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Stereoselctive Formation of β-Lactams with Acyl Ketenes Generated from 5-Acyl-Meldrum's Acids.

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Supporting Information

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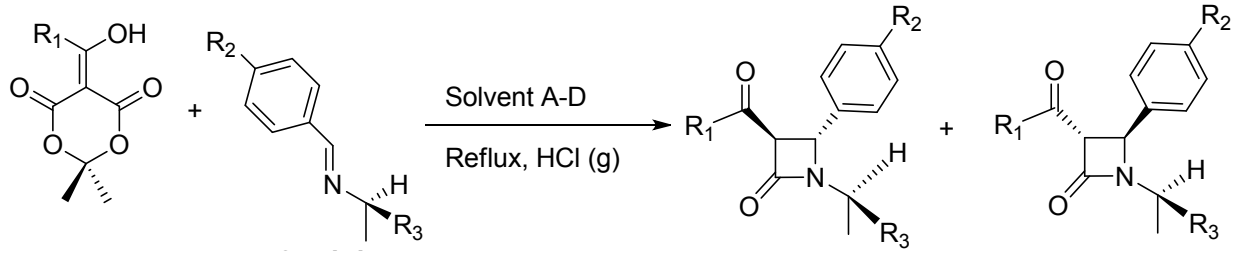
General Information

Reagents were purchased from Sigma-Aldrich or Acros. Toluene was distilled from potassium under argon and stored over molecular sieves. Chlorobenzene, DCE and nitromethane were distilled over P_4O_{10} and stored over molecular sieves. Commercially unavailable reagents were prepared according to literature procedures: 5-[hydroxy(phenyl)methylene]-2,2-dimethyl-1,3-dioxa-4,6-dione **1a** [1], 5-[hydroxy(1-naphtylmethyl)methylene]-2,2-dimethyl-1,3-dioxa-4,6-dione **1b** [2], (*R*)-N-benzylidene-1-phenylethanamine **2aa**, racemic N-benzylidenebutan-2-amine **2ab**, (R)-N-(4-chlorobenzylidene)-1-phenylethanamine **2ba**, (*R*)-N-benzylidene-1-(naphthalen-1-yl)ethanamine **2ac**, (*R*)-N-benzylidene-1-(naphthalen-2-yl)ethanamine **2ad** [3]. Analytical TLC was performed on aluminum sheets of silica gel UV-254 Merck. Flash chromatography was performed using 40-63 microns of Zeochem silica gel. The ^1H, ^{13}C were recorded on Varian Gemini 200 and Varian Unity Plus 500, chemical shifts (δ) in ppm rel. to internal Me_4Si; coupling constants *J* in Hz. High-resolution (HRMS) was recorded on *MicroMas Quattro LCT* mass spectrometer. Melting points were determined with *Warsztat Elektromechaniczny W-wa* apparatus and are not corrected. Ratio of diasteroisomers was determined based on isolated yields as well as by integration of ^1H spectra.

Experimental Procedures and Characterization Data

Stereoselective preparation of azetidin-2-ones. General Procedure



To a solution of **1a-b** (1 mmol) in dry solvent (toluene (A), DCE (B), chlorobenzene (C) or nitromethane (D)) was added aldimine **2aa-bd** (1.5 mmol). The reaction mixture was cooled to 0°C and saturated with dry HCl over 20 min. The resulting mixture was stirred and heated to reflux for the time specified in Table 1 and 2. After completion of the reaction, the solvent was removed under vacuum, and the residue was purified as specified below.

3-Benzoyl-4-phenyl-1-(1-phenylethyl)azetidin-2-one (3aaa), (4aaa)

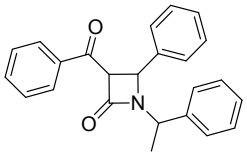


Table 2, Entry 1. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:8, SiO_2) gave (71 mg, 0.20 mmol, 20%) as three fractions respectively, I diasteroisomer (**3aaa**) (9 mg, 0.03 mmol, 3%, yellow oil), mixture of two diasteroisomers (51 mg, 0.14 mmol, 14%, ratio **3aaa** : **4aaa**, 13:87, yellow oil) and II diasteroisomer (**4aaa)** (11 mg, 0.03 mmol, 3%, yellow oil). Overall *de* = 54%. Table 2, Entry 2. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:8, SiO_2) gave one fraction, mixture of two diasteroisomers (89 mg, 0.25 mmol, 25%, ratio **3aaa** : **4aaa**, 65:35, yellow oil). Overall *de* = 30%. **I diasteroisomer (3aaa) ^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 8.08-8.07 (m, 2H, ArH), 7.60-7.57 (m, 1H, ArH), 7.50-7.47 (m, 2H, ArH), 7.35-7.31 (m, 3H, ArH), 7.30-7.23 (m, 5H, ArH), 7.23-7.18 (m, 2H, ArH), 5.09 (d, J = 2.4 Hz, 1H, H-4), 4.74 (d, J = 2.4 Hz, 1H, H-4), 4.41 (q, J = 7.4 Hz, 1H, CH_3*CH*), 1.81 (d, J = 7.4 Hz, 1H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 191.6, 163.3, 140.8, 137.3, 136.2, 134.0, 129.4, 129.2, 128.9, 127.9, 127.4, 127.3, 127.1, 127.0, 68.3, 55.5, 55.1, 20.1; **HRMS** (ESI+): m/z calcd for $C_{24}H_{21}NO_2Na$ $[M+Na]^+$ 378.1470, found. 378.1478. **II diasteroisomer (4aaa) ^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 8.06-8.04 (m, 2H, ArH), 7.58-7.55 (m, 1H, ArH), 7.48-7.45 (m, 2H, ArH), 7.36-7.30 (m, 7H, ArH), 7.29-7.24 (m, 3H, ArH), 5.10 (d, J = 2.4 Hz, 1H, H-4), 4.93 (q, J = 7.3 Hz, 1H, CH_3*CH*), 4.74 (d, J = 2.4 Hz, 1H, H-3), 1.41 (d, J = 7.4 Hz, 1H, CH_3);**^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 191.4, 163.3, 139.8, 138.4, 136.2, 134.0, 129.4, 129.2, 129.1, 129.0, 128.8, 128.1, 127.5, 127.2, 68.2, 55.9, 53.6, 19.5; **HRMS** (ESI+): m/z calcd for $C_{24}H_{21}NO_2Na$ $[M+Na]^+$ 378.1470, found. 378.1478

2,6-diphenyl-3-(1-phenylethyl)-2H-1,3-oxazin-4(3H)-one (5aaa), (6aaa)

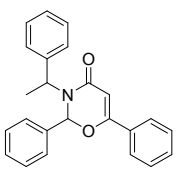


Table 1, Entry 5. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:4, SiO_2) gave (78 mg, 0.22 mmol, 22%) as two fractions respectively, I diastereoisomer (**5aaa)** (28 mg, 0.08 mmol, 22%, yellow oil), II diastereoisomer (**6aaa)** (50 mg, 0.014 mmol, 14%, white solid). **I diastereoisomer (5aaa) ^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 7.56-7.52 (m, 5H, ArH), 7.42-7.26 (m, 10H, ArH), 6.29 (s, 1H, NCHO), 6.14 (q, J = 7.3 Hz, 1H, CH_3*CH*), 5.97 (s, 1H, CH), 1.47 (d, J = 7.3 Hz, 3H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 163.6, 160.5, 141.0, 137.6, 132.1, 131.3, 129.5, 129.0, 128.7, 128.6, 127.9, 127.6, 127.3, 126.6, 98.7, 84.8, 50.5, 17.9; **HRMS** (ESI+): m/z calcd for

$C_{24}H_{21}NO_2Na$ $[M+Na]^+$ 378.1470, found. 378.1473. **II diastereoisomer (6aaa)** **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 7.82-7.75 (m, 1H, ArH), δ: 7.59 (d, J = 7.2 Hz, 2H, ArH), 7.50-7.33 (m, 6H, ArH), 7.19-7.09 (m, 6H), 6.50 (s, 1H, NCHO), 5.94 (q, J = 7.1 Hz, 1H, CH_3*CH*), 5.93 (s, 1H, CH), 1.76 (d, J = 7.1 Hz, 3H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 163.7, 160.7, 139.2, 136.6, 132.1, 131.3, 128.9, 128.8, 128.6, 128.4, 128.0, 127.9, 127.3, 126.6, 99.3, 85.1, 52.1, 18.0; **HRMS** (ESI+): m/z calcd for $C_{24}H_{21}NO_2Na$ $[M+Na]^+$ 378.1470, found. 378.1473.

3-Benzoyl-1-(sec-butyl)-4-phenylazetidin-2-one (3aab), (4aab)

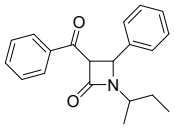


Table 1, Entry 7. Purification with flash chromatography R_f 0.31 (EtOAc:Hex 1:8, SiO_2) gave one fraction, mixture of two diasteroisomers (89 mg, 0.32 mmol, 32%, ratio 55:45, yellow oil). Overall *de* = 10 %. **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 8.08-8.06 (m, 2H, ArH), 7.58-7.55 (m, 1H, ArH), 7.49-7.45 (m, 4H, ArH), 7.42-7.33 (m, 3H, ArH), 5.28-5.27 (m, 1H, H-4), 4.74-7.73 (m, 1H, H-3), 3.66-3.60 (m, 0.55H, NCH), 3.46-3.42 (m, 0.45H, NCH), 1.82-1.73 (m, 0.55H, CH_2), 1.60-1.49 (m, 0.9H, CH_2), 1.39-1.32 (m, 1.9H, CH_2+CH_3), 0.99-0.95 (m, 3.3H, CH_3), 0.92-0.89 (m, 1.35H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 191.8, 163.4, 163.3, 138.7 (min), 138.4 (maj), 136.3, 134.0, 129.5 (maj), 129.3 (min), 129.2, 129.1 (min), 129.0 (maj), 128.8, 127.3, 127.1, 68.2 (maj), 68.1 (min), 55.5 (min), 54.6 (maj), 52.3 (min), 51.7 (maj), 28.4 (min), 27.8 (maj), 19.3 (maj), 18.0 (min), 11.3 (maj), 11.0 (min); **HRMS** (ESI+): m/z calcd for $C_{20}H_{21}NO_2Na$ $[M+Na]^+$ 330.1470, found. 330.1466.

3-benzoyl-4-(4-chlorophenyl)-1-(1-phenylethyl)azetidin-2-one (3aba), (4aba)

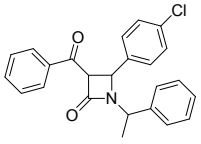


Table 2, Entry 3. Purification with flash chromatography R_f 0.31 (EtOAc:Hex 1:7, SiO_2) gave 109 mg, 0.28 mmol, 28% as two fractions respectively, mixture of two diasteroisomers (45 mg, 0.11 mmol, 11%, ratio **3aba** : **4aba**, 63:37, yellow oil) and II diasteroisomer (**4aba)** (64 mg, 0,17 mmol, 17%, yellow oil). Overall *de* = 48%. Table 2, Entry 4. Purification with flash chromatography R_f 0.31 (EtOAc:Hex 1:7, SiO_2) gave one fraction, mixture of two diasteroisomers (109 mg, 0,28 mmol, 28%, ratio **3aba** : **4aba**, 64:36, yellow oil). Overall *de* = 28%. **Mixture of diasteroisomers** (**3aba)**, (**4aba)**, ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 8.07 (d, J = 8.3 Hz 1.26H, ArH), 8.04 (d, J = 8.3 Hz 0.74H, ArH), 7.58 (q, J = 6.8 Hz, 1H, ArH), 7.55-7.44 (m, 3H, ArH), 7.38-7.22 (m, 6H, ArH), 7.17 (d, J = 7.8 Hz, 2H, ArH), 5.09-5.08 (m, 1H, H-4), 4.90 (q, J = 7.3 Hz, 0.37H, CH_3*CH*), 4.70-4.68 (m, 1H, H-3), 4.45 (q, J = 7.3 Hz, 0.63H, CH_3*CH*), 1.78 (d, J = 7.3 Hz, 1.89H, CH_3), 1.43 (d, J = 7.3 Hz, 1.11H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 191.3 (maj), 191.1 (min), 163.1 (min), 163.0 (maj), 142.1 (maj), 140.4 (maj), 139.6 (min), 137.0 (min), 136.0 (min), 135.8 (maj), 134.7 (min), 134.6 (maj), 134.1 (min), 129.7 (maj), 129.6 (maj), 129.5 (min), 129.4 (maj), 129.3 (min), 129.3 (maj), 129.0 (min), 128.9 (min), 128.6 (min), 128.5 (maj), 128.2 (min), 128.0 (maj), 127.4 (min), 127.0 (maj), 68.4 (min), 68.3 (maj), 55.3 (min), 55.0 (maj), 54.7 (maj), 53.7 (min), 20.1 (maj), 19.5 (min); **HRMS** (ESI+): m/z calcd for $C_{24}H_{20}ClNO_2Na$ $[M+Na]^+$ 412.1080, found. 412.1090. **II diasteroisomer** (**4aba)** **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 8.04 (d, J = 7.8 Hz, 2H, ArH), 7.57 (t, J = 6.8 Hz, 1H, ArH), 7.47 (t, J = 7.8 Hz, 2H, ArH), 7.44-7.26 (m, 7H, ArH), 7.22 (d, J = 6.8 Hz, 2H, ArH), 5.08 (d, J = 1.9 Hz, 1H, H-4), 4.90 (q, J = 6.8 Hz, 1H, CH_3*CH*), 4.70 (d, J = 1.9 Hz, 1H, H-3), 1.43 (d, J = 6.8 Hz, 3H, CH_3); ^{13}C-NMR (125 MHz, $CDCl_3$) δ: 191.3, 163.1, 139.6, 137.0, 136.0, 134.7, 134.1, 129.5, 129.3, 129.0, 128.9, 128.6, 128.2, 127.4, 68.4, 55.3, 53.7, 19.5; **HRMS** (ESI+): m/z calcd for $C_{24}H_{20}ClNO_2Na$ $[M+Na]^+$ 412.1080, found. 412.1090

2-(4-chlorophenyl)-6-phenyl-3-(1-phenylethyl)-2H-1,3-oxazin-4(3H)-one (5aba), (6aba)

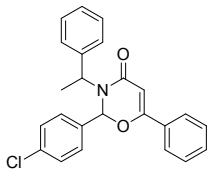


Table 2, Entry 4. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:3, SiO_2) gave one fraction, mixture of two diasteroisomers (109 mg, 0.28 mmol, 28%, white solid). **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 7.89-7.83 (m, 1H, ArH), 7.61-7.50 (m, 1H, ArH), 7.49-7.40 (m, 1H, ArH), 7.39-7.24 (m, 6H, ArH), 7.23-7.16 (m, 1H, ArH), 7.14-7.07 (m, 4H, ArH), 6.46 (s, 0.56H, NCHO), 6.23 (s, 0.44H, NCHO), 6.12 (q, J = 7.1 Hz, 0.44H, CH_3*CH*), 6.00 (q, J = 7.1 Hz, 0.56H, CH_3*CH*), 5.97 (s, 0.44H, CH), 5.93 (s, 0.56H, CH), 1.75 (d, J = 7.1 Hz, 1.32H, CH_3), 1.50 (d, J = 7.1 Hz, 1.68H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 163.4 (maj), 163.3 (min), 160.6 (min), 160.4 (maj), 140.7 (maj), 138.9 (min), 136.2 (maj), 135.6 (min), 135.4 (min), 135.3 (maj), 131.9 (min), 131.5 (maj), 131.4 (min), 129.1 (maj), 129.0 (min), 128.9 (min), 128.8 (maj), 128.8 (maj), 128.7 (min), 128.5 (maj), 128.4 (min), 128.3 (min), 128.2 (maj), 128.0 (min), 127.3 (maj), 127.1 (min), 126.5 (maj), 126.4 (min), 99.3 (min), 98.6 (maj), 84.2, 52.0 (min), 50.8 (maj), 18.0 (min), 17.9 (maj); **HRMS** (ESI+): m/z calcd for $C_{24}H_{20}ClNO_2Na$ $[M+Na]^+$ 412.1080, found. 412.1085.

3-Benzoyl-1-(1-(naphthalen-1-yl)ethyl)-4-phenylazetidin-2-one (3aac), (4aac)

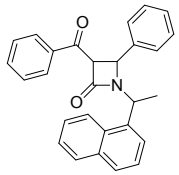


Table 2, Entry 6. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:6, SiO_2) gave one fraction, mixture of two diasteroisomers (105 mg, 0.26 mmol, 26%, ratio **3aac** : **4aac**, 63:37, yellow oil). Overall *de* = 26%. Table 2, Entry 5. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:6, SiO_2) gave one fraction, mixture of two diasteroisomers (97 mg, 0.24 mmol, 24%, ratio **3aac** : **4aac**, 58:42, yellow oil). Overall *de* = 16%. **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 8.09-8.07 (m, 1H, ArH), 8.04-8.03 (m, 0.65H, ArH), 8.00-7.99 (m, 1.35H, ArH), 7.90-7.89 (m, 0.65H, ArH), 7.83-7.82 (m, 1H, ArH), 7.69-7.67 (m, 0.35H, ArH), 7.60-7.23 (m, 10.35H, ArH), 7.16-7.13 (m, 0.35H, ArH), 7.08 (t, J = 7.3 Hz, 0.65H, ArH), 7.03 (d, J = 6.8 Hz, 0.65H, ArH), 5.82 (q, J = 7.3 Hz, 0.65H, CH_3*CH*), 5.44 (q, J = 7.3 Hz, 0.35H, CH_3*CH*), 5.17 (d, J = 1.9 Hz, 0.35H, H-4), 4.77 (d, J = 2.4 Hz, 0.65H, H-4), 4.76 (d, J = 2.4 Hz, 0.35H, H-3), 4.68 (d, J = 1.9 Hz, 0.65H, H-3), 1.95 (d, J = 7.3 Hz, 1.05H, CH_3), 1.58 (d, J = 6.8 Hz, 1.95H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 191.7 (min), 191.1 (maj), 163.6 (min), 163.4 (maj), 138.3 (maj), 136.8 (min), 136.3 (min), 136.2 (maj), 135.2, 134.0 (maj), 133.9 (min), 131.3 (maj), 130.9 (min), 129.5 (min), 129.3 (maj), 129.2 (min), 129.1 (maj), 129.0 (min), 129.0 (maj), 128.9 (min), 128.8 (maj), 128.7 (maj), 128.6 (min), 128.6, 127.5 (maj), 127.2 (min), 126.9 (maj), 126.7 (min), 126.2 (maj), 125.9 (min), 125.5 (min), 125.1 (maj), 124.6 (maj), 124.3 (min), 123.0, 122.9, 68.3 (min), 67.8 (maj), 56.0 (maj), 55.3 (min), 49.9 (min), 48.5 (maj), 19.4 (min), 19.3 (maj); **HRMS** (ESI+): m/z calcd for $C_{28}H_{23}NO_2Na$ $[M+Na]^+$ 428.1626, found. 428.1638.

3-Benzoyl-1-(1-(naphthalen-2-yl)ethyl)-4-phenylazetidin-2-one (3aad), (4aad)

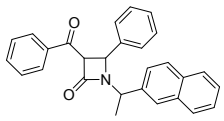


Table 2, Entry 7. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:6, SiO_2) gave one fraction, mixture of two diasteroisomers (73 mg, 0.18 mmol, 18%, ratio **3aad** : **4aad**, 68:32, yellow oil). overall *de* = 36%. Table 2, Entry 8. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:6, SiO_2) gave (102 mg, 0,25 mmol, 25%) as three fractions respectively, I diasteroisomer **3aad** (28 mg, 0.07 mmol, 7%, yellow oil), mixture of two diasteroisomers (46 mg, 0.11 mmol, 11%, ratio **3aad** : **4aad**, 42:58, yellow oil) and II diasteroisomer **4aad** (28 mg, 0.07 mmol, 7%, yellow oil). Overall *de* = 6%. **I diasteroisomer** (**3aad**) **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 8.04 (d, J = 7.4 Hz, 2H, ArH), 7.84-7.82 (m, 2H, ArH), 7.78-7.76 (m, 1H, ArH), 7.63 (s, 1H, ArH), 7.56 (t, J = 7.7 Hz, 1H, ArH), 7.49-7.42 (m, 4H, ArH), 7.36-7.32 (m, 5H,

ArH), 7.28-7.26 (m, 1H, ArH), 5.12 (q, J = 7.2 Hz, 1H, CH_3CH), 5.11 (d, J = 2.0 Hz, 1H, H-4), 4.78 (d, J = 2.0 Hz, 1H, H-3), 1.51 (d, J = 7.2 Hz, 3H, CH_3); **II diasteroisomer** (**4aad**) ^{1}H-NMR (500 MHz, $CDCl_3$) δ: 8.08 (d, J = 7.4 Hz 2H, ArH), 7.82-7.78 (m, 2H, ArH), 7.75-7.73 (m, 1H, ArH), 7.60-7.50 (m, 2H, ArH), 7.48-7.45 (m, 4H, ArH), 7.41-7.38 (m, 1H, ArH), 7.33-7.26 (m, 5H, ArH), 5.11 (d, J = 2.2 Hz, 1H, H-4), 4.77 (d, J = 2.2 Hz, 1H, H-3), 4.58 (q, J = 7.1 Hz, 1H, CH_3CH), 1.91 (d, J = 7.1 Hz, 3H, CH_3); **Mixture of diasteroisomers (3aad), (4aad) ^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 8.08 (d, J = 7.4 Hz 0.84H, ArH), 8.04 (d, J = 7.4 Hz 1.16H, ArH), 7.85-7.82 (m, 1.16H, ArH), 7.82-7.78 (m, 0.84H, ArH), 7.78-7.76 (m, 0.58H, ArH), 7.76-7.73 (m, 0.42H, ArH), 7.64 (s, 0.58H, ArH), 7.60-7.54 (m, 1.42H, ArH), 7.49-7.42 (m, 4H, ArH), 7.41-7.33 (m, 3.32H, ArH), 7.33-7.26 (m, 2.68H, ArH), 5.14-5.10 (m, 1.58H, CH_3CH+H4), 4.79-4.78 (m, 1H, H-3), 4.58 (q, J = 6.9 Hz, 0.42H, CH_3CH), 1.91 (d, J = 6.9 Hz, 1.26H, CH_3), 1.51 (d, J = 6.9 Hz, 1.74H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 191.7 (min), 191.4 (maj), 163.5, 163.4 (maj), 138.4 (maj), 138.2 (min), 137.3 (min), 137.2 (maj), 136.2 (min), 136.1 (maj), 134.0, 133.4, 133.1, 129.5 (min), 129.4 (maj), 129.2 (min), 129.1 (maj), 129.0, 128.9 (min), 128.9 (maj), 128.8, 128.3 (maj), 128.2 (min), 127.9 (maj), 127.8 (min), 127.3 (maj), 127.2 (min), 126.4 (maj), 126.3 (min), 126.2 (min), 126.1 (maj), 125.9 (min), 125.7 (maj), 125.0, 68.3 (maj), 68.2 (min), 56.0 (maj), 55.5 (min), 55.3 (min), 53.6 (maj), 20.3 (min), 19.4 (maj); **HRMS** (ESI+): m/z calcd for $C_{28}H_{23}NO_2Na$ $[M+Na]^+$ 428.1626, found. 428.1630.

3-((Naphthalen-1-yl)acetyl)-4-phenyl-1-(1-phenylethyl)azetidin-2-one (3baa), (4baa)

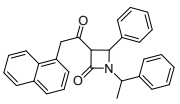


Table 2, Entry 9. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:3, SiO_2) gave one fraction, mixture of two diasteroisomers (176 mg, 0.42 mmol, 42%, ratio **3baa** : **4baa**, 52:48, yellow oil). Overall *de* = 4%. Table 2, Entry 10. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:3, SiO_2) gave one fraction, mixture of two diasteroisomers (185 mg, 0.44 mmol, 44%, ratio **3baa** : **4baa** 67:33, yellow oil). Overall *de* = 34%. Table 2, Entry 11. Purification with flash chromatography R_f 0.30 (EtOAc:Hex 1:3, SiO_2) gave one fraction, mixture of two diasteroisomers (147 mg, 0.35 mmol, 35%, ratio **3baa** : **4baa**, 67:33, yellow oil). Overall *de* = 34%. **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 7.93-7.83 (m, 2.33H, ArH), 7.78-7.75 (m, 1H, ArH), 7.56-7.46 (m, 2.67H, ArH), 7.45-7.38 (m, 1.67H, ArH), 7.34-7.22 (m, 6.66H, ArH), 7.17-7.14 (m, 2H, ArH), 7.06-7.04 (m, 0.67H, ArH), 4.94 (q, J = 7.3 Hz, 0.67H, CH_3CH), 4.79 (d, J = 2.0 Hz, 0.67H, H-4), 4.78 (d, J = 2.0 Hz, 0.33H, H-4), 4.48 (d, J = 16.1 Hz, 0.33H, CH_2), 4.33 (d, J = 16.1 Hz, 0.67H, CH_2), 4.36 (q, J = 7.3 Hz, 0.33H, CH_3CH), 4.28 (d, J = 16.1 Hz, 0.33H, CH_2), 4.25 (d, J = 16.1 Hz, 0.67H, CH_2), 4.19 (d, J = 2.0 Hz, 0.67H, H-3), 4.17 (d, J = 2.0 Hz, 0.33H, H-3), 1.80 (d, J = 7.3 Hz, 0.99H, CH_3), 1.37 (d, J = 7.3 Hz, 2.01H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 199.8 (maj), 199.5 (min), 163.6, 140.8 (maj), 139.7 (min), 137.9 (min), 136.7 (maj) 134.1, 132.5 (maj), 132.4 (min), 129.9, 129.1, 129.0 (min), 128.9 (maj), 128.9 (maj), 128.8 (min), 128.5, 128.1 (min), 127.9 (maj), 127.4, 127.2, 127.1, 126.9, 126.8 (maj), 126.8 (min), 126.1 (maj), 126.1 (min), 125.8 (maj), 125.8 (min), 124.2, 69.5 (min), 69.4 (maj), 55.9 (min), 55.5 (maj), 55.1 (maj), 53.5 (min), 48.1 (maj), 48.0 (min), 20.4 (maj), 19.4 (min); **HRMS** (ESI+): m/z calcd for $C_{29}H_{25}NO_2Na$ $[M+Na]^+$ 442.1783, found. 442.1786.

4-(4-Chlorophenyl)-3-((naphthalen-1-yl)acetyl)-1-(1-phenylethyl)azetidin-2-one (3bba), (4bba)

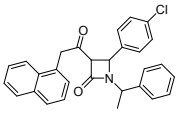


Table 2, Entry 12. Purification with flash chromatography R_f 0.32 (EtOAc:Hex 1:3, SiO_2) gave one fraction, mixture of two diasteroisomers (222 mg, 0.49 mmol, 49%, ratio **3bba** : **4bba**, 66:34, yellow oil). Overall *de* = 32%. **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 7.90-7.89 (m, 0.33H, ArH), 7.86-7.82 (m, 1.66H, ArH), 7.77-7.74 (m, 1H, ArH), 7.53-7.32 (m, 4H, ArH), 7.32-7.23 (m, 3H, ArH), 7.20-7.20 (m, 4H, ArH), 7.00 (d, J = 8.3 Hz, 1.33H, ArH), 6.90 (d, J = 8.3 Hz, 0.66H, ArH), 4.89 (q, J = 6.8 Hz, 0.66H, CH_3CH), 4.70 (d, J = 2.4 Hz, 0.33H, H-4), 4.68 (d, J = 2.4 Hz, 0.66H, H-4), 4.46-4.36 (m, 1.33H, CH_3CH +CH_2), 4.27-4.20 (m, 1H, CH_2), 4.09 (d, J = 2.4 Hz, 0.66H, H-3), 4.08 (d, J = 2.4 Hz, 0.33H, H-3), 1.75 (d, J = 7.3 Hz, 1H, CH_3), 1.37 (d, J = 6.8 Hz, 2H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 199.7 (min), 199.4 (maj), 163.4 (maj),

163.3 (min), 140.4 (min), 139.4 (maj), 136.4 (maj), 135.3 (min) 134.7 (maj), 134.6 (min),134.1, 132.4, 129.7 (min), 129.2 (maj), 129.2 (min), 128.9 (maj), 128.8, 128.7, 128.5 (min), 128.4 (maj), 128.3, 128.2 (maj), 128.0 (min), 127.5, 127.3 (maj), 127.0 (min), 126.9 (min), 126.8 (maj), 126.1, 125.8 (min), 125.8 (maj), 124.1, 69.3, 55.3 (maj), 54.9 (min), 54.8 (min), 53.6 (maj), 48.2 (min), 48.1 (maj), 20.1 (min), 19.4 (maj); **HRMS** (ESI+): m/z calcd for $C_{29}H_{24}ClNO_2Na$ $[M+Na]^+$ 476.1393, found. 476.1396.

3-((Naphthalen-1-yl)acetyl)-1-(1-(naphthalen-1-yl)ethyl)-4-phenylazetidin-2-one (3bac), (4bac)

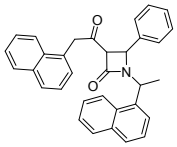


Table 2, Entry 13. Purification with flash chromatography R_f 0.29 (EtOAc:Hex 1:3, SiO_2) gave one fraction, mixture of two diasteroisomers (174 mg, 0.37 mmol, 37%, ratio **3bac** : **4bac**, 61:39). Overall *de* = 22%. **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 8.09 (d, J = 8.3 Hz, 0.61H, ArH), 8.04 (d, J = 8.3 Hz, 0.39H, ArH), 7.94-7.73 (m, 4.61H, ArH), 7.67 (d, J = 8.3 Hz, 0.39H, ArH), 7.57-7.33 (m, 7H, ArH), 7.29-7.20 (m, 3H, ArH), 7.10 (t, J = 7.3 Hz, 0.39H, ArH), 7.00 (d, J = 7.8 Hz, 2H, ArH), 6.81 (d, J = 7.3 Hz, 0.61H, ArH), 5.84 (q, J = 7.3 Hz, 0.61H, CH_3*CH*), 5.37 (q, J = 6.8 Hz, 0.39H, CH_3*CH*), 4.83 (d, J = 2.0 Hz, 0.39H, H-4), 4.51 (d, J = 16.1 Hz, 0.39H, CH_2), 4.37 (d, J = 16.1 Hz, 0.61H, CH_2), 4.33 (d, J = 2.0 Hz, 0.61H, H-4), 4.28 (d, J = 16.1 Hz, 0.39H, CH_2), 4.21 (d, J = 16.1 Hz, 0.61H, CH_2), 4.20 (d, J = 2.0 Hz, 0.39H, H-3), 4.19 (d, J = 2.0 Hz, 0.61H, H-3), 1.93 (d, J = 6.8 Hz, 1.17H, CH_3), 1.52 (d, J = 6.8 Hz, 1.83H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 199.8 (min), 199.0 (maj), 163.8 (min), 163.6 (maj), 137.8 (maj), 136.2 (min), 135.2 (min), 134.1 (maj), 134.1, 134.0, 133.9, 132.5 (min), 132.4 (maj), 131.2 (maj), 130.9 (min), 129.8, 129.2, 129.1 (min), 129.0 (maj), 128.9, 128.8 (maj), 128.7 (min), 128.6 (maj), 128.6 (min), 128.5 (min), 128.4 (maj), 127.3 (maj), 127.2 (min), 126.9 (min), 126.8 (maj), 126.7 (maj), 126.6 (min), 126.3 (maj), 126.1 (min), 126.0 (maj), 125.9 (min), 125.8 (min), 125.7 (maj), 125.5 (min), 125.1 (maj), 124.5, 124.2 (min), 124.1 (maj), 122.9 (maj), 122.8 (min), 69.5 (min), 69.0 (maj), 55.9 (maj), 55.3 (min), 48.4, 48.2 (min), 47.7 (maj), 19.4 (min), 19.2 (maj); **HRMS** (ESI+): m/z calcd for $C_{33}H_{27}NO_2Na$ $[M+Na]^+$ 492.1939, found. 492.1938.

3-((Naphthalen-1-yl)acetyl)-1-(1-(naphthalen-2-yl)ethyl)-4-phenylazetidin-2-one (3bad), (4bad)

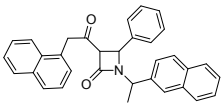
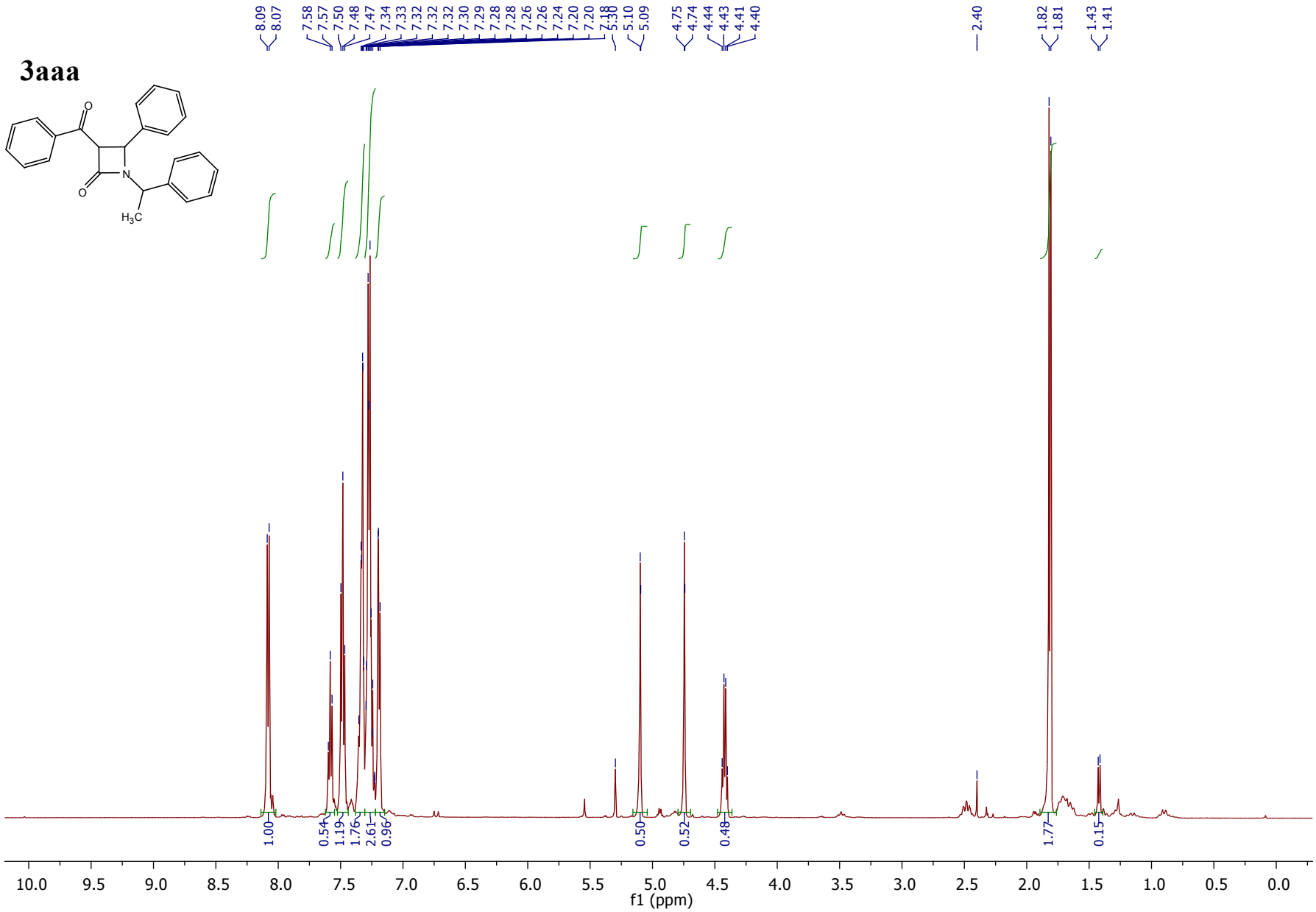
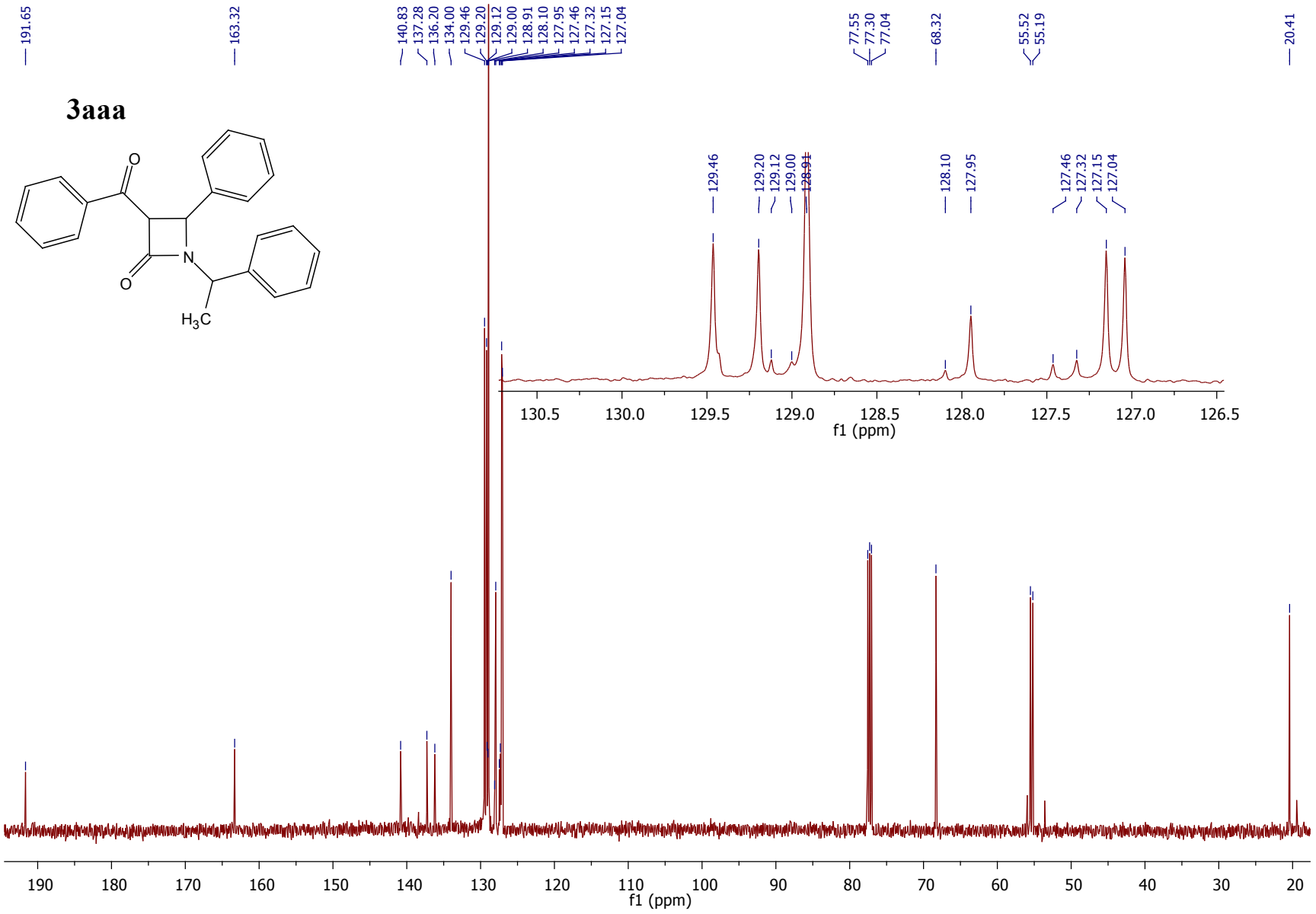


Table 2, Entry 14. Purification with flash chromatography R_f 0.29 (EtOAc:Hex 1:3, SiO_2) gave one fraction, mixture of two diasteroisomers (206 mg, 0.48 mmol, 48%, ratio **3bad** : **4bad**, 62:38, yellow oil). Overall *de* = 24%. **^{1}H-NMR** (500 MHz, $CDCl_3$) δ: 7.93-7.00 (m, 6H, ArH), 7.59 (s, 0.62H, ArH), 7.52-7.42 (m, 4.38H, ArH), 7.42-7.34 (m, 2.62H, ArH), 7.28-7.13 (m, 3.38H, ArH), 7.11 (d, J = 1.5 Hz, 1.38H, ArH), 7.04-7.02 (m, 0.62H, ArH), 5.10 (q, J = 7.3 Hz, 0.62H, CH_3*CH*), 4.79-4.77 (m, 1H, H-4), 4.51 (q, J = 7.3 Hz, 0.38H, CH_3*CH*), 4.38 (d, J = 16.1 Hz, 0.38H, CH_2), 4.42 (d, J = 16.1 Hz, 0.62H, CH_2), 4.28 (d, J = 16.1 Hz, 0.38H, CH_2), 4.25 (d, J = 16.1 Hz, 0.62H, CH_2), 4.20 (d, J = 1.9 Hz, 0.62H, H-3), 4.18 (d, J = 1.9 Hz, 0.38H, H-3), 1.87 (d, J = 7.3 Hz, 1.14H, CH_3), 1.45 (d, J = 7.3 Hz, 1.86H, CH_3); **^{13}C-NMR** (125 MHz, $CDCl_3$) δ: 199.8 (min), 199.6 (maj), 163.7 (maj), 163.6 (min), 138.2 (min), 137.9 (maj), 137.2 (maj), 136.7 (min) 134.1 (min), 134.0 (maj), 133.4, 133.2 (maj), 133.1 (min), 132.5 (min), 132.4 (maj), 129.9 (min), 129.8 (maj), 129.1 (min), 129.0 (maj), 128.9 (min), 128.9 (maj), 128.5, 128.3 (maj), 128.2 (min), 127.9 (maj), 127.8 (min), 127.3 (maj), 127.1 (min), 126.8 (min), 126.8 (maj), 126.5 (maj), 126.5 (min), 126.4, 126.3, 126.2 (min), 126.1 (maj), 126.0, 125.9 (maj), 125.8 (min), 125.7, 125.6, 124.9, 124.2, 69.5 (maj), 69.4 (min), 56.0 (maj), 55.6 (min), 55.3 (min), 53.5 (maj), 48.2 (min), 48.0 (maj), 20.4 (min), 19.4 (maj); **HRMS** (ESI+): m/z calcd for $C_{33}H_{27}NO_2Na$ $[M+Na]^+$ 492.1939, found. 492.1942.

^{1}H and ^{13}C-NMR Spectra

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127.15
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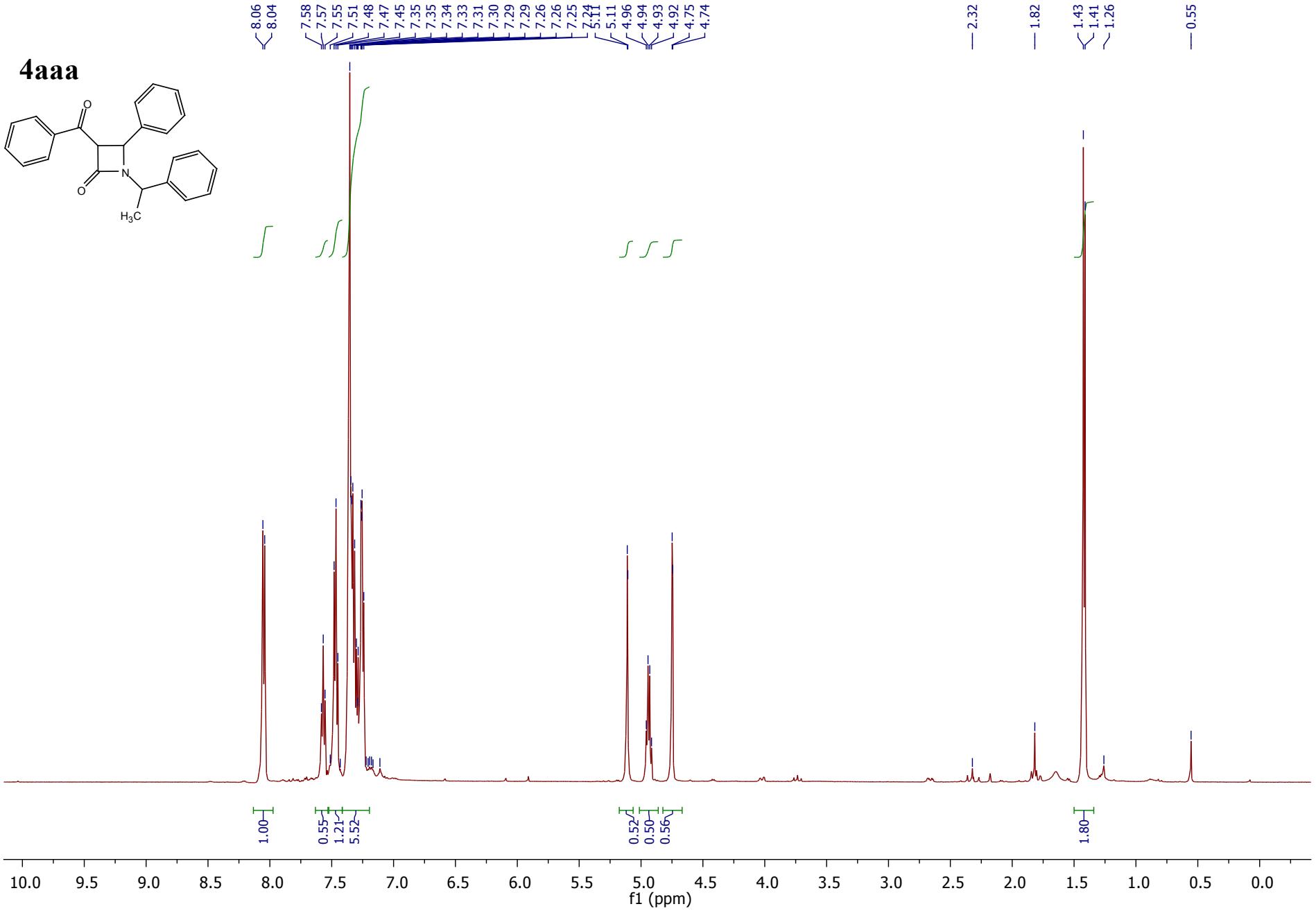
f1 (ppm)
f1 (ppm)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4.92
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1.41
1.26
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0.55
1.21
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0.56
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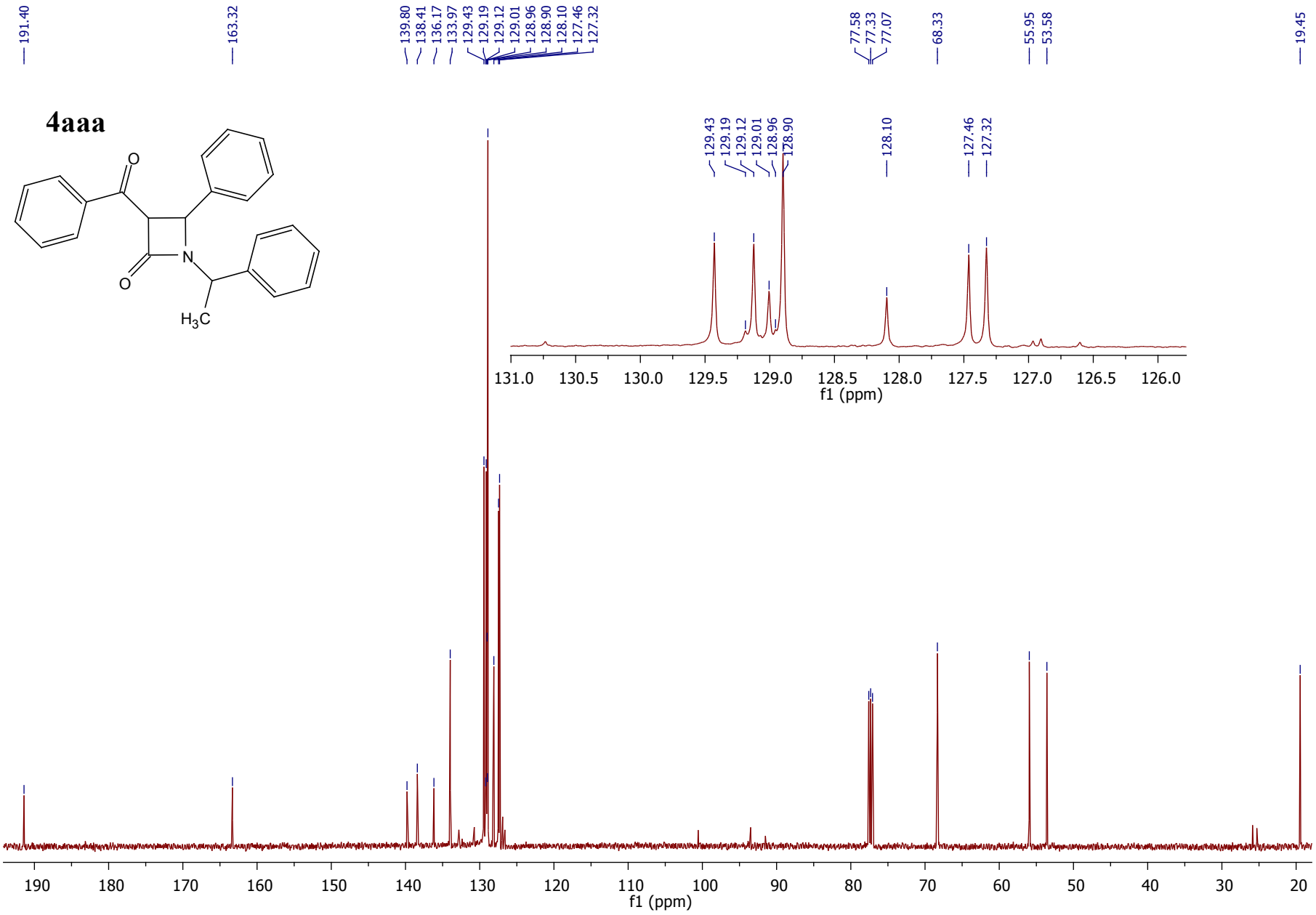
f1 (ppm)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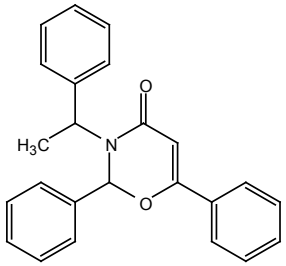
N
H3C
f1 (ppm)
f1 (ppm)

5aaa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N
O

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9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
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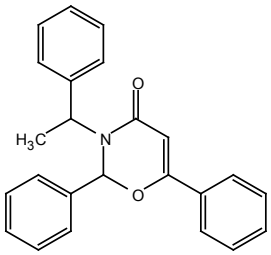
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H3C
N
O

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17.88
190
180
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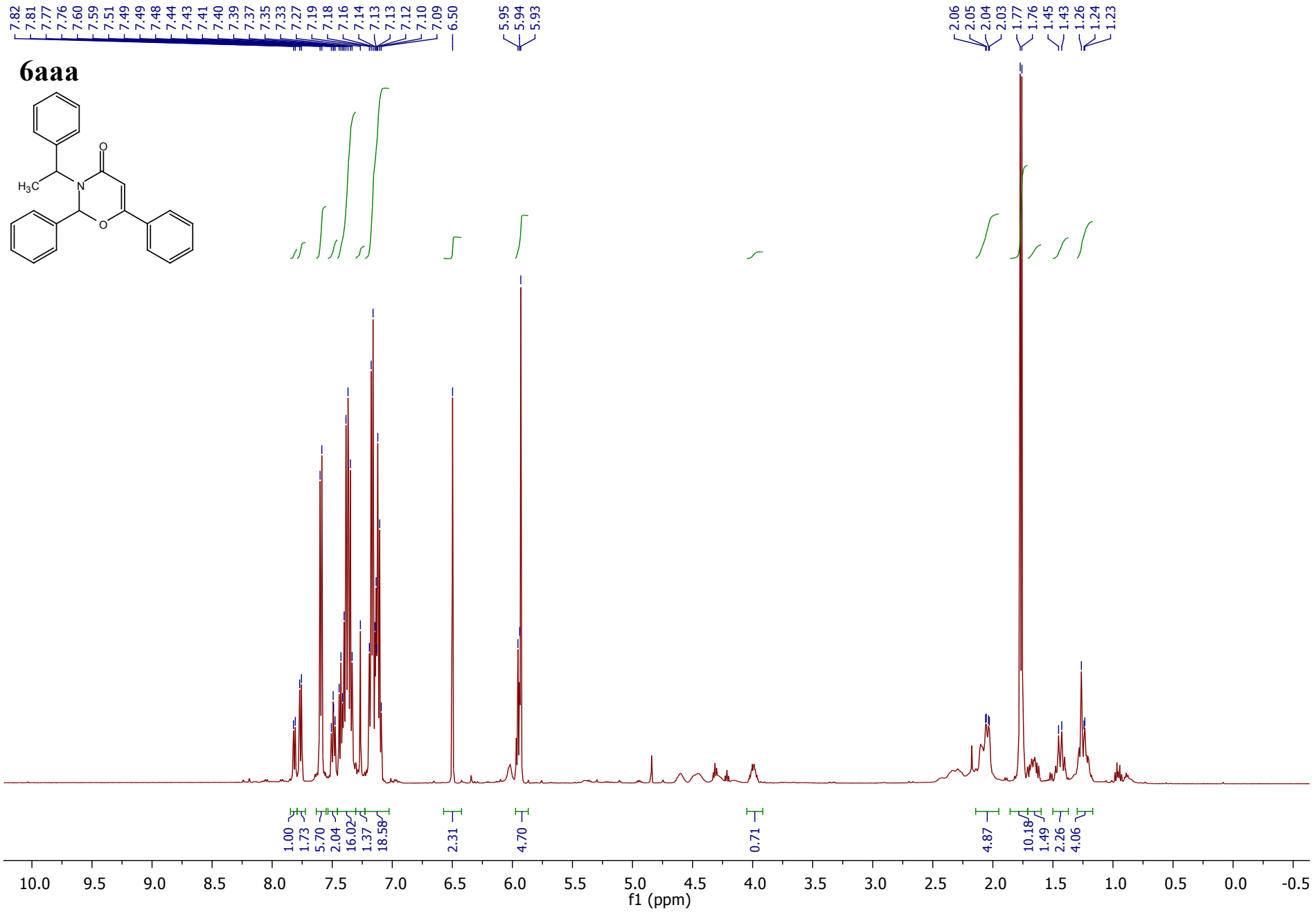
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5.95 5.94 5.93
2.06 2.05 2.04 2.03 1.77 1.76 1.45 1.43 1.26 1.24 1.23
O
H3C
N
O
1.00 1.73 5.70 2.04 16.02 1.37 18.58 2.31 4.70 0.71 4.87 10.18 1.49 2.26 4.06
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
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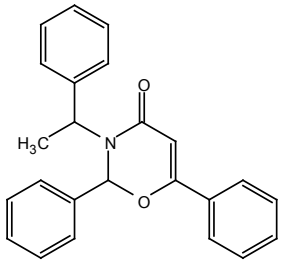
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H3C
N
O

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80
70
60
50
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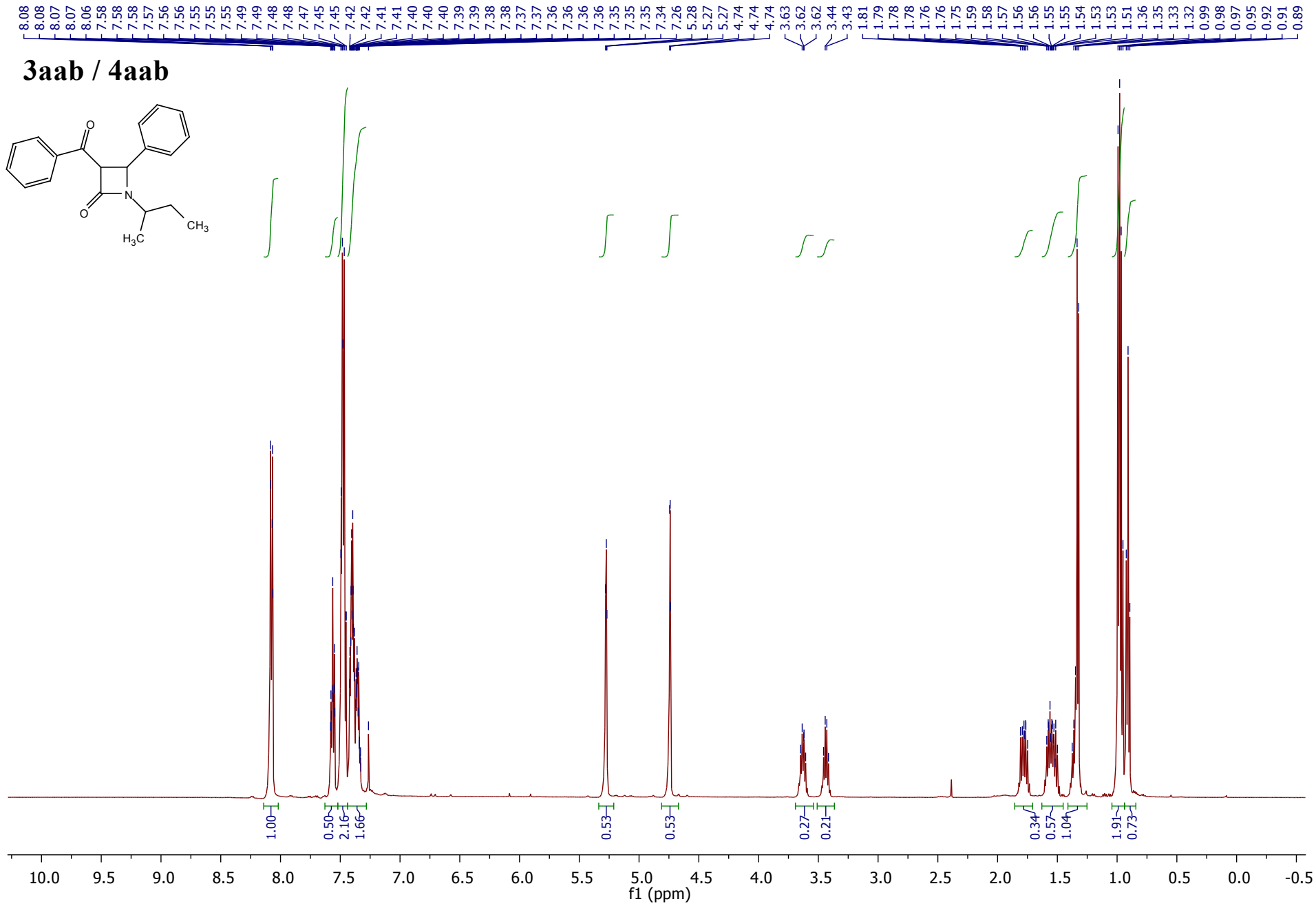
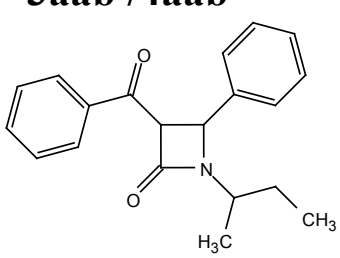
30
20
10
f1 (ppm)


3aab / 4aab
8.08 8.08 8.07 8.07 8.06 7.58 7.58 7.58 7.57 7.56 7.55 7.55 7.55 7.49 7.49 7.48 7.48 7.47 7.45 7.45 7.42 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.38 7.38 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.34 7.26 5.28 5.27 4.74 4.74 4.74 3.63 3.62 3.62 3.44 3.43 1.81 1.79 1.78 1.78 1.76 1.76 1.75 1.59 1.58 1.57 1.56 1.56 1.55 1.55 1.54 1.53 1.53 1.51 1.36 1.35 1.33 1.32 0.99 0.98 0.97 0.95 0.92 0.91 0.89
O
O
N
CH3
H3C
1.00 0.50 2.16 1.66 0.53 0.53 0.27 0.21 0.34 0.57 1.04 1.91 0.73
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

3aab /4aab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O
CH3
H3C

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129.20
129.05
129.03
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127.26
127.22
77.59
77.34
77.08
68.30
68.08
55.64
54.73
52.26
51.69
28.44
27.76
19.25
18.14
11.28
10.98
220
210
200
190
180
170
160
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90
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70
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50
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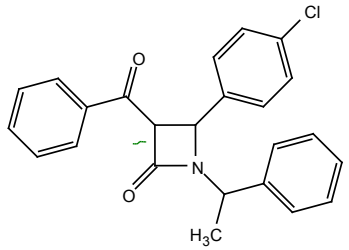
30
20
10
0
f1 (ppm)

3aba / 4aba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O
N
H_3C

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7.61
22.81
13.19
7.80
55.41
32.44
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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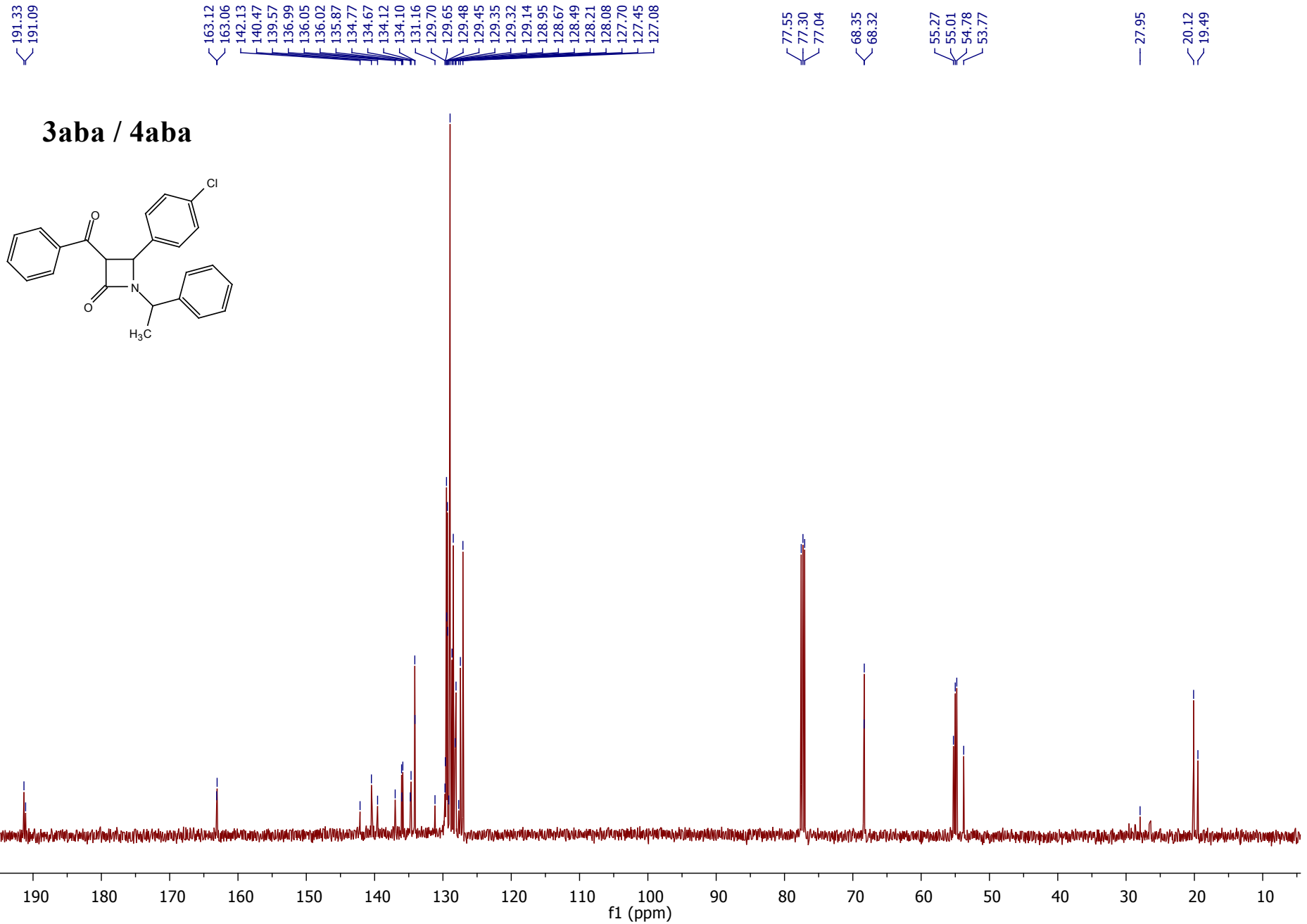
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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134.10
131.16
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129.65
129.48
129.45
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129.32
129.14
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128.67
128.49
128.21
128.08
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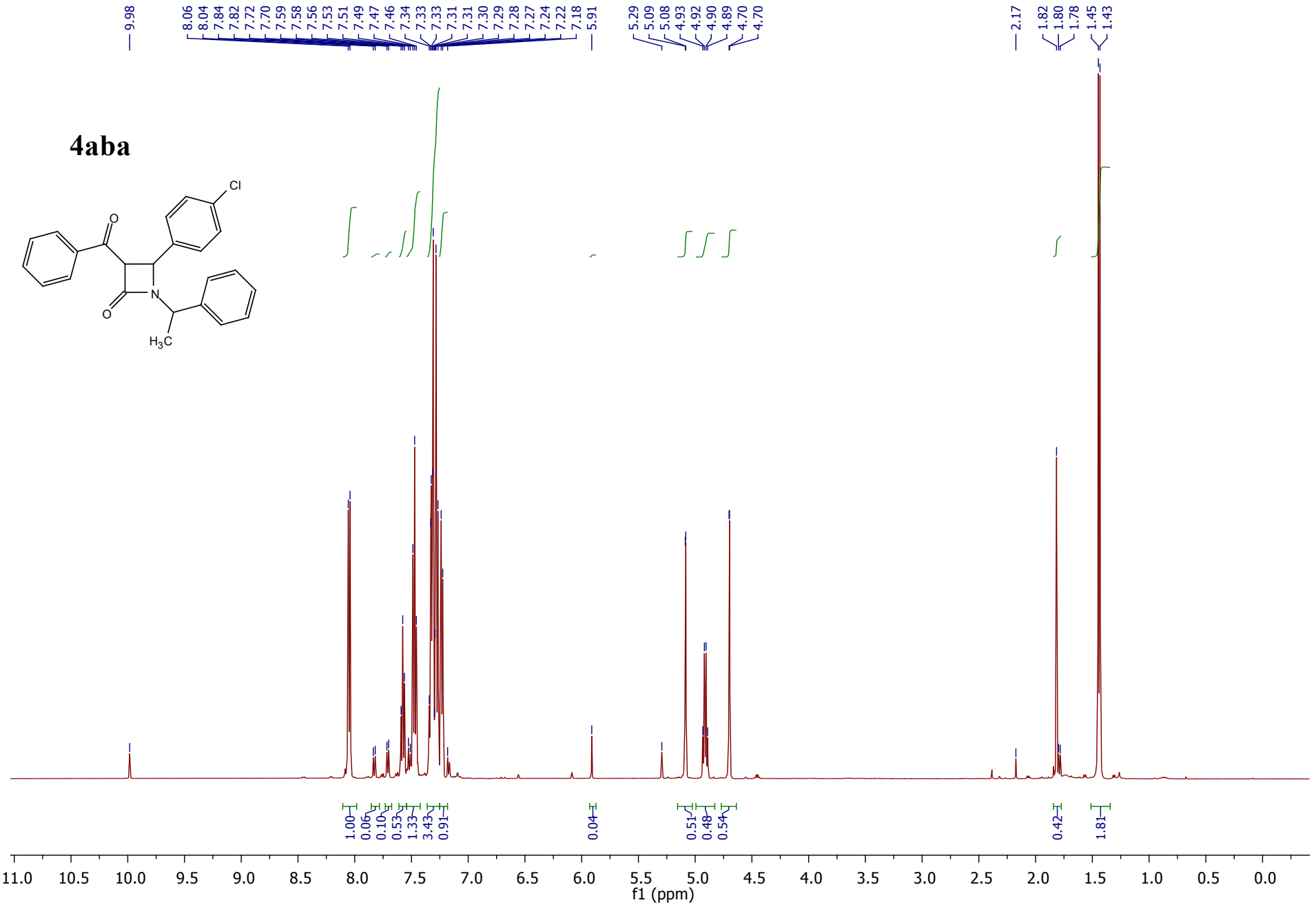
127.45
127.08
77.55
77.30
77.04
68.35
68.32
55.27
55.01
54.78
53.77
27.95
20.12
19.49
3aba / 4aba
Cl
O
O
N
H3C
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


4aba
Cl
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O
N
H_3C
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8.06
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7.72
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7.58
7.56
7.53
7.51
7.49
7.47
7.46
7.34
7.33
7.33
7.31
7.31
7.30
7.29
7.28
7.27
7.24
7.22
7.18
5.91
5.29
5.09
5.08
4.93
4.92
4.90
4.89
4.70
4.70
2.17
1.82
1.80
1.78
1.45
1.43
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0.10
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1.33
3.43
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0.04
0.51
0.48
0.54
0.42
1.81
11.0
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9.0
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8.0
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6.5
6.0
5.5
5.0
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3.0
2.5
2.0
1.5
1.0
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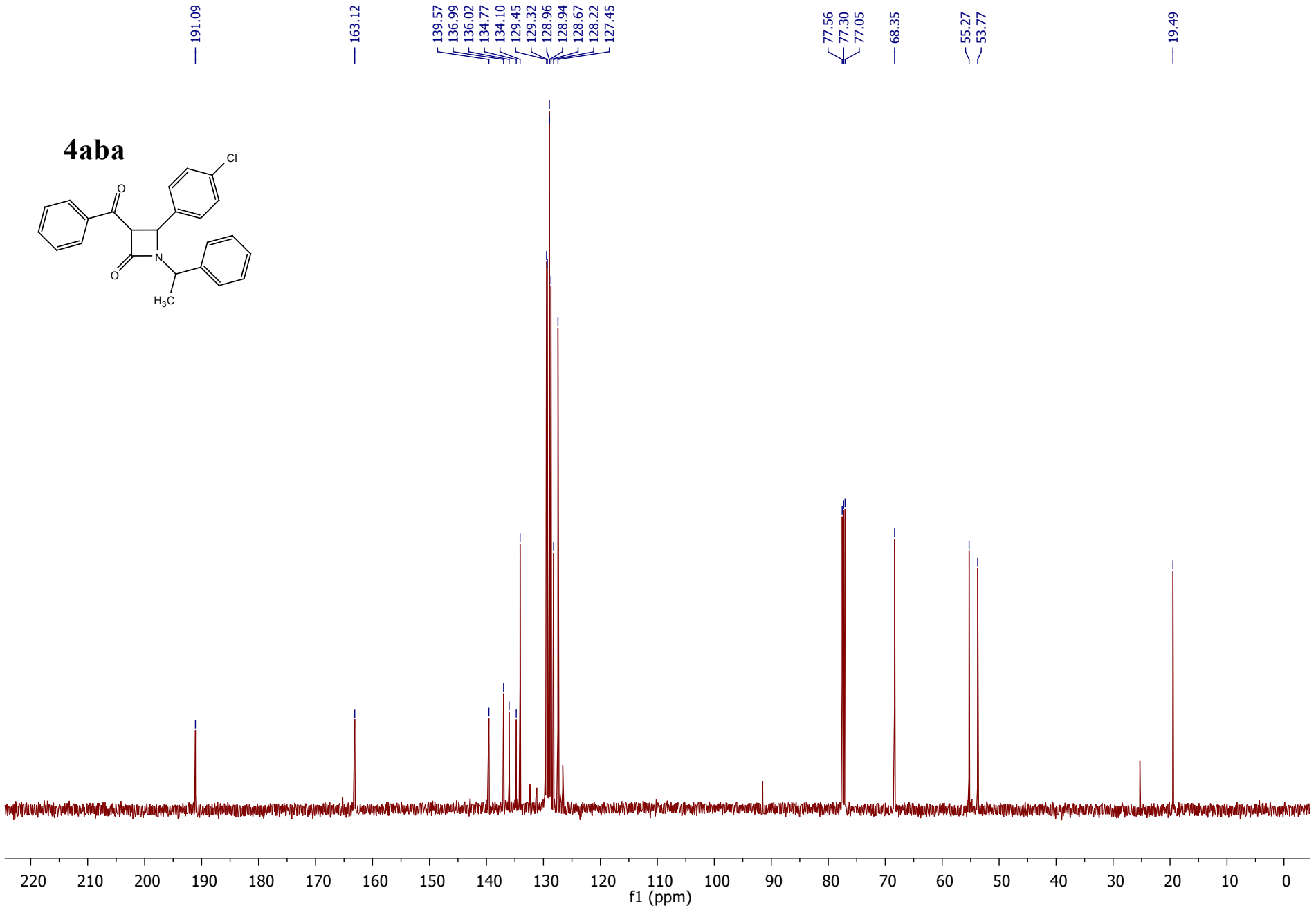
0.0
f1 (ppm)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136.99
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129.45
129.32
128.96
128.94
128.67
128.22
127.45
77.56
77.30
77.05
68.35
55.27
53.77
19.49
4aba
Cl
O
O
N
H3C
220
210
200
190
180
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150
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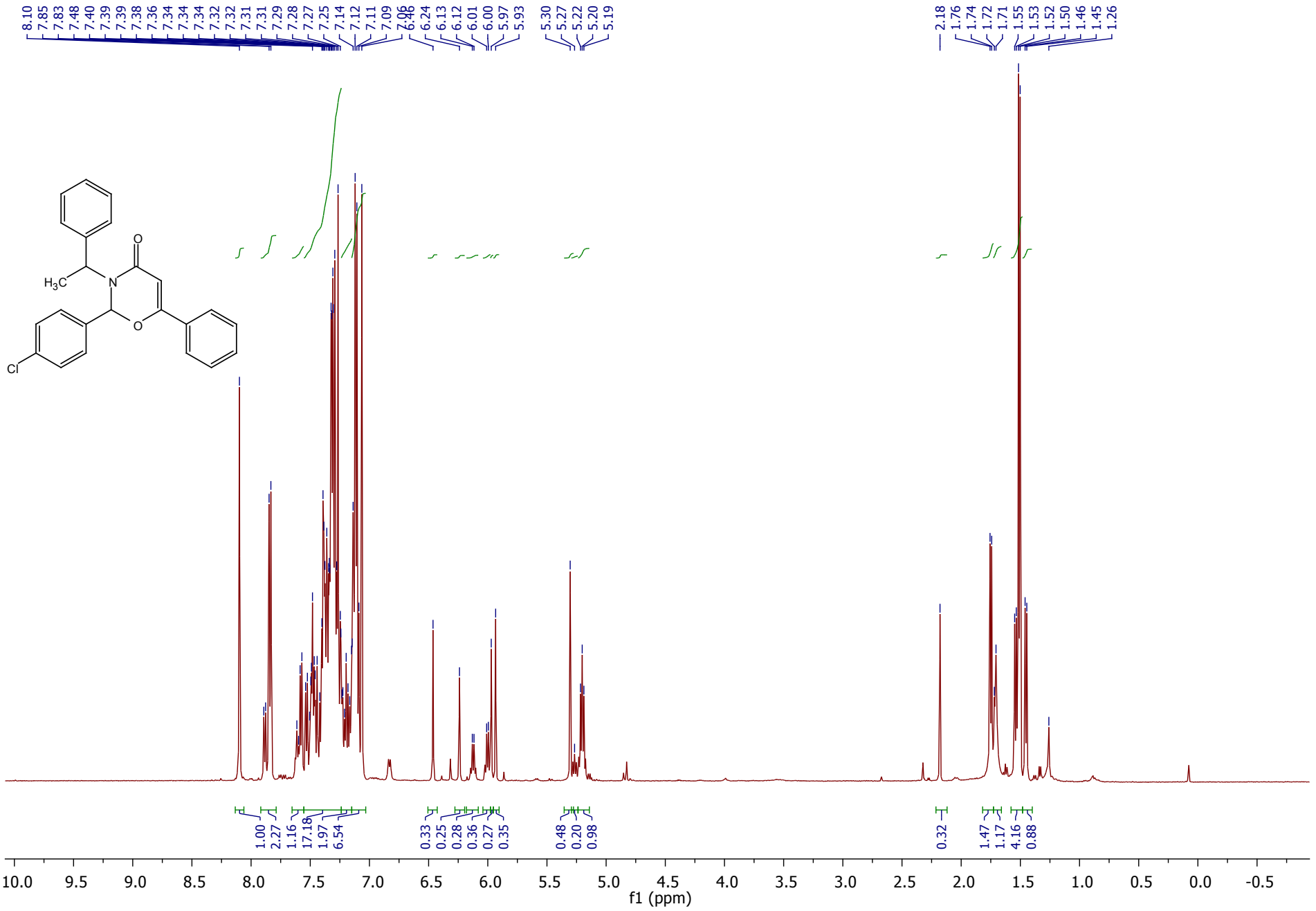
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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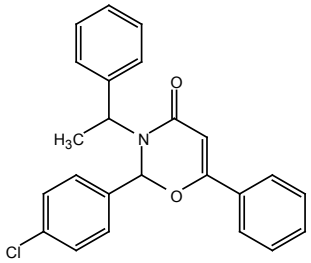
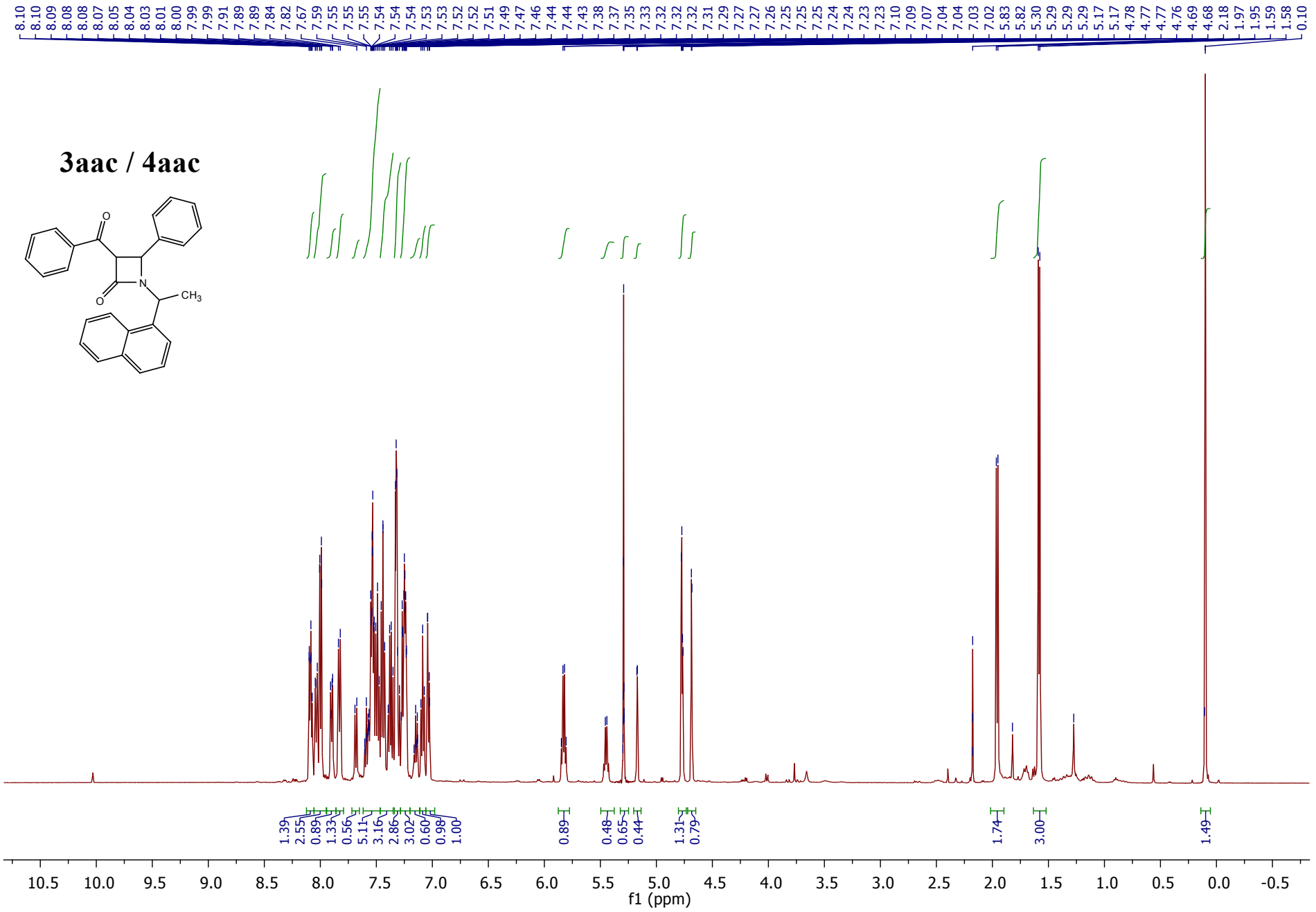
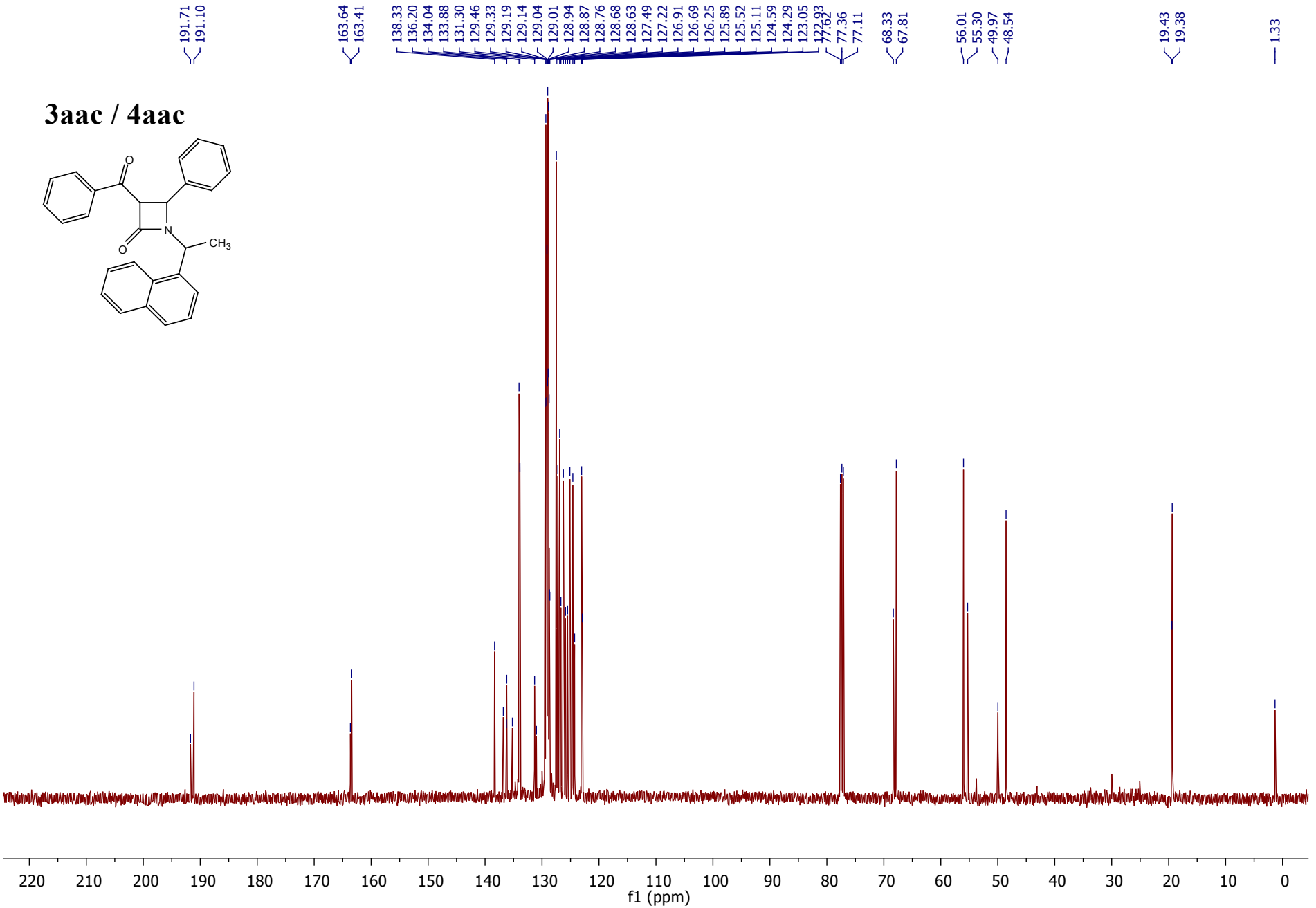
7.34
7.34
7.32
7.32
7.31
7.31
7.29
7.28
7.27
7.25
7.14
7.12
7.11
7.09
6.24
6.13
6.12
6.01
6.00
5.97
5.93
5.30
5.27
5.22
5.20
5.19
2.18
1.76
1.74
1.72
1.71
1.55
1.53
1.52
1.50
1.46
1.45
1.26
O
N
O
H3C
Cl
1.00
2.27
1.16
17.18
1.97
6.54
0.33
0.25
0.28
0.36
0.27
0.35
0.48
0.20
0.98
0.32
1.47
1.17
4.16
0.88
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

5aba / 6aba


O
H_3C
N
O
Cl

163.35
163.31
160.60
160.47
136.24
131.90
131.51
131.44
129.05
129.00
128.95
128.88
128.82
128.71
128.57
128.34
128.27
128.19
128.05
127.28
127.14
126.52
126.48
99.31
98.66
84.21
77.61
77.36
77.10
51.75
50.54
48.94
33.46
25.83
25.21
17.93
17.89
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


3aac / 4aac
O
O
N
CH3
f1 (ppm)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128.63
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127.22
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126.69
126.25
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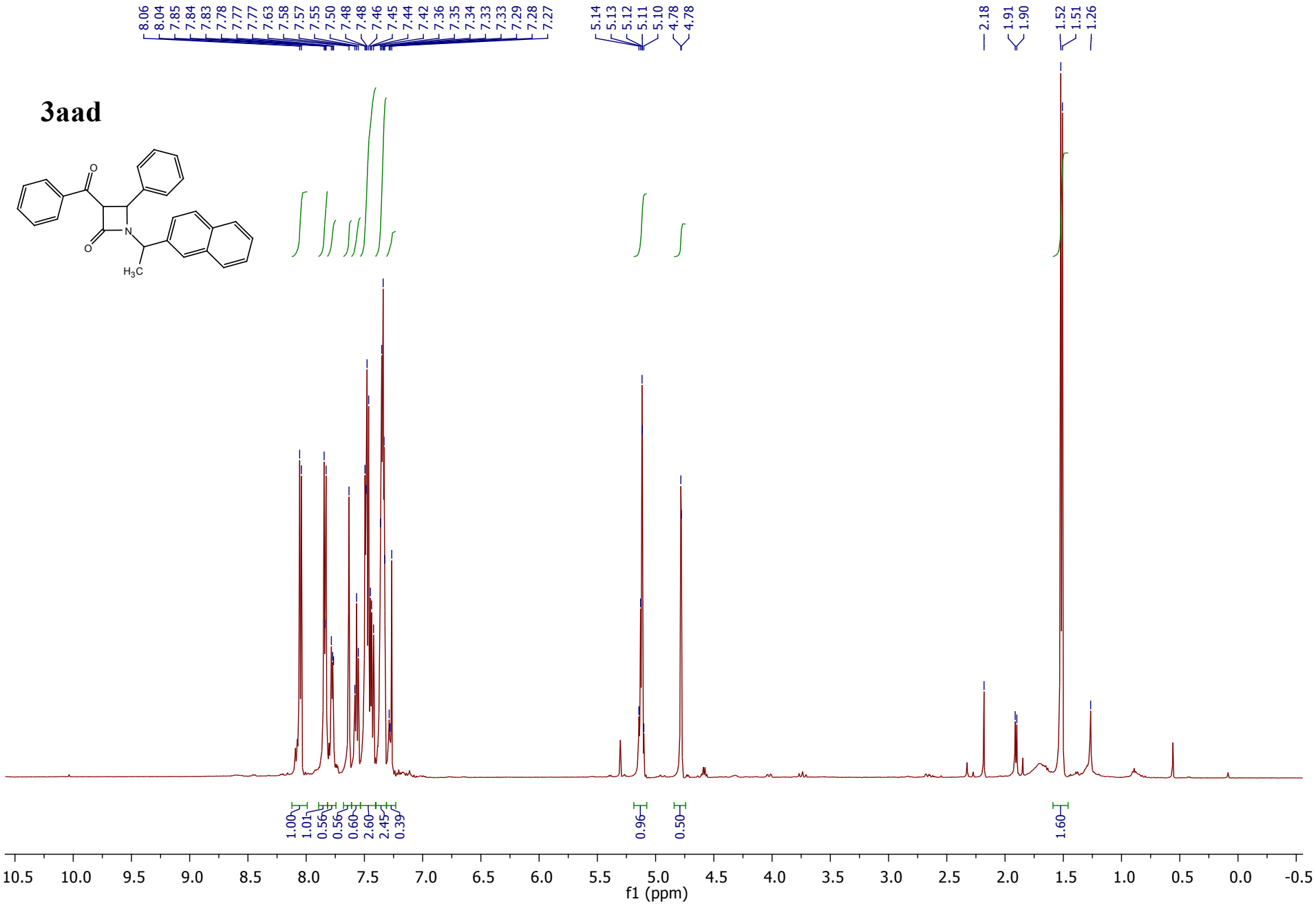
125.52
125.11
124.59
124.29
123.05
77.62
77.36
77.11
68.33
67.81
56.01
55.30
49.97
48.54
19.43
19.38
1.33
3aac / 4aac
O
O
N
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.57
7.55
7.50
7.48
7.48
7.46
7.45
7.44
7.42
7.36
7.35
7.34
7.33
7.33
7.29
7.28
7.27
5.14
5.13
5.12
5.11
5.10
4.78
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2.18
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1.90
1.52
1.51
1.26
1.00
1.01
0.56
0.56
0.60
2.60
2.45
0.39
0.96
0.50
1.60
10.5
10.0
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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2.5
2.0
1.5
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0.5
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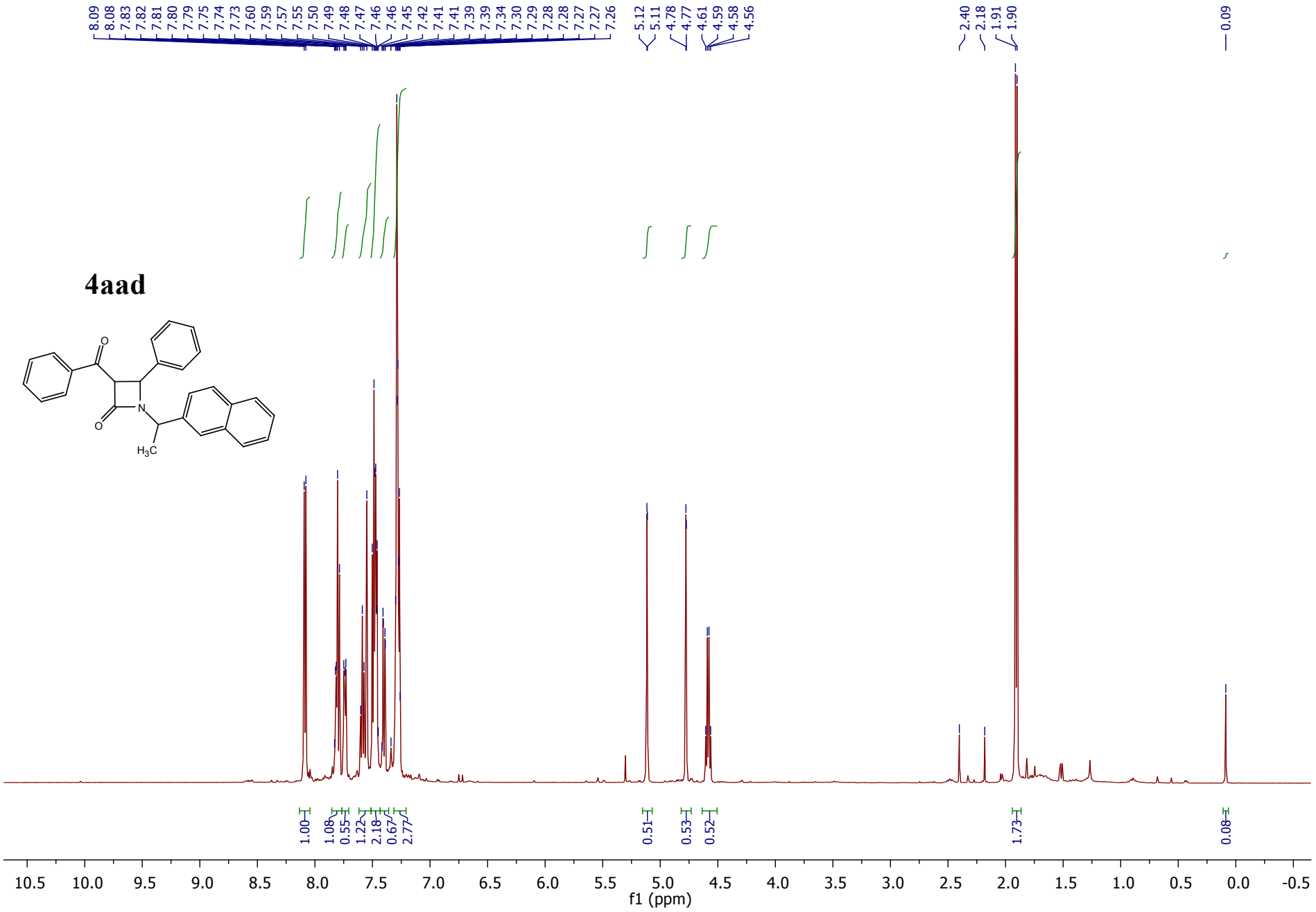
-0.5
f1 (ppm)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7.59
7.57
7.55
7.50
7.49
7.48
7.47
7.46
7.46
7.45
7.42
7.41
7.41
7.39
7.39
7.34
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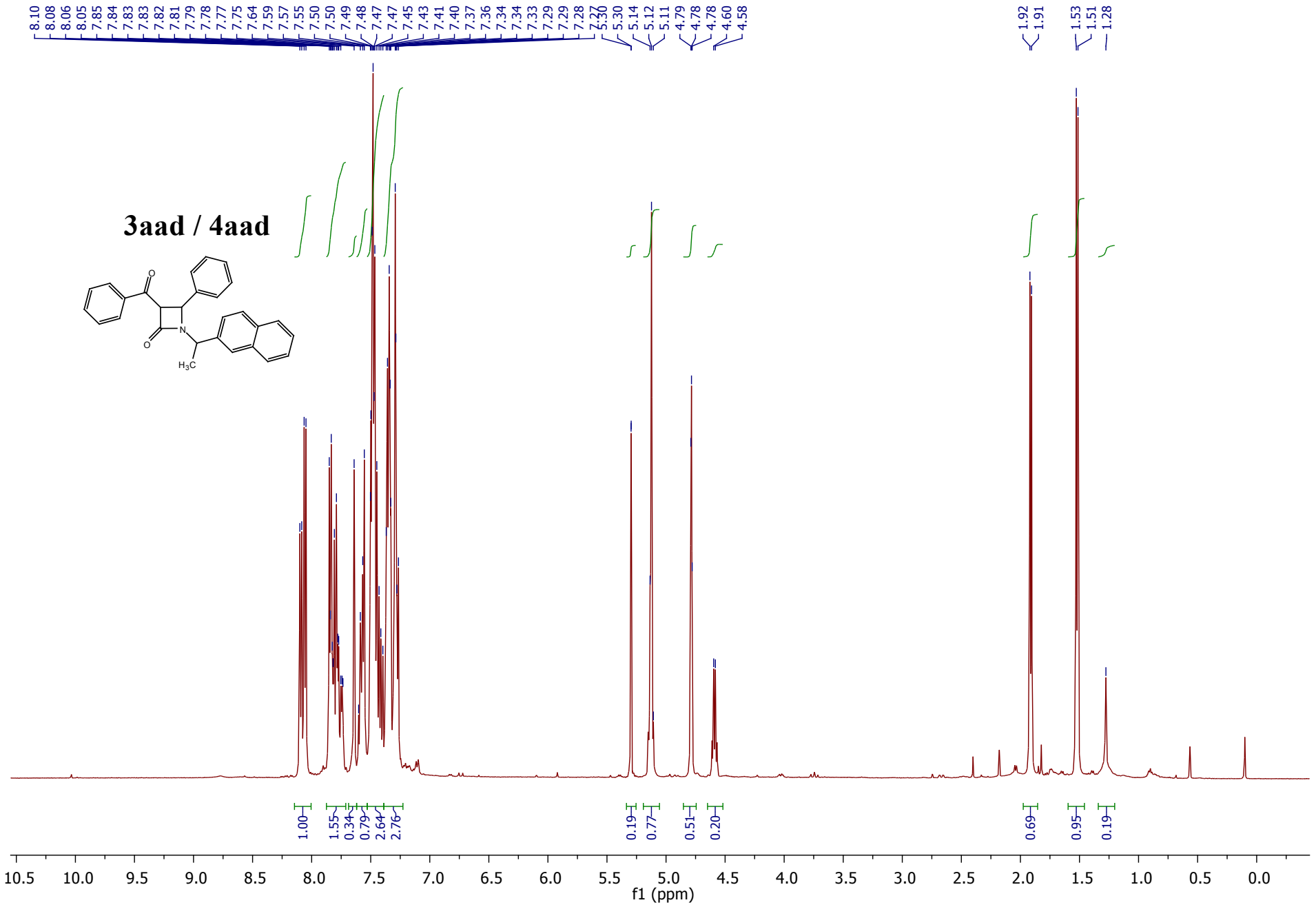
7.28
7.28
7.27
7.27
7.26
5.12
5.11
4.78
4.77
4.61
4.59
4.58
4.56
2.40
2.18
1.91
1.90
0.09
4aad
O
O
N
H3C
1.00
1.08
0.55
1.22
2.18
0.67
2.77
0.51
0.53
0.52
1.73
0.08
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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8.05
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7.83
7.82
7.81
7.79
7.78
7.77
7.75
7.64
7.59
7.57
7.55
7.50
7.50
7.49
7.48
7.47
7.47
7.45
7.43
7.41
7.40
7.37
7.36
7.34
7.34
7.33
7.29
7.29
7.28
5.30
5.30
5.14
5.12
5.11
4.79
4.78
4.78
4.60
4.58
1.92
1.91
1.53
1.51
1.28
3aad / 4aad
O
O
N
H3C
1.00
1.55
0.34
0.79
2.64
2.76
0.19
0.77
0.51
0.20
0.69
0.95
0.19
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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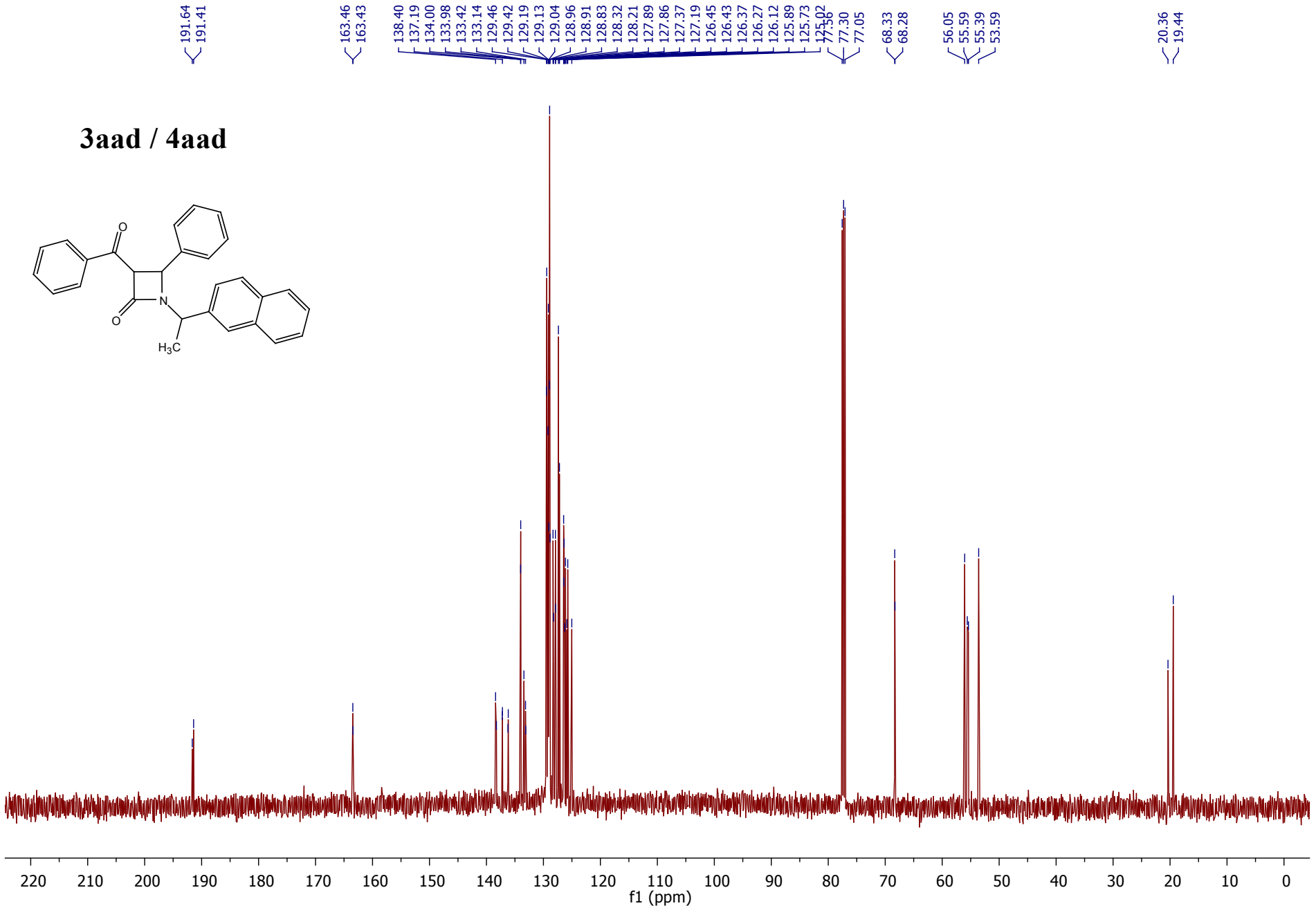
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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128.91
128.83
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128.21
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127.19
126.45
126.43
126.37
126.27
126.12
125.89
125.73
125.02
77.56
77.30
77.05
68.33
68.28
56.05
55.59
55.39
53.59
20.36
19.44
3aad / 4aad
O
O
N
H3C
220
210
200
190
180
170
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150
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130
120
110
100
90
80
70
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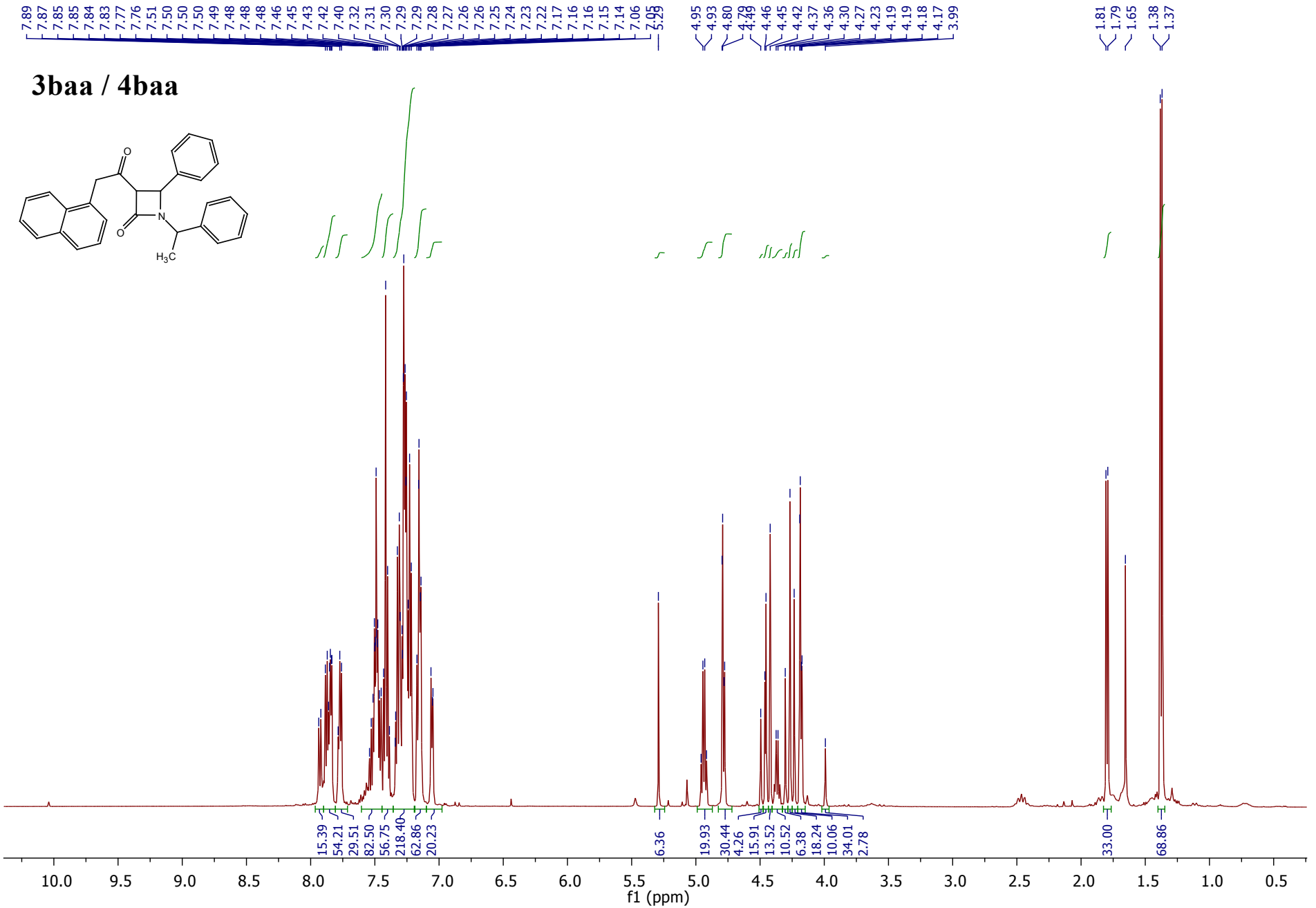
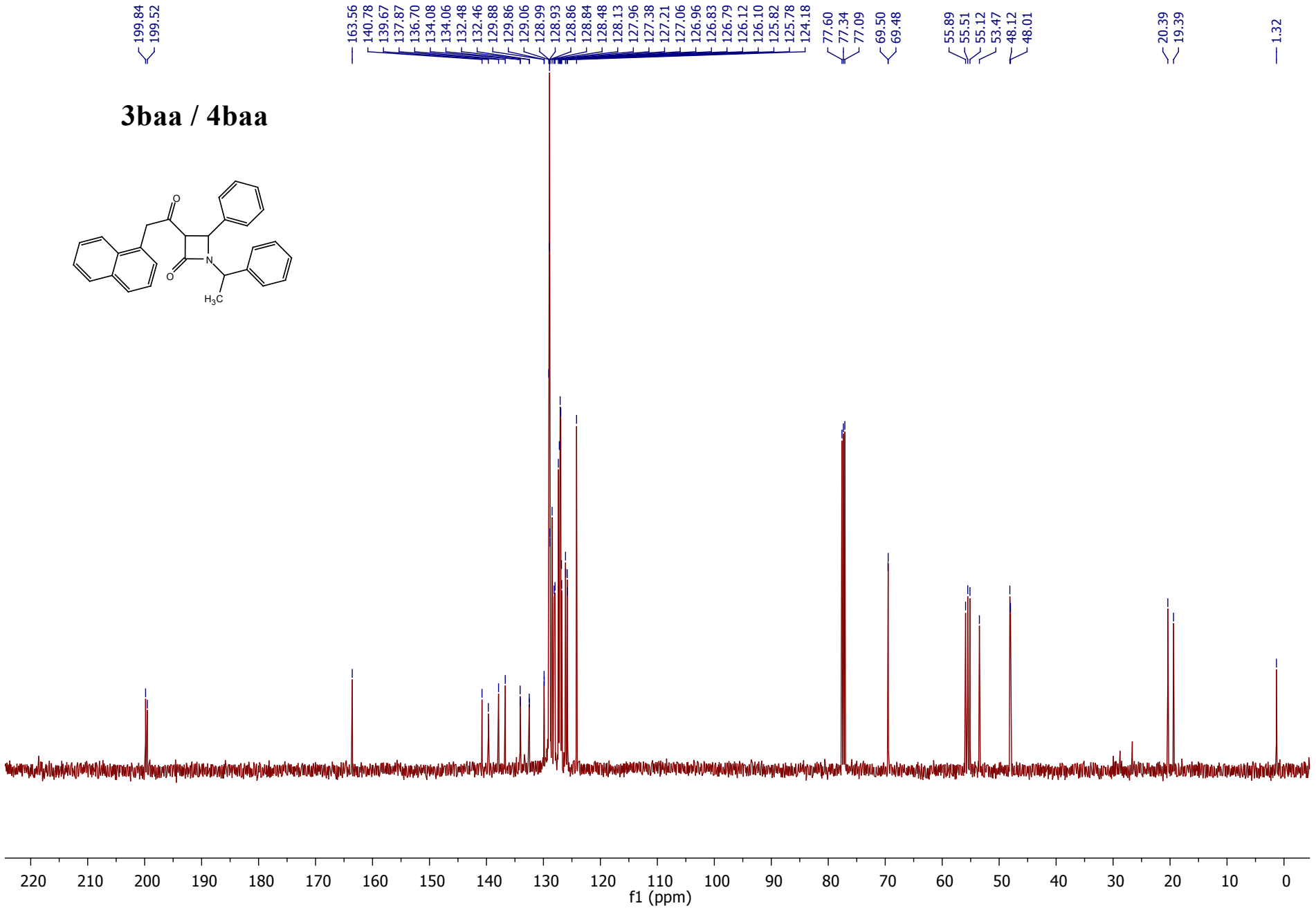
50
40
30
20
10
0
f1 (ppm)


7.89 7.87 7.85 7.85 7.84 7.83 7.77 7.76 7.51 7.50 7.50 7.50 7.49 7.48 7.48 7.48 7.46 7.45 7.43 7.42 7.40 7.32 7.31 7.30 7.29 7.29 7.28 7.27 7.26 7.26 7.25 7.24 7.23 7.22 7.17 7.16 7.16 7.15 7.14 7.06 7.05 5.29
4.95 4.93 4.80 4.79 4.46 4.45 4.42 4.37 4.36 4.30 4.27 4.23 4.19 4.19 4.18 4.17 3.99
1.81 1.79 1.65 1.38 1.37
3baa / 4baa
O
O
N
H3C
15.39 54.21 29.51 82.50 56.75 218.40 62.86 20.23 6.36 19.93 30.44 4.26 15.91 13.52 10.52 6.38 18.24 10.06 34.01 2.78 33.00 68.86
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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132.48
132.46
129.88
129.86
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128.99
128.93
128.86
128.84
128.48
128.13
127.96
127.38
127.21
127.06
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126.79
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125.82
125.78
124.18
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77.34
77.09
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69.48
55.89
55.51
55.12
53.47
48.12
48.01
20.39
19.39
1.32
3baa / 4baa
O
O
N
H3C
220
210
200
190
180
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110
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90
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70
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50
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30
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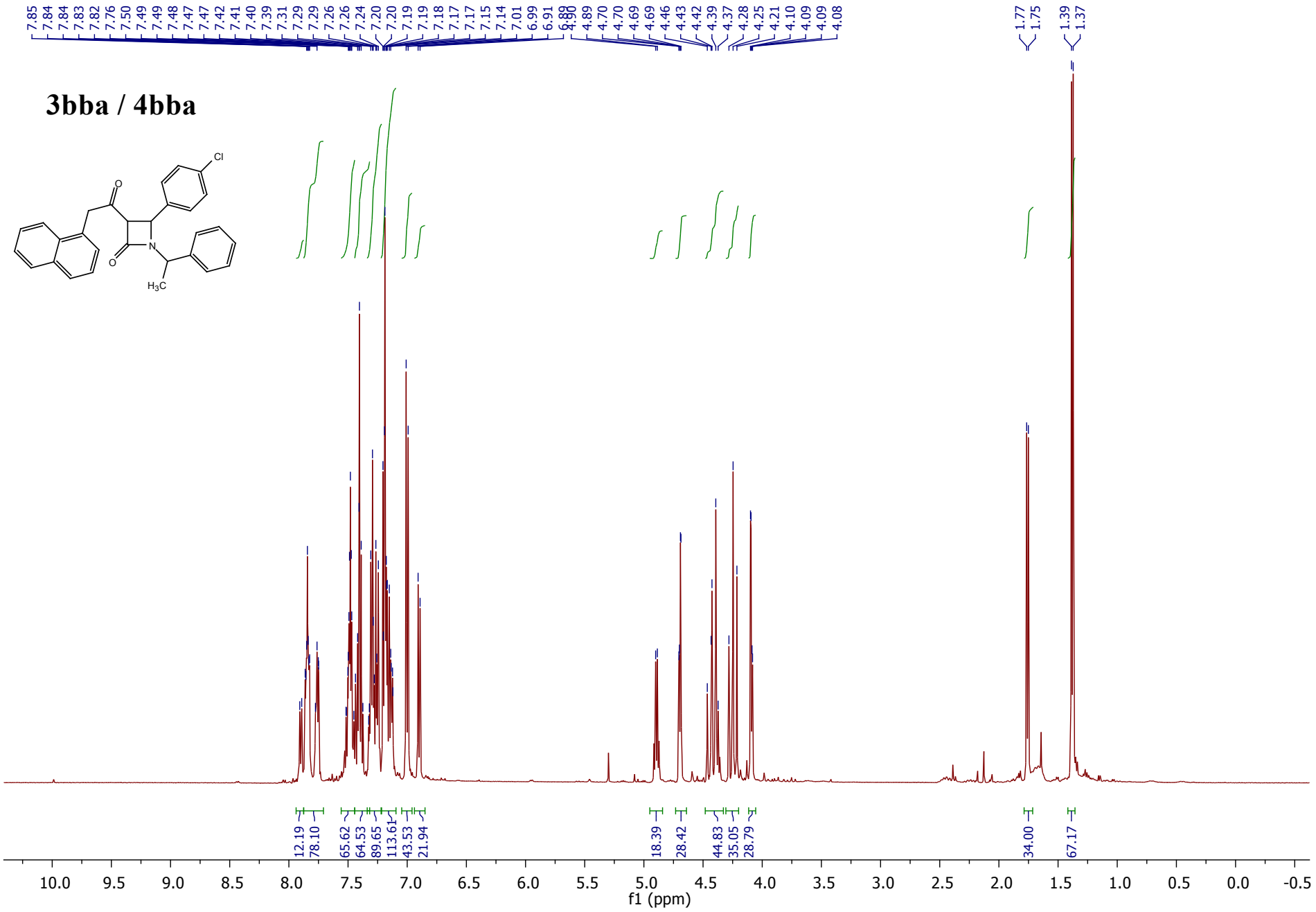
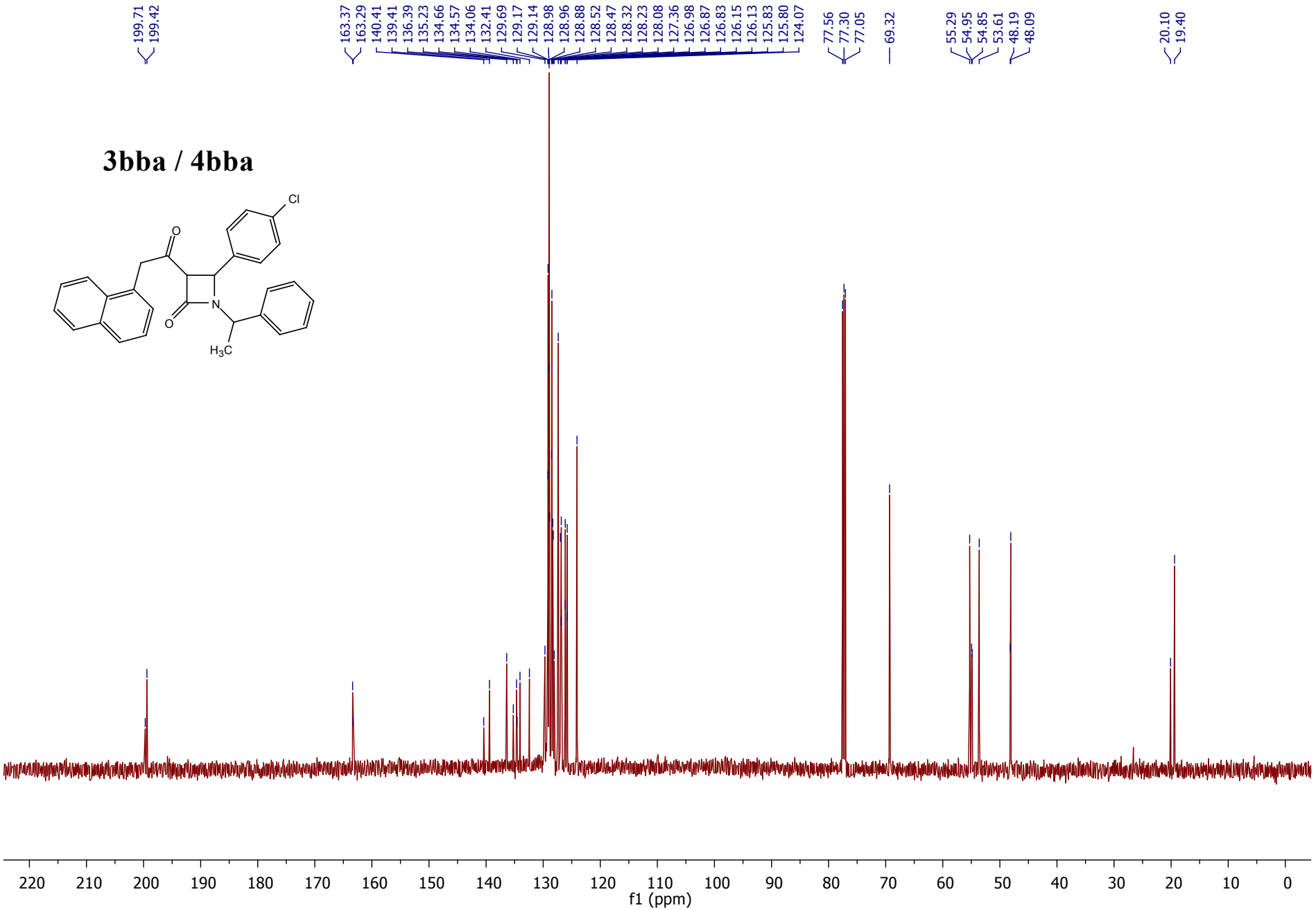
10
0
f1 (ppm)


7.85 7.84 7.84 7.83 7.82 7.76 7.50 7.49 7.49 7.48 7.47 7.47 7.42 7.41 7.40 7.39 7.31 7.29 7.29 7.26 7.26 7.24 7.20 7.20 7.19 7.19 7.18 7.17 7.17 7.15 7.14 7.01 6.99 6.91 4.90 4.89 4.70 4.70 4.69 4.69 4.46 4.43 4.42 4.39 4.37 4.28 4.25 4.21 4.10 4.09 4.09 4.08
1.77 1.75 1.39 1.37
3bba / 4bba
Cl
O
O
N
H3C
12.19 78.10 65.62 64.53 89.65 113.61 43.53 21.94 18.39 28.42 44.83 35.05 28.79 34.00 67.17
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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134.06
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129.14
128.98
128.96
128.88
128.52
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128.23
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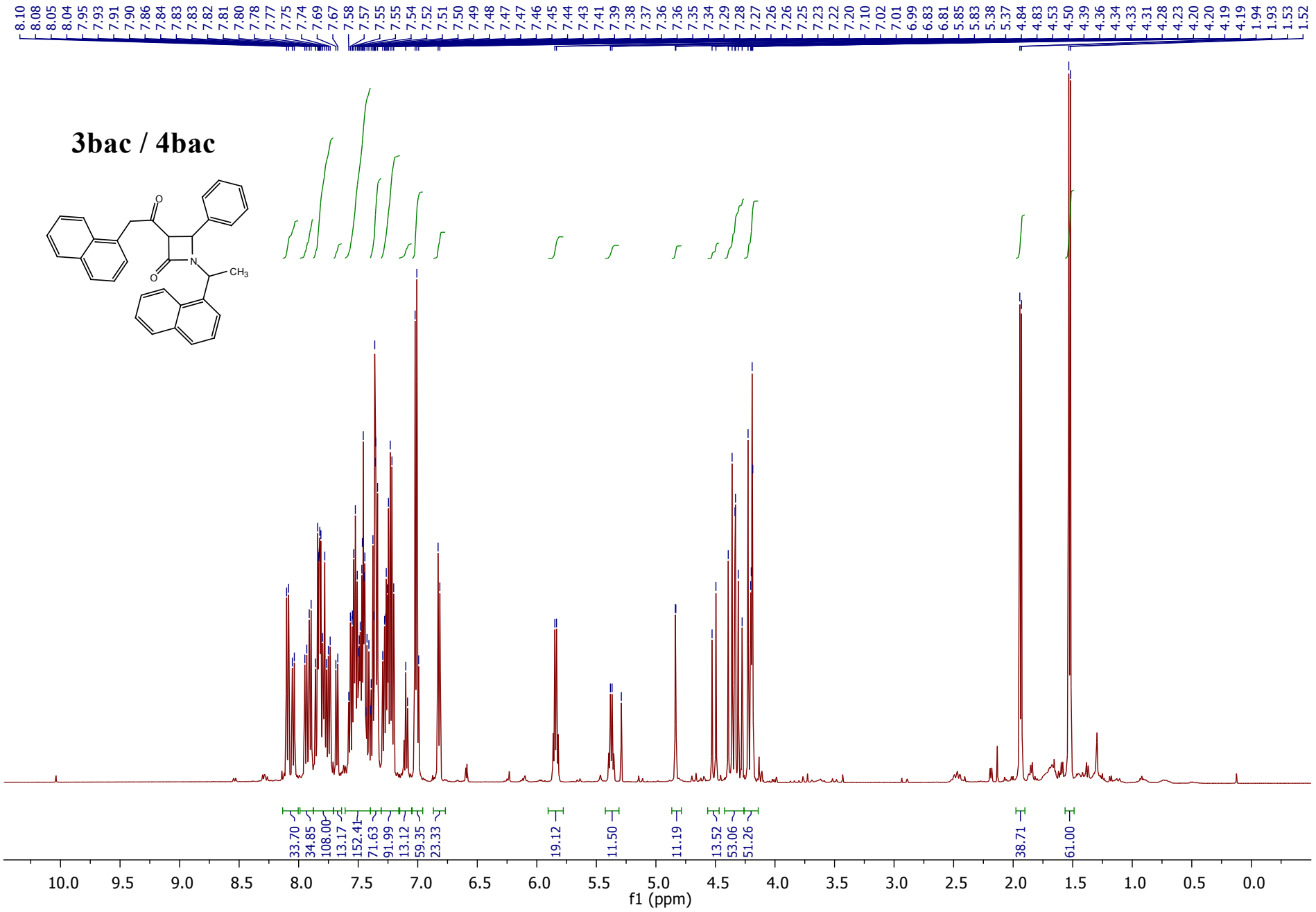
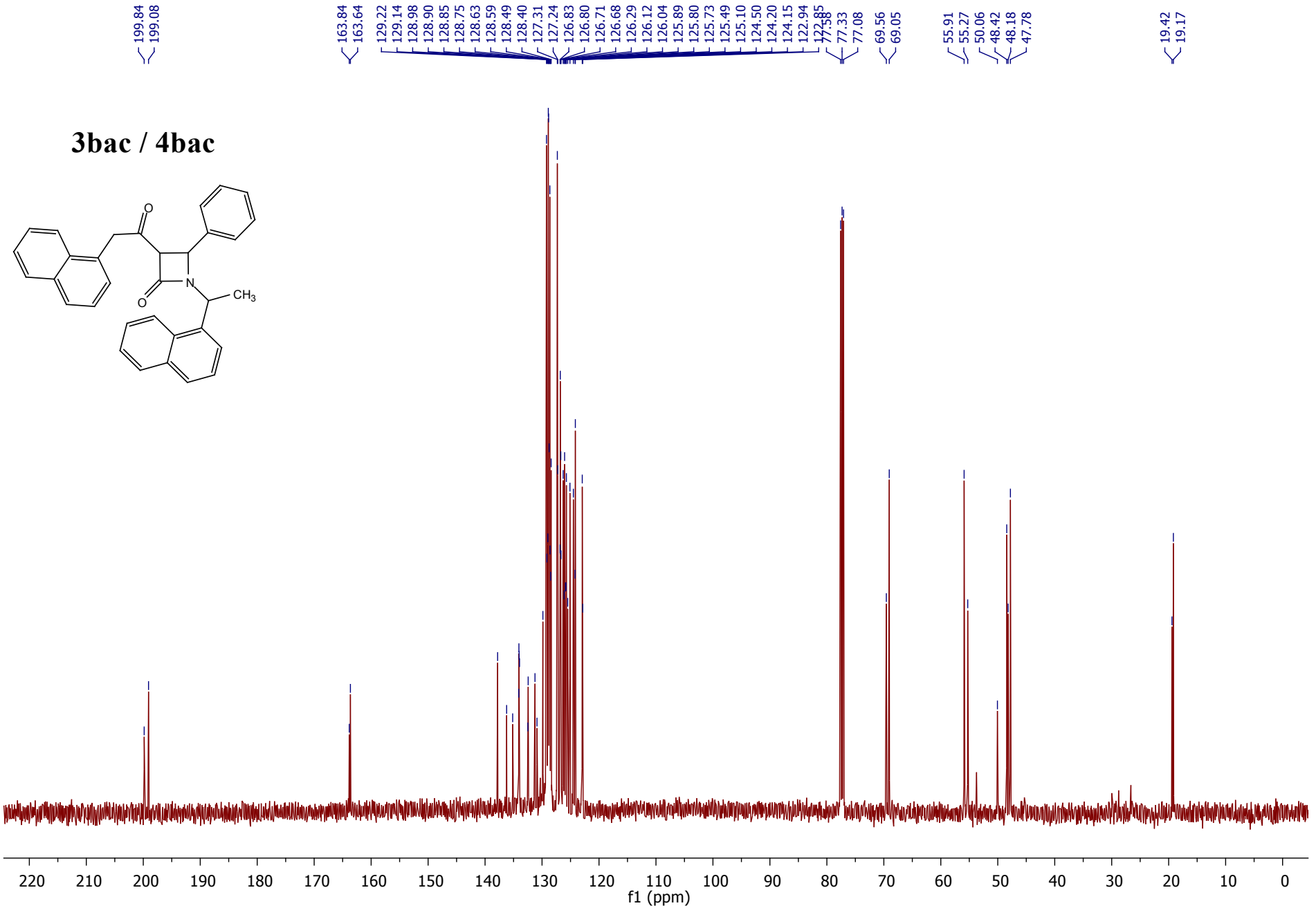
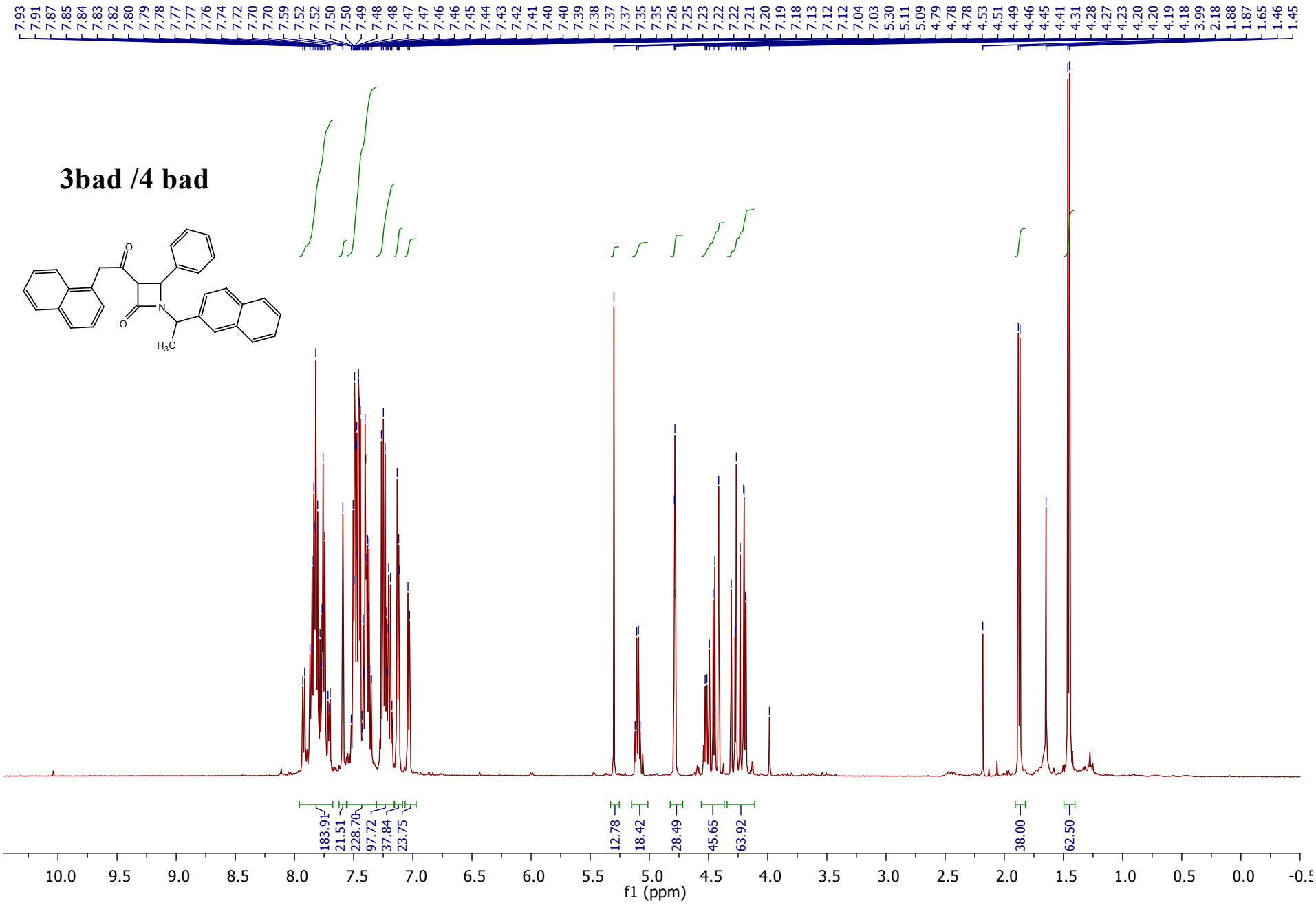
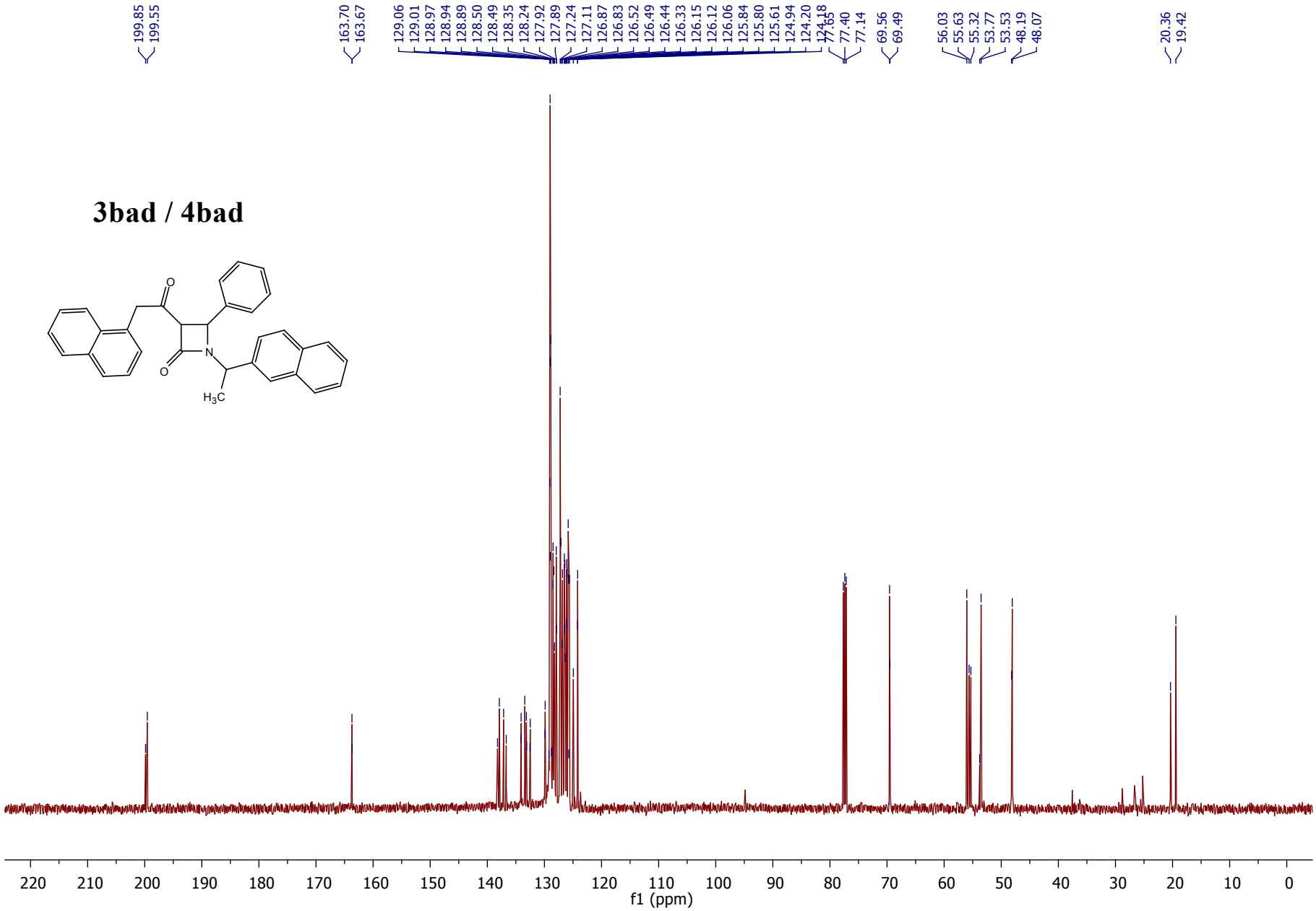
77.05
69.32
55.29
54.95
54.85
53.61
48.19
48.09
20.10
19.40
3bba / 4bba
Cl
O
O
N
H3C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


3bac / 4bac
O
O
N
CH3
8.10 8.08 8.05 8.04 7.95 7.93 7.91 7.90 7.86 7.84 7.83 7.83 7.82 7.81 7.80 7.78 7.77 7.75 7.74 7.69 7.67 7.58 7.57 7.55 7.55 7.54 7.52 7.51 7.50 7.49 7.48 7.47 7.47 7.46 7.45 7.44 7.43 7.41 7.39 7.38 7.37 7.36 7.36 7.35 7.34 7.29 7.28 7.27 7.26 7.26 7.25 7.23 7.22 7.20 7.10 7.02 7.01 6.99 6.83 6.81 5.85 5.83 5.38 5.37 4.84 4.83 4.53 4.50 4.39 4.36 4.34 4.33 4.31 4.28 4.23 4.20 4.20 4.19 4.19 1.94 1.93 1.53 1.52
33.70 34.85 108.00 13.17 152.41 71.63 91.99 13.12 59.35 23.33 19.12 11.50 11.19 13.52 53.06 51.26 38.71 61.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


199.84
199.08
163.84
163.64
129.22
129.14
128.98
128.90
128.85
128.75
128.63
128.59
128.49
128.40
127.31
127.24
126.83
126.80
126.71
126.68
126.29
126.12
126.04
125.89
125.80
125.73
125.49
125.10
124.50
124.20
124.15
122.94
77.58
77.33
77.08
69.56
69.05
55.91
55.27
50.06
48.42
48.18
47.78
19.42
19.17
3bac / 4bac
O
O
N
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


3bad /4 bad
O
O
N
H3C
7.93 7.91 7.87 7.85 7.84 7.83 7.82 7.80 7.79 7.78 7.77 7.77 7.76 7.74 7.72 7.70 7.70 7.59 7.52 7.52 7.50 7.50 7.49 7.48 7.48 7.47 7.47 7.46 7.46 7.45 7.44 7.43 7.42 7.41 7.40 7.40 7.39 7.38 7.37 7.37 7.35 7.35 7.26 7.25 7.23 7.22 7.22 7.21 7.20 7.19 7.18 7.13 7.12 7.12 7.04 7.03 5.30 5.11 5.09 4.79 4.78 4.53 4.51 4.49 4.46 4.45 4.41 4.31 4.28 4.27 4.23 4.20 4.20 4.19 4.18 3.99 2.18 1.88 1.87 1.65 1.46 1.45
183.91
21.51
228.70
97.72
37.84
23.75
12.78
18.42
28.49
45.65
63.92
38.00
62.50
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


199.85
199.55
163.70
163.67
129.06
129.01
128.97
128.94
128.89
128.50
128.49
128.35
128.24
127.92
127.89
127.24
127.11
126.87
126.83
126.52
126.49
126.44
126.33
126.15
126.12
126.06
125.84
125.80
125.61
124.94
124.20
124.18
77.65
77.40
77.14
69.56
69.49
56.03
55.63
55.32
53.77
53.53
48.19
48.07
20.36
19.42
3bad / 4bad
O
O
N
H3C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

References

[1] Raillard, S. P.; Chen, W.; Sullivan, E.; Bajjalieh, W.; Bhandari, A.; Baer, T. A. *J. Comb. Chem.* **2002**, 4, 470.

[2] Emtenäs, H.; Alderin, L.; Almqvist, F.; *J. Org. Chem.* **2001**, 66, 6756.

[3] Boyer, N.; Gloanec, P.; Nanteuil, G.; Jubaulta, P.; Quiriona, J-Ch. *Tetrahedron* 2007, 63, 12352.