Generation of 6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxides via a palladium-catalyzed reaction of 2-(2-alkynyl)benzenesulfonamide

Huanhuan Wang,[a] Yong Luo,[a] Xiufeng Hou,*[,a] and Jie Wu*[,ab]

[a] *Department of Chemistry, Fudan University, 220 Handan Road, Shanghai 200433, China*

[b] *State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Road, Shanghai 200032, China*

jie_wu@fudan.edu.cn

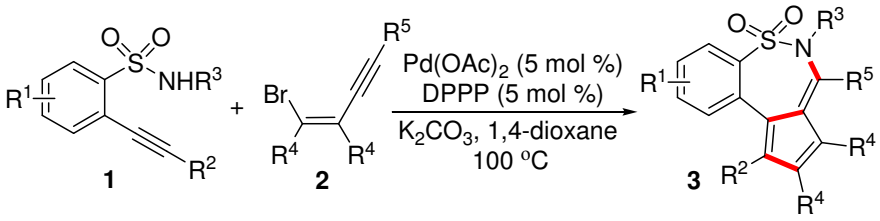
Supporting Information

1. General experimental methods (S2).
2. General experimental procedure and characterization data (S2-S10).
3. ^{1}H and ^{13}C NMR spectra of compound **3** (S11-S40).

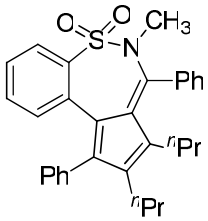
General Materials and Methods:

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.

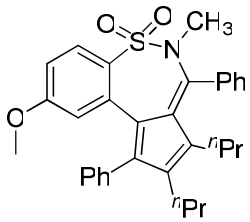
General procedure of the synthesis of 6H-benzo[f]cyclopenta[d][1,2]thiazepine 5,5-dioxides ***3*** *via a palladium-catalyzed reaction of 2-(2-alkynyl)benzenesulfonamide* ***1*** *with 2-alkynylvinyl bromide* ***2***



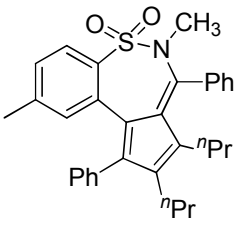
2-(2-Alkynyl)benzenesulfonamide **1** (0.30 mmol) was added to a mixture of $Pd(OAc)_2$ (5 mol%), DPPP (5 mol %), K_2CO_3 (0.60 mmol), and 2-alkynylvinyl bromide **2** (0.36 mmol) in 1,4-dioxane (2.0 mL). The mixture was stirred under 100 °C. After completion of the reaction as indicated by TLC (~12 hours), the mixture was cooled and diluted by EtOAc (10 mL), washed with saturated brine (2 × 10 mL), and dried by anhydrous Na_2SO_4. Evaporation of the solvent followed by purification on silica gel provided the product **3**.



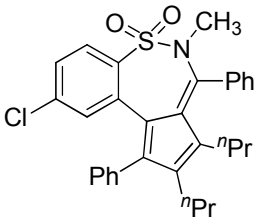
6-Methyl-7,10-diphenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3a**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.43 (t, *J* = 7.3 Hz, 3H), 0.73 (t, *J* = 6.9 Hz, 3H), 1.13-1.26 (m, 4H), 1.43-1.56 (m, 1H), 1.87-1.92 (m, 1H), 2.16-2.24 (m,1H), 2.29-2.36 (m, 2H), 2.68 (s, 3H), 7.02 (d, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 7.8 Hz, 1H), 7.18-7.25 (m, 4H), 7.39-7.44 (m, 3H), 7.50-7.56 (m, 2H), 7.77 (d, *J* = 6.4 Hz, 1H), 7.90 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 14.5, 23.5, 25.5, 28.5, 29.0, 36.9, 125.3, 126.3, 127.0, 127.6, 128.3, 128.5, 128.8, 130.0, 130.6, 131.5, 133.2, 133.7, 134.0, 136.4, 136.0, 137.8, 142.6, 146.1, 146.5; HRMS (ESI) calcd for $C_{31}H_{31}NO_2S$: 504.1968 ($M + Na^+$), found: 504.1970.



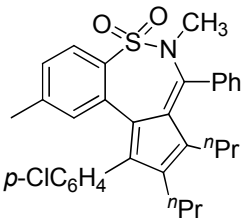
2-Methoxy-6-methyl-7,10-diphenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3b**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.43 (t, *J* = 6.9 Hz, 3H), 0.73 (t, *J* = 7.3 Hz, 3H), 1.14-1.27 (m, 4H), 1.48-1.53 (m, 1H), 1.91-1.96 (m, 1H), 2.20-2.26 (m, 1H), 2.30-2.36 (m, 1H), 2.70 (s, 3H), 3.25 (s, 3H), 6.50 (s, 1H), 6.71 (d, *J* = 8.2 Hz, 2H), 7.26-7.54 (m, 9H), 7.80 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 14.5, 23.6, 25.6, 28.5, 29.0, 36.9, 55.1, 113.6, 116.7, 126.9, 127.6, 128.2, 128.3, 128.7, 128.8, 130.1, 130.6, 132.8, 133.7, 134.1, 135.8, 136.5, 138.4, 142.3, 146.0, 147.1, 161.6; HRMS (ESI) calcd for $C_{32}H_{33}NO_3S$: 534.2073 ($M + Na^+$), found: 534.2077.



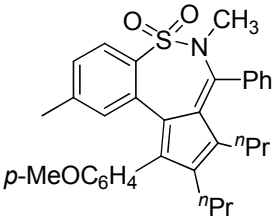
2,6-Dimethyl-7,10-diphenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3c**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.42 (t, *J* = 7.3 Hz, 3H), 0.72 (t, *J* = 7.3 Hz, 3H), 1.11-1.27 (m, 4H), 1.47-1.52 (m, 1H), 1.87-1.93 (m, 1H), 1.97 (s, 3H), 2.18-2.26 (m, 1H), 2.29-2.36 (m, 1H), 2.67 (s, 3H), 6.78 (s, 1H), 6.99 (d, *J* = 7.8 Hz, 2H), 7.23-7.52 (m, 9H), 7.77 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 14.5, 21.3, 23.6, 25.5, 28.5, 29.0, 36.8, 126.2, 126.4 126.9, 127.6, 128.2, 128.4, 128.8, 130.0, 130.5, 133.2, 133.6, 133.7, 133.9, 134.4, 136.5, 138.0, 142.0, 142.5, 145.9, 146.5; HRMS (ESI) calcd for $C_{32}H_{33}NO_2S$: 518.2124 (M + Na^+), found: 518.2142.



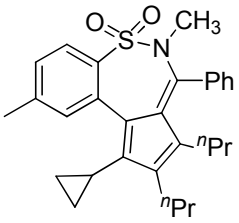
2-Chloro-6-methyl-7,10-diphenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3d**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.43 (t, *J* = 7.3 Hz, 3H), 0.74 (t, *J* = 7.3 Hz, 3H), 1.10-1.29 (m, 4H), 1.48-1.54 (m, 1H), 1.89-1.94 (m, 1H), 2.18-2.27 (m, 1H), 2.28-2.36 (m, 1H), 2.69 (s, 3H), 6.94 (s, 1H), 7.14 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 6.4 Hz, 2H), 7.40-7.41 (m, 3H), 7.52-7.54 (m, 2H), 7.74-7.76 (m, 1H), 7.81 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 14.5, 23.5, 25.5, 28.4, 29.0, 36.9, 125.6, 126.1 127.4, 127.7, 128.4, 128.7, 128.9, 129.9, 130.7, 133.1, 133.3, 133.7, 134.6, 134.8, 135.6, 136.1, 137.2, 137.8, 142.7, 146.7, 147.3; HRMS (ESI) calcd for $C_{31}H_{30}ClNO_2S$: 538.1578 (M + Na^+), found:538.1569.



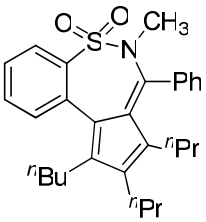
10-(4-Chlorophenyl)-2,6-dimethyl-7-phenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3e**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.42 (t, *J* = 6.9 Hz, 3H), 0.74 (t, *J* = 7.3 Hz, 3H), 1.09-1.25 (m, 4H), 1.42-1.53 (m, 1H), 1.85-1.92 (m, 1H), 2.03 (s, 3H), 2.17-2.24 (m, 1H), 2.26-2.34 (m, 1H), 2.67 (s, 3H), 6.78 (s, 1H), 7.02 (d, *J* = 7.8 Hz, 2H), 7.25-7.39 (m, 5H), 7.49-7.54 (m, 2H),7.74-7.79 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 14.5, 21.4, 23.6, 25.5, 28.5, 29.0, 36.9, 126.4, 126.5 128.0, 128.3, 128.6, 128.9, 130.0, 130.6, 132.7, 133.4, 133.7, 133.9, 134.0, 134.3, 136.3, 136.6, 141.9, 142.2, 144.3 147.1; HRMS (ESI) calcd for $C_{32}H_{32}ClNO_2S$: 552.1734 ($M + Na^+$), found: 552.1734.



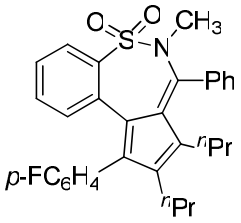
10-(4-Methoxyphenyl)-2,6-dimethyl-7-phenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3f**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.42 (t, *J* = 7.4 Hz, 3H), 0.74 (t, *J* = 7.3 Hz, 3H), 1.10-1.25 (m, 4H), 1.47-1.54 (m, 1H), 1.86-1.92 (m, 1H), 2.01 (s, 3H), 2.18-2.25 (m, 1H), 2.29-2.38 (m, 1H), 2.66 (s, 3H), 6.83 (s, 1H), 6.99 (d, *J* = 7.8 Hz, 2H), 7.35-7.42 (m, 2H), 7.47-7.53 (m, 2H), 7.75-7.78 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 14.6, 21.5, 23.6, 25.5, 28.6, 29.0, 36.8, 55.3, 113.9, 126.2, 126.4 127.3, 128.2, 128.8, 130.0, 130.1, 130.5, 133.3, 133.6, 133.7, 133.8, 133.9, 134.3, 136.6, 136.6, 142.0, 142.7, 145.6 146.2, 158.7; HRMS (ESI) calcd for $C_{33}H_{35}NO_3S$: 548.2230 ($M + Na^+$), found: 548.2239.



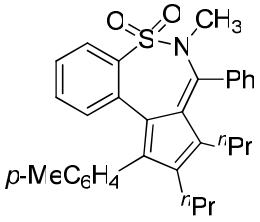
10-Cyclopropyl-2,6-dimethyl-7-phenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3g**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ -1.20- -0.15 (m, 1H), 0.37 (t, *J* = 7.3 Hz, 3H), 0.40-0.45 (m, 1H), 0.60-0.66 (m, 1H), 0.92-1.77 (m, 6H), 1.47-1.54 (m, 2H), 1.66-1.80 (m, 3H), 2.32-2.40 (m, 1H), 2.45 (s, 3H), 2.50-2.58 (m, 1H), 2.60 (s, 3H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.33-7.36 (m, 2H), 7.47 (d, *J* = 6.9 Hz, 2H), 7.65-7.69 (m, 2H), 7.8 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.8, 10.1, 10.5, 14.1, 14.8, 21.7, 24.0, 25.3, 28.5, 28.8, 36.6, 126.1, 126.4, 127.3, 128.0, 128.8, 130.0, 130.1, 132.4, 133.0, 133.3, 133.4, 134.1, 136.8, 141.7, 144.5, 144.7, 146.0; HRMS (ESI) calcd for $C_{29}H_{33}NO_2S$: 482.2124 ($M + Na^+$), found: 482.2132.



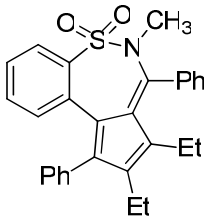
10-Butyl-6-methyl-7-phenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3h**): Dark red oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.39 (t, *J* = 6.9 Hz, 3H) 0.90 (t, *J* = 7.3 Hz, 3H), 0.98 (t, *J* = 7.3 Hz, 3H), 1.05-1.12 (m, 2H), 1.36-1.42 (m, 2H), 1.49-1.58 (m, 5H), 1.76-1.82 (m, 1H), 2.30 (t, *J* = 7.8 Hz, 2H), 2.44-2.49 (m, 2H), 2.61 (s, 3H), 7.34-7.36 (m, 3H), 7.47-7.60 (m, 4H), 7.71 (d, *J* = 6.0 Hz, 1H), 7.96 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 14.0, 14.7, 22.8, 24.2, 25.2, 26.9, 28.3, 28.7, 32.3, 36.4, 125.5, 126.6, 126.7, 127.9, 128.6, 129.8, 130.1, 131.2, 132.0, 133.3, 133.4, 133.7, 134.3, 136.3, 136.5, 142.8, 144.0, 146.1; HRMS (ESI) calcd for $C_{29}H_{35}NO_2S$: 500.2020 ($M + K^+$), found: 500.2001.



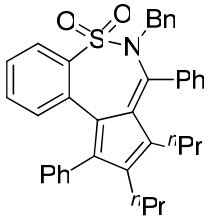
10-(4-Fluorophenyl)-6-methyl-7-phenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3i**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.42 (t, *J* = 6.9 Hz, 3H), 0.73 (t, *J* = 6.8 Hz, 3H), 1.12-1.25 (m, 4H), 1.47-1.54 (m, 1H), 1.86-1.90 (m, 1H), 2.03 (s, 3H), 2.20-2.24 (m, 1H), 2.28-2.34 (m, 1H), 2.68 (s, 3H), 7.00 (d, *J* = 7.8 Hz, 2H), 7.12 (t, *J* = 7.8 Hz, 2H), 7.22 (t, *J* = 7.3 Hz, 2H), 7.36-7.55 (m, 5H), 7.76 (d, *J* = 5.5 Hz, 1H), 7.90 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 14.5, 23.5, 25.5, 28.6, 29.0, 36.9, 115.5 (d, $^{2}J_{CF}$ = 21.0 Hz); 125.7, 126.4 127.8, 128.3, 128.9, 130.0, 130.7, 131.5, 131.7, 133.0, 133.5, 133.6, 133.8 (d, $^{3}J_{CF}$ = 8.6 Hz), 136.3, 136.7, 142.2, 144.8, 146.8, 162.0 (d, $^{1}J_{CF}$ = 245.0 Hz); HRMS (ESI) calcd for $C_{31}H_{30}FNO_2S$: 522.1873 (M + Na^+), found: 522.1874.



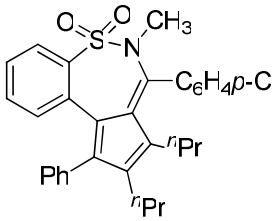
6-Methyl-7-phenyl-8,9-dipropyl-10-(*p*-tolyl)-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3j**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.43 (t, *J* = 7.4 Hz, 3H), 0.74 (t, *J* = 7.3 Hz, 3H), 1.12-1.27 (m, 4H), 1.49-1.56 (m, 1H), 1.87-1.95 (m, 1H), 2.18-2.24 (m,1H), 2.30-2.35 (m, 4H), 2.67 (s, 3H), 7.05-7.12 (m, 5H), 7.20 (t, *J* = 7.8 Hz, 2H), 7.37-7.44 (m, 2H), 7.76-7.54 (m, 2H), 7.77 (d, *J* = 6.0 Hz, 1H), 7.90 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 14.6, 21.4, 23.6, 25.5, 28.5, 29.0, 36.9, 125.4, 126.3 127.4, 128.2, 128.8, 129.2, 130.0, 130.5, 131.6, 133.4, 133.7, 134.2, 134.7, 136.4, 136.5, 136.6, 142.8, 146.1, 146.2; HRMS (ESI) calcd for $C_{32}H_{33}NO_2S$: 518.2124 (M + Na^+), found: 518.2117.



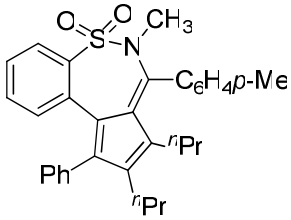
8,9-Diethyl-6-methyl-7,10-diphenyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3k**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.70 (t, *J* = 7.4 Hz, 3H), 0.80 (t, *J* = 6.9 Hz, 3H), 1.60-1.65 (m, 1H), 1.95-2.00 (m, 1H), 2.26-2.38 (m, 2H), 2.68 (s, 3H), 7.02 (d, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 7.3 Hz, 1H), 7.18-7.25 (m, 4H), 7.38-7.44 (m, 3H), 7.51 (d, *J* = 7.3 Hz, 2H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.90 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.8, 16.7, 19.3, 19.6, 36.9, 125.6, 126.3, 127.0, 127.6, 128.2, 128.5, 128.8, 129.9, 130.6, 131.6, 132.9, 133.6, 133.7, 134.0, 134.9, 136.5, 137.7, 143.8, 146.1, 146.4; HRMS (ESI) calcd for $C_{29}H_{27}NO_2S$: 476.1655 ($M + Na^+$), found: 476.1655.



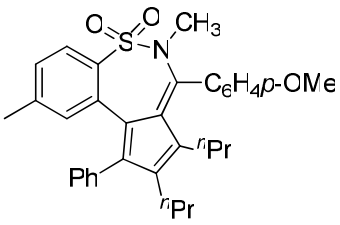
6-Benzyl-7,10-diphenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3l**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.43 (t, *J* = 7.3 Hz, 3H), 0.73 (t, *J* = 6.8 Hz, 3H), 1.13-1.28 (m, 4H), 1.42-1.49 (m, 1H), 1.84-1.91 (m, 1H), 2.18-2.24 (m, 1H), 2.29-2.38 (m, 1H), 4,31 (d, *J* = 16.0 Hz, 2H), 4,82 (d, *J* = 16.0 Hz, 2H), 6.45 (d, *J* = 7.8, 2), 6.80 (t, *J* = 6.8 Hz, 2H), 6.86-6.91 (m, 5H), 7.22-7.26 (m, 2H), 7.32-7.33 (m, 3H), 7.50-7.55 (m, 3H), 7.77 (d, *J* = 6.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 14.6, 23.6, 25.6, 28.5, 29.1, 53.3, 125.5, 126.9 127.2, 127.8, 127.9, 128.2, 128.3, 128.4, 128.6, 130.0, 130.4, 131.1, 133.2, 133.5, 133.7, 133.8, 133.9, 135.3, 136.0, 137.9, 139.2, 142.3, 145.7, 146.1; HRMS (ESI) calcd for $C_{37}H_{35}NO_2S$: 580.2281 ($M + Na^+$), found: 580.2281.



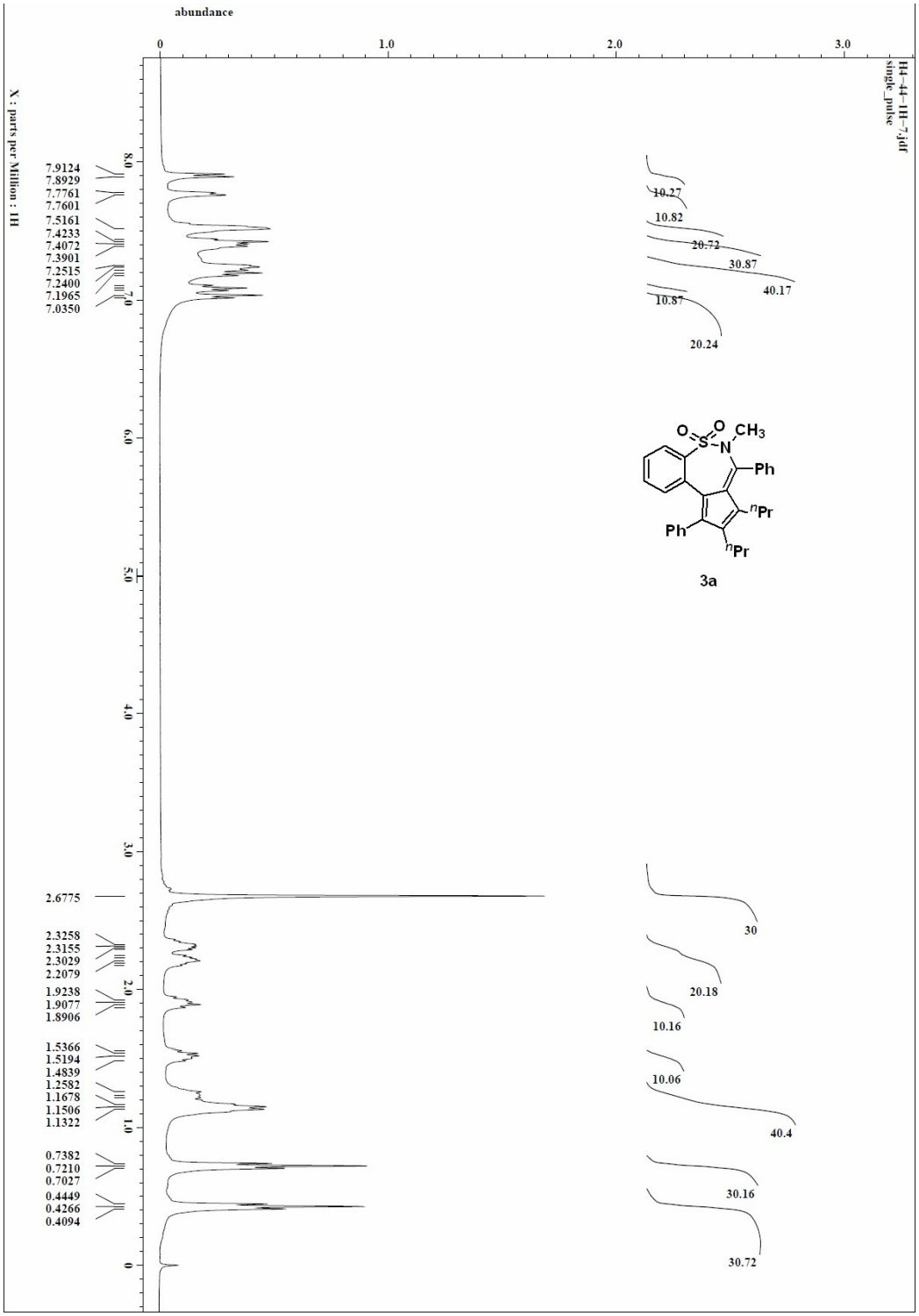
7-(4-Chlorophenyl)-6-methyl-10-phenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3m**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.47 (t, *J* = 6.9 Hz, 3H), 0.72 (t, *J* = 7.3 Hz, 3H), 1.11-1.28 (m, 4H), 1.51-1.59 (m, 1H), 1.91-1.97 (m, 1H), 2.18-2.24 (m,1H), 2.28-2.34 (m, 1H), 2.66 (s, 3H), 7.00 (d, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 7.8 Hz, 1H), 7.18-7.38 (m, 7H), 7.52 (d, *J* = 8.2 Hz, 1H), 7.71 (d, *J* = 8.2 Hz, 1H), 7.89 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 14.5, 23.5, 25.4, 28.5, 29.0, 36.9, 125.7, 126.4, 127.1, 127.6, 128.6, 129.2, 131.3, 131.6, 133.2, 133.6, 133.7, 133.8, 134.8, 134.9, 136.4, 136.9, 137.6, 143.2, 144.9, 146.4; HRMS (ESI) calcd for $C_{31}H_{30}ClNO_2S$: 538.1578 ($M + Na^+$), found: 538.1572.

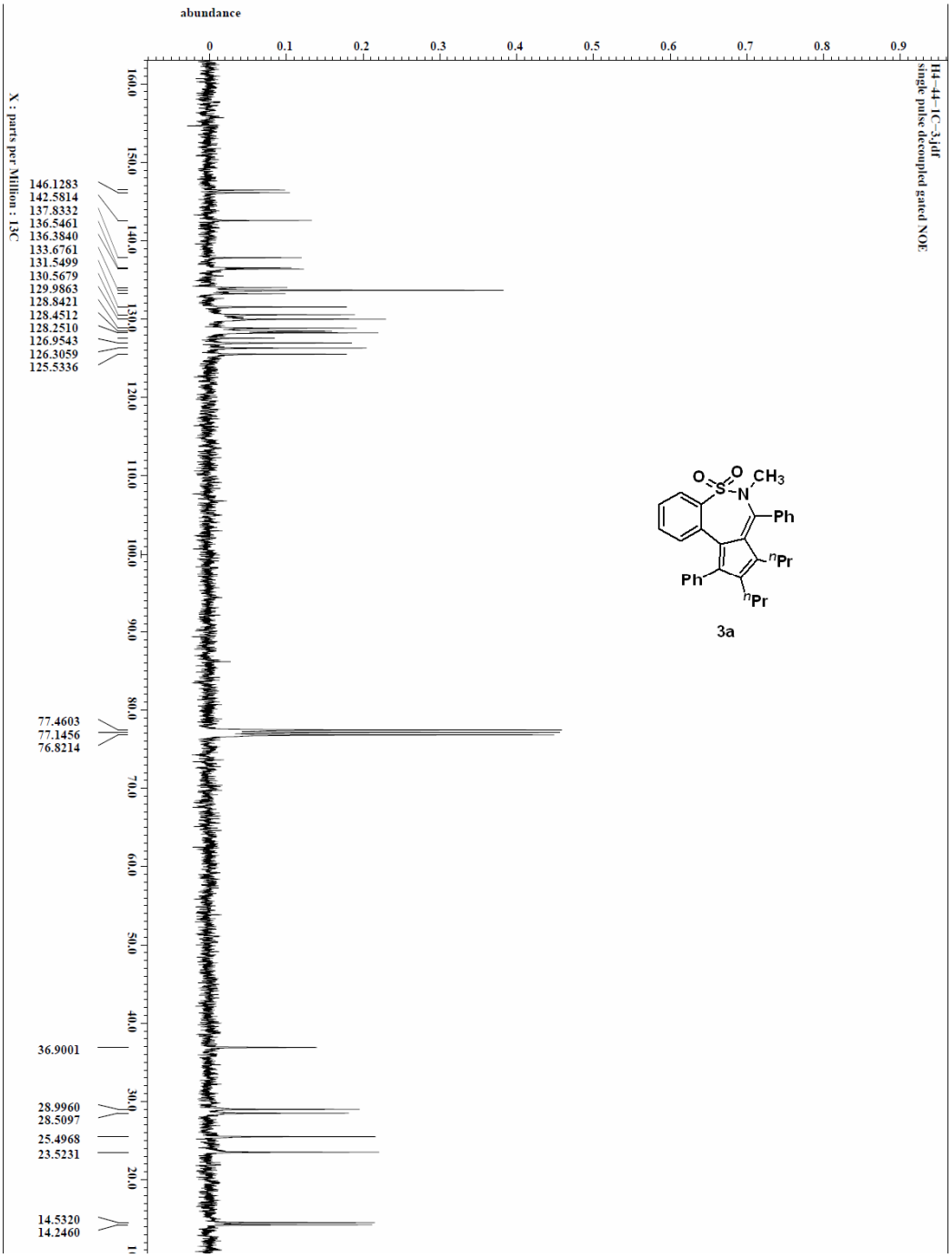


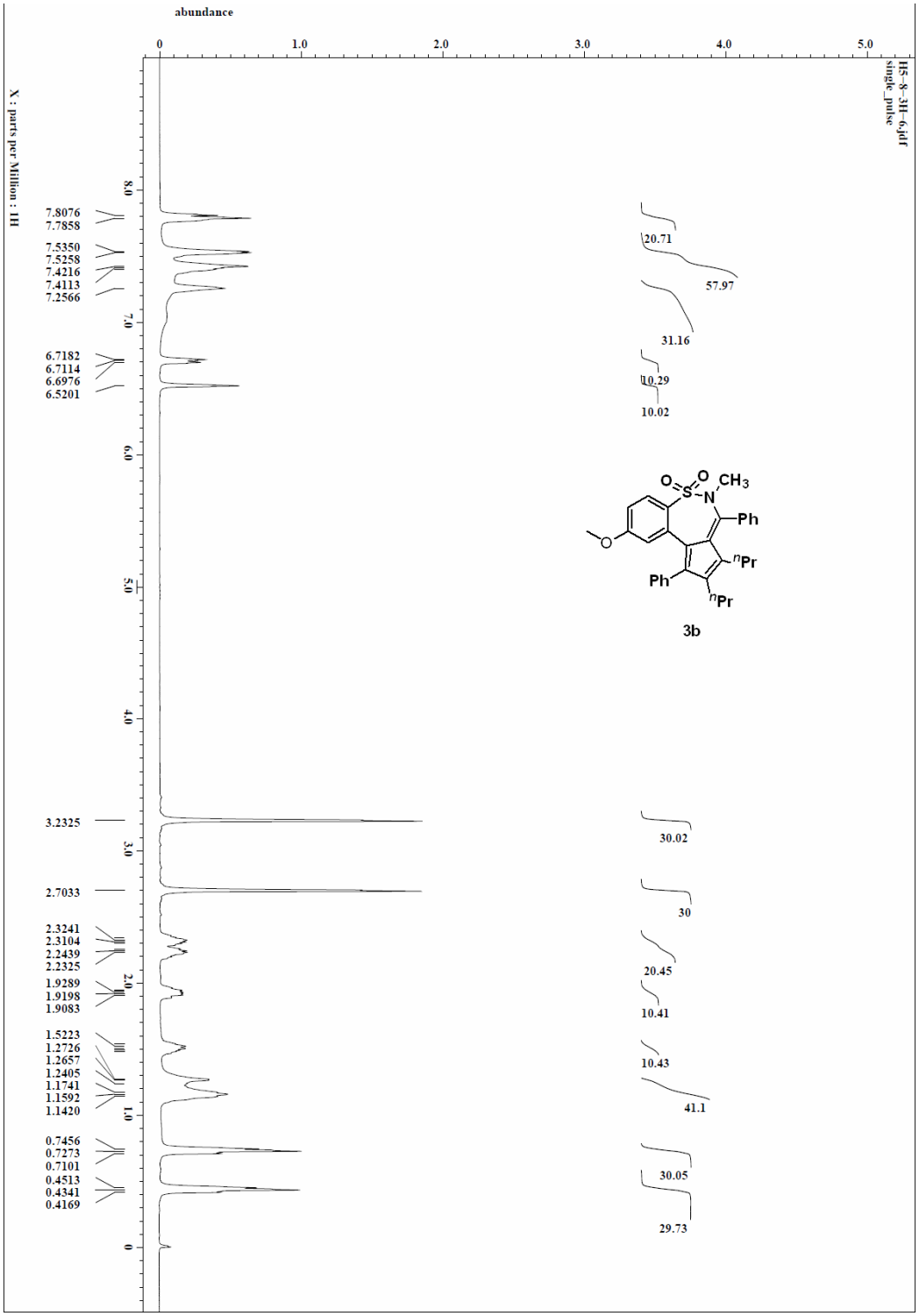
6-Methyl-10-phenyl-8,9-dipropyl-7-(*p*-tolyl)-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3n**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.45 (t, *J* = 6.8 Hz, 3H), 0.73 (t, *J* = 7.3 Hz, 3H), 1.13-1.27 (m, 4H), 1.55-1.61 (m, 1H), 1.90-1.98 (m, 1H), 2.19-2.26 (m, 1H), 2.30-2.38 (m, 1H), 2.45 (s, 3H), 2.69 (s, 3H), 7.01 (d, *J* = 7.8 Hz, 2H), 7.08 (t, *J* = 7.4 Hz, 1H), 7.14-7.26 (m, 4H), 7.31-7.36 (m, 3H), 7.65 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 14.5, 21.6, 23.6, 25.5, 28.5, 29.0, 37.0, 125.4, 126.3, 126.9, 127.5, 128.5, 128.9, 129.6, 129.9, 131.5, 132.9, 133.5, 133.7, 133.8, 134.1, 136.6, 138.0, 141.0, 142.3, 145.8, 146.9; HRMS (ESI) calcd for $C_{32}H_{33}NO_2S$: 518.2124 ($M + Na^+$), found: 518.2127.

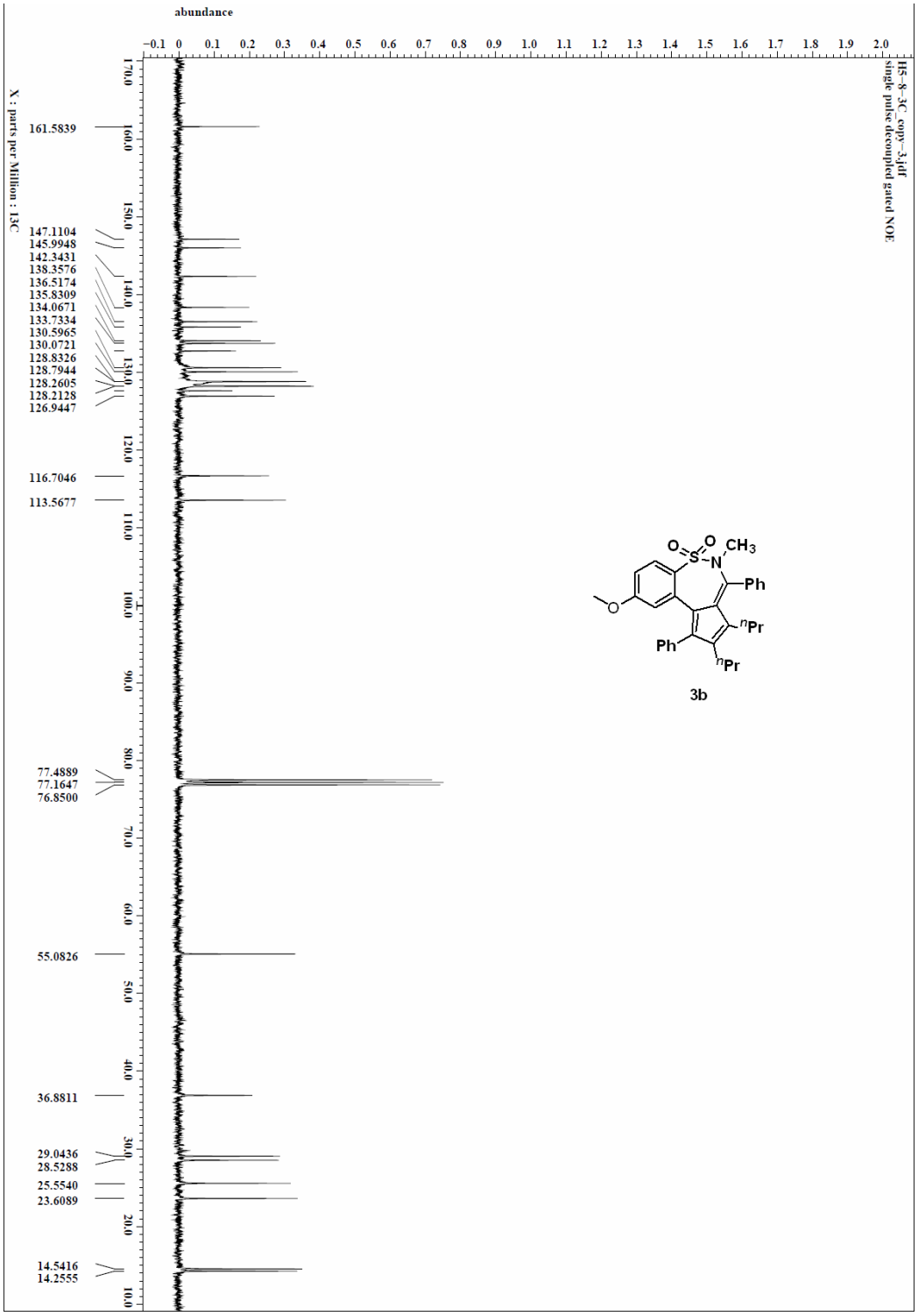


7-(4-Methoxyphenyl)-2,6-dimethyl-10-phenyl-8,9-dipropyl-6*H*-benzo[*f*]cyclopenta[*d*][1,2]thiazepine 5,5-dioxide (**3o**): Dark red solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.47 (t, *J* = 6.8 Hz, 3H), 0.73 (t, *J* = 7.3 Hz, 3H), 1.12-1.25 (m, 4H), 1.56-1.63 (m, 1H), 1.96-2.22 (m, 4H), 2.20-2.26 (m, 1H), 2.30-2.36 (m, 1H), 2.68 (s, 3H), 6.77 (s, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 6.98 (d, *J* = 7.8 Hz, 1H), 7.05 (d, *J* = 8.2 Hz, 1H), 7.23-7.35 (m, 6H), 7.68 (d, *J* = 8.2 Hz, 1H), 7.77 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 14.6, 21.3, 23.7, 25.4, 28.5, 29.0, 37.0, 55.6, 113.4, 114.6, 126.1, 126.4, 126.8, 127.5, 128.3, 128.6, 130.2, 131.5, 132.5, 133.7, 133.8, 134.3, 135.6, 138.1, 141.9, 142.0, 145.4, 146.8; HRMS (ESI) calcd for $C_{33}H_{35}NO_3S$: 548.2230 (M + Na^+), found: 548.2230.

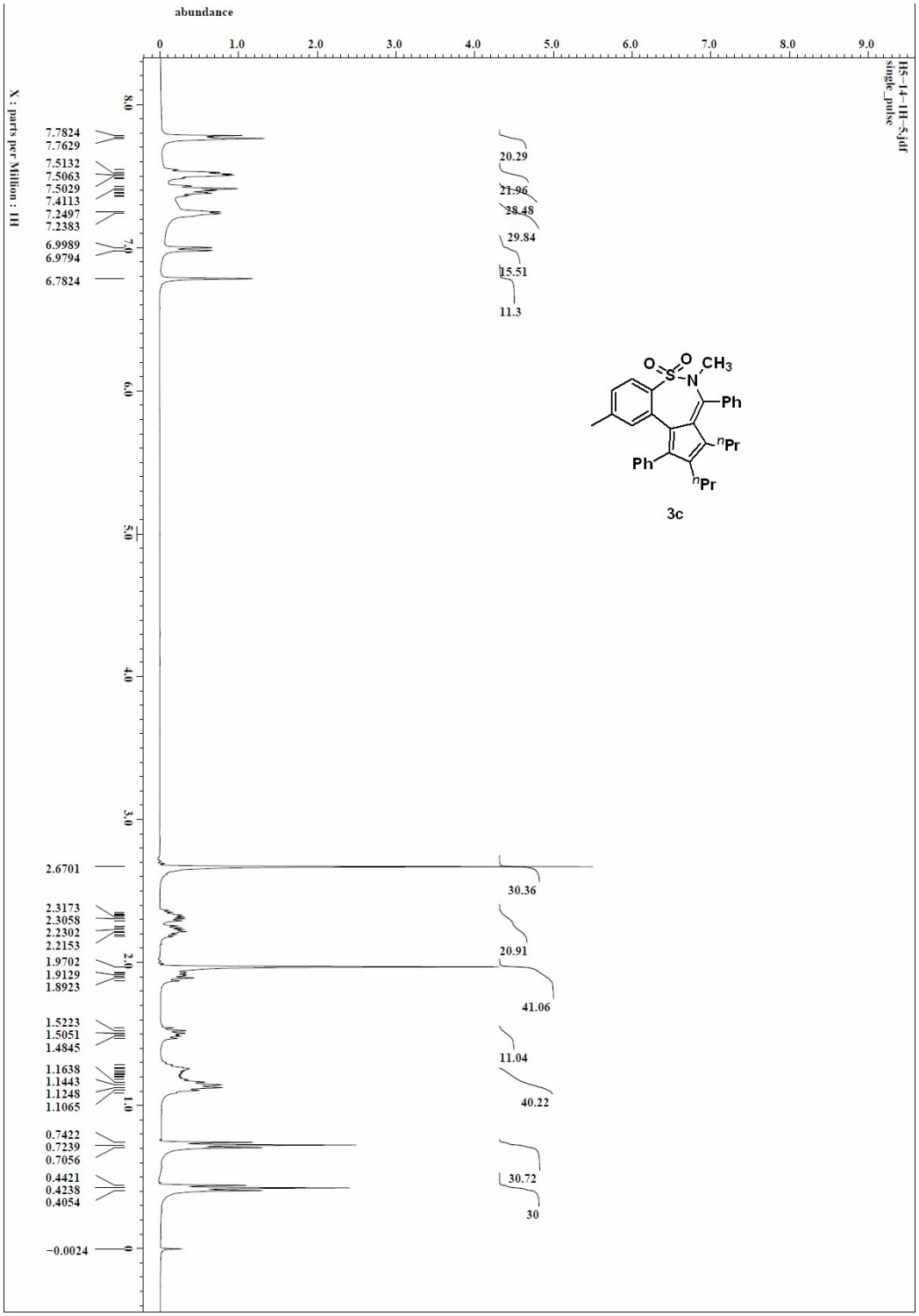

abundance
X : parts per Million : 1H
H4--44--1H--7.jdf
single_pulse
7.9124
7.8929
7.7761
7.7601
7.5161
7.4233
7.4072
7.3901
7.2515
7.2400
7.1965
7.0350
2.6775
2.3258
2.3155
2.3029
2.2079
1.9238
1.9077
1.8906
1.5366
1.5194
1.4839
1.2582
1.1678
1.1506
1.1322
0.7382
0.7210
0.7027
0.4449
0.4266
0.4094
10.27
10.82
20.72
30.87
40.17
10.87
20.24
30
20.18
10.16
10.06
40.4
30.16
30.72
Ph
nPr
Ph
nPr
3a

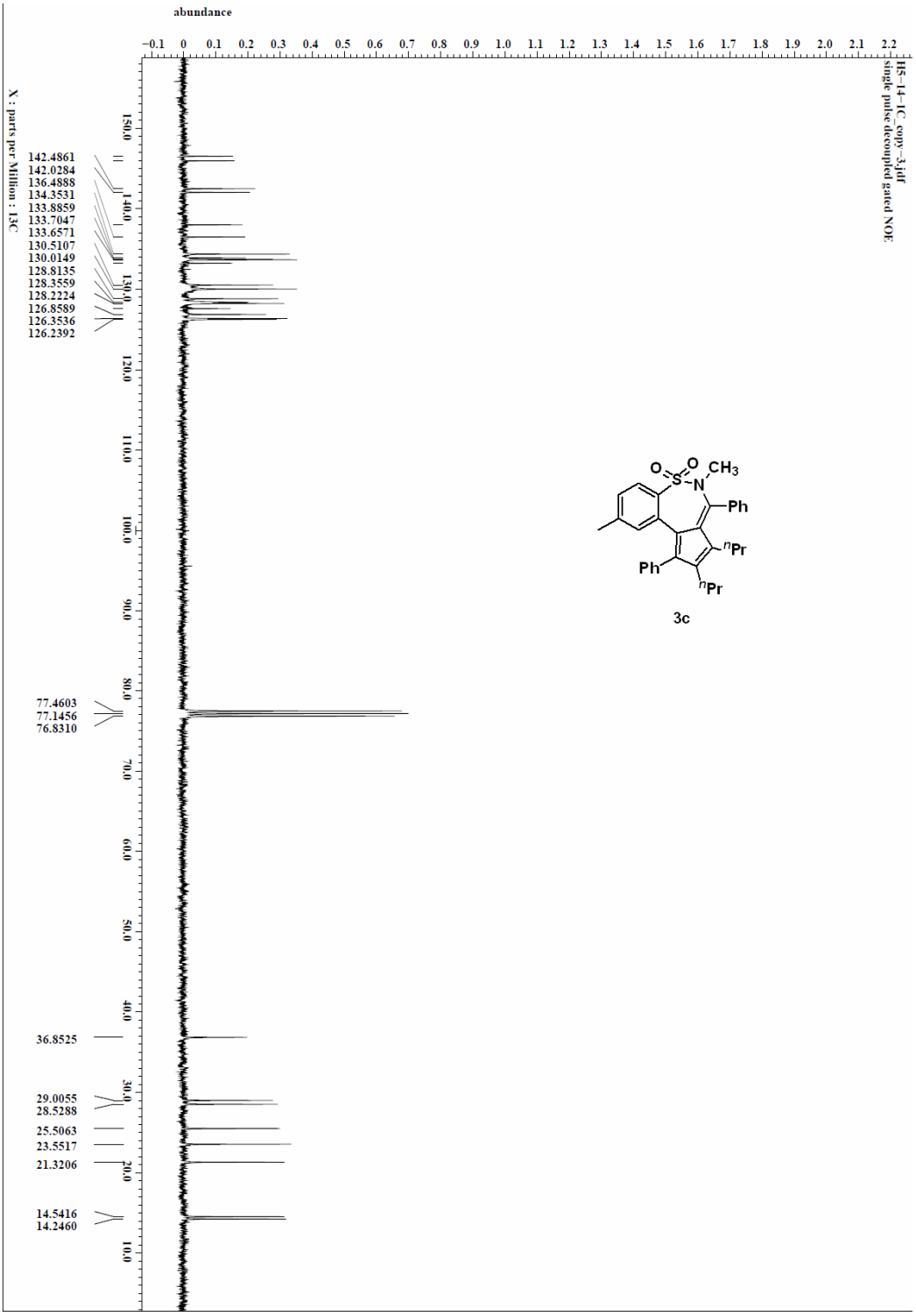

abundance
X : parts per Million : 13C
H4-44-1C-3.jdf
single pulse decoupled gated NOE
146.1283
142.5814
137.8332
136.5461
136.3840
133.6761
131.5499
130.5679
129.9863
128.8421
128.4512
128.2510
126.9543
126.3059
125.5336
77.4603
77.1456
76.8214
36.9001
28.9960
28.5097
25.4968
23.5231
14.5320
14.2460
Ph
nPr
Ph
nPr
3a


abundance
X : parts per Million : 1H
HS-8-3H-6.jdf
single_pulse
7.8076
7.7858
7.5350
7.5258
7.4216
7.4113
7.2566
6.7182
6.7114
6.6976
6.5201
3.2325
2.7033
2.3241
2.3104
2.2439
2.2325
1.9289
1.9198
1.9083
1.5223
1.2726
1.2657
1.2405
1.1741
1.1592
1.1420
0.7456
0.7273
0.7101
0.4513
0.4341
0.4169
20.71
57.97
31.16
10.29
10.02
30.02
30
20.45
10.41
10.43
41.1
30.05
29.73
3b

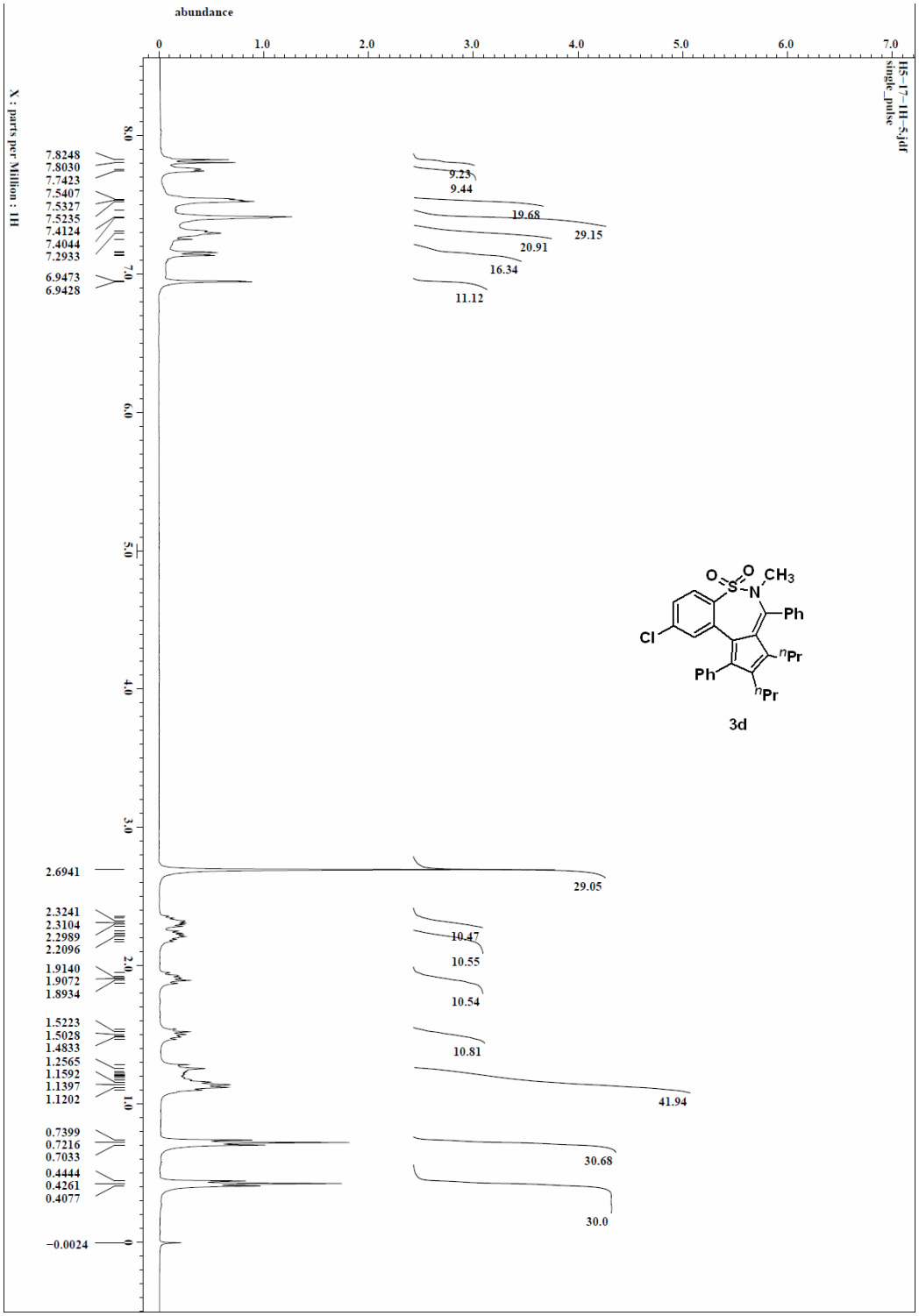

abundance
X : parts per Million : 13C
H5-8-3C_copy-3.jdf
single pulse decoupled gated NOE
161.5839
147.1104
145.9948
142.3431
138.3576
136.5174
135.8309
134.0671
133.7334
130.5965
130.0721
128.8326
128.7944
128.2605
128.2128
126.9447
116.7046
113.5677
77.4889
77.1647
76.8500
55.0826
36.8811
29.0436
28.5288
25.5540
23.6089
14.5416
14.2555
O
S
O
N
CH3
Ph
nPr
Ph
nPr
O

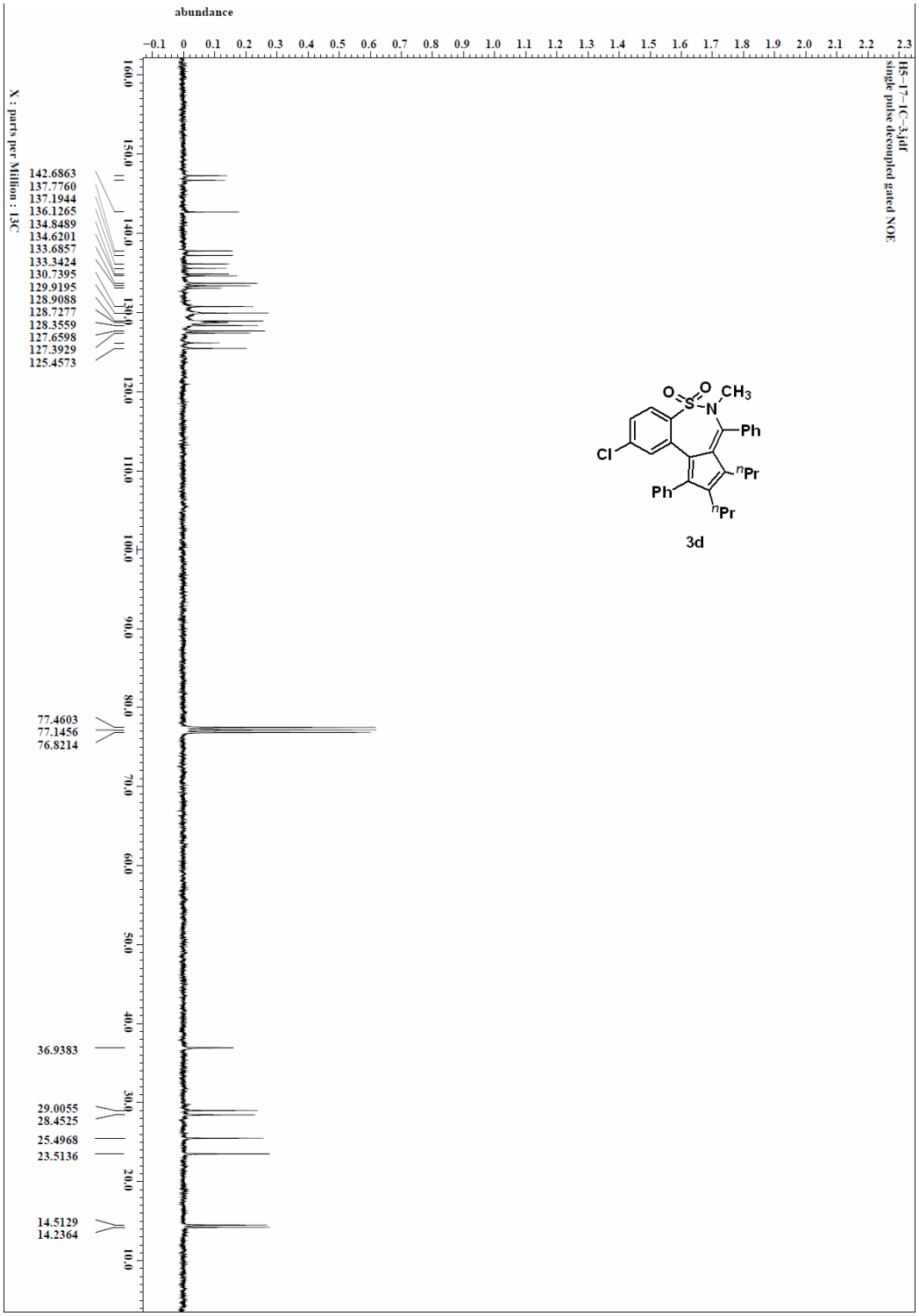
3b


abundance
X : parts per Million : 1H
H5-14-1H-5.jdf
single_pulse
7.7824
7.7629
7.5132
7.5063
7.5029
7.4113
7.2497
7.2383
6.9989
6.9794
6.7824
2.6701
2.3173
2.3058
2.2302
2.2153
1.9702
1.9129
1.8923
1.5223
1.5051
1.4845
1.1638
1.1443
1.1248
1.1065
0.7422
0.7239
0.7056
0.4421
0.4238
0.4054
−0.0024
20.29
21.96
38.48
29.84
15.51
11.3
30.36
20.91
41.06
11.04
40.22
30.72
30
3c

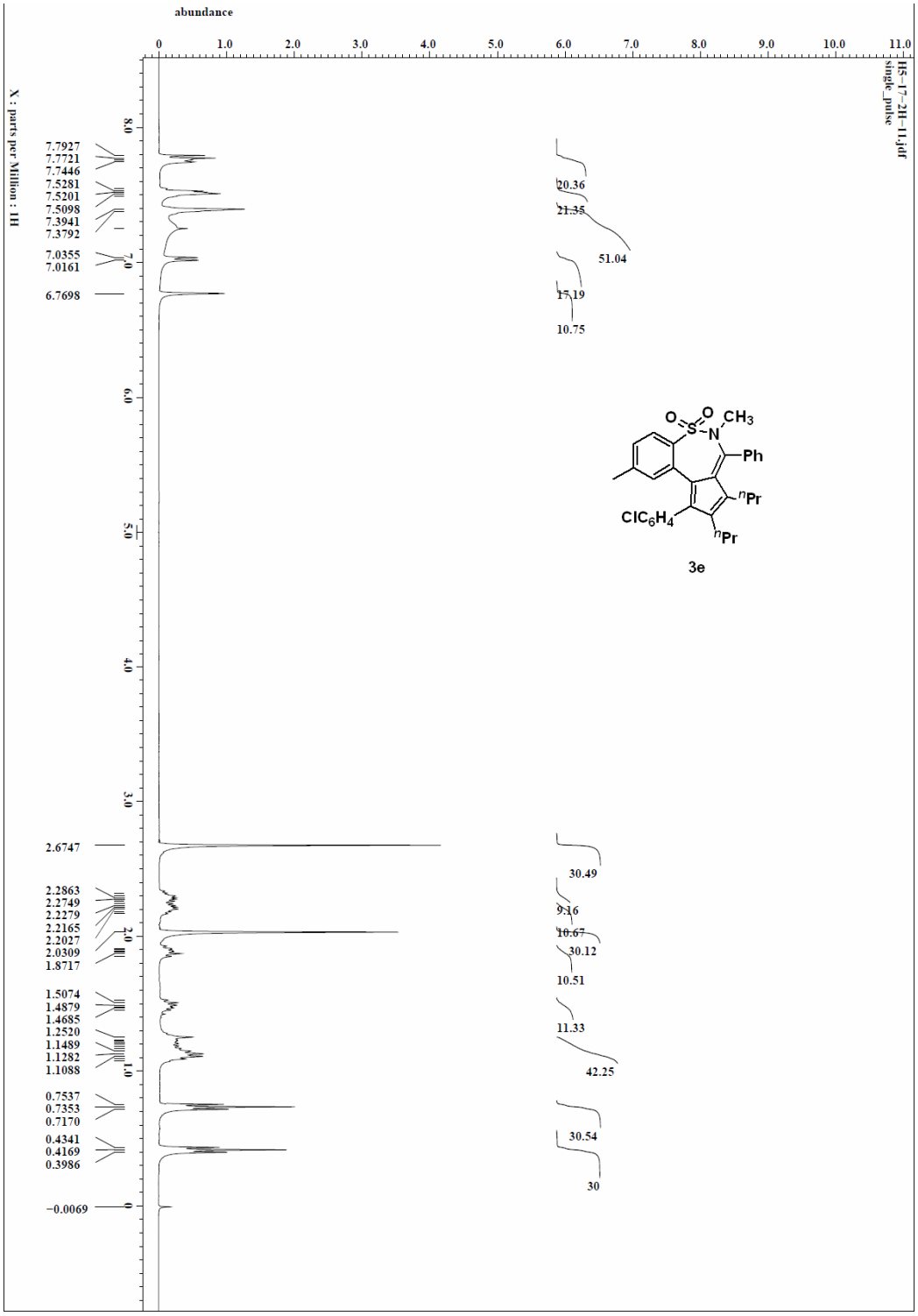

abundance
X : parts per Million : 13C
142.4861
142.0284
136.4888
134.3531
133.8859
133.7047
133.6571
130.5107
130.0149
128.8135
128.3559
128.2224
126.8589
126.3536
126.2392
77.4603
77.1456
76.8310
36.8525
29.0055
28.5288
25.5063
23.5517
21.3206
14.5416
14.2460
H5-14-1C copy-3.jdf
single pulse decoupled gated NOE
O
O
S
N
CH3
Ph
nPr
Ph
nPr

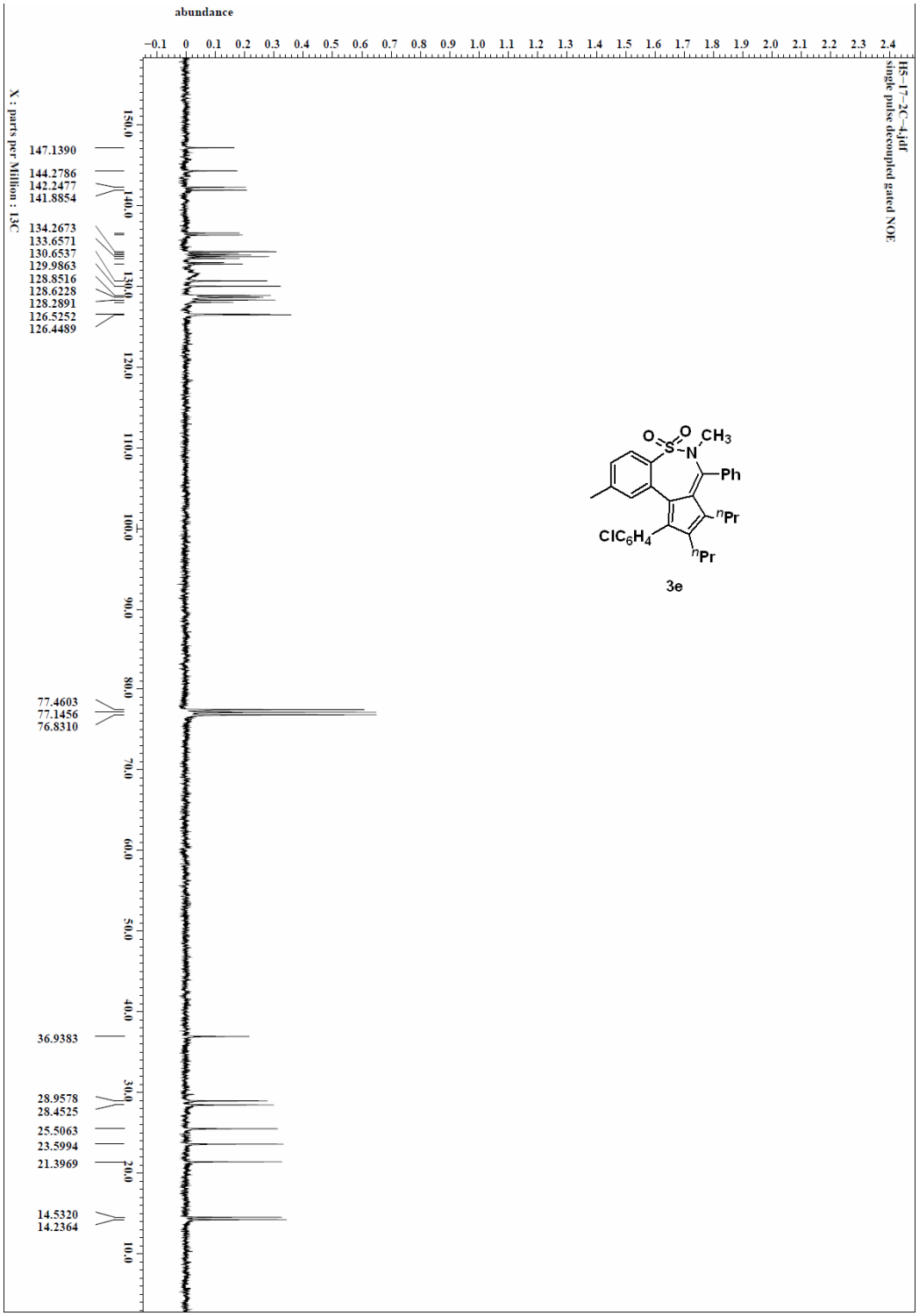
3c

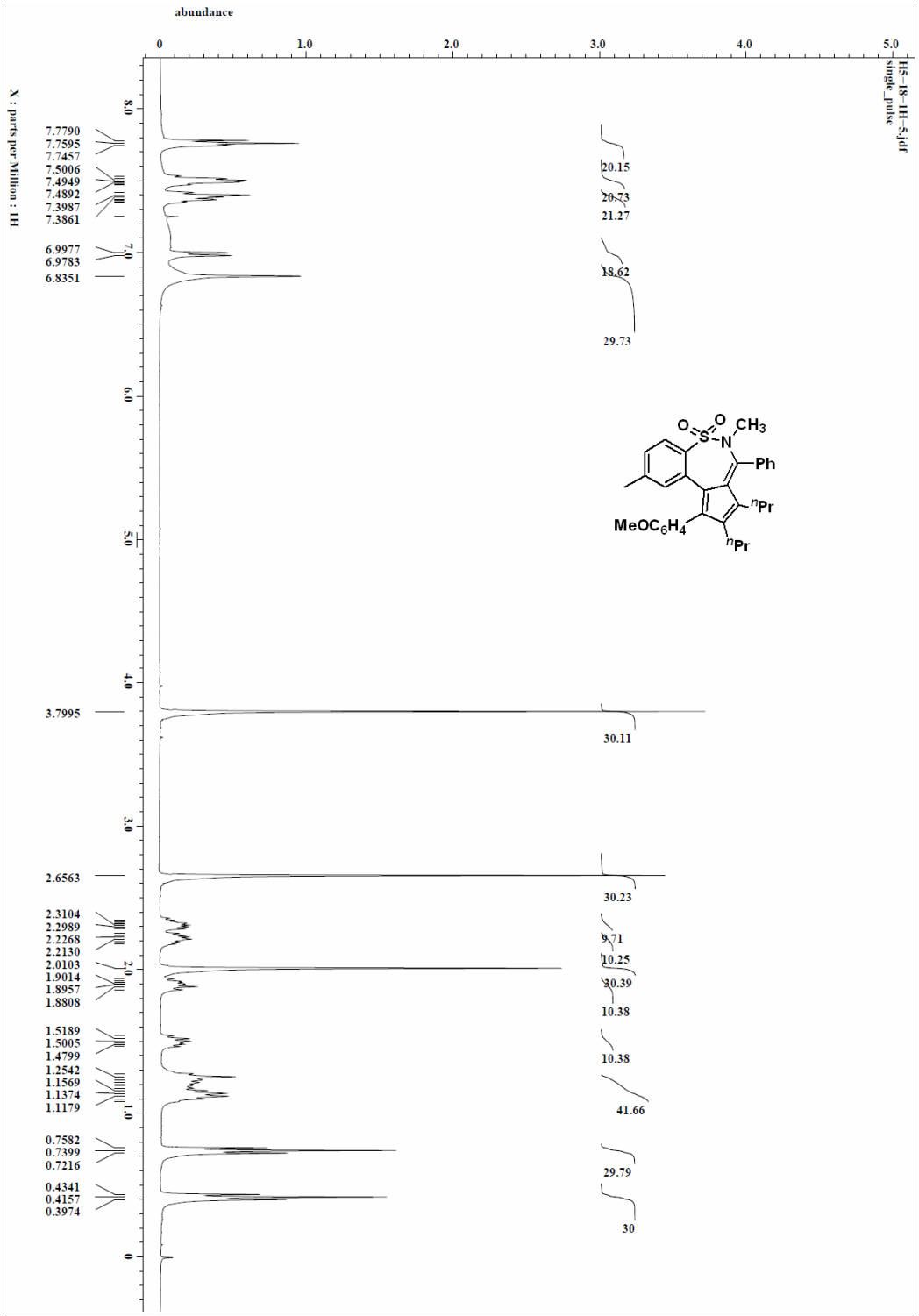

abundance
X : parts per Million : 1H
H5-17-1H-5.jdf
single_pulse
7.8248
7.8030
7.7423
7.5407
7.5327
7.5235
7.4124
7.4044
7.2933
6.9473
6.9428
2.6941
2.3241
2.3104
2.2989
2.2096
1.9140
1.9072
1.8934
1.5223
1.5028
1.4833
1.2565
1.1592
1.1397
1.1202
0.7399
0.7216
0.7033
0.4444
0.4261
0.4077
−0.0024
9.23
9.44
19.68
29.15
20.91
16.34
11.12
29.05
10.47
10.55
10.54
10.81
41.94
30.68
30.0
Cl
Ph
Ph
nPr
nPr
3d

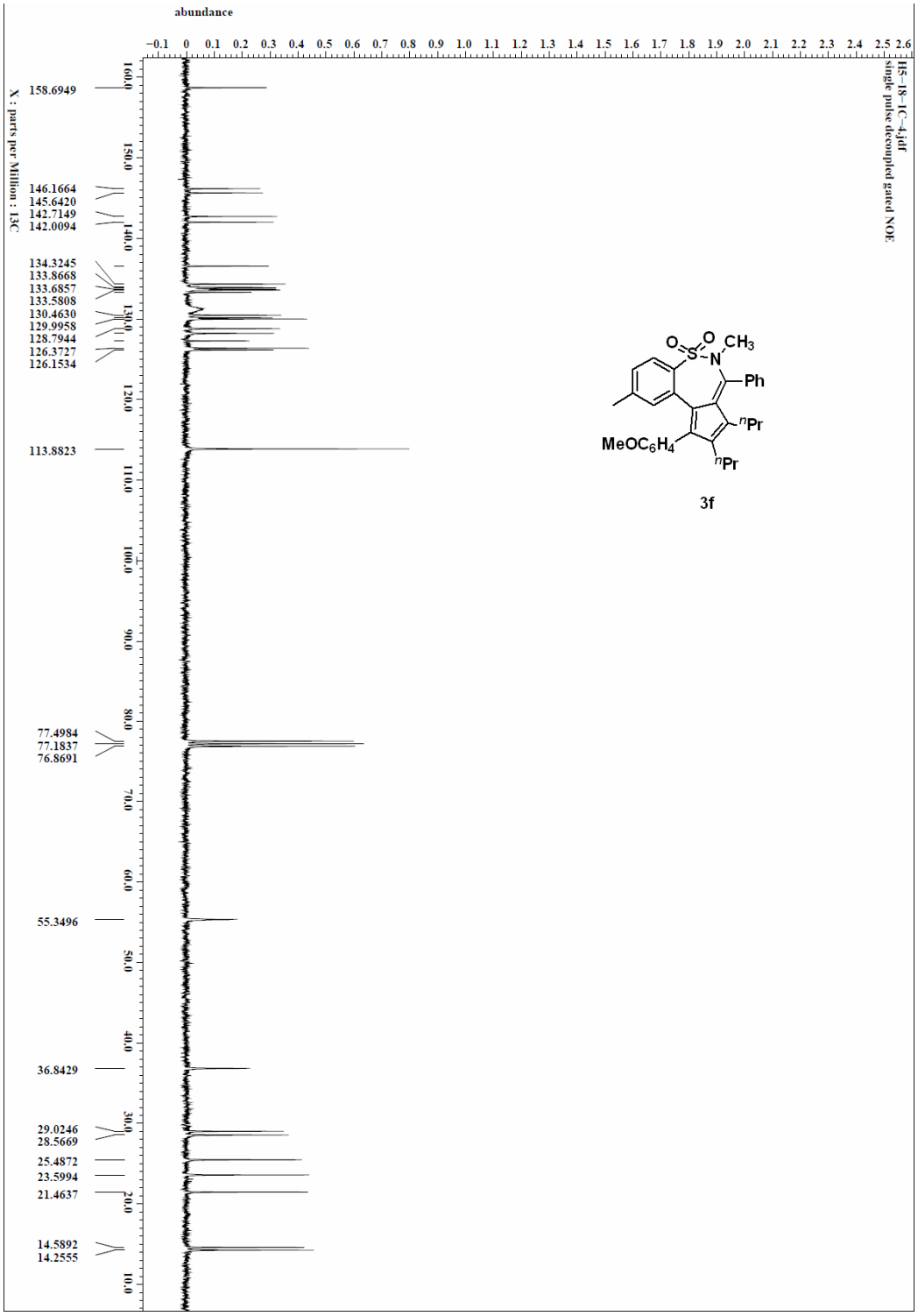

abundance
X : parts per Million : 13C
H5-17-1C-3.jdf
single pulse decoupled gated NOE
142.6863
137.7760
137.1944
136.1265
134.8489
134.6201
133.6857
133.3424
130.7395
129.9195
128.9088
128.7277
128.3559
127.6598
127.3929
125.4573
77.4603
77.1456
76.8214
36.9383
29.0055
28.4525
25.4968
23.5136
14.5129
14.2364
O
O
S
N
CH3
Ph
Cl
nPr
Ph
nPr

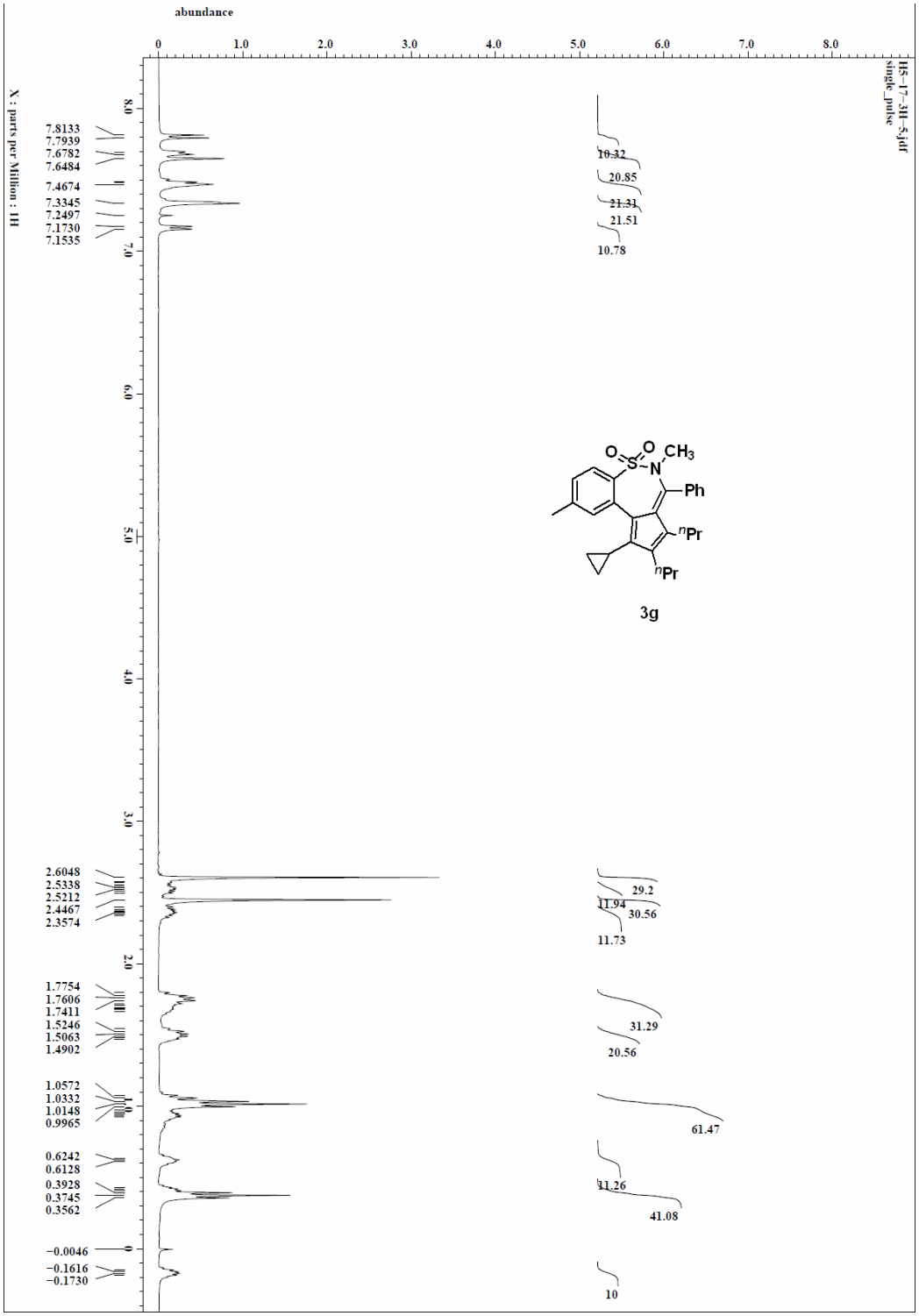
3d

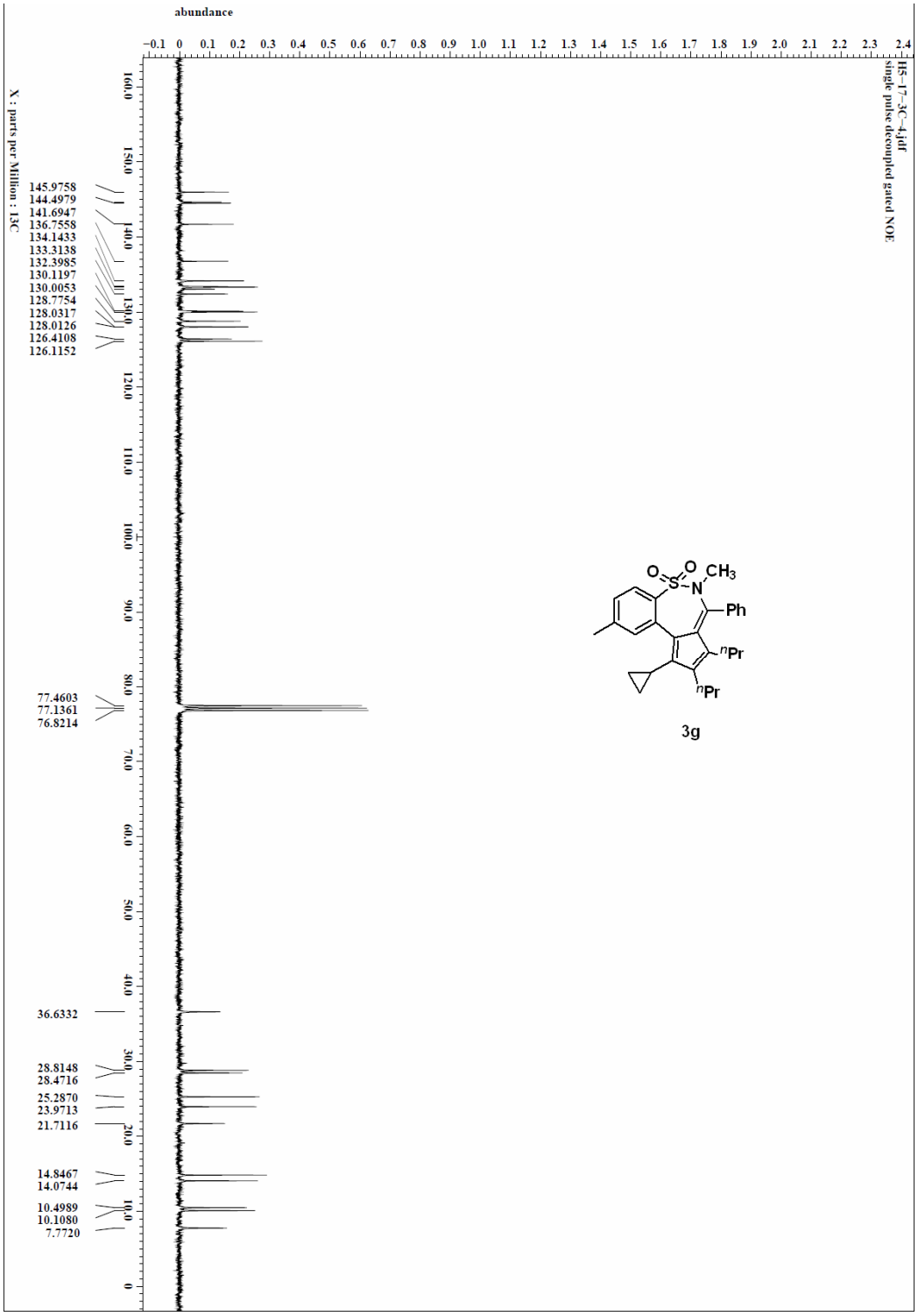

abundance
X : parts per Million : 1H
H5-17-2H-11.jdf
single_pulse
7.7927
7.7721
7.7446
7.5281
7.5201
7.5098
7.3941
7.3792
7.0355
7.0161
6.7698
2.6747
2.2863
2.2749
2.2279
2.2165
2.2027
2.0309
1.8717
1.5074
1.4879
1.4685
1.2520
1.1489
1.1282
1.1088
0.7537
0.7353
0.7170
0.4341
0.4169
0.3986
−0.0069
20.36
21.35
51.04
17.19
10.75
30.49
9.16
10.67
30.12
10.51
11.33
42.25
30.54
30
CH3
Ph
nPr
ClC6H4
nPr
3e


abundance
−0.1 0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 2.0 2.1 2.2 2.3 2.4
H5−17−2C−4.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
147.1390
144.2786
142.2477
141.8854
134.2673
133.6571
130.6537
129.9863
128.8516
128.6228
128.2891
126.5252
126.4489
77.4603
77.1456
76.8310
36.9383
28.9578
28.4525
25.5063
23.5994
21.3969
14.5320
14.2364
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
O
O
S
N
CH3
Ph
nPr
ClC6H4
nPr
3e

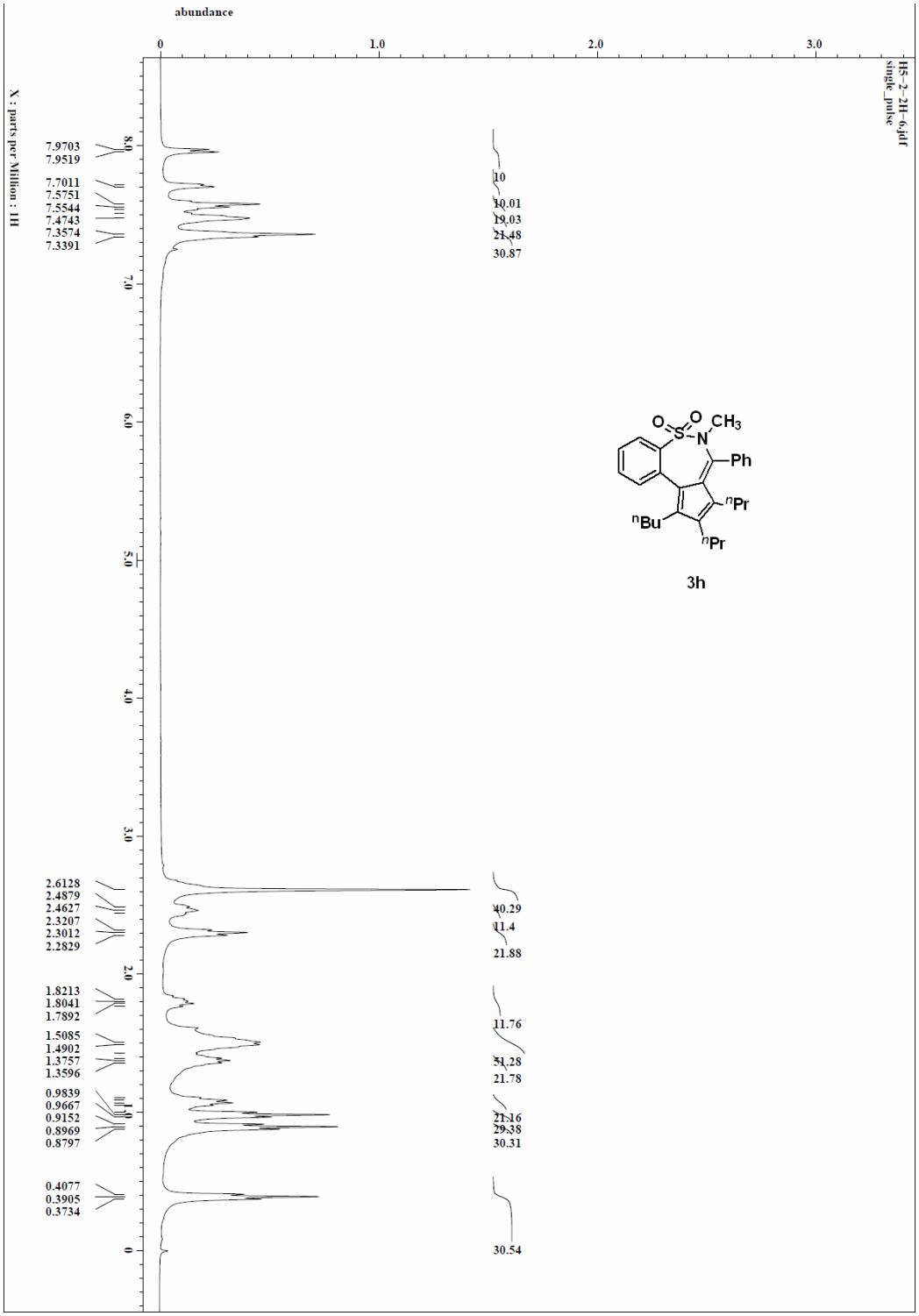

abundance
X : parts per Million : 1H
H5-18-1H-5.jdf
single_pulse
7.7790
7.7595
7.7457
7.5006
7.4949
7.4892
7.3987
7.3861
6.9977
6.9783
6.8351
3.7995
2.6563
2.3104
2.2989
2.2268
2.2130
2.0103
1.9014
1.8957
1.8808
1.5189
1.5005
1.4799
1.2542
1.1569
1.1374
1.1179
0.7582
0.7399
0.7216
0.4341
0.4157
0.3974
20.15
20.73
21.27
18.62
29.73
30.11
30.23
9.71
10.25
30.39
10.38
10.38
41.66
29.79
30
Ph
nPr
MeOC6H4
nPr

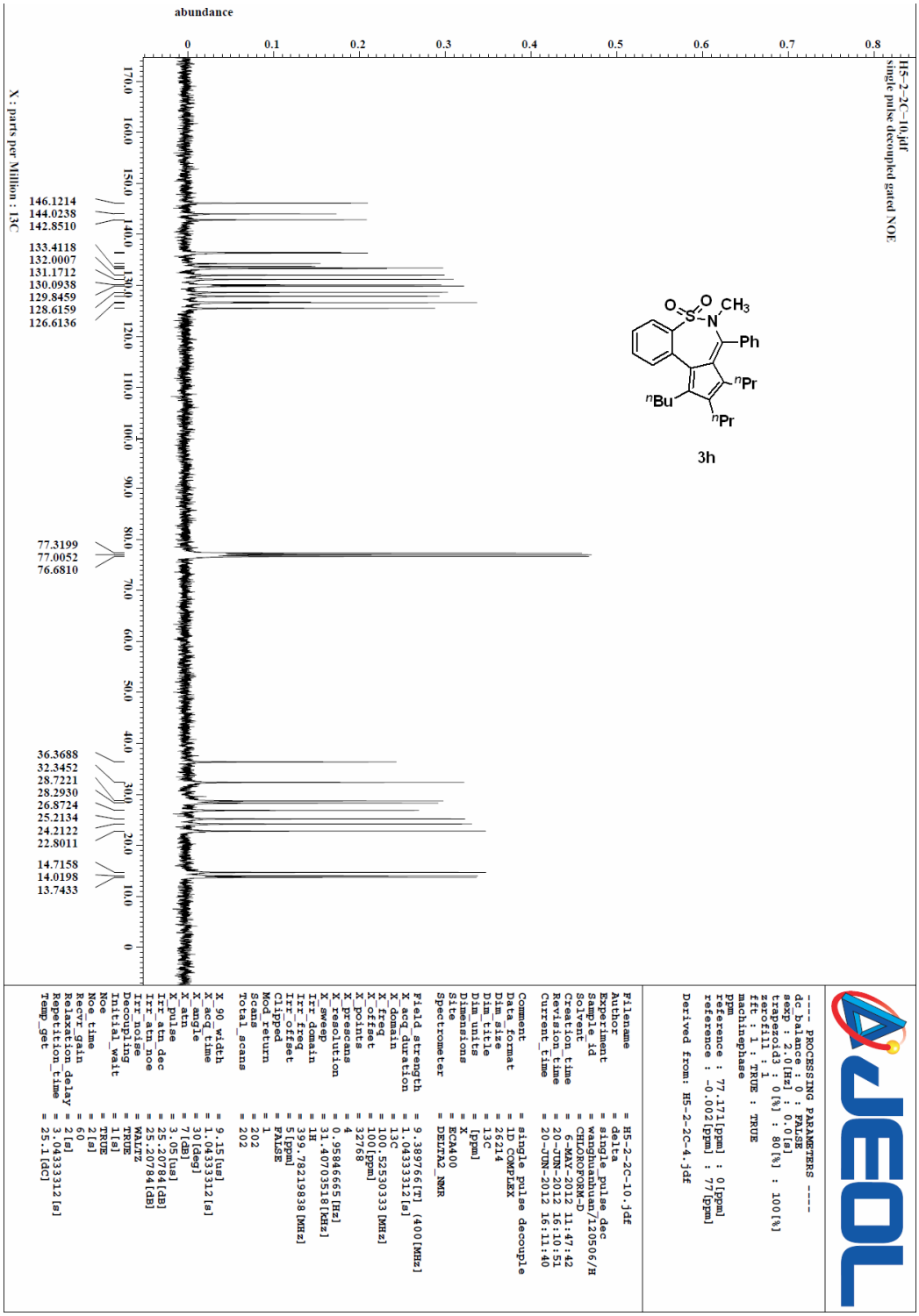

abundance
X : parts per Million : 13C
158.6949
146.1664
145.6420
142.7149
142.0094
134.3245
133.8668
133.6857
133.5808
130.4630
129.9958
128.7944
126.3727
126.1534
113.8823
77.4984
77.1837
76.8691
55.3496
36.8429
29.0246
28.5669
25.4872
23.5994
21.4637
14.5892
14.2555
H5-18-1C-4.jdf
single pulse decoupled gated NOE
Ph
nPr
MeOC6H4
nPr
3f

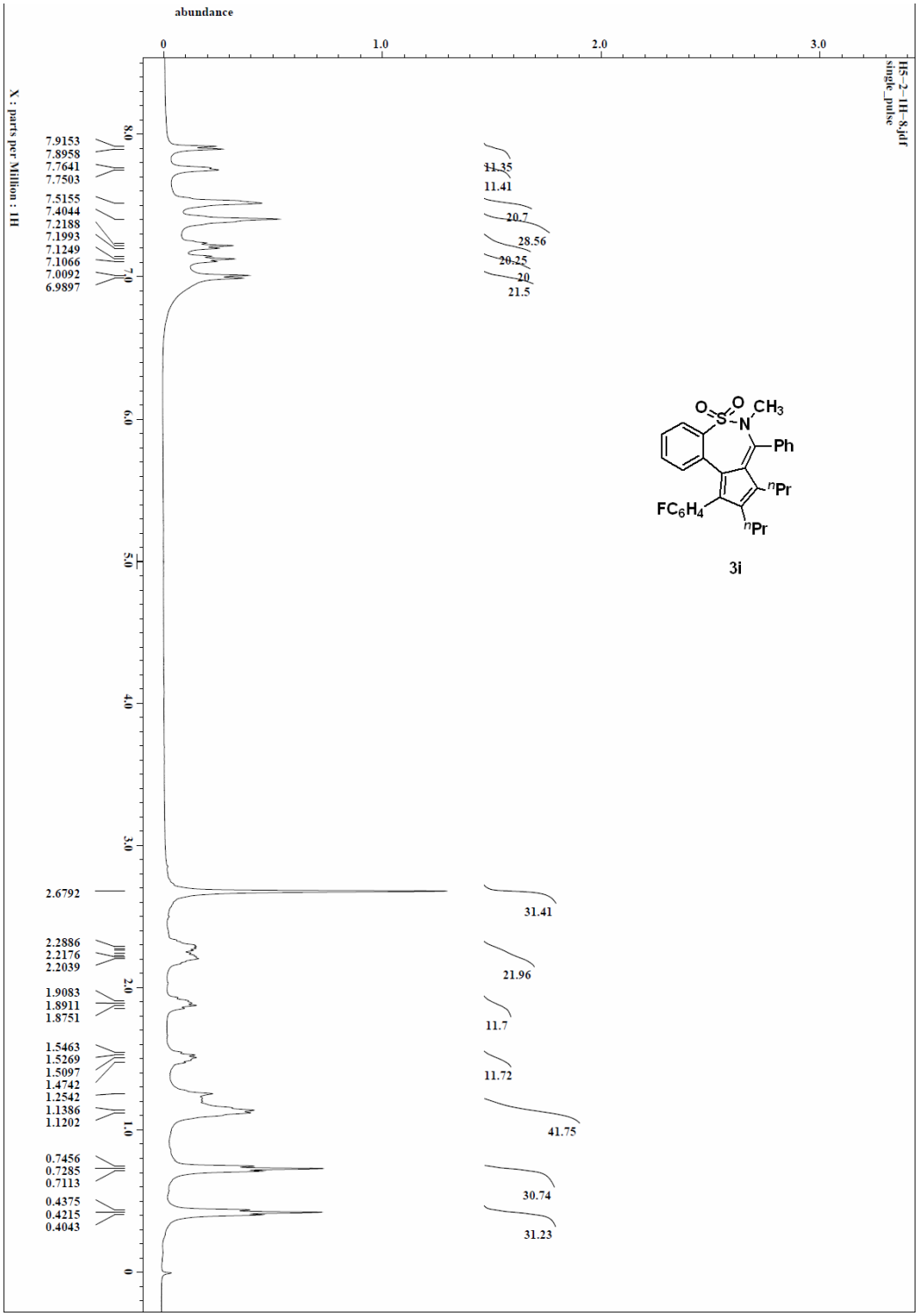

abundance
X : parts per Million : 1H
H5-17-3H-5.jdf
single_pulse
7.8133
7.7939
7.6782
7.6484
7.4674
7.3345
7.2497
7.1730
7.1535
2.6048
2.5338
2.5212
2.4467
2.3574
1.7754
1.7606
1.7411
1.5246
1.5063
1.4902
1.0572
1.0332
1.0148
0.9965
0.6242
0.6128
0.3928
0.3745
0.3562
−0.0046
−0.1616
−0.1730
10.32
20.85
21.31
21.51
10.78
29.2
11.94
30.56
11.73
31.29
20.56
61.47
11.26
41.08
10
Ph
nPr
nPr
3g

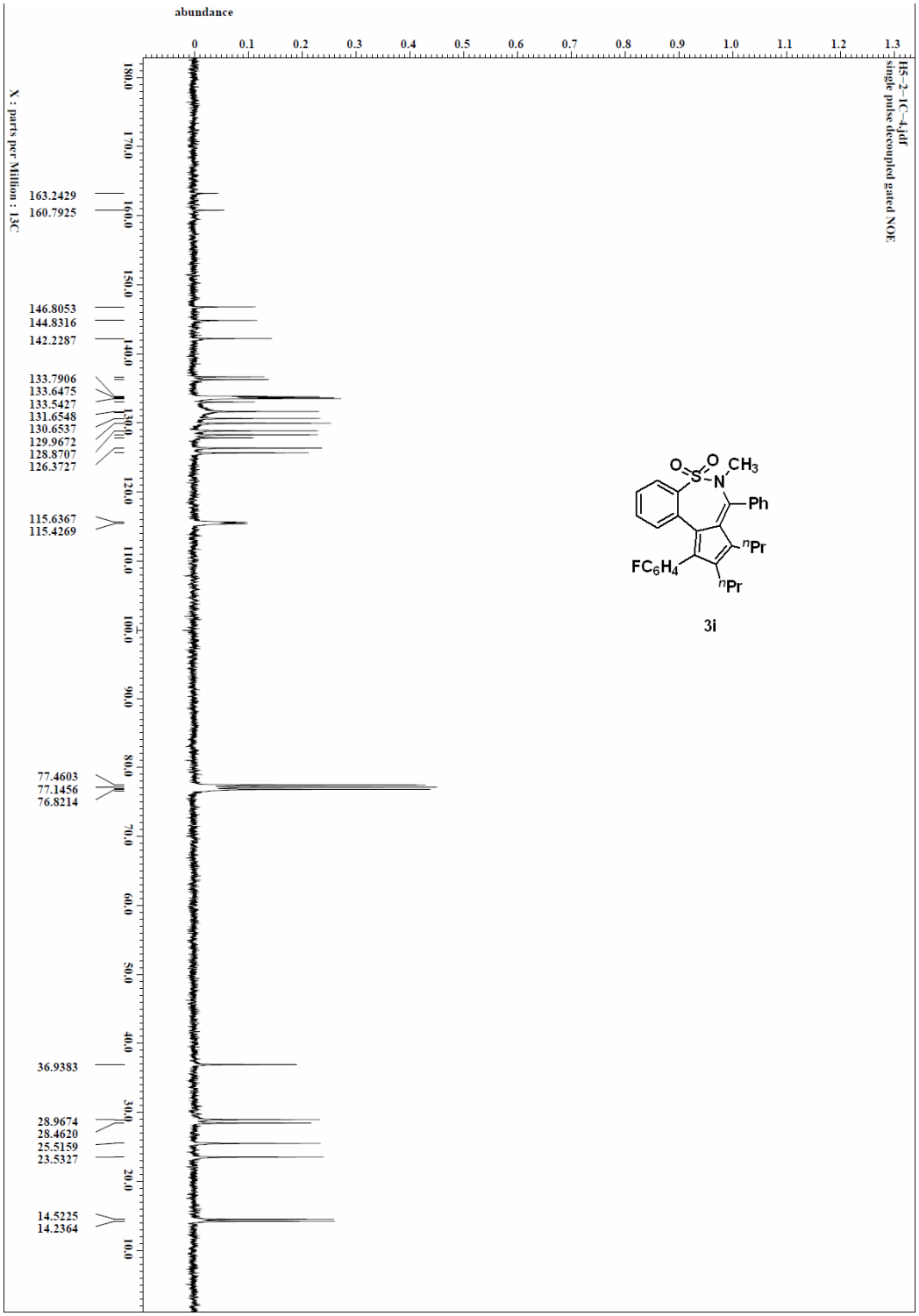

abundance
X : parts per Million : 13C
H5-17-3C-4.jdf
single pulse decoupled gated NOE
145.9758
144.4979
141.6947
136.7558
134.1433
133.3138
132.3985
130.1197
130.0053
128.7754
128.0317
128.0126
126.4108
126.1152
77.4603
77.1361
76.8214
36.6332
28.8148
28.4716
25.2870
23.9713
21.7116
14.8467
14.0744
10.4989
10.1080
7.7720
O
O
S
N
CH3
Ph
nPr
nPr

3g

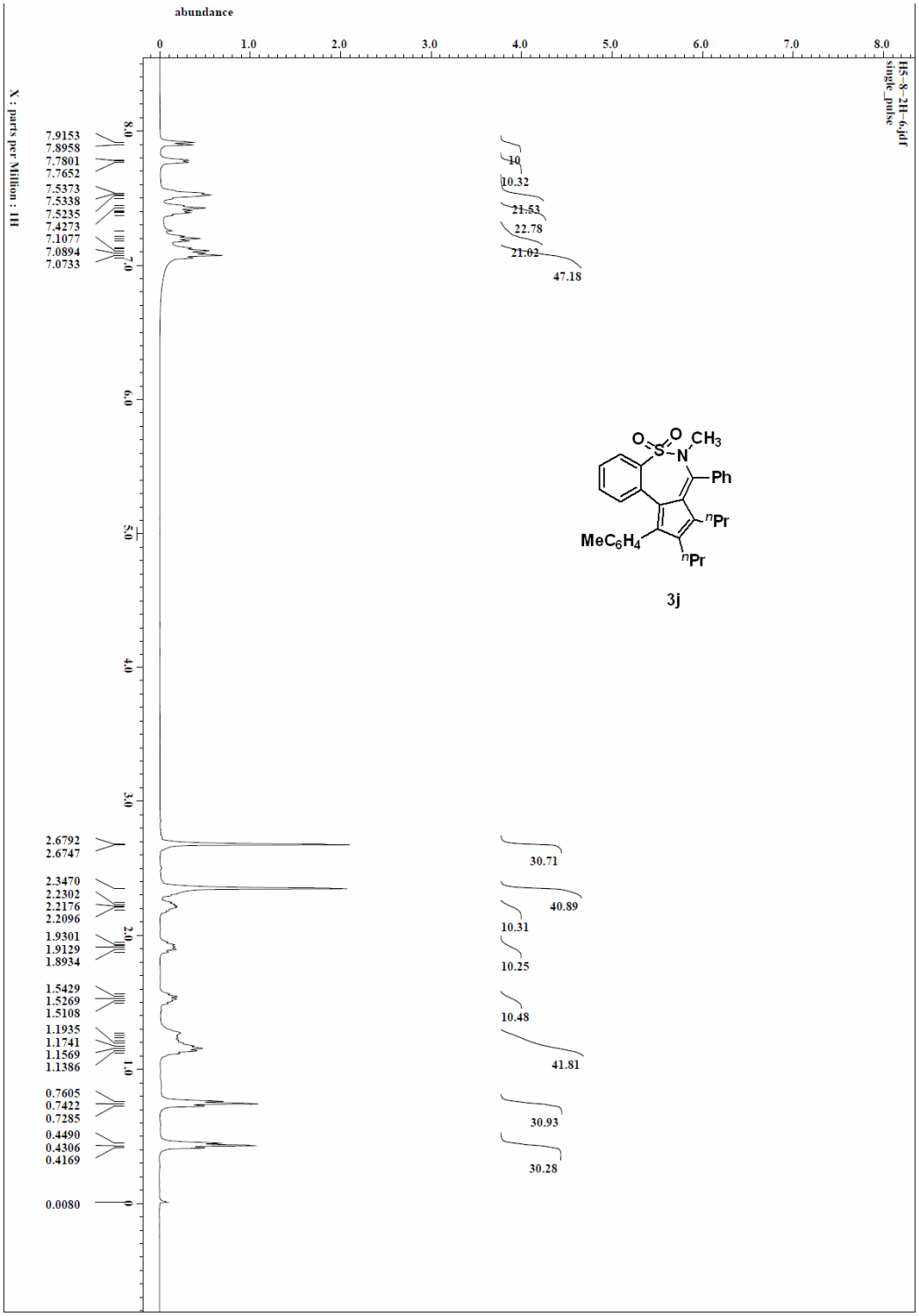

abundance
X : parts per Million : 1H
H5-2-2H-6.jdf
single_pulse
7.9703
7.9519
7.7011
7.5751
7.5544
7.4743
7.3574
7.3391
2.6128
2.4879
2.4627
2.3207
2.3012
2.2829
1.8213
1.8041
1.7892
1.5085
1.4902
1.3757
1.3596
0.9839
0.9667
0.9152
0.8969
0.8797
0.4077
0.3905
0.3734
10
10.01
19.03
21.48
30.87
40.29
11.4
21.88
11.76
31.28
21.78
21.16
29.38
30.31
30.54
Ph
nPr
nBu
nPr
3h

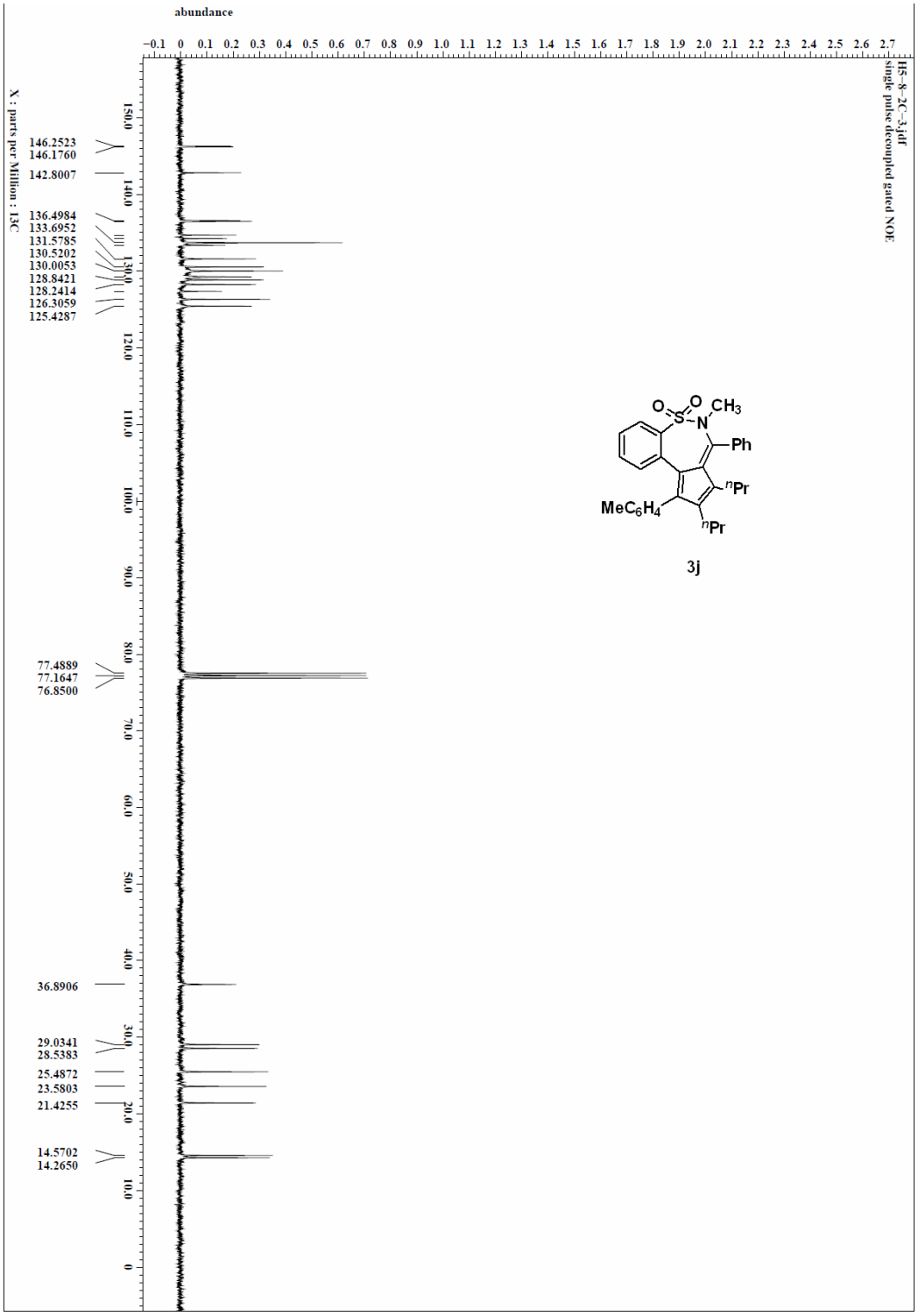

H5-2-2C-10.jdf
single pulse decoupled gated NOE
abundance
X : parts per Million : 13C
146.1214
144.0238
142.8510
133.4118
132.0007
131.1712
130.0938
129.8459
128.6159
126.6136
77.3199
77.0052
76.6810
36.3688
32.3452
28.7221
28.2930
26.8724
25.2134
24.2122
22.8011
14.7158
14.0198
13.7433
Ph
nPr
nBu
nPr
3h
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 1 0[%] : 80[%] : 100[%]
fft : 1 : TRUE : TRUE
machinephase
ppm
reference : 77.1711[ppm] : 0[ppm]
reference : -0.002[ppm] : 77[ppm]
Derived from: H5-2-2C-4.jdf
Filename = H5-2-2C-10.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = wanghuanhuan/120506/H
Solvent = CHLOROFORM-D
Creation_time = 20-JUN-2012 11:47:42
Revision_time = 20-JUN-2012 16:10:51
Current_time = 20-JUN-2012 16:11:40
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA400
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333 [MHz]
X_offset = 109[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838 [MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 202
Total_scans = 202
X_90_width = 9.15[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 7[dB]
X_pulse = 3.05[us]
Irr_atn_dec = 25.20784[dB]
Irr_atn_noe = 25.20784[dB]
Irr_mode = WALTZ2
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recyc_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get = 25.1[dC]

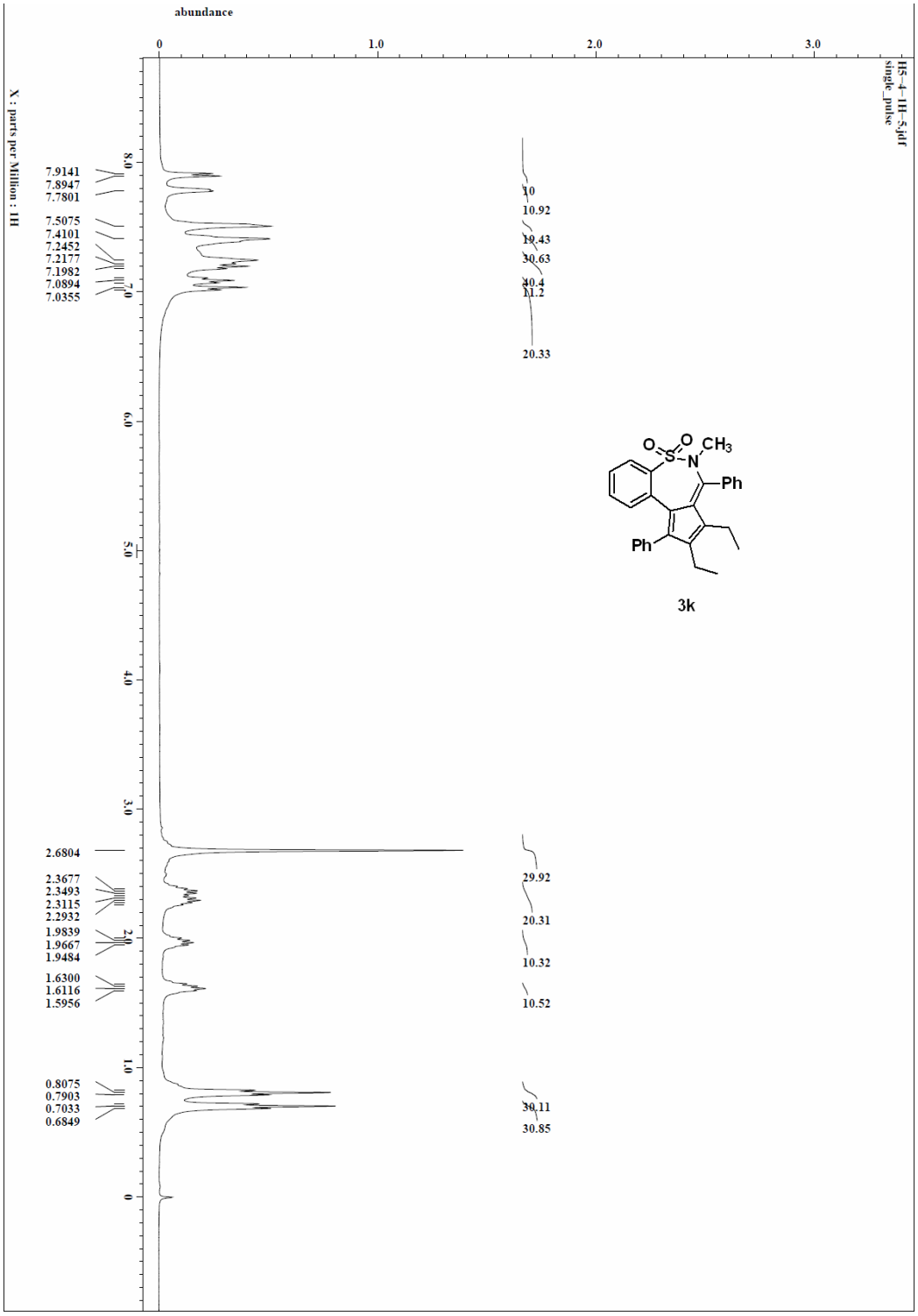

abundance
0
1.0
2.0
3.0
HS-2-1H-8.jdf
single_pulse
X : parts per Million : 1H
7.9153
7.8958
7.7641
7.7503
7.5155
7.4044
7.2188
7.1993
7.1249
7.1066
7.0092
6.9897
2.6792
2.2886
2.2176
2.2039
1.9083
1.8911
1.8751
1.5463
1.5269
1.5097
1.4742
1.2542
1.1386
1.1202
0.7456
0.7285
0.7113
0.4375
0.4215
0.4043
11.35
11.41
20.7
28.56
20.25
20
21.5
31.41
21.96
11.7
11.72
41.75
30.74
31.23
O
S
O
N
CH3
Ph
nPr
FC6H4
nPr
3i


abundance
X : parts per Million : 13C
H5-2-1C-4.jdf
single pulse decoupled gated NOE
163.2429
160.7925
146.8053
144.8316
142.2287
133.7906
133.6475
133.5427
131.6548
130.6537
129.9672
128.8707
126.3727
115.6367
115.4269
77.4603
77.1456
76.8214
36.9383
28.9674
28.4620
25.5159
23.5327
14.5225
14.2364
O
S
O
N
CH3
Ph
nPr
FC6H4
nPr

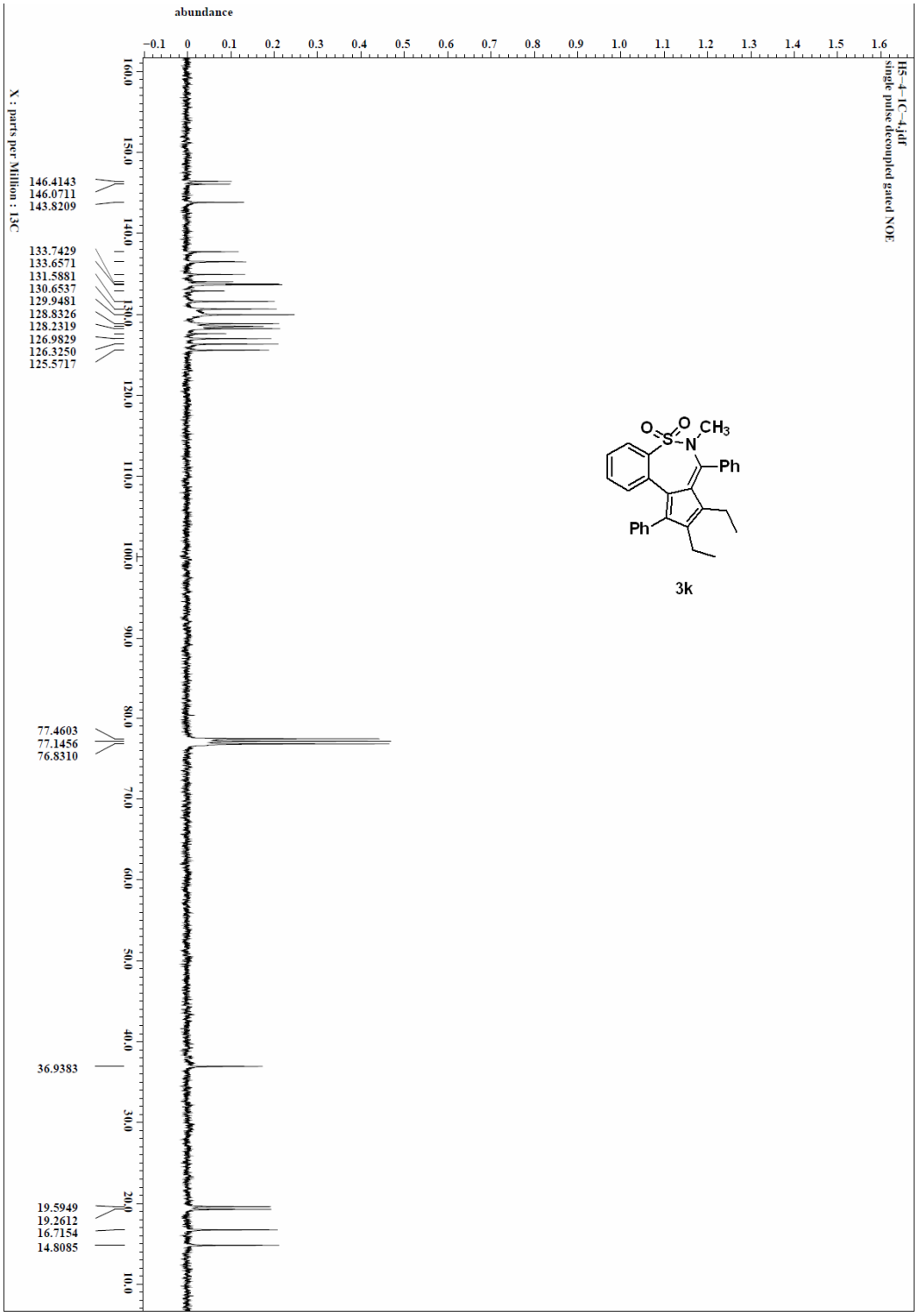
3i


abundance
X : parts per Million : 1H
H5-8-2H-6.jdf
single_pulse
7.9153
7.8958
7.7801
7.7652
7.5373
7.5338
7.5235
7.4273
7.1077
7.0894
7.0733
2.6792
2.6747
2.3470
2.2302
2.2176
2.2096
1.9301
1.9129
1.8934
1.5429
1.5269
1.5108
1.1935
1.1741
1.1569
1.1386
0.7605
0.7422
0.7285
0.4490
0.4306
0.4169
0.0080
10
10.32
21.53
22.78
21.02
47.18
30.71
40.89
10.31
10.25
10.48
41.81
30.93
30.28
Ph
nPr
MeC6H4
nPr
3j

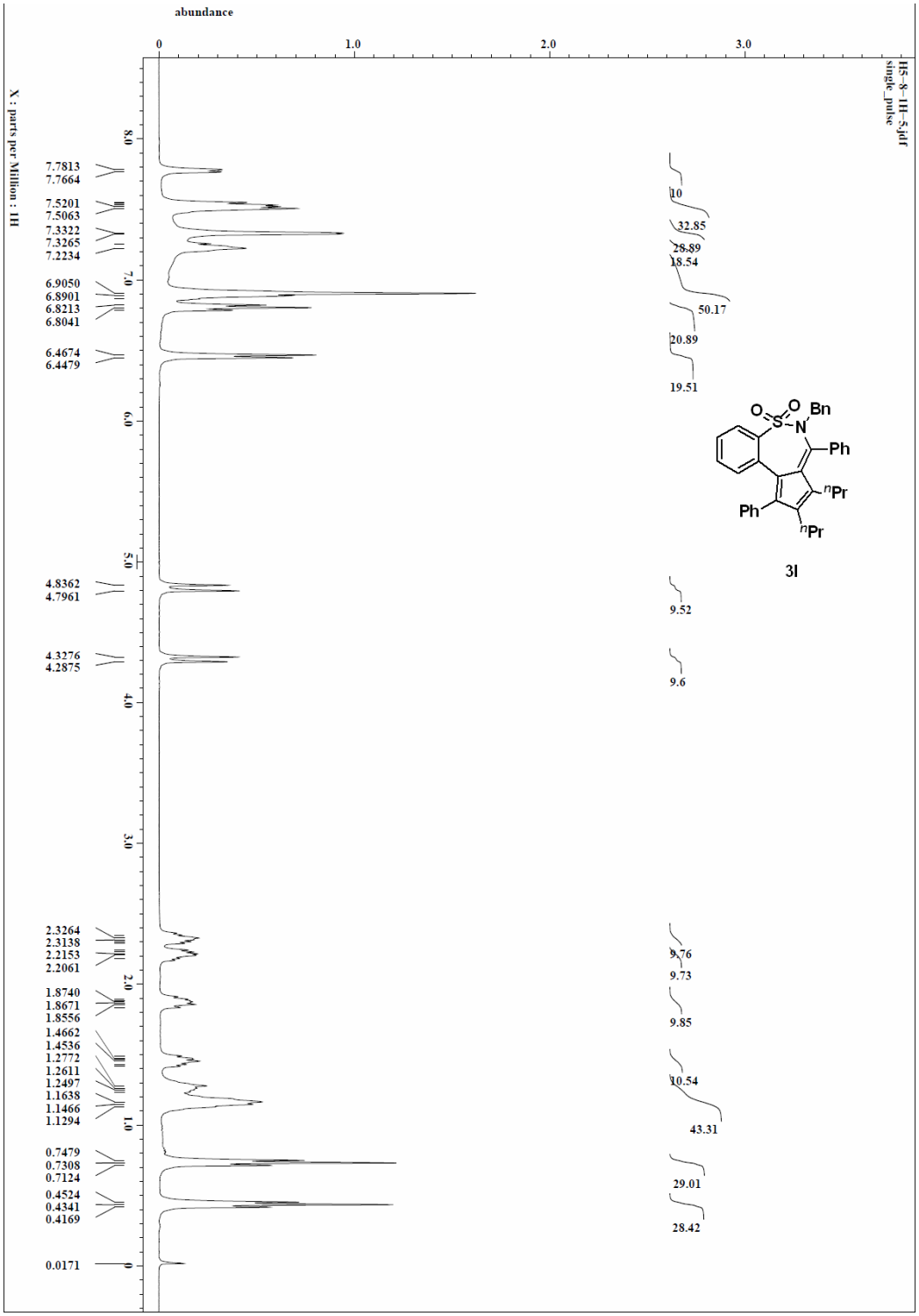

abundance
H5-8-2C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
146.2523
146.1760
142.8007
136.4984
133.6952
131.5785
130.5202
130.0053
128.8421
128.2414
126.3059
125.4287
77.4889
77.1647
76.8500
36.8906
29.0341
28.5383
25.4872
23.5803
21.4255
14.5702
14.2650
Ph
nPr
MeC6H4
nPr
3j

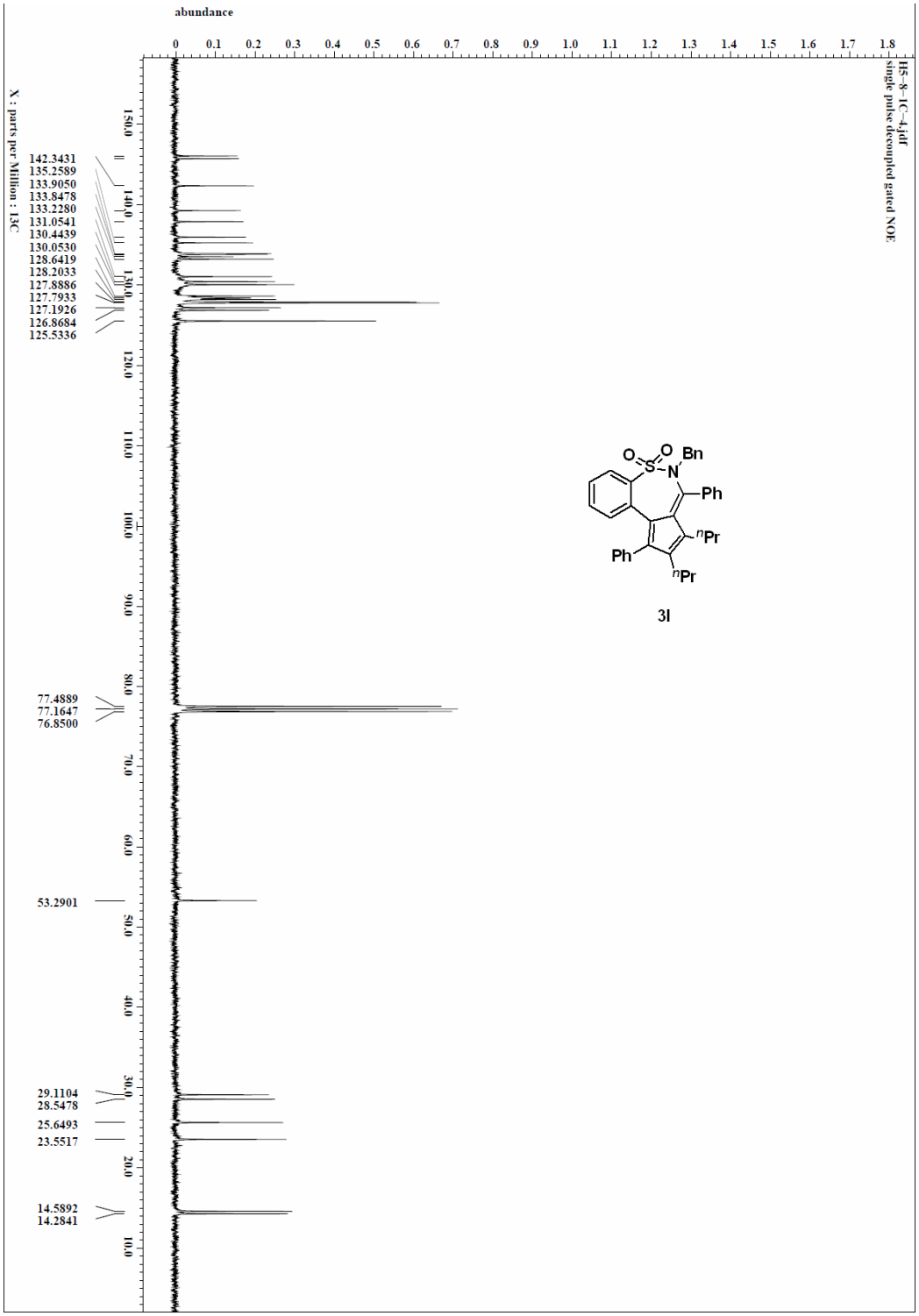

abundance
X : parts per Million : 1H
HS-4-1H-5.jdf
single_pulse
7.9141
7.8947
7.7801
7.5075
7.4101
7.2452
7.2177
7.1982
7.0894
7.0355
2.6804
2.3677
2.3493
2.3115
2.2932
1.9839
1.9667
1.9484
1.6300
1.6116
1.5956
0.8075
0.7903
0.7033
0.6849
10
10.92
19.43
30.63
40.4
41.2
20.33
29.92
20.31
10.32
10.52
30.11
30.85
O
S
O
N
CH3
Ph
Ph

3k

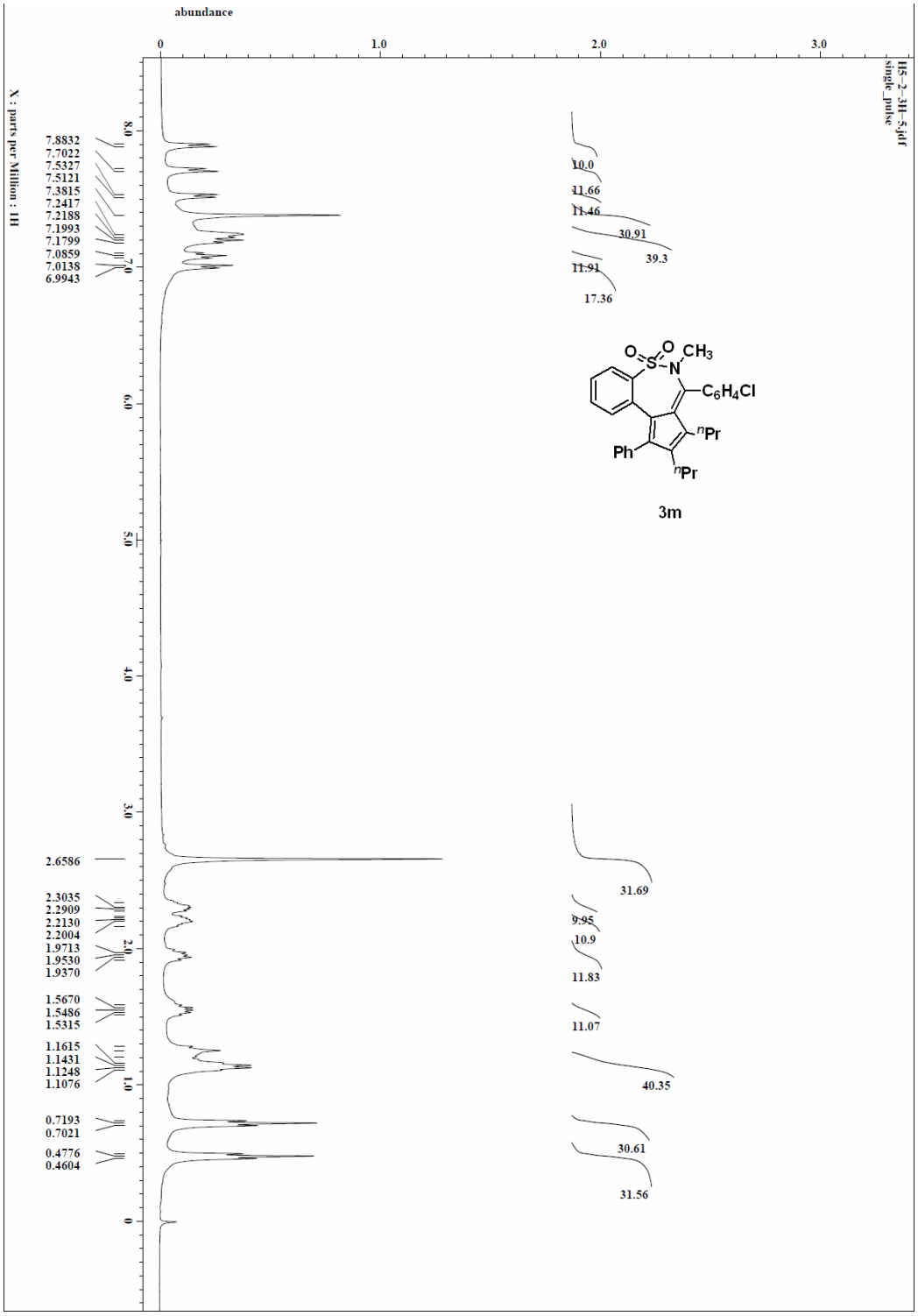

abundance
X : parts per Million : 13C
H5-4-1C-4.jdf
single pulse decoupled gated NOE
146.4143
146.0711
143.8209
133.7429
133.6571
131.5881
130.6537
129.9481
128.8326
128.2319
126.9829
126.3250
125.5717
77.4603
77.1456
76.8310
36.9383
19.5949
19.2612
16.7154
14.8085
O
O
S
N
CH3
Ph
Ph

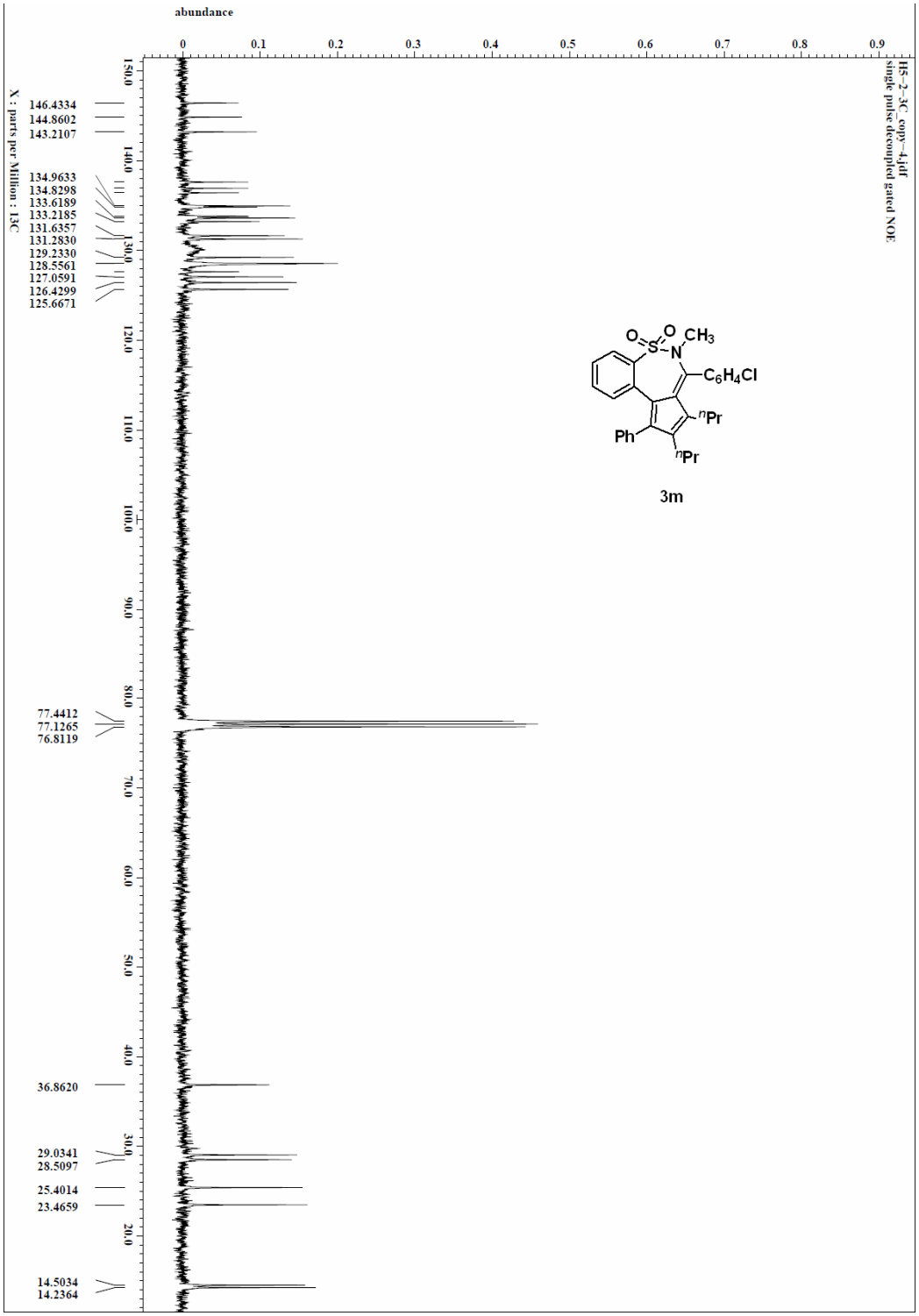
3k


abundance
X : parts per Million : 1H
HS-8-1H-5.jdf
single_pulse
7.7813
7.7664
7.5201
7.5063
7.3322
7.3265
7.2234
6.9050
6.8901
6.8213
6.8041
6.4674
6.4479
4.8362
4.7961
4.3276
4.2875
2.3264
2.3138
2.2153
2.2061
1.8740
1.8671
1.8556
1.4662
1.4536
1.2772
1.2611
1.2497
1.1638
1.1466
1.1294
0.7479
0.7308
0.7124
0.4524
0.4341
0.4169
0.0171
10
32.85
28.89
18.54
50.17
20.89
19.51
9.52
9.6
9.76
9.73
9.85
10.54
43.31
29.01
28.42
Bn
Ph
Ph
3l

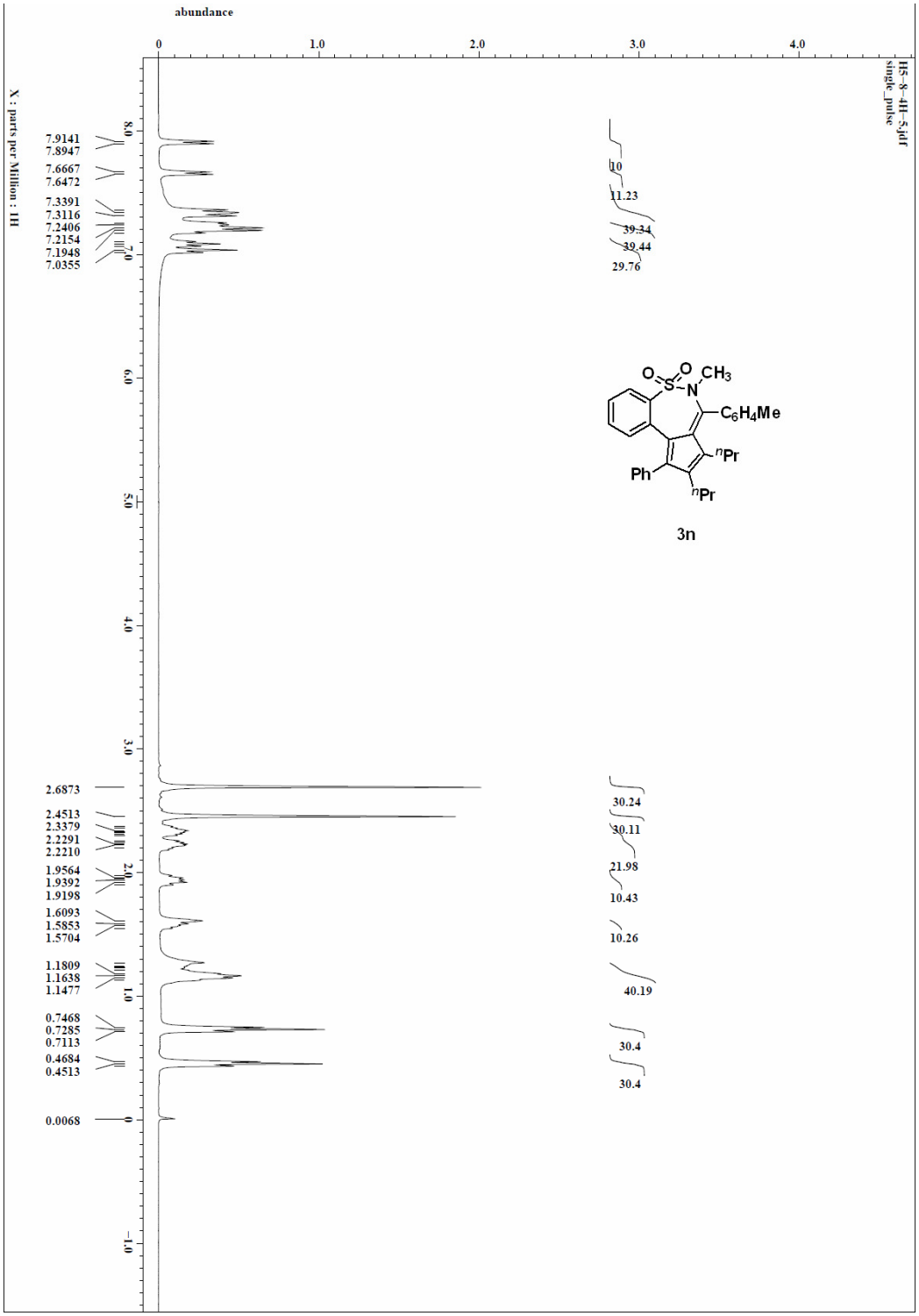

abundance
X : parts per Million : 13C
H5-8-1C-4.jdf
single pulse decoupled gated NOE
142.3431
135.2589
133.9050
133.8478
133.2280
131.0541
130.4439
130.0530
128.6419
128.2033
127.8886
127.7933
127.1926
126.8684
125.5336
77.4889
77.1647
76.8500
53.2901
29.1104
28.5478
25.6493
23.5517
14.5892
14.2841
O
O
S
N
Bn
Ph
nPr
Ph
nPr

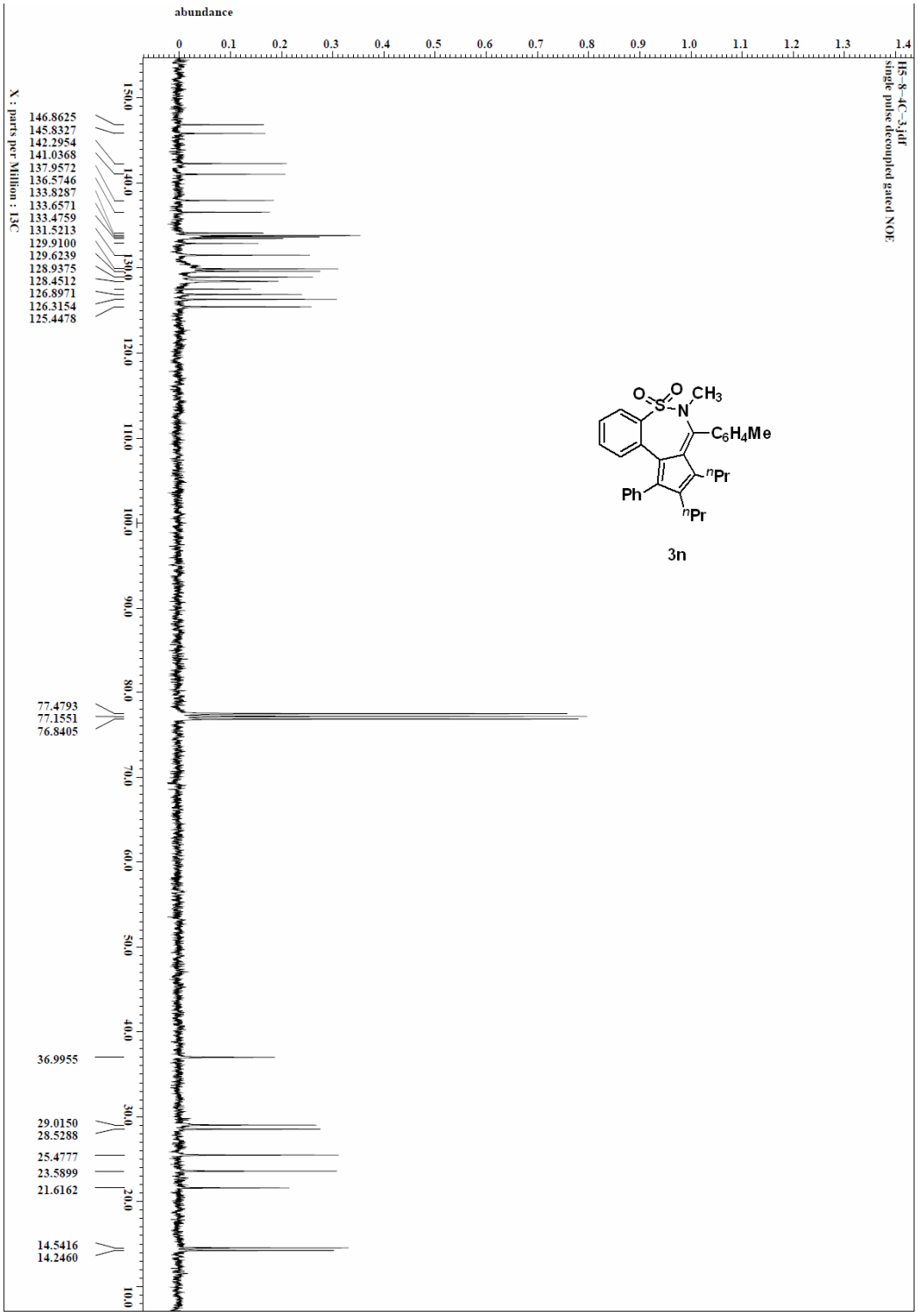
3l


abundance
X : parts per Million : 1H
H5-2-3H-5.jdf
single_pulse
7.8832
7.7022
7.5327
7.5121
7.3815
7.2417
7.2188
7.1993
7.1799
7.0859
7.0138
6.9943
2.6586
2.3035
2.2909
2.2130
2.2004
1.9713
1.9530
1.9370
1.5670
1.5486
1.5315
1.1615
1.1431
1.1248
1.1076
0.7193
0.7021
0.4776
0.4604
10.0
11.66
11.46
30.91
39.3
11.91
17.36
31.69
9.95
10.9
11.83
11.07
40.35
30.61
31.56
3m

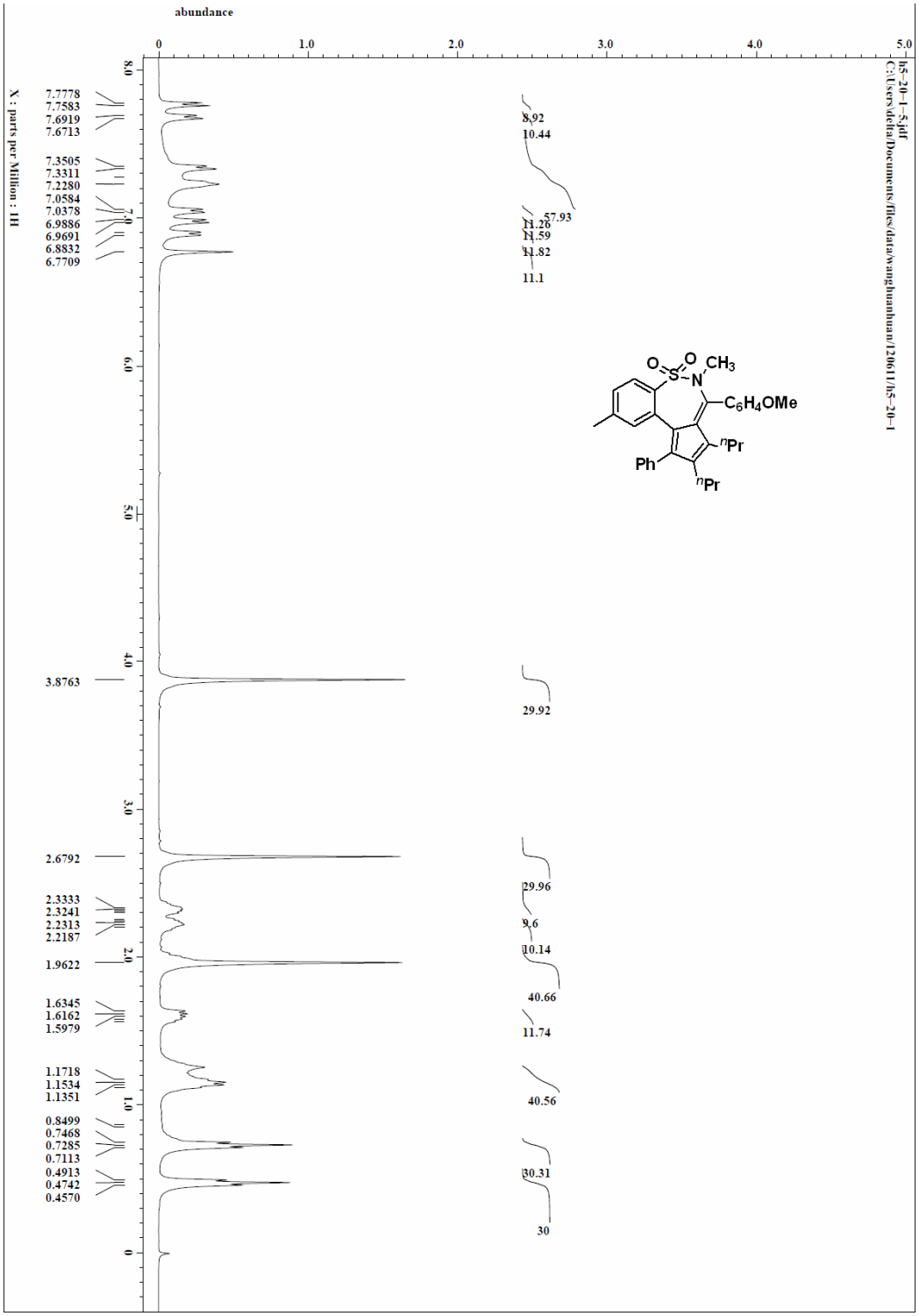

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
H5-2-3C_copy-4.jdf
Single pulse decoupled gated NOE
X : parts per Million : 13C
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
146.4334
144.8602
143.2107
134.9633
134.8298
133.6189
133.2185
131.6357
131.2830
129.2330
128.5561
127.0591
126.4299
125.6671
77.4412
77.1265
76.8119
36.8620
29.0341
28.5097
25.4014
23.4659
14.5034
14.2364
O
O
S
N
CH3
C6H4Cl
nPr
Ph
nPr

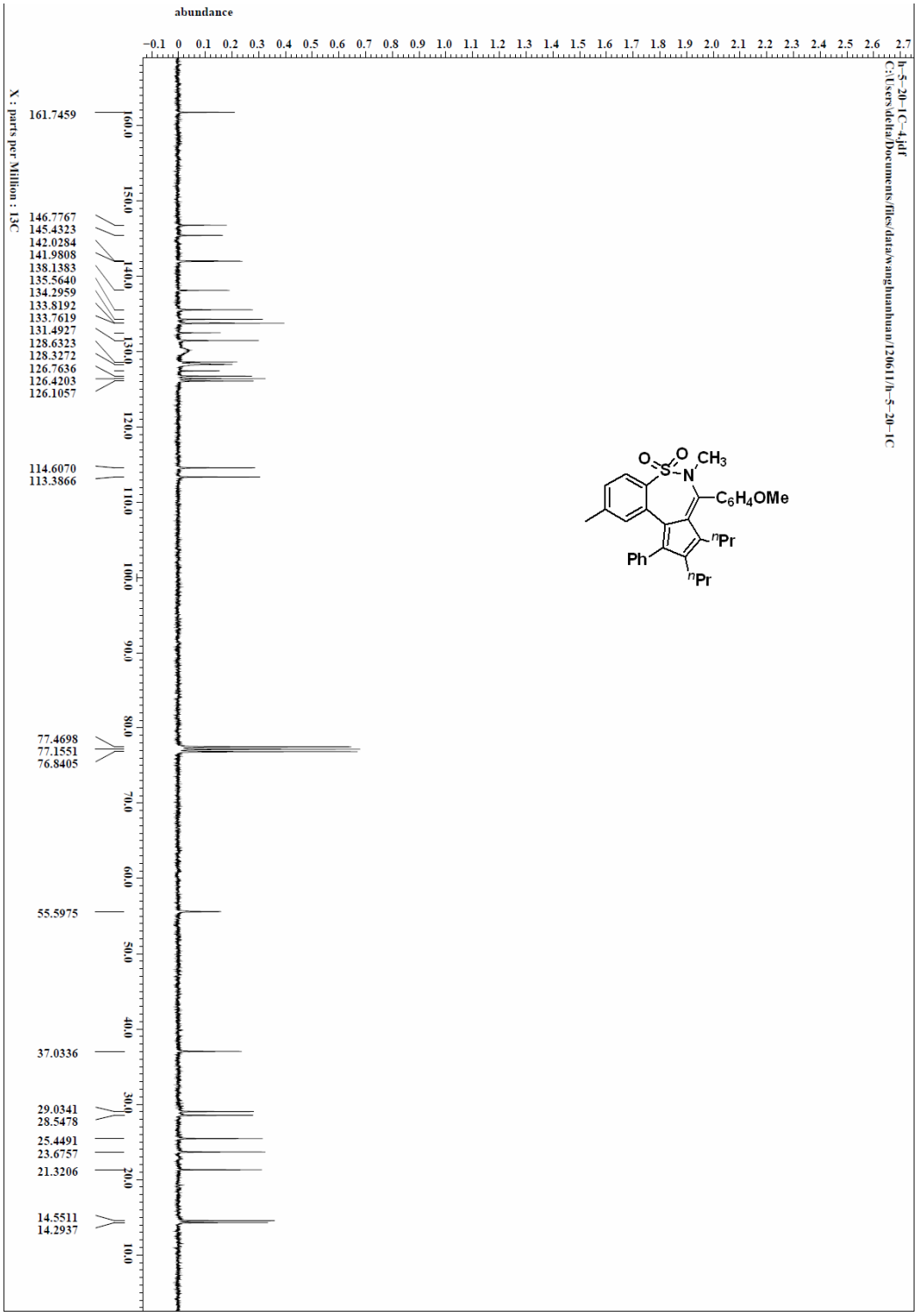
3m


abundance
X : parts per Million : 1H
H5-8-4H-5.jdf
single_pulse
7.9141
7.8947
7.6667
7.6472
7.3391
7.3116
7.2406
7.2154
7.1948
7.0355
2.6873
2.4513
2.3379
2.2291
2.2210
1.9564
1.9392
1.9198
1.6093
1.5853
1.5704
1.1809
1.1638
1.1477
0.7468
0.7285
0.7113
0.4684
0.4513
0.0068
10
11.23
39.34
39.44
29.76
30.24
30.11
21.98
10.43
10.26
40.19
30.4
30.4
O
O
S
N
CH3
C6H4Me
nPr
Ph
nPr
3n


abundance
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4
H5-8-4C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
146.8625
145.8327
142.2954
141.0368
137.9572
136.5746
133.8287
133.6571
133.4759
131.5213
129.9100
129.6239
128.9375
128.4512
126.8971
126.3154
125.4478
77.4793
77.1551
76.8405
36.9955
29.0150
28.5288
25.4777
23.5899
21.6162
14.5416
14.2460
O
S
O
N
CH3
C6H4Me
nPr
Ph
nPr

3n


abundance
X : parts per Million : 1H
C:\Users\delfa\Documents\files\data\wanghuanhuan\120611\h5-20-1-5.ddf
h5-20-1-5.ddf
7.7778
7.7583
7.6919
7.6713
7.3505
7.3311
7.2280
7.0584
7.0378
6.9886
6.9691
6.8832
6.7709
3.8763
2.6792
2.3333
2.3241
2.2313
2.2187
1.9622
1.6345
1.6162
1.5979
1.1718
1.1534
1.1351
0.8499
0.7468
0.7285
0.7113
0.4913
0.4742
0.4570
8.92
10.44
57.93
11.26
11.59
11.82
11.1
29.92
29.96
9.6
10.14
40.66
11.74
40.56
30.31
30
O
O
S
N
CH3
C6H4OMe
nPr
Ph
nPr


abundance
X : parts per Million : 13C
161.7459
146.7767
145.4323
142.0284
141.9808
138.1383
135.5640
134.2959
133.8192
133.7619
131.4927
128.6323
128.3272
126.7636
126.4203
126.1057
114.6070
113.3866
77.4698
77.1551
76.8405
55.5975
37.0336
29.0341
28.5478
25.4491
23.6757
21.3206
14.5511
14.2937
h-5-20-1C-4.jdf
C:\Users\delta\Documents\files\data\wanghuanhuan\120611\h-5-20-1C
O
O
S
N
CH3
C6H4OMe
nPr
Ph
nPr