Generation of 3-(1*H*-pyrrol-3-yl)-1*H*-inden-1-ones via a reaction of 1-(2-alkynylphenyl)-2-enone, 2-isocyanoacetate, with water

Danqing Zheng,[†] Shaoyu Li,[†] and Jie Wu*[,†,‡]

[†] *Department of Chemistry, Fudan University, 220 Handan Road, Shanghai 200433, China* [‡] *State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China*

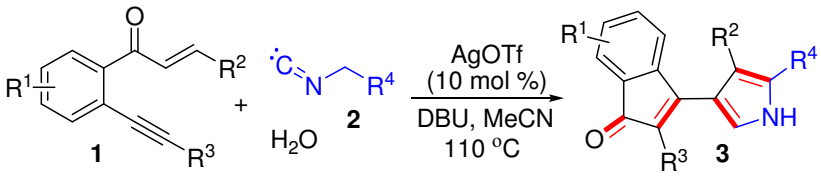
jie_wu@fudan.edu.cn

Supporting Information

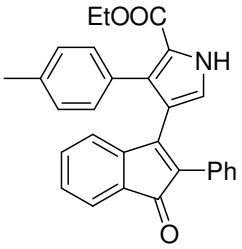
General experimental methods:

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.

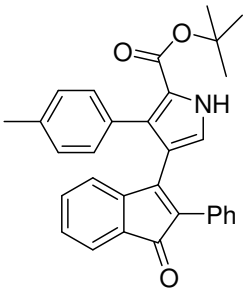
*General procedure of the silver(I)-catalyzed tandem reaction of the reaction of 1-(2-alkynylphenyl)-2-enone **1** with 2-isocyanoacetate **2***



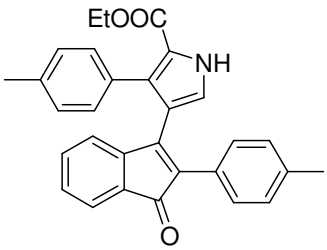
Silver triflate (0.02 mmol, 10 mol %), 2-isocyanoacetate **2** (0.24 mmol, 1.2 equiv), and DBU (0.1 mmol, 0.5 equiv) were added to a solution of 1-(2-alkynylphenyl)-2-enone **1** (0.20 mmol) in CH_3CN (1.0 mL). The mixture was stirred at 110 °C in a sealed tube for 4~6 hours. After completion of reaction as indicated by TLC, the mixture was purified by flash column chromatograph (EtOAc/n-hexane, 1:5) to give the desired product **3**.



Ethyl 4-(1-oxo-2-phenyl-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3a**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.70 (s, 1H), 7.45 (d, *J* = 6.0 Hz, 1H), 7.13-7.20 (m, 5H), 7.02-7.06 (m, 3H), 6.94 (d, *J* = 6.8 Hz, 2H), 6.83-6.88 (m, 3H), 4.19-4.21 (m, 2H), 2.22 (s, 3H), 1.18 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.5, 161.0, 150.2, 145.2, 136.4, 133.3, 133.1, 131.4, 131.0, 130.8, 130.2, 129.7, 129.4, 128.6, 128.0, 127.9, 127.8, 127.2, 122.3, 121.3, 120.2, 117.7, 60.6, 21.2, 14.2; HRMS (ESI) calcd for $C_{29}H_{23}NO_3$: 434.1756 (M + H^+), found: 434.1737.

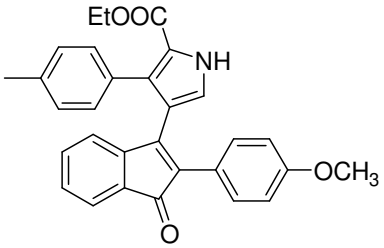


tert-Butyl 4-(1-oxo-2-phenyl-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3b**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H), 7.45 (d, *J* = 6.4 Hz, 1H), 7.14-7.24 (m, 5H), 7.02-7.06 (m, 3H), 6.82-6.91 (m, 5H), 2.22 (s, 3H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ196.5, 160.8, 150.3, 145.4, 136.1, 133.2, 133.0, 131.5, 130.8, 130.6, 130.2, 129.8, 129.4, 128.6, 127.9, 127.8, 127.2, 122.3, 121.7, 121.6, 121.3, 117.6, 81.7, 28.3, 21.2; HRMS (ESI) calcd for $C_{37}H_{21}NO_3$: 462.2069 (M + H^+), found: 462.2072.



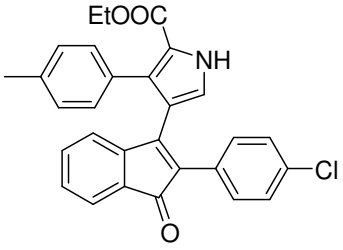
Ethyl 4-(1-oxo-2-*p*-tolyl-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3c**): ^{1}H

NMR (400 MHz, $CDCl_3$) δ 9.53 (s, 1H), 7.44 (d, *J* = 6.4 Hz, 1H), 7.13-7.17 (m, 2H), 6.96-6.99 (m, 6H), 6.87 (d, *J* = 7.2 Hz, 2H), 6.80 (d, *J* = 6.4 Hz, 2H), 4.21 (d, *J* = 6.8 Hz, 2H), 2.29 (s, 3H), 2.23 (s, 3H), 1.20 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.7, 160.9, 149.6, 145.3, 137.1, 136.5, 133.3, 133.2, 130.9, 130.7, 130.3, 129.7, 129.3, 128.6, 128.4, 128.0, 122.2, 121.3, 121.2, 120.1, 117.9, 60.6, 21.4, 21.2, 14.2; HRMS (ESI) calcd for $C_{30}H_{25}NO_3$: 448.1913 (M + H^+), found: 448.1911.



Ethyl

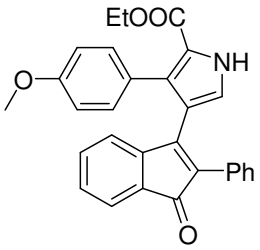
4-(2-(4-methoxyphenyl)-1-oxo-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3d**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.62 (s, 1H), 7.43 (d, *J* = 6.4 Hz, 1H), 7.12-7.19 (m, 3H), 7.04 (d, *J* = 7.2 Hz, 2H), 6.98 (d, *J* = 6.8 Hz, 2H), 6.82-6.87 (m, 3H), 6.69 (d, *J* = 8.0 Hz, 2H), 4.21 (d, *J* = 6.8 Hz, 2H), 3.77 (s, 3H), 2.23 (s, 3H), 1.19 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 161.0, 159.0, 148.8, 145.5, 136.5, 133.3, 132.8, 130.9, 130.8,130.7, 130.3, 129.7, 128.3, 128.0, 123.9, 122.3, 122.2, 121.0, 120.1, 117.9, 60.6, 55.3, 21.2, 14.2; HRMS (ESI) calcd for $C_{30}H_{25}NO_4$: 464.1862 (M + H^+), found: 464.1854.



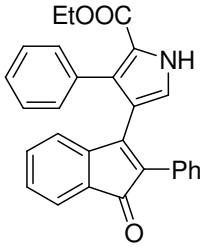
Ethyl

4-(2-(4-chlorophenyl)-1-oxo-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3e**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.52 (s, 1H), 7.46-7.48 (d, *J* = 6.8 Hz, 1H), 7.19-7.27 (m, 2H), 7.13 (s, 1H), 7.06 (d, *J* = 7.2 Hz, 2H), 6.98 (d, *J* = 6.8 Hz, 1H), 6.91 (d, *J* = 6.8 Hz, 2H), 6.85-6.88 (m, 4H), 4.21 (d, *J* = 6.8 Hz, 2H), 2.23 (s, 3H), 1.18 (t, *J* = 6.8

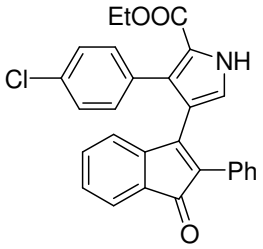
Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.1, 160.8, 145.0, 136.5, 133.4, 133.1, 130.8, 130.7, 130.6, 129.9, 129.8, 128.9, 128.0, 127.9, 127.8, 122.5, 122.1, 122.0, 121.2, 120.5, 117.4, 60.7, 21.2, 14.2; HRMS (ESI) calcd for $C_{29}H_{22}ClNO_3$: 468.1366 ($M + H^+$), found: 468.1352.



Ethyl 3-(4-methoxyphenyl)-4-(1-oxo-2-phenyl-1*H*-inden-3-yl)-1*H*-pyrrole-2-carboxylate (**3f**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.63 (s, 1H), 7.46 (d, *J* = 6.4 Hz, 1H), 7.13-7.24 (m, 5H), 7.04-7.06 (m, 3H), 6.97 (d, *J* = 7.6 Hz, 2H), 6.88 (d, *J* = 6.8 Hz, 1H), 6.59 (d, *J* = 7.6 Hz, 2H), 4.20 (d, *J* = 6.8 Hz, 2H), 3.72 (s, 3H), 1.19 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.5, 161.0, 158.6, 150.1, 145.2, 133.3, 133.1, 131.4, 131.0, 130.8, 130.6, 129.4, 128.6, 127.8, 127.3, 125.7, 122.4, 122.3, 121.2, 120.1, 117.7, 112.9, 60.6, 55.2, 14.2; HRMS (ESI) calcd for $C_{29}H_{23}NO_4$: 450.1705 ($M + H^+$), found: 450.1686.

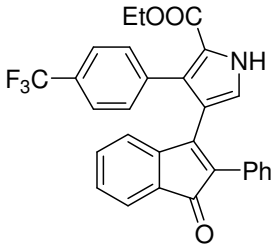


Ethyl 4-(1-oxo-2-phenyl-1*H*-inden-3-yl)-3-phenyl-1*H*-pyrrole-2-carboxylate (**3g**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.66 (s, 1H), 7.46 (d, *J* = 6.4 Hz, 1H), 7.13-7.24 (m, 5H), 7.04-7.10 (m, 8H), 6.88 (d, *J* = 6.8 Hz, 1H), 4.19 (d, *J* = 6.8 Hz, 2H), 1.16 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.4, 161.0, 149.9, 145.2, 133.2, 131.3, 130.8, 130.7, 129.9, 129.4, 129.3, 128.7, 127.9, 127.2, 126.8, 122.4, 122.3, 121.2, 120.4, 119.4, 118.6, 117.7, 60.7, 14.1; HRMS (ESI) calcd for $C_{28}H_{21}NO_3$: 420.1600 ($M + H^+$), found: 420.1586.



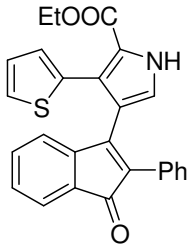
Ethyl

3-(4-chlorophenyl)-4-(1-oxo-2-phenyl-1*H*-inden-3-yl)-1*H*-pyrrole-2-carboxylate (**3h**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.80 (s, 1H), 7.49 (d, *J* = 6.4 Hz, 1H), 7.30 (t, *J* = 6.8 Hz, 1H), 7.22 (t, *J* = 7.2 Hz, 1H), 7.11-7.16 (m, 4H), 6.95-7.01 (m, 5H), 6.84 (d, *J* = 7.2 Hz, 2H), 4.19 (d, *J* = 6.8 Hz, 2H), 1.16 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.2, 160.8, 149.1, 145.1, 133.4, 133.1, 132.7, 131.6, 131.3, 131.0, 130.8, 129.5, 129.3, 128.9, 127.8, 127.4, 122.6, 122.4, 120.9, 120.5, 117.5, 60.8, 14.1; HRMS (ESI) calcd for $C_{28}H_{20}ClNO_3$: 454.1210 ($M + H^+$), found: 454.1194.

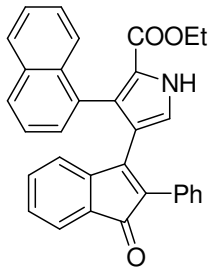


Ethyl

4-(1-oxo-2-phenyl-1*H*-inden-3-yl)-3-(4-(trifluoromethyl)phenyl)-1*H*-pyrrole-2-carboxylate (**3i**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.69 (s, 1H), 7.51 (d, *J* = 6.8 Hz, 1H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.20-7.27 (m, 4H), 7.03-7.10 (m, 4H), 6.94 (d, *J* = 7.6 Hz, 2H), 6.81 (d, *J* = 7.6 Hz, 2H), 4.17 (d, *J* = 6.8 Hz, 2H), 1.13 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.0, 160.6, 148.5, 145.1, 136.8, 133.4, 133.2, 131.1, 130.7, 130.0, 129.1, 129.0, 128.3, 127.9, 127.2, 125.8, 124.0 (f, $^{1}J_{CF}$ = 2.8Hz), 122.8, 122.3, 120.8, 120.7, 117.6, 60.9, 14.0; HRMS (ESI) calcd for $C_{29}H_{20}F_3NO_3$: 488.1474 ($M + H^+$), found: 488.1451.

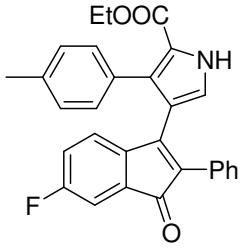


Ethyl 4-(1-oxo-2-phenyl-1*H*-inden-3-yl)-3-(thiophen-2-yl)-1*H*-pyrrole-2-carboxylate (**3j**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.59 (s, 1H), 7.47 (d, *J* = 5.6 Hz, 1H), 7.13-7.24 (m, 8H), 6.90-6.96 (m, 2H), 6.76-6.80 (m, 2H), 4.28 (d, *J* = 7.2 Hz, 2H), 1.27 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.5, 160.6, 150.1, 145.3, 134.1, 133.6, 133.4, 131.2, 130.5, 129.6, 128.6, 128.5, 128.0, 127.5, 126.5, 126.2, 123.0, 122.4, 122.1, 121.1, 120.5, 118.6, 60.9, 14.2; HRMS (ESI) calcd for $C_{26}H_{19}NO_3S$: 426.1164 ($M + H^+$), found: 426.1140.

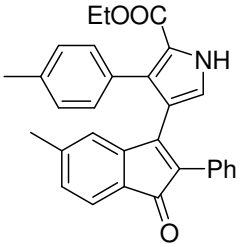


Ethyl

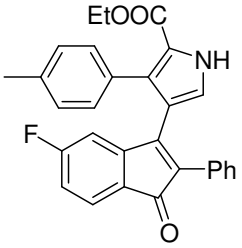
3-(naphthalen-1-yl)-4-(1-oxo-2-phenyl-1*H*-inden-3-yl)-1*H*-pyrrole-2-carboxylate (**3k**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.65 (s, 1H), 7.68 (d, *J* = 7.6 Hz, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.8 Hz, 1H), 7.37-7.39 (m, 1H), 7.24-7.30 (m, 2H), 7.09-7.13 (m, 4H), 6.87-6.99 (m, 7H), 3.89 (d, *J* = 6.8 Hz, 2H), 0.61 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 196.1, 160.5, 151.3, 145.4, 134.2, 133.1, 132.7, 132.3, 132.0, 131.5, 130.1, 129.4, 129.2, 128.2, 128.1, 127.5, 127.3, 126.2, 125.4, 125.2, 124.7, 122.3, 121.8, 121.6, 117.3, 59.9, 13.8; HRMS (ESI) calcd for $C_{32}H_{23}NO_3$: 470.1756 ($M + H^+$), found: 470.1734.



Ethyl 4-(6-fluoro-1-oxo-2-phenyl-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3l**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.58 (s, 1H), 7.10-7.15 (m, 6H), 6.98-7.00 (m, 3H), 6.74-6.89 (m, 4H), 4.22 (m, 2H), 2.24 (s, 3H), 1.19 (t, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 163.4 (d, $^{1}J_{CF}$ = 247.9 Hz), 160.9, 150.4, 140.4, 136.7, 133.4, 133.1 (d, $^{3}J_{CF}$ = 7.6 Hz), 131.1, 130.8, 130.1, 129.7, 129.4, 128.1, 127.9, 127.4, 122.5, 122.4 (d, $^{3}J_{CF}$ = 10.0 Hz), 120.2, 118.4 (d, $^{2}J_{CF}$ = 22.9 Hz), 117.6, 110.9 (d, $^{2}J_{CF}$ = 24.8Hz), 60.7, 21.2, 14.2; HRMS (ESI) calcd for $C_{29}H_{22}FNO_3$: 452.1662 (M + H^+), found: 452.1633.

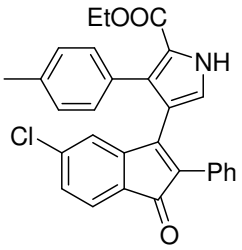


Ethyl 4-(5-methyl-1-oxo-2-phenyl-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3m**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H), 7.35 (d, J = 6.8 Hz, 1H), 7.02-7.13 (m, 6H), 6.94-6.96 (m, 3H), 6.86 (d, J = 8.0 Hz, 2H), 6.68 (s, 1H), 4.20 (d, J = 6.4 Hz, 2H), 2.24 (s, 3H), 2.23 (s, 3H), 1.19 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.3, 161.0, 149.7, 145.6, 144.1, 136.4, 133.5, 131.5, 131.0, 130.3, 129.8, 129.4, 128.6, 128.4, 128.0, 127.8, 127.2, 122.7, 122.4, 120.1, 117.8, 60.6, 21.9, 21.2, 14.2; HRMS (ESI) calcd for $C_{30}H_{25}NO_3$: 448.1913 (M + H^+)), found: 448.1884.

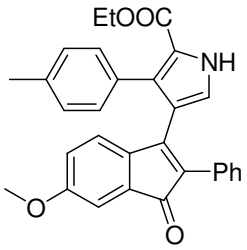


Ethyl 4-(5-fluoro-1-oxo-2-phenyl-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3n**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.70 (s, 1H), 7.43 (t, J = 5.2 Hz, 1H), 7.09-7.15 (m, 5H), 6.95-6.99 (m, 3H), 6.87 (d, J = 7.2 Hz, 2H), 6.79 (t, J = 8.4 Hz, 1H), 6.56 (d, J = 8.4 Hz, 1H), 4.21 (d, J = 7.2 Hz, 2H), 2.24 (s, 3H), 1.19 (t, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 166.5 (d, $^{1}J_{CF}$ = 251.7Hz), 160.9, 148.7 (d, $^{3}J_{CF}$ =

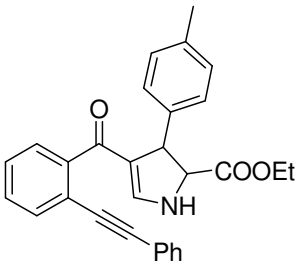
9.5Hz), 148.1, 136.6, 134.5, 131.0, 130.8, 130.1, 129.7, 129.4, 128.1, 127.9, 127.6, 126.5 (d, $^4J_{CF}$ = 2.9 Hz), 124.1 (d, $^3J_{CF}$ = 8.6 Hz), 122.2 (d, $^4J_{CF}$ = 1.9 Hz), 120.3, 117.3, 114.1 (d, $^2J_{CF}$ = 22.9 Hz), 110.2 (d, $^2J_{CF}$ = 24.8 Hz), 60.7, 21.2, 14.2; HRMS (ESI) calcd for $C_{29}H_{22}FNO_3$: 452.1662 (M + H^+), found: 452.1643.



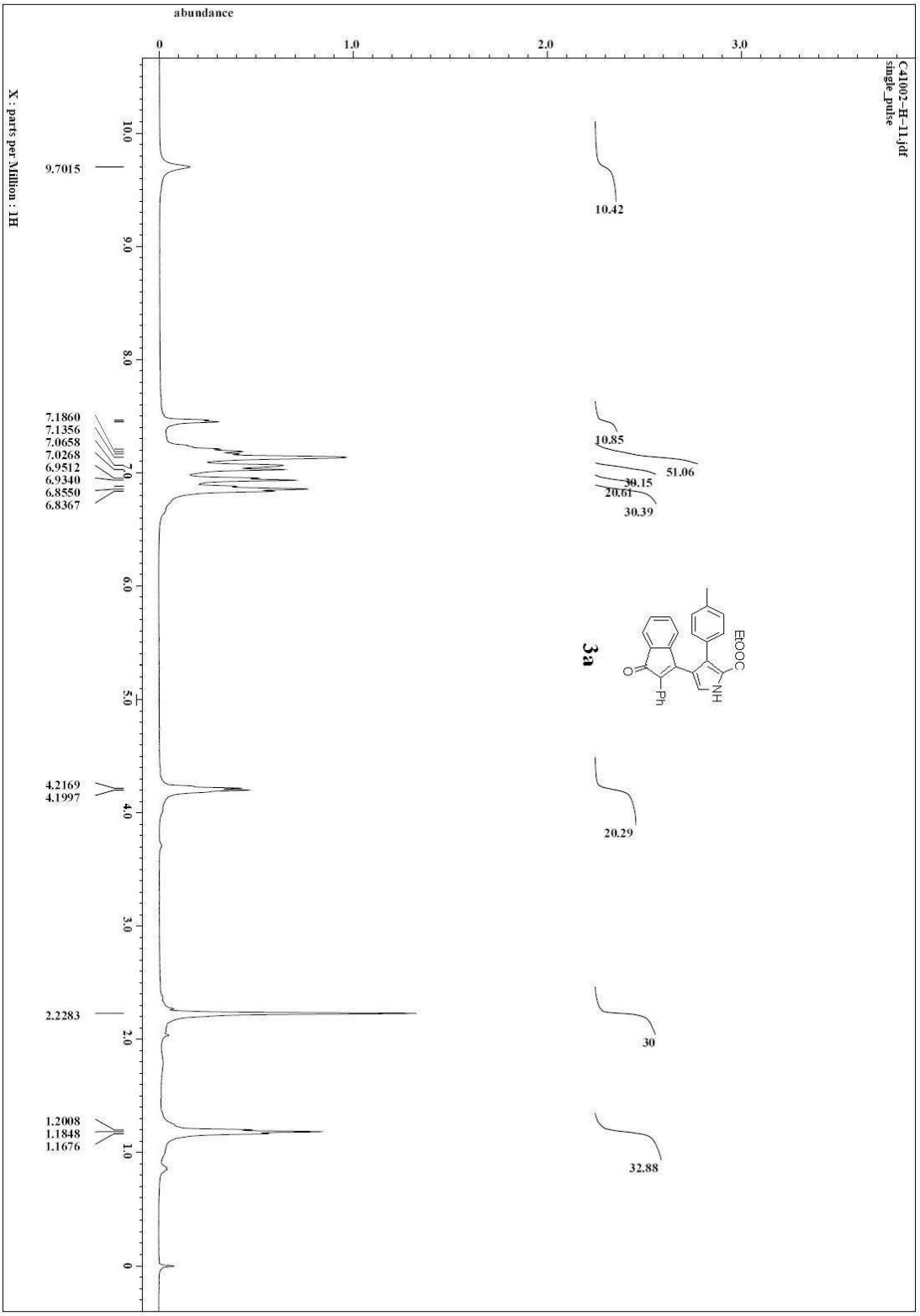
Ethyl 4-(5-chloro-1-oxo-2-phenyl-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3o**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H), 7.37 (d, *J* = 7.2 Hz, 1H), 7.08-7.15 (m, 6H), 7.01 (s, 1H), 6.94 (d, *J* = 7.2 Hz, 2H), 6.87 (d, *J* = 7.6 Hz, 2H), 6.82 (s, 1H), 4.21 (d, *J* = 7.2 Hz, 2H), 2.24 (s, 3H), 1.19 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.0, 160.9, 148.9, 147.2, 139.5, 136.7, 134.2, 131.0, 130.8, 130.1, 129.7, 129.4, 128.9, 128.1, 128.0, 127.9, 127.6, 123.2, 122.4, 122.1, 120.3, 117.2, 60.7, 21.2, 14.2; HRMS (ESI) calcd for $C_{29}H_{22}ClNO_3$: 468.1366 (M + H^+), found: 468.1342.

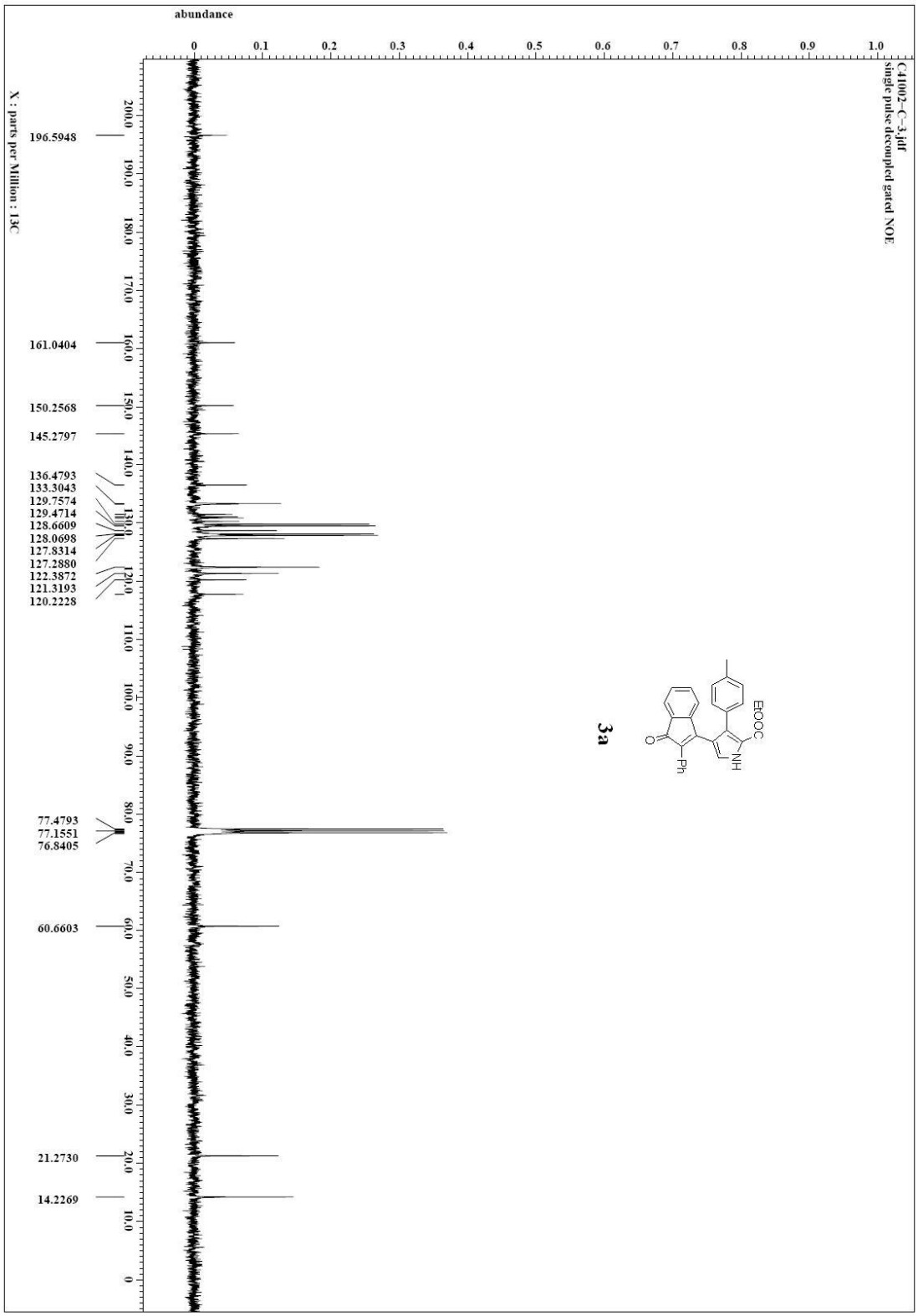


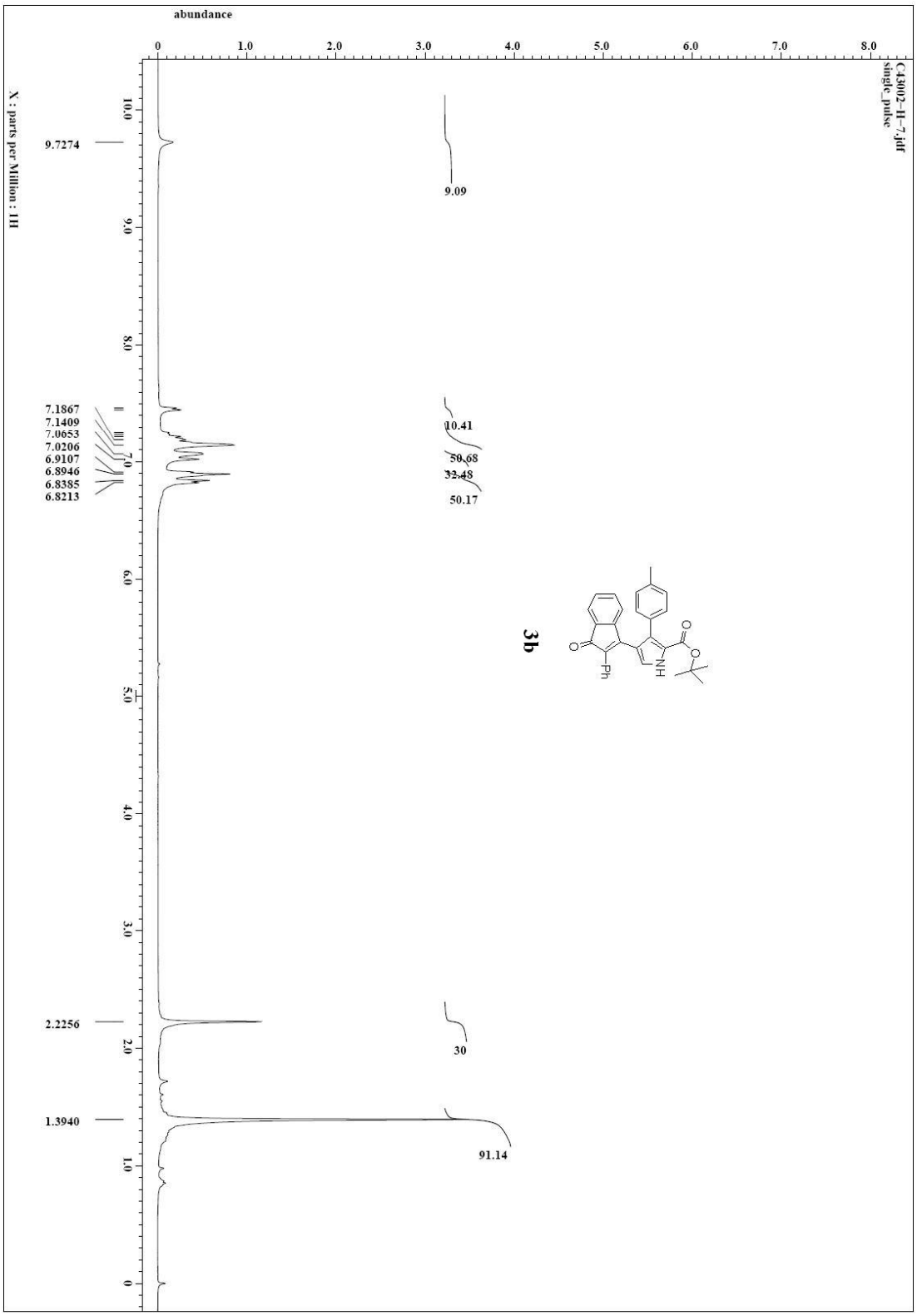
Ethyl 4-(6-methoxy-1-oxo-2-phenyl-1*H*-inden-3-yl)-3-*p*-tolyl-1*H*-pyrrole-2-carboxylate (**3p**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.65 (s, 1H), 7.06-7.12 (m, 6H), 6.95-7.00 (m, 3H), 6.86 (d, *J* = 7.2 Hz, 2H), 6.73 (d, *J* = 7.6 Hz, 1H), 6.62 (d, *J* = 7.6 Hz, 1H), 4.21 (d, *J* = 6.4 Hz, 2H), 3.78 (s, 3H), 2.24 (s, 3H), 1.19 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.0, 160.9, 148.9, 147.2, 139.5, 136.7, 134.2, 131.0, 130.8, 130.1, 129.7, 129.4, 128.9, 128.1, 128.0, 127.9, 127.6, 123.2, 122.4, 122.1, 120.3, 117.2, 60.7, 21.2, 14.2; HRMS (ESI) calcd for $C_{30}H_{25}NO_4$: 464.1862 (M + H^+), found: 464.1842.

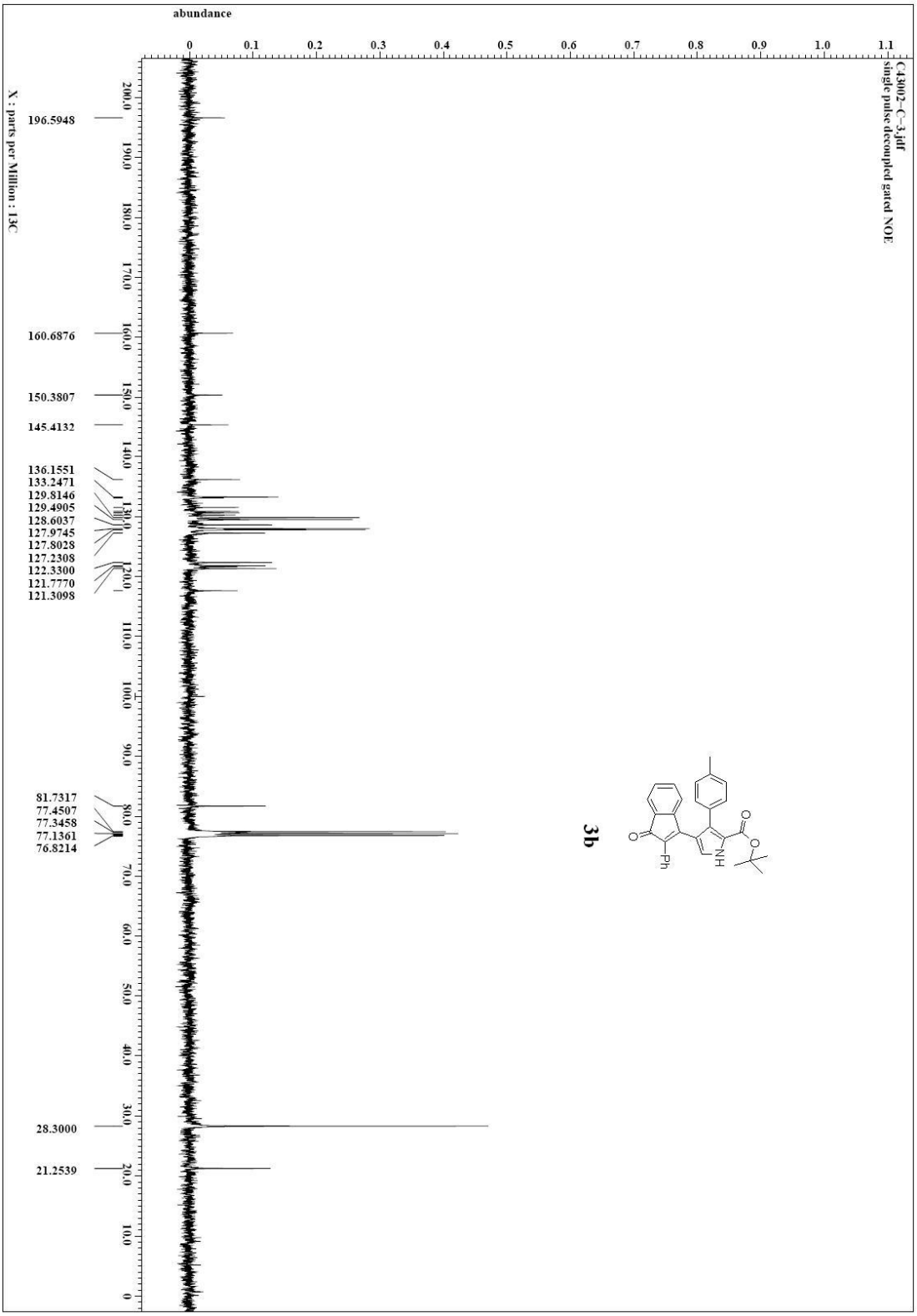


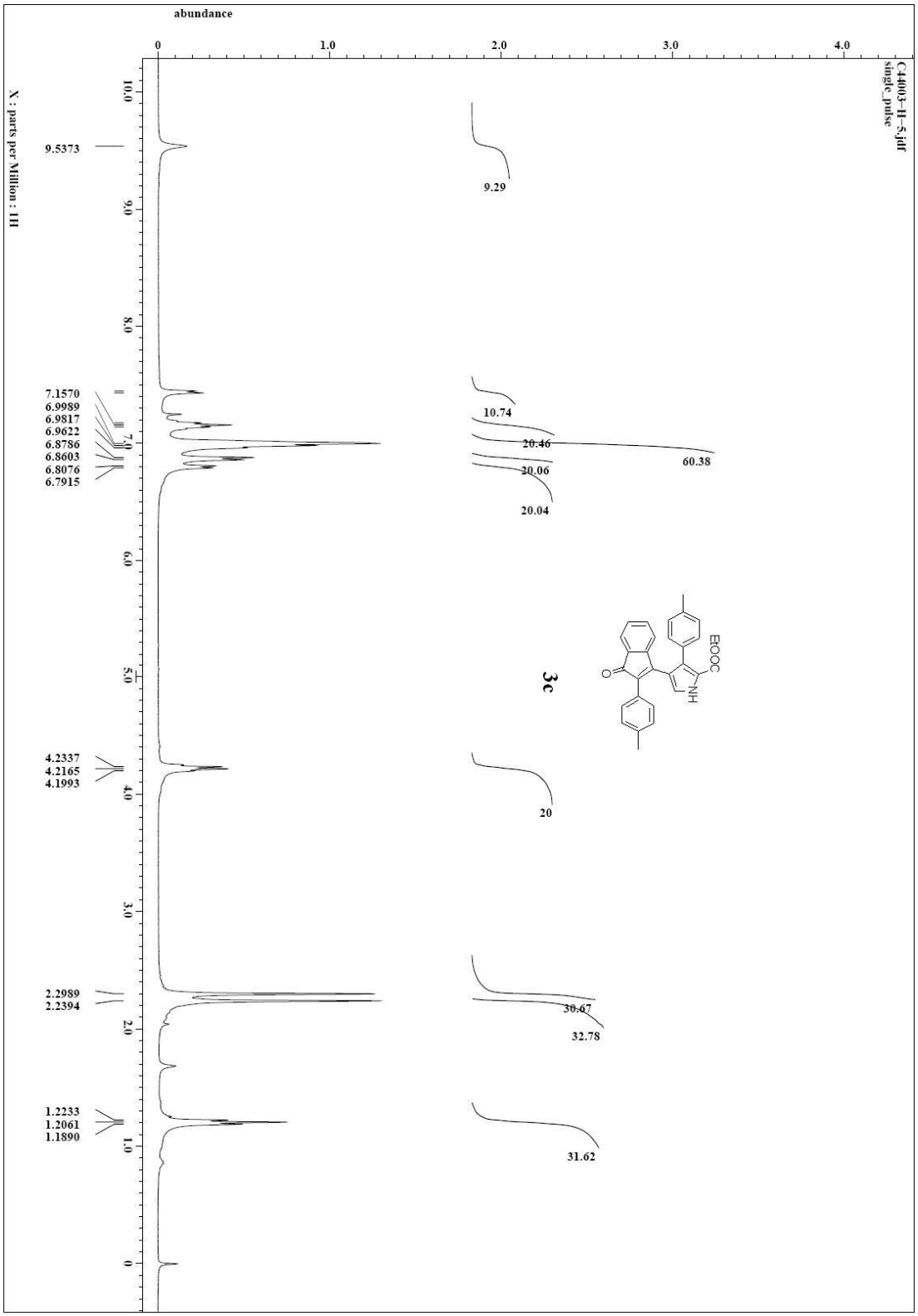
Ethyl 4-(2-(phenylethynyl)benzoyl)-3-*p*-tolyl-2,3-dihydro-1*H*-pyrrole-2-carboxylate (**B1**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 6.8 Hz, 1H), 7.23-7.34 (m, 10H), 7.08 (d, *J* = 6.8 Hz, 1H), 6.91 (d, *J* = 7.6 Hz, 2H), 5.39 (s, 1H), 4.68 (d, *J* = 4.4 Hz, 1H), 4.19-4.22 (m, 3H), 2.22 (s, 1H), 1.28 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.1, 172.1, 153.5, 143.7, 140.2, 136.2, 132.7, 131.9, 129.4, 128.7, 128.3, 128.1, 127.9, 127.6, 127.0, 123.1, 120.6, 118.1, 93.0, 87.8, 69.7, 61.9, 49.3, 21.1, 14.2; HRMS (ESI) calcd for $C_{29}H_{26}NO_3$: 436.1907 ($M + H^+$), found: 436.1906.

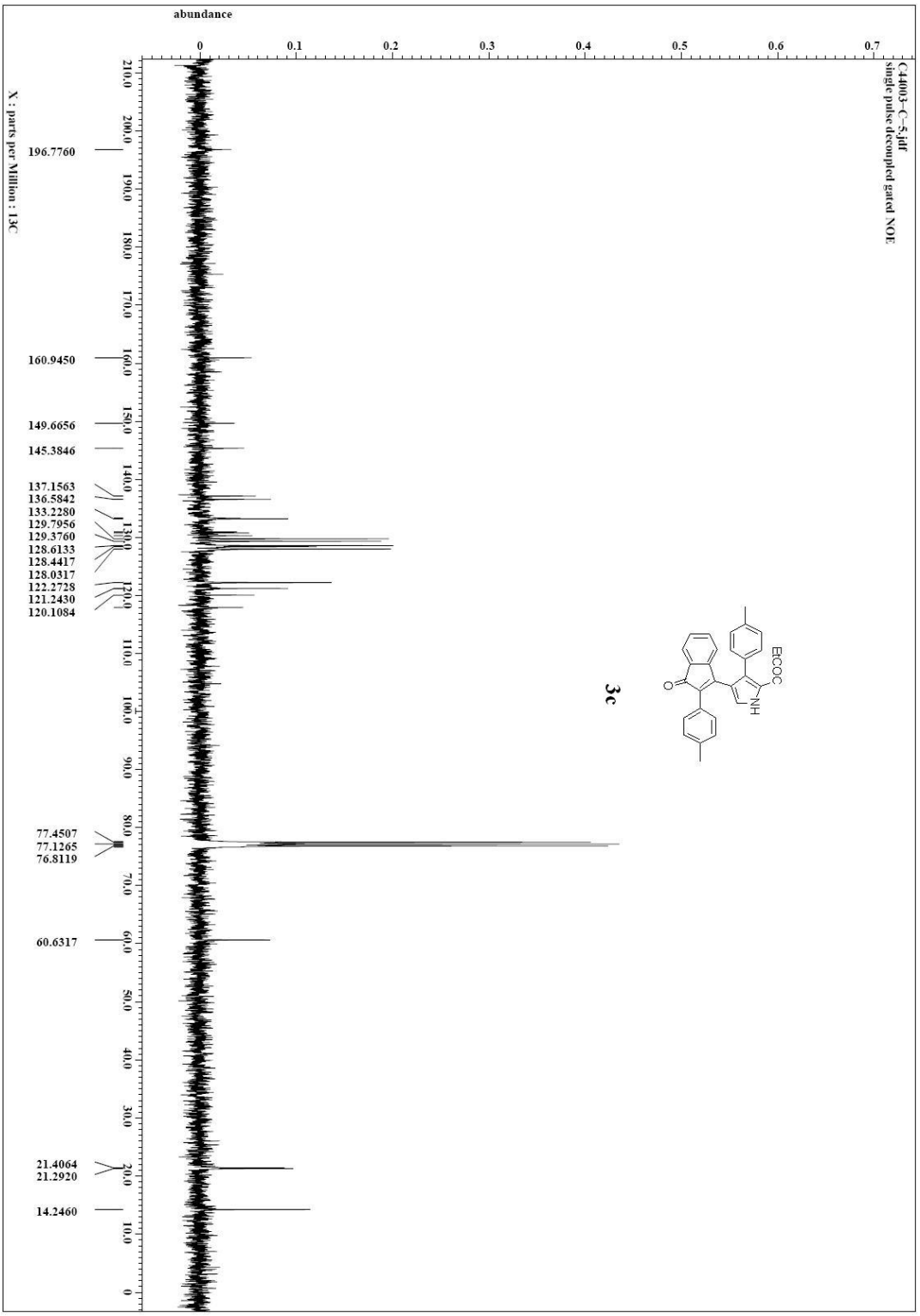

abundance
0
1.0
2.0
3.0
C41002-H-11.jdf
single_pulse
X : parts per Million : 1H
9.7015
7.1860
7.1356
7.0658
7.0268
6.9512
6.9340
6.8550
6.8367
4.2169
4.1997
2.2283
1.2008
1.1848
1.1676
10.42
10.85
51.06
30.15
20.61
30.39
20.29
30
32.88
3a
EtOOC
NH
Ph
O

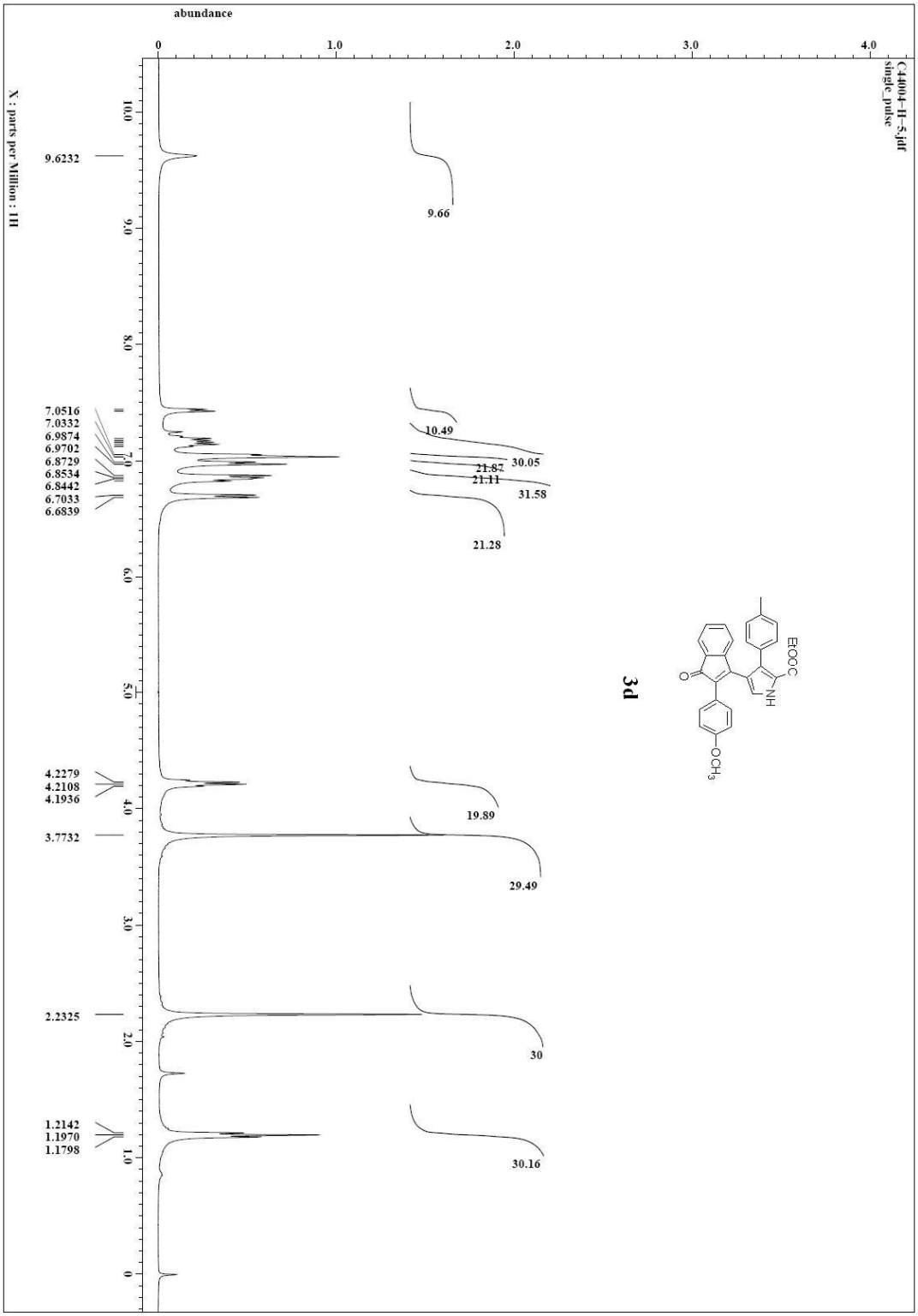

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
C41002-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.5948
161.0404
150.2568
145.2797
136.4793
133.3043
129.7574
129.4714
128.6609
128.0698
127.8314
127.2880
122.3872
121.3193
120.2228
77.4793
77.1551
76.8405
60.6603
21.2730
14.2269
3a
EtOOC
NH
Ph
O

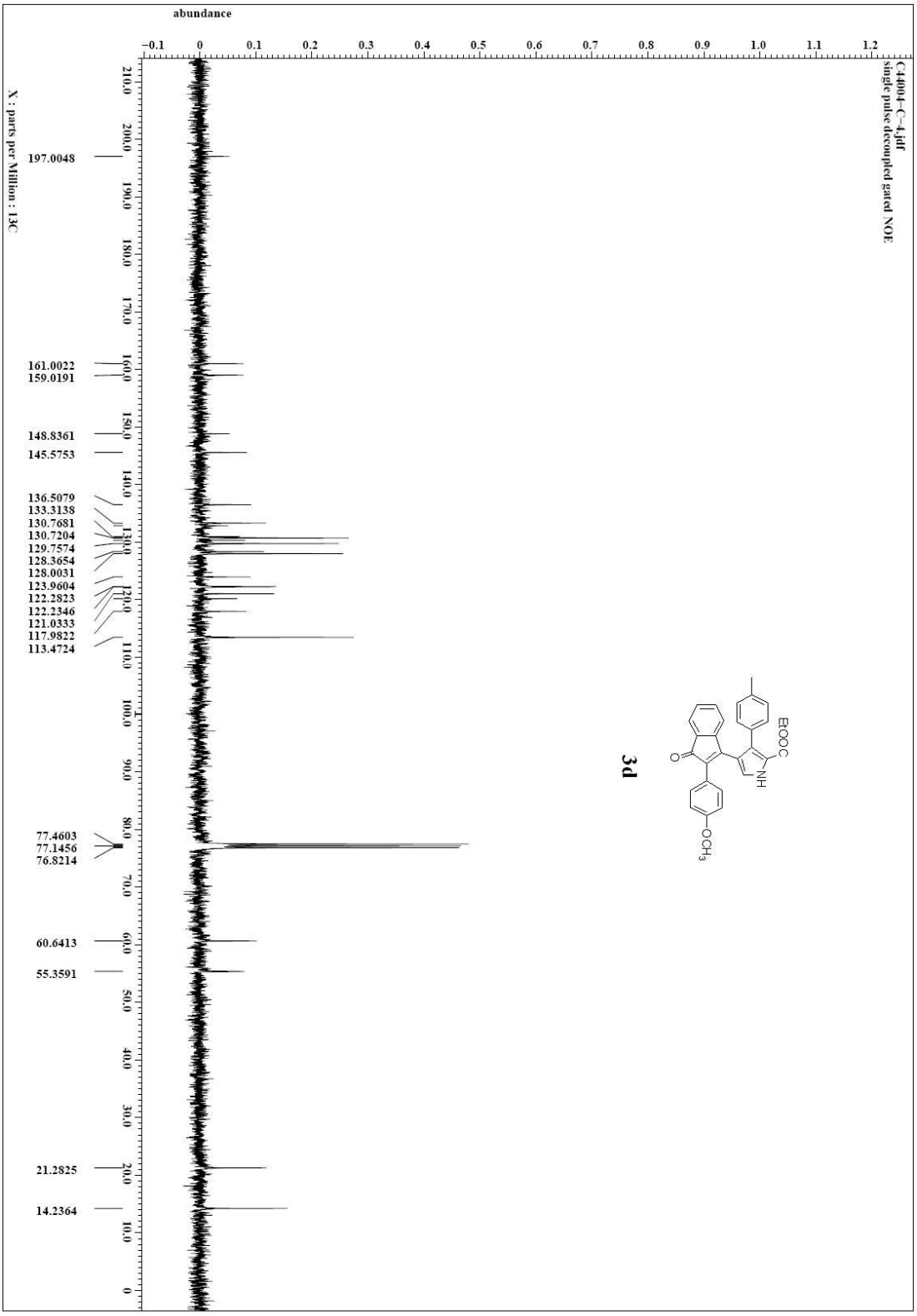

abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
C4302-H-7.jdf
single_pulse
X : parts per Million : 1H
9.7274
7.1867
7.1409
7.0653
7.0206
6.9107
6.8946
6.8385
6.8213
2.2256
1.3940
9.09
10.41
50.68
32.48
50.17
30
91.14
3b
Ph
NH

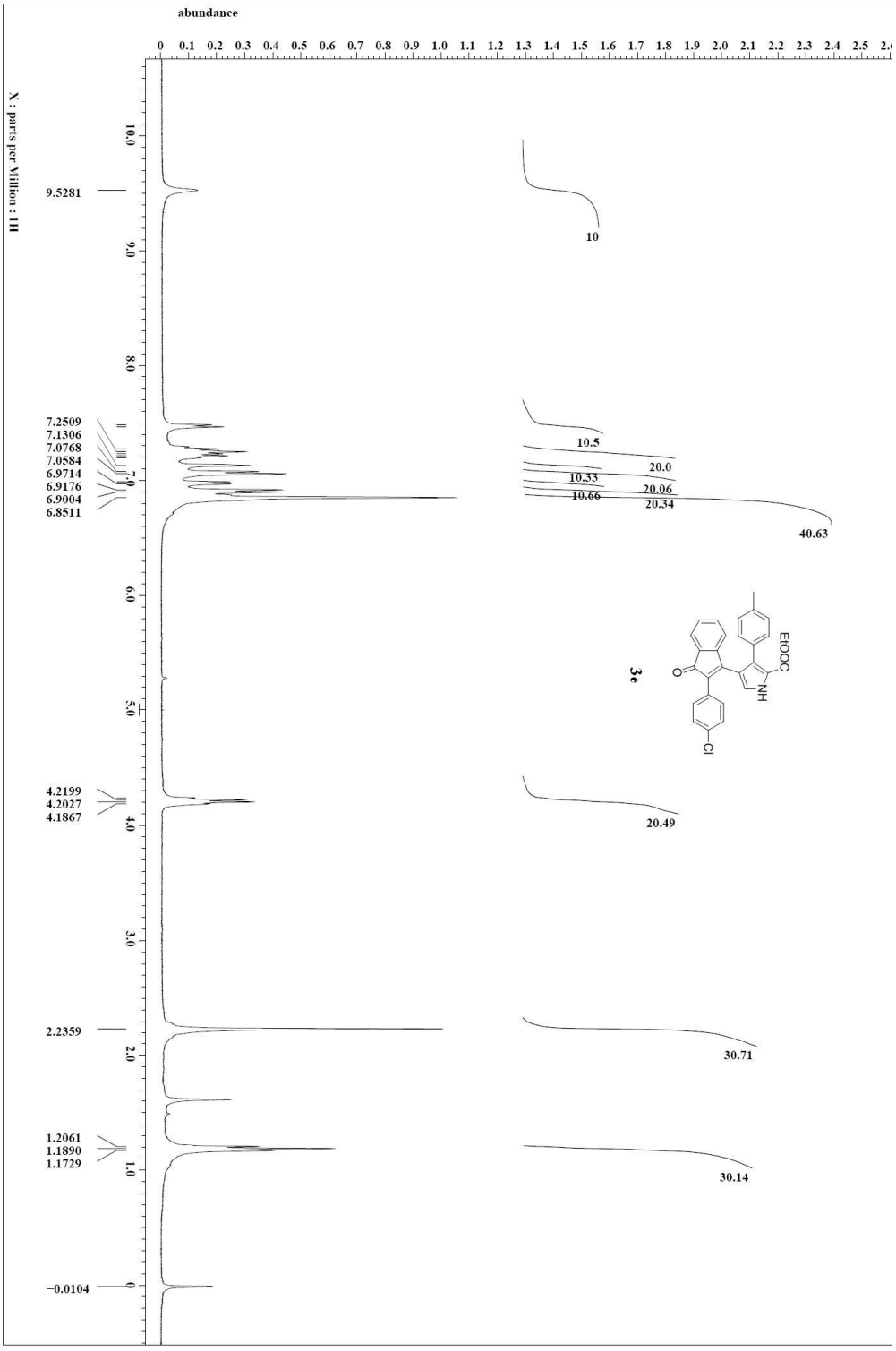

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
C43002-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.5948
160.6876
150.3807
145.4132
136.1551
133.2471
129.8146
129.4905
128.6037
127.9745
127.8028
127.2308
122.3300
121.7770
121.3098
81.7317
77.4507
77.3458
77.1361
76.8214
28.3000
21.2539
3b
O
O
O
NH
Ph

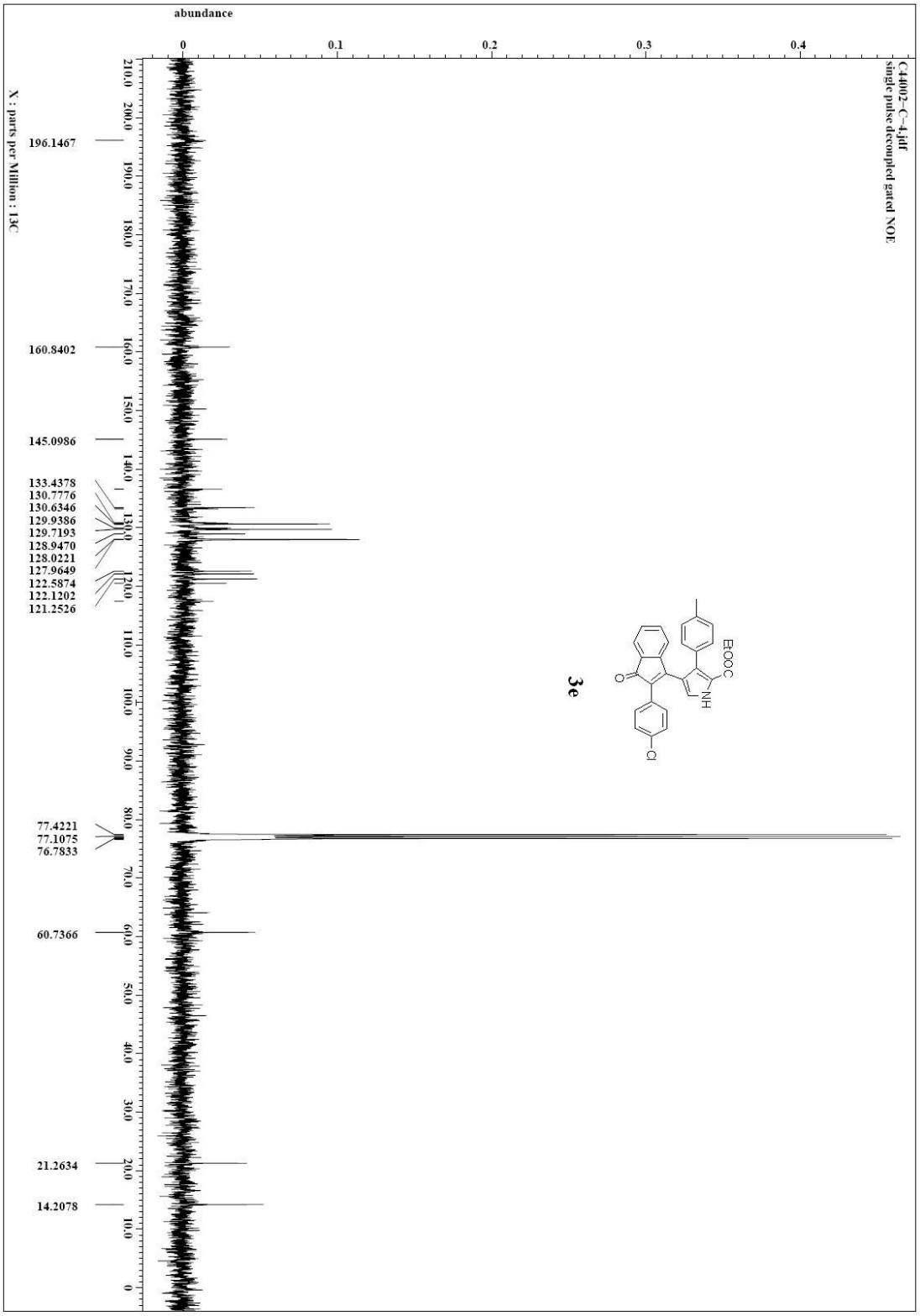

abundance
0
1.0
2.0
3.0
4.0
C4403-H-5.jdf
single_pulse
X : parts per Million : 1H
9.5373
7.1570
6.9989
6.9817
6.9622
6.8786
6.8603
6.8076
6.7915
4.2337
4.2165
4.1993
2.2989
2.2394
1.2233
1.2061
1.1890
9.29
10.74
20.46
60.38
20.06
20.04
20
30.67
32.78
31.62
3c
EtOOC
NH
O

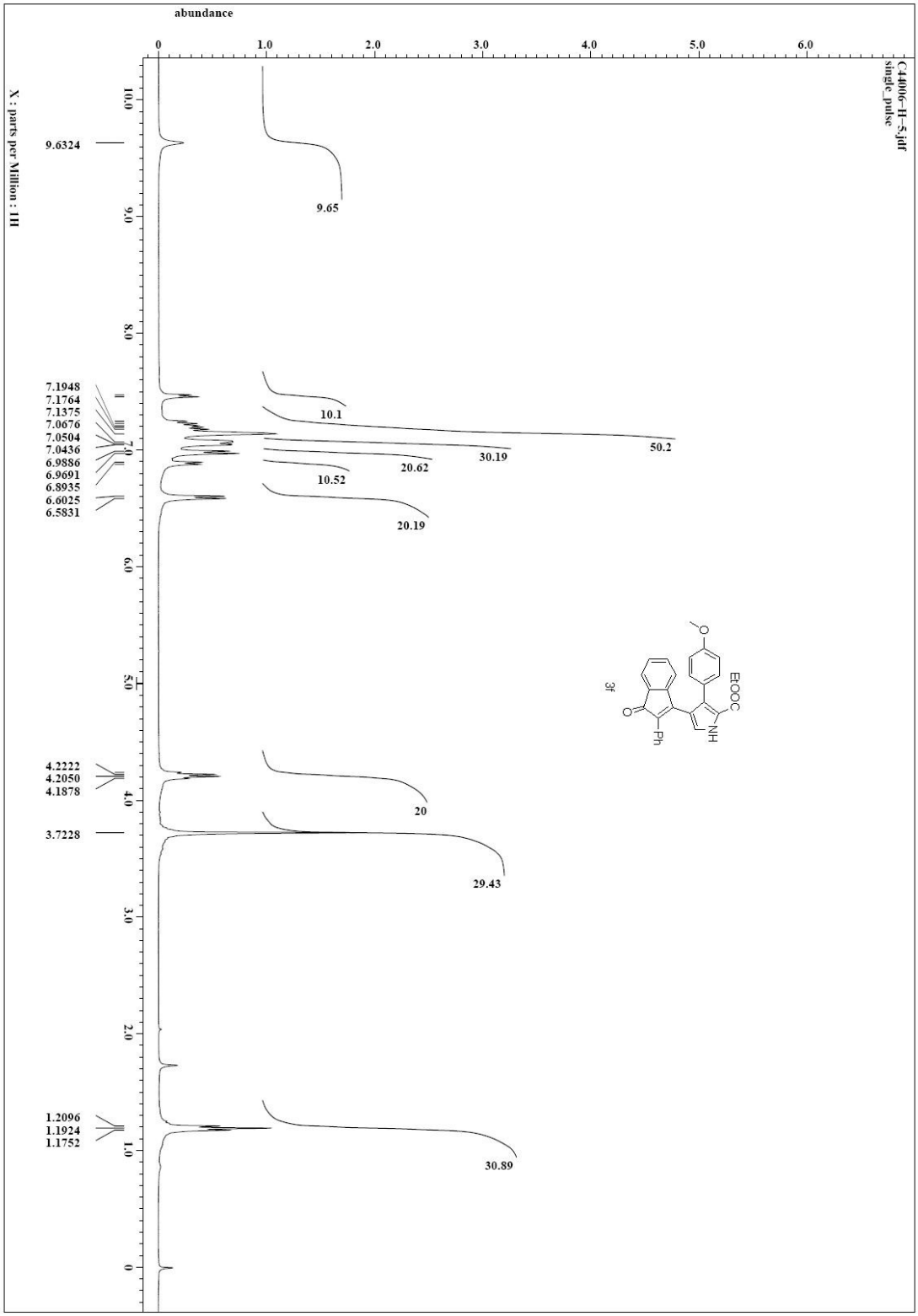

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
C44003--C--5.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.7760
160.9450
149.6656
145.3846
137.1563
136.5842
133.2280
129.7956
129.3760
128.6133
128.4417
128.0317
122.2728
121.2430
120.1084
77.4507
77.1265
76.8119
60.6317
21.4064
21.2920
14.2460
3c
EtCOO
NH
O

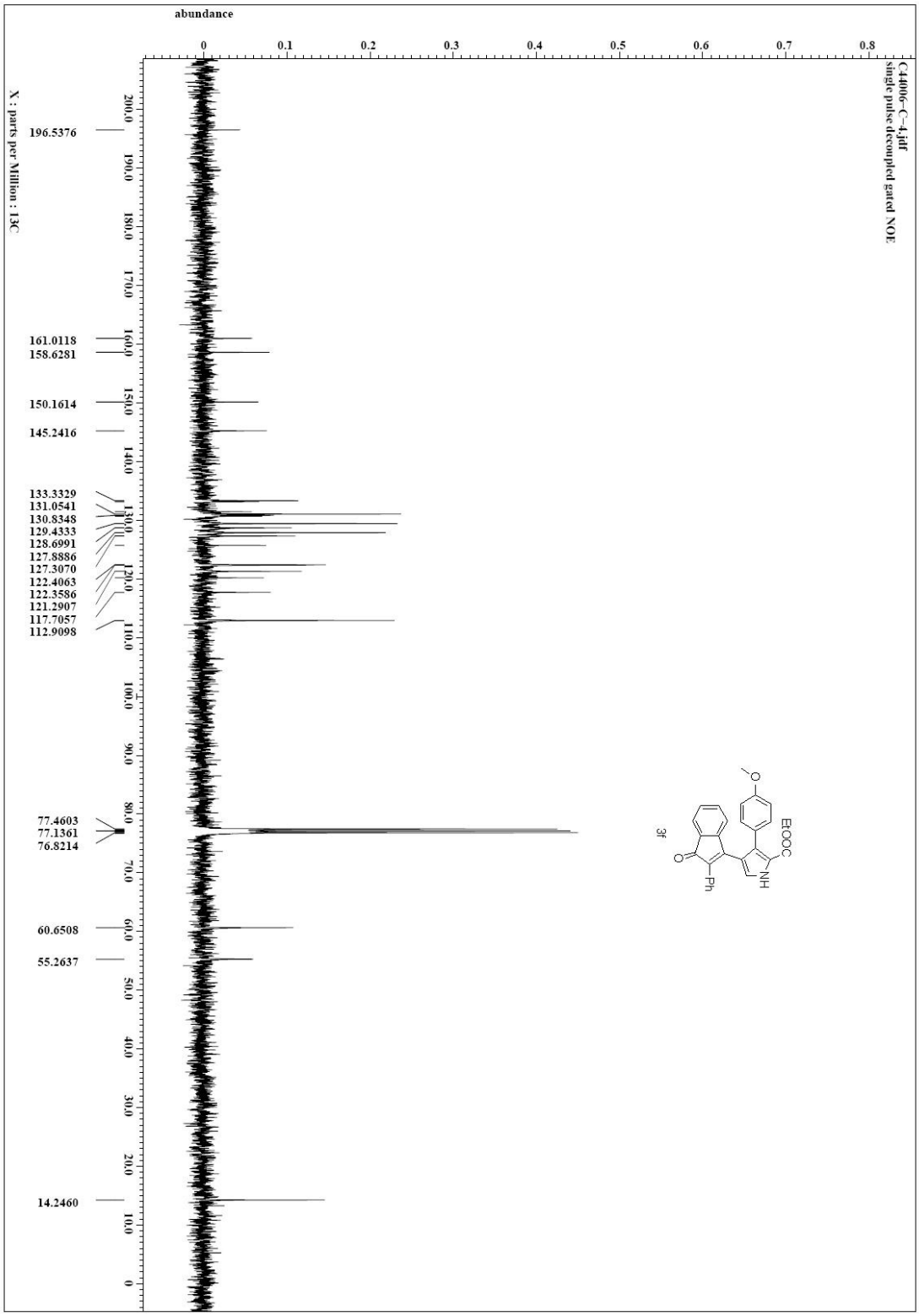

abundance
X : parts per Million : 1H
C4404-H-5.jdf
single_pulse
9.6232
7.0516
7.0332
6.9874
6.9702
6.8729
6.8534
6.8442
6.7033
6.6839
4.2279
4.2108
4.1936
3.7732
2.2325
1.2142
1.1970
1.1798
9.66
10.49
30.05
21.87
21.11
31.58
21.28
19.89
29.49
30
30.16
3d
EtOOC
NH
O
OCH3

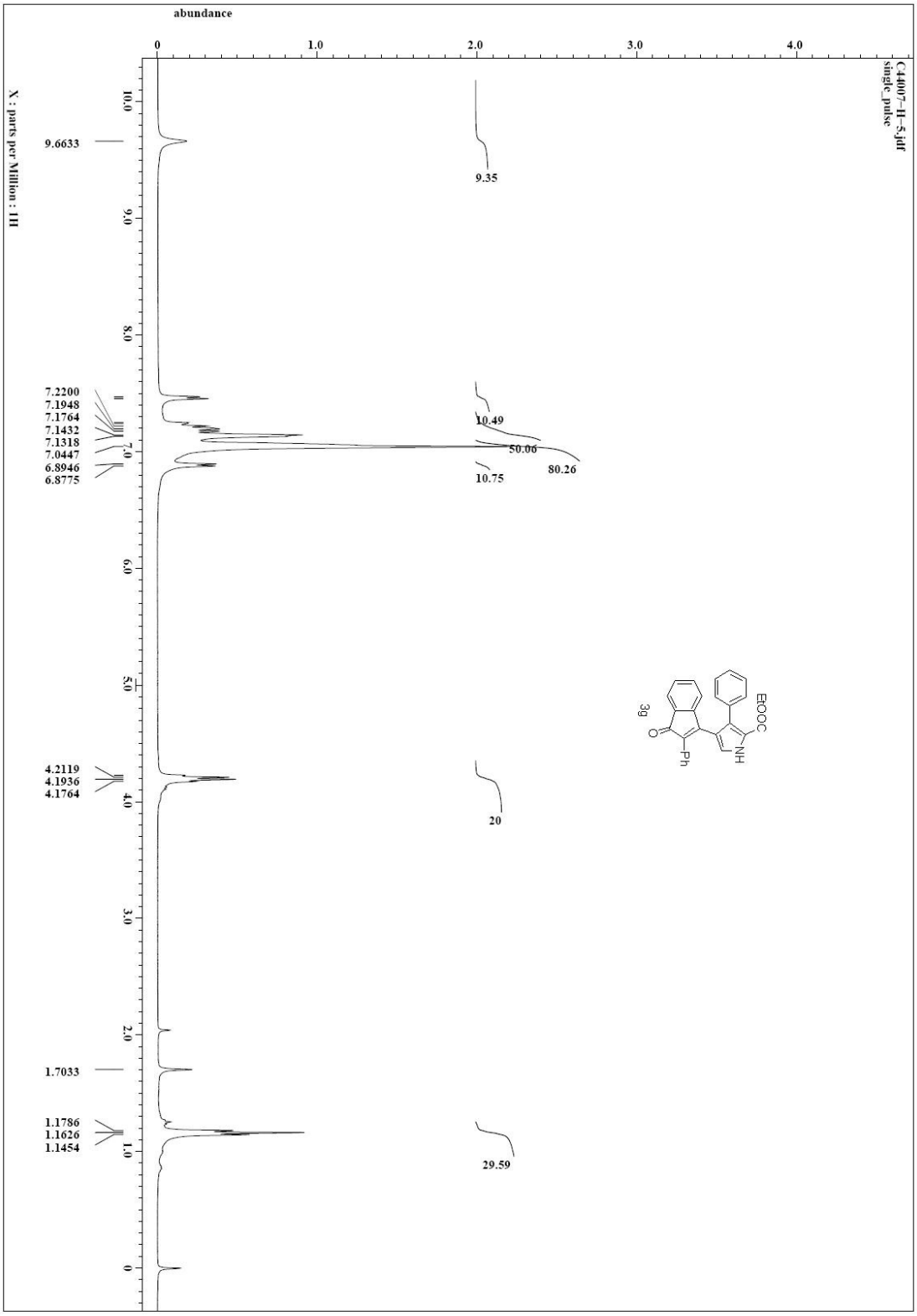

abundance
X : parts per Million : 13C
C:\4004-C-4.jdf
Single pulse decoupled gated NOE
197.0048
161.0022
159.0191
148.8361
145.5753
136.5079
133.3138
130.7681
130.7204
129.7574
128.3654
128.0031
123.9604
122.2823
122.2346
121.0333
117.9822
113.4724
77.4603
77.1456
76.8214
60.6413
55.3591
21.2825
14.2364
3d
EtOOC
NH
OCH3

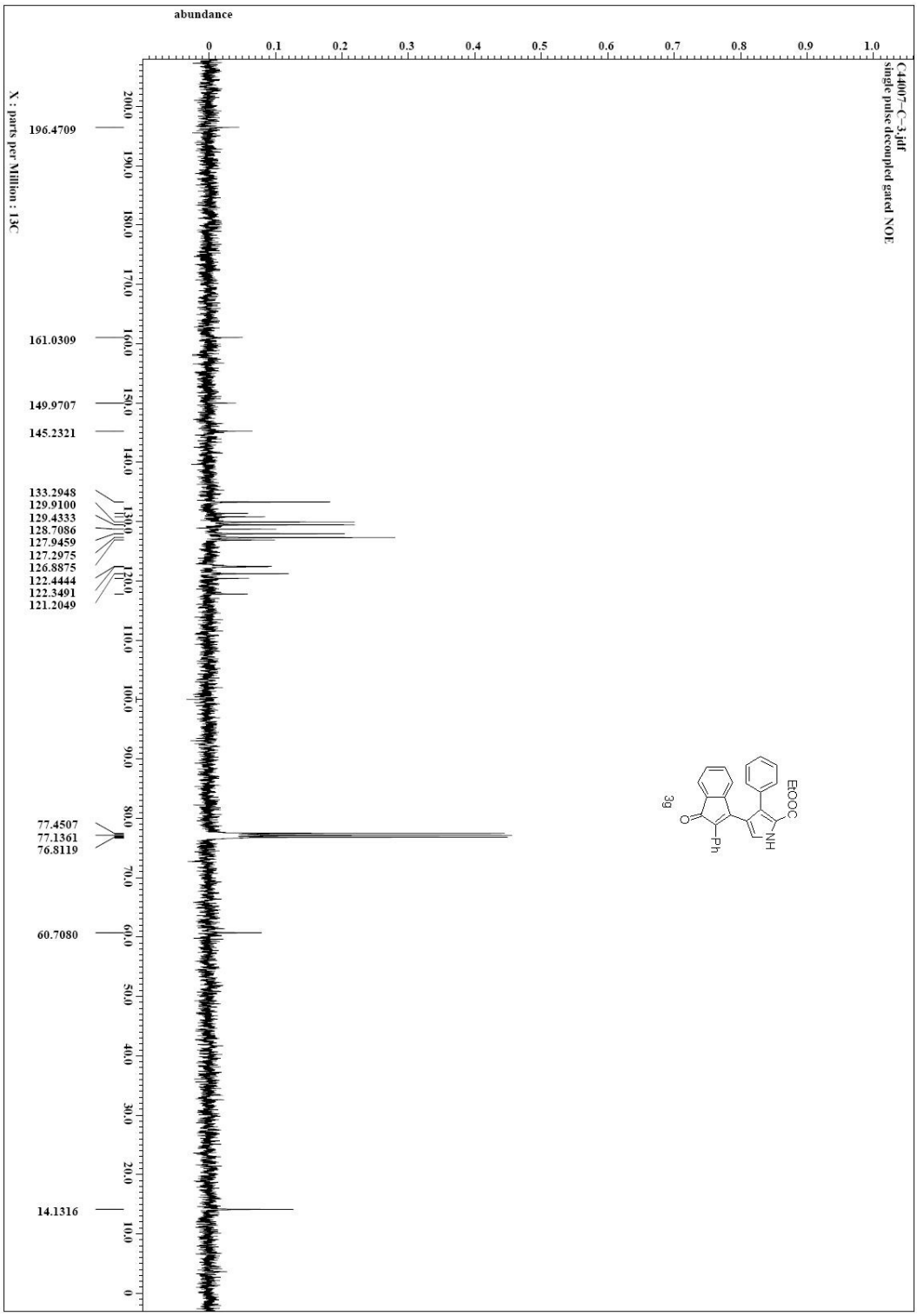

abundance
X : parts per Million : 1H
9.5281
7.2509
7.1306
7.0768
7.0584
6.9714
6.9176
6.9004
6.8511
4.2199
4.2027
4.1867
2.2359
1.2061
1.1890
1.1729
−0.0104
10
10.5
20.0
10.33
20.06
10.66
20.34
40.63
20.49
30.71
30.14
3e
EtOOC
NH
O
Cl

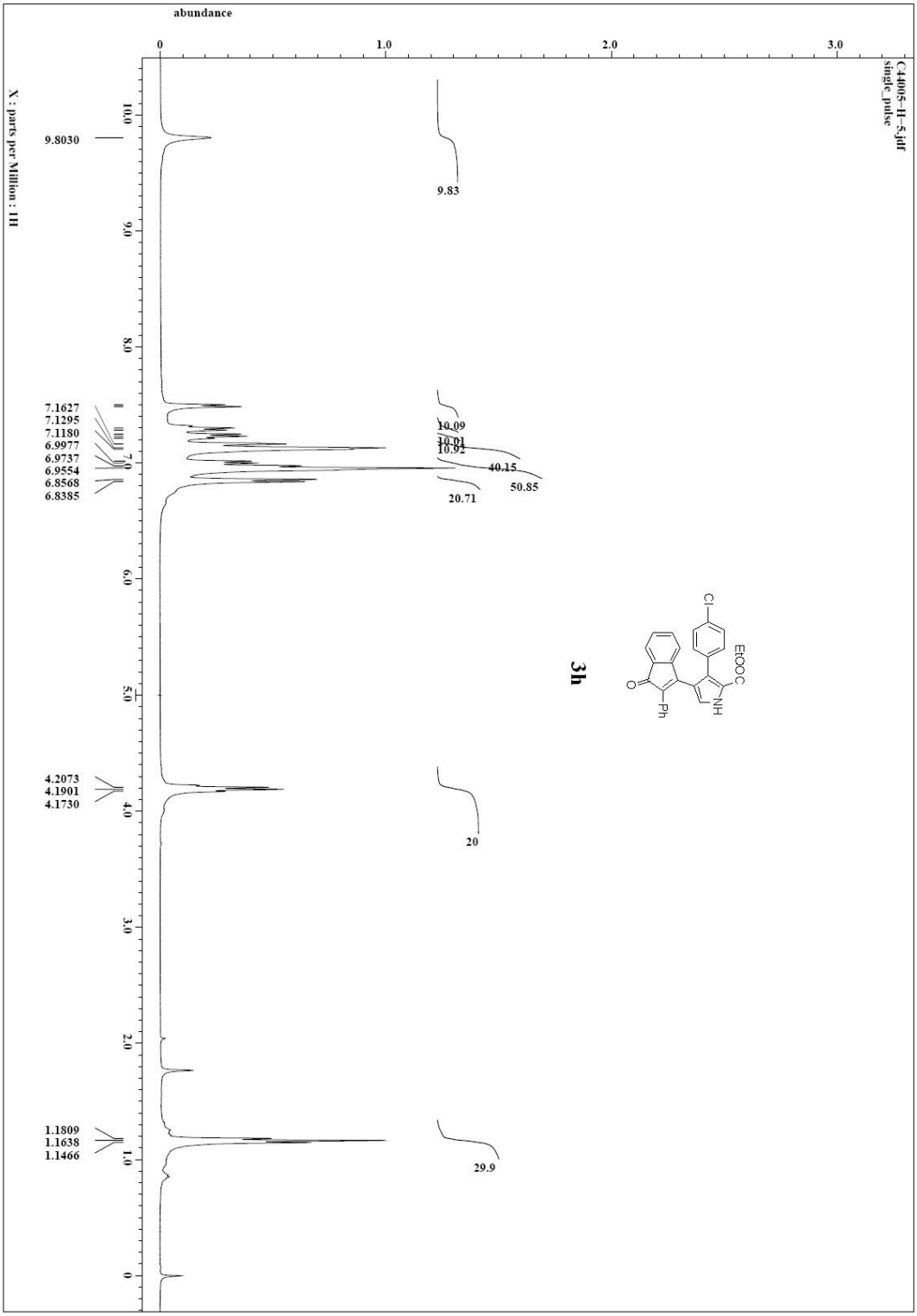

abundance
0
0.1
0.2
0.3
0.4
C:\4002-C-4.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.1467
160.8402
145.0986
133.4378
130.7776
130.6346
129.9386
129.7193
128.9470
128.0221
127.9649
122.5874
122.1202
121.2526
77.4221
77.1075
76.7833
60.7366
21.2634
14.2078
3e
EtOOC
NH
O
Cl


abundance
X : parts per Million : 1H
C44006-H-5.jdf
single_pulse
9.6324
7.1948
7.1764
7.1375
7.0676
7.0504
7.0436
6.9886
6.9691
6.8935
6.6025
6.5831
4.2222
4.2050
4.1878
3.7228
1.2096
1.1924
1.1752
9.65
10.1
50.2
30.19
20.62
10.52
20.19
20
29.43
30.89
3f
EtOOC
NH
Ph
O


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
C44006-C-4.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.5376
161.0118
158.6281
150.1614
145.2416
133.3329
131.0541
130.8348
129.4333
128.6991
127.8886
127.3070
122.4063
122.3586
121.2907
117.7057
112.9098
77.4603
77.1361
76.8214
60.6508
55.2637
14.2460
3f
EtOOC
NH
Ph
O


abundance
X : parts per Million : 1H
C44007-11-5.jdf
single_pulse
9.6633
7.2200
7.1948
7.1764
7.1432
7.1318
7.0447
6.8946
6.8775
4.2119
4.1936
4.1764
1.7033
1.1786
1.1626
1.1454
9.35
10.49
50.06
80.26
10.75
20
29.59
3g
EtOOC
NH
Ph
O


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
C:\4007-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.4709
161.0309
149.9707
145.2321
133.2948
129.9100
129.4333
128.7086
127.9459
127.2975
126.8875
122.4444
122.3491
121.2049
77.4507
77.1361
76.8119
60.7080
14.1316
3g
EtOOC
NH
Ph
O


abundance
X : parts per Million : 1H
C44005-11-5.jdf
single_pulse
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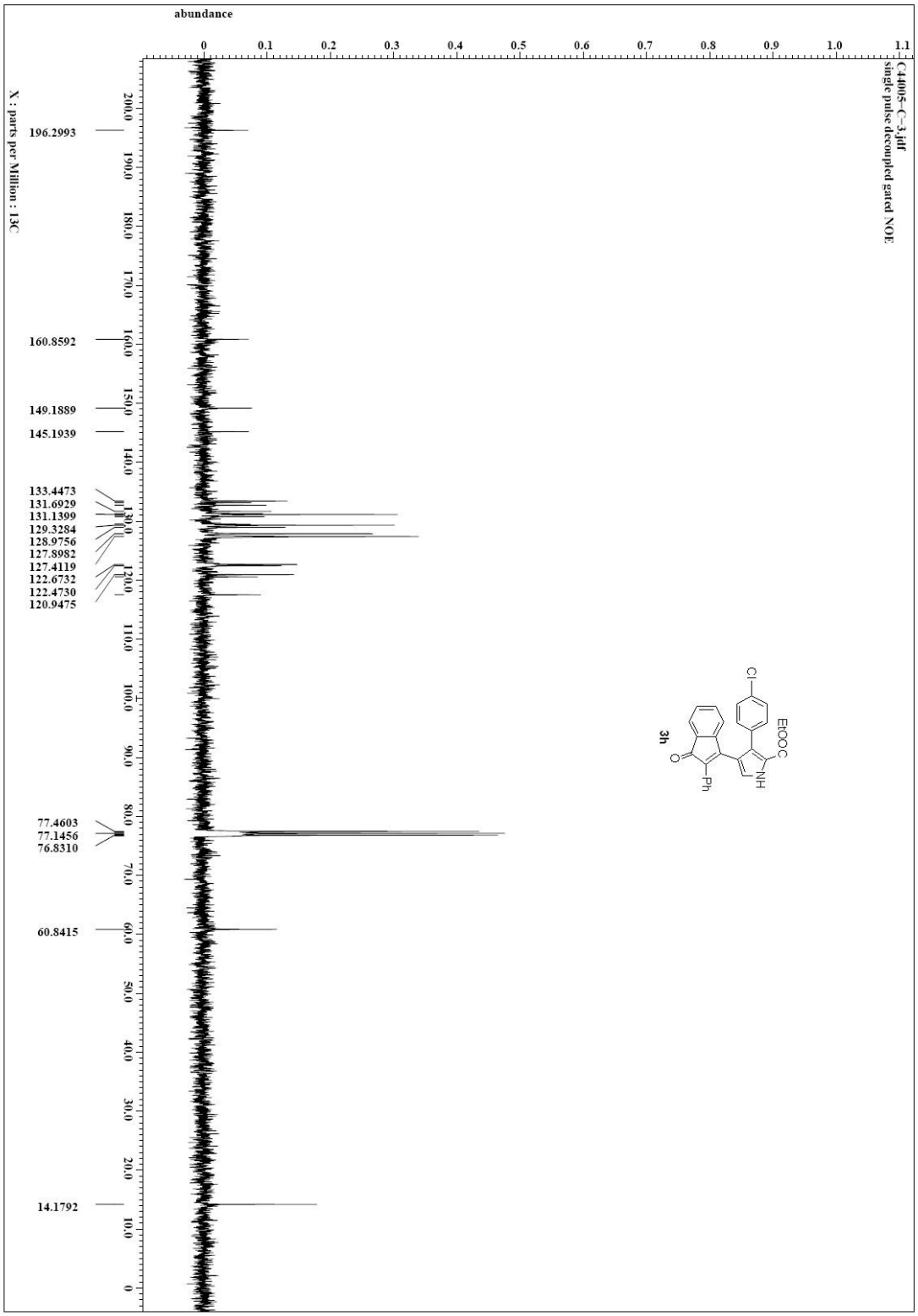
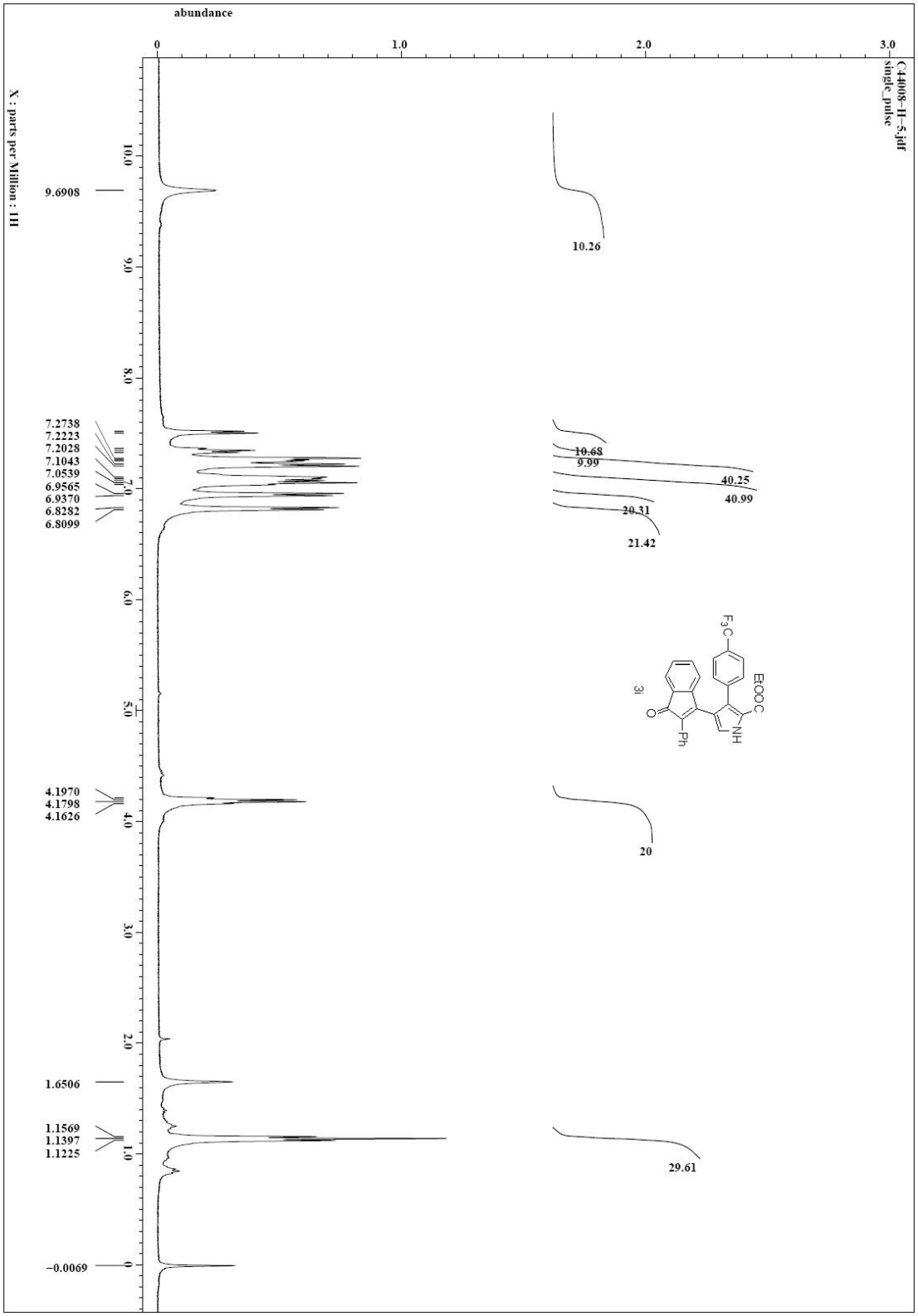
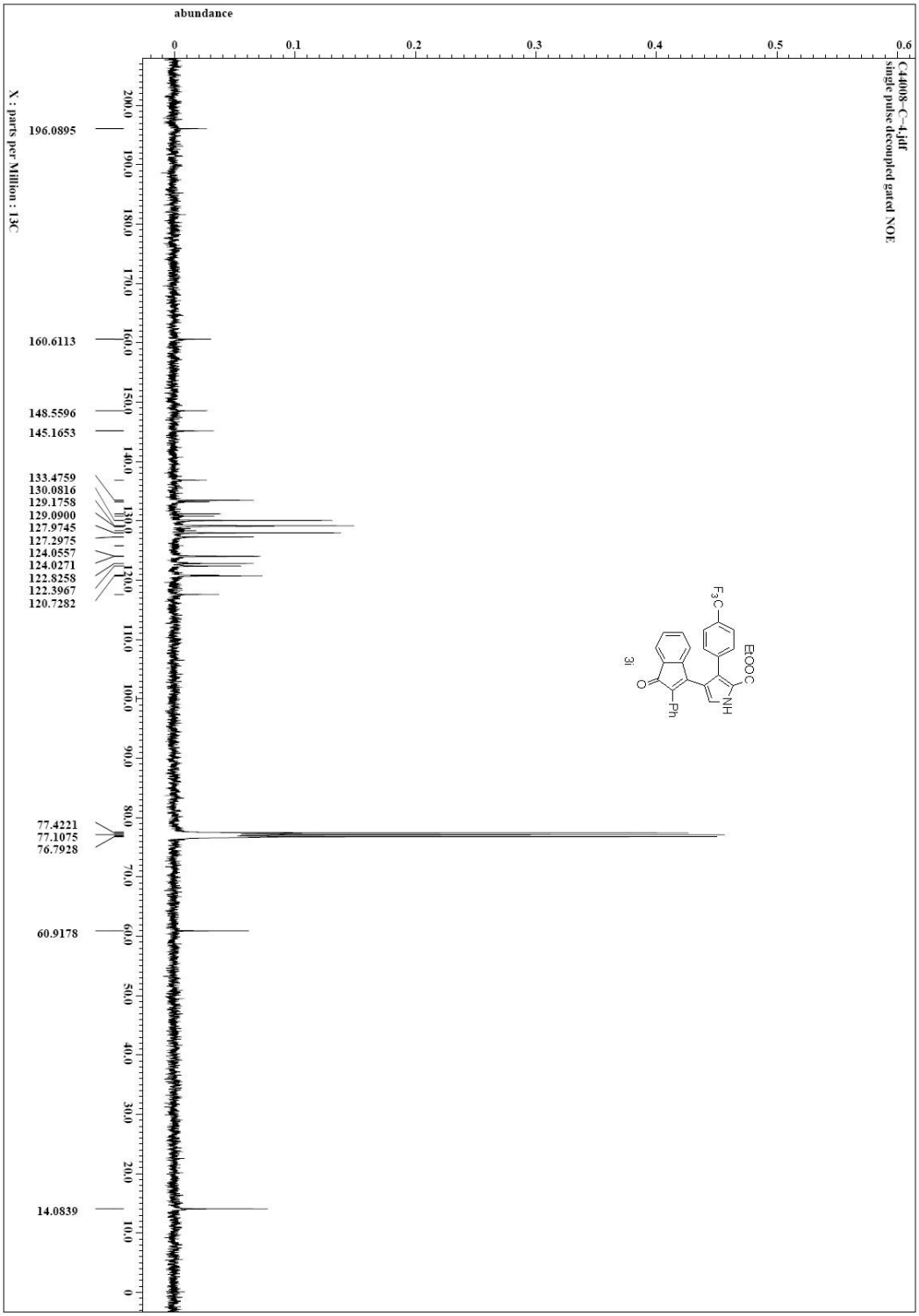
7.1627
7.1295
7.1180
6.9977
6.9737
6.9554
6.8568
6.8385
4.2073
4.1901
4.1730
1.1809
1.1638
1.1466
9.83
10.09
10.01
10.92
40.15
50.85
20.71
20
29.9
3h
Cl
EtOOC
NH
Ph
O


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
C:\4005-C--3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.2993
160.8592
149.1889
145.1939
133.4473
131.6929
131.1399
129.3284
128.9756
127.8982
127.4119
122.6732
122.4730
120.9475
77.4603
77.1456
76.8310
60.8415
14.1792
3h
Cl
EtOOC
NH
Ph
O


abundance
0
1.0
2.0
3.0
C44008-H-5.jdf
single_pulse
X : parts per Million : 1H
9.6908
7.2738
7.2223
7.2028
7.1043
7.0539
6.9565
6.9370
6.8282
6.8099
4.1970
4.1798
4.1626
1.6506
1.1569
1.1397
1.1225
-0.0069
10.26
10.68
9.99
40.25
40.99
20.31
21.42
20
29.61
3l
F3C
EtOOC
NH
Ph
O


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
C44008-C-4.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.0895
160.6113
148.5596
145.1653
133.4759
130.0816
129.1758
129.0900
127.9745
127.2975
124.0557
124.0271
122.8258
122.3967
120.7282
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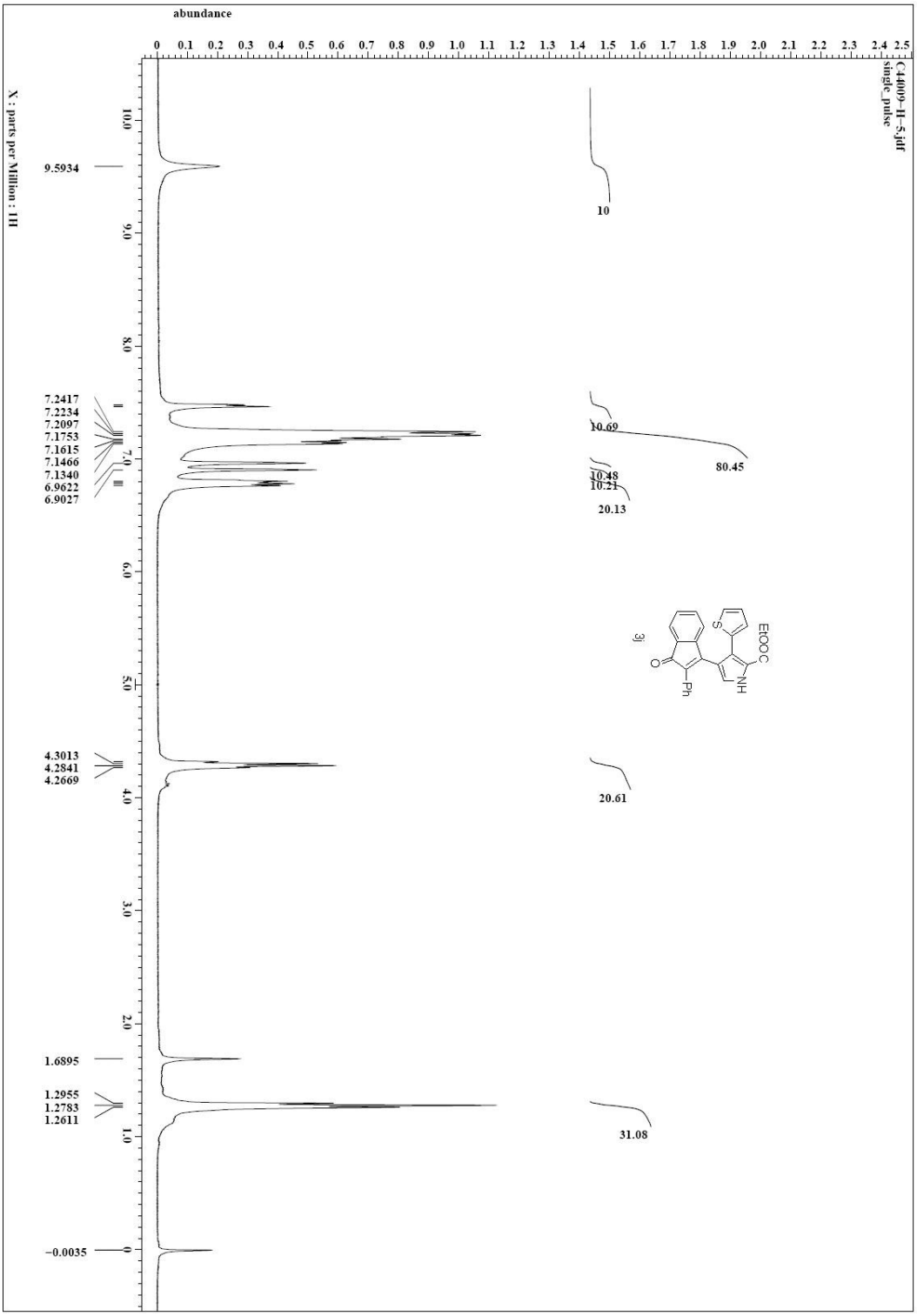
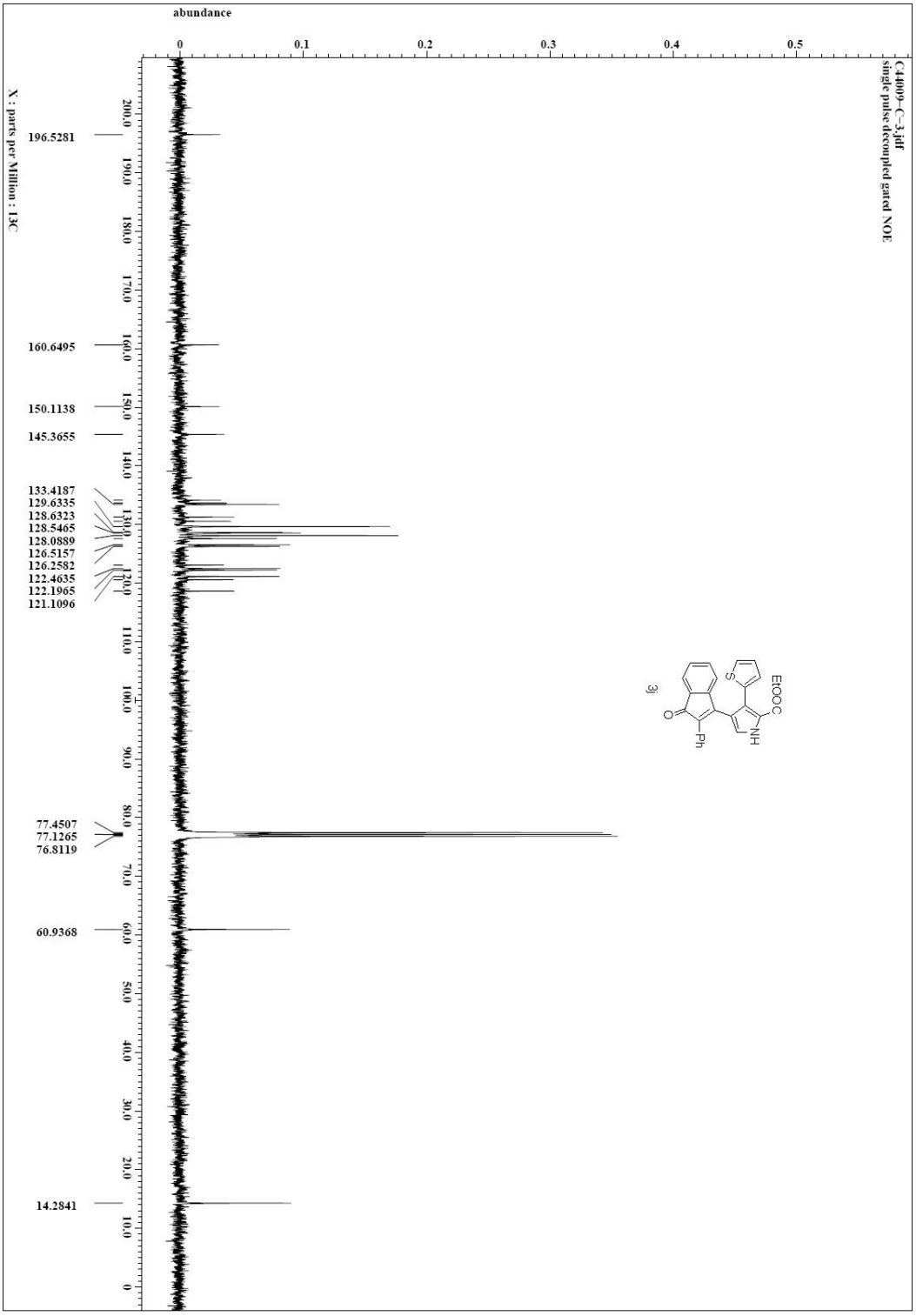
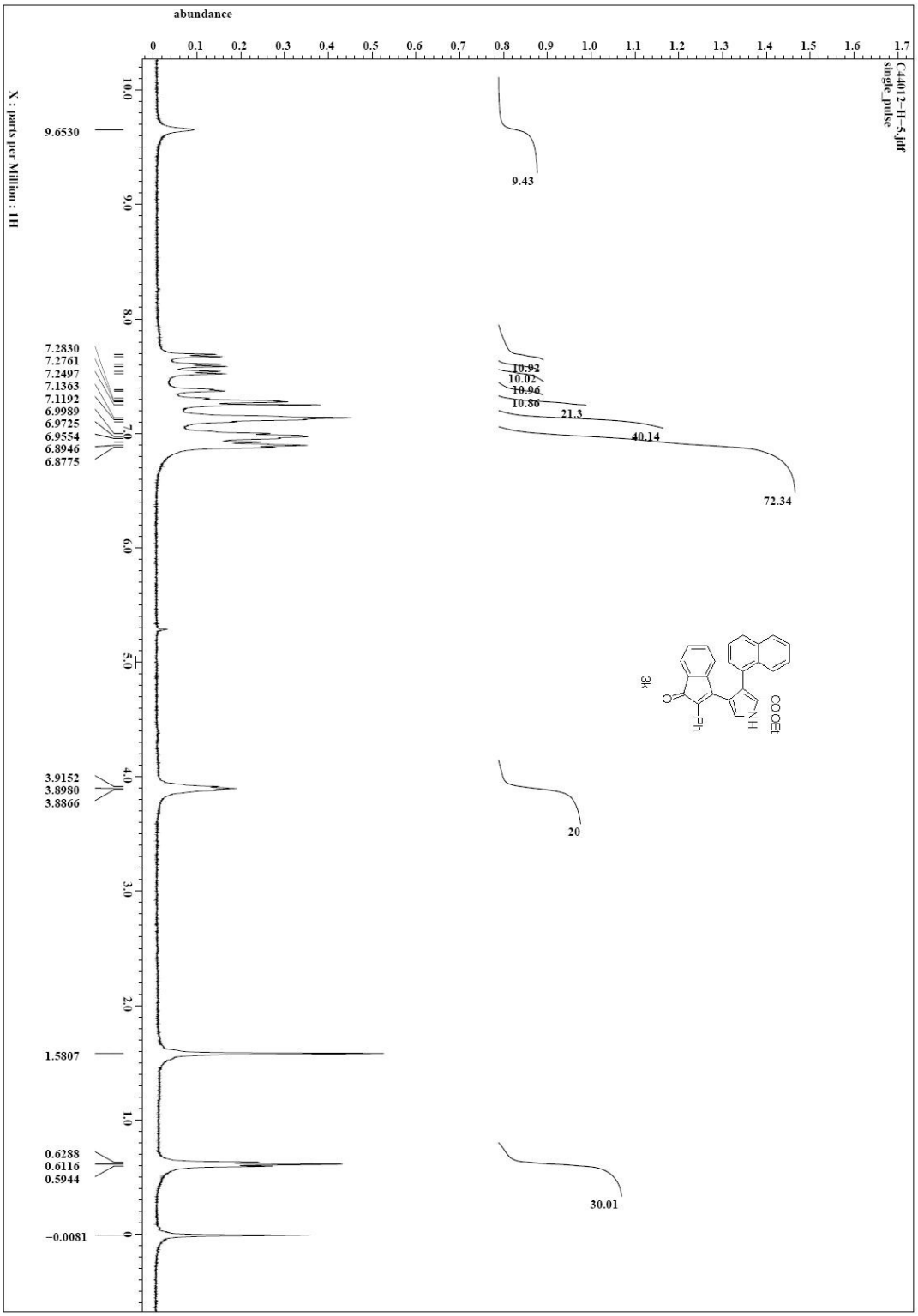
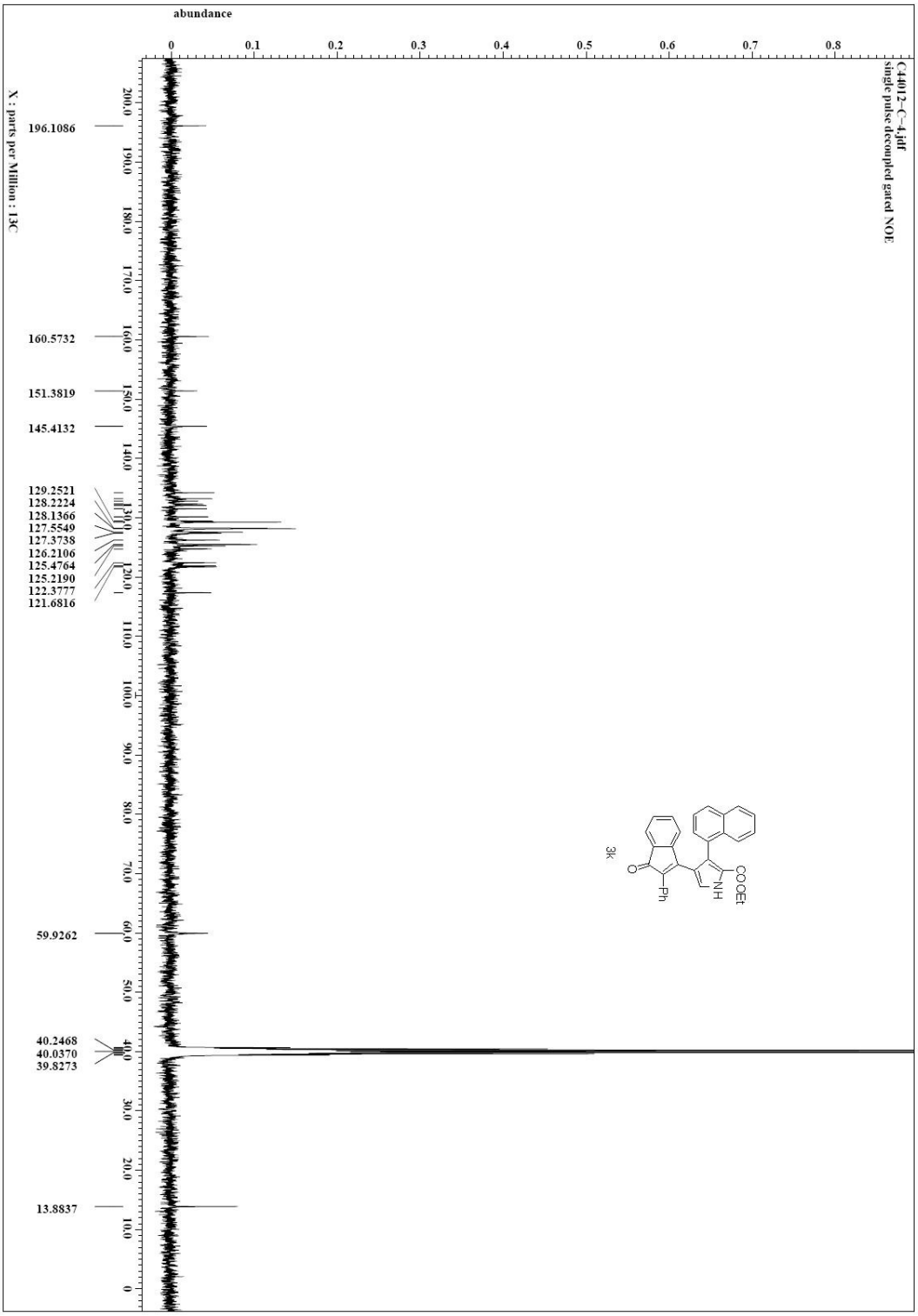
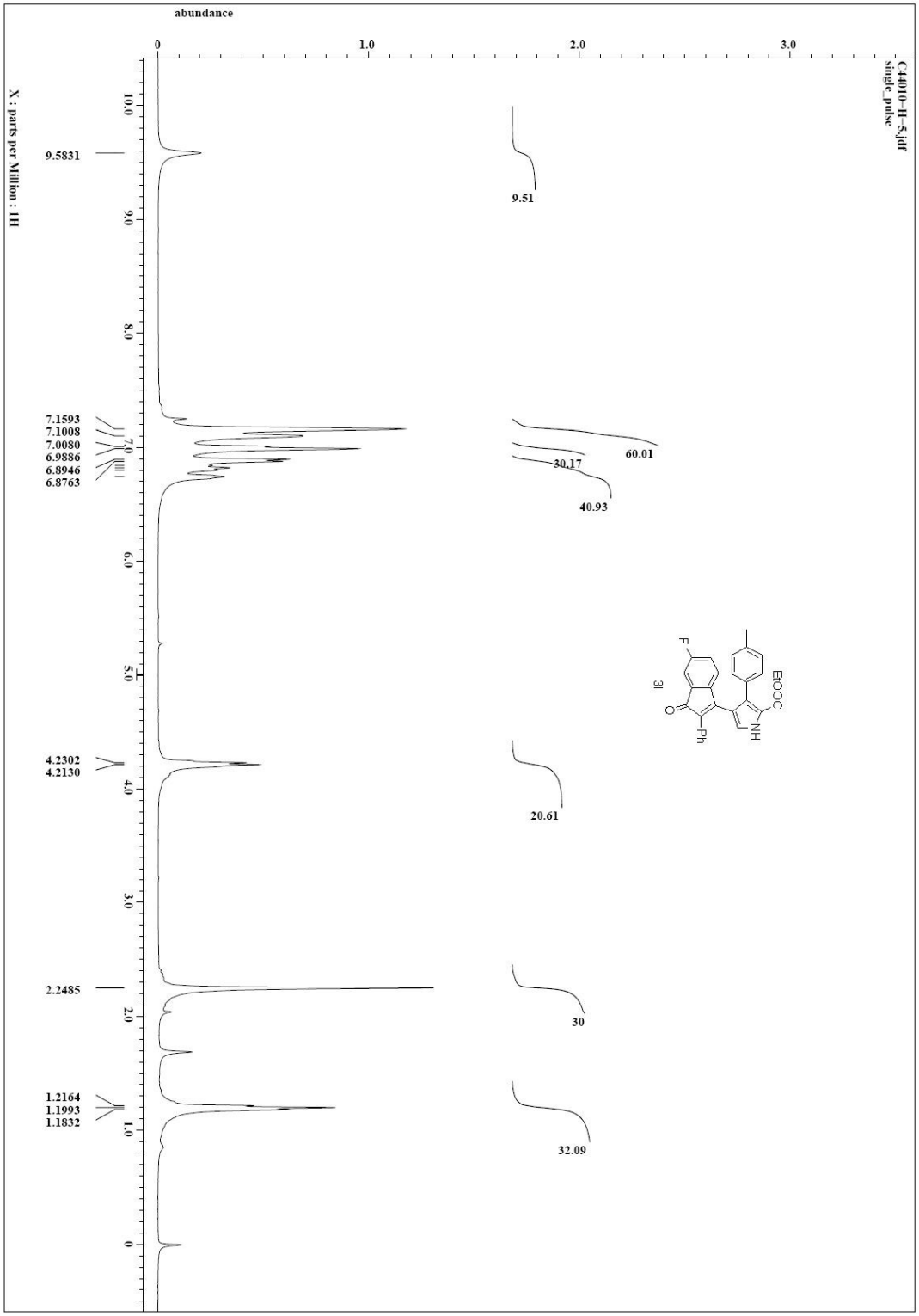
77.1075
76.7928
60.9178
14.0839
3i
F3C
EtOOC
NH
Ph
O

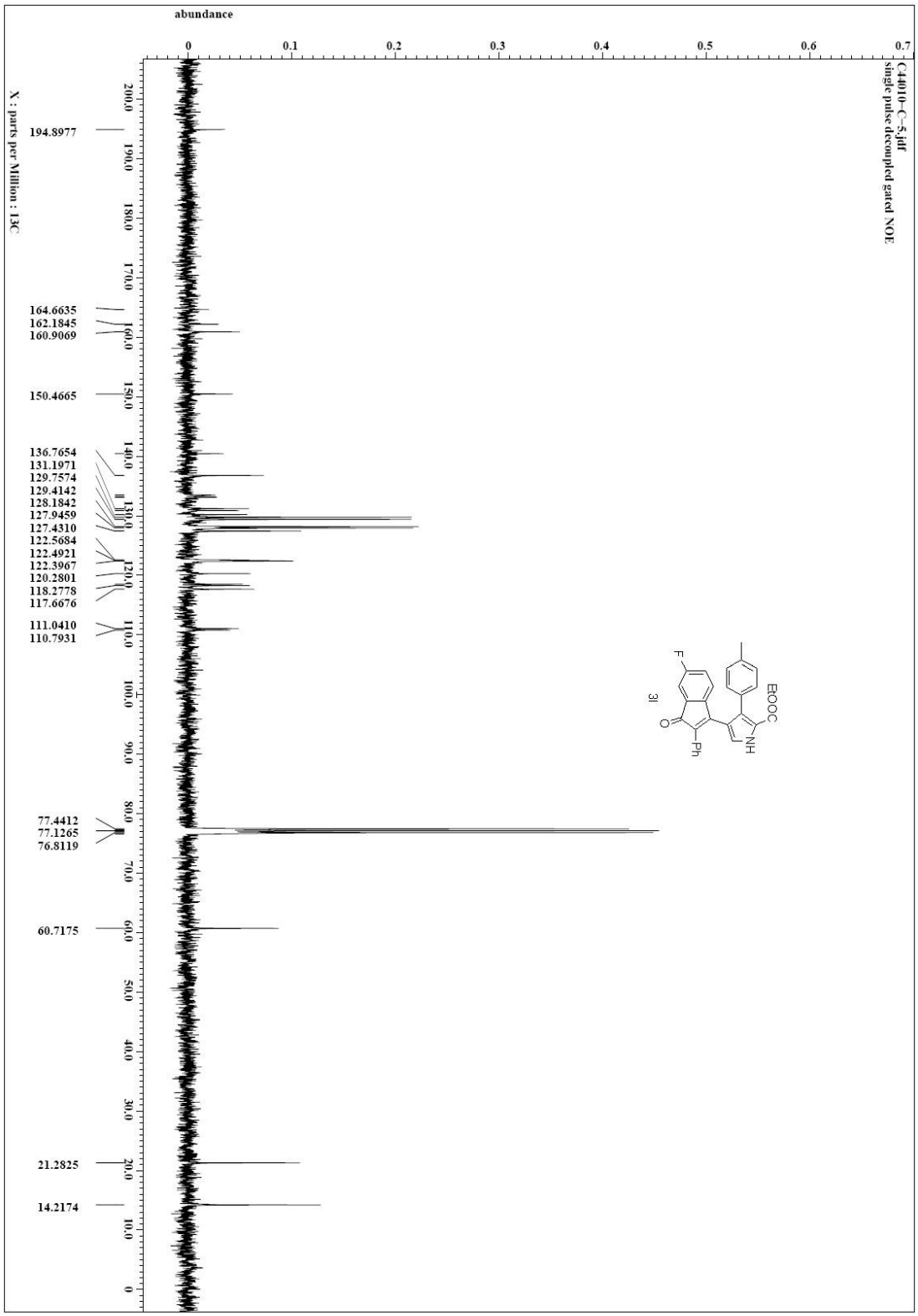

abundance
X : parts per Million : 1H
C:\4009-H-5.jdf
single_pulse
9.5934
7.2417
7.2234
7.2097
7.1753
7.1615
7.1466
7.1340
6.9622
6.9027
4.3013
4.2841
4.2669
1.6895
1.2955
1.2783
1.2611
-0.0035
10
10.69
80.45
10.48
10.21
20.13
20.61
31.08
3j
EtOOC
Ph
NH
O
S

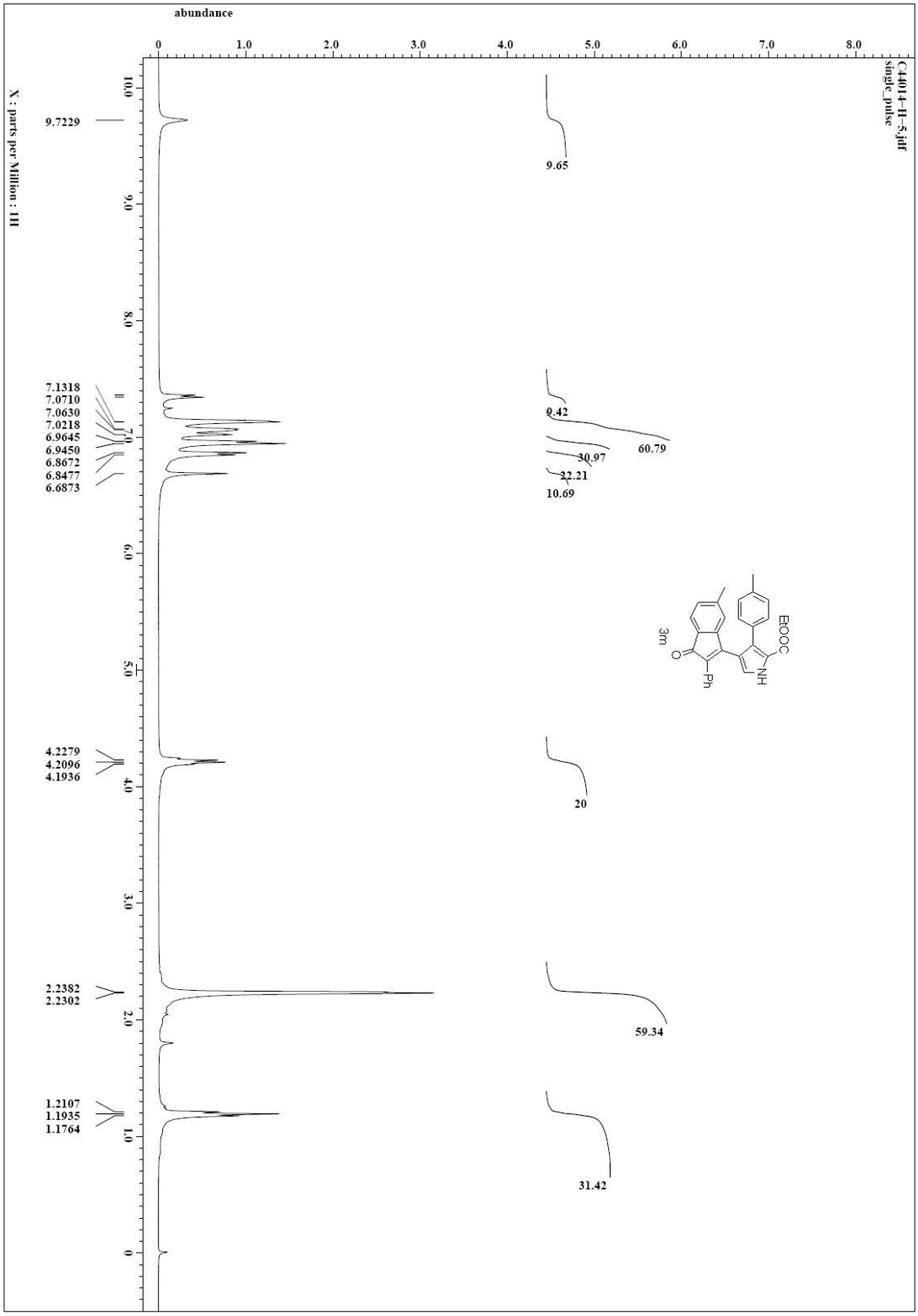

abundance
0
0.1
0.2
0.3
0.4
0.5
C44009-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.5281
160.6495
150.1138
145.3655
133.4187
129.6335
128.6323
128.5465
128.0889
126.5157
126.2582
122.4635
122.1965
121.1096
77.4507
77.1265
76.8119
60.9368
14.2841
3j
EtOOC
NH
S
O
Ph

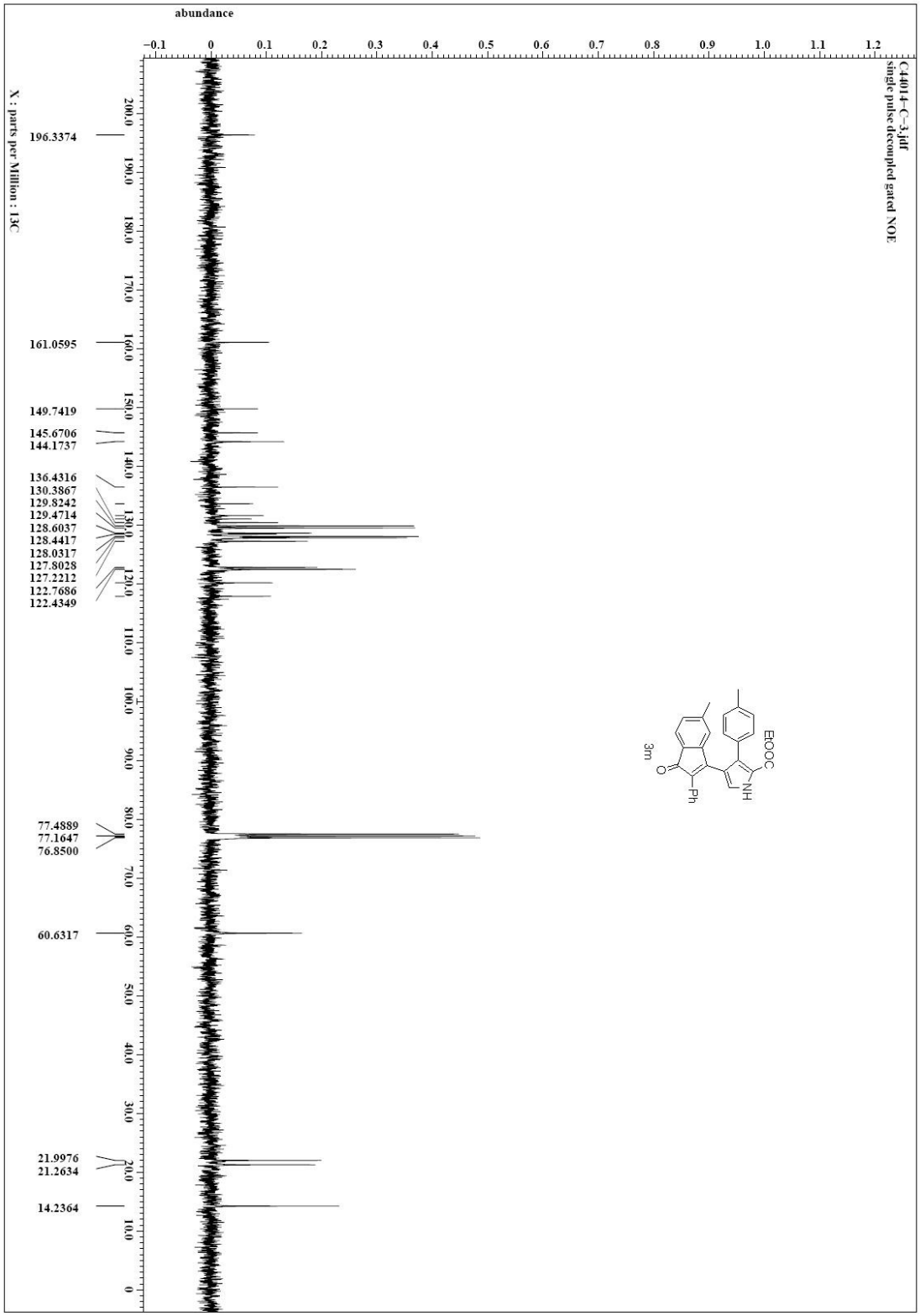

abundance
X : parts per Million : 1H
C:\40012-H-5.jdf
single_pulse
9.6530
7.2830
7.2761
7.2497
7.1363
7.1192
6.9989
6.9725
6.9554
6.8946
6.8775
3.9152
3.8980
3.8866
1.5807
0.6288
0.6116
0.5944
−0.0081
9.43
10.93
10.02
10.96
10.80
21.3
40.14
72.34
20
30.01
3k
O
Ph
NH
COOEt

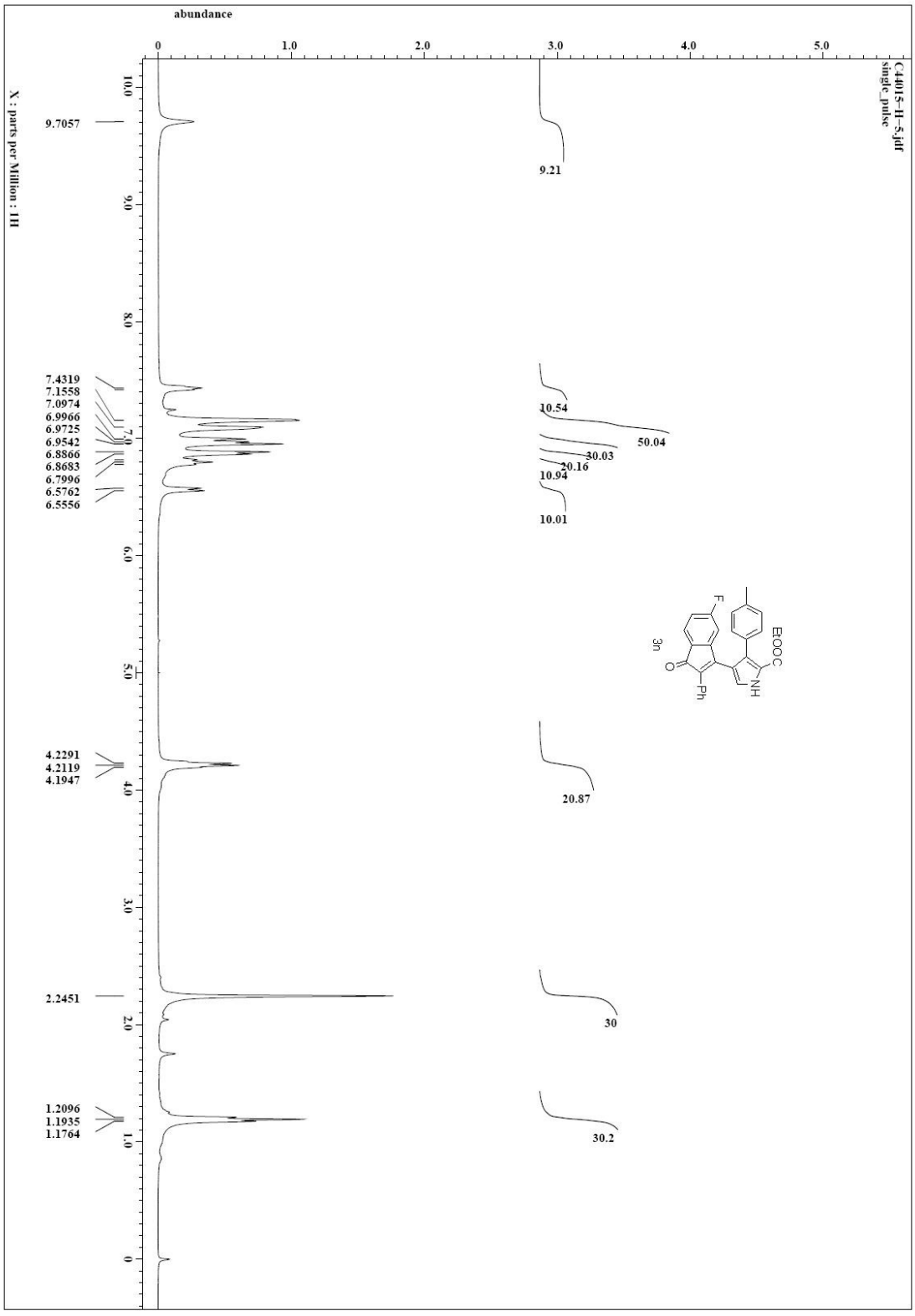

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
C:\4012-C--4.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
196.1086
160.5732
151.3819
145.4132
129.2521
128.2224
128.1366
127.5549
127.3738
126.2106
125.4764
125.2190
122.3777
121.6816
59.9262
40.2468
40.0370
39.8273
13.8837
3k
O
Ph
NH
COOEt

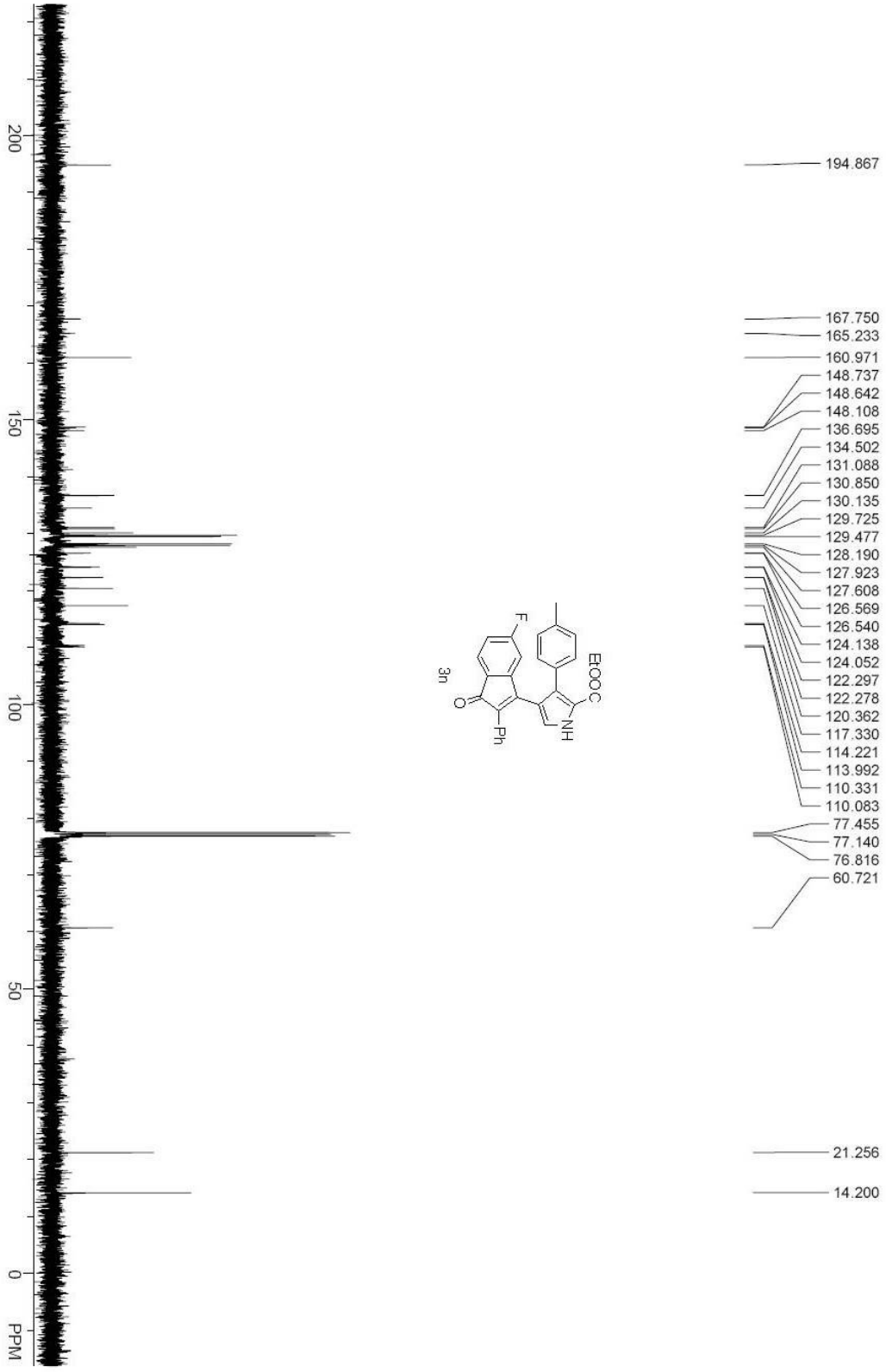

abundance
X : parts per Million : 1H
C44010-H-5.jdf
single_pulse
9.5831
7.1593
7.1008
7.0080
6.9886
6.8946
6.8763
4.2302
4.2130
2.2485
1.2164
1.1993
1.1832
9.51
60.01
30.17
40.93
20.61
30
32.09
3l
F
EtOOC
NH
Ph
O

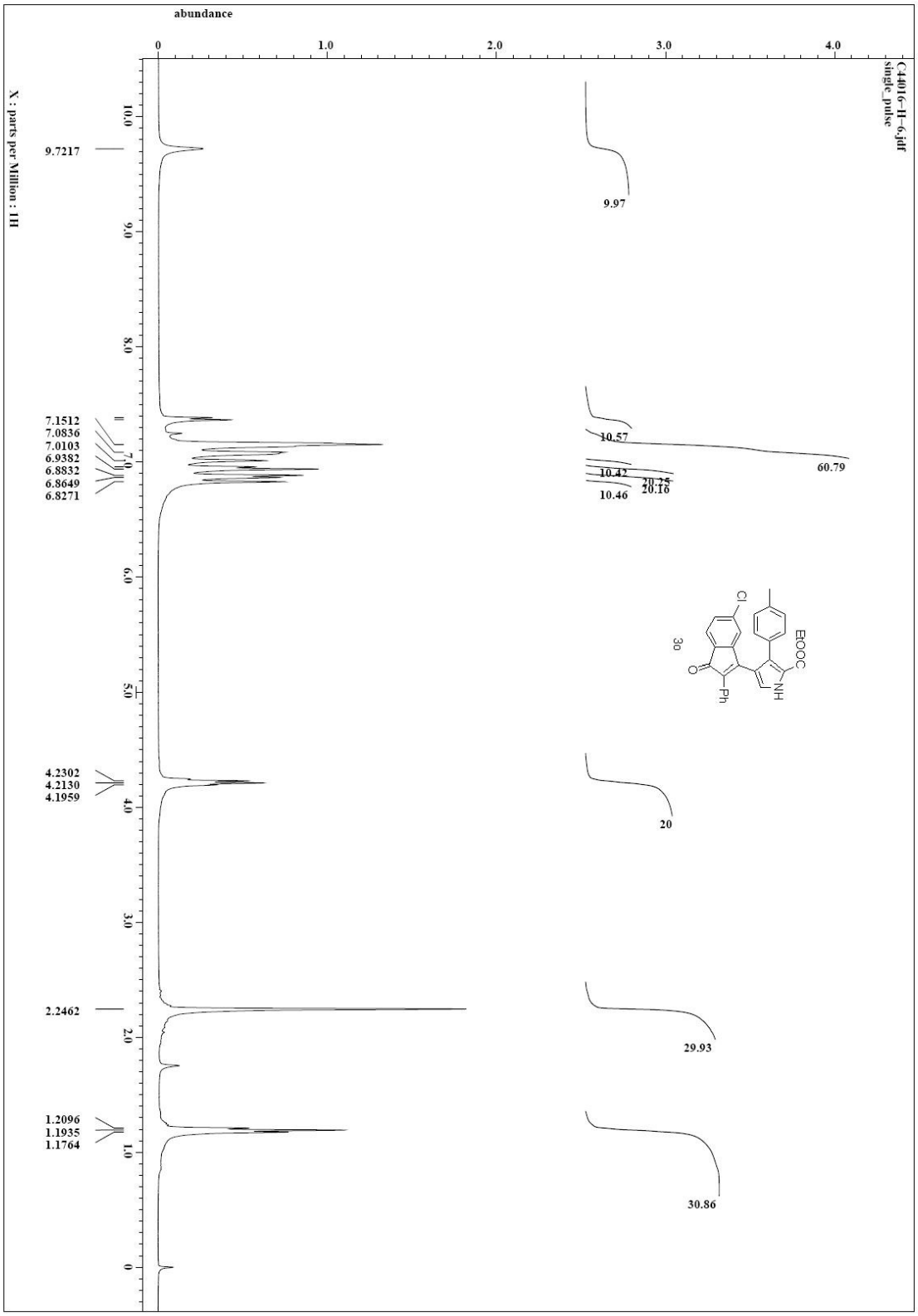

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
C:\4010-C-5.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
194.8977
164.6635
162.1845
160.9069
150.4665
136.7654
131.1971
129.7574
129.4142
128.1842
127.9459
127.4310
122.5684
122.4921
122.3967
120.2801
118.2778
117.6676
111.0410
110.7931
77.4412
77.1265
76.8119
60.7175
21.2825
14.2174
3l
F
O
Ph
NH
EtOOC

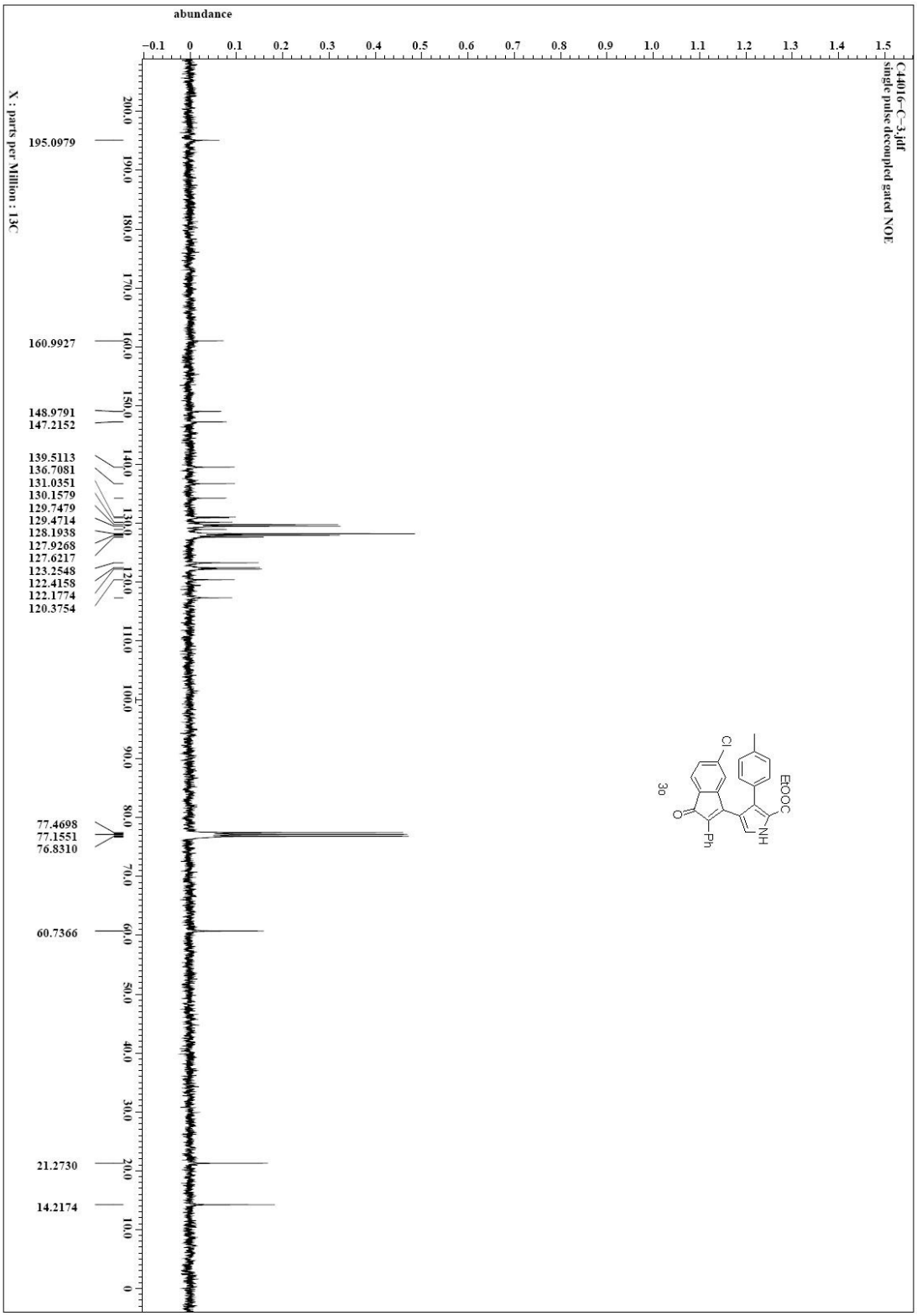

abundance
X : parts per Million : 1H
C14014-H-5.jdf
single_pulse
9.7229
7.1318
7.0710
7.0630
7.0218
6.9645
6.9450
6.8672
6.8477
6.6873
4.2279
4.2096
4.1936
2.2382
2.2302
1.2107
1.1935
1.1764
9.65
9.42
60.79
30.97
22.21
10.69
20
59.34
31.42
3m
EtOOC
NH
Ph
O

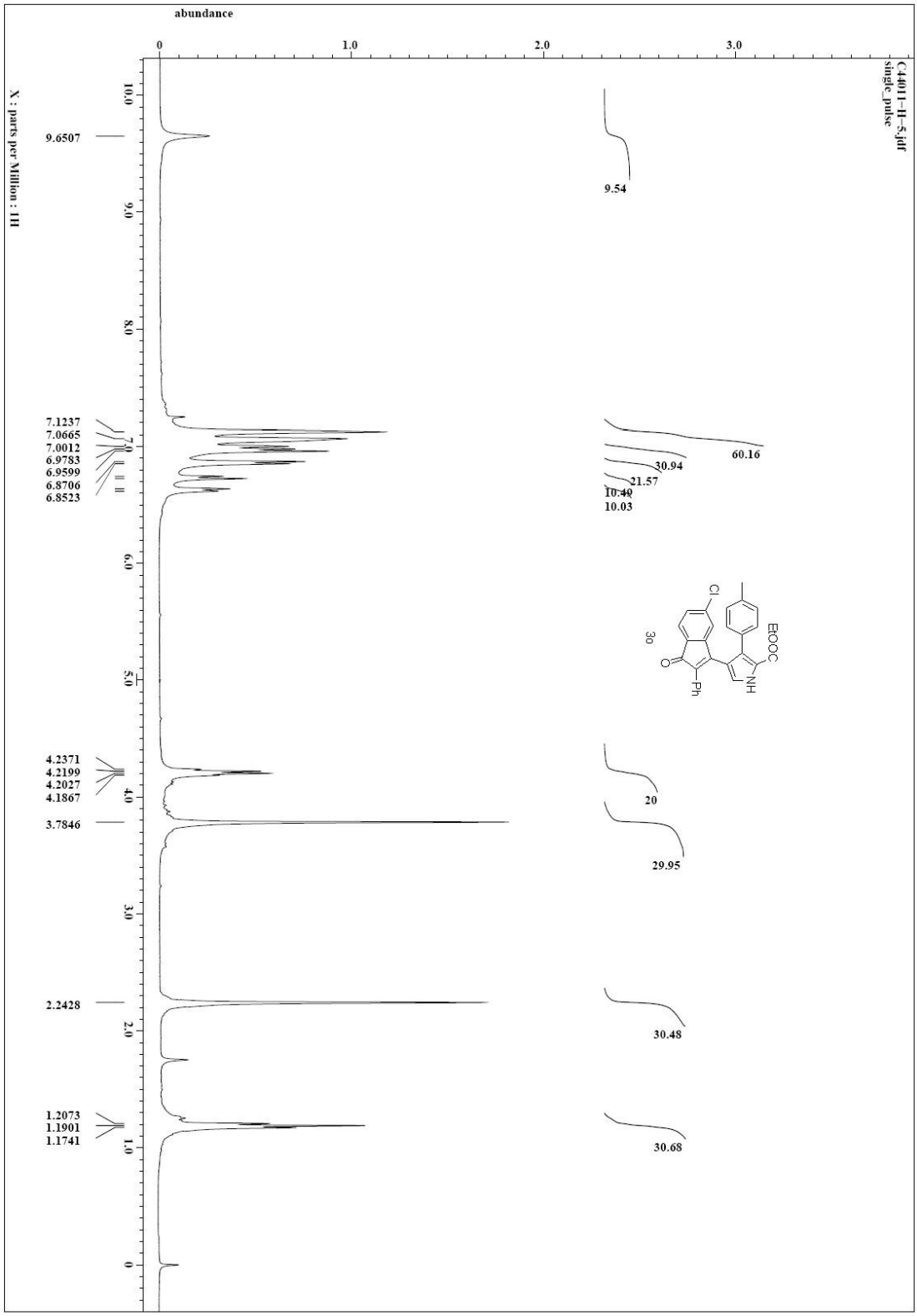

abundance
X : parts per Million : 13C
C:\4014-C-3.jdf
single pulse decoupled gated NOE
196.3374
161.0595
149.7419
145.6706
144.1737
136.4316
130.3867
129.8242
129.4714
128.6037
128.4417
128.0317
127.8028
127.2212
122.7686
122.4349
77.4889
77.1647
76.8500
60.6317
21.9976
21.2634
14.2364
3m
EtOOC
NH
Ph
O

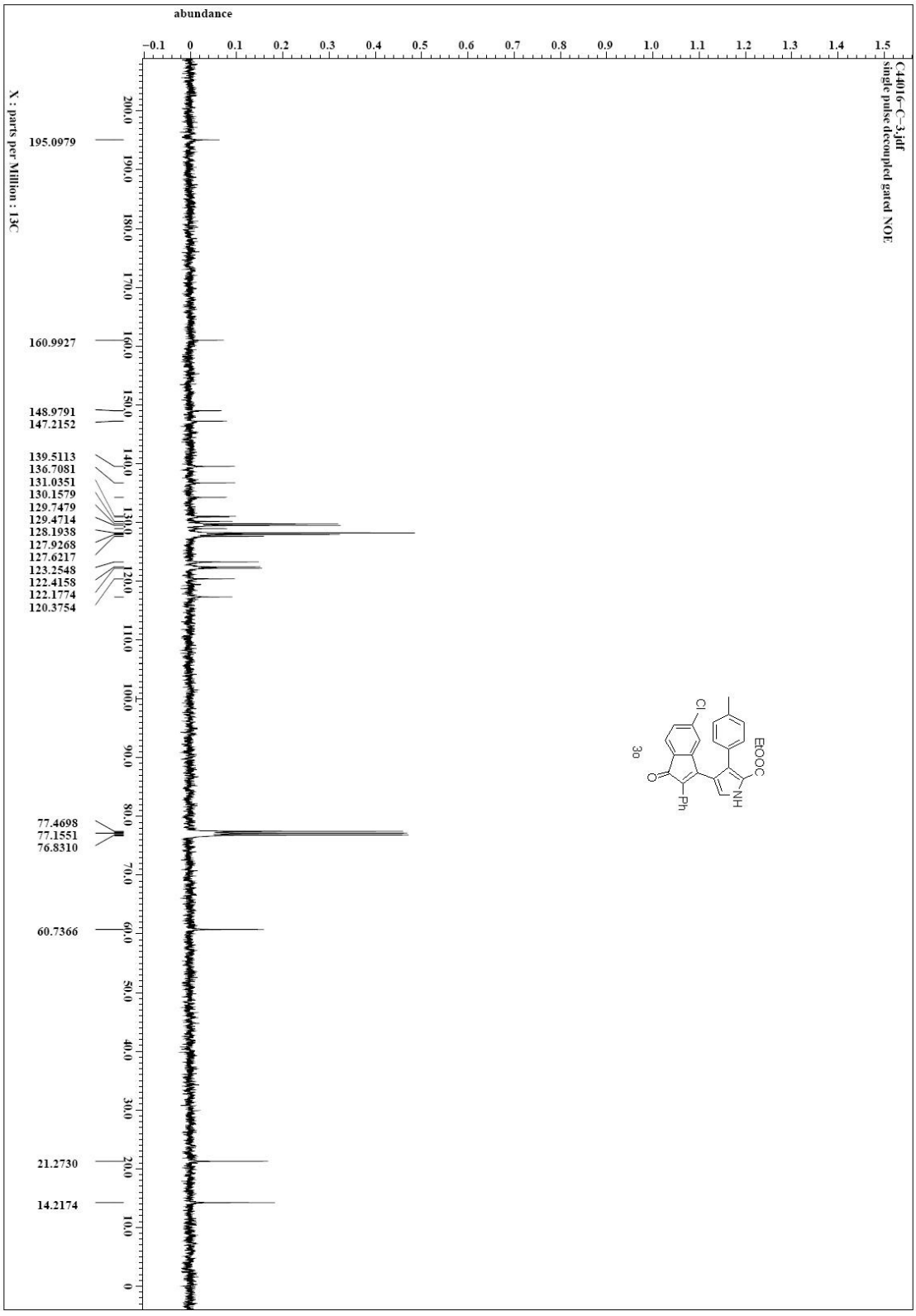

abundance
X : parts per Million : 1H
C:\4015-11-5.jdf
single_pulse
9.7057
7.4319
7.1558
7.0974
6.9966
6.9725
6.9542
6.8866
6.8683
6.7996
6.5762
6.5556
4.2291
4.2119
4.1947
2.2451
1.2096
1.1935
1.1764
9.21
10.54
50.04
30.03
20.16
10.94
10.01
20.87
30
30.2
3n
F
EtOOC
NH
Ph
O

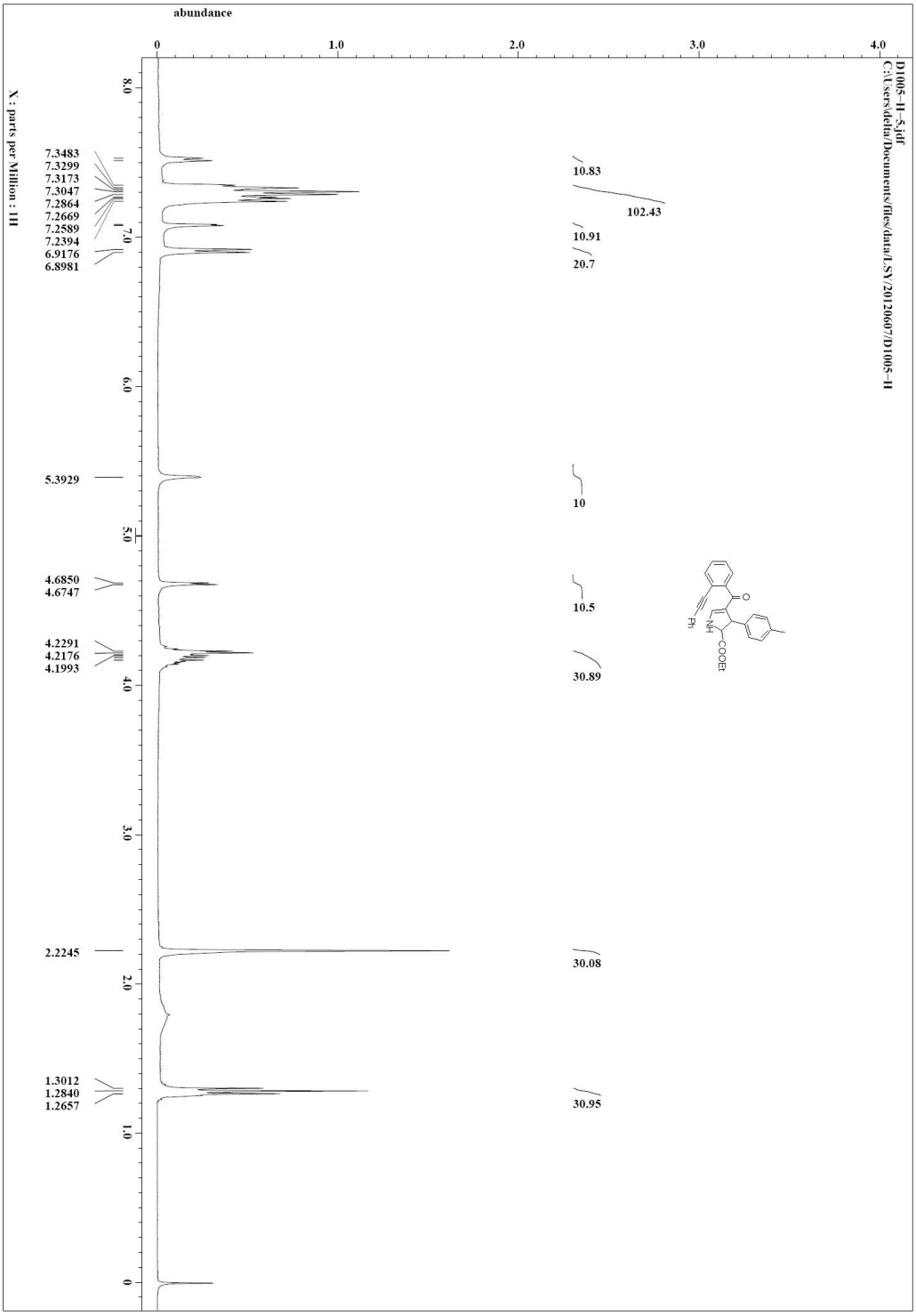

200
150
100
50
0
PPM
194.867
167.750
165.233
160.971
148.737
148.642
148.108
136.695
134.502
131.088
130.850
130.135
129.725
129.477
128.190
127.923
127.608
126.569
126.540
124.138
124.052
122.297
122.278
120.362
117.330
114.221
113.992
110.331
110.083
77.455
77.140
76.816
60.721
21.256
14.200
3n
F
O
Ph
NH
EtOOC

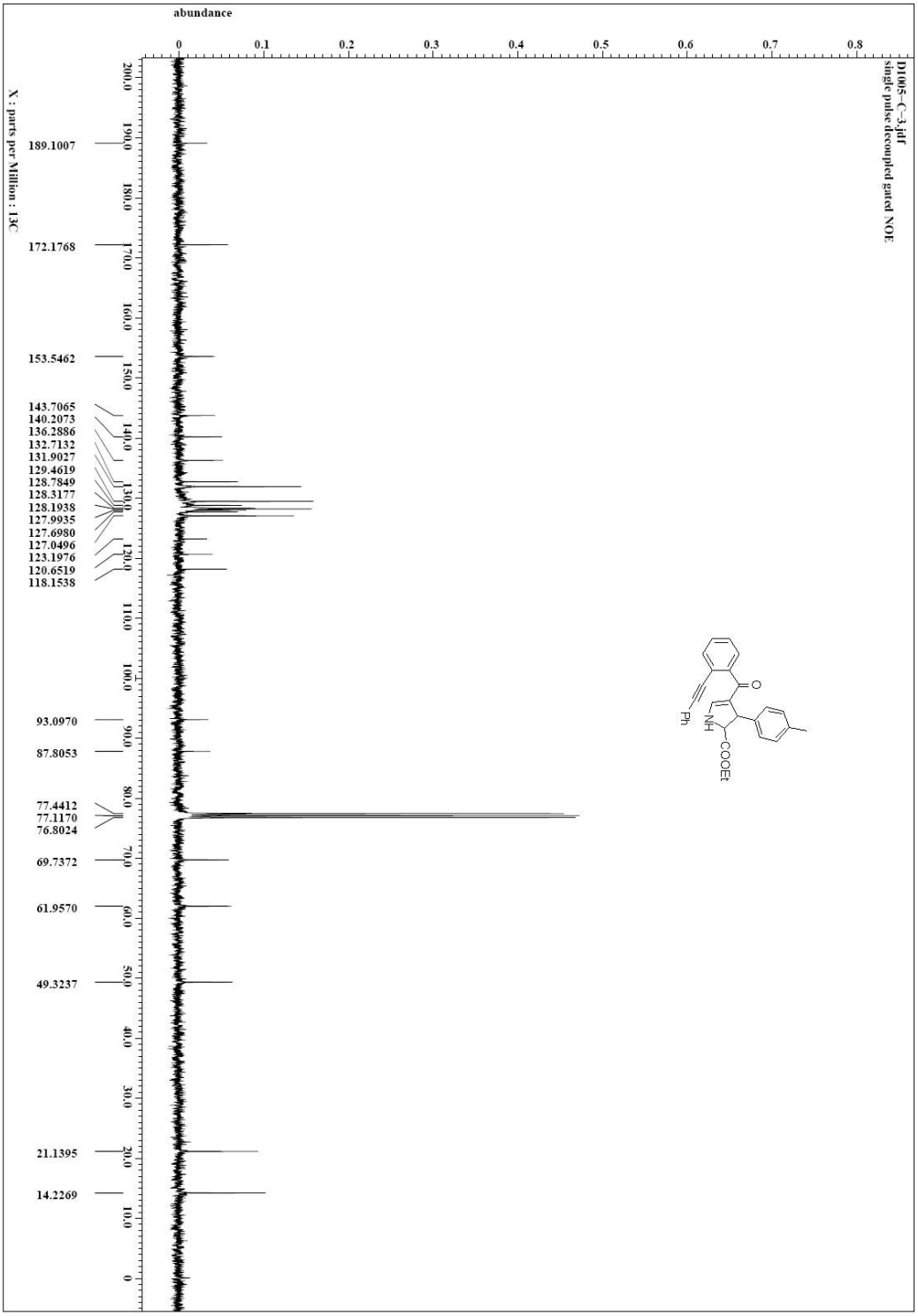

abundance
X : parts per Million : 1H
C44016-H-6.jdf
single_pulse
9.7217
7.1512
7.0836
7.0103
6.9382
6.8832
6.8649
6.8271
4.2302
4.2130
4.1959
2.2462
1.2096
1.1935
1.1764
9.97
10.57
60.79
10.42
20.75
20.16
10.46
20
29.93
30.86
30
Cl
EtOOC
NH
Ph
O


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
C4016-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
195.0979
160.9927
148.9791
147.2152
139.5113
136.7081
131.0351
130.1579
129.7479
129.4714
128.1938
127.9268
127.6217
123.2548
122.4158
122.1774
120.3754
77.4698
77.1551
76.8310
60.7366
21.2730
14.2174
3o
Cl
EtOOC
NH
Ph
O


abundance
0
1.0
2.0
3.0
X : parts per Million : 1H
C44011-1-5.jdf
single_pulse
9.6507
7.1237
7.0665
7.0012
6.9783
6.9599
6.8706
6.8523
4.2371
4.2199
4.2027
4.1867
3.7846
2.2428
1.2073
1.1901
1.1741
9.54
60.16
30.94
21.57
10.49
10.03
20
29.95
30.48
30.68
3o
Cl
EtOOC
NH
Ph
O


abundance
X : parts per Million : 13C
C44016-C--3.jdf
single pulse decoupled gated NOE
195.0979
160.9927
148.9791
147.2152
139.5113
136.7081
131.0351
130.1579
129.7479
129.4714
128.1938
127.9268
127.6217
123.2548
122.4158
122.1774
120.3754
77.4698
77.1551
76.8310
60.7366
21.2730
14.2174
Cl
EtOOC
NH
Ph
O
3o


abundance
0
1.0
2.0
3.0
4.0
X : parts per Million : 1H
D1005-H-5.jdf
C:\Users\delta\Documents\files\data\LSY\20120607\D1005-H
7.3483
7.3299
7.3173
7.3047
7.2864
7.2669
7.2589
7.2394
6.9176
6.8981
5.3929
4.6850
4.6747
4.2291
4.2176
4.1993
2.2245
1.3012
1.2840
1.2657
10.83
102.43
10.91
20.7
10
10.5
30.89
30.08
30.95
Ph
NH
COOEt


abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
D1005-C-3.jdf
single pulse decoupled gated NOE
X : parts per Million : 13C
189.1007
172.1768
153.5462
143.7065
140.2073
136.2886
132.7132
131.9027
129.4619
128.7849
128.3177
128.1938
127.9935
127.6980
127.0496
123.1976
120.6519
118.1538
93.0970
87.8053
77.4412
77.1170
76.8024
69.7372
61.9570
49.3237
21.1395
14.2269
O
Ph
NH
COOEt