Supporting Information: Graphene oxide catalyzed one-pot synthesis of α -aminophosphonates

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1. General information

All reagents were purchased from standard commercial sources and used as received. The presence of various functional groups on graphene oxide surface were identified using FTIR spectra on Bruker-VERTEX 80V instrument aligned with Ultra-Scan interferometer with a resolution of 1 cm^{-1} wave number. Crystalline nature of graphene oxide was determined by X-ray diffraction in a wide angle ($2\theta = 5^\circ$ to 80°) (Bruker X-ray Diffractometer, D8-Advance) data scanning rate of 0.016 sec/step, using monochromatized Cu K α radiation ($1.5406\text{ \AA}$). Zeta potential of dispersed graphene oxide was measured by using Zetasizer ZS90 (Malvern Instrument, UK). The size and morphology of graphene oxide was determined using high resolution transmission microscopy (HR-TEM, JEOL) images were collected with an operating voltage of 200 kV. Mass spectra of compound were obtained using a Finnigan LCD Advantage Max mass spectrometer (LCQAD 30000, Thermo Electron Corporation, USA). The instrument was equipped with a capillary column and a quadrupole detector. Nitrogen was used as a sheath gas with a flow rate of $40 \times 10^{-6}\text{ m}^3/\text{min}$ and auxiliary N₂ flow rate was maintained at $18 \times 10^{-6}\text{ m}^3/\text{min}$. Capillary temperature was maintained at 548 K with voltage of 420 V and ion spray voltage at 5 KV. (Note: Mass spectra was analysed by two different instruments). Compounds, synthesized were characterized by ¹H NMR (Varian 200 MHz NMR Spectrometer), ¹³C NMR spectra (50 MHz). Chemical shifts are reported in parts per million (δ) relative to tetramethylsilane as an internal standard. Elemental analysis were performed on Thermo Finnigan, (Italy) FLASH EA 1112 series analyser. Melting point of synthesized compounds was measured by using capillary method.

2. General procedure for synthesis of graphene oxide

Graphene oxide (GO) was prepared from natural graphite powder by modified Hummers method ^[1]. Natural graphite particles were intercalated by immersing the particles in a mixture of concentrated sulphuric acid (H₂SO₄) and concentrated nitric acid (HNO₃) in the ratio 3:1 v/v of H₂SO₄:HNO₃ for 24 h under ambient conditions. The reaction mass was washed with distilled water and air dried. Exfoliation was carried out by exposing the intercalated graphite to 800 °C for 2 min. Powdered exfoliated graphite was dried and further oxidation was carried out by dispersing exfoliated graphite (400 mg) in conc. H₂SO₄ followed by slow addition of KMnO₄ for 3 h. After the addition of KMnO₄ was complete, the solution was stirred for 30 min. De-ionized water (100 mL) was then added slowly and the solution was further stirred for another 24 h. Another 600 mL of water was added to the mixture followed by addition of 20 mL of 30% H₂O₂ for bleaching the reaction mass. Crude GO, thus obtained, was separated by centrifugation. The residue obtained from centrifugation was purified by sonication in 7 N HCl (100 mL) for the removal of MnO₂ impurities.

3. General procedure for synthesis of aminophosphonates

Aromatic aldehyde (1 mmole), amine (1 mmole) and trimethyl phosphite (1 mmole) were mixed with well dispersed graphene oxide (3mg) in methanol and stirred at room temperature. In the case of diamine (Entries 11k, Table 3) the molar ratio of carbonyl compound and phosphite was doubled with respect to amine. The progress of the reaction was monitored by thin layer chromatography using a solvent system consisting of a mixture of hexane and ethyl acetate (7:3). After completion, the catalyst was separated from the reaction mass by decantation. The reaction mass was then quenched with 5% NaHCO₃ solution (3 mL). Crude product was extracted with dichloromethane (5 mL × 3). The organic

layer was again washed with 5% brine (3 mL) solution and dried over sodium sulphate for appropriate time. The crude product was analyzed without further purification using various analytical techniques such as MS, ^1H NMR, ^{13}C NMR, IR and Elemental analysis techniques.

4. General Procedure for catalyst recycle

The reaction mixture was transferred to a separating funnel and the mixture was allowed to settle. Brown GO particles settled at the bottom of solution and was separated. This method avoids loss of catalyst associated other catalyst isolation processes such as filtration through paper or porous surface funnel. GO thus separated was washed with methanol ($1\text{ mL} \times 3$) and later with dichloromethane ($1\text{ mL} \times 2$) in order to remove trace amounts of reaction mixture adsorbed over catalyst surface. Dichloromethane was removed by vacuum drying at room temperature. Recovered catalyst was used in subsequent experiments.

5. Selective images during catalyst recycle experimentation

Figure 1. Selective images during catalyst recycling experiments

6. Spectral Data for Selected compounds

Compound 1a: MF = C₁₅H₁₈NO₃P White Solid; mp: 88 °C; ^[2-3] IR (KBr) ^[5] : $\nu_{\max}$ 3304 (-NH), 2952 (-CH), 1236 (P=O), 1188 (P-O-Me) Rocking vib., 1038 (P-O-C) out of phase, 751 (P-O-C) in phase; ¹H NMR(200MHz,CDCl₃,TMS): δ = δ 3.45-3.50 (d, J = 10 Hz, 3H), 3.74-3.79 (d, J = 10 Hz, 3H), 4.74-4.86 (d, J = 24 Hz, 1H), 6.57-6.62 (d, J = 8.0 Hz, 2H), 6.66-6.74 (t, J = 16 Hz, 1H), 7.07-7.15 (t, J = 16 Hz, 2H), 7.26-7.50 (m, 6H); ¹³C NMR(50MHz,CDCl₃,TMS): δ = δ 54.02 (OCH₃), 54.04 (OCH₃), 57.36 (CH), 114.03 (CH), 118.72 (CH), 122.6 (CH), 122.88 (CH), 128.85 (CH), 128.90 (CH), 135.76 (C), 140.02 (-C). Analytically calculated for C₁₅H₁₈NO₃P: C, 61.86; H, 6.22; N, 4.80; Found C, 62.01; H, 6.21; N, 4.78.

MS (ESI): m/z [M] = 291.27

Compound 2b: MF = C₁₅H₁₇NO₃PCl White solid; mp: 102 °C IR (KBr): $\nu_{\max}$ 3305 (-NH), 2955 (-CH), 1240 (-P=O), 1179 (P-O-Me) Rocking vib., 1034 (P-O-C) out of phase, 775 (P-O-C) in phase, 579 (C-Cl); ¹H NMR(200MHz,CDCl₃,TMS): δ = δ 3.52-3.58 (d, J = 12 Hz, 3H), 3.75-3.81 (d, J = 12 Hz, 3H), 4.70-4.82 (d, J = 24 Hz, 1H), 6.59-6.65 (d, J = 8.0 Hz, 2H), 6.69-6.77 (t, J = 16 Hz, 1H), 7.09-7.40 (m, 6H), 7.47 (s, 1H); ¹³C NMR(50MHz,CDCl₃,TMS): δ = δ 54.03 (OCH₃), 57.0 (CH), 113.97 (CH), 119.01 (CH), 126.02 (CH), 128.03 (CH), 129.44 (CH), 130.10 (CH), 134.88 (C), 138.22 (C). Analytically calculated for C₁₅H₁₇NO₃PCl: C, 55.07; H, 5.23; N, 4.28; Found C, 55.20; H, 5.18; N, 4.21.

MS (ESI): m/z [M + Na]⁺ = 348.04

Compound 3c: MF = $C_{17}H_{23}N_2O_3P$ white Solid; mp: 156 °C; IR (KBr): $\nu_{\max}$ 3446 (-NH), 2926 (-CH), 1350 (C-N) 1251 (-P=O), 1167 (P-O-Me), 1030 (P-O-Me) out of phase, 756 (P-O-Me) in phase; 1H NMR(200MHz,CDCl₃,TMS): δ = δ 2.92 (s, 6H), 3.45-3.50 (d, J = 10 Hz, 3H), 3.73-3.78 (d, J = 10 Hz, 3H), 4.66-4.79 (m, 2H), 6.59-6.72 (m, 5H), 7.06-7.14 (t, J = 2 Hz, 2H), 7.28-7.33 (dd, J = 2 Hz, 2H); ^{13}C NMR(50MHz,CDCl₃,TMS): δ = 30.39 (-CH₃), 53.12 (OCH₃), 58.77 (CH), 118.30 (CH), 119.62 (CH), 127.88 (CH), 128.92 (CH), 128.93 (CH), 145.74 (-C). Analytically calculated for $C_{17}H_{23}N_2O_3P$: C, 61.07; H, 6.92; N, 8.38; Found C, 61.00; H, 6.90; N, 8.36.
MS (ESI): m/z $[M + K]^+ = 373.10$

Compound 4d: MF = $C_{15}H_{18}NO_4P$. Brown liquid IR (KBr): $\nu_{\max}$ 3349 (-NH), 2960 (-CH), 1240 (-P=O), 1186 (P-O-Me), 1058 (P-O-Me) out of phase, 830 (P-O-Me) in phase; 1H NMR(200MHz,CDCl₃,TMS): δ = δ 3.43-3.49 (d, J = 12, 3H), 3.73-3.79 (d, J = 12 Hz, 3H), 4.65-4.83 (m, 2H), 6.49-6.53 (d, J = 8, 2H), 7.02-7.07 (d, J = 10 Hz, 2H), 7.32-7.46 (m, 5H); ^{13}C NMR(50MHz,CDCl₃,TMS): δ = δ 53.58 (OCH₃), 59.53 (CH), 113.79 (CH), 118.66 (CH), 126.89 (CH), 127.78 (CH), 128.72 (CH), 128.98 (CH), 129.33 (CH), 129.49, (CH) 129.86 (CH). Analytically calculated for $C_{15}H_{18}NO_4P$: C 58.70, H 5.88, N 4.55; Found C 58.80, H 5.90, N 4.52.

Compound 5e: MF = $C_{16}H_{20}NO_4P$ White solid; mp: 103 °C; IR (KBr): $\nu_{\max}$ 3322 (-NH), 2948 (-CH), 1240 (-P=O), 1183 (P-O-Me), 1030 (P-O-Me) out of phase, 754 (P-O-Me) in phase; 1H NMR(200MHz,CDCl₃,TMS): δ = δ 3.47-3.52 (d, J = 10 Hz, 3H), 3.73-3.78 (d, J = 10 Hz, 3H), 3.78 (s, 3H), 4.66-4.77 (m, 1H), 4.79-4.83 (d, J = 8 Hz, 1H), 6.57-6.61 (d, J = 8 Hz, 2H), 6.66-6.73 (t, J = 14, 1 H), 6.85-6.90 (d, J = 10, 2H), 7.07-7.15 (t, J = 16, 2H), 7.35-

7.41 (d, J = 12, 2H); ^{13}C NMR(50MHz,CDCl₃,TMS): δ = 53.97 (OCH₃), 55.88 (OCH₃), 114.06 (CH), 114.30 (CH), 118.16 (CH), 127.44 (CH), 129.03 (CH), 146.16 (-C), 159.52 (O-C=C). Analytically calculated for C₁₆H₂₀NO₄P: C 59.80, H 6.27, N 4.36; Found C 59.32, H 6.25, N 4.34.

MS (ESI): m/z [M + K]⁺ = 360.08

Compound 6f: MF = C₁₄H₂₂NO₃P White Solid; mp: 109 °C; ^{31}P IR (KBr): ν_{max} 3322 (-NH), 2948 (-CH), 1240 (-P=O), 1183 (P-O-Me), 1030 (P-O-Me) out of phase, 754 (P-O-Me) in phase; ^1H NMR(200MHz,CDCl₃,TMS): δ = δ 1.22-1.93 (m, 8H), 2.16-2.26 (Br, 2H), 3.68 (s, 3H), 3.70 (s, 3H), 6.78-6.85 (t, J = 14 Hz, 1H), 7.01-7.04 (d, J = 6 Hz, 2H), 7.14-7.22 (t, J = 16 Hz, 2H); ^{13}C NMR(50MHz,CDCl₃,TMS): δ = 19.86 (-CH₂), 25.11 (-CH₂), 30.32 (-CH₂), 53.02 (OCH₃), 118.43 (CH), 119.57 (CH), 128.86 (CH). Analytically calculated for C₁₄H₂₂NO₃P: C, 59.36; H, 7.81; N, 4.94; Found C, 59.53; H, 7.79; N, 4.92.

MS (ESI): m/z [M + K]⁺ = 322.04

Compound 7g: MF = C₁₅H₁₇N₂O₅P Yellowish Solid; mp: 101 °C; IR (KBr): ν_{max} 3366 (-NH), 2926 (-CH), 1316 (C-N), 1238 (P=O), 1180 (P-O-Me), 1034 (P-O-C) out of phase, 721 (P-O-C) in phase; ^1H NMR(200MHz,CDCl₃,TMS): δ = δ 3.43-3.48 (d, J = 10 Hz, 3H), 3.76-3.81 (d, J = 10 Hz, 3H), 4.77-4.89 (d, J = 24 Hz, 1H), 5.69 (s, 1H), 6.57-6.60 (d, J = 6 Hz, 3H), 7.27-7.46 (m, 5H), 8.01-8.09 (m, 1H); ^{13}C NMR(50MHz,CDCl₃,TMS): δ 53.02 (OCH₃), 53.13 (OCH₃), 118.43 (CH), 118.97 (CH), 119.23 (CH), 119.52 (CH), 128.86 (CH). Analytically calculated for C₁₅H₁₇N₂O₅P: C, 53.65; H, 5.08; N, 8.31; Found C, 53.59; H, 5.05; N, 8.31.

MS (ESI): m/z [M + Na]⁺ = 360.54

Compound 8h: MF = C₁₅H₁₇NO₃PCl White solid mp: 97 °C IR (KBr): $\nu_{\max}$ 3301 (-NH), 2953 (-CH), 1237 (-P=O), 1175 (P-O-Me), 1029 (P-O-Me) out of phase, 816 (P-O-Me) in phase, 570 (C-Cl); ¹H NMR(200MHz,CDCl₃,TMS): δ = δ 3.43-3.49 (d, J = 12 Hz, 3H), 3.74-3.79 (d, J = 10 Hz, 3H), 4.67-4.80 (d, J = 26 Hz, 1H), 6.49-6.53 (d, J = 8 Hz, 2H), 7.03-7.07 (d, J=8, 2H), 7.26-7.46 (m, 5H) (Note :- peak at δ 2.17 is due to acetone); ¹³C NMR(50MHz,CDCl₃,TMS): δ = δ 54.04 (OCH₃), 56.69 (CH), 115.18 (CH), 122.85 (CH), 127.91 (CH), 128.39 (CH), 128.97 (CH), 129.21 (CH), 135.25 (C), 144.69 (C). Analytically calculated for C₁₅H₁₇NO₃PCl: C 55.07, H 5.23, N 4.28; Found C 55.05, H 5.24, N 4.29. MS (ESI): m/z [M + K]⁺ = 366.04

Compound 9i: MF = C₁₅H₁₇NO₃PBr Brown Solid; mp: 76 °C IR (KBr): $\nu_{\max}$ 3447 (-NH), 2926 (-CH), 1241 (-P=O), 1191 (P-O-Me), 1049 (P-O-Me) out of phase, 741 (P-O-Me) in phase, 528 (C-Br); ¹H NMR(200MHz,CDCl₃,TMS): δ = δ 3.52-3.58 (d, J = 12, 3H), 3.74-3.79 (d, J = 10 Hz, 3H), 4.74-4.90 (d, J = 32, 1H), 5.39-5.48 (t, J = 18 1H), 6.43-6.47 (d, J = 8 Hz, 1H), 6.53-6.61 (t, J = 16 Hz, 1H), 6.99-7.06 (t, J = 14 Hz 1H), 7.31-7.49 (m, 6H); ¹³C NMR(50MHz,CDCl₃,TMS): δ δ = 53.04 (OCH₃), 56.26 (CH), 109.69 (-C), 111.65 (CH), 118.15 (CH), 126.58 (CH), 126.68 (CH), 127.32 (CH), 127.82 (C), 133.94 (CH), 147.86 (-C). Analytically calculated for C₁₅H₁₇NO₃PBr: C 48.61, H 4.62, N 3.77; Found C 48.80, H 4.61, N 3.76 MS (ESI): m/z [M + K]⁺ = 409.91

Compound 10j: MF = C₁₅H₁₇NO₃PI White Solid; mp: 81 °C; IR (KBr): $\nu_{\max}$ 3373 (-NH), 2925 (-CH), 1228 (-P=O), 1180 (P-O-Me), 1044 (P-O-Me) out of phase, 743 (P-O-Me) in phase, 537 (C-I); ¹H NMR(200MHz,CDCl₃,TMS): δ = δ 3.54-3.59 (d, J = 10 Hz, 3H), 3.75-3.80 (d, J = 10 Hz, 3H), 4.75-4.88 (d, J = 25 Hz, 1H), 6.36-6.48 (m, 2H), 7.01-7.09 (m, 1H),

7.27-7.49 (m, 6H), 7.65-7.69 (dd, 1H); ^{13}C NMR(50MHz,CDCl₃,TMS): δ = 54.88 (OCH₃), 57.87 (CH), 86.38 (-C), 112.18 (CH), 120.11 (CH), 122.78 (CH), 127.88 (CH), 128.93 (CH), 129.93 (C), 139.29 (CH), 142.52 (-C), 149.79 (-C). Analytically calculated for C₁₅H₁₇NO₃PI: C, 43.14; H, 4.10; N, 3.34; Found C, 43.12; H, 4.08; N, 3.30.
MS (ESI): m/z [M + K]⁺ = 455.9

Compound 11k: C₂₀H₃₀N₂O₆P₂ Yellowish Liquid; IR (KBr): ν_{max} 3421 (-NH), 2954 (-CH), 1217 (-P=O), 1050 (P-O-Me) out of phase, 704 (P-O-Me) in phase; ^1H NMR(200MHz,CDCl₃,TMS): δ = δ 2.33-2.59 (m, 4H), 3.51-3.57 (d, J = 12 Hz, 6H), 3.71-3.77 (d, J = 12 Hz, 6H), 3.86-3.92 (d, J= 12 1H), 4.80 (t, 2H), 7.31-7.40 (m, 10H); ^{13}C NMR(50MHz,CDCl₃,TMS): δ = 43.52 (-CH₂), 53.68 (OCH₃), 120.00 (CH), 128.71 (CH), 130.24 (CH), 132.49 (CH), 132.98 (CH). Analytically calculated for C₂₀H₃₀N₂O₆P₂: C 52.64, H 6.62, N 6.13; Found C 52.40, H 6.64, N 6.14.
MS (ESI): m/z [M+ K]⁺ = 495.1

7. References

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7. Copies of ^1H ^{13}C NMR, MS Elemental analysis spectra of selected products

1a. (Phenyl-phenylamino-methyl)-phosphonic acid dimethyl ester

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 18:37
 Printed: 09-17-2014 12:06
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-018-SAMPLE-1-CHN-42 (# 18)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-018-SAMPLE-1-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.671
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Componen
1	0.775	257564	4.787	Nitrogen
2	1.050	7103487	62.014	Carbon
3	3.667	2130002	6.215	Hydrogen
		9491053	73.016	

2b. 3-Chloro-phenyl-phenylamino-methyl-phosphonicacid dimethyl ester

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 17:11
 Printed: 09-17-2014 10:58
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-010-SAMPLE-2-CHN-42 (# 10)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-010-SAMPLE-2-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.906
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.775	246488	4.219	Nitrogen
2	1.058	6880312	55.208	Carbon
3	3.667	1939804	5.181	Hydrogen
		9066604	64.608	

3c. (4-Dimethylamino-phenyl)-phenylamino-methyl]-phosphonic acid dimethyl ester

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 17:21
 Printed: 09-17-2014 10:59
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-011-SAMPLE-3-CHN-42 (# 11)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-011-SAMPLE-3-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.245
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.783	356528	8.367	Nitrogen
2	1.083	5873655	61.008	Carbon
3	3.667	1995736	6.905	Hydrogen
		8225919	76.280	

4d. (2-Hydroxy-phenyl)-phenylamino-methyl]-phosphonic acid dimethyl ester

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 15:07
 Printed: 09-17-2014 10:56
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-007-CHN-42-SAMPLE-12 (# 7)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-007-CHN-42-SAMPLE-12.dat
 Calibration method: K Factors
 Sample weight: 2.652
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Componen
1	0.792	258437	4.520	Nitrogen
2	1.075	6688265	58.807	Carbon
3	3.667	2010353	5.902	Hydrogen
		8957055	69.229	

5e. (4-Methoxy-phenyl)-phenylamino-methyl]-phosphonic acid dimethyl ester)

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 18:26
 Printed: 09-17-2014 12:05
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-017-SAMPLE-7-CHN-42 (# 17)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-017-SAMPLE-7-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.282
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1* μ V*sec)	Element %	Componen
1	0.775	181711	4.340	Nitrogen
2	1.083	5805300	59.320	Carbon
3	3.233	1859174	6.250	Hydrogen
		7846185	69.910	

6f. ((1-Phenylamino-cyclohexyl)-phosphonic acid dimethyl ester)

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 17:55
 Printed: 09-17-2014 12:04
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-014-SAMPLE-4-CHN-42 (# 14)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-014-SAMPLE-4-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.158
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.775	219077	4.920	Nitrogen
2	1.075	5509556	59.533	Carbon
3	3.667	2160721	7.798	Hydrogen
		7889354	72.251	

7g. 4-Nitro-phenylamino-phenyl-methyl]-phosphonic acid dimethyl ester

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 17:42
 Printed: 09-17-2014 10:59
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-013-SAMPLE-5-CHN-42 (# 13)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-013-SAMPLE-5-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.291
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.775	547636	8.314	Nitrogen
2	1.083	5265864	53.596	Carbon
3	3.233	1509749	5.057	Hydrogen
		7323249	66.967	

8h. (4-Chloro-phenylamino)-phenyl-methyl]-phosphonic acid dimethyl ester

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 17:32
 Printed: 09-17-2014 10:59
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-012-SAMPLE-10-CHN-42 (# 12)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-012-SAMPLE-10-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.115
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1* μ V*sec)	Element %	Component
1	0.775	193286	4.290	Nitrogen
2	1.075	4993796	55.057	Carbon
3	3.225	1447268	5.245	Hydrogen
		6634350	64.592	

9i. [(2-bromo-phenylamino)-phenyl-methyl]-phosphonic acid dimethyl ester

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 17:00
 Printed: 09-17-2014 10:58
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-009-SAMPLE-8-CHN-42 (# 9)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-009-SAMPLE-8-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.273
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.775	185255	3.764	Nitrogen
2	1.083	4757143	48.802	Carbon
3	3.225	1382456	4.610	Hydrogen
		6324854	57.176	

10j. 2-iodo-phenylamino)-phenyl-methyl-phosphonic acid dimethyl ester

Mol. Wt.: 417.60

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 18:16
 Printed: 09-17-2014 12:05
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-016-SAMPLE-9-CHN-42 (# 16)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-016-SAMPLE-9-CHN-42.dat
 Calibration method: K Factors
 Sample weight: 2.118
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1* μ V*sec)	Element %	Component
1	0.783	158373	3.303	Nitrogen
2	1.100	3917369	43.128	Carbon
3	3.217	1144112	4.086	Hydrogen
		5219854	50.517	

11k. *2-Dimethoxy-phosphoryl)-phenyl-methyl]-amino}-ethylamino)-phenyl-methyl]-phosphonic acid dimethyl ester*

Operator ID: IIT-B
 Company name: ThermoFinnigan
 Method filename: C:\2013-2014-MTH\16-09-2014-chns.mth
 Method name: Nitrogen/Carbon/Hydrogen/Sulphur
 Analysed: 09/16/2014 15:23
 Printed: 09-17-2014 10:57
 Elemental Analyser method:
 Sampler method:
 Sample ID: 16-09-2014-008-CHN-42-SAMPLE-11 (# 8)
 Analysis type: UnkNown
 Chromatogram filename: 16-09-2014-008-CHN-42-SAMPLE-11.dat
 Calibration method: K Factors
 Sample weight: 2.247
 Protein factor: 6.25

Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Element %	Component
1	0.792	280306	6.145	Nitrogen
2	1.100	5049448	52.400	Carbon
3	3.667	1919500	6.641	Hydrogen
		7249254	65.186	