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Supporting Information

Redox Deracemization of Phosphonate-Substituted Dihydropyrimidines

Fan-Jie Meng, Bing-Ru Shao, Maria K. Velopolcek, Xuan Guo, Guang-Shou Feng* and Lei Shi*

E-mail: gf56@duke.edu

Email: shileichem@dlut.edu.cn

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1. General

General: All reactions were carried out under an atmosphere of nitrogen using the standard Schlenk techniques, unless otherwise noted. ¹H NMR and ¹³C NMR spectra were recorded at room temperature in CDCl₃ on 400 MHz instrument with tetramethylsilane (TMS) as internal standard. Enantiomeric excess was determined by HPLC analysis, using chiral column described below in detail. Optical rotations were measured by polarimeter. Flash column chromatography was performed on silica gel (200-300 mesh). All reactions were monitored by TLC analysis.

Materials: Commercially available reagents were used throughout without further purification. The anhydrous solvents for asymmetric transfer hydrogenation were also purchased without the further purification.

2. General Procedure for Synthesis of 3,4-Dihydropyrimidin-2-one

3,4-Dihydropyrimidin-2-one derivatives **1** can be conveniently prepared according to the known literature procedure with some minor modifications.^{1, 2} The compound **1m**, **11** are known.²



General procedure: A mixture of 2,4-dichloropyrimidine **S-1a** (2.959 g, 20 mmol), the corresponding arylboronic acid (20 mmol), $Pd(PPh_3)_4$ (0.231 g, 0.2 mmol) and sodium carbonate (2.119 g, 20 mmol) in acetonitrile (25 mL) and water (25 mL) was stirred at reflux for 8 h, then cooled to ambient temperature, then extracted with ethyl acetate (50 mL × 3). The combined organic layer was dried over anhydrous sodium sulfate. After filtration, the solvent was removed under the reduced pressure, and the residue was purified by flash chromatography on silica gel to give the corresponding products S-1b.

Subsequently, the above S-1b (10 mmol) in 38% hydrogen chloride aqueous solution (5.0 mL) was stirred at 100 °C for 5 h before the mixture was cooled and the pH adjusted to 7 with aqueous sodium hydroxide (1.0 M). The most water was removed under reduced pressure and the residue was extracted with chloroform (30 mL \times 3). The combined organic layer was dried over anhydrous sodium sulfate. After filtration, the solvent was removed under reduced pressure and the crude product purified by flash chromatography on silica gel to give the corresponding products S-1c.

A mixture of 2-hydroxypyrimidine **S-1c** (4 mmol), diisopropyl phosphonate (8 mmol, 2 equiv), and zirconium chloride (0.2 mmol, 5 mol%) in tetrahydrofuran (20 mL) was stirred at room temperature for 24 h. After the reaction was completed (determined by TLC), the solvent was removed under the reduced pressure. The residue was purified by flash chromatography on silica gel using the ethyl acetate/ triethylamine as eluent to give the racemic of 3,4-dihydropyrimidin- 2-one derivatives **1**.

3. General Procedure for Redox Deracemization



A racemic of 3,4-Dihydropyrimidin-2-one derivatives $\mathbf{1}$ (0.10 mmol), DDQ (25 mg, 0.11 mmol, 1.1 equiv) in ethyl acetate (4 mL) was stirred at room temperature for 5 min. After the reaction was completed (determined by TLC), chiral phosphoric acid (*R*)-2g (7.5 mg, 0.01 mmol, 10 mol%) and Hantzsch ester 3a (31.5 mg, 0.14 mmol, 1.4 equiv) was added. The mixture was stirred at room temperature for 24 h. The resulting mixture was concentrated in *vacuo* and further purification was performed by a silica gel column eluted with dichloromethane/methanol to give the optically active products 1. The enantiomeric excesses were determined by chiral HPLC.

Diisopropyl (*R*)-(2-oxo-6-phenyl-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (1a): 31 mg, 92% yield, new compound, white solid, mp: 157-158 °C, $R_f = 0.40$ (dichloromethane/ methanol



 $= 10/1), 96\% \text{ ee}, [\alpha]^{20}_{D} = -26.45 (c \ 0.31, \text{ MeOH}), ^{1}\text{H NMR (400 MHz, CDCl}_{3})$ $= 10/1), 96\% \text{ ee}, [\alpha]^{20}_{D} = -26.45 (c \ 0.31, \text{ MeOH}), ^{1}\text{H NMR (400 MHz, CDCl}_{3})$ $\delta 7.47-7.41 (m, 5\text{H}), 6.79 (s, 1\text{H}), 5.29 (s, 1\text{H}), 5.10 (s, 1\text{H}), 4.83-4.75 (m, 2\text{H}),$ $4.52-4.49 (m, 1\text{H}), 1.38-1.31 (m, 12\text{H}). ^{13}\text{C NMR (100 MHz, CDCl}_{3}) \delta 153.8,$ 138.3, 134.1 (d, J = 3.2 Hz), 129.3, 128.8, 125.1, 90.7 (d, J = 9.1 Hz), 72.2 (d, J = 7.7 Hz), 71.8 (d, J = 7.7 Hz), 51.8 (d, J = 160.5 Hz), 24.3 (d, J = 3.3 Hz), $24.1 (d, J = 3.2 \text{ Hz}), 24.1 (d, J = 4.0 \text{ Hz}), 23.9 (d, J = 5.1 \text{ Hz}). ^{31}\text{P NMR (162)}$

MHz, CDCl₃) δ 16.0. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 6.3 min (maj) and 7.2 min. HRMS (ESI) *m/z* calculated for C₁₆H₂₄N₂O₄P [M+H]⁺ 339.1464, found 339.1466.

Diisopropyl (*R*)-(2-oxo-6-(*p*-tolyl)-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (1b): 33 mg, 94% yield, new compound, white solid, mp: 168-169 °C, $R_f = 0.40$ (dichloromethane/ methanol



89.8 (d, J = 9.2 Hz), 72.2 (d, J = 7.6 Hz), 71.7 (d, J = 7.9 Hz), 51.7 (d, J = 160.5 Hz), 24.3 (d, J = 3.0 Hz), 24.1 (d, J = 3.3 Hz), 24.1 (d, J = 1.5 Hz), 23.9 (d, J = 5.2 Hz), 21.3. ³¹P NMR (162 MHz, CDCl₃) δ 16.2. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 6.9 min (maj) and 7.8 min. HRMS (ESI) *m/z* calculated for C₁₇H₂₆N₂O₄P [M+H]⁺ 353.1626, found 353.1625.

Diisopropyl (R)-(2-oxo-6-(m-tolyl)-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (1c): 34



mg, 96% yield, new compound, white solid, mp: 129-130 °C, $R_f = 0.40$ (dichloromethane/ methanol = 10/1), 92% ee, $[\alpha]^{20}_{D} = -52.43$ (*c* 0.34, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.53 (s, 1H), 7.29-7.24 (m, 3H), 7.18 (d, J = 5.9 Hz, 1H), 5.75 (s, 1H), 5.05 (s, 1H), 4.81-4.71 (m, 2H), 4.44 (d, J = 3.9 Hz, 1H), 2.37 (s, 3H), 1.35-1.28 (m, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 154.0, 138.5, 134.0 (d, J = 3.2 Hz), 130.0 (d, J = 4.2 Hz), 128.7 (d, J = 5.2 Hz), 125.8, 122.2, 90.4 (d, J = 7.8 Hz), 72.2 (d, J = 6.8 Hz), 71.8 (d, J = 7.2 Hz), 51.7 (d, J = 159.7 Hz), 24.3 (d, J = 3.1 Hz), 24.1(d, J = 3.0 Hz), 24.1 (d, J = 3.0 Hz), 24.1 (d, J = 8.5 Hz), 23.9 (d, J = 5.1 Hz), 21.4. ³¹P NMR (162 MHz, CDCl₃) δ 16.1. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 5.3 min (maj) and 6.3 min. HRMS (ESI) *m/z* calculated for C₁₇H₂₆N₂O₄P [M+H]⁺ 353.1626, found 353.1624.

Diisopropyl (R)-(6-(3,4-dimethylphenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)phosphon ate (1d): 36 mg, 98% yield, new compound, white solid, mp: 132-133 °C, $R_f = 0.40$



(dichloromethane/methanol = 10/1), 90% ee, $[\alpha]^{20}{}_{D}$ = -39.72 (*c* 0.36, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.22 (s, 1H), 7.17 (d, *J* = 0.8 Hz, 2H), 6.69 (s, 1H), 5.28 (s, 1H), 5.07-5.05 (m, 1H), 4.82-4.73 (m, 2H), 4.51-4.48 (m, 1H), 2.29 (d, *J* = 2.5 Hz, 6H), 1.38-1.30 (m, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 154.1, 138.3 (d, *J* = 10.7 Hz), 138.0, 137.1, 131.5 (d, *J* = 3.0 Hz), 130.0, 126.2, 122.4, 89.6 (d, *J* = 9.0 Hz), 72.2 (d, *J* = 7.6 Hz), 71.7

(d, J = 7.9 Hz), 51.7 (d, J = 160.2 Hz), 24.3 (d, J = 2.9 Hz), 24.1 (d, J = 4.1 Hz), 23.9 (d, J = 5.2 Hz), 19.8, 19.6. ³¹P NMR (162 MHz, CDCl₃) δ 16.2. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 5.9 min (maj) and 6.9 min. HRMS (ESI) *m/z* calculated for C₁₈H₂₈N₂O₄P [M+H]⁺ 367.1778, found 367.1777.

Diisopropyl (R)-(6-(4-isopropylphenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)phosphon ate (1e): 35 mg, 92% yield, new compound, white solid, mp: 158-159 °C, $R_f = 0.40$



(dichloromethane/methanol = 10/1), 94% ee, $[\alpha]^{20}{}_{D}$ = -39.43 (c 0.35, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.52 (s, 1H), 7.38 (d, J = 8.2 Hz, 2H), 7.24 (d, J = 8.3 Hz, 2H), 5.69 (s, 1H), 5.03 (dd, J = 5.2, 3.4 Hz, 1H), NH 4.77-4.74 (m, 2H), 4.48-4.46 (m, 1H), 2.94-2.88 (m, 1H), 1.35-1.28 (m, 12H), 1.24 (d, J = 6.9 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 154.0, 150.2,

138.2 (d, J = 10.9 Hz), 131.5 (d, J = 3.3 Hz), 126.9, 125.1 (d, J = 1.8 Hz), 89.9 (d, J = 9.1 Hz), 72.1 (d, J = 7.6 Hz), 71.8 (d, J = 7.6 Hz), 51.7 (d, J = 160.4 Hz), 33.9, 24.3 (d, J = 3.0 Hz), 24.2 (d, J = 3.1 Hz), 24.1 (d, J = 4.0 Hz), 23.9 (d, J = 5.2 Hz), 23.8. ³¹P NMR (162 MHz, CDCl₃) δ 16.2. HPLC: Chiracel OD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 7.1 min (maj) and 7.7 min. HRMS (ESI) *m*/*z* calculated for C₁₉H₃₀N₂O₄P [M+H]⁺ 381.1934, found 381.1932.

Diisopropyl (*R*)-(6-(4-(tert-butyl)phenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)phosph onate (1f): 37 mg, 94% yield, new compound, white solid, mp: 191-192 °C, $R_f = 0.40$



(dichloromethane/methanol = 10/1), 95% ee, $[\alpha]^{20}_{D}$ = -35.41 (*c* 0.37, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.53 (s, 1H), 7.40 (s, 4H), 5.70 (s, 1H), 5.06-5.04 (m, 1H), 4.81-4.71 (m, 2H), 4.48-4.46 (m, 1H), 1.36-1.28 (m, 21H). ¹³C NMR (100 MHz, CDCl₃) δ 154.0, 152.4, 138.1 (d, *J* = 10.8 Hz), 131.1, 125.7, 124.8, 90.0 (d, *J* = 9.3 Hz), 72.1 (d, *J* = 7.6 Hz), 71.8 (d, *J* = 7.7 Hz), 51.8 (d, *J* = 160.5 Hz), 34.7, 31.2, 24.3 (d, *J* = 3.2 Hz), 24.2 (d, *J* = 3.3 Hz), 24.1 (d, *J* = 4.3 Hz), 23.9 (d, *J* = 5.1 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 16.1. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 6.3 min and 7.1 min (maj). HRMS (ESI) *m/z* calculated for C₂₀H₃₂N₂O₄P [M+H]⁺ 395.2093, found 395.2092.

Diisopropyl (*R*)-(6-(4-chlorophenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (1g): 35 mg, 94% yield, new compound, white solid, mp: 184-185 °C, $R_f = 0.40$ (dichloromethane/



methanol = 10/1), 95% ee, $[\alpha]^{20}{}_{D}$ = -39.72 (*c* 0.35, MeOH). ¹H NMR (400 MHz, DMSO-d₆) δ 8.64 (s, 1H), 7.51 (d, *J* = 8.6 Hz, 2H), 7.46-7.44 (m, 2H), 6.95 (s, 1H), 5.00-4.98 (m, 1H), 4.65-4.56 (m, 2H), 4.34-4.30 (m, 1H), 1.30-1.18 (m, 12H). ¹³C NMR (100 MHz, DMSO-d₆) δ 153.7, 138.0 (d, *J* = 9.9 Hz), 133.8, 133.2 (d, *J* = 3.8 Hz), 129.0, 127.6 (d, *J* = 1.4 Hz), 91.9 (d,

J = 7.7 Hz), 71.4 (d, J = 7.6 Hz), 71.2 (d, J = 7.6 Hz), 50.9 (d, J = 158.5 Hz), 24.5 (d, J = 3.0 Hz), 24.4 (d, J = 3.5 Hz), 24.2 (d, J = 4.7 Hz), 24.1 (d, J = 5.0 Hz). ³¹P NMR (162 MHz, DMSO-d₆) δ 17.4. HPLC: Chiracel OD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 6.3 min and 7.4 min (maj). HRMS (ESI) *m/z* calculated for C₁₆H₂₃ClN₂O₄P [M+H]⁺ 373.0894, found 373.0893.

Diisopropyl (*R*)-(6-(3-chlorophenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (1h): 36 mg, 97% yield, new compound, white solid, mp: 165-166 °C, $R_f = 0.40$



(dichloromethane/methanol = 10/1), 93% ee, $[\alpha]^{20}{}_{D}$ = -56.39 (c 0.36, $\stackrel{P(O'Pr)_2}{\underset{H}{}}$ MeOH). ¹H NMR (400 MHz, CDCl₃) δ 8.20 (s, 1H), 7.44 (s, 1H), 7.32 (d, J = 7.2 Hz, 3H), 5.88 (s, 1H), 5.05 (s, 1H), 4.79 - 4.71 (m, 2H), 4.45-4.43 (m, 1H), 1.34-1.27 (m, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 154.2, 137.3 (d, J = 10.8 Hz), 135.7 (d, J = 2.9 Hz), 134.7, 130.0, 129.2, 125.5, 123.4,

91.7 (d, J = 9.3 Hz), 72.2(d, J = 7.5 Hz), 72.0 (d, J = 7.7 Hz), 51.7 (d, J = 160.8 Hz), 24.2 (d, J = 3.0 Hz), 24.1 (d, J = 3.8 Hz), 24.1 (d, J = 6.4 Hz), 23.9 (d, J = 5.1 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 15.9. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 5.7 min (maj) and 7.0 min. HRMS (ESI) *m/z* calculated for C₁₆H₂₃ClN₂O₄P [M+H]⁺ 373.0894, found 373.0891.

Diisopropyl (*R*)-(6-(4-ethylphenyl)-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (1i): 34 mg, 93% yield, new compound, white solid, mp: 156-157 °C, $R_f = 0.40$ (dichloromethane/

 $\begin{array}{c} & \text{methanol} = 10/1), 81\% \text{ ee}, [\alpha]^{20}{}_{\text{D}} = -34,72 \ (c \ 0.36, \text{ MeOH}). \ ^{1}\text{H NMR} \ (400) \\ & \text{MHz}, \text{CDCl}_3) \ \delta \ 7.51 \ (s, 1\text{H}), \ 7.37 \ (d, J = 6.5 \ \text{Hz}, 2\text{H}), \ 7.21 \ (d, J = 6.4 \ \text{Hz}, 2\text{H}), \ 5.69 \ (s, 1\text{H}), \ 5.03 \ (s, 1\text{H}), \ 4.75 \ (s, 2\text{H}), \ 4.45 \ (s, 1\text{H}), \ 2.64 \ (d, J = 6.7 \ \text{Hz}, 2\text{H}), \ 1.33 - 1.23 \ (m, 15\text{H}). \ ^{13}\text{C NMR} \ (100 \ \text{MHz}, \text{CDCl}_3) \ \delta \ 154.0, \ 145.6, \ 138.2 \ (d, J = 11.0 \ \text{Hz}), \ 131.4, \ 128.3, \ 125.0, \ 89.9 \ (d, J = 9.2 \ \text{Hz}), \ 7.2.2 \ (d, J = 6.4 \ \text{Hz}, 1.2 \ \text{Hz}) \ 7.2.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{Hz}) \ 7.2 \ (d, J = 6.4 \ \text{H$

= 7.7 Hz), 71.8 (d, J = 7.9 Hz), 51.7 (d, J = 160.5 Hz), 28.6, 24.3 (d, J = 2.8 Hz), 24.1 (d, J = 3.8 Hz), 23.9 (d, J = 5.2 Hz), 15.33 (s). ³¹P NMR (162 MHz, CDCl₃) δ 16.2. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 7.1 min (maj) and 7.7 min. HRMS (ESI) *m*/*z* calculated for C₁₈H₂₈N₂O₄P [M+H]⁺ 367.1784, found 367.1781.

Diisopropyl (*R*)-(6-(naphthalen-2-yl)-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (1j): 30 mg, 77% yield, new compound, white solid, mp: 156-157 °C, $R_f = 0.40$ (dichloromethane/

NH NH NH NH NH NH NH NH

methanol = 10/1), 49% ee, $[\alpha]^{20}{}_{D}$ = -74.58 (*c* 0.30, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 8.26-8.23 (m, 1H), 7.85-7.84 (m, 2H), 7.51-7.48 (m, 4H), 6.85 (s, 1H), 5.77 (s, 1H), 4.93-4.91 (m, 1H), 4.83-4.72 (m, 2H), 4.53-4.50 (m, 1H), 1.40-1.33 (m, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 153.2, 137.2 (d, *J* = 10.4 Hz), 133.7, 132.7 (d, *J* = 2.7 Hz), 130.8 (d, *J* = 1.9 Hz), 129.6,

128.4, 126.6, 126.3, 126.1 (d, J = 2.9 Hz), 125.2, 125.1, 94.2 (d, J = 9.1 Hz), 72.1 (d, J = 7.4 Hz), 71.7 (d, J = 7.7 Hz), 51.9 (d, J = 161.1 Hz), 24.3 (d, J = 3.6 Hz), 24.2, 24.1, 24.0. ³¹P NMR (162 MHz, CDCl₃) δ 16.3. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 5.9 min (maj) and 7.7 min. HRMS (ESI) *m/z* calculated for C₂₀H₂₆N₂O₄P [M+H]⁺ 389.1624, found 389.1627.

Diethyl (*R***)-(2-oxo-6-phenyl-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (1k):** 30 mg, 97% yield, new compound, white solid, mp: 157-158 °C, $R_f = 0.40$ (dichloromethane/methanol = 10/1),



90% ee, $[\alpha]^{20}_{D}$ = -35.68 (*c* 0.30, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.91 (s, 1H), 7.52-7.41 (m, 2H), 7.39-7.36 (m, 3H), 6.27 (s, 1H), 5.06 (s, 1H), 4.45-4.42 (m, 1H), 4.20-4.13 (m, 4H), 1.36-1.28 (m, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 154.5, 138.7 (d, *J* = 10.5 Hz), 133.8 (d, *J* = 3.4 Hz), 129.3, 128.8, 125.2 (d, *J* = 1.9 Hz), 90.4 (d, *J* = 8.8 Hz), 63.6 (d, *J* = 7.3 Hz), 63.0 (d, *J* = 7.5 Hz), 133.8 (d, *J* = 7.5 Hz), 133.8 (d, *J* = 7.5 Hz), 63.0 (d, *J* = 7.5 Hz), 63.0 (d, *J* = 7.5 Hz), 63.6 (d, *J* = 7.5 Hz), 63.0 (d, J = 7.5 Hz), 63

Hz), 51.0 (d, J = 158.8 Hz), 16.6 (d, J = 5.0 Hz), 16.5 (d, J = 5.3 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 18.0. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 8.2 min (maj) and 8.9 min. HRMS (ESI) *m/z* calculated for C₁₄H₂₀N₂O₄P [M+H]⁺ 311.1117, found 311.1116.

Diisopropyl (*R***)-(6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate (11):** 26 mg, 94% yield, white solid, mp: 122-123 °C, $R_f = 0.40$ (dichloromethane/methanol = 10/1), 5% ee, $\lceil \alpha \rceil^{20}$ _D

= -4.77 (*c* 0.26, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.40 (s, 1H), 5.24 (s, 1H), 4.79-4.72 (m, 2H), 4.53 (s, 1H), 4.31-4.30 (m, 1H), 1.80 (d, *J* = 4.5 Hz, 3H), 1.35-1.31 (m, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 154.3, 135.1 (d, *J* = 10.3 Hz), 88.9 (d, *J* = 8.4 Hz), 71.9 (d, *J* = 7.7 Hz), 71.6 (d, *J* = 7.8 Hz), 51.4 (d, *J* = 161.2 Hz), 24.2 (d, *J* = 3.4 Hz), 24.1 (d, *J* = 3.6 Hz), 24.0 (d, *J* = 4.6 Hz), 23.8 (d, *J* = 5.1

Hz), 18.5 (d, J = 2.5 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 16.6. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 5.2 min and 6.6 min (maj). HRMS (ESI) *m/z* calculated for C₁₁H₂₂N₂O₄P [M+H]⁺ 277.1313, found 277.1312.

Diisopropyl (R)-(5-methyl-2-oxo-6-phenyl-1,2,3,4-tetrahydropyrimidin-4-yl)phosphonate

(1m): 33 mg, 94% yield, white solid, mp: 158-159 °C, $R_f = 0.40$ (dichloromethane/methanol = 10/1), 84% ee, $[\alpha]^{20}_D = -18.21$ (*c* 0.33, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.31 (m, 5H), 6.50 (s,



 $\begin{array}{l} \text{1H}, 5.53 \ (\text{s}, 1\text{H}), 4.81\text{-}4.75 \ (\text{m}, 2\text{H}), 4.13 \ (\text{d}, J = 8.1 \ \text{Hz}, 1\text{H}), 1.82 \ (\text{s}, 3\text{H}), \\ 1.37\text{-}1.34 \ (\text{m}, 12\text{H}). \ ^{13}\text{C} \ \text{NMR} \ (100 \ \text{MHz}, \text{CDCl}_3) \ \delta \ 153.8 \ , 134.8 \ (\text{d}, J = 3.6 \ \text{Hz}), 133.2 \ (\text{d}, J = 9.8 \ \text{Hz}), 128.8 \ , 128.7, 128.4 \ (\text{d}, J = 2.8 \ \text{Hz}), 99.2 \ (\text{d}, J = 6.4 \ \text{Hz}), 72.1 \ (\text{d}, J = 7.9 \ \text{Hz}), 71.5 \ (\text{d}, J = 7.8 \ \text{Hz}), 56.3 \ (\text{d}, J = 155.6 \ \text{Hz}), 24.4 \ (\text{d}, J = 3.1 \ \text{Hz}), 24.2, 24.1, 24.0 \ (\text{d}, J = 5.0 \ \text{Hz}), 16.7 \ (\text{d}, J = 1.5 \ \text{Hz}). \ ^{31}\text{P} \ \text{NMR} \ (162 \ \text{MHz}, \text{CDCl}_3) \ \delta \ 16.9. \ \text{HPLC}: \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{nm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{mm}, 30 \ ^{\circ}\text{C}, n - 100 \ \text{Chiracel AD column}, 254 \ \text{Chiracel AD column},$

hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 5.2 min (maj) and 6.4 min. HRMS (ESI) m/z calculated for C₁₇H₂₆N₂O₄P [M+H]⁺ 353.1654, found 353.1652.

Diisopropyl (*R*)-(6-(3-methoxyphenyl)-5-methyl-2-oxo-1,2,3,4-tetrahydropyrimidin-4-yl) phosphonate (1n): 37 mg, 97% yield, new compound, white solid, mp: 153-154 °C, $R_f = 0.40$



(dichloromethane/methanol = 10/1), 80% ee, $[\alpha]^{20}{}_{D}$ = -11.62 (*c* 0.37, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.33-7.28 (m, 1H), 6.91-6.85 (m, 3H), 6.43 (s, 1H), 5.47 (s, 1H), 4.80-4.75 (m, 2H), 4.12 (d, *J* = 8.2 Hz, 1H), 3.81 (s, 3H), 1.83 (s, 3H), 1.36 (t, *J* = 6.2 Hz, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 159.7, 153.6, 136.1 (d, *J* = 3.4 Hz), 133.0 (d, *J* = 10.0 Hz), 129.8, 120.8 (d, *J* = 2.8 Hz), 114.5, 113.9 (d, *J* = 2.8 Hz), 99.3 (d, *J*

= 6.4 Hz), 72.1 (d, J = 7.9 Hz), 71.5 (d, J = 7.7 Hz), 56.3 (d, J = 155.6 Hz), 55.3, 24.4 (d, J = 3.1 Hz), 24.2, 24.1, 24.0 (d, J = 5.0 Hz), 16.8 (d, J = 1.4 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 16.9. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 5.9 min (maj) and 7.8 min. HRMS (ESI) *m/z* calculated for C₁₈H₂₈N₂O₅P [M+H]⁺ 383.1727, found 383.1729.

Diisopropyl (*R*)-(2-oxo-5,6-diphenyl-1,2,3,4-tetrahydropyrimidin-4-yl) phosphonate (10): 39 mg, 94% yield, new compound, white solid, mp: 188-189 °C, $R_f = 0.40$ (dichloromethane/



methanol = 10/1), 88% ee, $[\alpha]^{20}_{D}$ = -19.23 (*c* 0.39, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.26-7.11 (m, 10H), 6.44 (s, 1H), 5.57 (s, 1H), 4.77-4.71 (m, 1H), 4.70-4.60 (m, 2H), 1.36-1.30 (m, 6H), 1.16-1.10 (m, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 154.2, 137.0 (d, *J* = 4.7 Hz), 135.4 , 135.0 (d, *J* = 3.4 Hz), 130.3, 128.6, 128.4, 128.0, 126.7, 118.8, 105.3 (d, *J* = 4.0 Hz), 71.7 (d, *J* = 8.0 Hz),

56.0 (d, J = 157.5 Hz), 24.3 (d, J = 3.1 Hz), 24.1 (d, J = 4.4 Hz), 24.0 (d, J = 2.8 Hz), 23.6 (d, J = 4.9 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 17.4. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 5.8 min (maj) and 7.6 min. HRMS (ESI) *m*/*z* calculated for C₂₂H₂₈N₂O₄P [M+H]⁺ 415.1783, found 415.1781.

Failed example:

Under standard conditions, the redox deracemization of 4-(diphenylphosphoryl)-5-methyl- 6phenyl-3,4-dihydropyrimi-din-2(1H)-one, in which the isopropoxy group is replaced with a phenyl group, the reaction only underwent oxidation, without observation reduction products, the starting material was recovered.



4. Determination of Absolute Configuration of Products

4.1 Determination of Absolute Configuration of Products 4m

To determine the absolute configuration of 4m (93% ee), firstly, 4m was upgraded to >99% ee by recrystallization with *n*-hexane/ethyl acetate. Then, *n*-hexane was slowly added into the solution of 4m in ethyl acetate at 50 °C, then the solution was slowly cooled down to room temperature. The crystal was grown from the solution, which is suitable for X-ray diffraction analysis. The structure in **Figure S2** showed that the absolute configuration of 4m is (*R*). [CCDC 2080219] contains the structure and supplementary crystallographic data for (*R*)-4m. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc. cam.ac.uk.



Figure S2. X-ray crystallographic analysis of (*R*)-4m [CCDC 2080219]

ORTEP diagram of compound 4m, thermal ellipsoids are drawn on 30% probability level

Identification code	4m
Empirical formula	$C_{21}H_{29}N_2O_6P$
Formula weight	436.43
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	P 21
Volume	1162.84(4) Å ³
Z	2
Density (calculated)	1.246 Mg/m ³

Crystal data and structure refinement for 4m.

Absorption coefficient	0.155 mm ⁻¹	
F(000)	464	
Crystal size	$0.200 \ge 0.160 \ge 0.130 \text{ mm}^3$	
Theta range for data collection	2.832 to 26.000°.	
Goodness-of-fit on F ²	1.054	
Flack	0.02(3)	
Extinction coefficient	0.031(11)	

4.2 Determination of Absolute Configuration of Products 4a

A racemic of 3,4-Dihydropyrimidin-2-one derivatives **1a** (1.352 g, 4.0 mmol), DDQ (0.999 g, 4.4 mmol, 1.1 equiv) in ethyl acetate (50 mL) was stirred at room temperature for 10 min. After the reaction was completed (determined by TLC), chiral phosphoric acid (*S*)-**2g** (0.301 g, 0.4 mmol, 10 mol%) and Hantzsch ester **3a** (1.261 g, 5.6 mmol, 1.4 equiv) was added. The mixture was stirred at room temperature for 24 h. The resulting mixture was concentrated in *vacuo* and further purification was performed by a silica gel column eluted with dichloromethane /methanol to give the optically active products (*S*)-**1a** 1.243 g in 92% yield and 96% ee. $[\alpha]^{20}_{D} = +24.40$ (*c* 1.0, MeOH).

A solution of compound (S)-1a (1.056 g, 3 mmol) in dichloroethane (60 mL) was added DMAP (36.6 mg, 0.3 mmol), Et₃N (3.036 g, 30 mmol), Ac₂O (3.063g, 30 mmol). The mixture was stirred at room temperature for 10 h. After the reaction was completed (determined by TLC). The resulting mixture was concentrated in *vacuo* and further purification was performed by a silica gel column eluted with hexane/ethyl acetate to give the optically active products (S)-4a 1.203 g in 92% yield and 92% ee. To determine the absolute configuration of (S)-4a (96% ee), firstly, (S)-4a was upgraded to >99% ee by recrystallization with *n*-hexane/ethyl acetate. Then, *n*-hexane was slowly added into the solution of (S)-4a in ethyl acetate at 50 °C, then the solution was slowly cooled down to room temperature. The crystal was grown from the solution, which is suitable for X-ray diffraction analysis. The structure in **Figure S1** showed that the absolute configuration of 4a is (S). [CCDC 2100898] contains the structure and supplementary crystallographic data for (S)-4a. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc. cam.ac.uk.



Figure S1. X-ray Crystallographic Analysis of (S)-4a [CCDC 2100898]

ORTEP diagram of compound **4a**, thermal ellipsoids are drawn on 30% probability level Crystal data and structure refinement for **4a**.

Identification code	4a
Empirical formula	$C_{20}H_{27}N_2O_6P$
Formula weight	422.40
Temperature	190(0) K
Wavelength	1.34139 Å
Crystal system	triclinic
Space group	P 1
Volume	2756.2(4) Å ³
Z	5
Density (calculated)	1.272 Mg/m ³
Absorption coefficient	0.925 mm ⁻¹
F(000)	1120
Crystal size	0.120 x 0.100 x 0.100 mm ³
Goodness-of-fit on F ²	1.040
Flack	0.049(11)

5. Control Experiments



A compound of 2-hydroxypyrimindine **7a** (33.6 mg, 0.10 mmol, 1.0 equiv), chiral phosphoric acid (*R*)-**2g** (7.5 mg, 0.01 mmol, 10 mol%) and Hantzsch ester **3a** (31.5 mg, 0.14 mmol, 1.4 equiv) was added. The mixture was stirred at room temperature for 24 h. The resulting mixture was concentrated in *vacuo* and further purification was performed by a silica gel column chromatography eluted with dichloromethane/methanol to give the optically active product (*R*)-**1a** 31.1 mg in 92% yield and 96% ee. The enantiomeric excess was determined by chiral HPLC.



A compound of (*R*)-1a (33.8 mg, 0.10 mmol, 1.0 equiv), DDQ (25 mg, 0.11 mmol, 1.1 equiv) in ethyl acetate (4 mL) was stirred at room temperature for 5 min. After the reaction was completed (determined by TLC), chiral phosphoric acid (*S*)-2g (7.5 mg, 0.01 mmol, 10 mol%) and Hantzsch ester 3a (31.5 mg, 0.14 mmol, 1.4 equiv) was added. The mixture was stirred at room temperature for 24 h. The resulting mixture was concentrated in *vacuo* and further purification was performed by a silica gel column chromatography eluted with dichloromethane/methanol to give the optically active product (*S*)-1a 31.1 mg in 92% yield and 96% ee. The enantiomeric excess was determined by chiral HPLC.



The 4-(diisopropoxyphosphoryl)-6-phenylpyrimidin-2-yl 4-methylbenzenesulfonate **7a'** (0.10 mmol), chiral phosphoric acid (R)-**2g** (7.5 mg, 0.01 mmol, 10 mol%) and Hantzsch ester **3a** (31.5 mg, 0.14 mmol, 1.4 equiv) in ethyl acetate (4 mL) was stirred at room temperature for 24 hrs. The reaction was monitored by TLC. The reaction mixture was concentrated in *vacuo* and then tested the crude residue by ¹HNMR to find that starting material **7a'** remained >95% NMR yield.

6. Redox Deracemization of 3,4-Dihydropyrimidin-2-ones at Gram Scale



A racemic of 3,4-Dihydropyrimidin-2-one derivatives 1a (1.352 g, 4.0 mmol, 1.0 equiv), DDQ (0.999 g, 4.4 mmol, 1.1 equiv) in ethyl acetate (50 mL) was stirred at room temperature for 10 min. After the reaction was completed (determined by TLC), chiral phosphoric acid (*R*)-**2g** (0.301 g, 0.4 mmol, 10 mol%) and Hantzsch ester **3a** (1.261 g, 5.6 mmol, 1.4 equiv) was added. The mixture was stirred at room temperature for 24 h. The resulting mixture was concentrated in *vacuo* and further purification was performed by a silica gel column eluted with dichloromethane/methanol to give the optically active products (*R*)-(-)-**1a** 1.243 g in 92% yield and 96% ee. The enantiomeric excesses were determined by Chiral HPLC.

7. Synthesis of the Optically Active Products 5m and 6m

7.1 Synthesis DHPMs Derivative 4m



A solution of compound (*R*)-1m (0.352 g, 1 mmol) in dichloroethane (20 mL) was added DMAP (12.2 mg, 0.1 mmol), Et₃N (1.012 g, 10 mmol), Ac₂O (1.021g, 10 mmol). The mixture was stirred at room temperature for 10 h. After the reaction was completed (determined by TLC). The resulting mixture was concentrated in *vacuo* and further purification was performed by a silica gel column eluted with hexane/ethyl acetate to give the optically active products (*R*)-4m 0.401 g in 92% yield and 92% ee. The enantiomeric excesses were determined by chiral HPLC.

Diisopropyl (*R*)-(1,3-diacetyl-5-methyl-2-oxo-6-phenyl-1,2,3,4-tetrahydropyrimidin-4-yl) phosphonate (4m): 401 mg, 92% yield, new compound, white solid, mp: 103-104 °C, $R_f = 0.30$



(dichloromethane/methanol = 50/1), 92% ee, $[\alpha]^{20}_{D}$ = +36.30 (*c* 0.98, MeOH). ¹H NMR (400 MHz, DMSO-d₆) δ 7.37 (t, *J* = 7.4 Hz, 2H), 7.28 (t, *J* = 8.1 Hz, 3H), 5.26 (d, *J* = 20.0 Hz, 1H), 4.68-4.58 (m, 2H), 2.55 (s, 3H), 2.29 (s, 3H), 1.89 (d, *J* = 2.2 Hz, 3H), 1.30-1.20 (m, 12H). ¹³C NMR (100 MHz, DMSO-d₆) δ 170.9 (d, *J* = 1.6 Hz), 169.6 (d, *J* = 2.0 Hz), 153.3, 136.0 (d, *J* = 3.3 Hz), 135.5

(d, J = 9.0 Hz), 128.4, 128.2, 127.9, 120.7 (d, J = 1.4 Hz), 72.4 (d, J = 7.3 Hz), 71.9 (d, J = 7.3 Hz), 52.9 (d, J = 155.9 Hz), 26.4, 26.1, 24.3 (d, J = 3.5 Hz), 24.1 (d, J = 4.0 Hz), 23.9 (d, J = 3.0 Hz), 23.8 (d, J = 2.0 Hz), 18.0 (d, J = 4.9 Hz). ³¹P NMR (162 MHz, DMSO-d₆) δ 16.7. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 4.1 min (maj) and 4.8 min. HRMS (ESI) m/z Calculated for C₂₁H₃₀N₂O₆P [M+H]⁺ 437.1836, found 437.1837.

7.2 Synthesis DHPMs Derivative 5m



A solution of compound (R)-4m (43.6 mg, 0.10 mmol) in methanol (2.0 mL) was added sodium borohydride (15.1 mg, 0.40 mmol). The resulting solution was stirred at room temperature for 0.5 h. After the reaction was completed (determined by TLC). Methanol was removed in *vacuo* and the residue was diluted with water. The aqueous mixture was extracted with dichloromethane (10 mL×3). The combined organic layer was washed twice with brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure to give viscous oil. The crude product was purified by flash column chromatography using dichloromethane/methanol as eluent to give the desired products (+)-**5m** 38.9 mg in 98% yield and 92% ee.

Diisopropyl (*R*)-(1-acetyl-5-methyl-2-oxo-6-phenyl-1,2,3,4-tetrahydropyrimidin-4-yl) phosphonate (5m): 38.9 mg, 98% yield, new compound, white solid, mp: 181-182 °C, $R_f = 0.30$

(hexanes/ethyl acetate = 20/1), 92% ee, $[\alpha]^{20}_{D}$ = +86.73 (*c* 1.74, MeOH). ¹H NMR (400 MHz, DMSO-d₆) δ 8.64 (d, *J* = 5.3 Hz, 1H), 7.34 (t, *J* = 7.4 Hz, 2H), 7.24 (t, *J* = 7.2 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 2H), 4.67-4.62 (m, 2H), 4.01-3.96 (m, 1H), 2.21 (s, 3H), 1.85 (d, *J* = 2.1 Hz, 3H), 1.32-1.25 (m, 12H). ¹³C NMR (100 MHz, DMSO-d₆) δ 168.4 (d, *J* = 2.4 Hz), 154.7, 137.1 (d, *J* = 3.5 Hz),

135.7 (d, J = 9.4 Hz), 128.3, 128.0 (d, J = 1.2 Hz), 127.5, 120.0 (d, J = 1.2 Hz), 71.5 (d, J = 7.4 Hz), 71.3 (d, J = 7.5 Hz), 54.0 (d, J = 153.6 Hz), 25.7, 24.2 (d, J = 3.7 Hz), 24.1, 24.1 (d, J = 1.5 Hz), 24.0 (d, J = 4.3 Hz), 17.7 (d, J = 4.2 Hz). ³¹P NMR (162 MHz, DMSO-d₆) δ 18.2. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 90/10, flow = 1.0 mL/min, retention time 16.0 min (maj) and 18.0 min. HRMS (ESI) m/z Calculated for C₁₉H₂₈N₂O₅P [M+H]⁺ 395.1729, found 395.1728.

7.3 Synthesis DHPMs Derivative 6m



A mixture of compound (*R*)-1m (70.4 mg, 0.20 mmol) in ethyl acetate (5.0 mL) was added Pd/C (47.3 mg, 0.01mmol, 5 mol%) in a Schlenk tube. The Schlenk tube was carefully and quickly vacuum purged before being filled with hydrogen using an hydrogen balloon. The reaction mixture was then stirred at 50 °C until the the reaction was completed (determined by TLC). Ethyl acetate was removed in vacuo and further purification was performed by a silica gel column eluted with dichloromethane /methanol to give the optically active products (+)-6m 67.3 mg in 95% yield and 92% ee. The enantiomeric excesses were determined by chiral HPLC.

Diisopropyl ((*R***)-5-methyl-2-oxo-6-phenylhexahydropyrimidin-4-yl)phosphonate (6m):** 67.3 mg, 95% yield, new compound, colorless liquid, $R_f = 0.30$ (methanol/dichloromethane = 20/1), 92% ee, $[\alpha]^{20}_D = +73.11$ (*c* 0.70, MeOH). ¹H NMR (400 MHz, CDCl₃) δ 7.38 $P(O^{i}Pr)_2$ (t, *J* = 7.3 Hz, 2H), 7.33-7.26 (m, 3H), 5.27 (s, 1H), 5.00 (d, *J* = 7.9 Hz, 1H), Me NH 4.82-4.72 (m, 3H), 4.04 (dd, *J* = 14.2, 2.8 Hz, 1H), 2.42 (d, *J* = 3.3 Hz, 1H),

156.3 (d, J = 11.9 Hz), 138.9 (d, J = 3.7 Hz), 128.7 (s), 128.0 (s), 126.3 (s), 71.9 (d, J = 6.8 Hz), 71.6 (d, J = 7.5 Hz), 60.2 (d, J = 18.8 Hz), 53.6 (d, J = 159.1 Hz), 32.9 (d, J = 4.4 Hz), 53.6 (d, J = 159.1 Hz), 32.9 (d, J = 4.4 Hz), 53.6 (d, J = 159.1 Hz), 32.9 (d, J = 4.4 Hz), 53.6 (d, J = 159.1 Hz), 32.9 (d, J = 4.4 Hz), 53.6 (d, J = 159.1 Hz), 32.9 (d, J = 4.4 Hz), 53.6 (d, J = 159.1 Hz), 53.6 (d, J

1.38-1.34 (m, 12H), 0.89 (d, J = 6.9 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ

Hz), 24.2 (d, J = 3.6 Hz), 24.1 (d, J = 5.0 Hz), 24.0 (d, J = 3.6 Hz), 23.9 (d, J = 5.7 Hz), 7.6 (d, J = 1.9 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 18.5. HPLC: Chiracel AD column, 254 nm, 30 °C, *n*-hexane/*i*-propanol = 80/20, flow = 1.0 mL/min, retention time 7.9 min and 10.7 min (maj). HRMS (ESI) m/z Calculated for C₁₇H₂₈N₂O₄P [M+H]⁺ 355.1787, found 355.1788.

8. References

- 1. G.-S. Feng, M.-W. Chen, L. Shi and Y.-G. Zhou, Angew. Chem. Int. Ed., 2018, 57, 5853.
- 2. K.-R. Li, F.-J. Meng, W.-F. Jiang and L. Shi, *Tetrahedron Lett.* 2021, 73, 153149.

9. Copy of NMR, HPLC for Compounds









S18













13C NMR FM-5-34C in CDCl3



















31P NMR FM-5-34F in CDCI3

S35


-17.37

31P NMR FM-5-34G in DMSO







-15.90







31P NMR FM-5-34I in CDCI3

-16.16





13C NMR FM-5-34J in CDCI3









31P NMR FM-5-34J in CDCl3

-16.33



1H NMR FM-5-34K in CDCI3







-18.01



S49





S51















1H NMR FM-5-34N in CDCl3







-16.90



1H NMR FM-5-340 in CDCl3



10 ¹H NMR (400 MHz, CDCl₃)







31P NMR FM-5-340 in CDCI3

-17.35











1H NMR FM-5-66 in DMSO







-18.15











S69

Data file:	FM-5-34-1(+)-V-2020-10-28 17-31-31+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-34-1(+/-)	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2020-10-28 17:35:45+08:00	
Acq. method:	FM-4-41.amx	Туре:	Sample	
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



Signal:	VWD1A,Wavelength=2	254 nm			
RT [min]	Peak Width Base	Area	Height	Area%	
6.302	0.799	1045.78	80.34	50.15	
7.173	1.117	1039.68	70.97	49,85	
	Sum	2085.47			



O ⊨ P(OⁱPr)₂

'NH

Data file:	FM-5-34-1-V-2020-10-27 15-23-03+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-34-1	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2020-10-27 15:24:09+08:00	
Acq. method:	FM-4-41.amx	Туре:	Sample	
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	H(O [/] Pr) ₂
6.327	1.143	579,55	43,22	98.18	(1 /2
7,235	0.200	10,76	0,87	1.82	NH NH
	Sum	590.31			N HO
					(-)- 1 a

0
P(O [/] Pr) ₂
NH
N [×] O
L H
(+/) 1a
(1/-)-10

Data file:	FM-6-15-V-2021-04-24 09-30-51+08-00.dx		
Sequence Name:	SingleSample	Project Name:	1260
Sample name:	FM-6-15	Operator:	SYSTEM
Instrument:	Ic1260	Injection date:	2021-04-24 09:31:57+08:00
Acq. method:	FM-4-41.amx	Туре:	Sample
Processing method:	GC_LC Area Percent_DefaultMethod.pmx		
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm		



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr) ₂
6.595	0.176	14.14	1.29	2.10	
7.475	1.523	658.18	45.03	97.90	ŃH
	Sum	672.31			N HO
					(+)- 1a
Data file:	FM-5-34-2(+)-V-2020-10-28 16-18-09+08-00.dx				
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Sequence Name:	SingleSample	Project Name:	1260		
Sample name:	FM-5-34-2(+/-)	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2020-10-28 16:19:47+08:00		
Acq. method:	FM-4-41.amx	Туре:	Sample		
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				



VWD1A,Wavelength=2	54 nm			0
Peak Width Base	Area	Height	Area%	
1.004	1516.86	113.11	49.99	1 (011)2
1,260	1517,43	97.54	50.01	NH
Sum	3034.28			Me
	VWD1A,Wavelength=2 Peak Width Base 1.004 1.260 Sum	WD1A,Wavelength=254 nm Peak Width Base Area 1,004 1516,86 1,260 1517,43 Sum 3034,28	VWD1A,Wavelength=254 nm Peak Width Base Area Height 1,004 1516,86 113,11 1,260 1517,43 97,54 Sum 3034,28	VWD1A.Wavelength=254 nm Area Height Area% Peak Width Base Area Height Area% 1,004 1516.86 113,11 49,99 1,260 1517,43 97,54 50,01 Sum 3034.28 50,01 50,01



Data file:	FM-5-34-2-V-2020-10-28 16-0	FM-5-34-2-V-2020-10-28 16-05-23+08-00.dx				
Sequence Name:	SingleSample	Project Name:	1260			
Sample name:	FM-5-34-2	Operator:	SYSTEM			
Instrument:	lc1260	Injection date:	2020-10-28 16:05:42+08:00			
Acq. method:	FM-4-41.amx	FM-4-41.amx Type: Sample				
Processing method:	GC_LC Area Percent_Default	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 80/20,	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr) ₂
6.907	1.193	538.35	40.21	97.14	(2.1.)2
7.833	0.204	15.83	1.24	2.86	NH NH
	Sum	554.18			Ma
					(-)- 1b

Data file:	FM-5-34-3(+)-V-2020-10-28 17-01-24+08-00.dx				
Sequence Name:	SingleSample Project Name: 1260				
Sample name:	FM-5-34-3(+/-)	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2020-10-28 17:01:36+08:00		
Acq. method:	FM-4-41.amx Type: Sample				
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				



Signal:	VWD1A,Wavelength=2	254 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O ⁱ Pr)
5.277	0.656	1230.58	117.85	49.45	1(011)2
6.323	0.953	1257.85	99.45	50.55	NH
	Sum	2488.43			Me N H



Data file:	FM-5-34-3-V-2020-10-28 16-31-24+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-34-3	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2020-10-28 16:45:37+08:00	
Acq. method:	FM-4-41.amx Type: Sample			
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



Signal:	VWD1A,Wavelength=2	54 nm			Q
RT [min]	Peak Width Base	Area	Height	Area%	P(O ⁱ Pr) ₂
5.268	0.160	823.65	78.76	96.03	
6.307	0.199	34.07	2.68	3.97	<u>і</u> ŅН
	Sum	857.72			Me N O
					(-)-1c

Data file:	FM-5-34-10(+)-V-2020-10-31 10-05-18+08-00.dx				
Sequence Name:	SingleSample Project Name: 1260				
Sample name:	FM-5-34-10(+/-)	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2020-10-31 10:06:47+08:00		
Acq. method:	FM-4-41.amx	Туре:	Sample		
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				



Signal:	VWD1A,Wavelength=2	254 nm			
RT [min]	Peak Width Base	Area	Height	Area%	
5.858	0,973	546.78	47.18	50.32	
6.876	1,050	539.80	38.46	49.68	
	Sum	1086.58			Me Me



O ₽(OⁱPr)₂

NH

Data file:	FM-5-34-10-V-2020-10-31 09-50-52+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-34-10	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2020-10-31 09:53:08+08:00	
Acq. method:	FM-4-41.amx Type: Sample			
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr) ₂
5,869	0.874	886,25	74.86	95.19	(0.1.)2
6,910	0,205	44.79	3.40	4.81	NH NH
	Sum	931.04			
					(-)-1d



Data file:	FM-5-34-11(+)-V-2020-10-31 11-07	-10+08-00.dx	
Sequence Name:	SingleSample	Project Name:	1260
Sample name:	FM-5-34-11(+/-)	Operator:	SYSTEM
Instrument:	lc1260	Injection date:	2020-10-31 11:07:20+08:00
Acq. method:	FM-4-41.amx	Туре:	Sample
Processing method:	GC_LC Area Percent_DefaultMethod	.pmx	
Sample Info:	OD, Hexane/i-PrOH = 80/20, 1.0 mL	min, 30 oC, 254 nm	



Signal:	VWD1A,Wavelength=2	254 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	
7.092	1.196	1264,80	71.19	49.52	
7.807	1,508	1289,42	63,40	50.48	NH
	Sum	2554.22			Pr H O



Data file:	FM-5-34-11-V-2020-10-31	11-18-49+08-00.dx	
Sequence Name:	SingleSample	Project Name:	1260
Sample name:	FM-5-34-11	Operator:	SYSTEM
Instrument:	lc1260	Injection date:	2020-10-31 11:19:59+08:00
Acq. method:	FM-4-41.amx	Туре:	Sample
Processing method:	GC_LC Area Percent_Defa	aultMethod.pmx	
Sample Info:	OD, Hexane/i-PrOH = 80/2	20, 1.0 mL/min, 30 oC, 254 nm	



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr)
6.998	1.044	804.31	46.18	97.04	1
7.708	0.289	24.54	1,28	2,96	I NH
	Sum	828.85			
					(-)-1e

Data file:	FM-5-34-12(+)-V-2020-11-01 20-42	-35+08-00.dx	
Sequence Name:	SingleSample	Project Name:	1260
Sample name:	FM-5-34-12(+/-)	Operator:	SYSTEM
Instrument:	lc1260	Injection date:	2020-11-01 20:46:07+08:00
Acq. method:	FM-4-41.amx	Туре:	Sample
Processing method:	GC_LC Area Percent_DefaultMethod	.pmx	
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/r	min, 30 oC, 254 nm	



VWD1A,Wavelength=2	254 nm			0
Peak Width Base	Area	Height	Area%	
1.134	1364.83	100.31	50.62	
2,416	1331.25	51.68	49.38	NH
Sum	2696.08			
	VWD1A,Wavelength=2 Peak Width Base 1.134 2.416 Sum	VWD1A,Wavelength=254 nm Peak Width Base Area 1,134 1364.83 2,416 1331,25 Sum 2696.08	VWD1A,Wavelength=254 nm Peak Width Base Area Height 1,134 1364,83 100.31 2,416 1331,25 51,68 Sum 2696,08	VWD14,Wavelength=254 nm Peak Width Base Area Height Area% 1,134 1364,83 100,31 50,62 2,416 1331,25 51,68 49,38 Sum 2696.08

п	
(+/-)- 1f	

Data file:	FM-5-34-12-V-2020-11-01	21-08-33+08-00.dx	
Sequence Name:	SingleSample	Project Name:	1260
Sample name:	FM-5-34-12	Operator:	SYSTEM
Instrument:	lc1260	Injection date:	2020-11-01 21:32:51+08:00
Acq. method:	FM-4-41.amx	Туре:	Sample
Processing method:	GC_LC Area Percent_Defa	ultMethod.pmx	
Sample Info:	AD, Hexane/i-PrOH = 80/20	0, 1.0 mL/min, 30 oC, 254 nm	



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	H(O'Pr)
6.333	0.210	30.03	2,22	2.51	(* /2
7.148	0.376	1166,27	45.74	97.49	NH NH
	Sum	1196.30			N O
					ъи • (-)- 1f

Data file:	FM-5-34-6(+)-V-2020-10-30 10-02-2	2+08-00.dx	
Sequence Name:	SingleSample	Project Name:	1260
Sample name:	FM-5-34-6(+/-)	Operator:	SYSTEM
Instrument:	lc1260	Injection date:	2020-10-30 10:03:49+08:00
Acq. method:	FM-4-41.amx	Туре:	Sample
Processing method:	GC_LC Area Percent_DefaultMethod.	pmx	
Sample Info:	OD, Hexane/i-PrOH = 80/20, 1.0 mL/	min, 30 oC, 254 nm	



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	
6.234	1.027	273.60	18,35	50.57	F(0F1)2
7.289	1,000	267.38	15,58	49.43	NH
	Sum	540.98			CI NO

Data file:	FM-5-34-6-V-2020-10-30 10	FM-5-34-6-V-2020-10-30 10-27-24+08-00.dx					
Sequence Name:	SingleSample	SingleSample Project Name: 1260					
Sample name:	FM-5-34-6	Operator:	SYSTEM				
Instrument:	lc1260	Injection date:	2020-10-30 10:28:04+08:00				
Acq. method:	FM-4-41.amx Type: Sample						
Processing method:	GC_LC Area Percent_DefaultMethod.pmx						
Sample Info:	OD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm						



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	H(O [/] Pr) ₂
6.348	0.203	7.74	0.59	2.45	()2
7.419	1.410	308.38	17,55	97.55	NH NH
	Sum	316.12			
					(-)- 1g

Data file:	FM-5-34-7(+)-V-2020-10-30 11-08-55+08-00.dx					
Sequence Name:	SingleSample Project Name: 1260					
Sample name:	FM-5-34-7(+/-)	Operator:	SYSTEM			
Instrument:	lc1260	Injection date:	2020-10-30 11:09:43+08:00			
Acq. method:	FM-4-41.amx	Туре:	Sample			
Processing method:	GC_LC Area Percent_DefaultMethod.pmx					
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm					



Signal:	VWD1A,Wavelength=2	254 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	
5,684	1,273	519.72	45.55	50.04	(011)2
7.020	1.043	518,98	36,28	49.96	NH
	Sum	1038.70			



Data file:	FM-5-34-7-V-2020-10-30 11-20-48+08-00.dx						
Sequence Name:	SingleSample	SingleSample Project Name: 1260					
Sample name:	FM-5-34-7	Operator:	SYSTEM				
Instrument:	lc1260	Injection date:	2020-10-30 11:21:09+08:00				
Acq. method:	FM-4-41.amx Type: Sample						
Processing method:	GC_LC Area Percent_DefaultMethod.pmx						
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm						



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr)
5.671	1.220	430.29	38.07	96.54	(,2
6.964	0.259	15.43	0.93	3.46	NH NH
	Sum	445.72			
					(-)- 1h



Data file:	FM-5-34-9(+)-V-2020-10-30 18-52-33+08-00.dx					
Sequence Name:	SingleSample Project Name: 1260					
Sample name:	FM-5-34-9(+/-)	Operator:	SYSTEM			
Instrument:	lc1260	Injection date:	2020-10-30 18:54:39+08:00			
Acq. method:	FM-4-41.amx	Туре:	Sample			
Processing method:	GC_LC Area Percent_DefaultMethod.pmx					
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm					



VWD1A,Wavelength=2	54 nm			0
Peak Width Base	Area	Height	Area%	
0,224	444.15	30,55	49.95	
0.244	445.12	28,29	50.05	NH
Sum	889.27			Et NHO
	VWD1A,Wavelength=2 Peak Width Base 0,224 0,244 Sum	VWD1A,Wavelength=254 nm Peak Width Base Area 0.224 444,15 0.244 445,12 Sum 889,27	VWD1A,Wavelength=254 nm Peak Width Base Area Height 0.224 444,15 30,55 0.244 445,12 28,29 Sum 889,27	WUD1A,Wavelength=254 nm Peak Width Base Area Height Area% 0.224 444.15 30.55 49.95 0.244 445.12 28.29 50.05 Sum 889.27

Data file:	FM-5-34-9-V-2020-11-01 20-26-13+08-00.dx						
Sequence Name:	SingleSample	SingleSample Project Name: 1260					
Sample name:	FM-5-34-9	Operator:	SYSTEM				
Instrument:	lc1260	Injection date:	2020-11-01 20:27:45+08:00				
Acq. method:	FM-4-41.amx Type: Sample						
Processing method:	GC_LC Area Percent_DefaultMethod.pmx						
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm						



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr) ₂
7.017	0.844	503.17	33.70	90.38	I (01.7)2
7.627	0.560	53.57	3.45	9.62	NH NH
	Sum	556.74			
					(-)- 1 i

Data file:	FM-5-34-8(+)-V-2020-10-30 19-43-05+08-00.dx					
Sequence Name:	SingleSample Project Name: 1260					
Sample name:	FM-5-34-8(+/-)	Operator:	SYSTEM			
Instrument:	lc1260	Injection date:	2020-10-30 19:43:19+08:00			
Acq. method:	FM-4-41.amx	Туре:	Sample			
Processing method:	GC_LC Area Percent_DefaultMethod.pmx					
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm					



Signal:	VWD1A,Wavelength=2	254 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	
6.147	1.385	538.38	43.11	49.78	
8.059	1,800	543.24	30.90	50.22	NH
	Sum	1081.62			N ^L O



Data file:	FM-5-34-8-V-2020-10-30 1	FM-5-34-8-V-2020-10-30 17-31-47+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260		
Sample name:	FM-5-34-8	M-5-34-8 Operator:			
Instrument:	lc1260	Injection date:	2020-10-30 17:32:01+08:00		
Acq. method:	FM-4-41.amx	FM-4-41.amx Type: Sample			
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr) ₂
5.904	1,596	689,70	55,55	74.45	()/2
7,713	1,172	236,67	12,55	25,55	NH NH
	Sum	926.37			Ϋ́ς Ϋ́ς Ϋ́ς Ϋ́ς Ϋ́ς Ϋ́ς Ϋ́ς Ϋ́ς
					(-)- 1j



Data file:	FM-5-34-16(+)-V-2020-11-03 10-11-19+08-00.dx					
Sequence Name:	SingleSample Project Name: 1260					
Sample name:	FM-5-34-16(+/-)	Operator:	SYSTEM			
Instrument:	lc1260	Injection date:	2020-11-03 10:13:52+08:00			
Acq. method:	FM-4-41.amx Type: Sample					
Processing method:	GC_LC Area Percent_DefaultMethod.pmx					
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm					



Signal:	VWD1A,Wavelength=2	254 nm			
RT [min]	Peak Width Base	Area	Height	Area%	
8,185	0.839	807.43	45,02	49.75	
8,926	1.017	815.62	43,59	50,25	r
	Sum	1623.06			



O P(OEt)₂

NH

Data file:	FM-5-34-16-V-2020-11-03 11-19-14+08-00.dx				
Sequence Name:	SingleSample	Project Name:	1260		
Sample name:	FM-5-34-16	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2020-11-03 11:20:28+08:00		
Acq. method:	FM-4-41.amx Type: Sample				
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(OEt) ₂
8,172	0.881	290,13	16,91	95.04	(,2
8,892	0.261	15,13	0,88	4.96	<u>∭_NH</u>
	Sum	305.26			₩ ^L N ^L O
					(-)- 1k

Data file:	FM-5-34-17(+)-V-2020-11-04 10-27-28+08-00.dx					
Sequence Name:	SingleSample Project Name: 1260					
Sample name:	FM-5-34-17(+/-)	Operator:	SYSTEM			
Instrument:	lc1260	Injection date:	2020-11-04 10:28:14+08:00			
Acq. method:	FM-4-41.amx Type: Sample					
Processing method:	GC_LC Area Percent_DefaultMethod.pmx					
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm					







Data file:	FM-5-34-17-V-2020-11-04 10-42-23+08-00.dx				
Sequence Name:	SingleSample	Project Name:	1260		
Sample name:	FM-5-34-17	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2020-11-04 10:42:32+08:00		
Acq. method:	FM-4-41.amx Type: Sample				
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O ⁱ Pr) ₂
5,231	0.795	281.72	23.70	47.65	(* ,2
6,637	1,123	309.52	22,46	52.35	<u>∭</u> NH
	Sum	591.24			Me N O



Data file:	FM-5-34-14(+)-V-2020-11-02 17-01-18+08-00.dx					
Sequence Name:	SingleSample Project Name: 1260					
Sample name:	FM-5-34-14(+/-)	Operator:	SYSTEM			
Instrument:	Ic1260 Injection date: 2020-11-02 17:01:29+					
Acq. method:	FM-4-41.amx	Туре:	Sample			
Processing method:	GC_LC Area Percent_DefaultMethod.pmx					
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm					



Signal:	VWD1A,Wavelength=2	:54 nm			
RT [min]	Peak Width Base	Area	Height	Area%	
5,215	1,483	1089.61	100.35	50.67	
6.421	0.926	1060.73	79.57	49.33	
	Sum	2150.34			Me
					\bigcirc



0

P(O'Pr)2

Signal:	VWD1A,Wavelength=2	54 nm		
RT [min]	Peak Width Base	Area	Height	Area%
5,209	1,502	784.30	70.70	91.91
6.453	0.767	69.02	5,15	8.09
	Sum	853.33		

VWD1A,Wavelength=254 nm

x10²

3.4-3.2-3-2.8-2.6-0 2.4-2.2-2-1.8-1.6-1.4-1.2-

0.8-



Data file: FM-5-34-14-V-2020-11-02 17-19-03+08-00.dx SingleSample 1260 Sequence Name: Project Name: Sample name: FM-5-34-14 Operator: SYSTEM lc1260 2020-11-02 17:21:09+08:00 Instrument: Injection date: FM-4-41.amx Acq. method: Type: Sample GC_LC Area Percent_DefaultMethod.pmx Processing method: Sample Info: AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm

S83

Data file:	FM-5-34-15(+)-V-2020-11-03 09-39-02+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-34-15(+/-)	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2020-11-03 09:45:25+08:00	
Acq. method:	FM-4-41.amx	Туре:	Sample	
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			







Data file:	FM-5-34-15-V-2020-11-03 09-56-40+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-34-15	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2020-11-03 09:58:07+08:00	
Acq. method:	FM-4-41.amx	Туре:	Sample	
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



Signal:	VWD1A,Wavelength=2	54 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr) ₂
5.916	0.848	723.74	55.87	90.08	
7.796	0.252	79.69	4.89	9.92	Me
	Sum	803.43			MeO NHO
					(-)- 1n

Data file:	FM-5-34-13(+)-V-2020-11-02 15-58-37+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-34-13(+/-)	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2020-11-02 15:58:47+08:00	
Acq. method:	FM-4-41.amx	Туре:	Sample	
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



Signal:	VWD1A,Wavelength=2	54 nm			
RT [min]	Peak Width Base	Area	Height	Area%	~
5.869	1.062	714.51	62.72	50.02	
7.605	1,315	714.03	47.62	49.98	
	Sum	1428.54			~
					<u> </u>



O P(OⁱPr)₂

Data file:	FM-5-34-13-V-2020-11-02 16-14-13+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-34-13	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2020-11-02 16:16:27+08:00	
Acq. method:	FM-4-41.amx	Туре:	Sample	
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



ignal:	VWD1A,Wavelength=2	54 nm			
RT [min]	Peak Width Base	Area	Height	Area%	
5.702	1.813	789.50	61.78	93.89	
7.448	0.272	51.40	2.81	6.11	
	Sum	840.89			~



Data file:	FM-5-56-V-2021-01-20 20-56-01+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-56	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2021-01-20 20:56:16+08:00	
Acq. method:	FM-4-41.amx	Туре:	Sample	
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



Signal:	VWD1A,Wavelength=2	254 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	
4.083	0.654	730,62	98,50	49.80	I (011)2
4.887	0.939	736.47	80,84	50.20	Me
	Sum	1467.09			
					Ac O



Data file:	FM-5-57-V-2021-01-22 10-22-31+08-00.dx			
Sequence Name:	SingleSample	Project Name:	1260	
Sample name:	FM-5-57	Operator:	SYSTEM	
Instrument:	lc1260	Injection date:	2021-01-22 10:37:17+08:00	
Acq. method:	FM-4-41.amx	Туре:	Sample	
Processing method:	GC_LC Area Percent_DefaultMethod.pmx			
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm			



Signal:	VWD1A,Wavelength=2	254 nm			0
RT [min]	Peak Width Base	Area	Height	Area%	P(O [/] Pr) ₂
4.069	0.108	5491.10	770.44	96.17	
4.847	0.127	218.76	26.54	3.83	Me
	Sum	5709.86			



Data file:	FM-5-65(+)-V-2021-02-02 10-35-10+08-00.dx				
Sequence Name:	SingleSample Project Name: 1260				
Sample name:	FM-5-65(+/-)	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2021-02-02 10:40:26+08:00		
Acq. method:	FM-5-65.amx Type: Sample				
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm				



Signal:	VWD1A,Wavelength=2	254 nm			
RT [min]	Peak Width Base	Area	Height	Area%	
16.440	2,225	1794.88	48.41	50.06	
18,528	2,693	1790.30	42,31	49.94	Me
	Sum	3585.18			



Data file:	FM-5-66-V-2021-02-02 11-03-54+08-00.dx				
Sequence Name:	SingleSample	Project Name:	1260		
Sample name:	FM-5-66	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2021-02-02 11:04:21+08:00		
Acq. method:	FM-5-65.amx Type:		Sample		
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 90/10, 1.0 mL/min, 30 oC, 254 nm				



			54 nm	VWD1A,Wavelength=28	Signal:
	Area%	Height	Area	Peak Width Base	RT [min]
	95.91	21.63	740.92	0.528	16.007
M	4.09	0.88	31.58	0.470	18.013
~			772.50	Sum	



0 P(OⁱPr)₂

Data file:	FM-6-11-V-2021-04-13 20-03-02+08-00.dx				
Sequence Name:	SingleSample	Project Name:	1260		
Sample name:	FM-6-11	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2021-04-13 20:03:41+08:00		
Acq. method:	FM-6-10.amx	Type:	Sample		
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD. Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				







Data file:	FM-6-11-V-2021-04-13 20-56-45+08-00.dx				
Sequence Name:	SingleSample	Project Name:	1260		
Sample name:	FM-6-11	Operator:	SYSTEM		
Instrument:	lc1260	Injection date:	2021-04-13 20:59:20+08:00		
Acq. method:	FM-6-10.amx Type:		Sample		
Processing method:	GC_LC Area Percent_DefaultMethod.pmx				
Sample Info:	AD, Hexane/i-PrOH = 80/20, 1.0 mL/min, 30 oC, 254 nm				



Signal:	VWD1A,Wavelength=2	54 nm			
RT [min]	Peak Width Base	Area	Height	Area%	
7.356	0.250	20,15	1,28	3.96	
9,878	1,793	488.73	21.00	96.04	
	Sum	508.88			



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