Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

C-P bond construction catalyzed by Ni^{II} immobilized on aminated $Fe_3O_4@TiO_2$ yolk-shell NPs functionalized by (3-glycidyloxypropyl)trimethoxysilane ($Fe_3O_4@TiO_2$YS-GLYMO-UNNi^{II}) in green media

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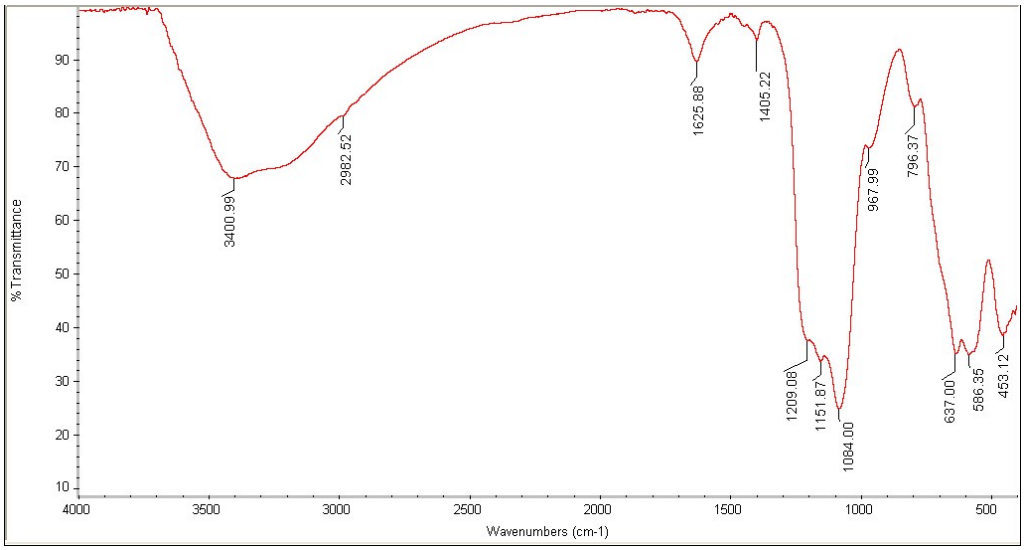

Figure 1: FT-IR (KBr) of Fe_3O_4@SiO_2 (**II**).

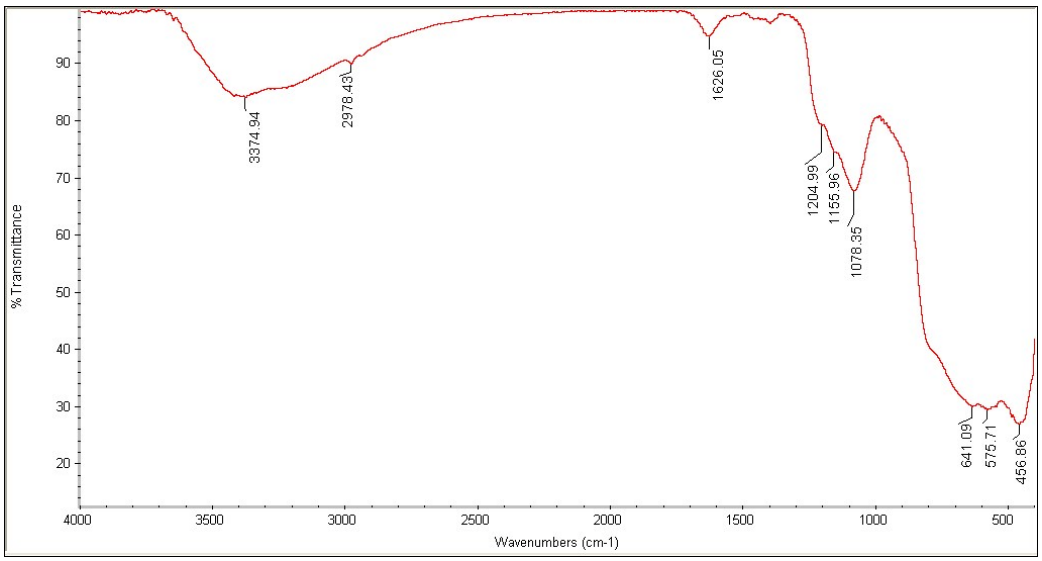

Figure 2: FT-IR (KBr) of Fe_3O_4@SiO_2@TiO_2 (**III**).

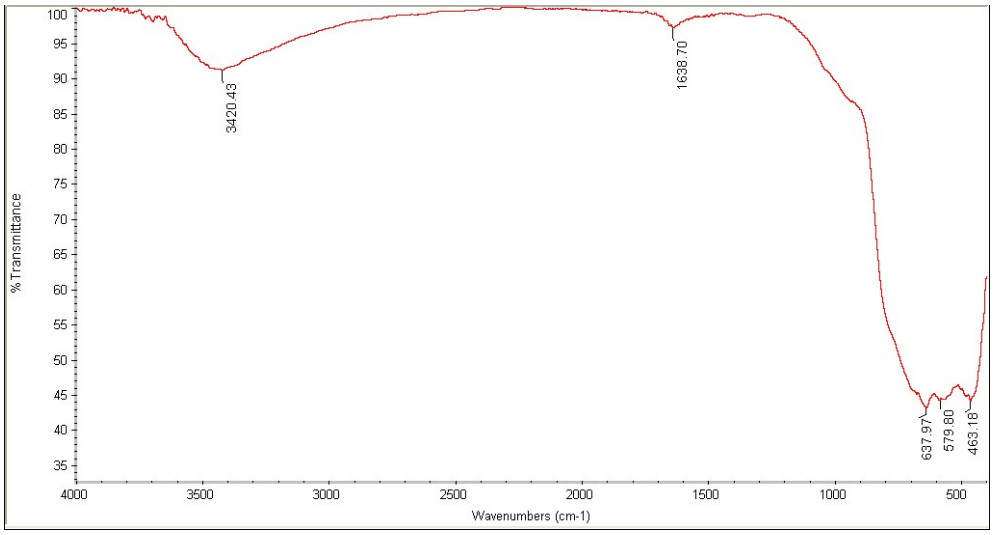

Figure 3: FT-IR (KBr) of Fe_3O_4@TiO_2 yolk-shell (**IV**).

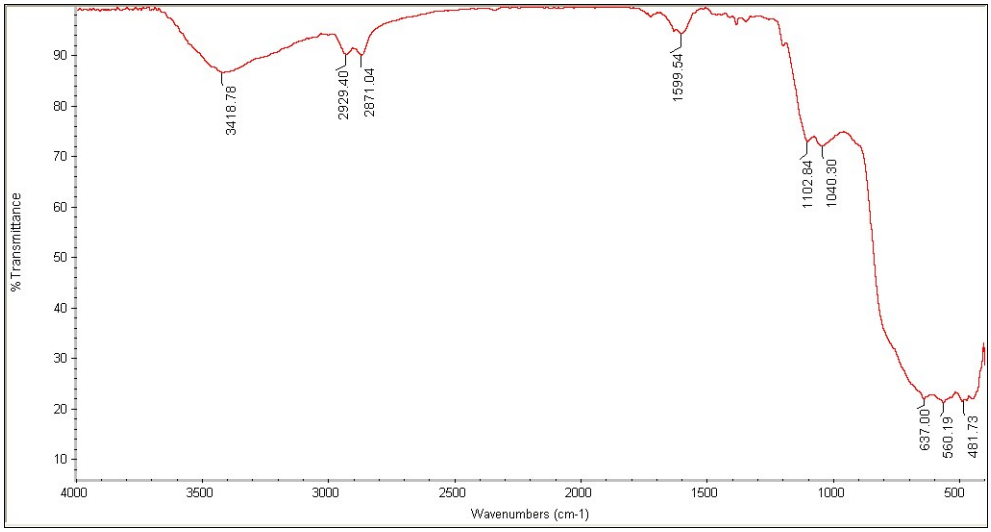

Figure 4: FT-IR (KBr) of Fe_3O_4@TiO_2 YS-GLYMO (**V**).

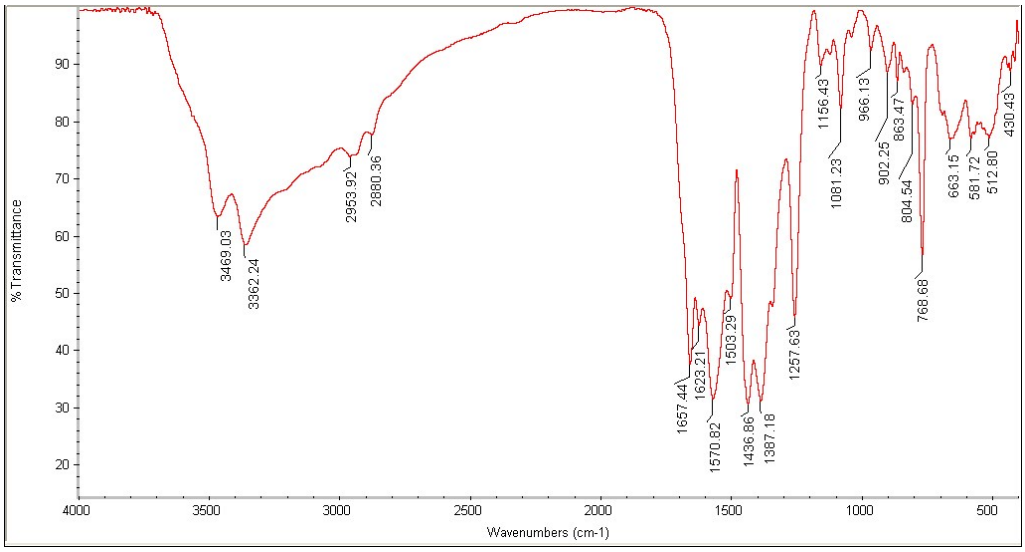

Figure 5: FT-IR (KBr) of UiO-66(Zr)-NH_2 (**V'**).

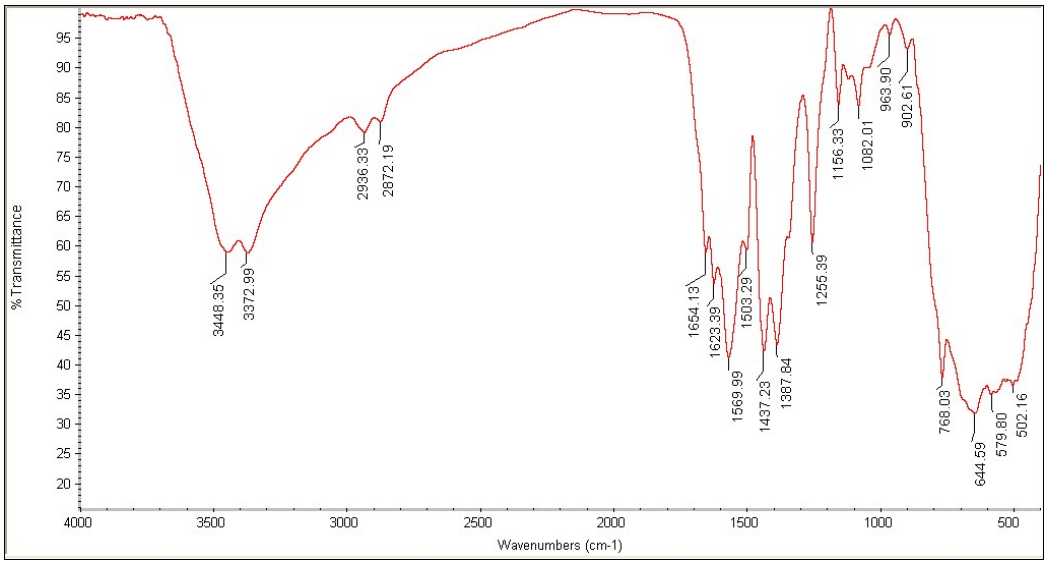

Figure 6: FT-IR (KBr) of Fe_3O_4@TiO_2 YS-GLYMO-UN (**VI**).

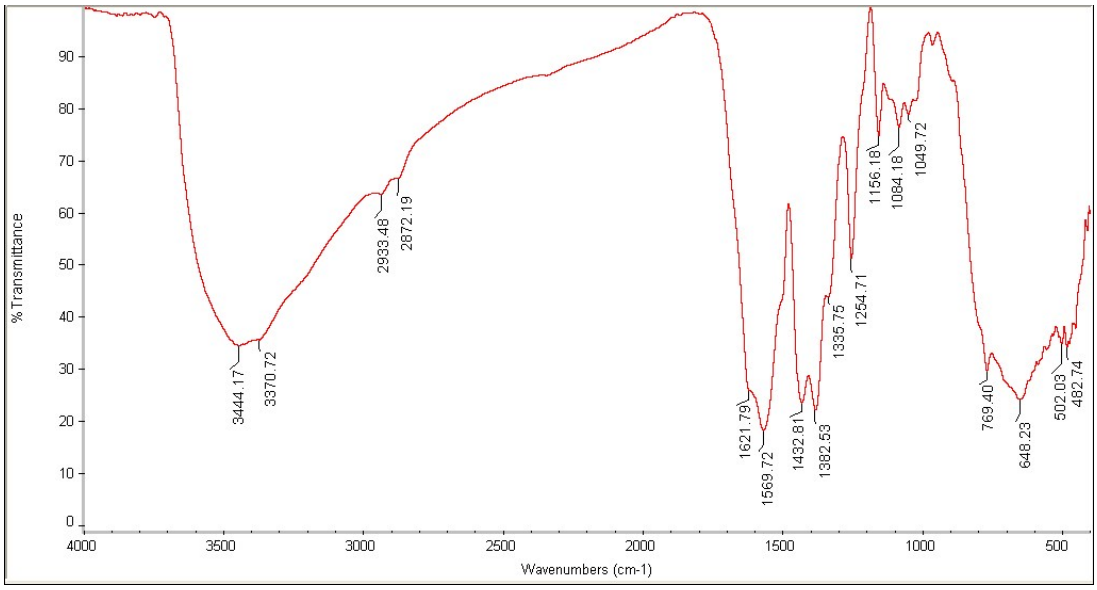

Figure 7: FT-IR (KBr) of Fe_3O_4@TiO_2 YS-GLYMO-UNNi^{II} (**VII**).

Diethyl phenylphosphonate (1a).[1] Oil; isolated yield: 95%; ^{1}H NMR: δH (400 MHz; $CDCl_3$; Me_4Si) 1.32-1.29 (t, *JH,H*= 6.8 Hz, 6 H), 4.16-4.05 (m, 4 H), 7.47-7.42 (m, 2 H), 7.53-7.51 (m, 1 H), 7.80 (dd, *JH,H*= 13.2, *JH,H*= 8.4, 2 H); ^{13}C NMR: δC (100 MHz; $CDCl_3$; Me_4Si) 16.3 (d, *JCP*= 7.0 Hz), 62.0 (d, *JCP*= 5.0 Hz), 131.7 (d, *JCP*= 10.0 Hz), 128.3 (d, *JCP*= 186.0 Hz), 128.4 (d, *JCP*= 15.0 Hz), 132.3 (d, *JCP*= 3.0 Hz); MS, *m/z* 214 (M^+, 5%).

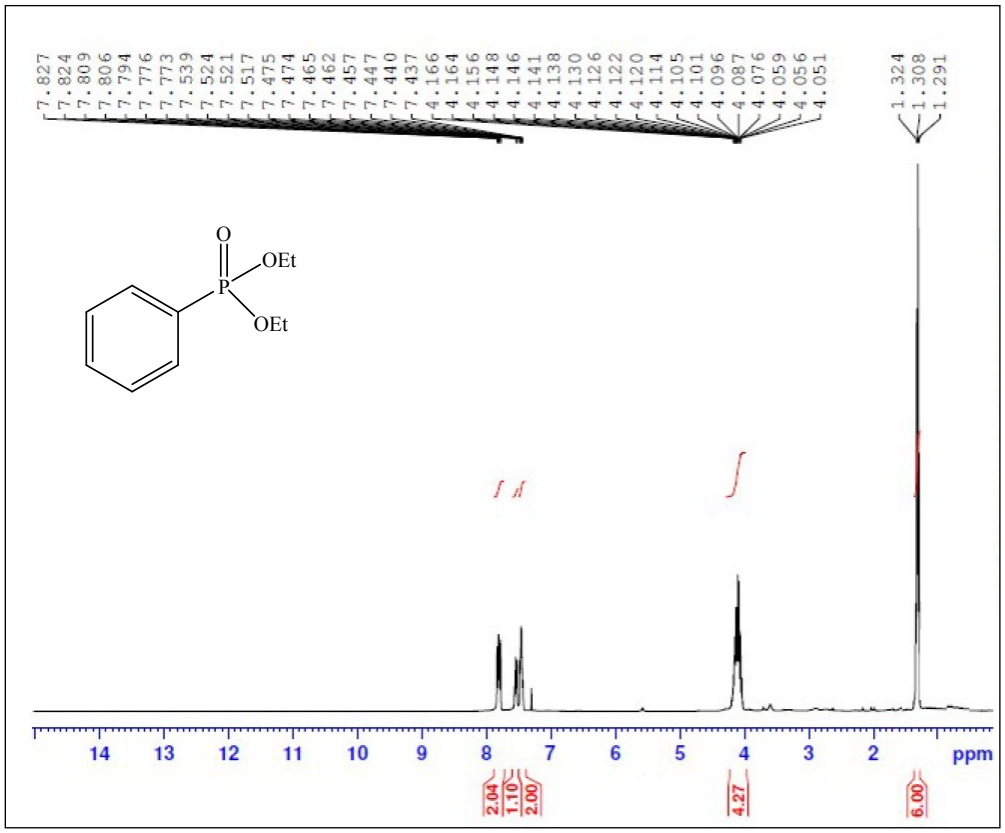

Figure 8: ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of diethyl phenylphosphonate (1a).

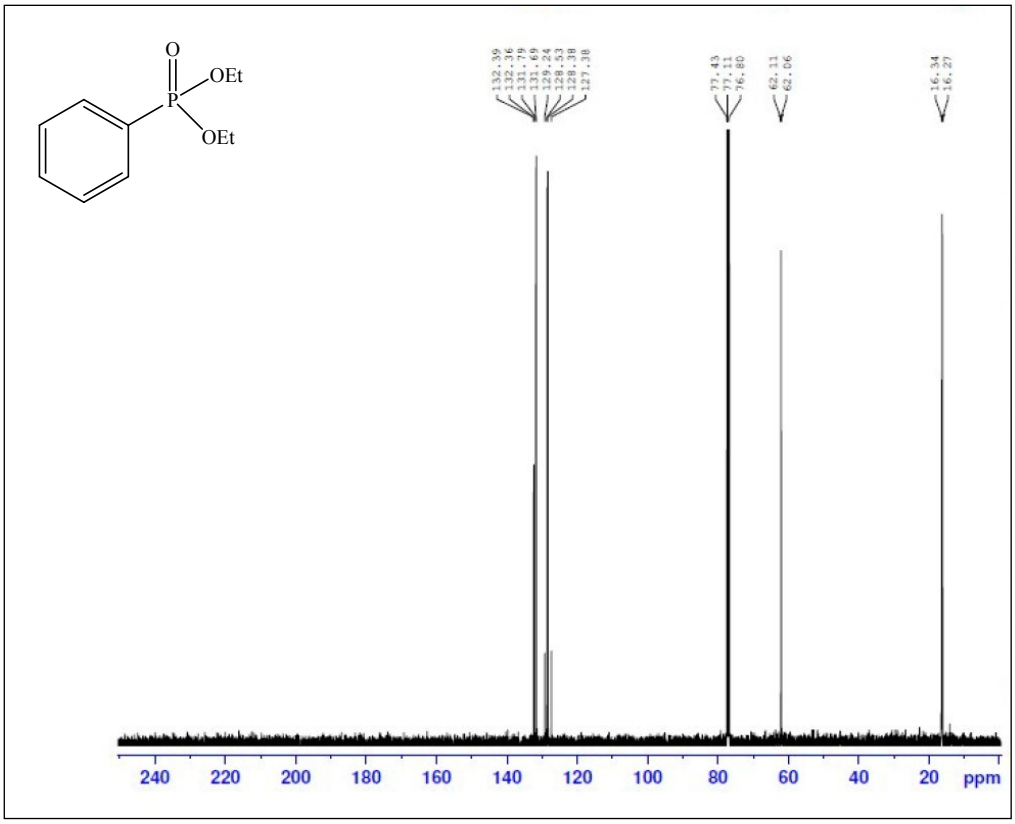

Figure 9: ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of diethyl phenylphosphonate (1a).

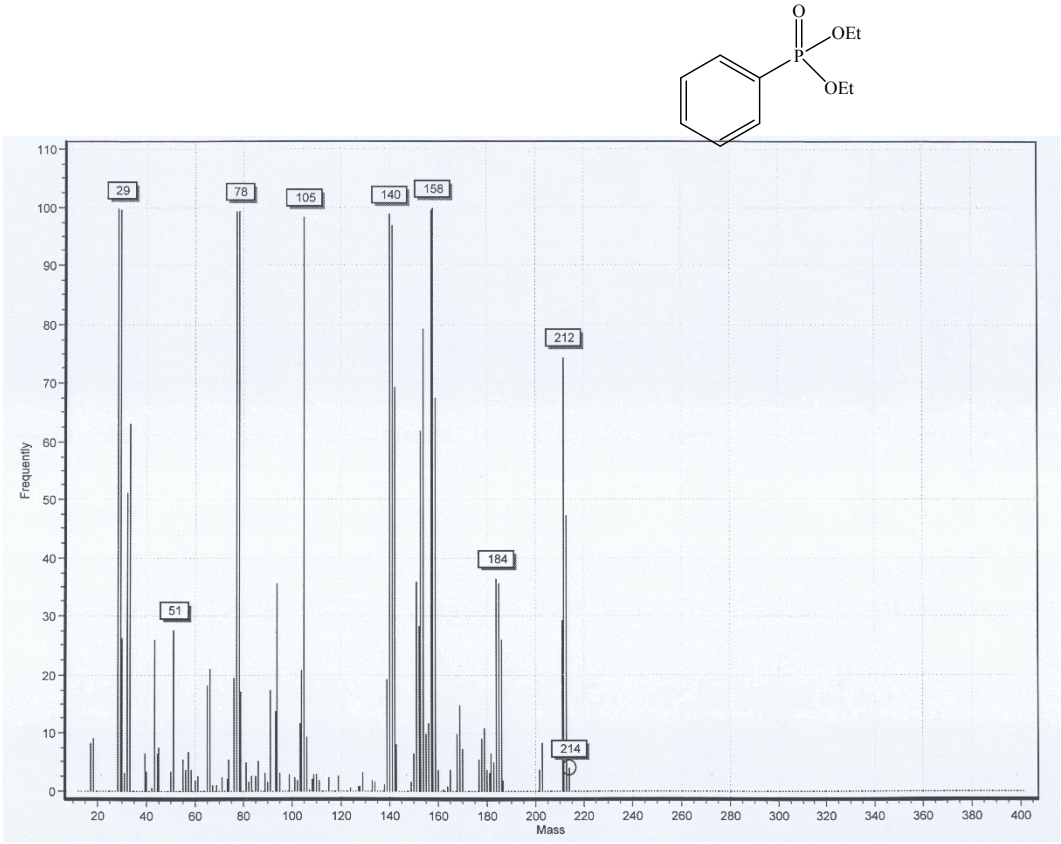

Figure 10: Mass spectrum of diethyl phenylphosphonate (1a).

Diethyl (4-methoxyphenyl)phosphonate (1d).[1] Oil; isolated yield: 90%; ^{1}H NMR: δH (400 MHz; $CDCl_3$; Me_4Si) 1.29-1.25 (t, *JHH*= 7.2 Hz, 6 H), 3.80 (s, 3 H), 4.09-3.97 (m, 4 H), 6.93 (dd, *JHH*= 8.8 Hz, *JHH*= 3.2 Hz, 2 H), 7.71 (dd, *JHH*= 12.8 Hz, *JHH*= 8.8 Hz, 2 H); ^{13}C NMR: δC (100 MHz, $CDCl_3$) 16.2 (d, *JCP*= 6.0 Hz), 55.2, 61.8 (d, *JCP*= 6.0 Hz), 113.9 (d, *JCP*= 16.0 Hz), 119.3 (d, *JCP*= 193.0 Hz), 133.7 (d, *JCP*= 12.0 Hz), 162.8 (d, *JCP*= 4.0 Hz); MS, *m/z* 244 (M^+, 8%).

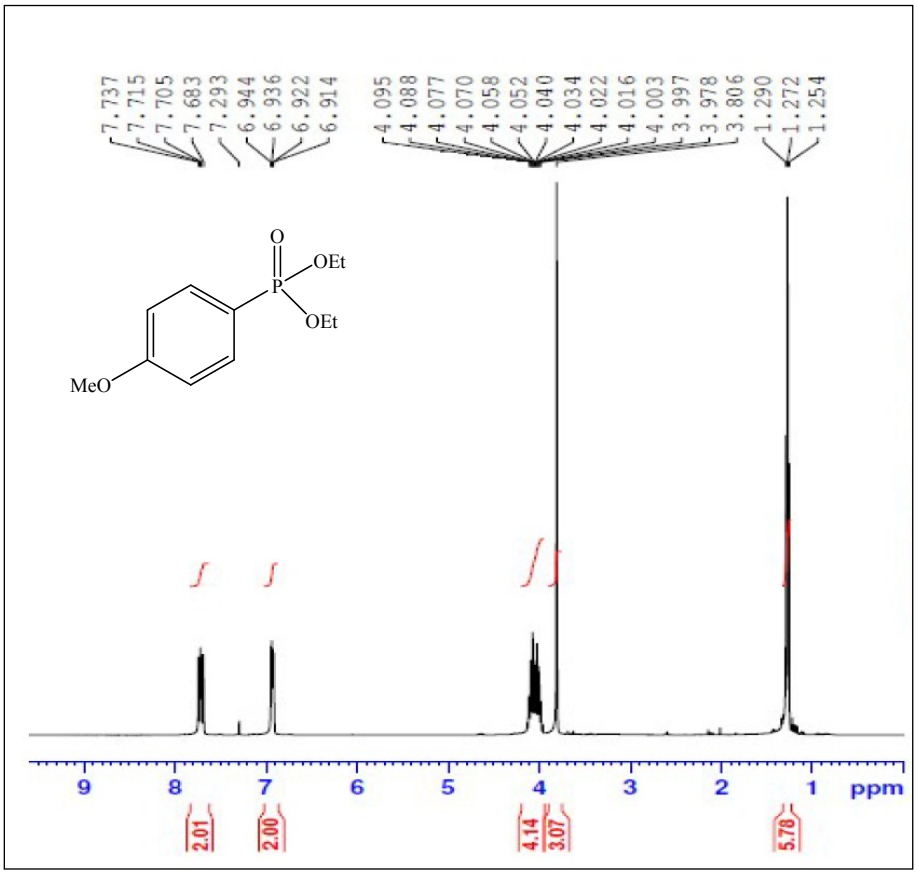

Figure 11: ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of diethyl (4-methoxyphenyl)phosphonate (1d).

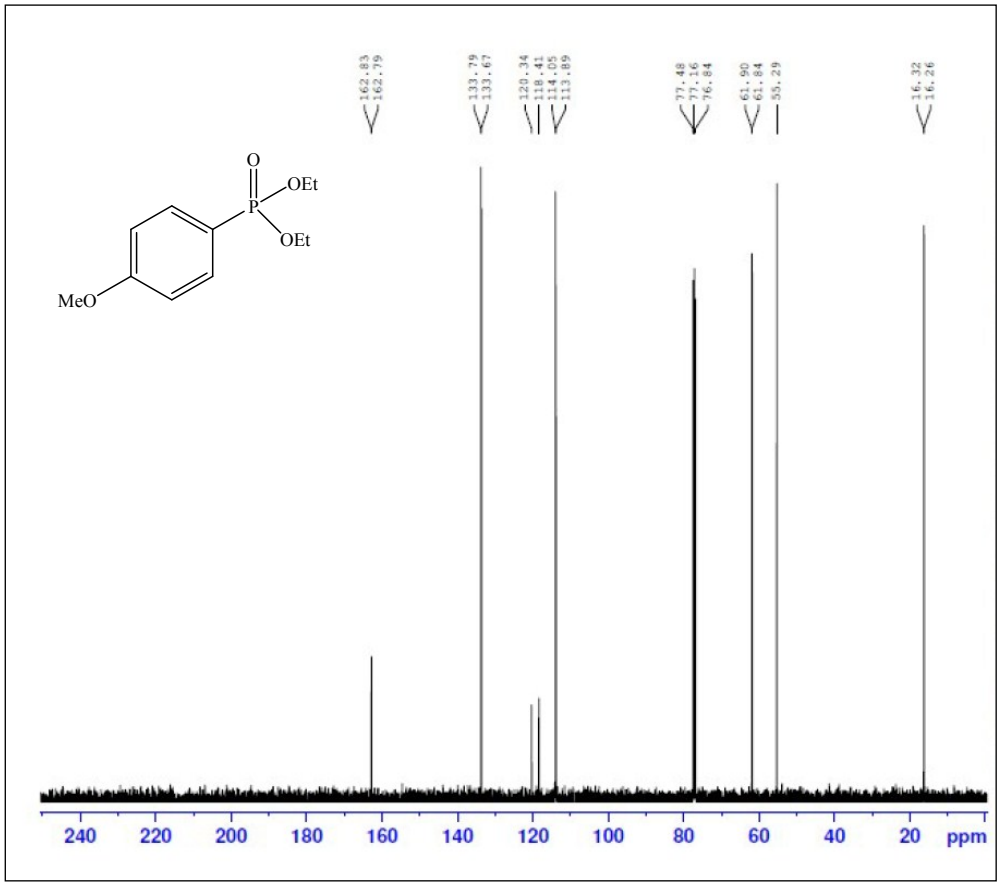

Figure 12: ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of diethyl (4-methoxyphenyl)phosphonate (1d).

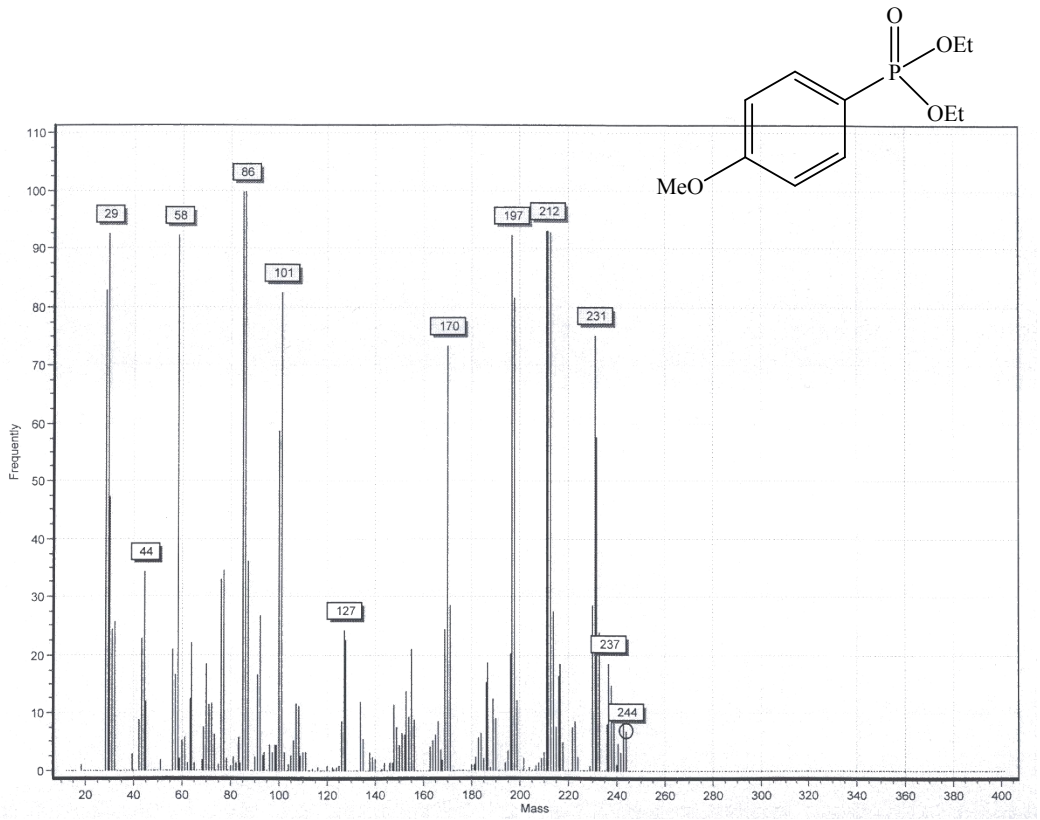

Figure 13: Mass spectrum of diethyl (4-methoxyphenyl)phosphonate (1d).

Diethyl *p*-tolylphosphonate (1e).[1] Oil; isolated yield: 90%; ^{1}H NMR: δH (300 MHz; $CDCl_3$; Me_4Si) 1.35-1.31 (t, *J*HH= 6.9 Hz, 6H), 2.42 (s, 3H), 4.21-4.01 (m, 4H), 7.29 (dd, *JHH*= 8.1 Hz, *JHH*= 3.3 Hz, 2H), 7.72 (dd, *JHH*= 13.2 Hz, *JHH*= 8.1 Hz, 2H); ^{13}C NMR: δC (75 MHz; $CDCl_3$; Me_4Si) 16.1 (d, *JCP*= 6.7 Hz), 16.3 (d, *JCP*= 6.7 Hz), 21.6, 61.9 (d, *JCP*= 5.2 Hz), 124.9 (d, *JCP*= 188.2 Hz), 129.2 (d, *JCP*= 15.0 Hz), 131.8 (d, *JCP*= 9.7 Hz), 142.9 (d, *JCP*= 3.0 Hz); MS, *m/z* 228 (M^+, 5%).

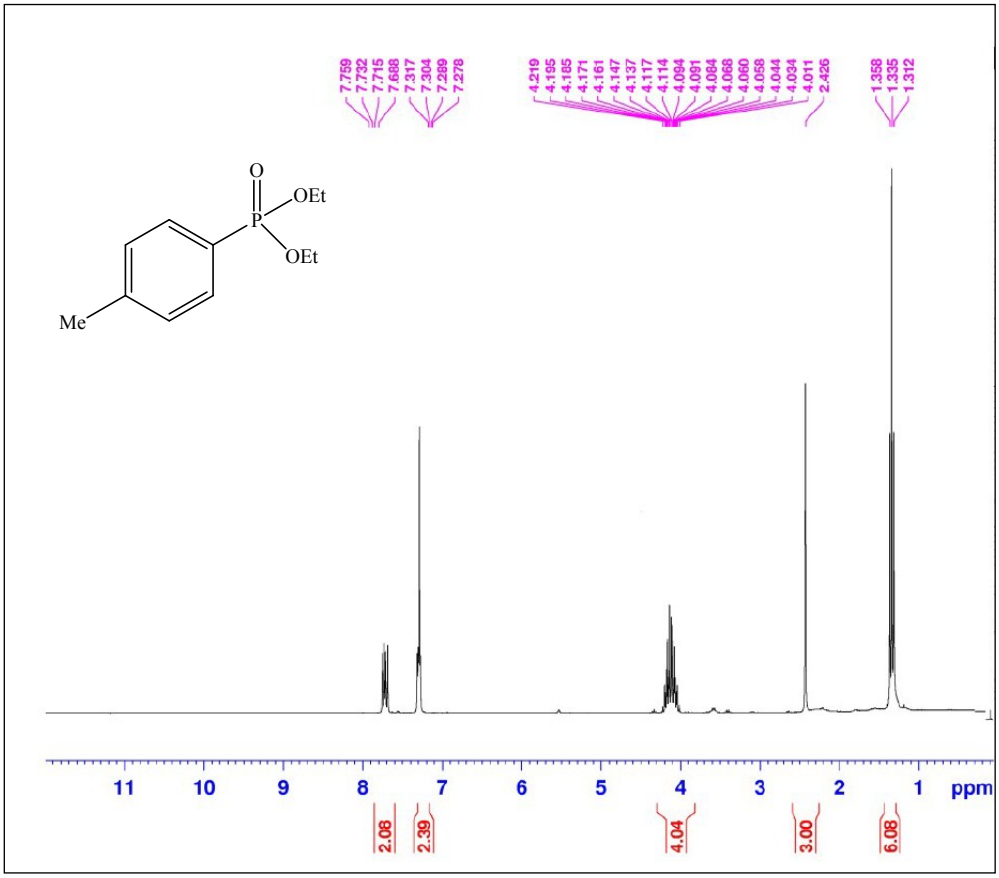

Figure 14: ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of diethyl *p*-tolylphosphonate (1e).

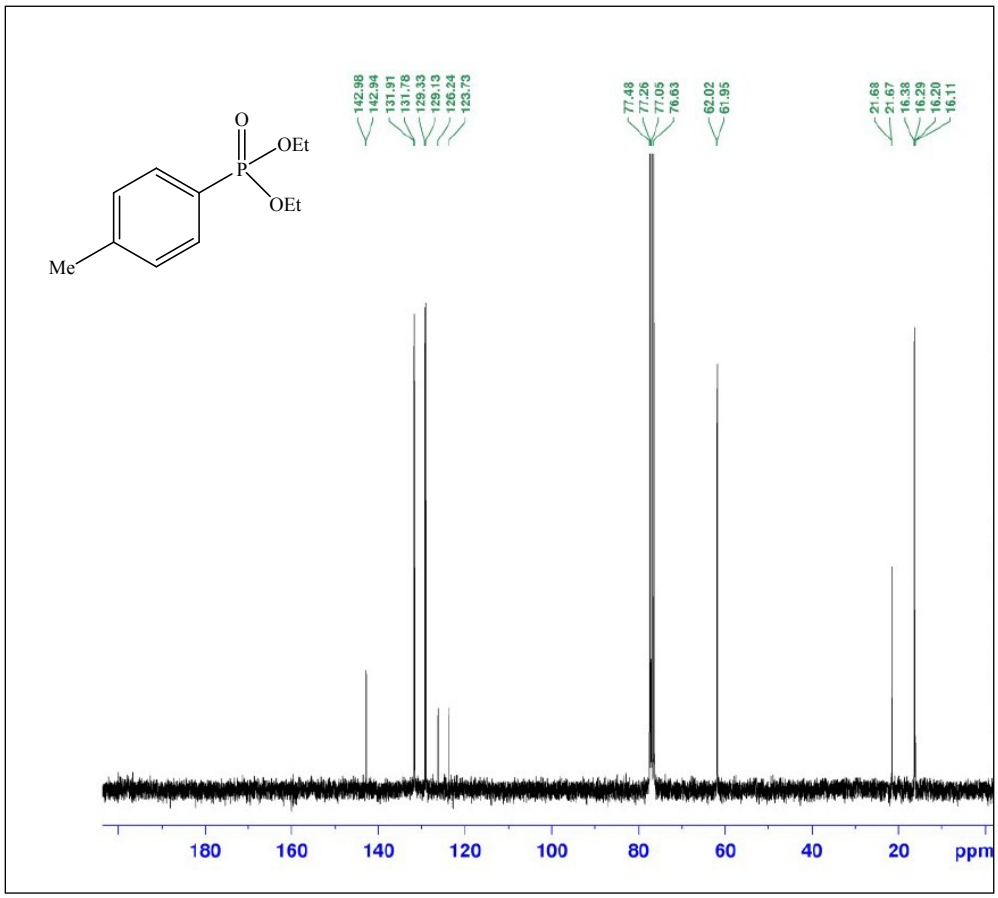

Figure 15: ^{13}C NMR spectrum (75 MHz, $CDCl_3$) of diethyl *p*-tolylphosphonate (1e).

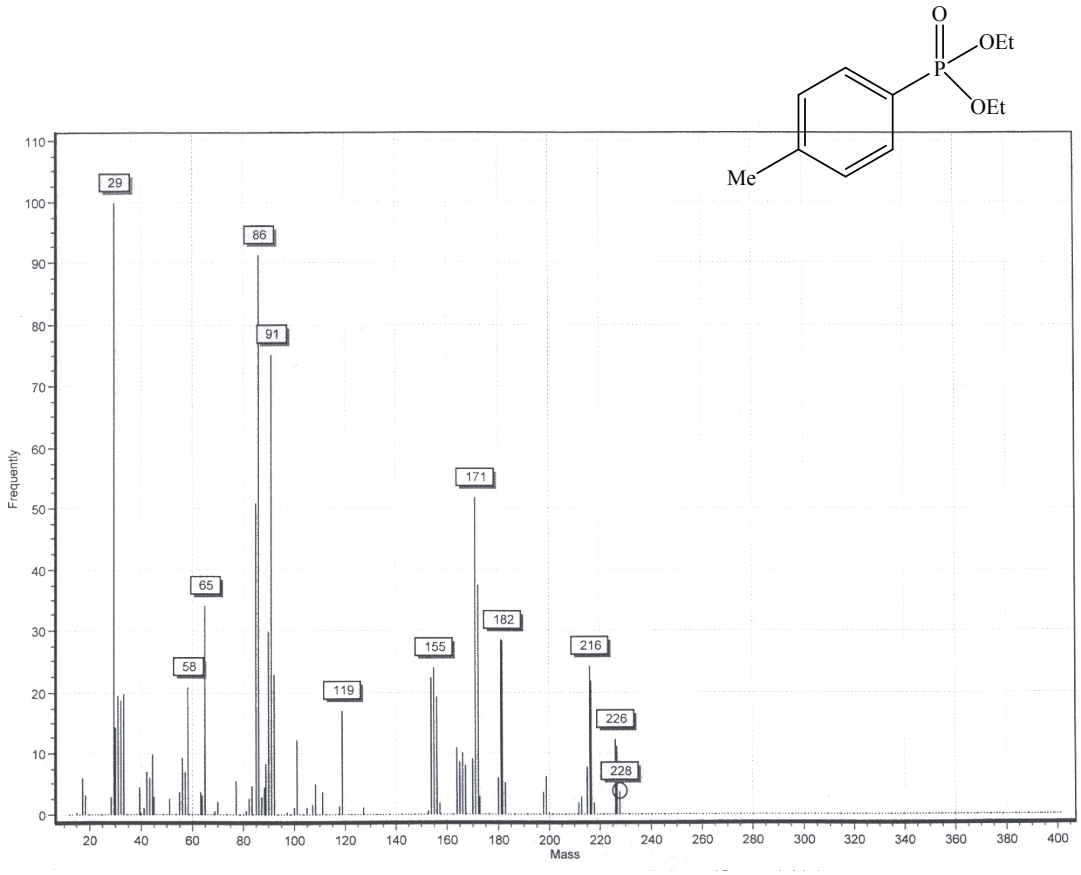

Figure 16: Mass spectrum of diethyl *p*-tolylphosphonate (1e).

Diethyl (4-nitrophenyl)phosphonate (1h).[1] Oil; isolated yield: 90%; ^{1}H NMR: δH (300 MHz; $CDCl_3$; Me_4Si) 1.36-1.32 (t, *JHH*= 6.9 Hz, 6H), 4.27-4.06 (m, 4H), 8.00 (dd, *JHH*= 12.7 Hz, *JHH* = 8.7 Hz, 1H), 8.3(dd, *JHH*= 8.7 Hz, *JHH* = 3.3 Hz, 1H); ^{13}C NMR: δC (75 MHz; $CDCl_3$; Me_4Si) 16.1 (d, *JCP*= 6.7 Hz), 16.3 (d, *JCP*= 6.0 Hz), 62.7 (d, *JCP*= 5.2 Hz), 123.3 (d, *JCP*= 15.0 Hz), 133.0 (d, *JCP*= 10.5 Hz), 135.8 (d, *JCP*=185.2 Hz), 150.2 (d, *JCP*= 3.7 Hz); MS, *m/z* 257 (M^+, 6%).

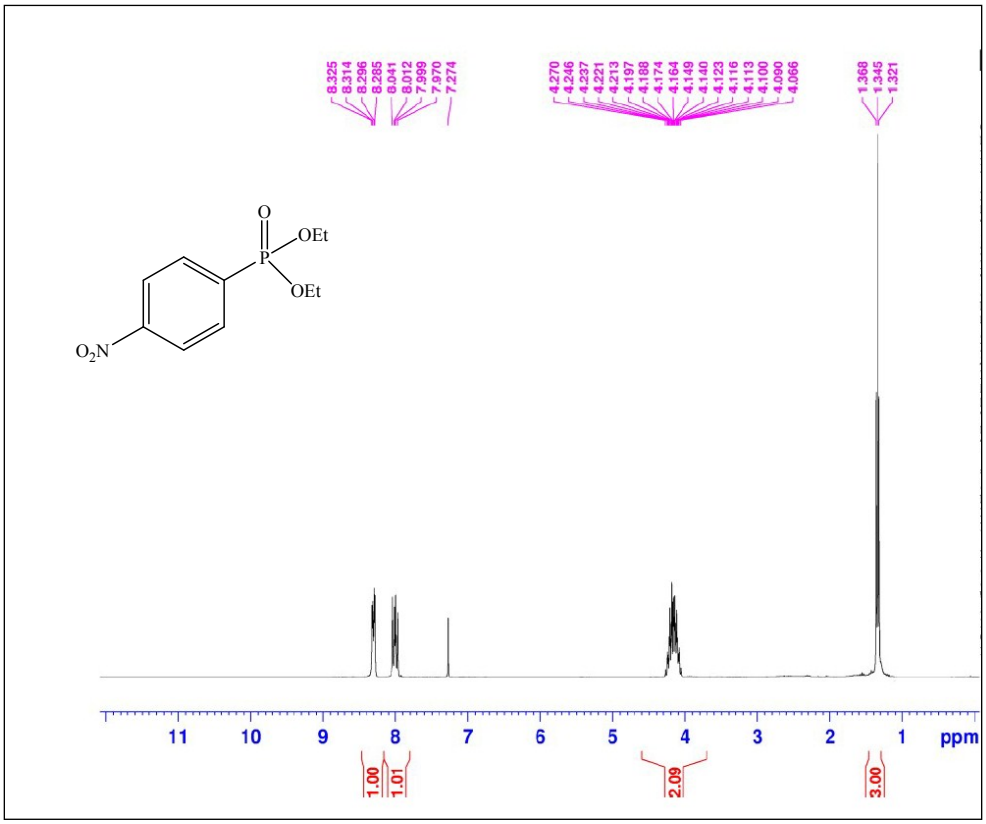

Figure 17: ^{1}H NMR spectrum (300 MHz, $CDCl_3$) of diethyl (4-nitrophenyl)phosphonate (1h).

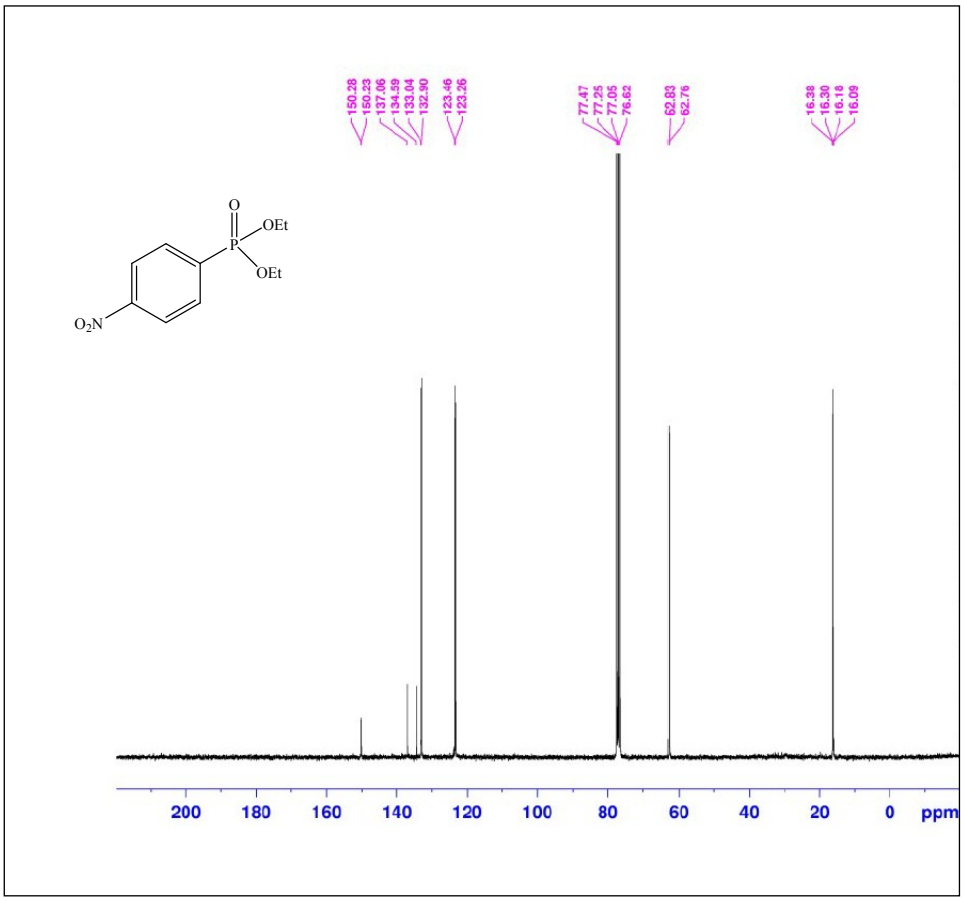

Figure 18: ^{13}C NMR spectrum (75 MHz, $CDCl_3$) of diethyl (4-nitrophenyl)phosphonate (1h).

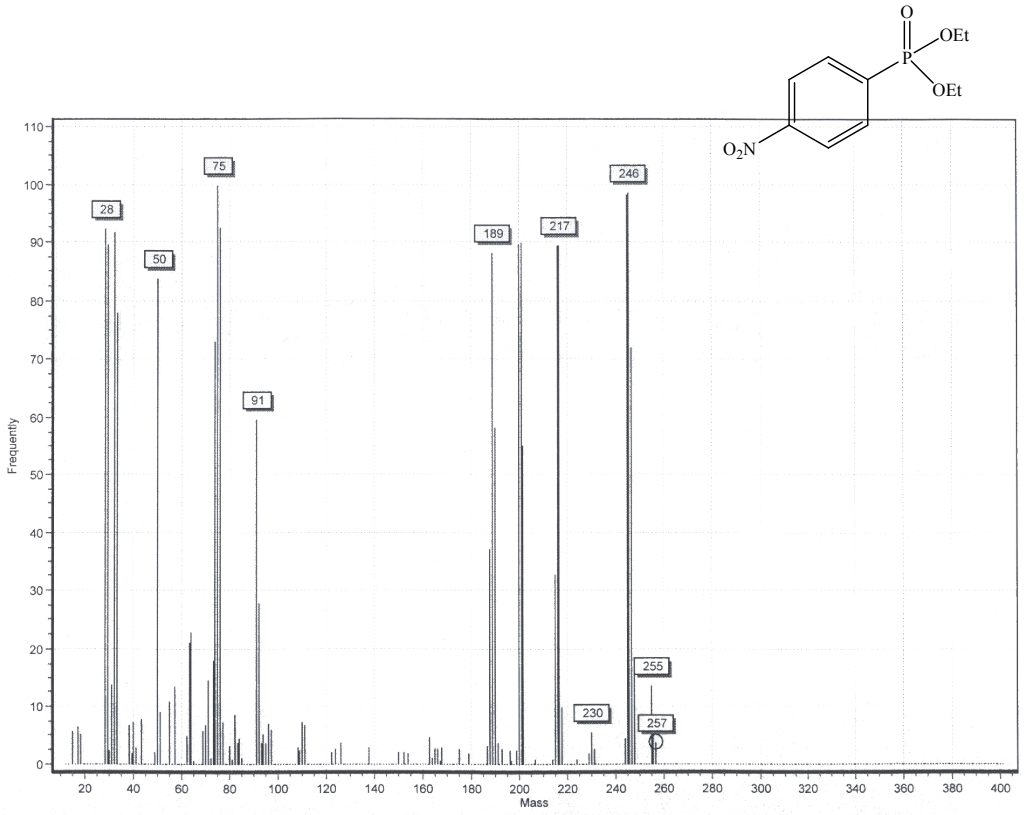

Figure 19: Mass spectrum of diethyl (4-nitrophenyl)phosphonate (1h).

Reference

1. N. Iranpoor, H. Firouzabadi, K. R. Moghadam and S. Motavalli, *RSC Adv.*, 2014, **4**, 55732-55737.