Supporting Information for

Manganese(III)-Mediated Direct Phosphonation of Arylalkenes and Arylakynes

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General information

All reactions were carried out under air. Solvents were dried by the standard procedures. ^{1}H and ^{13}C NMR spectra were determined in $CDCl_3$ on a Varian-Inova 300MHz or 400 MHz spectrometer and chemical shifts were reported in ppm from internal TMS(δ). High resolution mass spectra were recorded on a Finnigan MAT 95 mass spectrometer (ESI) or a MicroMass-TOF machine (EI). Column chromatography was performed with 200-300 mesh silica gel using flash column techniques. All of the reagents were used directly as obtained commercially unless otherwise noted.

Manganese triacetate[1], α, β-unsaturated ketones[2], conjugated alkenes[3], and conjugated alkynes[4] were prepared according to the reported procedures.

1. De Klein, W. J. In Organic Syntheses by oxidation with metal compounds, Eds. W. J. Mijs and C. R. H. I. DE Jonge **1986** Plenum Press, New York.
2. Fokas, D.; Ryan, W. J.; Casebier, D. S.; Coffen, D. L. *Tetrahedron Lett*. **1998**, *39*, 2235.
3. (a) Villieras, J.; Rambaud, M. *Synthesis*, **1983**, *4*, 300; (b) Sawaki, Y.; Ogata, Y. *Bull. Chem. Soc. Jpn*. **1981**, *54*, 793; (c) Bowman, R. K.; Johnson, J. S. *J. Org. Chem.* **2004**, *69*, 8537; (d) Dao, T. T.; Chi, Y. S.; Kim, J.; Kim, H. P.; Kim, S.; Park, H. *Bioorg. Med. Chem. Lett.* **2004**, *14*, 1165.
4. Luzung, M. R.; Toste, F. D. *J. Am. Chem. Soc*. **2003**, *125*, 15760.

General procedure for phosphonation of α,β-unsaturated ketones

To a stirred mixture of chalcone (1 mmol), diethylphosphite (2 mmol) in acetic acid (5 mL) was added $Mn(OAc)_3•2H_2O$ (0.81g, 3 mmol) in three portions and the resulting solution was heated at 60 ^oC for 1h. Then it was quenched with water (30 mL) and extracted with CH_2Cl_2 (10 mL×3). The combined organic layers were washed with Na_2CO_3 and brine (10 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography to afford the desired compound.

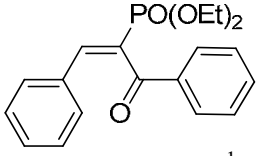
General procedure for phosphonation of conjugated alkenes

To a stirred mixture of conjugated alkene (1 mmol), diethylphosphite (2 mmol) in acetic acid (5 mL) was added $Mn(OAc)_3•2H_2O$ (0.81g, 3 mmol) in three portions and the resulting solution was heated at 60 ^oC for 0.5h. Then it was quenched with water (30 mL) and extracted with CH_2Cl_2 (10 mL×3). The combined organic layers were washed with Na_2CO_3 and brine (10 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography to afford the desired compound.

General procedure for phosphonation of conjugated alkynes

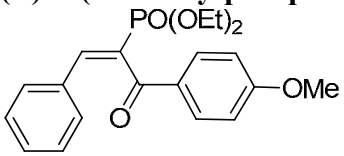
To a stirred mixture of conjugated alkyne (1 mmol), diethylphosphite (2 mmol) in acetic acid (5 mL) was added $Mn(OAc)_3•2H_2O$ (0.81g, 3 mmol) in three portions and the resulting solution was heated at 80 ^oC for 0.5h. Then it was quenched with water (30 mL) and extracted with CH_2Cl_2 (10 mL×3). The combined organic layers were washed with Na_2CO_3 and brine (10 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo. The residue was purified by column chromatography to afford the desired compound.

(*E*)-2-(Diethoxy-phosphoryl)-1,3-diphenyl-propenone



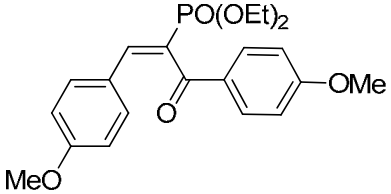
Light yellow oil, ^1H NMR (400 MHz, $CDCl_3$): δ 7.87-7.85 (d, 2H), 7.73 (d, $^3J_{P\text{-}H}$ = 25.7 Hz, 1H), 7.40-7.09 (m, 8H), 4.06 (m, 4H), 1.16 (t, $^3J_{H\text{-}H}$ = 5.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.5 (d, $^2J_{C\text{-}P}$ = 8.2 Hz), 141.0 (d, ^3J = 5.8 Hz), 130.3 (d, ^3J = 2.3 Hz), 128.7, 128.3 (d, ^2J = 21.4 Hz), 124.9, 124.6, 124.4, 123.5, 123.4, 57.5 (d, ^2J = 5.7 Hz), 10.9 (d, ^3J = 6.7 Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{19}H_{21}O_4P$ 344.1177, found 344.1162(M^+, 76.64).

(*E*)-2-(Diethoxy-phosphoryl)-1-(4-methoxyphenyl)-3-phenyl-propenone



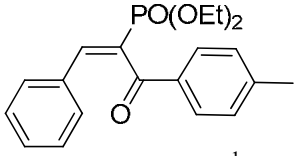
Light yellow oil, ^1H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, 2H), 7.73 (d, $^3J_{P\text{-}H}$ = 26.1 Hz, 1H), 7.28 (d, 2H), 7.19 (m, 3H), 6.81 (d, 2H), 4.12 (m, 4H), 3.78 (s, 3H), 1.24 (t, $^3J_{H\text{-}H}$ = 6.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 94.4 (d, ^2J = 8.8 Hz), 164.6, 146.1 (d, ^2J = 5.8 Hz), 134.1 (d, ^3J = 21.7 Hz), 132.5, 131.5 (d, ^1J = 171.5 Hz), 130.5, 130.2, 129.2 (d, ^3J = 2.3 Hz), 129.1, 114.3, 63.1 (d, ^2J = 5.7 Hz), 55.9, 16.5 (d, ^3J = 6.7 Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{20}H_{23}O_5P$ 374.1283, found 374.1281 (M^+, 39.13).

(*E*)-2-(Diethoxy-phosphoryl)-1,3-di(4-methoxyphenyl)-propenone



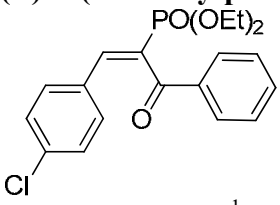
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, 2H), 7.65 (d, $^3J_{P\text{-}H}$ = 25.5 Hz, 1H), 7.24 (d, 2H), 6.80 (d, 2H), 6.66 (d, 2H), 4.09 (m, 4H), 3.77 (s, 3H), 3.67 (s, 3H), 1.21 (t, $^3J_{H\text{-}H}$=6.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.9 (d, ^2J = 6.7 Hz), 164.5, 161.4, 145.8 (d, ^2J = 5.0 Hz), 132.5, 132.2, 131.2 (d, ^1J = 150.2 Hz), 129.3, 126.7 (d, ^3J = 21.8 Hz), 114.5, 114.3, 63.0 (d, ^2J = 4.3 Hz), 55.8, 55.6, 16.6 (d, ^3J = 6.0 Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{21}H_{25}O_6P$ 404.1389, found 404.1396 (M^+, 30.72).

(*E*)-2-(Diethoxy-phosphoryl)-1-(*p*-tolyl)-3-phenyl-propenone



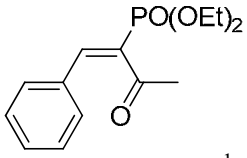
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, 2H), 7.74 (d, $^3J_{P\text{-}H}$ = 31.1 Hz, 1H), 7.30 (d, 2H), 7.17 (m, 5H), 4.14 (m, 4H), 2.34 (s, 3H), 1.25 (t, $^3J_{H\text{-}H}$ = 5.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.6 (d, ^2J = 7.5 Hz), 146.3 (d, ^2J = 4.1 Hz), 145.3, 133.8(d, ^3J = 21.3 Hz), 133.4, 130.4, 130.1, 130.0, 129.7, 129.0, 128.6 (d, ^3J = 28.2 Hz), 63.1, 22.1, 16.4 (d, ^3J = 5.2 Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{20}H_{23}O_4P$ 358.1334, found 358.1342(M^+, 48.92).

(*E*)-2-(Diethoxy-phosphoryl)-1-phenyl-3-(4-chlorophenyl)-propenone



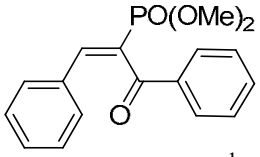
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, 2H), 7.73 (d, $^3J_{P\text{-}H}$ = 25.7 Hz, 1H), 7.30 (d, 2H), 7.51 (t, 1H), 7.37 (t, 2H), 7.22 (d, 2H), 7.15 (d, 2H), 4.14 (m, 4H), 1.24 (t, $^3J_{H\text{-}H}$=7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.0 (d, ^2J = 7.8 Hz), 145.1 (d, ^2J =5.0 Hz), 136.7, 135.8, 134.6, 132.4 (d, ^3J = 21.9 Hz), 131.4, 130.0, 129.4, 129.2, 63.3 (d, ^2J = 5.0 Hz), 16.5 (d, ^3J = 6.1 Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{19}H_{20}ClO_4P$ 378.0788, found 378.0780(M^+, 99.52).

(*E*)-3-(Diethoxy-phosphoryl)-4-phenyl-but-3-en-2-one



Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, $^3J_{P\text{-}H}$ = 25.7 Hz, 1H), 7.36 (m, 5H), 4.15(m, 4H), 2.24 (s, 3H), 1.34 (t, $^3J_{H\text{-}H}$ = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 203.5 (d, ^2J = 8.7 Hz), 145.3 (d, ^2J = 5.8 Hz), 133.9 (d, ^1J = 170.7 Hz), 133.8 (d, ^3J = 21.5 Hz), 130.7, 129.5, 129.2, 63.0 (d, ^2J = 5.5 Hz), 31.4, 16.5 (d, ^3J = 6.6 Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{14}H_{19}O_4P$ 282.1021, found 282.1017(M^+, 33.62).

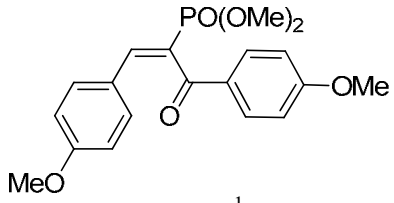
(*E*)-2-(Dimethoxy-phosphoryl)-1,3-diphenyl-propenone



Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, 2H), 7.85 (d, $^3J_{P\text{-}H}$ = 26.0 Hz, 1H), 7.53-7.18 (m, 8H), 3.79 (d, $^3J_{P\text{-}H}$ = 11.3 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.9 (d, ^2J = 8.3 Hz), 135.7 (d,

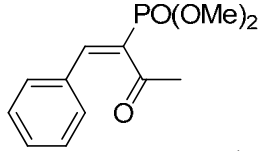
^2J = 2.7 Hz), 134.5, 133.7 (d, ^3J = 21.7 Hz), 130.8, 130.3, 130.0, 129.1, 129.8 (d, ^1J = 172.8 Hz), 53.6 (d, ^2J = 5.7 Hz). HRMS (M^+): *m/z* (%), calcd for $C_{17}H_{17}O_4P$ 316.0864 , found 316.0852 (M^+, 65.69).

(*E*)-2-(Dimethoxy-phosphoryl)-1,3-di(4-methoxyphenyl)-propenone



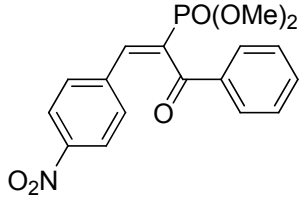
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, 2H), 7.72 (d, $^3J_{P\text{-}H}$ = 33.5 Hz, 1H), 7.27 (d, 2H), 6.85 (d, 2H), 6.71 (d, 2H), 3.83 (s, 3H), 3.74 (s, 3H), 3.76 (d, $^3J_{P\text{-}H}$ = 11.3 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 94.6 (d, ^2J = 8.2 Hz), 164.6, 161.5, 146.6 (d, ^2J = 6.1 Hz), 132.4, 128.3 (d, ^2J = 136.6 Hz), 126.4 (d, ^3J = 22.4 Hz), 125.9, 114.4, 114.3, 55.8, 55.6, 53.4 (d, ^2J = 5.6 Hz). HRMS (M^+): *m/z* (%), calcd for $C_{19}H_{21}O_6P$ 376.1076, found 376.1094(M^+, 68.60).

(*E*)-3-(Diethoxy-phosphoryl)-4-phenyl-but-3-en-2-one



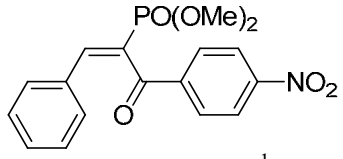
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, $^3J_{P\text{-}H}$ = 25.7 Hz, 1H), 7.37 (m, 5H), 3.81 (d, $^3J_{P\text{-}H}$ = 11.4 Hz, 6H), 2.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 203.5 (d, ^2J = 8.7 Hz), 146.3 (d, ^2J = 5.8 Hz), 132.7 (d, ^1J = 171.6 Hz), 133.7 (d, ^3J = 21.5 Hz), 130.9, 129.6, 129.3, 53.4 (d, ^2J = 5.6 Hz), 31.4. HRMS (M^+): *m/z* (%), calcd for $C_{12}H_{15}O_4P$ 254.0708, found 254.0715(M^+, 83.70).

(*E*)-2-(Diethoxy-phosphoryl)-1-(4-nitrophenyl)-3-phenyl-propenone



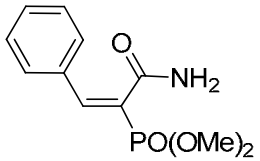
Light yellow solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, 2H), 7.82 (d, 2H), 7.78 (d, $^3J_{P\text{-}H}$ = 24.7 Hz, 1H), 7.39 (m, 5H), 3.73 (d, $^3J_{P\text{-}H}$ = 11.3 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.5, 144.11 (d, J = 5.9Hz), 139.9, 139.7, 135.9, 135.6, 134.9, 134.2, 130.6, 129.9, 129.3, 129.1, 124.2, 53.8(J = 5.9), HRMS (M^+): *m/z* (%), calcd for $C_{17}H_{16}NO_6P$ 361.0715, found 361.0715 (M^+, 29.69).

(*E*)-2-(Diethoxy-phosphoryl)-1-phenyl-3-(4-nitrophenyl)-propenone



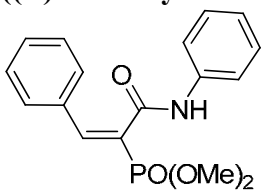
Light yellow solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (d, 2H), 7.97 (d, 2H), 7.86 (d, $^3J_{P\text{-}H}$ = 25.3Hz, 1H), 7.14 (m, 5H), 3.73 (d, $^3J_{P\text{-}H}$=11.4 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.9, 148.7 (d, J = 5.8Hz), 140.1, 133.3, 131.2, 130.8, 130.0, 129.3, 124.2, 53.7 (d, J = 5.9Hz). HRMS (M^+): *m/z* (%), calcd for $C_{17}H_{16}NO_6P$ 361.0715, found 361.0715 (M^+, 29.69).

((*E*)-1-Carbamoyl-2-phenylvinyl)-phosphonic acid dimethyl ester



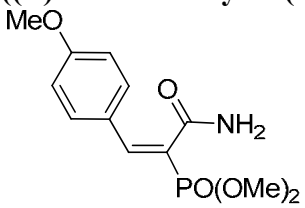
White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, $^3J_{P\text{-}H}$ = 25.3Hz, 1H), 7.58-7.35 (m, 5H), 6.11 (d, $^4J_{P\text{-}H}$ = 23.9Hz, 2H), 3.83 (d, $^3J_{P\text{-}H}$ = 11.3Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.1 (d, ^2J = 11.7 Hz), 147.1 (d, ^2J = 6.2 Hz), 133.4 (d, ^3J = 21.0 Hz), 130.8, 129.9, 129.0, 125.6 (d, ^1J = 177.3 Hz), 53.6 (d, ^2J = 5.6 Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{11}H_{14}NO_4P$ 255.0660, found 255.0660(M^+, 27.02).

((*E*)-1-Phenylcarbamoyl-2-phenylvinyl)-phosphonic acid dimethyl ester



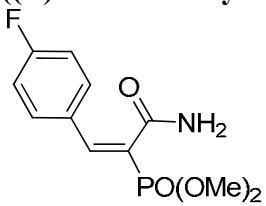
White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (s, 1H), 7.57-7.10 (m, 11H), 3.80 (d, $^3J_{P\text{-}H}$ = 11.3Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.0 (d, ^2J = 12.0Hz), 147.4 (d, ^2J = 6.9Hz), 138.1, 133.5 (d, ^3J = 21.0Hz), 131.1, 130.1, 129.4, 129.3, 126.29 (d, ^1J = 178.1Hz), 125.1, 120.4, 53.78 (d, ^2J = 5.9Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{17}H_{18}NO_4P$ 331.0973, found 331.0972(M^+, 15.32).

((*E*)-1-Carbamoyl-2-(4-methoxyphenyl)-vinyl)-phosphonic acid dimethyl ester



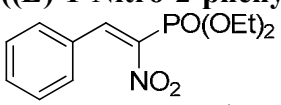
White solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56 (d, $J_{H\text{-}H}$ = 8.4Hz, 2H), 7.45 (d, $^3J_{P\text{-}H}$ =25.1Hz, 1H), 6.86 (d, $J_{H\text{-}H}$ = 8.2Hz, 2H), 6.09 (d, $^4J_{P\text{-}H}$ = 54.1Hz, 2H), 3.82 (d, $^3J_{P\text{-}H}$ =11.4Hz, 6H), 3.81 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 161.8, 147.44 (d, ^2J = 6.4Hz), 133.7, 132.2, 125.9 (d, ^3J =21.3Hz), 114.4, 113.8, 55.6, 53.5 (d, ^2J = 5.1Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{12}H_{16}NO_5P$ 285.0766, found 285.0767(M^+, 38.92).

((*E*)-1-Carbamoyl-2-(4-fluorophenyl)-vinyl)-phosphonic acid dimethyl ester



White solid , ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (dd, J_1 = 5.4Hz, J_2 = 8.6Hz, 2H), 7.47 (d, $^3J_{P\text{-}H}$ = 25.2Hz, 1H), 7.05 (t, J = 8.6Hz, 2H), 6.12 (d, $^4J_{P\text{-}H}$ = 55.8Hz, 2H), 3.83 (d, $^3J_{P\text{-}H}$ =11.3Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.74 (d, J =11.3Hz), 164.1 (d, 1H, J= 252.8Hz), 165.8, 162.4, 146.2 (d, J = 6.6Hz), 132.2 (d, J = 8.7Hz), 116.2 (d, J = 21.8Hz), 129.6 (dd, J = 3.4Hz, J = 21.5Hz), 125.0 (d, J = 177.3Hz), 53.62 (d, J = 5.8Hz). HRMS (M^+): *m*/*z* (%), calcd for $C_{11}H_{13}FNO_4P$ 273.0566, found 273.0567(M^+, 21.27).

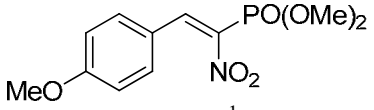
((*E*)-1-Nitro-2-phenylvinyl)-phosphonic acid diethyl ester



Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49 (m, 2H), 7.37 (m, 3H), 6.25 (t, $^3J_{P\text{-}H}$= 17.6 Hz, 1H), 4.13 (m, 4H), 1.34 (t, $^3J_{H\text{-}H}$ = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.2 (d, ^2J = 6.4 Hz),

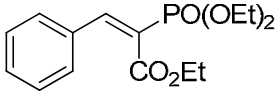
135.2 (d, ^3J = 23.2 Hz), 130.7, 129.5, 128.1, 114.5 (d, ^1J = 191.4 Hz), 62.5 (d, ^2J = 5.1 Hz), 16.8 (d, ^3J =6.6 Hz). HRMS (M^+): *m/z* (%), calcd for $C_{12}H_{16}NO_5P$ 284.0773, found 284.0713(M^+, 42.21).

((*E*)-1-Nitro-2-(4-methoxy-phenyl)-vinyl)-phosphonic acid dimethyl ester



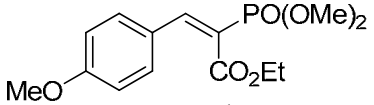
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41(d, 2H), 6.86(d, 2H), 6.01(t, $^3J_{P\text{-}H}$ = 17.7 Hz, 1H), 3.79(s, 3H), 3.72(d, $^3J_{P\text{-}H}$ = 11.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.8, 149.8(d, ^2J = 7.0 Hz), 129.8, 127.7, 114.7, 109.5(d, ^1J = 193.6 Hz), 55.8, 52.8(d, ^2J = 5.5 Hz). HRMS (M^+): *m/z* (%), calcd for $C_{11}H_{14}NO_6P$ 287.2057, found $C_{11}H_{15}O_4P$ 242.0664 (M^+, 86.39).

(*E*)-2-(Diethoxy-phosphoryl)-3-phenyl-acrylic acid ethyl ester



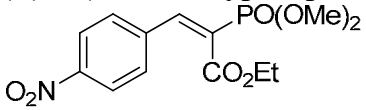
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7. 55 (d, $^3J_{P\text{-}H}$ = 24.2 Hz, 1H), 7.33-7.26 (m, 5H), 4.17 (m, 2H), 4.09 (m, 4H), 1.26 (m, 6H), 1.14 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.6 (d, ^2J= 12.7 Hz), 148.4 (d, ^2J= 6.4 Hz), 133.7 (d, ^3J= 20.1 Hz), 130.7, 129.4, 128.8, 124.5 (d, ^1J = 179.0 Hz), 62.9 (d, ^2J= 5.1 Hz), 61.9, 16.4 (d, ^3J= 6.7 Hz), 14.1. HRMS (M^+): *m/z* (%), calcd for $C_{15}H_{21}O_5P$ 312.1127, found 312.1129(M^+, 34.51).

(*E*)-2-(Dimethoxyphosphoryl)-3-(4-methoxyphenyl)-acrylic acid ethyl ester



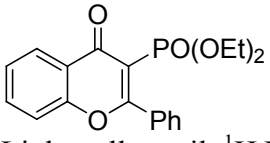
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, $^3J_{P\text{-}H}$ = 24.5 Hz, 1H), 7.37 (d, 2H), 6.84 (d, 2H), 4.26 (m, 2H), 3.78 (s, 3H), 3.77 (d, $^3J_{P\text{-}H}$ = 11.8 Hz, 6H), 1.24 (t, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.0, 149.2 (d, ^2J= 6.9 Hz), 133.4, 131.9, 126.3 (d, ^2J= 20.2 Hz), 119.9 (d, ^1J= 181.7 Hz), 114.5, 62.1, 55.8, 53.4 (d, ^2J= 5.2 Hz), 14.3. HRMS (M^+): *m/z* (%), calcd for $C_{14}H_{19}O_6P$ 314.0919, found 314.0936 (M^+, 57.65).

(*E*)-2-(Dimethoxyphosphoryl)-3-(4-nitrophenyl)-acrylic acid ethyl ester



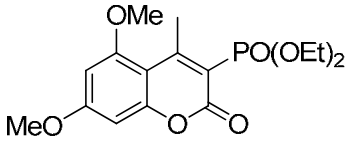
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.29 (d, 2H), 7.78 (d, $^3J_{P\text{-}H}$ = 23.9 Hz, 1H), 7.62 (d, 2H), 4.32 (q, 2H), 3.91 (d, $^3J_{P\text{-}H}$ = 11.4 Hz, 6H), 1.34 (t, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.1, 166.1 (J = 5.6Hz), 165.7 (J =12.0Hz), 148.9, 146.8 (J = 6.4Hz), 130.3, 124.3, 62.2, 53.8 (J = 6.3Hz), 14.5, HRMS (M^+): *m/z* (%), calcd for $C_{13}H_{16}NO_7P$ 329.0664, found 329.0664 (M^+,24.65).

Diethyl-4-oxo-2-phenyl-4H-chromen-3-ylphosphonate



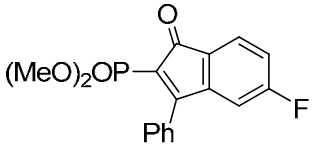
Light yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.18 (d, J = 7.7 Hz, 1H), 7.74 (d, J = 7.4 Hz, 2H), 7.68-7.64 (t, J = 7.7 Hz, 1H), 7.41-7.51 (m, 5H), 4.09-3.98 (m, 4H), 1.16 (t, J = 6.6 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 177.8, 147.8 ($^2J_{P\text{-}C}$ = 19.3 Hz), 128.9 ($^3J_{P\text{-}C}$ = 41.4 Hz), 155.9, 134.8, 134.3, 134.0, 131.6, 129.7, 128.4, 126.3, 123.5, 118.3, 62.9 ($^3J_{P\text{-}C}$ = 5.8 Hz) , 16.4 ($^3J_{P\text{-}C}$ = 6.5 Hz). HRMS (EI): calcd for $C_{19}H_{19}O_5P$ 358.0970 (M^+), found 358.0974.

Diethyl-5,7-dimethoxy-4-methyl-2-oxo-2H-chromen-3-ylphosphonate



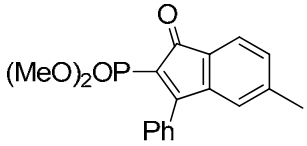
Yellow solid, mp 84-86℃, ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.40-6.39 (d, $^3J_{PH}$ = 2.3Hz, 1H), 4.26-4.18 (m, 4H), 3.88 (s, 3H), 3.85 (s, 3H), 3.04-3.03 (d, $^4J_{PH}$ = 2.4, 3H), 1.36 (t, J = 7.1, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.5-166.4 ($^2J_{P\text{-}C}$ = 11.3 Hz), 164.6, 160.6, 157.3, 111.6, 108.9, 105.7-105.5 ($^1J_{P\text{-}C}$ = 15.4 Hz), 96.1, 93.2, 63.5 (d, $^2J_{P\text{-}C}$ = 5.7Hz), 16.5 (d, $^3J_{P\text{-}C}$=6.1Hz). HRMS (EI): calcd for $C_{16}H_{21}O_7P$ 356.1025(M^+), found 356.1025.

Dimethyl-5-fluoro-1-oxo-3-phenyl-1*H*-inden-2-ylphosphonate



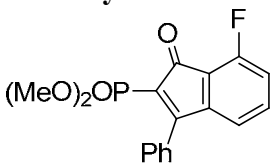
Yellow solid, Mp. 134-136 □; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.65 (d, $^3J_{PH}$ = 11.5 Hz, 6H), 6.8-6.89 (m, 1H), 7.04-7.09 (m, 1H), 7.54-7.63 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 53.0 (d, J = 5.9 Hz), 112.0 (d, J = 25.6 Hz), 117.2 (d, J = 23.2 Hz), 123.0 (d, J = 200.6 Hz), 125.5 (dd, J = 0.8 Hz, J =9.8 Hz), 126.9 (dd, J_1 = 3.0 Hz, J_2 = 10.6 Hz), 128.4 (d, J = 3.7 Hz), 128.7, 131.3 (d, J = 3.7 Hz), 147.3 (dd, J_1 = 9.2 Hz, J_2 = 19.5Hz), 164.7 (J = 2.5 Hz), 168.1 (J = 2.5 Hz), 169.5 (dd, J_1 = 2.1 Hz, J_2 = 11.5 Hz), 192.4 (d, J = 11.6 Hz); HRMS: calcd for $C_{17}H_{14}FO_4P$ 332.0614 [M^+], found 332.0618.

Dimethyl-5-methyl-1-oxo-3-phenyl-1*H*-inden-2-ylphosphonate



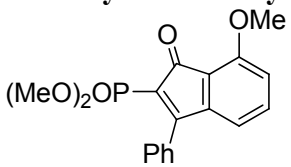
Yellow solid, Mp. 157-158 □; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.36 (s, 3H), 3.64 (d, $^3J_{PH}$ = 11.4 Hz, 6H), 6.94 (s, 1H), 7.21 (d, J = 7.4 Hz, 1H), 7.51 (d, J = 7.4 Hz, 1H), 7.53-7.61 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 22.2, 53.0 (d, $^2J_{P\text{-}C}$ = 5.3 Hz), 121.8 (d, $^1J_{P\text{-}C}$ = 199.7 Hz), 123.7, 124.9, 128.5, 128.5, 128.9 (d, $^3J_{P\text{-}C}$ = 10.0 Hz), 130.8, 131.6, 131.9 (d, $^3J_{P\text{-}C}$ = 2.5 Hz), 144.7 (d, $^3J_{P\text{-}C}$ = 18.9 Hz), 145.0, 171.8 (d, $^2J_{P\text{-}C}$ = 11.2 Hz), 194.0 (d, $^2J_{P\text{-}C}$ = 12.5 Hz); HRMS: calcd for $C_{18}H_{17}O_4P$ 328.0864 [M^+], found 328.0865.

Dimethyl-7-fluoro-1-oxo-3-phenyl-1*H*-inden-2-ylphosphonate



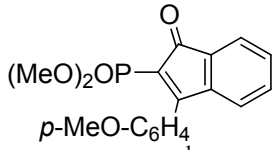
Yellow solid, Mp. 113-114 □; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.65 (d, $^3J_{PH}$ = 11.4 Hz, 6H), 6.96-7.69 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 53.2 (d, J = 6.0 Hz), 120.3 (d, J = 2.2Hz), 120.7 (d, J = 21.2 Hz), 122.6 (d, J = 201.4 Hz), 128.5 (d, J = 1.1 Hz), 128.6, 131.0, 131.6 (d, J = 3.5 Hz), 136.3 (d, J = 8.1 Hz), 146.4 (dd, J = 3.0 Hz, J = 19.6 Hz), 156.4, 159.9, 170.6 (dd, J = 4.3Hz, J = 11.4Hz), 190.5 (d, J = 12.8 Hz); HRMS: calcd for $C_{17}H_{14}FO_4P$ 332.0614 [M^+], found 332.0621.

Dimethyl-7-methoxy-1-oxo-3-phenyl-1*H*-inden-2-ylphosphonate



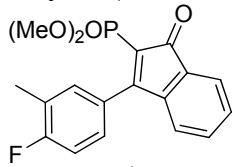
Yellow solid, Mp. 160-162 °C; ^{1}H NMR (400 MHz, CDCl$_3$): δ 3.65 (d, $^3J_{PH}$ = 11.4 Hz, 6H), 4.00 (s, 3H), 6.76 (d, J = 7.2 Hz, 1H), 7.00 (d, J = 8.4 Hz, 1H), 7.39 (dd, J =7.4, 8.4 Hz, 1H), 7.50-7.59 (m, 5H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 53.3 (d, $^2J_{P\text{-}C}$ = 6.0 Hz), 56.7, 116.7, 117.2, 121.8, 122.5 (d, $^1J_{P\text{-}C}$ = 200.4 Hz), 128.6, 128.8, 130.7, 132.2 (d, $^3J_{P\text{-}C}$ = 3.8 Hz), 136.1, 146.8 (d, $^3J_{P\text{-}C}$ = 19.4 Hz), 157.6, 169.8 (d, $^2J_{P\text{-}C}$ = 10.9 Hz), 192.6 (d, $^2J_{P\text{-}C}$ = 12.9 Hz); HRMS: calcd for $C_{18}H_{17}O_5P$ 344.0814 [M^+], found 344.0813.

Dimethyl-3-(4-methoxyphenyl)-1-oxo-1*H*-inden-2-ylphosphonate

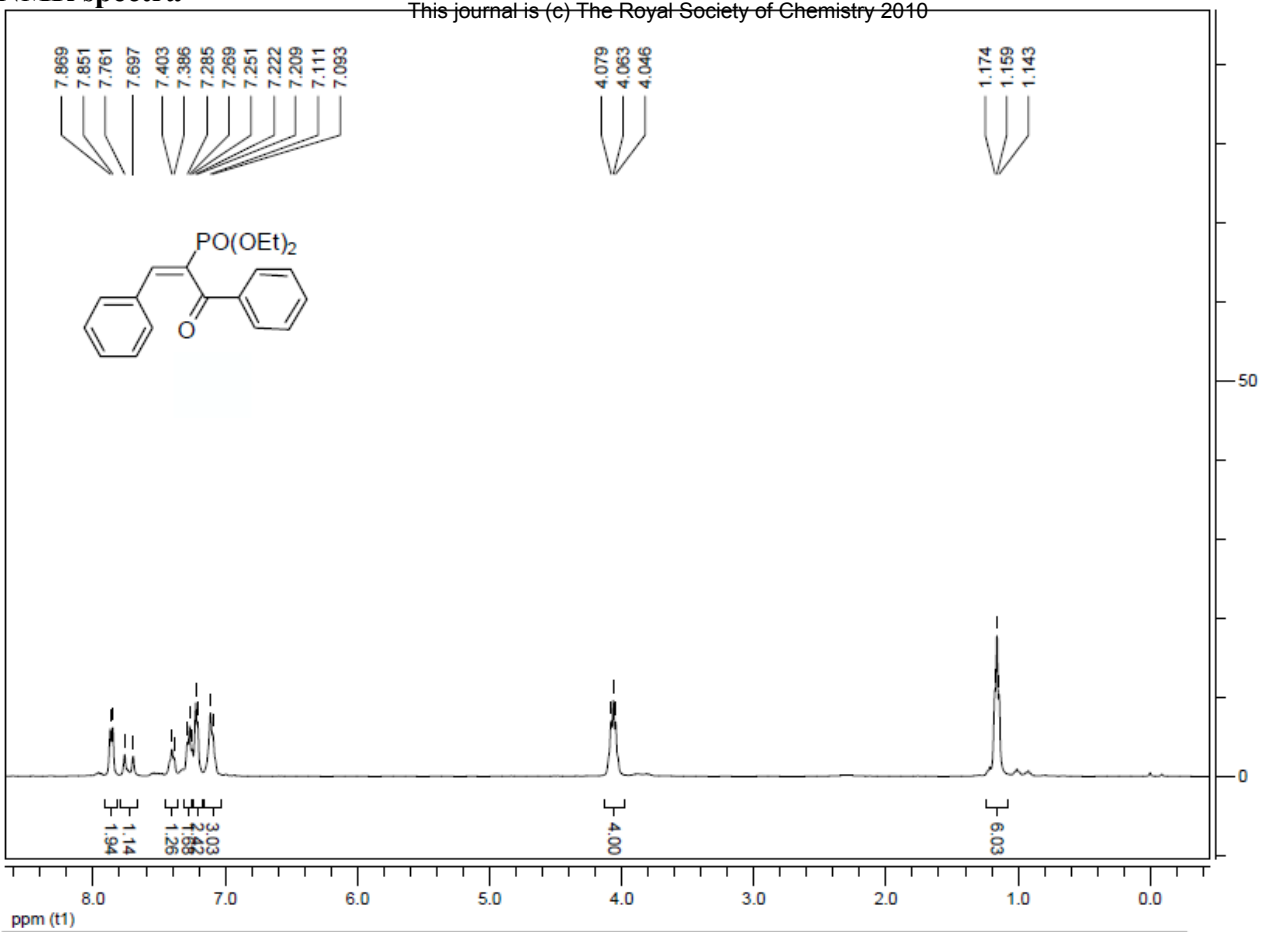


Yellow solid, ^{1}H NMR (300 MHz, CDCl$_3$): δ 3.69 (d, $^3J_{PH}$ =11.5 Hz, 6H), 3.90 (s, 3H, CH$_3$), 7.06 (d, J = 7.4 Hz, 2H), 7.66 (d, J = 7.4 Hz, 2H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 53.2 (d, $^2J_{P\text{-}C}$ = 5.9 Hz), 55.8, 114.1, 124.0, 128.5, 128.9, 131.1, 131.7, 131.9, 132.8, 133.8, 144.2 (d, $^2J_{P\text{-}C}$ = 19.0 Hz), 162.3, 172.2, 194.5 (d, $^2J_{P\text{-}C}$ = 12.4 Hz); HRMS: calcd for $C_{18}H_{17}O_5P$ 344.0814 [M^+], found 344.0815.

Dimethyl-3-(4-fluoro-3-methylphenyl)-1-oxo-1*H*-inden-2-ylphosphonate



Yellow solid, ^{1}H NMR (300 MHz, CDCl$_3$): δ 2.37 (s, 3H), 3.68 (d, $^3J_{PH}$ = 11.4 Hz, 6H), 7.16-7.68 (m, 2H), 7.43-7.62 (m, 5H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 15.1, 53.2 (d, J = 5.8 Hz), 115.6 (d, J = 23.0 Hz), 121.25 (d, J = 201.0 Hz), 123.9, 125.7 (d, J_1 = 17.9 Hz), 127.7, 128.5 (d, J_2 = 8.0 Hz), 131.4 (d, J = 10.4 Hz), 131.8, 132.2 (dd, J = 1.3 Hz, J = 5.9 Hz), 134.0 (d, J = 1.2 Hz), 144.3 (d, J = 18.9 Hz), 161.4, 164.8, 171.7 (d, J = 11.5 Hz), 194.3 (d, J = 11.4Hz); HRMS: calcd for $C_{18}H_{16}O_4FP$ 346.0770 [M^+], found 346.0768.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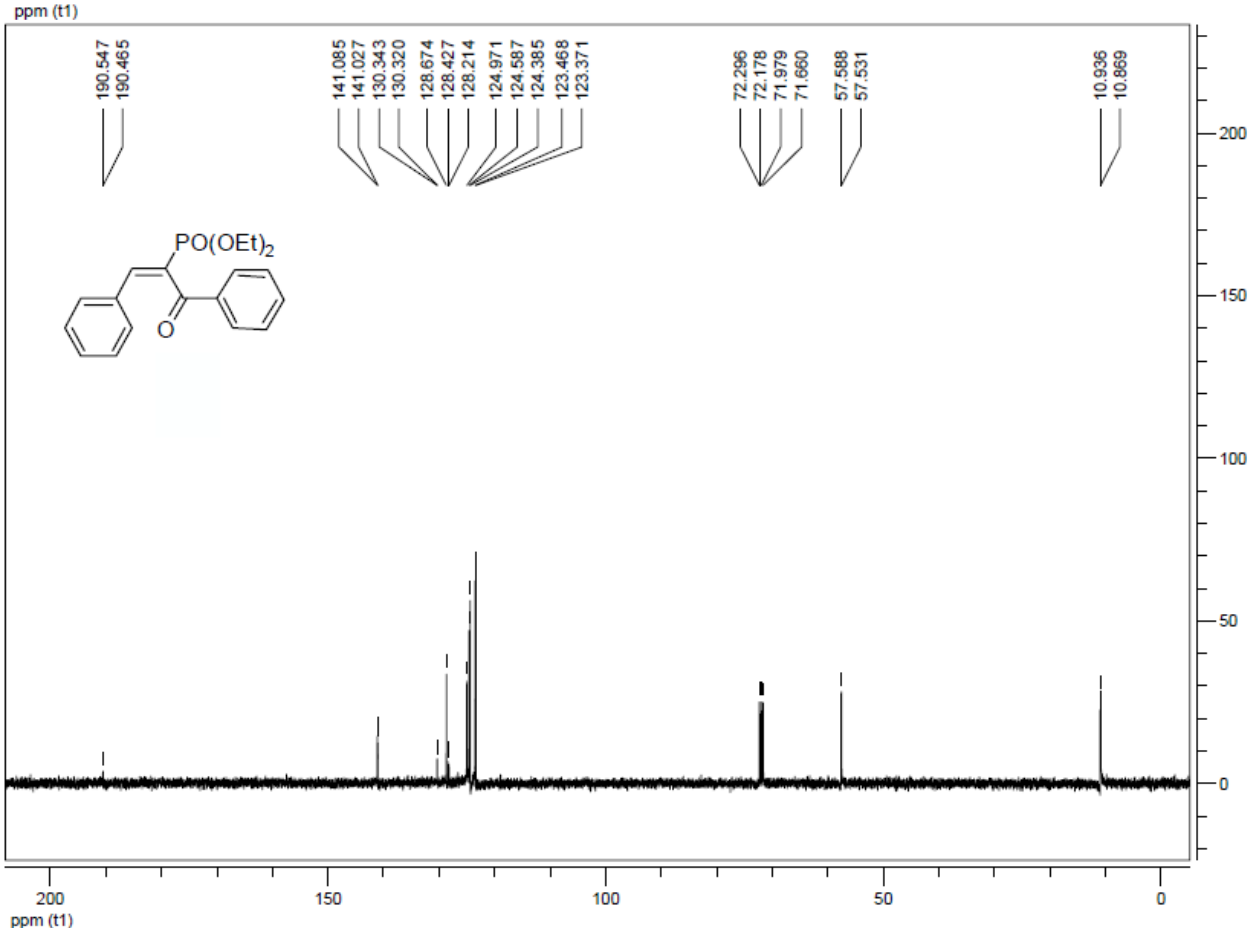
7.851
7.761
7.697
7.403
7.386
7.285
7.269
7.251
7.222
7.209
7.111
7.093
4.079
4.063
4.046
1.174
1.159
1.143
PO(OEt)2
O
1.94
1.14
1.26
3.03
4.00
6.03
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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130.320
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124.971
124.587
124.385
123.468
123.371
72.296
72.178
71.979
71.660
57.588
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O
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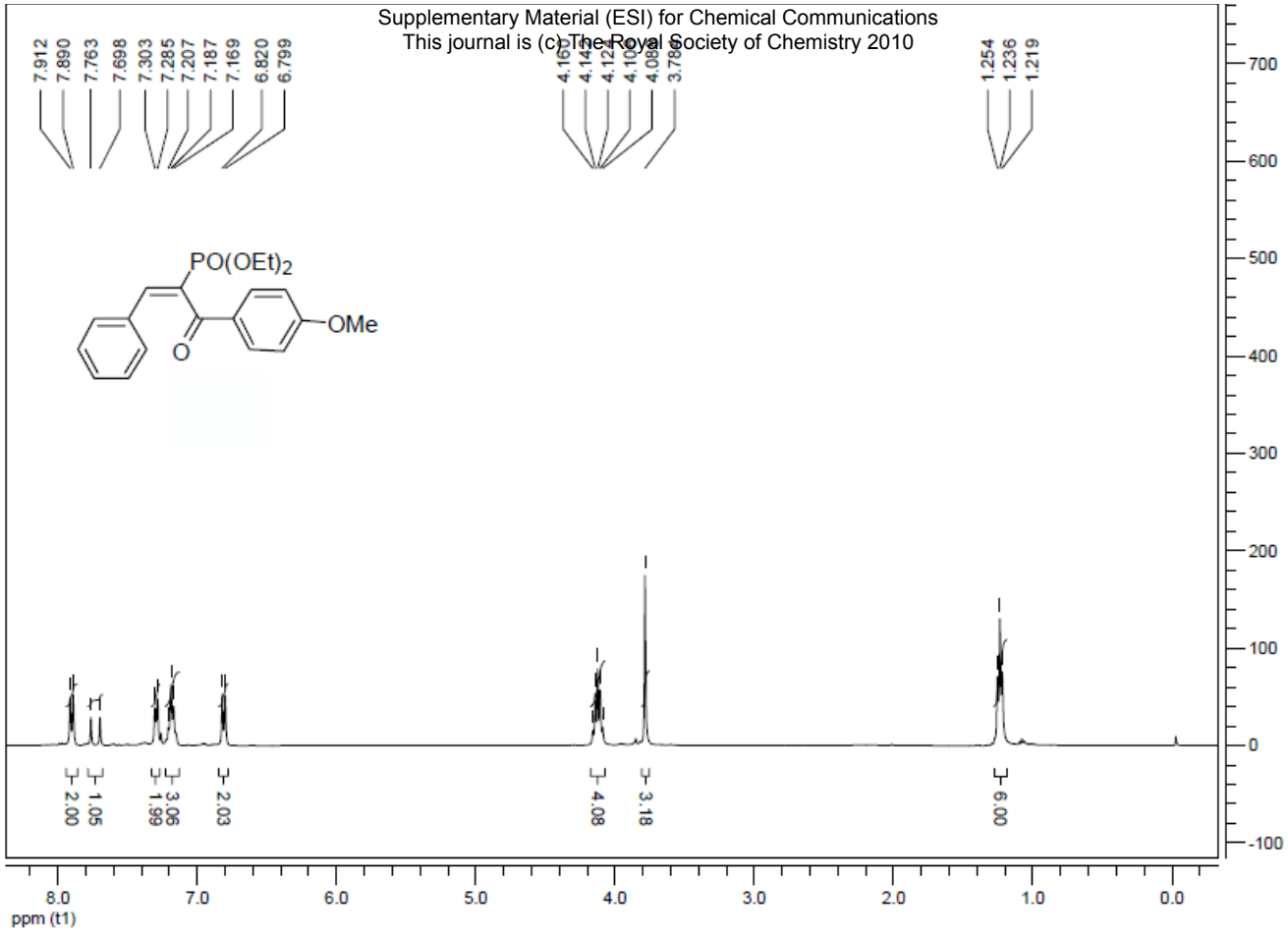
150
100
50
0
ppm (t1)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4.142
4.123
4.105
4.087
3.786
1.254
1.236
1.219
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OMe
O
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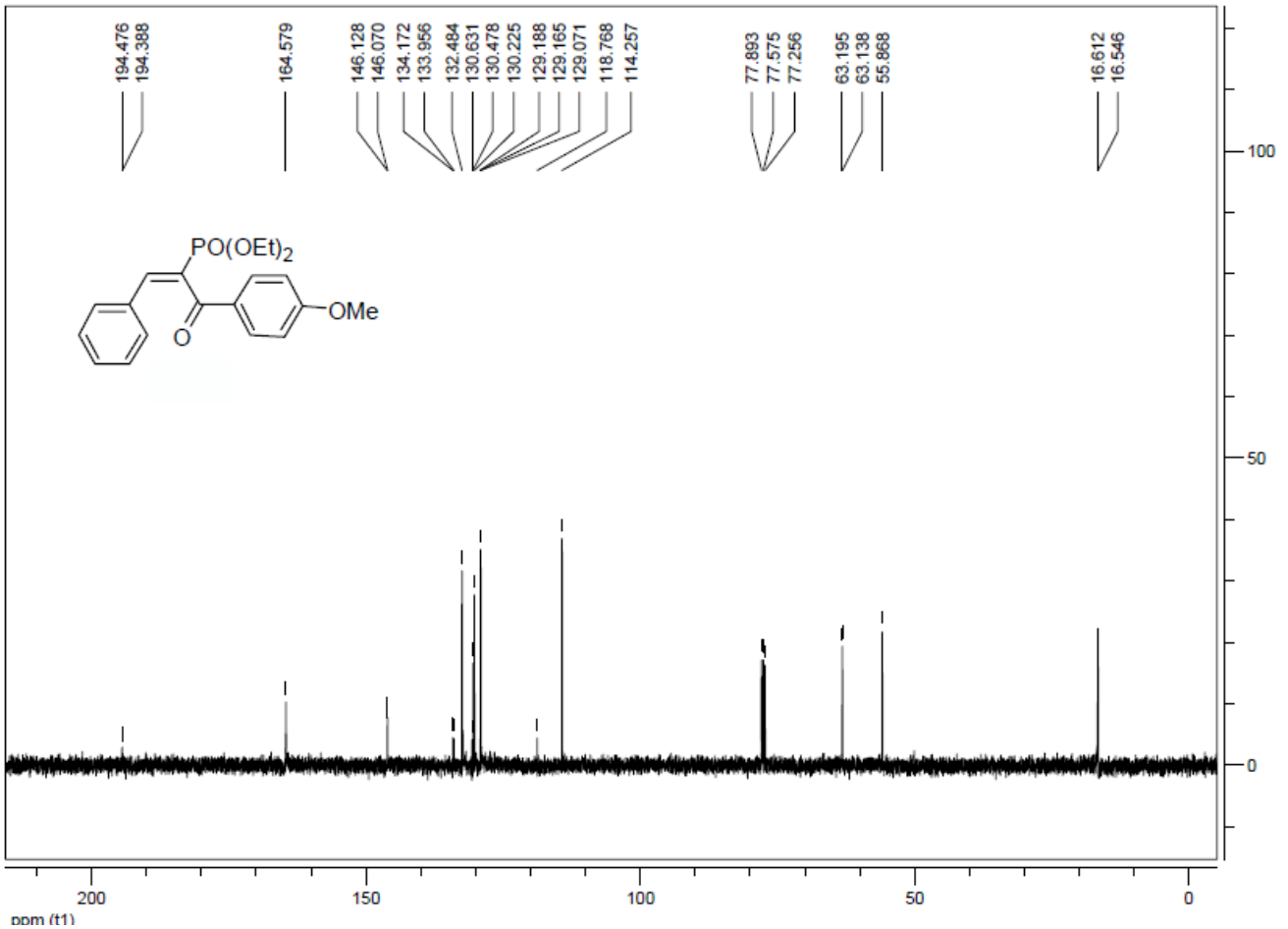
1.05
3.06
1.99
2.03
4.08
3.18
6.00
ppm (t1)


194.476
194.388
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129.165
129.071
118.768
114.257
77.893
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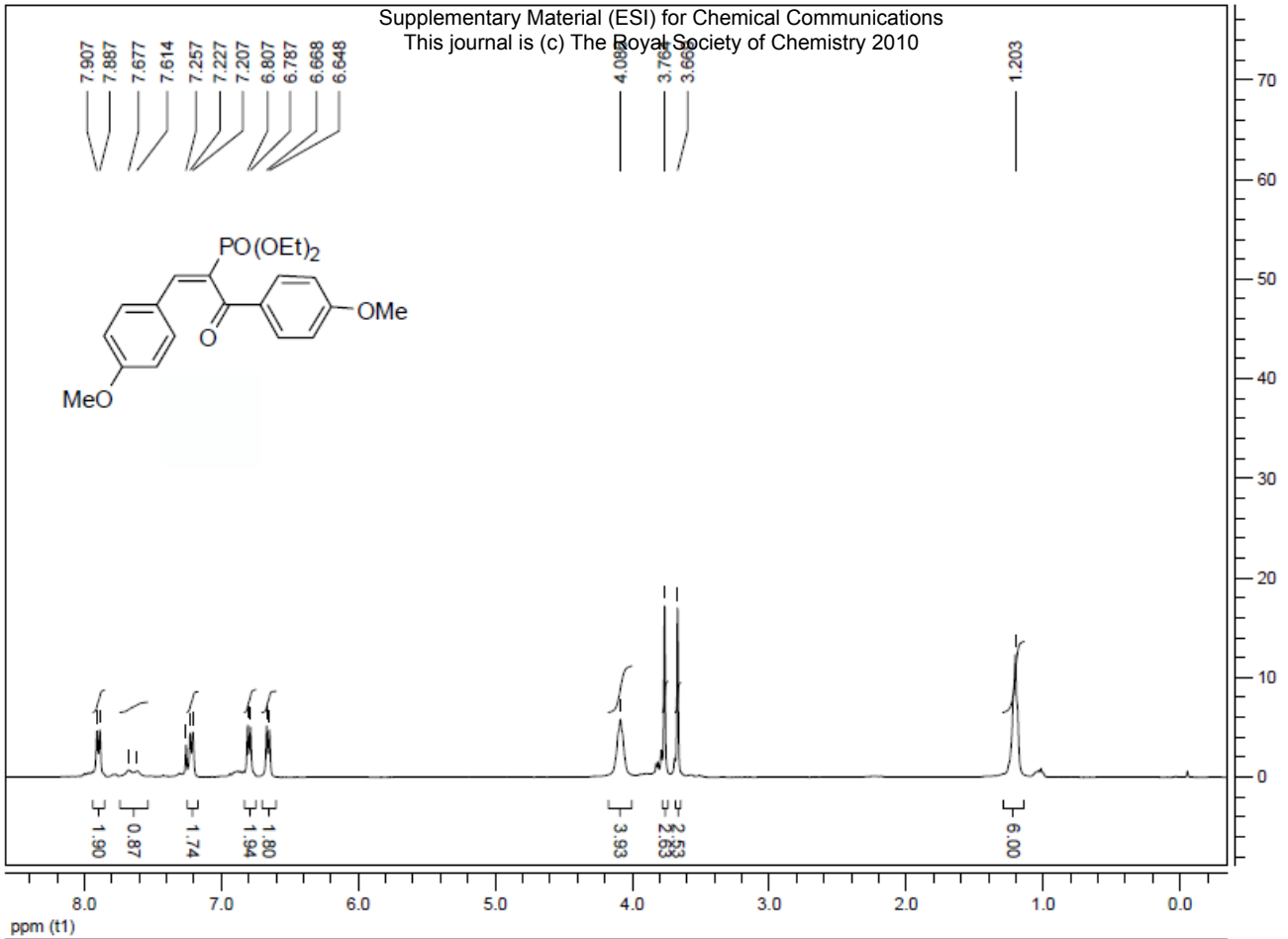
63.195
63.138
55.868
16.612
16.546
PO(OEt)2
OMe
O
ppm (t1)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7.207
6.807
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6.668
6.648
4.088
3.764
3.660
1.203
PO(OEt)2
OMe
O
MeO
1.90
0.87
1.74
1.94
1.80
3.93
2.93
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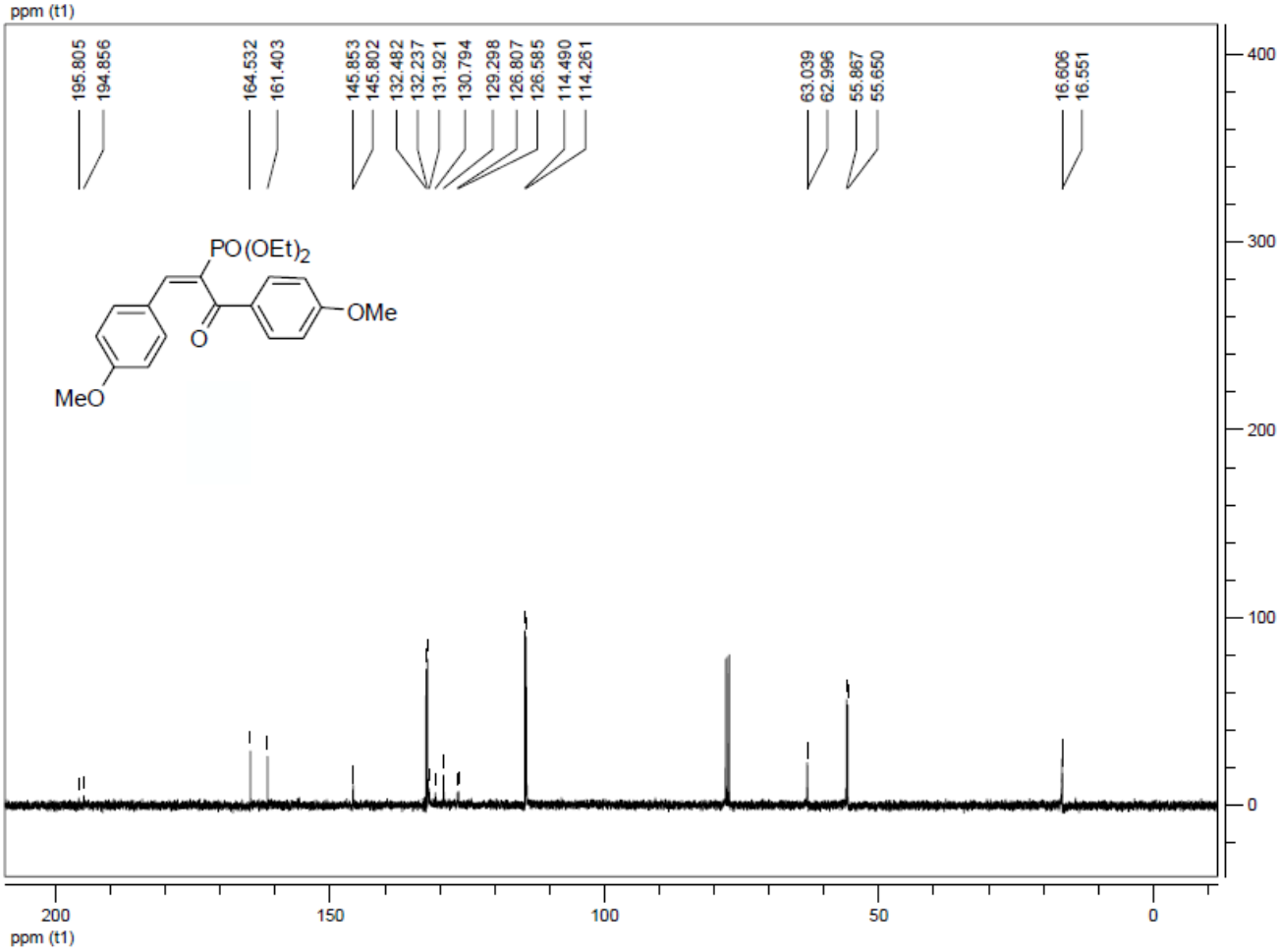
ppm (t1)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132.237
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114.490
114.261
63.039
62.996
55.867
55.650
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16.551
PO(OEt)2
OMe
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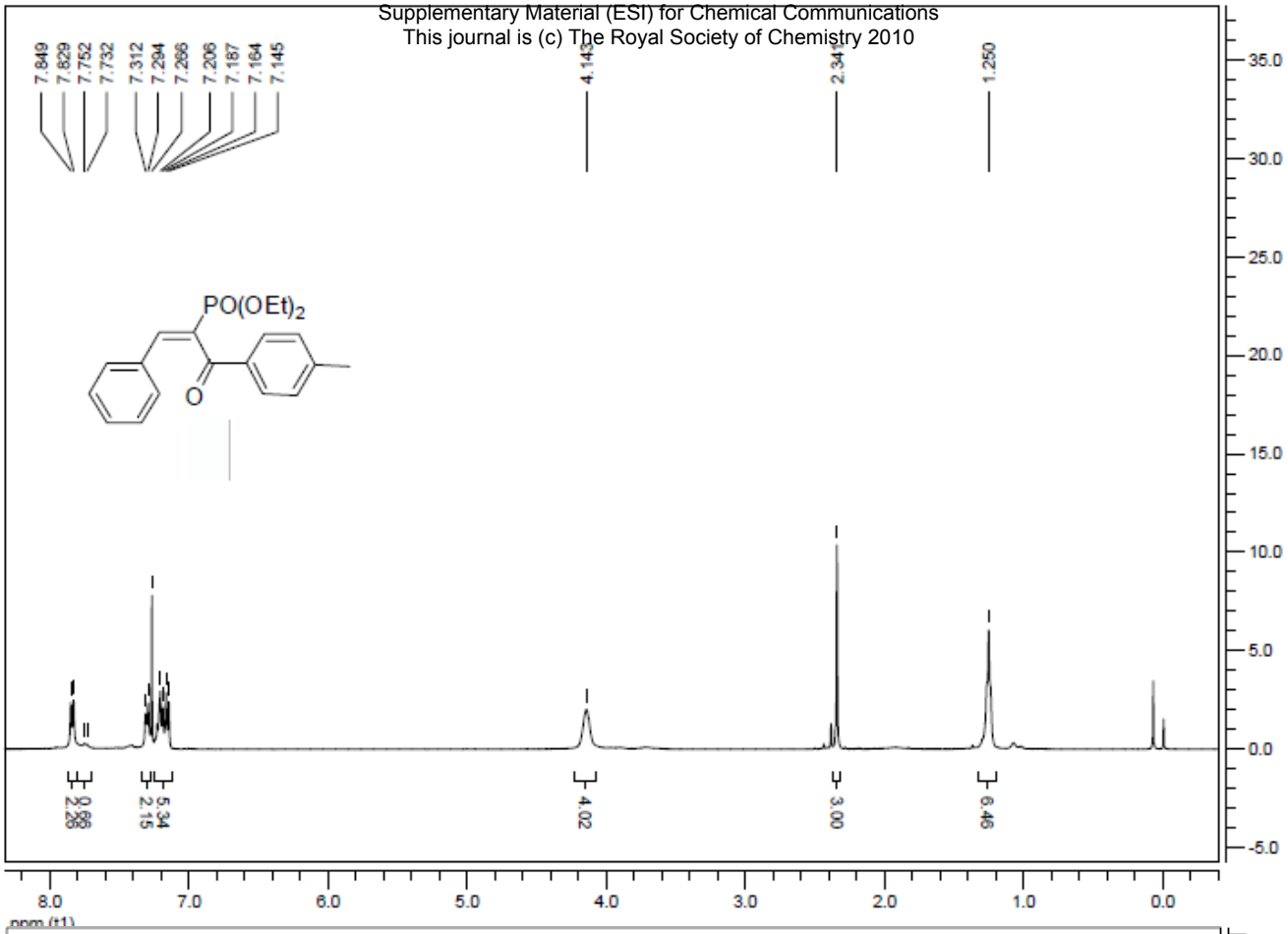
MeO
200
150
100
50
0
ppm (t1)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7.266
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7.164
7.145
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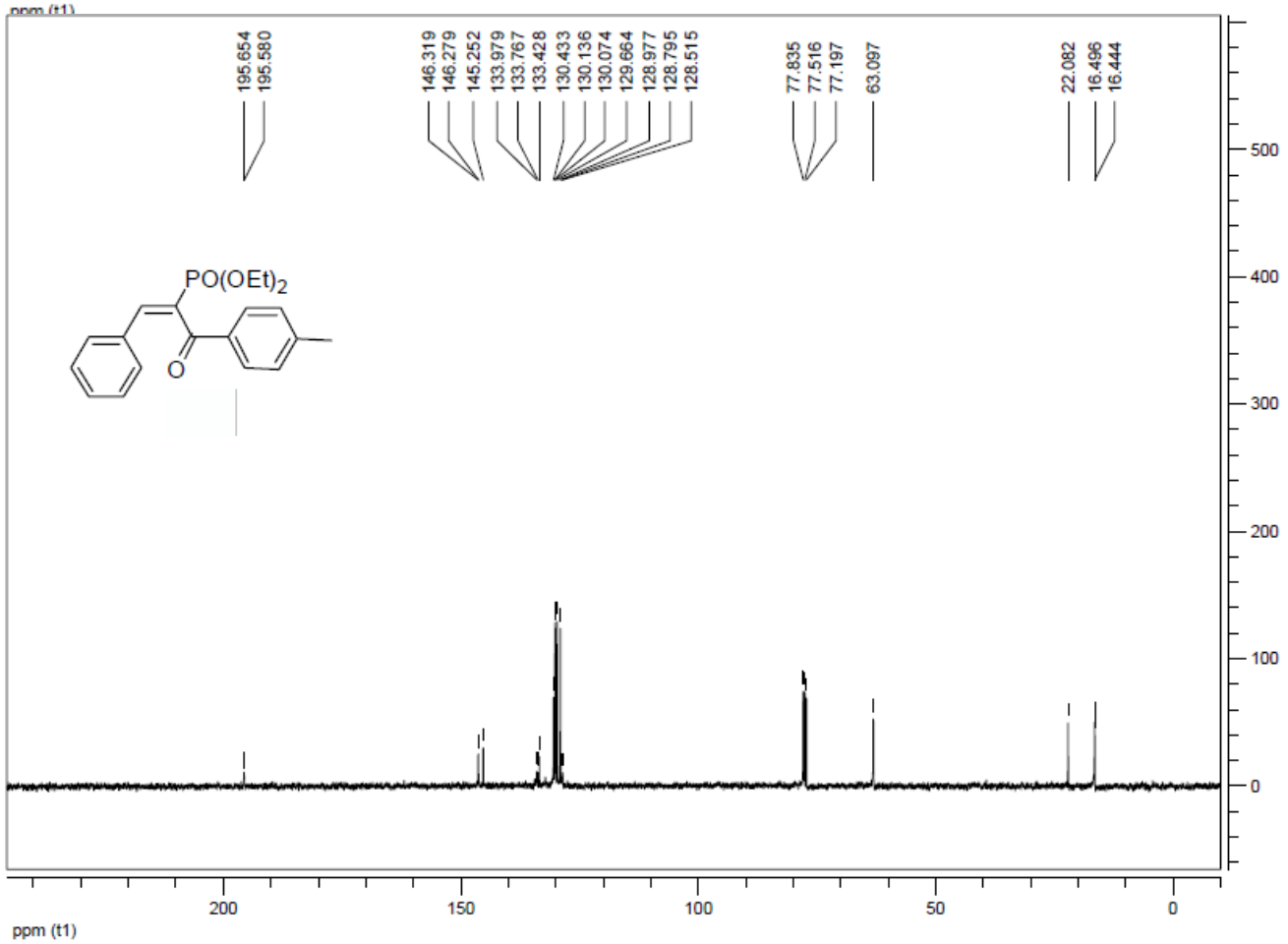
1.250
PO(OEt)2
O
9.26
9.66
5.34
2.15
4.02
3.00
6.46
35.0
30.0
25.0
20.0
15.0
10.0
5.0
0.0
-5.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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133.428
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130.136
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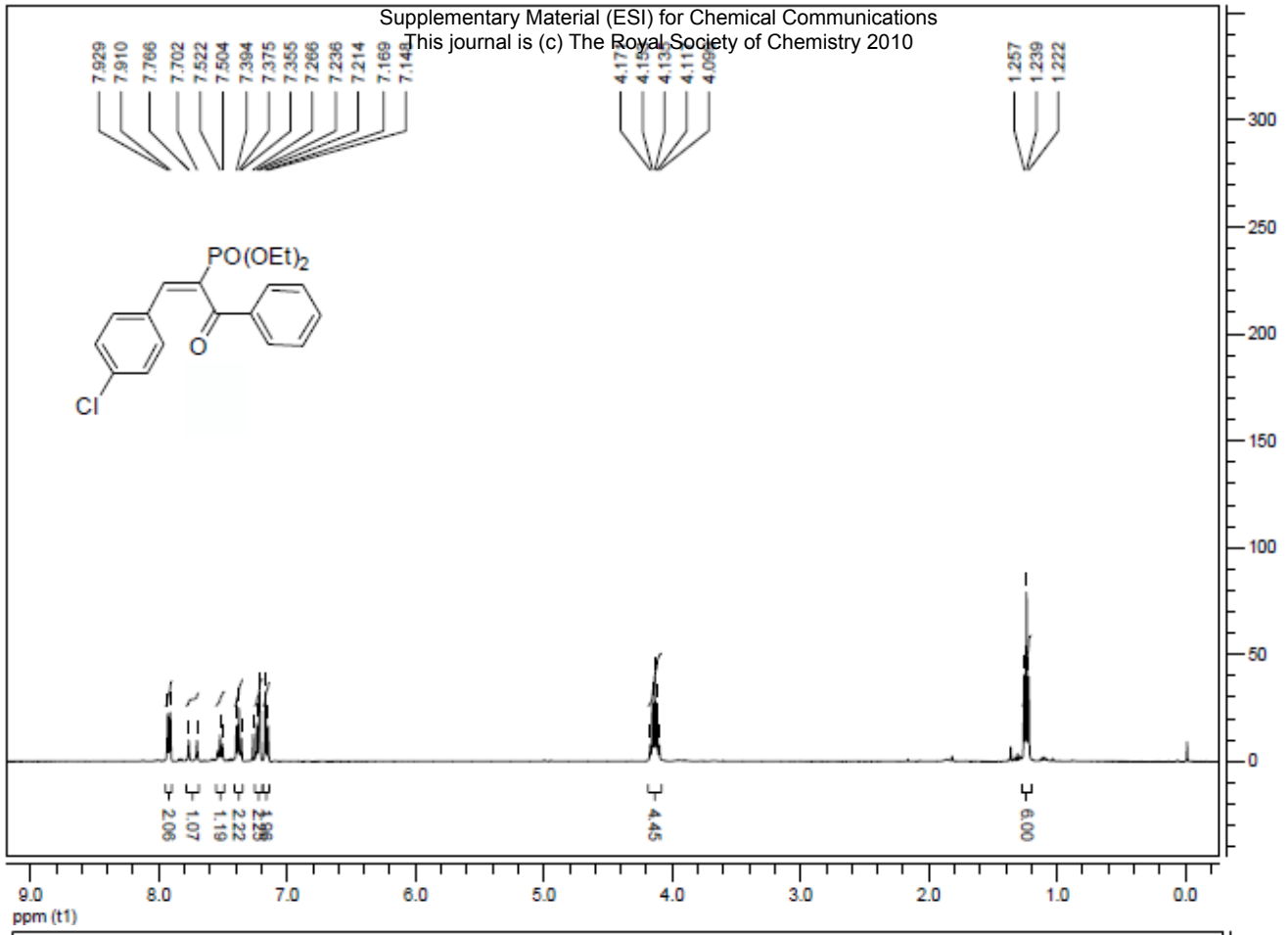
128.795
128.515
77.835
77.516
77.197
63.097
22.082
16.496
16.444
PO(OEt)2
O
500
400
300
200
100
0
200
150
100
50
0
ppm (t1)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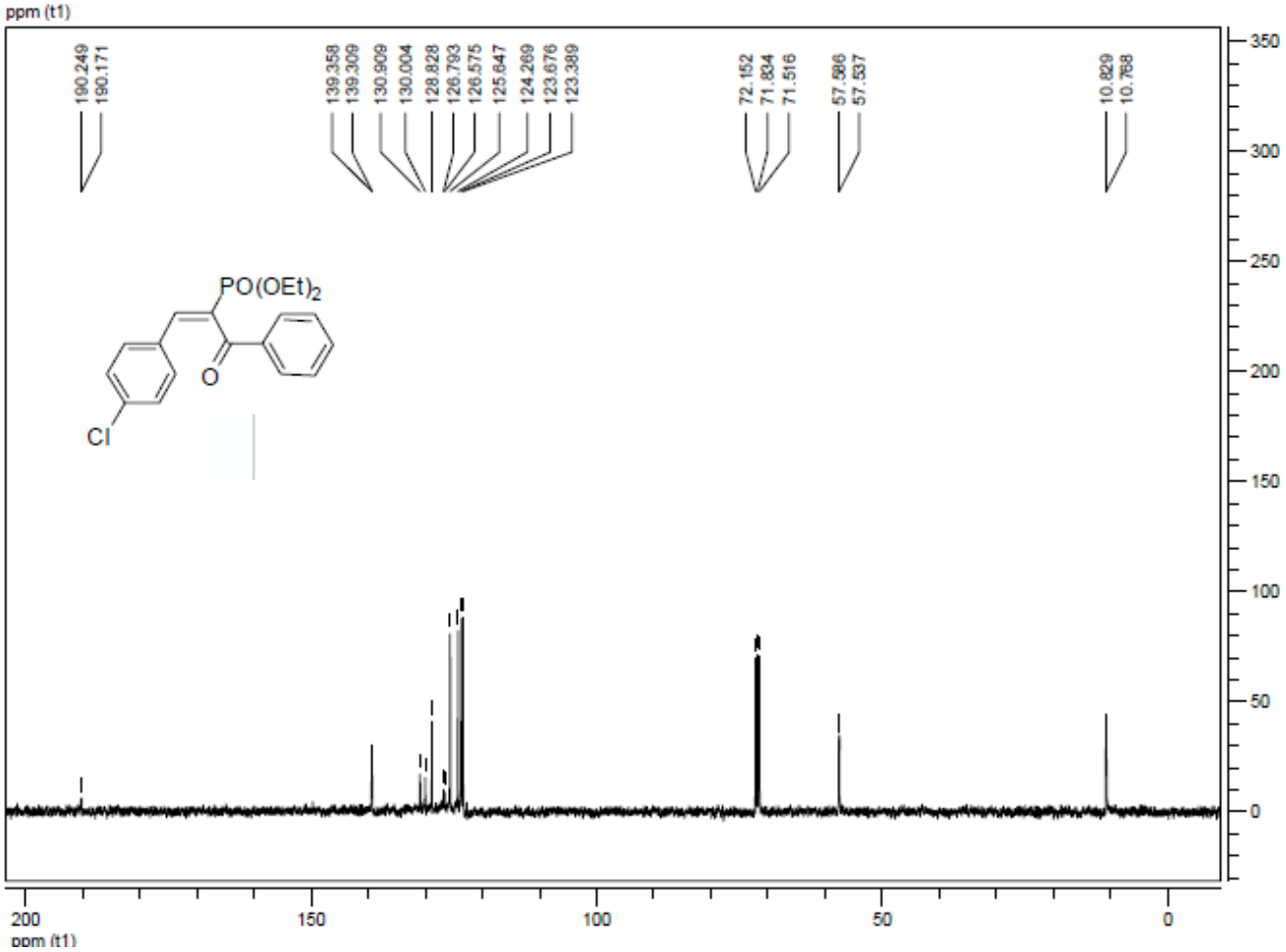
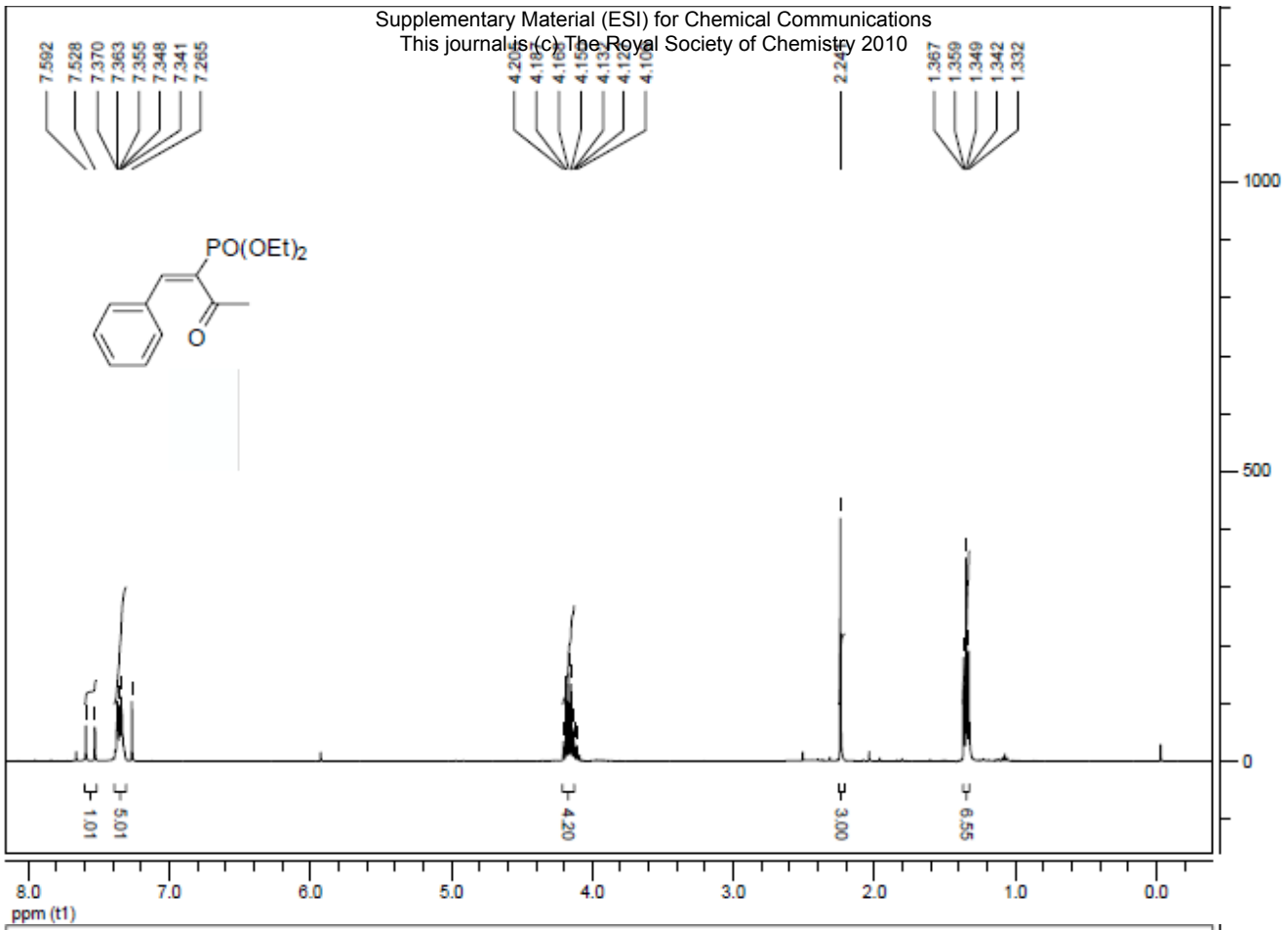
7.766
7.702
7.522
7.504
7.394
7.375
7.355
7.266
7.236
7.214
7.169
7.148
4.173
4.156
4.138
4.119
4.099
1.257
1.239
1.222
PO(OEt)2
O
Cl
2.06
1.07
1.19
2.22
2.29
3.98
4.45
6.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


190.249
190.171
139.358
139.309
130.909
130.004
128.828
126.793
126.575
125.647
124.269
123.676
123.389
72.152
71.834
71.516
57.586
57.537
10.829
10.768
PO(OEt)2
O
Cl
200
150
100
50
0
ppm (t1)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7.370
7.363
7.355
7.348
7.341
7.265
4.20
4.18
4.16
4.15
4.13
4.12
4.10
2.24
1.367
1.359
1.349
1.342
1.332
PO(OEt)2
O
1000
500
0
1.01
5.01
4.20
3.00
6.55
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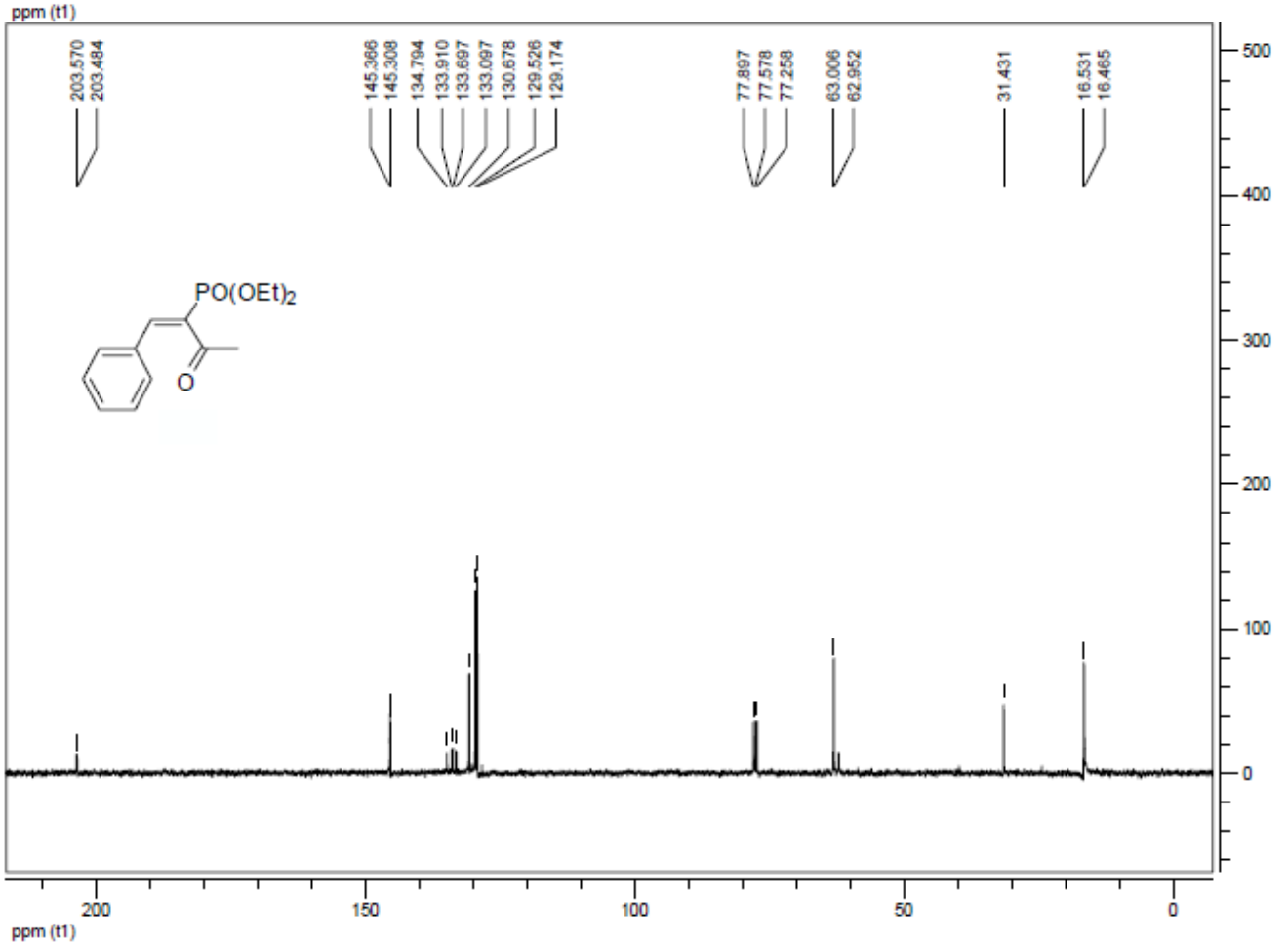
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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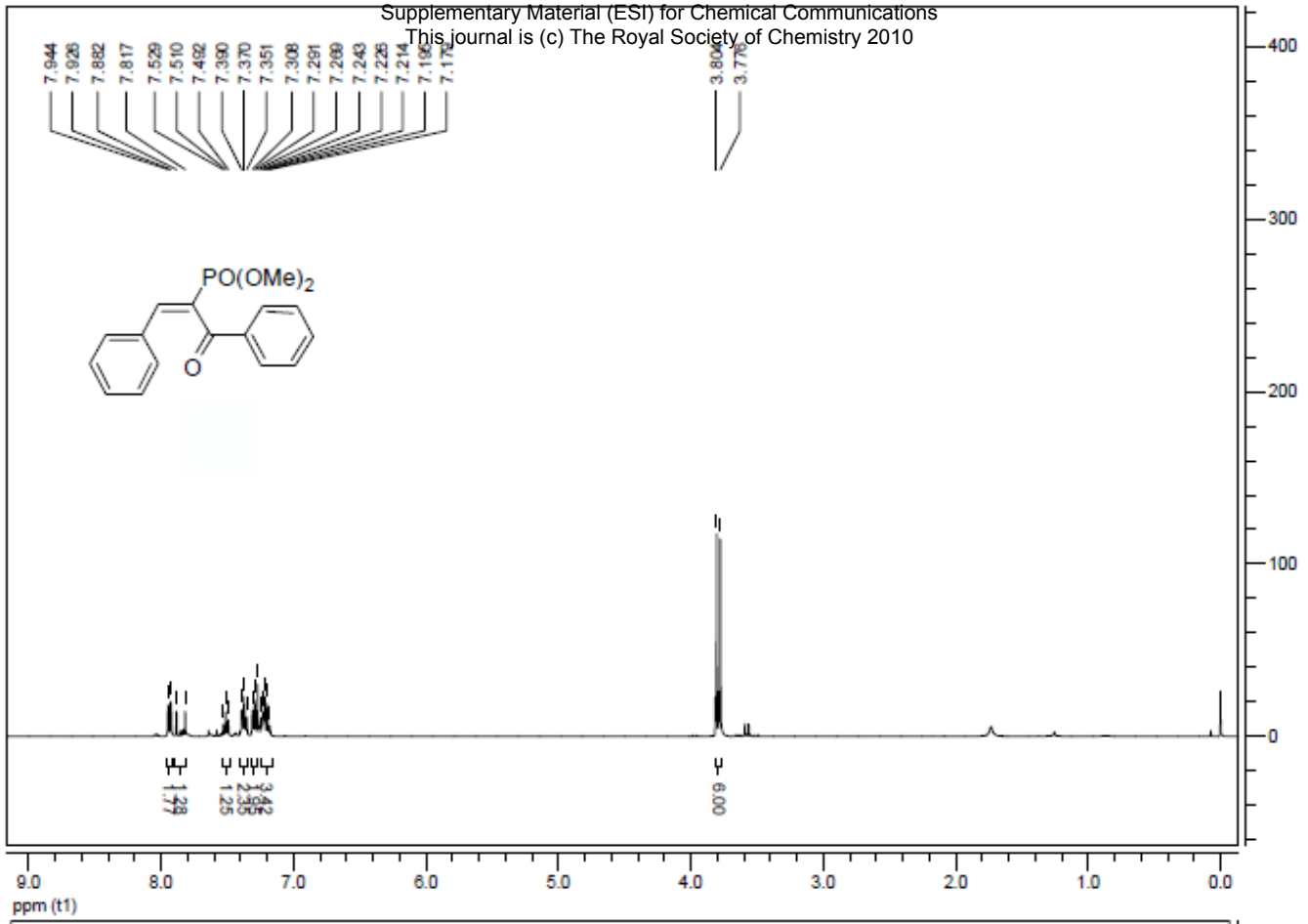
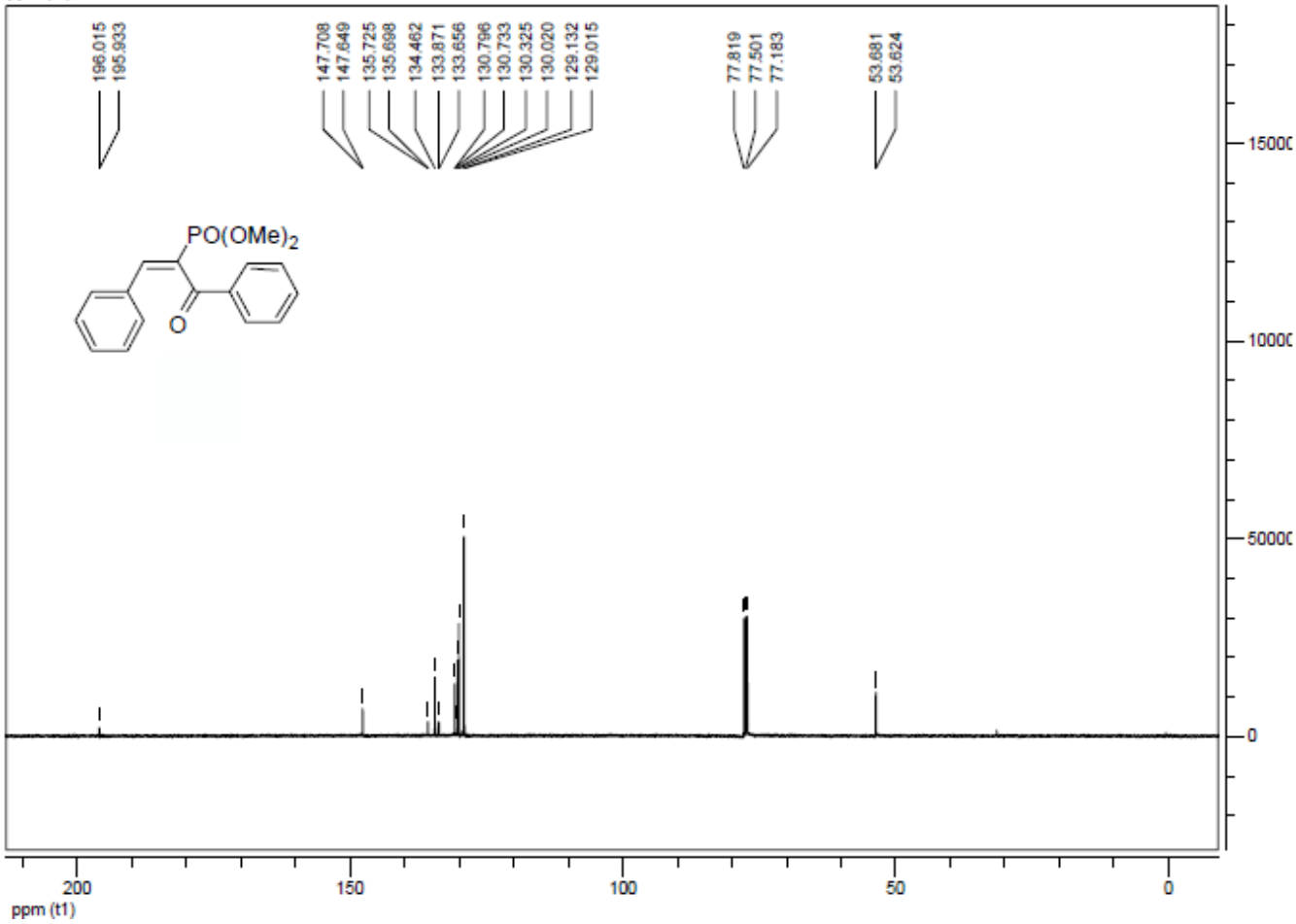
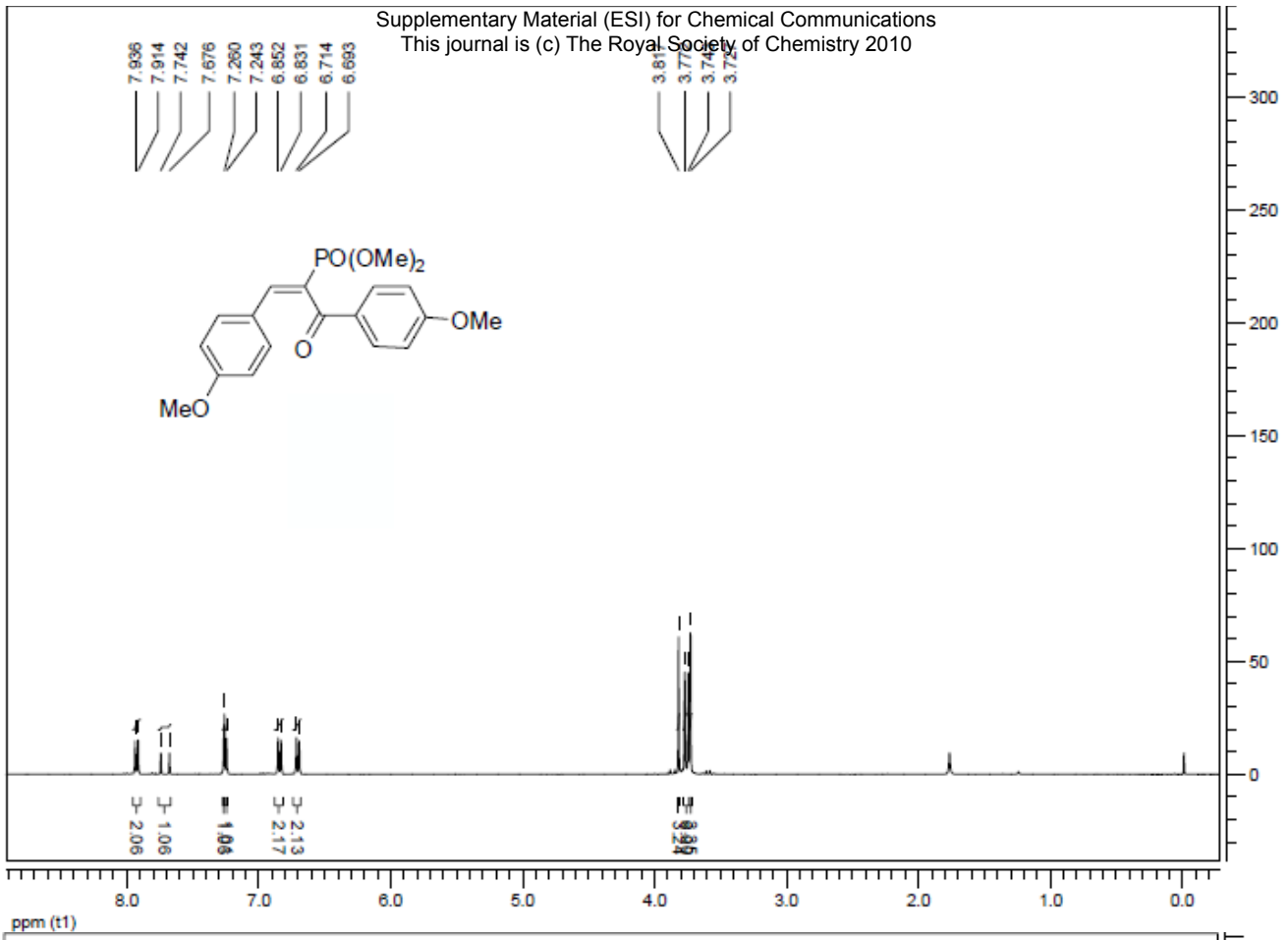
145.366
145.308
134.794
133.910
133.697
133.097
130.678
129.526
129.174
77.897
77.578
77.258
63.006
62.952
31.431
16.531
16.465
PO(OEt)2
O
500
400
300
200
100
0
200
150
100
50
0
ppm (t1)


7.944
7.926
7.882
7.817
7.529
7.510
7.492
7.390
7.370
7.351
7.308
7.291
7.269
7.243
7.226
7.214
3.804
3.776
PO(OMe)2
O
1.77
2.84
1.25
2.95
2.93
2.42
6.00
400
300
200
100
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


196.015
195.933
147.708
147.649
135.725
135.698
134.462
133.871
133.656
130.796
130.733
130.325
130.020
129.132
129.015
77.819
77.501
77.183
53.681
53.624
PO(OMe)2
O
15000
10000
50000
0
200
150
100
50
0
ppm (t1)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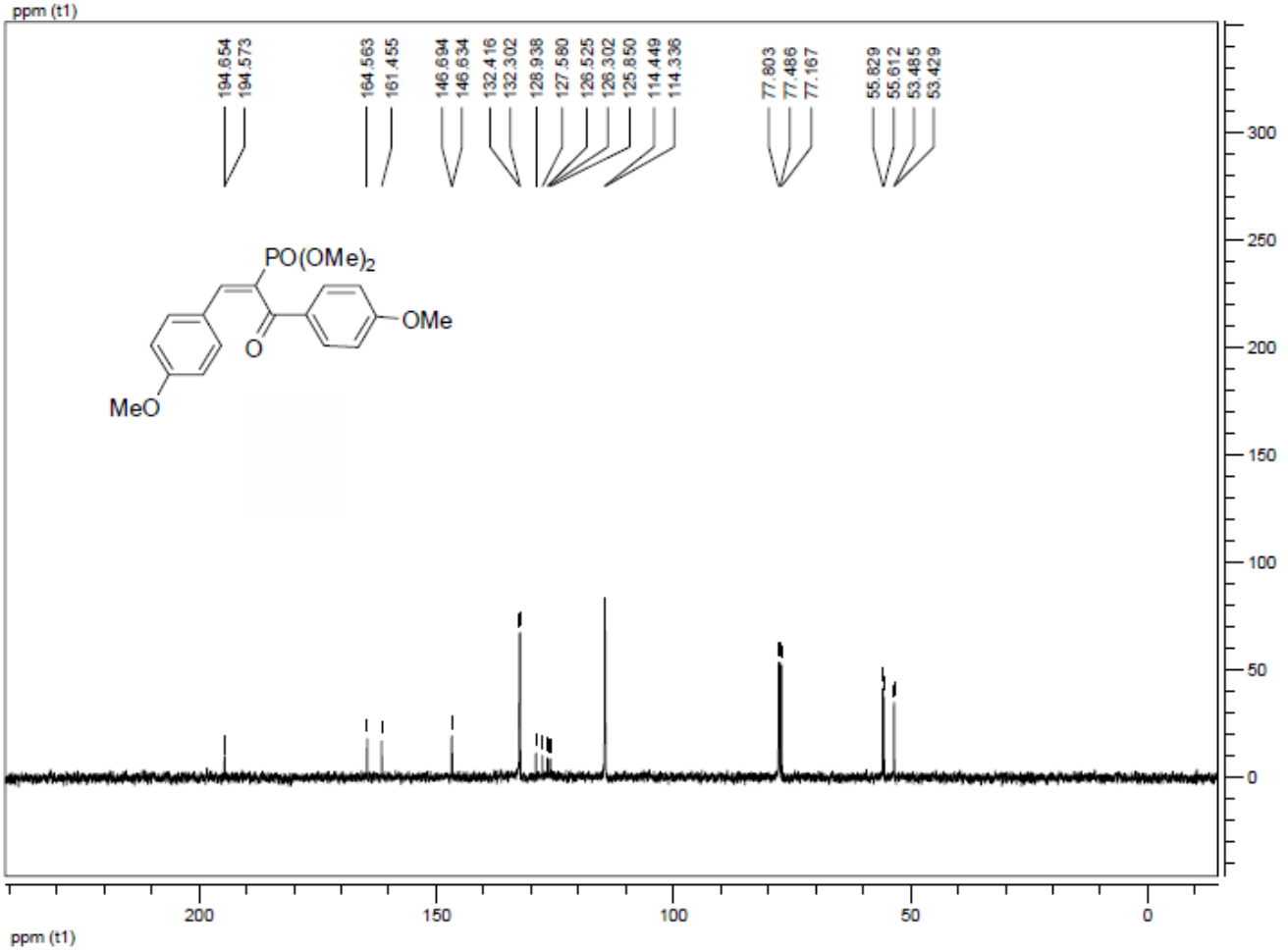
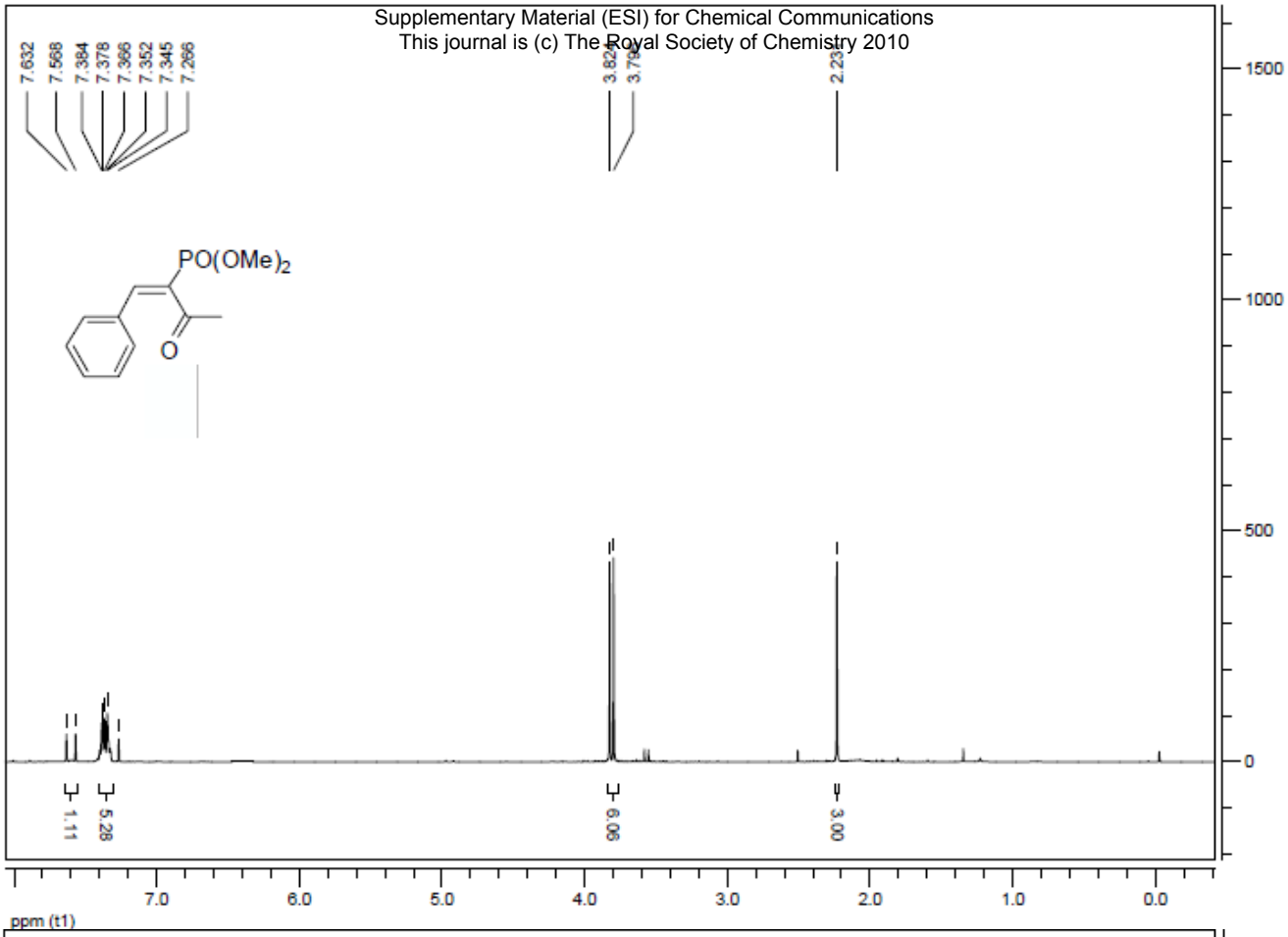
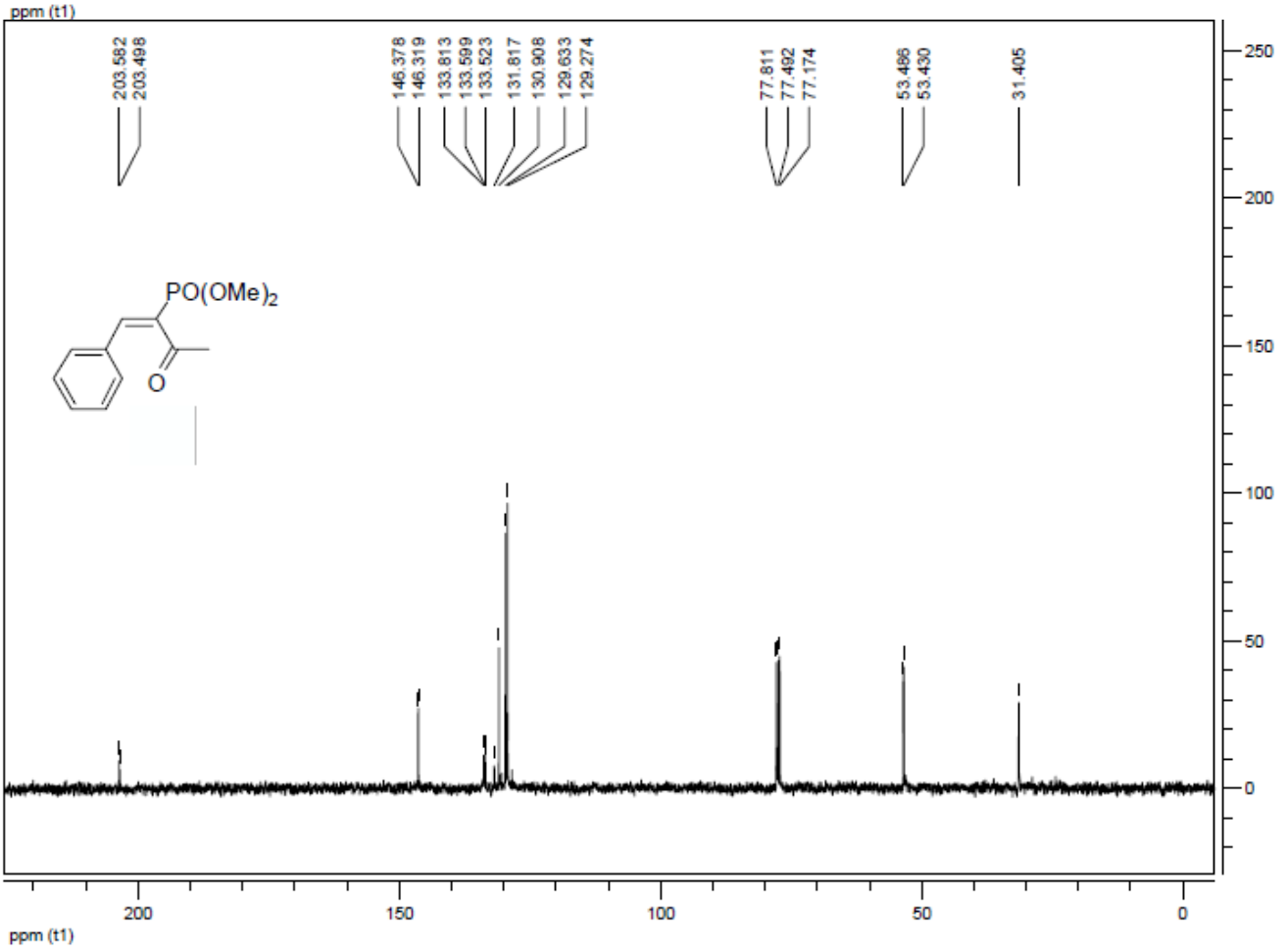
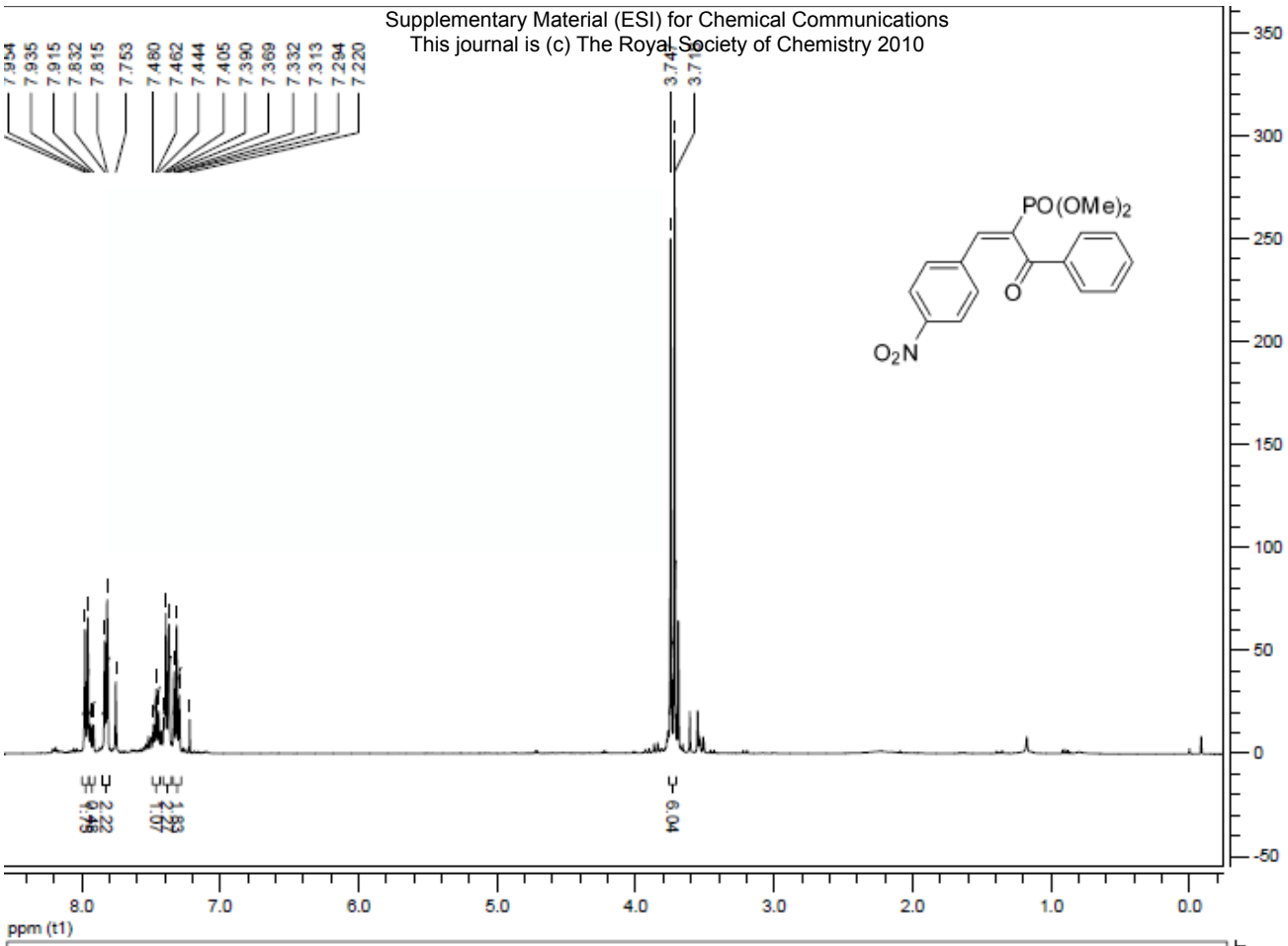
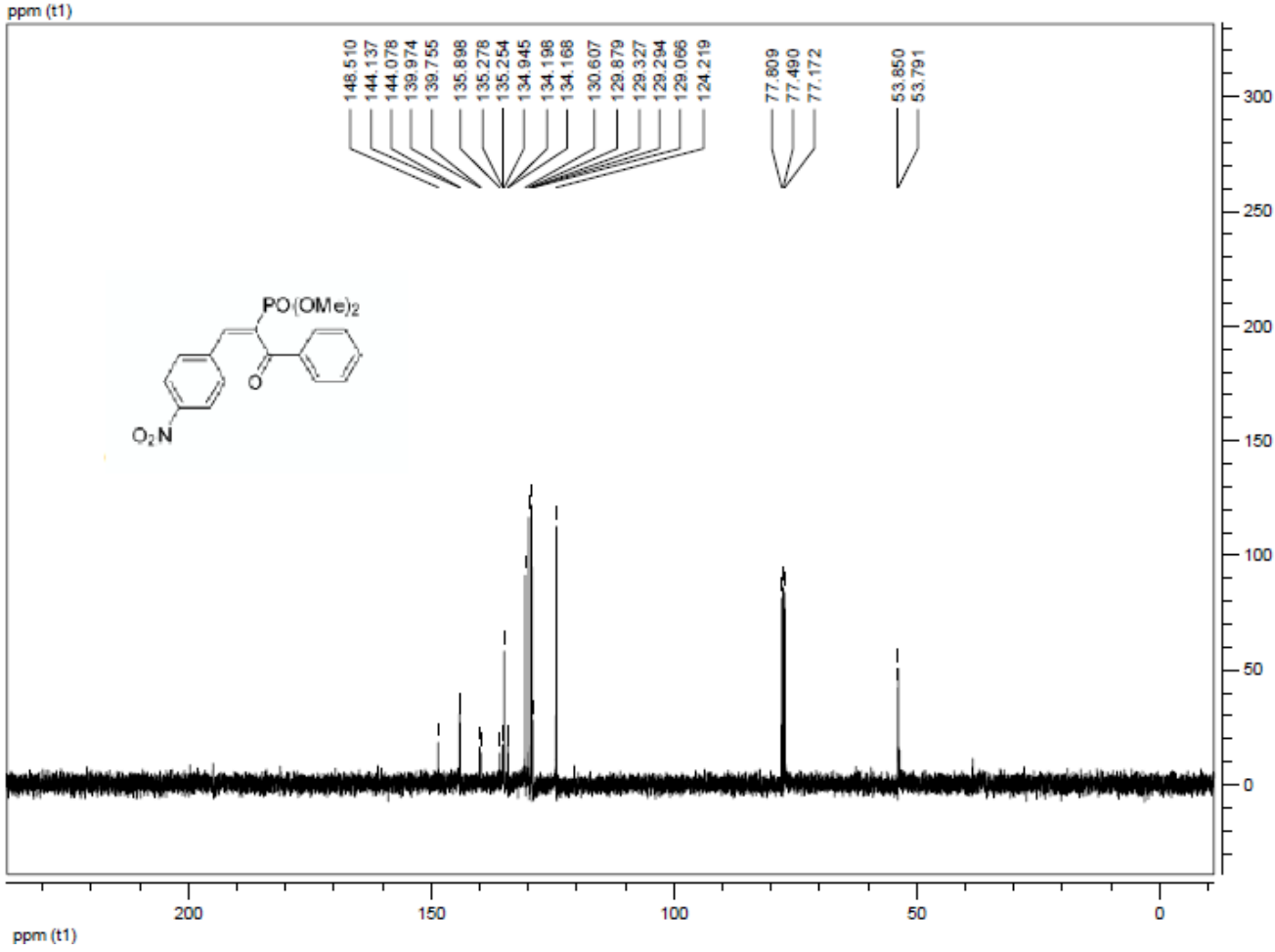
7.742
7.676
7.260
7.243
6.852
6.831
6.714
6.693
3.811
3.776
3.746
3.725
PO(OMe)2
OMe
O
MeO
2.06
1.06
1.06
2.17
2.13
3.24
ppm (t1)

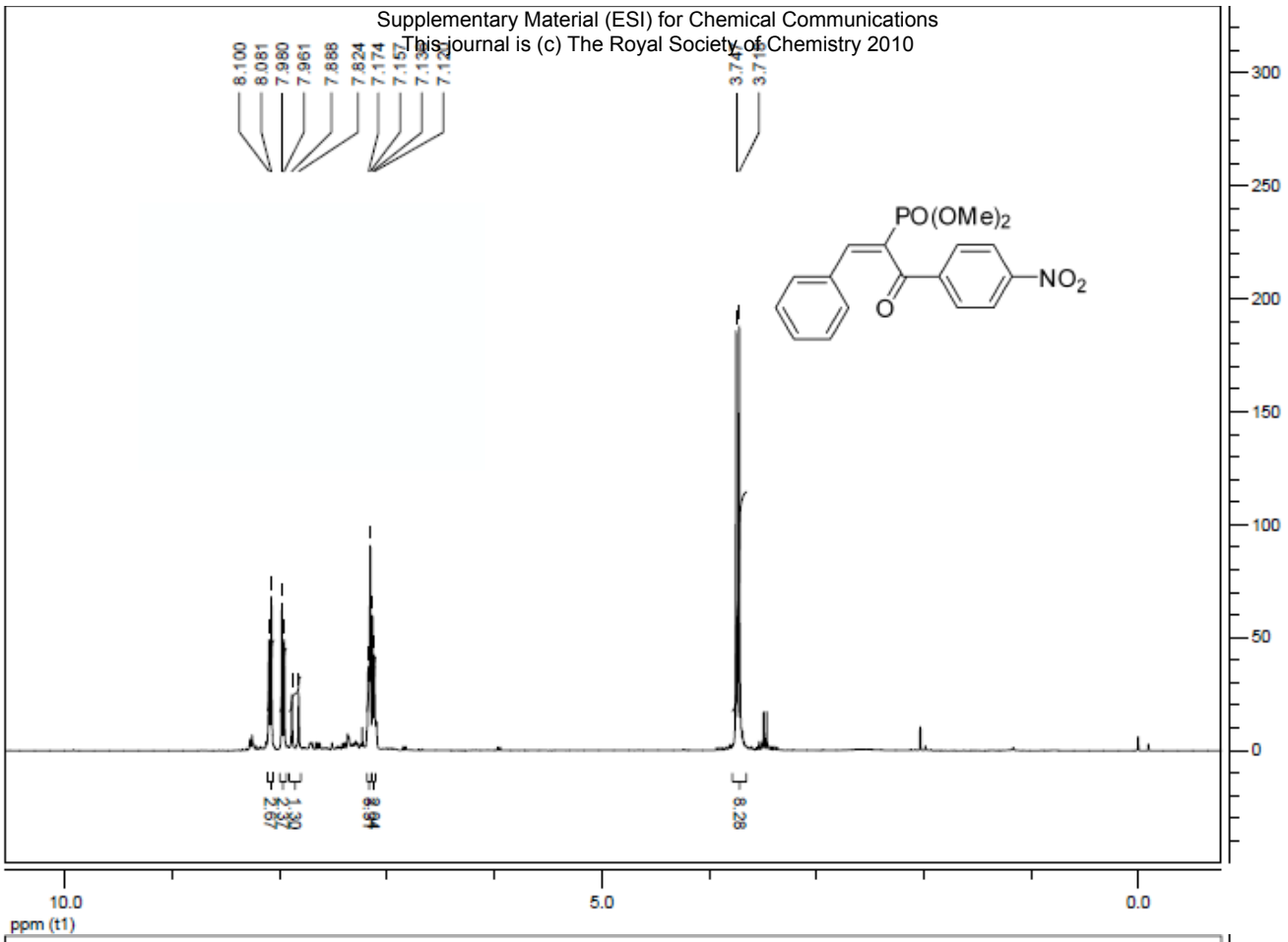

194.654
194.573
164.563
161.455
146.694
146.634
132.416
132.302
128.938
127.580
126.525
126.302
125.850
114.449
114.336
77.803
77.486
77.167
55.829
55.612
53.485
53.429
PO(OMe)2
OMe
O
MeO
ppm (t1)

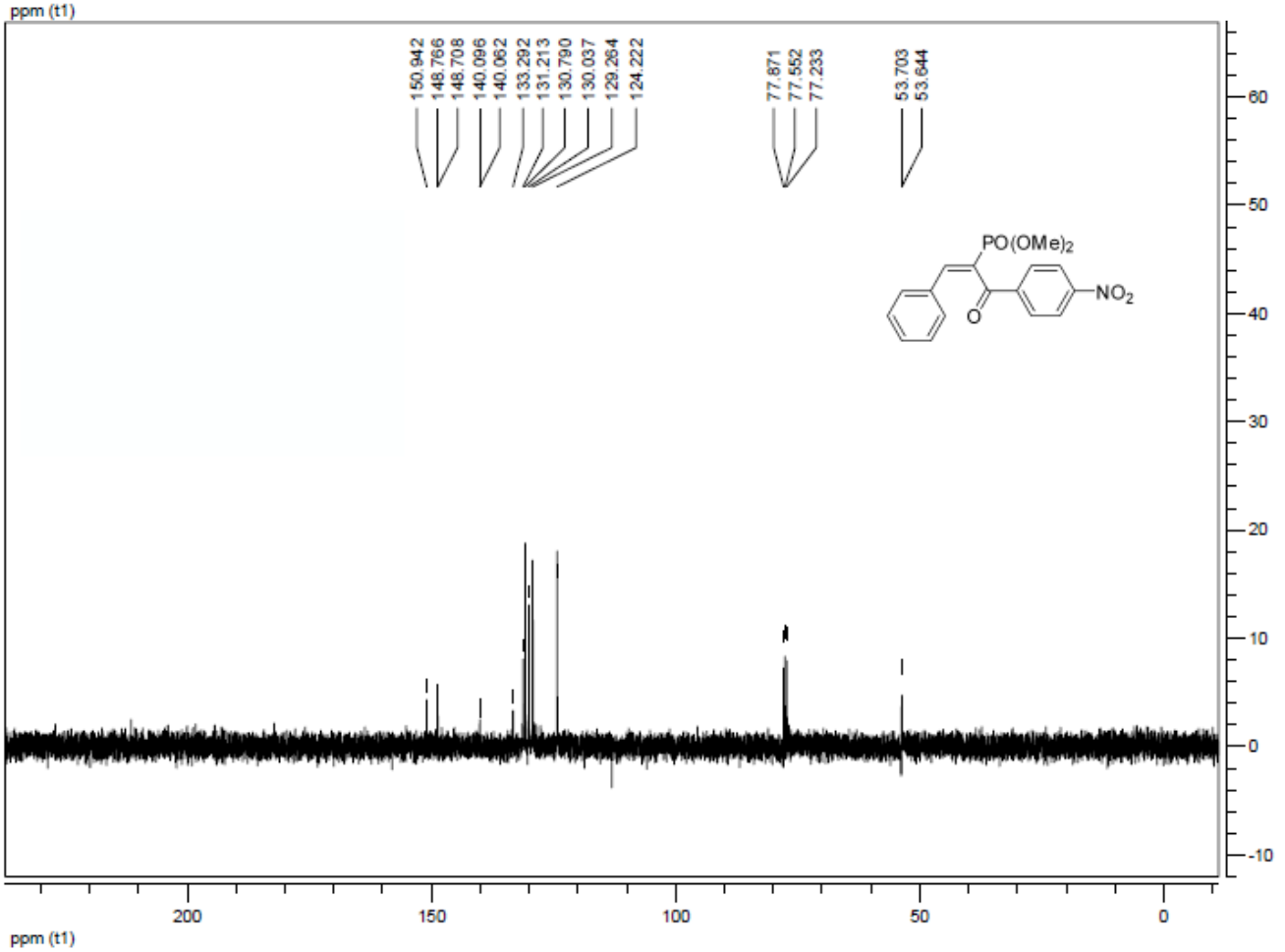

7.632
7.568
7.384
7.378
7.366
7.352
7.345
7.266
3.824
3.798
2.234
PO(OMe)2
O
1.11
5.28
6.06
3.00
ppm (t1)


203.582
203.498
146.378
146.319
133.813
133.599
133.523
131.817
130.908
129.633
129.274
77.811
77.492
77.174
53.486
53.430
31.405
PO(OMe)2
O
ppm (t1)


7.954
7.935
7.915
7.832
7.815
7.753
7.480
7.462
7.444
7.405
7.390
7.369
7.332
7.313
7.294
7.220
3.747
3.710
PO(OMe)2
O
O2N
6.04
ppm (t1)


148.510
144.137
144.078
139.974
139.755
135.898
135.278
135.254
134.945
134.198
134.168
130.607
129.879
129.327
129.294
129.066
124.219
77.809
77.490
77.172
53.850
53.791
PO(OMe)2
O
O2N
ppm (t1)


8.100
8.081
7.980
7.961
7.888
7.824
7.174
7.157
3.741
3.719
PO(OMe)2
NO2
O
10.0
5.0
0.0
ppm (t1)


150.942
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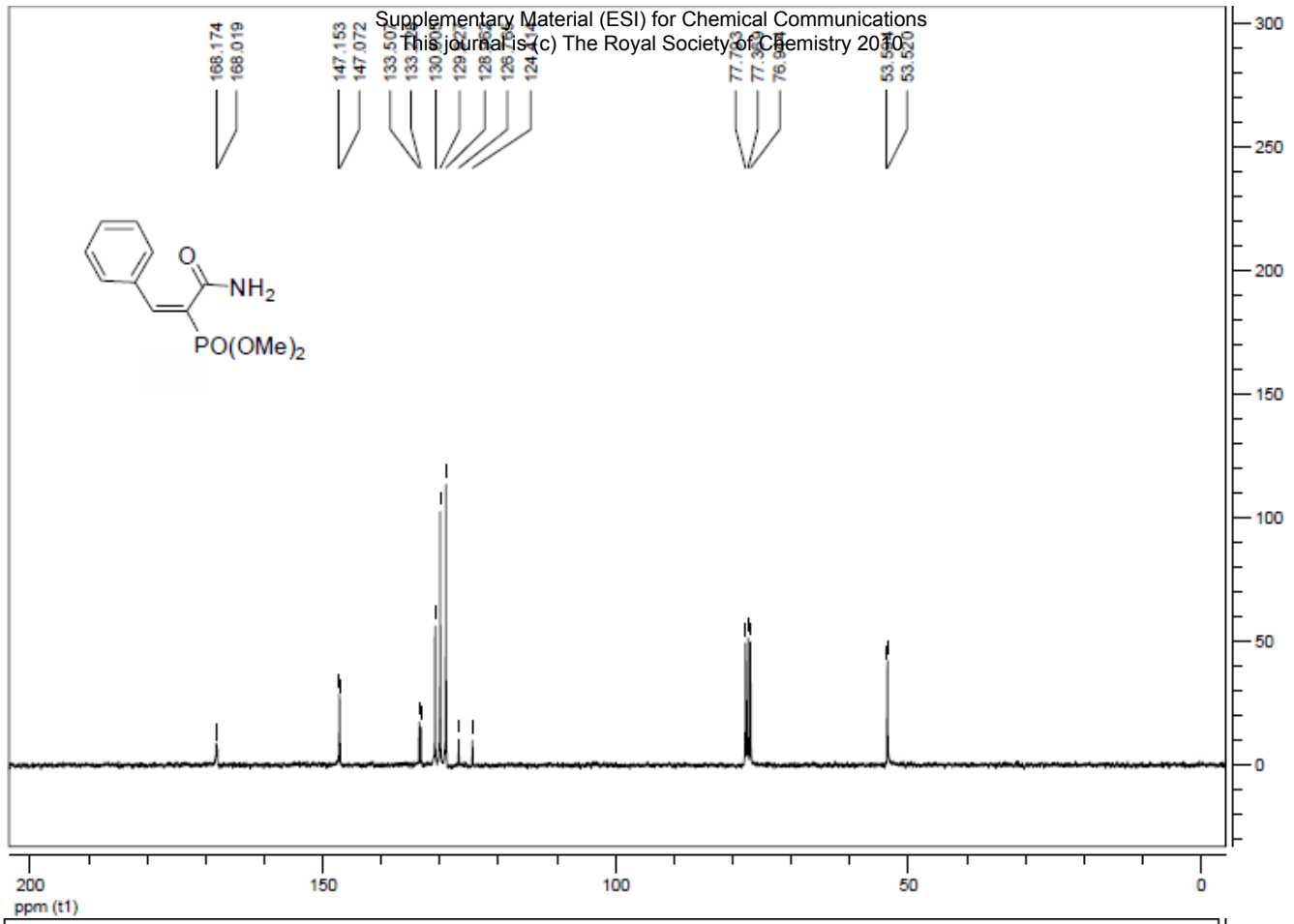
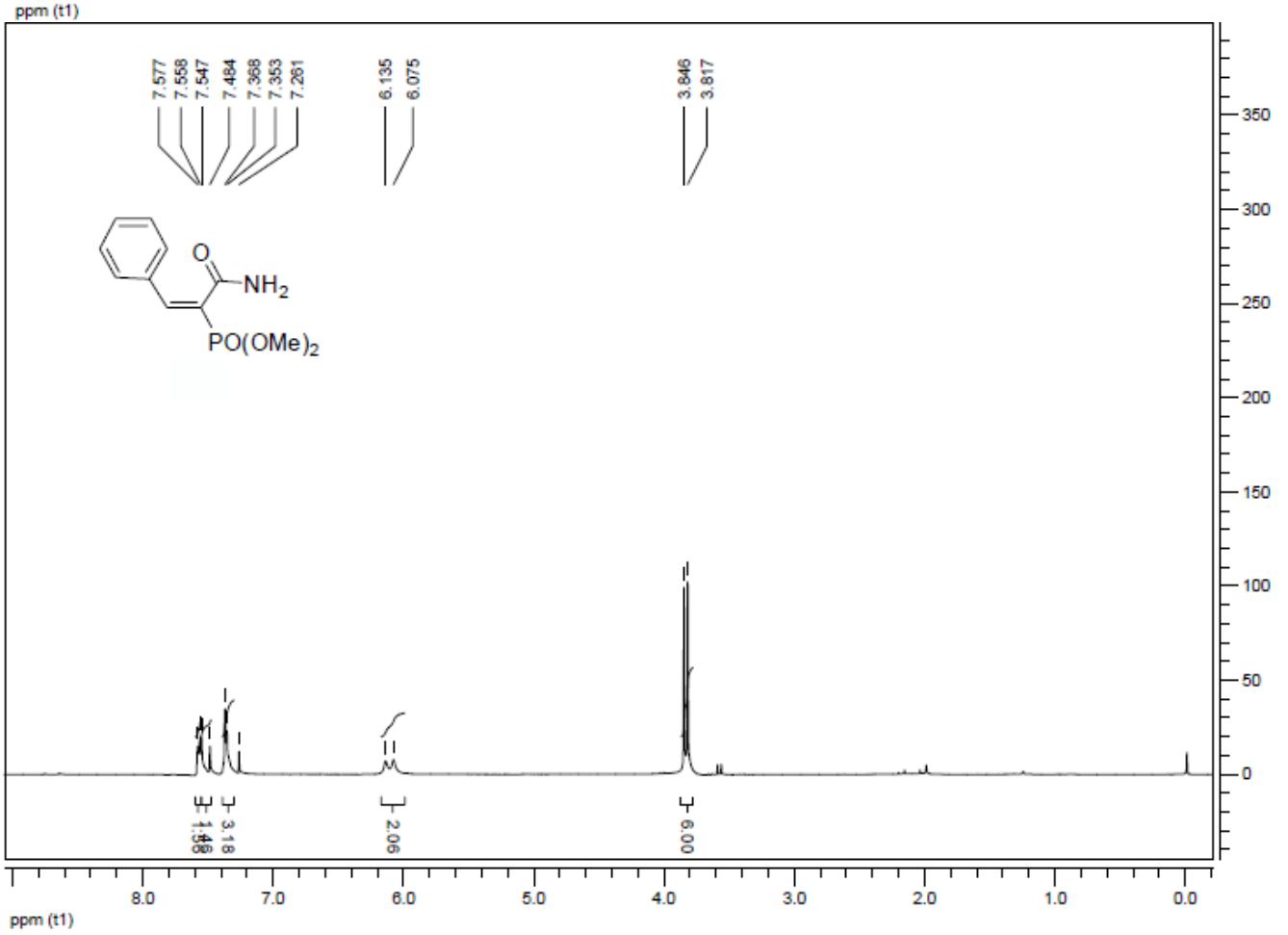
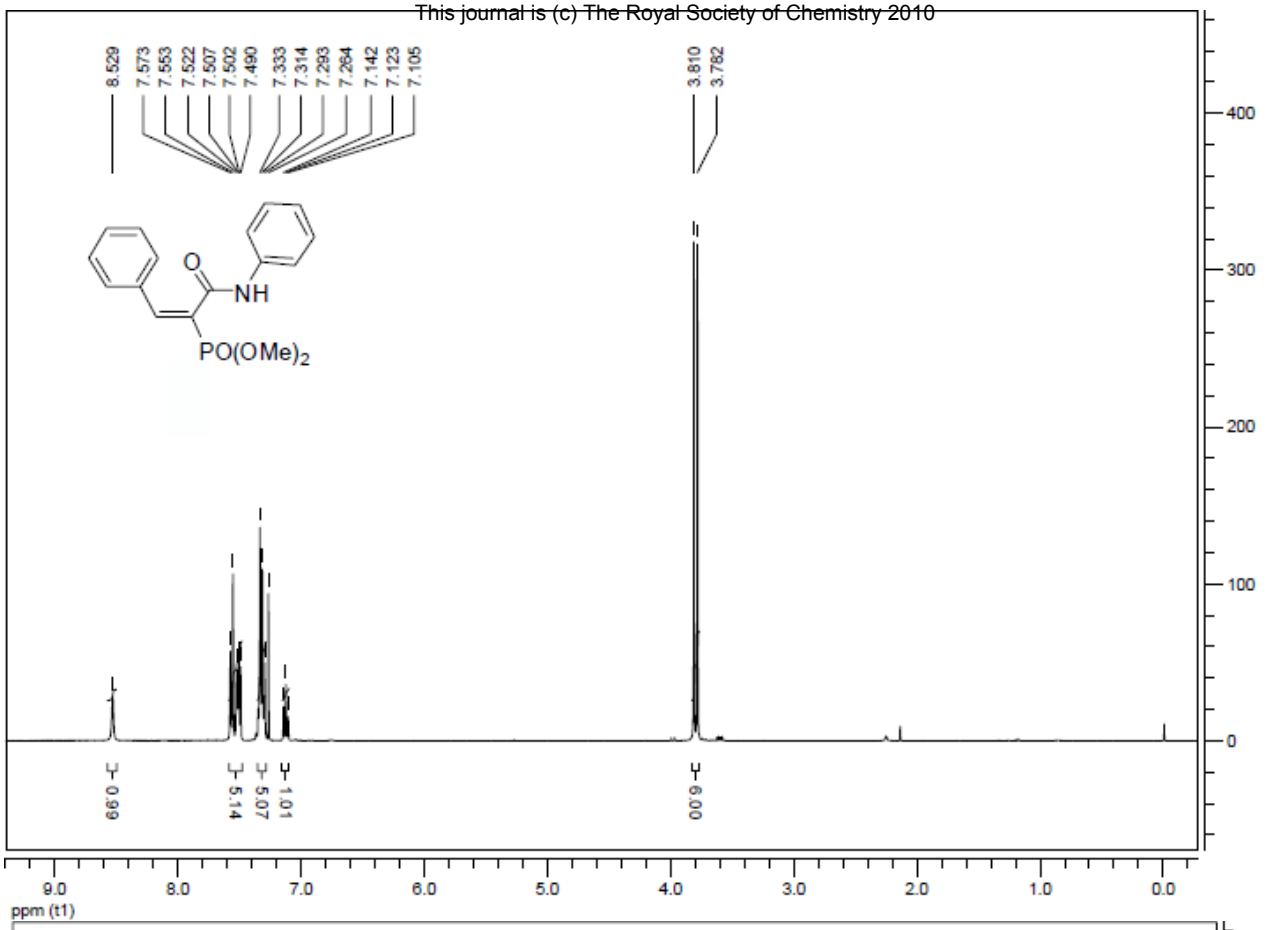
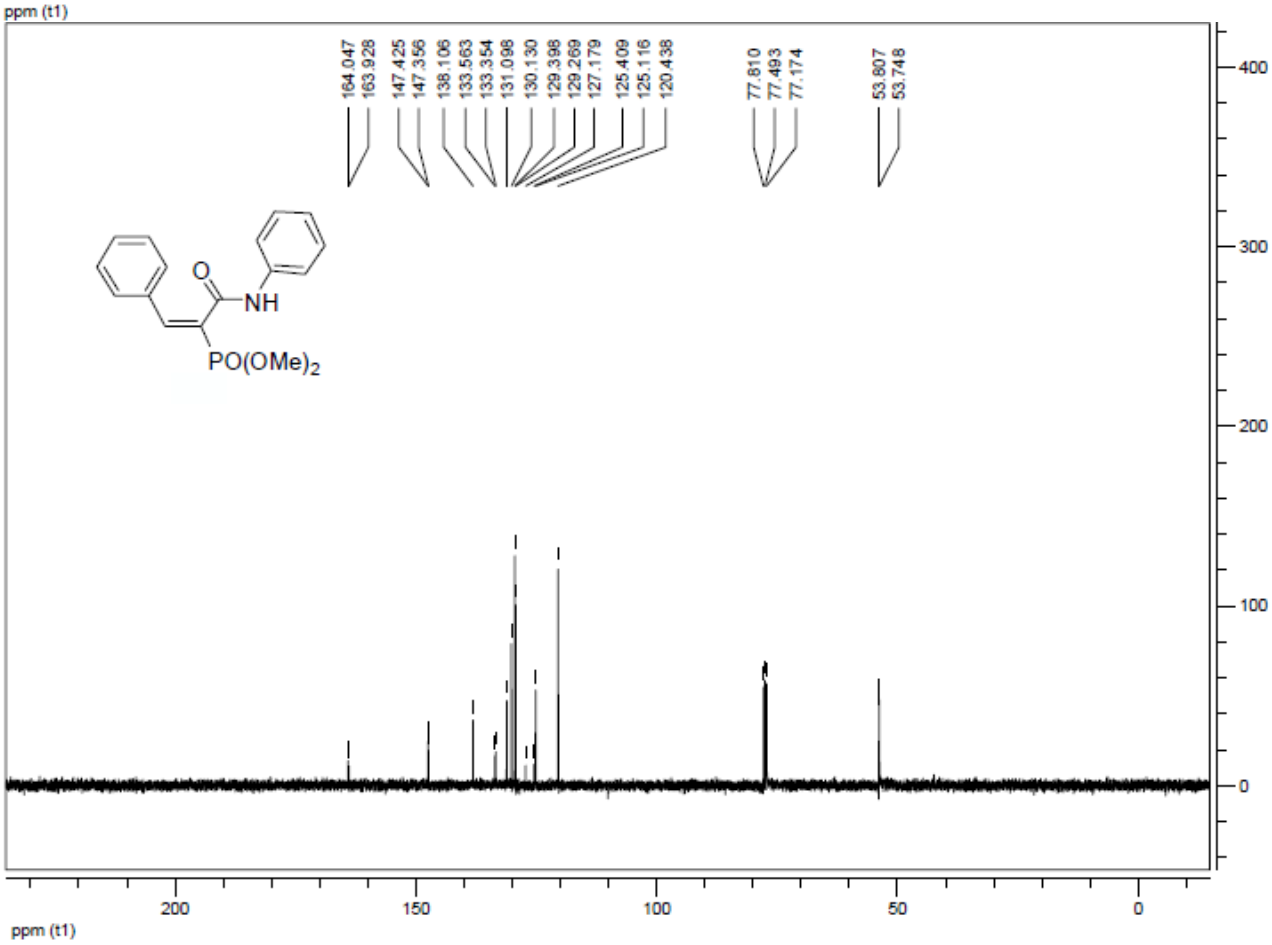
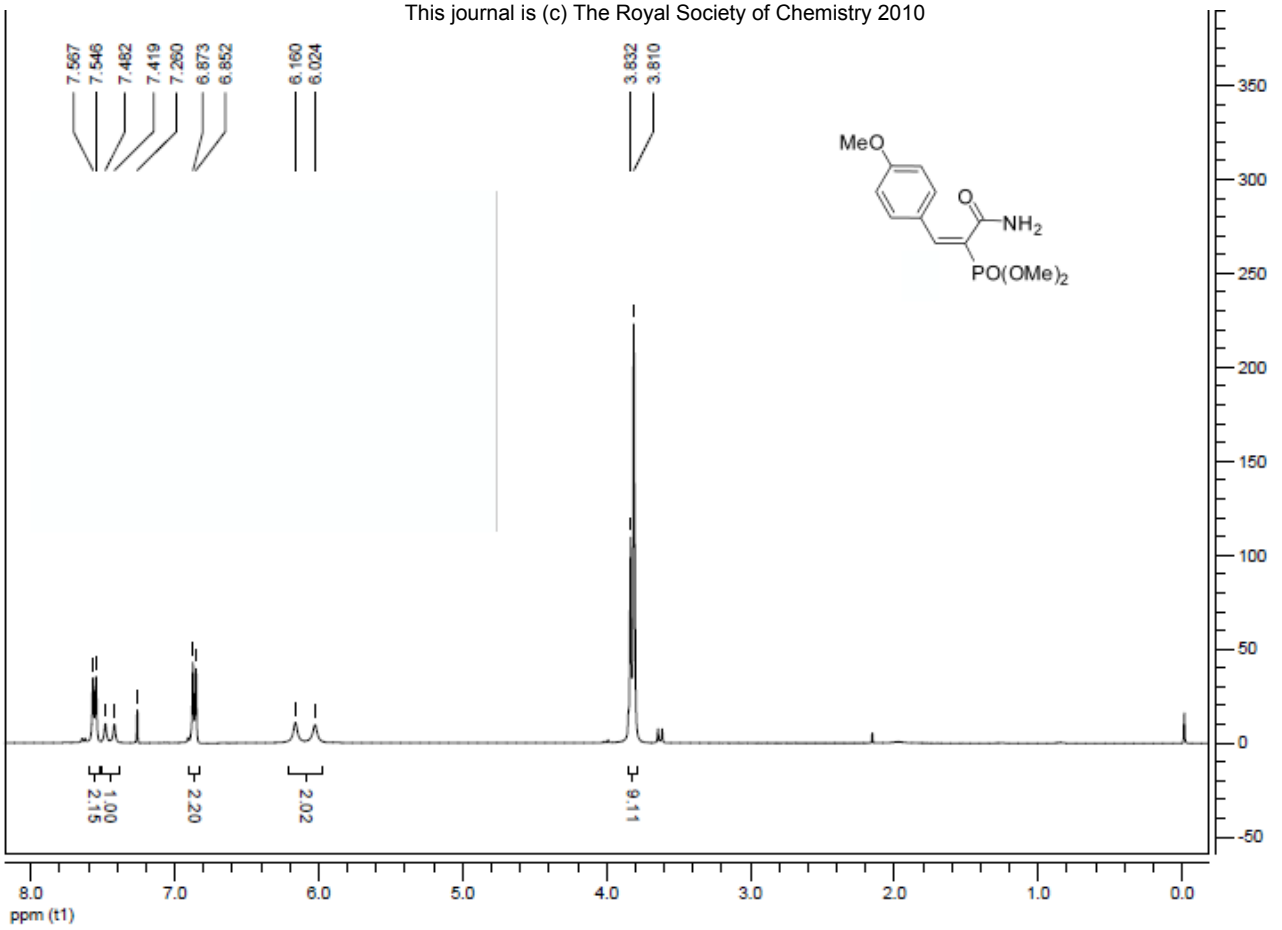
148.708
140.096
140.062
133.292
131.213
130.790
130.037
129.264
124.222
77.871
77.552
77.233
53.703
53.644
PO(OMe)2
NO2
O
200
150
100
50
0
ppm (t1)


168.174
168.019
147.153
147.072
133.507
133.411
130.045
129.227
128.463
126.963
124.140
77.793
77.380
76.968
53.594
53.529
O
NH2
PO(OMe)2
300
250
200
150
100
50
0
200
150
100
50
0
ppm (t1)


7.577
7.558
7.547
7.484
7.368
7.353
7.261
6.135
6.075
3.846
3.817
O
NH2
PO(OMe)2
1.98
3.18
2.06
6.00
350
300
250
200
150
100
50
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


8.529
7.573
7.553
7.522
7.507
7.502
7.490
7.333
7.314
7.293
7.264
7.142
7.123
7.105
3.810
3.782
O
NH
PO(OMe)2
0.99
5.14
5.07
1.01
6.00
ppm (t1)


164.047
163.928
147.425
147.356
138.106
133.563
133.354
131.098
130.130
129.398
129.269
127.179
125.409
125.116
120.438
77.810
77.493
77.174
53.807
53.748
O
NH
PO(OMe)2
ppm (t1)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7.546
7.482
7.419
7.260
6.873
6.852
6.160
6.024
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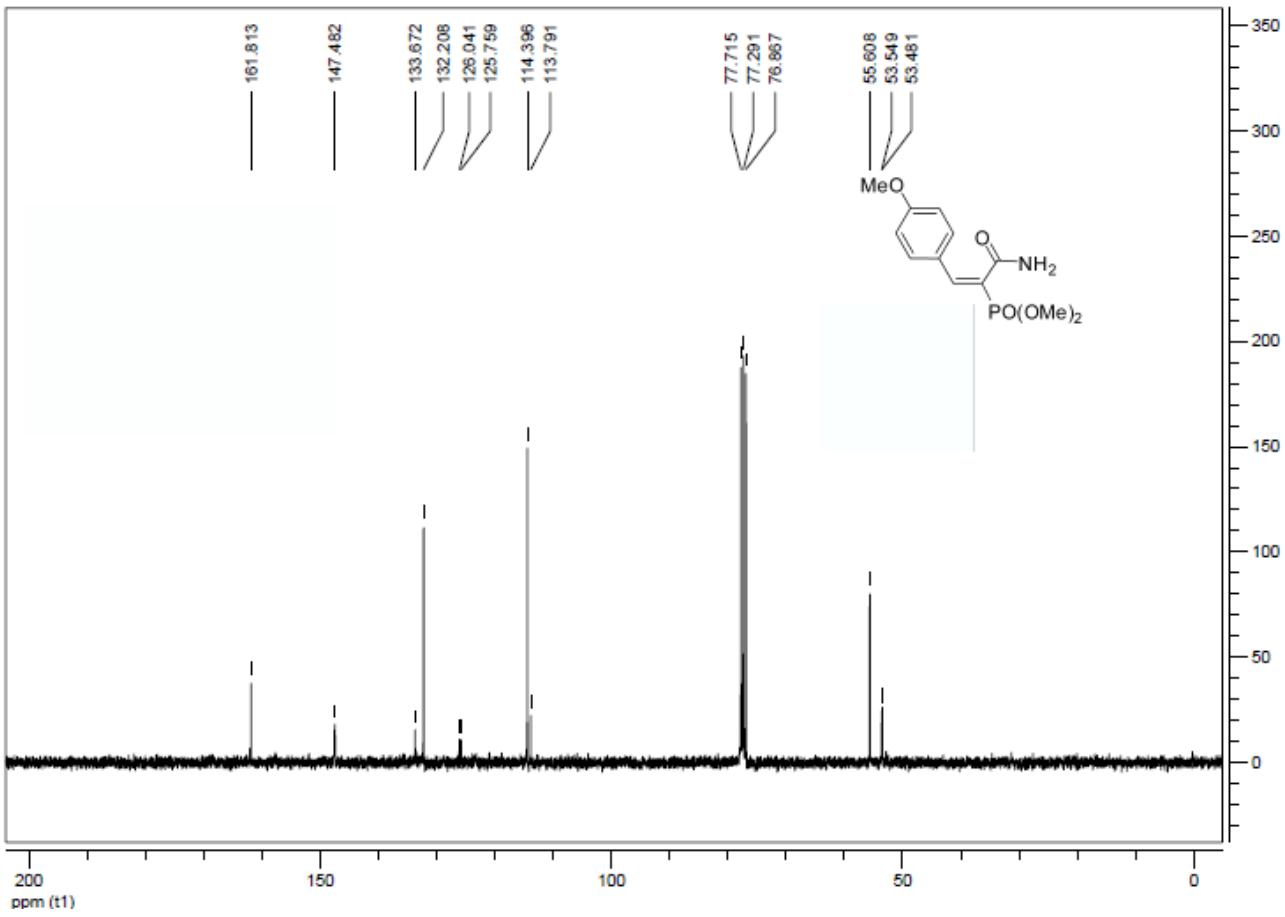
3.810
MeO
O
NH2
PO(OMe)2
2.15
1.00
2.20
2.02
9.11
ppm (t1)


161.813
147.482
133.672
132.208
126.041
125.759
114.396
113.791
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77.291
76.867
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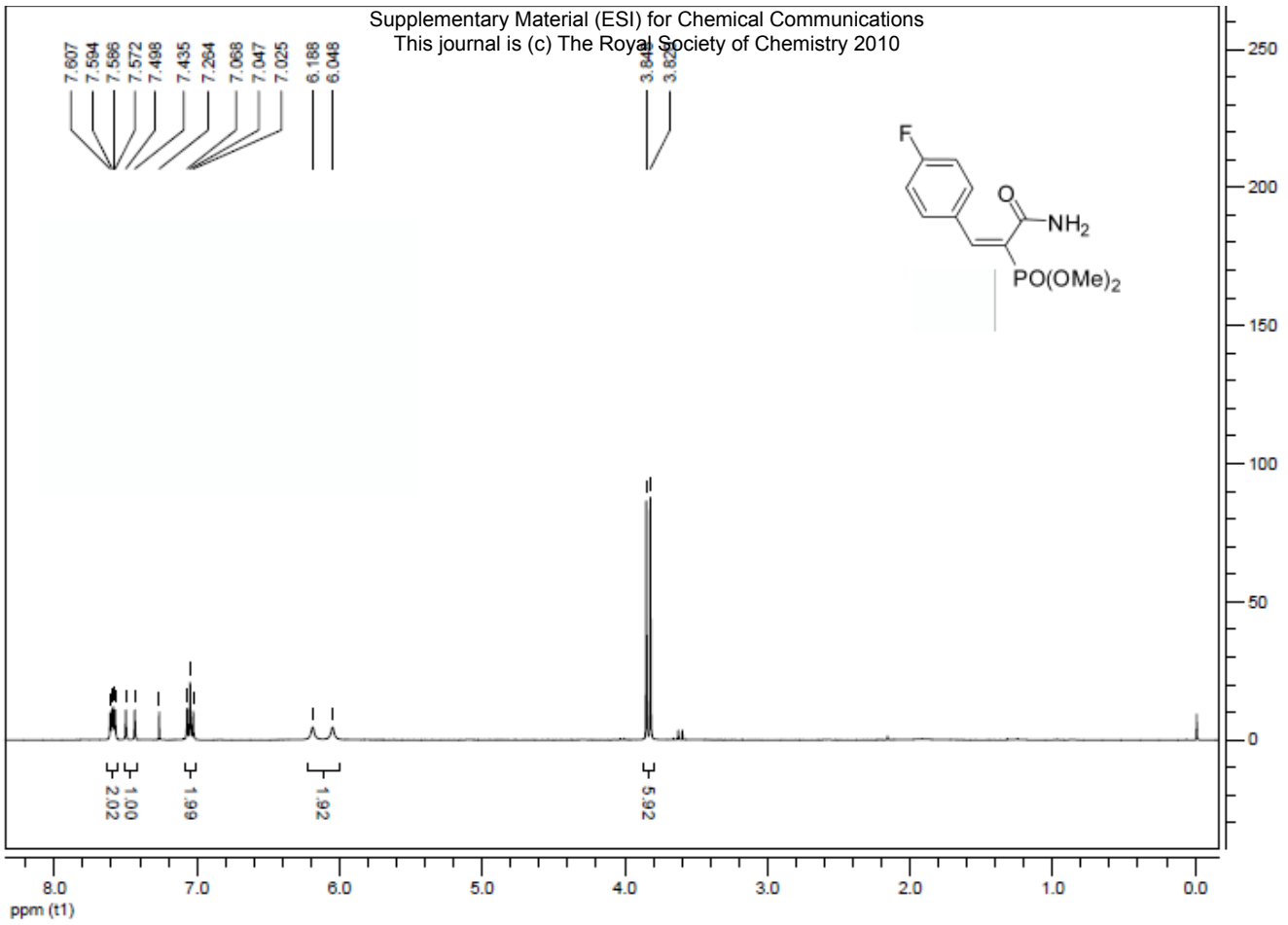
53.549
53.481
MeO
O
NH2
PO(OMe)2
ppm (t1)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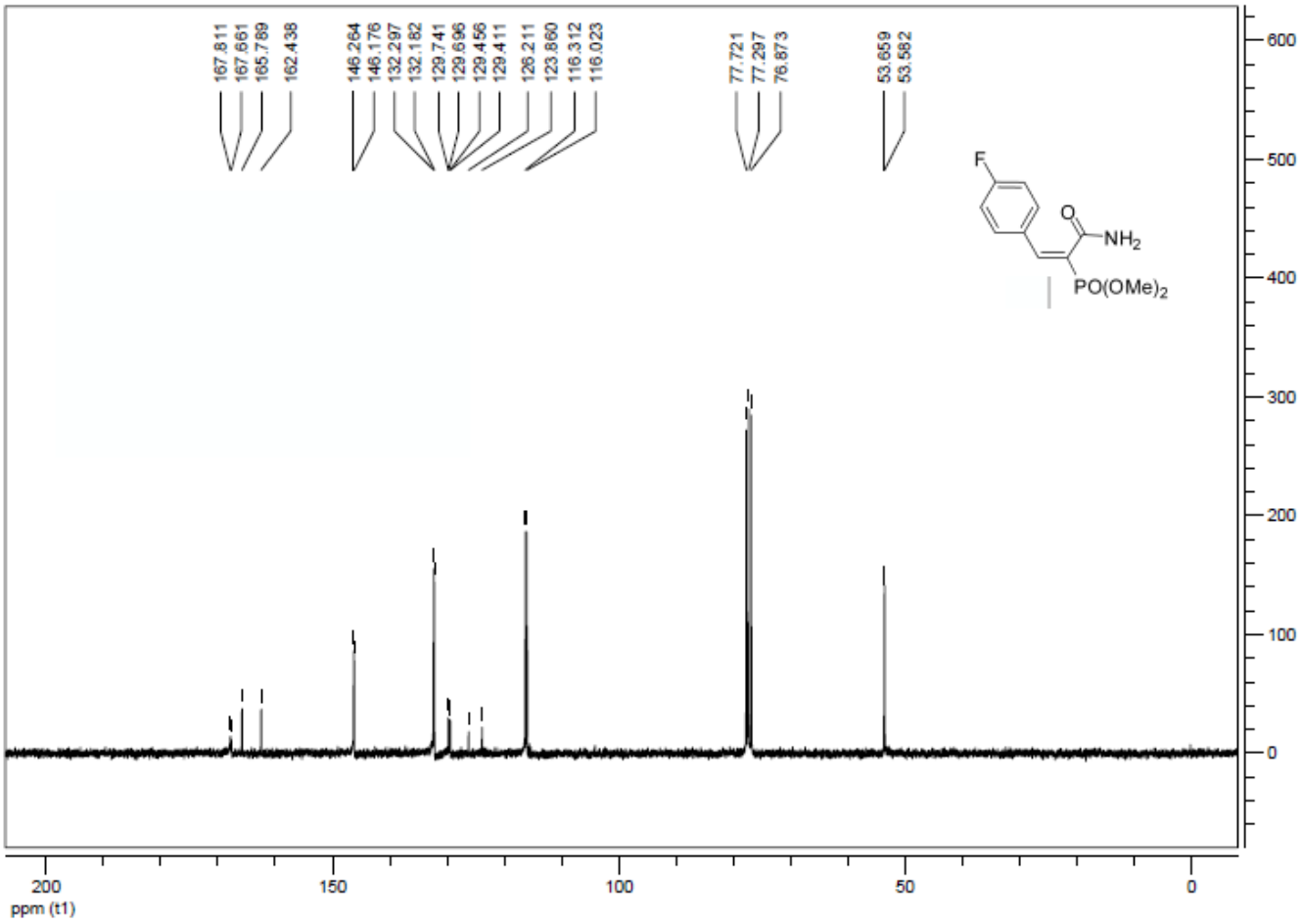
7.594
7.586
7.572
7.498
7.435
7.264
7.068
7.047
7.025
6.188
6.048
3.848
3.826
F
O
NH2
PO(OMe)2
2.02
1.00
1.99
1.92
5.92
ppm (t1)


167.811
167.661
165.769
162.438
146.264
146.176
132.297
132.182
129.741
129.696
129.456
129.411
128.211
123.860
116.312
116.023
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77.297
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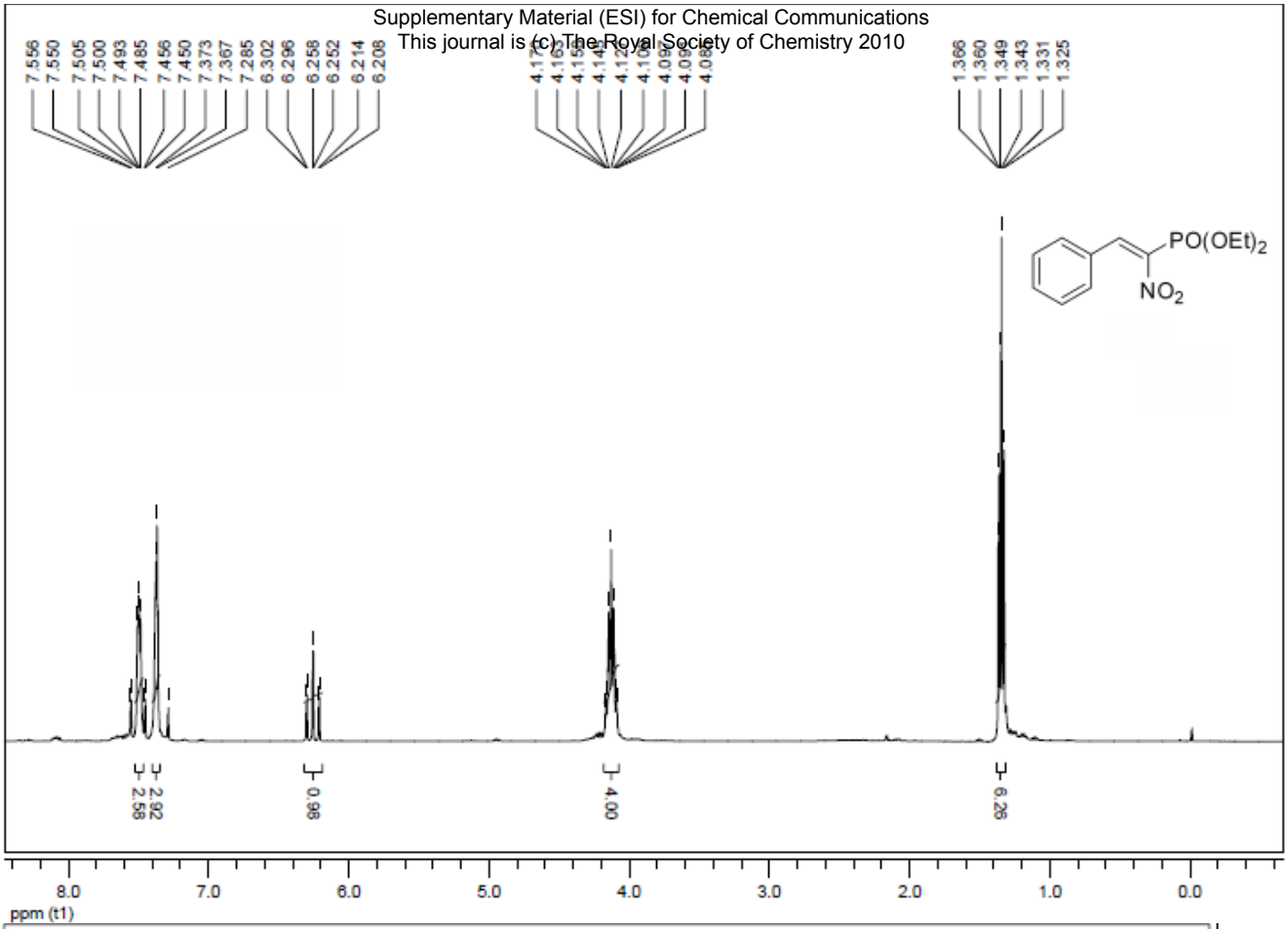
53.659
53.582
F
O
NH2
PO(OMe)2
ppm (t1)


7.556
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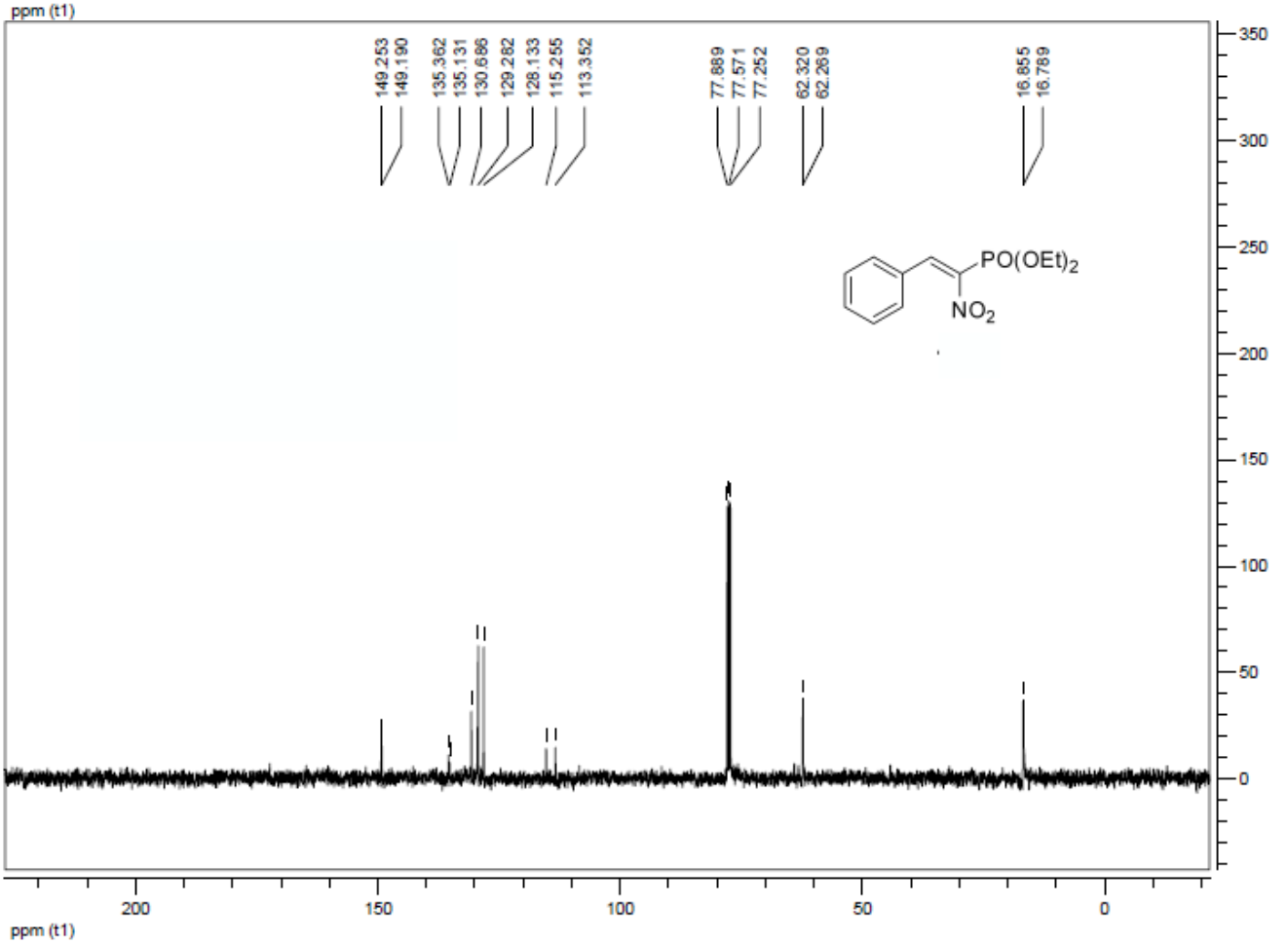
7.505
7.500
7.493
7.485
7.456
7.450
7.373
7.367
7.285
6.302
6.296
6.258
6.252
6.214
6.208
4.173
4.165
4.157
4.149
4.125
4.101
4.093
4.085
1.366
1.360
1.349
1.343
1.331
1.325
PO(OEt)2
NO2
2.58
2.92
0.96
4.00
6.26
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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135.362
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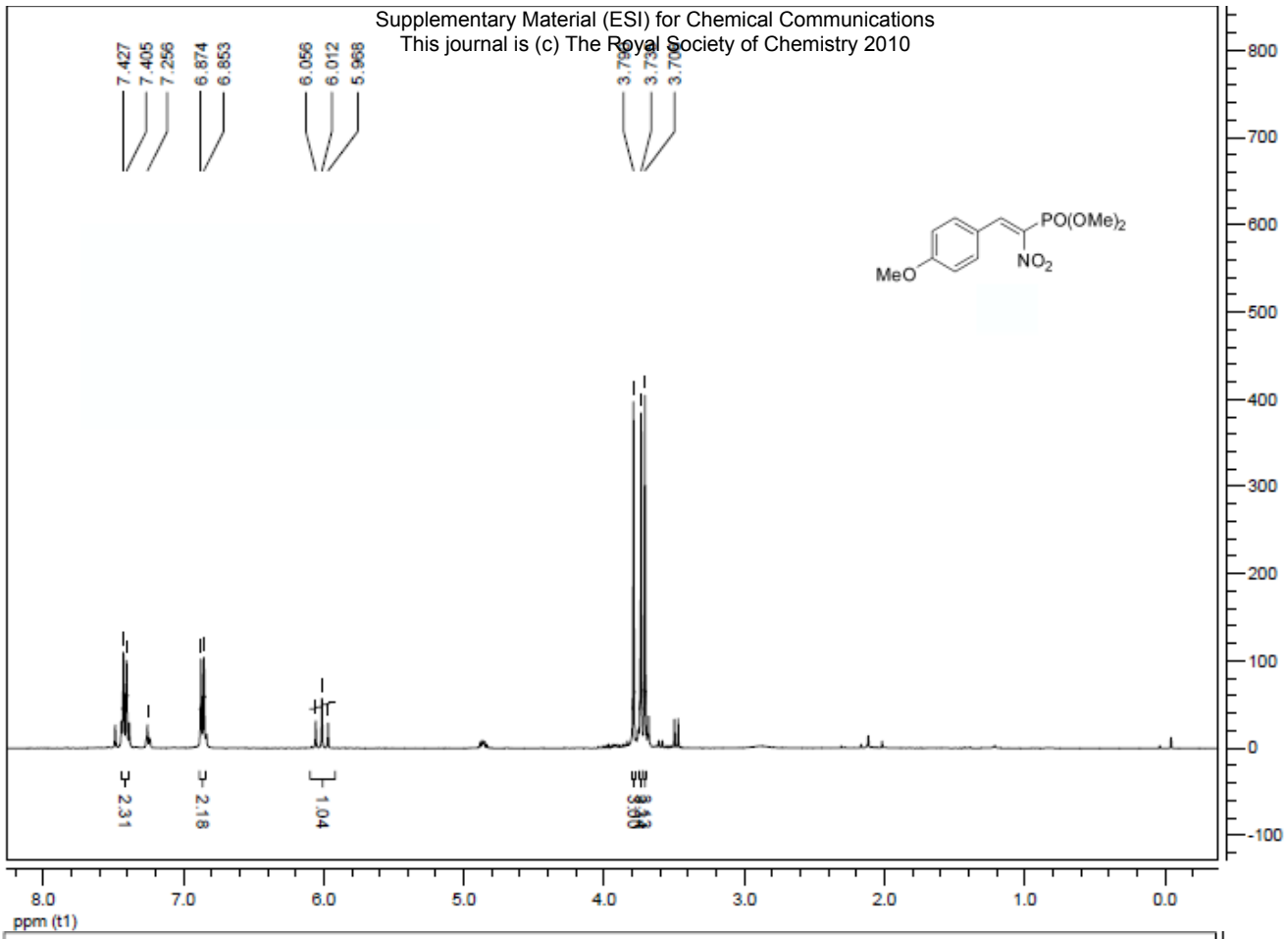
130.686
129.282
128.133
115.255
113.352
77.889
77.571
77.252
62.320
62.269
16.855
16.789
PO(OEt)2
NO2
350
300
250
200
150
100
50
0
200
150
100
50
0
ppm (t1)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6.853
6.056
6.012
5.968
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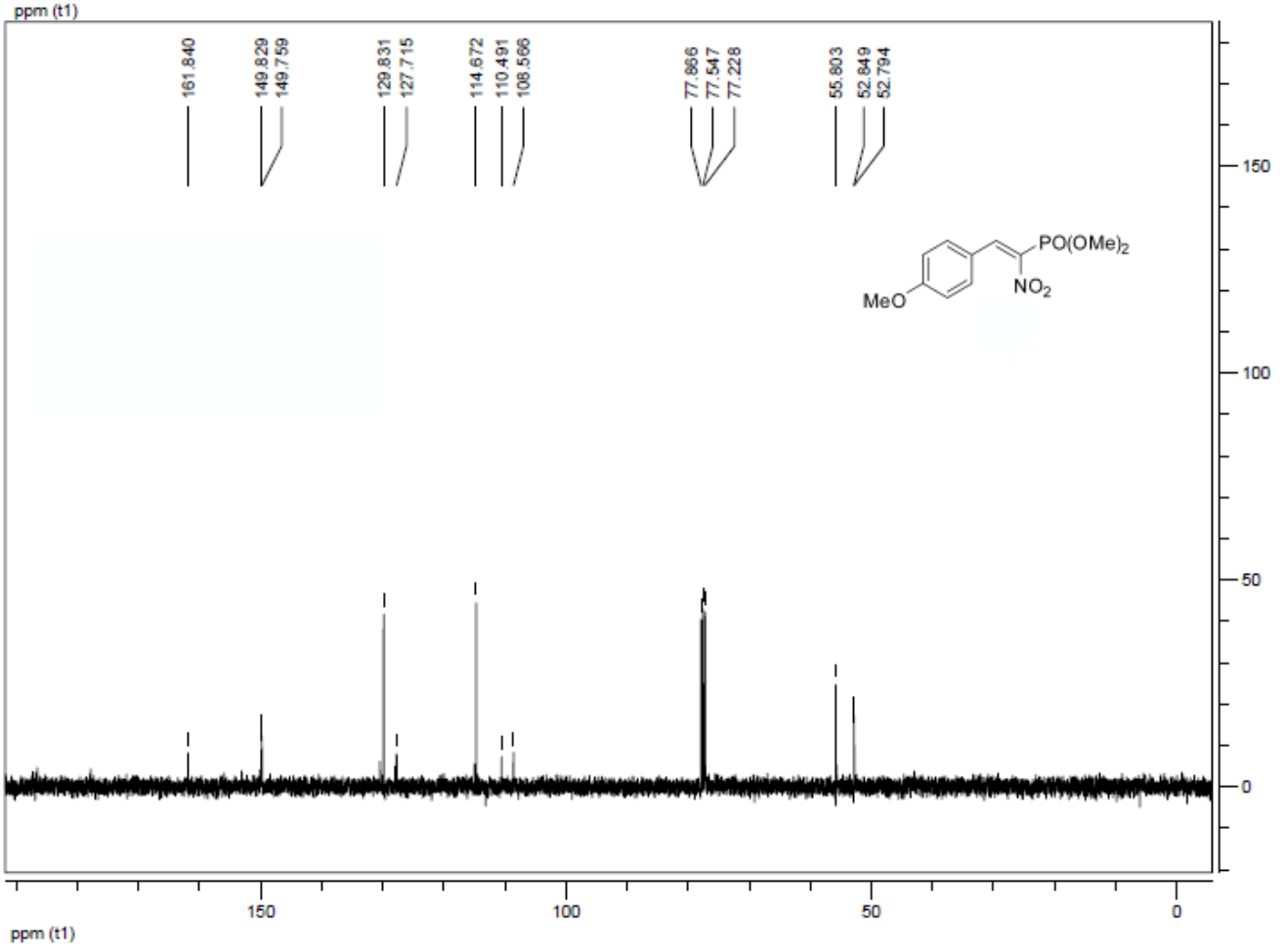
3.706
PO(OMe)2
NO2
MeO
2.31
2.18
1.04
6.00
ppm (t1)


161.840
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149.759
129.831
127.715
114.672
110.491
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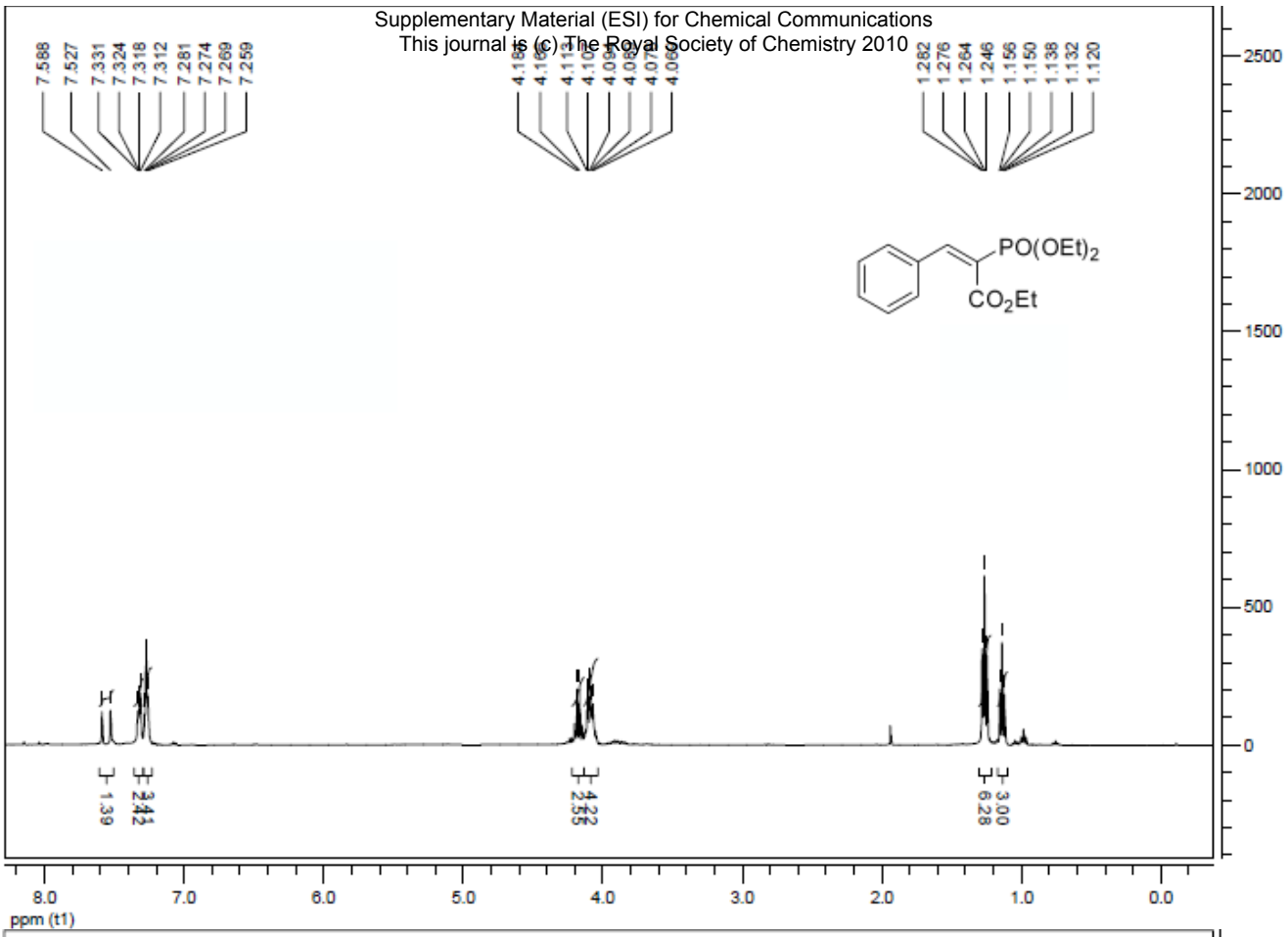
77.547
77.228
55.803
52.849
52.794
PO(OMe)2
NO2
MeO
ppm (t1)


7.588
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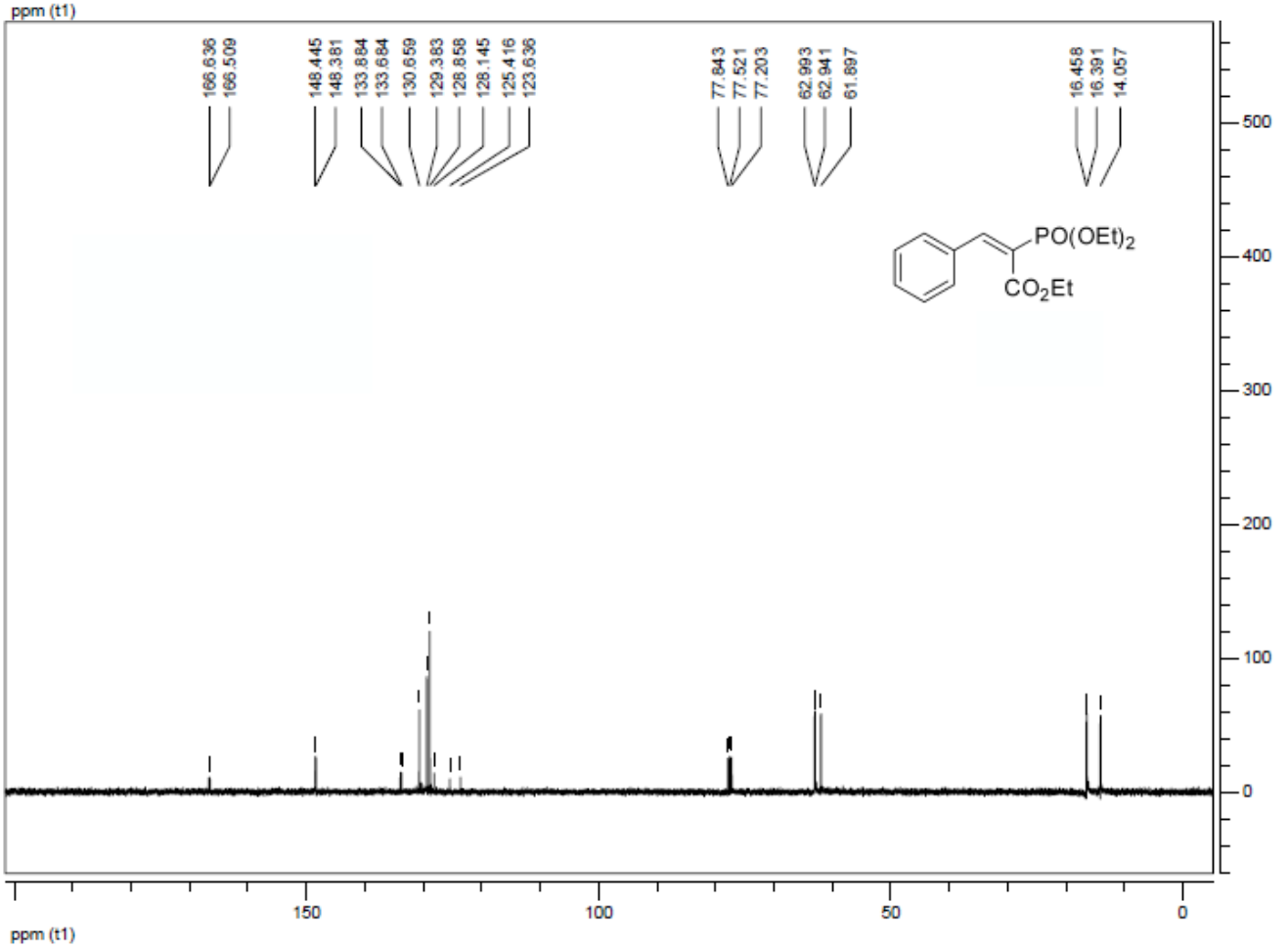
7.318
7.312
7.281
7.274
7.269
7.259
4.186
4.182
4.113
4.107
4.092
4.082
4.075
4.066
1.282
1.276
1.264
1.246
1.156
1.150
1.138
1.132
1.120
PO(OEt)2
CO2Et
1.39
2.42
4.22
9.28
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)
2500
2000
1500
1000
500
0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166.509
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133.684
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129.383
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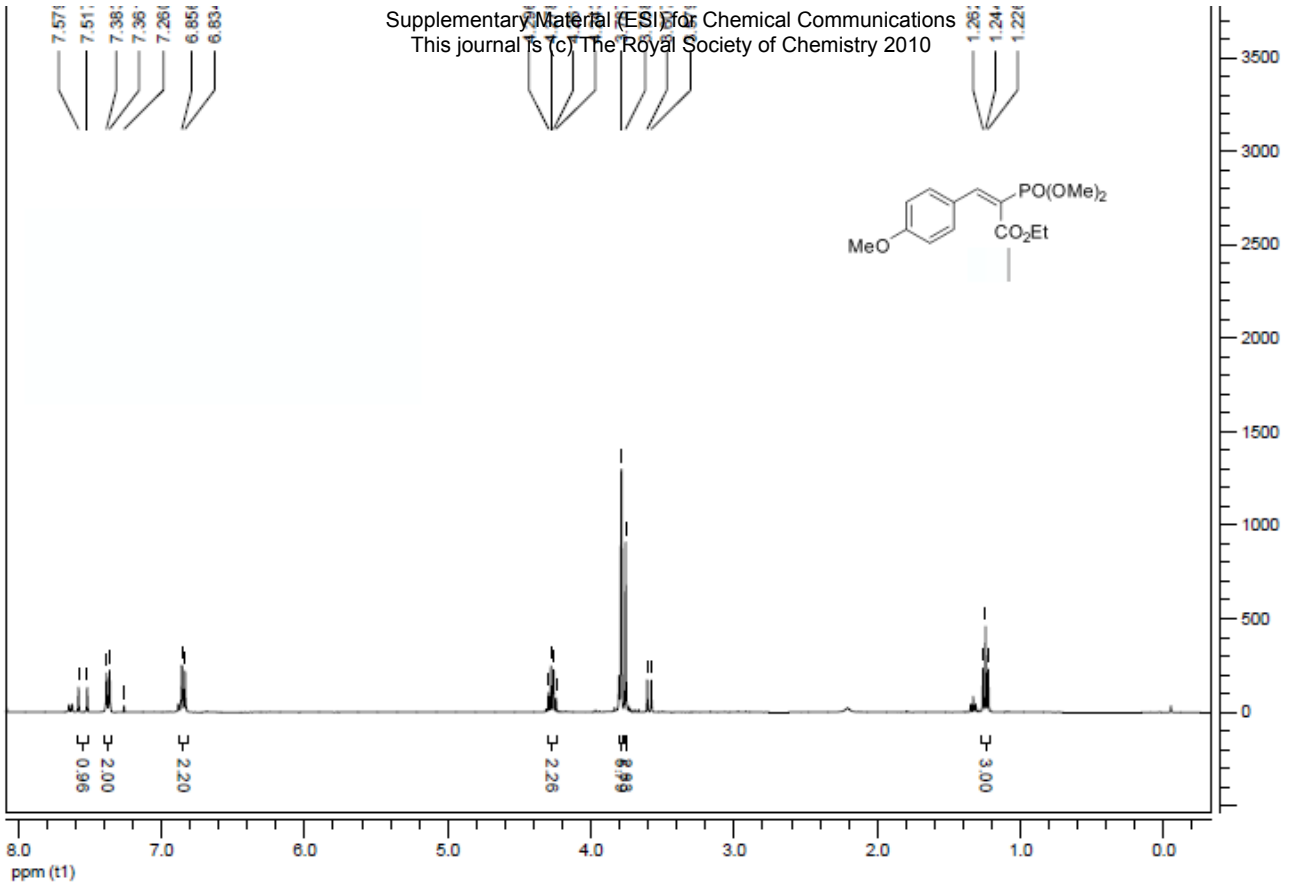
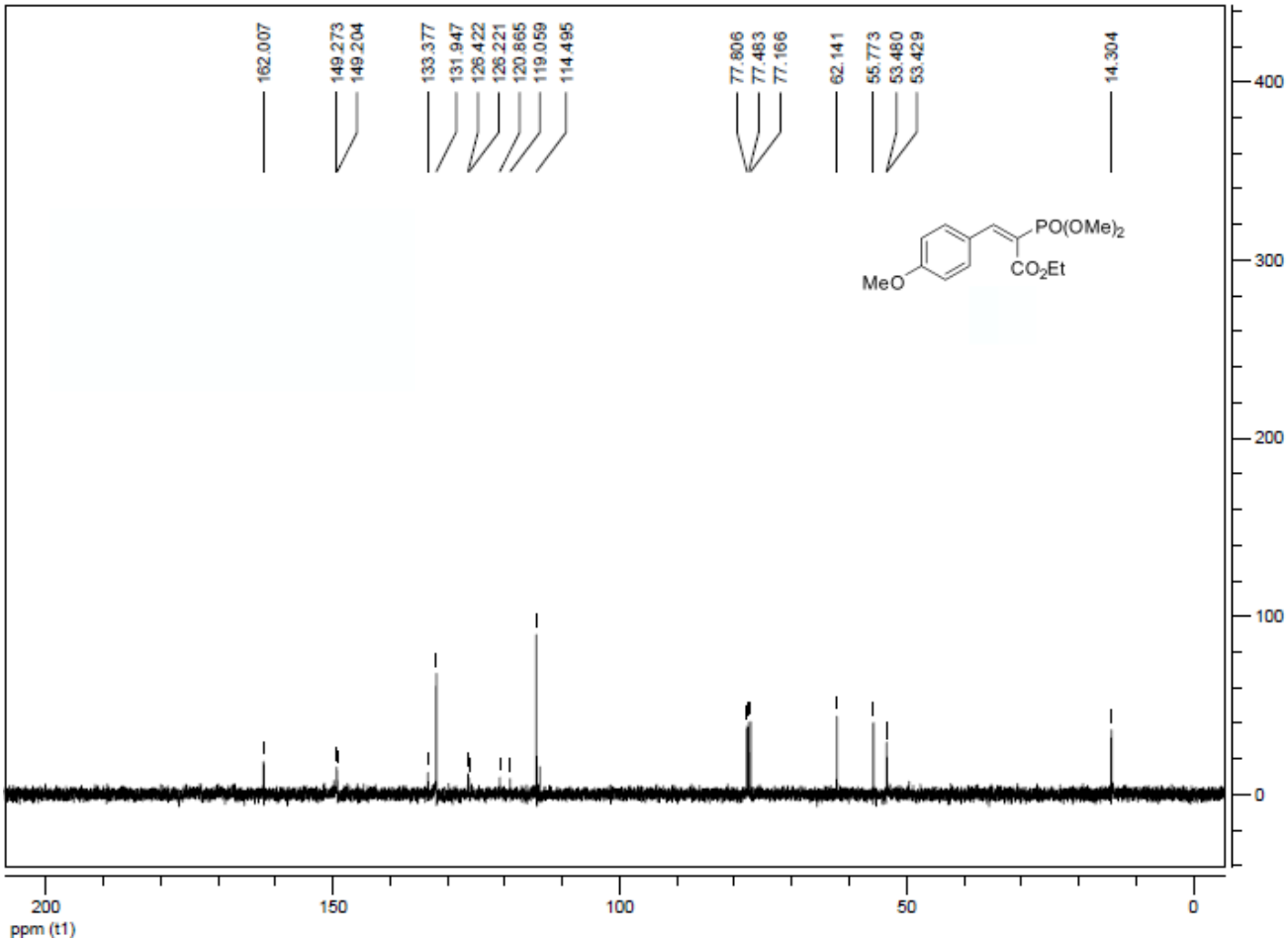
128.145
125.416
123.636
77.843
77.521
77.203
62.993
62.941
61.897
16.458
16.391
14.057
PO(OEt)2
CO2Et
150
100
50
0
ppm (t1)
500
400
300
200
100
0


7.571
7.511
7.388
7.366
7.266
6.858
6.834
1.263
1.244
1.226
PO(OMe)2
CO2Et
MeO
0.96
2.00
2.20
2.26
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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120.865
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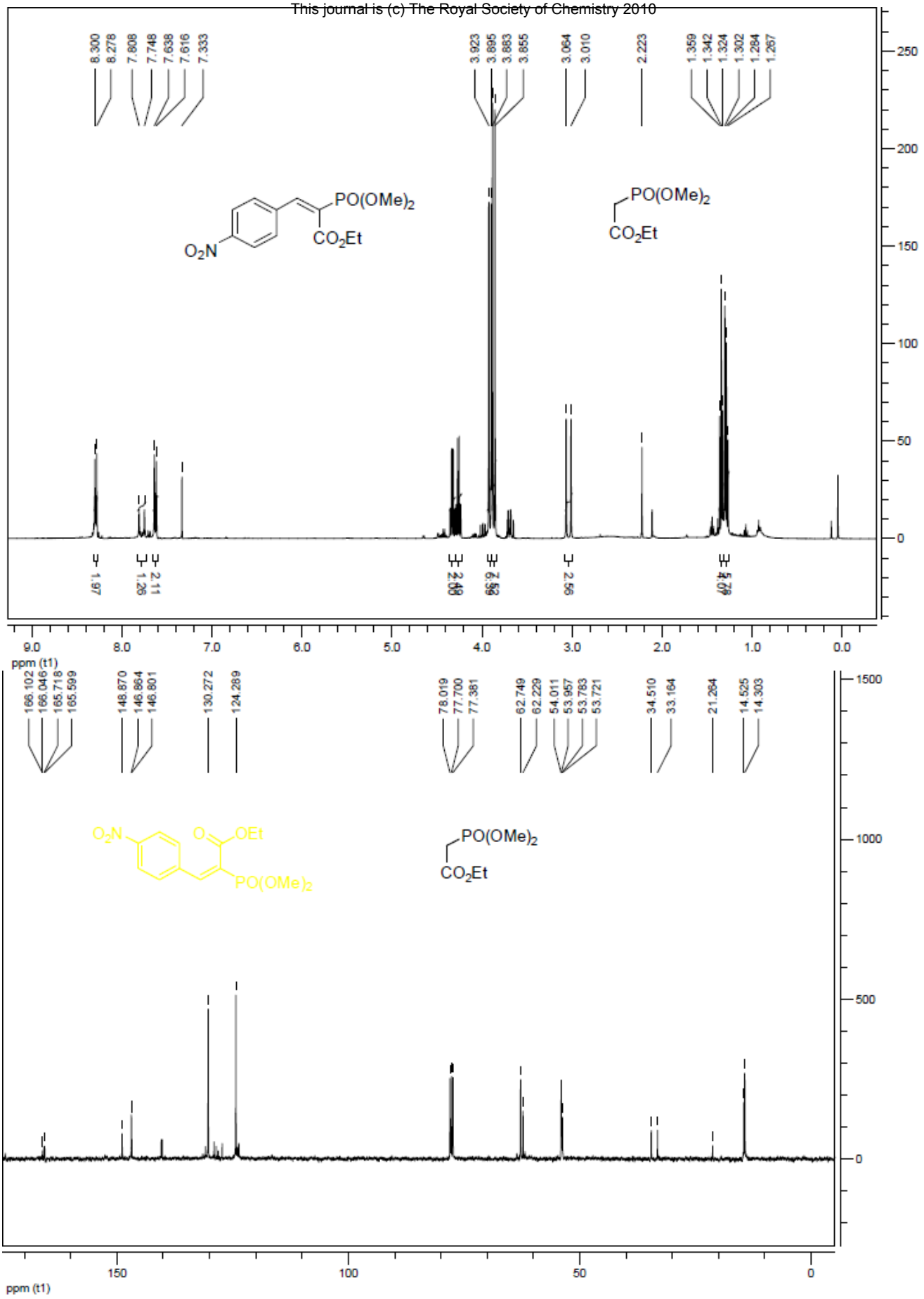
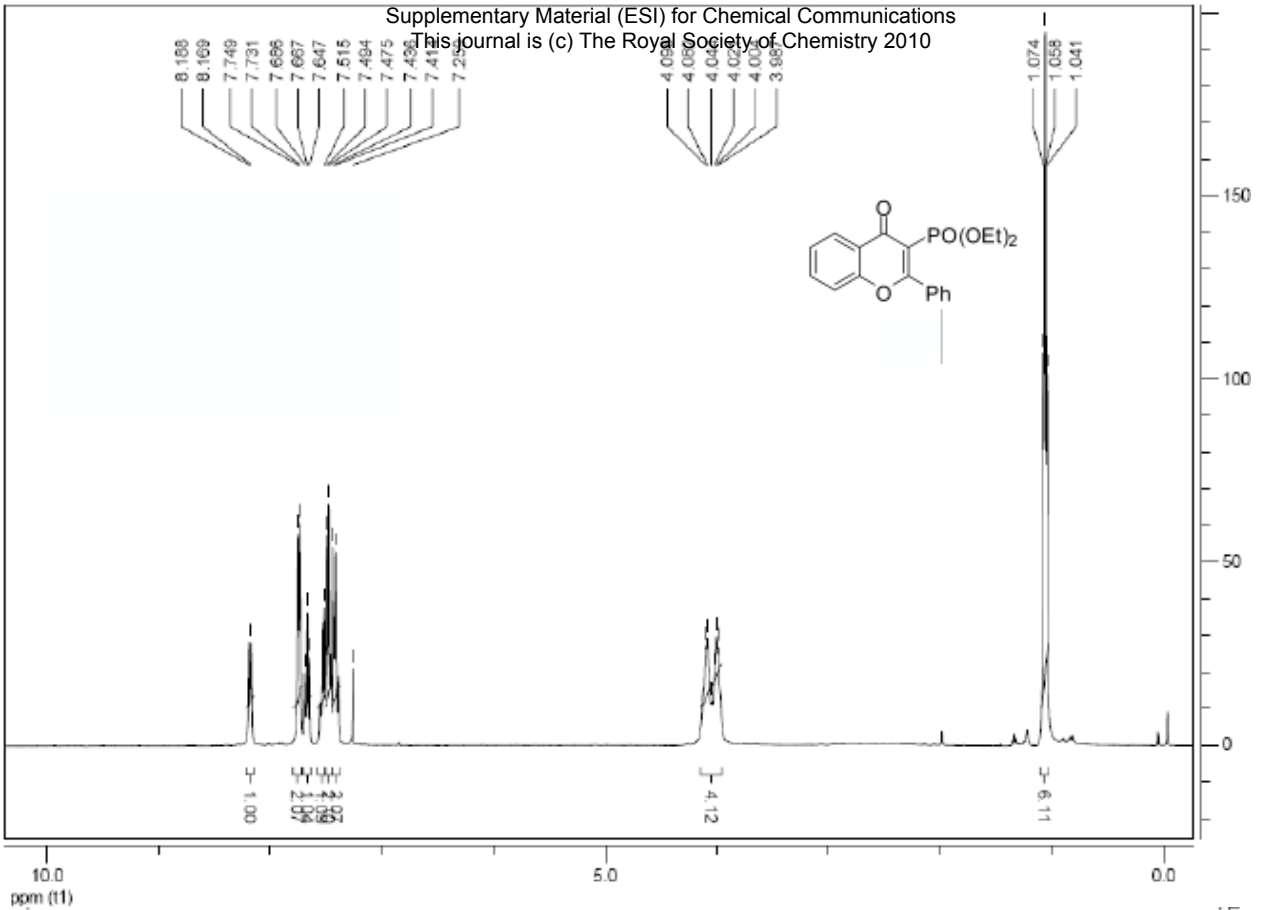
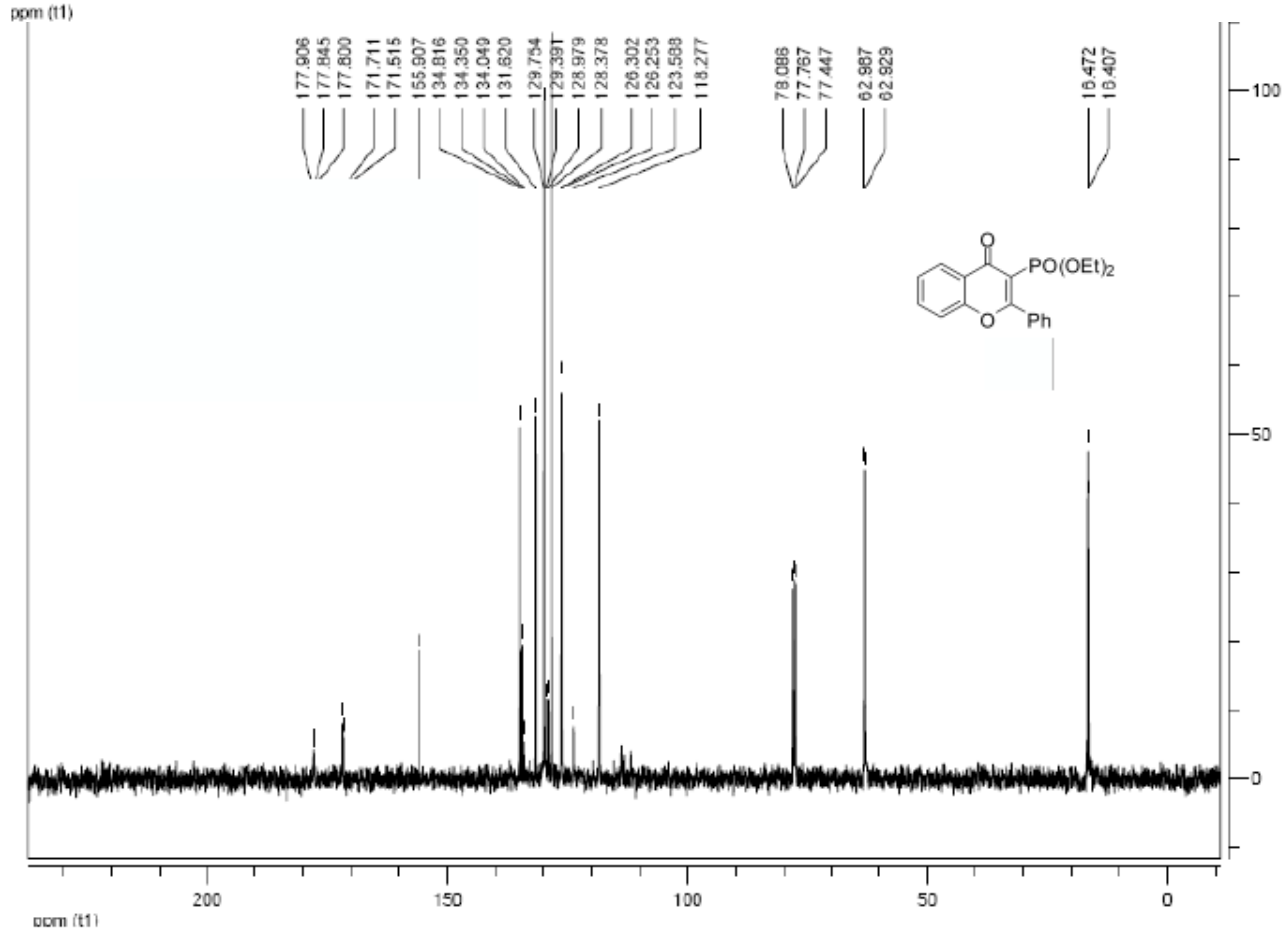
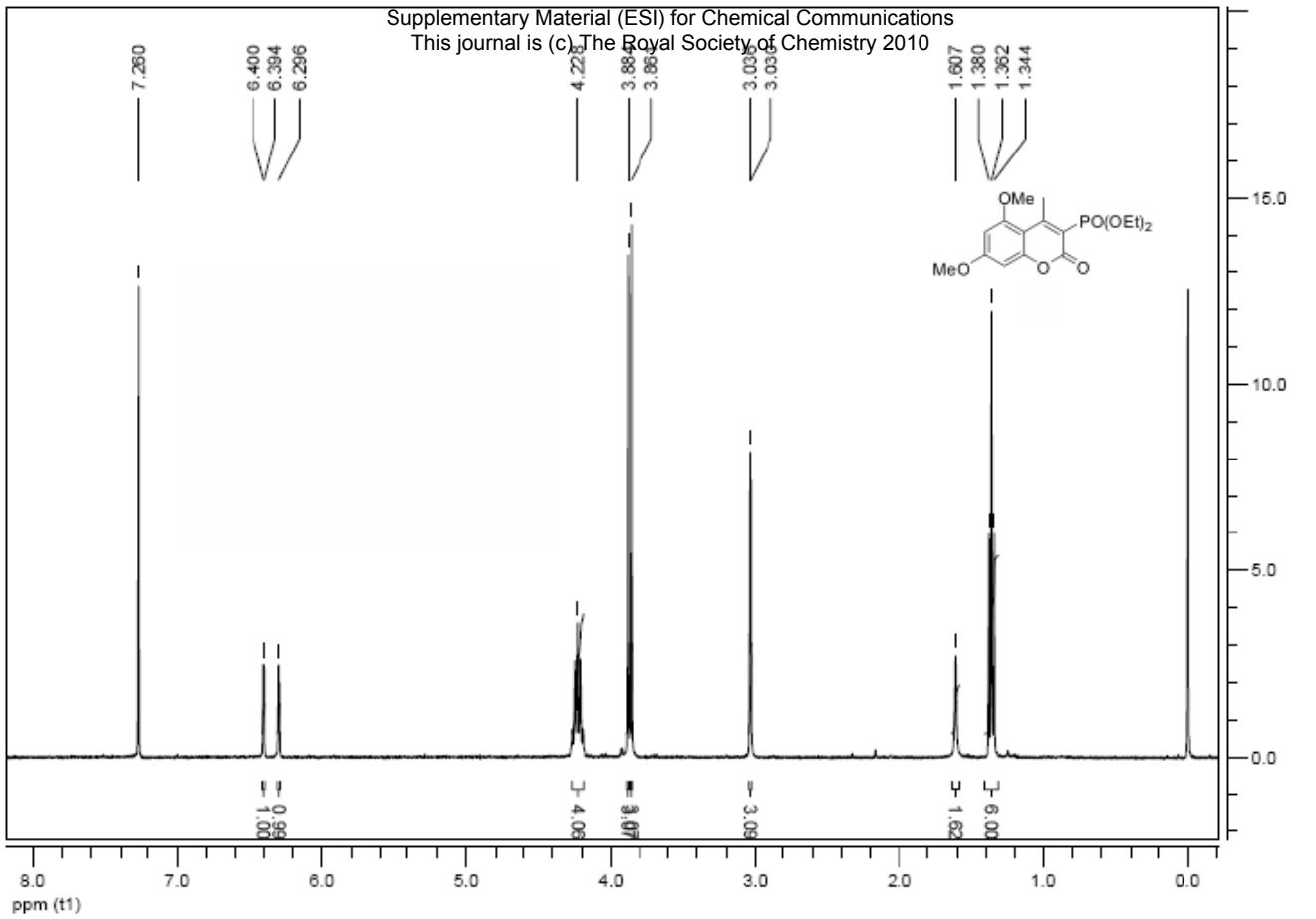
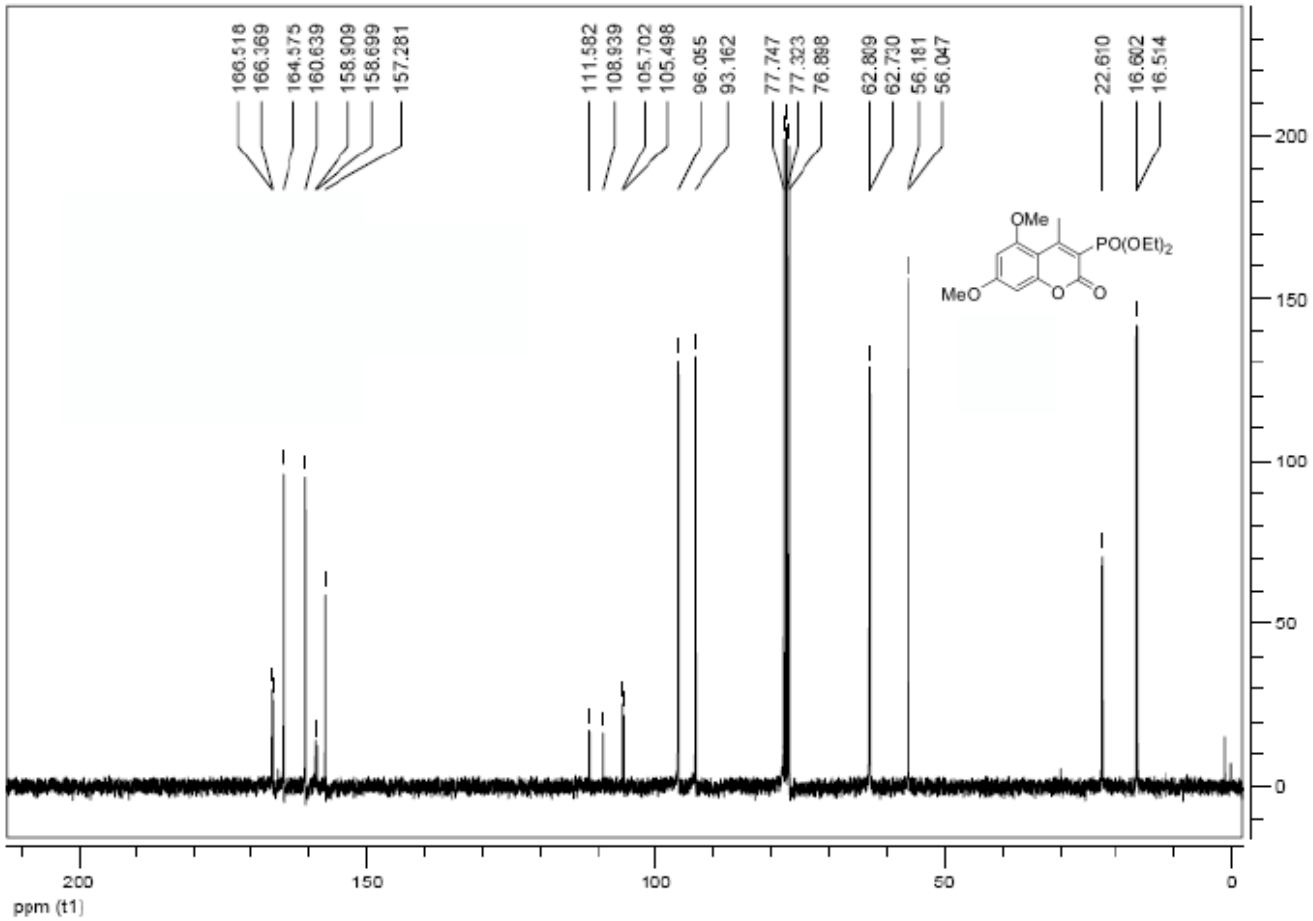
77.166
62.141
55.773
53.480
53.429
14.304
PO(OMe)2
CO2Et
MeO
200
150
100
50
0
ppm (t1)

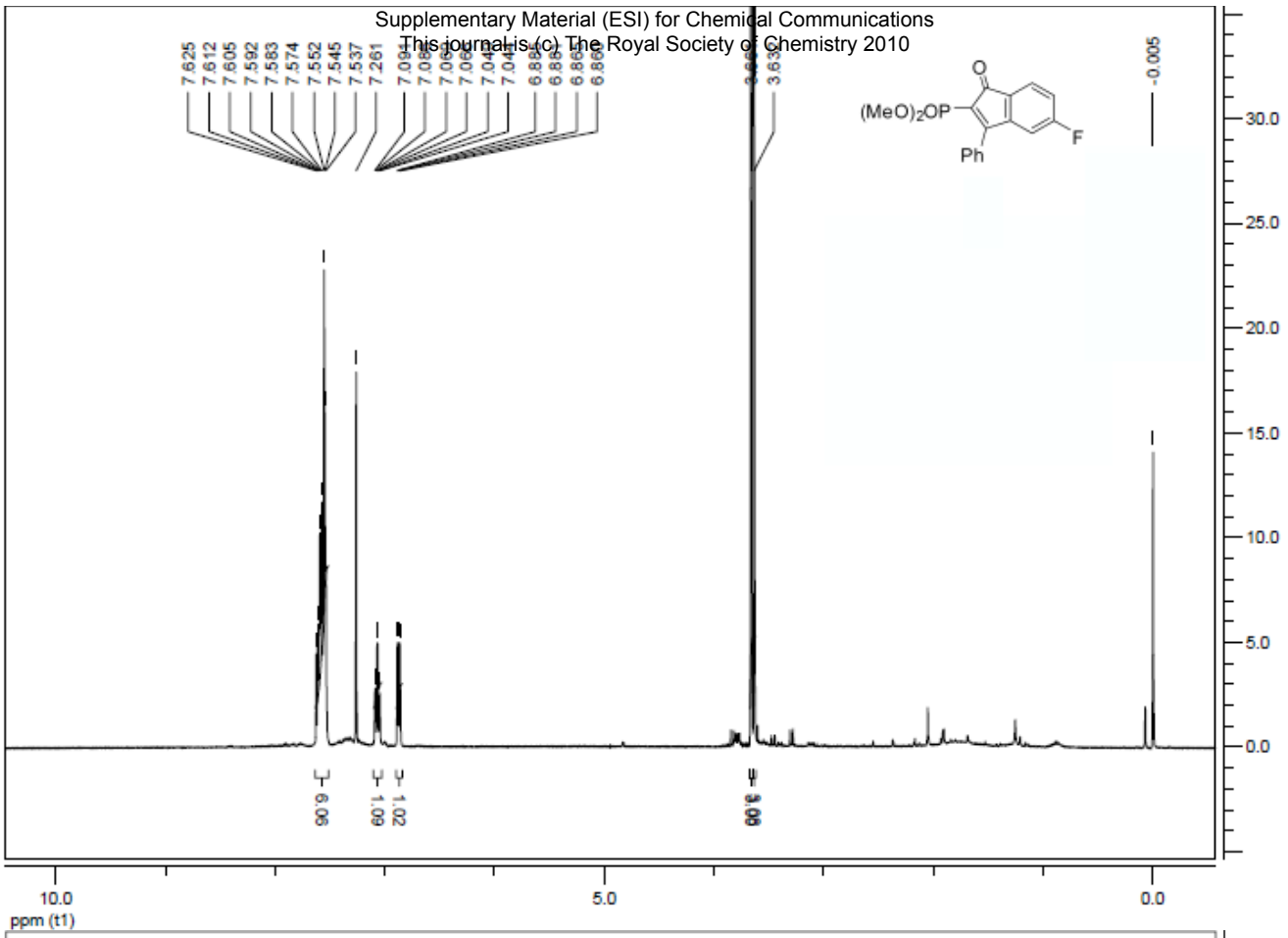

8.300
8.278
7.808
7.748
7.638
7.616
7.333
3.923
3.895
3.883
3.855
3.064
3.010
2.223
1.359
1.342
1.324
1.302
1.284
1.257
PO(OMe)2
CO2Et
O2N
ppm (t1)
166.102
166.046
165.718
165.599
148.870
146.864
146.801
130.272
124.289
78.019
77.700
77.381
62.749
62.229
54.011
53.957
53.783
53.721
34.510
33.164
21.264
14.525
14.303
OEt
O
ppm (t1)


8.168
8.169
7.749
7.731
7.686
7.667
7.647
7.515
7.494
7.475
7.436
7.415
7.259
4.098
4.080
4.044
4.026
4.008
3.989
1.074
1.058
1.041
PO(OEt)2
Ph
1.00
2.12
2.04
2.09
2.02
4.12
6.11
10.0
5.0
0.0
ppm (t1)


177.906
177.845
177.800
171.711
171.515
155.907
134.816
134.350
134.049
131.620
129.794
129.391
128.979
128.378
126.302
126.253
123.588
118.277
78.086
77.767
77.447
62.987
62.929
16.472
16.407
PO(OEt)2
Ph
200
150
100
50
0
ppm (t1)


7.260
6.400
6.394
6.296
4.228
3.884
3.863
3.038
3.034
1.607
1.380
1.362
1.344
OMe
PO(OEt)2
MeO
O
O
15.0
10.0
5.0
0.0
1.00
0.99
4.06
3.07
3.09
1.62
6.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


166.516
166.369
164.575
160.639
158.909
158.699
157.281
111.582
108.939
105.702
105.498
96.055
93.162
77.747
77.323
76.898
62.809
62.730
56.181
56.047
22.610
16.602
16.514
OMe
PO(OEt)2
MeO
O
O
200
150
100
50
0
200
150
100
50
0
ppm (t1)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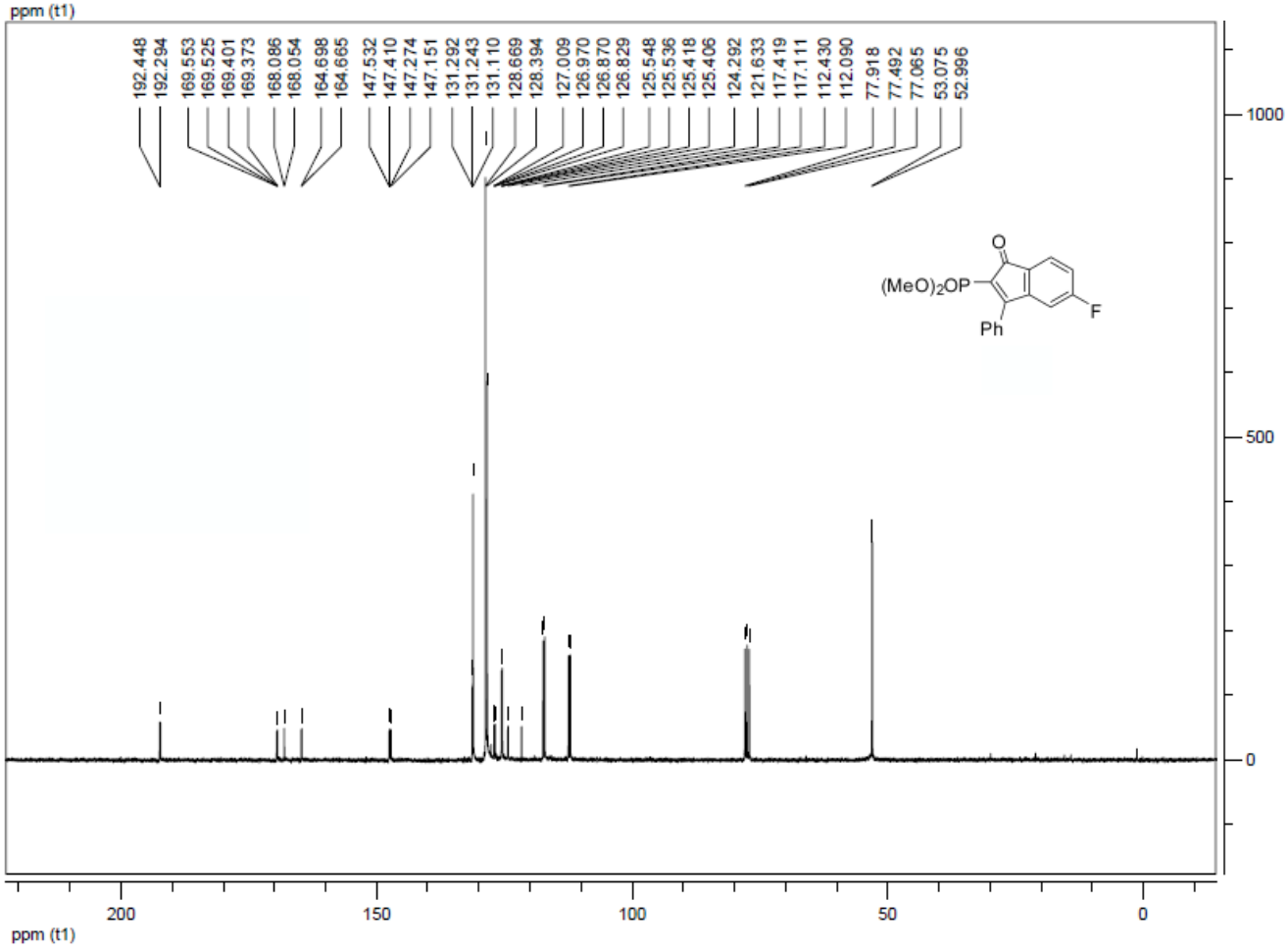
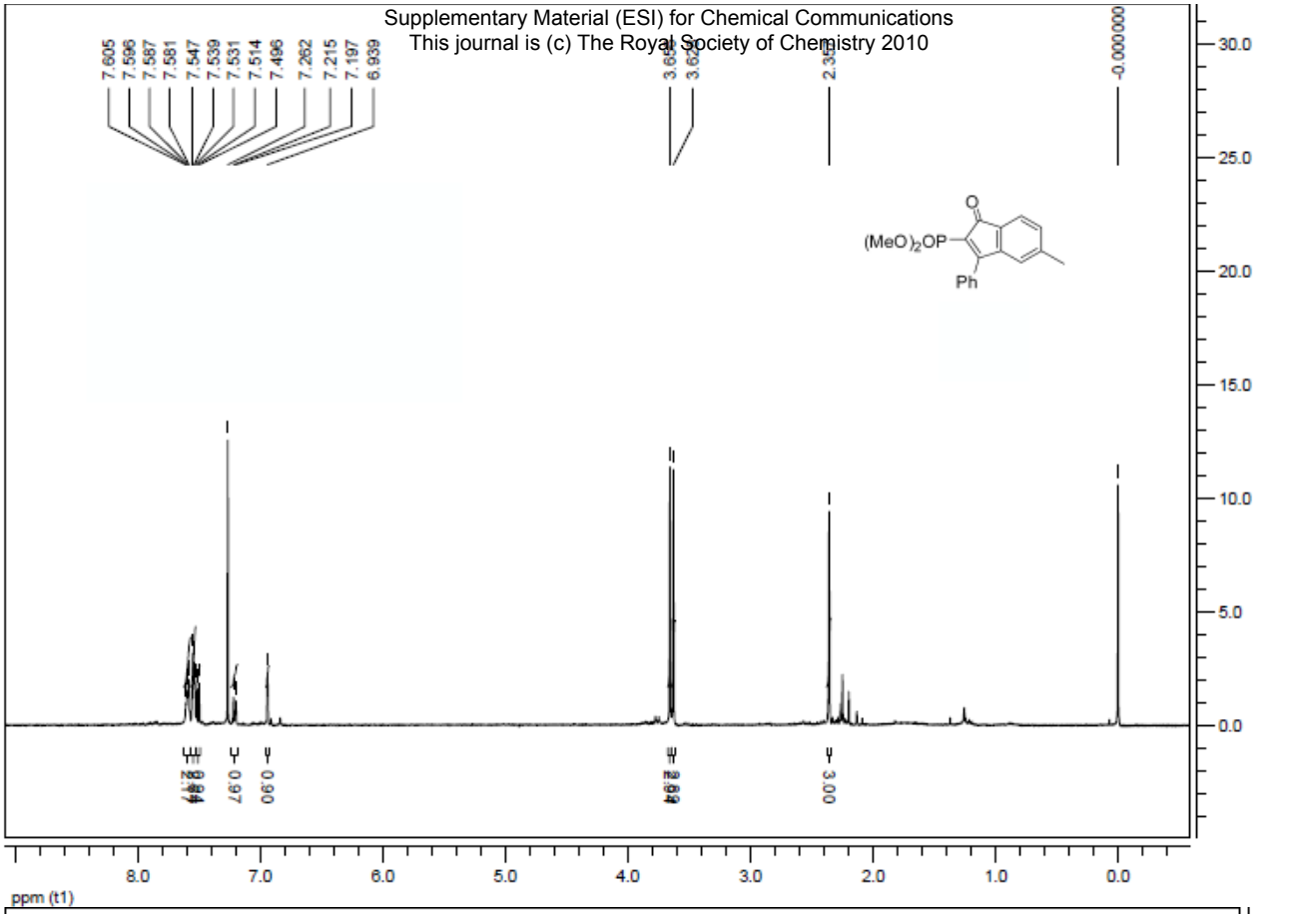
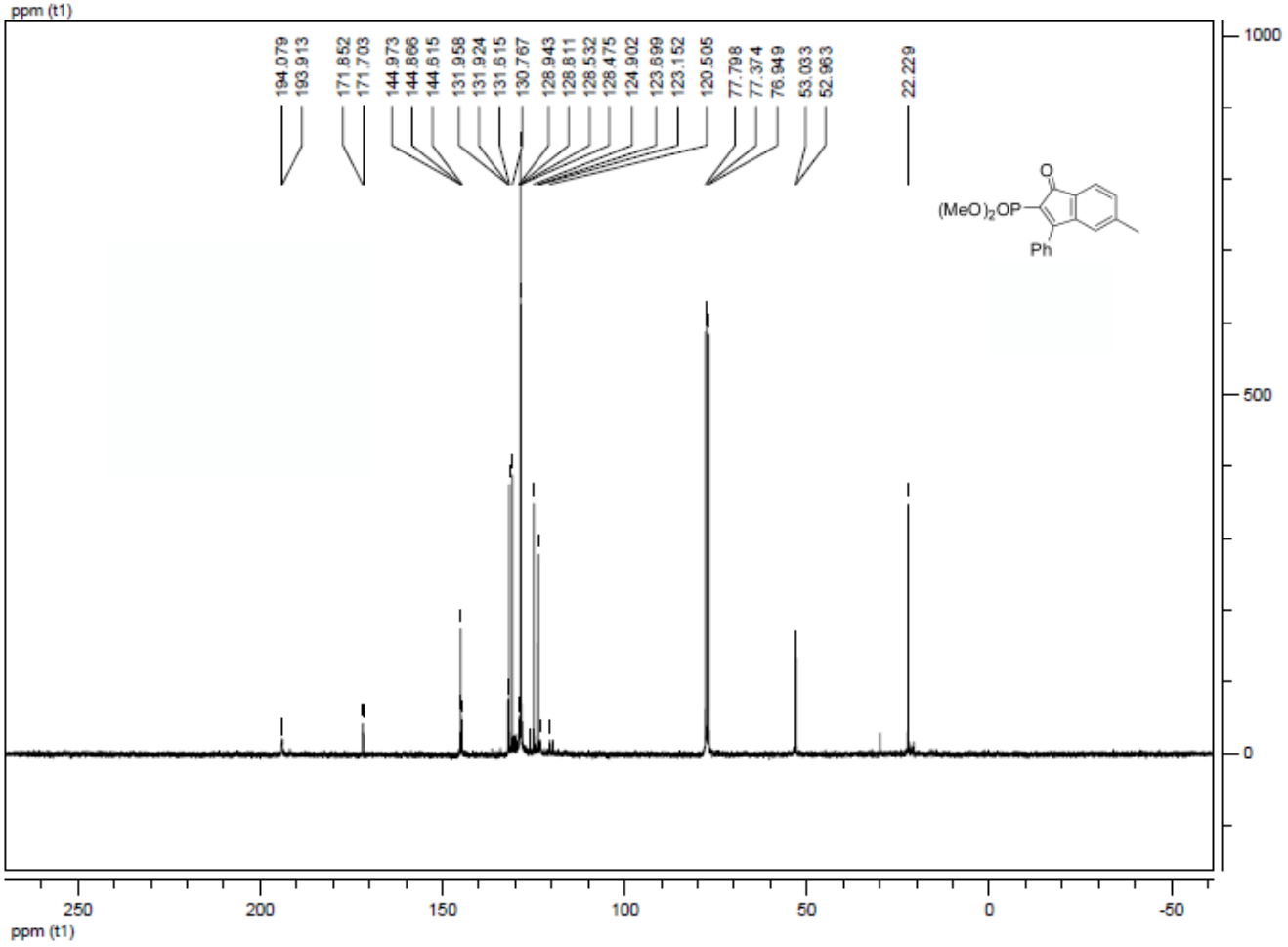
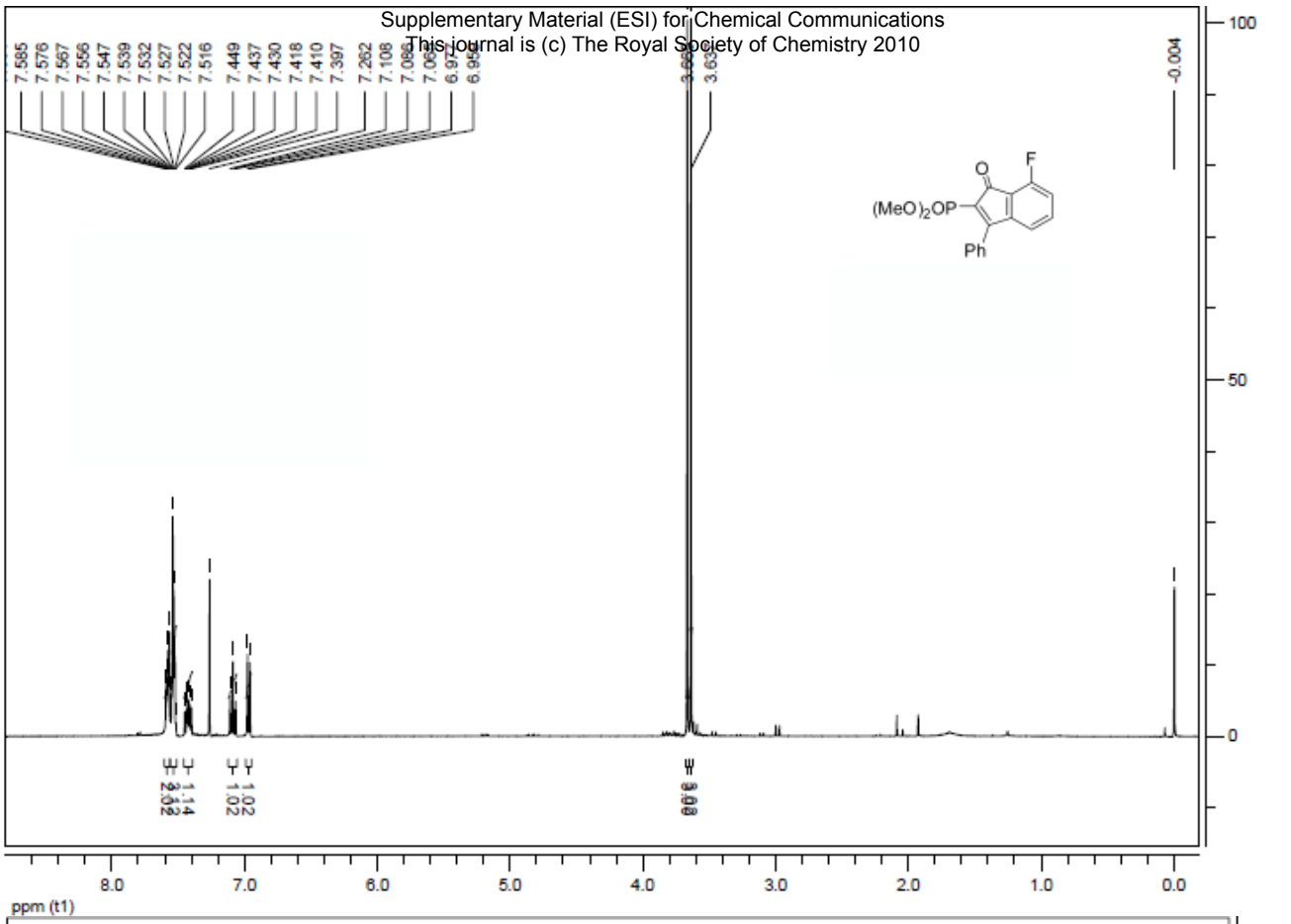
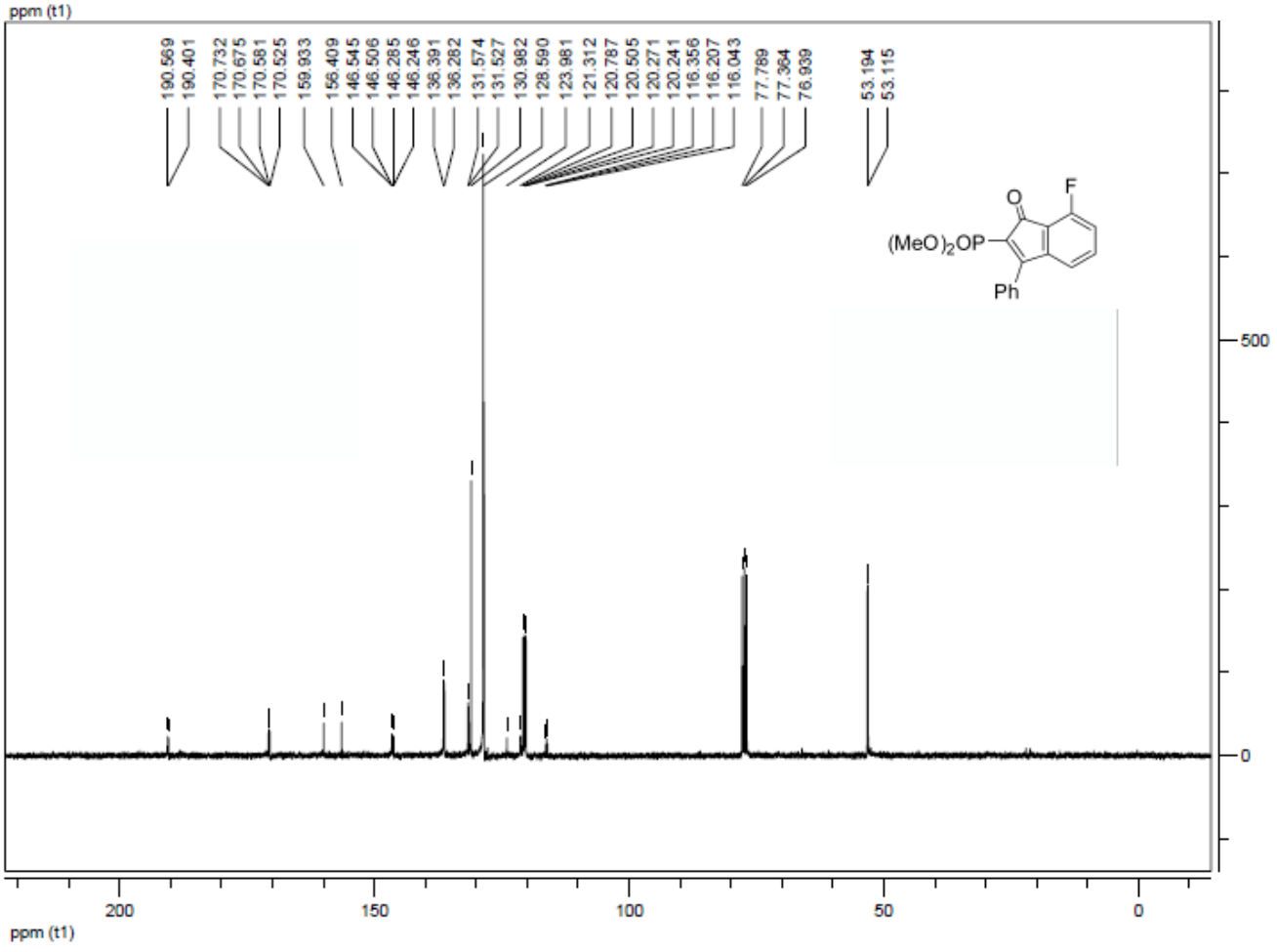
7.612
7.605
7.592
7.583
7.574
7.552
7.545
7.537
7.261
7.094
7.088
7.066
7.060
7.047
7.041
6.884
6.881
6.865
6.860
3.665
3.639
-0.005
(MeO)2OP
O
Ph
F
6.06
1.09
1.02
3.06
30.0
25.0
20.0
15.0
10.0
5.0
0.0
10.0
5.0
0.0
ppm (t1)


192.448
192.294
169.553
169.525
169.401
169.373
168.086
168.054
164.698
164.665
147.532
147.410
147.274
147.151
131.292
131.243
131.110
128.669
128.394
127.009
126.970
126.870
126.829
125.548
125.536
125.418
125.406
124.292
121.633
117.419
117.111
112.430
112.090
77.918
77.492
77.065
53.075
52.996
(MeO)2OP
O
Ph
F
1000
500
0
200
150
100
50
0
ppm (t1)


7.605
7.596
7.587
7.581
7.547
7.539
7.531
7.514
7.496
7.262
7.215
7.197
6.939
3.655
3.627
2.355
-0.000000
(MeO)2OP
O
Ph
0.97
0.90
2.84
3.00
ppm (t1)


194.079
193.913
171.852
171.703
144.973
144.866
144.615
131.958
131.924
131.615
130.767
128.943
128.811
128.532
128.475
124.902
123.699
123.152
120.505
77.798
77.374
76.949
53.033
52.963
22.229
(MeO)2OP
O
Ph
ppm (t1)


7.585
7.576
7.567
7.556
7.547
7.539
7.532
7.527
7.522
7.516
7.449
7.437
7.430
7.418
7.410
7.397
7.262
7.108
7.086
7.063
6.975
6.956
3.639
-0.004
O
F
(MeO)2OP
Ph
2.02
1.14
1.02
3.00
100
50
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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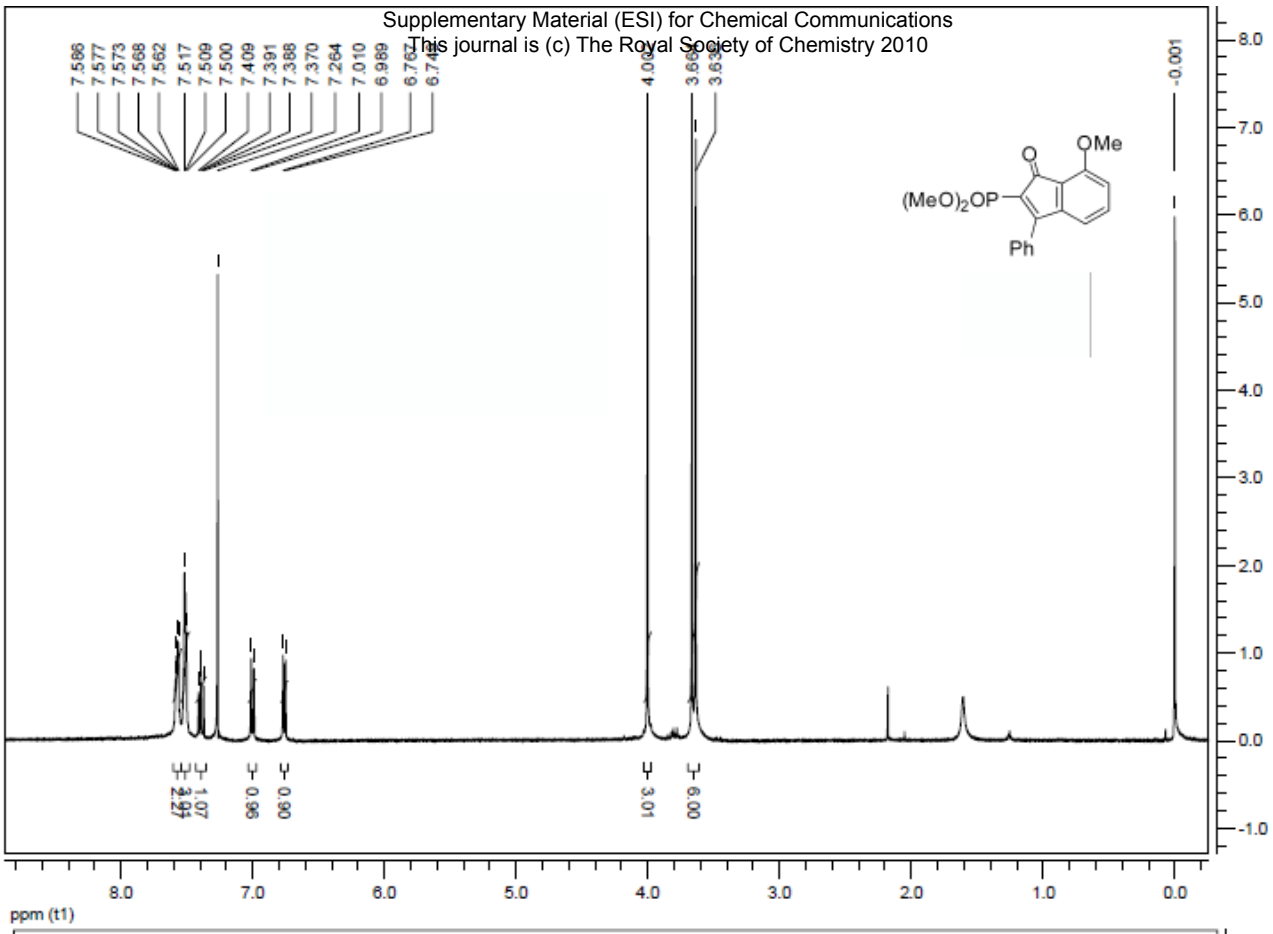
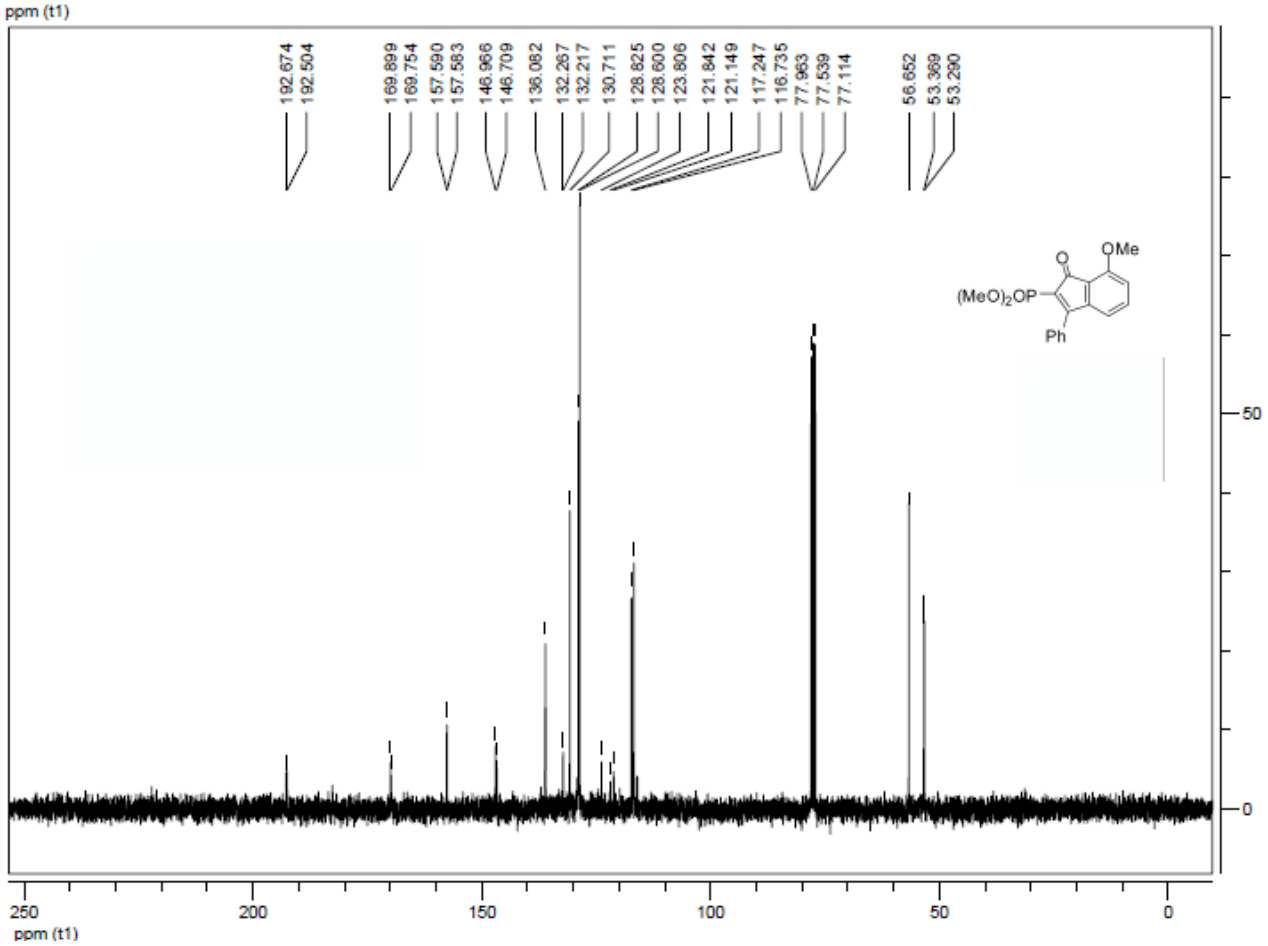
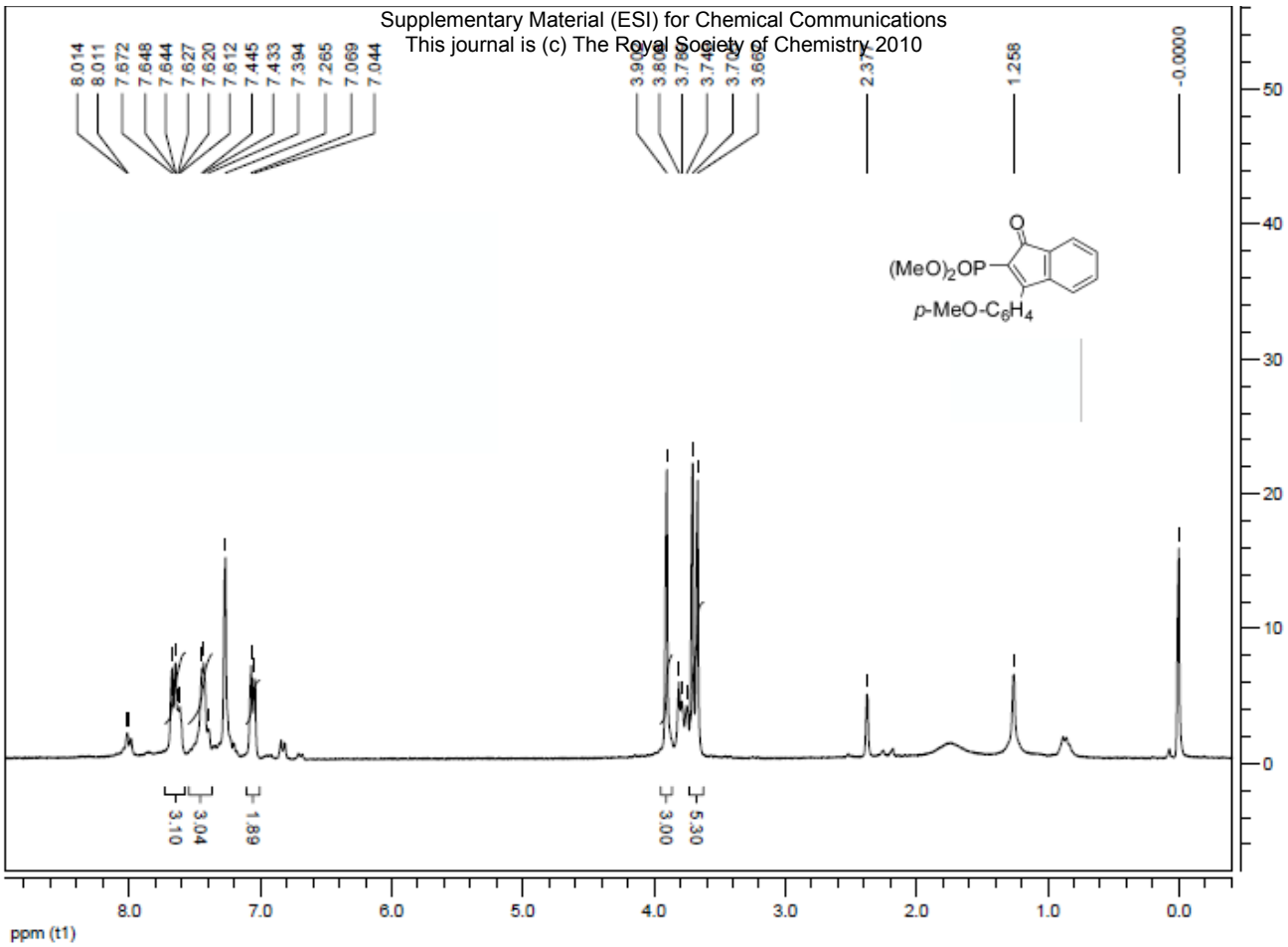
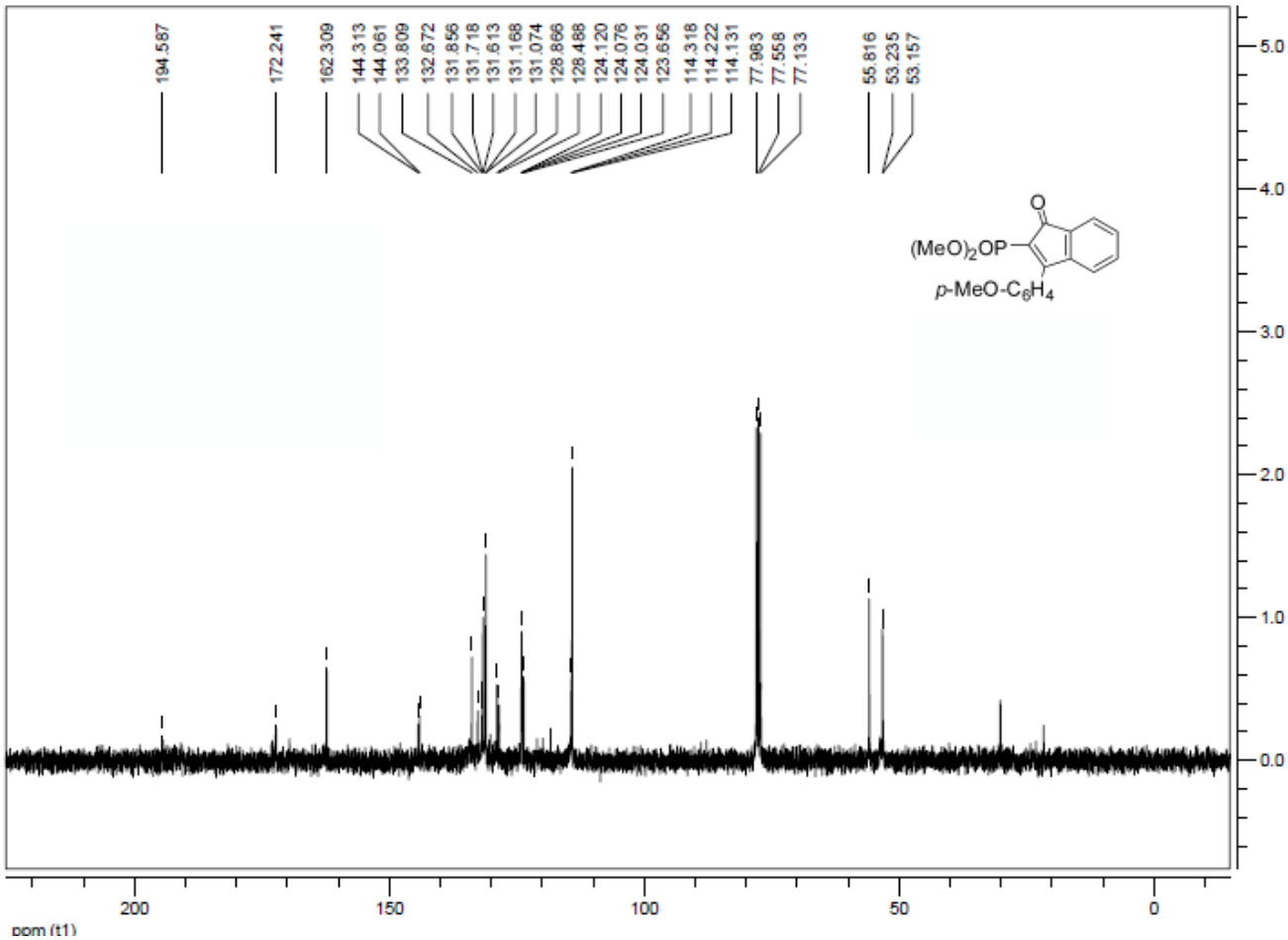
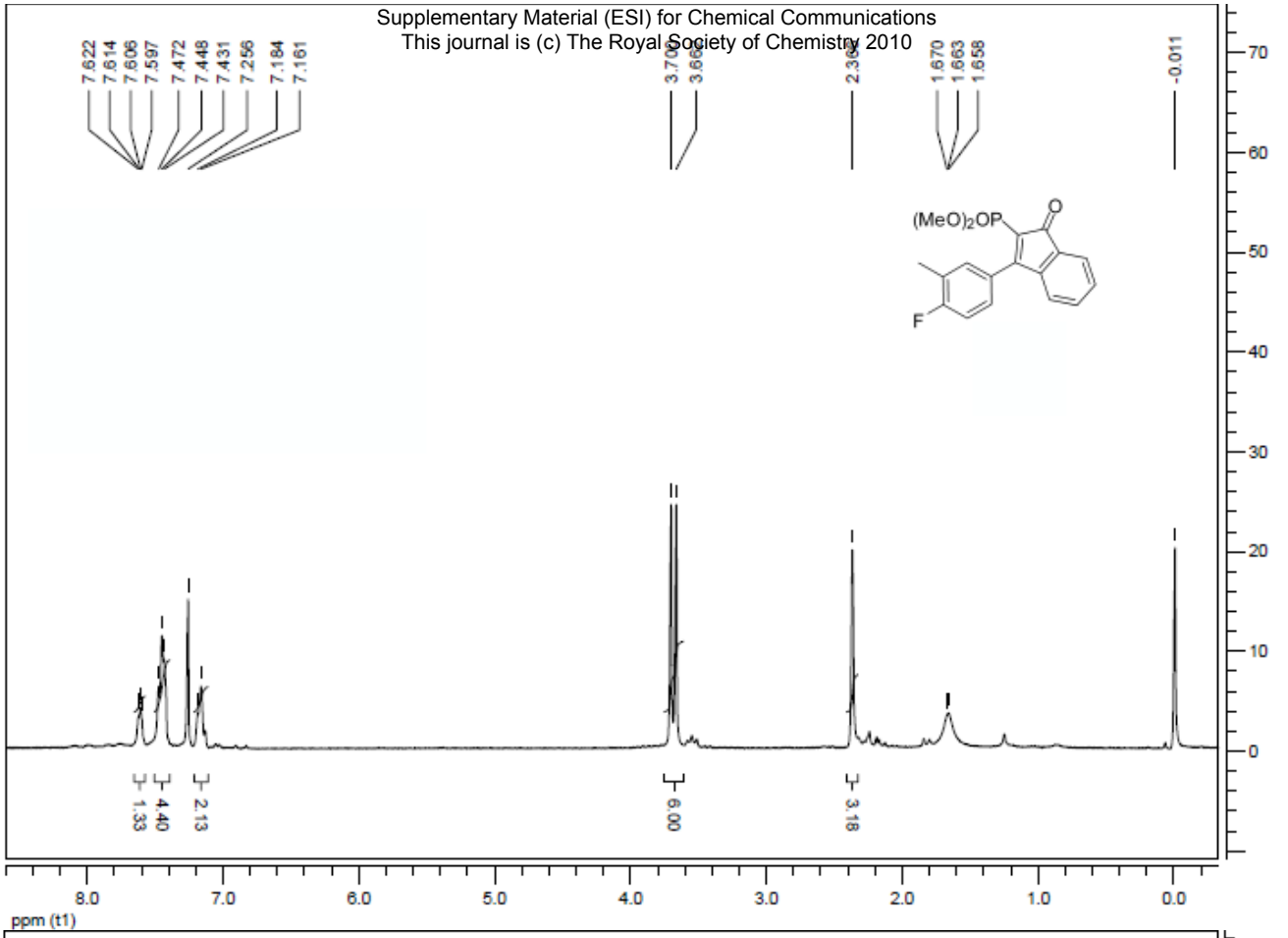
190.401
170.732
170.675
170.581
170.525
159.933
156.409
146.545
146.506
146.285
146.246
136.391
136.282
131.574
131.527
130.982
128.590
123.981
121.312
120.787
120.505
120.271
120.241
116.356
116.207
116.043
77.789
77.364
76.939
53.194
53.115
O
F
(MeO)2OP
Ph
500
0
200
150
100
50
0
ppm (t1)

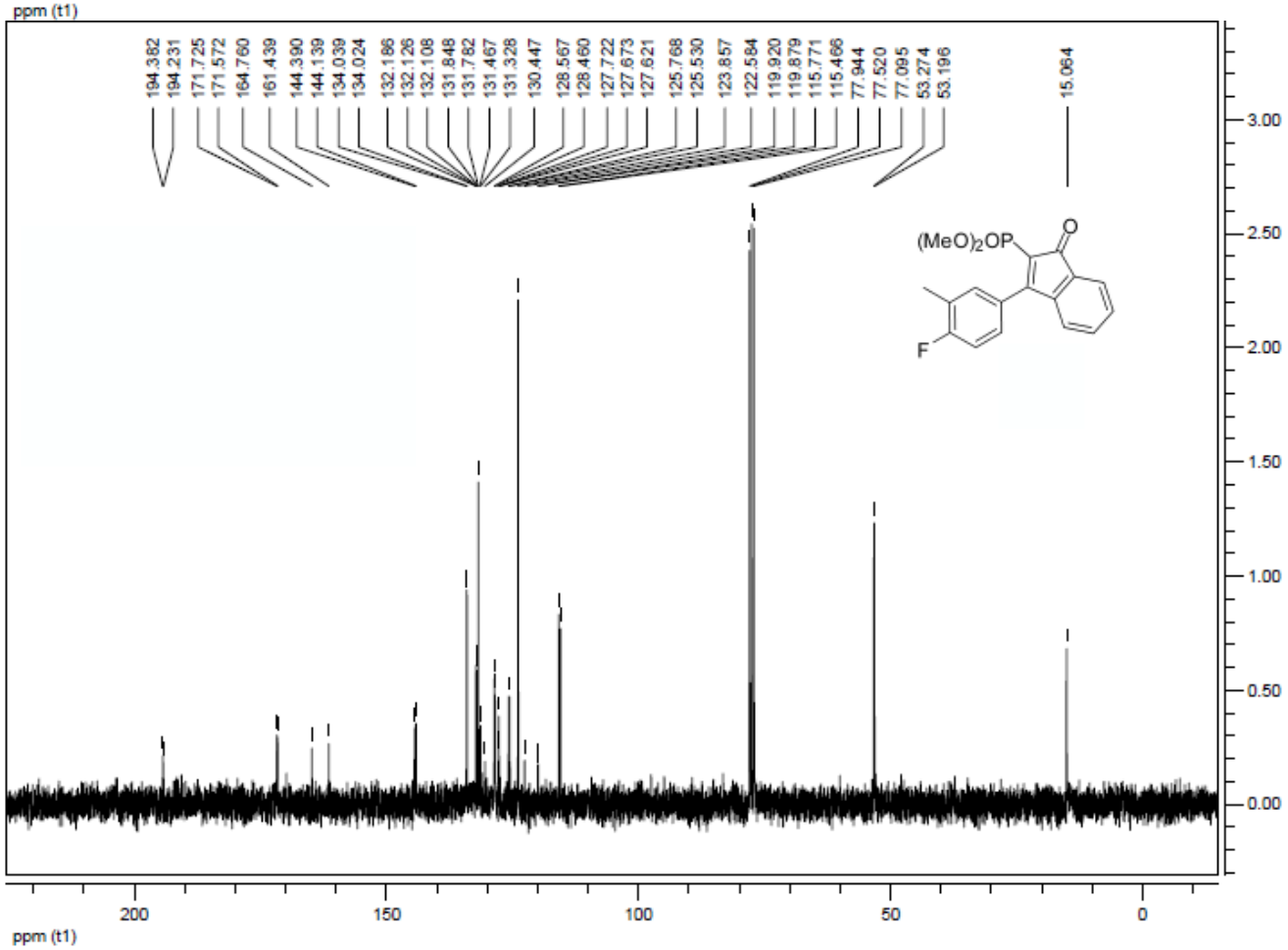

7.586
7.577
7.573
7.568
7.562
7.517
7.509
7.500
7.409
7.391
7.388
7.370
7.264
7.010
6.989
6.762
6.745
4.003
3.668
3.639
-0.001
OMe
O
(MeO)2OP
Ph
1.07
0.96
0.90
3.01
6.00
ppm (t1)


192.674
192.504
169.899
169.754
157.590
157.583
146.966
146.709
136.082
132.267
132.217
130.711
128.825
128.600
123.806
121.842
121.149
117.247
116.735
77.963
77.539
77.114
56.652
53.369
53.290
OMe
O
(MeO)2OP
Ph
ppm (t1)

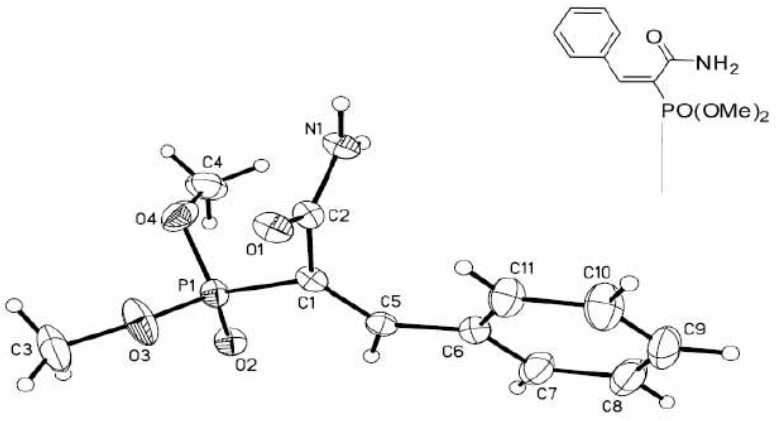

8.014
8.011
7.672
7.648
7.644
7.627
7.620
7.612
7.445
7.433
7.394
7.265
7.069
7.044
3.906
3.802
3.786
3.746
3.703
3.665
2.377
1.258
-0.0000
(MeO)2OP
p-MeO-C6H4
3.10
3.04
1.89
3.00
5.30
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


194.587
172.241
162.309
144.313
144.061
133.809
132.672
131.856
131.718
131.613
131.168
131.074
128.866
128.488
124.120
124.076
124.031
123.656
114.318
114.222
114.131
77.983
77.558
77.133
55.816
53.235
53.157
(MeO)2OP
p-MeO-C6H4
200
150
100
50
0
ppm (t1)


7.622
7.614
7.606
7.597
7.472
7.448
7.431
7.256
7.184
7.161
3.700
3.662
2.359
1.670
1.663
1.658
-0.011
(MeO)2OP
O
F
1.33
4.40
2.13
6.00
3.18
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

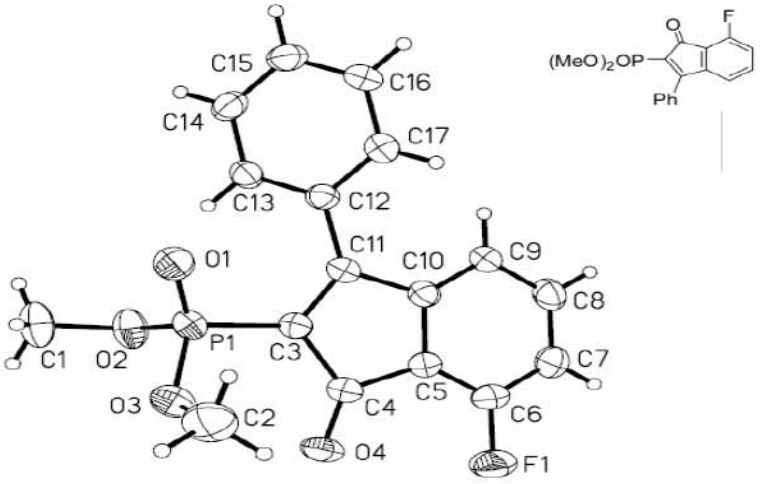

194.382
194.231
171.725
171.572
164.760
161.439
144.390
144.139
134.039
134.024
132.186
132.126
132.108
131.848
131.782
131.467
131.328
130.447
128.567
128.460
127.722
127.673
127.621
125.768
125.530
123.857
122.584
119.920
119.879
115.771
115.466
77.944
77.520
77.095
53.274
53.196
15.064
(MeO)2OP
O
F
200
150
100
50
0
ppm (t1)

X-ray crystal structures



CCDC 743887

Formula: C11 H14 N1 O4 P1;
Unit cell parameters: a 24.732(4) b 9.2832(11) c 18.052(3) beta 105.924(4); space group P21/c



CCDC 743888

Formula: C17 H17 O5 P1; Unit cell parameters: a 10.723(4) b 7.421(2) c 10.609(4) beta 103.992(8); space group P21