

SUPPORTING INFORMATION

Title: Synthesis of arylphosphonates catalyzed by Pd-imino-Py- γ -Fe₂O₃ as a new magnetically recyclable heterogeneous catalyst in pure water without requiring any additive

Author(s): Sara Sobhani,* Zohreh Ramezani

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General information

Chemicals were purchased from Merck Chemical Company. NMR spectra were recorded on a Bruker Avance DPX-400 and 300 using deuterated CDCl_3 as solvent and TMS as internal standard. The purity of the products and the progress of the reactions were accomplished by TLC on silica-gel polygram SILG/UV254 plates. The BET surface area measurements were performed on a BEL-MAX (Japan) instrument at liquid nitrogen temperature. TEM analysis was performed using TEM microscope (Philips CM30). FT-IR spectra were recorded on a Shimadzu Fourier Transform Infrared Spectrophotometer (FT-IR-8300). Thermo gravimetric analysis (TGA) was performed using a Shimadzu thermo gravimetric analyzer (TG-50). Elemental analysis was carried out on a Costech 4010 CHN elemental analyzer. The morphology of the products was determined by using Hitachi Japan, model s4160 Scanning Electron Microscopy (SEM) at accelerating voltage of 15 KV. Power X-ray diffraction (XRD) was performed on a Bruker D8-advance X-ray diffractometer with $\text{Cu K}\alpha$ ($\lambda = 0.154 \text{ nm}$) radiation. This system was equipped with a concentric hemispherical (CHA) electron energy analyzer (Specs model EA10 plus) suitable for X-ray photoelectron spectroscopy (XPS). The content of Pd in the catalyst was determined by OPTIMA 7300DV ICP analyzer. Room temperature magnetization isotherms were obtained using a vibrating sample magnetometer (VSM, Lake Shore 7400).

Synthesis of iminopyridine

Pyridine-2-carbaldehyde (0.5 g) was added to a magnetically stirring mixture of 4-aminophenol (0.5 g) in MeOH (10 mL). After refluxing for 3 h, the mixture was cooled to room temperature. The resulting yellow crystals (iminopyridine ligand) was separated by filtration, washed with MeOH ($3 \times 10 \text{ mL}$) and dried under vacuum.

Synthesis of Schiff base immobilized on $\gamma\text{-Fe}_2\text{O}_3$ (imino-Py- $\gamma\text{-Fe}_2\text{O}_3$)

The maghemite ($\gamma\text{-Fe}_2\text{O}_3$) nanoparticles were synthesized by a reported chemical co-precipitation technique of ferric and ferrous ions in alkali solution with minor modifications.¹ Using BET method, values of $91 \text{ m}^2 \text{ g}^{-1}$ and 14.3 nm were found for surface area and mean pore diameter of $\gamma\text{-Fe}_2\text{O}_3$, respectively (Figure 1). $\gamma\text{-Fe}_2\text{O}_3$ was chloro-functionalized by the reaction with 3-chloropropyltrimethoxysilane.² A solution of chloro-functionalized $\gamma\text{-Fe}_2\text{O}_3$ (1.7 g) in DMF (10 mL) was added dropwise to a stirring solution of iminopyridine (0.15 g) and NaH (0.005 g) in DMF (5 mL), under Ar atmosphere at $80 \text{ }^\circ\text{C}$ within 1 h. The reaction mixture was stirred at $80 \text{ }^\circ\text{C}$ for another 24 h. The resulting Schiff base immobilized on $\gamma\text{-Fe}_2\text{O}_3$ was separated by an external magnet, washed with acetone ($3 \times 10 \text{ mL}$) and dried under vacuum. The loading amount of Schiff base was 0.28 mmol g^{-1} catalyst based on elemental analysis.

Synthesis of palladium-Schiff base complex immobilized on $\gamma\text{-Fe}_2\text{O}_3$ (Pd-imino-Py- $\gamma\text{-Fe}_2\text{O}_3$)

The synthesized Schiff base immobilized on $\gamma\text{-Fe}_2\text{O}_3$ (1.7 g) was added to a solution of palladium acetate (0.12 g) in dry acetone (5 mL). The reaction mixture was stirred at room temperature for 24 h. The solid was separated by an external magnet, and washed with acetone ($3 \times 10 \text{ mL}$) and dried under vacuum to afford Pd-imino-Py- $\gamma\text{-Fe}_2\text{O}_3$.

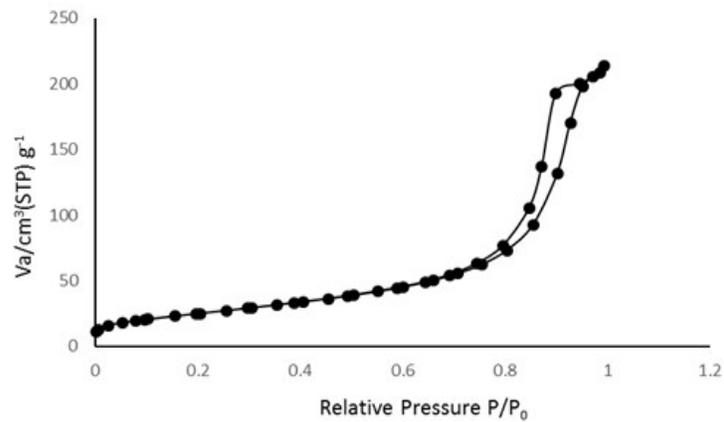


Figure 1. Nitrogen adsorption–desorption isotherms of Pd-imino-Py- γ -Fe₂O₃

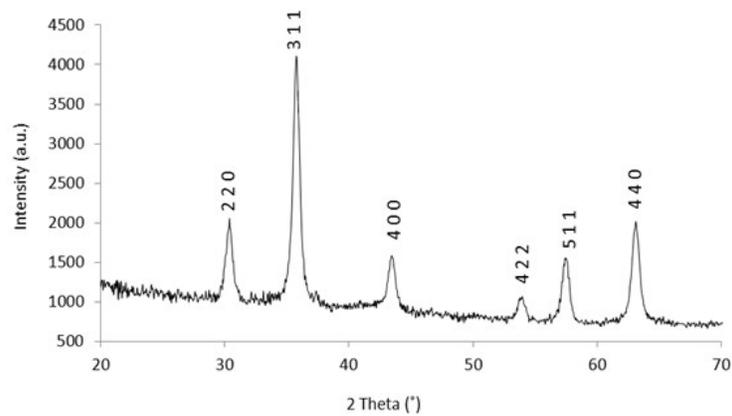


Figure S2. XRD patterns of Pd-imino-Py- γ -Fe₂O₃

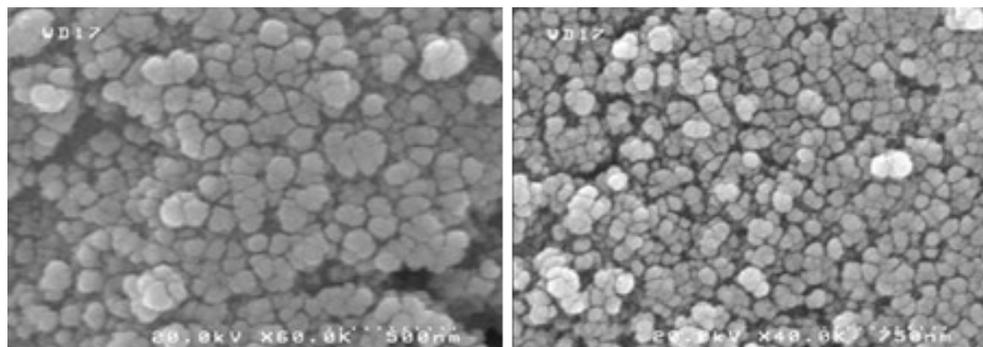


Figure S3. SEM of Pd-imino-Py- γ -Fe₂O₃

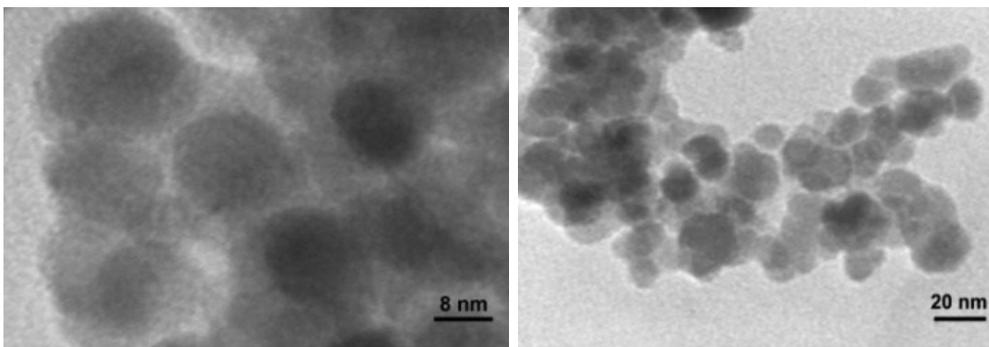


Figure S4. TEM of Pd-imino-Py- γ -Fe₂O₃

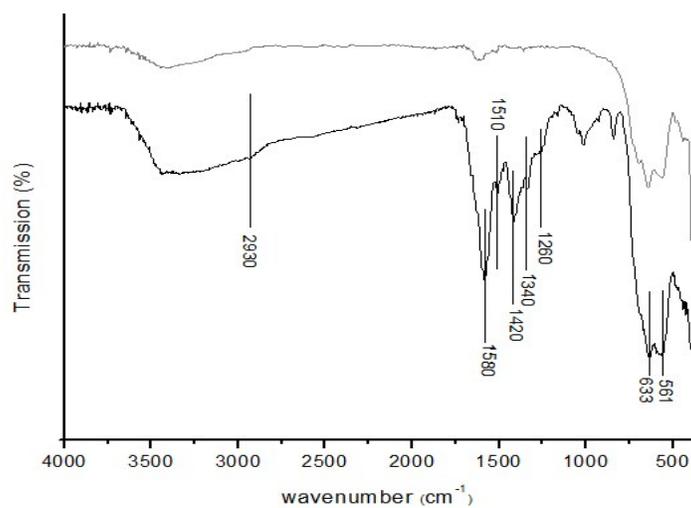


Figure S5. FT-IR spectra of γ -Fe₂O₃ (gray), Pd-imino-Py- γ -Fe₂O₃ (black)

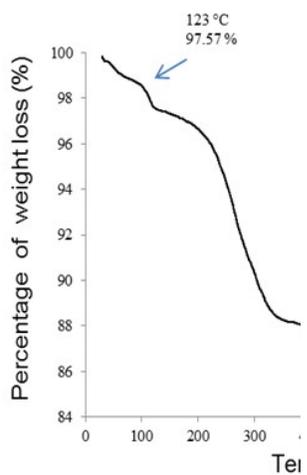
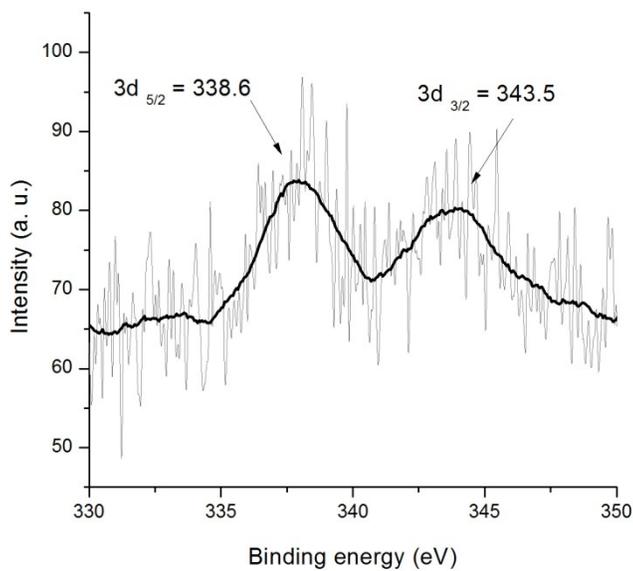


Figure (a)
imino-P



TGA of Pd-
Fe₂O₃

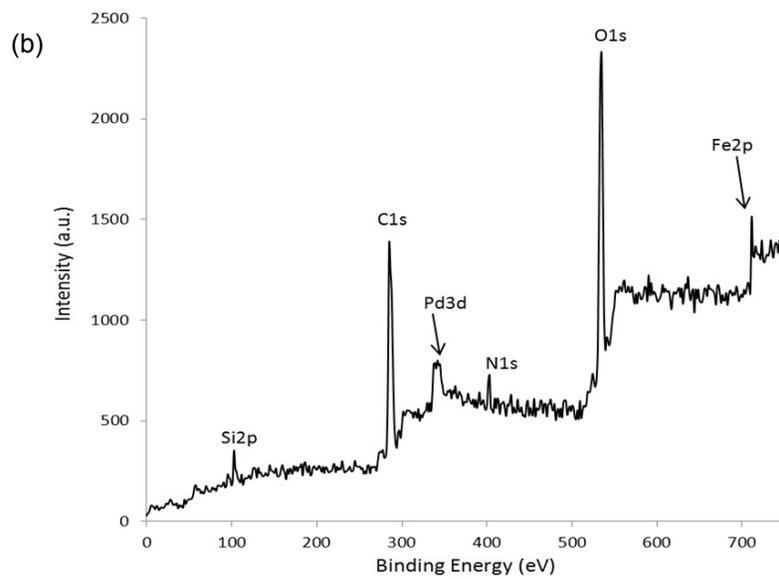


Figure S7. (a) XPS spectrum of Pd in Pd-imino-Py- γ -Fe₂O₃, (b) XPS spectrum of all elements of Pd-imino-Py- γ -Fe₂O₃

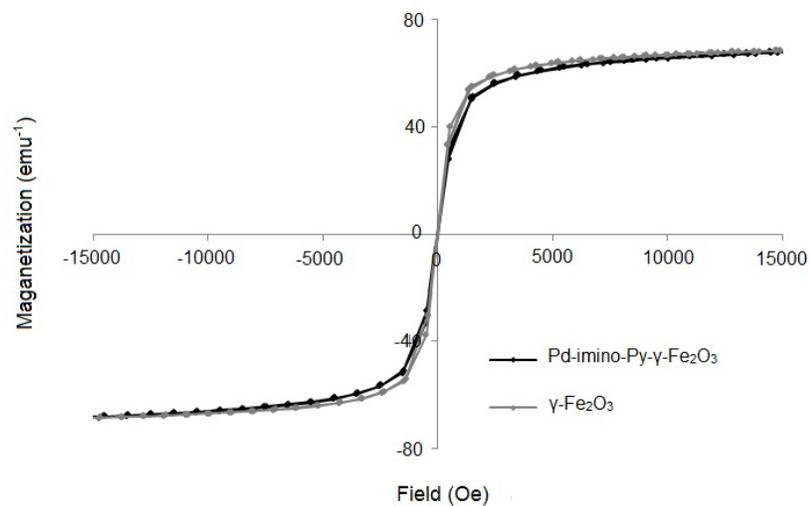
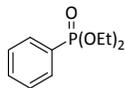


Figure S8. Magnetization curves of $\gamma\text{-Fe}_2\text{O}_3$ and Pd-imino-Py- $\gamma\text{-Fe}_2\text{O}_3$

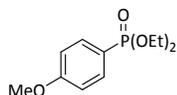
Characterization data of the products

Diethyl phenylphosphonate: Isolated as colorless oil (Table 1, entry 1: 98%, 209.7 mg; entry 3, 93%, 199.0 mg, entry 10: 88%, 188.3 mg; entry 20: 75%, 160.5 mg; entry 21: 80%, 171.2 mg)



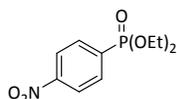
^1H NMR (400 MHz, CDCl_3): δ 1.30 (t, $J_{\text{HH}} = 6.8$ Hz, 6 H), 4.16-4.05 (m, 4 H), 7.47-7.42 (m, 2 H), 7.53-7.51 (m, 1 H), 7.80 (dd, $J_{\text{HH}} = 13.2$, $J_{\text{HH}} = 8.4$, 2 H), ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 16.3 (d, $J_{\text{CP}} = 7.0$ Hz), 62.0 (d, $J_{\text{CP}} = 5.0$ Hz), 131.7 (d, $J_{\text{CP}} = 10.0$ Hz), 128.3 (d, $J_{\text{CP}} = 186.0$ Hz), 128.4 (d, $J_{\text{CP}} = 15.0$ Hz), 132.3 (d, $J_{\text{CP}} = 3.0$ Hz) ppm.

Diethyl 4-methoxyphenylphosphonate: Isolated as colorless oil (Table 1, entry 2: 95%, 231.8 mg; entry 4: 83%, 202.5 mg)



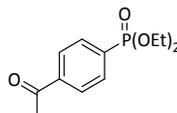
^1H NMR (400 MHz, CDCl_3): δ 1.27 (t, $J_{\text{HH}} = 7.2$ Hz, 6 H), 3.80 (s, 3 H), 4.09-3.97 (m, 4 H), 6.93 (dd, $J_{\text{HH}} = 8.8$ Hz, $J_{\text{HH}} = 3.2$ Hz, 2 H), 7.71 (dd, $J_{\text{HH}} = 12.8$ Hz, $J_{\text{HH}} = 8.8$ Hz, 2 H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 16.2 (d, $J_{\text{CP}} = 6.0$ Hz), 55.2, 61.8 (d, $J_{\text{CP}} = 6.0$ Hz), 113.9 (d, $J_{\text{CP}} = 16.0$ Hz), 119.3 (d, $J_{\text{CP}} = 193.0$ Hz), 133.7 (d, $J_{\text{CP}} = 12.0$ Hz), 162.8 (d, $J_{\text{CP}} = 4.0$ Hz) ppm.

Diethyl 4-nitrophenylphosphonate: Isolated as oil (Table 1, entry 5: 92%, 238.2 mg, entry 11: 89%, 230.5 mg).



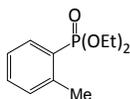
^1H NMR (300 MHz, CDCl_3): δ 1.34 (t, $J_{\text{HH}} = 6.9$ Hz, 6H), 4.27-4.06 (m, 4H), 8.00 (dd, $J_{\text{HH}} = 12.7$ Hz, $J_{\text{HH}} = 8.7$ Hz, 1H), 8.3 (dd, $J_{\text{HH}} = 8.7$ Hz, $J_{\text{HH}} = 3.3$ Hz, 1H) ppm. ^{13}C NMR (75 MHz, CDCl_3): δ 16.1 (d, $J_{\text{CP}} = 6.7$ Hz), 16.3 (d, $J_{\text{CP}} = 6.0$ Hz), 62.7 (d, $J_{\text{CP}} = 5.2$ Hz), 123.3 (d, $J_{\text{CP}} = 15.0$ Hz), 133.0 (d, $J_{\text{CP}} = 10.5$ Hz), 135.8 (d, $J_{\text{CP}} = 185.2$ Hz), 150.2 (d, $J_{\text{CP}} = 3.7$ Hz) ppm.

Diethyl 4-acetylphenylphosphonate: Isolated as oil (Table 1, entry 6: 85%, 217.6 mg).



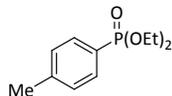
^1H NMR (300 MHz, CDCl_3): δ 1.32 (t, $J_{\text{HH}} = 6.9$ Hz, 6 H), 2.64 (s, 3H), 4.21-4.05 (m, 4H), 7.91 (dd, $J_{\text{HH}} = 8.1$ Hz, $J_{\text{HH}} = 12.7$ Hz, 2H), 8.04-8.00 (m, 2H) ppm. ^{13}C NMR (75 MHz, CDCl_3): δ 16.3 (d, $J_{\text{CP}} = 6.7$ Hz), 26.85 (s, 3H), 62.4 (d, $J_{\text{CP}} = 5.2$ Hz), 128.0 (d, $J_{\text{CP}} = 15.0$ Hz), 130.4 (d, $J_{\text{CP}} = 95.2$ Hz), 132.0 (d, $J_{\text{CP}} = 9.7$ Hz), 134.61, 139.8 (d, $J_{\text{CP}} = 3$), 197.5 ppm.

Diethyl 2-methylphenylphosphonate: Isolated as oil (Table 1, entry 7: 95%, 216.6 mg).



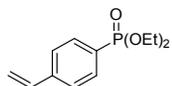
^1H NMR (300 MHz, CDCl_3): δ 1.24 (t, $J_{\text{HH}} = 6.6$ Hz, 6 H), 2.49 (s, 3H), 3.93-4.13 (m, 4H), 7.20-7.14 (m, 2H), 7.34 (t, $J_{\text{HH}} = 6.0$ Hz, 1H), 7.87-7.79 (m, 1H) ppm. ^{13}C NMR (75 MHz, CDCl_3): δ 16.3 (d, $J_{\text{CP}} = 6.7$ Hz), 21.1 (d, $J_{\text{CP}} = 3.0$ Hz), 61.8 (d, $J_{\text{CP}} = 6.0$ Hz), 125.3 (d, $J_{\text{CP}} = 15.0$ Hz), 126.8 (d, $J_{\text{CP}} = 183.0$ Hz), 131.1 (d, $J_{\text{CP}} = 14.2$ Hz), 132.4 (d, $J_{\text{CP}} = 3$), 133.8 (d, $J_{\text{CP}} = 9.7$), 141.7 (d, $J_{\text{CP}} = 9.7$) ppm.

Diethyl 4-tolylphosphonate: Isolated as oil (Table 1, entry 8: 89%, 202.9 mg, entry 13: 93%, 212.0 mg).



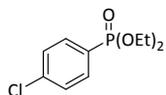
^1H NMR (300 MHz, CDCl_3): δ 1.33 (t, $J_{\text{HH}} = 6.9$ Hz, 6H), 2.42 (s, 3H), 4.21-4.01 (m, 4H), 7.29 (dd, $J_{\text{HH}} = 8.1$ Hz, $J_{\text{HH}} = 3.3$ Hz, 2H), 7.72 (dd, $J_{\text{HH}} = 13.2$ Hz, $J_{\text{HH}} = 8.1$ Hz, 2H) ppm. ^{13}C NMR (75 MHz, CDCl_3): δ 16.1 (d, $J_{\text{CP}} = 6.7$ Hz), 16.3 (d, $J_{\text{CP}} = 6.7$ Hz), 21.6, 61.9 (d, $J_{\text{CP}} = 5.2$ Hz), 124.9 (d, $J_{\text{CP}} = 188.2$ Hz), 129.2 (d, $J_{\text{CP}} = 15.0$ Hz), 131.8 (d, $J_{\text{CP}} = 9.7$ Hz), 142.9 (d, $J_{\text{CP}} = 3.0$ Hz) ppm.

Diethyl 4-vinylphenylphosphonate: Isolated as oil (Table 1, entry 9: 78%, 187.2 mg).



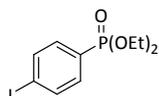
^1H NMR (300 MHz, CDCl_3): δ 1.33 (t, $J_{\text{HH}} = 6.9$ Hz, 6H), 4.20-4.04 (m, 4H), 5.4 (d, $J_{\text{HH}} = 10.8$ Hz, 1H), 5.87 (d, $J_{\text{HH}} = 17.4$ Hz, 1H), 6.75 (dd, $J_{\text{HH}} = 17.4$ Hz, $J_{\text{HH}} = 10.8$ Hz, 1H), 7.52-7.48 (m, 2H), 7.78 (dd, $J_{\text{HH}} = 12.9$ Hz, $J_{\text{HH}} = 8.1$ Hz, 2H) ppm. ^{13}C NMR (75 MHz, CDCl_3): δ 16.1 (d, $J_{\text{CP}} = 6.7$ Hz), 16.3 (d, $J_{\text{CP}} = 6.0$ Hz), 62.1 (d, $J_{\text{CP}} = 5.2$ Hz), 116.5, 63.6 (d, $J_{\text{CP}} = 6.0$ Hz), 126.1 (d, $J_{\text{CP}} = 15.0$ Hz), 127.2 (d, $J_{\text{CP}} = 187.5$ Hz), 132.1 (d, $J_{\text{CP}} = 9.7$ Hz), 135.9, 141.4 (d, $J_{\text{CP}} = 3.0$ Hz) ppm.

Diethyl 4-chlorophenylphosphonate: Isolated as colorless oil (Table 1, entry 14: 98%, 244.0 mg; entry 16: 91%, 226.5 mg)



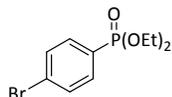
^1H NMR (400 MHz, CDCl_3): δ 1.34 (t, $J_{\text{HH}} = 7.2$ Hz, 6 H), 4.19-4.06 (m, 4 H), 7.46 (dd, $J_{\text{HH}} = 8.2$ Hz, $J_{\text{HH}} = 3.6$ Hz, 2 H), 7.76 (dd, $J_{\text{HH}} = 12.8$ Hz, $J_{\text{HH}} = 8.4$ Hz, 2 H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 16.3 (d, $J_{\text{CP}} = 7.0$ Hz), 62.2 (d, $J_{\text{CP}} = 5.0$ Hz), 126.9 (d, $J_{\text{CP}} = 190.0$ Hz), 128.8 (d, $J_{\text{CP}} = 16.0$ Hz), 133.2 (d, $J_{\text{CP}} = 10.0$ Hz), 138.9 (d, $J_{\text{CP}} = 4.0$ Hz) ppm.

Diethyl 4-iodophenylphosphonate: Isolated as colorless oil (Table 1, entry 15: 98%, 333.2 mg)



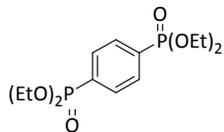
^1H NMR (400 MHz, CDCl_3): δ 1.34 (t, $J_{\text{HH}} = 6.8$ Hz, 6 H), 4.19-4.06 (m, 4 H), 7.54 (dd, $J_{\text{HH}} = 13$ Hz, $J_{\text{HH}} = 8.0$ Hz, 2 H), 7.85 (dd, $J_{\text{HH}} = 8.2$ Hz, $J_{\text{HH}} = 3.6$ Hz, 2 H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 16.3 (d, $J_{\text{CP}} = 7.0$ Hz), 62.3 (d, $J_{\text{CP}} = 5.0$ Hz), 100.1 (d, $J_{\text{CP}} = 4.0$ Hz), 127.4 (d, $J_{\text{CP}} = 189.0$ Hz), 133.1 (d, $J_{\text{CP}} = 10.0$ Hz), 137.7 (d, $J_{\text{CP}} = 16.0$ Hz) ppm.

Diethyl 4-bromophenylphosphonate: Isolated as colorless oil (Table 1, entry 17: 95%, 278.3 mg).



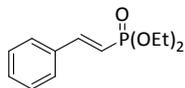
^1H NMR (400 MHz, CDCl_3): δ 1.20 (t, $J_{\text{HH}} = 7.2$ Hz, 6 H), 4.06-3.93 (m, 4 H), 7.32 (dd, $J_{\text{HH}} = 8.4$ Hz, $J_{\text{HH}} = 3.2$ Hz, 2 H), 7.63 (dd, $J_{\text{HH}} = 13.2$ Hz, $J_{\text{HH}} = 8.4$ Hz, 2 H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 16.2 (d, $J_{\text{CP}} = 7.0$ Hz), 62.1 (d, $J_{\text{CP}} = 5.0$ Hz), 126.9 (d, $J_{\text{CP}} = 190.0$ Hz), 128.7 (d, $J_{\text{CP}} = 15.0$ Hz), 133.0 (d, $J_{\text{CP}} = 10.0$ Hz), 138.7 (d, $J_{\text{CP}} = 4.0$ Hz) ppm.

Tetraethyl phenylbis(phosphonate): Isolated as white powder (Table 1, entry 18: 57%, 199.5 mg; entry 19: 78%, 273 mg)



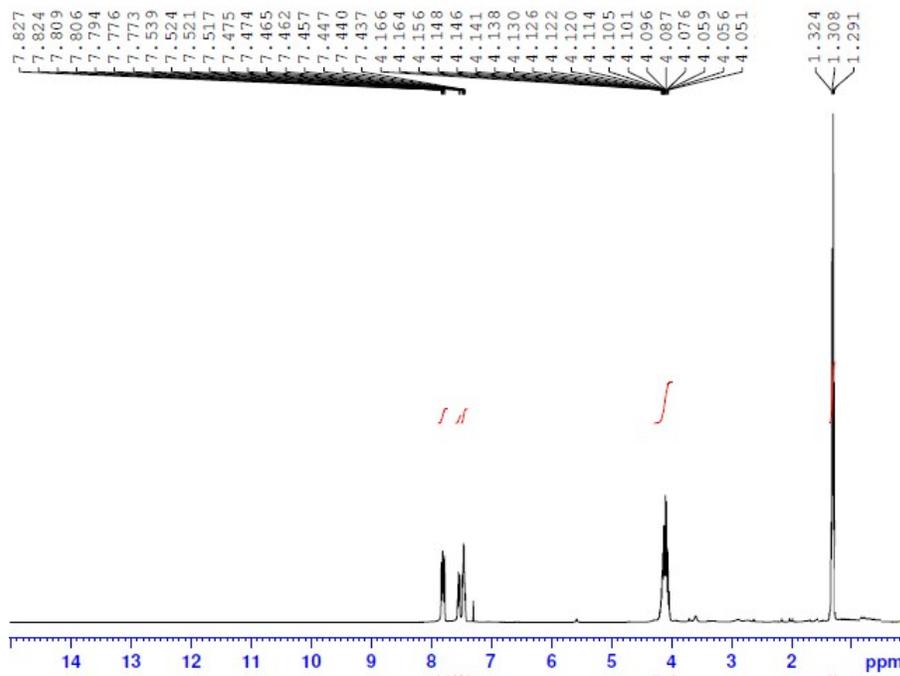
^1H NMR (400 MHz, CDCl_3): δ 1.33 (t, $J_{\text{HH}} = 7.2$ Hz, 12 H), 4.18 - 4.09 (m, 8 H), 7.90 (dd, $J_{\text{HH}} = 10.2$ Hz, $J_{\text{HH}} = 6.8$ Hz, 4 H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 16.3 (d, $J_{\text{CP}} = 7.0$ Hz), 62.4 (d, $J_{\text{CP}} = 5.0$ Hz), 128.0 (d, $J_{\text{CP}} = 155.0$ Hz), 131.6 (dd, $J_{\text{CP}} = 16.5$ Hz, $J_{\text{CP}} = 8.0$ Hz) ppm.

Diethyl 2-phenylvinylphosphonate: Isolated as colorless oil (Table 1, entry 22: 96%, 230.4 mg).



^1H NMR (400 MHz, CDCl_3): δ 1.37 (t, $J_{\text{HH}} = 6.8$ Hz, 6 H), 4.18-4.11 (m, 4 H), 6.3 (t, $J_{\text{HH}} = J_{\text{HP}} = 17.6$, 1H), 7.57-7.39 (m, 6H), ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 16.4 (d, $J_{\text{CP}} = 6.0$ Hz), 61.8 (d, $J_{\text{CP}} = 6.0$ Hz), 113.8 (d, $J_{\text{CP}} = 190.0$ Hz), 127.7, 128.1, 128.8, 130.2, 134.7, 134.9, 148.7 (d, $J_{\text{CP}} = 7.0$ Hz) ppm.

Copies of ^1H and ^{13}C NMR spectra

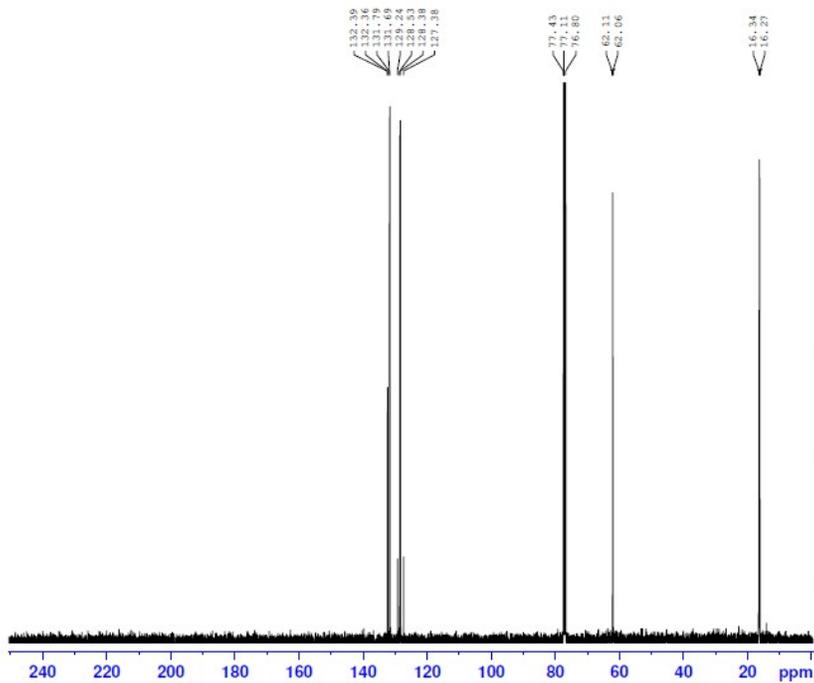


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LB        0.30 Hz
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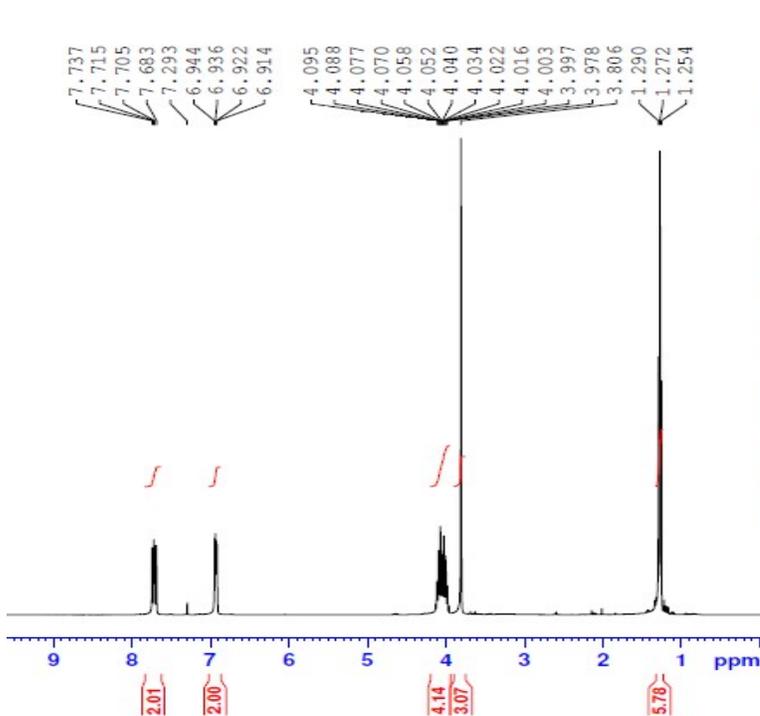
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PL12      14.16 dB
PL13      17.90 dB
PL2W      11.86359406 W
PL12W     0.28722104 W
PL13W     0.12139934 W
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SI        32768
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WDW       EM
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LB        1.00 Hz
GB        0
PC        1.40
  
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¹H NMR and ¹³C NMR of diethylphenylphosphonate

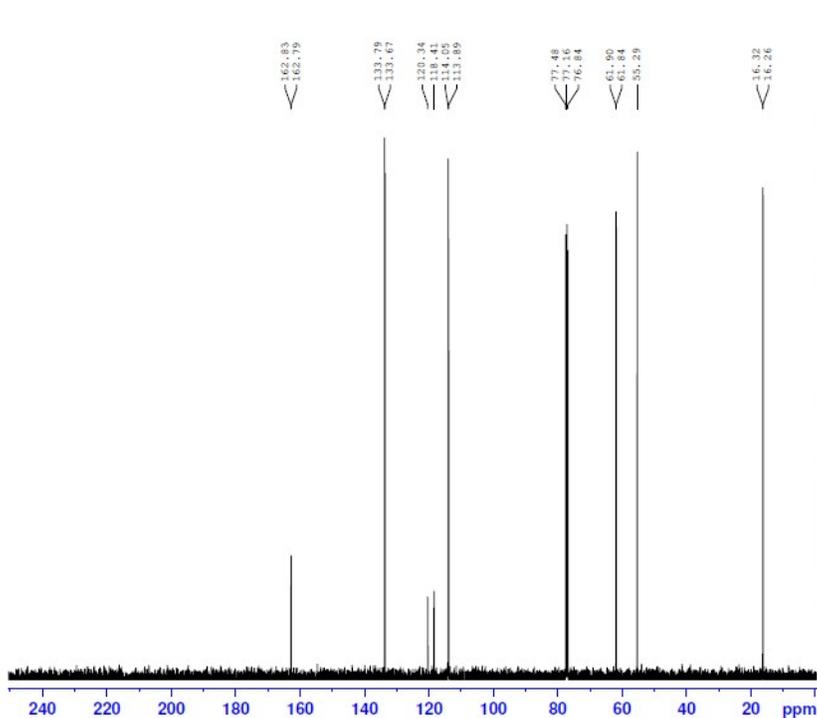


BRUKER

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NAME      Birjand UN
EXPNO     231
PROCNO    1
Date_     20141206
Time      8.06
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         20
DS         0
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894966 sec
RG         40.3
DW         62.400 usec
DE         6.50 usec
TE         292.6 K
D1         4.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.00 usec
PL1        -2.00 dB
PL1W       11.86359406 W
SFO1       400.2236020 MHz
SI         32768
SF         400.2200000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
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BRUKER

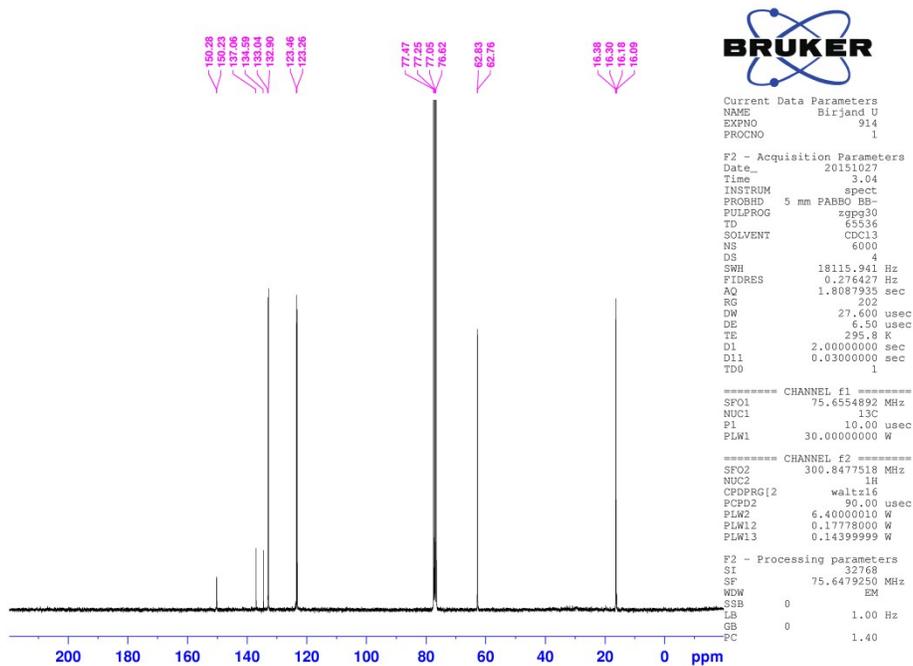
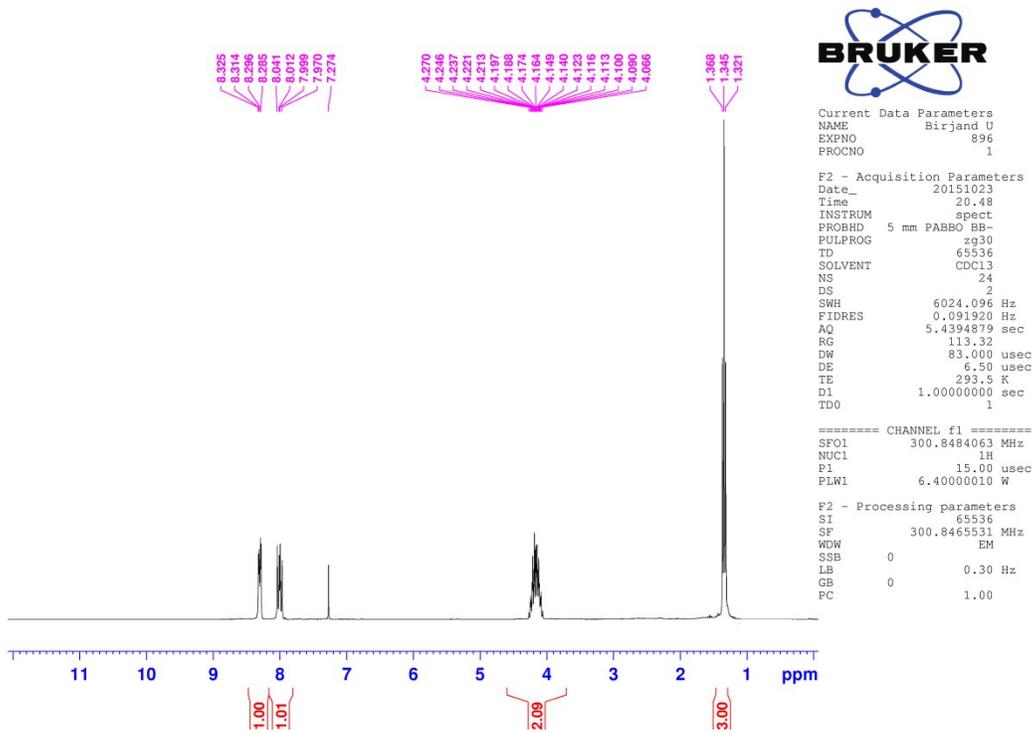
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NAME      Birjand UN
EXPNO     232
PROCNO    1
Date_     20141206
Time      8.10
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         30
DS         0
SWH        25252.525 Hz
FIDRES     0.385323 Hz
AQ         1.2976629 sec
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TE         292.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

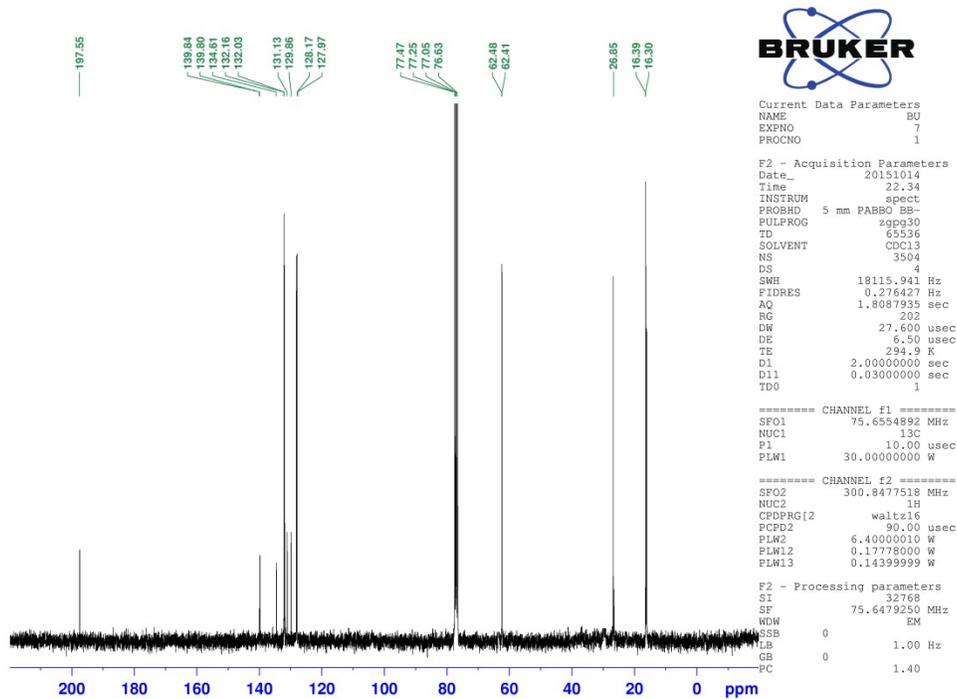
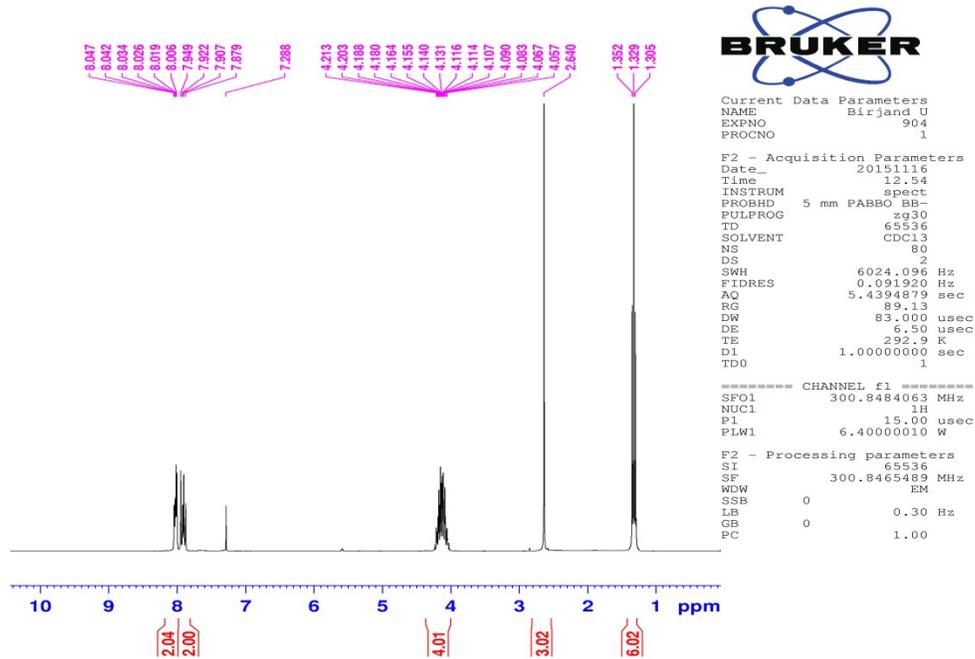
===== CHANNEL f1 =====
NUC1       13C
P1         9.00 usec
PL1        -0.90 dB
PL1W       42.02801895 W
SFO1       100.6479784 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2        -2.00 dB
PL12       14.16 dB
PL13       17.90 dB
PL2W       11.86359406 W
PL12W     0.28722104 W
PL13W     0.12139934 W
SFO2       400.2216009 MHz
SI         32768
SF         100.6353990 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
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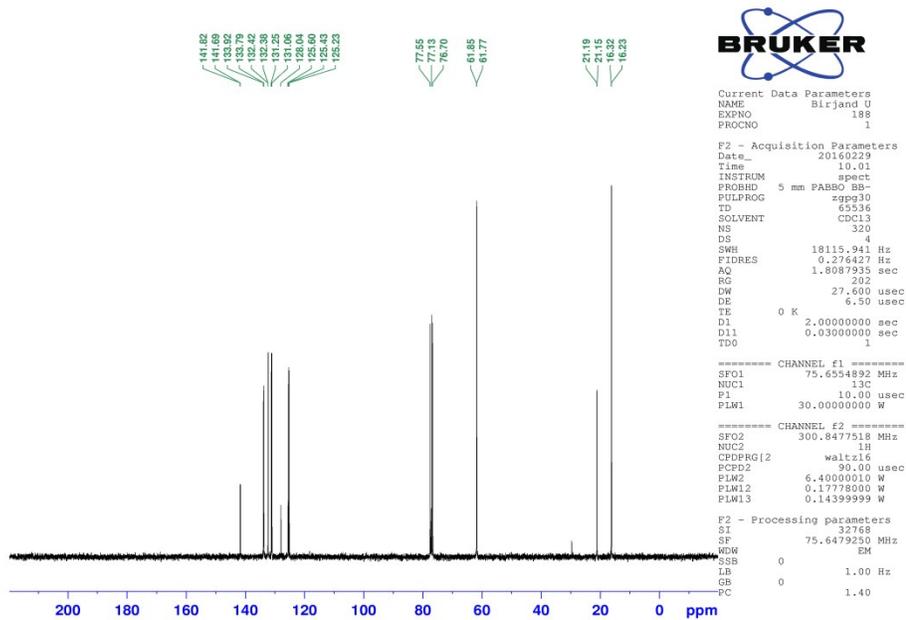
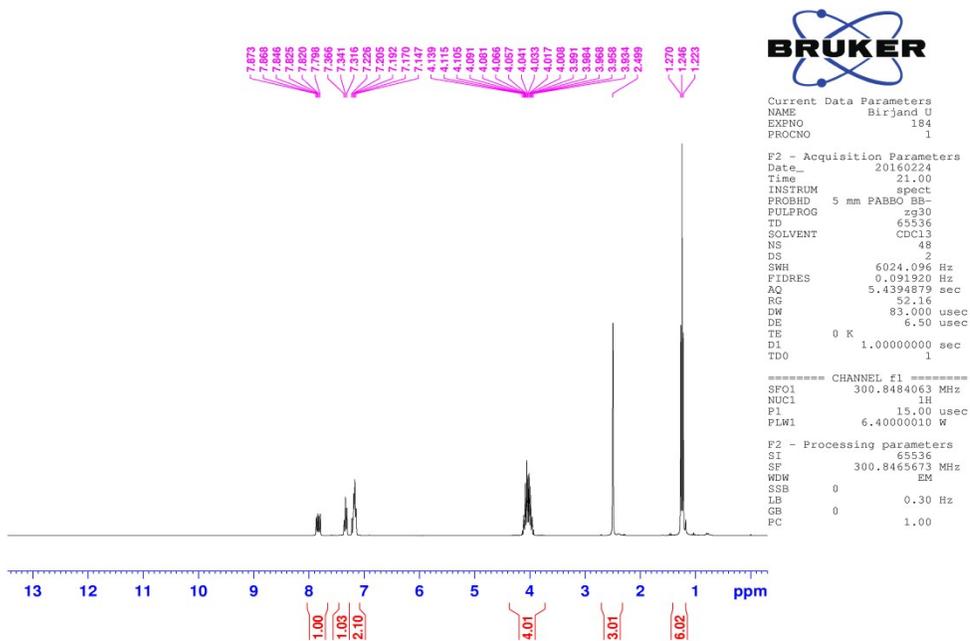
¹H NMR and ¹³C NMR of diethyl 4-methoxyphenylphosphonate



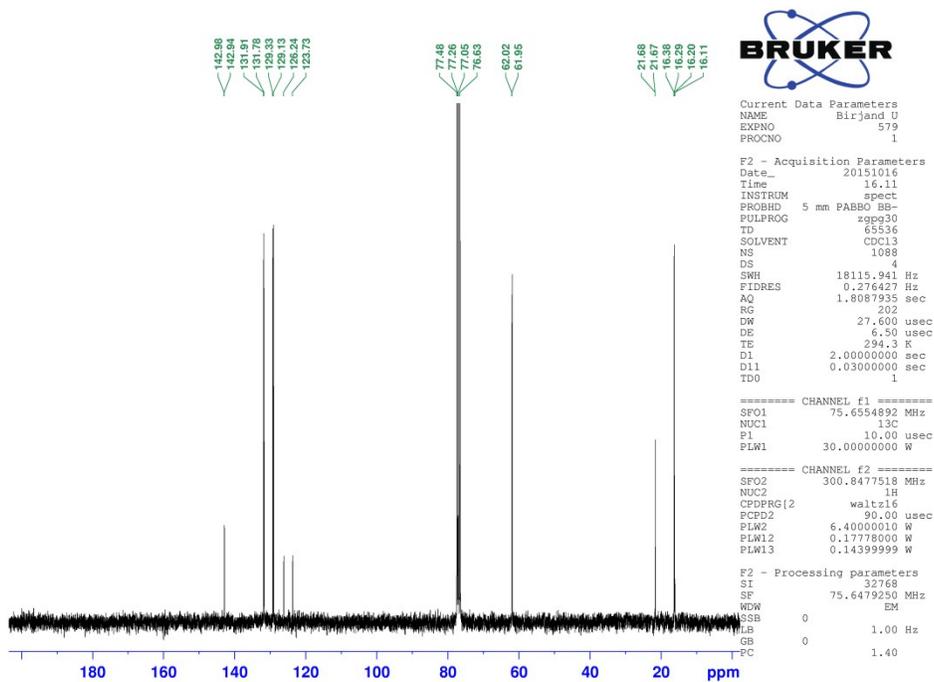
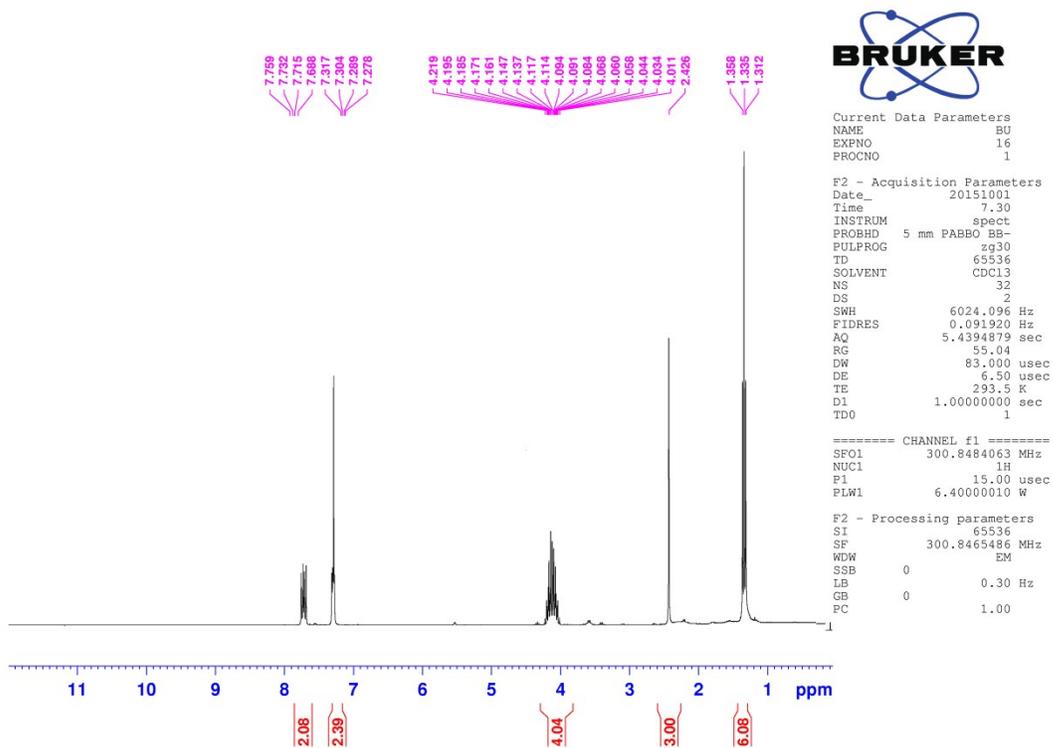
¹H NMR and ¹³C NMR of diethyl (4-nitrophenyl)phosphonate



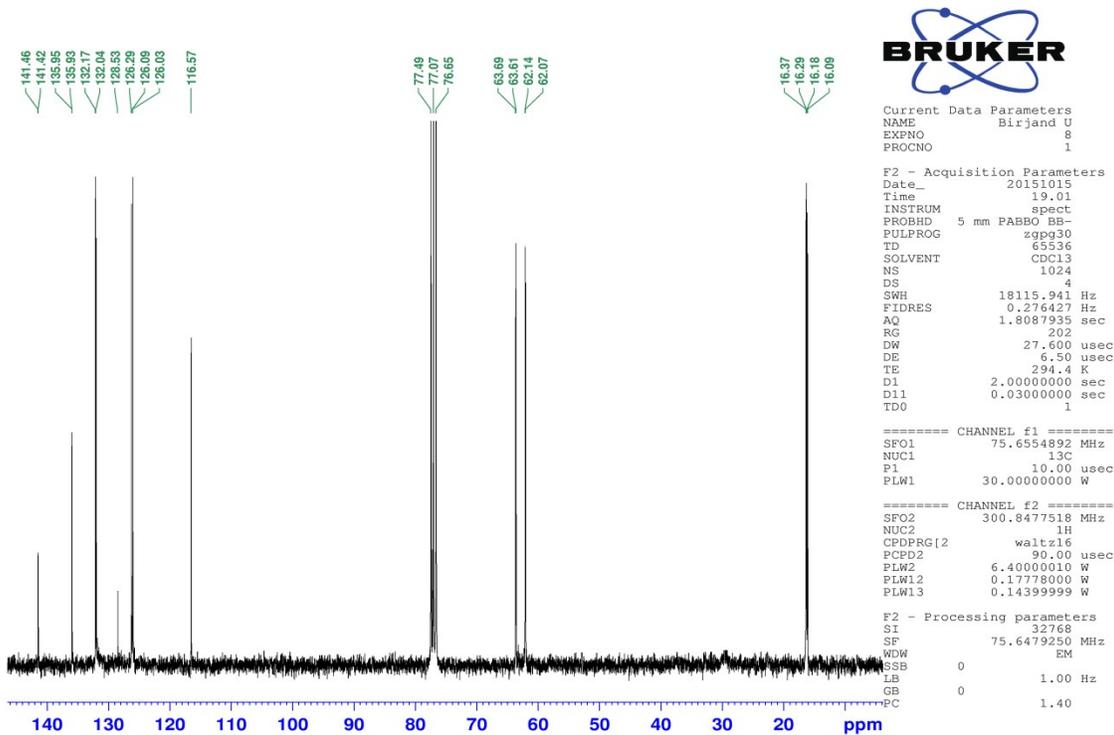
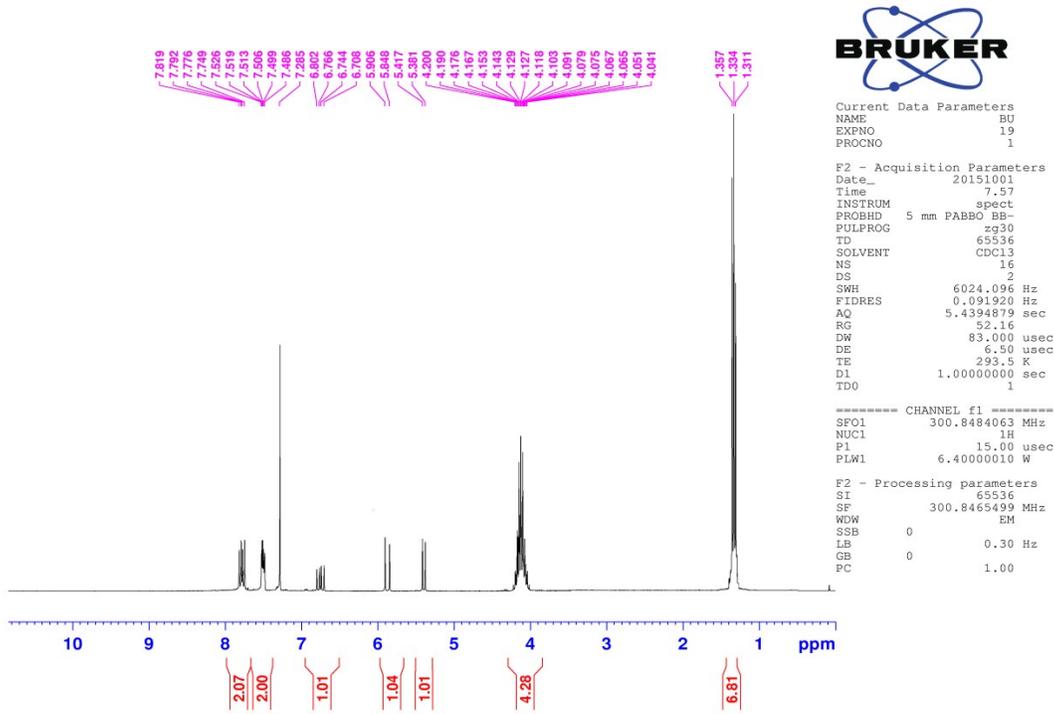
¹H NMR and ¹³C NMR of diethyl 4-acetylphenylphosphonate



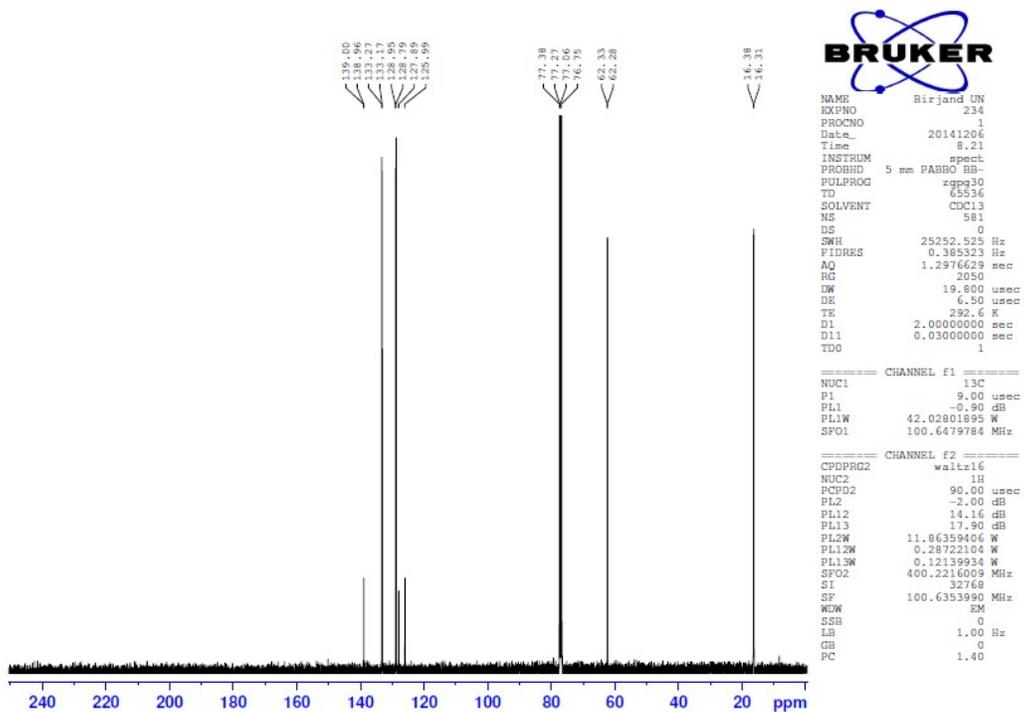
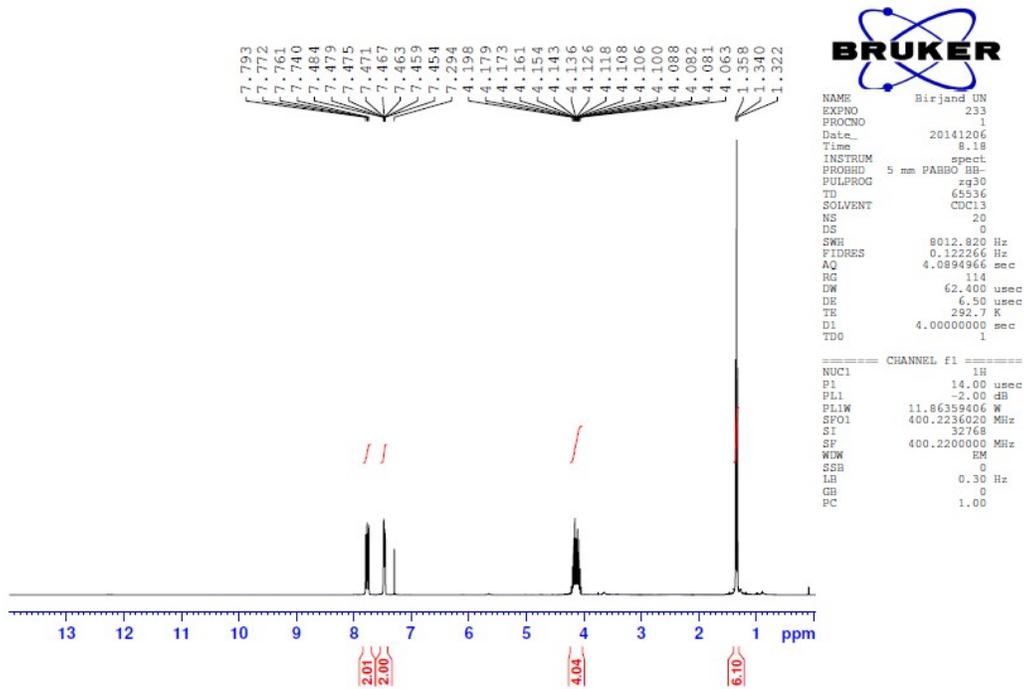
¹H NMR and ¹³C NMR of diethyl 2-methylphenylphosphonate



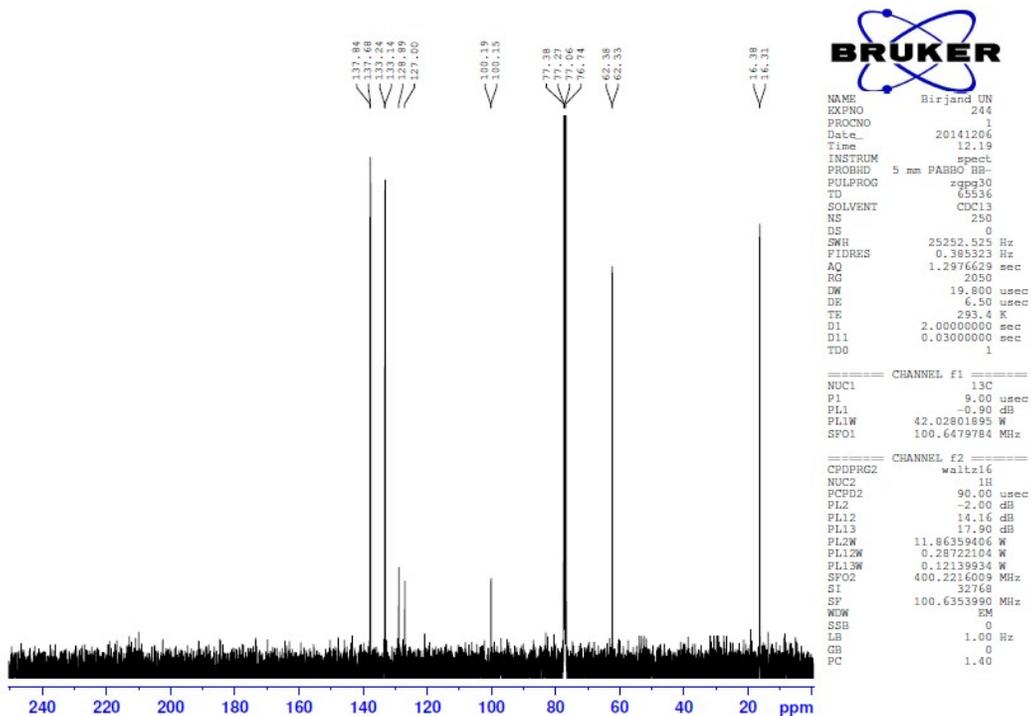
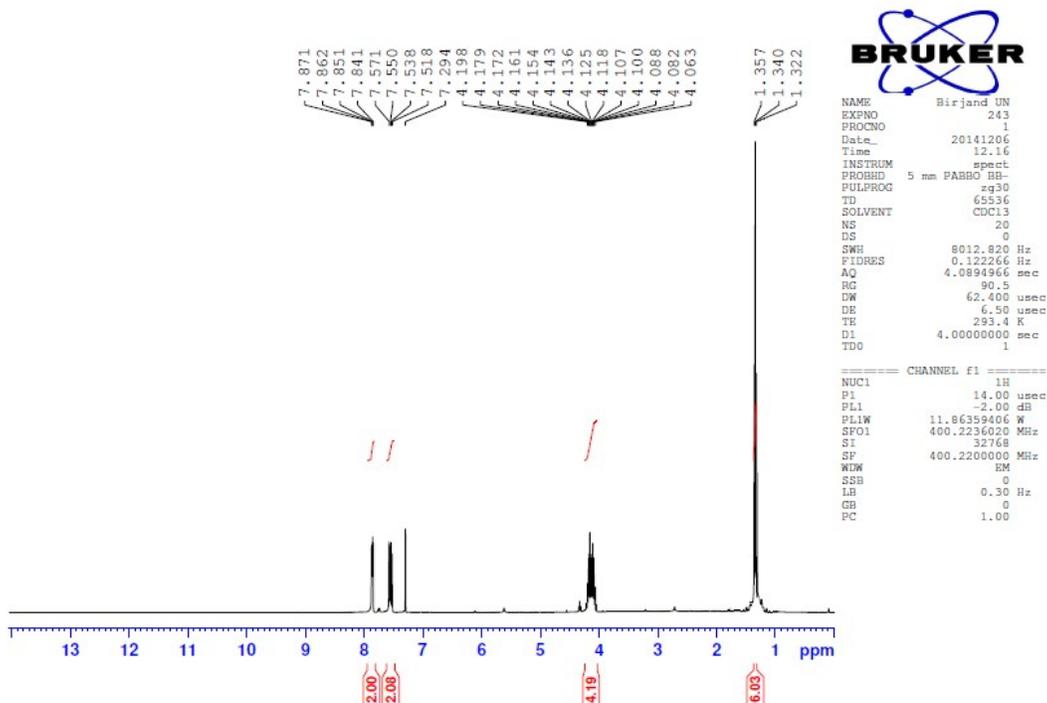
¹H NMR and ¹³C NMR of diethyl 4-tolylphosphonate



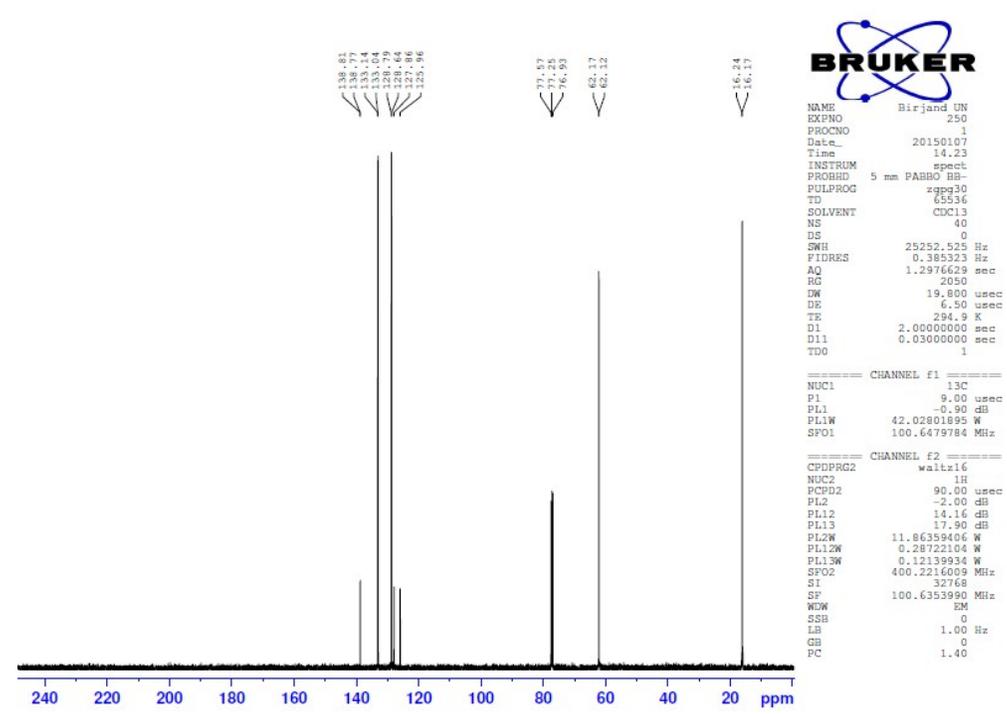
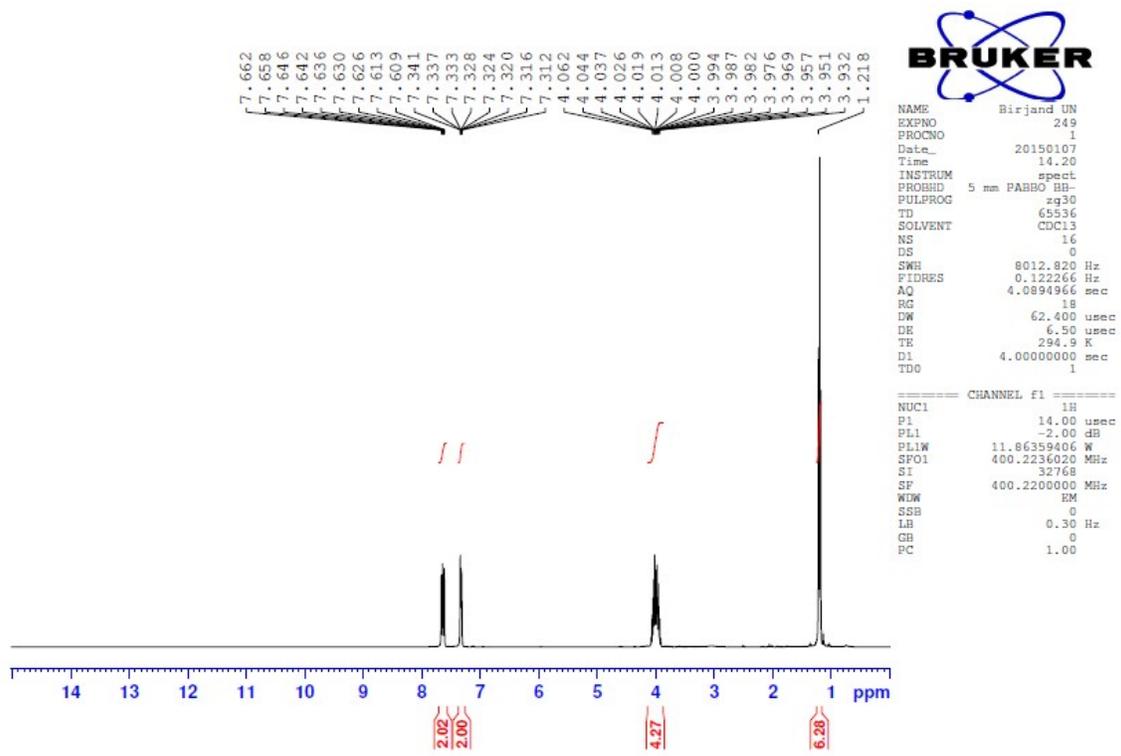
¹H NMR and ¹³C NMR of diethyl 4-vinylphenylphosphonate



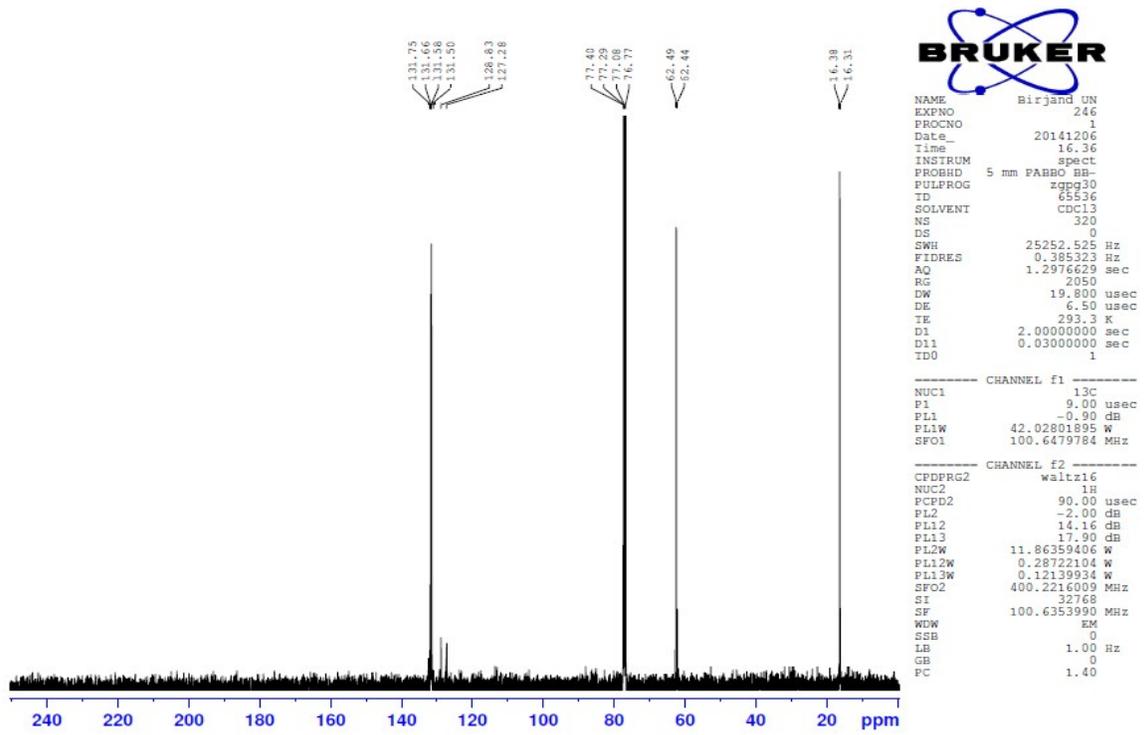
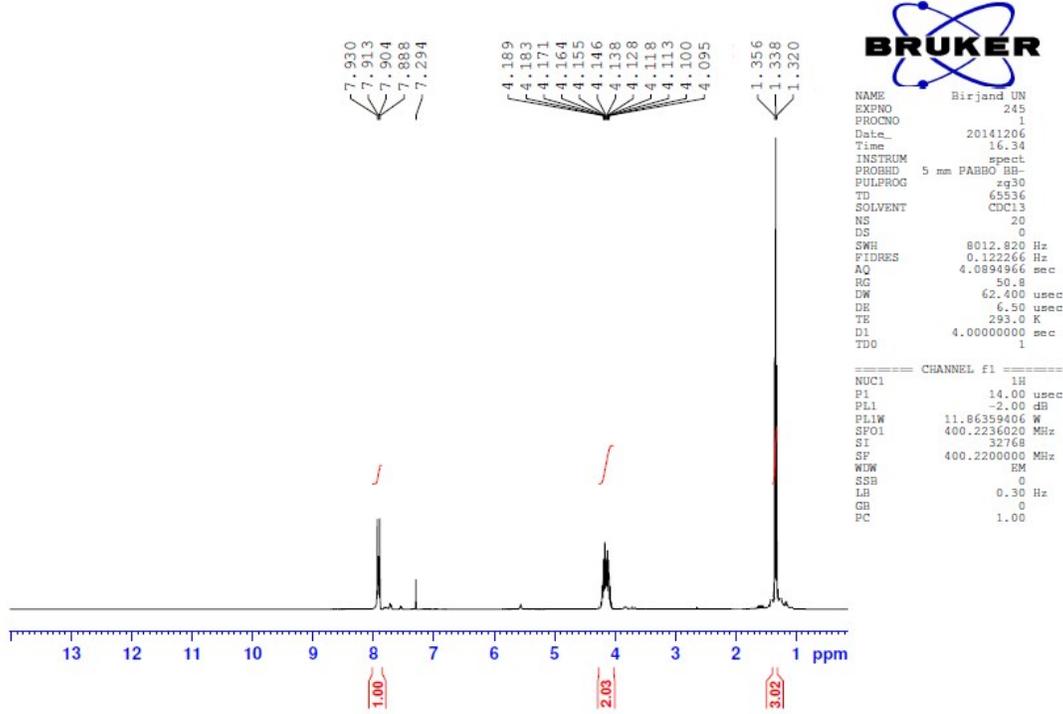
^1H NMR and ^{13}C NMR of diethyl 4-chlorophenylphosphonate



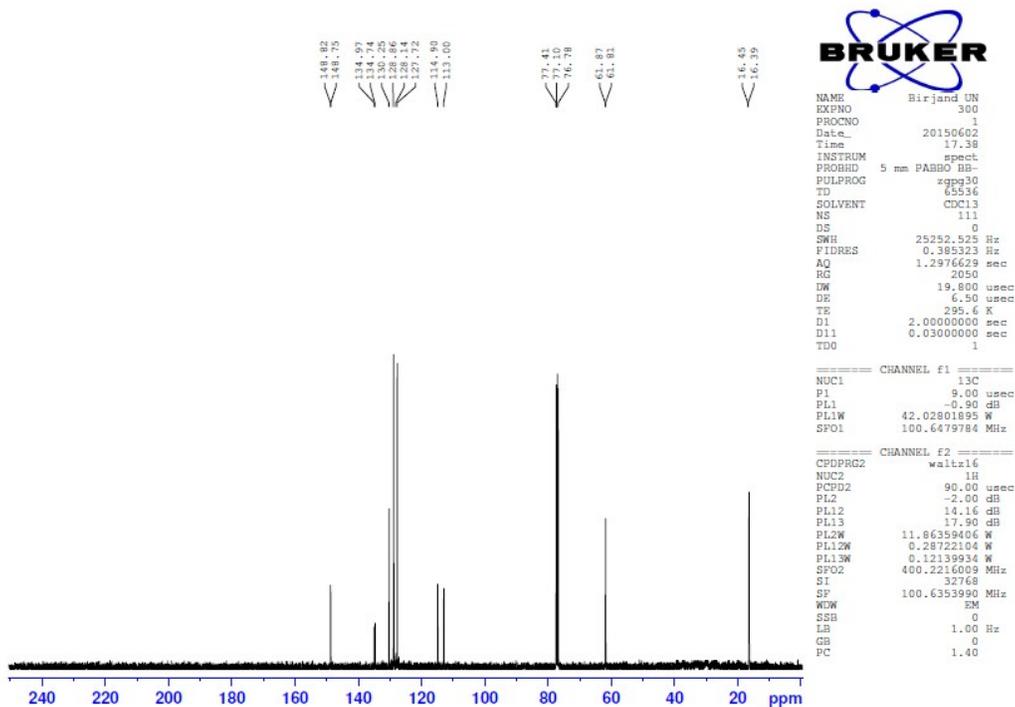
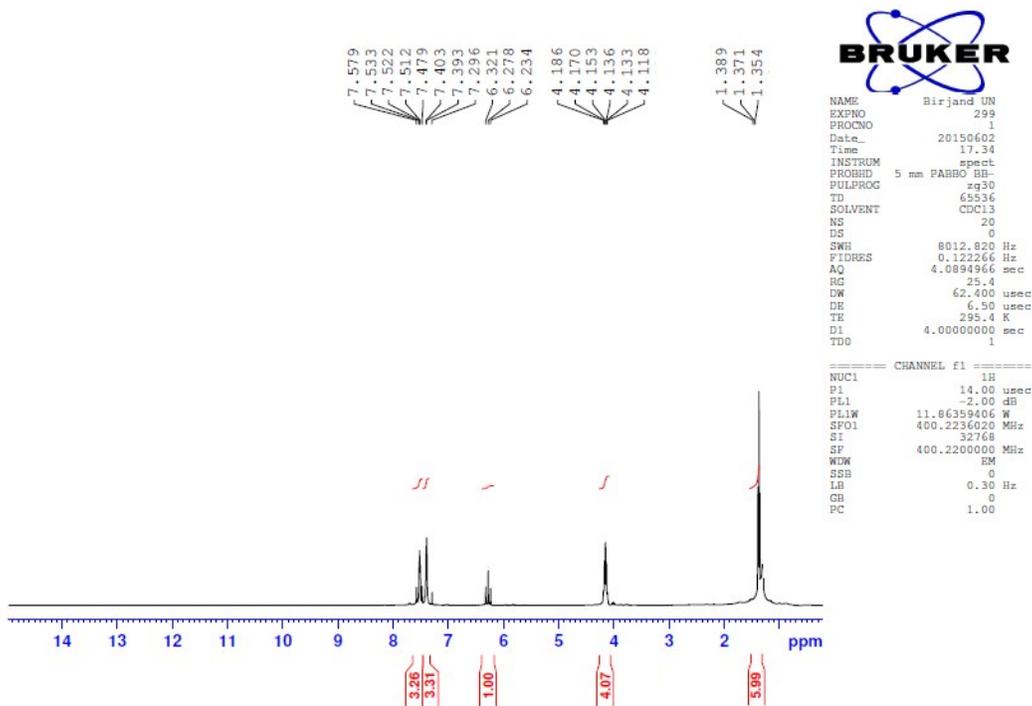
¹H NMR and ¹³C NMR of diethyl 4-iodophenylphosphonate



¹H NMR and ¹³C NMR of diethyl 4-bromophenylphosphonate



¹H NMR and ¹³C NMR of tetraethylphenylbis(phosphonate)



¹H NMR and ¹³C NMR of diethyl 2-phenylvinylphosphonate

References

- 1 (a) B. Z. Tang, Y. Geng, J. W. Y. Lam, B. Li, X. Jing, X. Wang, F. Wang, A. B. Pakhomov, X. X. Zhang, *Chem. Mater.* **1999**, *11*, 1581-1589; (b) K. M. Ho, P. Li, *Langmuir* **2008**, *24*, 1801-1807.
- 2 S. Sobhani, Z. Pakdin Parizi, *Appl. Catal. A: Gen.* **2014**, *479*, 112-120.