Microwave-assisted synthesis of α-aminophosphine oxides by the Kabachnik-Fields reaction applying amides as the starting materials

Anna Tripolszky,[1] Lili Zoboki, Erika Bálint, János Kóti[2] and György Keglevich[1,*]

Address:

[1]*Department of Organic Chemistry and Technology, Budapest University of Technology and Economics, H-1111 Budapest, Budafoki út 8., Hungary*

[2]*Gedeon Richter Plc. 1475 Budapest Hungary*

E-mail:

György Keglevich[*] - gkeglevich@mail.bme.hu

* Corresponding author

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General information

The reactions were carried out in a 300 W CEM Discover microwave reactor (CEM Microwave Technology Ltd., Buckingham, UK) equipped with a pressure controller applying 20–120 W under isothermal conditions.

HPLC-MS experiments were performed with an Agilent 1200 liquid chromatography system coupled with a 6130 quadrupole mass spectrometer equipped with an ESI ion source (Agilent Technologies, Palo Alto, CA, USA). Analysis was performed at 40 ºC on a Gemini C18 column (150 mm × 4.6 mm, 3 fµm; Phenomenex, Torrance, CA, USA) with a mobile phase flow rate of 0.6 ml/min. Composition of eluent A was 0.1% NH_4HCO_3 in water; eluent B was 0.1% HCO_2NH_4 and 8% water in acetonitrile. 0→3 min 5% B, 3→13 min gradient, 13→20 min 95% B. The injection volume was 5 µL. The chromatographic profile was registered at 222 nm. The MSD operating parameters were as follows: positive ionization mode, scan spectra from m/z 100 to 1000, drying gas temperature 300 ºC, nitrogen flow rate 12 L/min, nebulizer pressure 60 psi, capillary voltage 2500 V.

The ^{31}P, ^{13}C, ^{1}H NMR spectra were taken on a Bruker AV-300 or DRX-500 spectrometer operating at 121.5, 75.5 and 300 or 202.4, 125.7 and 500 MHz, respectively, in $CDCl_3$ solution. Chemical shifts are downfield relative to 85% H_3PO_4 and TMS. The couplings are given in Hz. Electrospray high-resolution MS measurements were performed on a Thermo Velos Pro Orbitrap Elite Hybrid Mass spectrometer (Thermo Fisher Scientific, Bremen, Germany). The ionization method was ESI and operated in positive ion mode. The capillary temperature was set at 275°C. Samples were infused into the ESI source MeOH solutions at a flow rate of 3 µL/min. Resolving power of 60,000 (FWHM) at *m/z* 400. Data acquisition and analysis were accomplished with Xcalibur software version 3.0 (Thermo Fisher Scientific Inc.).

General procedure for the synthesis of acylated α-aminophosphine oxides (1a-d, 2a-d and 3a-d)

A mixture of 5.0 mmol of the amide (0.29 g of acetamide, 0.37 g of propionamide or 0.61 g of benzamide), 0.50 mmol (0.02 g) of paraformaldehyde and 0.50 mmol of the secondary phosphine oxide (0.11 g of diphenylphosphine oxide, 0.12 g of di(*p*-tolyl)phosphine oxide, 0.13 g of bis(3,5-dimetylphenyl)phosphine oxide or 0.12 g of dibenzylphosphine oxide) was heated at 220 °C or 240 °C in a closed vial in a CEM Discover microwave reactor equipped with a pressure controller for 3.5 h. The crude product so obtained was dissolved in dichloromethane, and the excess of amide was removed by extraction with 3x15 mL of water. The combined organic phases were dried (Na_2SO_4). Evaporation of the solvent left a residue that was passed through a 1 cm silica gel layer using 10% methanol in dichloromethane as the eluent to afford the title products as white crystals. Most of the aminophosphine oxides were recrystallized from acetone. The following compounds were thus prepared:

[(Acetylamino)methyl]diphenylphosphine oxide (1a)

Yield: 72% (0.16 g); Mp: 174-175 °C; ^{31}P NMR ($CDCl_3$) δ 32.3; ^{13}C NMR ($CDCl_3$) δ 22.8 (CH_3), 39.1 (d, $^1J_{CP}$ = 78.9, P(O)CH_2), 128.8 (d, $^2J_{CP}$ = 11.9, C_2), 130.6 (d, $^1J_{CP}$ = 99.9, C_1), 131.0 (d, $^3J_{CP}$ = 9.7, C_3), 132.4 (d, $^4J_{CP}$ = 2.8, C_4), 170.6 (d, $^3J_{CP}$ = 4.9, C(O)); ^{1}H NMR ($CDCl_3$) δ 1.95 (s, 3H, CH_3), 4.24 (t, $^2J_{HP}$ = $^3J_{HH}$ = 5.9, 2H, NHCH_2), 7.36–7.60 (m, 6H, C_3, C_4), 7.65–7.83 (m, 4H, C_2), 7.93–8.09 (br, 1H, NH). $[M+H]^+_{found}$ = 274.09969, $C_{15}H_{17}NO_2P$ requires 274.09914.

[(Acetylamino)methyl]di-*p*-tolylphosphine oxide (1b)

Yield: 85% (0.13 g); Mp: 162-163 °C; ^{31}P NMR ($CDCl_3$) δ 31.4; ^{13}C NMR ($CDCl_3$) δ 21.6 (C_4CH_3), 22.9 (s, C(O)CH_3), 39.0 (d, $^1J_{CP}$ = 78.6, $NHCH_2$), 127.4 (d, $^1J_{CP}$ = 102.7, C_1), 129.6

(d, $^{2}J_{CP}$ = 12.3, C_2), 130.9 (d, $^{3}J_{CP}$ = 10.0, C_3), 142.9 (d, $^{4}J_{CP}$ = 2.7, C_4), 170.4 (d, $^{3}J_{CP}$ = 2.3, C(O)); ^{1}H NMR ($CDCl_3$) δ 1.94 (s, 3H, C(O)CH_3), 2.39 (s, 6H, C_4CH_3), 4.18 (t, $^{2}J_{HP}$ = $^{3}J_{HH}$ = 5.0, 2H, NHCH_2), 7.26 (d, $^{3}J_{HH}$ = 8.0, 4H, C_3H), 7.61 (dd, $^{3}J_{HP}$ = 11.5, $^{3}J_{HH}$ = 7.8, 4H, C_2H), 7.66–7.84 (br, 1H, NH). $[M+H]^{+}_{found}$ = 302.13000, $C_{17}H_{21}NO_2P$ requires 302.13044.

[(Acetylamino)methyl]bis-3,5-dimetylphenylphosphine oxide (1c)

Yield: 75% (0.09 g); Mp: 172-174 °C; ^{31}P NMR ($CDCl_3$) δ 31.8; ^{13}C NMR ($CDCl_3$) δ 21.3 (C_3CH_3), 22.9 (C(O)CH_3), 38.9 (d, $^{1}J_{CP}$ = 77.6 $NHCH_2$), 128.4 (d, $^{2}J_{CP}$ = 9.7, C_2), 130.3 (d, $^{1}J_{CP}$ = 99.6, C_1), 134.1 (d, $^{3}J_{CP}$ = 2.8, C_3), 138.6 (d, $^{4}J_{CP}$ = 12.7, C_4), 170.4 (d, $^{3}J_{CP}$ = 5.0, C(O)); ^{1}H NMR ($CDCl_3$) δ 1.95 (s, 3H, C(O)CH_3), 2.33 (s, 12H, C_3CH_3), 4.17 (t, $^{2}J_{HP}$ = $^{3}J_{HH}$ = 6.0, 2H, NHCH_2), 7.15 (s, 2H, C_4H), 7.35 (d, $^{3}J_{HP}$ = 12.0, 4H, C_2H). $[M+H]^{+}_{found}$ = 330.16148, $C_{19}H_{25}NO_2P$ requires 330.16174.

[(Acetylamino)methyl]dibenzylphosphine oxide (1d)

Yield: 93% (0.14 g); Mp: 110-112 °C; ^{31}P NMR ($CDCl_3$) δ 41.0; ^{13}C NMR ($CDCl_3$) δ 22.8 (CH_3), 35.0 (d, $^{1}J_{CP}$ = 60.1, P(O)CH_2), 36.9 (d, $^{1}J_{CP}$ = 72.9, $NHCH_2$), 127.2 (d, $^{5}J_{CP}$ = 2.9, C_4), 128.9 (d, $^{4}J_{CP}$ = 2.5, C_3), 129.7 (d, $^{3}J_{CP}$ = 5.2, C_2), 130.9 (d, $^{2}J_{CP}$ = 7.5, C_1), 170.9 (d, $^{3}J_{CP}$ = 4.0, C(O)); ^{1}H NMR ($CDCl_3$) δ 1.93 (s, 3H, CH_3), 2.97–3.17 (m, 4H, P(O)CH_2), 3.47 (t, $^{2}J_{HP}$ = $^{3}J_{HH}$ = 5.0, 2H, NHCH_2), 7.16–7.34 (m, 10H, ArH), 7.42–7.57 (br, 1H, NH). $[M+H]^{+}_{found}$ = 302.13022, $C_{17}H_{21}NO_2P$ requires 302.13044.

[(Propyonylamino)methyl]diphenylphosphine oxide (2a)

Yield: 91% (0.13 g); Mp: 153-154 °C; ^{31}P NMR ($CDCl_3$) δ 34.0; ^{13}C NMR ($CDCl_3$) δ 9.9 (CH_3), 29.4 (CH_2CH_3), 38.8 (d, $^{1}J_{CP}$ = 78.4, $NHCH_2$), 128.8 (d, $^{2}J_{CP}$ = 11.9, C_2), 130.6 (d, $^{1}J_{CP}$ = 99.7, C_1), 131.0 (d, $^{3}J_{CP}$ = 9.8, C_3), 132.4 (d, $^{4}J_{CP}$ = 2.8, C_4), 174.2 (d, $^{3}J_{CP}$ = 4.6, C(O)); ^{1}H

NMR ($CDCl_3$) δ 0.99 (t, $^3J_{HH}$ = 7.6, 3H, CH_3), 2.17 (q, $^3J_{HH}$ = 7.6, 2H, C*H*$_2$CH$_3$), 4.25 (t, $^2J_{HP}$ = $^3J_{HH}$ = 5.9, 2H, NHC*H*$_2$), 7.16–7.61 (m, 7H, C_3H, C_4H, NH), 7.67–7.87 (m, 4H, C_2H). $[M+H]^+_{found}$ = 288.11489, $C_{16}H_{19}NO_2P$ requires 288.11479.

[(Propyonylamino)methyl]di-*p*-tolylphosphine oxide (2b)

Yield: 62% (0.15 g); Mp: 146-148 °C; ^{31}P NMR ($CDCl_3$) δ 31.6; ^{13}C NMR ($CDCl_3$) δ 9.9 (CH_2CH_3), 21.6 (C_4CH_3), 29.4 (CH_2CH_3), 38.9 (d, $^1J_{CP}$ = 78.7, $NHCH_2$), 127.4 (d, $^1J_{CP}$ = 102.5, C_1), 129.5 (d, $^2J_{CP}$ = 129.5, C_2), 131.0 (d, $^3J_{CP}$ = 10.0, C_3), 142.8 (d, $^4J_{CP}$ = 1.7, C_4), 174.2 (d, $^3J_{CP}$ = 2.3, C(O)); ^1H NMR ($CDCl_3$) δ 1.00 (t, $^3J_{HH}$ = 7.5, 3H, C*H*$_3$CH$_2$), 2.16 (q, $^3J_{HH}$ = 7.6, 2H, C*H*$_2$CH$_3$), 2.38 (s, 6H, C_4CH_3), 4.19 (t, $^2J_{HP}$ = $^3J_{HH}$ = 6.1, 2H, NHC*H*$_2$), 7.26 (d, $^3J_{HH}$ = 7.8, 4H, C_3H), 7.31–7.44 (br, 1H, NH), 7.62 (dd, $^3J_{HP}$ =11.7, $^3J_{HH}$ =7.5, 4H, C_2H). $[M+H]^+_{found}$ = 316.14566, $C_{18}H_{23}NO_2P$ requires 316.14609.

[(Propyonylamino)methyl]bis-3,5-dimetylphenylphosphine oxide (2c)

Yield: 66% (0.10 g); Mp: 164-165 °C; ^{31}P NMR ($CDCl_3$) δ 32.0; ^{13}C NMR ($CDCl_3$) δ 9.9 (CH_2CH_3), 21.3 (C_3CH_3), 29.5 (CH_2CH_3), 38.8 (d, $^1J_{CP}$ = 77.7, $NHCH_2$), 128.4 (d, $^2J_{CP}$ = 9.7, C_2), 130.3 (d, $^1J_{CP}$ = 99.4, C_1), 134.1 (d, $^3J_{CP}$ = 2.9, C_3), 138.5 (d, $^4J_{CP}$ = 12.6, C_4), 174.1 (d, $^3J_{CP}$ = 4.9, C(O)); ^1H NMR ($CDCl_3$) δ 1.01 (t, $^3J_{HH}$ = 7.6, 3H, CH_2CH_3), 2.17 (q, $^3J_{HH}$ = 7.6, C*H*$_2$CH$_3$), 2.33 (s, 12H, C_3CH_3), 4.18 (t, $^2J_{HP}$ = $^3J_{HH}$ = 6.0, 2H, NHC*H*$_2$), 7.07–7.13 (br, 1H, NH), 7.15 (s, 2H, C_4H), 7.35 (d, $^3J_{HP}$ = 12.0, 4H, C_2H). $[M+H]^+_{found}$ = 344.17694, $C_{20}H_{27}NO_2P$ requires 344.17739.

[(Propyonylamino)methyl]dibenzylphosphine oxide (2d)

Yield: 57% (0.14 g) as white crystals. Mp: 142-144 °C. ^{31}P NMR ($CDCl_3$) δ 41.2; ^{13}C NMR ($CDCl_3$) δ 9.7 (CH_3), 29.3 (CH_2CH_3), 35.1 (d, $^1J_{CP}$ = 60.3, $P(O)CH_2$), 36.7 (d, $^1J_{CP}$ = 72.7,

$NHCH_2$), 127.3 (d, $^5J_{CP}$ = 2.9, C_4), 128.9 (d, $^4J_{CP}$ = 2.5, C_3), 129.8 (d, $^3J_{CP}$ = 5.2, C_2); 130.8 (d, $^2J_{CP}$ = 7.6, C_1), 174.5 (d, $^3J_{CP}$ = 4.0, C(O)); ^{1}H NMR ($CDCl_3$) δ 1.10 (t, $^3J_{HH}$ = 7.6, 3H, CH_3), 2.15 (q, $^3J_{HH}$ = 7.6, 2H, C*H*$_2$CH$_3$), 2.99–3.22 (m, 4H, P(O)CH_2), 3.56 (t, $^2J_{HP}$ = $^3J_{HH}$ = 5.4, 2H, NHC*H*$_2$), 6.70–6.81 (br, 1H, NH), 7.15–7.39 (m, 10H, ArH). $[M+H]^+_{found}$ = 316.14555, $C_{18}H_{23}NO_2P$ requires 316.14609.

[(Benzoylamino)methyl]diphenylphosphine oxide (3a)

Yield: 78% (0.14 g); Mp: 239-240 °C; ^{31}P NMR ($CDCl_3$) δ 31.2; ^{13}C NMR ($CDCl_3$) δ 39.5 (d, $^1J_{CP}$ = 78.1, $NHCH_2$), 127.3 ($C_{2'}$*), 128.4 ($C_{3'}$*), 128.8 (d, $^2J_{CP}$ = 12.0, C_2), 130.6 (d, $^1J_{CP}$ = 100.0, C_1), 131.0 (d, $^3J_{CP}$ = 9.7, C_3), 131.6 ($C_{1'}$), 132.3 (d, $^4J_{CP}$ = 2.9, C_4), 133.8 ($C_{4'}$), 167.6 (d, $^3J_{CP}$ = 4.6, C(O)), *may be reversed; ^{1}H NMR ($CDCl_3$) δ 4.48 (t, $^2J_{HP}$ = $^3J_{HH}$ = 6.0, 2H, NHC*H*$_2$), 7.25–7.33 (m, 2H, $C_{3'}$H), 7.37–7.58 (m, 7H, C_3H, C_4H, $C_{4'}$H), 7.72–7.85 (m, 6H, C_2H, $C_{2'}$H), 7.92–8.00 (br, 1H, NH). $[M+H]^+_{found}$ = 336.11470, $C_{20}H_{19}NO_2P$ requires 336.11479.

[(Benzoylamino)methyl]di-*p*-tolylphosphine oxide (3b)

Yield: 78% (0.15 g); Mp: 253-255 °C; ^{31}P NMR ($CDCl_3$) δ 31.4; ^{13}C NMR ($CDCl_3$) δ 21.6 (C_4*C*H$_3$), 39.6 (d, $^1J_{CP}$ = 77.9, $NHCH_2$), 126.8 ($C_{2'}$*), 127.3 ($C_{3'}$*), 127.5 (d, $^1J_{CP}$ = 102.7, C_1), 129.6 (d, $^2J_{CP}$ = 12.3, C_2), 131.1 (d, $^3J_{CP}$ = 10.1, C_3), 131.5 ($C_{1'}$), 133.8 ($C_{4'}$), 142.9 (d, $^4J_{CP}$ = 2.9, C_4), 167.6 (d, $^3J_{CP}$ = 4.6, C(O)), *may be reversed; ^{1}H NMR ($CDCl_3$) δ 2.37 (s, 6H, $C_{4'}CH_3$), 4.41 (t, $^2J_{HP}$ = $^3J_{HH}$ =6.1, 2H, NHC*H*$_2$), 7.11–7.48 (m, 7H, C_3H, $C_{3'}$H, $C_{4'}$H), 7.66 (dd, $^3J_{HP}$ =11.6, $^3J_{HH}$ =7.8, 4H, C_2H), 7.75 (d, $^3J_{HH}$ =7.1, 2H, $C_{2'}$H), 7.79–7.87 (br, 1H, NH). $[M+H]^+_{found}$ = 364.14580, $C_{22}H_{23}NO_2P$ requires 364.14609.

[(Benzoylamino)methyl]bis-3,5-dimetylphenylphosphine oxide (3c)

Yield: 58% (0.12 g); Mp: 200-201 °C; ^{31}P NMR ($CDCl_3$) δ 30.9; ^{13}C NMR ($CDCl_3$) δ 21.3 (C_3CH_3), 39.6 (d, $^1J_{CP}$ = 77.3, $NHCH_2$), 127.3 ($C_{2'}$*), 128.4 ($C_{3'}$*), 128.5 (d, $^2J_{CP}$ = 9.6, C_2), 130.5 (d, $^1J_{CP}$ = 99.4, C_1), 131.5 ($C_{1'}$), 134.0 ($C_{4'}$), 134.1 (d, $^3J_{CP}$ = 2.8, C_3), 138.5 (d, $^4J_{CP}$ = 12.6, C_4), 167.6 (d, $^3J_{CP}$ = 4.9, C(O)), *may be reversed; ^{1}H NMR (CDCl3) δ 2.35 (s, 12H, C_3CH_3), 4.45 (t, $^2J_{HP}$ = $^3J_{HH}$ = 5.2, 2H, NHCH_2), 7.18 (s, 2H, C_4H), 7.29–7.50 (m, 7H, C_2H, $C_{3'}H$, $C_{4'}H$), 7.56–7.69 (br, 1H, NH), 7.80 (d, $^3J_{HH}$ = 8.1, 2H, $C_{2'}H$); $[M+H]^+_{found}$ =392.17688, $C_{24}H_{27}NO_2P$ requires 392.17739.

[(Benzoylamino)methyl]dibenzylphosphine oxide (3d)

Yield: 64% (0.11 g); Mp: 148-149 °C; ^{31}P NMR ($CDCl_3$) δ 43.3; ^{13}C NMR ($CDCl_3$) δ 35.1 (d, $^1J_{CP}$ = 60.1, $P(O)CH_2$), 37.5 (d, $^1J_{CP}$ = 72.3, $NHCH_2$), 127.2 (d, $^5J_{CP}$ = 2.9, C_4), 127.4 ($C_{2'}$*), 128.4 ($C_{3'}$*), 128.9 (d, $^4J_{CP}$ = 2.4, C_3), 129.8 (d, $^3J_{CP}$ = 5.2, C_2), 130.8 (d, $^2J_{CP}$ = 7.7, C_1), 131.7 ($C_{1'}$), 133.4 ($C_{4'}$), 167.7 (d, $^3J_{CP}$ = 3.6. C(O)), *may be reversed; ^{1}H NMR ($CDCl_3$) δ 3.10–3.22 (m, 4H, $P(O)CH_2$), 3.74 (t, $^2J_{HP}$ = $^3J_{HH}$ = 5.4, 2H, NHCH_2), 7.21–7.27 (m, 6H, C_2H, C_4H), 7.27–7.32 (m, 4H, C_3H), 7.35–7.41 (m, 4H, $C_{3'}H$), 7.45–7.50 (m, 1H, $C_{4'}H$) 7.69–7.74 (br, 1H, NH), 7.81 (d, $^3J_{HH}$ = 7.4, 2H, $C_{2'}H$). $[M+H]^+_{found}$ = 364.14580, $C_{22}H_{23}NO_2P$ requires 364.14609.

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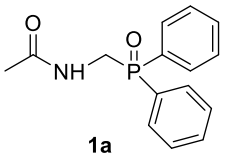
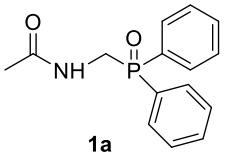


1a

^{13}C NMR ($CDCl_3$)



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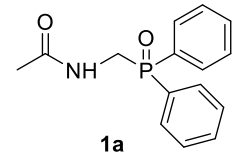

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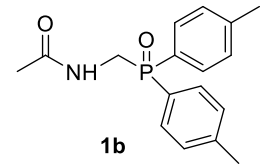
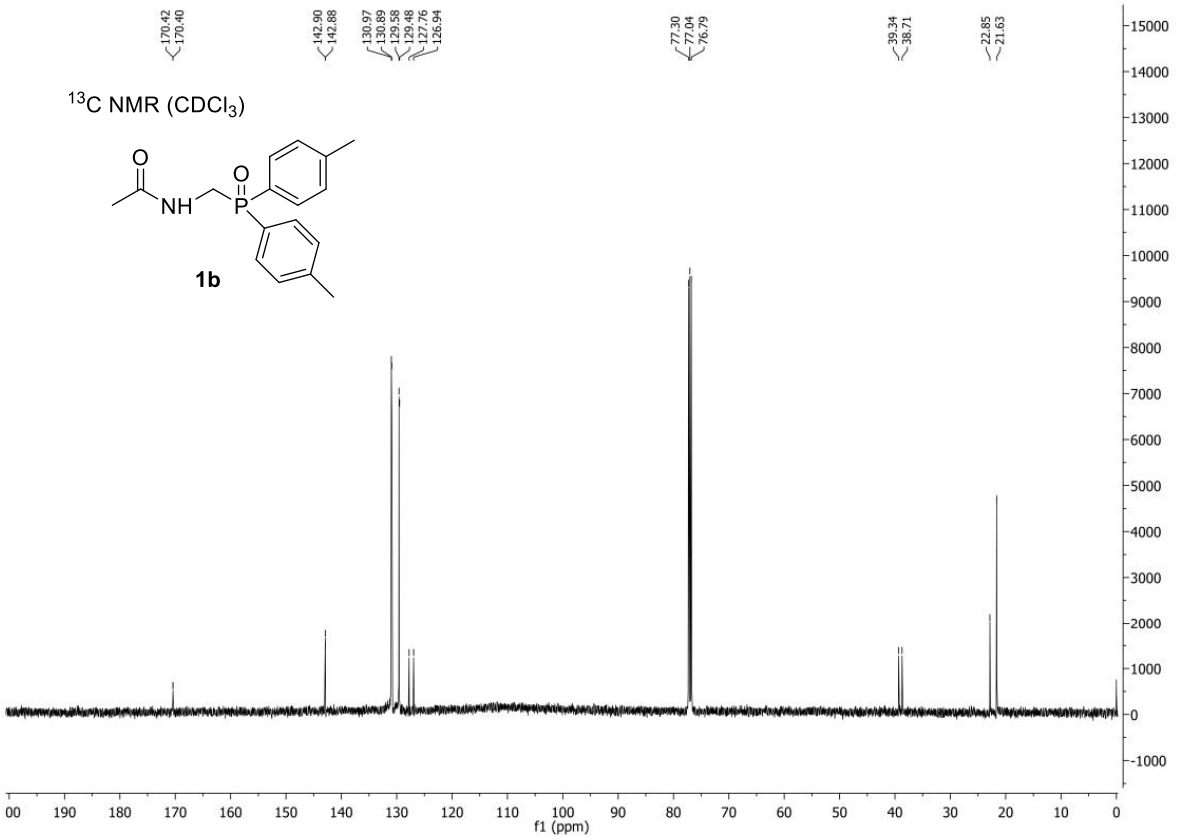


1b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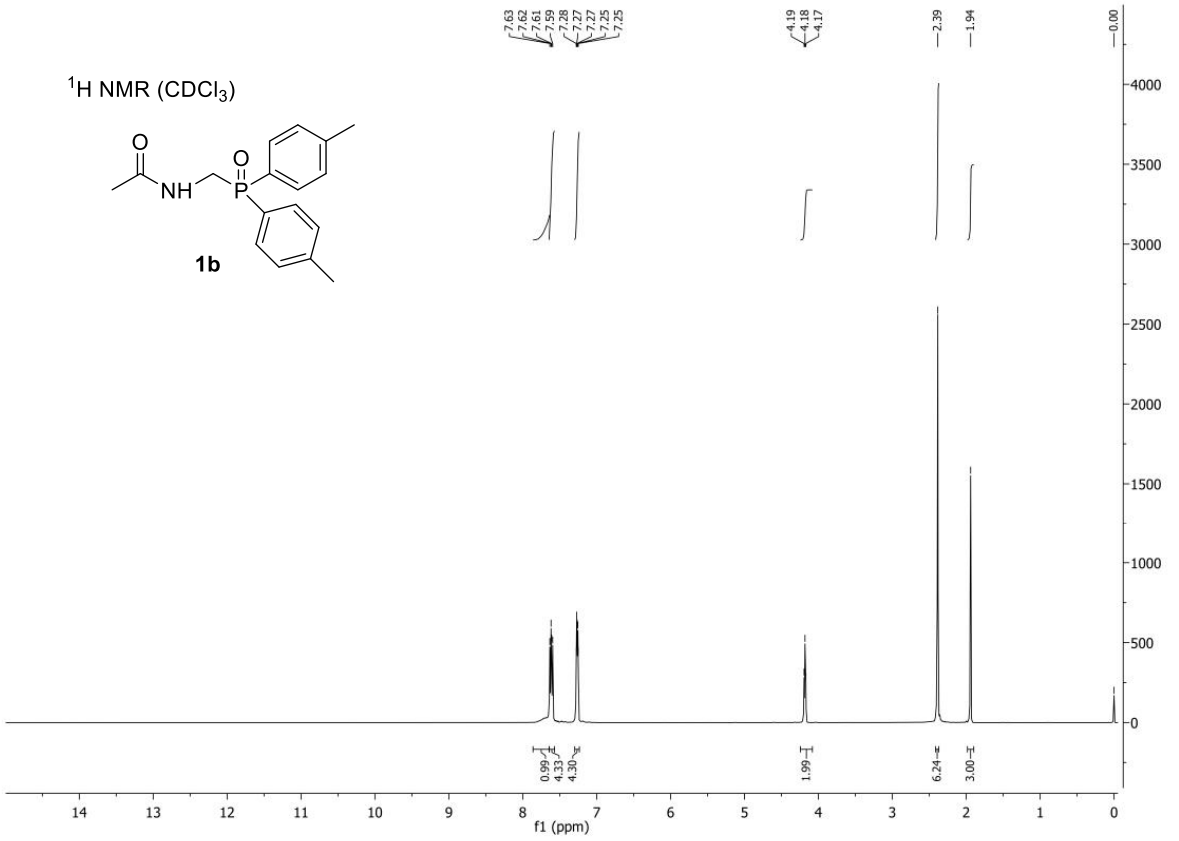
22.85
21.63
^{13}C NMR ($CDCl_3$)
1b
f1 (ppm)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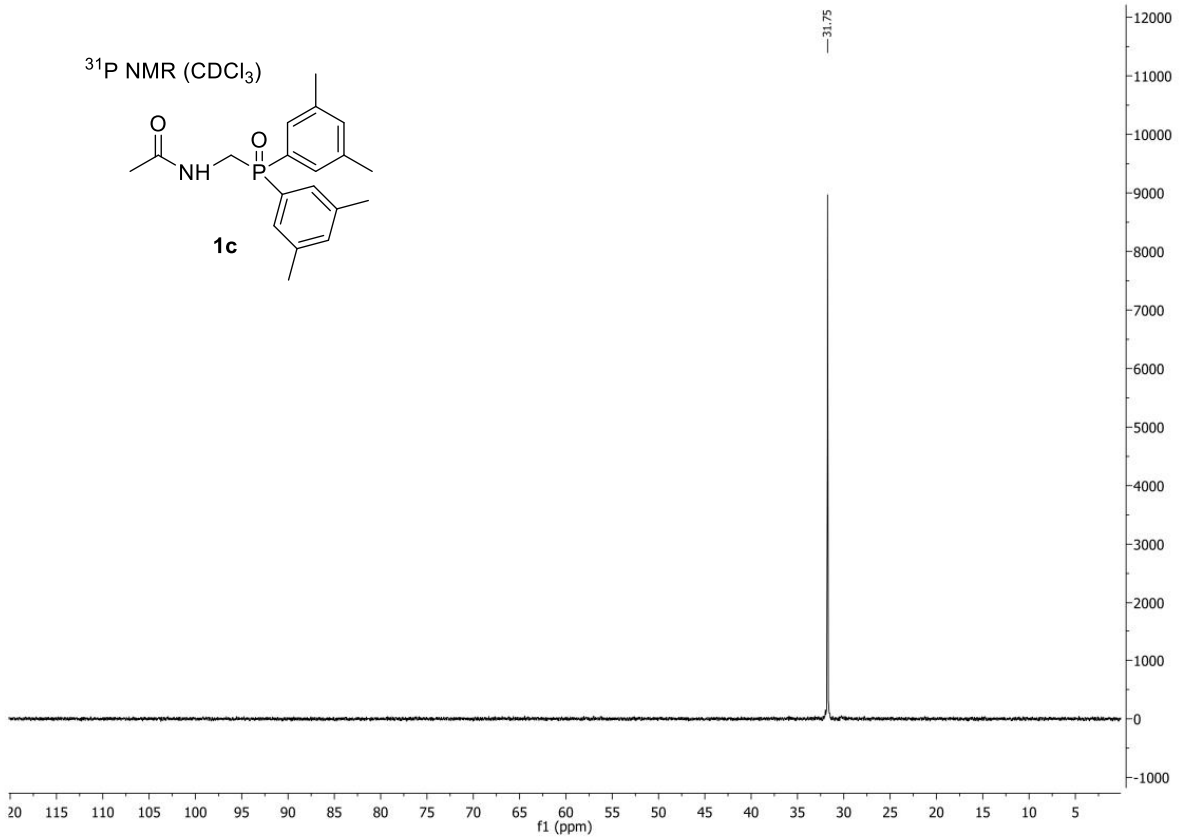
3.00
f1 (ppm)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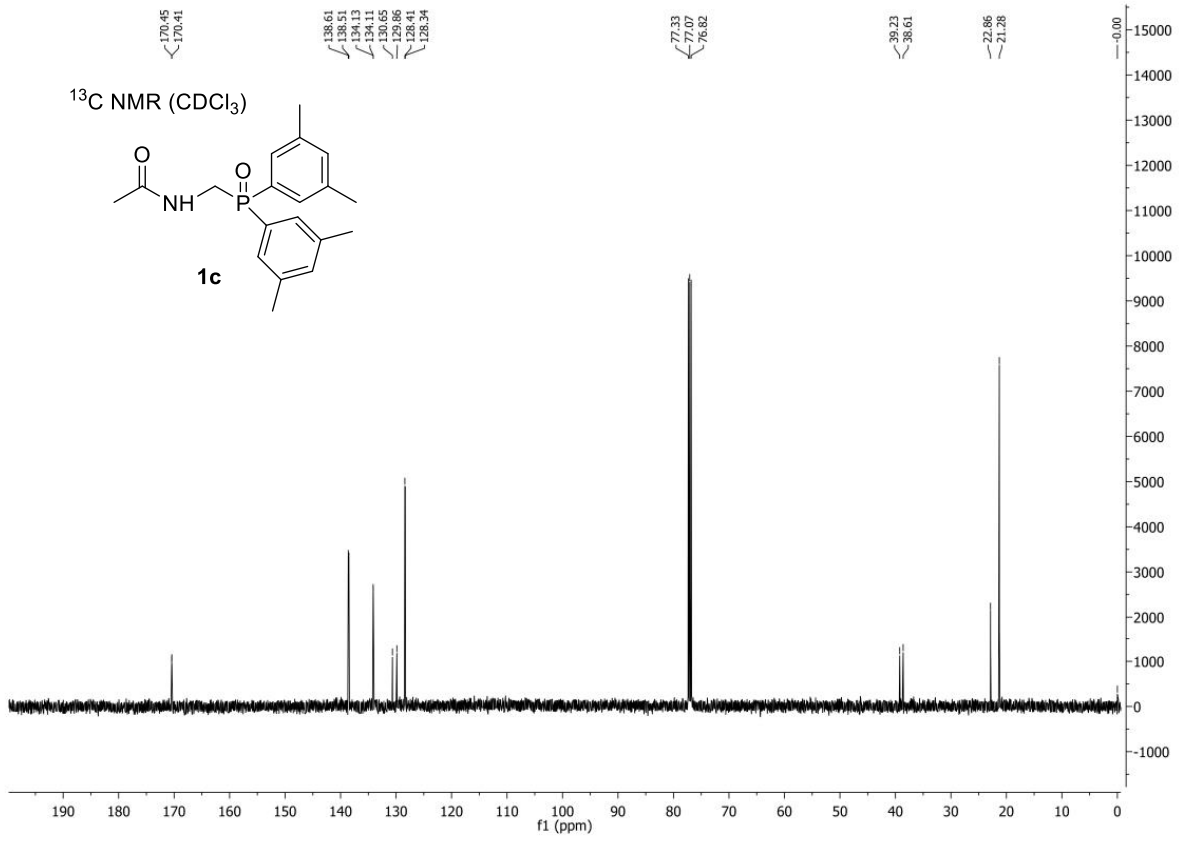
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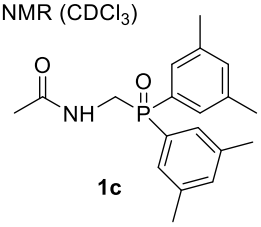
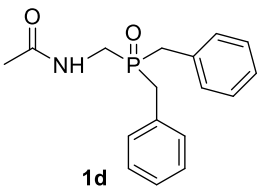
f1 (ppm)

^{1}H NMR ($CDCl_3$)



1c

^{31}P NMR ($CDCl_3$)



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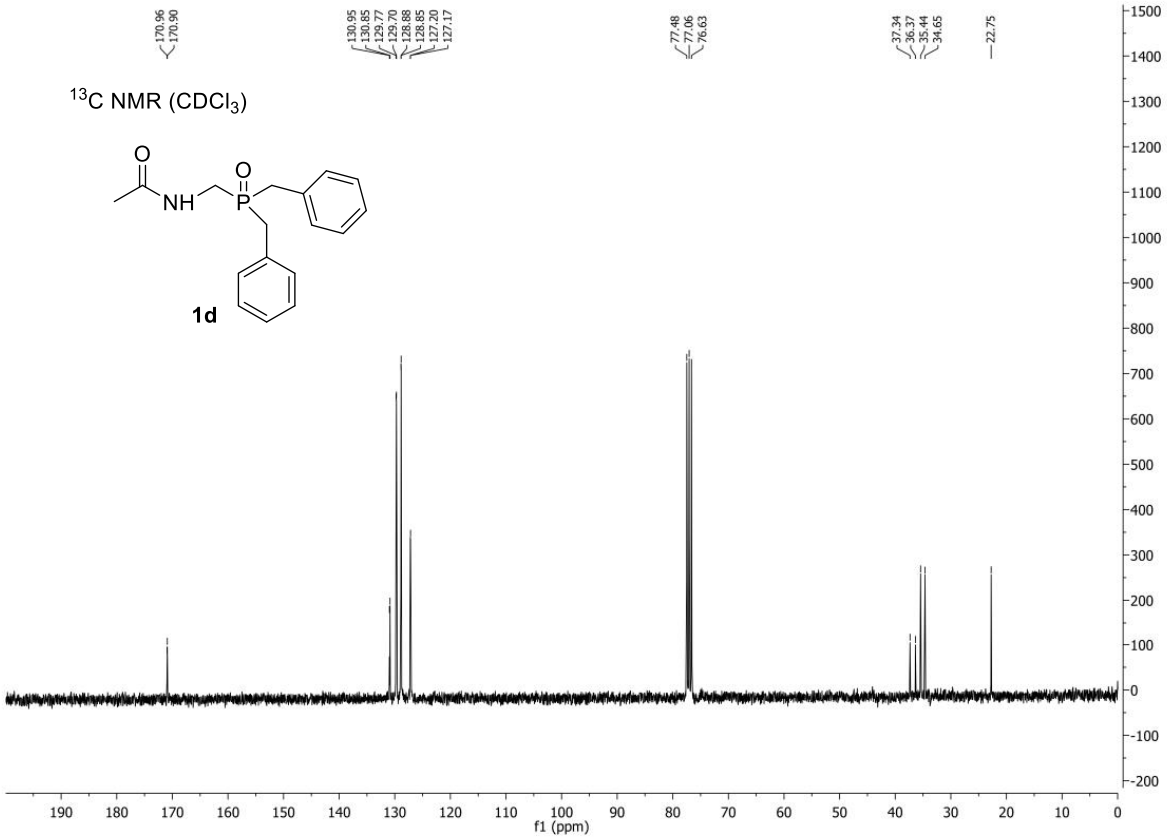
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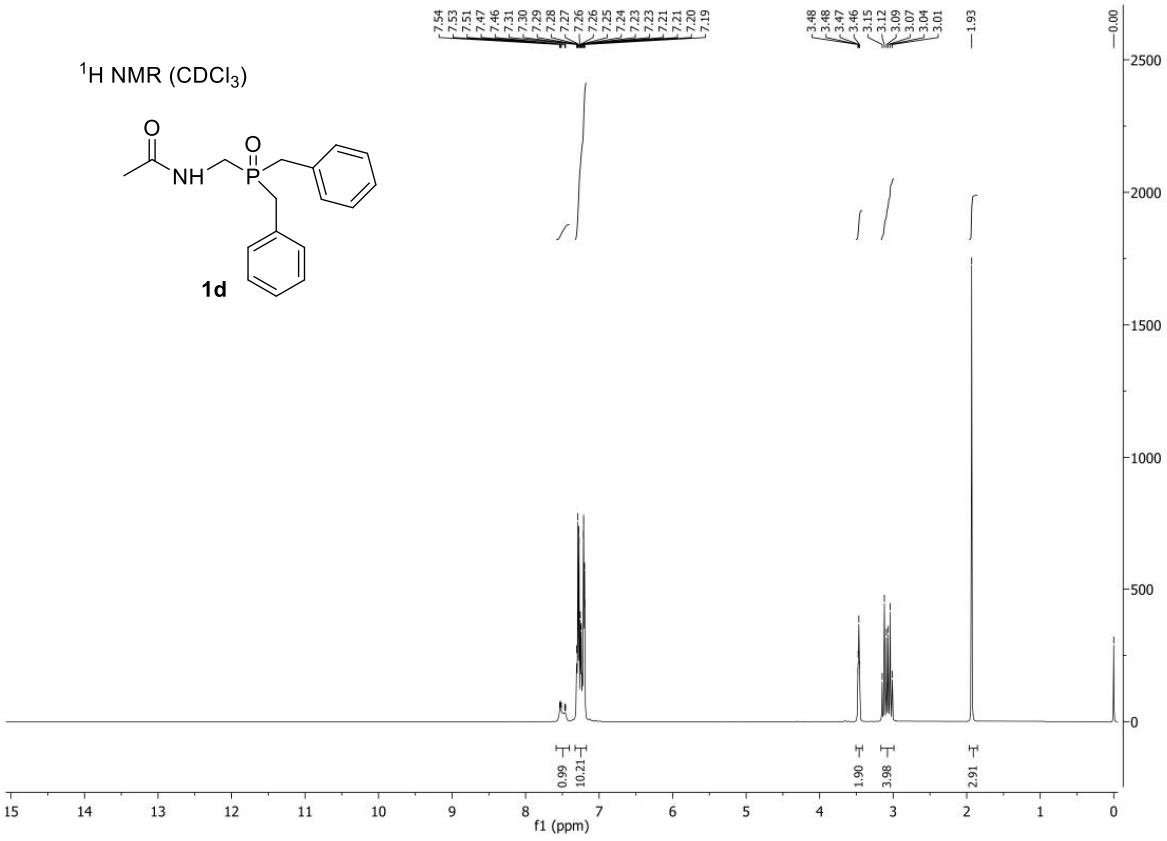
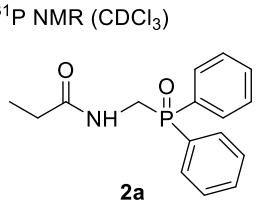
37.34
36.37
35.44
34.65
22.75
13C NMR (CDCl3)
O
NH
O
P
1d
f1 (ppm)

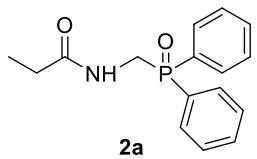

7.54
7.53
7.51
7.47
7.46
7.31
7.30
7.29
7.28
7.27
7.26
7.26
7.25
7.24
7.23
7.23
7.21
7.21
7.20
7.19
3.48
3.48
3.47
3.46
3.15
3.12
3.09
3.07
3.04
3.01
1.93
0.00
1H NMR (CDCl3)
O
NH
O
P
1d
0.99
10.21
1.90
3.98
2.91
f1 (ppm)

^{31}P NMR ($CDCl_3$)



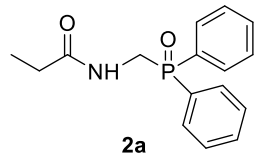
2a

^{13}C NMR ($CDCl_3$)



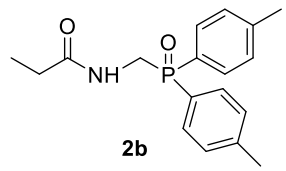
2a

^{1}H NMR ($CDCl_3$)

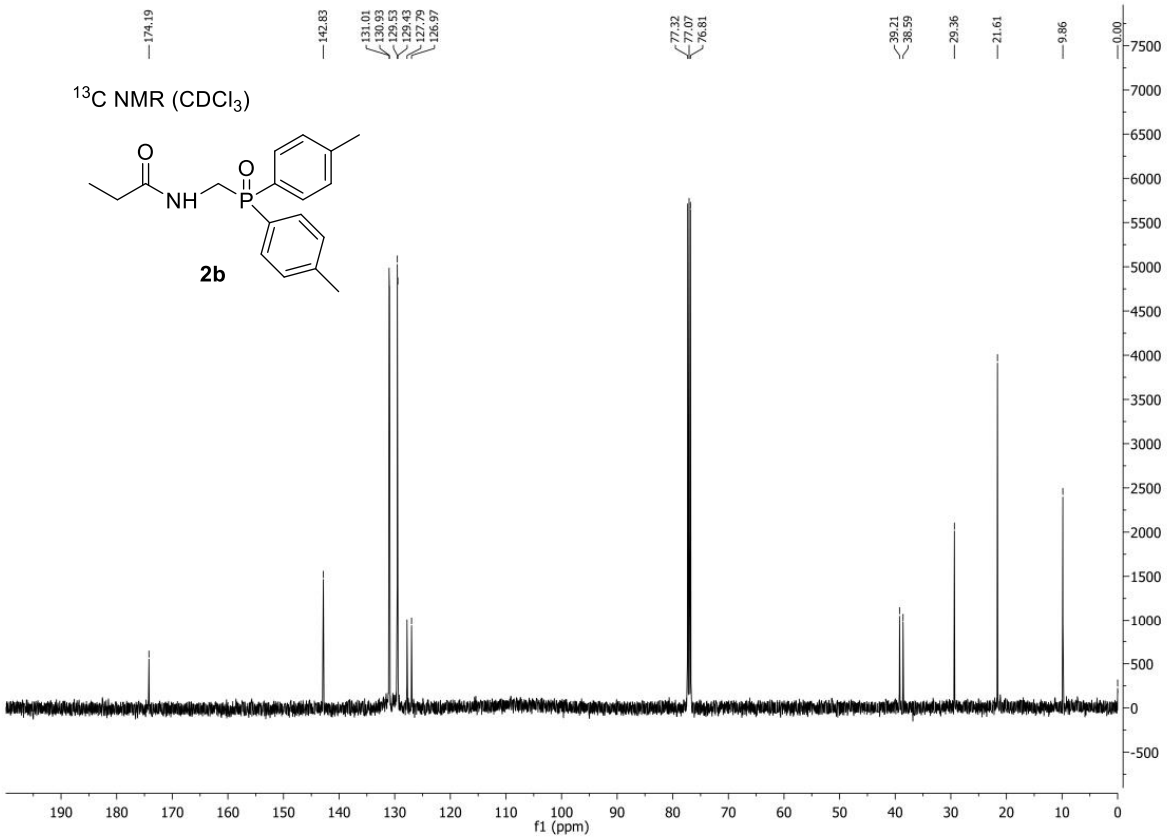


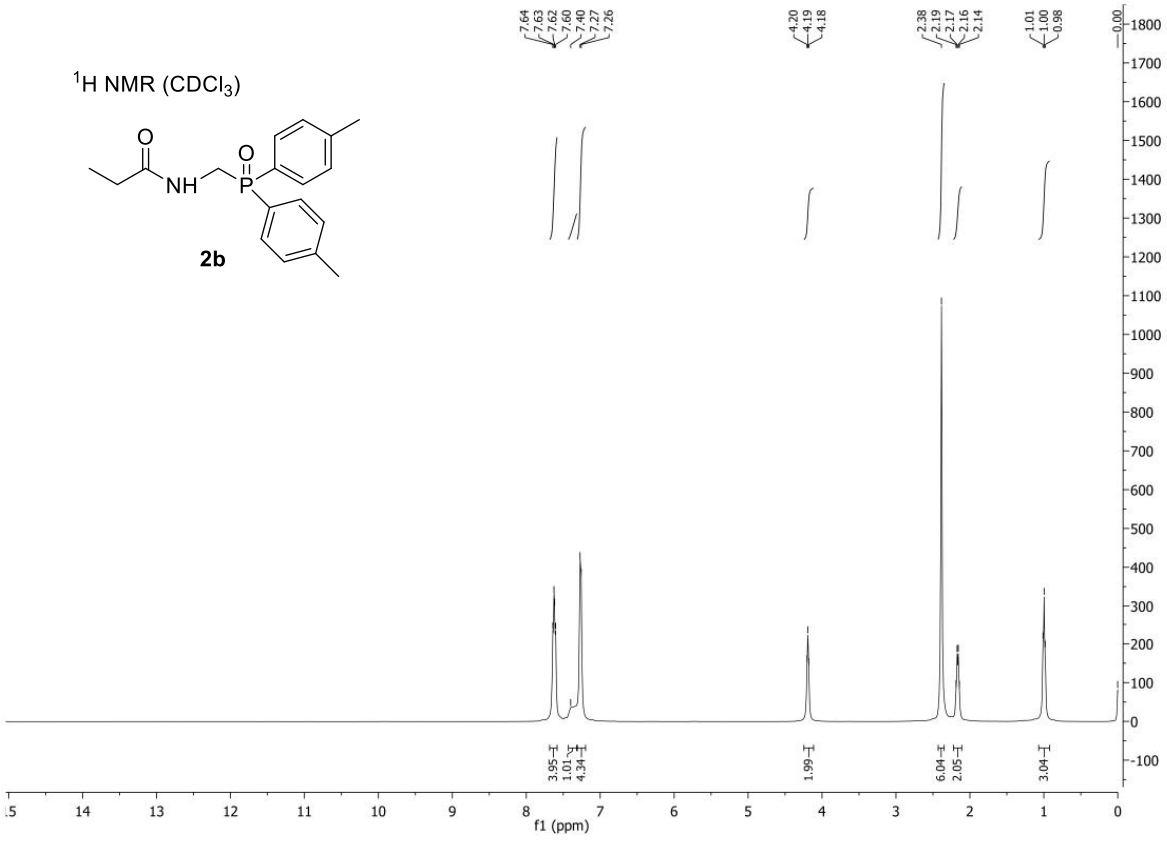
2a

^{31}P NMR ($CDCl_3$)

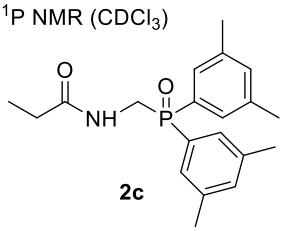


2b


174.19
142.83
131.01
130.93
129.53
129.43
127.79
126.97
77.32
77.07
76.81
39.21
38.59
29.36
21.61
9.86
0.00
13C NMR (CDCl3)
O
NH
O
P
2b
f1 (ppm)

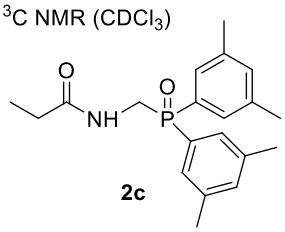

7.64
7.63
7.62
7.60
7.40
7.27
7.26
4.20
4.19
4.18
2.38
2.19
2.17
2.16
2.14
1.01
1.00
0.98
0.00
1H NMR (CDCl3)
O
NH
O
P
2b
3.95
1.01
4.34
1.99
6.04
2.05
3.04
f1 (ppm)

^{31}P NMR ($CDCl_3$)



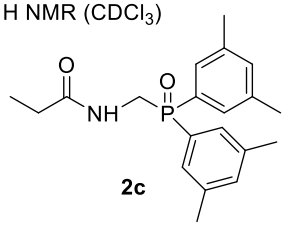
2c

^{13}C NMR ($CDCl_3$)



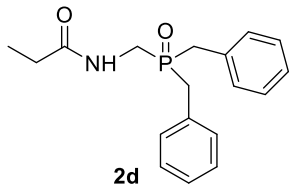
2c

^{1}H NMR ($CDCl_3$)

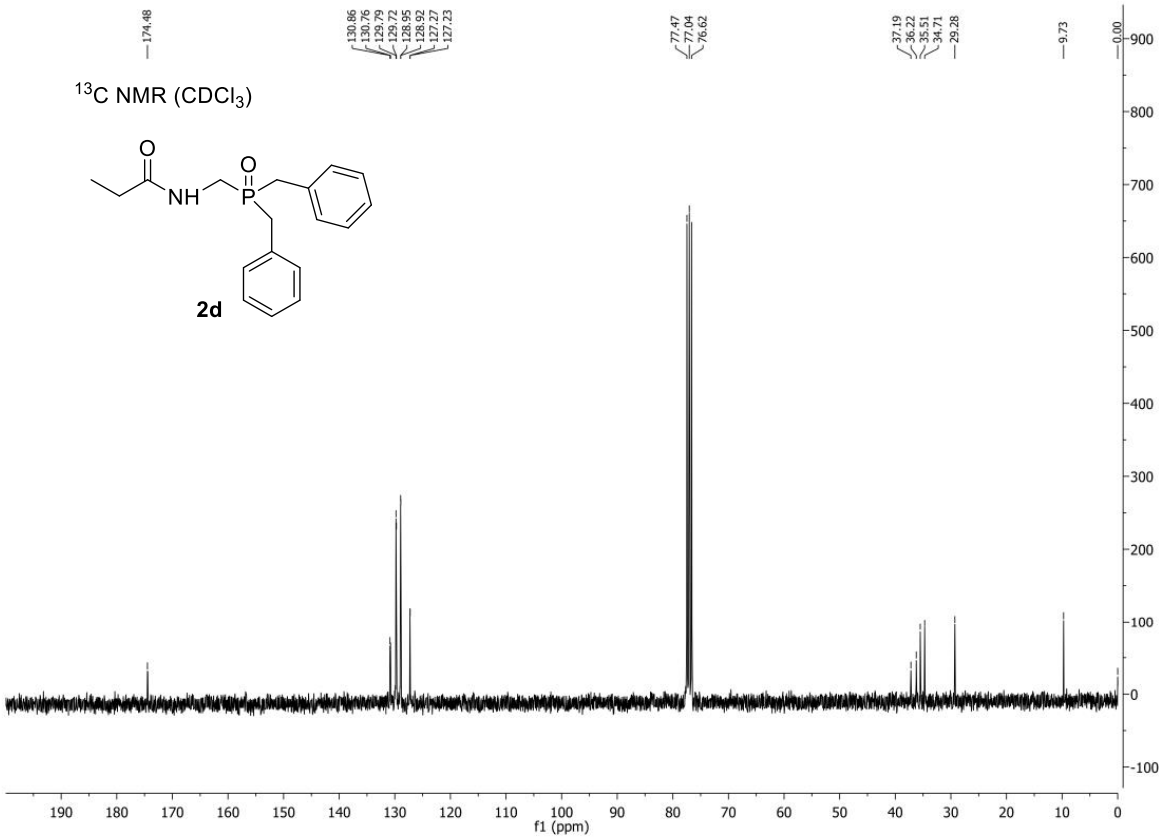


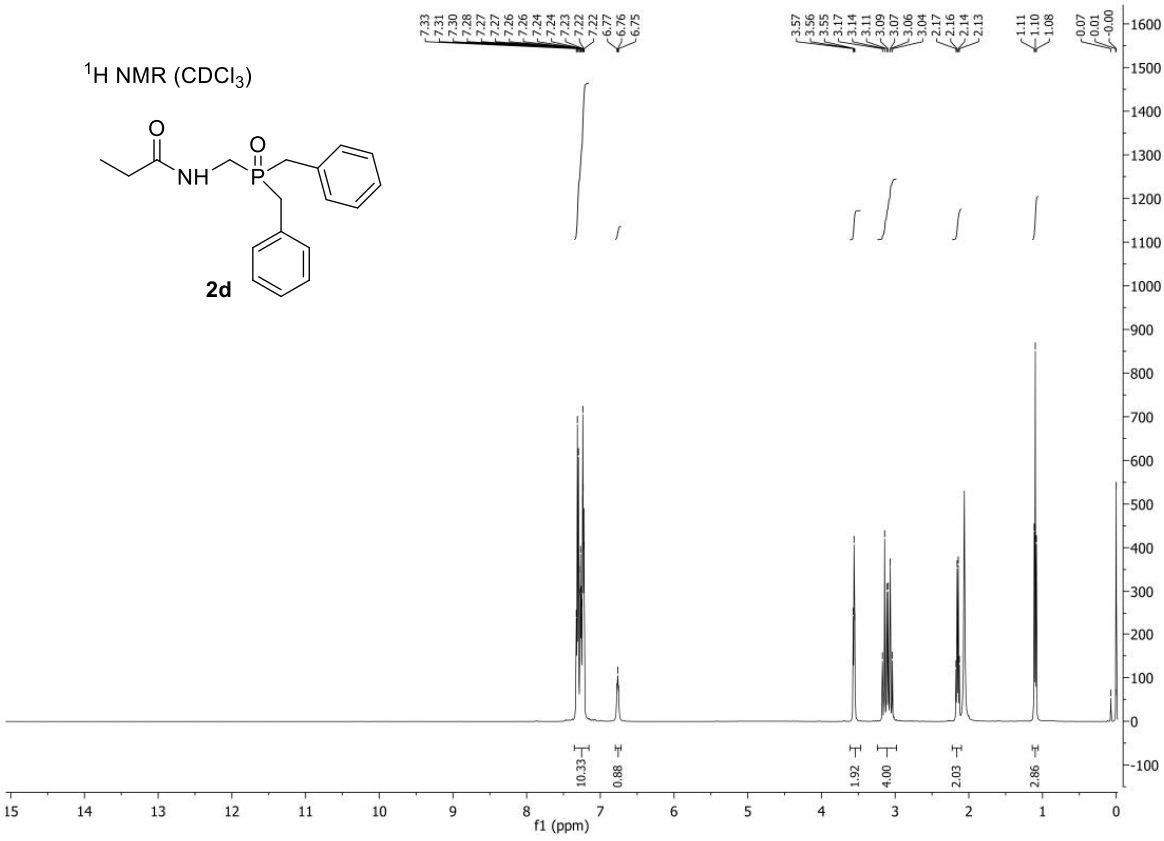
2c

^{31}P NMR ($CDCl_3$)

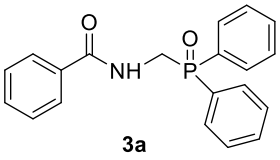


2d


174.48
130.86
130.76
129.79
129.72
128.95
128.92
127.27
127.23
77.47
77.04
76.62
37.19
36.22
35.51
34.71
29.28
9.73
0.00
13C NMR (CDCl3)
O
NH
O
P
2d
f1 (ppm)

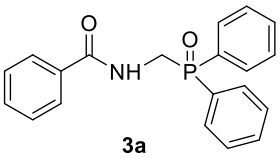

7.33
7.31
7.30
7.28
7.27
7.27
7.26
7.26
7.24
7.24
7.23
7.22
7.22
6.77
6.76
6.75
3.57
3.56
3.55
3.17
3.14
3.11
3.09
3.07
3.06
3.04
2.17
2.16
2.14
2.13
1.11
1.10
1.08
0.07
0.01
-0.00
1H NMR (CDCl3)
O
NH
O
P
2d
10.33
0.88
1.92
4.00
2.03
2.86
f1 (ppm)

^{31}P NMR ($CDCl_3$)



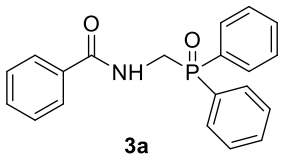
3a

^{13}C NMR ($CDCl_3$)



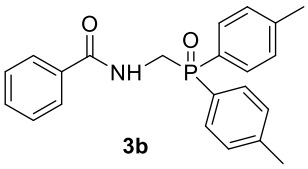
3a

^{1}H NMR ($CDCl_3$)

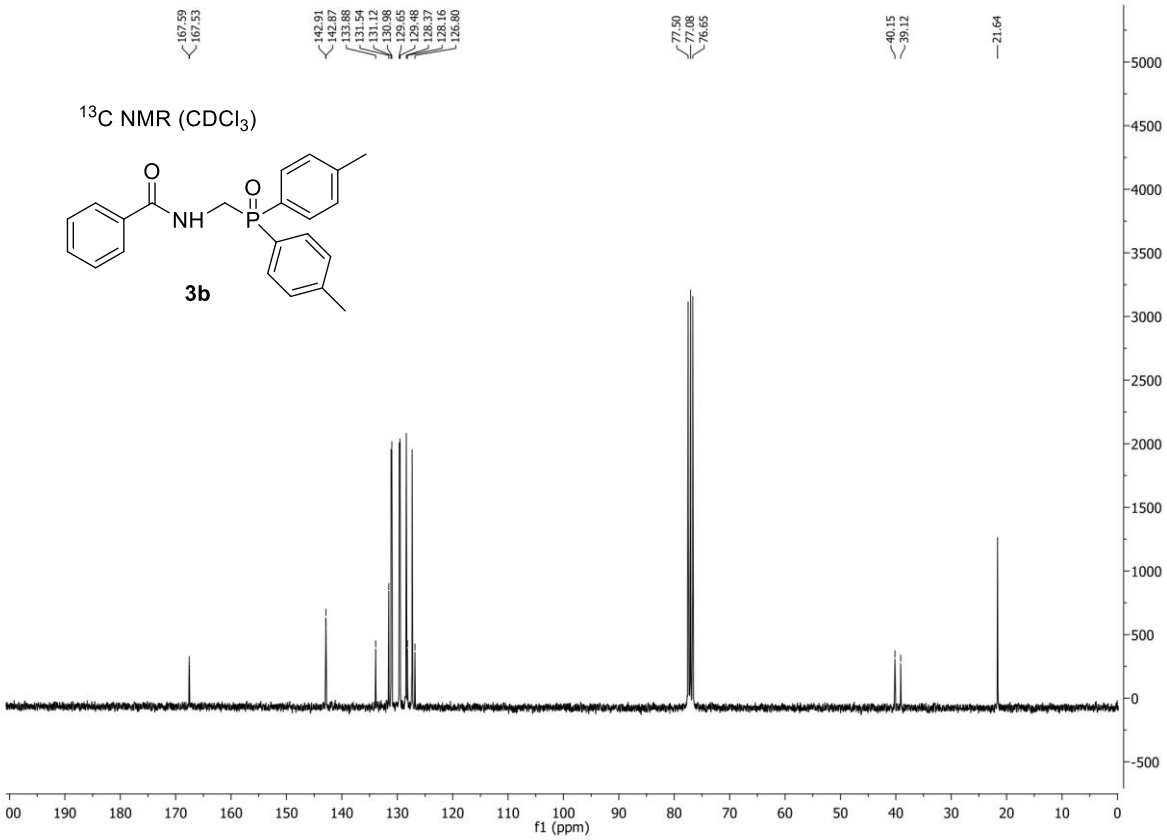


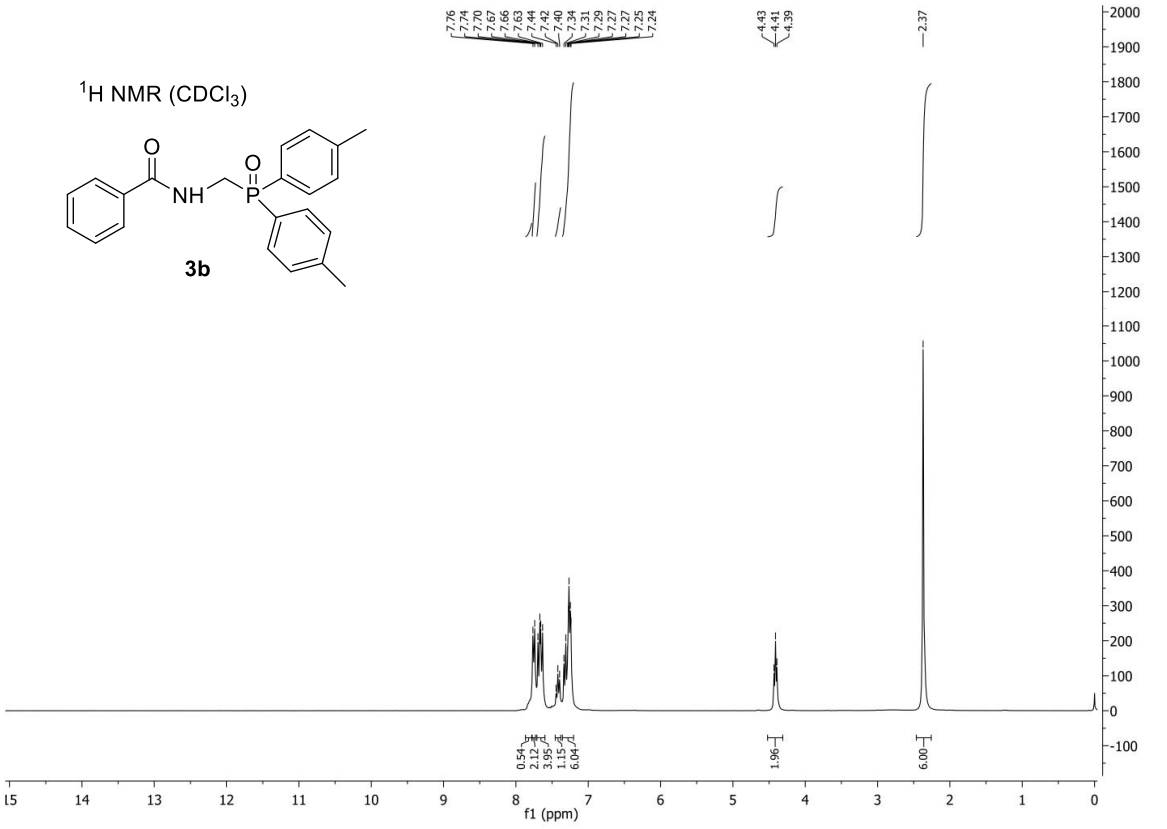
3a

^{31}P NMR ($CDCl_3$)

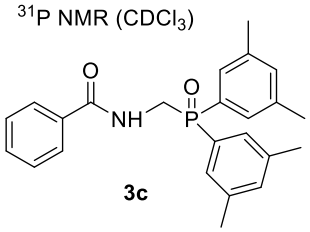


3b


167.59
167.53
142.91
142.87
133.88
131.54
131.12
130.98
129.65
129.48
128.37
128.16
126.80
77.50
77.08
76.65
40.15
39.12
21.64
13C NMR (CDCl3)
O
NH
O
P
3b
f1 (ppm)

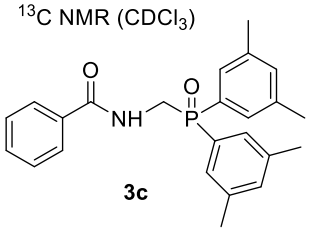

7.76
7.74
7.70
7.67
7.66
7.63
7.44
7.42
7.40
7.34
7.31
7.29
7.27
7.27
7.25
7.24
4.43
4.41
4.39
2.37
1H NMR (CDCl3)
O
NH
O
P
3b
0.54
2.12
3.95
1.15
6.04
1.96
6.00
f1 (ppm)

^{31}P NMR ($CDCl_3$)



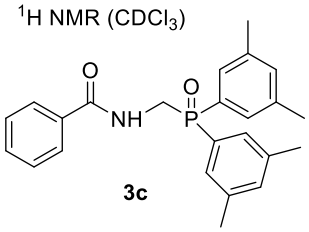
3c

^{13}C NMR ($CDCl_3$)



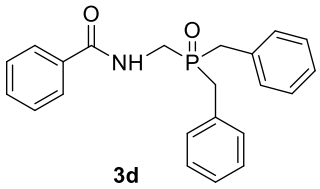
3c

^{1}H NMR ($CDCl_3$)

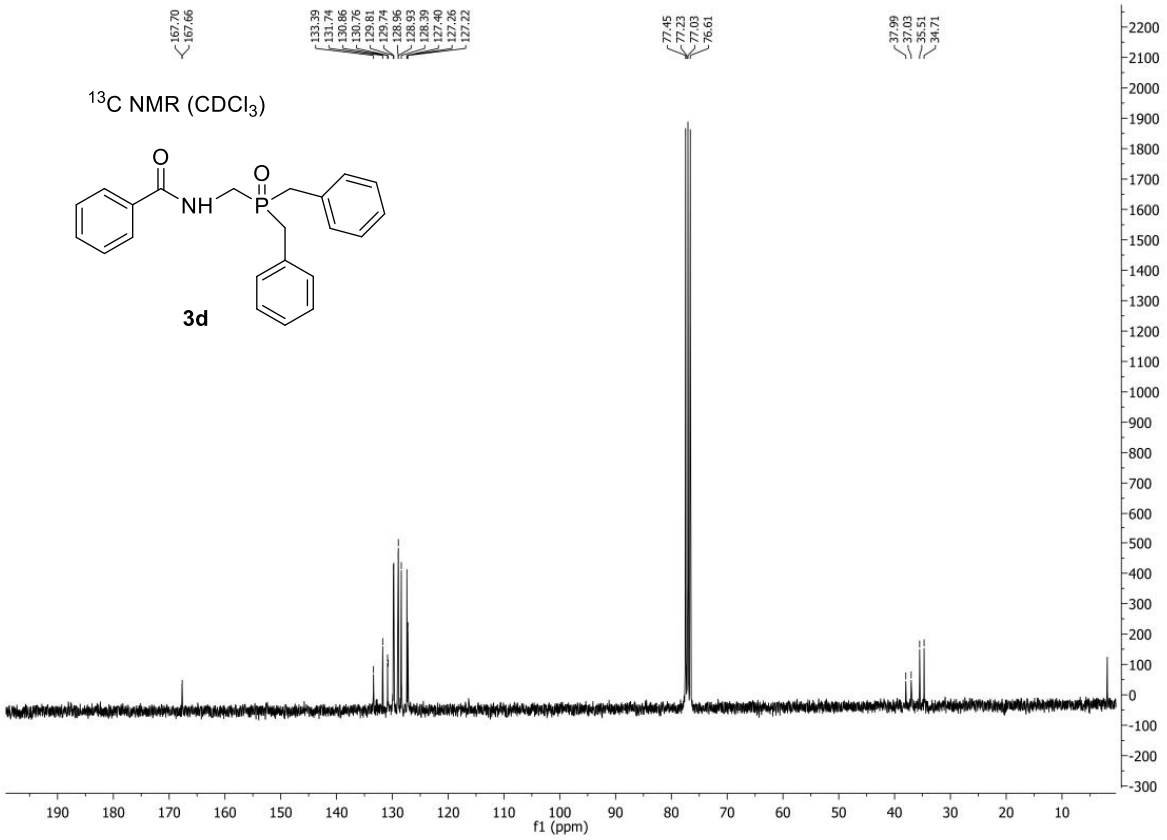


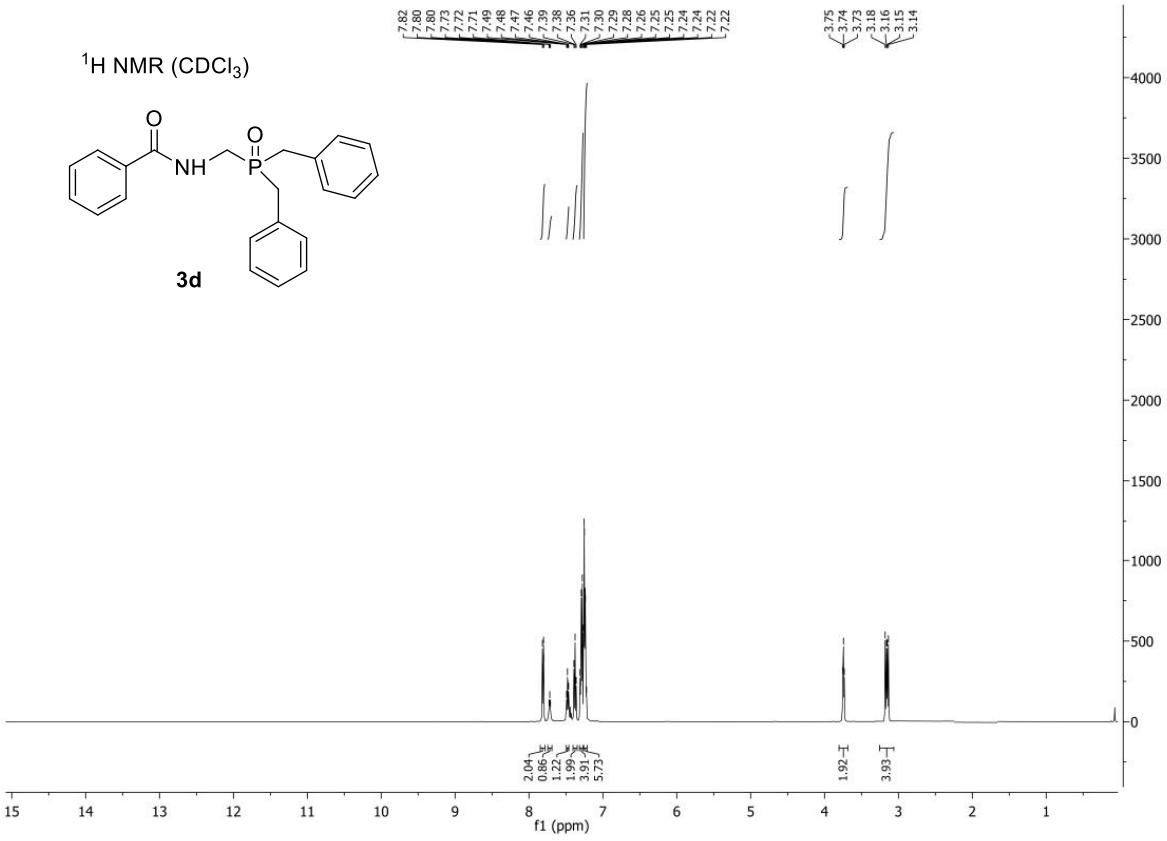
3c

^{31}P NMR ($CDCl_3$)



3d


167.70
167.66
133.39
131.74
131.66
130.76
129.81
129.74
128.96
128.93
128.39
127.40
127.26
127.22
77.45
77.23
77.03
76.61
37.99
37.03
35.51
34.71
13C NMR (CDCl3)
O
NH
O
P
3d
f1 (ppm)


7.82
7.80
7.80
7.73
7.72
7.71
7.49
7.48
7.47
7.46
7.39
7.38
7.36
7.31
7.30
7.29
7.28
7.26
7.25
7.25
7.24
7.24
7.22
7.22
3.75
3.74
3.73
3.18
3.16
3.15
3.14
1H NMR (CDCl3)
O
NH
O
P
3d
2.04
0.86
1.22
1.99
3.91
5.73
1.92
3.93
f1 (ppm)