A Copper(I)-Catalyzed Reaction of 2-(2-Ethynylphenyl)oxirane, Sulfonyl Azide, with 2-Isocyanoacetate

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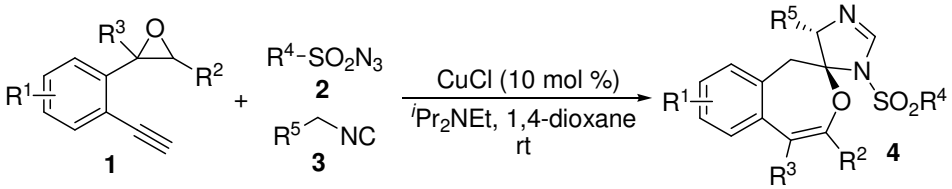
Supporting Information

General experimental methods:

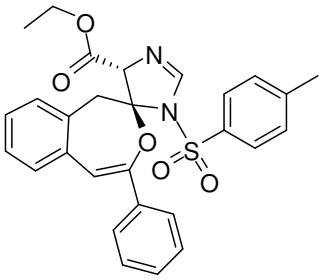
Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 25–35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument. The starting material 2-(2-ethynylphenyl)oxiranes **1** were synthesized according to the reference (R. J. Madhushaw, M.-Y. Lin, S. M. A. Sohel, R.-S. Liu, *J. Am. Chem. Soc.* **2004**, *126*, 6895.) and the detailed characterizations of the starting materials are available from the above reference.

*General experimental procedure for the copper-catalyzed reaction of 2-(2-ethynylphenyl)oxirane **1**, sulfonyl azide **2**, and 2-isocyanoacetate **3**.*

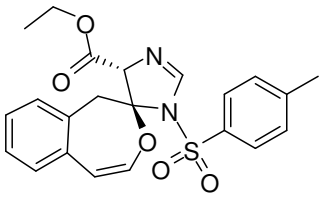


Diisopropylethylamine (58 mg, 0.45 mmol) was added to a solution of 2-(2-ethynylphenyl)oxirane **1** (0.3 mmol), sulfonyl azide **2** (0.36 mmol), and copper(I) chloride (3 mg, 0.03 mmol) in 1,4-dioxane (1.5 mL). The resulting mixture was stirred at room temperature for 2 hours under N_2 atmosphere, then 2-isocyanoacetate **3** (0.45 mmol) was added to the reaction mixture through a syringe. After completion

of the reaction as indicated by TLC, the mixture was diluted with CH_2Cl_2 (10 mL), and filtered through a thin layer of silica gel. The solvent was evaporated and the residue was purified by column chromatography on silica gel (eluted with PE/EA = 4:1) to provide the pure products **4**.

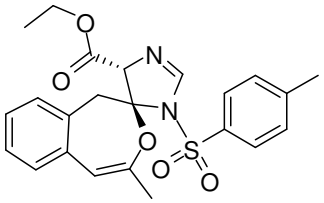


Ethyl 4-phenyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4a**). 8h. White solid; melting point: 168.1–169.0 °C. Yield: 63%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.88 (t, *J* = 6.8 Hz, 3H), 2.22 (s, 3H), 3.41-3.47 (m, 1H), 3.77-3.81 (m, 1H), 3.92 (d, *J* = 16.0 Hz, 1H), 4.08 (d, *J* = 16.0 Hz, 1H), 4.58 (s, 1H), 6.15 (s, 1H), 6.96-7.03 (m, 5H), 7.08-7.12 (m, 3H), 7.16-7.22 (m, 3H), 7.71 (d, *J* = 7.2 Hz, 2H), 7.86 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 21.6, 43.0, 62.0, 98.3, 105.3, 126.1, 126.2, 127.5, 127.8, 128.4, 129.8, 130.4, 131.5, 131.6, 134.0, 136.5, 137.5, 144.8, 146.3, 152.0, 167.3. HRMS (ESI) calcd for $C_{28}H_{26}N_2O_5S$: 503.1635 ($M+H^+$), found: 503.1643.



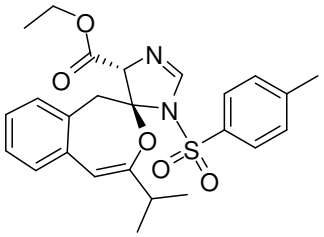
Ethyl 3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4b**). 6h. White solid; melting point: 155.8–156.8 °C. Yield: 50%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.87 (t, *J* = 6.8 Hz, 3H), 2.44 (s, 3H), 3.40-3.42 (m, 1H), 3.73-3.81 (m, 3H), 4.37 (s, 1H), 5.52 (d, *J* = 8.0 Hz, 1H), 5.83 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 7.2 Hz, 1H), 7.04-7.09 (m, 2H), 7.16-7.20 (m, 1H), 7.34 (d, *J* = 7.2 Hz, 2H), 7.70 (s, 1H), 7.81 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 21.7, 42.2, 62.0, 77.6, 98.7, 106.7, 126.1, 127.4, 127.6, 129.1, 129.8, 131.5, 132.2, 133.9, 137.4, 137.5,

145.0, 151.6, 167.4. HRMS (ESI) calcd for $C_{22}H_{22}N_2O_5S$: 427.1322 ($M+H^+$), found: 427.1327.



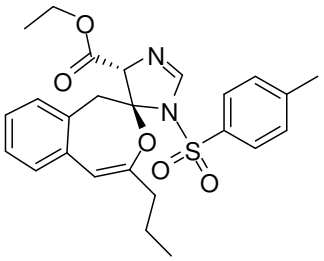
Ethyl

4-methyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxyl ate (**4c**). 6h. White solid; melting point: 183.0–184.0 °C. Yield: 70%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.69 (t, *J* = 7.2 Hz, 3H), 1.49 (s, 3H), 2.45 (s, 3H), 3.41-3.43 (m, 1H), 3.75-3.81 (m, 3H), 4.42 (s, 1H), 5.48 (s, 1H), 6.92 (d, *J* = 6.8 Hz, 1H), 6.99-7.03 (m, 2H), 7.14-7.17 (m, 1H), 7.35 (d, *J* = 7.6 Hz, 2H), 7.76 (s, 1H), 7.83 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 21.7, 22.3, 42.3, 61.9, 77.5, 98.0, 103.8, 125.3, 127.3, 127.6, 128.9, 129.8, 131.4, 131.4, 134.4, 137.8, 144.8, 146.0, 151.7, 167.4. HRMS (ESI) calcd for $C_{23}H_{24}N_2O_5S$: 441.1479 ($M+H^+$), found: 441.1476.



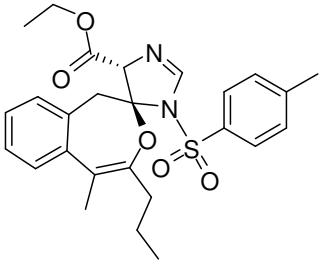
Ethyl

4-isopropyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carbo xylate (**4d**). 6h. White solid; melting point: 161.0–162.0 °C. Yield: 70%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.70 (d, *J* = 6.4 Hz, 3H), 0.85-0.88 (m, 6H), 1.94-1.97 (m, 1H), 2.43 (s, 3H), 3.36-3.40 (m, 1H), 3.66-3.83 (m, 3H), 4.43 (s, 1H), 5.48 (s, 1H), 6.87 (d, *J* = 7.2 Hz, 1H), 6.97-7.03 (m, 2H), 7.12-7.15 (m, 1H), 7.34 (d, *J* = 7.2 Hz, 2H), 7.79 (s, 1H), 7.82 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 20.5, 21.1, 21.6, 34.7, 42.3, 61.9, 76.9, 97.4, 101.4, 125.4, 127.3, 127.4, 129.5, 129.9, 131.3, 134.3, 137.8, 144.8, 151.7, 154.4, 167.5. HRMS (ESI) calcd for $C_{25}H_{28}N_2O_5S$: 469.1792 ($M+H^+$), found: 469.1788.



Ethyl

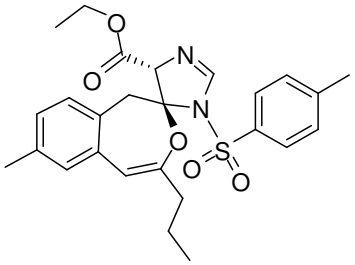
4-propyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxyl ate (**4e**). 6h. White solid; melting point: 143.0–144.0 °C. Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.70 (t, *J* = 6.8 Hz, 3H), 0.89 (t, *J* = 6.8 Hz, 3H), 0.98-1.03 (m, 2H), 1.61-1.79 (m, 2H), 2.45 (s, 3H), 3.40-3.42 (m, 1H), 3.75-3.86 (m, 3H), 4.42 (s, 1H), 5.48 (s, 1H), 6.92 (d, *J* = 7.2 Hz, 1H), 7.00-7.02 (m, 2H), 7.13-7.17 (m, 1H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.78 (s, 1H), 7.82 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 13.5, 20.9, 21.6, 38.5, 42.5, 61.9, 77.2, 97.7, 103.6, 125.4, 127.3, 127.5, 129.2, 129.8, 131.4, 131.5, 134.3, 138.0, 144.7, 149.3, 151.8, 167.4. HRMS (ESI) calcd for $C_{25}H_{28}N_2O_5S$: 469.1792 ($M+H^+$), found: 469.1799.



Ethyl

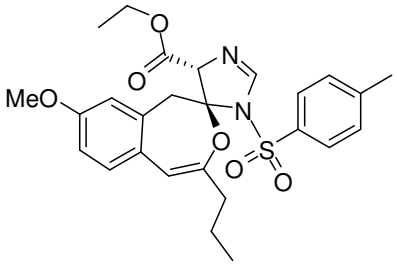
5-methyl-4-propyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5' -carboxylate (**4f**). 6h. White solid; melting point: 158.1–159.0 °C. Yield: 60%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90 (t, *J* = 7.2 Hz, 3H), 1.13 (t, *J* = 7.2 Hz, 3H), 1.45-1.52 (m, 2H), 2.07 (s, 3H), 2.31-2.36 (m, 1H), 2.44 (s, 3H), 2.52-2.57 (m, 1H), 2.84 (d, *J* = 14.0 Hz, 1H), 3.81 (d, *J* = 14.0 Hz, 1H), 3.99-4.09 (m, 2H), 4.47 (s, 1H), 6.82 (d, *J* = 7.2 Hz, 1H), 7.04-7.05 (m, 1H), 7.27-7.32 (m, 4H), 7.72-7.76 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 13.9, 17.8, 20.9, 21.7, 34.7, 35.5, 61.6, 80.6, 107.0, 117.3, 125.7, 126.6, 127.0, 127.5, 129.2, 130.0, 132.7, 137.4, 140.9, 144.7, 150.6, 168.4. HRMS (ESI) calcd for $C_{26}H_{30}N_2O_5S$: 483.1948 ($M+H^+$), found:

483.1956.



Ethyl

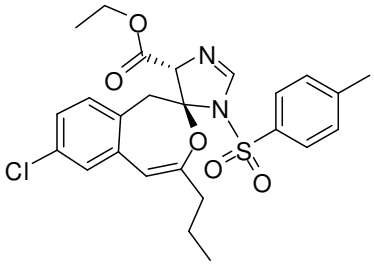
7-methyl-4-propyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4g**). 5h. White solid; melting point: 154.4–155.2 °C. Yield: 75%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.69 (t, *J* = 6.8 Hz, 3H), 0.90 (t, *J* = 6.8 Hz, 3H), 0.98-1.03 (m, 2H), 1.60-1.77 (m, 2H), 2.26 (s, 3H), 2.45 (s, 3H), 3.44-3.48 (m, 1H), 3.71-3.77 (m, 3H), 4.41 (s, 1H), 5.43 (s, 1H), 6.81-6.83 (m, 3H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.78 (s, 1H), 7.81 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.3, 13.5, 20.9, 20.9, 21.6, 38.6, 42.1, 62.0, 77.2, 97.8, 103.6, 126.2, 127.5, 128.6, 129.8, 131.3, 134.1, 136.8, 138.0, 144.7, 149.2, 151.8, 167.4. HRMS (ESI) calcd for $C_{26}H_{30}N_2O_5S$: 483.1948 ($M+H^+$), found: 483.1959.



Ethyl

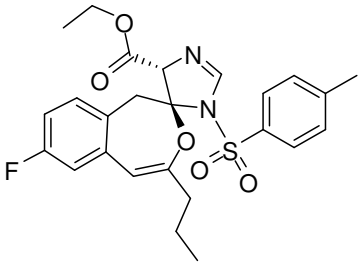
8-methoxy-4-propyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4h**). 4h. White solid; melting point: 108.3–109.2 °C. Yield: 81%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.68 (t, *J* = 7.2 Hz, 3H), 0.91-0.99 (m, 5H), 1.57-1.74 (m, 2H), 2.45 (s, 3H), 3.46-3.49 (m, 1H), 3.70-3.84 (m, 6H), 4.45 (s, 1H), 5.41 (s, 1H), 6.50 (s, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.77 (s, 1H), 7.81 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 13.5, 20.8, 21.6, 38.4, 42.9, 55.3, 61.8, 77.0, 97.5, 103.1, 113.1, 116.5, 127.2, 127.5, 129.7, 130.5, 132.9, 138.0, 144.7, 147.3, 151.8, 157.4, 167.6. HRMS (ESI) calcd for

$C_{26}H_{30}N_2O_6S$: 499.1897 ($M+H^+$), found: 499.1906.



Ethyl

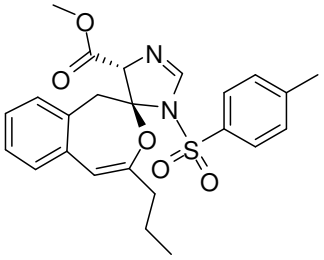
7-chloro-4-propyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4i**). 8h. White solid; melting point: 188.0–189.0 °C. Yield: 61%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.70 (t, *J* = 6.8 Hz, 3H), 0.95 (t, *J* = 6.8 Hz, 3H), 0.99-1.05 (m, 2H), 1.61-1.79 (m, 2H), 2.46 (s, 3H), 3.46-3.49 (m, 1H), 3.76-3.81 (m, 3H), 4.39 (s, 1H), 5.40 (s, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.98-7.01 (m, 2H), 7.35 (d, *J* = 7.6 Hz, 2H), 7.78 (s, 1H), 7.81 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 13.5, 20.8, 21.6, 38.6, 42.0, 62.2, 77.2, 97.7, 102.4, 125.1, 127.4, 128.6, 129.8, 129.9, 132.7, 133.1, 136.1, 137.8, 144.8, 150.9, 151.8, 167.3. HRMS (ESI) calcd for $C_{25}H_{27}ClN_2O_5S$: 503.1402 ($M+H^+$), found: 503.1402.



Ethyl

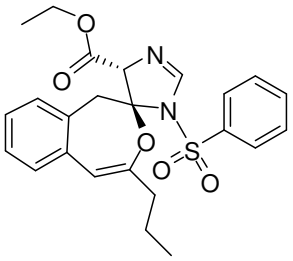
7-fluoro-4-propyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4j**). 8h. White solid; melting point: 180.0–181.0 °C. Yield: 62%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.70 (t, *J* = 7.2 Hz, 3H), 0.95 (t, *J* = 6.8 Hz, 3H), 1.00-1.05 (m, 2H), 1.61-1.79 (m, 2H), 2.45 (s, 3H), 3.47-3.52 (m, 1H), 3.77-3.83 (m, 3H), 4.39 (s, 1H), 5.41 (s, 1H), 6.71 (d, *J* = 8.0 Hz, 2H), 6.86-6.90 (m, 1H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.78-7.82 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 13.5, 20.8, 21.6, 38.6, 41.7, 62.1, 77.3, 97.8, 102.6, 112.1 (d, $^2J_{CF}$ = 21.0 Hz), 115.0 (d, $^2J_{CF}$ = 21.9 Hz), 127.3 (d, J_{CF} = 1.9 Hz), 127.4, 129.8, 133.0 (d, $^3J_{CF}$ = 8.6 Hz), 136.4 (d, $^3J_{CF}$

= 7.7 Hz), 137.9, 144.8, 150.7, 151.8, 162.1 (d, $^{1}J_{CF}$ = 244.1 Hz), 167.3 (d, J_{CF} = 4.7 Hz). HRMS (ESI) calcd for $C_{25}H_{27}ClN_2O_5S$: 487.1697 ($M+H^+$), found: 487.1696.



Methyl

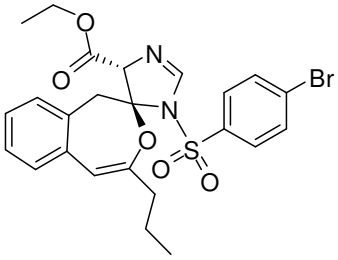
4-propyl-3'-tosyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxyl ate (**4k**). 6h. White solid; melting point: 150.5–151.5 °C. Yield: 75%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.69 (t, *J* = 7.2 Hz, 3H), 0.99-1.02 (m, 2H), 1.62-1.77 (m, 2H), 2.46 (s, 3H), 3.20 (s, 3H), 3.70-3.82 (m, 2H), 4.43 (s, 1H), 5.48 (s, 1H), 6.93 (d, *J* = 7.2 Hz, 1H), 7.02-7.06 (m, 2H), 7.14-7.17 (m, 1H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.79 (s, 1H), 7.82 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 20.9, 21.7, 38.5, 42.7, 52.7, 77.1, 97.6, 103.6, 125.4, 127.4, 127.5, 129.2, 129.8, 131.3, 131.4, 134.2, 137.9, 144.8, 149.3, 151.9, 167.7. HRMS (ESI) calcd for $C_{24}H_{26}N_2O_5S$: 455.1635 ($M+H^+$), found: 455.1627.



Ethyl

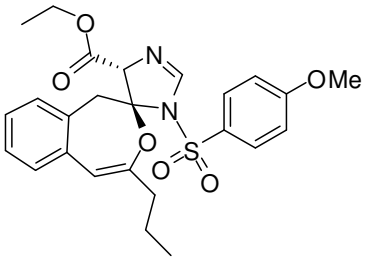
3'-(phenylsulfonyl)-4-propyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4l**). 6h. White solid; melting point: 135.0–136.0 °C. Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.68 (t, *J* = 6.8 Hz, 3H), 0.90 (t, *J* = 6.8 Hz, 3H), 0.97-1.01 (m, 2H), 1.55-1.77 (m, 2H), 3.40-3.44 (m, 1H), 3.69-3.87 (m, 3H), 4.43 (s, 1H), 5.48 (s, 1H), 6.93 (d, *J* = 7.2 Hz, 1H), 7.01 (d, *J* = 7.2 Hz, 2H), 7.14-7.18 (m, 1H), 7.55-7.58 (m, 2H), 7.64-7.66 (m, 1H), 7.81 (s, 1H), 7.95 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 13.5, 20.8, 38.4, 42.6, 62.0, 77.2, 97.7, 103.7,

125.4, 127.3, 127.4, 129.2, 131.4, 133.6, 134.3, 140.9, 149.2, 151.6, 167.3. HRMS (ESI) calcd for $C_{24}H_{26}N_2O_5S$: 455.1635 ($M+H^+$), found: 455.1633.



Ethyl

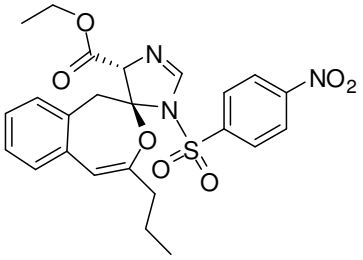
3'-(4-bromophenylsulfonyl)-4-propyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4m**). 6h. White solid; melting point: 150.1–152.0 °C. Yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.74 (t, *J* = 6.8 Hz, 3H), 0.89 (t, *J* = 6.8 Hz, 3H), 0.98-1.04 (m, 2H), 1.58-1.80 (m, 2H), 3.40-3.44 (m, 1H), 3.69-3.79 (m, 3H), 4.43 (s, 1H), 5.49 (s, 1H), 6.94 (d, *J* = 6.8 Hz, 1H), 7.01-7.05 (m, 2H), 7.15-7.18 (m, 1H), 7.70 (d, *J* = 7.2 Hz, 2H), 7.78-7.81 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 13.6, 20.9, 38.5, 42.6, 62.0, 77.2, 97.7, 103.8, 125.5, 127.4, 128.8, 128.9, 129.3, 131.3, 131.4, 132.5, 134.1, 139.9, 149.0, 151.4, 167.3. HRMS (ESI) calcd for $C_{24}H_{25}BrN_2O_5S$: 533.0740 ($M+H^+$), found: 533.0732.



Ethyl

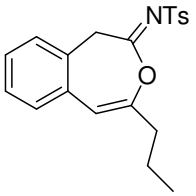
3'-(4-methoxyphenylsulfonyl)-4-propyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4n**). 6h. White solid; melting point: 128.0–129.0 °C. Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.71 (t, *J* = 6.8 Hz, 3H), 0.89 (t, *J* = 6.8 Hz, 3H), 1.01-1.08 (m, 2H), 1.70-1.82 (m, 2H), 3.40-3.42 (m, 1H), 3.75-3.82 (m, 3H), 3.88 (s, 3H), 4.42 (s, 1H), 5.49 (s, 1H), 6.92-7.02 (m, 5H), 7.14-7.18 (m, 1H), 7.77 (s, 1H), 7.87 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 13.5, 20.9, 38.7, 42.6, 55.8, 55.8, 61.9, 77.2, 97.7, 103.6, 114.3, 125.4, 127.3, 129.2, 129.7, 131.4, 131.5,

134.3, 149.3, 151.9, 163.8, 167.4. HRMS (ESI) calcd for $C_{25}H_{28}N_2O_6S$: 485.1741 ($M+H^+$), found: 485.1738.

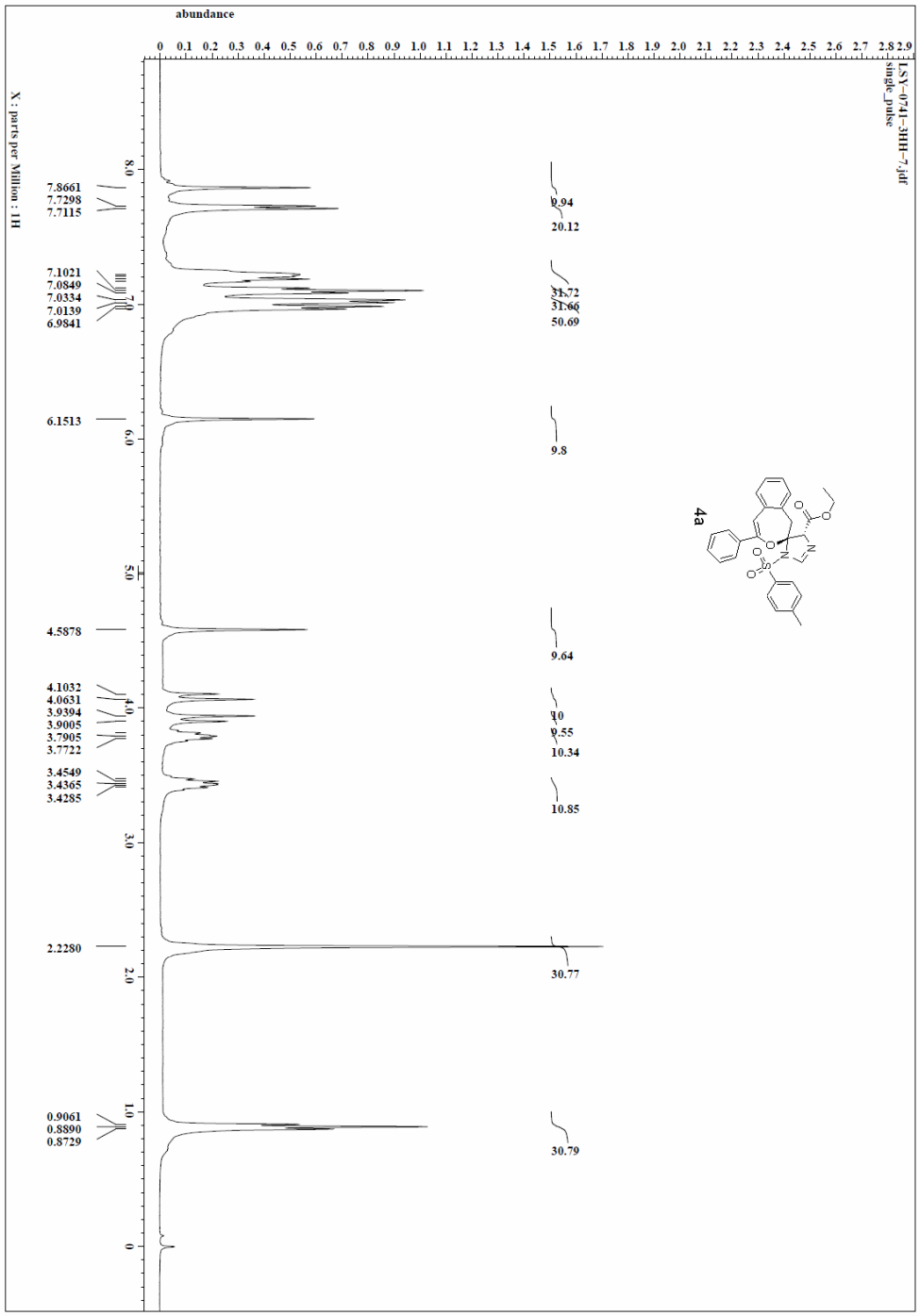


Ethyl

3'-(4-nitrophenylsulfonyl)-4-propyl-3',5'-dihydro-1*H*-spiro[benzo[*d*]oxepine-2,4'-imidazole]-5'-carboxylate (**4o**). 8h. Yellow solid; melting point: 169.3–170.3 °C. Yield: 40%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.67 (t, *J* = 7.2 Hz, 3H), 0.90 (t, *J* = 7.2 Hz, 3H), 0.95-1.02 (m, 2H), 1.55-1.77 (m, 2H), 3.40-3.44 (m, 1H), 3.74-3.81 (m, 3H), 4.46 (s, 1H), 5.51 (s, 1H), 6.93 (d, *J* = 7.2 Hz, 1H), 7.03-7.07 (m, 2H), 7.16-7.20 (m, 1H), 7.81 (s, 1H), 8.15 (d, *J* = 7.6 Hz, 2H), 8.41 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 20.8, 38.4, 42.5, 62.2, 67.1, 77.2, 97.9, 104.2, 124.4, 125.7, 127.5, 128.8, 129.4, 131.0, 131.4, 134.0, 146.3, 148.7, 150.5, 150.8, 167.1. HRMS (ESI) calcd for $C_{24}H_{25}N_3O_7S$: 500.1486 ($M+H^+$), found: 500.1482.



4-Methyl-*N*-((Z)-4-propylbenzo[*d*]oxepin-2(1*H*)-ylidene)benzenesulfonamide (**5a**). 2h. White solid; melting point: 158.0–159.0 °C. Yield: 64%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90 (s, 3H), 1.50 (m, 2H), 2.32-2.38 (m, 5H), 3.55 (s, 2H), 6.23 (s, 1H), 7.17-7.23 (m, 2H), 7.24 (d, *J* = 7.2 Hz, 2H), 7.25-7.31 (m, 2H), 7.79 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.5, 20.5, 21.6, 36.9, 40.7, 111.7, 127.3, 127.7, 128.3, 128.9, 129.1, 129.3, 130.1, 132.4, 138.4, 143.5, 152.8, 164.8. HRMS (ESI) calcd for $C_{20}H_{21}NO_3S$: 356.1315 ($M+H^+$), found: 356.1314.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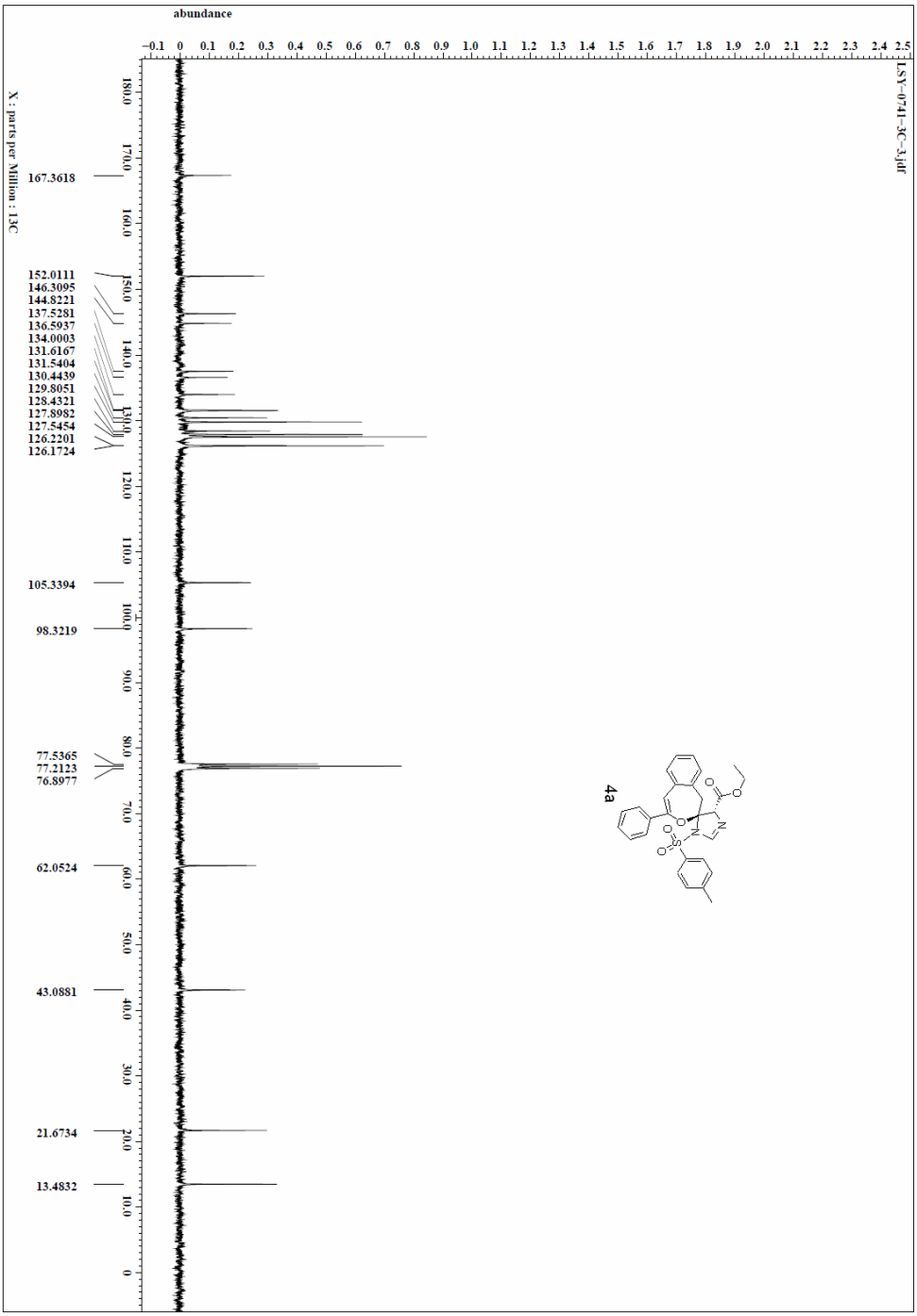
7.7115
7.1021
7.0849
7.0334
7.0139
6.9841
6.1513
4.5878
4.1032
4.0631
3.9394
3.9005
3.7905
3.7722
3.4549
3.4365
3.4285
2.2280
0.9061
0.8890
0.8729
9.94
20.12
31.72
31.60
50.69
9.8
9.64
10
9.55
10.34
10.85
30.77
30.79
4a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136.5937
134.0003
131.6167
131.5404
130.4439
129.8051
128.4321
127.8982
127.5454
126.2201
126.1724
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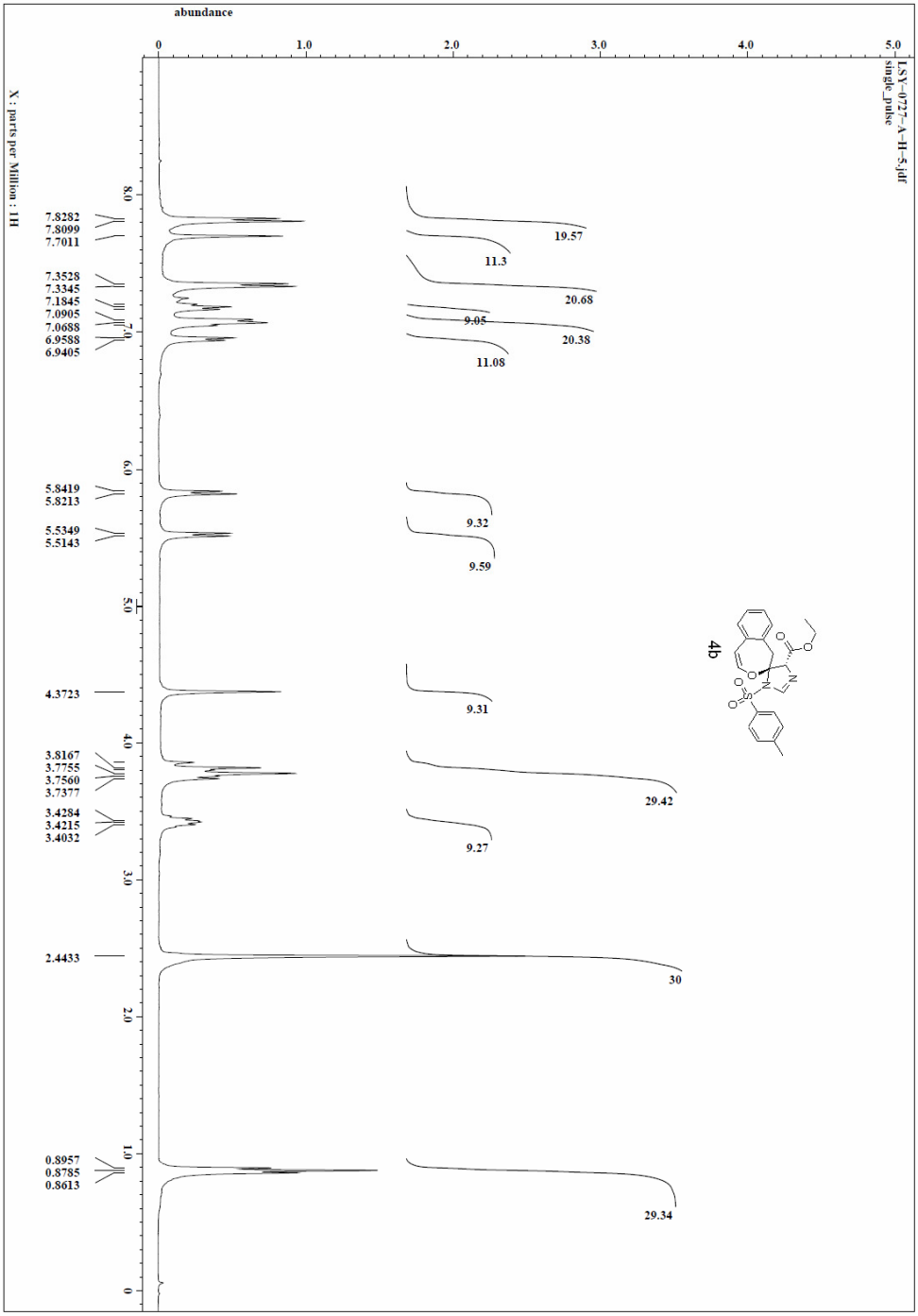
77.5365
77.2123
76.8977
62.0524
43.0881
21.6734
13.4832
4a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2.0
3.0
4.0
5.0
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single_pulse
X : parts per Million : 1H
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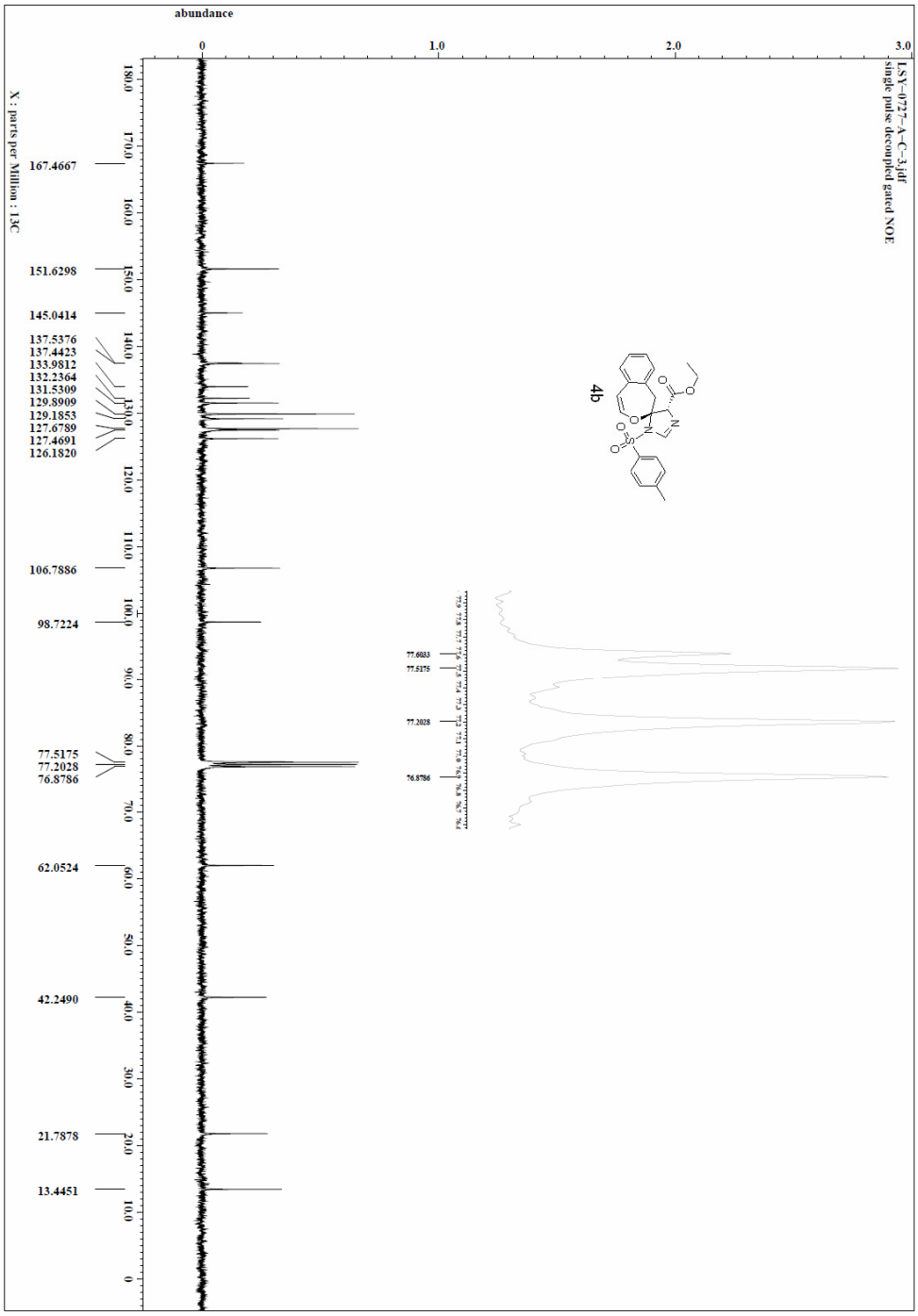
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.8282
7.8099
7.7011
7.3528
7.3345
7.1845
7.0905
7.0688
6.9588
6.9405
5.8419
5.8213
5.5349
5.5143
4.3723
3.8167
3.7755
3.7560
3.7377
3.4284
3.4215
3.4032
2.4433
0.8957
0.8785
0.8613
19.57
11.3
20.68
9.05
20.38
11.08
9.32
9.59
9.31
29.42
9.27
30
29.34
4b


abundance
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2.0
3.0
LSY-0727-A-C-3.jdf
single pulse decoupled gated NOE
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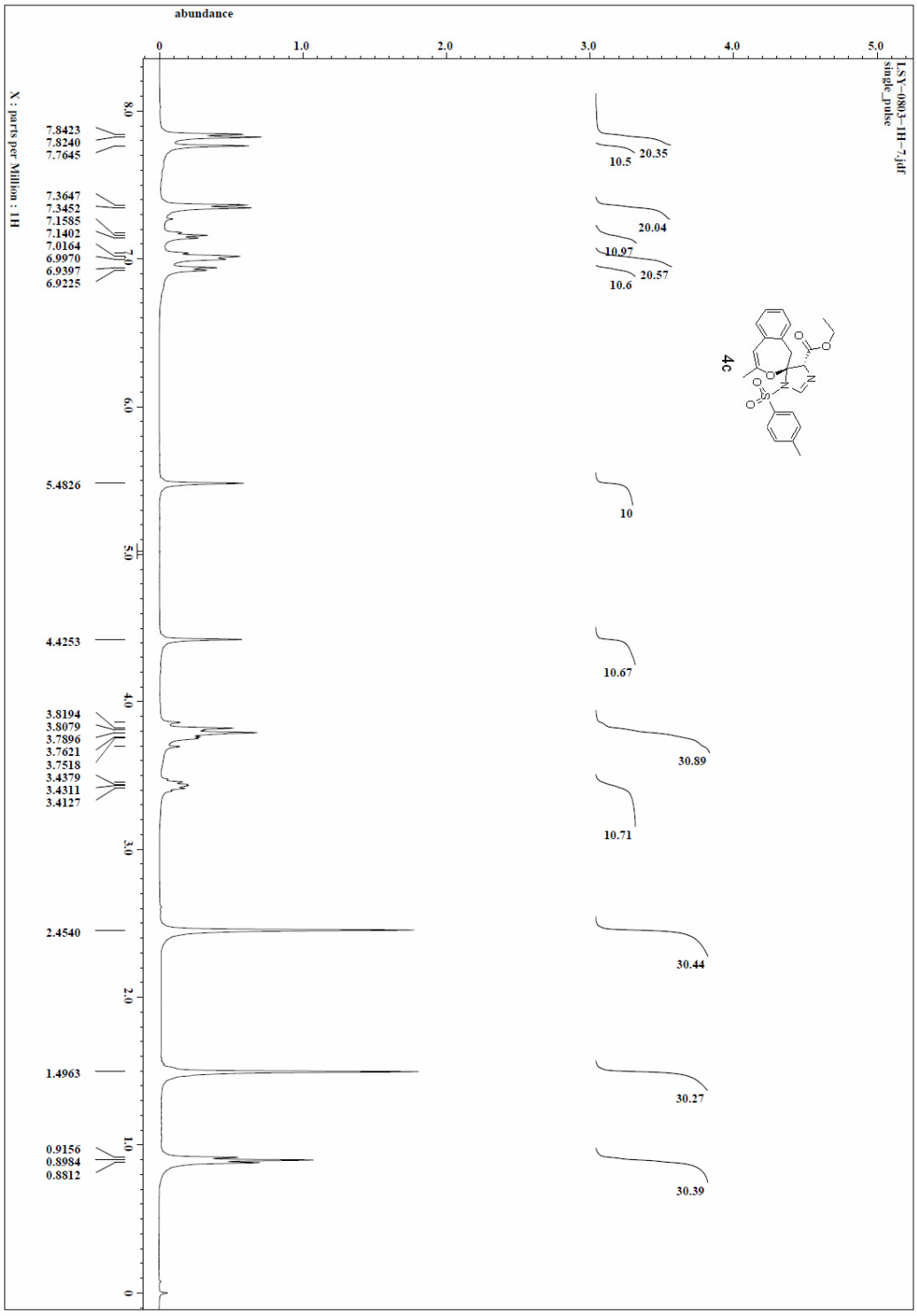
145.0414
137.5376
137.4423
133.9812
132.2364
131.5309
129.8909
129.1853
127.6789
127.4691
126.1820
106.7886
98.7224
77.5175
77.2028
76.8786
62.0524
42.2490
21.7878
13.4451
4b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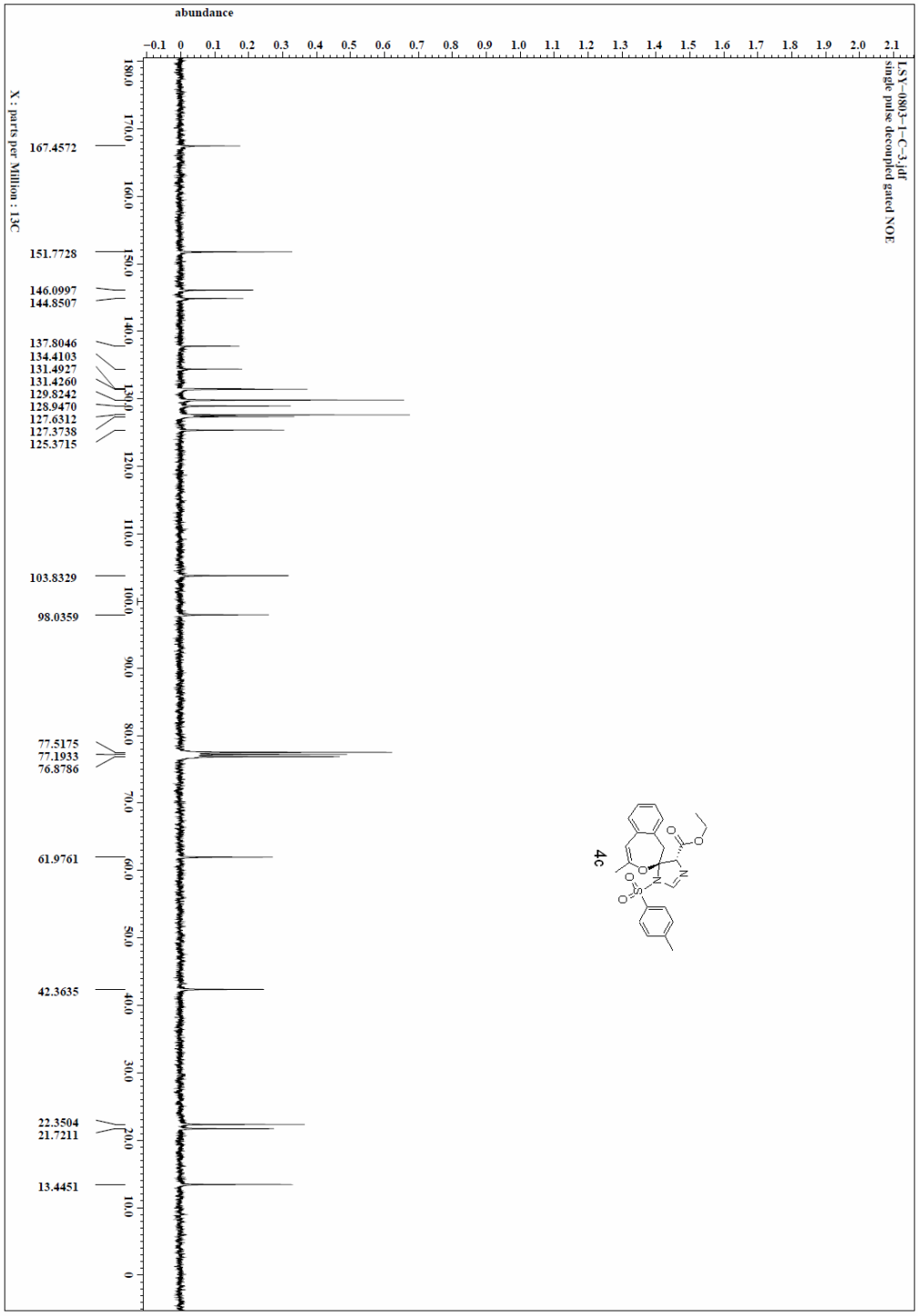
7.3647
7.3452
7.1585
7.1402
7.0164
6.9970
6.9397
6.9225
5.4826
4.4253
3.8194
3.8079
3.7896
3.7621
3.7518
3.4379
3.4311
3.4127
2.4540
1.4963
0.9156
0.8984
0.8812
10.5
20.35
20.04
10.97
20.57
10.6
10
10.67
30.89
10.71
30.44
30.27
30.39
4c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129.8242
128.9470
127.6312
127.3738
125.3715
103.8329
98.0359
77.5175
77.1933
76.8786
61.9761
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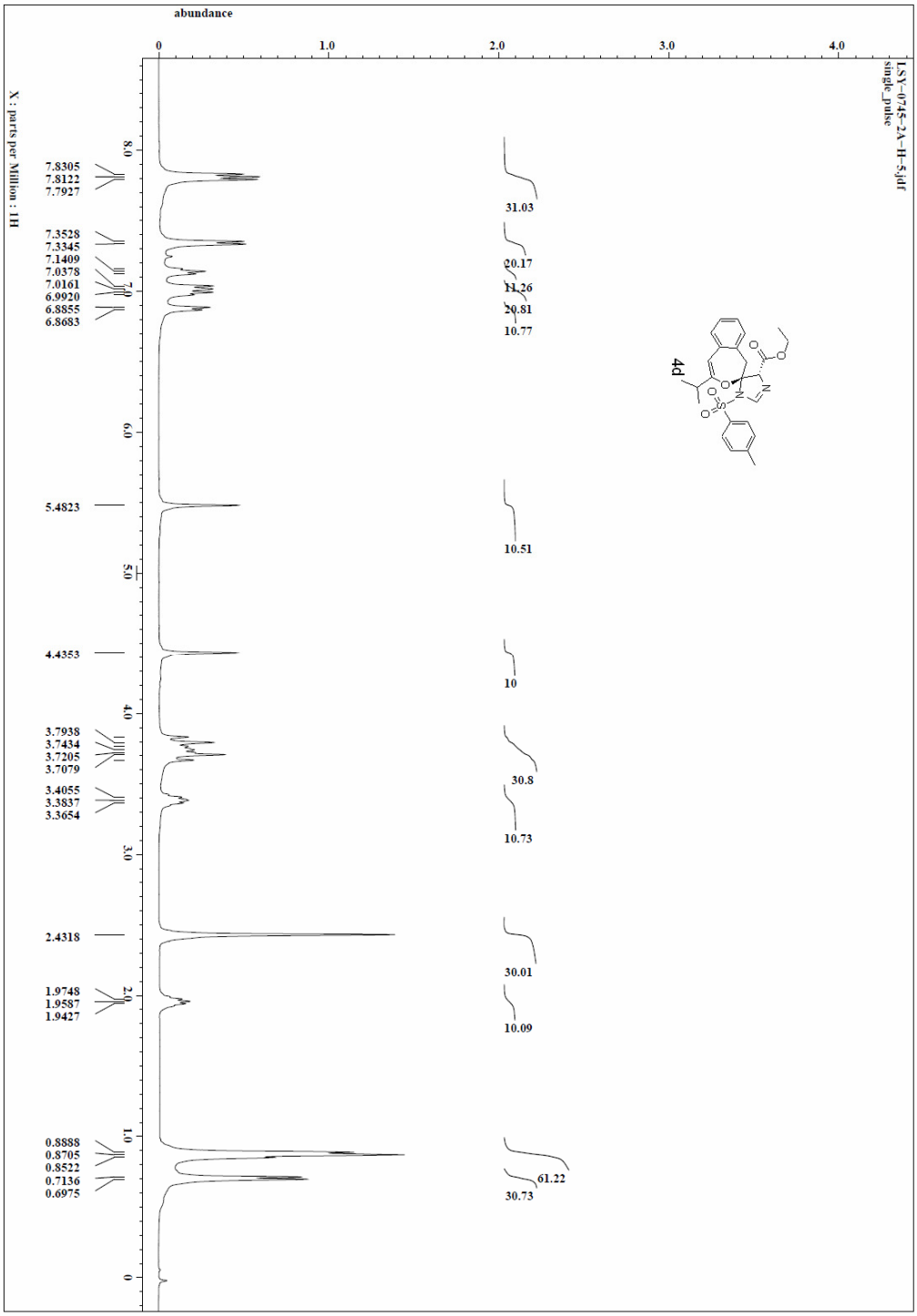
22.3504
21.7211
13.4451
LSY-0803-1-C-3.jdf
single pulse decoupled gated NOE
4c


abundance
X : parts per Million : 1H
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7.3528
7.3345
7.1409
7.0378
7.0161
6.9920
6.8855
6.8683
5.4823
4.4353
3.7938
3.7434
3.7205
3.7079
3.4055
3.3837
3.3654
2.4318
1.9748
1.9587
1.9427
0.8888
0.8705
0.8522
0.7136
0.6975
31.03
20.17
11.26
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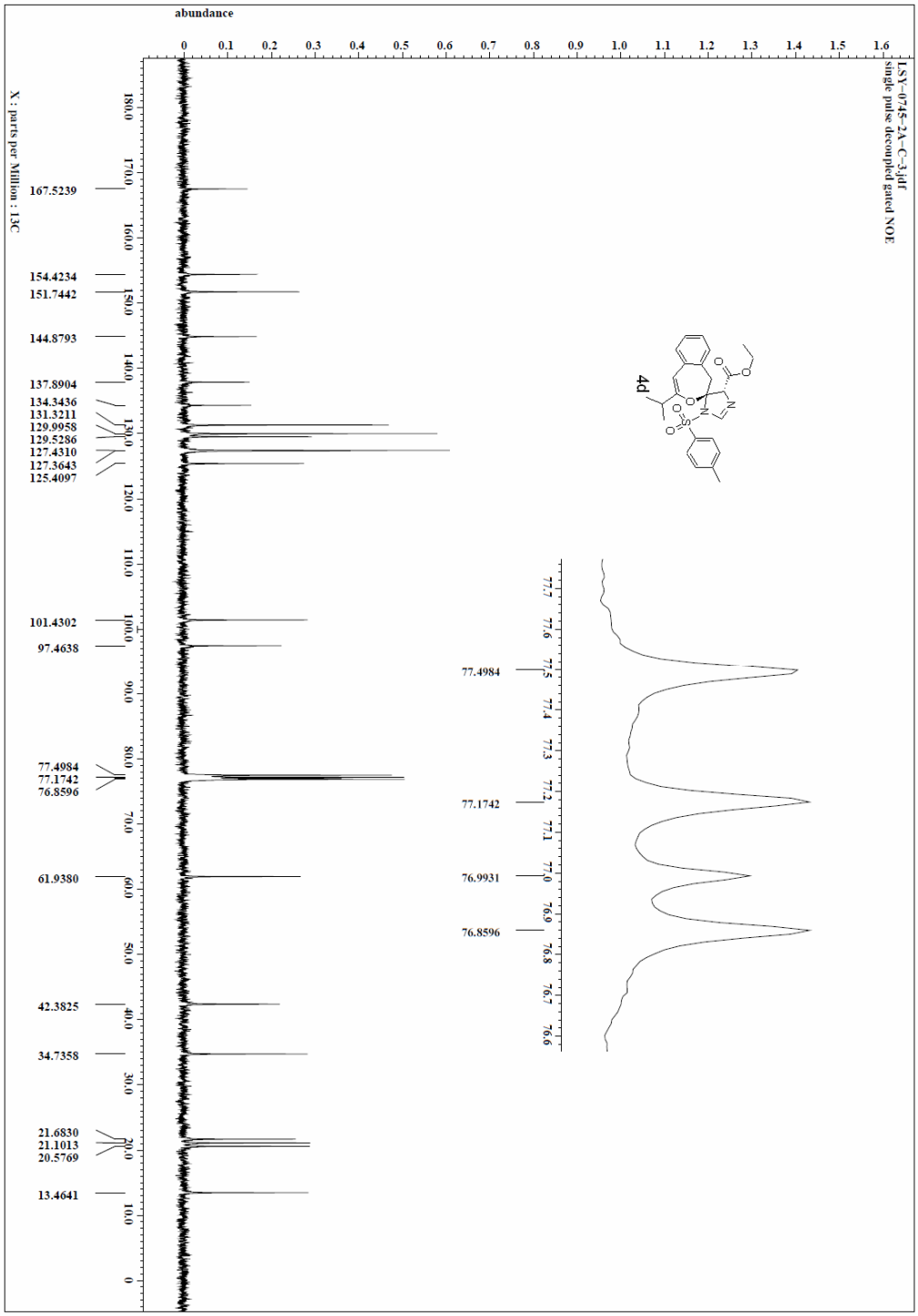
10.77
10.51
10
30.8
10.73
30.01
10.09
61.22
30.73
4d


abundance
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single pulse decoupled gated NOE
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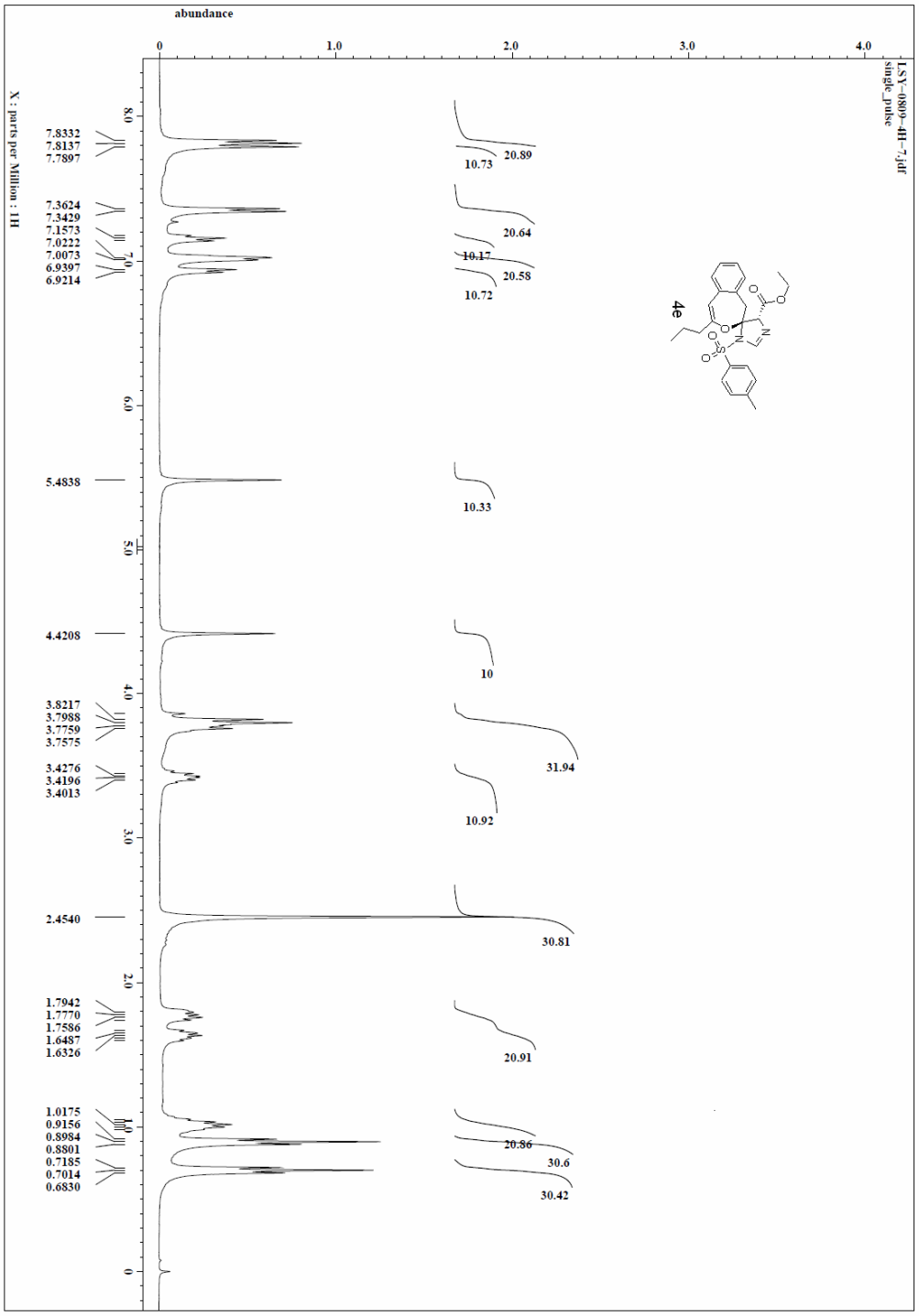
151.7442
144.8793
137.8904
134.3436
131.3211
129.9958
129.5286
127.4310
127.3643
125.4097
101.4302
97.4638
77.4984
77.1742
76.8596
61.9380
42.3825
34.7358
21.6830
21.1013
20.5769
13.4641
4d


abundance
X : parts per Million : 1H
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7.3429
7.1573
7.0222
7.0073
6.9397
6.9214
5.4838
4.4208
3.8217
3.7988
3.7759
3.7575
3.4276
3.4196
3.4013
2.4540
1.7942
1.7770
1.7586
1.6487
1.6326
1.0175
0.9156
0.8984
0.8801
0.7185
0.7014
0.6830
10.73
20.89
20.64
10.17
20.58
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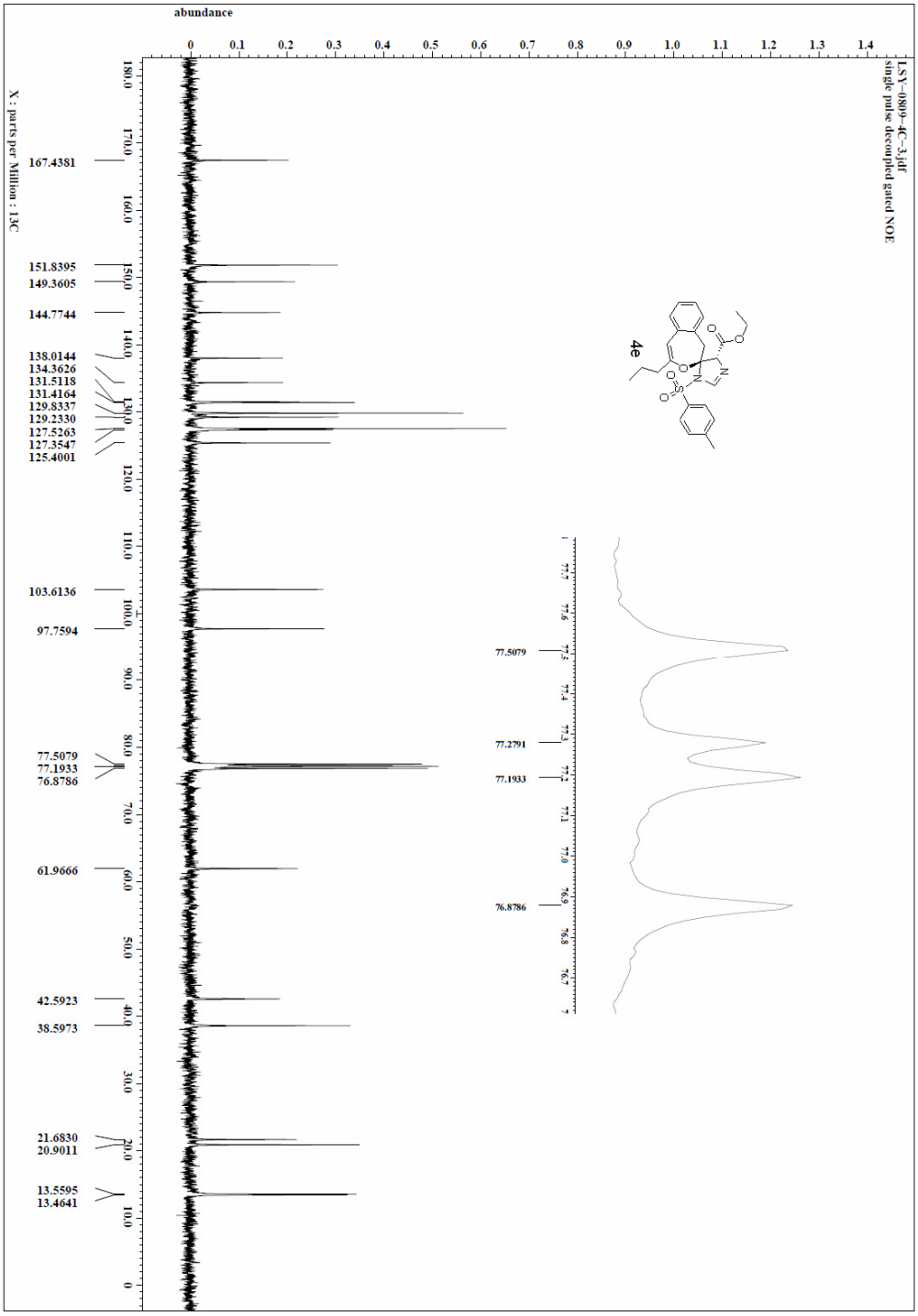
10.33
10
31.94
10.92
30.81
20.91
20.86
30.6
30.42
4e


abundance
LSY-0809-4C-3.jdf
single pulse decoupled gated NOE
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131.5118
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127.3547
125.4001
103.6136
97.7594
77.5079
77.1933
76.8786
61.9666
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4e
77.5079
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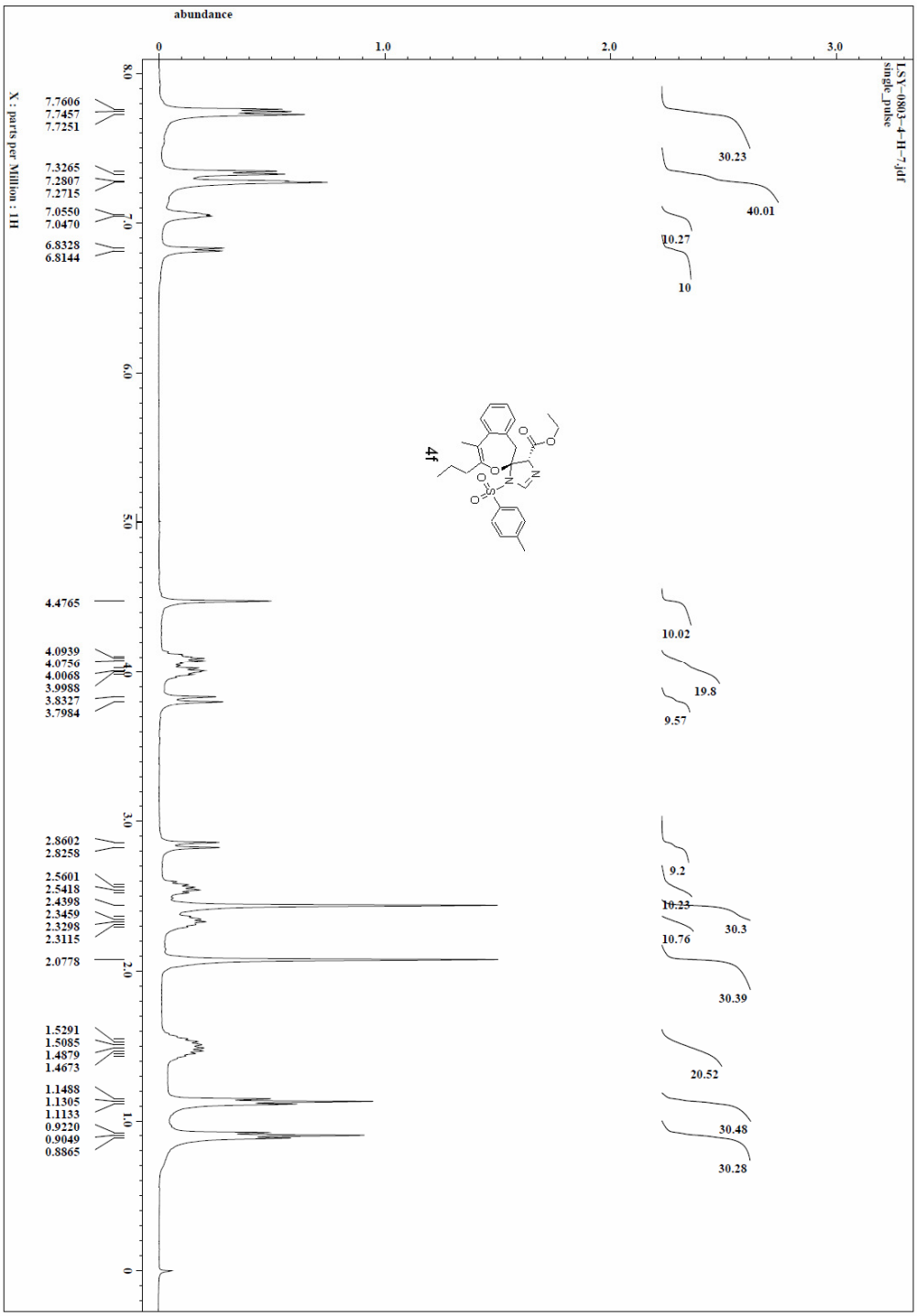
77.1933
76.8786


abundance
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single_pulse
4f
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7.3265
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7.2715
7.0550
7.0470
6.8328
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4.4765
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4.0756
4.0068
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3.8327
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2.4398
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2.3115
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1.5085
1.4879
1.4673
1.1488
1.1305
1.1133
0.9220
0.9049
0.8865
30.23
40.01
10.27
10
10.02
19.8
9.57
9.2
10.23
30.3
10.76
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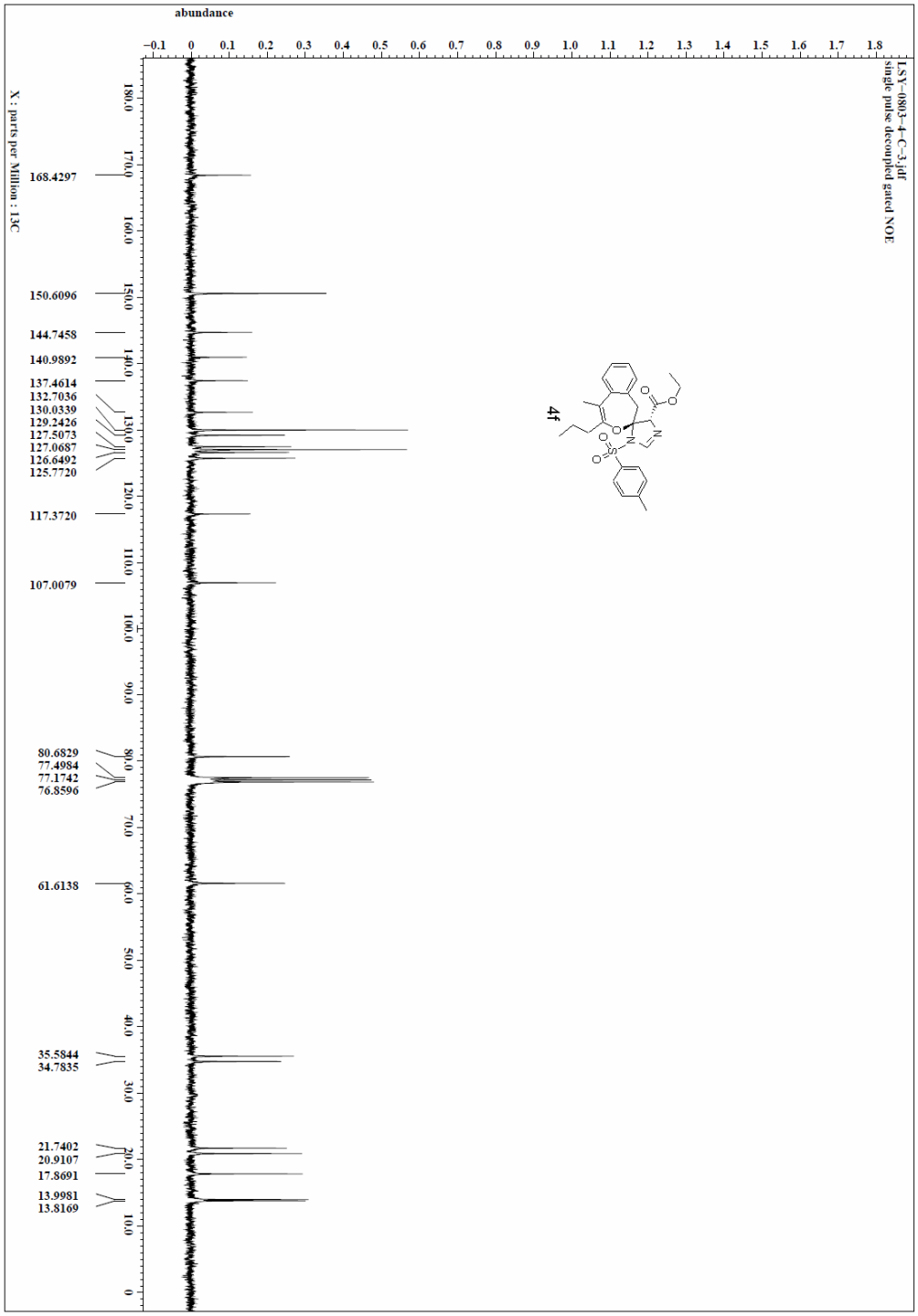
20.52
30.48
30.28


abundance
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LSY-0803-4-C-3.jdf
single pulse decoupled gated NOE
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137.4614
132.7036
130.0339
129.2426
127.5073
127.0687
126.6492
125.7720
117.3720
107.0079
80.6829
77.4984
77.1742
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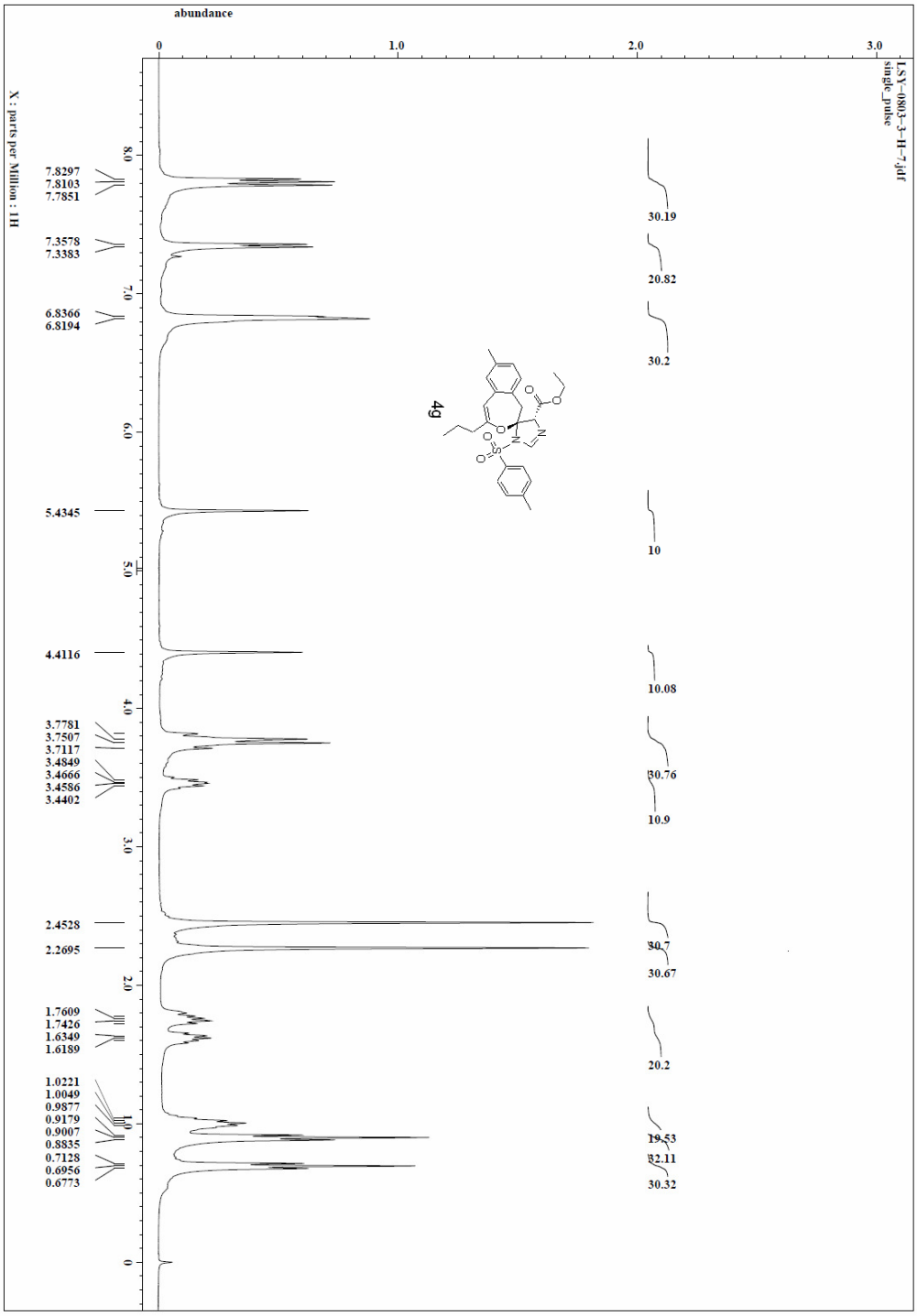
4f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LSY-0803-3-H-7.jdf
single_pulse
X : parts per Million : 1H
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7.3578
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6.8194
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3.7117
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3.4586
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1.7426
1.6349
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0.9007
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20.2
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32.11
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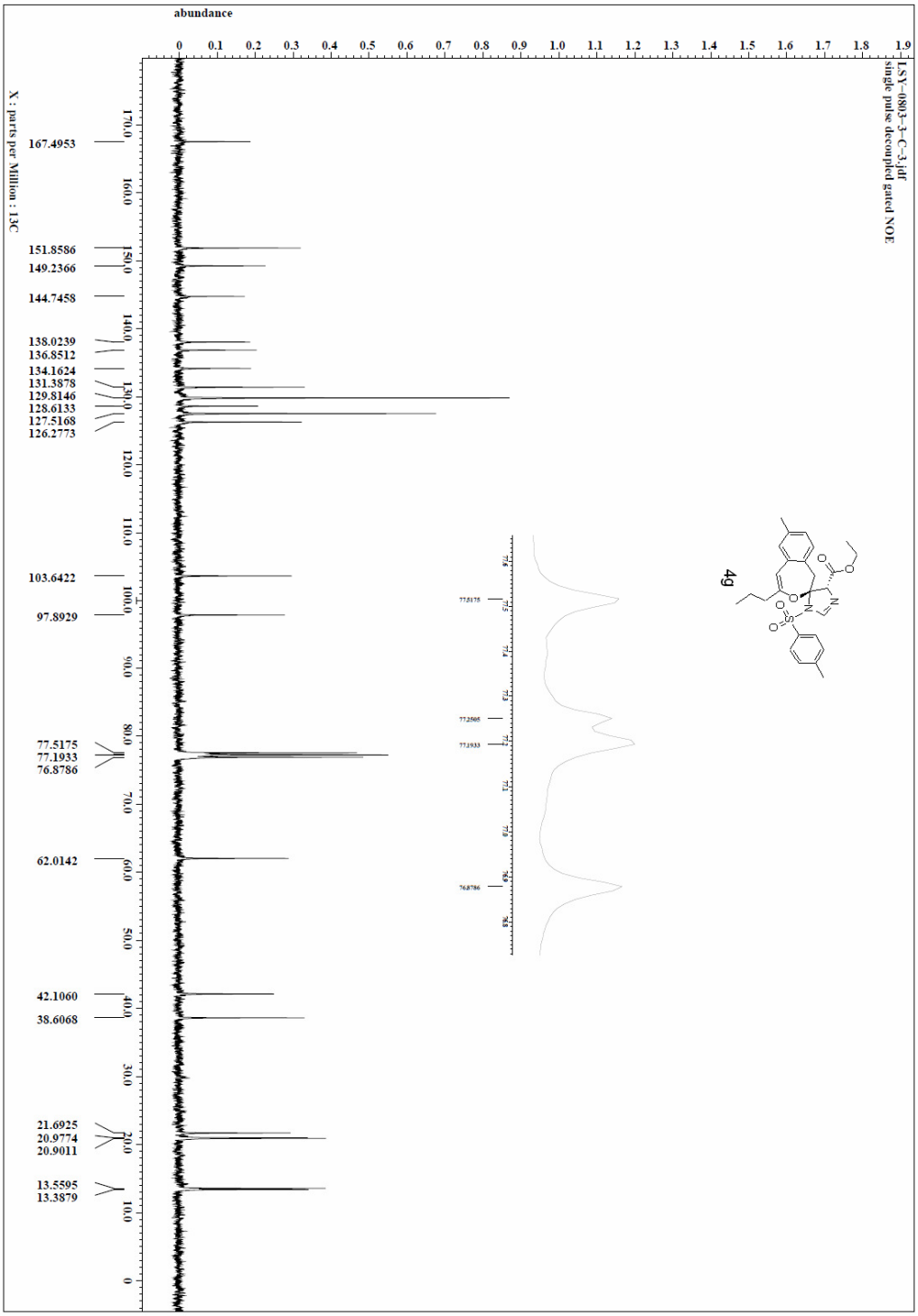
abundance
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single pulse decoupled gated NOE
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136.8512
134.1624
131.3878
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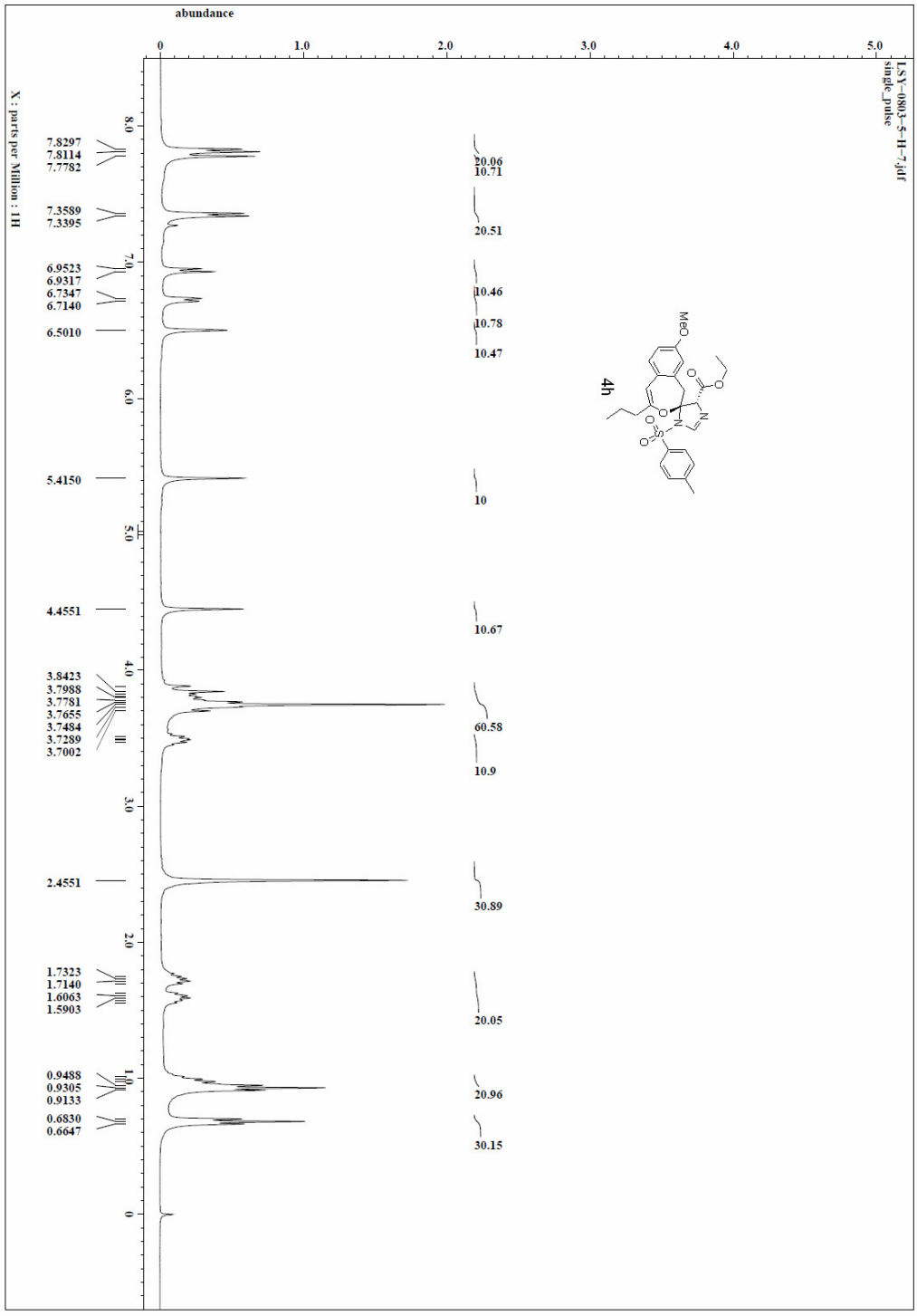
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5.4150
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3.7781
3.7655
3.7484
3.7289
3.7002
2.4551
1.7323
1.7140
1.6063
1.5903
0.9488
0.9305
0.9133
0.6830
0.6647
20.06
10.71
20.51
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10.78
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10
10.67
60.58
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20.05
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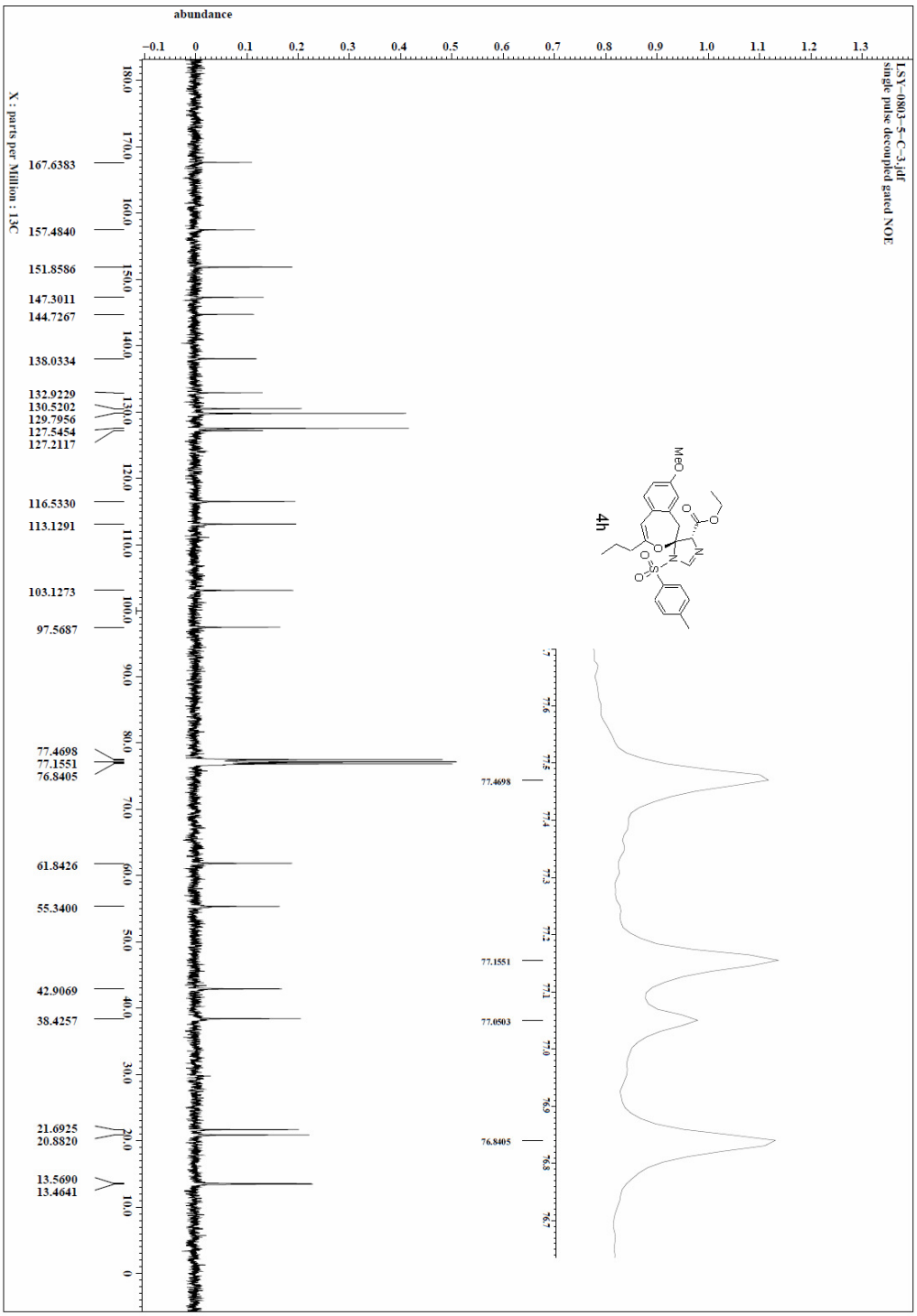
30.15
MeO
4h


abundance
LSY-0803-5-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
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151.8586
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144.7267
138.0334
132.9229
130.5202
129.7956
127.5454
127.2117
116.5330
113.1291
103.1273
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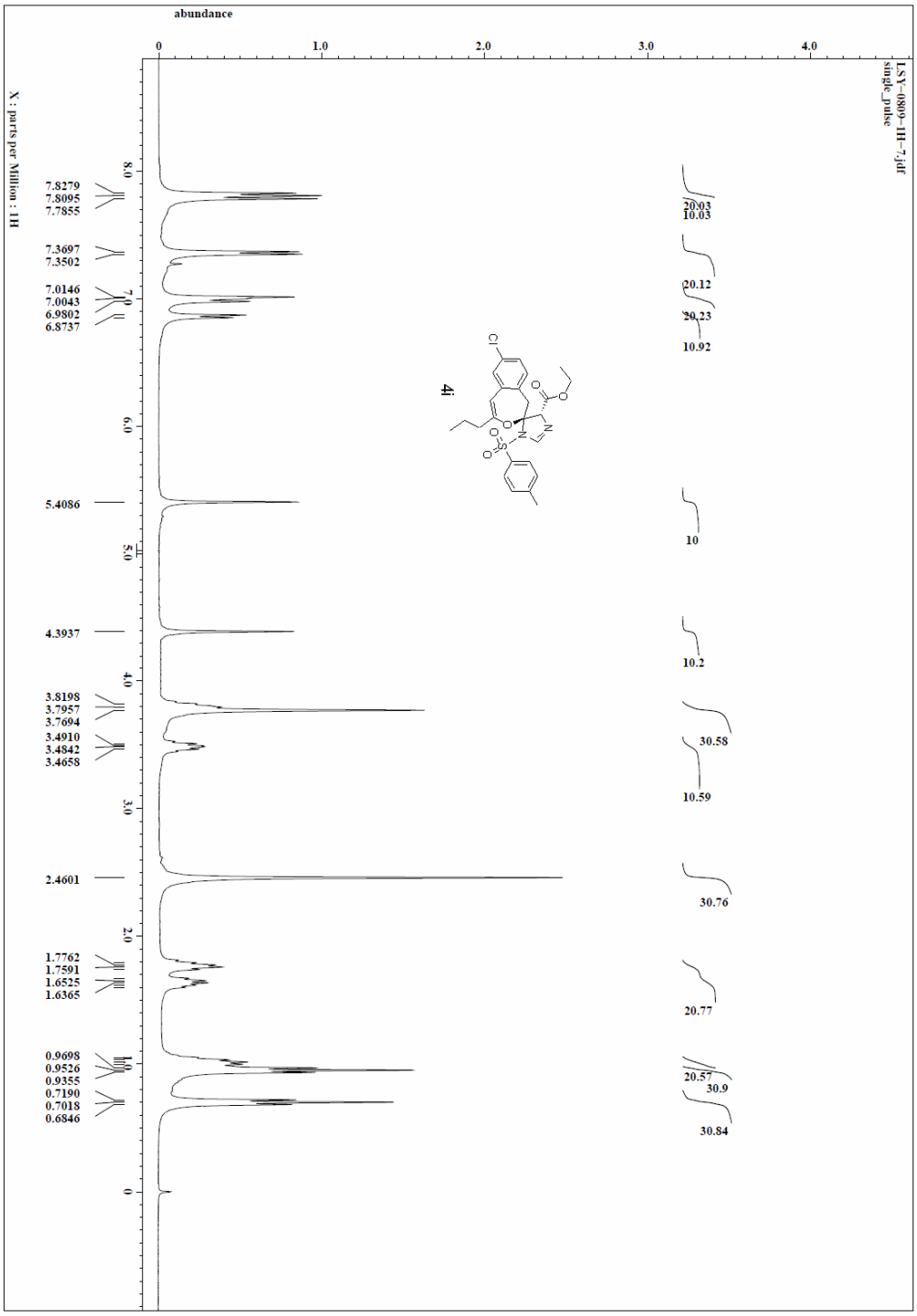
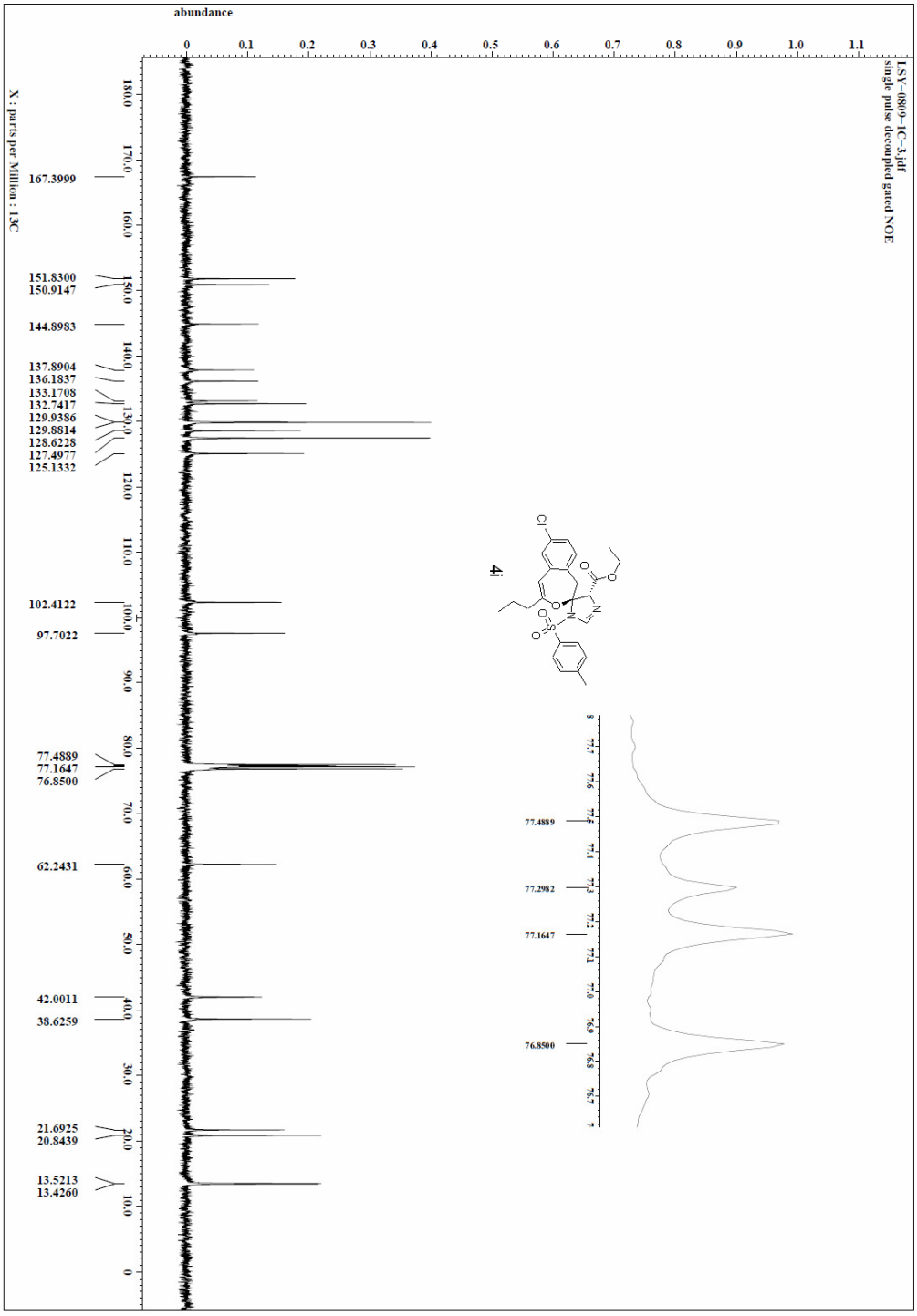
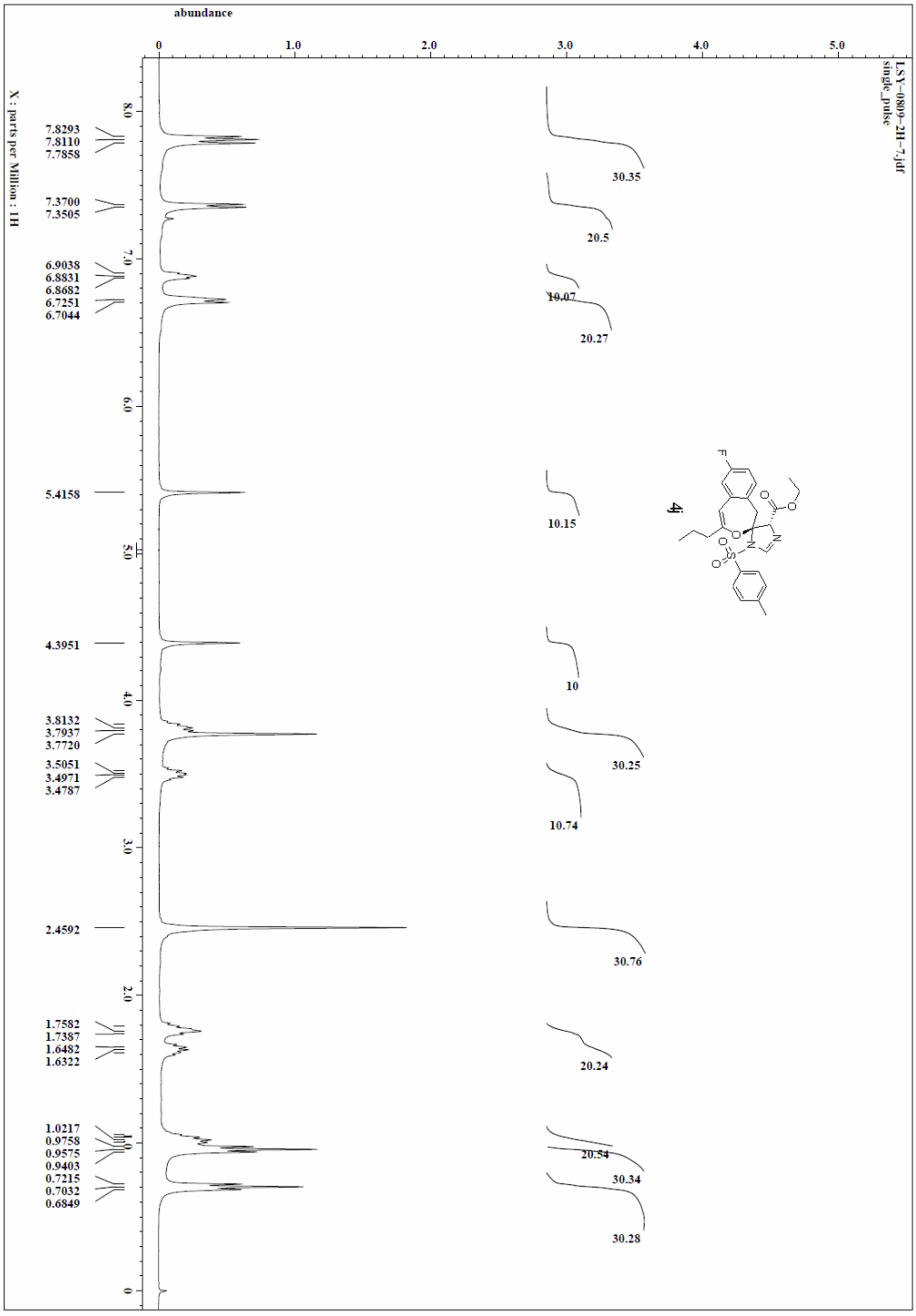
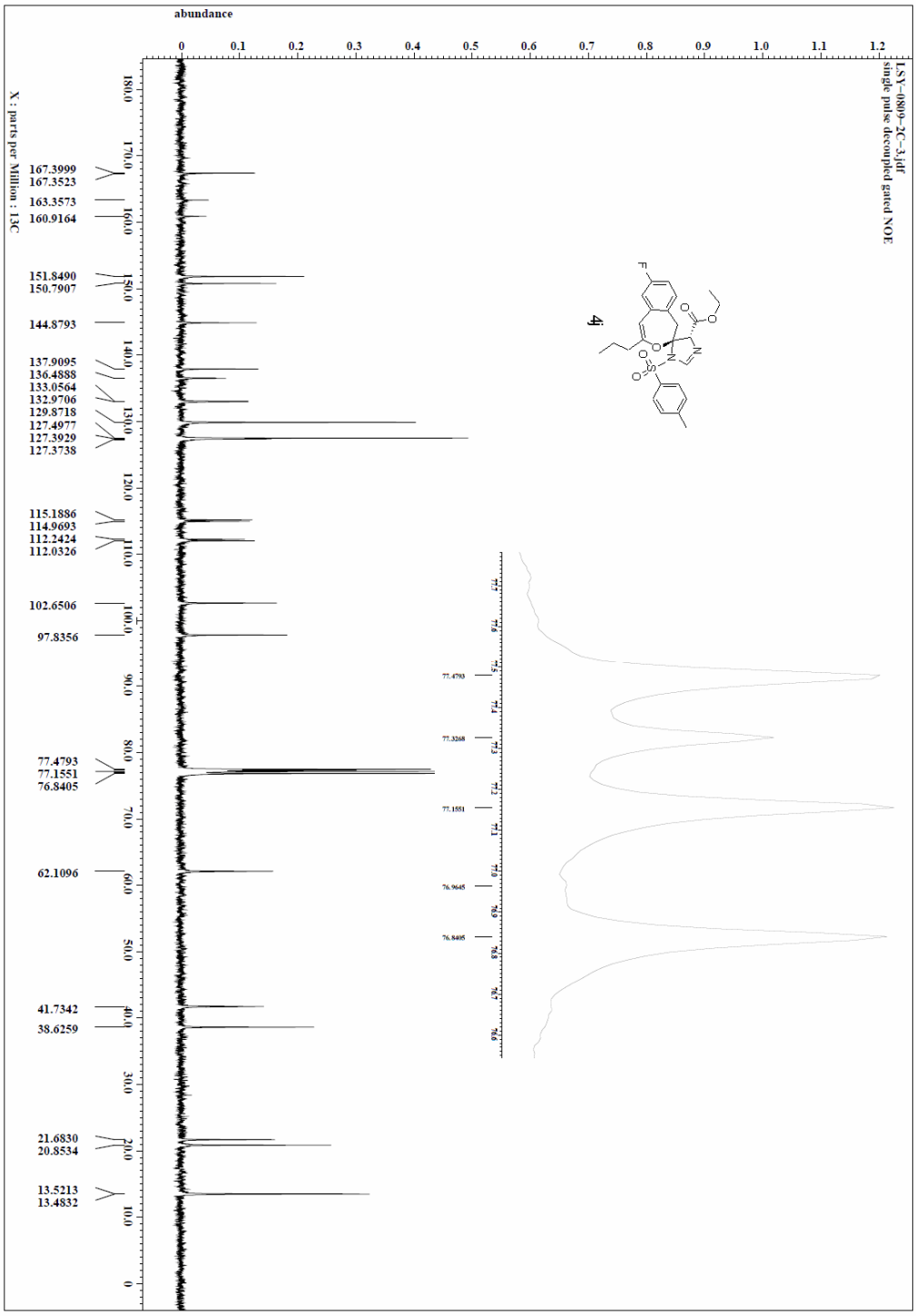
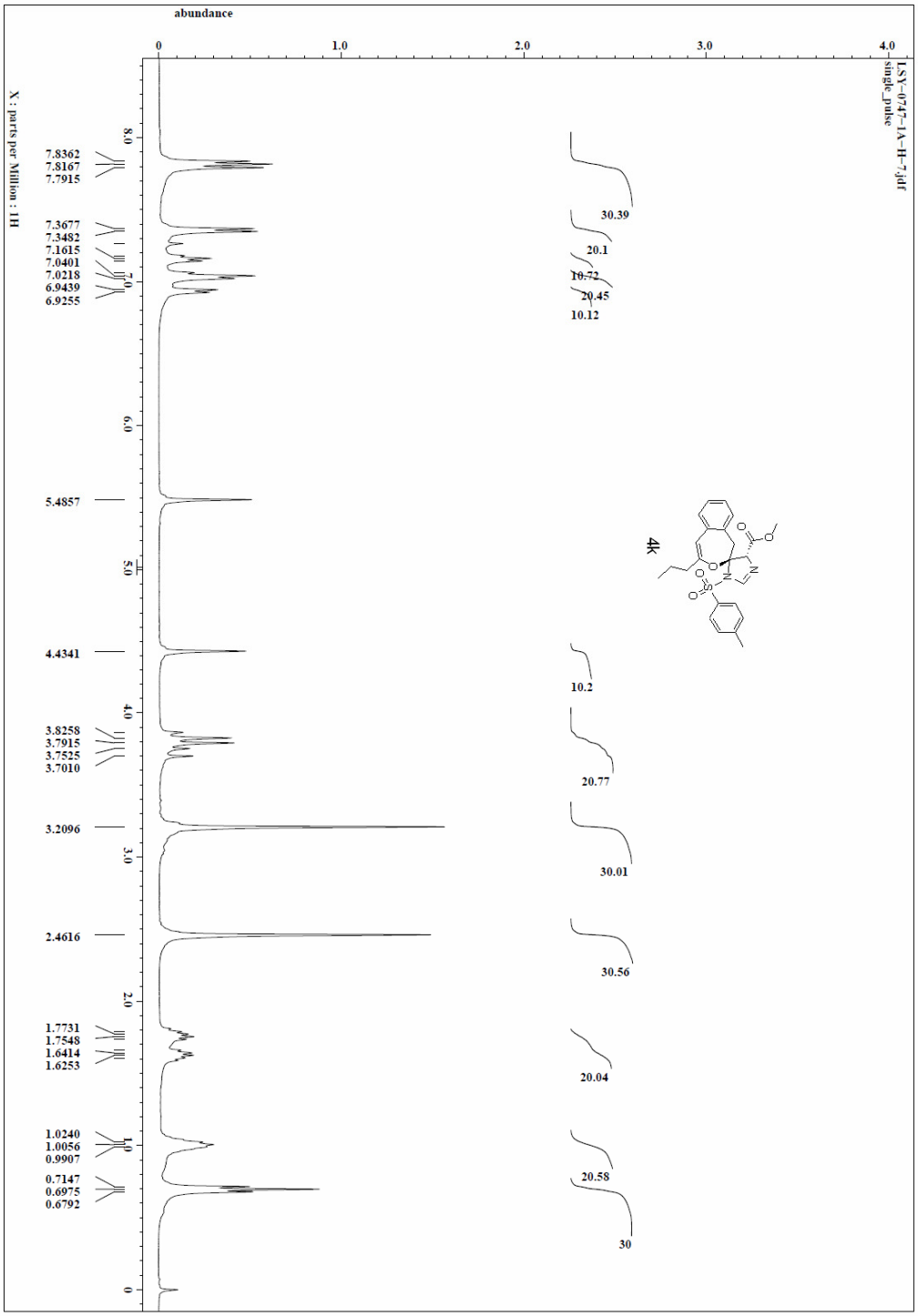
77.4698
77.1551
76.8405
61.8426
55.3400
42.9069
38.4257
21.6925
20.8820
13.5690
13.4641
MeO
4h

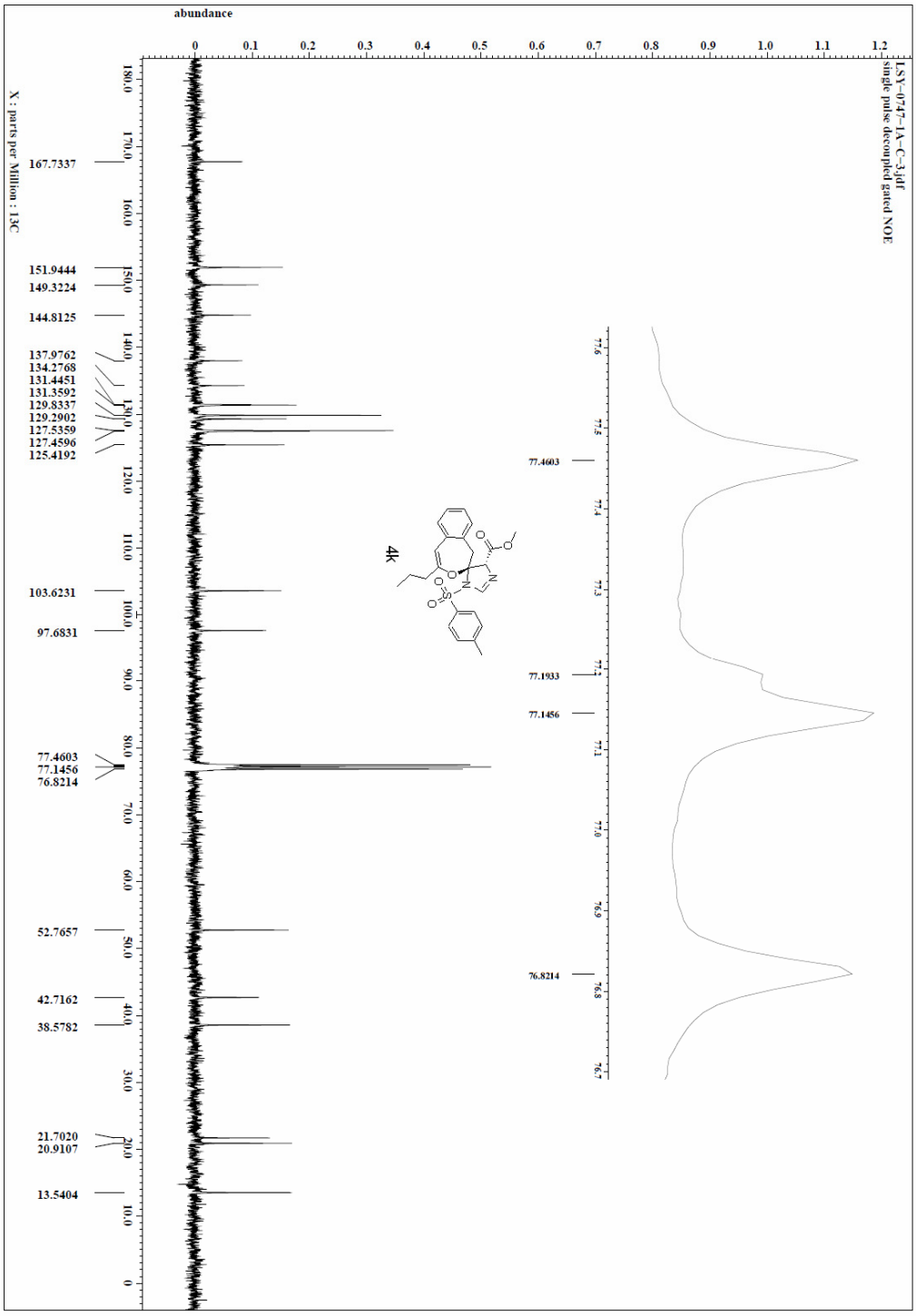

abundance
X : parts per Million : 1H
LSY-0809-1H-7.jdf
single_pulse
7.8279
7.8095
7.7855
7.3697
7.3502
7.0146
7.0043
6.9802
6.8737
5.4086
4.3937
3.8198
3.7957
3.7694
3.4910
3.4842
3.4658
2.4601
1.7762
1.7591
1.6525
1.6365
0.9698
0.9526
0.9355
0.7190
0.7018
0.6846
20.03
10.03
20.12
20.23
10.92
10
10.2
30.58
10.59
30.76
20.77
20.57
30.9
30.84
4i

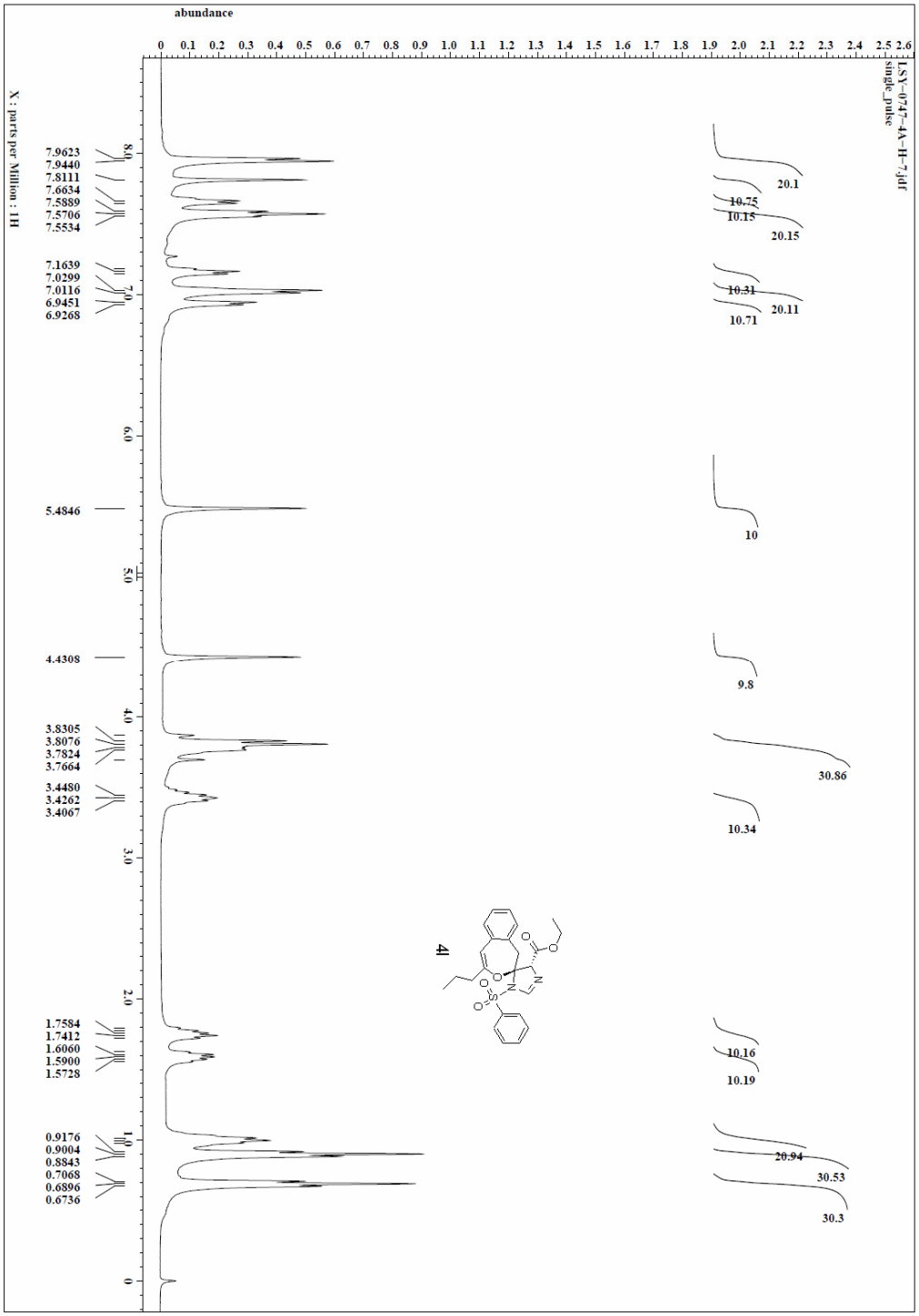

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
LSY-0809-1C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
167.3999
151.8300
150.9147
144.8983
137.8904
136.1837
133.1708
132.7417
129.9386
129.8814
128.6228
127.4977
125.1332
102.4122
97.7022
77.4889
77.1647
76.8500
62.2431
42.0011
38.6259
21.6925
20.8439
13.5213
13.4260
4i
77.4889
77.2982
77.1647
76.8500

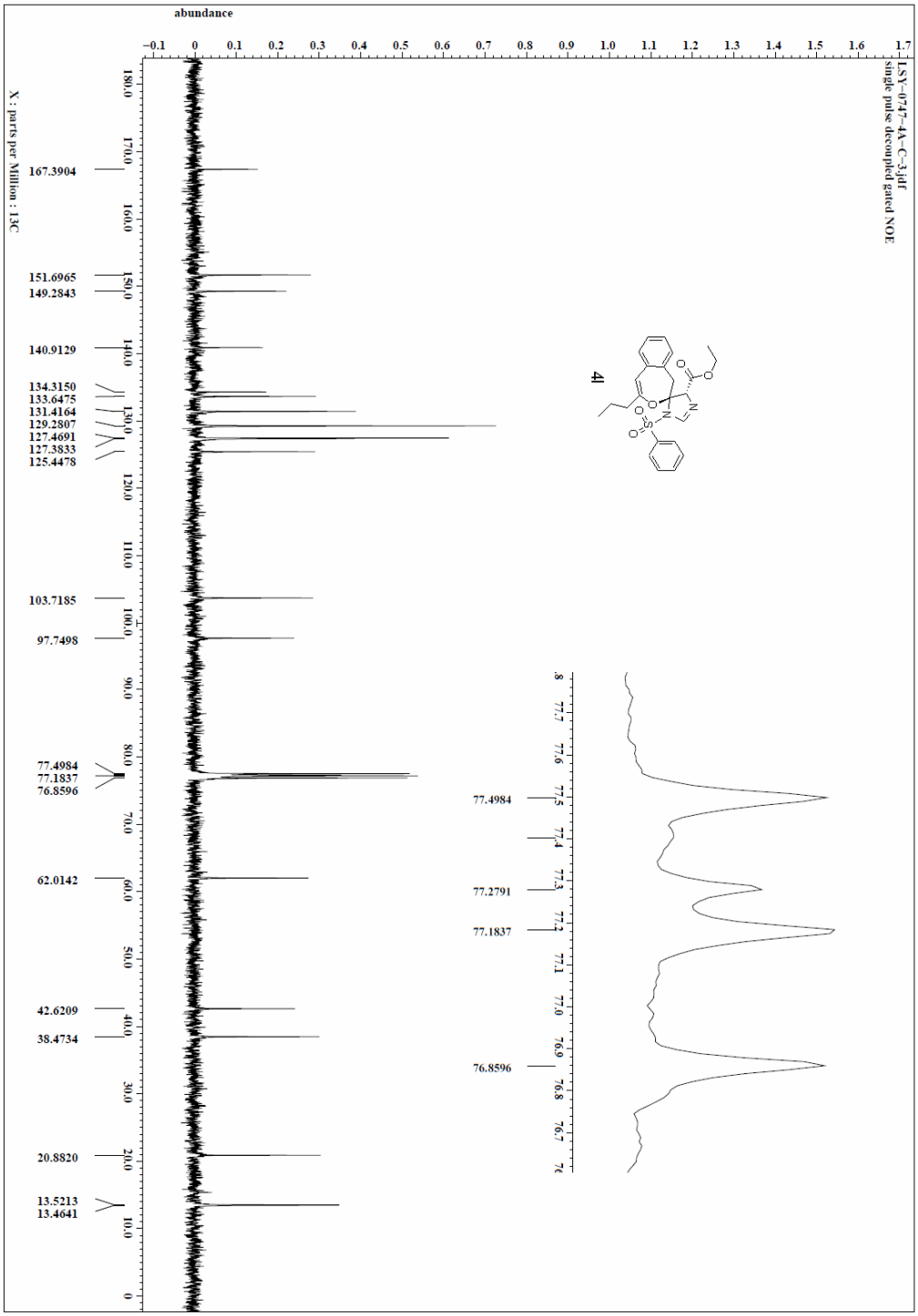

abundance
X : parts per Million : 1H
LSY-0809-2H-7.jdf
single_pulse
7.8293
7.8110
7.7858
7.3700
7.3505
6.9038
6.8831
6.8682
6.7251
6.7044
5.4158
4.3951
3.8132
3.7937
3.7720
3.5051
3.4971
3.4787
2.4592
1.7582
1.7387
1.6482
1.6322
1.0217
0.9758
0.9575
0.9403
0.7215
0.7032
0.6849
30.35
20.5
10.07
20.27
10.15
10
30.25
10.74
30.76
20.24
20.54
30.34
30.28
4j

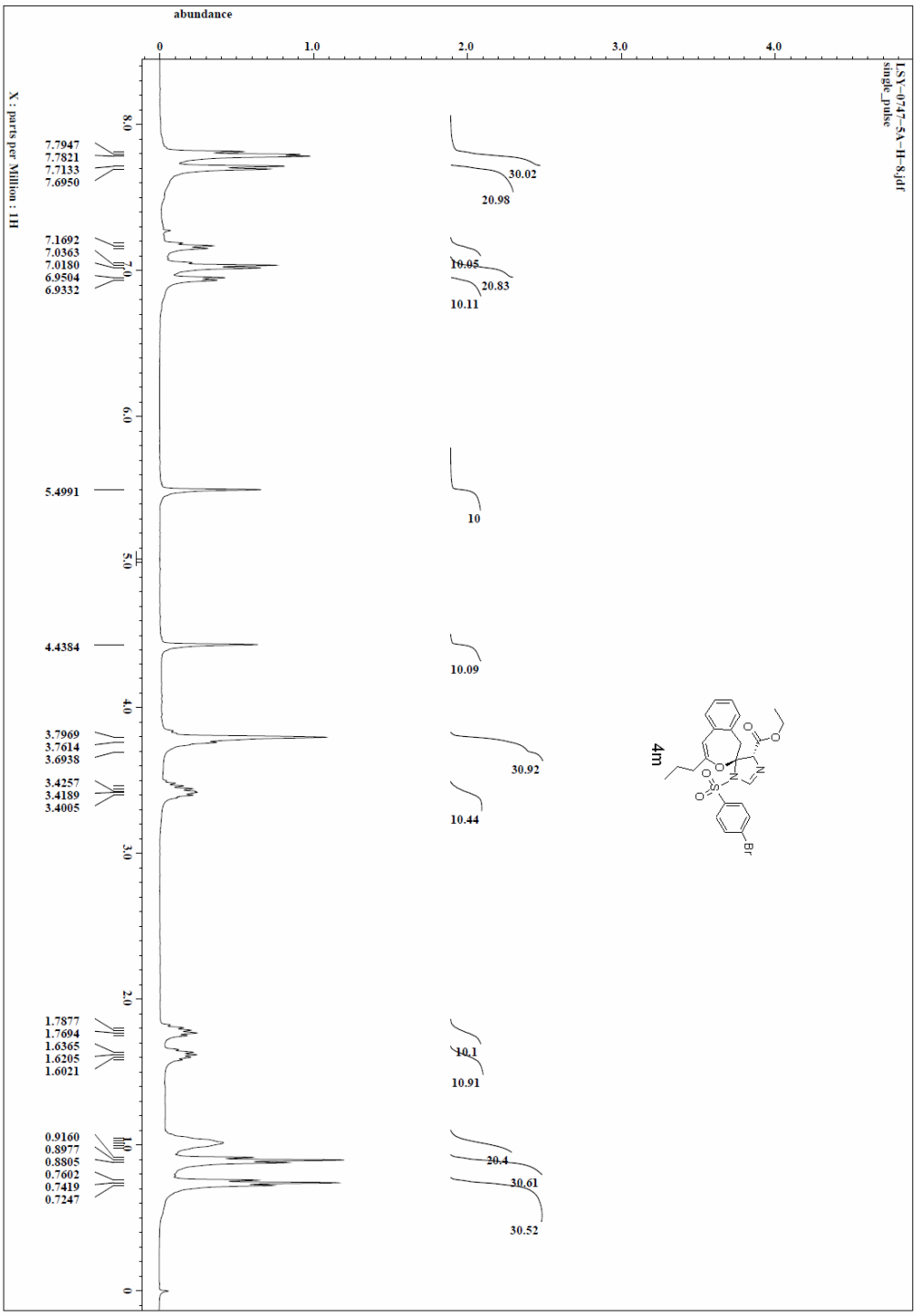

abundance
X : parts per Million : 13C
LSY-0809-2C-3.jdf
single pulse decoupled gated NOE
4j
167.3999
167.3523
163.3573
160.9164
151.8490
150.7907
144.8793
137.9095
136.4888
133.0564
132.9706
129.8718
127.4977
127.3929
127.3738
115.1886
114.9693
112.2424
112.0326
102.6506
97.8356
77.4793
77.1551
76.8405
62.1096
41.7342
38.6259
21.6830
20.8534
13.5213
13.4832

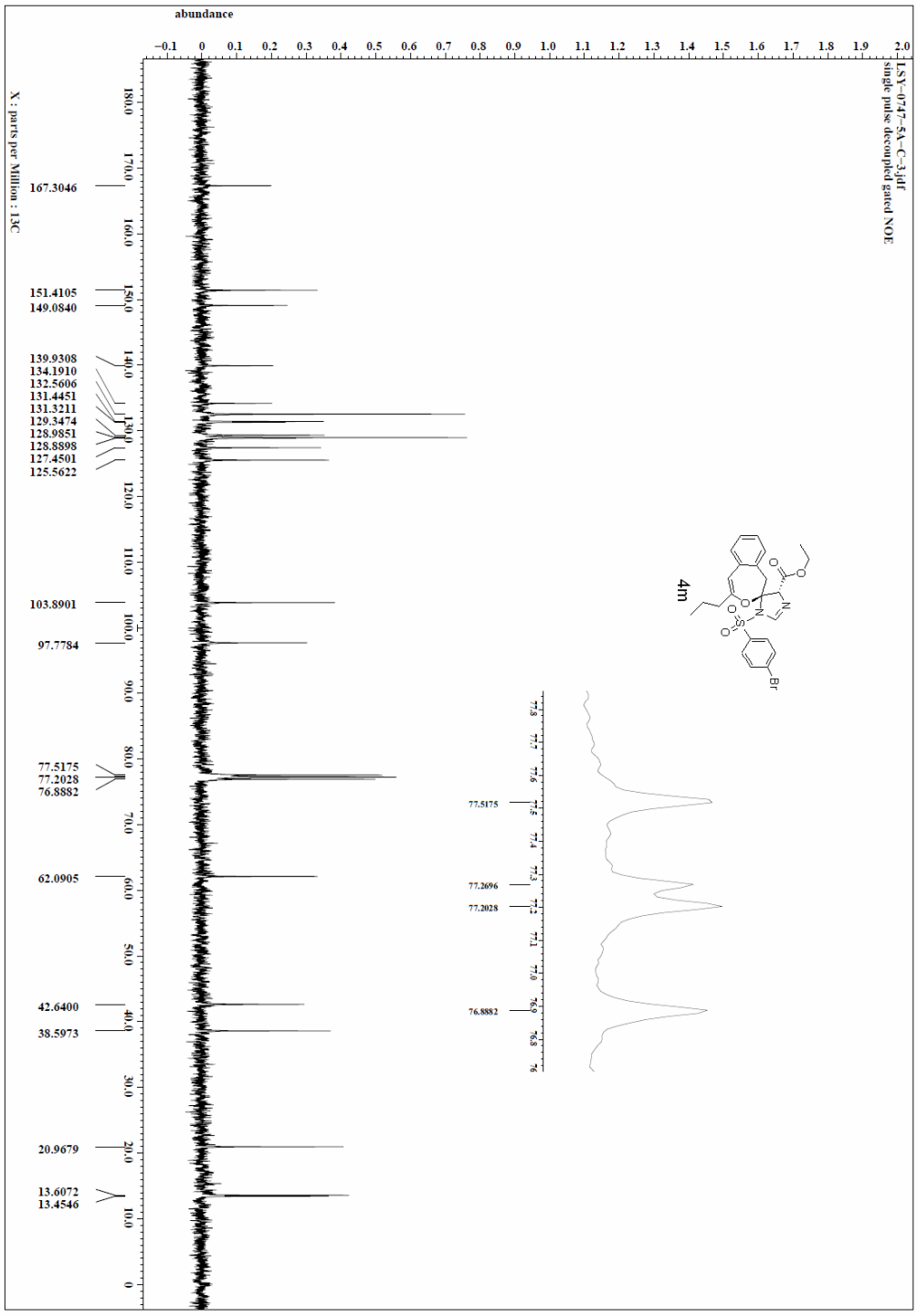

abundance
X : parts per Million : 1H
LSY-0747-1A-H-7.jdf
single_pulse
7.8362
7.8167
7.7915
7.3677
7.3482
7.1615
7.0401
7.0218
6.9439
6.9255
5.4857
4.4341
3.8258
3.7915
3.7525
3.7010
3.2096
2.4616
1.7731
1.7548
1.6414
1.6253
1.0240
1.0056
0.9907
0.7147
0.6975
0.6792
30.39
20.1
10.72
20.45
10.12
10.2
20.77
30.01
30.56
20.04
20.58
30
4k

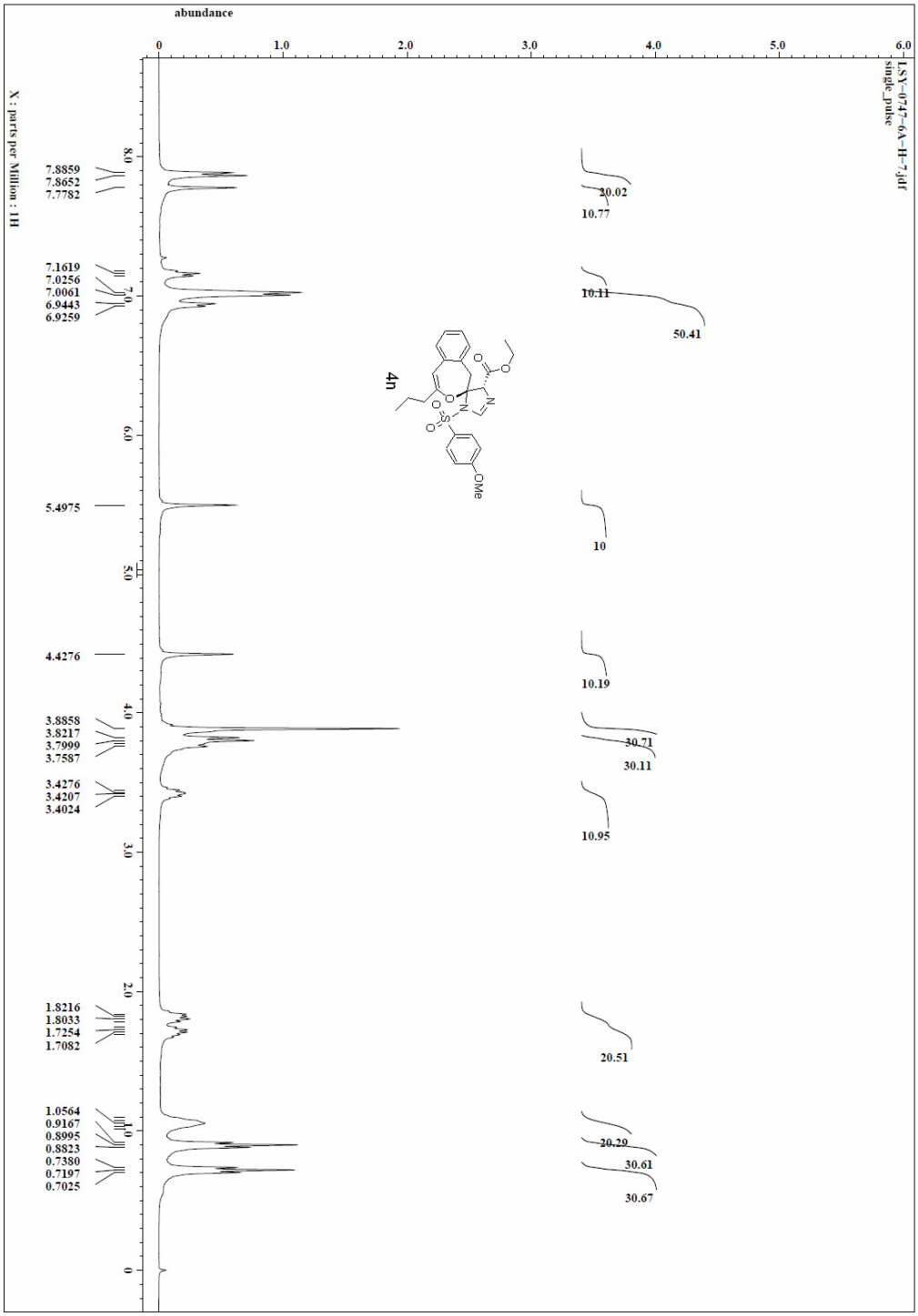

abundance
X : parts per Million : 13C
LSY-0747-1A-C-3.jdf
single pulse decoupled gated NOE
167.7337
151.9444
149.3224
144.8125
137.9762
134.2768
131.4451
131.3592
129.8337
129.2902
127.5350
127.4596
125.4192
103.6231
97.6831
77.4603
77.1456
76.8214
52.7657
42.7162
38.5782
21.7020
20.9107
13.5404
4k

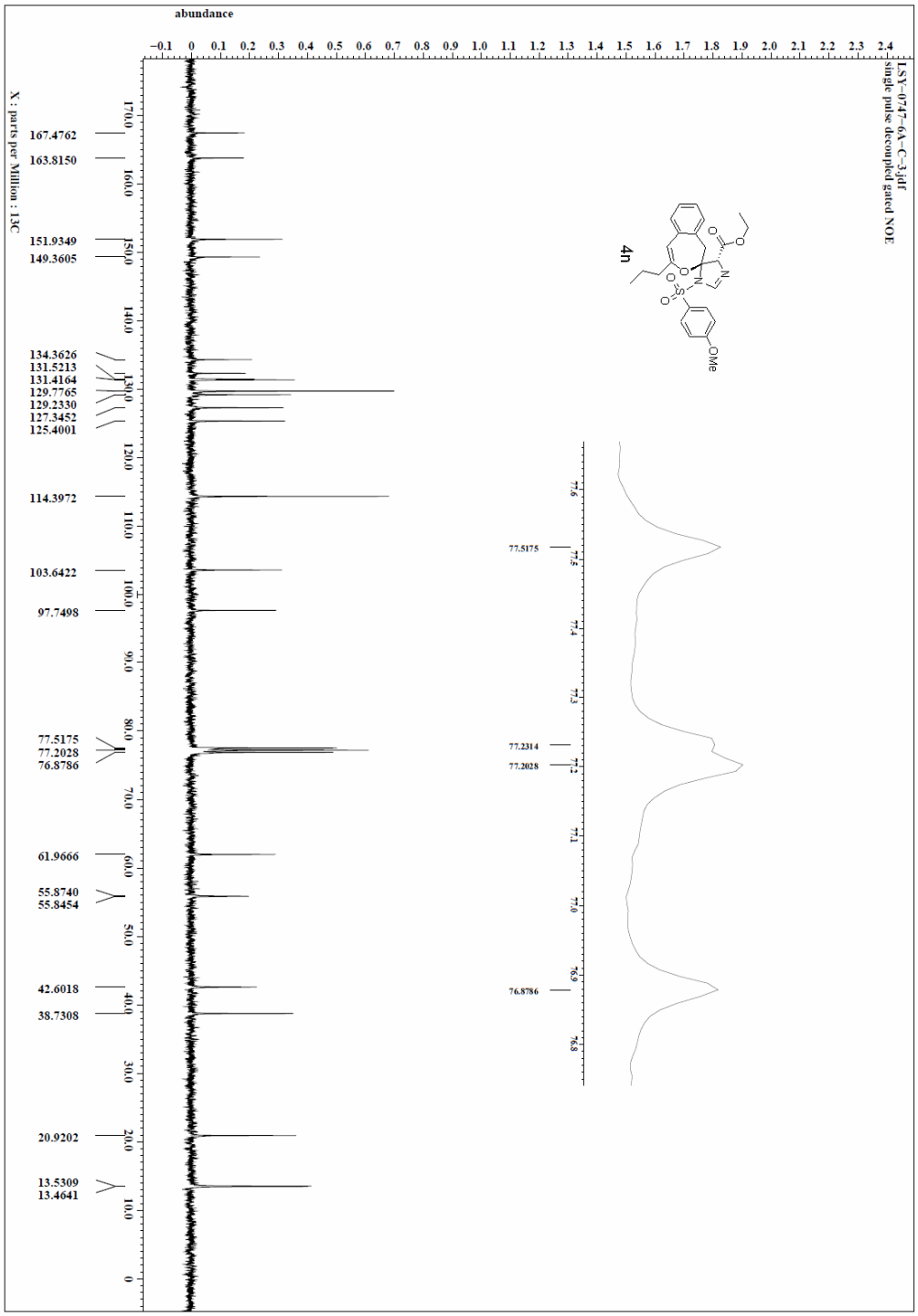

abundance
X : parts per Million : 1H
LSY-0747-4A-H-7.jdf
single_pulse
7.9623
7.9440
7.8111
7.6634
7.5889
7.5706
7.5534
7.1639
7.0299
7.0116
6.9451
6.9268
5.4846
4.4308
3.8305
3.8076
3.7824
3.7664
3.4480
3.4262
3.4067
1.7584
1.7412
1.6060
1.5900
1.5728
0.9176
0.9004
0.8843
0.7068
0.6896
0.6736
20.1
10.75
10.15
20.15
10.31
20.11
10.71
10
9.8
30.86
10.34
10.16
10.19
20.94
30.53
30.3
4l

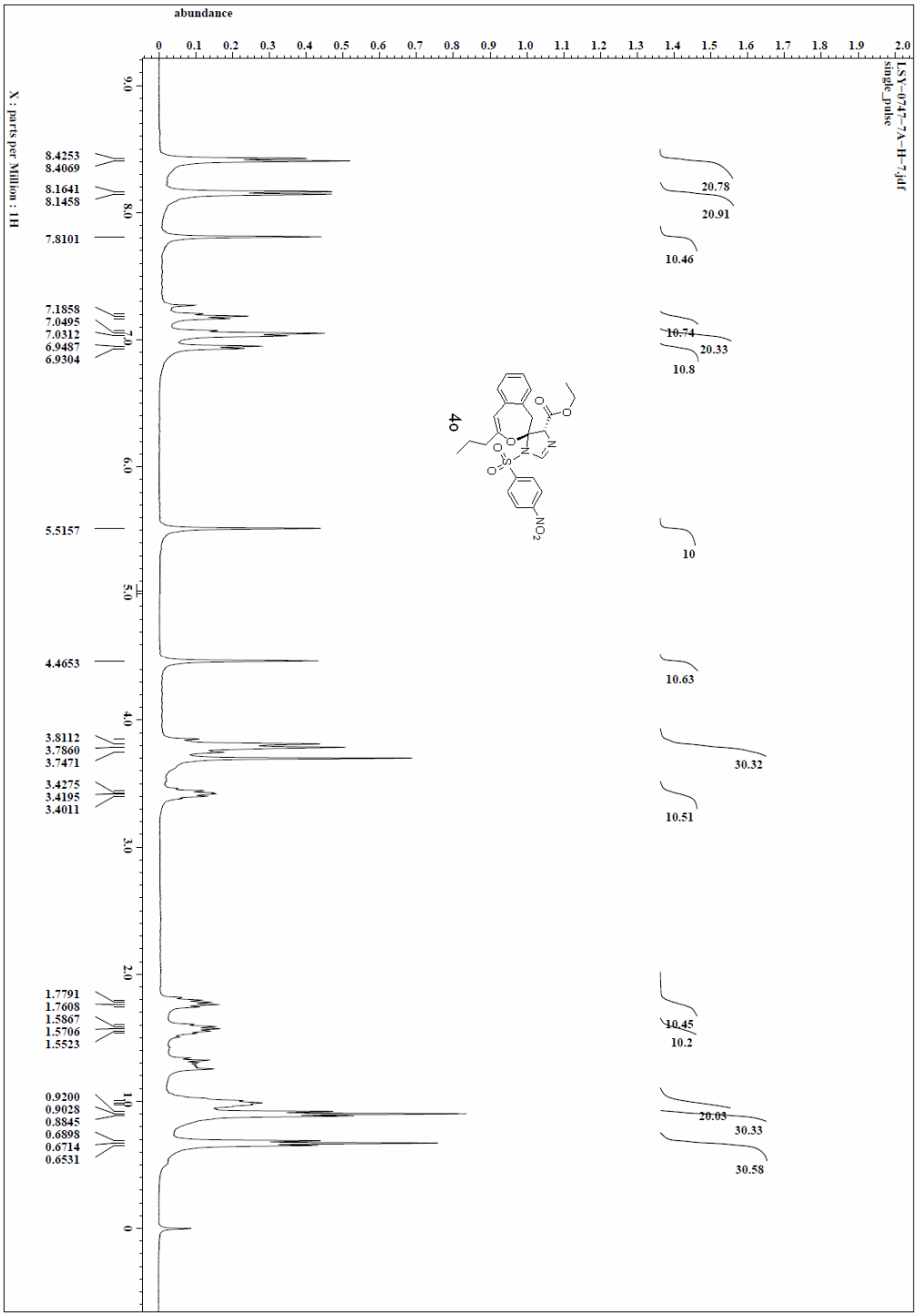

abundance
X : parts per Million : 13C
LSY-0747-4A-C-3.jdf
single pulse decoupled gated NOE
167.3904
151.6965
149.2843
140.9129
134.3150
133.6475
131.4164
129.2807
127.4691
127.3833
125.4478
103.7185
97.7498
77.4984
77.1837
76.8596
62.0142
42.6209
38.4734
20.8820
13.5213
13.4641
4l
77.4984
77.2791
77.1837
76.8596

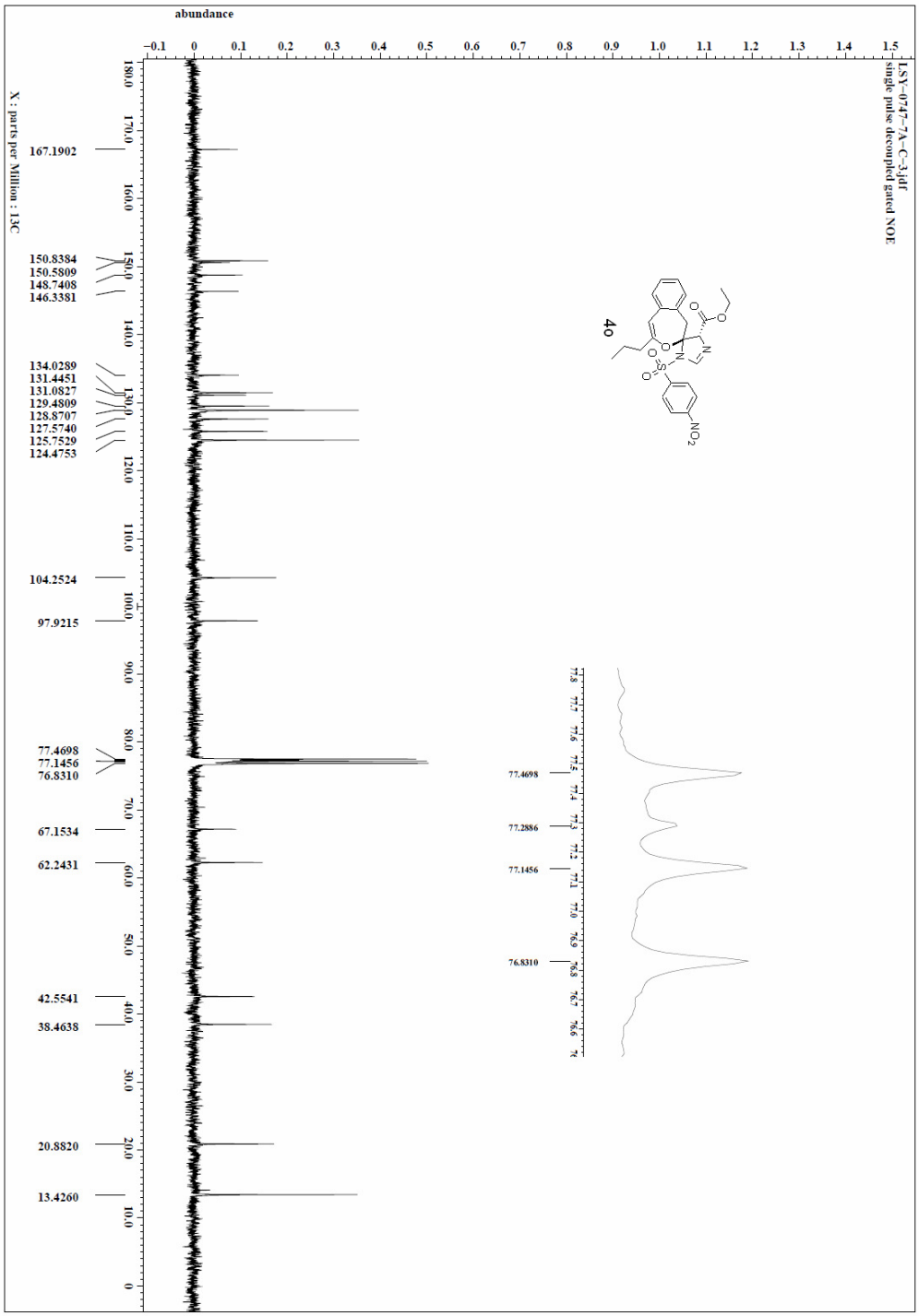

abundance
X : parts per Million : 1H
LSY-074-5A-H-8.jdf
single_pulse
7.7947
7.7821
7.7133
7.6950
7.1692
7.0363
7.0180
6.9504
6.9332
5.4991
4.4384
3.7969
3.7614
3.6938
3.4257
3.4189
3.4005
1.7877
1.7694
1.6365
1.6205
1.6021
0.9160
0.8977
0.8805
0.7602
0.7419
0.7247
30.02
20.98
10.05
20.83
10.11
10
10.09
30.92
10.44
10.1
10.91
20.4
30.61
30.52
4m
Br

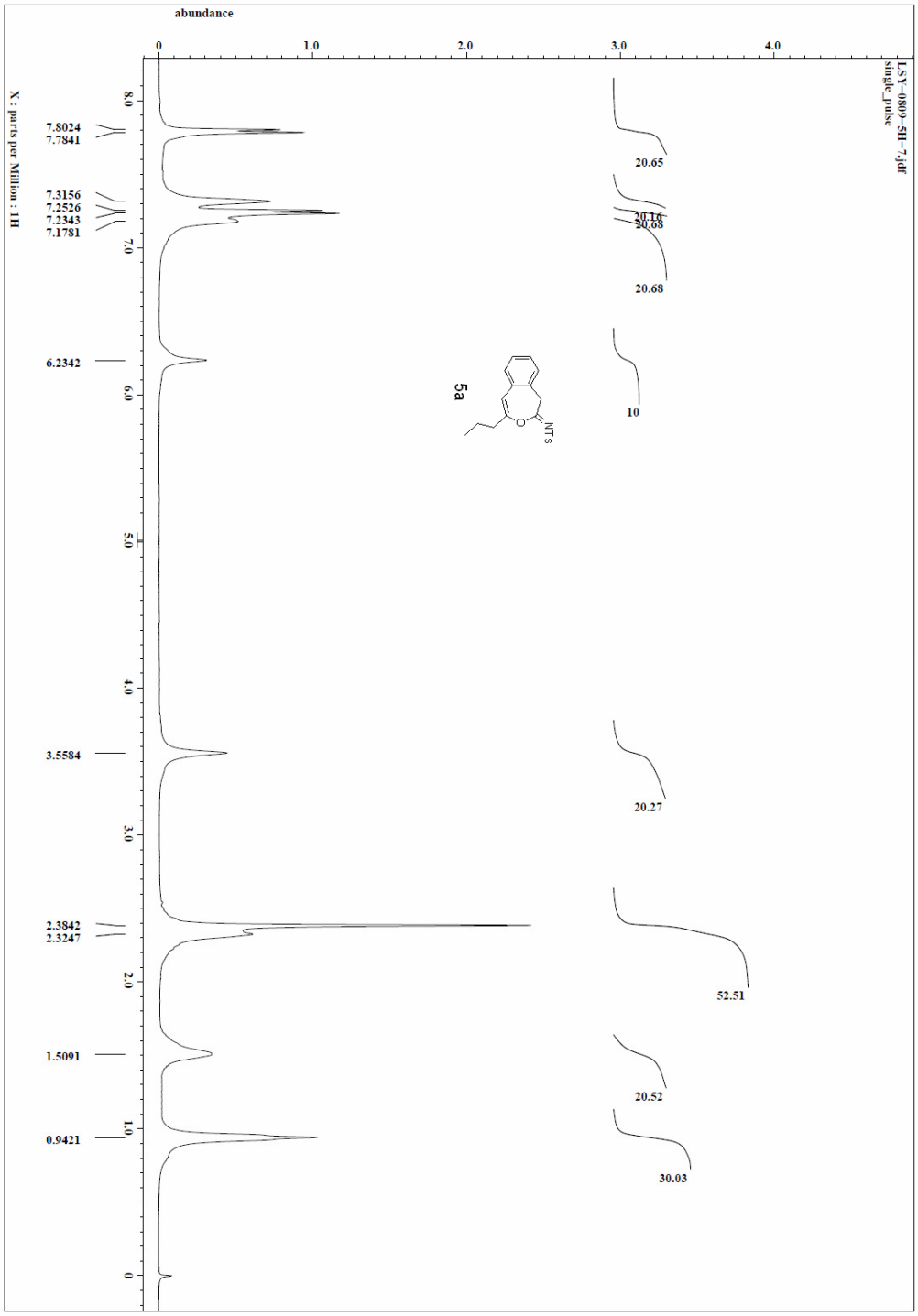

abundance
X : parts per Million : 13C
LSY-0747-5A-C-3.jdf
single pulse decoupled gated NOE
167.3046
151.4105
149.0840
139.9308
134.1910
132.5606
131.4451
131.3211
129.3474
128.9851
128.8898
127.4501
125.5622
103.8901
97.7784
77.5175
77.2028
76.8882
62.0905
42.6400
38.5973
20.9679
13.6072
13.4546
4m
Br

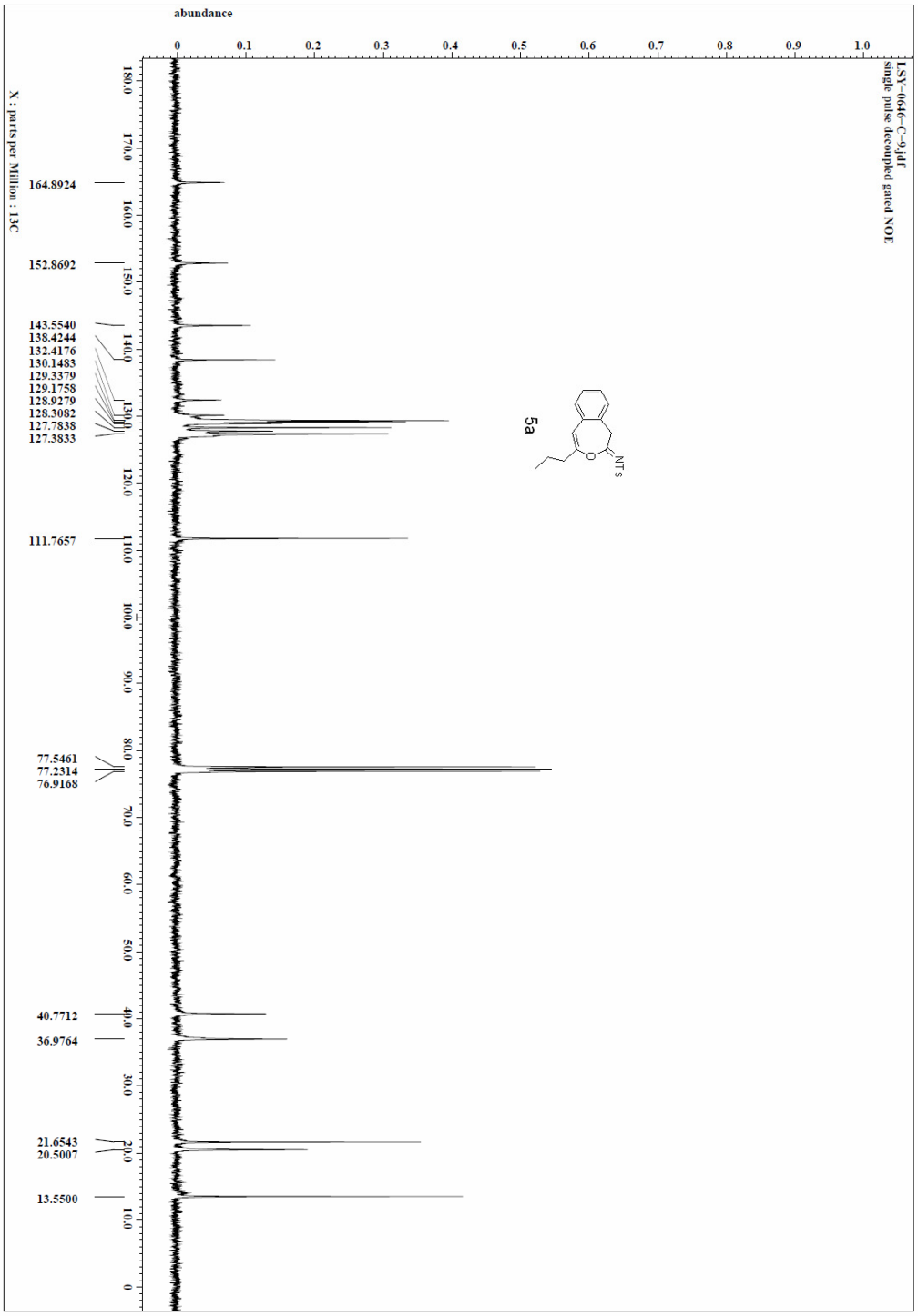

abundance
X : parts per Million : 1H
LSY-0747-6A-H-7.jdf
single_pulse
7.8859
7.8652
7.7782
7.1619
7.0256
7.0061
6.9443
6.9259
5.4975
4.4276
3.8858
3.8217
3.7999
3.7587
3.4276
3.4207
3.4024
1.8216
1.8033
1.7254
1.7082
1.0564
0.9167
0.8995
0.8823
0.7380
0.7197
0.7025
20.02
10.77
10.11
50.41
10
10.19
30.71
30.11
10.95
20.51
20.29
30.61
30.67
4n
OMe

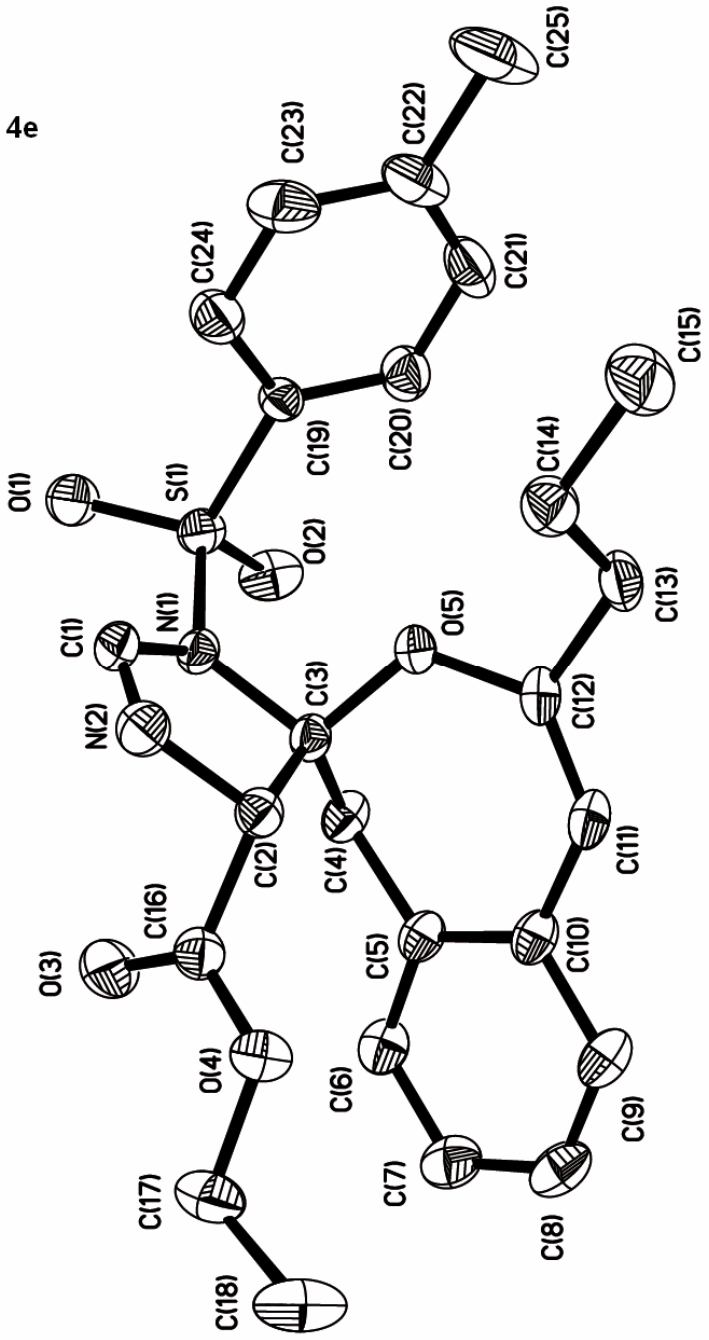

abundance
LSY-0747-6A-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
167.4762
163.8150
151.9349
149.3605
134.3626
131.5213
131.4164
129.7765
129.2330
127.3452
125.4001
114.3972
103.6422
97.7498
77.5175
77.2028
76.8786
61.9666
55.8740
55.8454
42.6018
38.7308
20.9202
13.5309
13.4641
4h
OMe
77.5175
77.2314
77.2028
76.8786

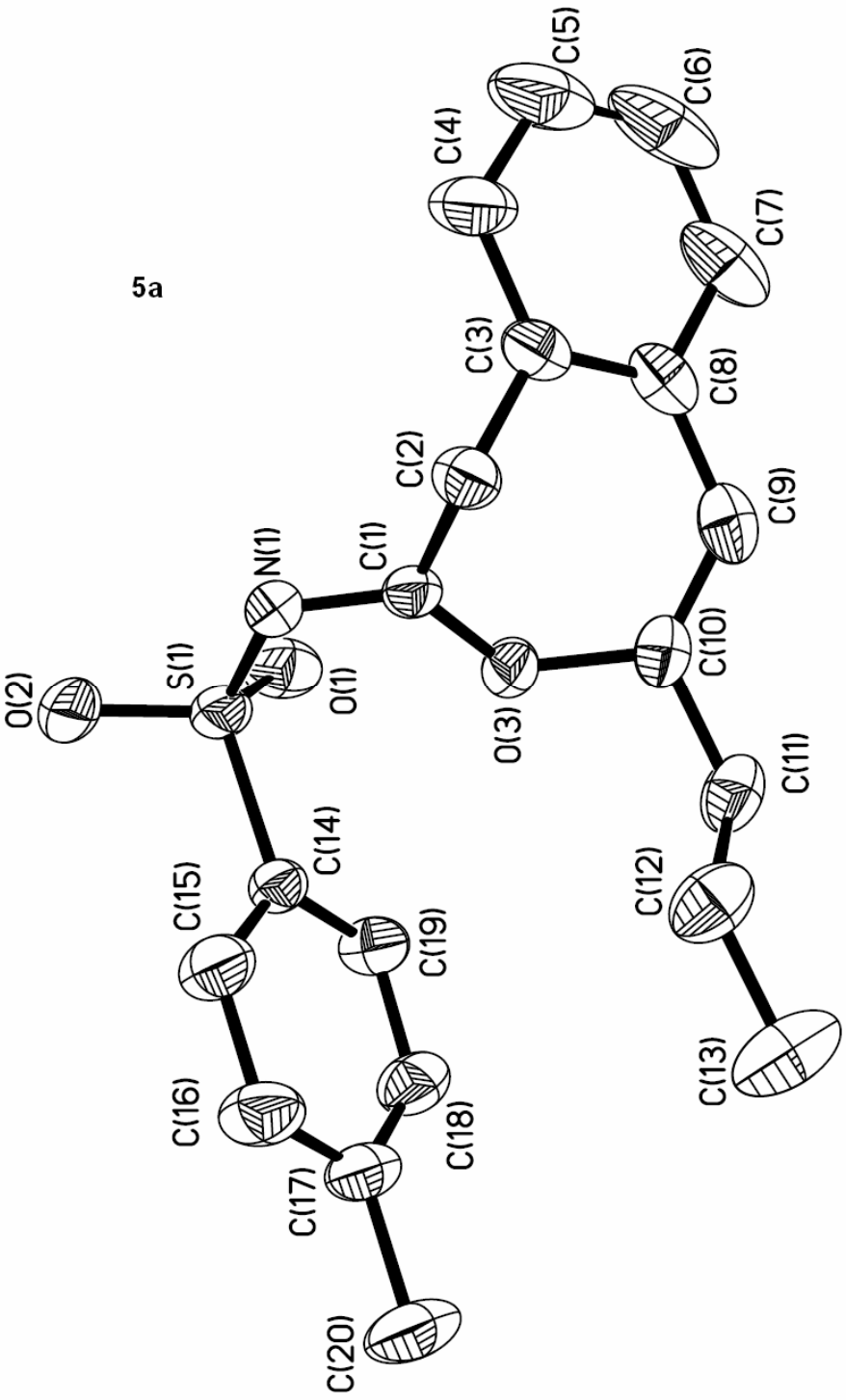

abundance
X : parts per Million : 1H
LSY-074-7A-H-7.jdf
single_pulse
8.4253
8.4069
8.1641
8.1458
7.8101
7.1858
7.0495
7.0312
6.9487
6.9304
5.5157
4.4653
3.8112
3.7860
3.7471
3.4275
3.4195
3.4011
1.7791
1.7608
1.5867
1.5706
1.5523
0.9200
0.9028
0.8845
0.6898
0.6714
0.6531
20.78
20.91
10.46
10.74
20.33
10.8
10
10.63
30.32
10.51
10.45
10.2
20.03
30.33
30.58
40


abundance
LSY-074-7A-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
167.1902
150.8384
150.5809
148.7408
146.3381
134.0289
131.4451
131.0827
129.4809
128.8707
127.5740
125.7529
124.4753
104.2524
97.9215
77.4698
77.1456
76.8310
67.1534
62.2431
42.5541
38.4638
20.8820
13.4260
40
77.4698
77.2886
77.1456
76.8310


abundance
0
1.0
2.0
3.0
4.0
X : parts per Million : 1H
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.8024
7.7841
7.3156
7.2526
7.2343
7.1781
6.2342
3.5584
2.3842
2.3247
1.5091
0.9421
20.65
20.16
20.08
20.68
10
20.27
52.51
20.52
30.03
5a
NTs
LSY-0809-SH-7.jdf
single_pulse


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
X : parts per Million : 13C
164.8924
152.8692
143.5540
138.4244
132.4176
130.1483
129.3379
129.1758
128.9279
128.3082
127.7838
127.3833
111.7657
77.5461
77.2314
76.9168
40.7712
36.9764
21.6543
20.5007
13.5500
5a
NTs
1SY-0646-C-9.jdf
single pulse decoupled gated NOE


4e
C(25)
C(22)
C(23)
C(24)
C(21)
C(15)
C(19)
C(20)
C(14)
O(1)
S(1)
O(2)
C(13)
C(1)
N(1)
O(5)
C(3)
C(12)
N(2)
C(2)
C(4)
C(11)
C(16)
C(5)
C(10)
O(3)
O(4)
C(6)
C(9)
C(7)
C(17)
C(8)
C(18)


5a
C(5)
C(6)
C(4)
C(7)
C(3)
C(8)
C(2)
C(9)
N(1)
C(1)
S(1)
C(10)
O(2)
O(1)
O(3)
C(11)
C(14)
C(12)
C(15)
C(19)
C(13)
C(16)
C(18)
C(17)
C(20)