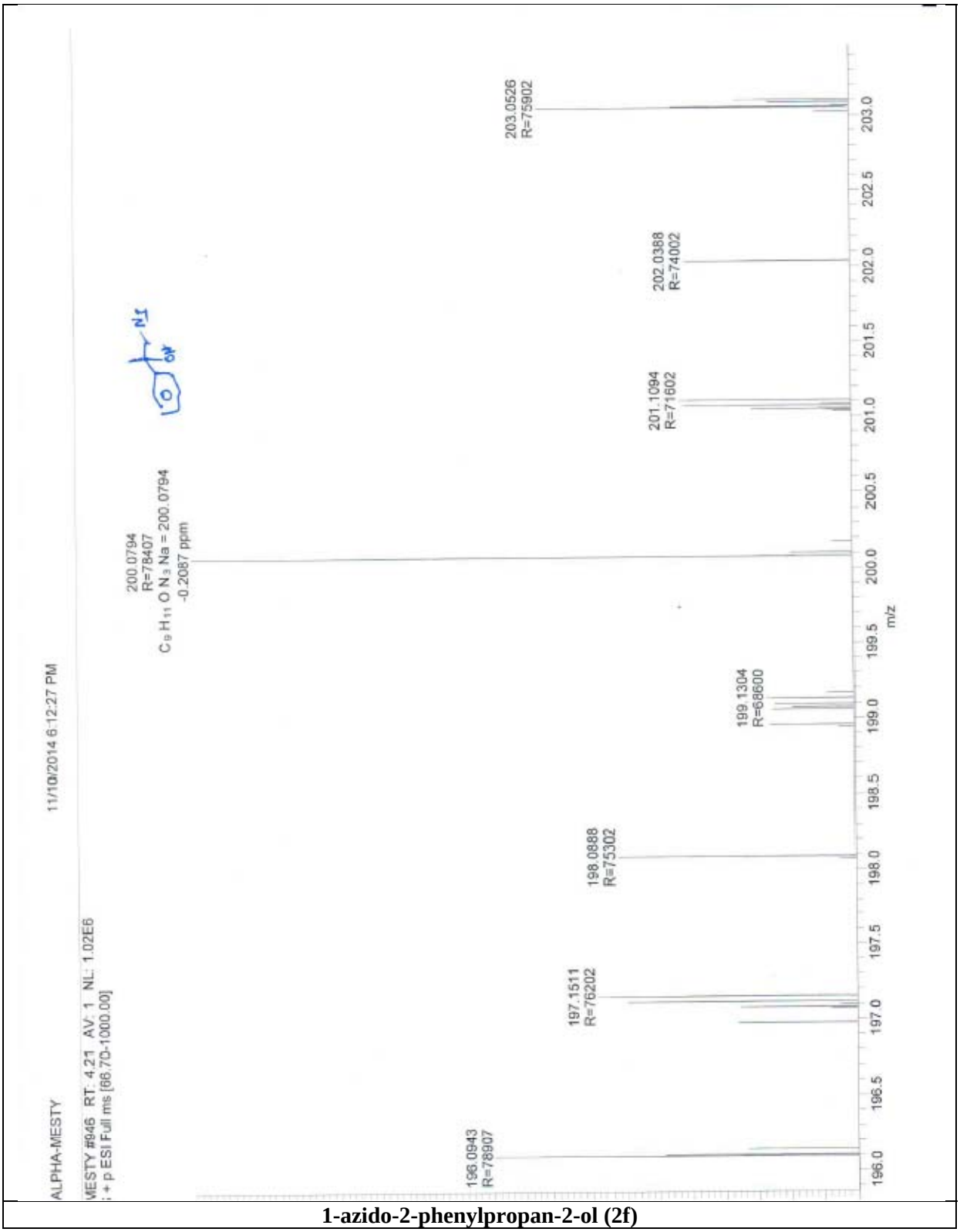
NOESY and ^{1}H ^{1}HCOSY spectra *Anti*(1*RS*,2*RS*)-1-azido-2,3-dihydro-1H-inden-2-ol (3k)



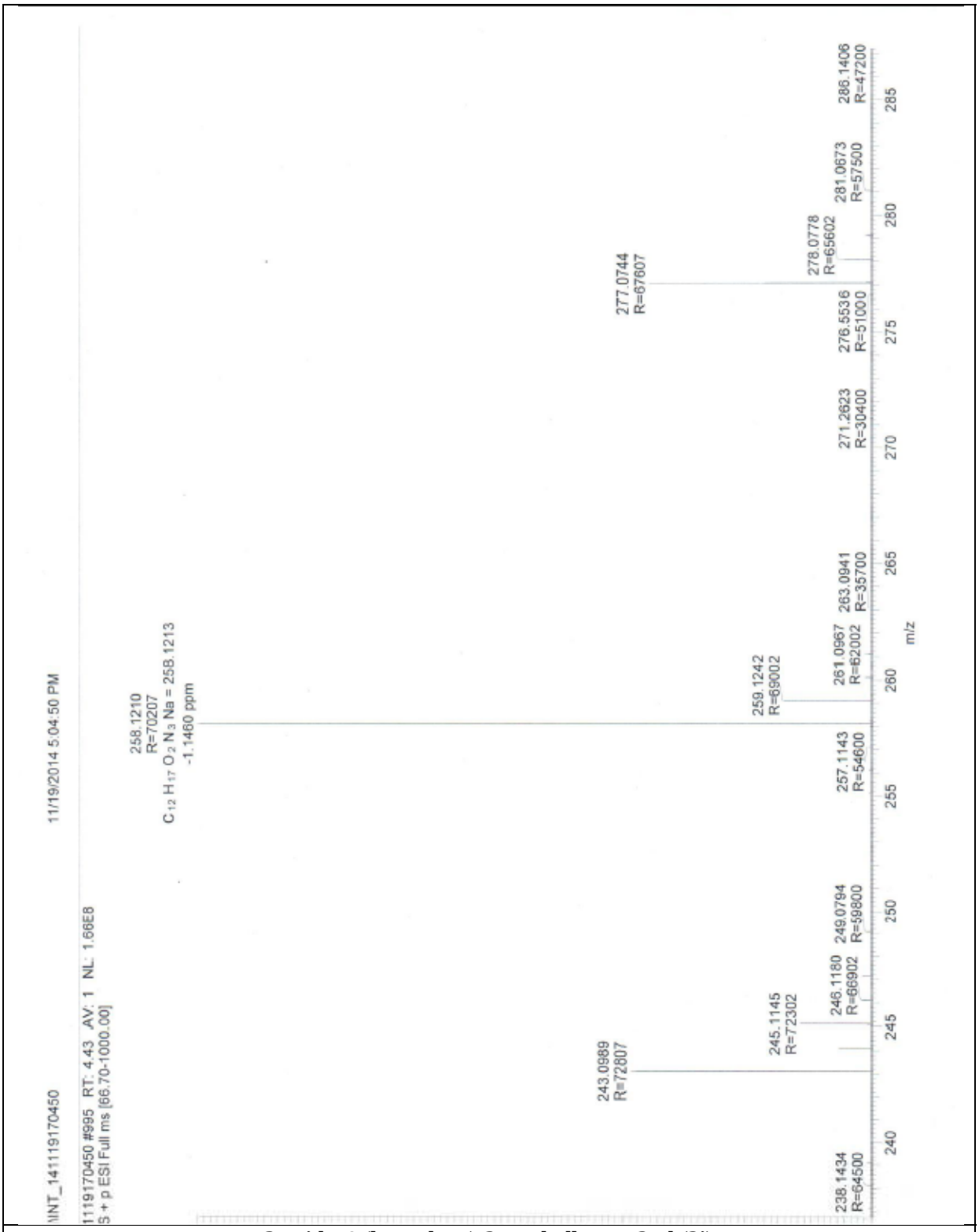
2-azido-1-phenylethan-1-ol (2a)



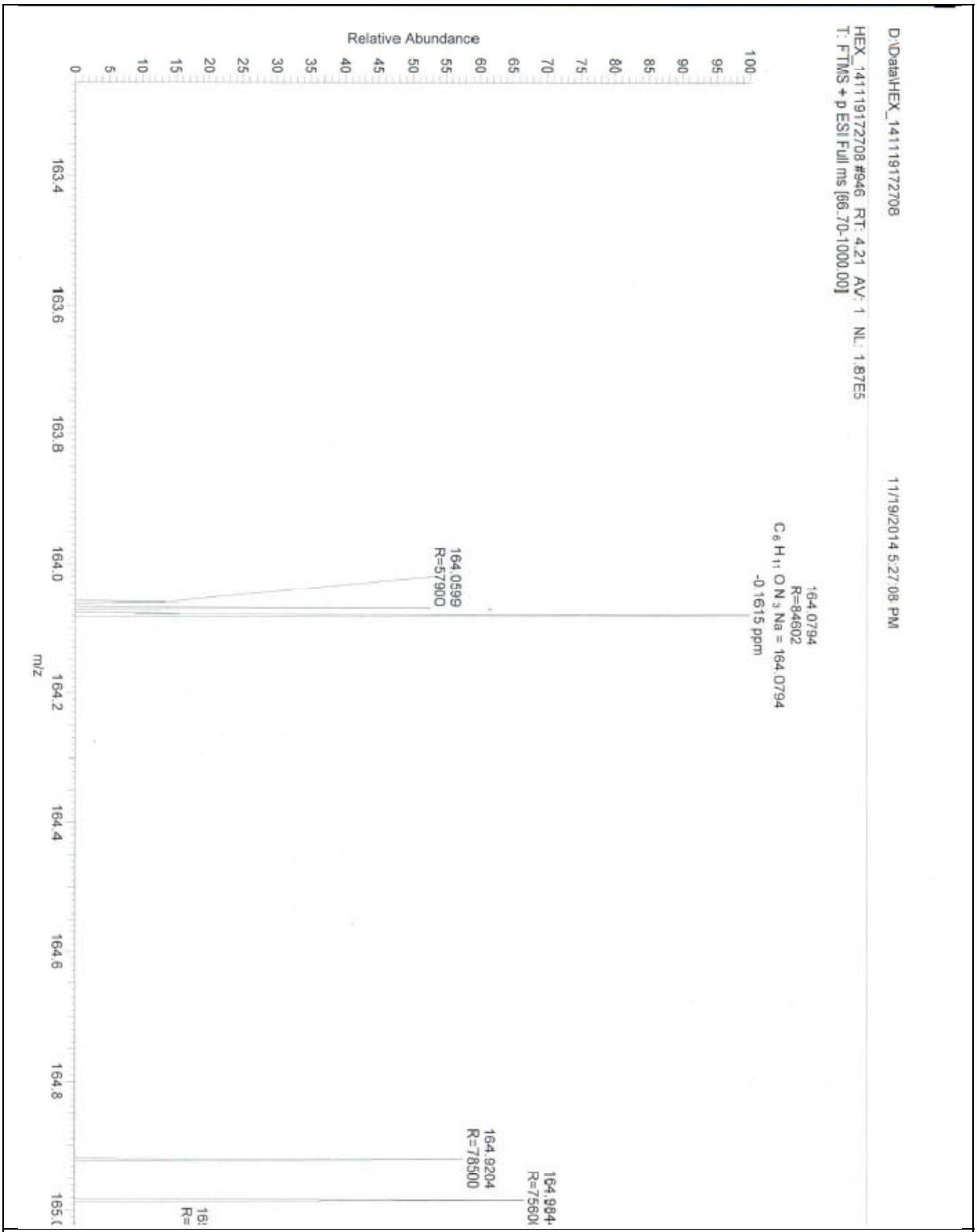
2-azido-1-(p-tolyl)ethan-1-ol (2b)



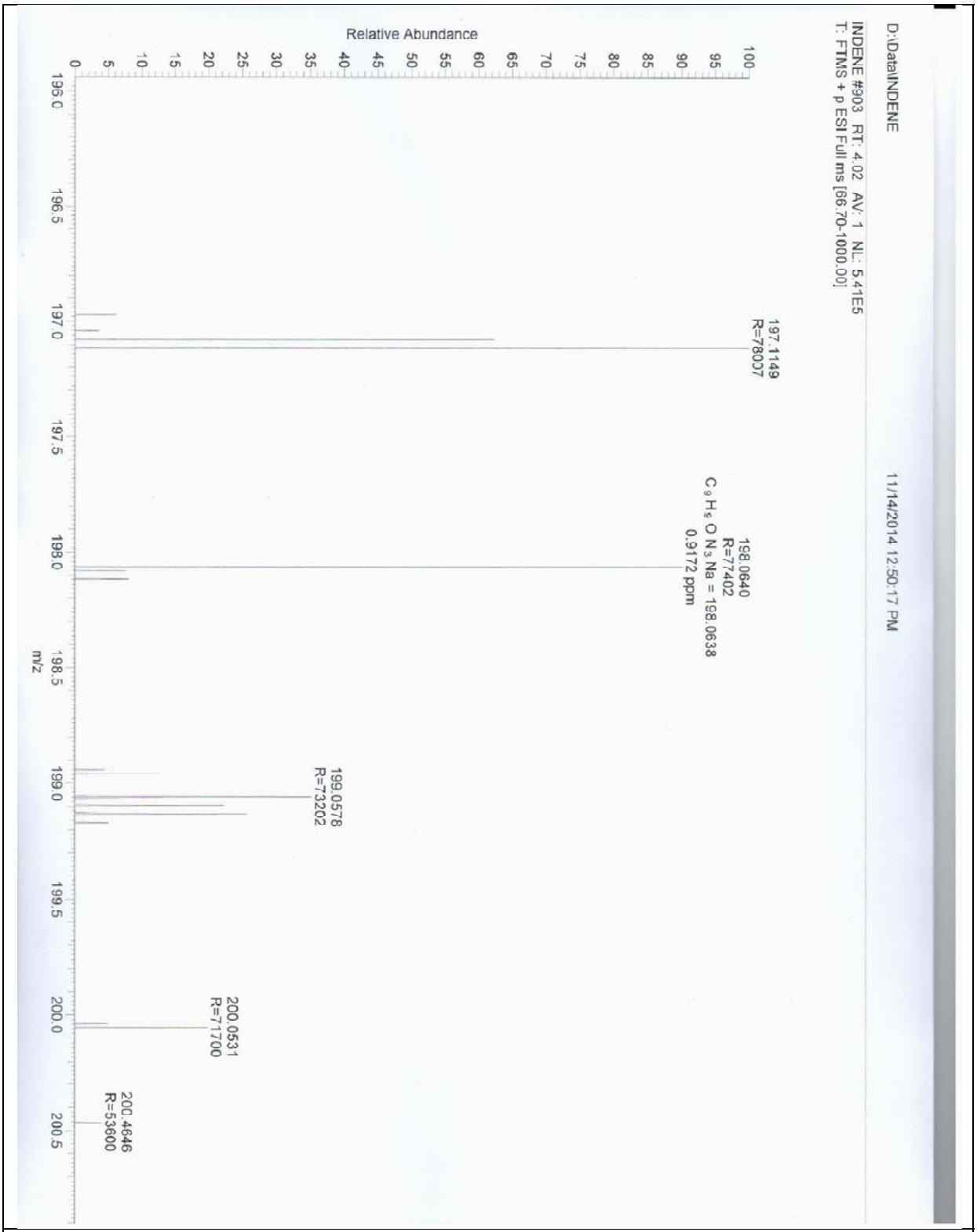
1-azido-2-phenylpropan-2-ol (2f)



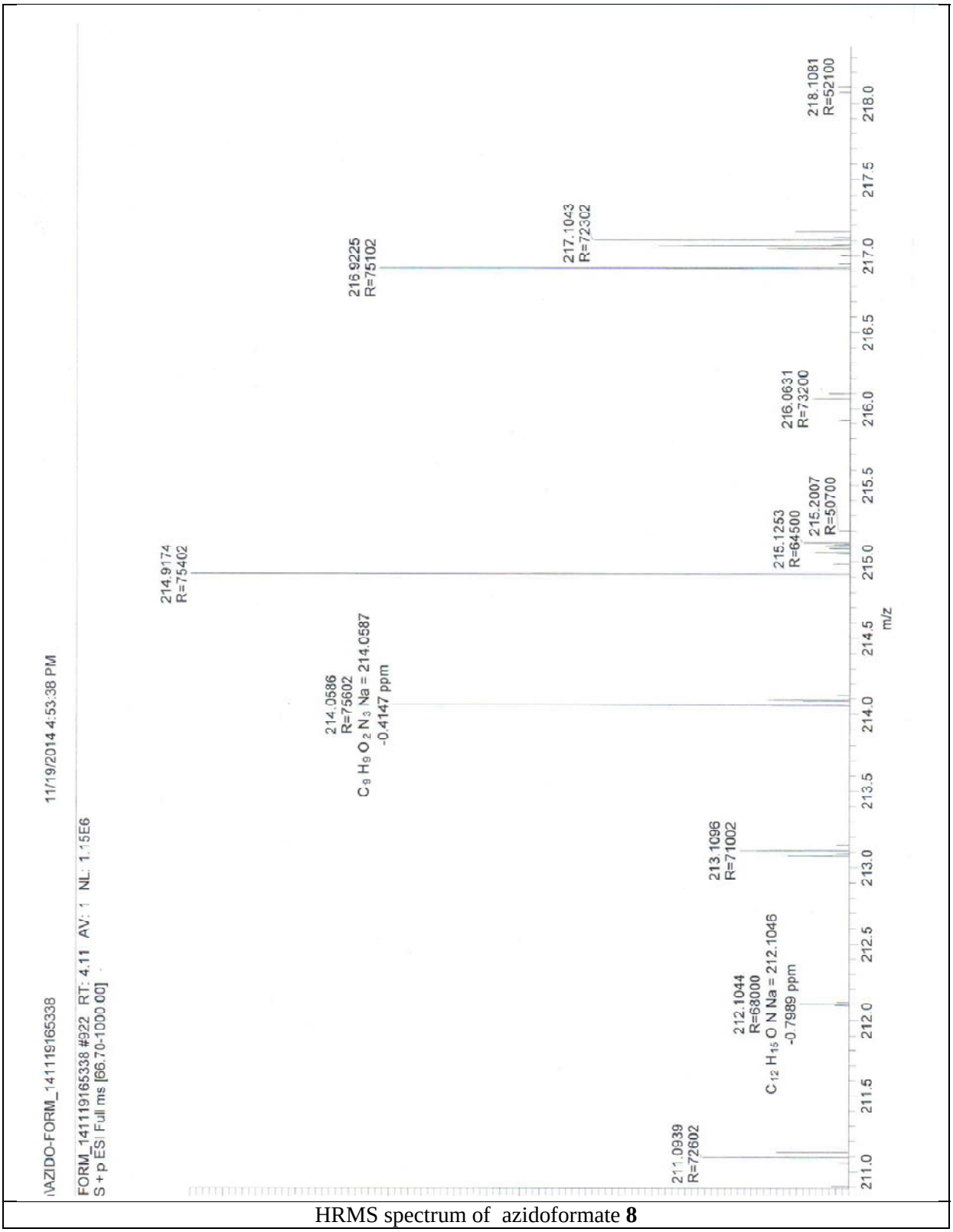
3-azido-4-(benzyloxy)-2-methylbutan-2-ol (2i)



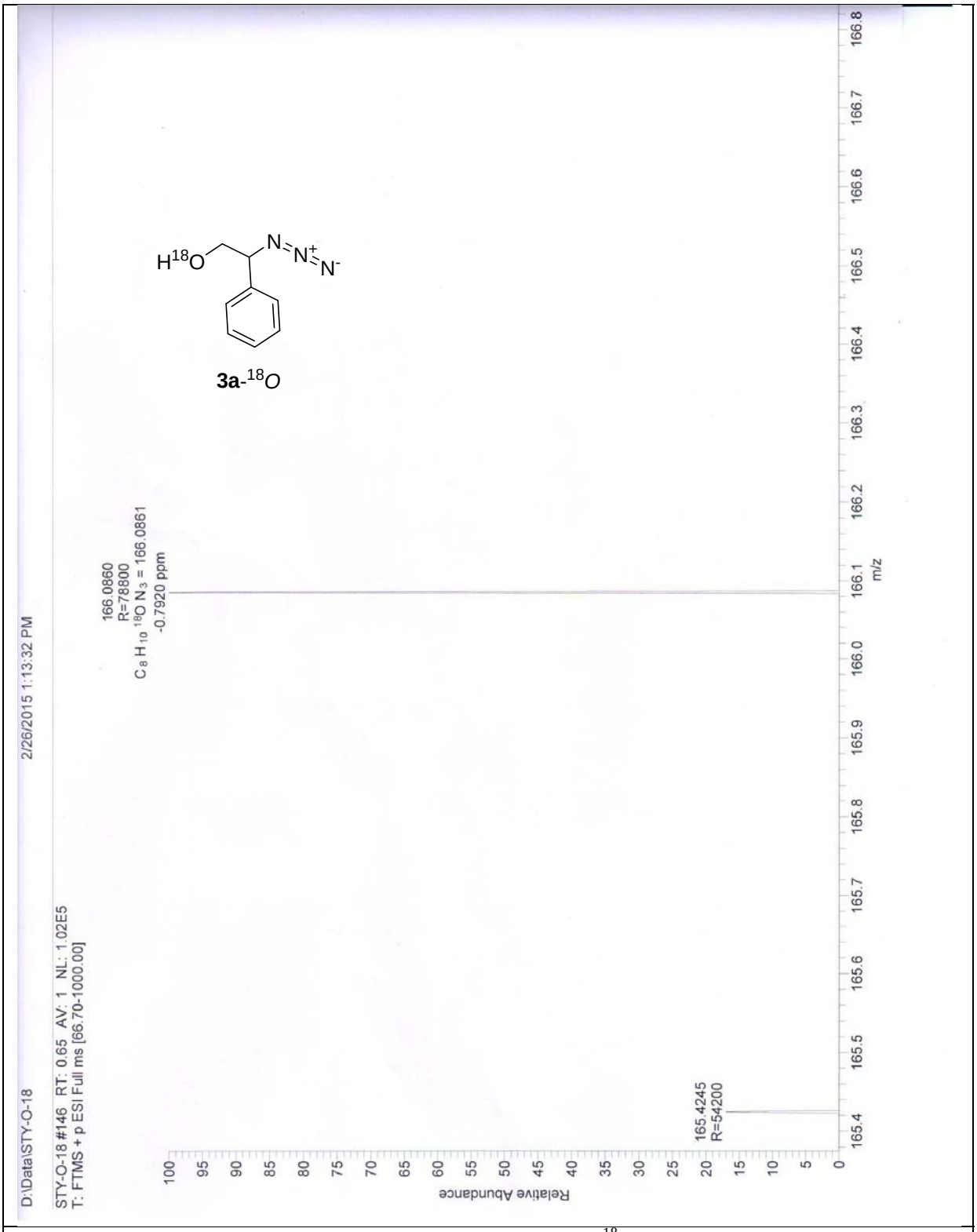
***cis*-(1*RS*,2*SR*)-2-azidocyclohexan-1-ol (2j)**



***cis*-(1*RS*,2*SR*)-2-azido-2,3-dihydro-1H-inden-1-ol (2k)**

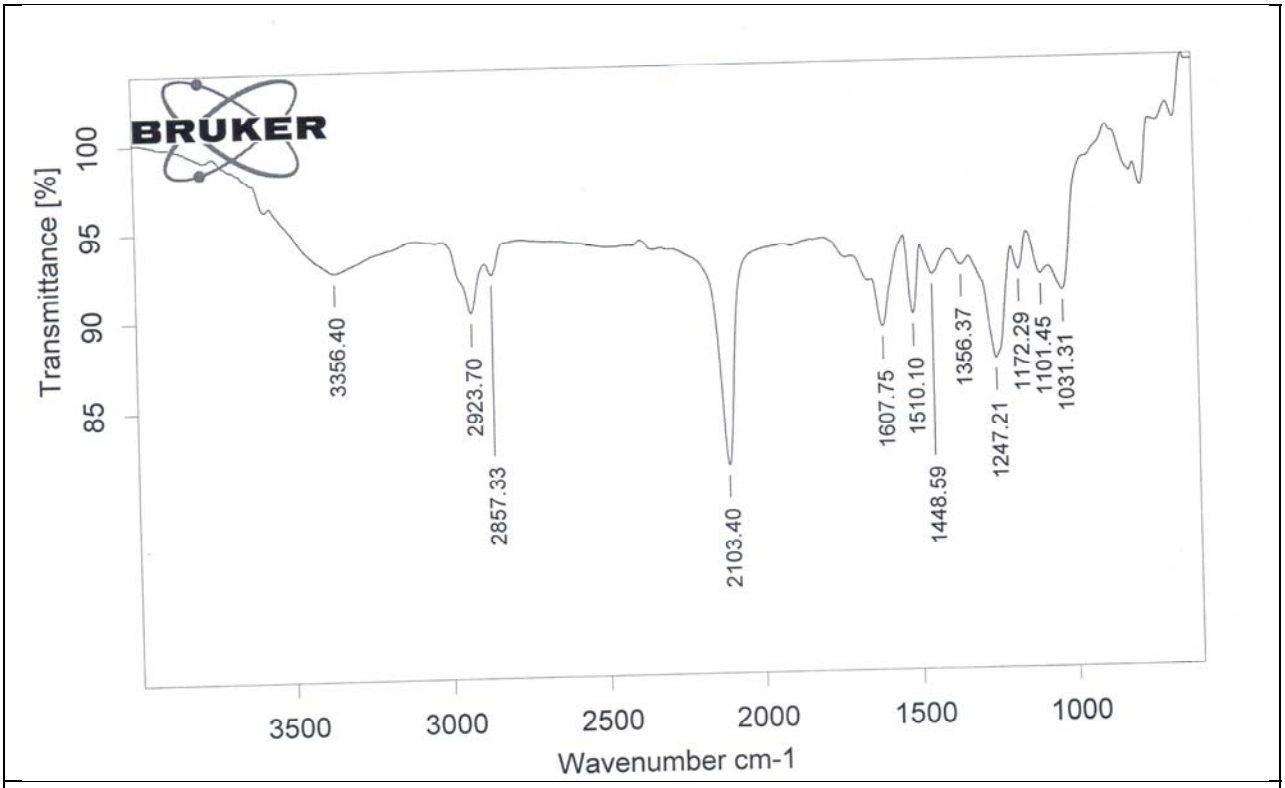


HRMS spectrum of azidoformate **8**

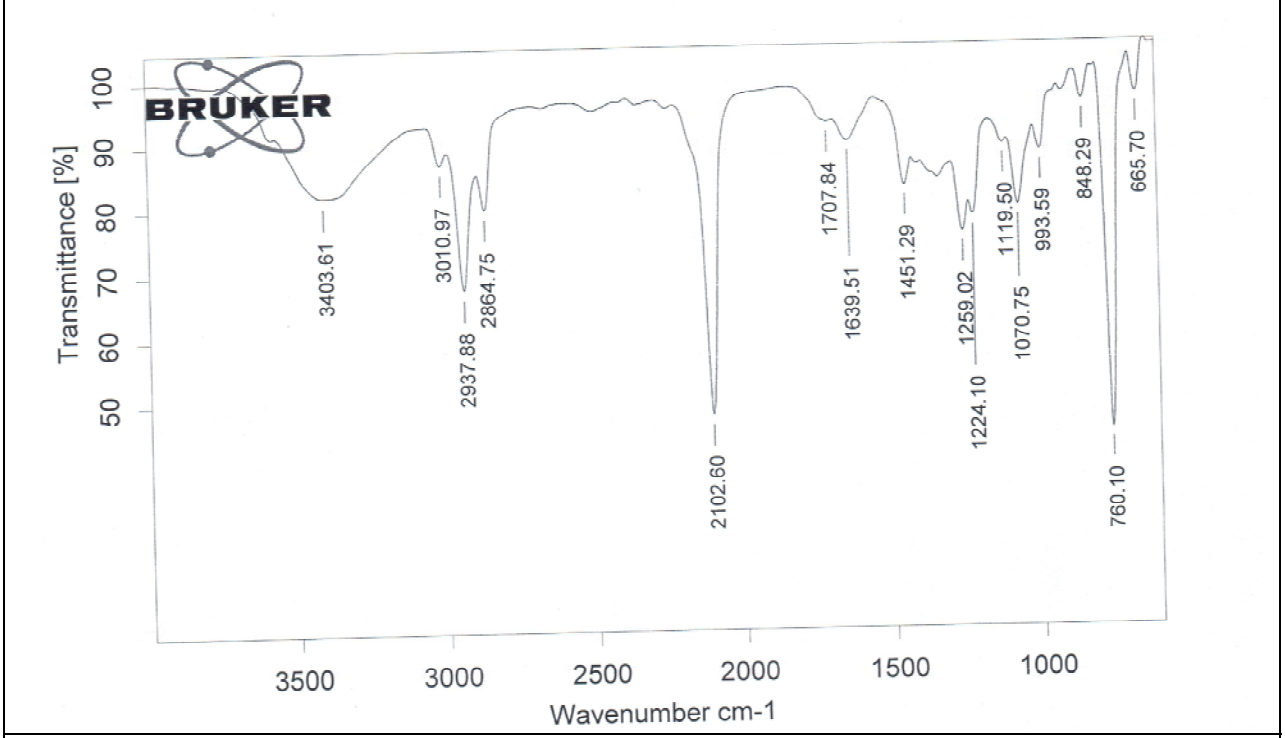


3a-^{18}O

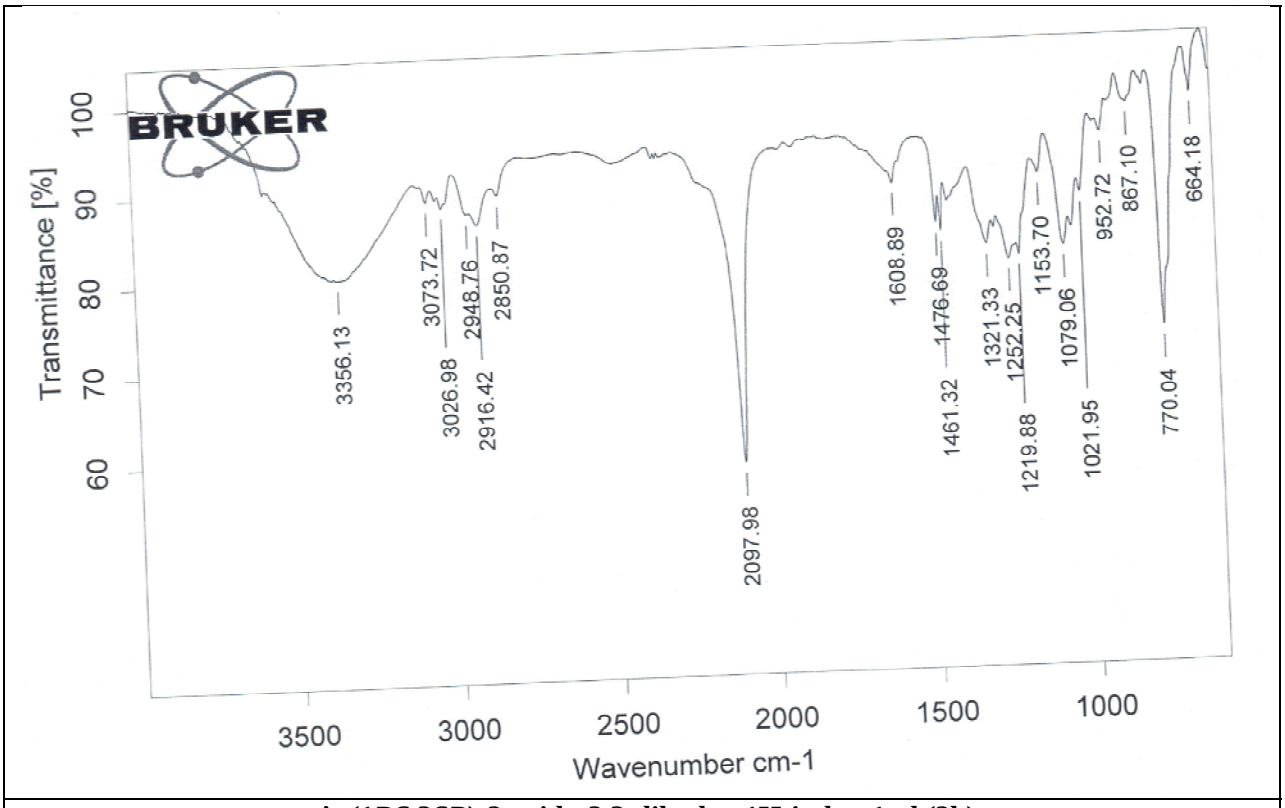
2-azido-2-phenylethan-1-ol-^{18}O



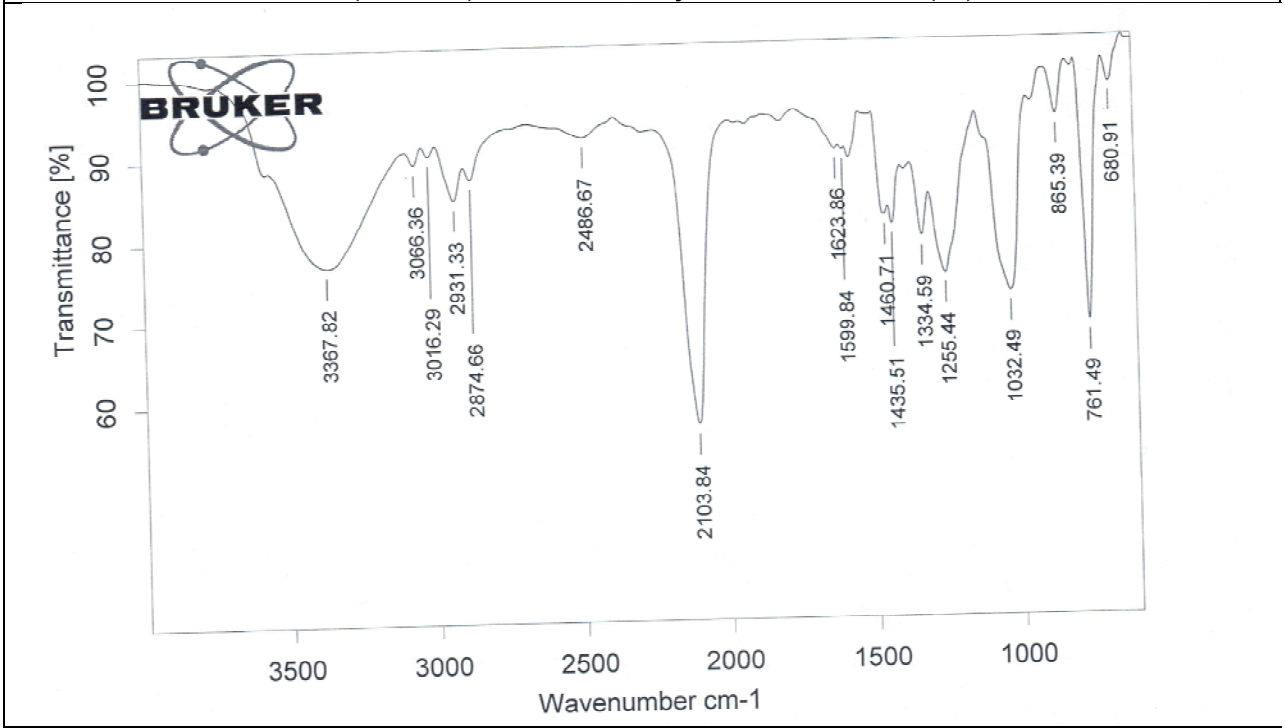
4-(2-azido-1-hydroxyethyl)phenol (2c)



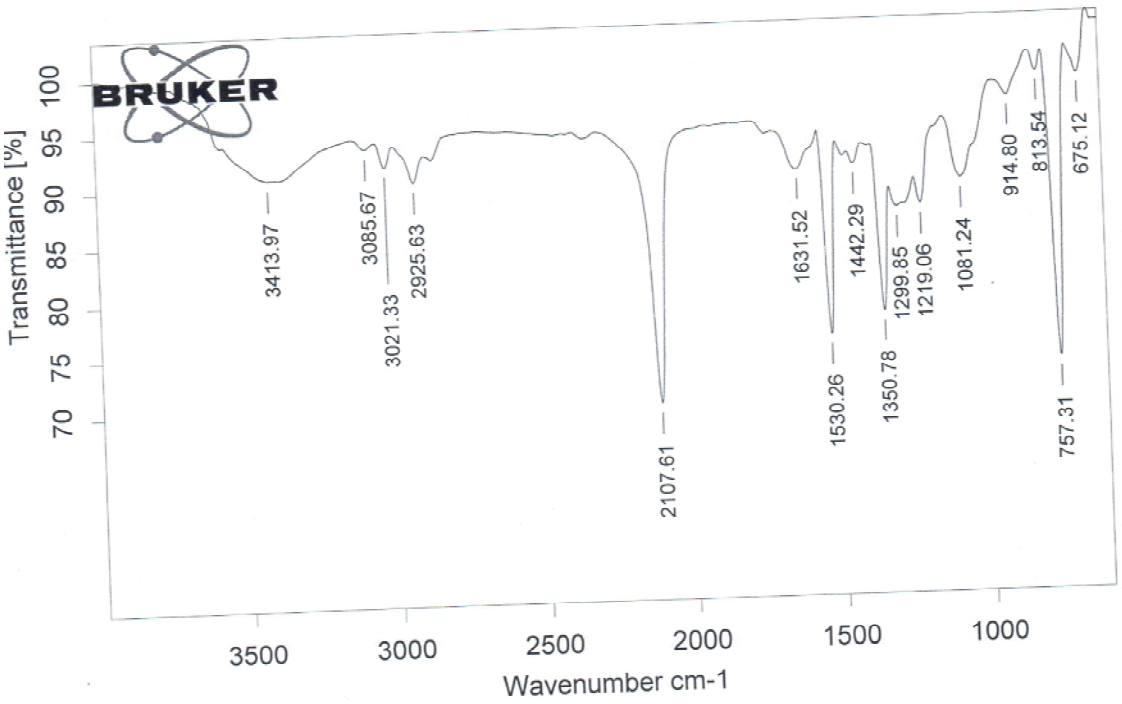
***cis*-(1*RS*,2*SR*)-2-azidocyclohexan-1-ol (2j)**



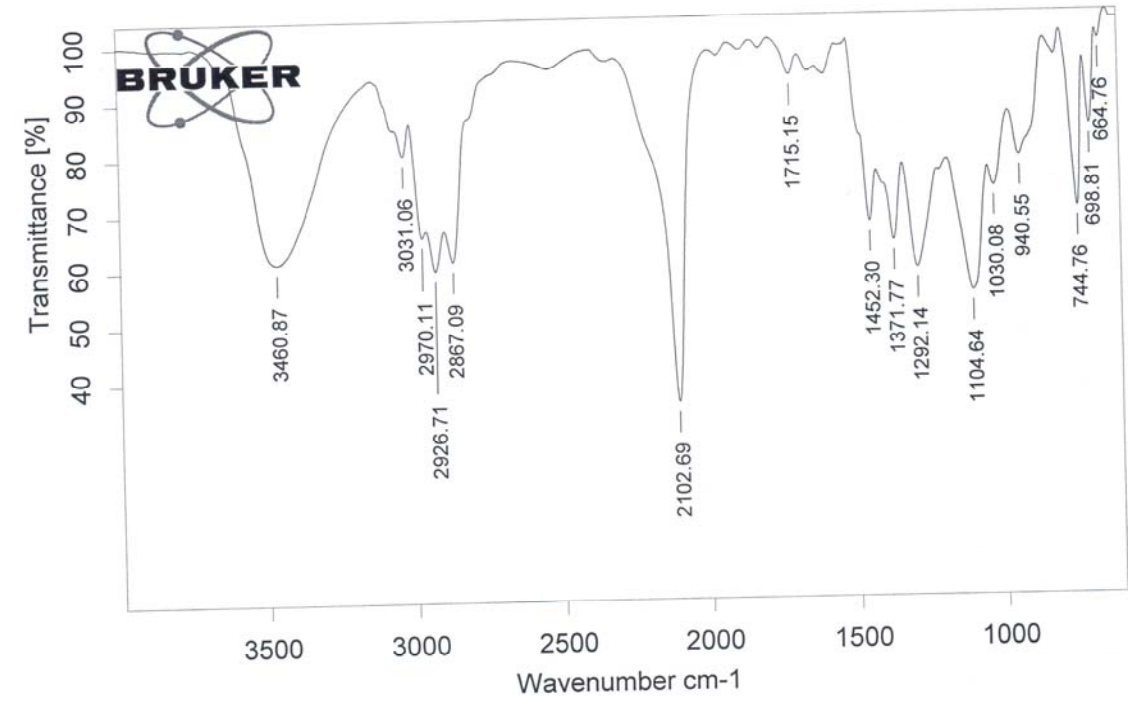
***cis*-(1*RS*,2*SR*)-2-azido-2,3-dihydro-1H-inden-1-ol (2k)**



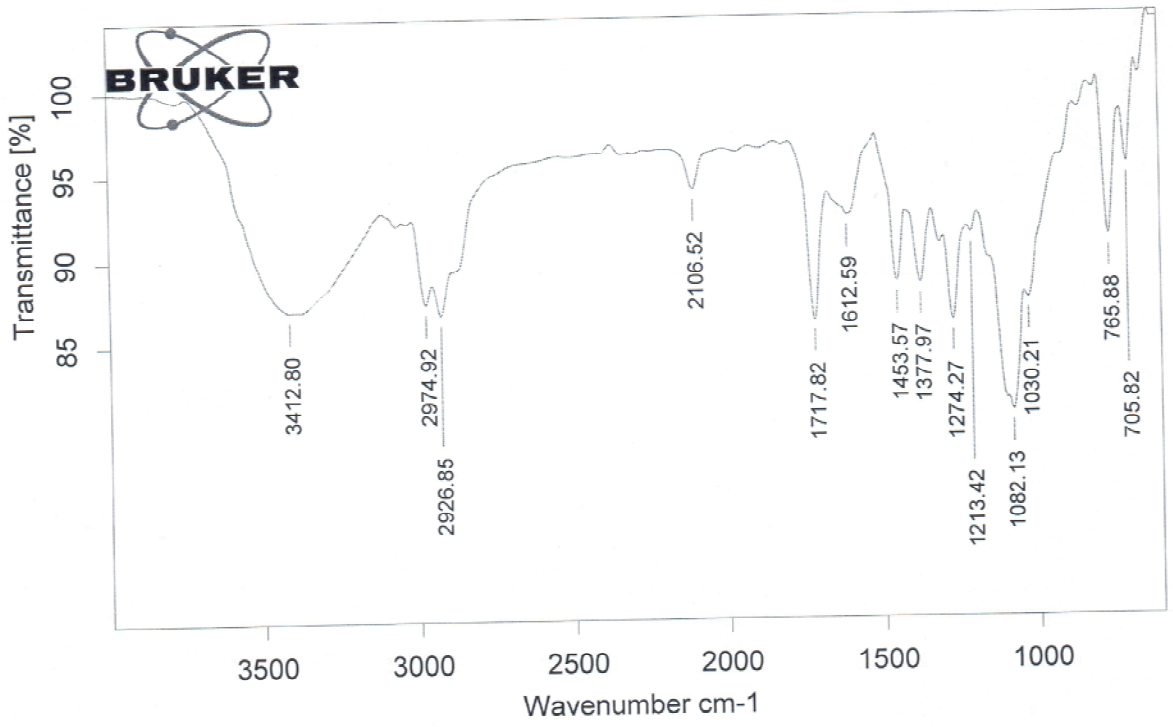
2-azido-2-(2-bromophenyl)ethan-1-ol (3d)



2-azido-2-(3-nitrophenyl)ethan-1-ol (3e)



2-azido-4-(benzyloxy)-2-methylbutan-1-ol (3h)



3-azido-1-(benzyloxy)-3-methylbutan-2-ol (3i)