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Supporting information

Lanthanum (III) triflate supported on nanomagnetic γ-Fe_2O_3: A new magnetically recyclable heterogeneous Lewis acid for the one-pot synthesis of β-phosphonomalonates

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Contents

1) General Information

Chemicals were purchased from Merck Chemical Company. NMR spectra were recorded on a Bruker Avance DPX-250 and 400 in $CDCl_3$ 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/UV_{254} plates. HRTEM analysis was performed using HRTEM microscope (Philips CM30). FT-IR spectra were recorded on a Shimadzu Fourier Transform Infrared Spectrophotometer (FT-IR-8300). IR spectra were run on a Perkin Elmer 780 instrument. Mass spectra were recorded on a Shimadzu GCMS-QP5050A. Thermo gravimetric analysis (TGA) was performed using a Shimadzu thermo gravimetric analyser (TG-50). Power X-ray diffraction (XRD) was performed on a Bruker D8-advance X-ray diffractometer with Cu K_α (λ = 0.154 nm) radiation. Room temperature magnetization isotherms were obtained using a vibrating sample magnetometer (VSM, LakeShore 7400).The content of La in the catalyst was determined by ICP-OES VISTA-PRO inductively coupled plasma analyzer.

2) Spectral Data of Unknown β-Phosphonomalonates

[1-(3-Bromophenyl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (6)

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.14 (t, $^3J_{HH}$ = 7.2 Hz, 3 H, CH_3), 1.31 (t, $^3J_{HH}$ = 7.2 Hz, 3 H, CH_3), 3.65 (dd, $^3J_{HH}$ = 7.6 Hz, $^2J_{HP}$ = 21.6 Hz, 1 H, CH), 3.81-4.19 (m, 4 H, CH_2), 4.7 (dd, $^3J_{HH}$ = 8.0 Hz, $^2J_{HP}$ = 9.2 Hz, 1 H, CH), 7.28 (t, $^3J_{HH}$ = 8.0 Hz, 1 H, Ar), 7.44 (d, $^3J_{HH}$ = 7.2 Hz, 1 H, Ar), 7.52 (d, $^3J_{HH}$ = 8.0 Hz, 1 H, Ar), 7.62 (s, 1 H, Ar). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 16.1 (d, $^3J_{CP}$ = 5.0 Hz), 16.2 (d, $^3J_{CP}$ = 6.0 Hz), 25.3, 43.8 (d, $^1J_{CP}$ = 143.0 Hz), 63.6 (d, $^2J_{CP}$ = 7.0 Hz), 64.4 (d, $^2J_{CP}$ = 7.0 Hz), 111.3 (d, $^3J_{CP}$ = 12.1 Hz), 111.4 (d, $^3J_{CP}$ = 11.1 Hz), 123.1, 127.9 (d, $^3J_{CP}$ = 6.0 Hz), 130.8, 132.4 (d, $^2J_{CP}$ = 6.0 Hz), 132.6, 132.8 (d, $^3J_{CP}$ = 6.0 Hz). MS (EI, 70 eV): *m/z* (%) = 370 (22 M^+), 372 (22 M^++2), 207 (80), 209 (80), 138 (100%). IR (KBr): 2242 (CN), 1003 (P=O)

cm^{-1}. Anal. Calcd for $C_{14}H_{16}BrN_2O_3P$: C, 45.30; H, 4.34; N, 7.5. Found: C, 44.33; H, 4.12; N, 7.39.

[1,1-Dicyanopentan-2-yl] Phosphonic Acid Diethyl Ester (13)

^1H NMR (250 MHz, $CDCl_3$): δ = 1.00 (t, $^3J_{HH}$ = 7.0 Hz, 3 H, CH_3), 1.37 (t, $^3J_{HH}$ =7.0 Hz, 6 H, CH_3), 1.61 (q, $^3J_{HH}$ = 7.0 Hz, 2 H, CH_2), 1.73-2.10 (m, 2 H, CH_2), 2.29-2.34 (m, 1 H, CH), 4.16-4.25 (m, 4 H, CH_2), 4.29-4.35 (m, 1 H, CH). ^{13}C NMR (62.9 MHz, $CDCl_3$): δ = 13.7, 16.3 (d, $^3J_{CP}$ = 5.6 Hz), 20.7 (d, $^2J_{CP}$ = 7.6 Hz), 23.7, 29.2, 37.8 (d, $^1J_{CP}$ = 144.9 Hz), 63.2 (d, $^2J_{CP}$ = 11.0 Hz), 110.7 (d, $^3J_{CP}$ = 5.0 Hz), 112.2 (d, $^3J_{CP}$ = 17.0 Hz). MS (EI, 70 eV): *m/z* (%) = 259 (5 M^++1), 138 (100), 121 [20 M^+-P(O)$(OEt)_2$]. IR (KBr): 2240 (CN), 1011 (P=O) cm^{-1}. Anal. Calcd for $C_{11}H_{19}N_2O_3P$: C, 51.16; H, 7.42; N, 10.85. Found: C, 50.99; H, 7.57; N, 10.71.

[1, 1-Dicyanooctan-2-yl] Phosphonic Acid Diethyl Ester (14)

^1H NMR (400 MHz, $CDCl_3$): δ = 0.81 (t, $^3J_{HH}$ = 6.8 Hz, 3 H, CH_3), 1.23-1.25 (m, 6 H, CH_3), 1.29 (t, $^3J_{HH}$ = 7.2 Hz, 6 H, CH_2), 1.45-1.50 (m, 2 H, CH_2), 1.66-1.79 (m, 1 H), 1.86-1.98 (m, 1 H, CH_2), 2.25-2.34 (m, 1 H, CH), 4.08-4.16 (m, 4 H, CH_2), 4.36 (dd, $^3J_{HP}$ = 13.2 Hz, $^3J_{HH}$ = 3.6 Hz, 1 H, CH). ^{13}C NMR (62.9 MHz, $CDCl_3$): δ = 13.9, 16.21 (d, $^3J_{CP}$ = 5.0 Hz), 22.3, 23.7, 27.1, 27.2 (d, $^3J_{CP}$ = 6.0 Hz), 28.8, 31.2, 37.7 (d, $^1J_{CP}$ = 143.0 Hz), 63.1 (d, $^2J_{CP}$ = 7.0 Hz), 111.1 (d, $^3J_{CP}$ = 3.0 Hz), 112.3 (d, $^3J_{CP}$ = 17.0 Hz). MS (EI, 70 eV): *m/z* (%) = 301 (2 M^++1), 163 [21 M^+-P(O)$(OEt)_2$], 138 (100), 111 (58), 81 (70). IR (KBr): 2238 (CN), 1011 (P=O) cm^{-1}. Anal. Calcd for $C_{14}H_{25}N_2O_3P$: C, 55.99; H, 8.39; N, 9.33. Found: C, 55.36; H, 8.03; N 9.20.

3) Spectral Data of Known β-Phosphonomalonates

[1-Phenyl-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (1)

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.11 (t, $^{3}J_{HH}$ = 6.8 Hz, 3 H, CH_3), 1.33 (t, $^{3}J_{HH}$ = 7.0 Hz, 3 H, CH_3), 3.65 (dd, $^{3}J_{HH}$ = 8.0, $^{2}J_{HP}$ = 21.0 Hz, 1 H, CH), 3.91-4.21 (m, 4 H, CH_2), 4.55 (t, $^{3}J_{HH}$ = 8.3 Hz, 1 H, CH), 7.43 (s, 5 H, Ar).

[1-(4-Chlorophenyl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (2)

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.16 (t, $^{3}J_{HH}$ = 7.0 Hz, 3 H, CH_3), 1.33 (t, $^{3}J_{HH}$ = 7.0 Hz, 3 H, CH_3), 3.62 (dd, $^{2}J_{HP}$ = 21.5, $^{3}J_{HH}$ = 7.5 Hz, 1 H, CH), 3.82–4.19 (m, 4 H, CH_2), 4.55 (t, $^{3}J_{HH}$ = 7.7 Hz, 1 H, CH), 7.42 (s, 4 H, Ar).

[1-(3-Chlorophenyl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (3)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.20 (t, $^{3}J_{HH}$ = 7.2 Hz, 3 H, CH_3), 1.38 (t, $^{3}J_{HH}$ = 7.2 Hz, 3 H, CH_3), 3.60 (dd, $^{3}J_{HH}$ = 8.0 Hz, $^{2}J_{HP}$ = 21.2 Hz, 1 H, CH), 3.83–3.93 (m, 1 H, CH), 4.03-4.26 (m, 3 H, CH_2), 4.56 (dd, $^{3}J_{HP}$ = 9.2 Hz, $^{3}J_{HH}$ = 8.0 Hz,1 H, CH), 7.41-7.45 (m, 3 H, Ar), 7.50 (s, 1 H, Ar).

[1-(2-Chlorophenyl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (4)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.11 (t, $^{3}J_{HH}$ = 7.0 Hz, 3 H, CH_3), 1.36 (t, $^{3}J_{HH}$ = 7.0 Hz, 3 H, CH_3), 3.75-4.30 (m, 4 H, CH_2), 4.46 (dd, $^{2}J_{HP}$ = 21.2 Hz, $^{3}J_{HH}$ = 8.2, 1 H, CH), 4.61 (t, $^{3}J_{HH}$ = 8.5 Hz, 1 H, CH), 7.35 (d, $^{3}J_{HH}$ = 4 Hz, 2 H, Ar), 7.47 (s, 1 H, Ar), 7.75 (d, $^{3}J_{HH}$ = 5.3 Hz, 1 H, Ar).

[1-(4-Bromophenyl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (5)

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.16 (t, $^{3}J_{HH}$ = 7.0 Hz, 3 H, CH_3), 1.33 (t, $^{3}J_{HH}$ = 7.0 Hz, 3 H, CH_3), 3.58 (dd, $^{2}J_{HP}$ = 21.5 Hz, $^{3}J_{HH}$ = 7.5 Hz, 1 H, CH), 3.79–4.18 (m, 4 H, CH_2), 4.56 (t, $^{3}J_{HH}$ = 7.75 Hz, 1 H, CH), 7.36 (d, $^{3}J_{HH}$ = 8.25 Hz, 2 H, Ar), 7.56 (d, $^{3}J_{HH}$ = 8.0 Hz, 2 H, Ar).

^{13}C NMR (62.9 MHz, $CDCl_3$): δ = 16.1 (d, $^{3}J_{CP}$ = 4.4 Hz), 16.2 (d, $^{3}J_{CP}$ = 5.7 Hz), 25.4, 43.9 (d, $^{1}J_{CP}$ = 144.7 Hz), 63.6 (d, $^{2}J_{CP}$ = 7.5 Hz), 64.4 (d, $^{2}J_{CP}$ = 7.5 Hz), 111.0 (d, $^{3}J_{CP}$ = 11.9 Hz), 111.2 (d, $^{3}J_{CP}$ = 10.7 Hz), 123.9, 129.4, 131.0, 132.5. ^{31}P NMR (101 MHz, $CDCl_3$): δ = 19.27. MS (EI,

70 eV): *m/z* (%) = 370 (13 M^+), 372 (8 M^++2), 233 [8 M^+-P(O)$(OEt)_2$], 235 [9 (M^++2)-P(O)$(OEt)_2$], 207 (100), 209 (97), 138 (64). IR (KBr): 2242 (CN), 1000 (P=O) cm^{-1}. Anal. Calcd for $C_{14}H_{16}BrN_2O_3P$: C, 45.30; H, 4.34; N, 7.55. Found: C, 44.46; H, 4.15; N, 7.39.

[1-(4-Methoxy)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (7)

^{1}H NMR (400 MHz, $CDCl_3$): δ =1.17 (t, $^3J_{HH}$ = 7.2 Hz, 3 H, CH_3), 1.37 (t, $^3J_{HH}$ = 7.2 Hz, 3 H, CH_3), 3.57 (dd, $^2J_{HP}$ = 21.2, $^3J_{HH}$ = 8.0 Hz, Hz, 1 H, CH), 3.84 (s, 3 H, CH_3), 4.00–4.24 (m, 4 H, CH_2), 4.51 (dd, $^3J_{HP}$ = 8.8 Hz, $^3J_{HH}$ = 8.0 Hz, 1 H, CH), 6.97 (d, $^3J_{HH}$ = 8.8 Hz, 2 H, Ar), 7.41-7.44 (m, 2 H, Ar).

[1-(3-Methoxyphenyl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (8)

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.11 (t, $^3J_{HH}$ = 6.7 Hz, 3 H, CH_3), 1.31 (t, $^3J_{HH}$ = 6.5 Hz, 3H, CH_3), 3.56 (dd, $^2J_{HP}$ = 21.0 Hz, $^3J_{HH}$ = 7.8 Hz, 1H, CH), 3.78 (s, 3H, CH_3), 3.94-4.16 (m, 4H, CH_2), 4.57 (t, $^3J_{HH}$, $^3J_{HP}$ = 8.0 Hz, 1H, CH), 6.91 (d, $^3J_{HH}$ = 7.5 Hz, 2H, Ar), 7.00 (s, 1H, Ar), 7.30 (t, $^3J_{HH}$ = 7.5 Hz, 1H, Ar). ^{13}C NMR (62.9 MHz, $CDCl_3$, TMS, ppm): δ = 16.1 (d, $^3J_{CP}$ = 6.3 Hz), 16.2 (d, $^3J_{CP}$ = 6.9 Hz), 25.5, 44.5 (d, $^1J_{CP}$ = 144.9 Hz), 55.3, 63.4 (d, $^2J_{CP}$ = 7.6 Hz), 64.4 (d, $^2J_{CP}$ = 6.9 Hz), 111.2 (d, $^3J_{CP}$ = 11.2 Hz), 111.3 (d, $^3J_{CP}$ = 10.0 Hz),114.9, 121.4, 130.4, 131.7, 160.1, 160.3. MS (EI, 70 eV): *m/z* (%) = 322 (M^+), 185 [M^+– P(O)$(OEt)_2$]. Anal. Calcd for $C_{15}H_{19}N_2O_4P$: C, 55.90; H, 5.94; N, 8.69. Found: C, 55.67; H, 5.92; N, 8.67.

[1-(4-Hydroxyphenyl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (9)

^{1}H NMR (250 MHz, $CDCl_3$): 1.19 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3), 1.38 (t, $^3J_{HH}$ = 7.0 Hz, 3H, CH_3), 3.53 (dd, $^3J_{HH}$ = 7.8 Hz, $^2J_{HP}$ = 21.3 Hz, 1H, CH), 3.79-4.23 (m, 4H, CH_2), 4.43 (t, 1H, $^3J_{HH}$, $^3J_{HP}$ = 8.0 Hz), 6.8 (d, $^3J_{HH}$ = 8.4 Hz, 2H, Ar), 7.26 (d, $^3J_{HH}$ = 8.4 Hz, 2H, Ar). ^{13}C NMR (62.9 Hz, $CDCl_3$, TMS, ppm): δ = 16.3, 25.7, 43.9 (d, $^1J_{CP}$ = 144.7 Hz), 63.8 (d, $^2J_{CP}$ = 7.5 Hz), 64.6 (d, $^2J_{CP}$ = 7.5 Hz), 111.0 (d, $^3J_{CP}$ = 11.2 Hz), 111.2 (d, $^3J_{CP}$ = 10.0 Hz) 116.6, 120.7, 130.5,

157.6. MS (EI, 70 eV): *m/z* (%) = 308 (M^+), 171 [M^+– $P(O)(OEt)_2$]. Anal. Calcd for $C_{14}H_{17}N_2O_4P$: C, 54.55; H, 5.56; N, 9.09. Found: C, 54.54; H, 5.54; N, 9.06.

[1-(4-Methyl)-2,2-dicyanoethyl] Phosphonic Pcid Diethyl Ester (10)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.16 (t, $^3J_{HH}$ = 7.2 Hz, 3 H, CH_3), 1.38 (t, $^3J_{HH}$ = 7.2 Hz, 3 H, CH_3), 2.40 (s, 3 H, CH_3), 3.57 (dd, $^3J_{HH}$ = 8.0 Hz, $^2J_{HP}$ = 21.2 Hz ,1 H, CH), 3.74–3.84 (m, 2 H, CH_2), 3.99- 4.06 (m, 2 H, CH_2), 4.50 (dd, $^3J_{HH}$ = 8.4 Hz, $^3J_{HP}$ = 9.0 Hz, 1 H, CH), 7.25-7.29 (m, 2 H, Ar), 7.37-7.39 (m, 2 H, Ar).

[1-(Pyridin-3-yl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (11)

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.18 (t, $^3J_{HH}$ = 6.8 Hz, 3 H, CH_3), 1.33 (t, $^3J_{HH}$ =7.0 Hz, 3 H, CH_3), 3.65 (dd, $^2J_{HP}$ = 21.6 Hz, $^3J_{HH}$ = 6.8 Hz, 1 H, ch), 3.92-4.21 (m, 4 H, CH_2), 4.63 (t, $^3J_{HH}$ = 8.5 Hz, 1 H, CH), 7.39 (t, $^3J_{HH}$ = 6.5 Hz, 1 H, CH), 7.95 (d, $^3J_{HH}$ = 6.5 Hz, 1 H, Ar), 8.67 (s, 2 H, Ar). ^{13}C NMR (62.9 MHz, $CDCl_3$): δ = 16.1 (d, $^3J_{CP}$ = 5.0 Hz), 16.2 (d, $^3J_{CP}$ = 5.0 Hz), 25.3, 42.1 (d, $^1J_{CP}$ = 144.6 Hz), 63.8 (d, $^2J_{CP}$ = 7.0 Hz), 64.5 (d, $^2J_{CP}$ = 7.0 Hz), 110.8 (d, $^3J_{CP}$ = 10.7 Hz), 111.0 (d, $^3J_{CP}$ = 11.9 Hz), 124.0, 126.7, 136.5, 150.5, 150.8. ^{31}P NMR (101 MHz, $CDCl_3$): δ = 19.03. MS (EI, 70 eV): *m/z* (%) = 293 (9 M^+), 156 [100 M^+-$P(O)(OEt)_2$], 138 (94), 130 (25), 111 (93). IR (KBr): 2236 (CN), 1011 (P=O) cm^{-1}. Anal. Calcd for $C_{13}H_{16}N_3O_3P$: C, 53.24; H, 5.50; N, 14.33. Found: C, 52.71; H, 5.32; N, 14.04.

[1-(Furan-2-yl)-2,2-dicyanoethyl] Phosphonic Acid Diethyl Ester (12)

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.24-1.37 (m, 6 H, CH_3), 3.87 (dd, $^2J_{HP}$ = 22.7 Hz, $^3J_{HH}$ = 6.5 Hz, 1 H, CH), 3.98- 4.23 (m, 4 H, CH_2), 4.51 (t, $^3J_{HH}$ = 8.7 Hz, 1 H, CH), 6.44 (s, 1 H, Ar), 6.62 (s, 1 H, Ar), 7.49 (s, 1 H, Ar).

[1-(4-Chlorophenyl)-2,2-dicyanoethyl] Phosphonic Acid Dimethyl Ester (15)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.61-3.68 (m, 4 H, CH_3), 3.86 (d, $^{3}J_{HP}$ = 11.2 Hz, 3 H, CH_3, CH), 4.51 (t, $^{3}J_{HH}$ = 8.0 Hz, 1 H, CH), 7.47 (s, 4 H, Ar).

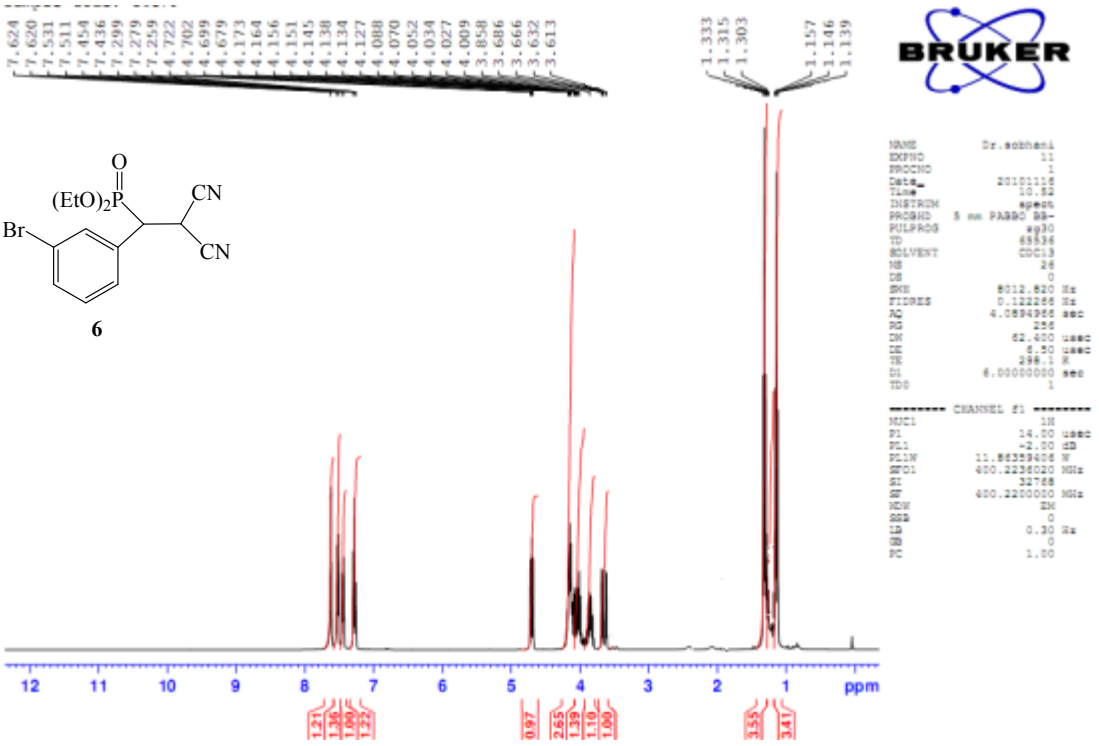
[1-(4-Chlorophenyl)-2,2-dicyanoethyl] Phosphonic Acid Di-*iso*-propyl Ester (16)

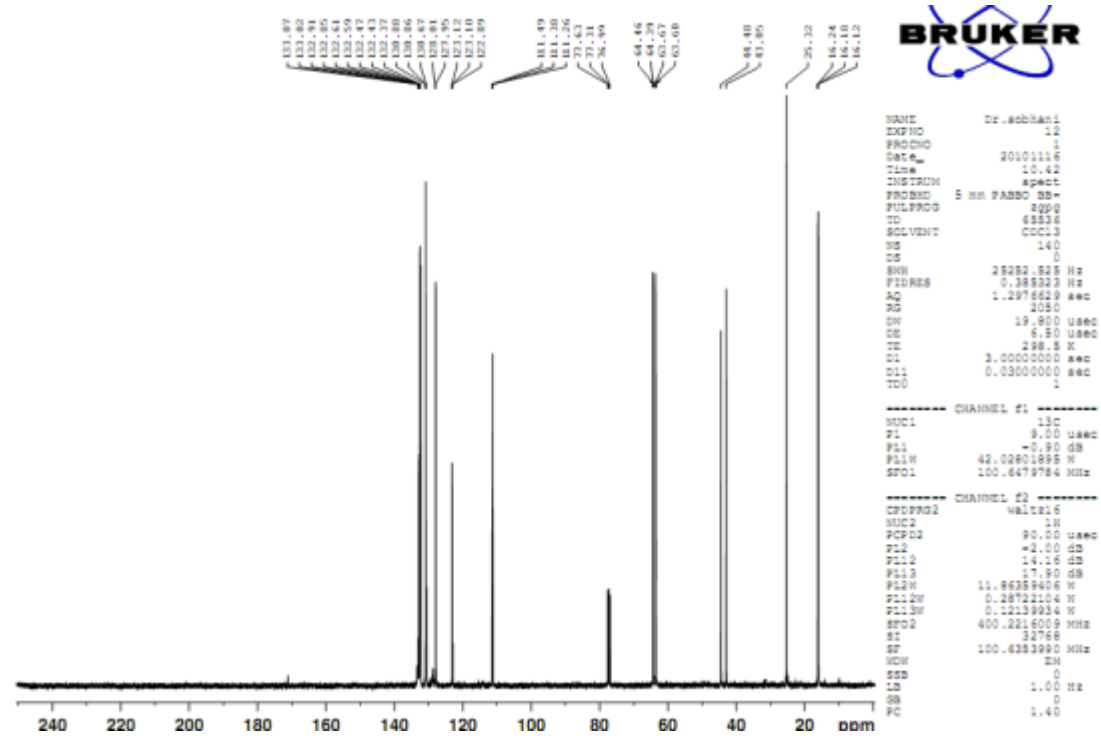
^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.00 (d, $^{3}J_{HH}$ = 6.4 Hz, 3 H, CH_3), 1.32 (d, $^{3}J_{HH}$ = 6.0 Hz, 3 H, CH_3), 1.39 (d, $^{3}J_{HH}$ = 6.0 Hz, 6 H, CH_3), 3.51 (dd, $^{2}J_{HP}$ = 21.6 Hz, $^{3}J_{HH}$ = 7.2 Hz, 1 H, CH), 4.51–4.57 (m, 2 H, CH), 4.75–4.83 (m, 1 H, CH), 7.43-7.50 (m, 4 H, Ar).

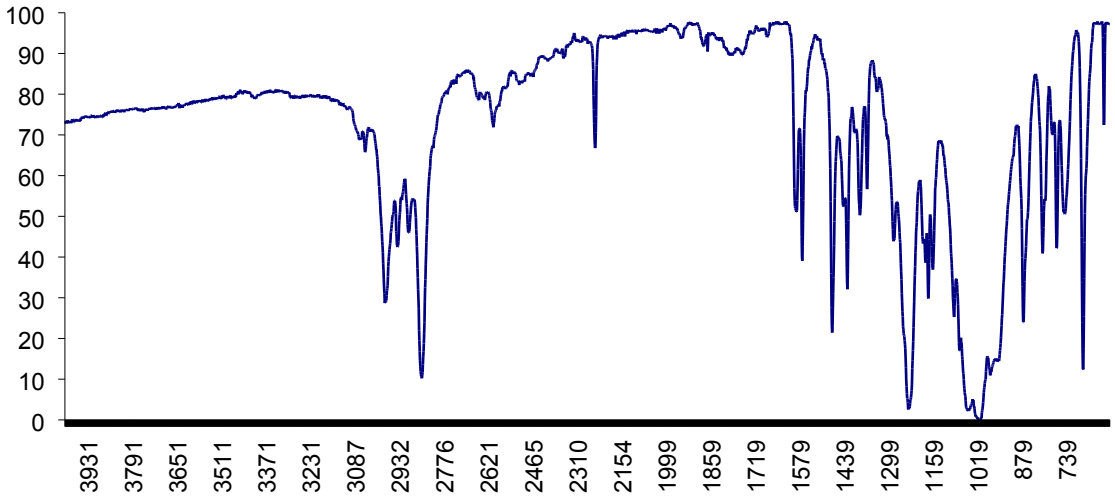
[1-(4-Chlorophenyl)-2-cyano-2-ethylcarboxylic Acid Ethyl Ester] Phosphonic Acid Diethyl Ester (17)

^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.10-1.15 (m, 3 H, CH_3), 1.19-1.30 (m, 12 H, CH_3), 1.35 (t, $^{3}J_{HH}$ = 7.2 Hz, 3 H, CH_3), 3.77-3.80 (m, 1 H, CH), 3.81-3.85 (m, 1 H, CH), 3.96-4.21 (m, 13 H, CH_2, CH), 4.27 (dd, $^{3}J_{HH}$ = 6.0 Hz, $^{3}J_{PH}$ = 8.4 Hz, 1 H, CH), 7.33-7.35 (m, 6 H, Ar), 7.46-7.48 (m, 2 H, Ar).

^{1}H NMR, ^{13}C NMR, IR and mass spectra of β-Phosphonomalonates



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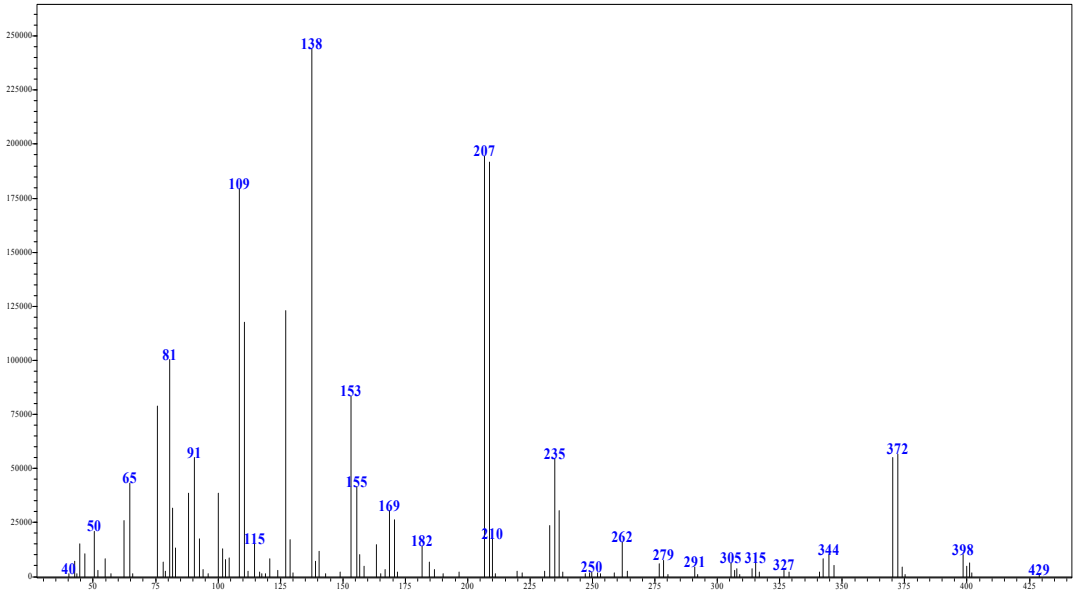
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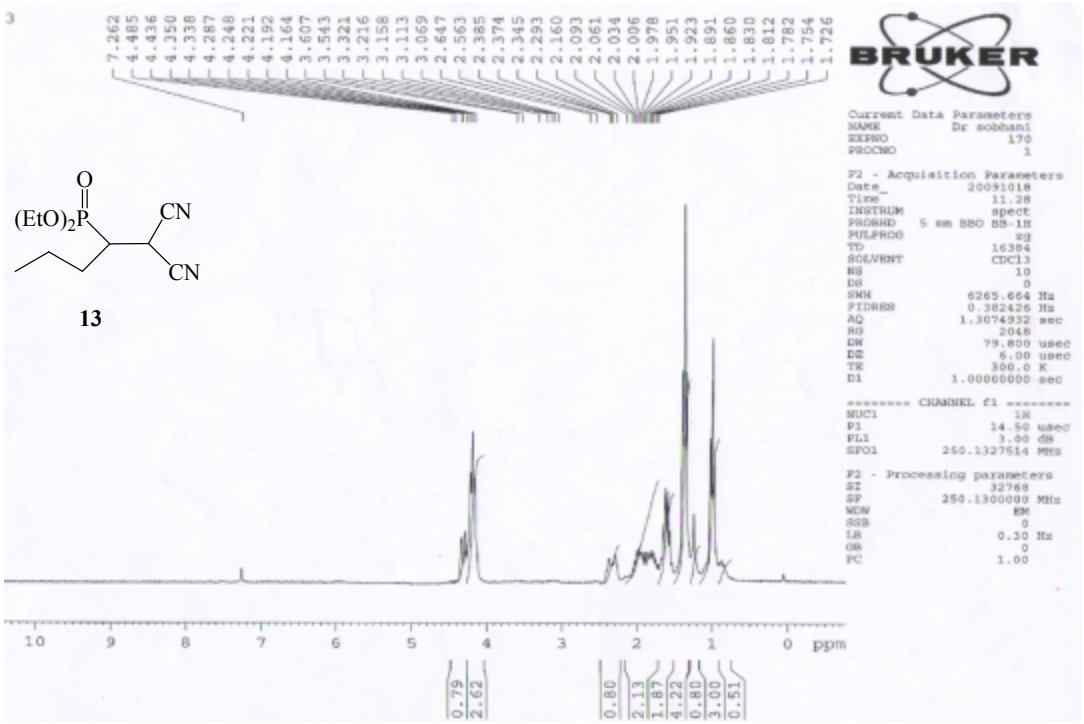
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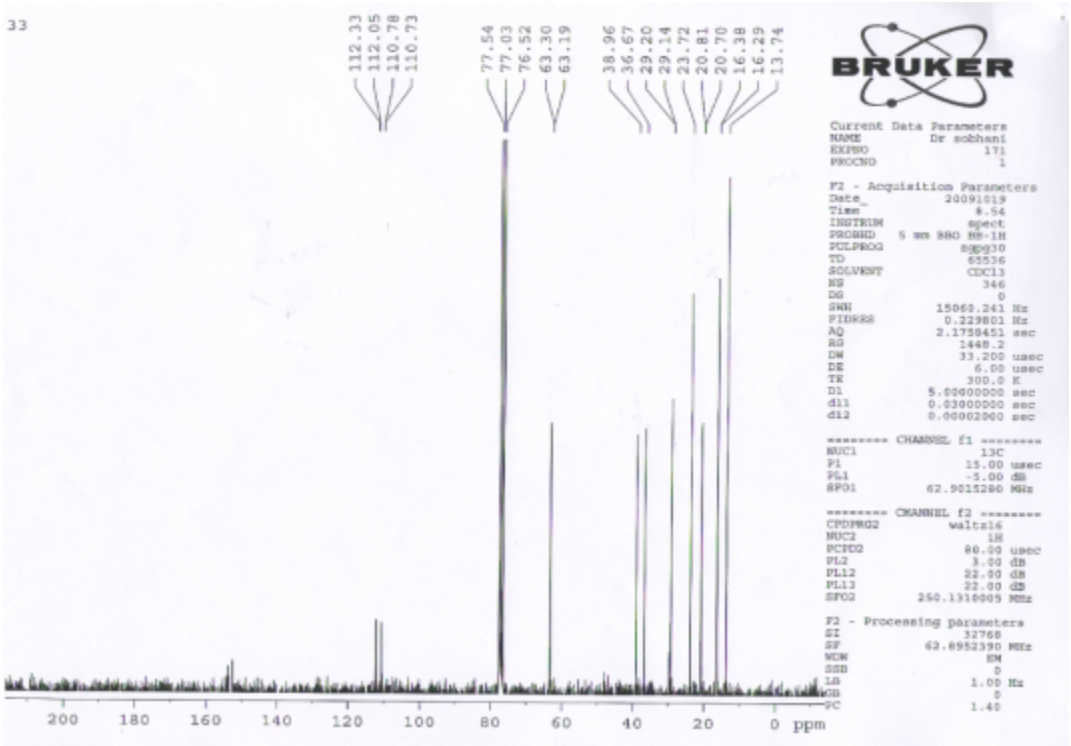
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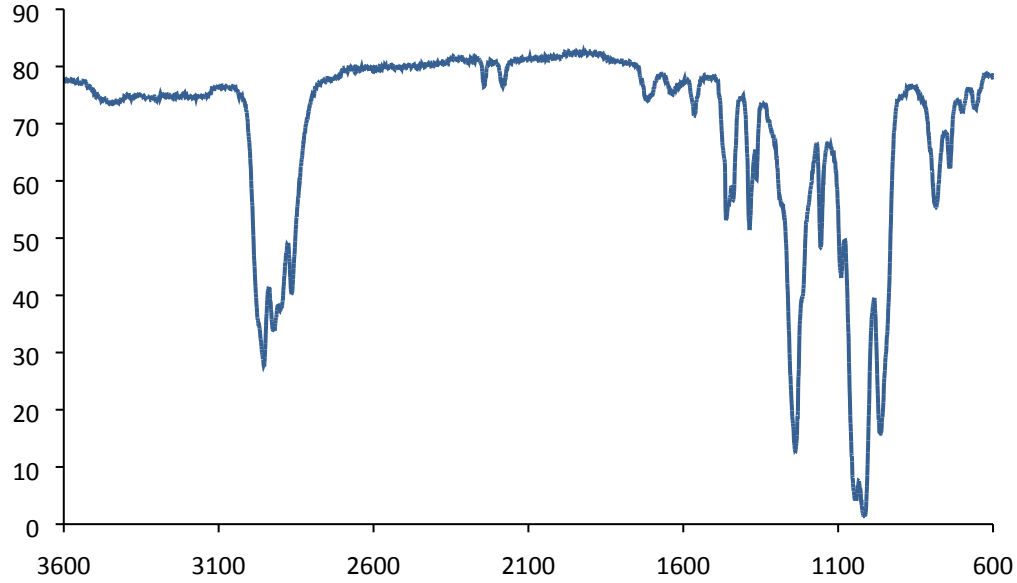
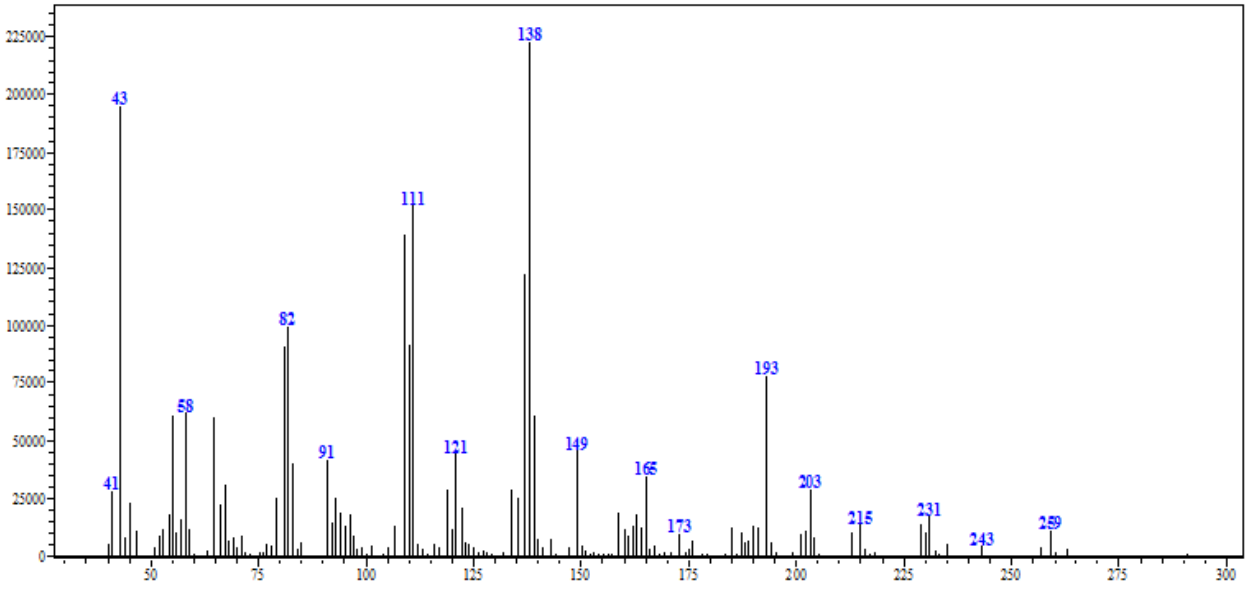
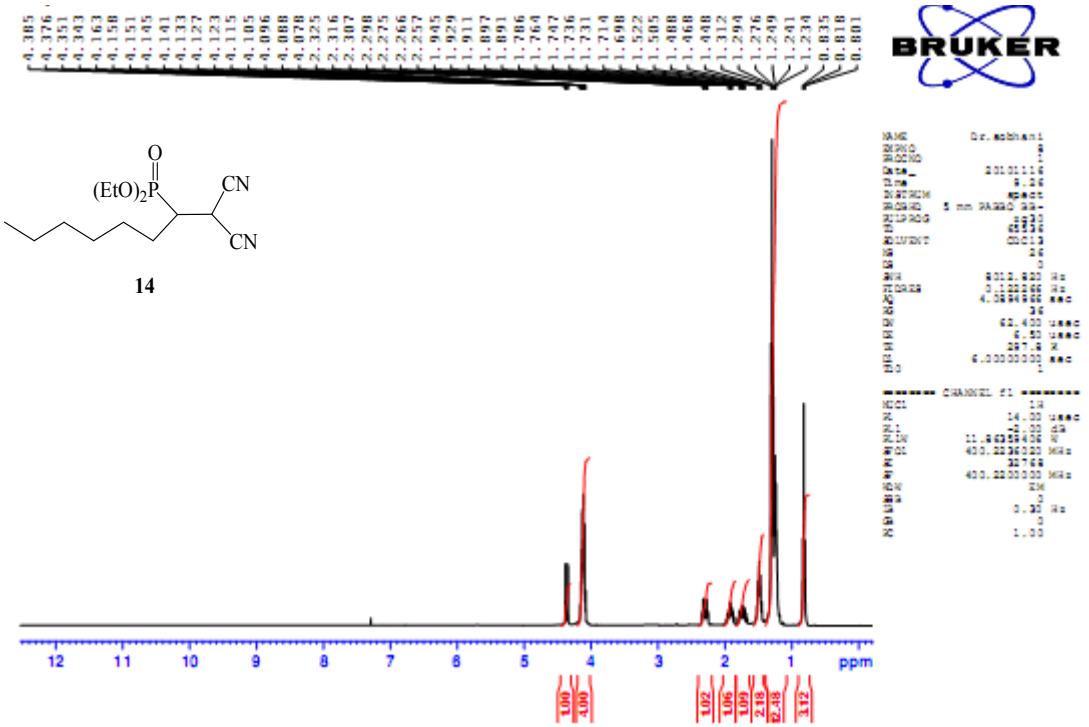
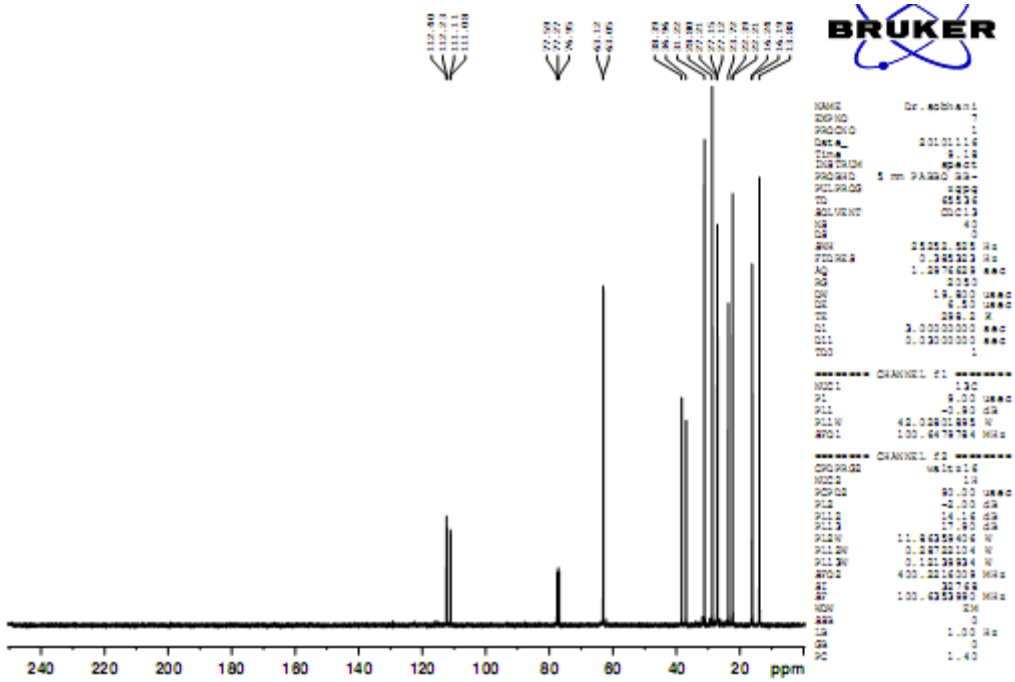
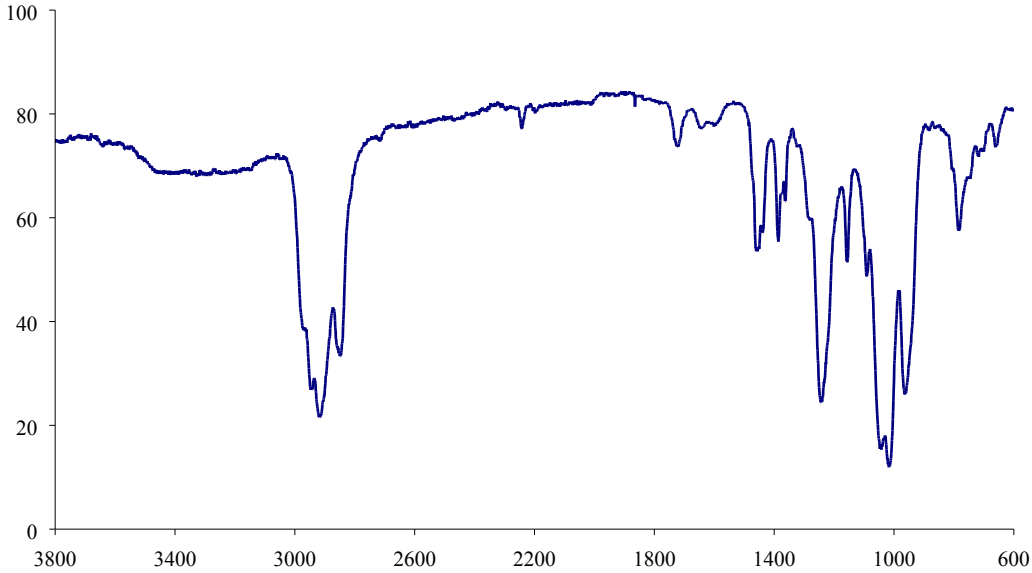
SFO2 250.1310005 MHz
F2 - Processing parameters
SI 32768
SF 62.8952390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

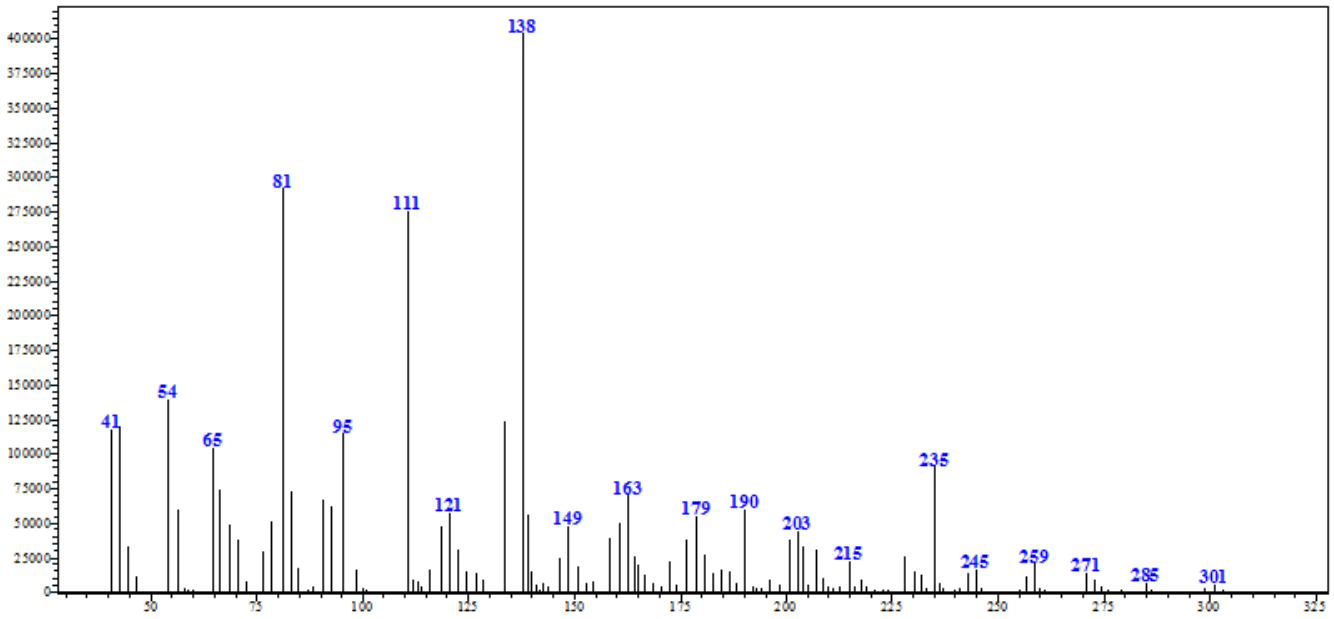

90
80
70
60
50
40
30
20
10
0
3600
3100
2600
2100
1600
1100
600


225000
200000
175000
150000
125000
100000
75000
50000
25000
0
50
75
100
125
150
175
200
225
250
275
300
41
43
58
82
91
111
121
138
149
165
173
193
203
215
231
243
259


4.385
4.376
4.351
4.343
4.163
4.158
4.151
4.145
4.141
4.133
4.127
4.123
4.115
4.105
4.096
4.088
4.078
4.325
2.316
2.307
2.298
2.275
2.266
2.257
1.945
1.929
1.911
1.897
1.891
1.786
1.764
1.747
1.736
1.731
1.714
1.698
1.522
1.505
1.488
1.468
1.448
1.312
1.294
1.276
1.249
1.241
1.234
0.835
0.818
0.801
BRUKER
O
(EtO)2P
CN
CN
14
ppm
100
400
102
106
109
218
248
312


BRUKER
240
220
200
180
160
140
120
100
80
60
40
20
ppm


100
80
60
40
20
0
3800
3400
3000
2600
2200
1800
1400
1000
600


138
81
111
54
41
65
95
121
149
163
179
190
203
215
235
245
259
271
285
301