



Supporting Information

for

Highly regio- and stereoselective phosphinylphosphination of terminal alkynes with tetraphenyldiphosphine monoxide under radical conditions

Dat Phuc Tran, Yuki Sato, Yuki Yamamoto, Shin-ichi Kawaguchi, Shintaro Kodama, Akihiro Nomoto and Akiya Ogawa

Beilstein J. Org. Chem. **2021**, *17*, 866–872. doi:10.3762/bjoc.17.72

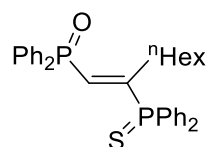
Characterization data and copies of NMR spectra

CONTENTS

1. Characterization data	S3–S10
2. Copies of NMR spectra	S11–S50

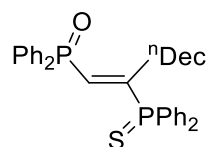
1. Characterization Data

(*E*)- 1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-oct-1-ene (3a)



Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 7.78-7.72 (m, 4H), 7.70-7.65 (m, 4H), 7.54-7.48 (m, 4H), 7.46-7.41 (m, 8H), 7.19 (dd, $J_{\text{H-P}} = 27.9, 24.3$ Hz, 1H), 2.90-2.82 (m, 2H), 1.09-0.86 (m, 8H), 0.72 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 158.1 (d, $J_{\text{C-P}} = 58.5$ Hz), 135.1 (dd, $J_{\text{C-P}} = 88.2, 7.7$ Hz), 133.7 (d, $J = 105.4$ Hz), 132.3 (d, $J = 10.5$ Hz), 131.5 (d, $J = 115.0$ Hz), 131.7, 130.9 (d, $J = 9.6$ Hz), 128.84 (dd, $J = 12.5, 8.6$ Hz), 31.3 (dd, $J = 10.0, 5.8$ Hz), 31.2, 31.1, 29.5, 22.4, 14.03; ^{31}P NMR (162 MHz, CDCl_3): δ 49.6 (d, $J_{\text{P-P}} = 56.4$ Hz), 20.26 (d, $J_{\text{P-P}} = 56.4$ Hz); IR (KBr, cm^{-1}): 3055, 2954, 2928, 2855, 1437, 1201, 1117, 1102, 744, 719, 693, 640; HRMS (ESI+) m/z calcd for $\text{C}_{32}\text{H}_{34}\text{NaOP}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 551.1703, found: 551.1696.

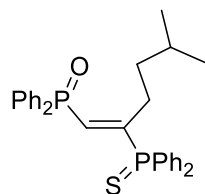
(*E*)- 1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)dodec-1-ene (3b)



Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 7.79-7.71 (m, 4H), 7.70-7.65 (m, 4H), 7.54-7.48 (m, 4H), 7.46-7.41 (m, 8H), 7.20 (dd, $J_{\text{H-P}} = 27.9, 24.0$ Hz, 1H), 2.90-2.81 (m, 2H), 1.33-0.90 (m, 16H), 0.86 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 158.1 (d, $J_{\text{C-P}} = 58.5$ Hz), 135.1 (dd, $J_{\text{C-P}} = 88.2, 7.6$ Hz), 133.7 (d, $J = 104.9$ Hz), 132.3 (d, $J = 10.5$ Hz), 131.5 (d, $J = 115.4$ Hz), 131.7, 130.9 (d, $J = 9.5$ Hz), 128.8 (dd, $J = 11.4, 8.6$ Hz), 32.0, 31.3 (dd, $J = 9.5, 6.7$ Hz), 31.1, 29.9, 29.6, 29.44, 29.36, 29.0, 22.8, 14.2; ^{31}P NMR (162 MHz, CDCl_3): δ 48.9 (d, $J_{\text{P-P}} = 58.6$ Hz), 19.6 (d, $J_{\text{P-P}} = 58.6$ Hz); IR (KBr, cm^{-1}): 3054, 2924, 2852, 1436, 1202,

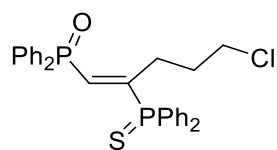
1117, 1102, 744, 719, 693, 640, 545, 527, 498; HRMS (ESI+) m/z calcd for $C_{36}H_{42}NaOP_2S$ $[M+Na]^+$: 607.2329, found: 607.2328.

(E)- 1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-5-methylhex-1-ene (3c)



Colorless oil; 1H NMR (400 MHz, $CDCl_3$): δ 7.77-7.72 (m, 4H), 7.70-7.66 (m, 4H), 7.54-7.49 (m, 4H), 7.47-7.42 (m, 8H), 7.27 (dd, $J_{H-P} = 27.9, 23.8$ Hz, 1H), 2.90-2.81 (m, 2H), 1.28-1.17 (m, 1H), 0.94-0.87 (m, 2H), 0.56 (d, $J = 6.9$ Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.3 (d, $J_{C-P} = 57.5$ Hz), 135.3 (dd, $J_{C-P} = 88.2, 7.7$ Hz), 133.8 (d, $J = 105.4$ Hz), 132.4 (d, $J = 9.6$ Hz), 131.5 (d, $J = 115.0$ Hz), 131.7, 130.9 (d, $J = 10.5$ Hz), 128.8 (dd, $J = 12.5, 6.7$ Hz), 39.6, 29.5 (dd, $J = 9.6, 7.7$ Hz), 28.6, 22.0; ^{31}P NMR (162 MHz, $CDCl_3$): δ 49.6 (d, $J_{P-P} = 56.4$ Hz), 20.0 (d, $J_{P-P} = 56.4$ Hz); IR (KBr, cm^{-1}): 2955, 1436, 1198, 1102, 745, 719, 693; HRMS (ESI+) m/z calcd for $C_{31}H_{32}NaOP_2S$ $[M+Na]^+$: 537.1547, found: 537.1547.

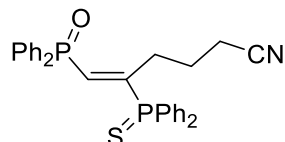
(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-5-chloropent-1-ene (3d)



Colorless oil; 1H NMR (400 MHz, $CDCl_3$): δ 7.77-7.71 (m, 4H), 7.69-7.34 (m, 4H), 7.56-7.52 (m, 4H), 7.48-7.44 (m, 8H), 7.27 (dd, $J_{H-P} = 28.0, 24.0$ Hz, 1H), 3.28 (t, $J = 6.3$ Hz, 2H), 3.03-2.94 (m, 2H), 1.63-1.56 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.7 (d, $J_{C-P} = 59.1$ Hz), 136.0 (dd, $J_{C-P} = 87.3, 6.7$ Hz), 133.1 (d, $J = 104.9$ Hz), 132.3 (d, $J = 9.5$ Hz), 130.9 (d, $J = 9.5$ Hz), 130.7 (d, $J = 83.9$ Hz), 129.0 (dd, $J = 12.4, 3.8$ Hz), 44.73, 33.7, 28.9 (dd, $J = 10.0, 6.7$ Hz); ^{31}P NMR (162 MHz, $CDCl_3$): δ 48.9 (d, $J_{P-P} = 54.1$ Hz), 20.4 (d, $J_{P-P} = 54.1$ Hz); IR (KBr,

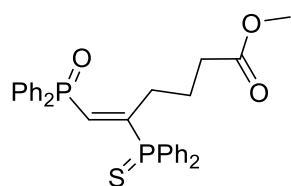
cm⁻¹): 3054, 2925, 1436, 1198, 1101, 720, 693, 640, 527, 544; HRMS (ESI+) *m/z* calcd for C₂₉H₂₇ClNaOP₂S [M+Na]⁺: 543.0844, found: 543.0843.

(*E*)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-5-cyanopent-1-ene (3e)



Colorless oil; ¹H NMR (400 MHz, CDCl₃): δ 7.77-7.70 (m, 4H), 7.67-7.62 (4H, m), 7.58-7.52 (m, 4H), 7.50-7.44 (m, 8H), 7.22 (dd, *J*_{H-P} = 27.1, 24.0 Hz, 1H), 3.02-2.94 (m, 2H), 2.18 (t, *J* = 7.2 Hz, 2H), 1.55-1.47 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 155.8 (d, *J*_{C-P} = 60.1 Hz), 136.3 (dd, *J*_{C-P} = 86.3, 7.6 Hz), 133.1 (d, *J* = 105.9 Hz), 132.4 (d, *J* = 2.9 Hz), 132.3 (d, *J* = 10.5 Hz), 130.9 (d, *J* = 9.5 Hz), 130.6 (d, *J* = 83.9 Hz), 129.1 (d, *J* = 12.4 Hz), 119.2, 29.9 (dd, *J* = 10.0, 6.7 Hz), 27.0, 17.2; ³¹P NMR (162 MHz, CDCl₃): δ 48.6 (d, *J*_{P-P} = 55.2 Hz), 20.0 (d, *J*_{P-P} = 55.2 Hz); IR (KBr, cm⁻¹): 3054, 2937, 2245, 1436, 1197, 1102, 745, 720, 693, 642; HRMS (ESI+) *m/z* calcd for C₃₀H₂₇NNaOP₂S [M+Na]⁺: 534.1186, found: 534.1186.

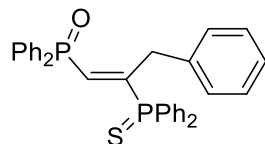
(*E*)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-methylhex-1-enoate (3g)



Colorless oil; ¹H NMR (400 MHz, CDCl₃): δ 7.76-7.71 (m, 4H), 7.67-7.62 (m, 4H), 7.53-7.49 (m, 4H), 7.47-7.42 (m, 8H), 7.14 (dd, *J*_{H-P} = 27.6, 24.0 Hz, 1H), 3.52 (s, 3H), 2.97-2.88 (m, 2H), 2.09 (t, *J* = 7.2 Hz, 2H), 1.51-1.43 (m, 2H); ¹³C NMR (100 MHz, CDCl₃): δ 173.2, 157.1 (d, *J*_{C-P} = 58.2 Hz), 135.7 (dd, *J*_{C-P} = 87.7, 6.7 Hz), 133.3 (d, *J* = 104.9 Hz), 132.3, 132.2 (d, *J* = 4.8 Hz), 130.87 (d, *J* = 83.9 Hz), 130.88 (d, *J* = 9.5 Hz), 128.9 (dd, *J* = 12.4, 7.6 Hz), 51.4, 34.1, 30.2 (dd, *J* = 9.5, 6.7 Hz), 26.5; ³¹P NMR (162 MHz, CDCl₃): δ 48.9 (d, *J*_{P-P} = 58.2 Hz),

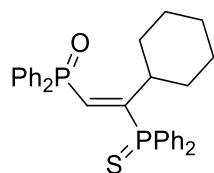
20.0 (d, $J_{\text{P-P}} = 58.2$ Hz); IR (KBr, cm^{-1}): 3054, 2937, 1732, 1436, 1196, 1101, 744, 719, 693; HRMS (ESI+) m/z calcd for $\text{C}_{31}\text{H}_{30}\text{NaO}_3\text{P}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 567.1289, found: 567.1289.

(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-3-phenylprop-1-ene (3i)



White solide; mp. 124-125 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.63-7.56 (m, 8H), 7.52-7.48 (m, 2H), 7.44-7.38 (m, 6H), 7.31-7.27 (m, 4H), 7.04 (dd, $J_{\text{H-P}} = 25.8, 23.1$ Hz, 1H), 6.99 (d, $J = 5.9$ Hz, 2H), 6.87-6.80 (m, 3H), 4.89 (d, $J = 19.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 156.4 (d, $J_{\text{C-P}} = 59.1$ Hz), 136.6, 136.0 (dd, $J_{\text{C-P}} = 87.7, 6.7$ Hz), 133.3 (d, $J = 104.9$ Hz), 132.2 (d, $J = 10.5$ Hz), 132.1 (d, $J = 2.9$ Hz), 131.8 (d, $J = 2.9$ Hz), 130.9 (d, $J = 9.5$ Hz), 130.7 (d, $J = 83.0$ Hz), 129.6, 128.9 (d, $J = 12.4$ Hz), 128.5 (d, $J = 12.4$ Hz), 127.8, 126.1, 35.3 (dd, $J = 10.5, 6.7$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 49.8 (d, $J_{\text{P-P}} = 54.2$ Hz), 19.4 (d, $J_{\text{P-P}} = 54.2$ Hz); IR (KBr, cm^{-1}): 3054, 2923, 1587, 1436, 1206, 1109, 741, 718, 692, 528; HRMS (ESI+) m/z calcd for $\text{C}_{33}\text{H}_{28}\text{NaOP}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 557.1234, found: 557.1228.

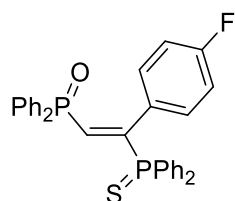
(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-(2-cyclohexyl)ethene (3j)



Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 7.83-7.75 (m, 4H), 7.63-7.56 (m, 4H), 7.53-7.48 (m, 4H), 7.47-7.39 (m, 8H), 6.82 (dd, $J_{\text{H-P}} = 30.4, 21.3$ Hz, 1H), 3.02-2.91 (m, 1H), 2.28-2.18 (m, 2H), 1.65-1.46 (m, 3H), 1.39-1.30 (m, 2H), 1.28-1.18 (m, 1H), 1.03-0.92 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 161.3 (d, $J_{\text{C-P}} = 56.3$ Hz), 134.6 (dd, $J_{\text{C-P}} = 87.7, 8.6$ Hz), 134.1 (d, $J = 105.9$ Hz), 132.3 (d, $J = 10.5$ Hz), 132.9 (dd, $J = 13.4, 1.9$ Hz), 131.1 (d, $J = 83.0$ Hz),

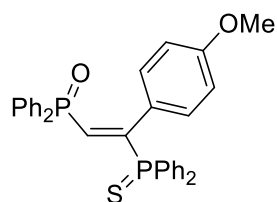
130.8 (d, $J = 9.5$ Hz), 128.7 (dd, $J = 12.4, 6.7$ Hz), 43.1 (dd, $J = 11.4, 5.7$ Hz), 32.6, 29.4, 25.2; ^{31}P NMR (162 MHz, CDCl_3): δ 51.1 (d, $J_{\text{P-P}} = 60.6$ Hz), 18.2 (d, $J_{\text{P-P}} = 60.6$ Hz); IR (KBr, cm^{-1}): 3055, 2927, 2852, 2226, 1436, 1200, 1117, 1102, 734, 720, 694, 645; HRMS (ESI+) m/z calcd for $\text{C}_{32}\text{H}_{32}\text{NaOP}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 549.1547, found: 549.1547.

(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-fluorophenyl)ethene (3k)



Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 7.78-7.73 (m, 4H), 7.56 (dd, $J_{\text{H-P}} = 24.9, 20.1$ Hz, 1H), 7.53-7.44 (m, 5H), 7.40-7.35 (m, 6H), 7.28-7.24 (m, 5H), 6.90 (td, $J = 6.9, 1.4$ Hz, 2H), 6.51 (t, $J = 8.7$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 162.7 (d, $J_{\text{C-F}} = 248.9$ Hz), 156.2 (d, $J_{\text{C-P}} = 60.1$ Hz), 138.3 (d, $J_{\text{C-P}} = 88.7, 9.5$ Hz), 132.5 (d, $J_{\text{C-P}} = 105.9$ Hz), 132.6 (d, $J_{\text{C-P}} = 10.5$ Hz), 132.1 (d, $J = 2.9$ Hz), 130.0 (t, $J = 4.8$ Hz), 131.8 (d, $J = 1.9$ Hz), 130.9, 130.7 (d, $J = 9.5$ Hz), 129.5 (d, $J_{\text{C-P}} = 84.9$ Hz), 128.57, 128.58 (d, $J = 21.9$ Hz), 128.0 (d, $J = 12.4$ Hz), 127.6 (d, $J = 12.4$ Hz), 114.4 (d, $J_{\text{C-F}} = 21.0$ Hz); ^{19}F NMR (377 MHz, CDCl_3): δ -112.43; ^{31}P NMR (162 MHz, CDCl_3): δ 47.8 (d, $J_{\text{P-P}} = 50.7$ Hz), 18.4 (d, $J_{\text{P-P}} = 50.7$ Hz); IR (KBr, cm^{-1}): 3057, 1600, 1503, 1436, 1229, 1187, 1099, 838, 747, 726, 692, 639, 548, 528, 503; HRMS (ESI+) m/z calcd for $\text{C}_{32}\text{H}_{25}\text{FNaOP}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 561.0983, found: 561.0982.

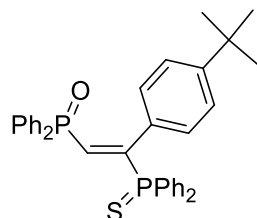
(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-methoxyphenyl)ethene (3l)



Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 7.77-7.72 (m, 4H), 7.58 (dd, $J_{\text{H-P}} = 25.4, 20.1$ Hz, 1H), 7.56-7.51 (m, 4H), 7.50-7.45 (m, 2H), 7.41-7.36 (m, 6H), 7.32-7.27 (m, 4H), 6.85 (dd, $J = 8.7, 1.8$ Hz, 2H), 6.39 (d, $J = 8.2$ Hz, 2H), 3.65 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 159.4, 152.6 (d, $J_{\text{C-P}} = 59.4$ Hz), 138.9 (dd, $J_{\text{C-P}} = 73.3, 11.0$ Hz), 132.9 (d, $J = 105.9$ Hz), 132.7 (d, $J = 10.5$ Hz), 131.63 (d, $J = 2.9$ Hz), 131.57 (d, $J = 3.8$ Hz), 132.0 (d, $J = 2.9$ Hz), 131.9, 131.7 (dd, $J = 8.6, 2.9$ Hz), 131.4, 131.3, 130.2 (d, $J = 83.4$ Hz), 128.6 (d, $J = 12.5$ Hz), 128.4 (d, $J = 13.4$ Hz), 124.8, 112.7, 55.2; ^{31}P NMR (162 MHz, CDCl_3): δ 47.5 (d, $J_{\text{P-P}} = 52.0$ Hz), 17.90 (d, $J_{\text{P-P}} = 52.0$ Hz); IR (KBr, cm^{-1}): 3053, 2924, 1605, 1505, 1436, 1250, 1180, 1099, 748, 719, 693, 640; HRMS (ESI+) m/z calcd for $\text{C}_{33}\text{H}_{28}\text{NaO}_2\text{P}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 573.1183, found: 573.1183.

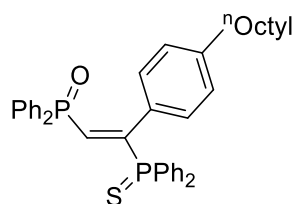
(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-tert-butylphenyl)ethene

(3m)



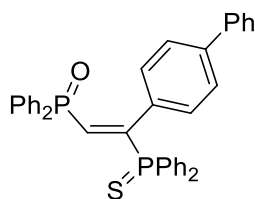
White solid; mp. > 250 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.74-7.68 (m, 4H), 7.64 (dd, $J_{\text{H-P}} = 25.7, 19.7$ Hz, 1H), 7.53-7.44 (m, 6H), 7.39-7.32 (m, 6H), 7.28-7.24 (m, 4H), 6.83 (d, $J = 8.2$ Hz, 2H), 6.78 (dd, $J = 8.7, 1.4$ Hz, 2H), 1.41 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3): δ 156.8 (d, $J_{\text{C-P}} = 70.9$ Hz), 151.3, 138.2 (dd, $J_{\text{C-P}} = 89.1, 8.6$ Hz), 133.0 (d, $J = 106.4$ Hz), 132.7 (d, $J = 10.54$ Hz), 132.0 (d, $J = 2.9$ Hz), 131.5 (d, $J = 1.9$ Hz), 130.8 (d, $J = 9.6$ Hz), 130.1 (d, $J = 84.35$ Hz), 129.9 (d, $J = 3.8$ Hz), 128.5 (dd, $J = 12.5, 7.7$ Hz), 124.2, 34.5, 31.2; ^{31}P NMR (162 MHz, CDCl_3): δ 48.5 (d, $J_{\text{P-P}} = 52.0$ Hz), 18.9 (d, $J_{\text{P-P}} = 52.0$ Hz); IR (KBr) 3051, 2959, 1607, 1548, 1479, 1437, 847, 750, 715, 691, 644; HRMS (ESI+) m/z calcd for $\text{C}_{36}\text{H}_{34}\text{NaOP}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 599.1703, found: 599.1702.

(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-octylphenyl)ethene (3n)



Colorless oil; ^1H NMR (400 MHz, CDCl_3): δ 7.74-7.68 (m, 4H), 7.66 (dd, $J_{\text{H-P}} = 25.4, 20.2$ Hz, 1H), 7.55-7.49 (m, 4H), 7.46-7.43 (m, 2H), 7.38-7.33 (m, 6H), 7.29-7.24 (m, 4H), 6.76 (dd, $J = 7.8, 1.4$ Hz, 2H), 6.63 (d, $J = 7.8$ Hz, 2H), 2.36 (t, $J = 7.6$ Hz, 2H), 1.46-1.36 (m, 2H), 1.31-1.21 (m, 10H), 0.88 (t, $J = 6.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 156.9 (d, $J = 58.5$ Hz), 143.4, 138.0 (dd, $J_{\text{C-P}} = 89.1, 9.6$ Hz), 133.0 (d, $J_{\text{C-P}} = 106.4$ Hz), 132.7 (d, $J = 9.6$ Hz), 131.9 (d, $J = 1.9$ Hz), 131.5 (d, $J = 2.9$ Hz), 130.8 (d, $J = 10.5$ Hz), 130.6 (d, $J = 8.6$ Hz), 129.99 (d, $J = 84.4$ Hz), 129.95 (d, $J = 3.8$ Hz), 128.4 (dd, $J = 12.5, 6.7$ Hz), 127.3, 35.6, 32.0, 31.3, 29.5, 29.4, 29.3, 22.8, 14.2; ^{31}P NMR (162 MHz, CDCl_3): δ 48.4 (d, $J_{\text{P-P}} = 52.0$ Hz), 18.9 (d, $J_{\text{P-P}} = 52.0$ Hz); IR (KBr, cm^{-1}): 2925, 2854, 1437, 1186, 1106, 720, 693; HRMS (ESI+) m/z calcd for $\text{C}_{40}\text{H}_{42}\text{NaOP}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 655.2329, found: 655.2329.

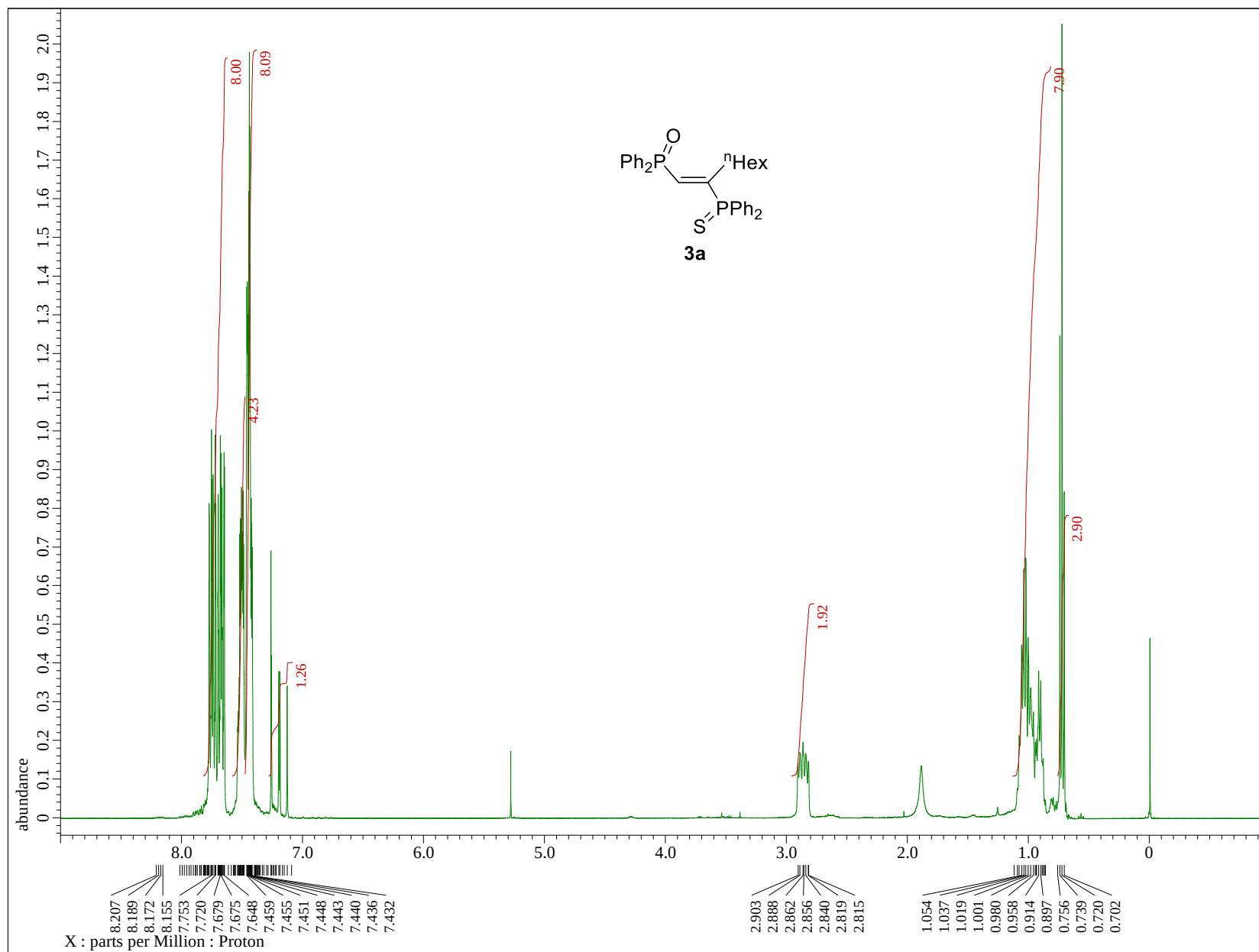
(E)-1-(diphenylphosphinyl)-2-(diphenylthiophosphinyl)-2-(4-diphenyl)ethene (3o)

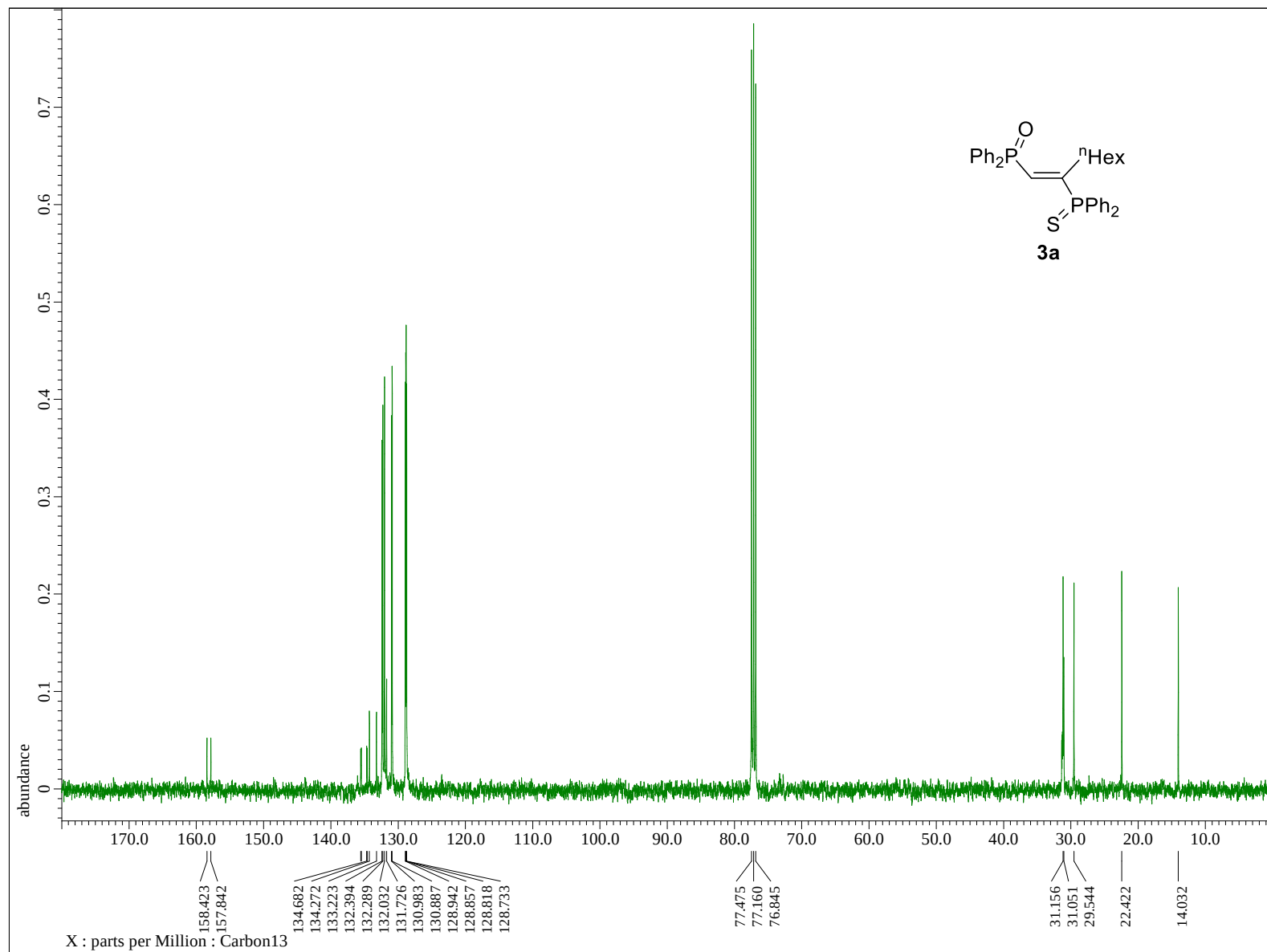


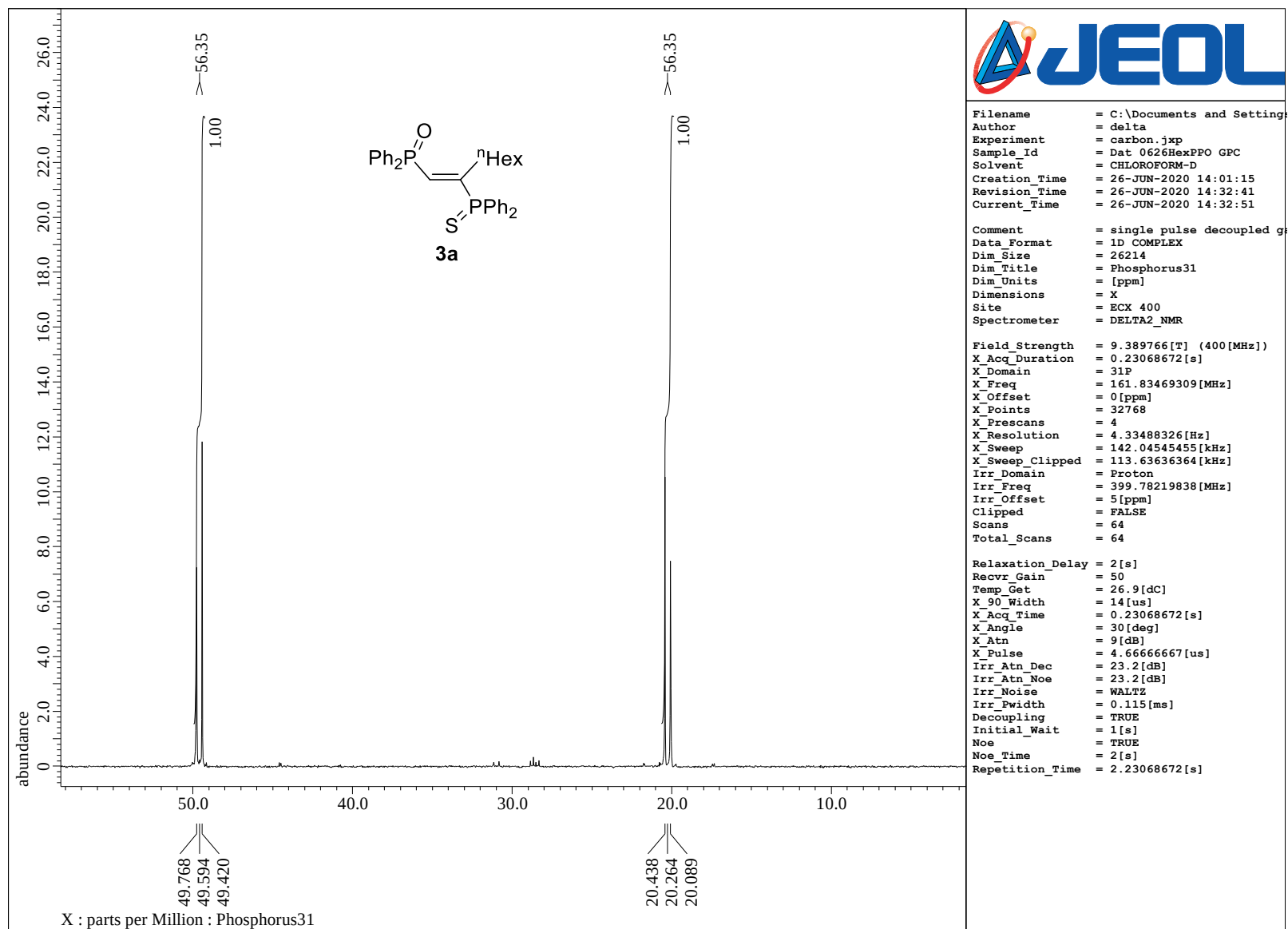
White solid; mp. 89-90 $^{\circ}\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 7.80-7.75 (m, 4H), 7.68 (dd, $J_{\text{H-P}} = 30.2, 19.2$ Hz, 1H), 7.56-7.52 (m, 4H), 7.45-7.47 (m, 2H), 7.43-7.39 (m, 8H), 7.30-7.27 (m, 7H), 7.06 (d, $J = 8.2$ Hz, 2H), 6.93 (dd, $J = 8.2, 1.4$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 152.3 (d, $J_{\text{C-P}} = 59.1$ Hz), 141.0 (dd, $J_{\text{C-P}} = 72.5, 9.5$ Hz), 132.8 (d, $J = 10.5$ Hz), 132.7 (d, $J = 4.8$ Hz), 132.4, 132.3 (d, $J = 5.7$ Hz), 132.1, 131.8, 131.7 (d, $J = 78.2$ Hz), 131.6 (d, $J = 8.6$ Hz), 131.5 (d, $J = 10.5$ Hz), 131.3 (d, $J = 2.9$ Hz), 130.8 (d, $J = 2.9$ Hz), 130.2 (d, $J = 84.9$ Hz),

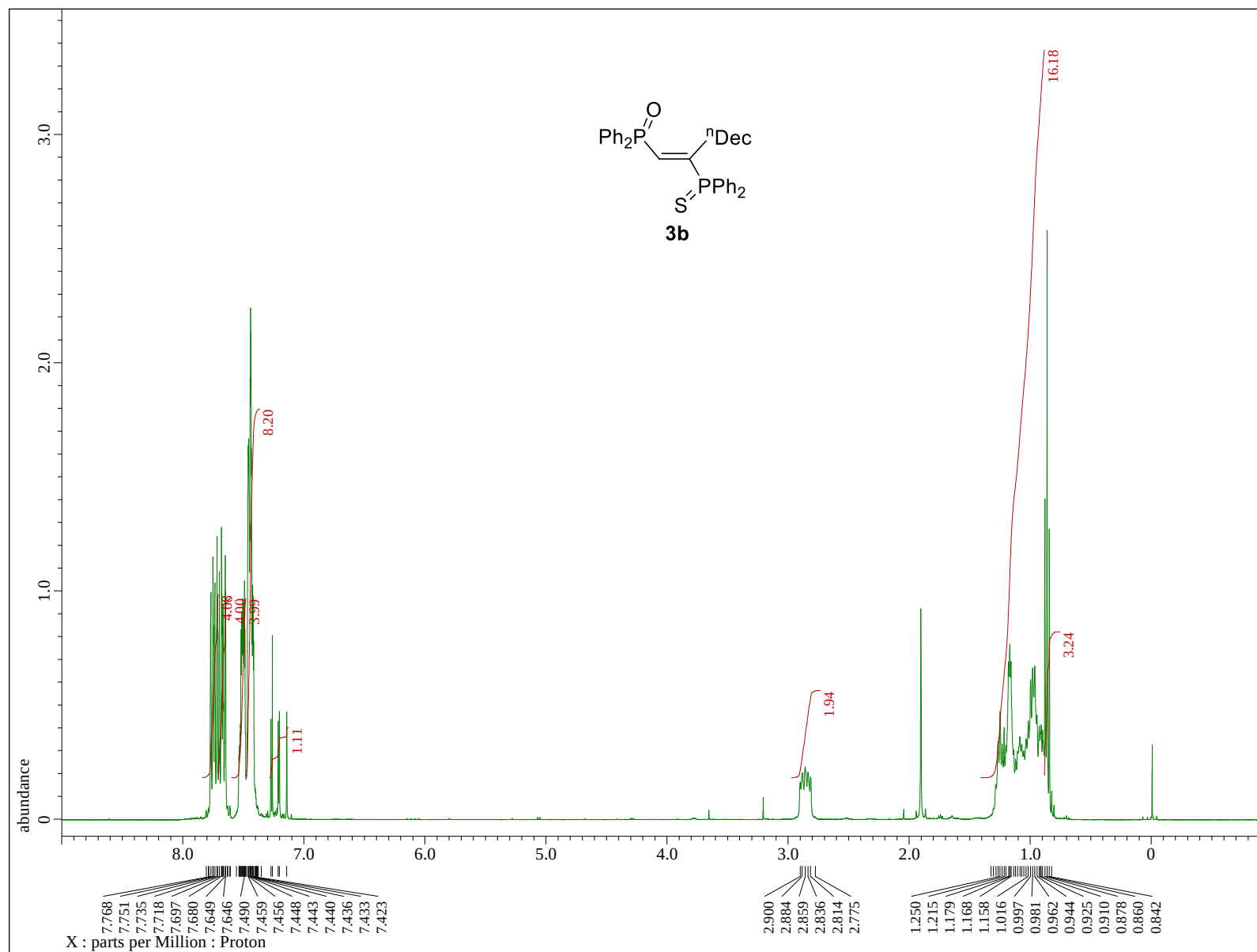
128.9, 128.6 (d, $J = 12.4$ Hz), 128.5 (d, $J = 12.4$ Hz), 128.4 (d, $J = 13.4$ Hz), 127.6, 127.0, 126.8 (d, $J = 68.6$ Hz), 125.8; ^{31}P NMR (162 MHz, CDCl_3): δ 48.4 (d, $J_{\text{P-P}} = 52.0$ Hz), 18.9 (d, $J_{\text{P-P}} = 52.0$ Hz); IR (KBr, cm^{-1}): 3053, 1482, 1436, 1185, 1099, 719, 692, 642, 545, 509; HRMS (ESI+) m/z calcd for $\text{C}_{40}\text{H}_{42}\text{NaOP}_2\text{S}$ $[\text{M}+\text{Na}]^+$: 655.2329, found: 655.2329.

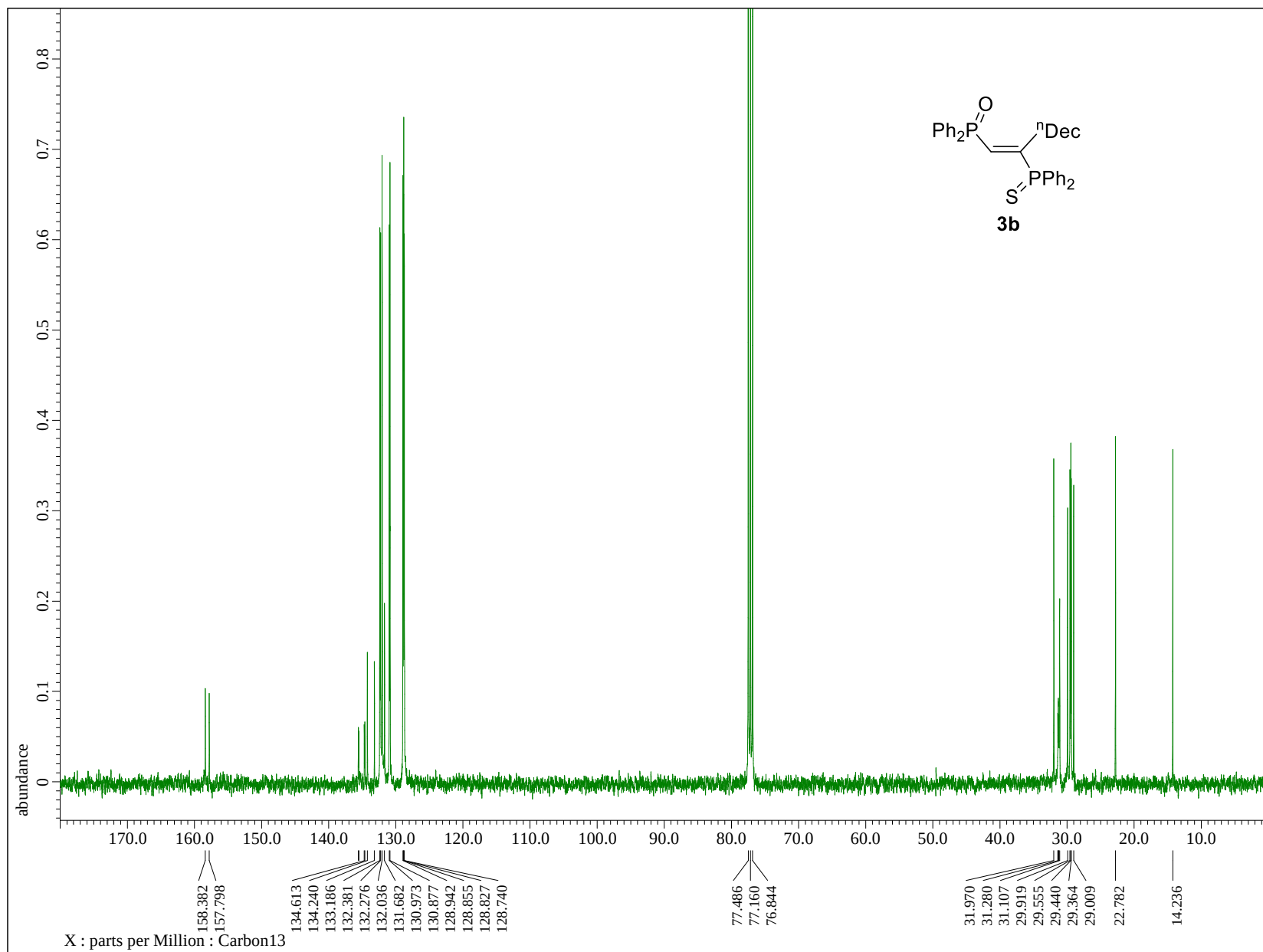
2. Copies of NMR spectra



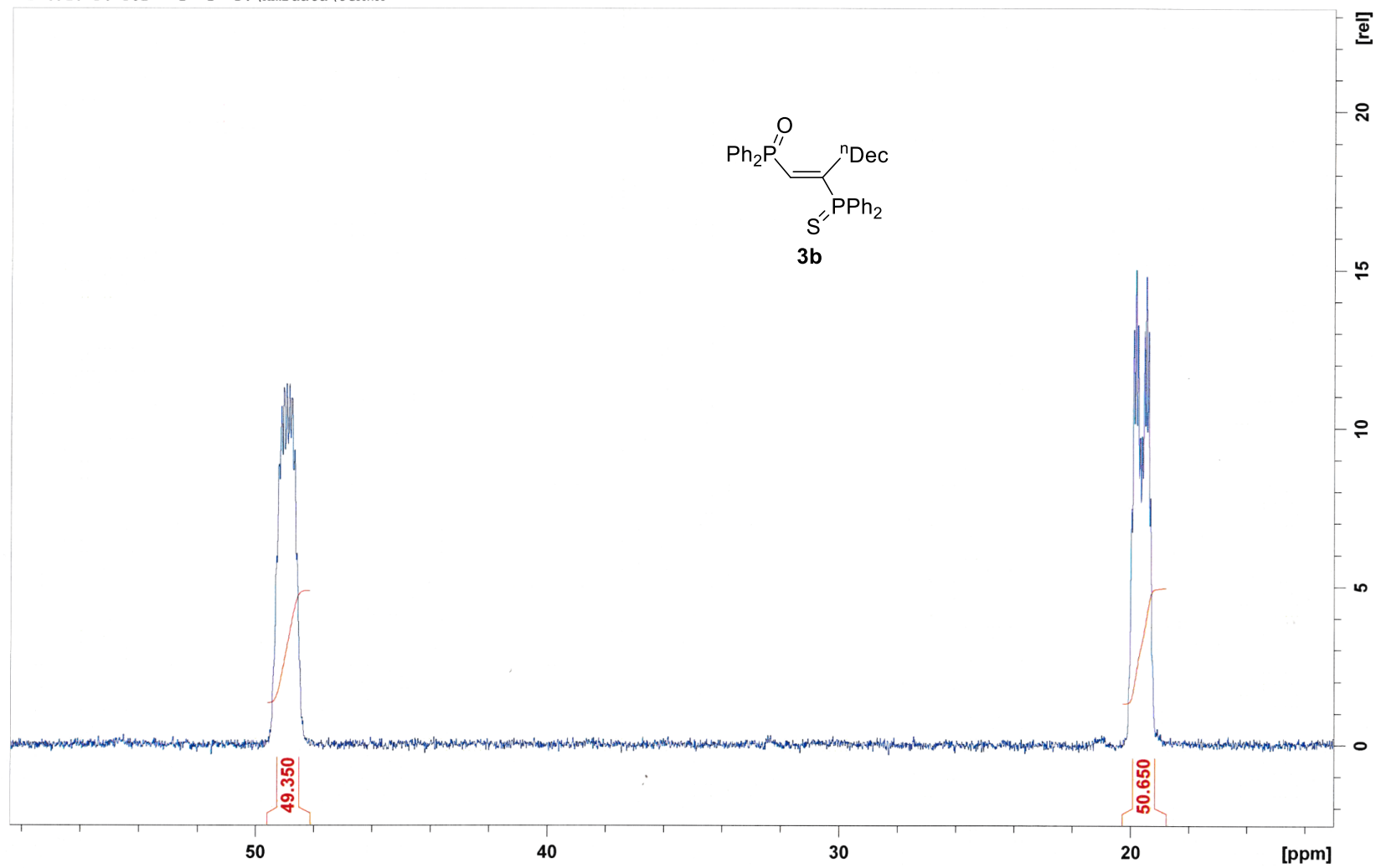


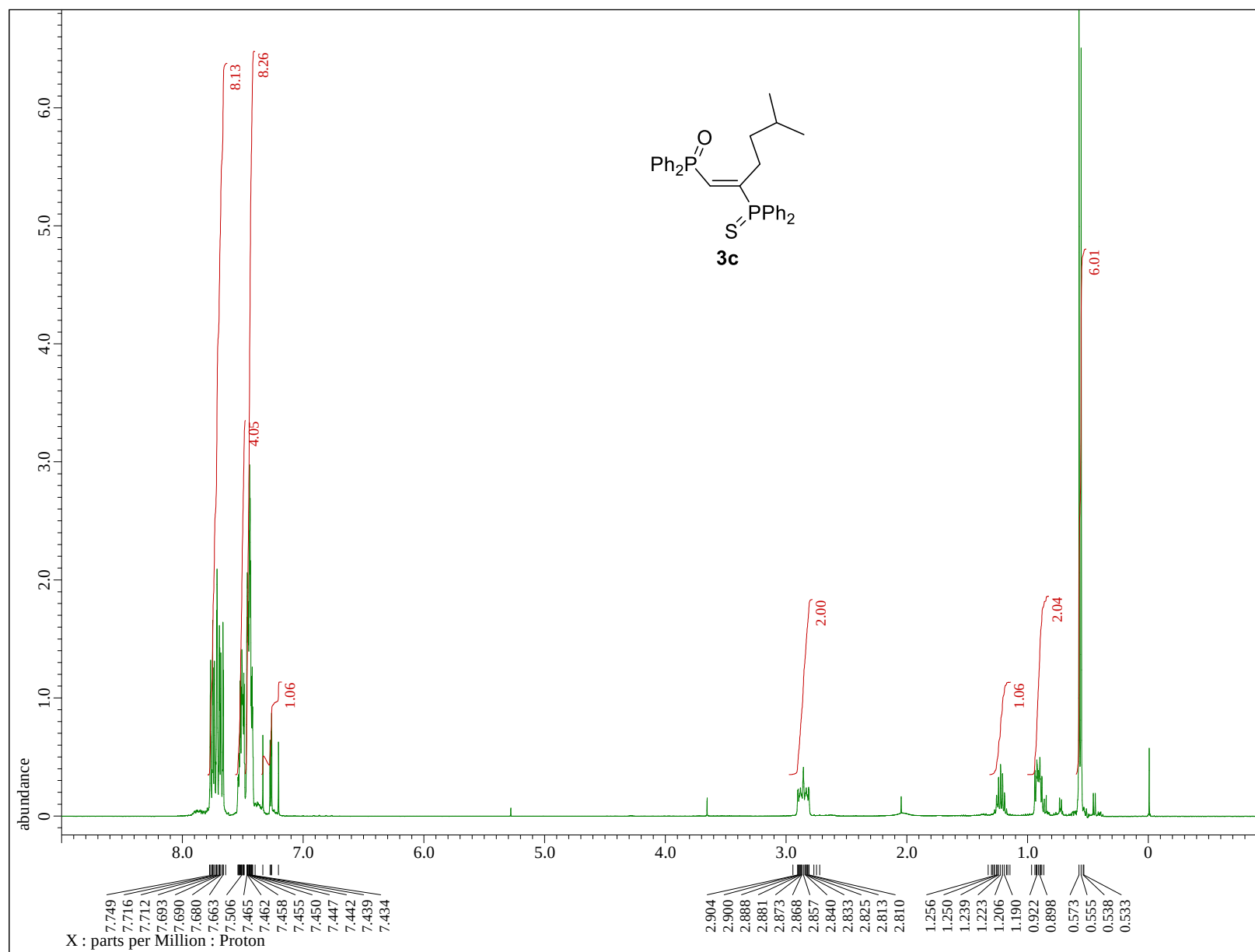


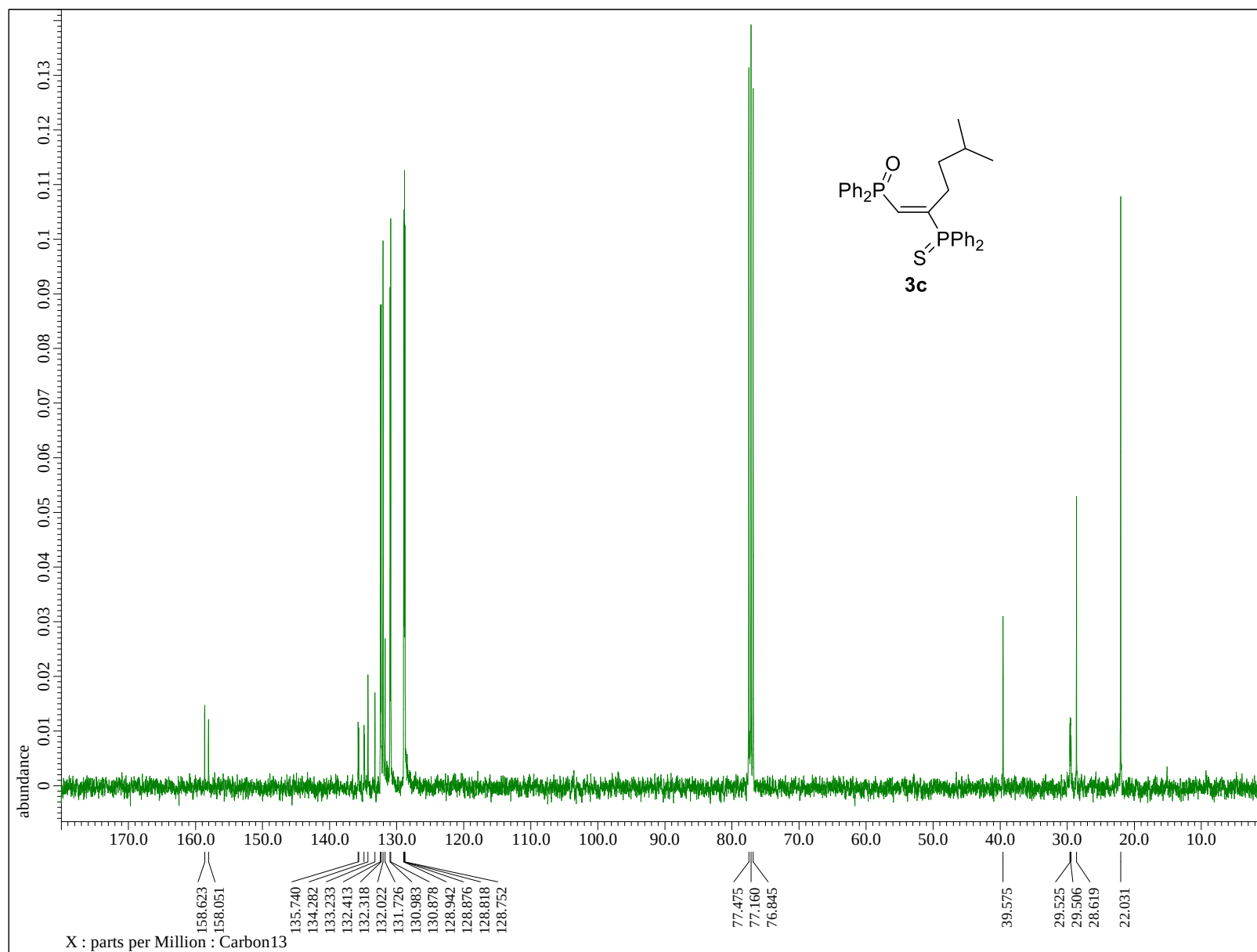


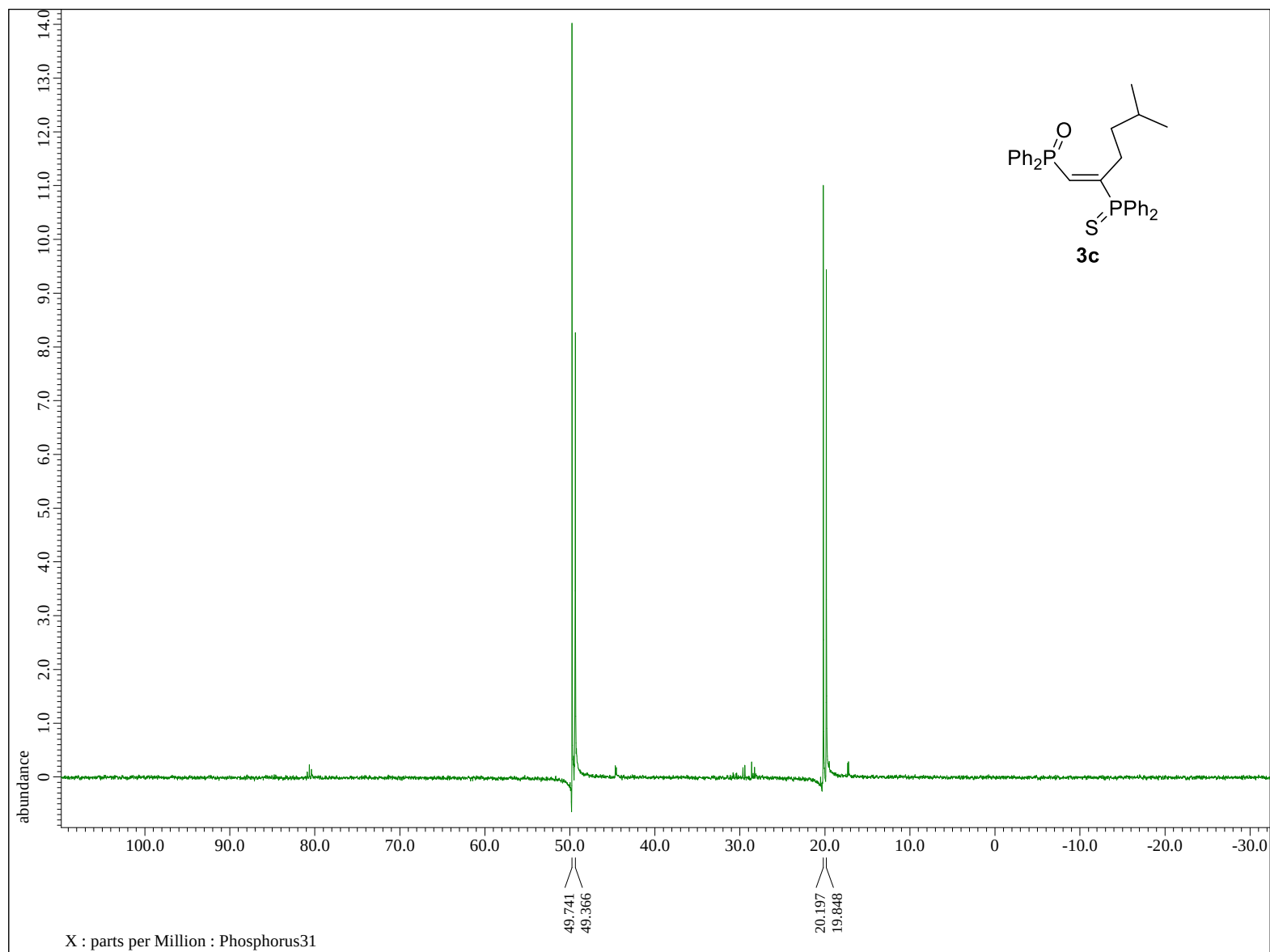


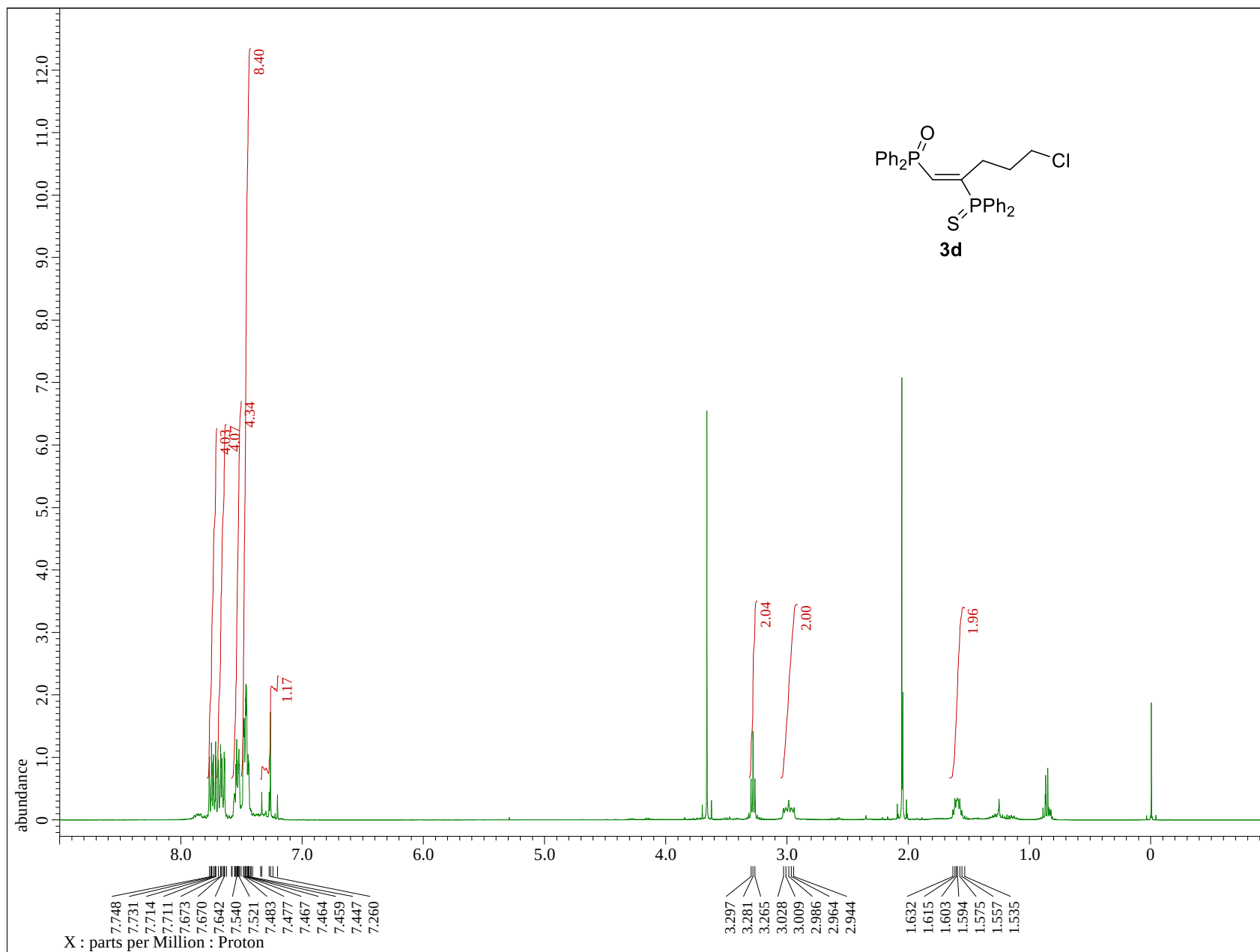
"D 0918 D6 TC2" 1 1 D:\nmrdata\OGAWA

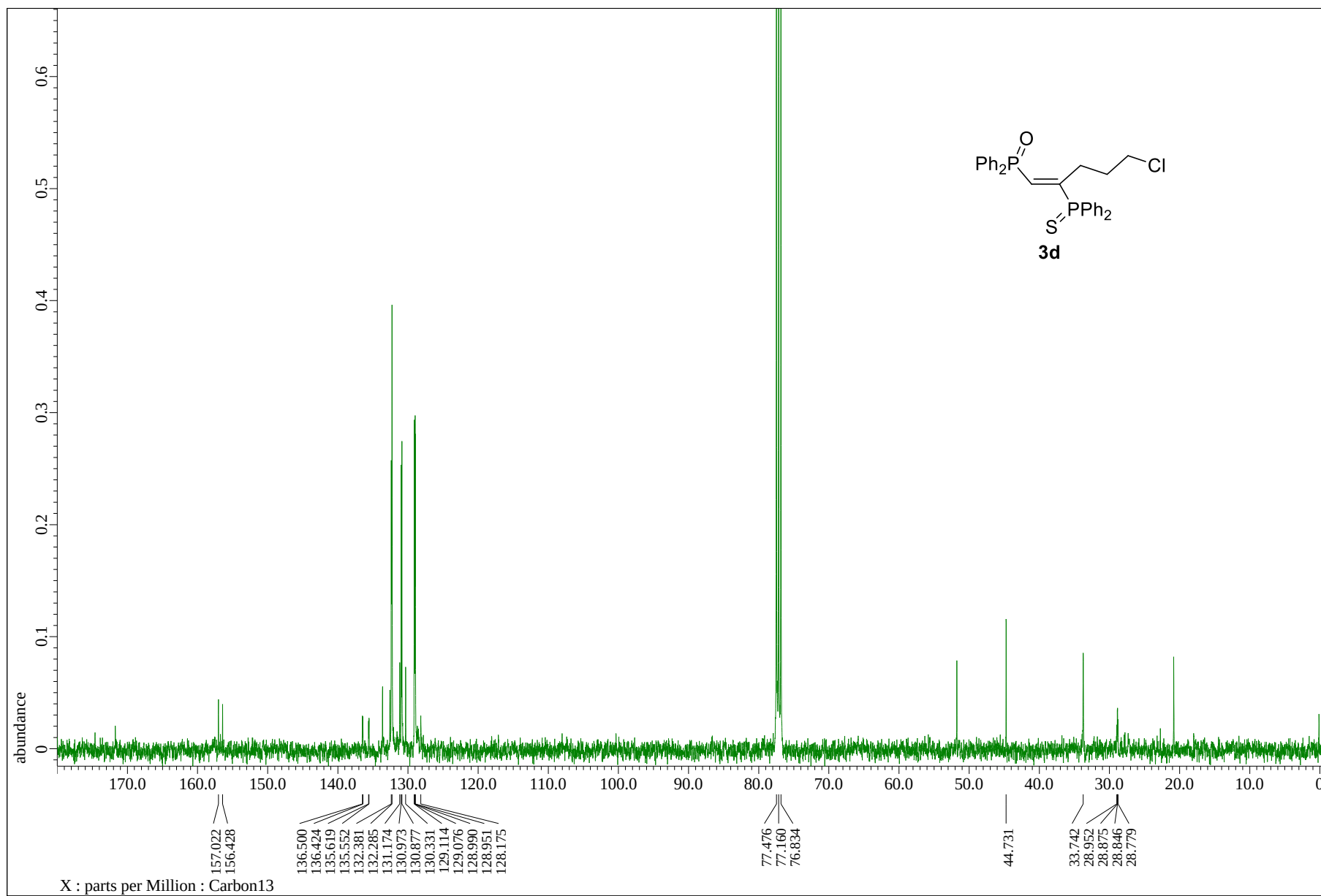




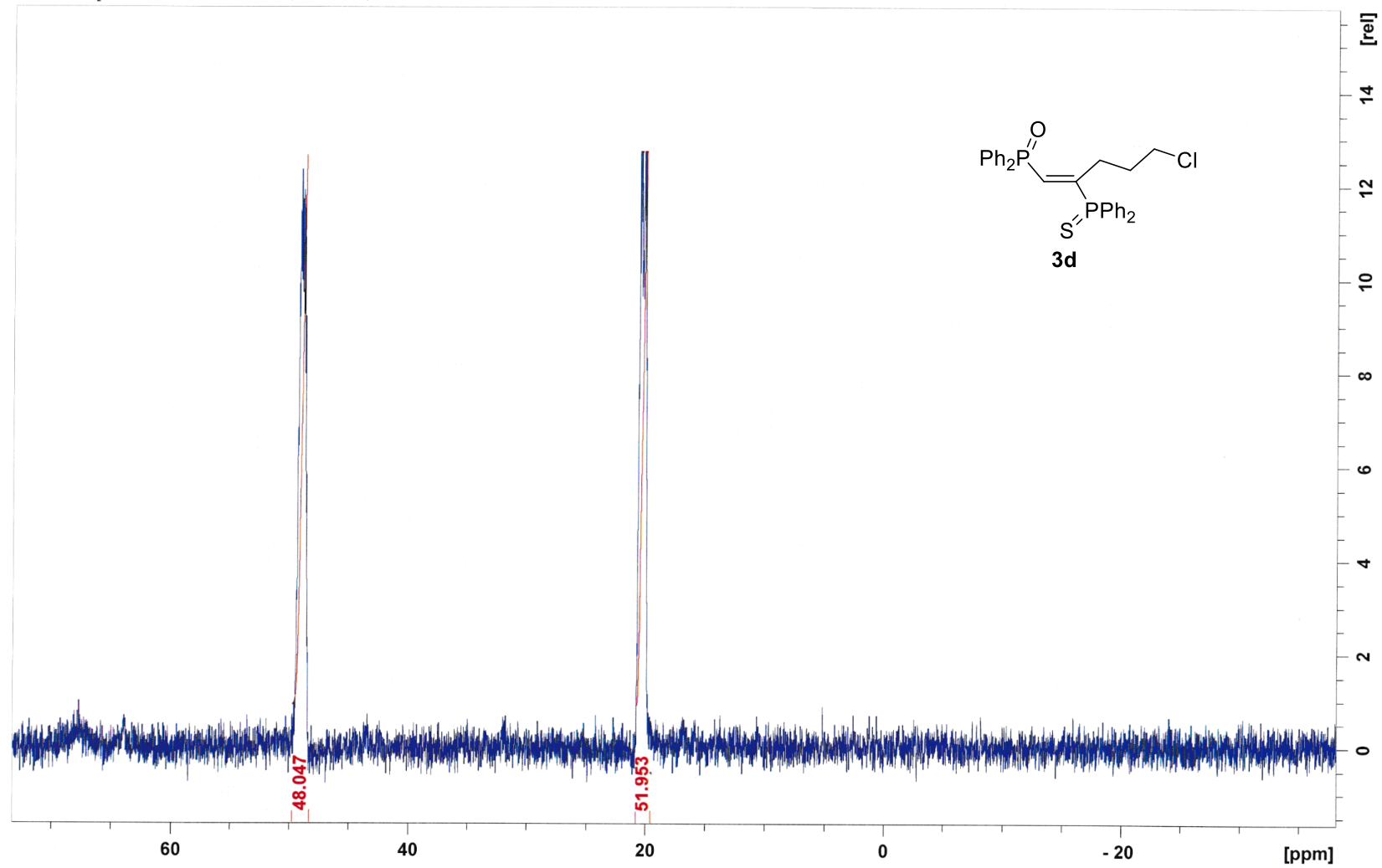


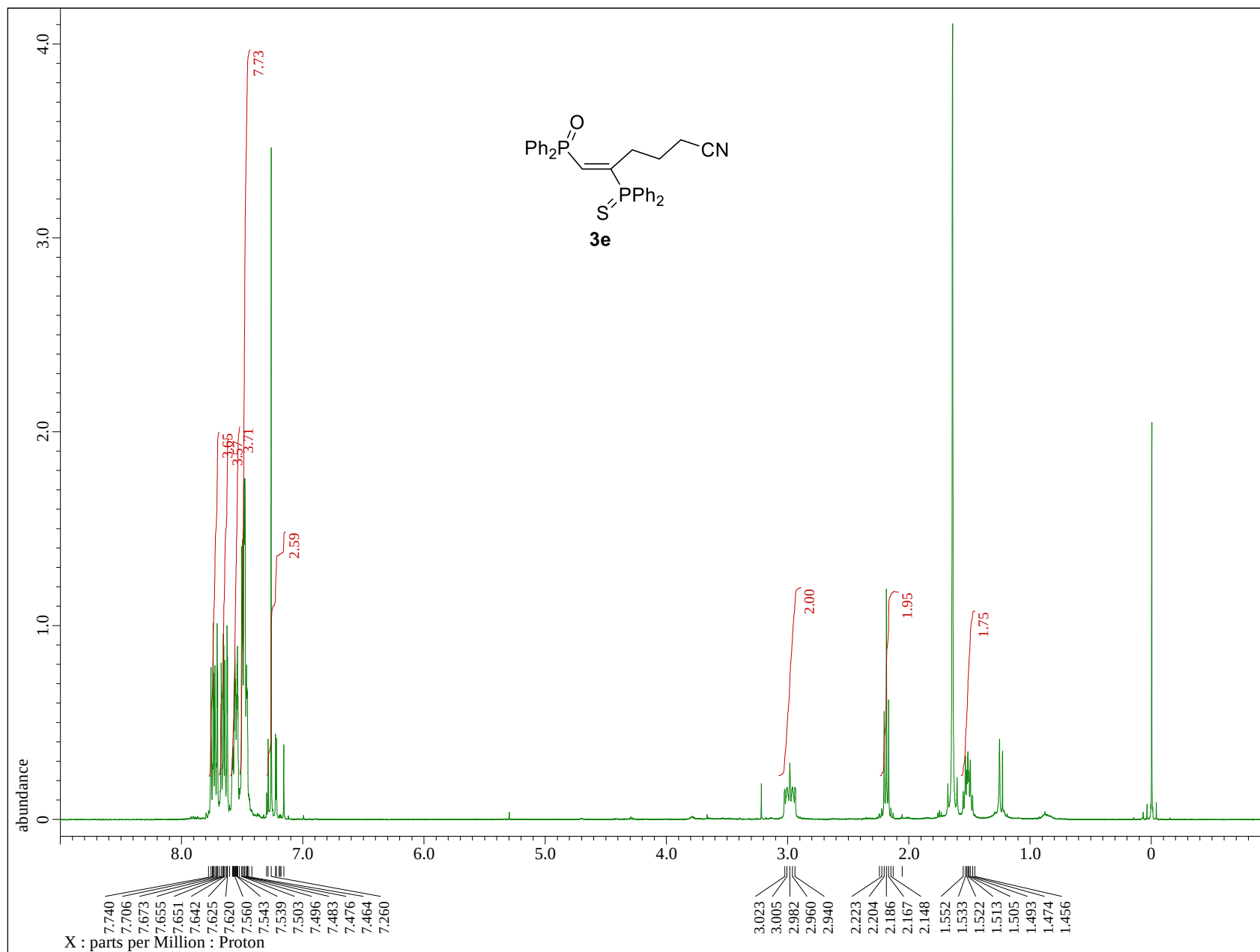


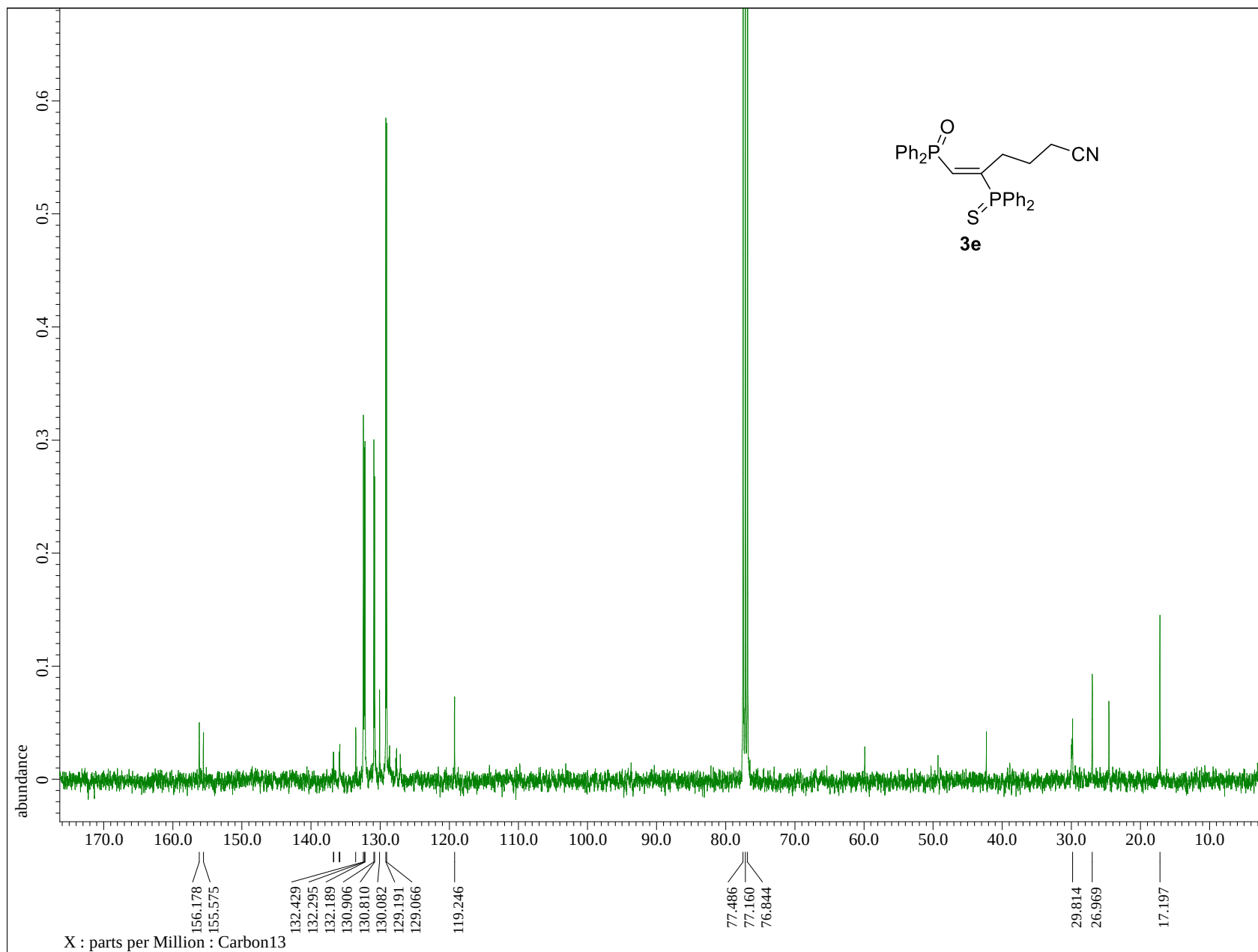




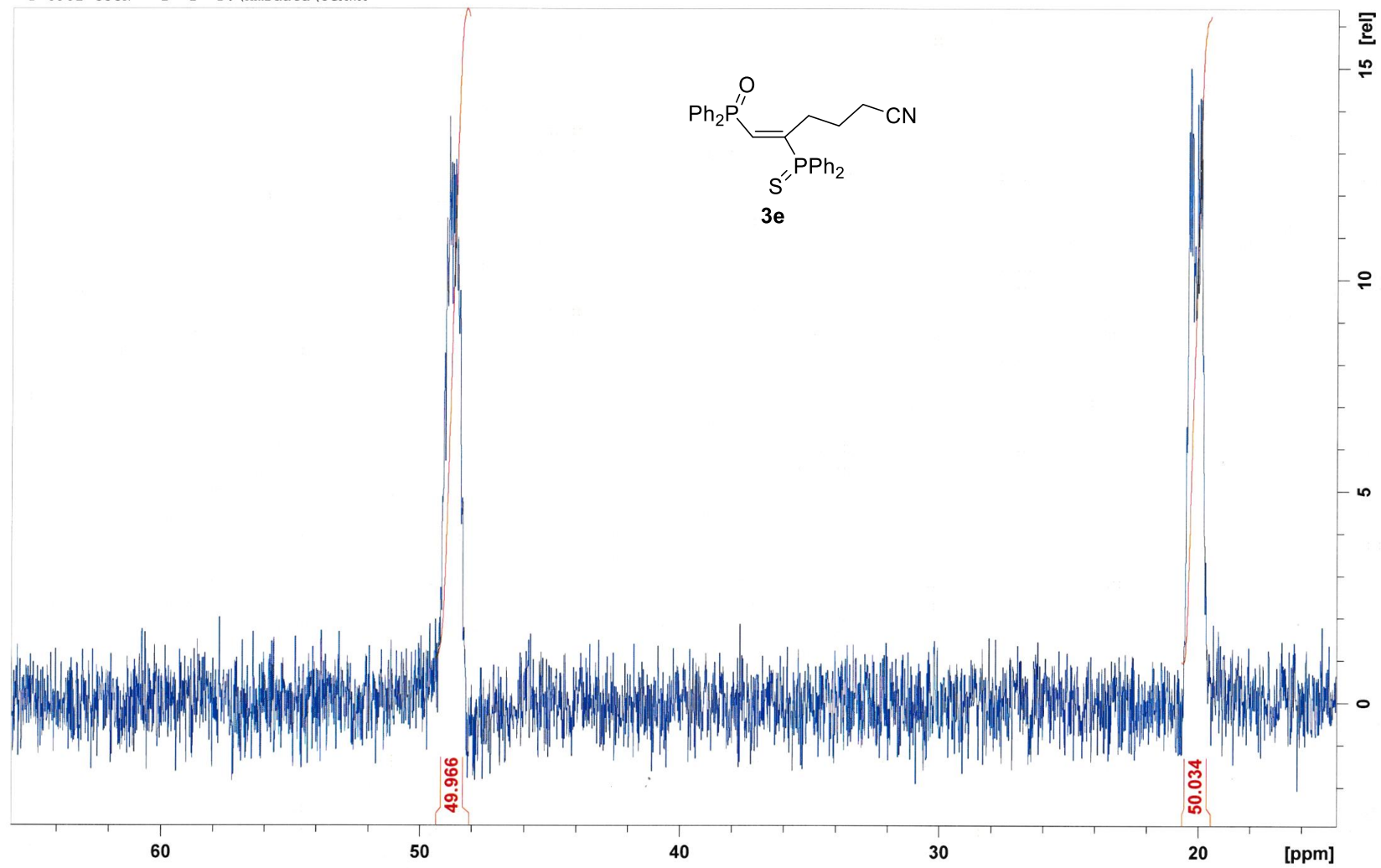
"D 0925 penclo S1" 1 1 D:\nmrdata\OGAWA

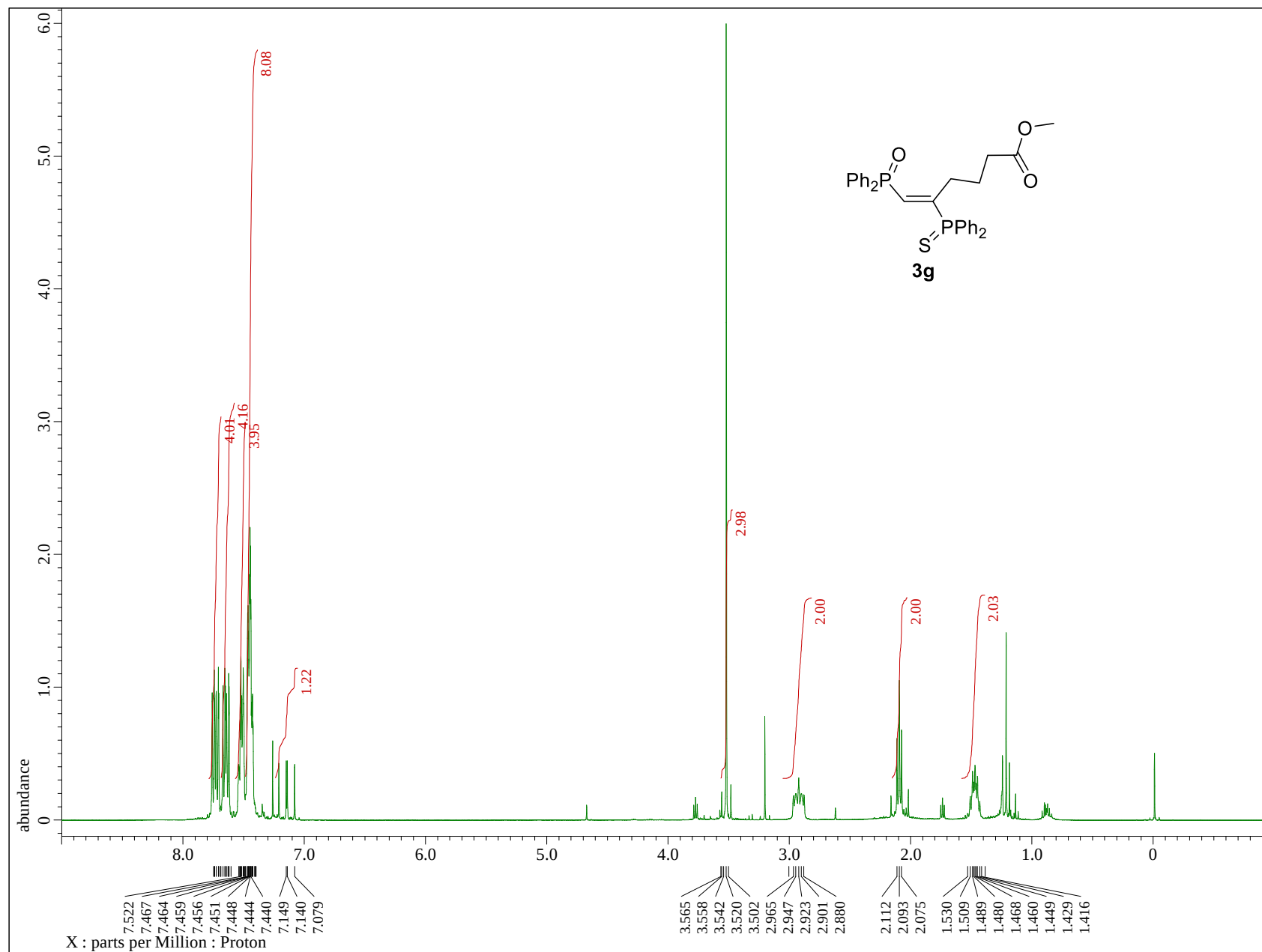


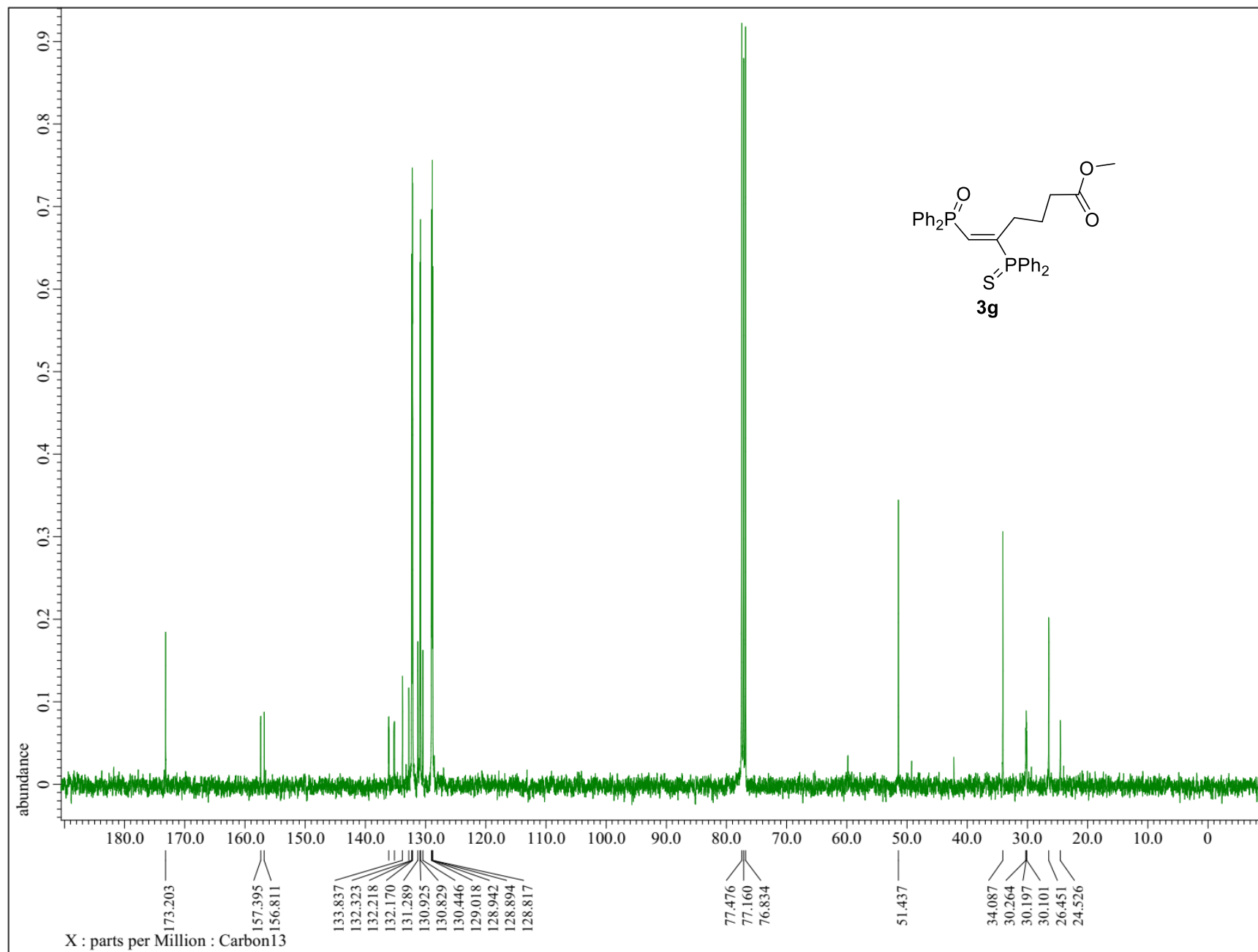




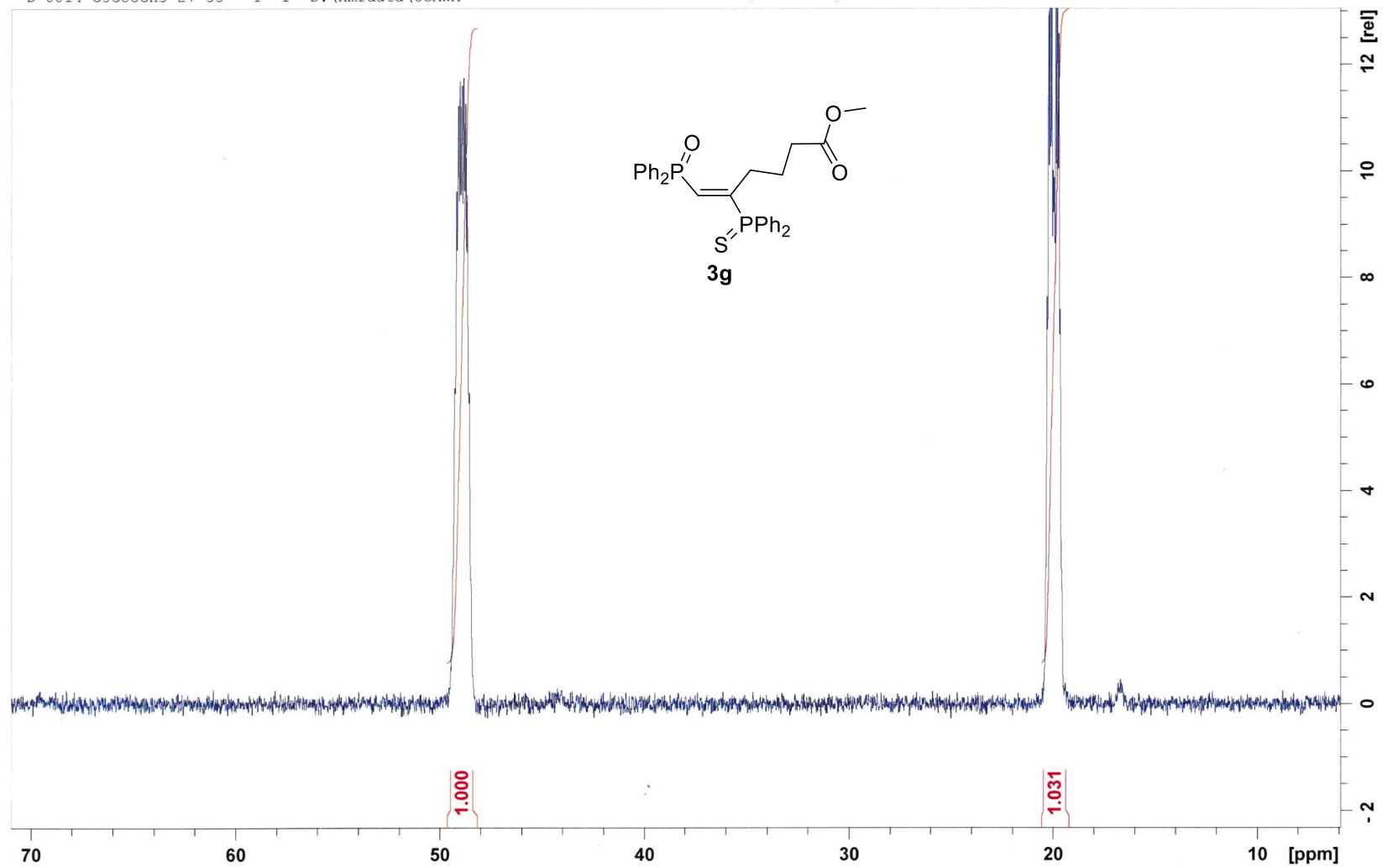
"D 0902 C5CN" 1 1 D:\nmrdata\OGAWA

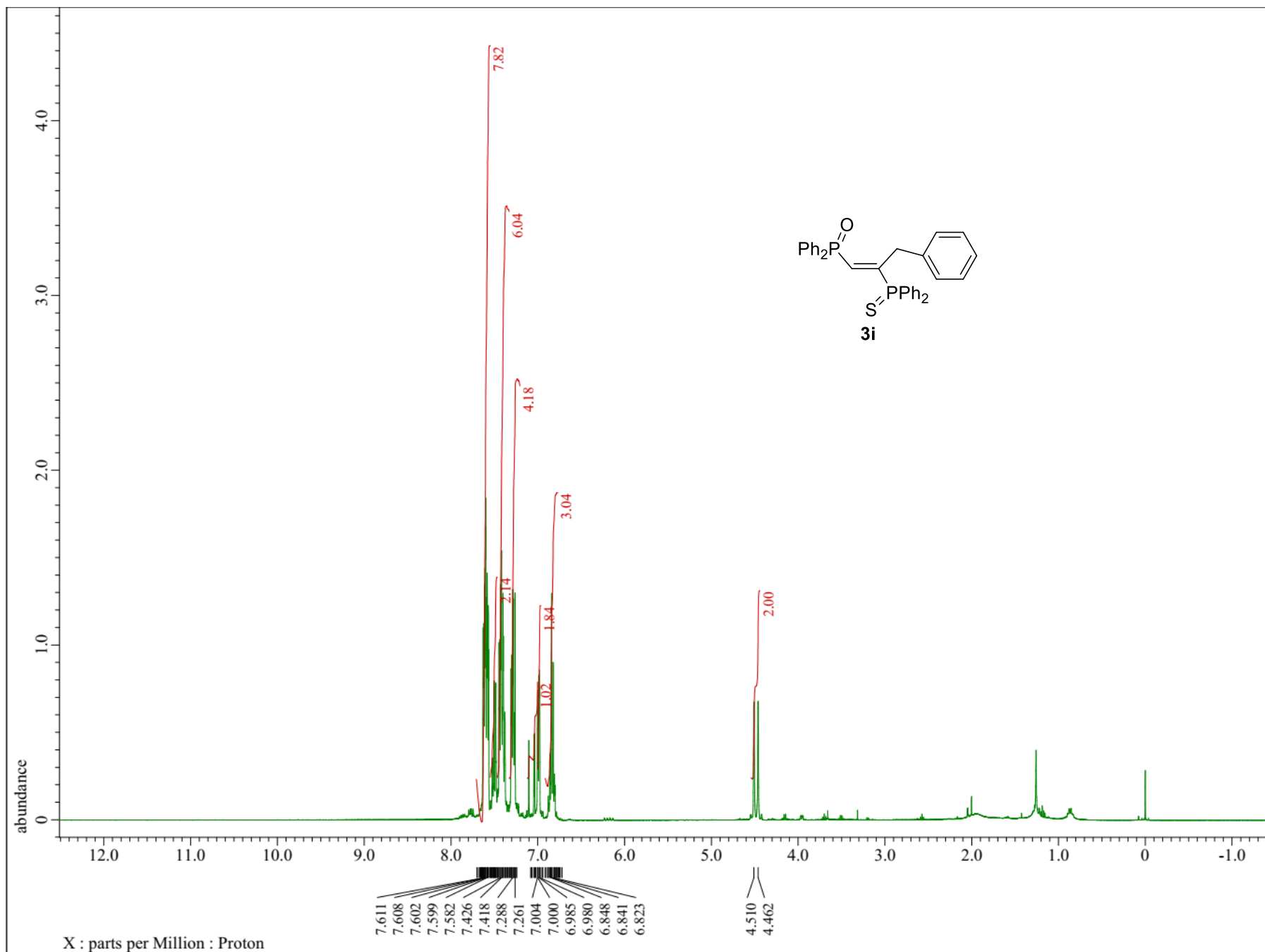


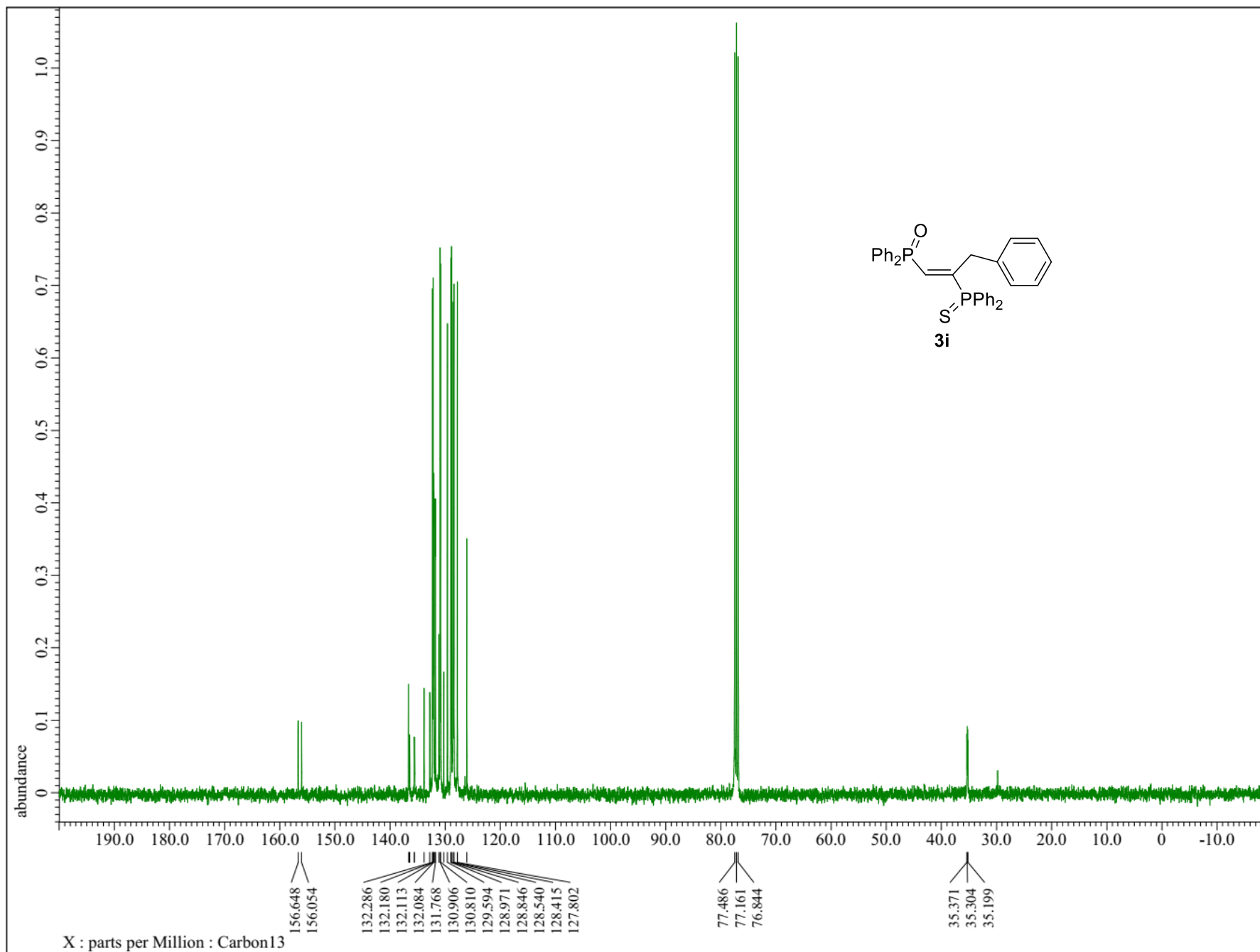




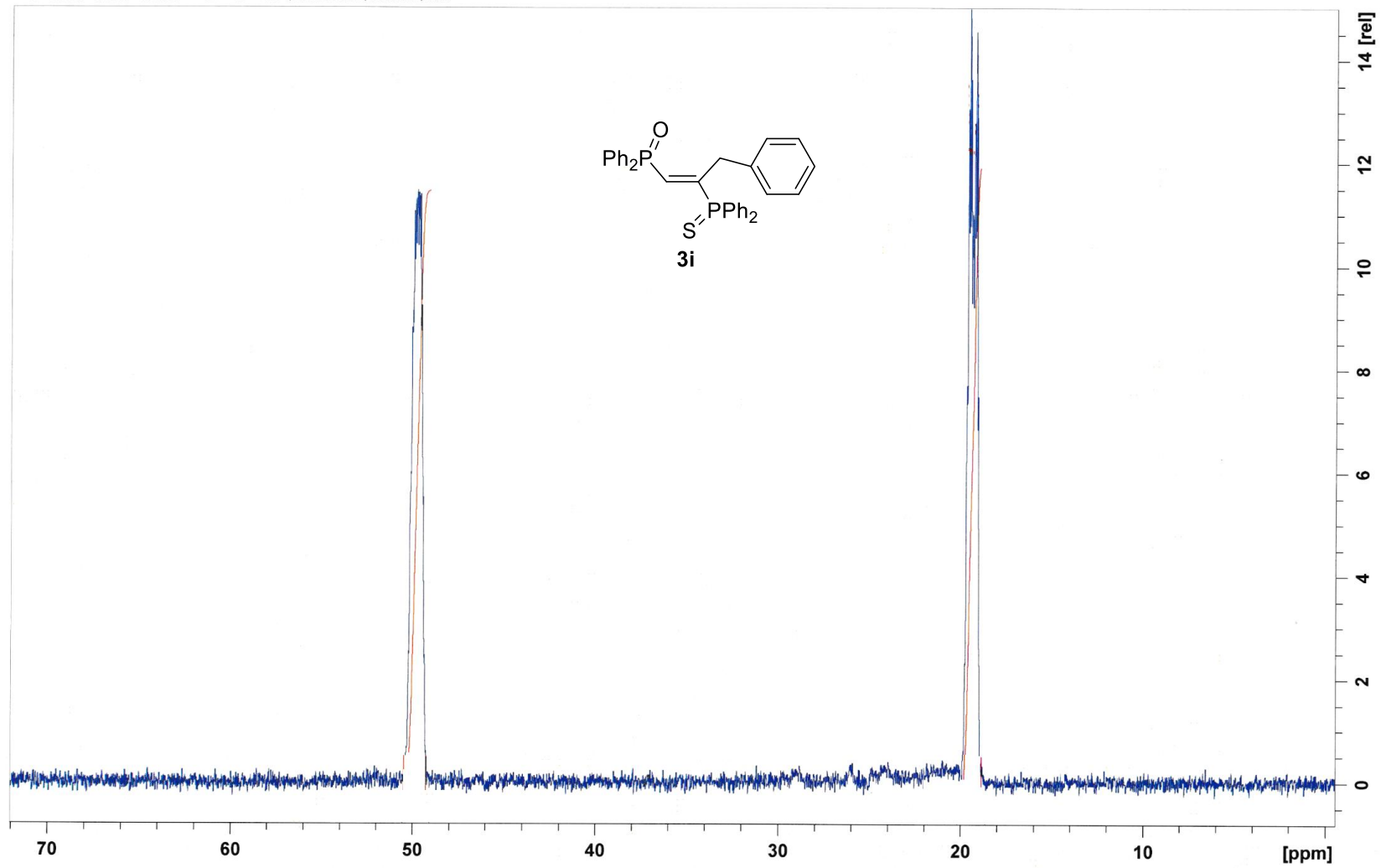
"D 0814 C5COOCh3 27 35" 1 1 D:\nmrdata\OGAWA

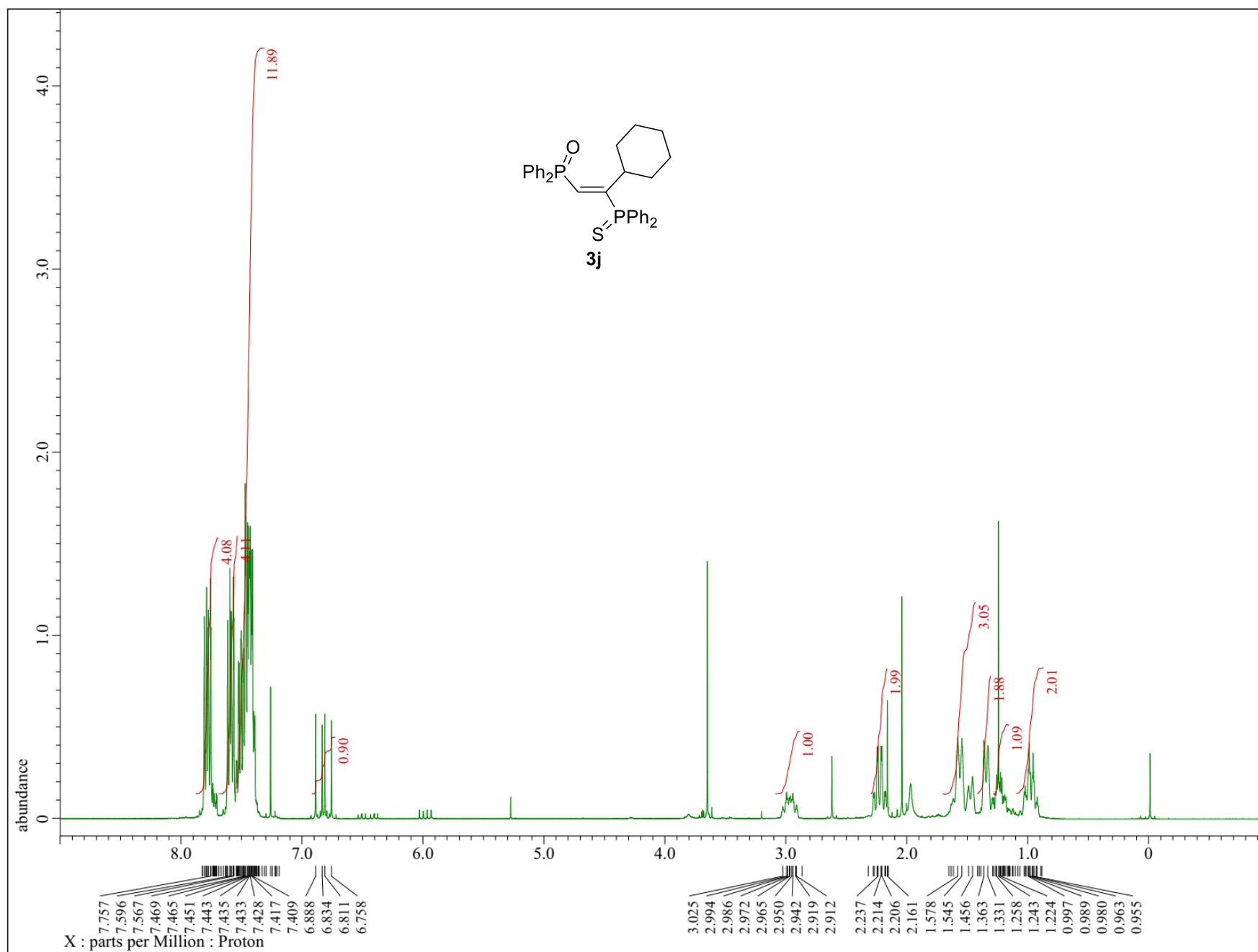


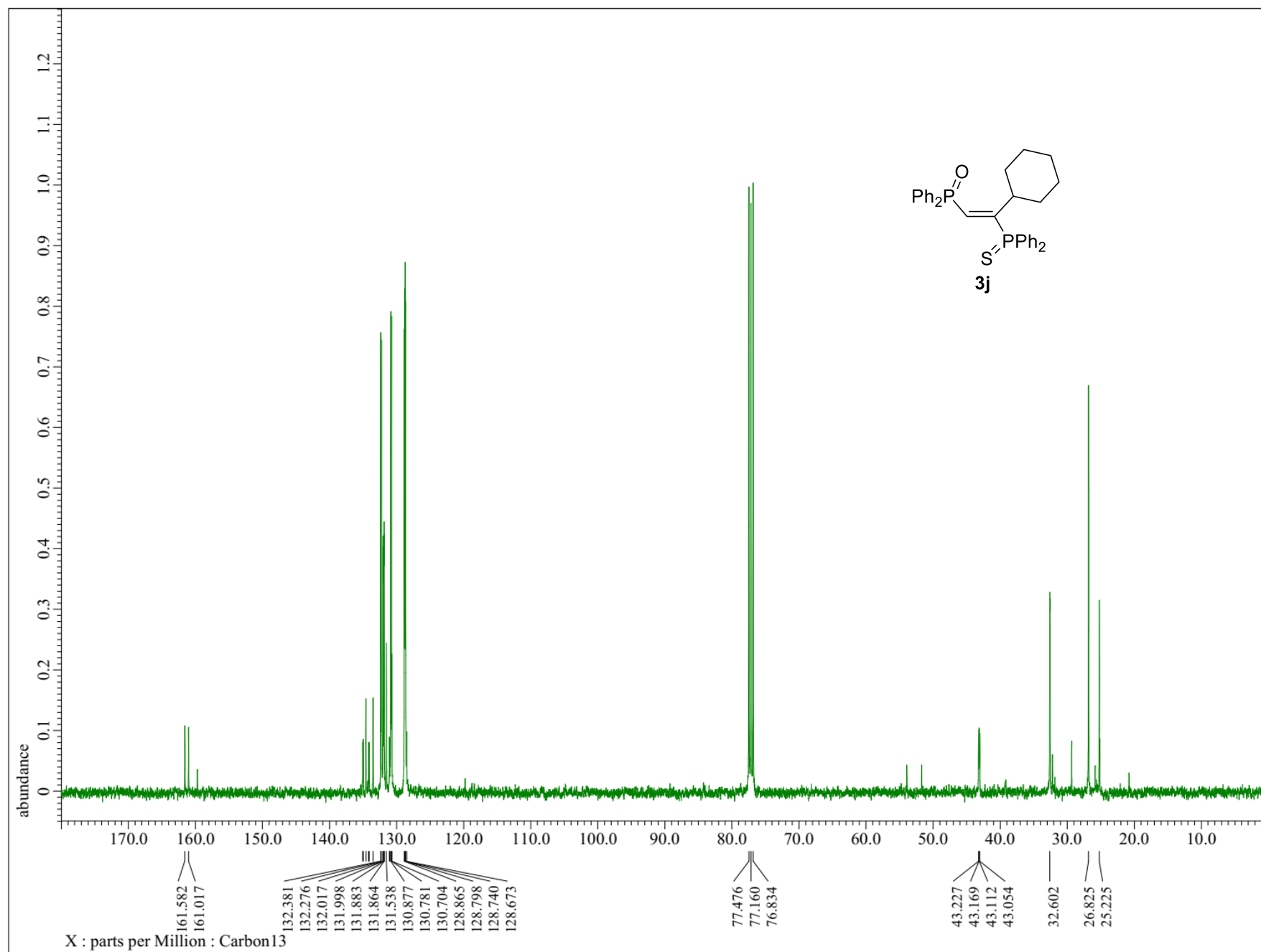




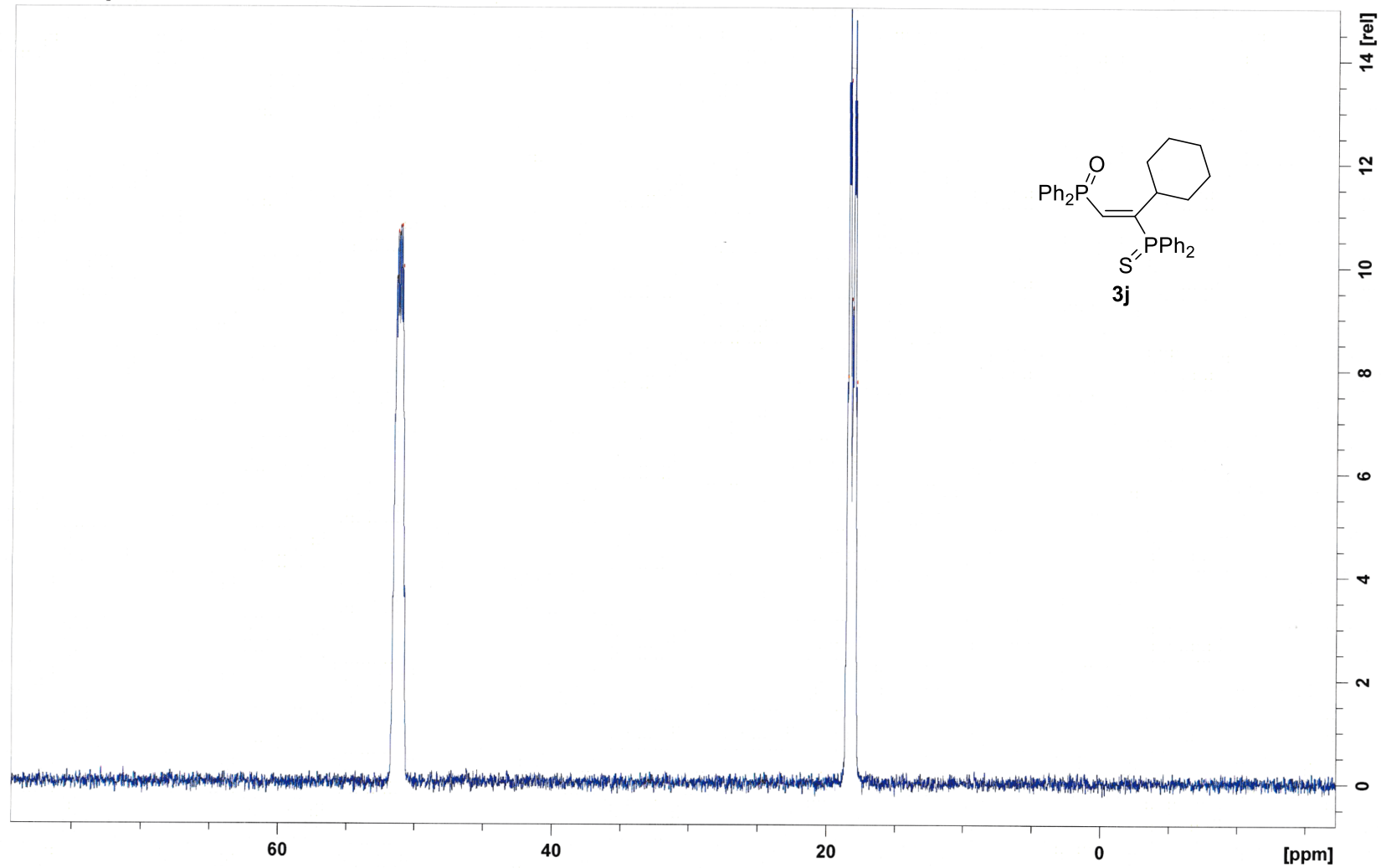
"D 1009 PhC3 CC11" 1 1 D:\nmrdata\OGAWA\Dat

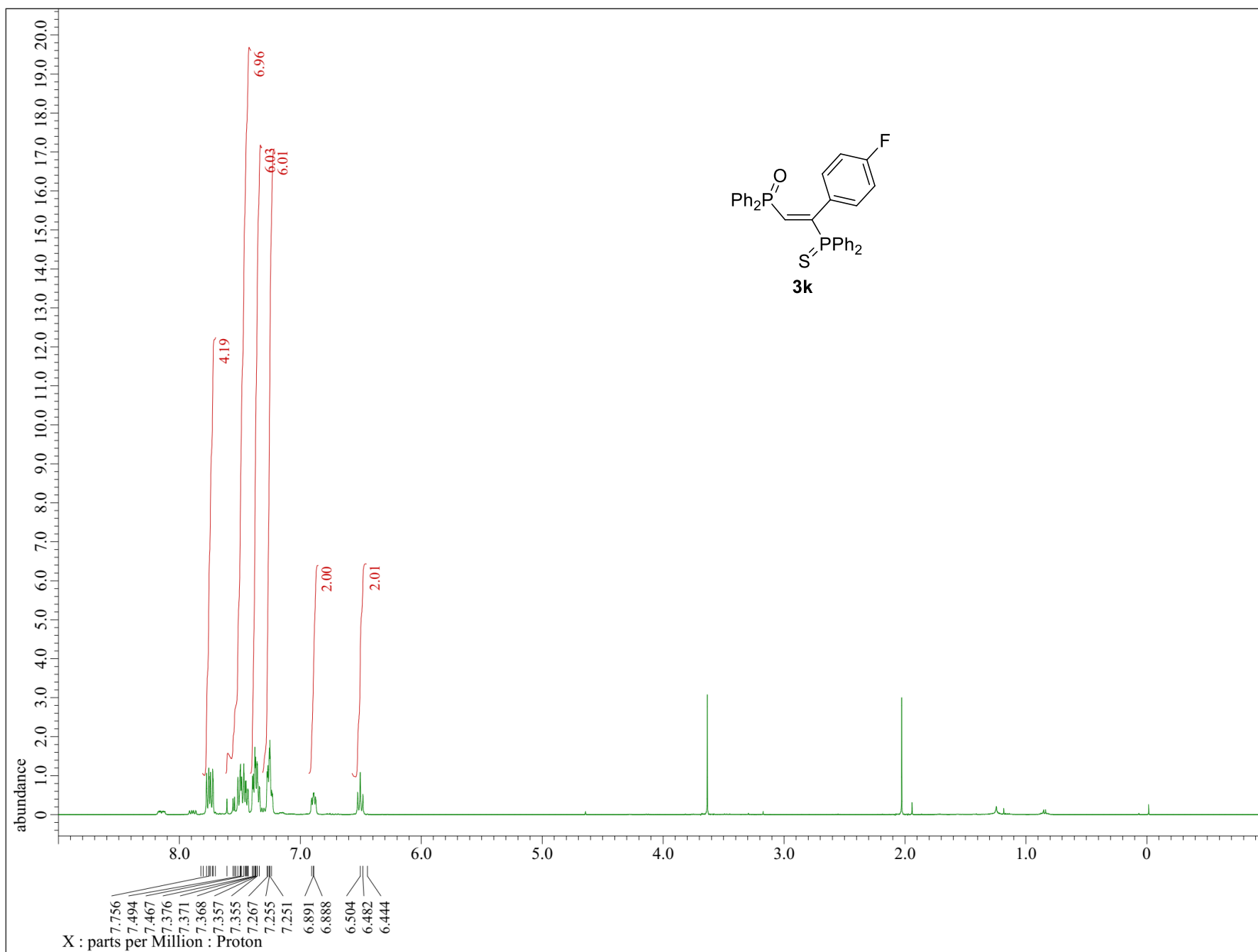


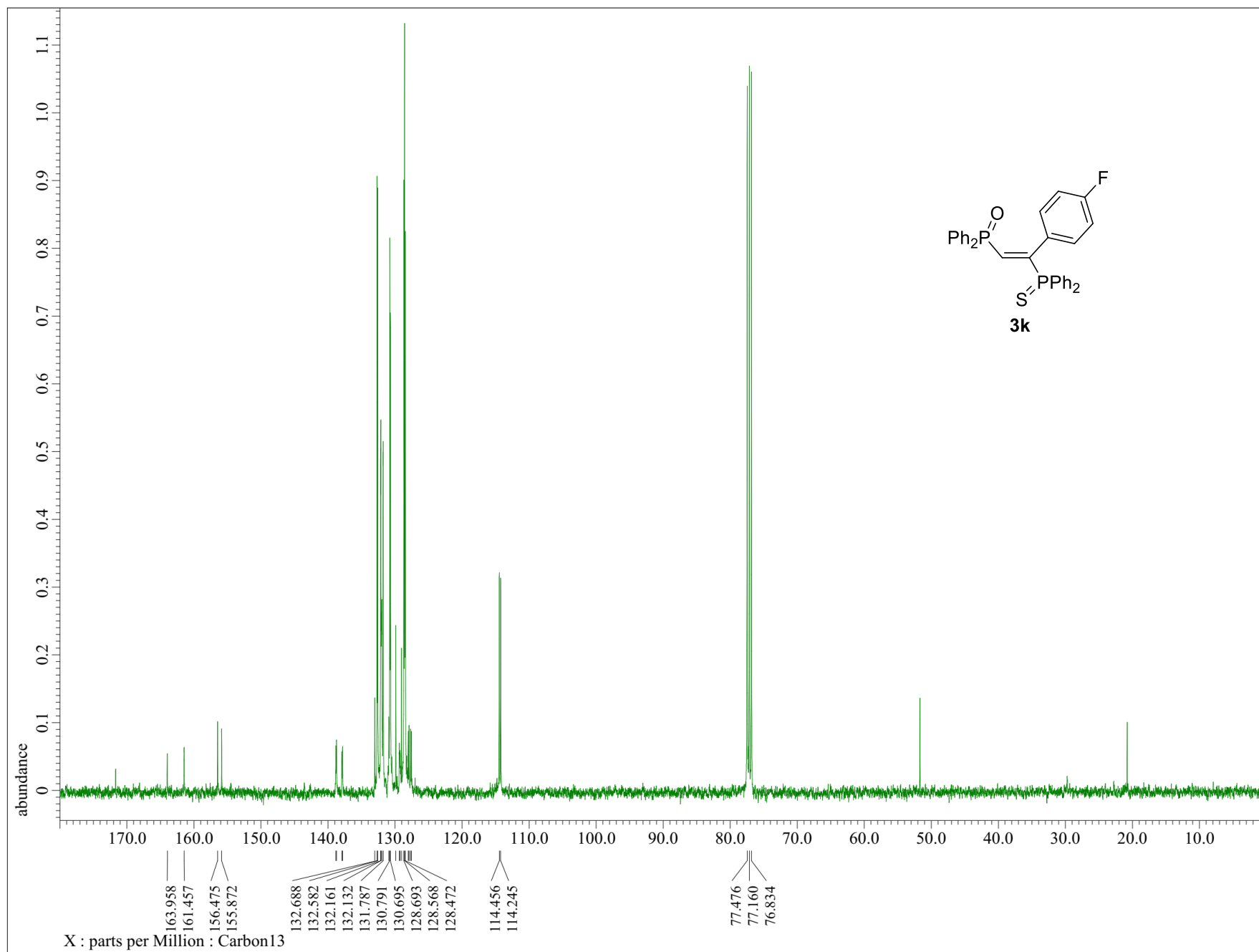




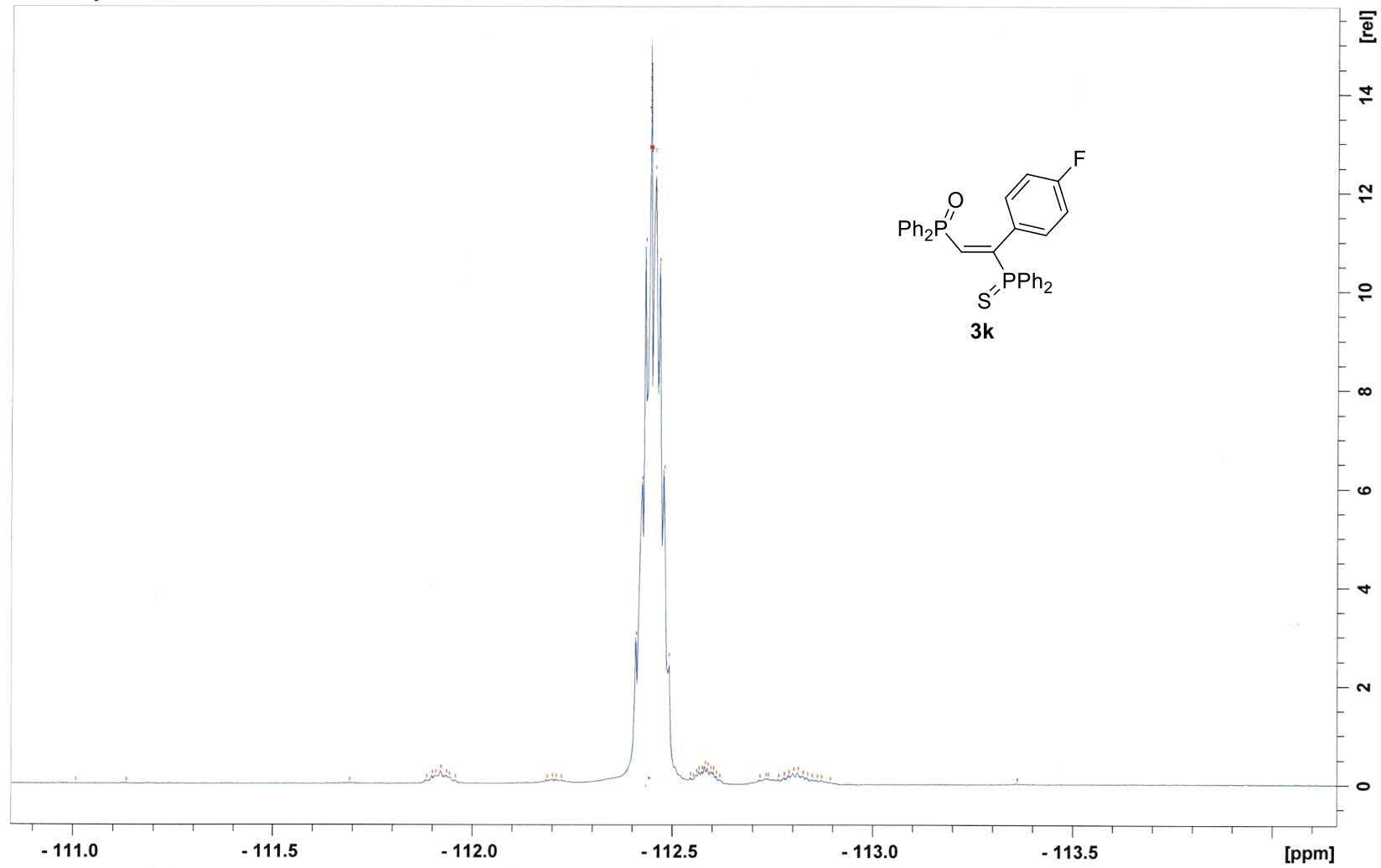
"D 0903 cyclohex" 1 1 D:\nmrdata\OGAWA



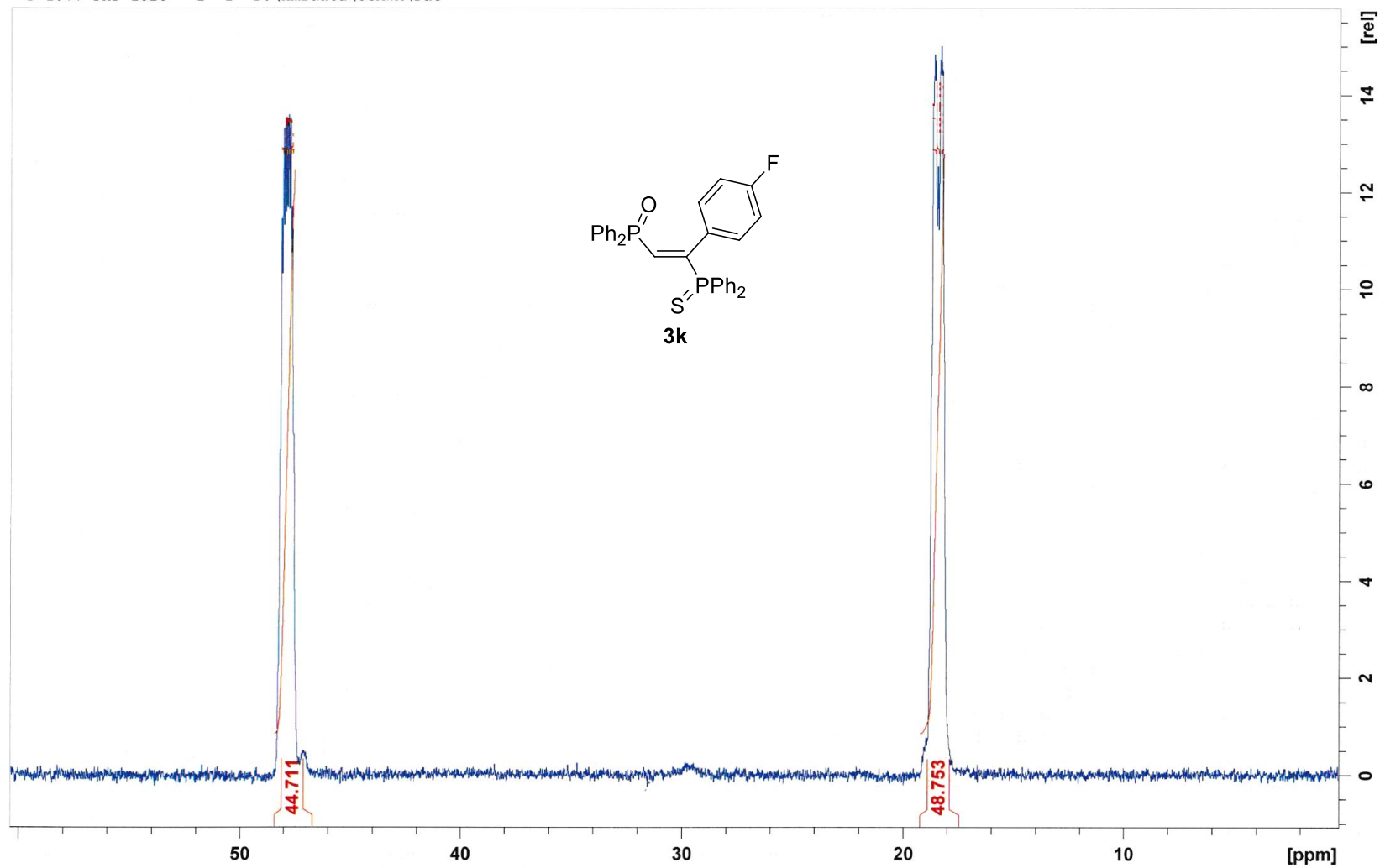


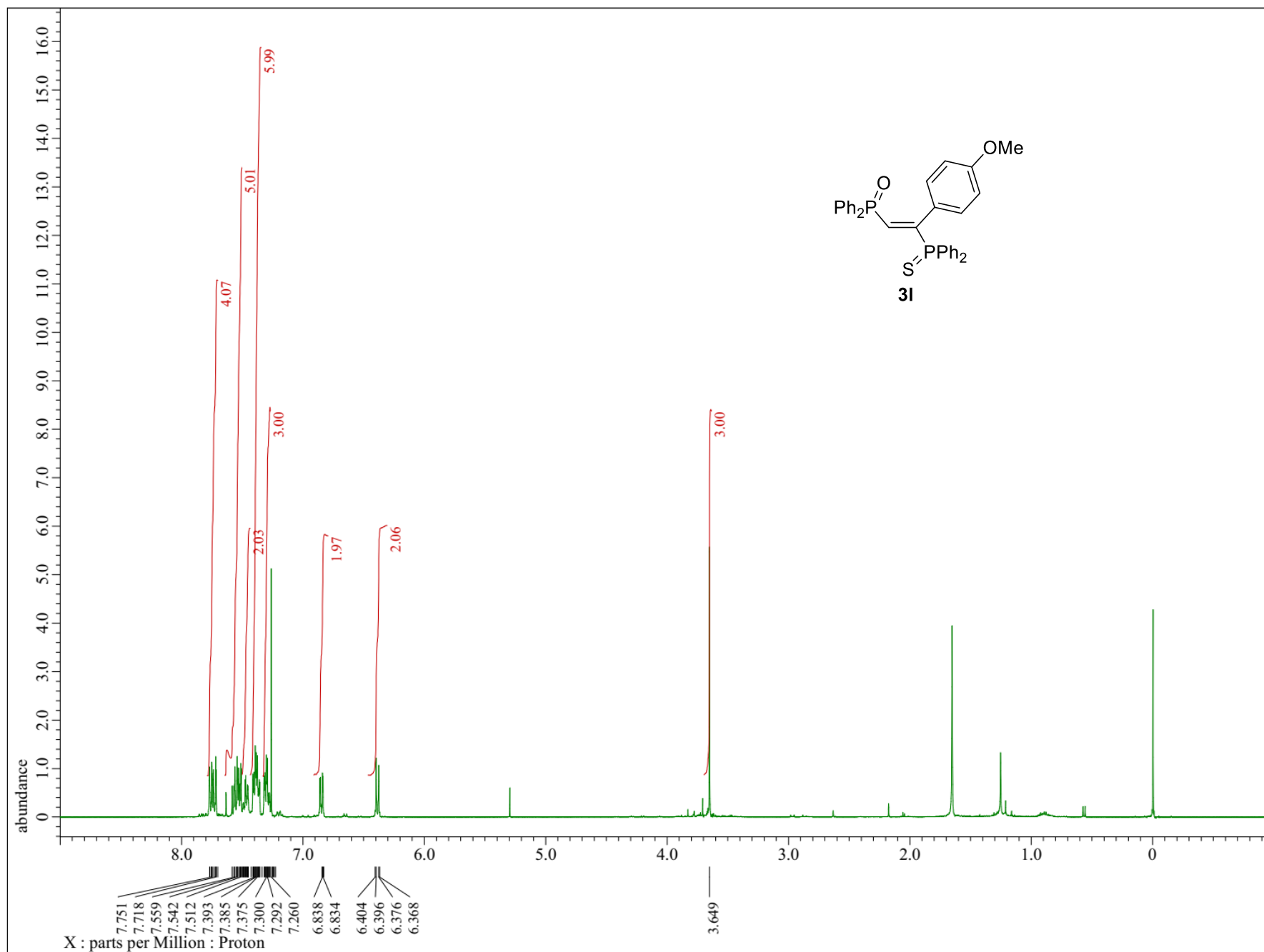


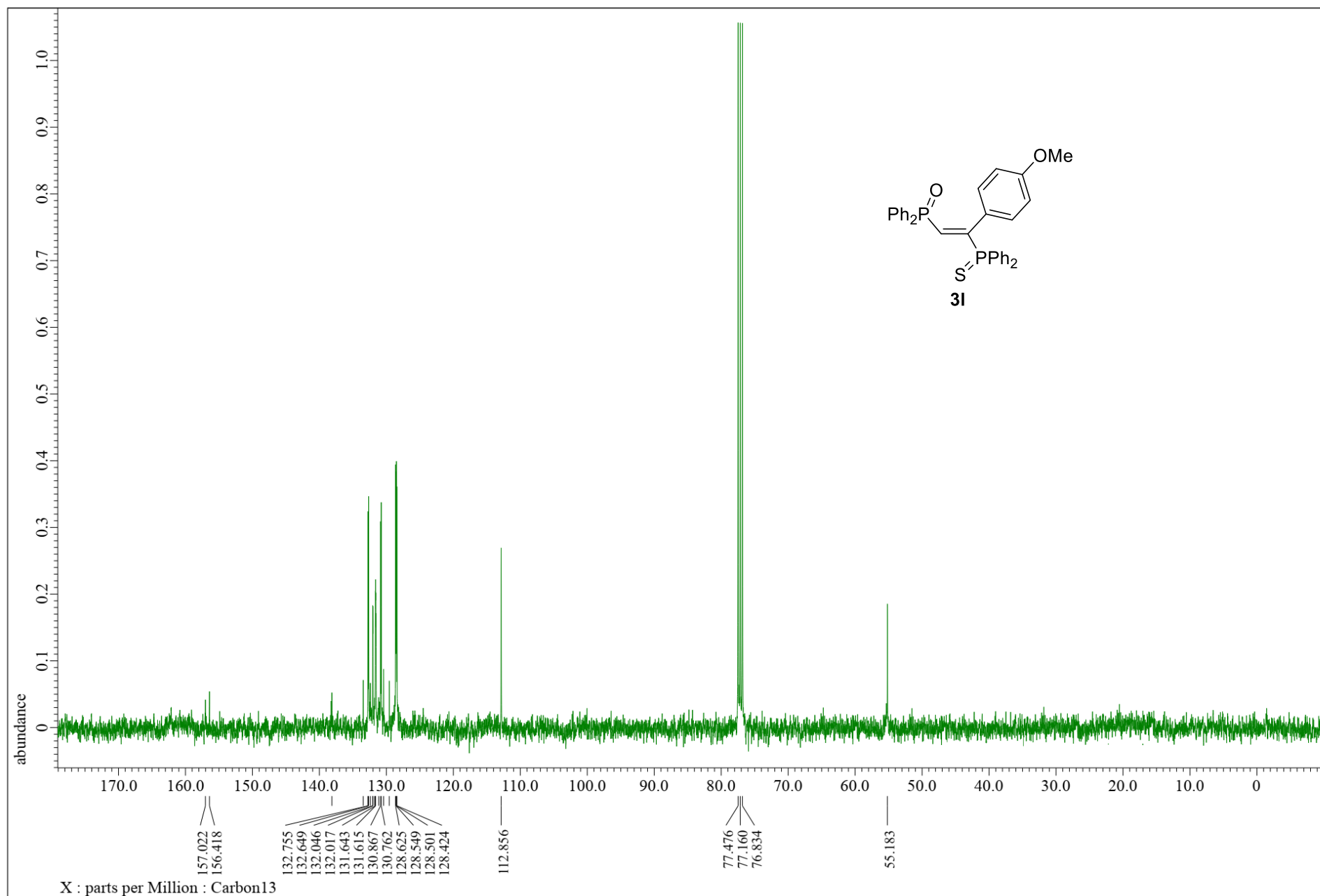
"D 1015 phF 3" 1 1 D:\nmrdata\OGAWA\Dat



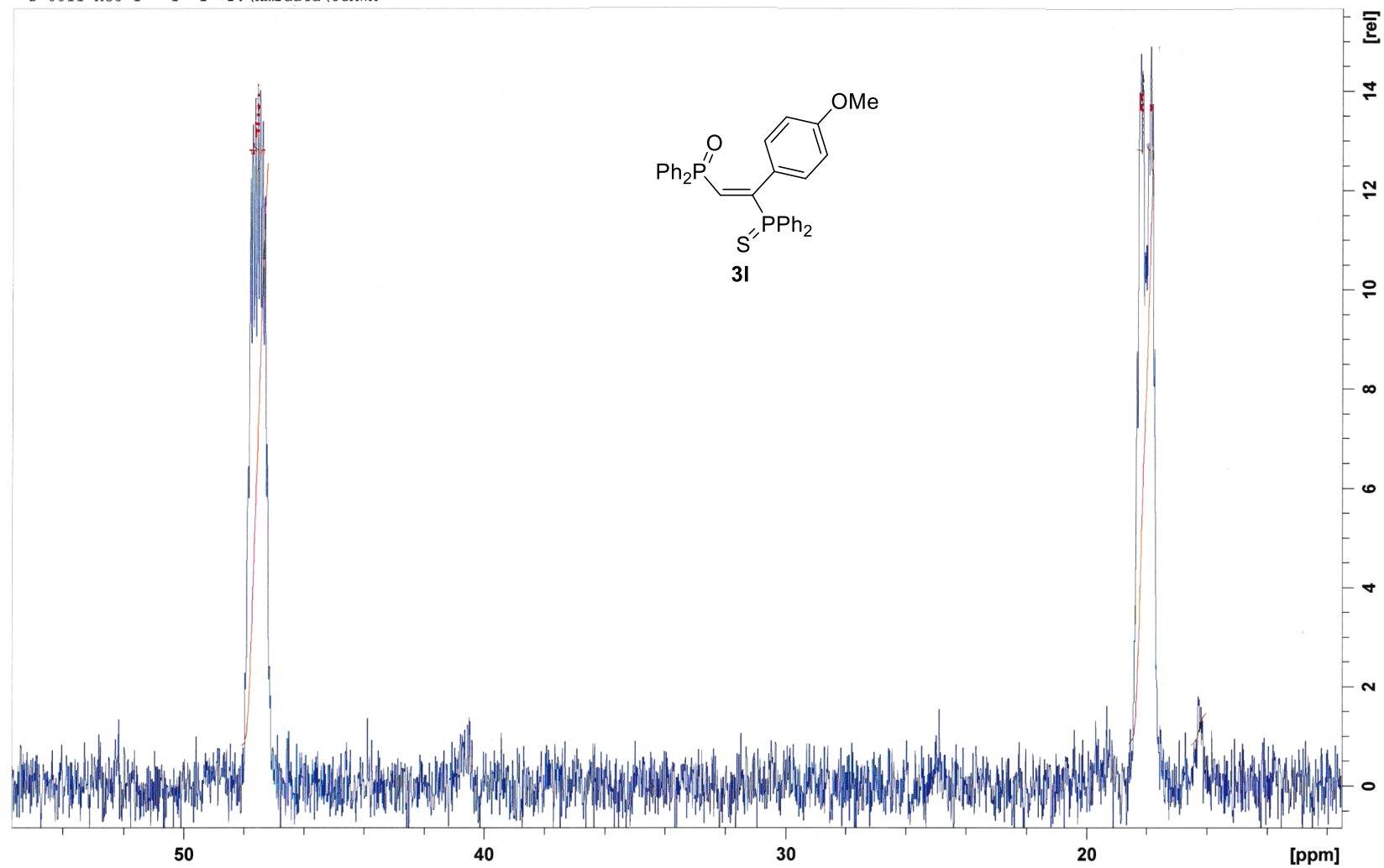
"D 10vv PhF 1820" 1 1 D:\nmrdata\OGAWA\Dat

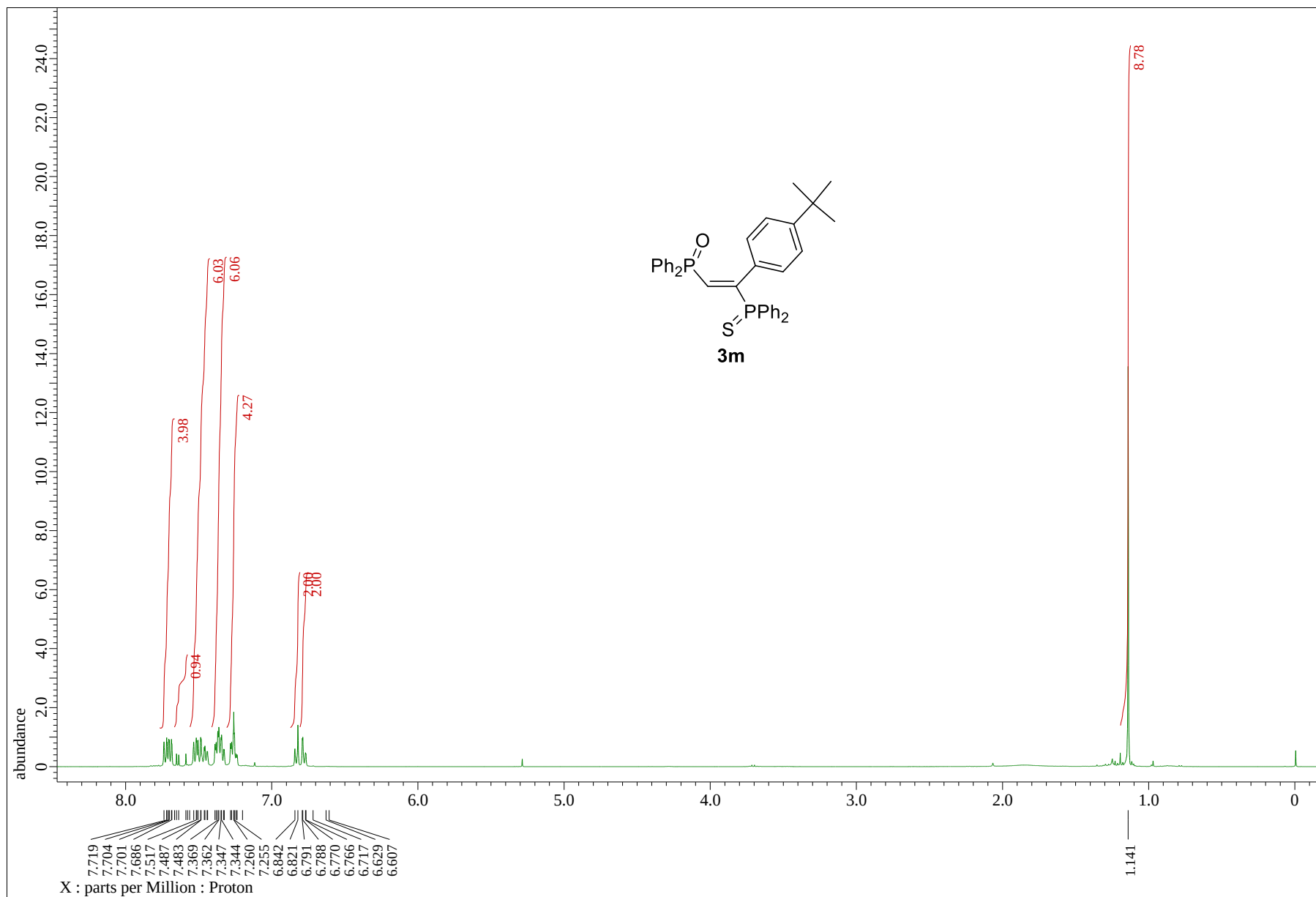


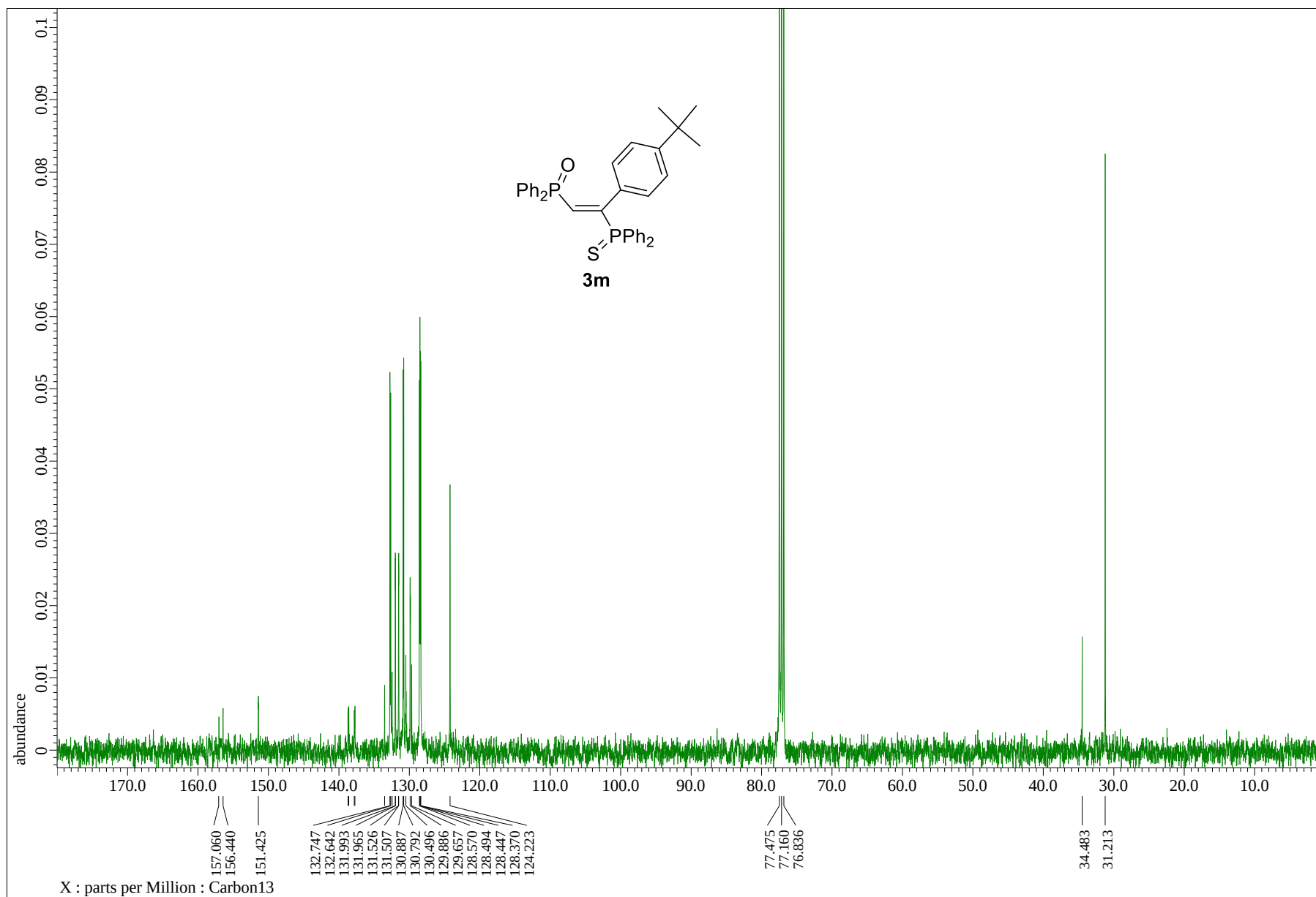


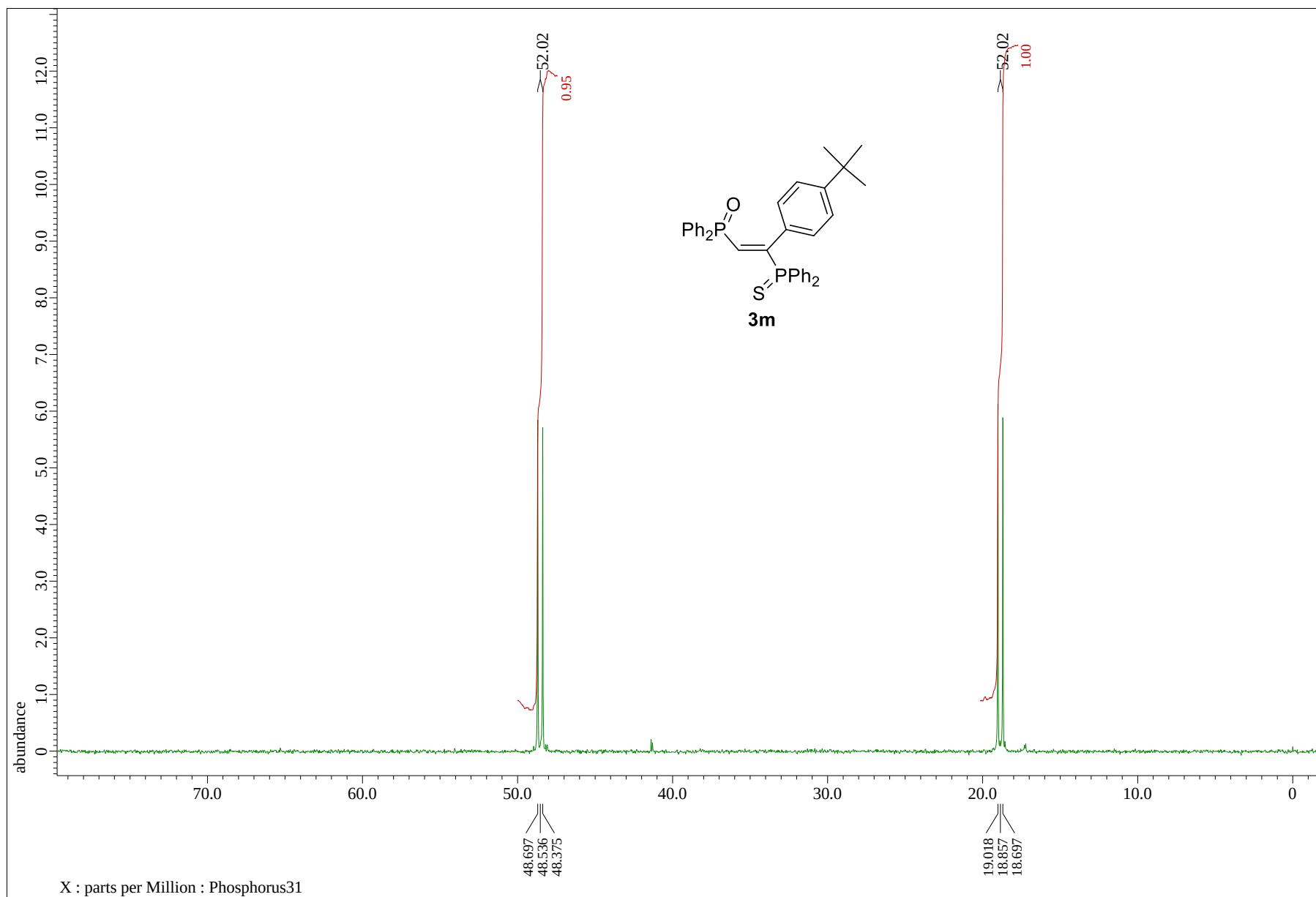


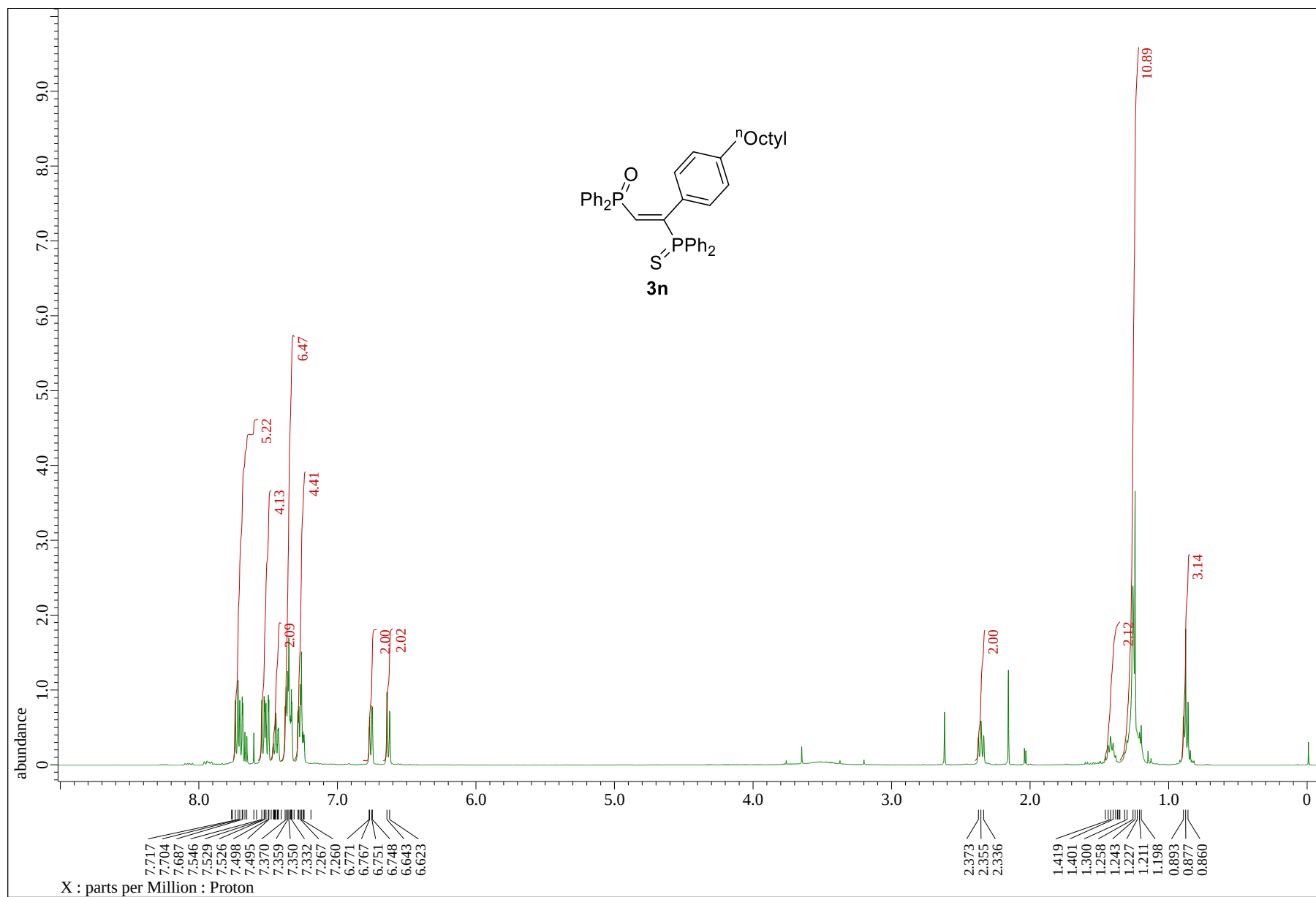
"D 0811 MeO 1" 1 1 D:\nmrdata\OGAWA

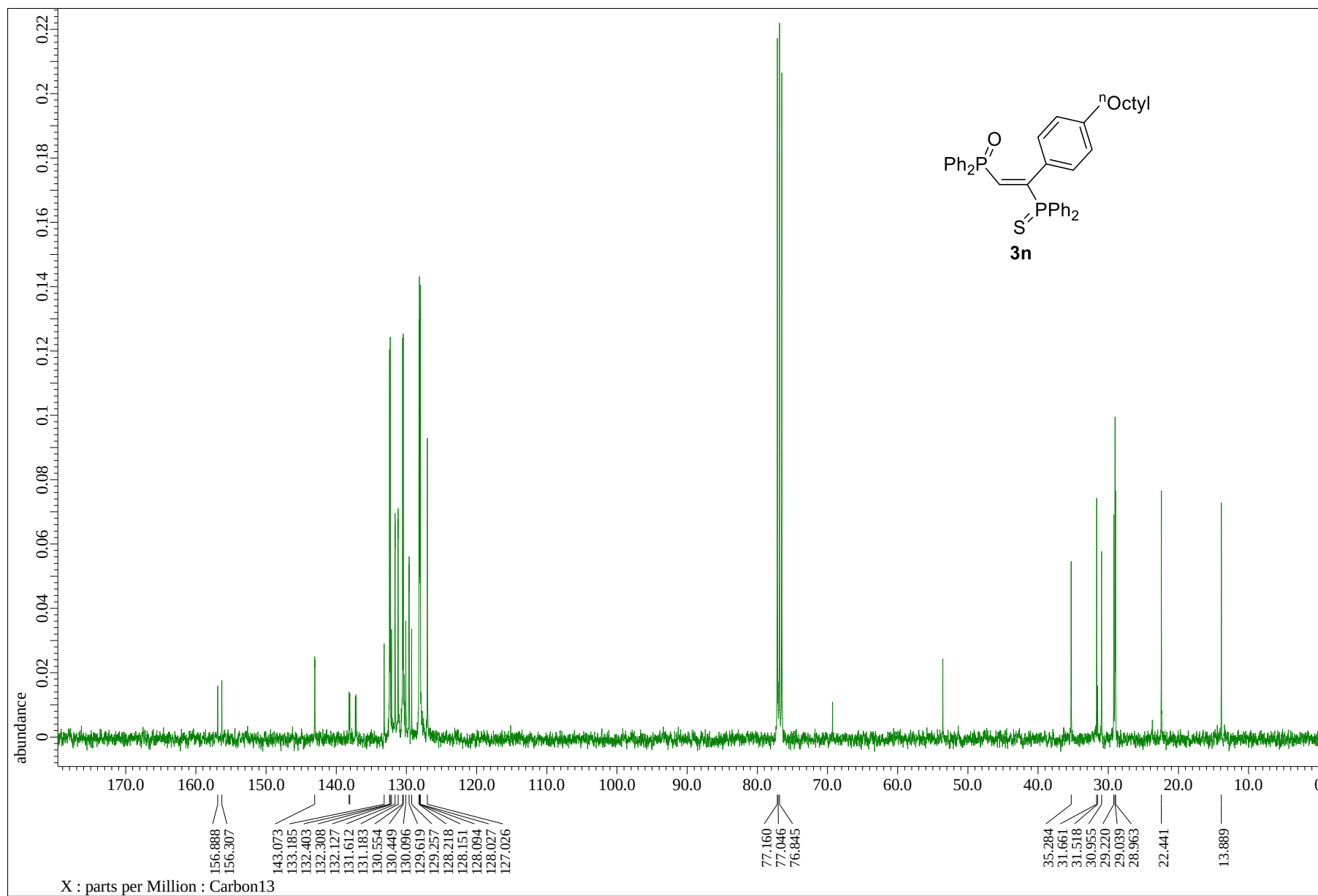


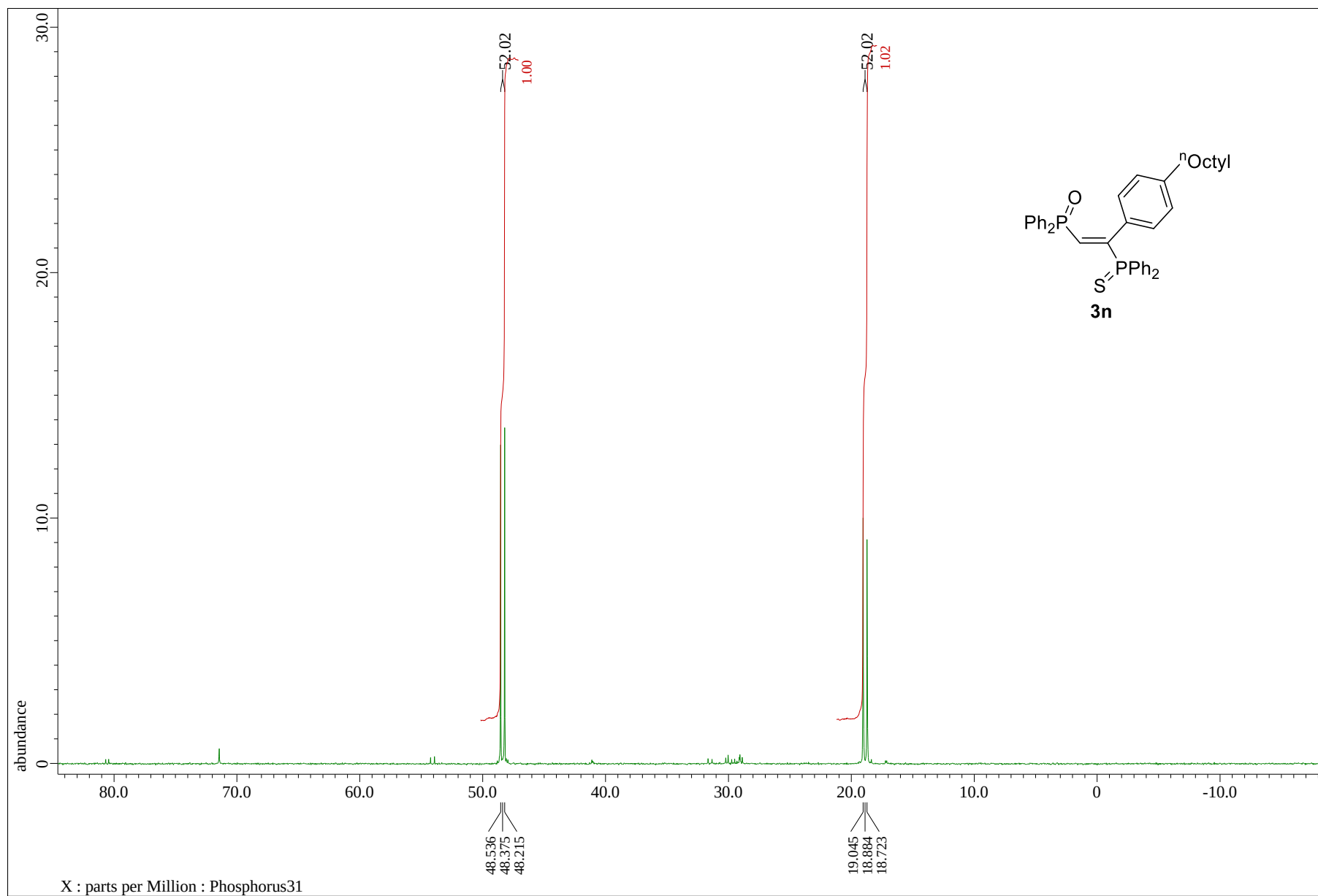


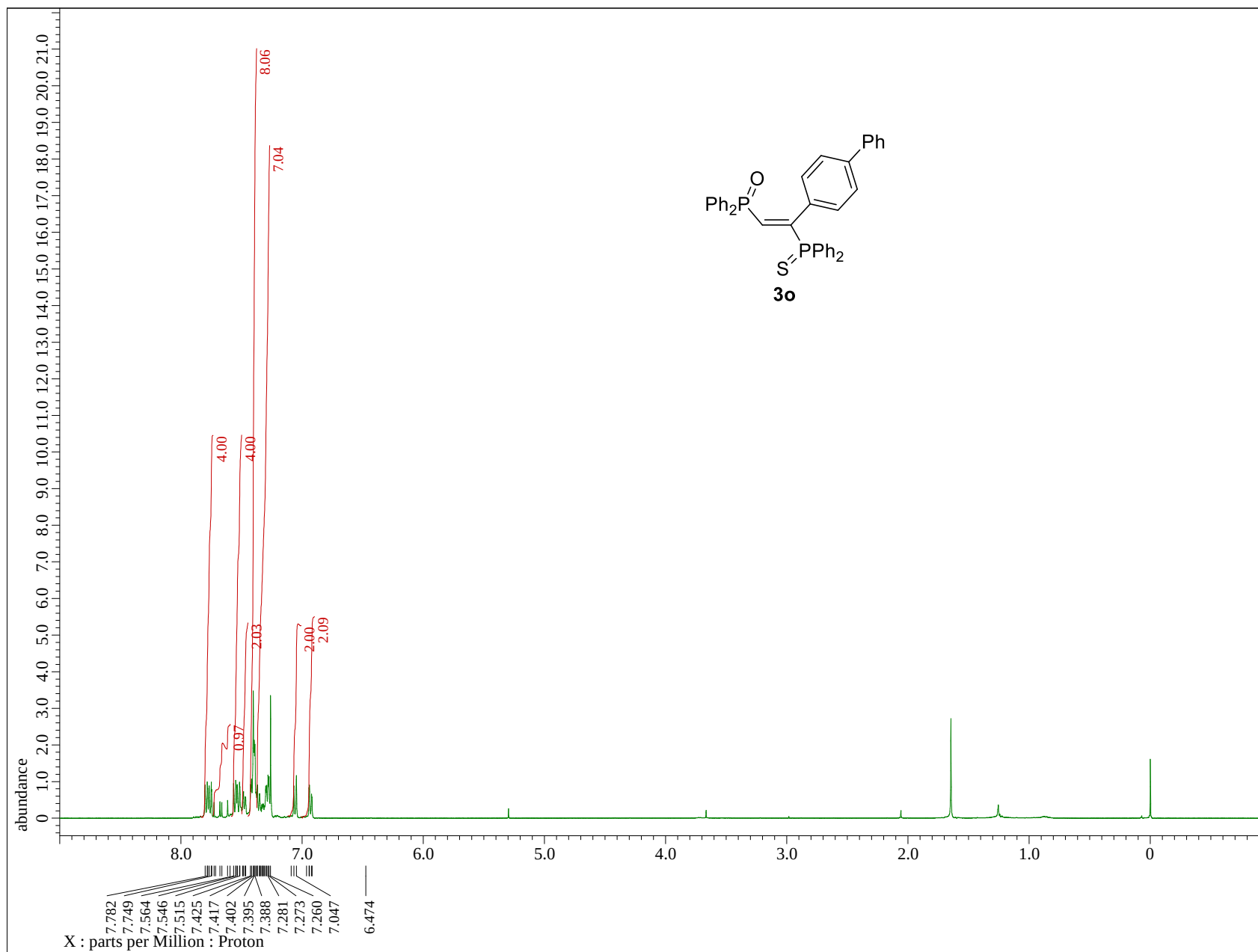


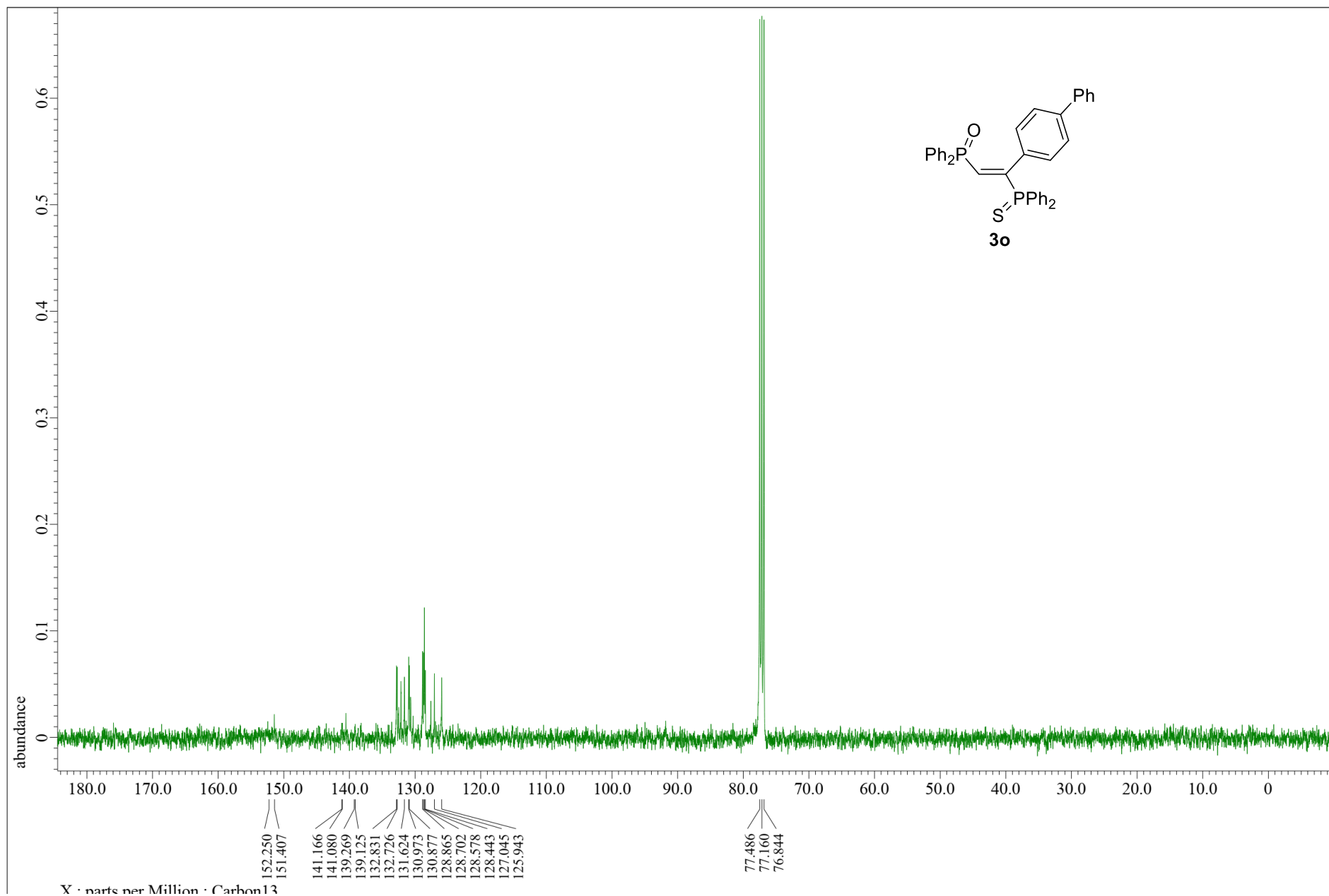












"D 1006 BiPh" 1 1 D:\nmrdata\OGAWA\Dat

