Supplementary Material

Highly efficient and extremely simple protocol for the oxidation α-hydroxyphosphonates to α-ketophosphonates using Dess-Martin periodinane

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Spectral data
Diethyl (4-chlorobenzoyl) phosphonate, 2a: $^1$H-NMR (300 MHz, CDCl$_3$): $\delta$ 1.34 (t, $J = 7.2$ Hz, 6H, $2 \times$ OCH$_2$CH$_3$), 4.25 (m, 4H, $2 \times$ OCH$_2$CH$_3$), 7.44 (d, $J = 8.4$ Hz, 2H, ArHs), 8.18 (d, $J = 8.4$ Hz, 2H, ArHs); $^{13}$C-NMR (75 MHz, CDCl$_3$): $\delta$ 16.25 (d, $^3$J$_{C-P} = 6$ Hz, OCH$_2$CH$_3$), 64.26 (d, $^2$J$_{C-P} = 7.5$ Hz, OCH$_2$CH$_3$), 129.24, 131.17, 133.35, 134.21, 141.47, 175.30 (ArCs), 197.63 (d, $^1$J$_{C-P} = 177$ Hz, ArCO) ppm.

Figure S 1: $^1$H - NMR of compound 2a
Figure S 2: $^{13}$C - NMR of compound 2a

Figure S 3: DEPT of compound 2a
Figure S 4: $^1$H - NMR of compound 2d

Figure S 5: $^{13}$C - NMR of compound 2d
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Figure S 13: $^1$H - NMR of compound 2l
Figure S 14: $^{13}$C - NMR of compound 2l

Figure S 15: DEPT of compound 2l
Figure S 16: $^1$H - NMR of compound 2n

Figure S 17: $^{13}$C - NMR of compound 2n
Figure S 18: DEPT of compound 2n
Spectroscopic data of α-hydroxyphosphonates

**AHP-1  Dethyl-1-hydroxy-1-(4-chlorophenyl) methyl phosphonate**

\(^1\)H NMR (300 MHz, CDCl\(_3\)): δ 1.23 (t, J = 8Hz, 3H) 1.26 (t, J = 8Hz, 3H), 4.01- 4.08 (m, 4H), 4.59(bs, 1H), 5.00(d, \(^1\)J\(_{\text{PC}}\) 12Hz, 1H), 7.31 (d J = 6Hz 2H), 7.41(d, J = 6Hz, 2H).

**AHP-2  Dethyl-1-hydroxy-1-(4-methylphenyl) methyl phosphonate**

\(^1\)H NMR (200 MHz, CDCl\(_3\)): δ 1.22(t, J = 8Hz, 3H) 1.27(t, J = 8Hz, 3H), 2.34 (s, 3H), 2.05(bs, 1H), 3.90-4.20 (m, 4H), 4.98(d, \(^1\)J\(_{\text{PH}}\) 11Hz, 1H), 7.16 (d J = 8Hz 2H), 7.36(dd, J=8Hz, 2Hz);
\(^{13}\)CNMR (75MHz, CDCl\(_3\)): δ 16.18, 21.04, 63.11, 70.37(d, \(^1\)J\(_{\text{PC}}\) = 169.5Hz), 127.02, 128.72, 133.70, 137.52

**AHP-3  Dethyl-1-hydroxy-1-(4-isopropylphenyl) methyl phosphonate**

\(^1\)H NMR (300 MHz, CDCl\(_3\)): δ 1.17-1.31(m, 6H), 2.89 (septet, J=6.9Hz, 1H ), 3.96-4.08(m, 4H), 4.98(d, \(^1\)J\(_{\text{PH}}\) 10.5Hz, 1H), 5.41(bs, 1H), 7.19(d, J = 7.8Hz, 2H);7.38(d, J = 7.8Hz,2H);
\(^{13}\)CNMR (75MHz, CDCl\(_3\)): δ 16.20, 23.59, 23.82, 33.69, 63.13 (d, \(^2\)J\(_{\text{PC}}\) = 7.0Hz), 63.45(d, \(^2\)J\(_{\text{PC}}\) = 7.0Hz), 126.20, 127.06, 133.78, 148.62

**AHP-4  Diethyl [(2,6-dimethylphenyl)(hydroxy)methyl]phosphonate: 1H -NMR (300 MHz, CDCl\(_3\)): δ 1.22 (t, J = 7.2 Hz, 3H, OCH2CH3), 1.30 (t, J = 7.2 Hz, 3H, OCH2CH3), 2.50 (s, 6H, 2 x ArCH3), 3.68 (s, CHOH), 3.92 – 4.15 (m, 4H, 2 x OCH2CH3), 5.51 (d, J = 15.9 Hz, CH-P), 6.98 – 7.10 (m, 3H, ArHs); 13C-NMR (75 MHz, CDCl3): δ 16.18 (d, 3JC-P = 6 Hz, OCH2CH3), 16.40 (d, 3JC-P = 6 Hz, OCH2CH3), 21.12 (2 x CH3), 62.64 (d, 2JC-P = 7.5 Hz, OCH2CH3), 63.05 (d, 2JC-P = 7.5 Hz, OCH2CH3), 68.78 (d, 1JC-P = 158.25 Hz, ArCH), 127.81, 127.86, 129.39, 132.43, 137.77 (ArCs) ppm.AHP-5 5.

**AHP-7  Dethyl-1-hydroxy-1-(3-nitrophenyl) methyl phosphonate**

\(^1\)H NMR (200 MHz, CDCl\(_3\)): δ 1.25 (t, J = 8Hz, 3H) 1.28 (t, J = 8Hz, 3H), 4.00-4.25 (m, 4H ), 5.16 (dd, \(^1\)J\(_{\text{PH}}\) =12Hz, \(^1\)J\(_{\text{H-OH}}\) = 6Hz, 1H), 5.50 (t, J = 6Hz, 1H), 7.49 (t, J = 8Hz, 1H), 7.80 (d, J = 8Hz, 1H), 8.14 (d, J = 8Hz, 1H), 8.40(bs, 1H);

**AHP-8  Dethyl-1-hydroxy-1-(cinnamyl) methyl phosphonate**
$^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 1.20-1.40 (2 X t, 6H), 4.0-4.25 (m 1H), 4.5 (bs, 1H), 4.68-4.78 6.26-6.40 (m, 1H), 6.75-6.87 (m, 1H), 7.28-7.38 (m, 5H); $^{13}$CNMR (75MHz, CDCl$_3$): $\delta$ 53.47, 53.61, 53.80, 67.29, 70.52, 123.67, 126.47, 128.37, 132.10, 132.37, 136.15;

AHP-9 Diethyl-1-hydroxy-1-(4-benzloxyphenyl) methyl phosphonate

$^1$H NMR (200 MHz, CDCl$_3$): $\delta$ 1.21 (t, $J = 8$ Hz, 3H) 1.26 (t, $J = 8$ Hz, 3H), 3.9-4.2 (m, 4H), 4.95 (d, $J_{PH}$ 10 Hz, 1H), 5.06 (s, 2H), 6.96 (d, $J = 8$ Hz, 2H), 7.31 (m, 5H), 7.39 (d, $J = 8$ Hz, 1H),

$^{13}$CNMR (50 MHz, CDCl$_3$): 16.20, 63.02, 69.80, 70.08 (d, $J_{PH}$ = 160.5 Hz), 114.44, 127.31, 128.40, 127.87, 129.10, 136.74, 158.47;

AHP-10 Dethyl-1-hydroxy-1-(4-allyloxyphenyl) methyl phosphonate

$^1$H NMR (200 MHz, CDCl$_3$): $\delta$ 1.20 (t, $J = 8$ Hz, 3H) 1.26 (t, $J = 8$ Hz, 3H), 2.2 (bs, 1H), 3.9-4.2 (m, 4H), 4.52 (d, $J = 6$ Hz, 2H), 4.93 (d, $J_{PH}$ = 10 Hz, 1H), 5.30 (d, $J = 9$ Hz, 1H), 5.37 (dd, $J = 16$ Hz, 2Hz, 2H), 5.9-6.3 (m, 1H), 7.00 (d, $J = 8$ Hz, 2H), 7.39 (d, $J = 8$ Hz, 2H), $^{13}$CNMR (50 MHz, CDCl$_3$): 16.20, 62.80, 68.64, 70.18 (d, $J_{PH}$ = 154 Hz), 114.32, 117.54, 128.32, 128.78, 130.04, 158.33

AHP-12 Diethyl [hydroxy(thiophen-2-yl)methyl]phosphonate

$^1$H -NMR (300 MHz, CDCl$_3$): $\delta$ 1.17 – 1.24 (m, 6H, 2 x OCH$_2$CH$_3$), 3.99 - 4.09 (m, 4H, 2 x OCH$_2$CH$_3$), 5.20 (d, $J = 11.4$ Hz, CH - P), 5.62 (s, OH), 6.92 (t, $J = 4.8$ Hz, 1H, ArH), 7.01 (t, $J = 3.0$ Hz, 1H, ArH), 7.22 (d, $J = 5.1$ Hz, 1H, ArH);

$^{13}$C -NMR (75 MHz, CDCl$_3$): $\delta$ 16.30 (d, 3JC-P = 5. 2 Hz, 2 x OCH$_2$CH$_3$), 63.40 (d, 2JC-P = 7.5 Hz, OCH$_2$CH$_3$), 63.74 (d, 2JC-P = 6.75 Hz, OCH$_2$CH$_3$), 66.70 (d, 1JC-P = 168.00 Hz, ArCH), 125.46, 125.88, 126.67, 139.93 (ArCs) ppm.
Figure AHP-1: PMR Spectrum α-hydroxyphosphonate-1

![PMR Spectrum α-hydroxyphosphonate-1](image)

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Figure AHP-2: PMR Spectrum α-hydroxyphosphonate-2
Figure AHP-3: PMR Spectrum α-hydroxyphosphonate-3

Figure AHP-7: PMR Spectrum α-hydroxyphosphonate-7
Figure AHP-8: PMR Spectrum α-hydroxyphosphonate-8

Figure AHP-9: PMR Spectrum α-hydroxyphosphonate-9
Figure AHP-10: PMR Spectrum α-hydroxyphosphonate-10

Figure AHP-13: PMR Spectrum α-hydroxyphosphonate-13
Figure AHP-14: PMR Spectrum α-hydroxyphosphonate-14