Supporting Information

Staudinger ketene-imine cycloaddition, RCM approach to macrocrocyclic bisazetidinones

Yehia A. Ibrahim,* Talal F. Al-Azemi, Mohamed D. Abd El-Halim and Elizabeth John

Chemistry Department, Faculty of Science, Kuwait University, P.O. Box 5969, Safat 13060, Kuwai.

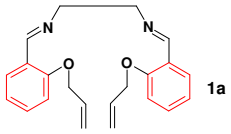
yehiaai@kuc01.kuniv.edu.kw

I. General Experimental Information

Experimental

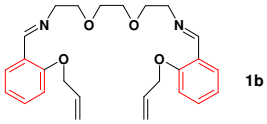
All melting points are uncorrected. IR spectra were recorded in KBr disks on a Perkin Elmer System 2000 FT-IR spectrophotometer. ^{1}H, ^{13}C and ^{15}N NMR spectra were recorded on a Bruker DPX 400, 400 MHz, Avance^{II} 600, 600 MHz super-conducting NMR spectrometers. Mass spectra were measured on VG Auto-spec-Q (high resolution, high performance, tri-sector GC/MS/MS) and with LCMS using Agilent 1100 series LC/MSD with an API-ES/APCI ionization mode. Microanalyses were performed on LECO CH NS-932 Elemental Analyzer. The starting compounds **1a**[8], **14**[6h] and **16**[6h] were prepared as reported.

General procedure for synthesis of compound 1a,b, 15 and 17.
A mixture of each of *o*-allyloxysalicylaldehyede (324 mg, 2 mmol) or bis-aldehydes **14, 16** (296 mg, 1 mmol) and the appropiate diamine (1 mmol) in methanol (15 ml) was refluxed for 2 h. The solvent was then removed in vacuo.



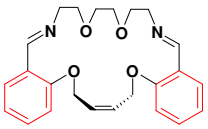
1a

Compound 1a.
Yield 0.3 g (85%); yellow crystals (EtOH), mp 87 ^{o}C (Lit.[8] mp 87 ^{o}C), R_f = 0.70 (pet. ether/EtOAc 3:1). IR: 3090, 3077, 3012, 2988, 2898, 2843, 1638, 1601, 1489, 1452, 1372, 1287, 1250, 1164, 998, 932, 759. ^{1}H NMR ($CDCl_3$): δ 4.01 (s, 4H), 4.56 (m, 4H), 5.30 (dd, 2H, *J* 10.4, 1.2), 5.41 (dd, 2H, *J* 17.2, 1.2), 6.03 (m, 2H), 6.88 (d, 2H, *J* 8.0), 6.98 (t, 2H, *J* 7.6), 7.35 (dt, 2H, *J* 7.8, 2.0), 7.95 (dd, 2H, *J* 7.6, 2.0), 8.79 (s, 2H). ^{13}C NMR ($CDCl_3$): δ 62.0, 69.0, 112.3, 117.5, 120.9, 125.0, 127.3, 131.6, 133.0, 157.7, 158.6.



1b

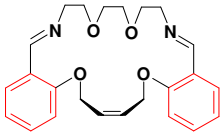
Compound 1b.
Yield 0.35 g (80%); yellow oil (EtOH), R_f = 0.65 (pet. ether/EtOAc 3:1). MS: *m/z* = 436 (M+, 5%). IR: 3036, 3076, 3019, 2893, 2867, 1638, 1600, 1486, 1454, 1295, 1244, 1119, 1019, 997, 926, 755. ^{1}H NMR ($CDCl_3$): δ 3.64 (s, 4H), 3.77 (m, 8H), 4.56 (m, 4H), 5.28 (dd, 2H, *J* 10.4, 1.2), 5.40 (dd, 2H, *J* 17.2, 1.6), 6.06 (m, 2H), 6.86 (d, 2H, *J* 8.4), 6.96 (t, 2H, *J* 7.6), 7.32 (dt, 2H, *J* 7.2, 1.6), 7.96 (dd, 2H, *J* 7.6, 1.6), 8.77 (s, 2H). ^{13}C NMR ($CDCl_3$): δ 61.2, 68.9, 70.3, 70.8, 112.1, 117.4, 120.7, 124.7, 127.2, 131.5, 132.8, 157.5, 158.4. HRMS = 436.2356 ($C_{26}H_{32}N_2O_4$ requires 436.2356).



15

Compound 15.

Yield 0.34 g (85%); yellow oil, R_f = 0.43 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 408 (M^+, 5%). IR: 3073, 3034, 2969, 2887, 1638, 1560, 1485, 1453, 1376, 1295, 1241, 1160, 1128, 1018, 954, 756. ^{1}H NMR ($CDCl_3$): δ 3.55 (s, 4H), 3.65 (t, 4H, *J* 4.8), 3.72 (t, 4H, *J* 4.8), 4.73 (d, 4H, *J* 4.8), 6.06 (t, 2H, *J* 4.8), 6.92 (d, 2H, *J* 8.4), 7.00 (t, 2H, *J* 7.8), 7.35 (dt, 2H, *J* 8.4, 1.8), 7.92 (dd, 2H, *J* 7.8, 1.8), 8.70 (s, 2H). HRMS = 408.2044 ($C_{24}H_{28}N_2O_4$ requires 408.2043).



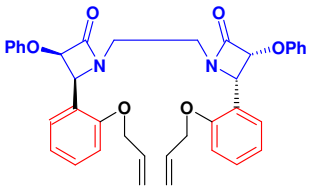
17

Compound 17.

Yield 0.33 g (81%); yellow oil, R_f = 0.44 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 408 (M^+, 10%). IR: 3073, 3034, 2969, 2887, 1638, 1560, 1485, 1453, 1376, 1295, 1241, 1160, 1128, 1018, 954, 756. ^{1}H NMR ($CDCl_3$): δ 3.60 (s, 4H), 3.81 (s, 8H), 4.66 (s, 4H), 6.16 (s, 2H), 6.91 (d, 2H, *J* 8.4), 7.01 (t, 2H, *J* 7.6), 7.38 (dt, 2H, *J* 8.8, 1.6), 7.93 (dd, 2H, *J* 7.6, 1.6), 8.80 (s, 2H). HRMS = 408.2044 ($C_{24}H_{28}N_2O_4$ requires 408.2043).

Synthesis of β-Lactams 2-7 by staudinger reaction: General Procedure.

A solution of aryloxyacetyl chloride (4 mmol) in dry CH_2Cl_2 (5 mL) was purged with nitrogen and cooled to 0 ^{o}C, then a solution of triethylamine (8 mmol) in dry CH_2Cl_2 (5 mL) was added dropwise with a syringe. The mixture was stirred for 30 min and a solution of the corresponding diimine **1a,b, 15, 17** (1 mmol) in dry CH_2Cl_2 (5 mL) was added dropwise over a period of 2 h. The reaction mixture was then stirred overnight at room temperature. The organic layer was washed with water, Na_2CO_3 solution (10%) till no effervescence and then dried over anhydrous Na_2SO_4. The solvent was removed in vacuo and the crude product was separated by chromatography. All *anti* products **2a**, **3a**, **4a** were readily separated from their corresponding *syn* isomers **5a**, **6a**, **7a**. The other *anti and syn* isomers could not be separated and identified as a mixture of each of these two isomers where ^{1}H NMR is almost completely identical except for the azetidinones ring which is assigned for each isomer.

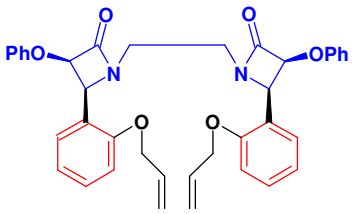


2a

Compound 2a:

Yield 0.28 g (45%); colorless crystals, mp130-132 ^{o}C, R_f = 0.54 (pet. ether/EtOAc 1:1). MS: *m/z* = 616 (M^+, 5%). IR: 3067, 3040, 2969, 2929, 1759, 1600, 1590, 1494, 1415, 1353, 12407, 1231, 1055, 1002, 926, 752, 691. ^{1}H NMR ($CDCl_3$): δ 2.90 (d, 2H, *J* 11.4), 3.90 (d, 2H, *J* 10.8), 4.34 (dd, 2H, *J* 13.2, 4.8), 4.50 (dd, 2H, *J* 13.2, 4.8), 5.34 (dd, 2H, *J* 10.8, 1.2), 5.48 (dd, 2H, *J* 17.7, 1.2), 5.51 (d, 2H, *J* 4.4), 5.84 (d, 2H, *J* 4.4), 6.06 (m, 2H), 6.73 (d, 2H, *J* 8.4), 6.80 (d, 4H, *J* 7.8), 6.87 (t, 2H, *J* 7.2), 6.94 (t, 2H, *J* 7.8), 7.11 (t, 4H, *J* 7.2), 7.20 (dt, 2H, *J* 7.8, 1.2), 7.32 (dd, 2H, *J* 7.8, 1.2). ^{13}C NMR ($CDCl_3$): δ 37.9, 55.5, 68.7, 82.5, 111.4, 115.6, 117.4, 120.4, 121.4, 121.7,

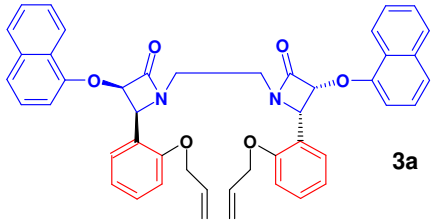
127.9, 128.9, 129.4, 133.0, 156.9, 157.0, 167.4. HRMS = 616.2568 ($C_{38}H_{36}N_2O_6$ requires 616.2567).



5a

Compound 5a:

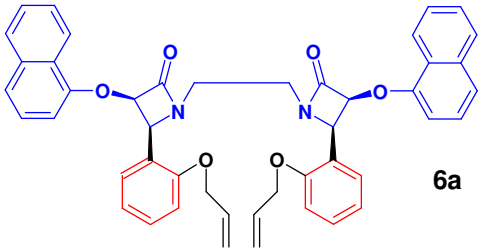
Yield 0.25 g (40%); yellow oil, R_f = 0.39 (pet. ether/EtOAc 1:1). MS: *m/z* = 616 (M^+, 5 %). IR: 3068, 3015, 2917, 2850, 1761, 1599, 1590, 1493, 1455, 1408, 1357, 1291, 1238, 997, 753, 691. ^{1}H NMR ($CDCl_3$): δ 3.11 (m, 2H), 3.85 (m, 2H), 4.28 (ddt, 2H, *J* 12.6, 5.4, 1.2), 4.44 (ddt, 2H, *J* 12.6, 5.4, 1.8), 5.29 (ddd, 2H, *J* 10.2, 2.0, 1.2), 5.32 (d, 2H, *J* 4.2), 5.38 (ddd, 2H, *J* 17.4, 2.4, 1.2), 5.44 (d, 2H, *J* 4.2), 6.00 (m, 2H), 6.71 (m, 6H), 6.86 (t, 2H, *J* 7.2), 6.98 (t, 2H, *J* 7.8), 7.09 (dt, 4H, *J* 7.2, 1.8), 7.22 (dt, 2H, *J* 8.4, 1.8), 7.30 (dd, 2H, *J* 7.8, 1.8). ^{13}C NMR ($CDCl_3$): δ 37.7, 55.8, 68.5, 81.9, 111.3, 115.2, 117.4, 120.4, 120.5, 121.5, 128.1, 128.7, 129.4, 132.7, 156.5, 156.6, 166.0. HRMS = 616.2568 ($C_{38}H_{36}N_2O_6$ requires 616.2567).



3a

Compound 3a:

Yield 0.32 g (46%); yellow oil, R_f = 0.52 (pet. ether/EtOAc 1:1). MS: *m/ z* = 716 (M^+, 10%). IR: 3061, 2958, 2928, 2859, 1760, 1727, 1598, 1580, 1491, 1459, 1396, 1350, 1285, 1268, 1244, 1124, 1072, 793, 771, 753. ^{1}H NMR ($CDCl_3$): δ 3.07 (d, 2H, *J* 11.2), 4.04 (d, 2H, *J* 11.2), 4.37 (ddt, 2H, *J* 12.8, 5.5, 1.2), 4.52 (ddt, 2H, *J* 12.8, 5.2, 1.2), 5.29 (ddd, 2H, *J* 10.4, 2.4, 1.2), 5.44 (ddd, 2H, *J* 17.2, 3.2, 1.6), 5.74 (d, 2H, *J* 4.4), 5.95 (d, 2H, *J* 4.4), 6.03 (m, 2H), 6.76 (d, 2H, *J* 7.6), 6.95 (t, 2H, *J* 6.8), 7.09 (d, 2H, *J* 7.2), 7.21 (dt, 2H, *J* 1.6, 8.4), 7.30 (m, 4H), 7.41 (m, 6H), 7.71 (d, 4H, *J* 8.4). ^{13}C NMR ($CDCl_3$): δ 38.1, 55.6, 68.8, 82.8, 107.2, 111.5, 117.5, 120.5, 121.4, 121.7, 121.9, 125.0, 125.2, 125.4, 126.1, 127.0, 127.8, 129.4, 132.9, 134.1, 152.8, 156.9, 167.7. HRMS = 716.2881 ($C_{46}H_{40}N_2O_6$ requires 716.2880)

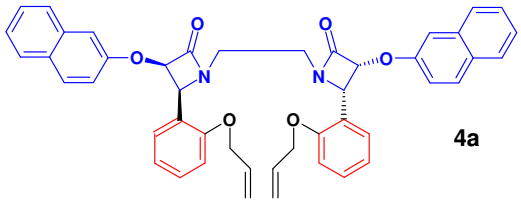


6a

Compound 6a:

Yield 0.32g (45%); colourless crystals, mp191 °C R_f = 0.64 (pet. ether/EtOAc 1:1). MS: *m/ z* = 716 (M^+, 10%). IR: 3081, 3058, 2949, 2910, 2866, 1758, 1595, 1579, 1490, 1455, 1394, 1352, 1289, 1265, 1240, 1181, 1159, 1111, 1086, 1014, 995, 923, 768, 751. ^{1}H NMR ($CDCl_3$): δ 3.25 (m, 2H), 3.98 (m, 2H), 4.33 (ddt, 2H, *J* 12.8, 5.2, 1.6), 4.49 (ddt, 2H, *J* 12.8, 5.2, 1.6), 5.26 (ddd, 2H, *J* 13.2, 2.4, 1.2), 5.35 (ddd, 2H, *J* 17.2, 2.8, 1.2), 5.50 (d, 2H, *J* 4.6), 5.54 (d, 2H, *J* 4.6), 5.98

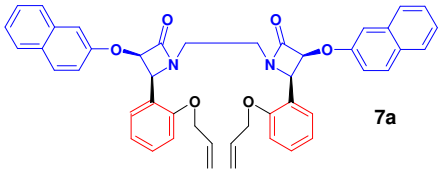
(m, 2H), 6.74 (d, 2H, *J* 8.0), 6.92 (d, 2H, *J* 7.6), 6.98 (t, 2H, *J* 7.2), 7.20 (dt, 2H, *J* 8.0, 1.6), 7.27 (m, 4H), 7.40 (m, 6H), 7.69 (m, 4H). ^{13}C NMR ($CDCl_3$): δ 38.1, 56.1, 68.9, 82.4, 106.9, 111.6, 117.7, 120.7, 121.1, 121.5, 121.9, 125.1, 125.2, 126.3, 127.0, 128.2, 129.6, 132.9, 134.1, 152.7, 156.8, 166.4. HRMS = 716.2882 ($C_{46}H_{40}N_2O_6$ requires 716.2880)



4a

Compound 4a:

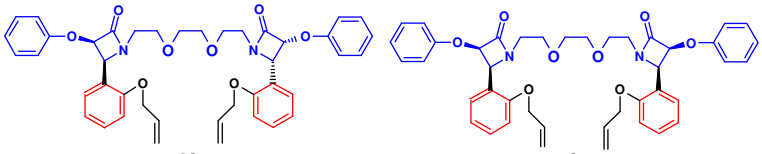
Yield 0.33 g (46%); yellow oil, R_f = 0.58 (pet. ether/EtOAc 1:1). MS: *m/ z* = 716 (M^+, 5%). IR: 3059, 2926, 2854, 1759, 1629, 1600, 1510, 1491, 1466, 1455, 1359, 1253, 1217, 1183, 840, 751. ^{1}H NMR ($CDCl_3$): δ 3.00 (d, 2H, *J* 10.8), 4.00 (d, 2H, *J* 10.8), 4.25 (dd, 2H, *J* 12.8, 4.8), 4.43 (dd, 2H, *J* 12.8, 4.4), 5.35 (d, 2H, *J* 10.8), 5.50 (d, 2H, *J* 17.2), 5.68 (d, 2H, *J* 4.4), 5.98 (d, 2H, J 4.4), 6.05 (m, 2H), 6.62 (d, 2H, *J* 8.4), 6.93 (m, 4H), 7.15 (m, 4H), 7.32 (t, 2H, *J* 7.4), 7.40 (m, 4H), 7.55 (d, 2H, *J* 8.8), 7.69 (d, 4H, *J* 8.4). ^{13}C NMR ($CDCl_3$): δ 37.9, 55.4, 68.4, 82.4, 108.7, 111.2, 117.3, 118.5, 120.3, 121.1, 123.9, 126.1, 126.9, 127.4, 127.9, 129.0, 129.3, 129.4, 132.9, 133.9, 154.6, 156.7, 167.3. HRMS = 716.2881 ($C_{46}H_{40}N_2O_6$ requires 716.2880)



7a

Compound 7a:

Yield 0.32 g (45%); yellow oil, R_f = 0.55 (pet. ether/EtOAc 1:1). MS: *m/ z* = 716 (M^+, 10%). IR: 3060, 2958, 2922, 2856, 1760, 1711, 1629, 1600, 1510, 1491, 1466, 1455, 1407, 1392, 1359, 1289, 1253, 1217, 1183, 1120, 839, 751. ^{1}H NMR ($CDCl_3$): δ 3.16 (m, 2H), 3.92 (m, 2H), 4.20 (dd, 2H, *J* 12.8, 5.2), 4.40 (dd, 2H, *J* 12.8, 4.8), 5.29 (dd, 2H, *J* 10.8, 1.2), 5.37 (dd, 2H, *J* 17.2, 1.2), 5.48 (d, 2H, *J* 4.4), 5.58 (d, 2H, J 4.4), 6.02 (m, 2H), 6.62 (d, 2H, *J* 8.4), 6.89 (dd, 2H, *J* 8.8, 2.4), 7.01 (m, 4H), 7.18 (dt, 2H, *J* 8.8, 1.2), 7.32 (t, 2H, *J* 7.2), 7.38 (m, 4H), 7.55 (d, 2H, *J* 8.8), 7.64 (d, 4H, *J* 8.0), 7.69 (d, 2H, *J* 8.0). ^{13}C NMR ($CDCl_3$): δ 37.8, 55.8, 68.6, 82.0, 108.3, 111.3, 117.6, 118.4, 120.4, 120.5, 123.9, 126.1, 126.7, 127.5, 128.3, 129.0, 129.3, 129.6, 132.9, 133.8, 154.5, 156.6, 166.0. HRMS = 716.2881 ($C_{46}H_{40}N_2O_6$ requires 716.2880)

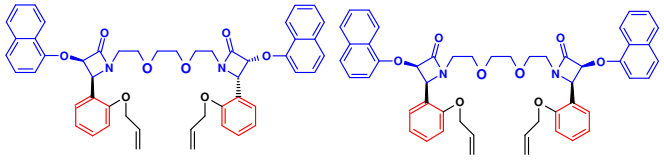


2b **5b**

Compound 2b, 5b:

Yield 0.52 g (74%), obtained as inseparable yellow oily mixture containing **2b** (38%) and **5b** (36%) from column chromatography (using pet. ether/EtOAc/DCM), R_f = 0.44 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 704 (M^+, 5%). IR: 3067, 3040, 2918, 2868, 1761, 1599, 1493, 1454, 1406, 1356, 1290, 1238, 1117, 754. ^{1}H NMR ($CDCl_3$): δ 3.15-3.17 (m, 2H), 3.41 (m, 2H), 3.47-4.58 (m, 6H), 3.71 (m, 2H), 4.26-4.29 (m, 2H), 4.4-4.45 (m, 2H), 5.28 (dm, 2H, *J* 10.8), 5.39 (d, 2H, *J* 16.2), 5.41 (d, 2H, *J* 4.8, azetidinone H-3 of **5b**), 5.44 (d, 2H, *J* 4.2, azetidinone H-3 of **2b**), 5.57 (d, 2H, *J* 4.8, H-4 of **5b**), 5.58 (d, 2H, *J* 4.2, azetidinone H-4 of **2b**), 6.02 (m, 2H), 6.65 (d, 2H, *J* 7.8, ArH **5b**), 6.66 (d, 2H, *J* 7.8, ArH **2b**), 6.73 (m, 4H), 6.83 (t, 2H, *J* 7.2), 6.90 (t,

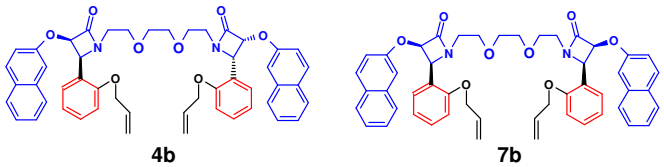
2H, *J* 7.8), 7.06 (t, 4H, *J* 8.1), 7.13 (m, 2H), 7.31 (dd, 2H, *J* 7.4, 1.8, ArH **2b**), 7.33 (dd, 2H, *J* 7.8, 1.6, ArH **5b**). ^{13}C NMR ($CDCl_3$): δ 39.83 (39.86) (azetidinone C-4), 56.6, 67.7, 68.3, 69.74 (69.75), 81.74 (azetidinone C-3), 110.92 (110.94), 115.1, 117.1, 120.0, 121.3, 121.38 (121.41), 127.8, 128.6, 129.0, 132.8, 156.2, 156.6, 166.04 (166.06) (isomer signals). HRMS = 704.3088 ($C_{42}H_{44}N_2O_8$ requires 704.3092).



3b **6b**

Compound 3b, 6b:

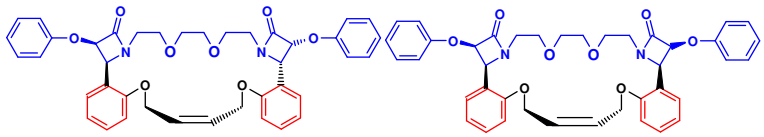
Yield 0.72 g (90%), obtained as inseparable yellow oily mixture containing **3b** (45%) and **6b** (45%) from column chromatography (using pet. ether/EtOAc/DCM), R_f = 0.48 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 804 (M^+, 10%). IR: 3062, 3011, 2957, 2925, 2870, 1763, 1492, 1457, 1398, 1267, 1243, 1119, 794, 772, 756. ^{1}H NMR ($CDCl_3$): δ 3.22-3.30 (m, 2H), 3.46-69 (m, 8H), 3.78-3.86 (m, 2H), 4.30 (dd, 2H, *J* 12.8, 5.2), 4.46 (dd, 2H, *J* 12.8, 4.8), 5.25 (d, 2H, *J* 10.8), 5.35 (d, 2H, *J* 17.2), 5.63 (d, 2H, *J* 4.8, azetidinone H-3 of **6b**),), 5.65 (d, 2H, *J* 4.8, azetidinone H-3 of **3b**), 5.67, 5.68 (2d, 2H, *J* 4.8, azetidinone H-4 of **6b**, **3b**), 5.99 (m, 2H), 6.71 (d, 2H, *J* 8.0), 6.92 (t, 2H, *J* 7.6), 6.99 (t, 2H, *J* 6.8), 7.16 (m, 2H), 7.26 (m, 4H), 7.39 (m, 6H), 7.67 (m, 4H). ^{13}C NMR ($CDCl_3$): δ 40.2 (40.3) (azetidinone C-4), 56.9, 67.97 (68.0), 69.0, 70.0 (70.04), 82.2 (azetidinone C-3), 106.92 (106.94), 111.2, 117.3, 120.4, 121.3, 121.8, 121.98 (122.0), 124.9, 125.1, 125.2, 126.1, 126.9, 128.0, 129.1, 133.0, 134.0, 152.8, 156.5, 166.63 (166.65) (isomer signals). HRMS = 804.3404 ($C_{50}H_{48}N_2O_8$ requires 804.3405).



4b **7b**

Compound 4b, 7b:

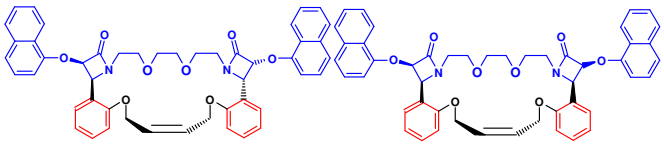
Yield 0.64 g (80%), obtained as inseparable yellow oily mixture containing **4b** (40%) and **7b** (40%) from column chromatography (using pet. ether/EtOAc/DCM), R_f = 0.48 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/ z* = 804 (M^+, 10%). IR: 3060, 2918, 2869, 1763, 1629, 1600, 1492, 1455, 1360, 1254, 1218, 1183, 1120, 752. ^{1}H NMR ($CDCl_3$): δ 3.20-3.23 (m, 2H), 3.45-3.49 (m, 2H), 3.54-3.65 (m, 6H), 3.73-3.79 (m, 2H), 4.22 (dd, 2H, *J* 12.6, 3.6), 4.39 (dd, 2H, *J* 12.6, 3.6), 5.31 (d, 2H, *J* 10.2), 5.42 (d, 2H, *J* 17.4), 5.57 (d, 2H, *J* 3.6, azetidinone H-3 of **7b**),), 5.60 (d, 2H, *J* 3.6, azetidinone H-3 of **4b**), 5.71 (d, 2H, *J* 3.6, overlaped azetidinone H-4 of **7b**, **4b**),), 6.02 (m, 2H), 6.57 (d, 2H, *J* 7.8), 6.91 (m, 4H), 7.10 (m, 4H), 7.31 (t, 2H, *J* 7.2), 7.39 (m, 4H), 7.53 (d, 2H, *J* 9.0), 7.64 (d, 2H, *J* 8.4), 7.68 (d, 2H, *J* 7.8). ^{13}C NMR ($CDCl_3$): δ 40.12 (40.16) (azetidinone C-4), 56.7, 67.99 (68.01), 68.4, 70.02 (70.05), 81.91 (81.95) (azetidinone C-3), 108.39 (108.41), 111.02 (111.04), 117.2, 118.5, 120.2, 121.28 (121.31), 123.8, 126.1, 126.7, 127.5, 128.2, 129.0, 129.20, 129.24, 133.1, 133.8, 154.6, 156.4, 166.2 (isomer signals). HRMS = 804.3404 ($C_{50}H_{48}N_2O_8$ requires 804.3405).



E-8b *E*-11b

Compound *E*-8b, *E*-11b:

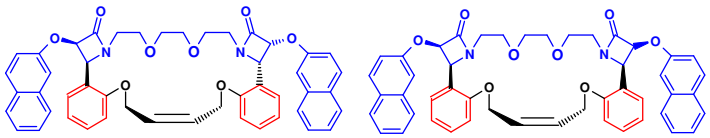
Yield 0.53 g (78%), isolated as a yellow oil of *anti/syn* (64:36) mixture after column chromatography, R_f = 0.41 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 676 (M^+, 7%). HRMS = 676.2778 ($C_{40}H_{40}N_2O_8$ requires 676.2779). IR: 3065, 3011, 2918, 2868, 1761, 1599, 1493, 1454, 1408, 1358, 1290, 1240, 1113, 752, 690. Compound **8b** ^{1}H NMR ($CDCl_3$): δ 3.07 (m, 2H), 3.45-3.55 (m, 4H), 3.57-3.60 (m, 2H), 3.65-3.71 (m, 2H), 3.91 (ddd, 2H, *J* 14.6, 6.8, 3.6), 4.36 (dd, 2H, *J* 11.4, 2.5), 4.59 (dd, 2H, *J* 11.4, 2.5), 5.45 (d, 2H, *J* 4.5), 5.69 (d, 2H, *J* 4.5), 6.17 (t, 2H, *J* 2.5), 6.74 (d, 4H, *J* 7.8), 6.77 (d, 2H, *J* 7.8), 6.86 (m, 2H), 6.97 (t, 2H, *J* 7.2), 7.08 (dt, 4H, *J* 7.2, 1.8), 7.21 (dt, 2H, *J* 8.0, 1.6), 7.36 (dd, 2H, *J* 7.6, 1.6). Compound **8b** ^{13}C NMR ($CDCl_3$): δ 39.5, 56.2, 67.2, 67.9, 69.5, 82.0, 111.0, 115.4, 120.59, 121.6, 121.7, 127.7, 128.3, 128.8, 129.3, 156.4, 156.9, 166.3. Compound **11b:**^{1}H NMR ($CDCl_3$): δ 3.07 (m, 2H), 3.45-3.55 (m, 4H), 3.57-3.60 (m, 2H), 3.65-3.71 (m, 2H), 3.84 (ddd, 2H, *J* 14.6, 6.2, 3.6), 4.36 (dd, 2H, *J* 12.9, 2.5), 4.56 (dd, 2H, *J* 12.9, 2.5), 5.48 (d, 2H, *J* 4.5), 5.70 (d, 2H, *J* 4.5), 6.08 (t, 2H, *J* 2.5), 6.70 (d, 4H, *J* 8.0), 6.73 (d, 2H, *J* 7.2), 6.86 (m, 2H), 6.95 (t, 2H, *J* 7.2), 7.11 (dt, 4H, *J* 7.6, 2.0), 7.18 (dt, 2H, *J* 8.0, 1.6), 7.36 (dd, 2H, *J* 7.2, 1.6). Compound **11b** ^{13}C NMR ($CDCl_3$): δ 39.6, 56.6, 67.3, 68.2, 69.6, 82.1, 111.3, 115.5, 120.6, 121.6, 122.0, 127.8, 128.5, 128.9, 129.2, 156.4, 157.0, 166.3.



E-9b *E*-12b

Compound *E*-9b, *E*-12b:

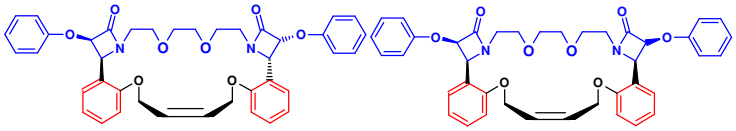
Yield 0.66 g (85%), isolated as a yellow oil of *anti/syn* (55:45) mixture after column chromatography, R_f = 0.37 (DCM/pet. ether/EtOAc 2:1:3). MS: *m/z* = 776 (M^+, 10%). HRMS = 776.3092 ($C_{48}H_{44}N_2O_8$ requires 776.3092) IR: 3054, 2918, 2867, 1759, 1492, 1456, 1396, 1267, 1242, 1112, 910, 773, 754, 730. Compound **9b** ^{1}H NMR ($CDCl_3$): δ 3.05 (m, 2H), 3.40-3.50 (m, 4H), 3.54 (m, 2H), 3.63 (m, 2H), 3.90 (m, 2H), 4.23 (d, 2H, *J* 11.4), 4.42 (d, 2H, *J* 11.4), 5.54 (d, 2H, *J* 4.8), 5.67 (d, 2H, *J* 4.8), 5.97 (s, 2H), 6.63 (d, 2H, *J* 8.4), 6.87 (m, 4H), 7.06-7.16 (m, 6H), 7.26 (d, 2H, *J* 8.4), 7.29 (dt, 4H, *J* 7.8, 1.2), 7.58 (t, 4H, *J* 7.8). Compound **9b** ^{13}C NMR ($CDCl_3$): δ 39.7, 56.2, 67.3, 67.8, 69.6, 82.2, 107.0, 111.1, 120.7, 121.4, 121.9, 122.1, 125.1, 125.2, 125.3, 126.2, 127.0, 127.6, 128.2, 129.3, 134.1, 152.9, 156.4, 166.6. Compound **12b:** ^{1}H NMR ($CDCl_3$): δ 3.05 (m, 2H), 3.40-3.50 (m, 4H), 3.54 (m, 2H), 3.63 (m, 2H), 3.81 (m, 2H), 4.33 (d, 2H, *J* 12.0), 4.43 (d, 2H, *J* 12.0), 5.56 (d, 2H, *J* 4.2), 5.68 (d, 2H, *J* 4.2), 5.91 (s, 2H), 6.62 (d, 2H, *J* 7.8), 6.87 (m, 4H), 7.06-7.16 (m, 6H), 7.18 (d, 2H, *J* 8.4), 7.29 (td, 4H, *J* 7.8, 1.2), 7.58 (t, 4H, *J* 7.8). Compound **12b** ^{13}C NMR ($CDCl_3$): δ 39.8, 56.6, 67.3, 68.2, 69.7, 82.3, 107.1, 111.4, 120.7, 121.4, 121.9, 122.3, 125.1, 125.2, 125.3, 126.2, 127.1, 127.8, 128.4, 129.2, 134.2, 153.0, 156.5, 166.6.



***E*-10b** ***E*-13b**

Compound *E*-10b, *E*-13b:

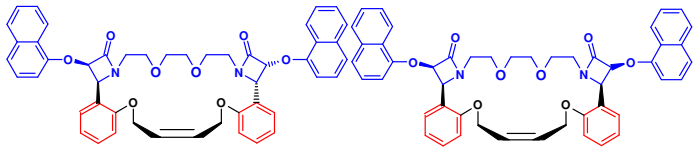
Yield 0.65 g (84%), isolated as a yellow oil of *anti/syn* (55:45) mixture after column chromatography, R_f = 0.29 (DCM/pet. ether/EtOAc 2:1:3). MS: *m/z* = 776 (M^+, 10%). HRMS = 776.3092 ($C_{48}H_{44}N_2O_8$ requires776.3092) IR: 3059, 2919, 2868, 1761, 1629, 1600, 1492, 1455, 1407, 1253, 1218, 1115, 913, 747. Compound **10b** ^{1}H NMR ($CDCl_3$): δ 3.11 (m, 2H), 3.59-3.66 (m, 6H), 3.74 (m, 2H), 3.96 (m, 2H), 4.25 (d, 2H, *J* 12.0), 4.58 (d, 2H, *J* 12.0), 5.60 (d, 2H, *J* 4.2), 5.83 (d, 2H, *J* 4.2), 6.21 (s, 2H), 6.62 (d, 2H, *J* 7.8), 6.89 (dd, 2H, *J* 7.2, 1.8), 6.97 (t, 2H, *J* 7.2), 7.08 (d, 2H, *J* 7.2), 7.15 (dt, 2H, *J* 8.4, 1.8), 7.32 (dt, 2H, *J* 7.2, 1.2), 7.38 (m, 4H), 7.53 (d, 2H, *J* 9), 7.58 (d, 2H, *J* 8.4), 7.68 (d, 2H, *J* 7.8). Compound **10b** ^{13}C NMR ($CDCl_3$): δ 39.5, 56.3, 67.1, 68.1, 69.5, 81.9, 108.4, 110.8, 118.6, 120.5, 121.3, 123.8, 126.1, 126.5, 127.6, 127.7, 128.2, 129.0, 129.3, 129.4, 133.9, 154.7, 156.3, 166.1. Compound **13b:** ^{1}H NMR ($CDCl_3$): δ 3.13 (m, 2H), 3.49-3.55 (m, 6H), 3.71 (m, 2H), 3.88 (m, 2H), 4.41(d, 2H, *J* 13.8), 4.54 (d, 2H, *J* 13.8), 5.63 (d, 2H, *J* 4.2), 5.85 (d, 2H, *J* 4.2), 6.09 (s, 2H), 6.57 (d, 2H, *J* 7.8), 6.93 (m, 4H), 7.10 (dt, 2H, *J* 9.0, 1.8), 7.13 (d, 2H, *J* 7.2), 7.31(td, 2H, *J* 7.8, 1.8), 7.39 (m, 4H), 7.55 (d, 2H, *J* 9.0), 7.65 (d, 2H, *J* 7.8), 7.67 (d, 2H, *J* 7.8). Compound **13b** ^{13}C NMR ($CDCl_3$): δ 39.7, 56.5, 67.1, 68.4, 69.6, 82.1, 108.5, 111.1, 118.6, 120.5, 123.9, 126.2, 126.6, 127.6, 127.8 128.4, 129.0, 129.2, 129.3, 130.9, 133.9, 154.8, 156.2, 166.1.



***Z*-8b** ***Z*-11b**

Compound *Z*-8b, *Z*-11b:

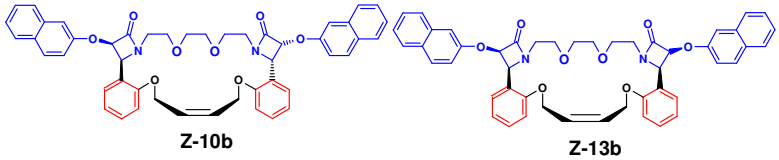
Yield 0.54 g (80%), isolated as a yellow oil of *anti/syn* (48:52) mixture after column chromatography, R_f = 0.41 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 676 (M^+, 15%). HRMS = 676.2778 ($C_{40}H_{40}N_2O_8$ requires 676.2779). IR: 3065, 3038, 2921, 2871, 1761, 1595, 1492, 1456, 1407, 1356, 1293, 1237, 1114, 1080, 1020, 755, 732, 692. Compound ***Z*-8b** ^{1}H NMR ($CDCl_3$): δ 3.18 (m, 2H), 3.13-349 (m, 6H), 3.59 (m, 2H), 3.77 (m, 2H), 4.43(dd, 2H, *J* 12.0, 3.6), 4.72 (dd, 2H, *J* 12.0, 3.6), 5.39 (d, 2H, *J* 4.4), 5.59 (d, 2H, *J* 4.4), 5.95 (t, 2H, *J* 3.6), 6.62 (d, 2H, *J* 7.6), 6.76 (td, 4H, *J* 8.0, 0.8), 6.88 (td, 2H, *J* 7.2, 0.8), 6.96 (m, 2H), 7.13 (m, 4H), 7.18 (m, 2H), 7.33 (m, 2H). Compound ***Z*-8b** ^{13}C NMR ($CDCl_3$): δ 39.6, 56.8, 64.6, 68.3, 69.6, 82.1, 111.4, 115.5, 120.7, 121.8, 121.9, 128.2, 128.7, 128.9, 129.3, 156.3, 157.0, 166.3. Compound ***Z*-11b:** ^{1}H NMR ($CDCl_3$): δ 3.08 (m, 2H), 3.13-349 (m, 6H), 3.59 (m, 2H), 3.77 (m, 2H), 4.62 (dd, 2H, *J* 11.6, 3.2), 4.69 (d, 2H, *J* 11.6, 3.2), 5.48 (d, 2H, *J* 4.4), 5.68 (d, 2H, *J* 4.4), 5.87 (t, 2H, *J* 3.2), 6.62 (d, 2H, *J* 7.6), 6.76 (td, 4H, *J* 8.0, 0.8), 6.88 (td, 2H, *J* 7.2, 0.8), 6.96 (m, 2H), 7.13 (m, 4H), 7.18 (m, 2H), 7.33 (m, 2H). Compound ***Z*-11b** ^{13}C NMR ($CDCl_3$): δ 39.5, 56.6, 64.1, 68.5, 69.6, 82.1, 111.1, 115.4, 120.7, 121.7, 121.8, 128.4, 128.5, 128.9, 129.2, 156.4, 156.9, 166.2.



Z-9b *Z*-12b

Compound *Z*-9b, *Z*-12b:

Yield 0.65 g (84%), isolated as a yellow oil of *anti/syn* (48:52) mixture after column chromatography, R_f = 0.41 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 776 (M^+, 10%). HRMS = 776.3092 ($C_{48}H_{44}N_2O_8$ requires 776.3092). IR: 3057, 2923, 2866, 1761, 1596, 1491, 1457, 1397, 1353, 1289, 1267, 1240, 1114, 1018, 794, 770, 732. Compound ***Z*-9b** ^{1}H NMR ($CDCl_3$): δ 3.24 (m, 2H), 3.39 (m, 2H), 3.46 (m, 2H), 3.54 (m, 2H), 3.65 (m, 2H), 3.85 (m, 2H), 4.42 (dd, 2H, *J* 12.0, 4.8), 4.68 (dd, 2H, *J* 12.0, 4.8), 5.55 (d, 2H, *J* 4.8), 5.66 (d, 2H, *J* 4.8), 5.89 (t, 2H, *J* 4.8), 6.67 (d, 2H, *J* 8.4), 6.95 (m, 4H), 7.12 (m, 2H), 7.26 (m, 4H), 7.40 (m, 6H), 7.18 (d, 2H, *J* 7.8), 7.70 (t, 2H, *J* 7.8). Compound ***Z*-9b** ^{13}C NMR ($CDCl_3$): δ 39.7, 56.6, 64.1, 68.5, 69.7, 82.4, 107.0, 111.2, 120.8, 121.5, 121.9, 122.2, 125.1, 125.23, 125.24, 126.2, 127.1, 128.3, 128.5, 129.2, 134.2, 152.9, 156.5, 166.4. Compound ***Z*-12b:** ^{1}H NMR ($CDCl_3$): δ 3.15 (m, 2H), 3.39 (m, 2H), 3.46 (m, 2H), 3.54 (m, 2H), 3.65 (m, 2H), 3.85 (m, 2H), 4.56 (dd, 2H, *J* 12.6, 3.6), 4.65 (dd, 2H, *J* 12.6, 3.6), 5.65 (d, 2H, *J* 4.8), 5.74 (d, 2H, *J* 4.8), 5.81 (t, 2H, *J* 3.6), 6.70 (d, 2H, *J* 7.8), 6.95 (m, 4H), 7.12 (m, 2H), 7.26 (m, 4H), 7.40 (m, 6H), 7.18 (d, 2H, *J* 7.8), 7.70 (t, 2H, *J* 7.8). Compound ***Z*-12b** ^{13}C NMR ($CDCl_3$): δ 39.8, 56.8, 64.5, 68.3, 69.8, 82.3, 107.2, 111.5, 120.9, 121.5, 121.8, 122.3, 125.1, 125.2, 125.4, 126.2, 127.1, 128.4, 128.5, 129.3, 134.2, 152.9, 156.5, 166.5.



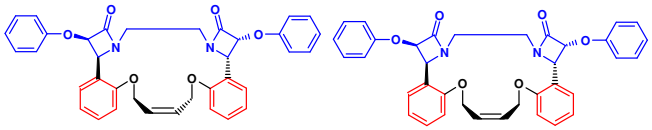
Z-10b Z-13b

Compound *Z*-10b, *Z*-13b:

Yield 0.61 g (78%), isolated as a yellow oil of *anti/syn* (48:52) mixture after column chromatography, R_f = 0.41 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 776 (M^+, 10%). HRMS = 776.3092 ($C_{48}H_{44}N_2O_8$ requires 776.3092). IR: 3059, 2923, 2862, 1762, 1597, 1491, 1461, 1402, 1357, 1254, 1220, 1119, 911, 776, 733. Compound ***Z*-10b** ^{1}H NMR ($CDCl_3$): δ 3.12 (m, 2H), 3.23-3.41 (m, 6H), 3.49-3.57 (m, 2H), 3.68-3.74 (m, 2H), 4.26 (dd, 2H, *J* 12.4, 3.2), 4.54 (dd, 2H, *J* 12.4, 3.2), 5.45 (d, 2H, *J* 4.1), 5.62 (d, 2H, *J* 4.1), 5.63 (t, 2H, *J* 3.2), 6.58 (d, 2H, *J* 7.6), 6.83 (m, 4H), 7.01 (d, 2H, *J* 7.2), 7.03 (td, 2H, *J* 1.4, 8.0), 7.22-7.36 (m, 6H), 7.53 (d, 2H, , *J* 8.0), 7.61 (dd, 4H, *J* 8.0, 2.2). Compound ***Z*-10b** ^{13}C NMR ($CDCl_3$): δ 39.6, 56.7, 64.7, 68.6, 69.6, 82.0, 108.6, 111.3, 118.6, 120.6, 121.6, 123.9, 126.2, 126.7, 127.6, 128.4, 128.6, 129.1, 129.2, 129.3, 133.9, 154.7, 156.2, 165.9. Compound ***Z*-13b:** ^{1}H NMR ($CDCl_3$): δ 3.12 (m, 2H), 3.23-3.41 (m, 6H), 3.49-3.57 (m, 2H), 3.68-3.74 (m, 2H), 4.26 (dd, 2H, *J* 12.4, 3.2), 4.54 (dd, 2H, *J* 12.4, 3.2), 5.53 (d, 2H, *J* 4.2), 5.71 (d, 2H, *J* 4.2), 5.79 (t, 2H, *J* 3.2), 6.35 (d, 2H, *J* 8.2), 6.83 (m, 4H), 5.95 (td, 2H, *J* 1.7, 8.2), 6.98 (d, 2H, *J* 7.2), 7.22-7.36 (m, 6H), 7.47 (d, 4H, , *J* 9.0), 7.59 (d, 2H, *J* 8.0). Compound ***Z*-13b** ^{13}C NMR ($CDCl_3$): δ 39.5, 56.5, 64.2, 68.5, 69.6, 82.1, 108.4, 111.0, 118.6, 120.7, 121.4, 123.9, 126.2, 126.6, 127.6, 128.1, 128.4, 129.1, 129.11, 129.3, 133.8, 154.7, 156.3, 166.0.

General Procedures of the RCM

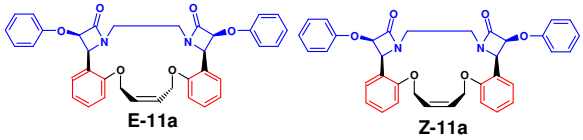
To a solution of each of the appropriate β-Lactam **2-7** (1 mmol) in DCM (10 ml), Grubb's catalyst **I** or **II** (mol% indicated in Tables 2, 3) was added. The reaction mixture was heated under reflux (for time indicated in Tables 2, 3) and the solvent was removed in vacuo and the product was purified by column chromatography with eluent DCM/pet. ether (60-80)/EtOAc



***E*-8a** ***Z*-8a**

Compound 8a:

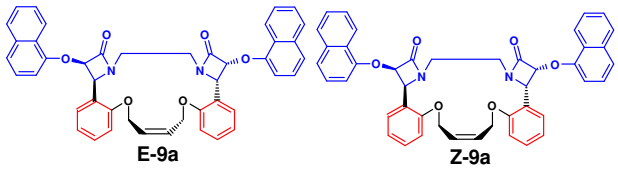
Yield 0.56 g (95%), isolated as *cis/trans* (8:92) mixture after column chromatography [of RCM reaction with Grubbs' **I** (5 mol%)] as colorless crystals, mp 123-125 ^{o}C, R_f = 0.61 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 588 (M^+, 25%). HRMS = 588.2254 ($C_{36}H_{32}N_2O_6$ requires 588.2254). IR: 3065, 3041, 2922, 2853, 1764, 1599, 1590, 1492, 1456, 1404, 1355, 1237, 912, 752, 734. ***Trans* 8a:** ^{1}H NMR ($CDCl_3$): δ 3.06 (d, 2H, *J* 9.6), 3.58 (d, 2H, *J* 9.6), 4.58 (dd, 2H, *J* 11.0, 2.8), 4.71 (dd, 2H, *J* 11.0, 2.8), 5.17 (d, 2H, *J* 4.6), 5.36 (d, 2H, *J* 4.6), 5.96 (t, 2H, *J* 2.8), 6.70 (d, 4H, *J* 7.8), 6.86 (m, 4H), 7.00 (t, 2H, *J* 7.2), 7.11 (m, 4H), 7.22 (td, 2H, *J* 8.4, 1.2), 7.39 (dd, 2H, *J* 7.8, 1.2). ***Trans* 8a:** ^{13}C NMR ($CDCl_3$): δ 39.1, 55.7, 68.4, 81.5, 114.7, 115.3, 121.6, 121.7, 122.6, 129.1, 129.3, 129.75, 129.9, 156.6, 156.9, 166.6. ***Cis* 8a:** ^{1}H NMR ($CDCl_3$): δ 3.32 (d, 2H, *J* 13.0), 3.73 (d, 2H, *J* 13.0), 4.63 (dd, 2H, *J* 11.0, 4.0), 4.67 (dd, 2H, *J* 11.0, 4.0), 4.73 (d, 2H, *J* 4.6), 5.42 (d, 2H, *J* 4.6), 6.18 (t, 2H, *J* 4.0), 6.64 (d, 4H, *J* 7.8), 6.86 (m, 4H), 7.06 (t, 2H, *J* 7.3), 7.12 (m, 4H), 7.27 (td, 2H, *J* 8.0, 2.0), 7.35 (dd, 2H, *J* 7.7, 1.6). ***Cis* 8a:** ^{13}C NMR ($CDCl_3$): δ 39.4, 56.0, 64.6, 81.4, 111.9, 115.5, 121.4, 121.68, 121.8, 128.9, 129.2, 129.6, 129.8, 156.5, 156.7, 166.3.



E-11a **Z-11a**

Compound 11a:

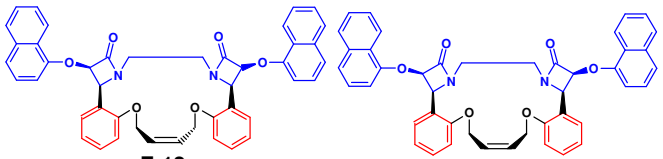
Yield 0.55 g (94%), isolated as *cis/trans* (11:89) mixture after column chromatography [of RCM reaction with Grubbs' **I** (5 mol%)] as white crystals, mp123-125 ^{o}C, R_f = 0.69 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 588 (M^+, 25%). HRMS = 588.2256 ($C_{36}H_{32}N_2O_6$ requires 588.2261). IR: 3063, 3013, 2926, 1771, 1599, 1493, 1456, 1406, 1360, 1236, 1047, 866, 752, 690. ***Trans* 11a** ^{1}H NMR ($CDCl_3$): δ 2.95 (m, 2H), 3.78 (m, 2H), 4.62 (d, 2H, *J* 14.0), 4.75 (d, 2H, *J* 14.0), 5.43 (d, 2H, *J* 4.5), 5.48 (d, 2H, *J* 4.5), 5.95 (s, 2H), 6.78 (m, 6H), 6.89 (m, 4H), 7.15 (m, 6H), 7.28 (dd, 2H, *J* 7.6, 1.6). ***Trans* 11a** ^{13}C NMR ($CDCl_3$): δ 38.2, 55.8, 68.6, 81.9, 114.4, 115.5, 121.4, 121.9, 122.1, 128.9, 129.1, 129.4, 129.7, 156.6, 157.0, 166.2. ***Cis* 11a** ^{1}H NMR ($CDCl_3$): δ 2.93 (m, 2H), 3.97 (m, 2H), 4.46 (dd, 2H, *J* 10.4, 5.6), 4.64 (dd, 2H, *J* 10.4, 5.6), 5.44 (d, 2H, *J* 4.5), 5.56 (d, 2H, *J* 4.5), 6.22 (t, 2H, *J* 5.6), 6.78 (m, 6H), 6.89 (m, 4H), 7.15 (m, 6H), 7.28 (dd, 2H, *J* 7.6, 1.6). ***Cis* 11a** ^{13}C NMR ($CDCl_3$): δ 38.0, 55.7, 63.6, 81.8, 112.3, 115.6, 121.1, 121.6, 121.7, 128.8, 129.0, 129.8, 130.7, 156.9, 157.2, 166.5.



E-9a **Z-9a**

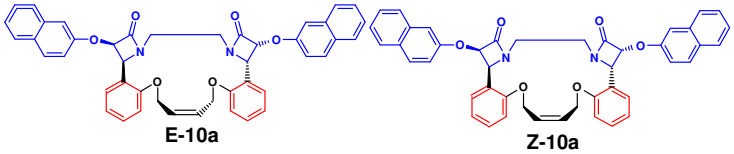
Compound 9a:

Yield 0.63 g (92%), isolated as *cis/trans* (21:79) mixture after column chromatography [of RCM reaction with Grubbs' **I** (5 mol%)] as yellow oil, R_f = 0.68 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 688 (M^+, 10%). HRMS = 688.2568 ($C_{44}H_{36}N_2O_6$ requires 688.2567). IR: 3056, 2925, 1768, 1629, 1600, 1490, 1458, 1396, 1266, 1217, 1183, 755, 733. ***Trans* 9a** ^{1}H NMR ($CDCl_3$): δ 3.23 (d, 2H, *J* 10.0), 3.70 (d, 2H, *J* 10.0), 4.60 (d, 2H, *J* 11.6), 4.70 (d, 2H, *J* 11.6), 5.35 (d, 2H, *J* 4.8), 5.50 (d, 2H, *J* 4.8), 6.02 (s, 2H), 6.83 (d, 2H, *J* 7.2), 6.92 (d, 2H, *J* 8.0), 7.03 (t, 2H, *J* 7.2), 7.22-7.34 (m, 6H), 7.41(m, 4H), 7.51 (dd, 2H, *J* 7.6, 1.6), 7.70 (d, 2H, *J* 8.0), 7.79 (d, 2H, *J* 8.4). ***Trans* 9a** ^{13}C NMR ($CDCl_3$): δ 39.3, 55.9, 68.6, 81.6, 106.8, 114.9, 121.4, 121.7, 121.8, 122.9, 125.1, 125.2, 125.5, 126.3, 127.1, 129.1, 129.8, 129.9, 134.2, 152.7, 156.7, 166.8. ***Cis* 9a** ^{1}H NMR ($CDCl_3$): δ 3.44 (d, 2H, *J* 13.2), 3.84 (d, 2H, *J* 13.2), 4.60 (dd, 2H, *J* 11.6, 4.0), 4.70 (dd, 2H, *J* 11.6, 4.0), 4.92 (d, 2H, *J* 4.8), 5.56 (d, 2H, *J* 4.8), 6.19 (t, 2H, *J* 4.0), 6.78 (d, 2H, *J* 7.6), 6.90 (d, 2H, *J* 8.0), 7.07 (t, 2H, *J* 7.6), 7.22-7.34 (m, 6H), 7.41(m, 4H), 7.47 (dd, 2H, *J* 7.6, 1.6), 7.74 (d, 2H, *J* 8.0), 7.69 (d, 2H, *J* 8.4). ***Cis* 9a** ^{13}C NMR ($CDCl_3$): δ 39.5, 55.9, 64.7, 81.5, 106.9, 111.9, 121.5, 121.7, 121.8, 122.9, 125.1, 125.2, 125.5, 126.3, 127.0, 128.8, 129.6, 130.9, 134.1, 152.6, 156.6, 166.5



Compound 12a:

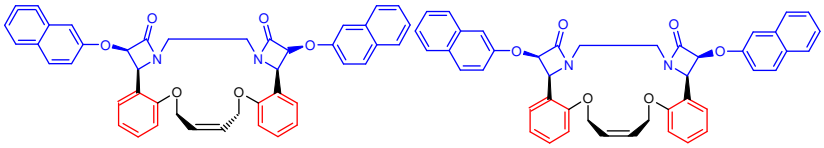
Yield 0.64 g (93%), isolated as *cis/trans* (19:81) mixture after column chromatography [of RCM reaction with Grubbs' **I** (5 mol%)] as yellow oil, R_f = 0.72 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 688 (M^+, 15%). HRMS = 688.2567 ($C_{44}H_{36}N_2O_6$ requires 688.2567). IR: 3058, 2954, 2925, 1770, 1629, 1600, 1490, 1458, 1396, 1359, 1254, 1218, 1183, 912, 752, 731. ***Trans* 12a** ^{1}H NMR ($CDCl_3$): δ 3.07 (m, 2H), 3.89 (m, 2H), 4.62 (d, 2H, *J* 12.8), 4.75 (d, 2H, *J* 12.8), 5.59 (d, 2H, *J* 4.4), 5.65 (d, 2H, *J* 4.4), 5.97 (s, 2H), 6.84 (d, 2H, *J* 8.0), 6.94 (t, 4H, *J* 8.0), 7.18 (dt, 2H, *J* 8.0, 1.6), 7.26-7.33 (m, 4H), 7.40 (m, 6H), 7.71 (d, 2H, *J* 8.0), 7.79 (d, 2H, *J* 8.4). ***Trans* 12a** ^{13}C NMR ($CDCl_3$): δ 38.3, 56.0, 68.8, 82.1, 107.2, 114.6, 121.5, 121.6, 121.8, 122.3, 125.2, 125.3, 125.5, 126.3, 127.2, 128.9, 129.3, 129.8, 134.2, 152.8, 156.7, 166.3. ***Cis* 12a** ^{1}H NMR ($CDCl_3$): δ 3.05 (m, 2H), 3.89 (m, 2H), 4.46 (dd, 2H, *J* 10.0, 4.8), 4.61 (d, 2H, *J* 10.0, 4.8), 5.58 (d, 2H, *J* 4.4), 5.68 (d, 2H, *J* 4.4), 6.08 (t, 2H, *J* 4.8), 6.89 (d, 2H, *J* 8.0), 6.98 (t, 4H, *J* 7.6), 7.18 (td, 2H, *J* 8.0, 1.6), 7.26-7.33 (m, 4H), 7.40 (m, 6H), 7.71 (d, 4H, *J* 8.0). ***Cis* 12a** ^{13}C NMR ($CDCl_3$): δ 38.1, 55.7, 63.9, 82.1, 107.3, 112.5, 121.3, 121.6, 121.7, 121.9, 125.2, 125.3, 125.4, 126.3, 127.2, 128.7, 129.3, 129.8, 130.6, 152.9, 157.4, 166.7.



Compound 10a:

Yield 0.40 g (58%), isolated as *cis/trans* (17:83) mixture after column chromatography [of RCM reaction with Grubbs' **I** (5 mol%)] as yellow oil, R_f = 0.64 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 688 (M^+, 10%). HRMS = 688.2567 ($C_{44}H_{36}N_2O_6$ requires 688.2567). IR: 3058, 2921, 2852 1766, 1629, 1600, 1489, 1456, 1395, 1359, 1254, 1217, 1183, 913, 752, 732. ***Trans* 10a** ^{1}H NMR ($CDCl_3$): δ 3.14 (d, 2H, *J* 9.7), 3.67 (d, 2H, *J* 9.7), 4.63 (d, 2H, *J* 12.0), 4.73 (d, 2H, *J* 12.0), 5.32 (d, 2H, *J* 4.4), 5.48 (d, 2H, *J* 4.4), 6.03 (s, 2H), 6.83 (d, 2H, *J* 7.6), 6.98 (m, 4H), 7.19

(t, 4H, *J* 7.2), 7.32 (t, 2H, *J* 7.2), 7.41 (t, 2H, *J* 7.2), 7.45 (t, 2H, *J* 7.6), 7.58 (d, 2H, *J* 8.8), 7.63 (d, 2H, *J* 7.6), 7.69 (d, 2H, *J* 8.1). ***Trans* 10a** ^{13}C NMR ($CDCl_3$): δ 39.2, 55.7, 68.2, 81.5, 108.6, 114.6, 118.4, 118.5, 121.6, 122.5, 123.9, 126.2, 126.28, 127.5, 129.2, 129.3, 129.4, 129.7, 134.0, 154.7, 156.5, 166.5. ***Cis* 10a** ^{1}H NMR ($CDCl_3$): δ 3.33 (d, 2H, *J* 13.5), 3.79 (d, 2H, *J* 13.5), 4.63 (d, 2H, *J* 12.0), 4.73 (d, 2H, *J* 12.0), 4.85 (d, 2H, *J* 4.5), 5.54 (d, 2H, *J* 4.5), 6.24 (s, 2H), 6.83 (d, 2H, *J* 7.6), 6.98 (m, 4H), 7.19 (t, 4H, *J* 7.2), 7.32 (t, 2H, *J* 7.2), 7.41 (t, 2H, *J* 7.2), 7.45 (t, 2H, *J* 7.6), 7.75 (d, 2H, *J* 8.8), 7.77 (d, 2H, *J* 7.6), 7.79 (d, 2H, *J* 8.1) ***Cis* 10a** ^{13}C NMR ($CDCl_3$): δ 39.4, 56.1, 64.8, 81.4, 107.2, 111.8, 118.5, 118.6, 121.4, 122.2, 124.0, 126.3, 126.9, 127.6, 129.1, 129.3, 129.6, 129.9, 134.3, 154.5, 156.5, 166.1.



E-13a **Z-13a**

Compound 13a:

Yield 0.41 g (60%), isolated as *cis/trans* (19:81) mixture after column chromatography [of RCM reaction with Grubbs' **I** (5 mol%)] as yellow oil, R_f = 0.67 (DCM/pet. ether/EtOAc 1:1:1). MS: *m/z* = 688 (M^+, 10%). HRMS = 688.2568 ($C_{44}H_{36}N_2O_6$ requires 688.2567). IR: 3060, 2927, 2879, 1772, 1630, 1601, 1511, 1490, 1395, 1360, 1255, 1218, 1184, 911, 732. ***Trans* 13a** ^{1}H NMR ($CDCl_3$): δ 2.99 (m, 2H), 3.88 (m, 2H), 4.62 (d, 2H, *J* 14.0), 4.79 (d, 2H, *J* 14.0), 5.61 (d, 2H, *J* 4.4), 5.65(d, 2H, *J* 4.4), 6.03 (s, 2H), 6.71 (d, 2H, *J* 8.0), 6.89 (t, 2H, *J* 7.6), 6.99 (dd, 2H, *J* 8.8, 2.4), 7.07-7.19 (m, 4H), 7.30-7.49 (m, 6H) 7.59 (d, 2H, *J* 8.8), 7.68 (t, 4H, *J* 8.8). ***Trans* 13a** ^{13}C NMR ($CDCl_3$): δ 38.2, 55.8, 68.4, 81.9, 108.8, 114.4, 118.5, 121.0, 121.4, 124.0, 126.2, 126.8, 127.5, 128.3, 128.9, 129.2, 129.4, 129.7, 134.0, 154.7, 156.3, 166.0. ***Cis* 13a** ^{1}H NMR ($CDCl_3$): δ 2.92 (m, 2H), 4.06 (m, 2H), 4.31 (dd, 2H, *J* 10.0, 4.8), 4.52 (d, 2H, *J* 10.0, 4.8), 5.67 (d, 2H, *J* 4.4), 5.76 (d, 2H, *J* 4.4), 6.33 (t, 2H, *J* 4.8), 6.70 (d, 2H, *J* 7.6), 6.89 (t, 2H, *J* 7.6), 6.99 (dd, 2H, *J* 8.8, 2.4), 7.07-7.19 (m, 4H), 7.30-7.49 (m, 6H) 7.59 (d, 2H, *J* 8.8), 7.74 (t, 4H, *J* 8.8). ***Trans* 13a** ^{13}C NMR ($CDCl_3$): δ 38.0, 55.7, 63.1, 81.6, 108.7, 111.9, 118.6, 121.2, 121.8, 124.0, 126.3, 126.6, 127.7, 128.5, 129.1, 129.21, 129.24, 131.0, 133.9, 154.7, 157.0, 166.3.

II. NMR Experiments of 8a with Chiral Shift Reagent Eu(hfc)$_3$

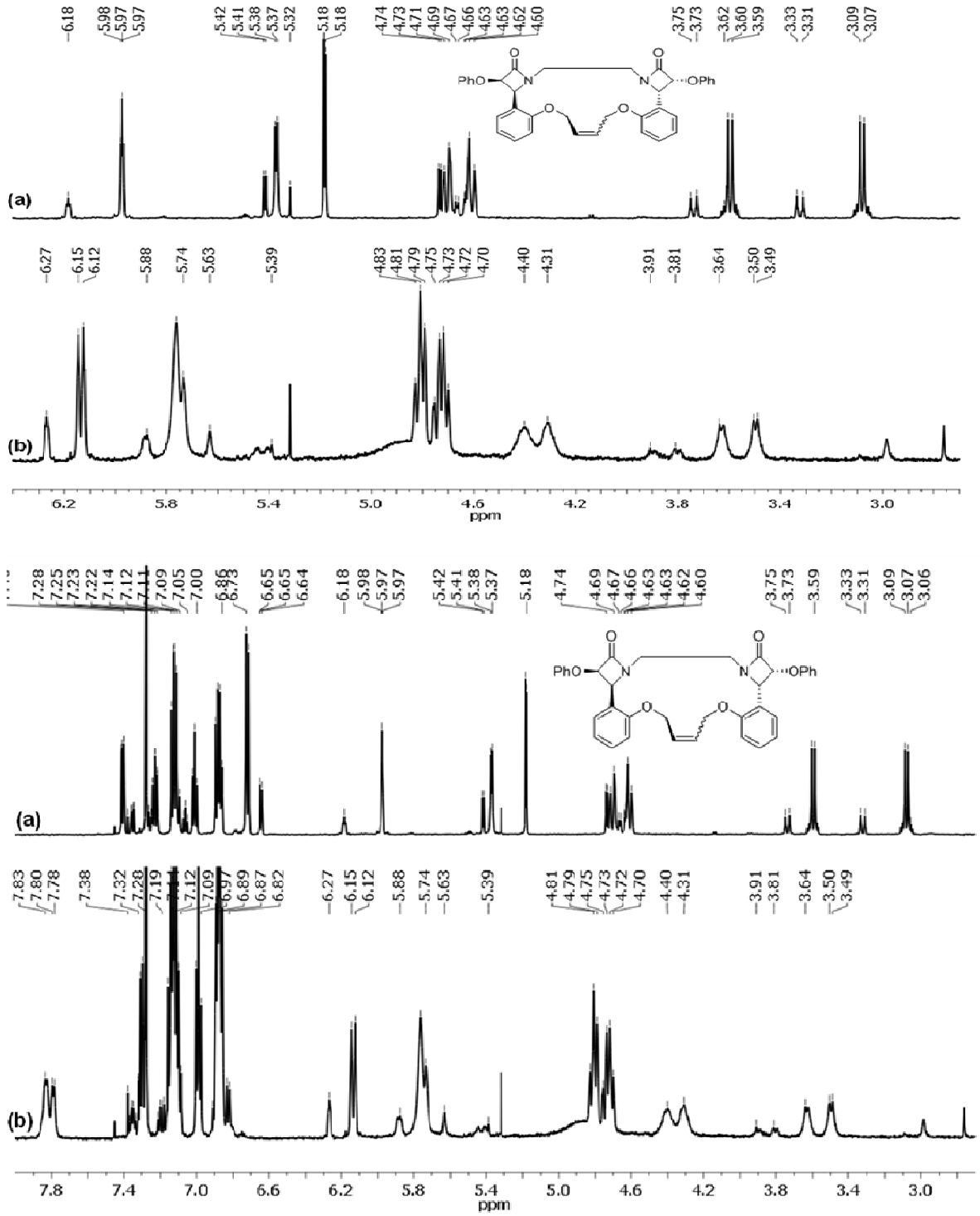


Figure 3. ^{1}H-NMR (600 MHz, $CDCl_3$) Spectra of *cis-anti-cis* compound **8a** (*racemic*): (a) before addition of chiral shift reagent, Eu(hfc)$_3$; (b) after addition of chiral shift reagent, Eu(hfc)$_3$.

III. ^{1}H, ^{13}C NMR and ^{15}N Full Spectral Assignment of 2a, 5a, 8a and 11a

Full Assignments of protons, carbons and nitrogen NMR of compounds **2a**, **5a**, **8a** and **11a** along with the numbering used in the NMR correlations are shown in Figure 4, 5. These assignments were made based on H,H-COSY, HMQC (or HSQC) and HMBC experiments, ^{15}N NMR signals were measured using ^{1}H-^{15}N HMBC experiments. The important ^{1}H-^{13}C and ^{1}H-^{15}N long range correlations found in the HMBC experiments are indicated in each figure.

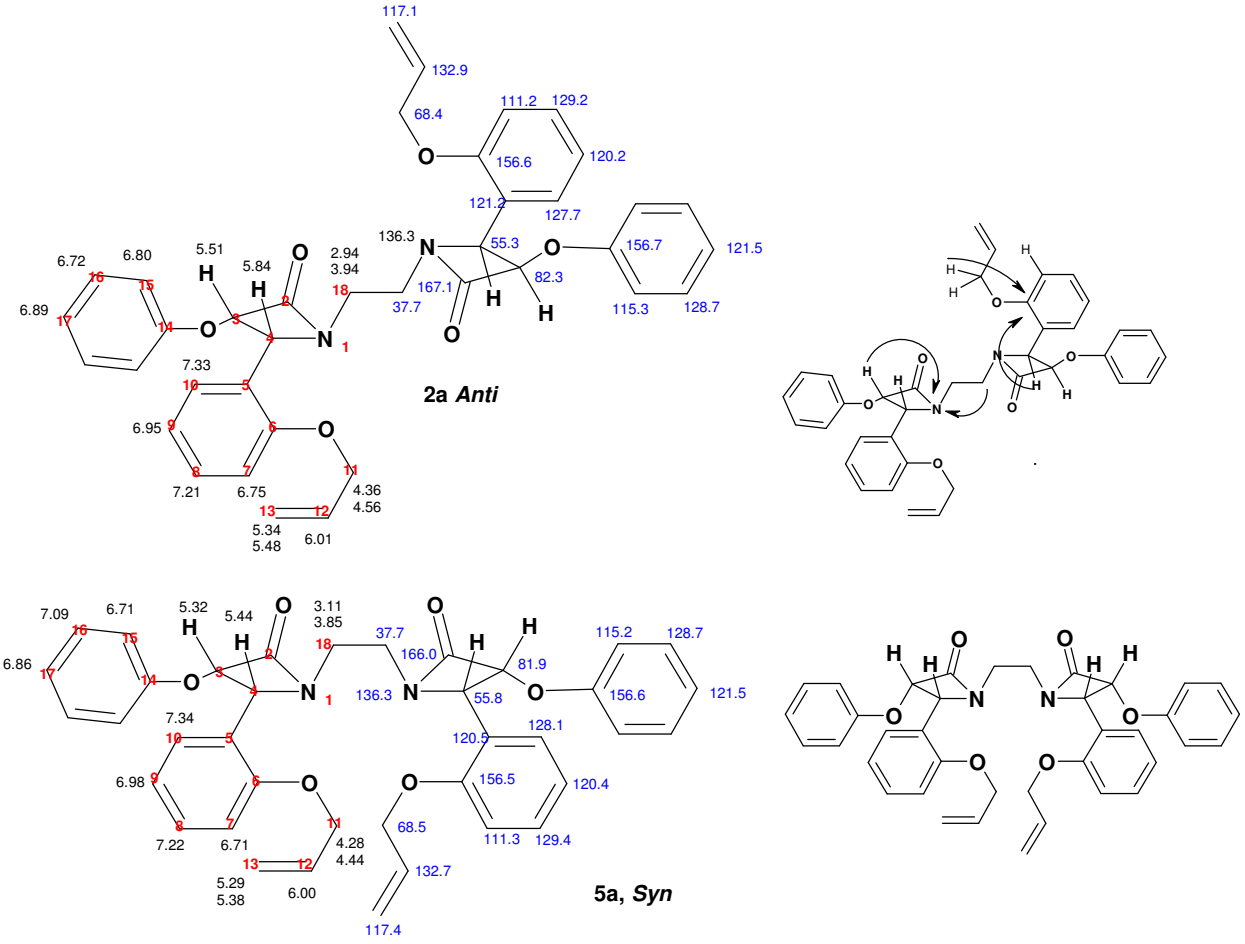


Figure 4: ^{1}H, ^{13}C and ^{15}N NMR spectroscopy assignment of **2a, 5a**

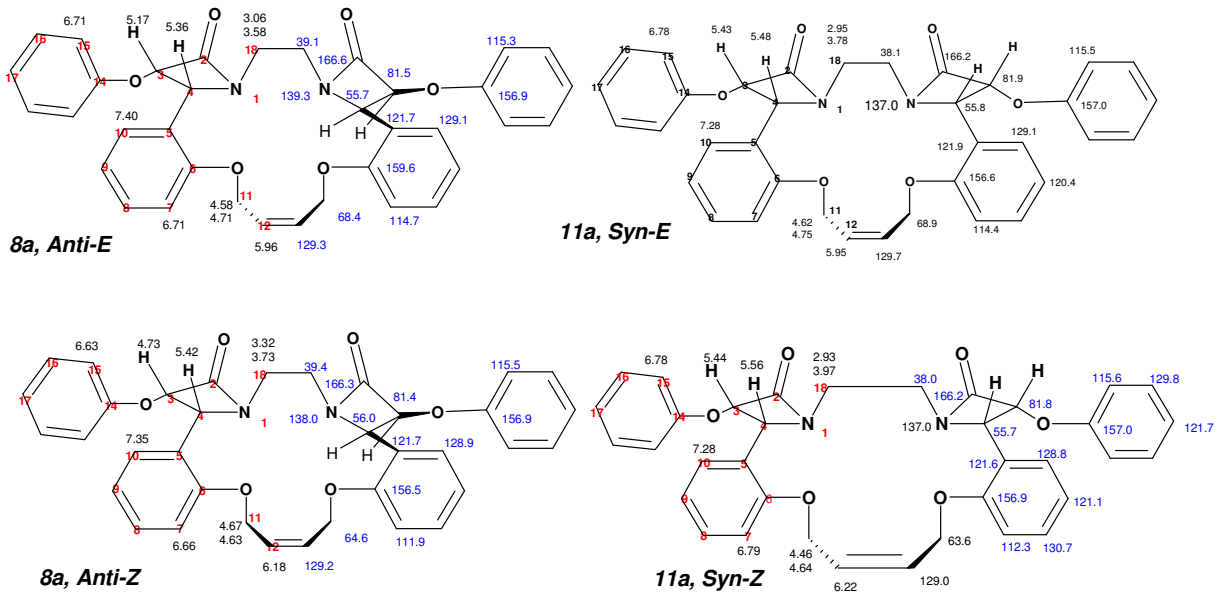
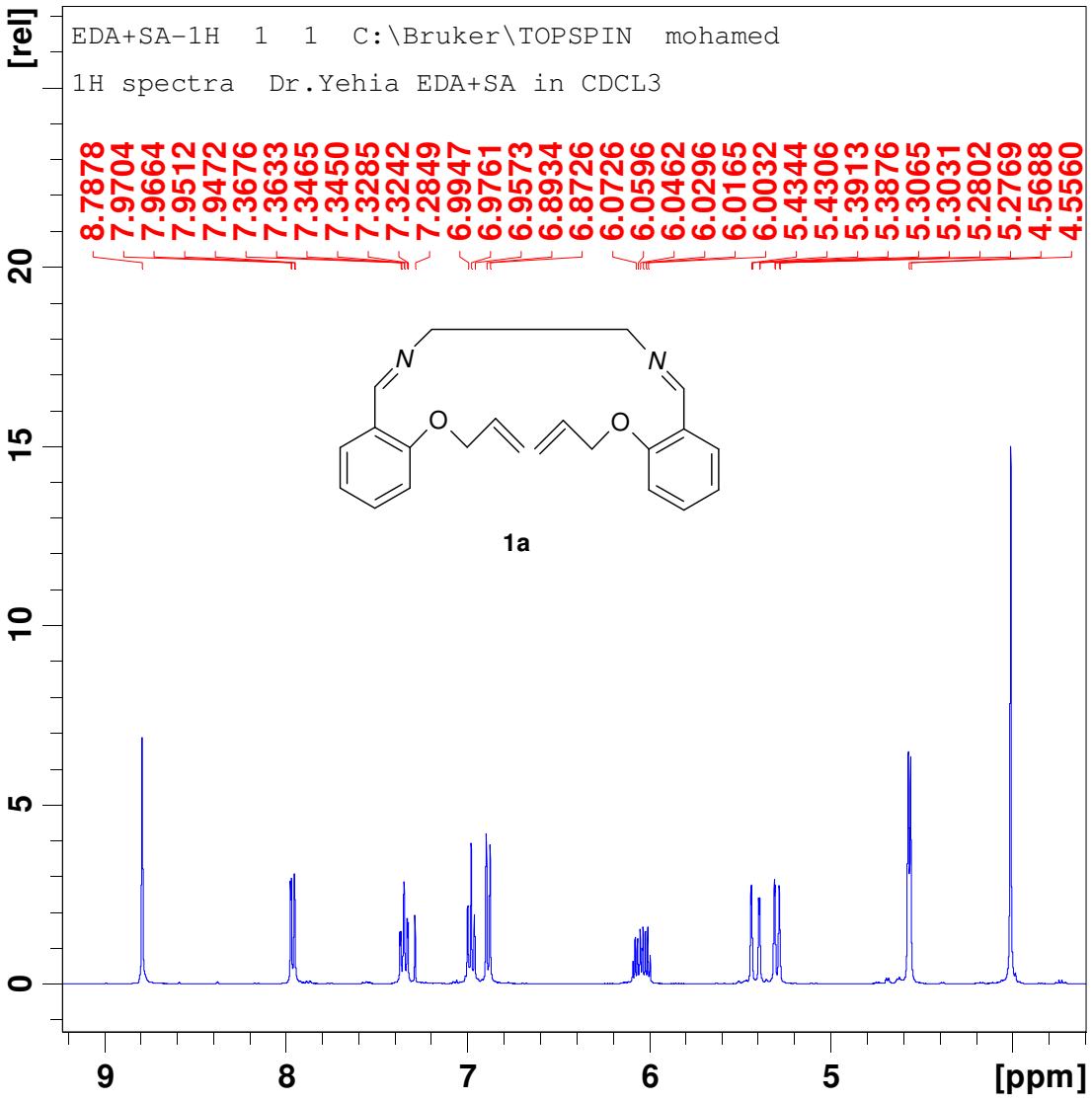
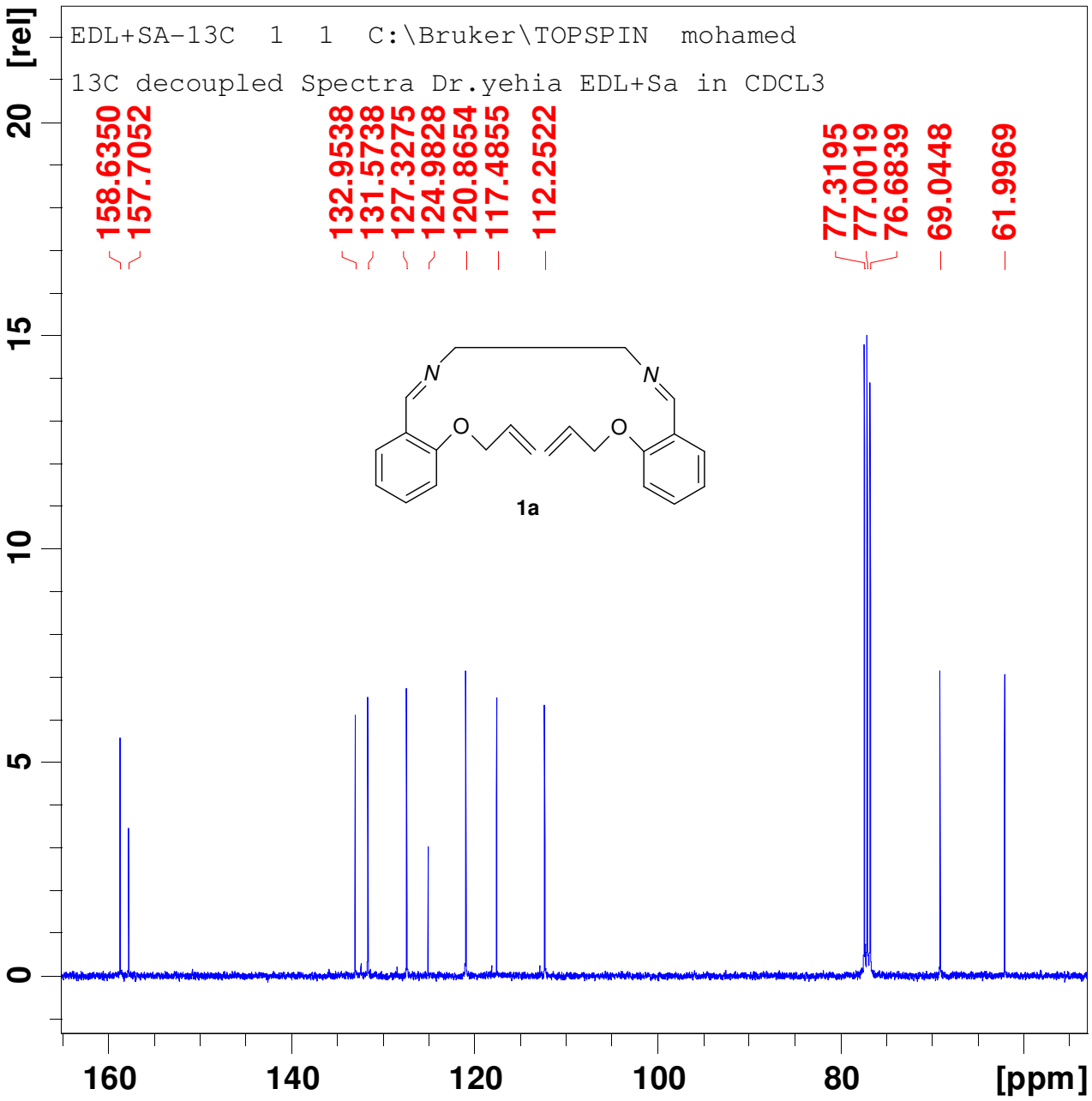
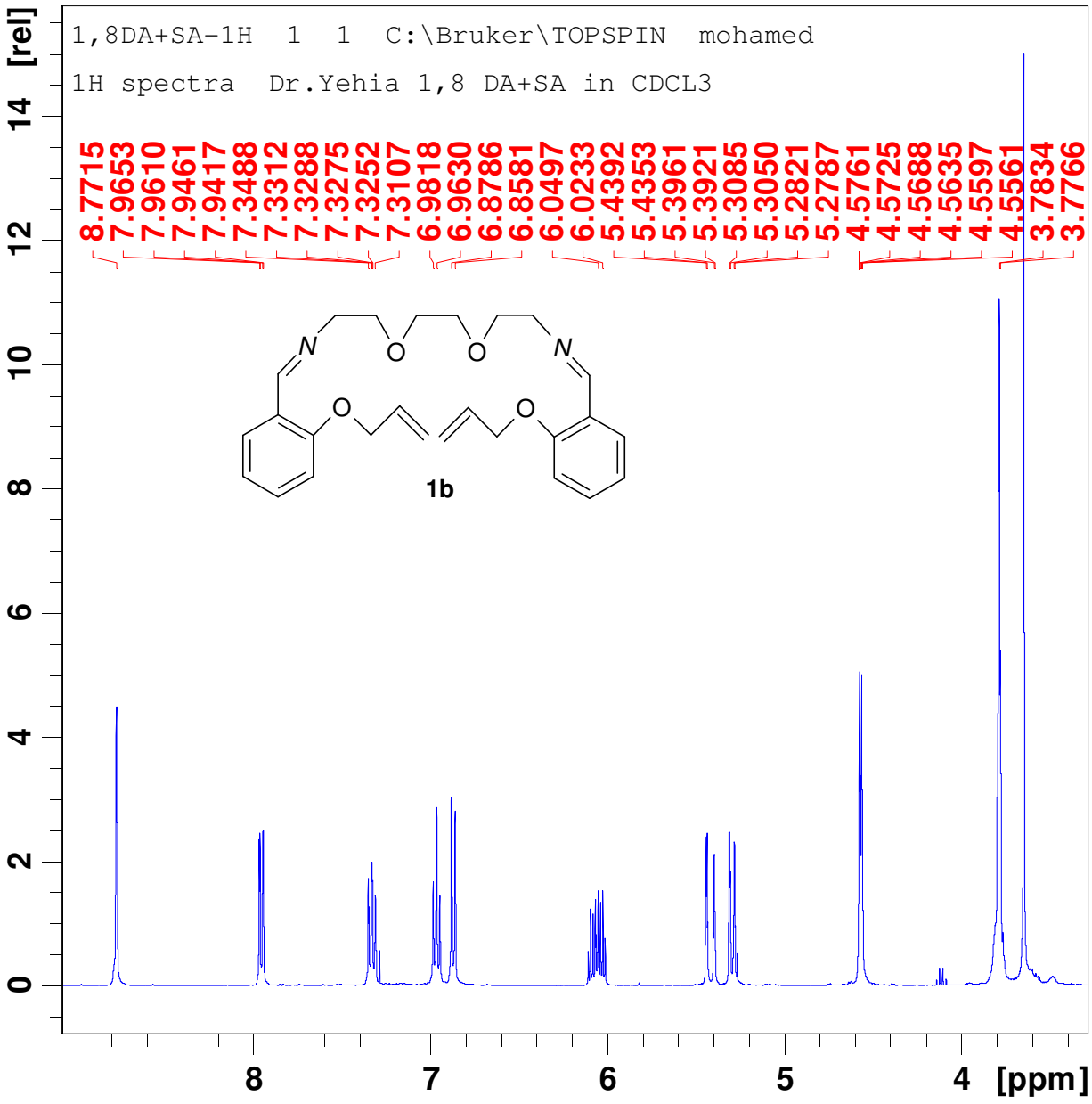


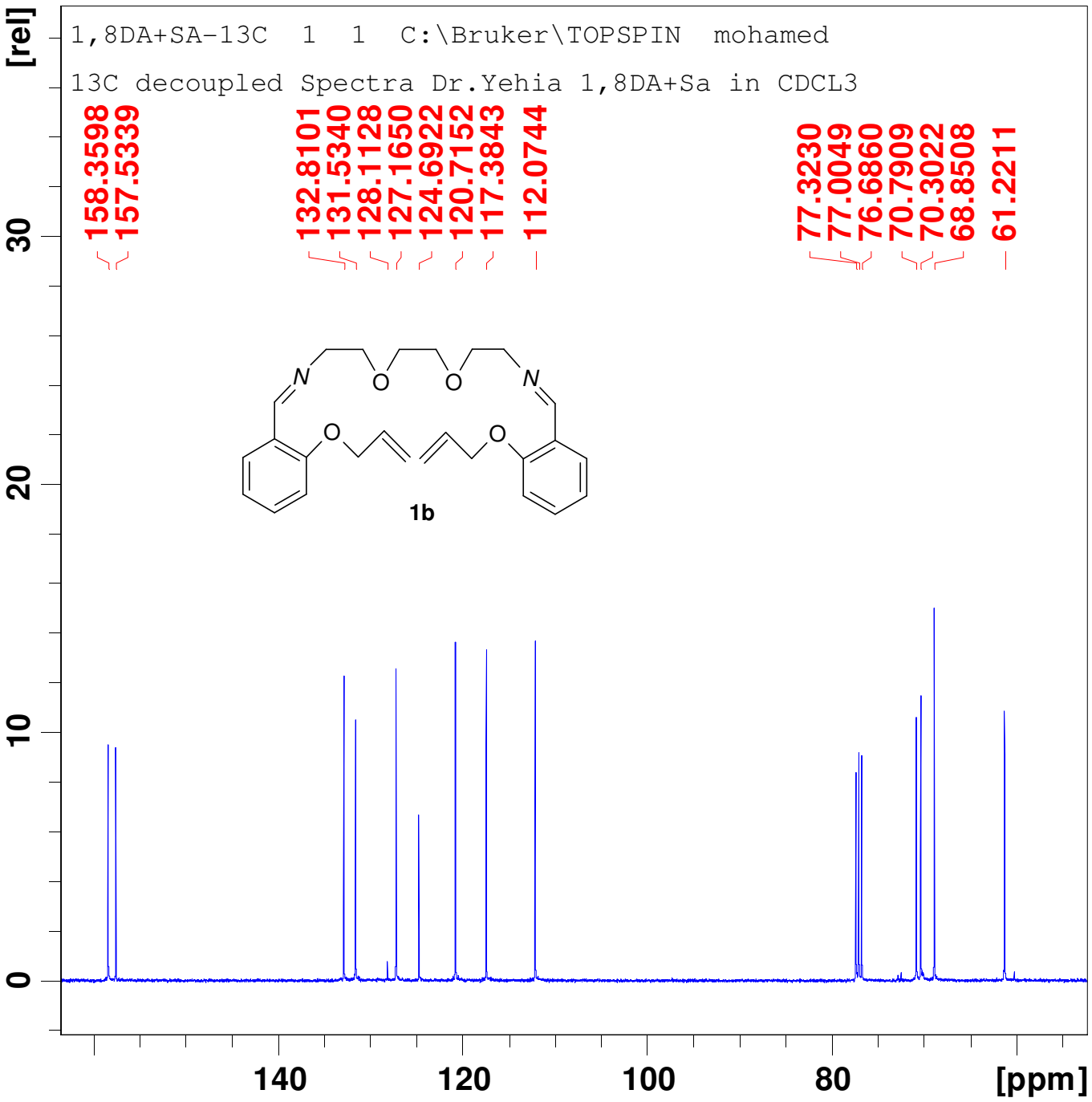
Figure 5: ^{1}H, ^{13}C and ^{15}N NMR spectroscopy assignment of ***E*-8a, *E*-11a, *Z*-8a, *Z*-11a**

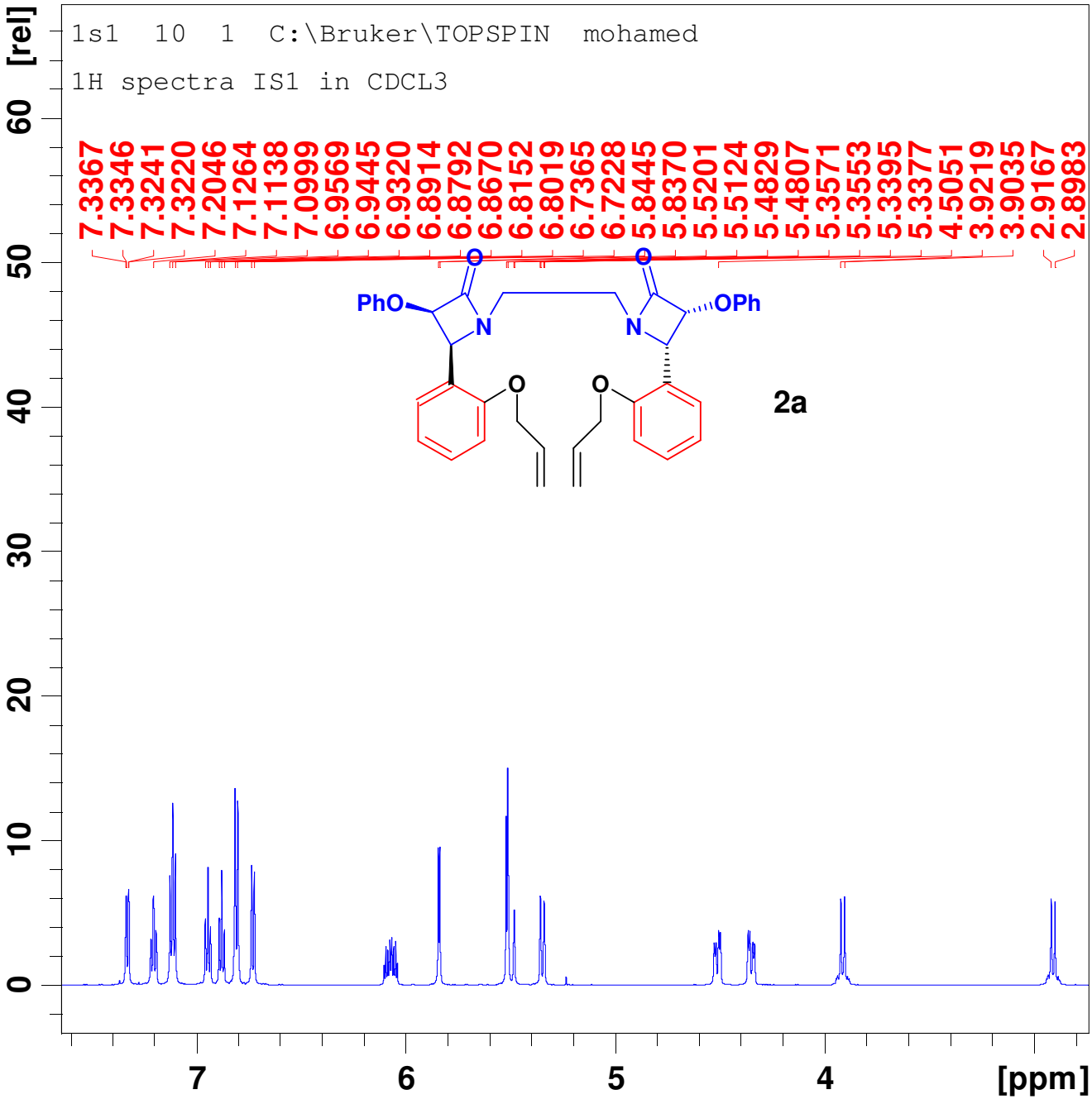
IV. ^{1}H and ^{13}C NMR spectra

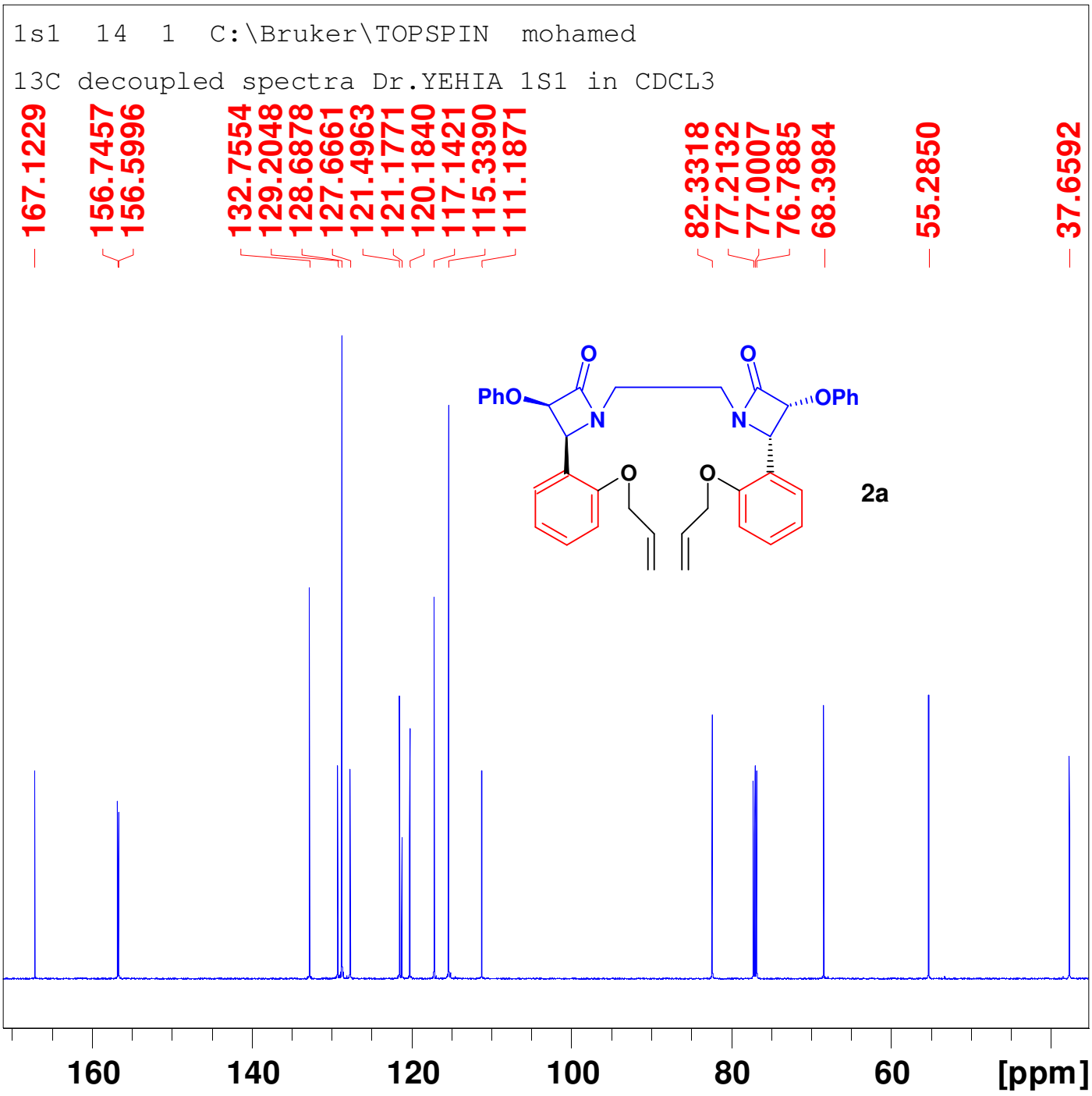


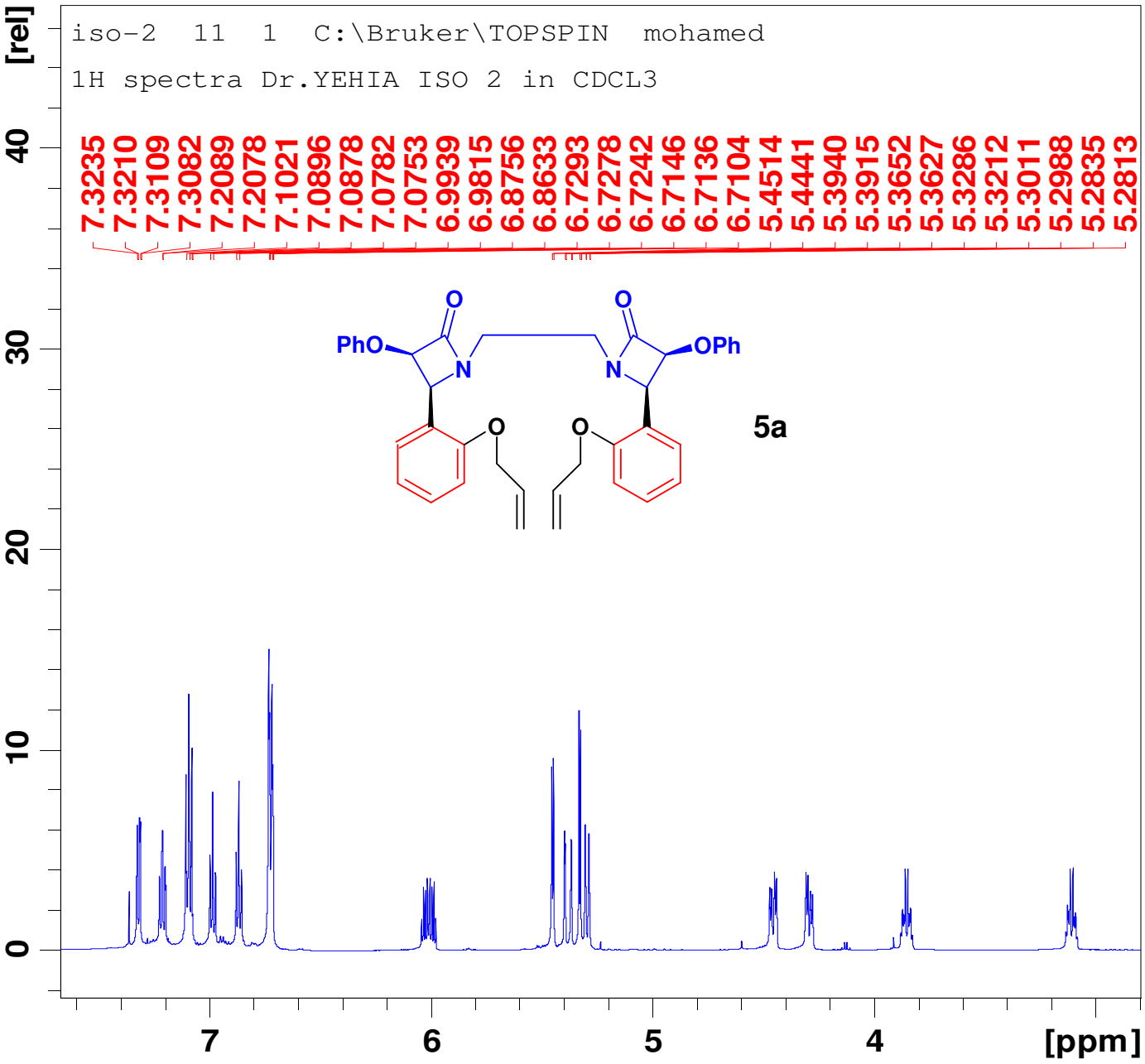

EDL+SA-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.yehia EDL+Sa in CDCL3
158.6350
157.7052
132.9538
131.5738
127.3275
124.9828
120.8654
117.4855
112.2522
77.3195
77.0019
76.6839
69.0448
61.9969
N
N
O
O
1a
[rel]
20
15
10
5
0
160
140
120
100
80
[ppm]

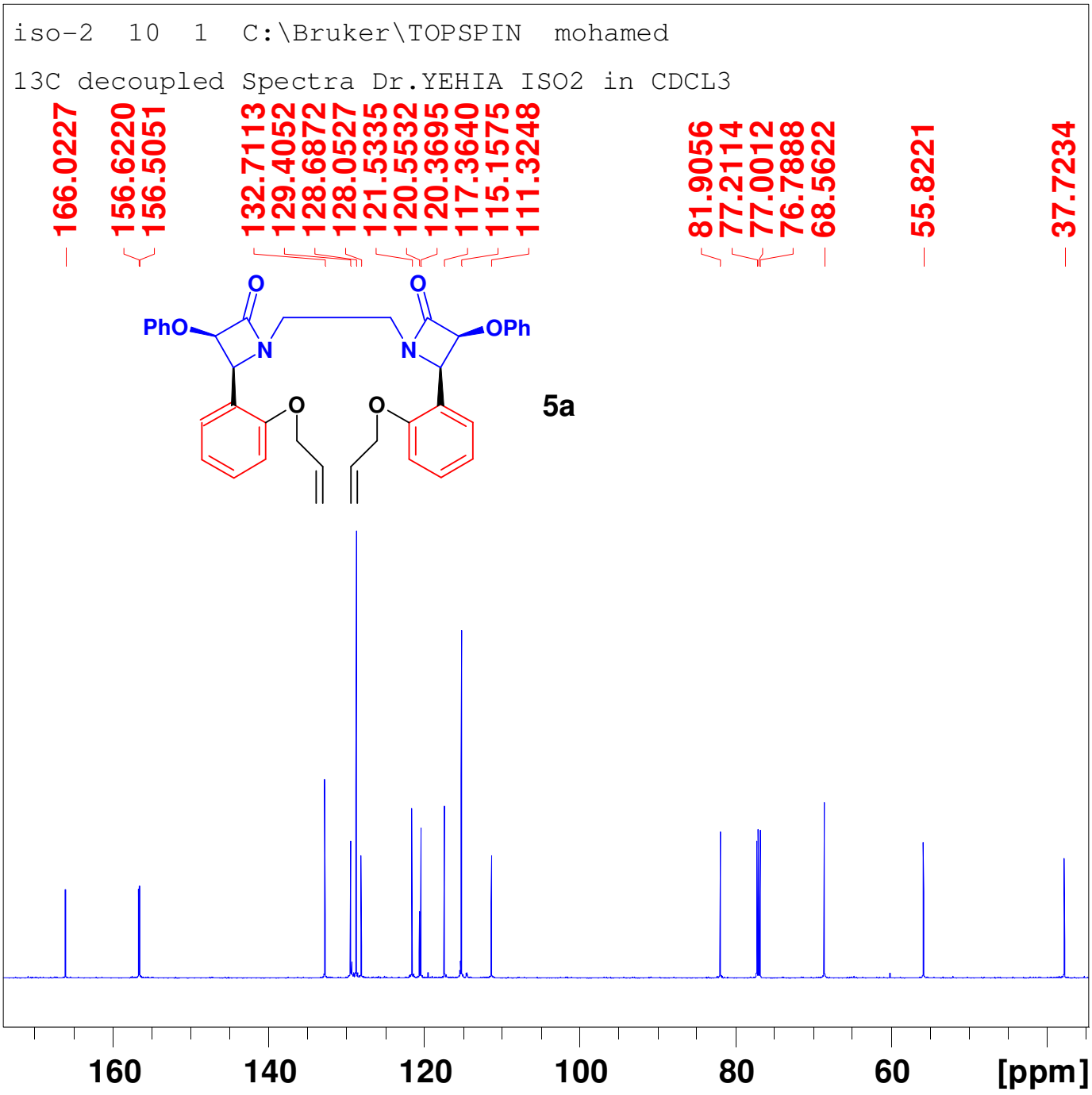

1,8DA+SA-1H 1 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia 1,8 DA+SA in CDCL3
8.7715
7.9653
7.9610
7.9461
7.9417
7.3488
7.3312
7.3288
7.3275
7.3252
7.3107
6.9818
6.9630
6.8786
6.8581
6.0497
6.0233
5.4392
5.4353
5.3961
5.3921
5.3085
5.3050
5.2821
5.2787
4.5761
4.5725
4.5688
4.5635
4.5597
4.5561
3.7834
3.7766
N
O
O
N
O
O
1b
[rel]
0
2
4
6
8
10
12
14
8
7
6
5
4
[ppm]

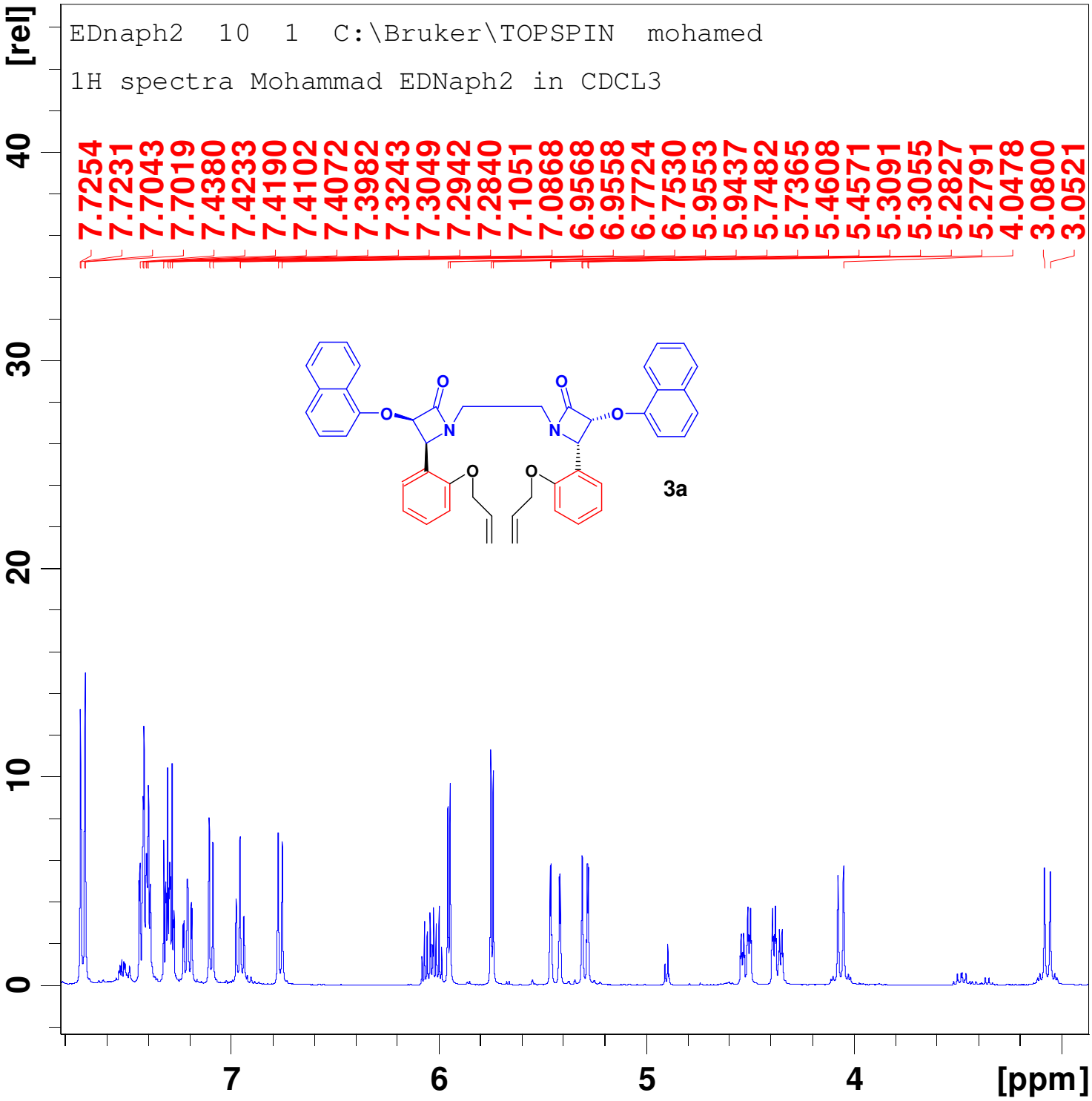

1,8DA+SA-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.Yehia 1,8DA+Sa in CDCL3
158.3598
157.5339
132.8101
131.5340
128.1128
127.1650
124.6922
120.7152
117.3843
112.0744
77.3230
77.0049
76.6860
70.7909
70.3022
68.8508
61.2211
1b
[rel]
0
10
20
30
140
120
100
80
[ppm]

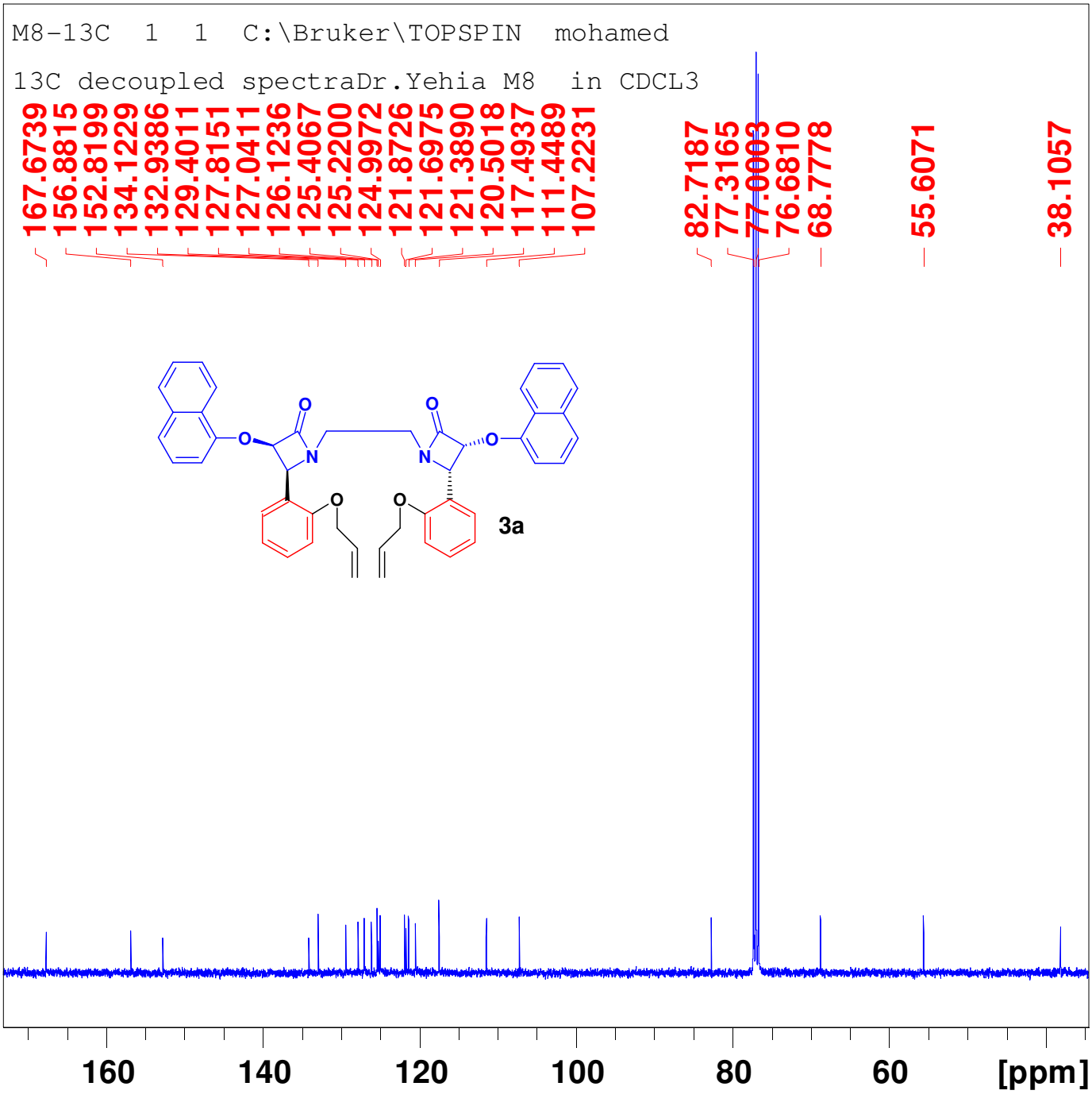

1s1 10 1 C:\Bruker\TOPSPIN mohamed
1H spectra IS1 in CDCL3
7.3367
7.3346
7.3241
7.3220
7.2046
7.1264
7.1138
7.0999
6.9569
6.9445
6.9320
6.8914
6.8792
6.8670
6.8152
6.8019
6.7365
6.7228
5.8445
5.8370
5.5201
5.5124
5.4829
5.4807
5.3571
5.3553
5.3395
5.3377
4.5051
3.9219
3.9035
2.9167
2.8983
PhO
OPh
N
N
O
O
O
O
2a
[rel]
0
10
20
30
40
50
60
7
6
5
4
[ppm]

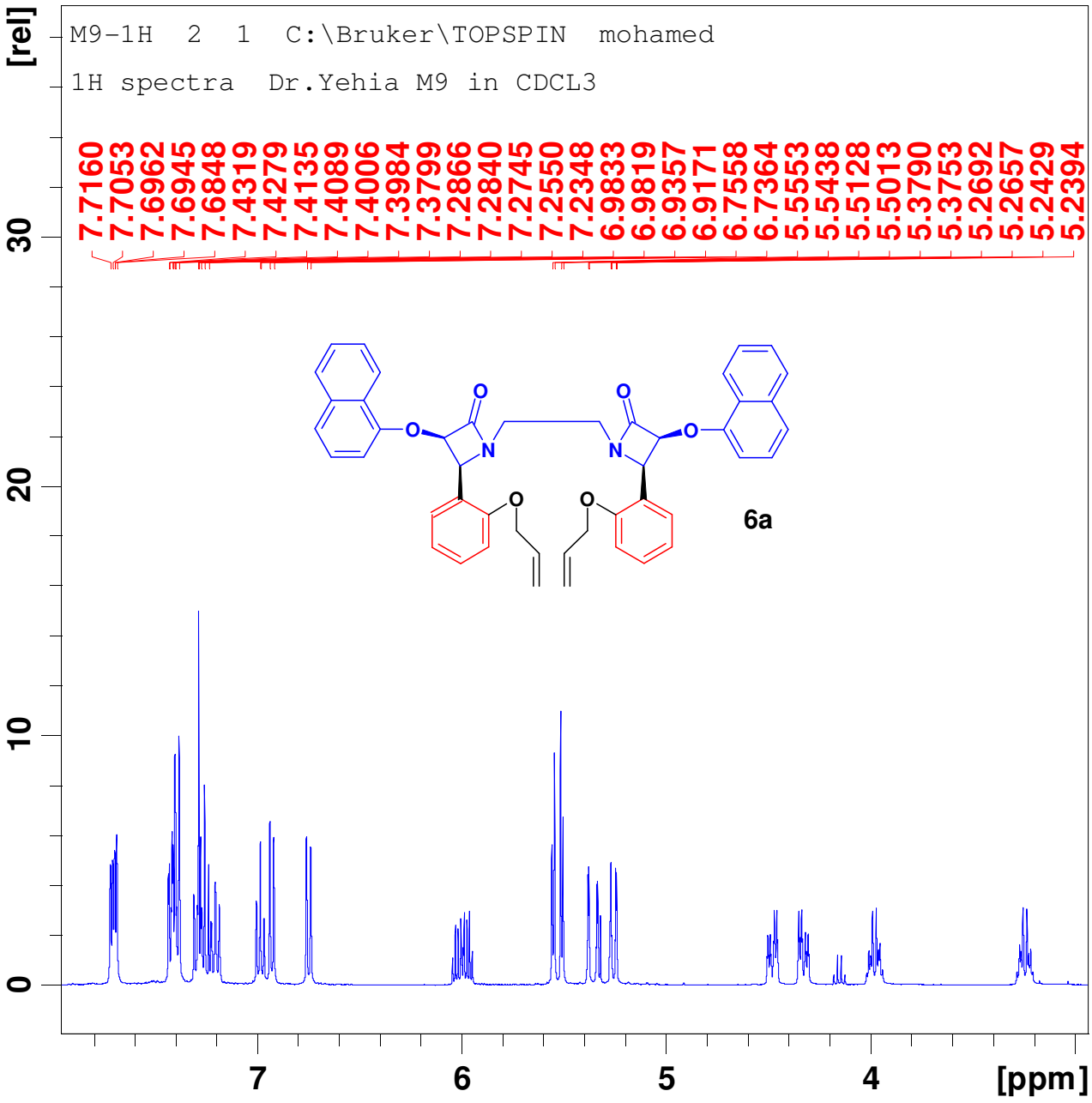

1s1 14 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra Dr.YEHIA 1S1 in CDCL3
167.1229
156.7457
156.5996
132.7554
129.2048
128.6878
127.6661
121.4963
121.1771
120.1840
117.1421
115.3390
111.1871
82.3318
77.2132
77.0007
76.7885
68.3984
55.2850
37.6592
PhO
O
N
O
N
OPh
O
O
2a
160
140
120
100
80
60
[ppm]

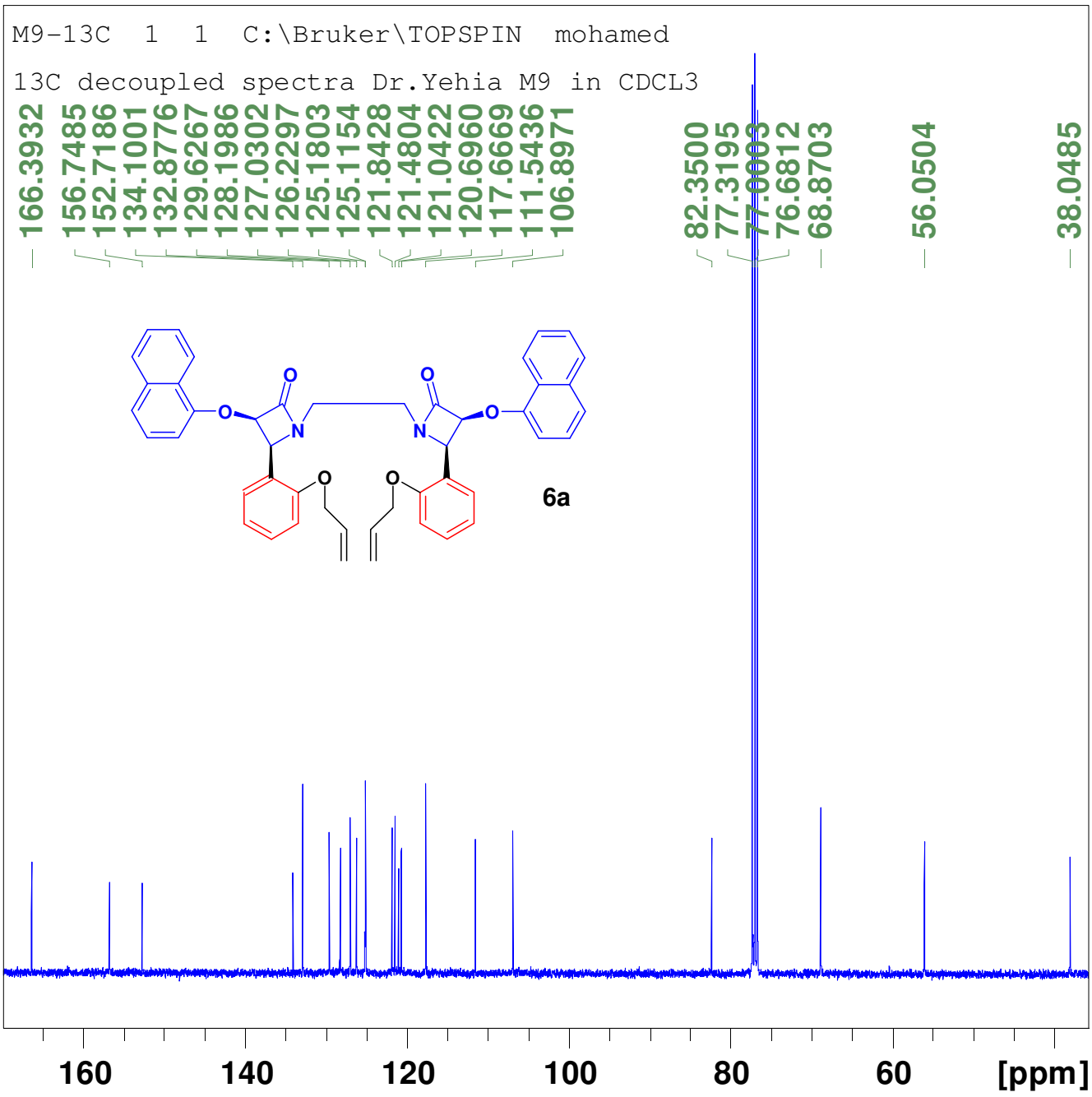

iso-2 11 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.YEHIA ISO 2 in CDCL3
7.3235
7.3210
7.3109
7.3082
7.2089
7.2078
7.1021
7.0896
7.0878
7.0782
7.0753
6.9939
6.9815
6.8756
6.8633
6.7293
6.7278
6.7242
6.7146
6.7136
6.7104
5.4514
5.4441
5.3940
5.3915
5.3652
5.3627
5.3286
5.3212
5.3011
5.2988
5.2835
5.2813
PhO
OPh
N
N
O
O
O
O
5a
[rel]
0
10
20
30
40
7
6
5
4
[ppm]

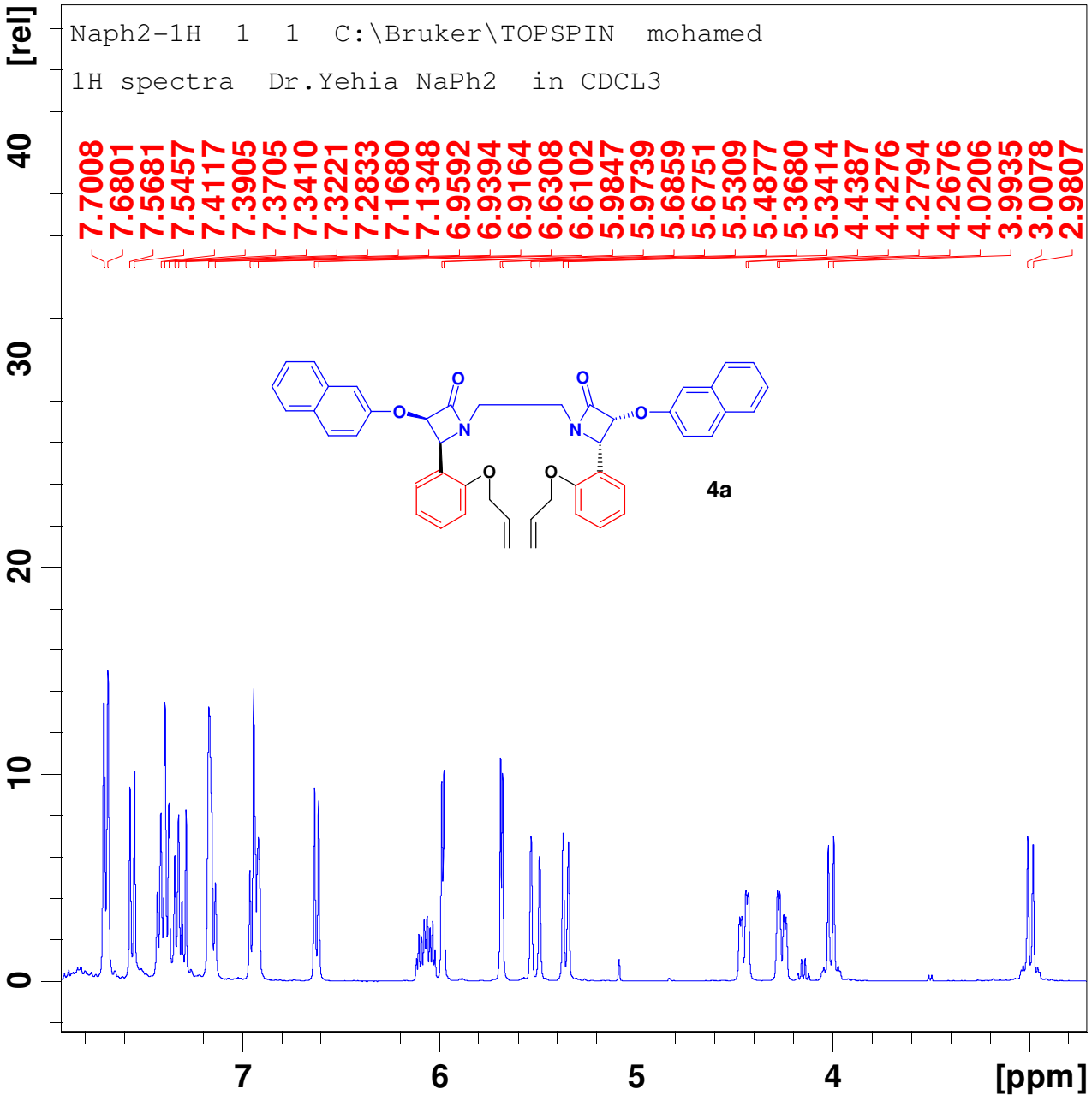

iso-2 10 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.YEHIA ISO2 in CDCL3
166.0227
156.6220
156.5051
132.7113
129.4052
128.6872
128.0527
121.5335
120.5532
120.3695
117.3640
115.1575
111.3248
81.9056
77.2114
77.0012
76.7888
68.5622
55.8221
37.7234
PhO
OPh
N
N
O
O
O
O
5a
160
140
120
100
80
60
[ppm]

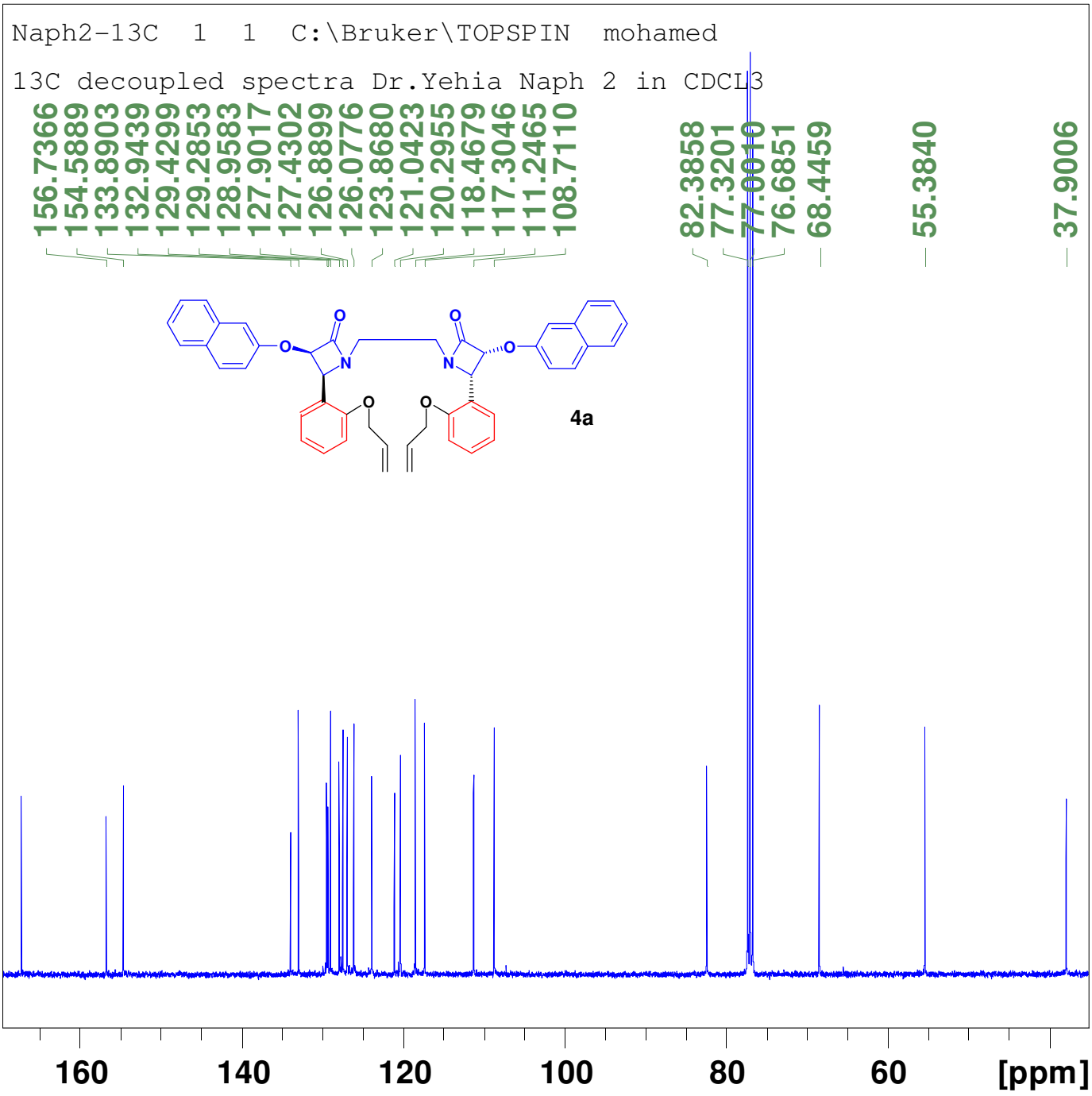

EDnaph2 10 1 C:\Bruker\TOPSPIN mohamed
1H spectra Mohammad EDNaph2 in CDCL3
7.7254
7.7231
7.7043
7.7019
7.4380
7.4233
7.4190
7.4102
7.4072
7.3982
7.3243
7.3049
7.2942
7.2840
7.1051
7.0868
6.9568
6.9558
6.7724
6.7530
5.9553
5.9437
5.7482
5.7365
5.4608
5.4571
5.3091
5.3055
5.2827
5.2791
4.0478
3.0800
3.0521
3a
[rel]
40
30
20
10
0
7
6
5
4
[ppm]

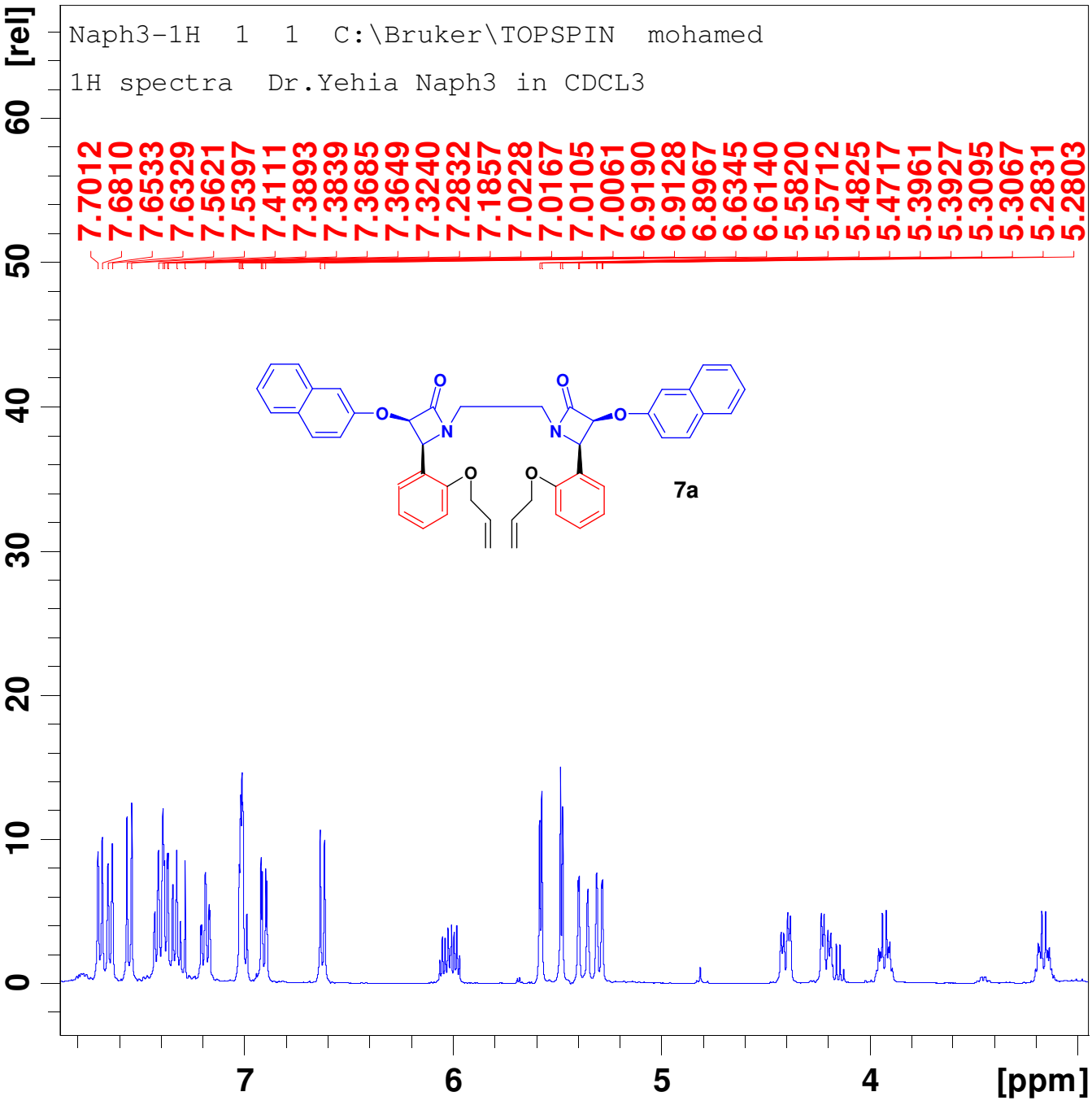

M8-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectraDr.Yehia M8 in CDCL3
167.6739
156.8815
152.8199
134.1229
132.9386
129.4011
127.8151
127.0411
126.1236
125.4067
125.2200
124.9972
121.8726
121.6975
121.3890
120.5018
117.4937
111.4489
107.2231
82.7187
77.3165
77.0003
76.6810
68.7778
55.6071
38.1057
3a
160
140
120
100
80
60
[ppm]

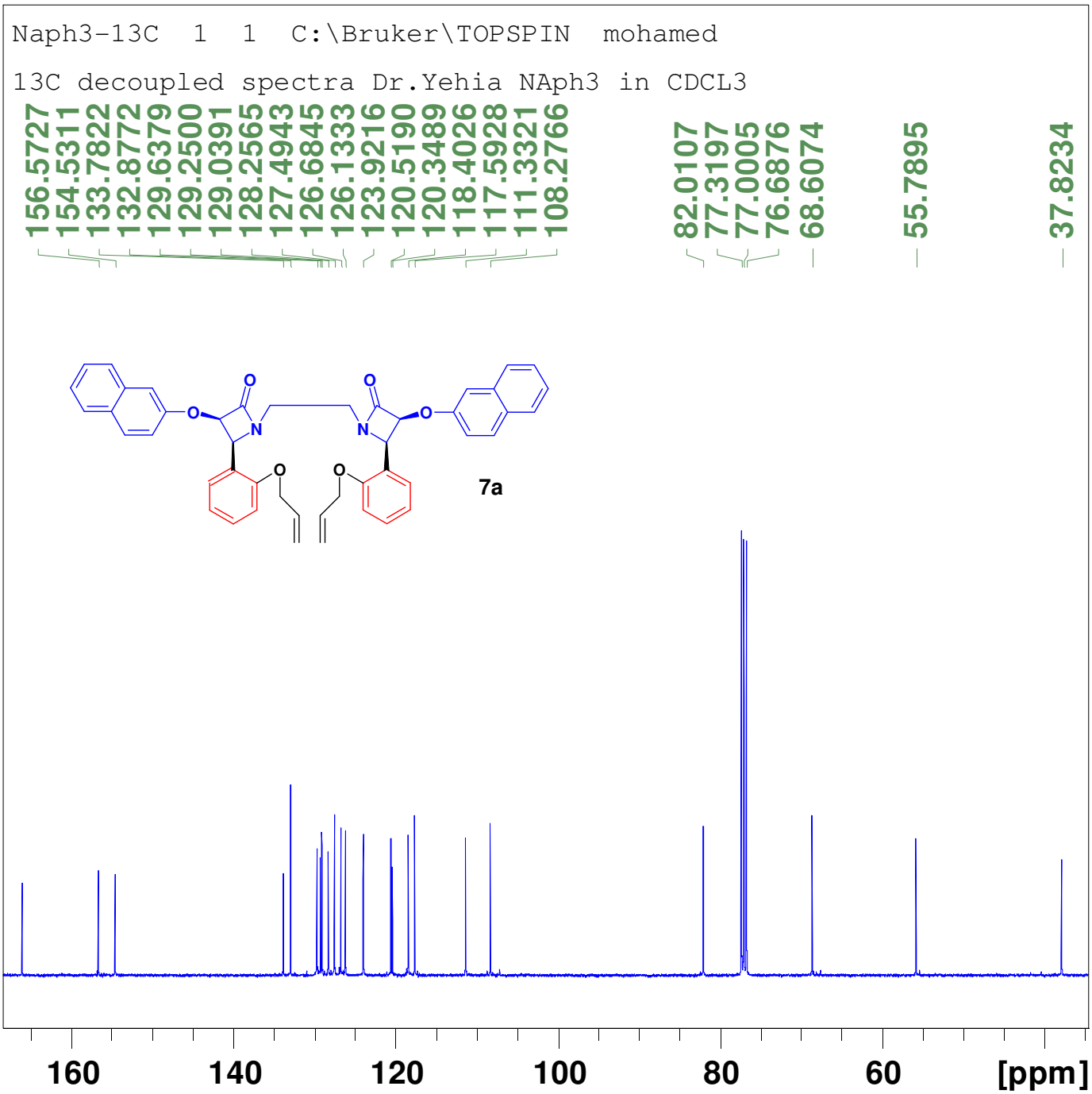

M9-1H 2 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia M9 in CDCL3
7.7160
7.7053
7.6962
7.6945
7.6848
7.4319
7.4279
7.4135
7.4089
7.4006
7.3984
7.3799
7.2866
7.2840
7.2745
7.2550
7.2348
6.9833
6.9819
6.9357
6.9171
6.7558
6.7364
5.5553
5.5438
5.5128
5.5013
5.3790
5.3753
5.2692
5.2657
5.2429
5.2394
[rel]
30
20
10
0
7
6
5
4
[ppm]
6a

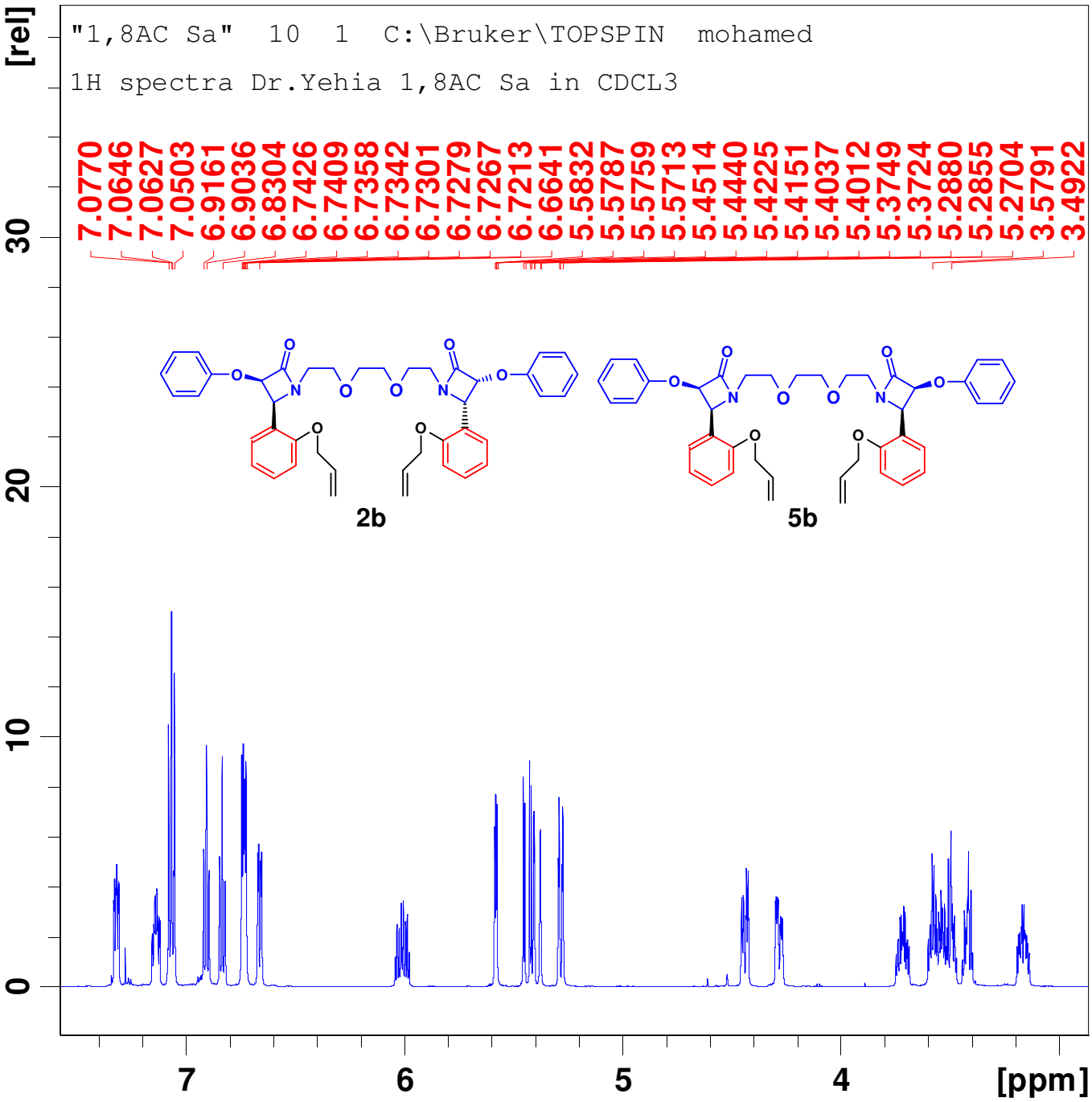

M9-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra Dr.Yehia M9 in CDCL3
166.3932
156.7485
152.7186
134.1001
132.8776
129.6267
128.1986
127.0302
126.2297
125.1803
125.1154
121.8428
121.4804
121.0422
120.6960
117.6669
111.5436
106.8971
82.3500
77.3195
77.0003
76.6812
68.8703
56.0504
38.0485
6a
160
140
120
100
80
60
[ppm]

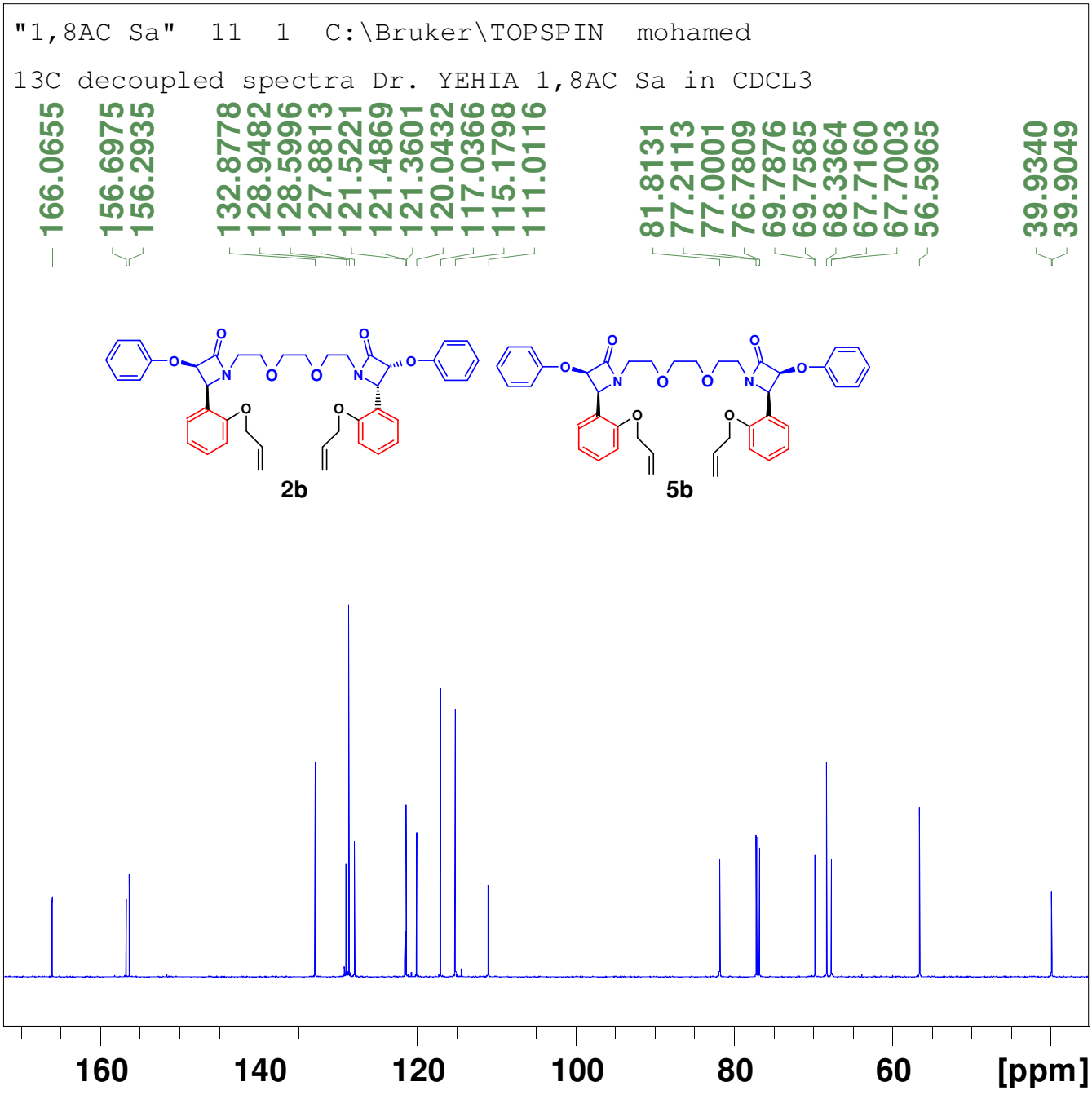

Naph2-1H 1 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia NaPh2 in CDCL3
7.7008
7.6801
7.5681
7.5457
7.4117
7.3905
7.3705
7.3410
7.3221
7.2833
7.1680
7.1348
6.9592
6.9394
6.9164
6.6308
6.6102
5.9847
5.9739
5.6859
5.6751
5.5309
5.4877
5.3680
5.3414
4.4387
4.4276
4.2794
4.2676
4.0206
3.9935
3.0078
2.9807
4a
[rel]
40
30
20
10
0
7
6
5
4
[ppm]

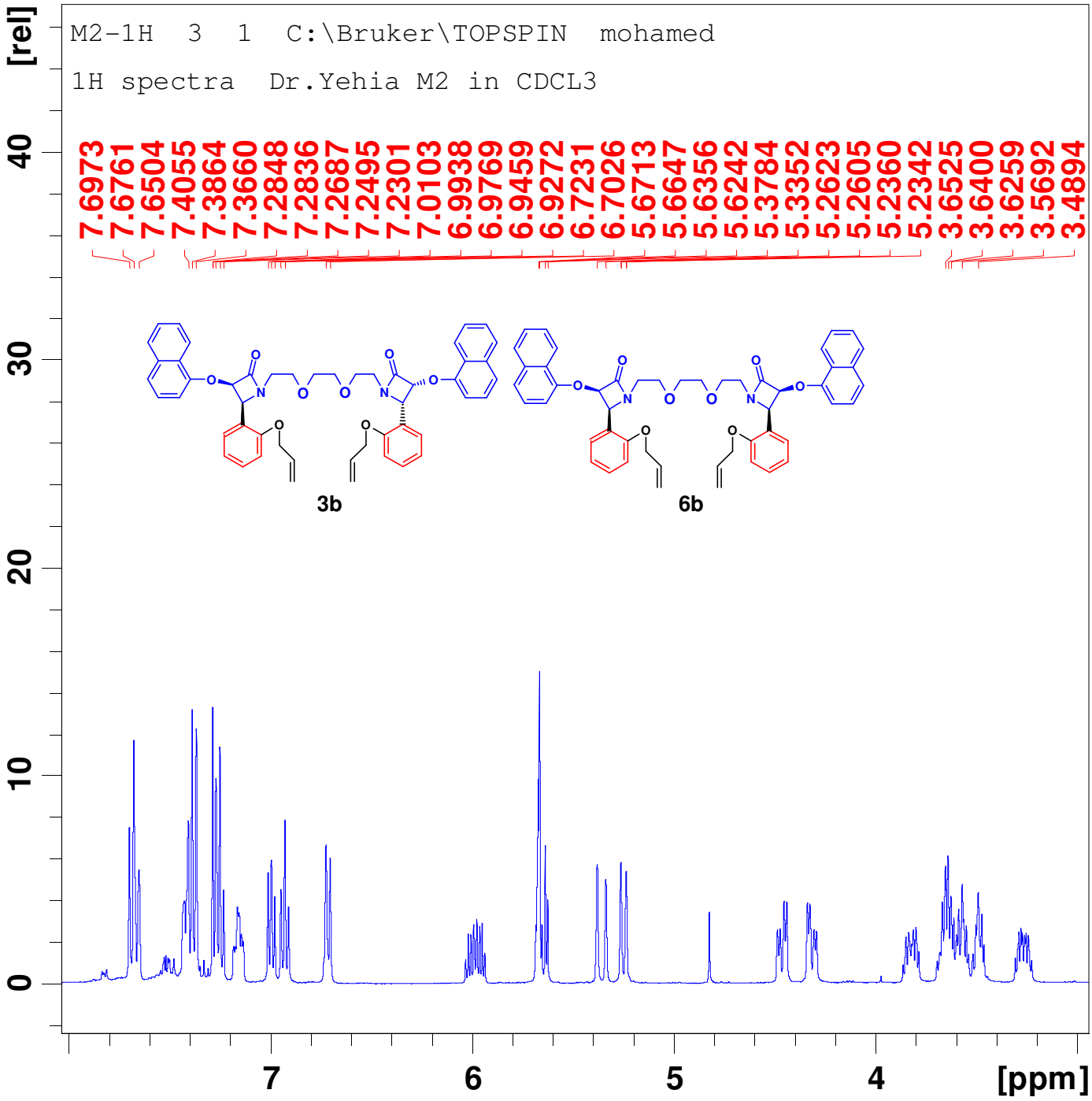

Naph2-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra Dr.Yehia Naph 2 in CDCL3
156.7366
154.5889
133.8903
132.9439
129.4299
129.2853
128.9583
127.9017
127.4302
126.8899
126.0776
123.8680
121.0423
120.2955
118.4679
117.3046
111.2465
108.7110
82.3858
77.3201
77.0010
76.6851
68.4459
55.3840
37.9006
4a
160
140
120
100
80
60
[ppm]

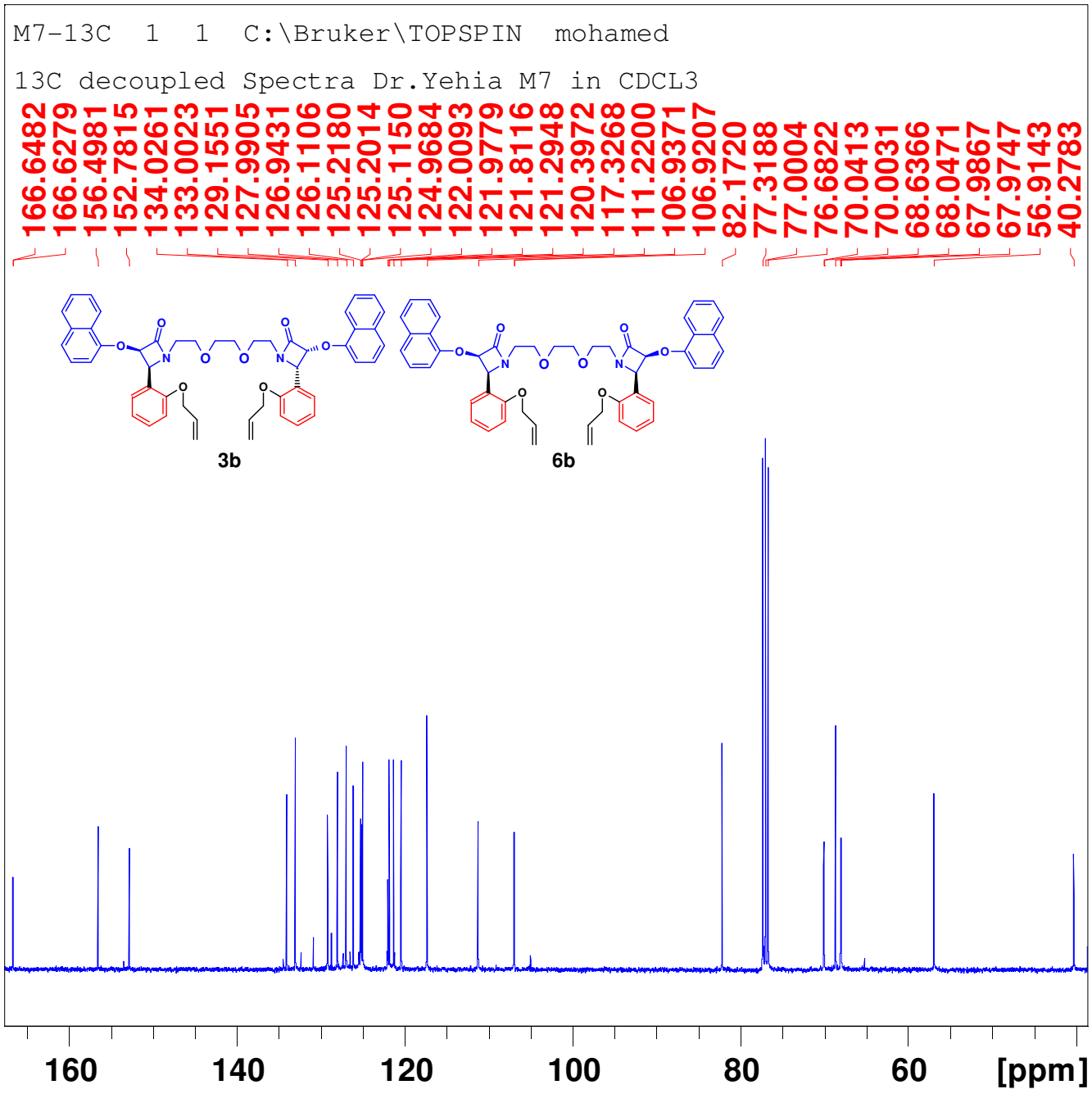

Naph3-1H 1 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia Naph3 in CDCL3
7.7012
7.6810
7.6533
7.6329
7.5621
7.5397
7.4111
7.3893
7.3839
7.3685
7.3649
7.3240
7.2832
7.1857
7.0228
7.0167
7.0105
7.0061
6.9190
6.9128
6.8967
6.6345
6.6140
5.5820
5.5712
5.4825
5.4717
5.3961
5.3927
5.3095
5.3067
5.2831
5.2803
7a
[rel]
60
50
40
30
20
10
0
7
6
5
4
[ppm]

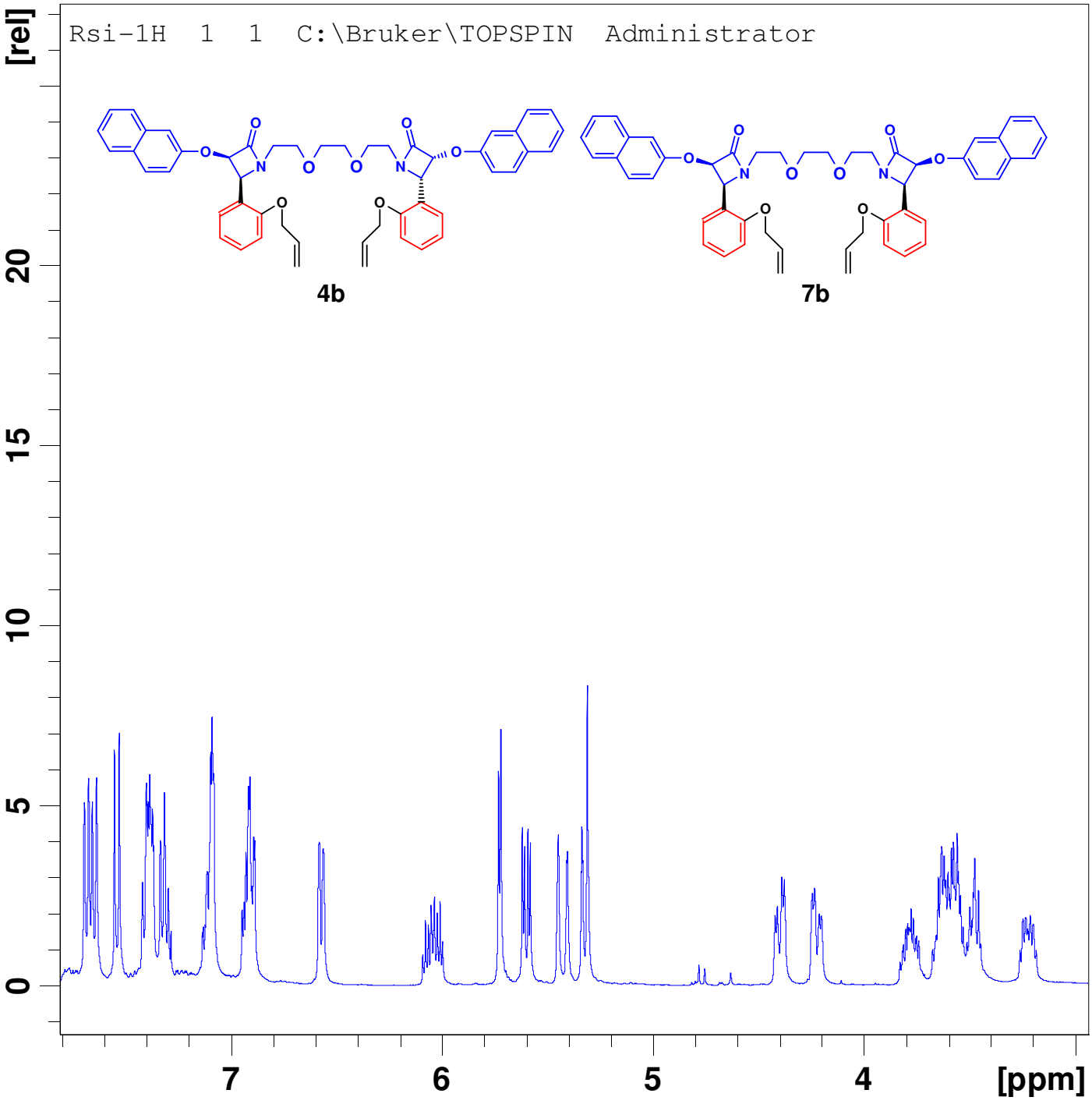

Naph3-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra Dr.Yehia NAph3 in CDCL3
156.5727
154.5311
133.7822
132.8772
129.6379
129.2500
129.0391
128.2565
127.4943
126.6845
126.1333
123.9216
120.5190
120.3489
118.4026
117.5928
111.3321
108.2766
82.0107
77.3197
77.0005
76.6876
68.6074
55.7895
37.8234
7a
160
140
120
100
80
60
[ppm]

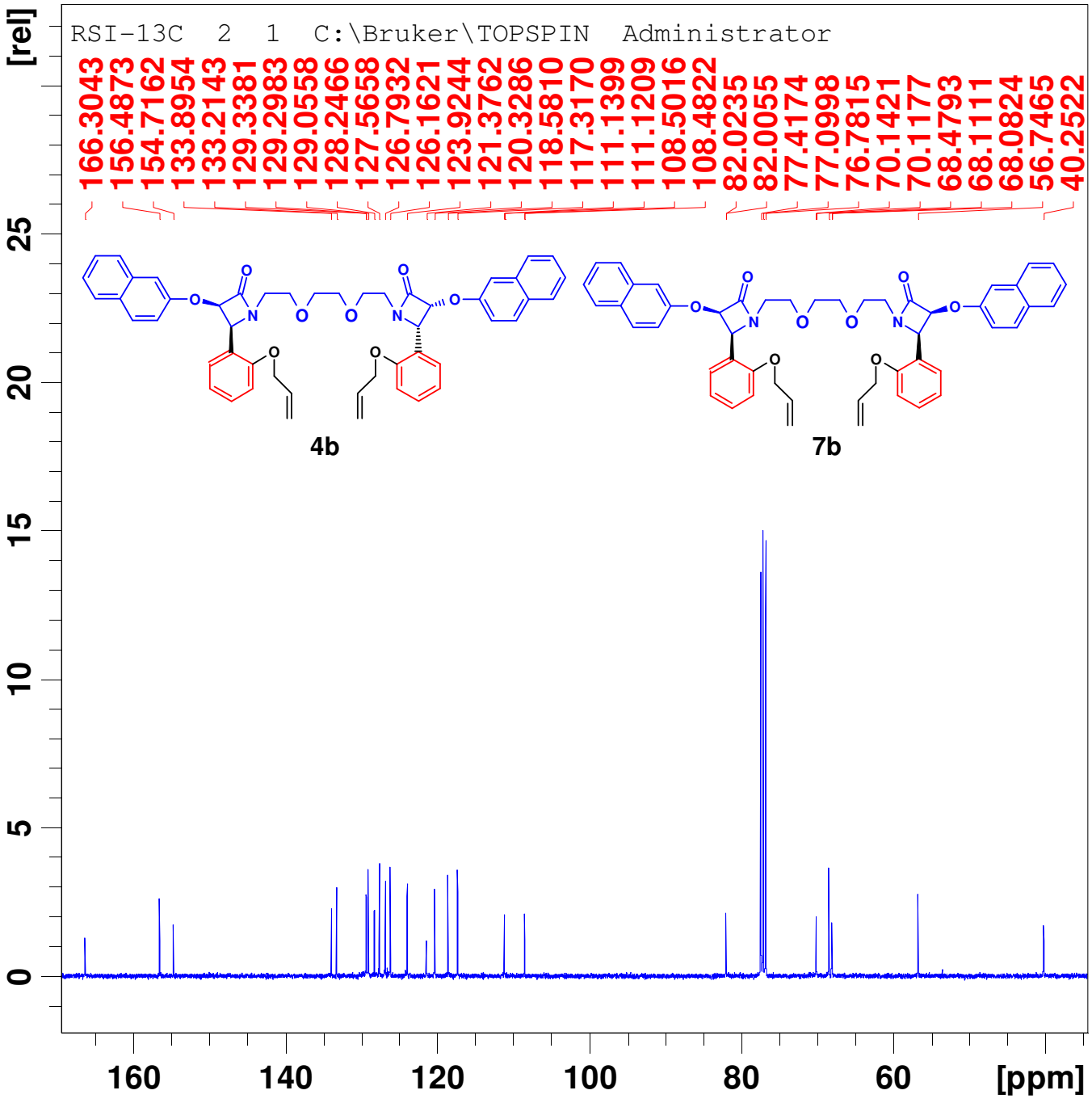

"1,8AC Sa" 10 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia 1,8AC Sa in CDCL3
7.0770
7.0646
7.0627
7.0503
6.9161
6.9036
6.8304
6.7426
6.7409
6.7358
6.7342
6.7301
6.7279
6.7267
6.7213
6.6641
5.5832
5.5787
5.5759
5.5713
5.4514
5.4440
5.4225
5.4151
5.4037
5.4012
5.3749
5.3724
5.2880
5.2855
5.2704
3.5791
3.4922
2b
5b
[rel]
0
10
20
30
7
6
5
4
[ppm]

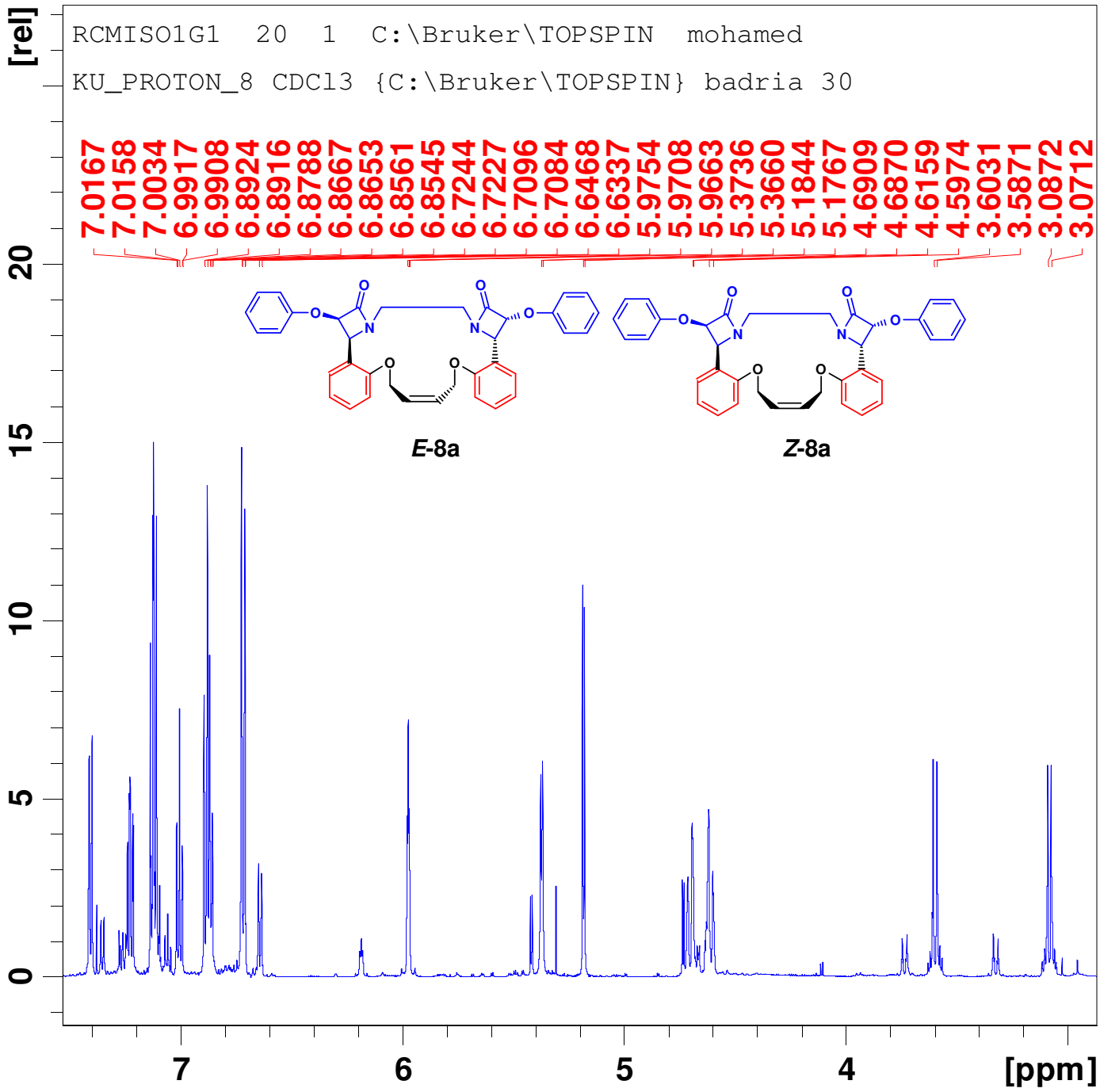

"1,8AC Sa" 11 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra Dr. YEHIA 1,8AC Sa in CDCL3
166.0655
156.6975
156.2935
132.8778
128.9482
128.5996
127.8813
121.5221
121.4869
121.3601
120.0432
117.0366
115.1798
111.0116
81.8131
77.2113
77.0001
76.7809
69.7876
69.7585
68.3364
67.7160
67.7003
56.5965
39.9340
39.9049
2b
5b
160
140
120
100
80
60
[ppm]

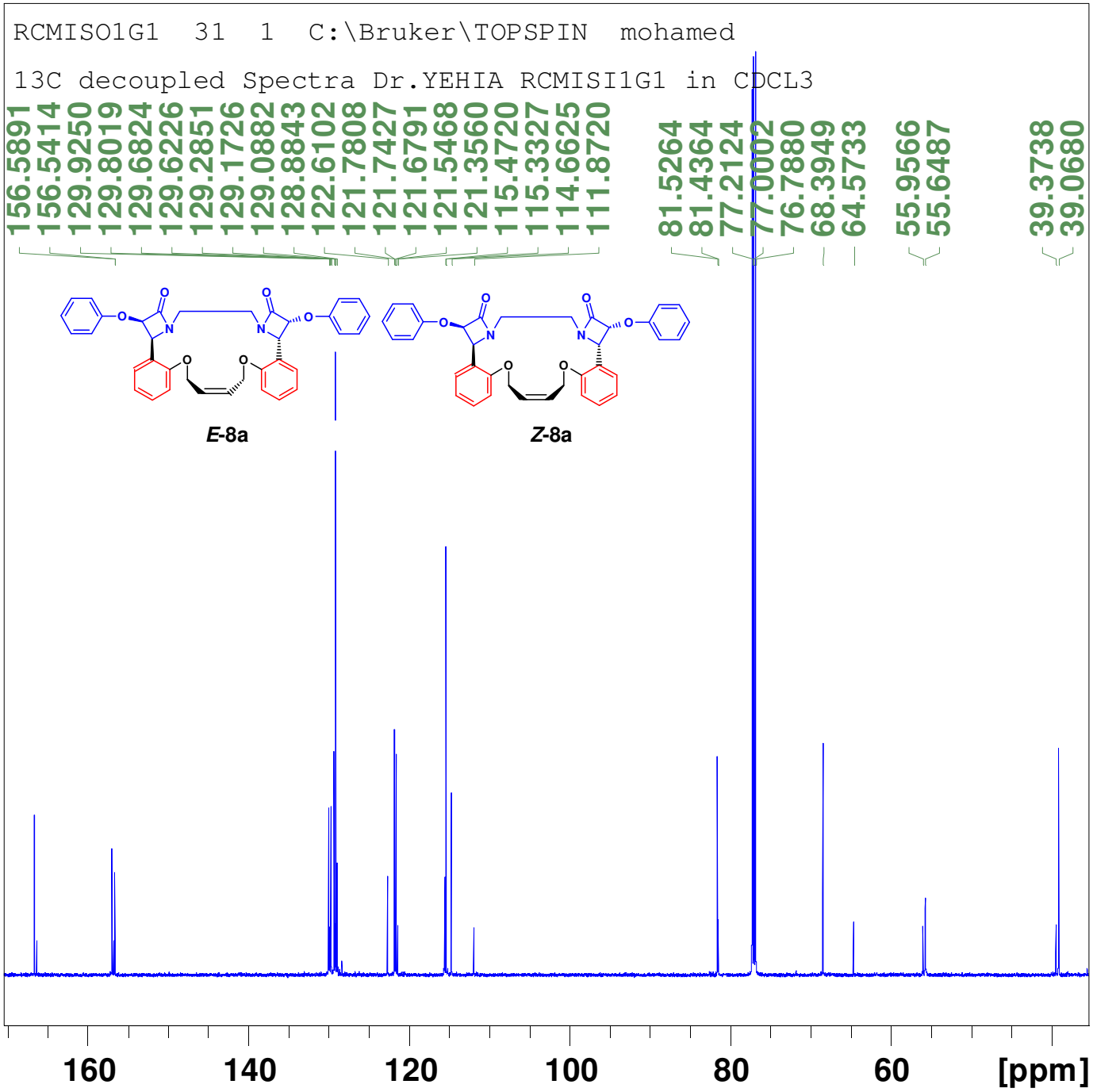

M2-1H 3 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia M2 in CDCL3
7.6973
7.6761
7.6504
7.4055
7.3864
7.3660
7.2848
7.2836
7.2687
7.2495
7.2301
7.0103
6.9938
6.9769
6.9459
6.9272
6.7231
6.7026
6.6713
5.6647
5.6356
5.6242
5.3784
5.3352
5.2623
5.2605
5.2360
5.2342
3.6525
3.6400
3.6259
3.5692
3.4894
3b
6b
[rel]
40
30
20
10
0
7
6
5
4
[ppm]

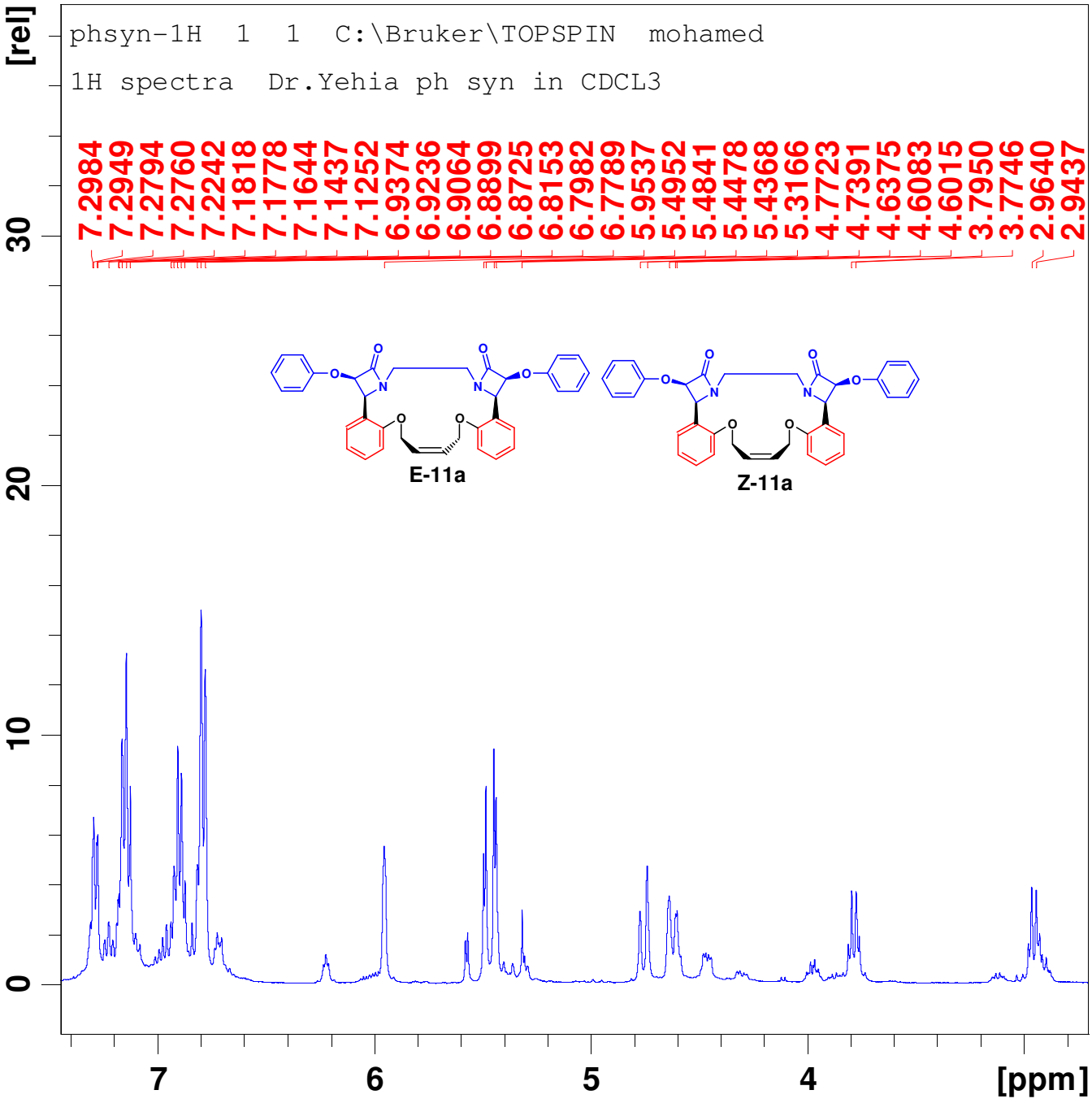

M7-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.Yehia M7 in CDCL3
166.6482
166.6279
156.4981
152.7815
134.0261
133.0023
129.1551
127.9905
126.9431
126.1106
125.2180
125.2014
125.1150
124.9684
122.0093
121.9779
121.8116
121.2948
120.3972
117.3268
111.2200
106.9371
106.9207
82.1720
77.3188
77.0004
76.6822
70.0413
70.0031
68.6366
68.0471
67.9867
67.9747
56.9143
40.2783
3b
6b
160
140
120
100
80
60
[ppm]

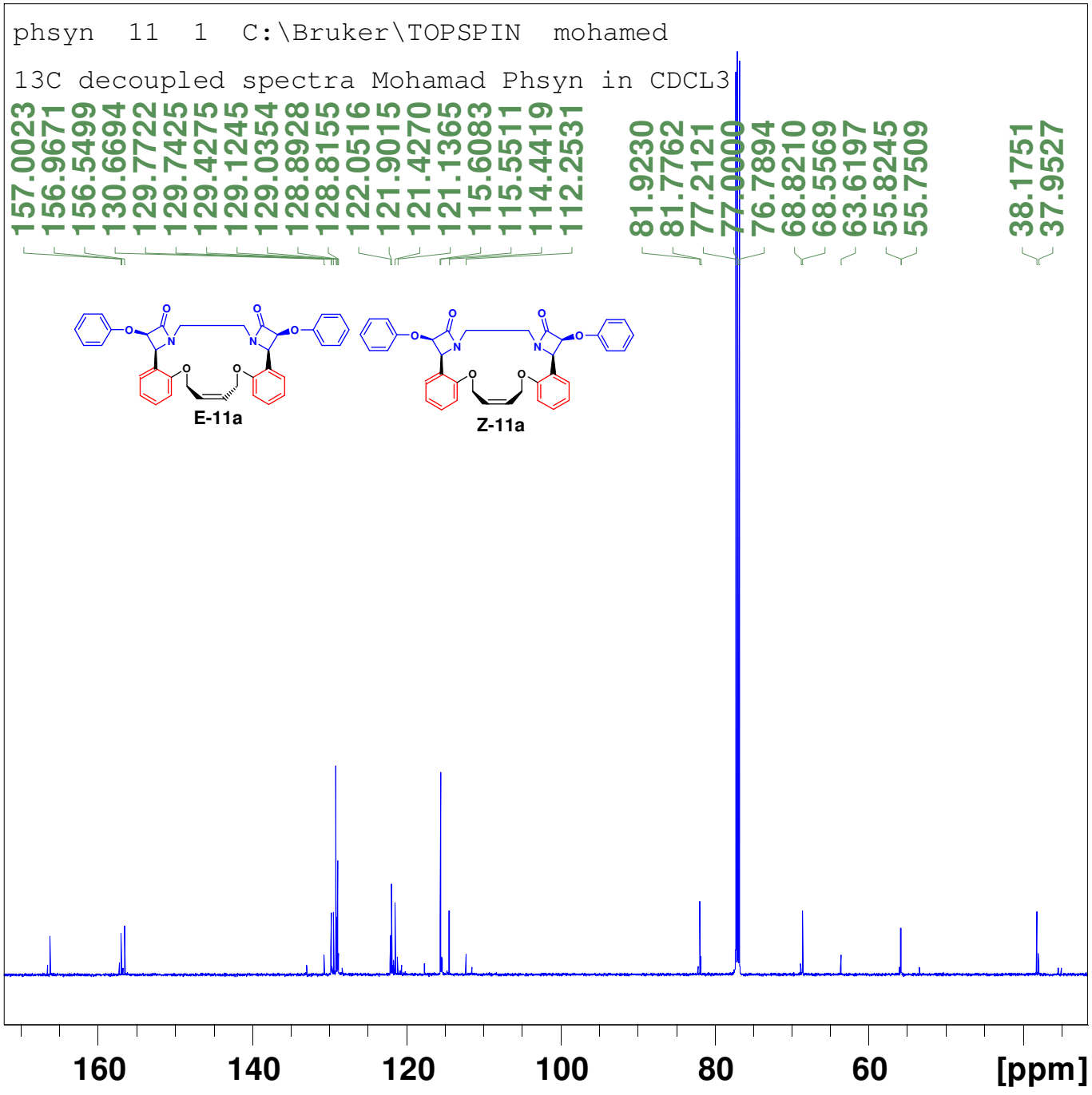

Rsi-1H 1 1 C:\Bruker\TOPSPIN Administrator
4b
7b
[rel]
0
5
10
15
20
7
6
5
4
[ppm]

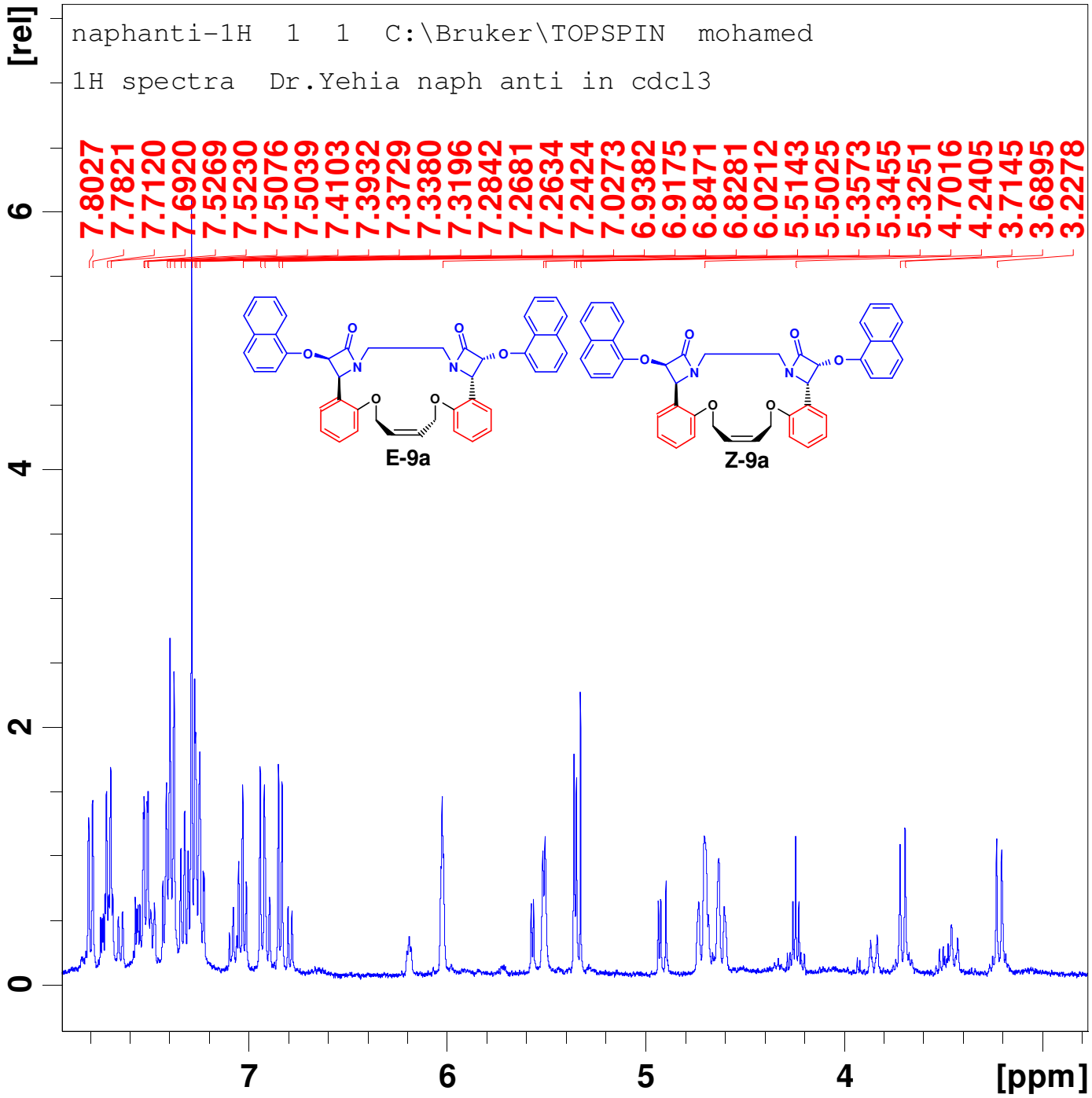

RSI-13C 2 1 C:\Bruker\TOPSPIN Administrator
166.3043
156.4873
154.7162
133.8954
133.2143
129.3381
129.2983
129.0558
128.2466
127.5658
126.7932
126.1621
123.9244
121.3762
120.3286
118.5810
117.3170
111.1399
111.1209
108.5016
108.4822
82.0235
82.0055
77.4174
77.0998
76.7815
70.1421
70.1177
68.4793
68.1111
68.0824
56.7465
40.2522
4b
7b
[rel]
0
5
10
15
20
25
160
140
120
100
80
60
[ppm]

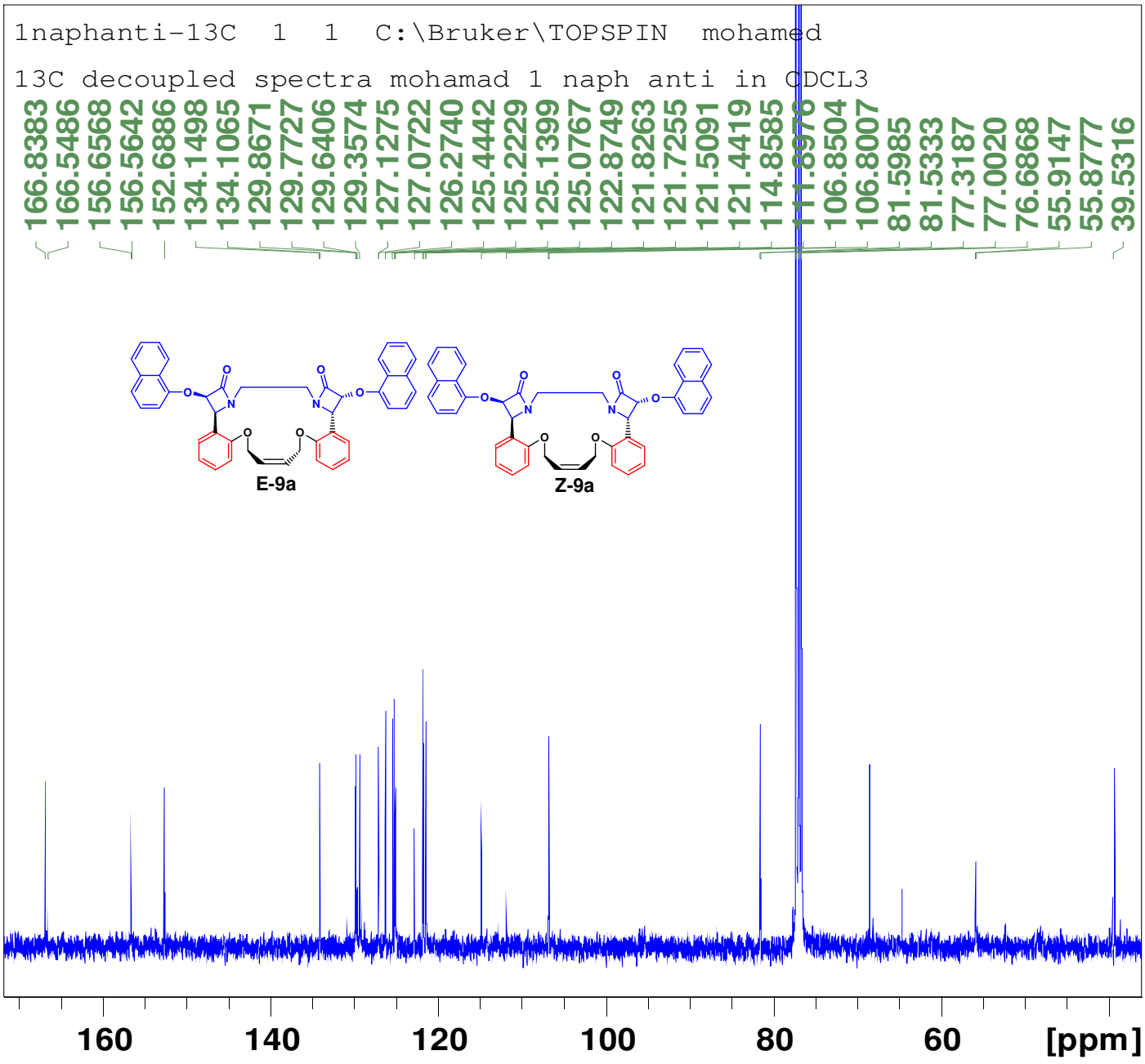

RCMISO1G1 20 1 C:\Bruker\TOPSPIN mohamed
KU_PROTON_8 CDCl3 {C:\Bruker\TOPSPIN} badria 30
7.0167
7.0158
7.0034
6.9917
6.9908
6.8924
6.8916
6.8788
6.8667
6.8653
6.8561
6.8545
6.7244
6.7227
6.7096
6.7084
6.6468
6.6337
5.9754
5.9708
5.9663
5.3736
5.3660
5.1844
5.1767
4.6909
4.6870
4.6159
4.5974
3.6031
3.5871
3.0872
3.0712
E-8a
Z-8a
[rel]
0
5
10
15
20
7
6
5
4
[ppm]

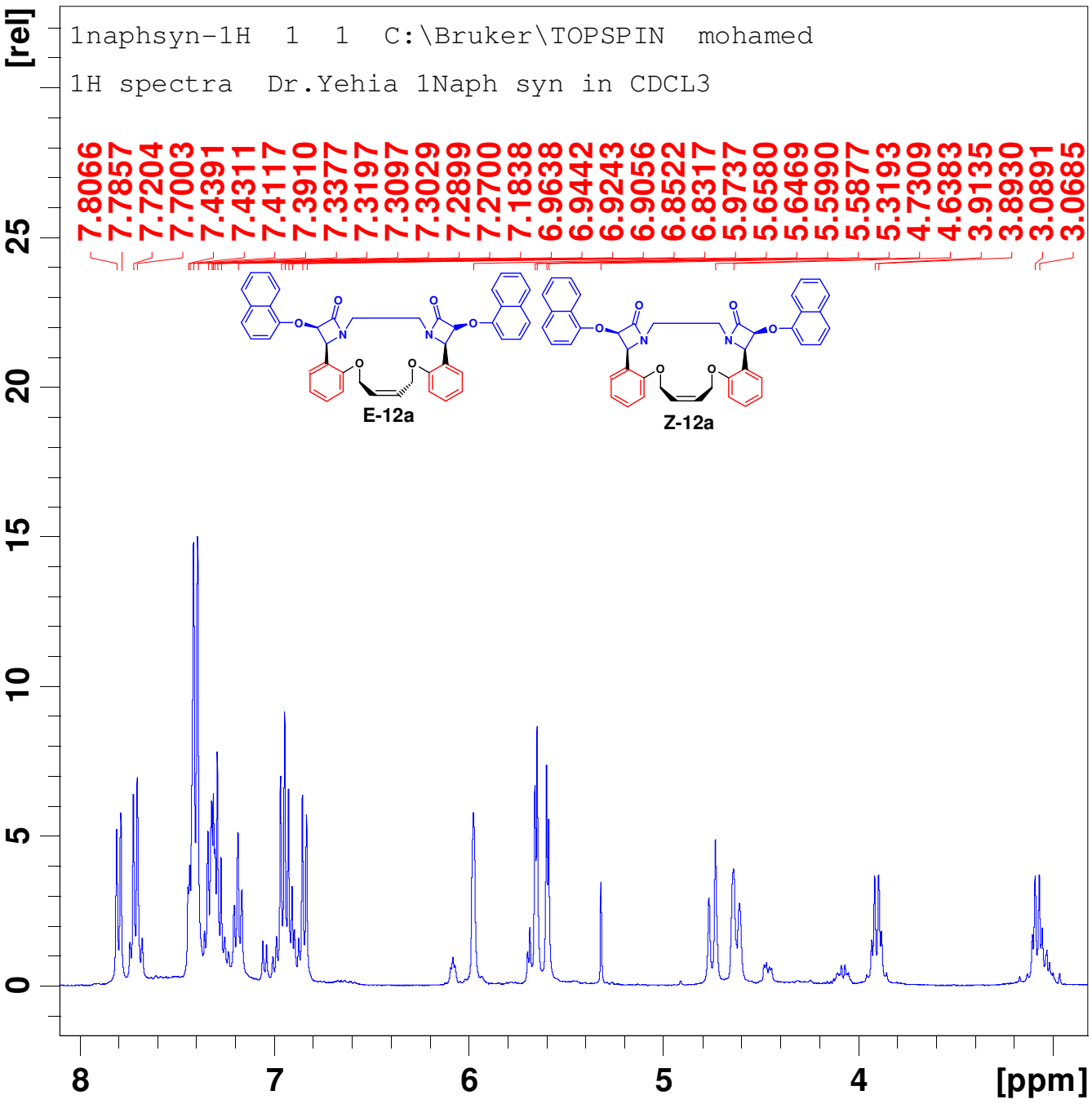

RCMISO1G1 31 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.YEHIA RCMISI1G1 in CDCL3
156.5891
156.5414
129.9250
129.8019
129.6824
129.6226
129.2851
129.1726
129.0882
128.8843
122.6102
121.7808
121.7427
121.6791
121.5468
121.3560
115.4720
115.3327
114.6625
111.8720
81.5264
81.4364
77.2124
77.0002
76.7880
68.3949
64.5733
55.9566
55.6487
39.3738
39.0680
E-8a
Z-8a
160
140
120
100
80
60
[ppm]

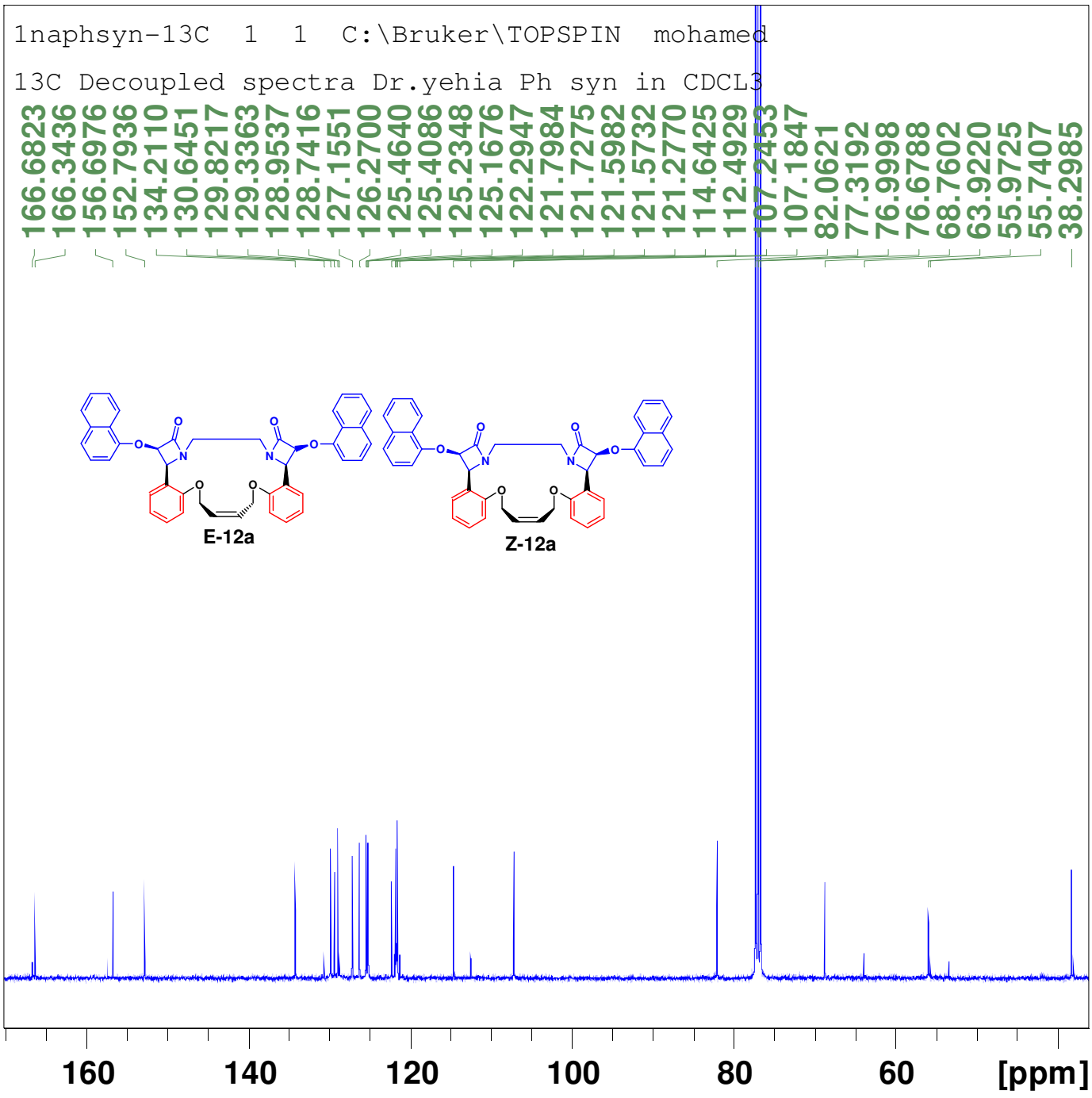

phsyn-1H 1 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia ph syn in CDCL3
7.2984
7.2949
7.2794
7.2760
7.2242
7.1818
7.1778
7.1644
7.1437
7.1252
6.9374
6.9236
6.9064
6.8899
6.8725
6.8153
6.7982
6.7789
5.9537
5.4952
5.4841
5.4478
5.4368
5.3166
4.7723
4.7391
4.6375
4.6083
4.6015
3.7950
3.7746
2.9640
2.9437
E-11a
Z-11a
[rel]
30
20
10
0
7
6
5
4
[ppm]

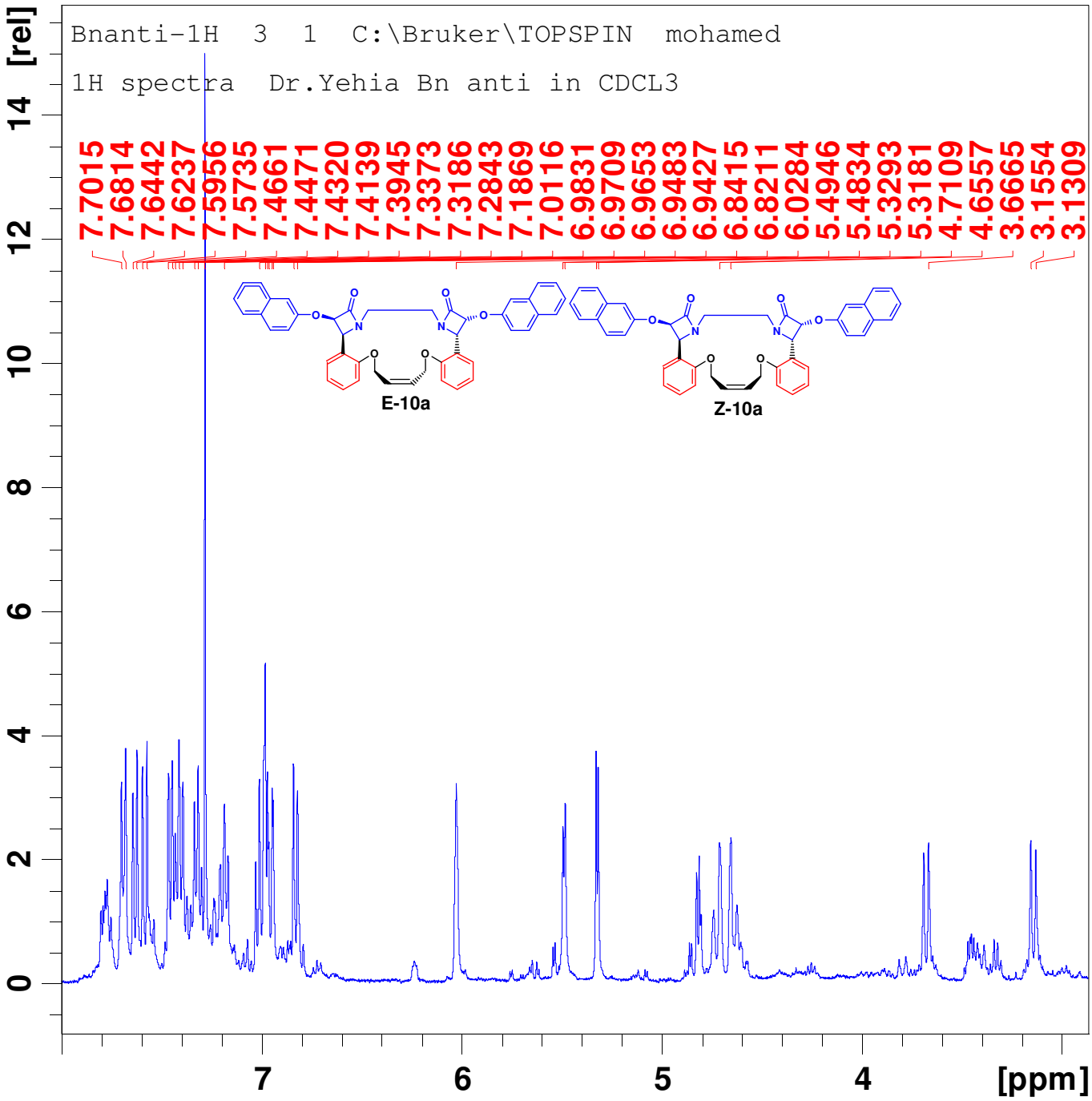

phsyn 11 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra Mohamad Phsyn in CDCL3
157.0023
156.9671
156.5499
130.6694
129.7722
129.7425
129.4275
129.1245
129.0354
128.8928
128.8155
122.0516
121.9015
121.4270
121.1365
115.6083
115.5511
114.4419
112.2531
81.9230
81.7762
77.2121
77.0000
76.7894
68.8210
68.5569
63.6197
55.8245
55.7509
38.1751
37.9527
E-11a
Z-11a
160
140
120
100
80
60
[ppm]

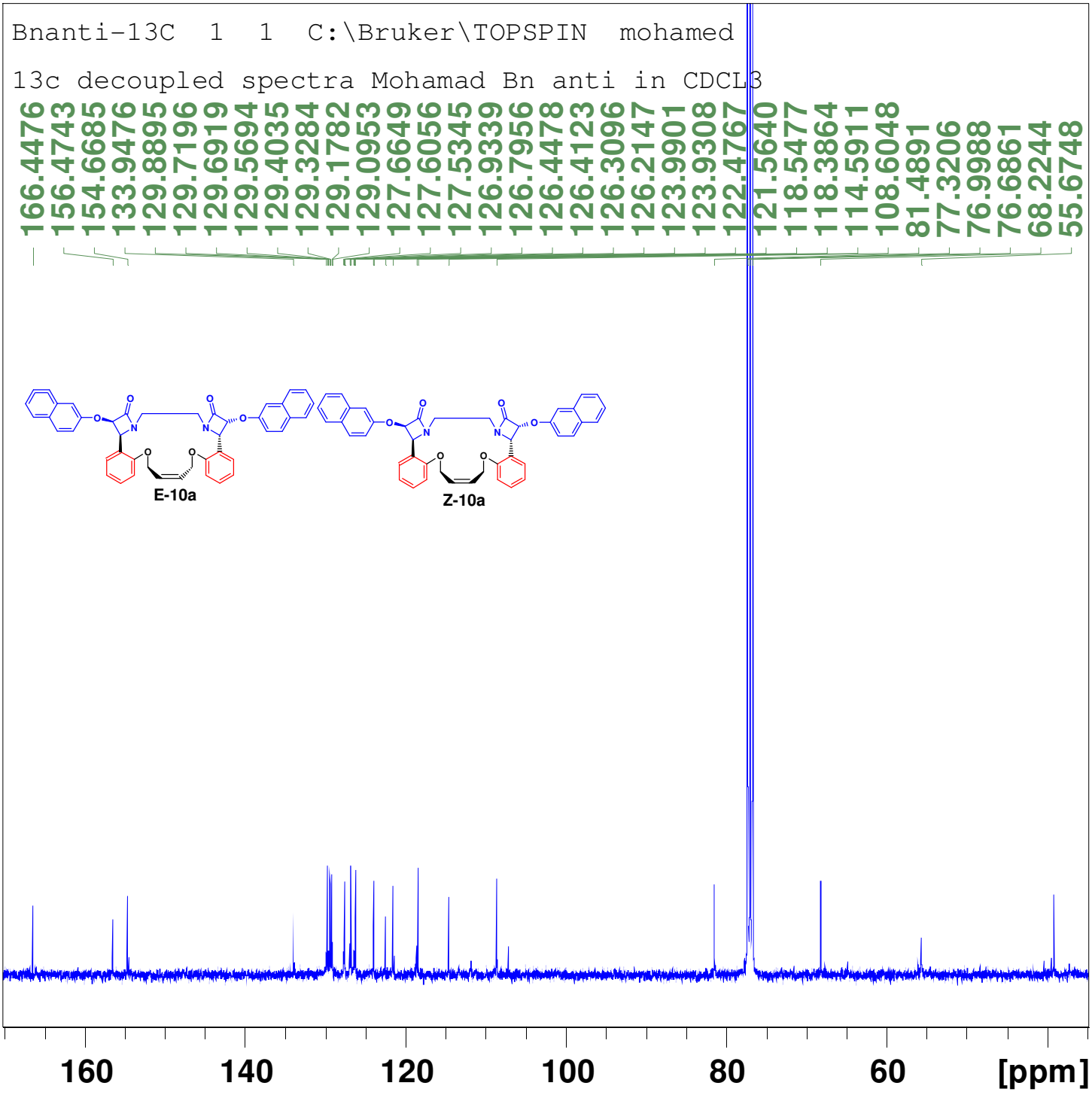

naphanti-1H 1 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia naph anti in cdcl3
7.8027
7.7821
7.7120
7.6920
7.5269
7.5230
7.5076
7.5039
7.4103
7.3932
7.3729
7.3380
7.3196
7.2842
7.2681
7.2634
7.2424
7.0273
6.9382
6.9175
6.8471
6.8281
6.0212
5.5143
5.5025
5.3573
5.3455
5.3251
4.7016
4.2405
3.7145
3.6895
3.2278
E-9a
Z-9a
[rel]
0
2
4
6
7
6
5
4
[ppm]

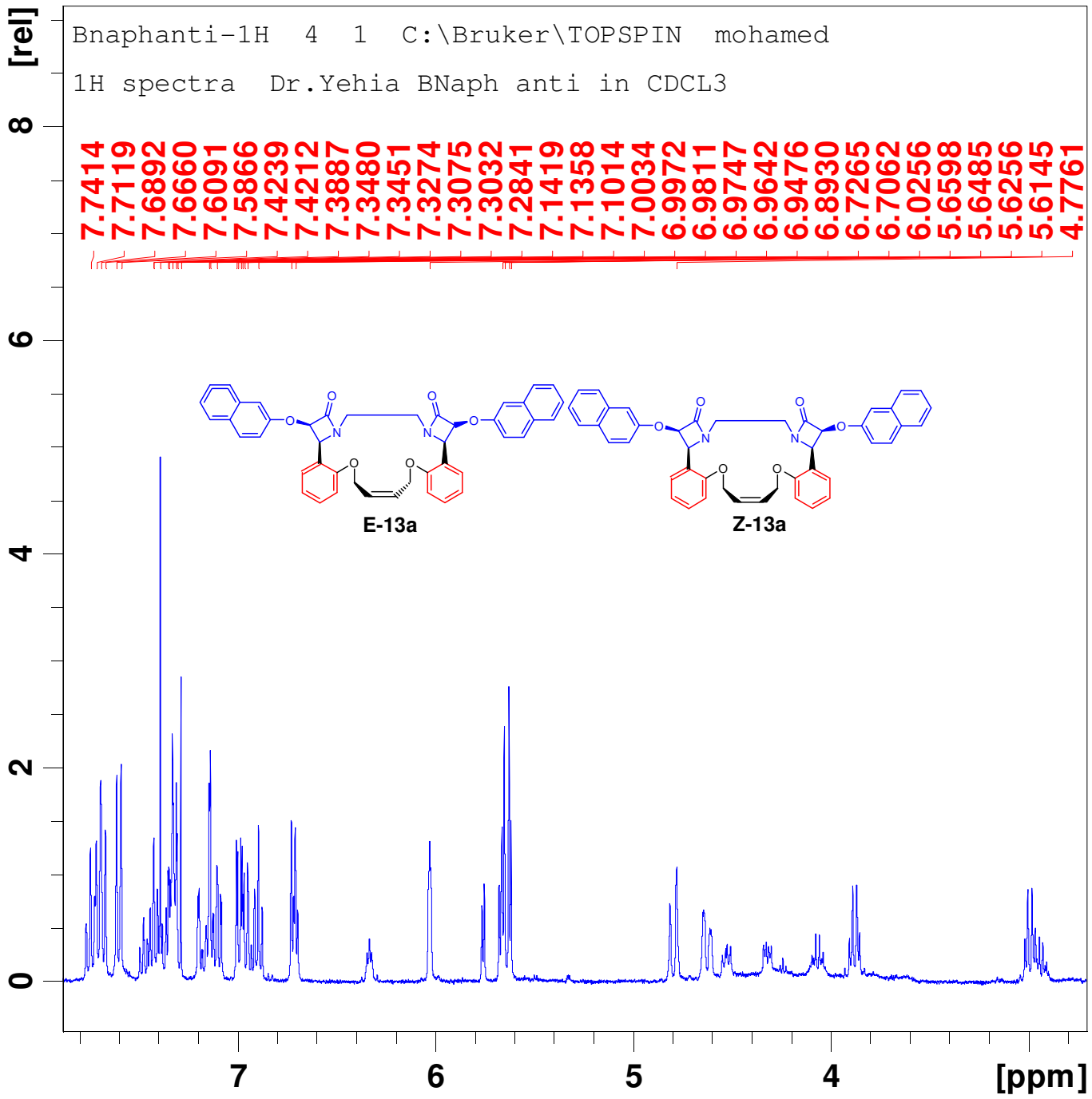

1naphanti-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra mohamad 1 naph anti in CDCL3
166.8383
166.5486
156.6568
156.5642
152.6886
134.1498
134.1065
129.8671
129.7727
129.6406
129.3574
127.1275
127.0722
126.2740
125.4442
125.2229
125.1399
125.0767
122.8749
121.8263
121.7255
121.5091
121.4419
114.8585
111.8976
106.8504
106.8007
81.5985
81.5333
77.3187
77.0020
76.6868
55.9147
55.8777
39.5316
E-9a
Z-9a
160
140
120
100
80
60
[ppm]

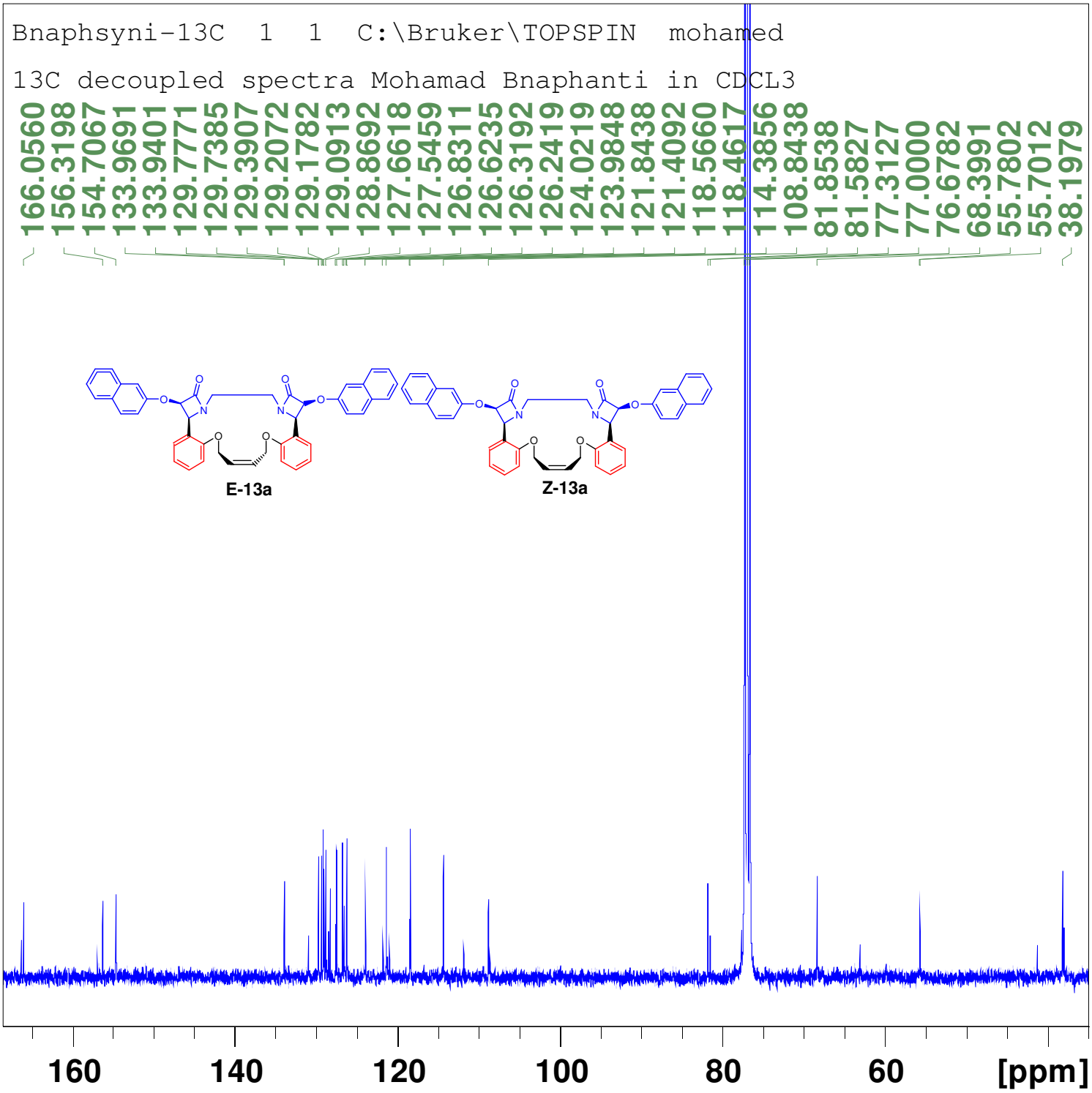

1naphsyn-1H 1 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia 1Naph syn in CDCL3
7.8066
7.7857
7.7204
7.7003
7.4391
7.4311
7.4117
7.3910
7.3377
7.3197
7.3097
7.3029
7.2899
7.2700
7.1838
6.9638
6.9442
6.9243
6.9056
6.8522
6.8317
5.9737
5.6580
5.6469
5.5990
5.5877
5.3193
4.7309
4.6383
3.9135
3.8930
3.0891
3.0685
E-12a
Z-12a
[rel]
25
20
15
10
5
0
8
7
6
5
4
[ppm]

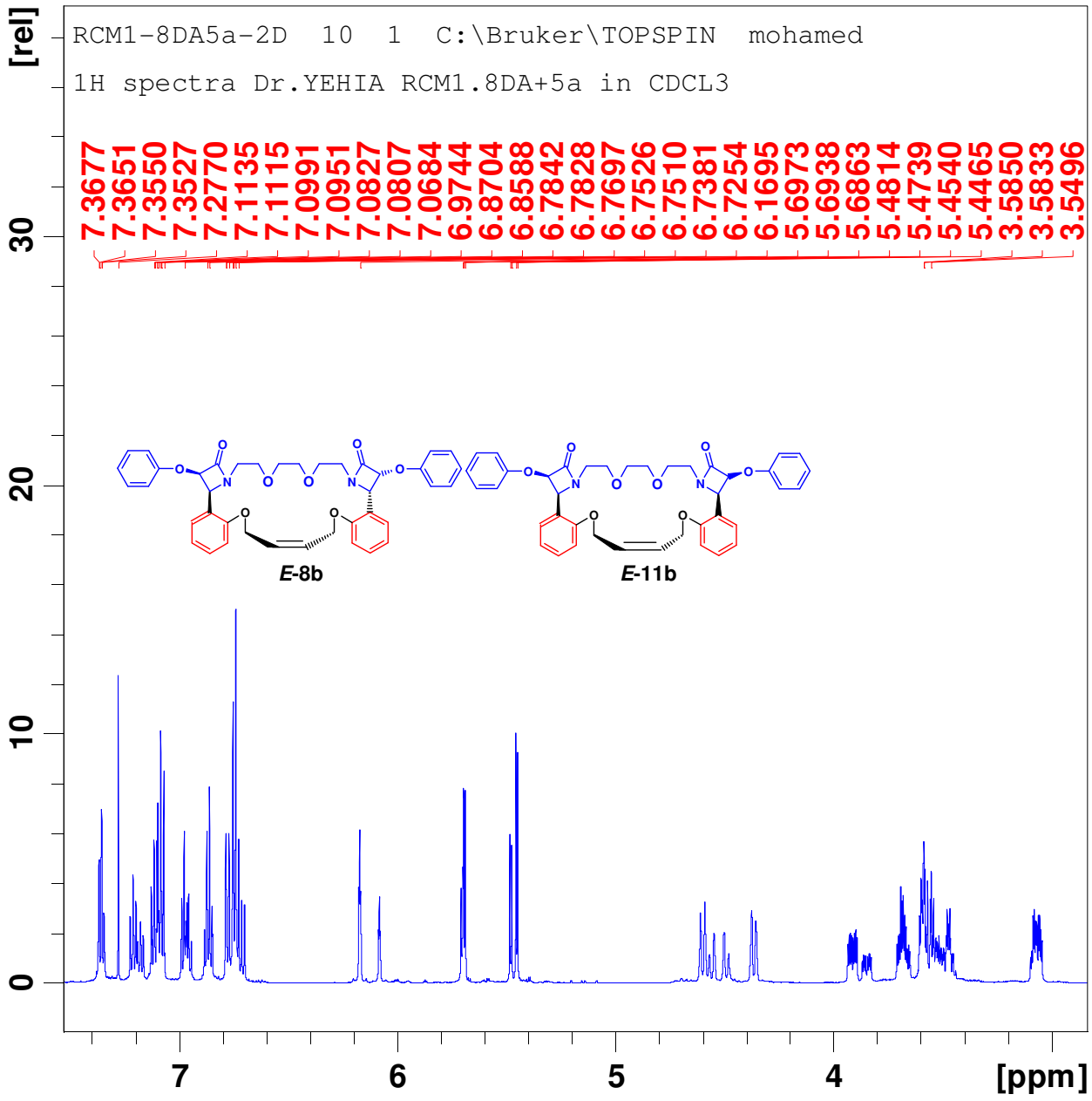

1naphsyn-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C Decoupled spectra Dr.yehia Ph syn in CDCL3
166.6823
166.3436
156.6976
152.7936
134.2110
130.6451
129.8217
129.3363
128.9537
128.7416
127.1551
126.2700
125.4640
125.4086
125.2348
125.1676
122.2947
121.7984
121.7275
121.5982
121.5732
121.2770
114.6425
112.4929
107.2453
107.1847
82.0621
77.3192
76.9998
76.6788
68.7602
63.9220
55.9725
55.7407
38.2985
E-12a
Z-12a
160
140
120
100
80
60
[ppm]

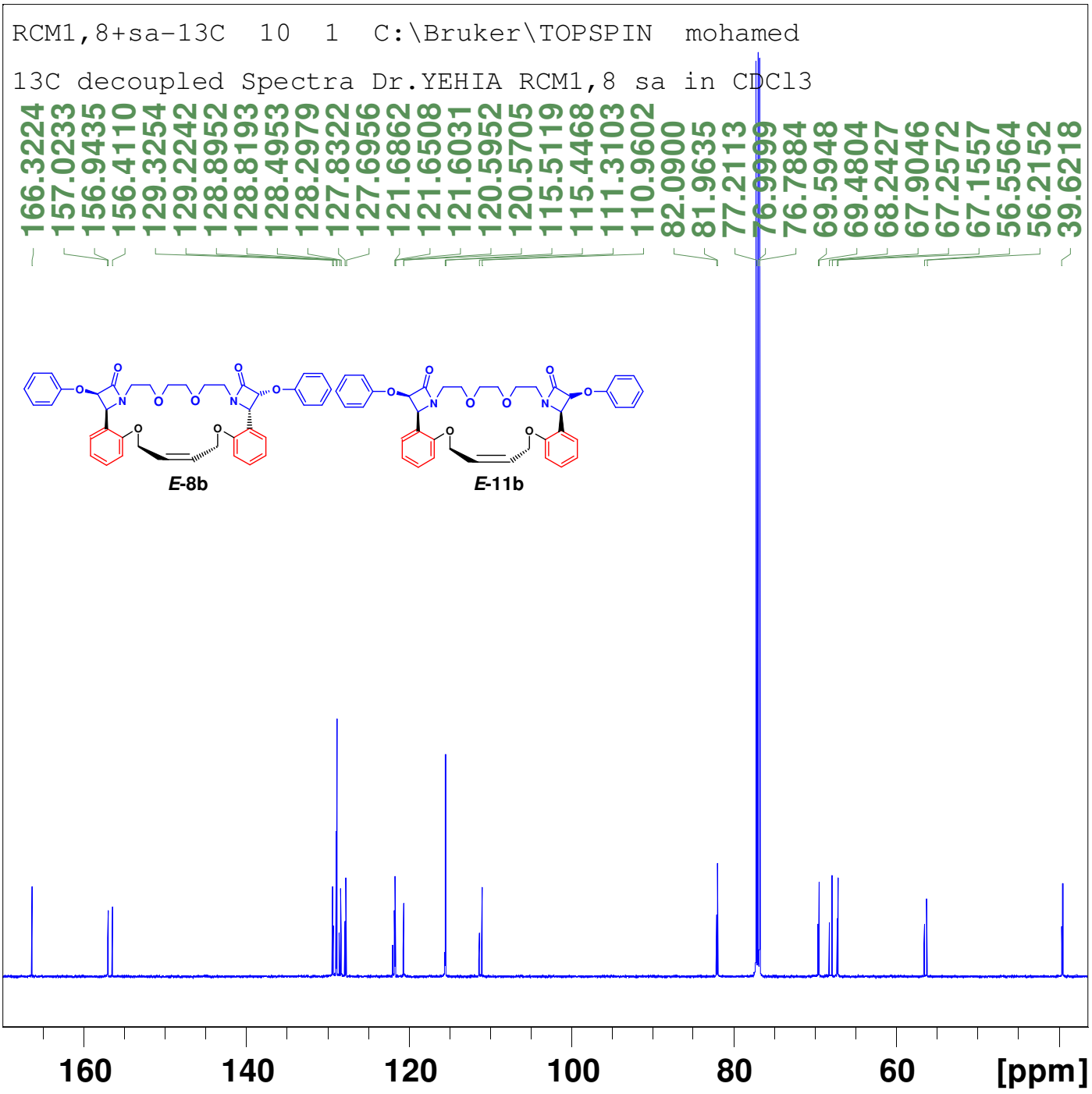

Bnanti-1H 3 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia Bn anti in CDCL3
7.7015
7.6814
7.6442
7.6237
7.5956
7.5735
7.4661
7.4471
7.4320
7.4139
7.3945
7.3373
7.3186
7.2843
7.1869
7.0116
6.9831
6.9709
6.9653
6.9483
6.9427
6.8415
6.8211
6.0284
5.4946
5.4834
5.3293
5.3181
4.7109
4.6557
3.6665
3.1554
3.1309
E-10a
Z-10a
[rel]
[ppm]

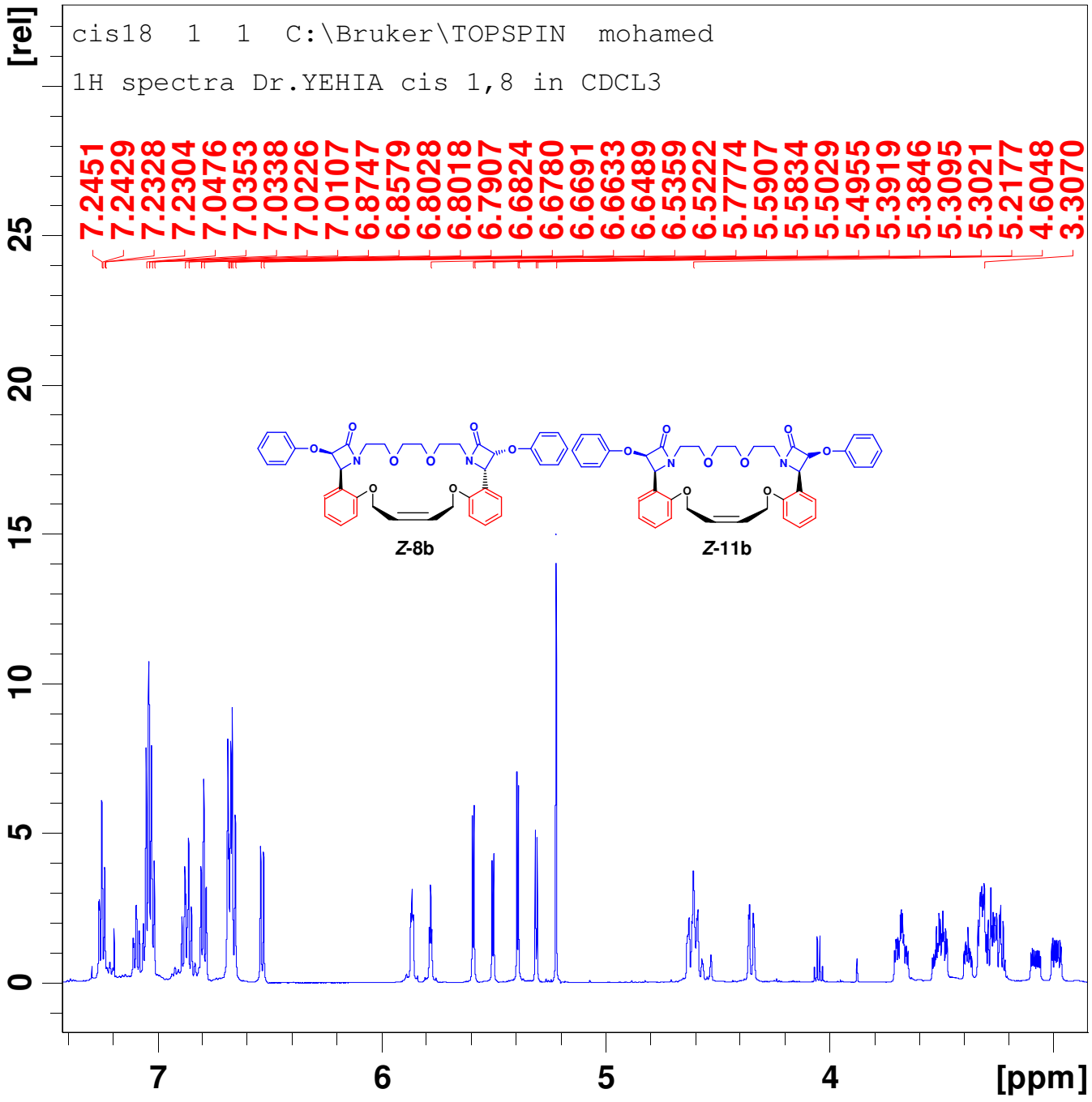

Bnanti-13C 1 1 C:\Bruker\TOPSPIN mohamed
13c decoupled spectra Mohamad Bn anti in CDCL3
166.4476
156.4743
154.6685
133.9476
129.8895
129.7196
129.6919
129.5694
129.4035
129.3284
129.1782
129.0953
127.6649
127.6056
127.5345
126.9339
126.7956
126.4478
126.4123
126.3096
126.2147
123.9901
123.9308
122.4767
121.5640
118.5477
118.3864
114.5911
108.6048
81.4891
77.3206
76.9988
76.6861
68.2244
55.6748
E-10a
Z-10a
160
140
120
100
80
60
[ppm]

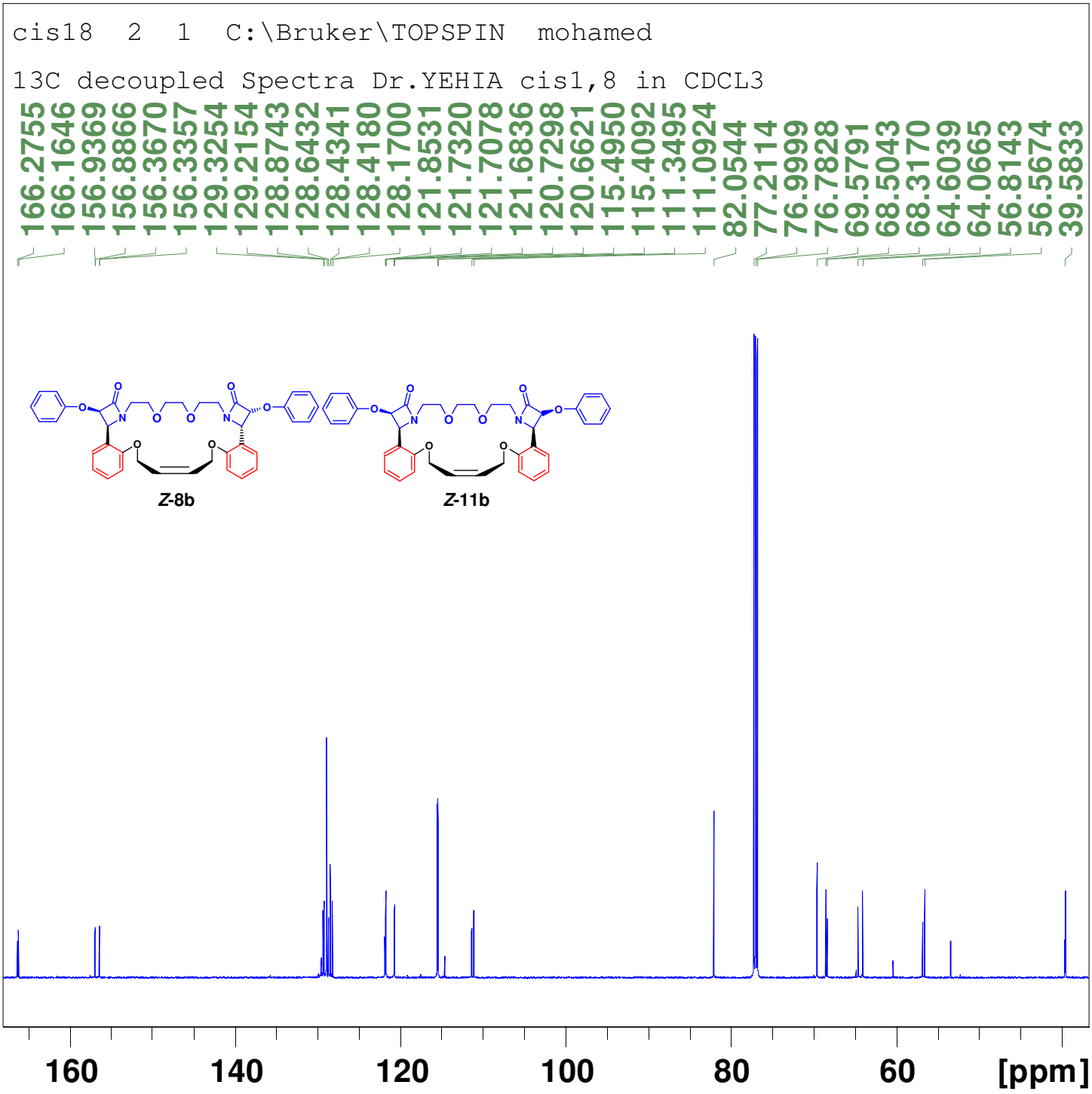

Bnaphanti-1H 4 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.Yehia BNaph anti in CDCL3
7.7414
7.7119
7.6892
7.6660
7.6091
7.5866
7.4239
7.4212
7.3887
7.3480
7.3451
7.3274
7.3075
7.3032
7.2841
7.1419
7.1358
7.1014
7.0034
6.9972
6.9811
6.9747
6.9642
6.9476
6.8930
6.7265
6.7062
6.0256
5.6598
5.6485
5.6256
5.6145
4.7761
E-13a
Z-13a
[rel]
[ppm]

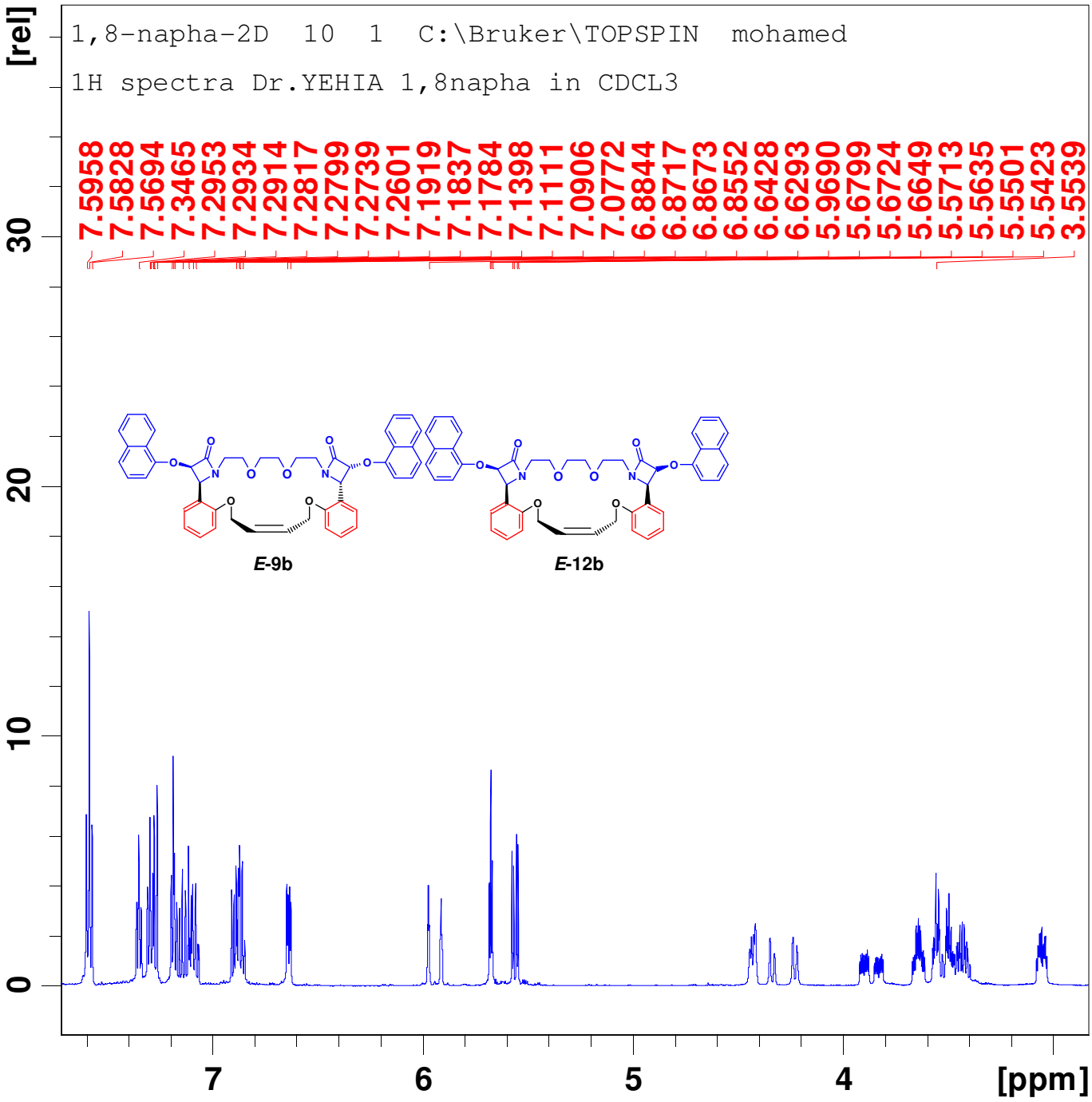

Bnaphsyni-13C 1 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra Mohamad Bnaphanti in CDCL3
166.0560
156.3198
154.7067
133.9691
133.9401
129.7771
129.7385
129.3907
129.2072
129.1782
129.0913
128.8692
127.6618
127.5459
126.8311
126.6235
126.3192
126.2419
124.0219
123.9848
121.8438
121.4092
118.5660
118.4617
114.3856
108.8438
81.8538
81.5827
77.3127
77.0000
76.6782
68.3991
55.7802
55.7012
38.1979
E-13a
Z-13a
160
140
120
100
80
60
[ppm]

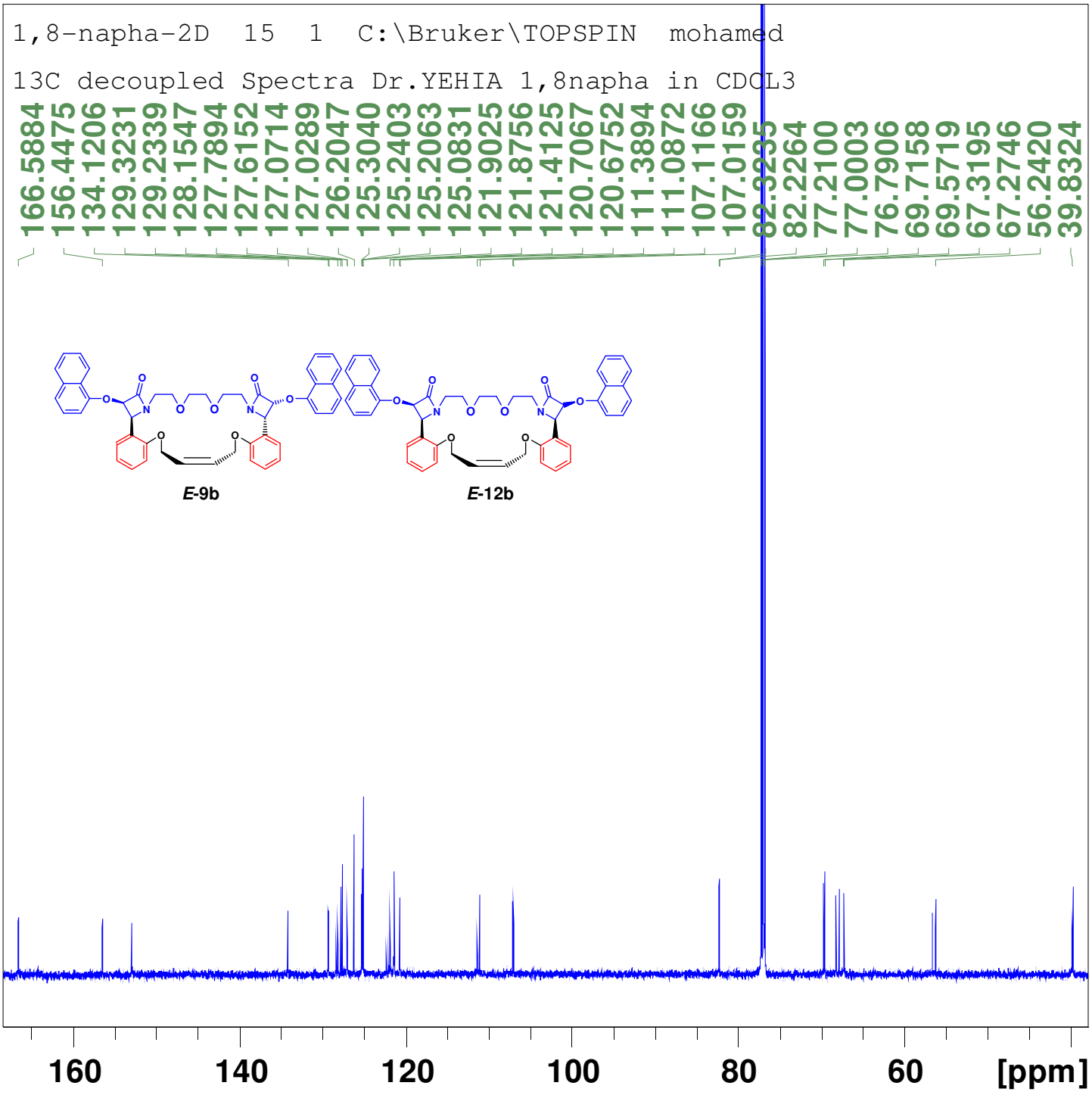

RCM1-8DA5a-2D 10 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.YEHIA RCM1.8DA+5a in CDCL3
[rel]
30
20
10
0
7.3677
7.3651
7.3550
7.3527
7.2770
7.1135
7.1115
7.0991
7.0951
7.0827
7.0807
7.0684
6.9744
6.8704
6.8588
6.7842
6.7828
6.7697
6.7526
6.7510
6.7381
6.7254
6.1695
5.6973
5.6938
5.6863
5.4814
5.4739
5.4540
5.4465
3.5850
3.5833
3.5496
E-8b
E-11b
7
6
5
4
[ppm]

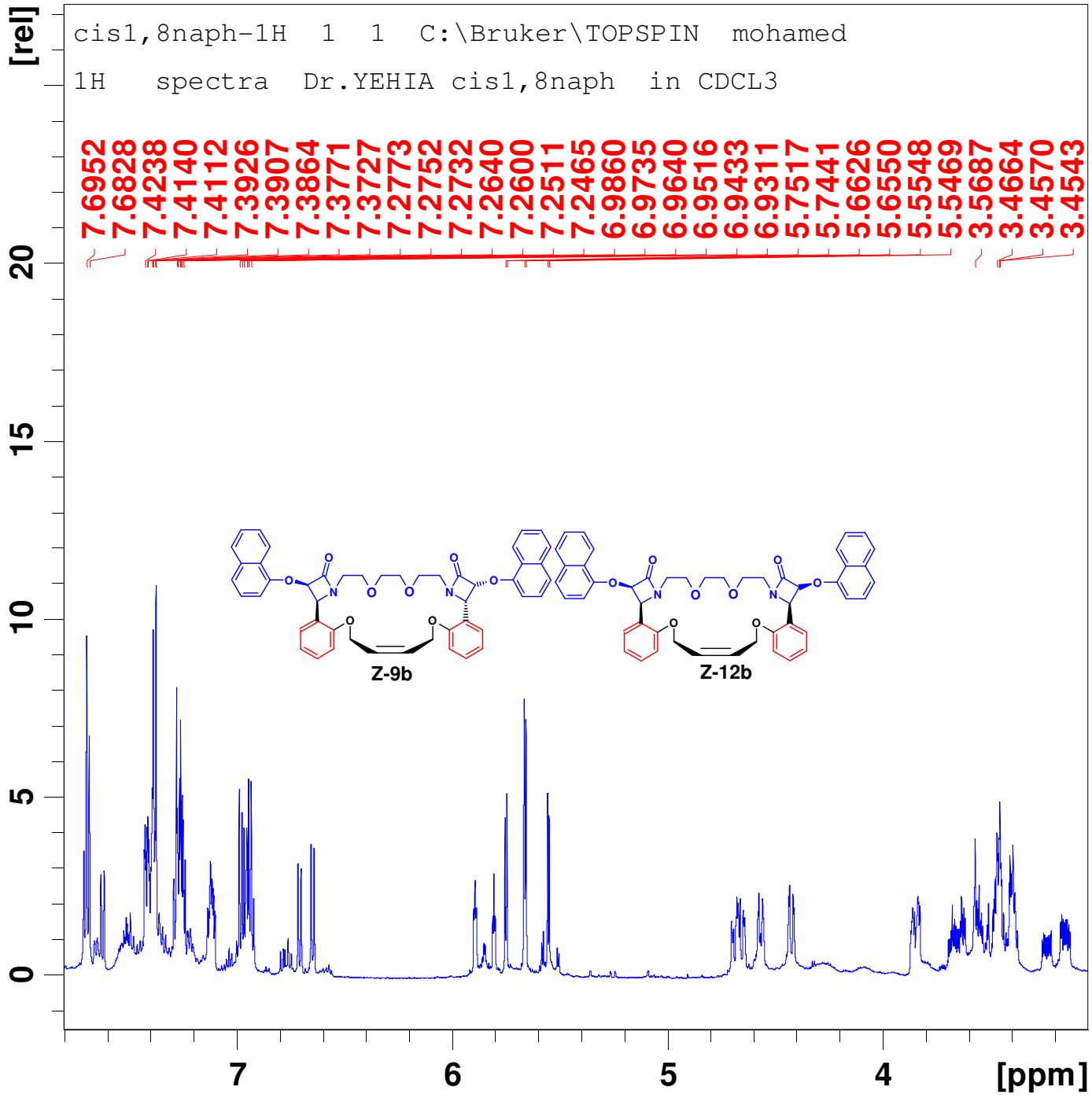

RCM1,8+sa-13C 10 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.YEHIA RCM1,8 sa in CDCl3
166.3224
157.0233
156.9435
156.4110
129.3254
129.2242
128.8952
128.8193
128.4953
128.2979
127.8322
127.6956
121.6862
121.6508
121.6031
120.5952
120.5705
115.5119
115.4468
111.3103
110.9602
82.0900
81.9635
77.2113
76.9999
76.7884
69.5948
69.4804
68.2427
67.9046
67.2572
67.1557
56.5564
56.2152
39.6218
E-8b
E-11b
160
140
120
100
80
60
[ppm]

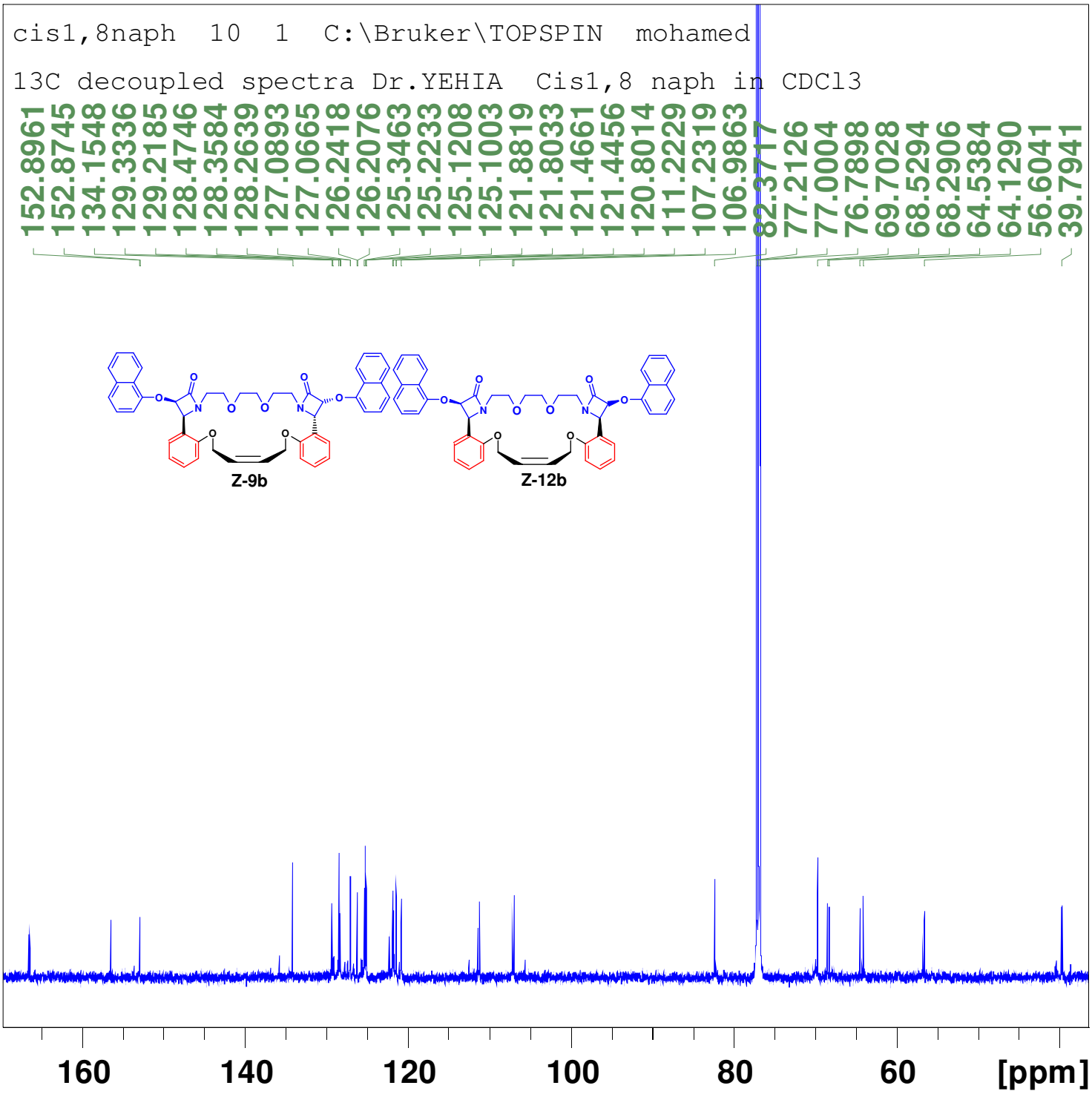

cis18 1 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.YEHIA cis 1,8 in CDCL3
7.2451
7.2429
7.2328
7.2304
7.0476
7.0353
7.0338
7.0226
7.0107
6.8747
6.8579
6.8028
6.8018
6.7907
6.6824
6.6780
6.6691
6.6633
6.6489
6.5359
6.5222
5.7774
5.5907
5.5834
5.5029
5.4955
5.3919
5.3846
5.3095
5.3021
5.2177
4.6048
3.3070
Z-8b
Z-11b
[rel]
0
5
10
15
20
25
7
6
5
4
[ppm]

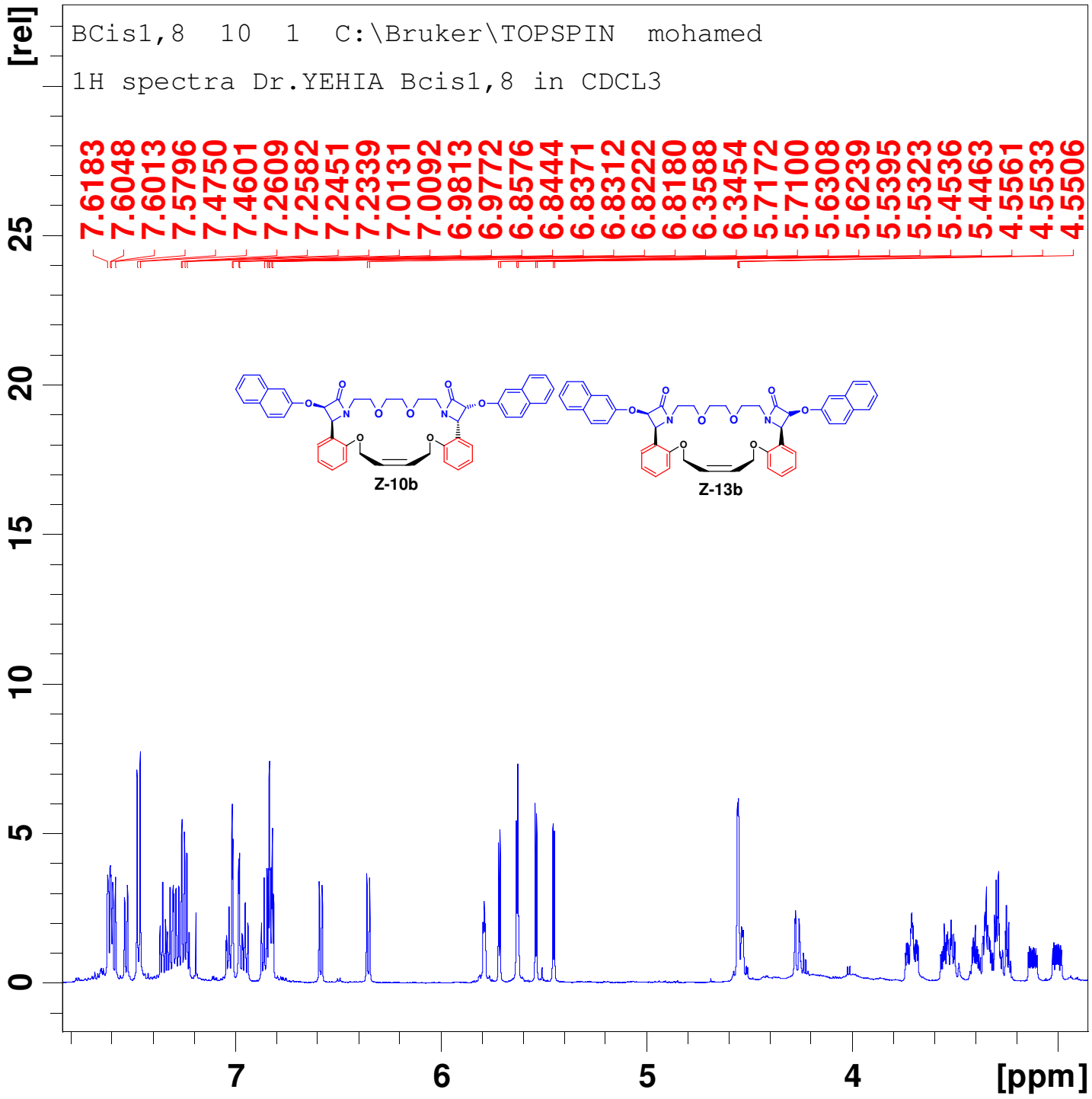

cis18 2 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.YEHIA cis1,8 in CDCL3
166.2755
166.1646
156.9369
156.8866
156.3670
156.3357
129.3254
129.2154
128.8743
128.6432
128.4341
128.4180
128.1700
121.8531
121.7320
121.7078
121.6836
120.7298
120.6621
115.4950
115.4092
111.3495
111.0924
82.0544
77.2114
76.9999
76.7828
69.5791
68.5043
68.3170
64.6039
64.0665
56.8143
56.5674
39.5833
Z-8b
Z-11b
160
140
120
100
80
60
[ppm]

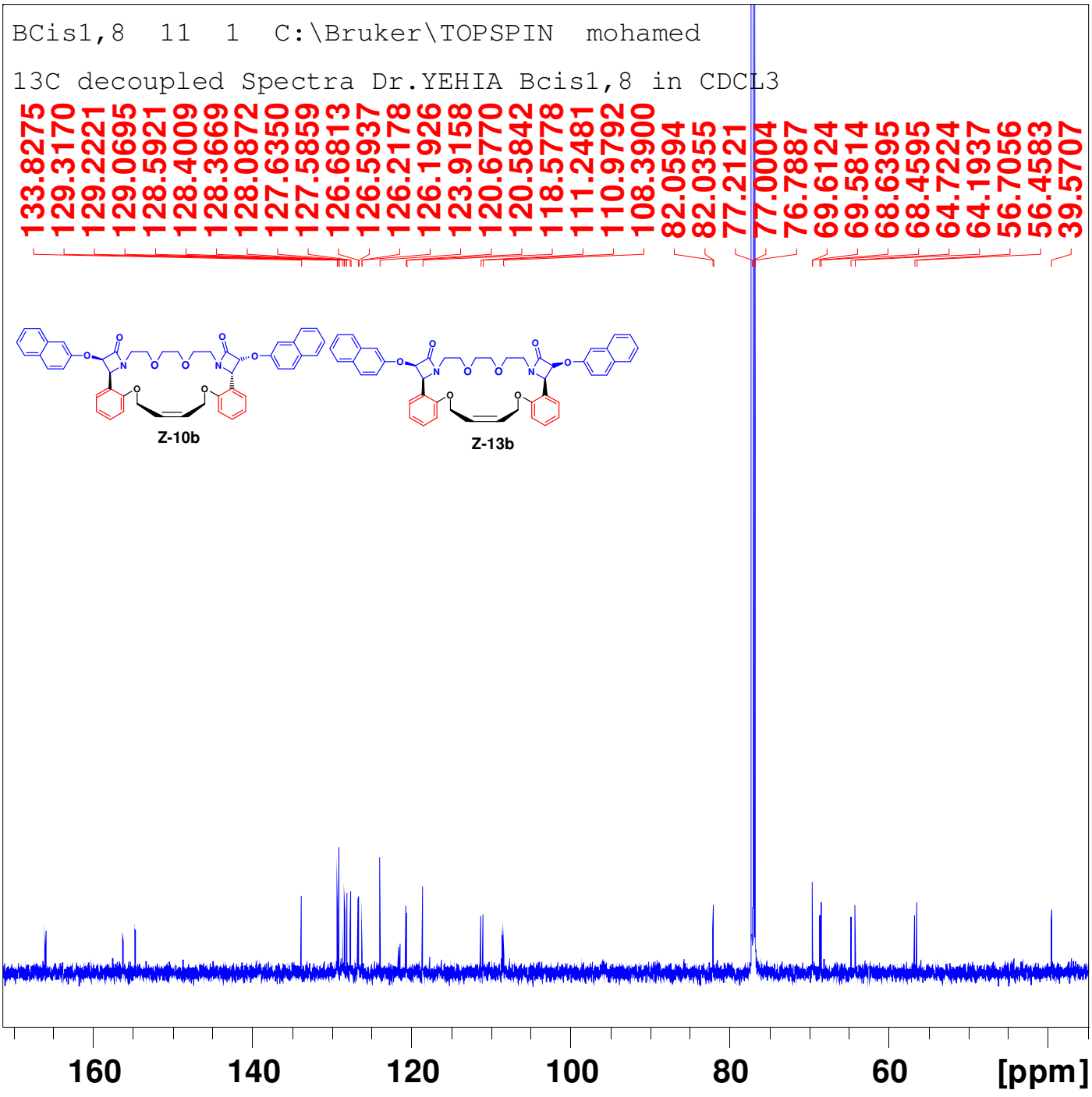

1,8-napha-2D 10 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.YEHIA 1,8napha in CDCL3
7.5958
7.5828
7.5694
7.3465
7.2953
7.2934
7.2914
7.2817
7.2799
7.2739
7.2601
7.1919
7.1837
7.1784
7.1398
7.1111
7.0906
7.0772
6.8844
6.8717
6.8673
6.8552
6.6428
6.6293
5.9690
5.6799
5.6724
5.6649
5.5713
5.5635
5.5501
5.5423
3.5539
E-9b
E-12b
[rel]
0
10
20
30
7
6
5
4
[ppm]


1,8-napha-2D 15 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.YEHIA 1,8napha in CDCL3
166.5884
156.4475
134.1206
129.3231
129.2339
128.1547
127.7894
127.6152
127.0714
127.0289
126.2047
125.3040
125.2403
125.2063
125.0831
121.9025
121.8756
121.4125
120.7067
120.6752
111.3894
111.0872
107.1166
107.0159
82.3235
82.2264
77.2100
77.0003
76.7906
69.7158
69.5719
67.3195
67.2746
56.2420
39.8324
E-9b
E-12b
160
140
120
100
80
60
[ppm]


cis1,8naph-1H 1 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.YEHIA cis1,8naph in CDCL3
7.6952
7.6828
7.4238
7.4140
7.4112
7.3926
7.3907
7.3864
7.3771
7.3727
7.2773
7.2752
7.2732
7.2640
7.2600
7.2511
7.2465
6.9860
6.9735
6.9640
6.9516
6.9433
6.9311
5.7517
5.7441
5.6626
5.6550
5.5548
5.5469
3.5687
3.4664
3.4570
3.4543
[rel]
20
15
10
5
0
7
6
5
4
[ppm]
Z-9b
Z-12b


cis1,8naph 10 1 C:\Bruker\TOPSPIN mohamed
13C decoupled spectra Dr.YEHIA Cis1,8 naph in CDCl3
152.8961
152.8745
134.1548
129.3336
129.2185
128.4746
128.3584
128.2639
127.0893
127.0665
126.2418
126.2076
125.3463
125.2233
125.1208
125.1003
121.8819
121.8033
121.4661
121.4456
120.8014
111.2229
107.2319
106.9863
82.3717
77.2126
77.0004
76.7898
69.7028
68.5294
68.2906
64.5384
64.1290
56.6041
39.7941
Z-9b
Z-12b
160
140
120
100
80
60
[ppm]


BCis1,8 10 1 C:\Bruker\TOPSPIN mohamed
1H spectra Dr.YEHIA Bcis1,8 in CDCL3
7.6183
7.6048
7.6013
7.5796
7.4750
7.4601
7.2609
7.2582
7.2451
7.2339
7.0131
7.0092
6.9813
6.9772
6.8576
6.8444
6.8371
6.8312
6.8222
6.8180
6.3588
6.3454
5.7172
5.7100
5.6308
5.6239
5.5395
5.5323
5.4536
5.4463
4.5561
4.5533
4.5506
Z-10b
Z-13b
[rel]
0
5
10
15
20
25
7
6
5
4
[ppm]


BCis1,8 11 1 C:\Bruker\TOPSPIN mohamed
13C decoupled Spectra Dr.YEHIA Bcis1,8 in CDCL3
133.8275
129.3170
129.2221
129.0695
128.5921
128.4009
128.3669
128.0872
127.6350
127.5859
126.6813
126.5937
126.2178
126.1926
123.9158
120.6770
120.5842
118.5778
111.2481
110.9792
108.3900
82.0594
82.0355
77.2121
77.0004
76.7887
69.6124
69.5814
68.6395
68.4595
64.7224
64.1937
56.7056
56.4583
39.5707
Z-10b
Z-13b
160
140
120
100
80
60
[ppm]