Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry. This journal is © The Royal Society of Chemistry 2023

Supporting Information

Copper(II)-catalyzed cascade Csp²–P/C–C bond formation to

construct benzo[d]thiazol-2-ylphosphonates

Han Wang, Le Huang, Jun Li, Wenyan Hao*

College of Chemistry and Chemical Engineering, Jiangxi Normal University, Nanchang 330022, People's Republic of China. E-mail: wenyanhao@jxnu.edu.cn

Table of contents

1. Experimental Section	S1
2. ¹ H and ¹³ C data of the compound 1n	
3. ¹ H, ¹³ C and ³¹ P data of the compounds 3	
4. References	
5. Copies of ¹ H, ¹³ C, and ³¹ P NMR Spectra	

1. Experimental Section

1.1. General Information

All reagents, ligands, and metal catalysts were purchased from commercial suppliers without further purification, and commercially available solvents were purified before use. All nuclear magnetic resonance (NMR) spectra were recorded on Bruker ADVANCE 400 spectrometer. (¹H NMR at 400 MHz and ¹³C NMR at 100 MHz). NMR spectra are reported in parts per million (ppm) from internal tetramethylsilane on the δ scale. Thin layer chromatography plates were visualized by exposure to ultraviolet light with 254 nm and 365 nm of wavelength. HPTLC Silica gel 60 GF254 were used for thin layer chromatography (TLC) and column chromatography was performed in 300-400 mesh silica gels.

1.2. General procedure for the synthesis of 1



Add **5** (1.0 equiv.) and CSCl_2 (1.5 equiv.) in DCM/H₂O (30mL, 3:7) for 3h, at room temperature. After completion of reaction as indicated by TLC, the mixture was washed by DCM, then the liquid was separated, and the organic layer was combined. Then the organic layer was dried by anhydrous Na₂SO₄ and concentrated in vacuo. The crude product was purified by column chromatography on 300-400 mesh silica gels (ethyl acetate/petroleum ether, 1:20) to get desired product **1**.¹

1.3. General procedure for the synthesis of 1n



In a 150 mL round bottom flask equipped with a magnetic stirring bar, 4(685mg, 5.0mmol) and NaHCO₃ (1.26g, 15.0mmol) was added. Then, DCM/H₂O (33mL, 2:1)

was added into the reaction. Nitrogen was bubbled through the mixture for 15 min while stirring. Then, Iodine (1.27g, 5mmol) was transferred to the reaction vessel and the mixture was stirred under nitrogen gas for a night. When the reaction completed (indicated by TLC), the mixture was separated and the aqueous phase was extracted with DCM (50 mL). The combined organic layers were washed with Na₂S₂O₃ (25 mL, aq./H₂O, 1:3), and dried over anhydrous Na₂SO₄, filtered and concentrated. The mixture was purified by flash column chromatography (EtOAc/petroleum ether, 1:3) to obtain a brown solid **5n** (986mg, 75%yield).²

A mixture of **5n** (788mg, 3.0mmol), and $CSCl_2(517mg, 4.5mmol)$, DCM/H₂O (30mL, 3:7) was stirred at room temperature for 3h. Then, the crude was extracted with DCM, and dried over anhydrous Na₂SO₄ and concentrated in vacuo. The crude product was purified by column chromatography on 300-400 mesh silica gels (ethyl acetate/petroleum ether, 1:20) to get **1n**.¹

1.4 General procedure for the synthesis of 3



In a schlenk sealed tube equipped with a stirrer, **1** (0.20 mmol, 1.0 equiv.) and **2** (0.4mmol, 2.0 equiv.), $Cu(OTf)_2(20 \text{ mol}\%)$, 1,10-phen (20 mol%), CsCO₃ (0.6 mmol, 3 equiv.) and DCM (2mL) were added. Place the sealed tube in an oil bath which is preheated to 60 °C. The mixture was stirred for 12h. Then, the mixture was concentrated in vacuo, and purified by column chromatography on silica gels (ethyl acetate/ petroleum ether, 4:5) to get **3**.

2. ¹H and ¹³C data of the compound 1n



5-iodo-6-isothiocyanatobenzo[d][1,3]dioxole (1n)

¹H NMR (400 MHz, CDCl3) δ 7.15 (s, 1H), 6.76 (s, 1H), 6.02 (s, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 148.8, 147.5, 128.4, 117.8, 107.3, 102.6, 83.3. HRMS calcd for C₈H₄INO₂S⁺ (M + H⁺): 305.9080, Found: 305.9082.

3. ¹H, ¹³C and ³¹P data of the compounds 3



diethyl benzo[d]thiazol-2-ylphosphonate (3aa)³

Yield: 85%; light yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 7.9 Hz, 1H), 8.02 (d, *J* = 8.1 Hz, 1H), 7.56 (dt, *J* = 14.9, 7.1 Hz, 2H), 4.42 - 4.26 (m, 4H), 1.40 (t, *J* = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 160.1 (d, *J*_{*C-P*} =237.0 Hz), 154.6 (d, *J* = 29.0 Hz), 136.4, 127.0, 126.9, 124.9, 122.0, 64.1 (d, *J*_{*C-P*} = 5.9 Hz), 16.3 (d, *J*_{*C-P*} = 6.3 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 3.96.



diethyl (6-fluorobenzo[d]thiazol-2-yl)phosphonate (3ba)

Yield: 42%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.20 (dd, J = 9.0, 4.8 Hz, 1H), 7.68 (dd, J = 8.0, 2.4 Hz, 1H), 7.33 (td, J = 8.9, 2.5 Hz, 1H), 4.42 - 4.25 (m, 4H), 1.41 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 161.5 (d, ¹ $J_F = 247.0$ Hz), 159.9 (d, $J_{C-P}=238.0$ Hz), 151.3 (d, ² $J_F = 29.0$ Hz), 137.6 (d, ³ $J_F = 12.0$ Hz), 126.1 (d, ³ $J_F = 10.0$ Hz), 116.1 (d, ² $J_F = 25.0$ Hz), 107.9 (d, ² $J_F = 26.0$ Hz), 64.2 (d, $J_{C-P} = 5.9$ Hz), 16.2 (d, $J_{C-P} = 6.2$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 3.39. ¹⁹F NMR (376 MHz, CDCl₃) δ -112.66. HRMS calcd for C₁₁H₁₃FNO₃PS⁺ (M + H⁺): 290.0411, Found: 290.0412.



diethyl (6-chlorobenzo[d]thiazol-2-yl)phosphonate (3ca)³

Yield: 54%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.15 (d, *J* = 8.8 Hz, 1H), 7.99 (d, *J* = 1.7 Hz, 1H), 7.54 (dd, *J* = 8.8, 1.9 Hz, 1H), 4.42 - 4.21 (m, 4H), 1.41 (t, *J* = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 160.7 (d, *J*_{C-P}=237.0 Hz), 153.0 (d, *J*=29.0 Hz), 137.5, 133.3, 127.8, 125.6, 121.5, 64.2 (d, *J*_{C-P} = 5.9 Hz), 16.3 (d, *J*_{C-P} = 6.2 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 3.31.



diethyl (6-bromobenzo[d]thiazol-2-yl)phosphonate (3da)³

Yield: 57%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, J = 1.5 Hz, 1H), 8.00 (d, J = 8.8 Hz, 1H), 7.59 (dd, J = 8.8, 1.7 Hz, 1H), 4.32 - 4.19 (m, 4H), 1.32 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 160.8 (d, $J_{C-P} = 237.0$ Hz), 153.4 (d, J=28.0 Hz), 138.0, 130.5, 125.9, 124.5, 121.1, 64.2 (d, $J_{C-P} = 6.0$ Hz), 16.3 (d, $J_{C-P} = 6.2$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 3.25.



diethyl (6-iodobenzo[d]thiazol-2-yl)phosphonate (3ea)

Yield: 42%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.36 (s, 1H), 7.96 (d, J = 8.7 Hz, 1H), 7.85 (d, J = 8.7 Hz, 1H), 4.41 - 4.25 (m, 4H), 1.40 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 160.8 (d, $J_{C-P} = 237.0$ Hz), 153.9 (d, J = 28.0 Hz), 138.4, 136.1, 130.5, 126.2, 92.2, 64.3 (d, $J_{C-P} = 6.0$ Hz), 16.3 (d, $J_{C-P} = 6.2$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 3.23. HRMS calcd for C₁₁H₁₃INO₃PS⁺ (M + H⁺): 397.9471, Found: 397.9470.



diethyl (7-chlorobenzo[d]thiazol-2-yl)phosphonate (3fa)

Yield: 17%; orange oil; ¹H NMR (400 MHz, CDCl₃) δ 8.14 (dd, J = 6.6, 2.5 Hz, 1H), 7.57 - 7.51 (m, 2H), 4.41 - 4.29 (m, 4H), 1.42 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 161.2 (d, $J_{C-P} = 236.0$ Hz), 155.1 (d, J=28.0 Hz), 136.8, 127.9, 127.3, 126.6, 123.2, 64.4 (d, $J_{C-P} = 5.9$ Hz), 16.3 (d, $J_{C-P} = 6.2$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 3.23. HRMS calcd for C₁₁H₁₃ClNO₃PS⁺ (M + H⁺): 306.0115, Found: 306.0113.



diethyl (5-chlorobenzo[d]thiazol-2-yl)phosphonate (3ga)⁴

Yield: 63%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, J = 1.6 Hz, 1H), 7.93 (d, J = 8.6 Hz, 1H), 7.50 (d, J = 8.6 Hz, 1H), 4.42 - 4.26 (m, 4H), 1.41 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 162.3 (d, $J_{C-P}=236.0$ Hz), 155.3 (d, J=28.0 Hz), 134.6, 133.0, 127.6, 124.5, 122.7, 64.3 (d, $J_{C-P}=6.0$ Hz), 16.3 (d, $J_{C-P}=6.2$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 3.16.



diethyl (4-chlorobenzo[d]thiazol-2-yl)phosphonate (3ha)

Yield: 55%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.23 (d, J = 1.8 Hz, 1H), 7.93 (d, J = 8.6 Hz, 1H), 7.51 (dd, J = 8.6, 1.1 Hz, 1H), 4.42 - 4.25 (m, 4H), 1.41 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 162.2 (d, $J_{C-P} = 236.0$ Hz), 155.3 (d, J = 28.0 Hz), 134.6, 133.0, 127.7, 124.5, 122.7, 64.3 (d, $J_{C-P} = 6.0$ Hz), 16.3 (d, $J_{C-P} = 6.3$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 3.19. HRMS calcd for C₁₁H₁₃ClNO₃PS⁺ (M + H⁺): 306.0115, Found: 306.0117.



diethyl (6-(trifluoromethyl)benzo[d]thiazol-2-yl)phosphonate (3ia)

Yield: 51%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.35 (d, J = 9.9 Hz, 2H), 7.82 (dd, J = 8.7, 1.2 Hz, 1H), 4.44 - 4.28 (m, 4H), 1.42 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 163.9 (d, $J_{C-P} = 235$ Hz), 156.3 (d, ² $J_{CF} = 27$ Hz), 136.4 (d, ⁴ $J_{CF} = 2$ Hz), 129.1 (dd, ⁴ $J_{CF} = 3.3$ Hz), 125.5, 123.8 (d, ⁴ $J_{CF} = 3$ Hz), 122.5, 119.8 (d, ⁴ $J_{CF} = 5$ Hz), 64.4 (d, $J_{C-P} = 6.0$ Hz), 16.3 (d, $J_{C-P} = 6.2$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 2.86. HRMS calcd for C₁₂H₁₃F₃NO₃PS⁺ (M + H⁺): 340.0379, Found: 340.0378.



diethyl (7-methylbenzo[d]thiazol-2-yl)phosphonate (3ja)

Yield: 85%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, J = 8.3 Hz, 1H), 7.49 (t, J = 7.8 Hz, 1H), 7.31 (d, J = 7.2 Hz, 1H), 4.42 - 4.26 (m, 4H), 2.62 (s, 3H), 1.41 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 159.4 (d, $J_{C-P} = 237.0$ Hz), 154.5 (d, J=28.0 Hz), 137.1, 132.1, 127.1, 126.9, 122.3, 64.0 (d, $J_{C-P} = 5.9$ Hz), 21.4, 16.3 (d, $J_{C-P} = 6.2$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 4.13. HRMS calcd for C₁₂H₁₆NO₃PS⁺ (M + H⁺): 286.0661, Found: 286.0662.



diethyl (6-methylbenzo[d]thiazol-2-yl)phosphonate (3ka)³

Yield: 88%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, J = 8.4 Hz, 1H), 7.75 (s, 1H), 7.35 (d, J = 8.4 Hz, 1H), 4.37 - 4.21 (m, 4H), 2.49 (s, 3H), 1.36 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 158.7 (d, $J_{C-P} = 236.0$ Hz), 152.8 (d, J=29.0 Hz), 137.5, 136.7, 128.6, 124.3, 121.4, 64.0 (d, $J_{C-P} = 5.9$ Hz), 21.6, 16.2 (d, $J_{C-P} = 6.2$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 4.26.



diethyl (5-methylbenzo[d]thiazol-2-yl)phosphonate (3la)⁴

Yield: 70%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.04 (s, 1H), 7.87 (d, *J* = 8.2 Hz, 1H), 7.35 (d, *J* = 8.2 Hz, 1H), 4.42 - 4.24 (m, 4H), 2.53 (s, 3H), 1.40 (t, *J* = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 159.9 (d, *J*_{*C-P*} = 238.0 Hz), 155.1 (d, *J* = 28.0 Hz), 137.1, 133.5, 128.9, 124.6, 121.4, 64.0 (d, *J*_{*C-P*} = 5.9 Hz), 21.4, 16.3 (d, *J*_{*C-P*} = 6.3 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 4.19.



diethyl (5-methoxybenzo[d]thiazol-2-yl)phosphonate (3ma)⁵

Yield: 73%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, J = 8.9 Hz, 1H), 7.68 (d, J = 2.0 Hz, 1H), 7.18 (d, J = 8.9 Hz, 1H), 4.42 - 4.25 (m, 4H), 3.90 (s, 3H), 1.41 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 160.7 (d, $J_{C-P} = 237.0$ Hz), 156.0 (d, J=28.0 Hz), 128.5, 122.1, 118.2, 106.0, 64.0 (d, $J_{C-P} = 5.9$ Hz), 55.6, 16.3 (d, $J_{C-P} = 6.3$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 4.02.



diethyl [1,3]dioxolo[4',5':4,5]benzo[1,2-d]thiazol-6-ylphosphonate(3na)

Yield: 64%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 7.56 (s, 1H), 7.30 (s, 1H), 6.11 (s, 2H), 4.39 - 4.22 (m, 4H), 1.39 (t, *J* = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 157.2 (d, *J*_{*C*-*P*} = 241.0 Hz), 150.2 (d, *J* = 28.0 Hz), 148.8, 130.8, 103.3, 102.3, 99.7, 63.9 (d, *J*_{*C*-*P*} = 5.9 Hz), 16.2 (d, *J*_{*C*-*P*} = 6.3 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 4.21. HRMS calcd for C₁₂H₁₄NO₅PS⁺ (M + H⁺): 316.0403, Found: 316.0400.



diethyl thiazolo[4,5-b]pyridin-2-ylphosphonate (30a)

Yield: 58%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.89 (d, J = 4.4 Hz, 1H), 8.45 (dd, J = 8.1, 1.5 Hz, 1H), 7.50 (dd, J = 8.1, 4.5 Hz, 1H), 4.47 - 4.31 (m, 4H), 1.42 (t, J = 7.1 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 164.6 (d, $J_{C-P} = 31.0$ Hz), 164.1 (d, J=235.0 Hz), 149.5, 131.5, 129.9, 121.4, 64.7 (d, $J_{C-P} = 6.3$ Hz), 16.2 (d, $J_{C-P} = 6.4$ Hz). ³¹P NMR (162 MHz, CDCl₃) δ 2.26. HRMS calcd for C₁₀H₁₃N₂O₃PS⁺ (M + H⁺): 273.0457, Found: 273.0456.



dimethyl benzo[d]thiazol-2-ylphosphonate (3ab)⁶

Yield: 49%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, J = 8.2 Hz, 1H), 7.94 (d, J = 7.8 Hz, 1H), 7.49 (dt, J = 15.0, 7.2 Hz, 2H), 3.88 (d, J = 11.3 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 158.6 (d, J_{C-P} = 239.0 Hz), 154.5 (d, J = 29.0 Hz), 136.4, 127.2, 127.0, 124.9, 122.0, 54.2 (d, J_{C-P} = 6.0 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 6.67.



diisopropyl benzo[d]thiazol-2-ylphosphonate (3ac)³

Yield: 51%; white solid; ¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, *J* = 8.1 Hz, 1H), 8.00 (d, *J* = 7.9 Hz, 1H), 7.54 (dt, *J* = 14.9, 7.2 Hz, 2H), 4.98 - 4.88 (m, 2H), 1.43 (d, *J* = 6.2 Hz, 6H), 1.35 (d, *J* = 6.2 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 161.5 (d, *J*_{C-P} = 240.0 Hz), 154.6 (d, *J* = 29.0 Hz), 136.4, 126.8, 126.7, 124.9, 121.9, 73.2 (d, *J*_{C-P} = 6.0 Hz), 24.0 (d, *J*_{C-P} = 4.1 Hz), 23.8 (d, *J*_{C-P} = 4.9 Hz). ³¹P NMR (162 MHz, CDCl₃) δ 1.92.



dibutyl benzo[d]thiazol-2-ylphosphonate (3ad)⁷

Yield: 90%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 8.1 Hz, 1H), 8.01 (d, *J* = 7.9 Hz, 1H), 7.54 (dt, *J* = 15.0, 7.2 Hz, 2H), 4.35 - 4.19 (m, 4H), 1.78 - 1.67 (m, 4H), 1.49 - 1.37 (m, 4H), 0.92 (t, *J* = 7.4 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 160.0 (d, *J*_{C-P} = 237.0 Hz), 154.6 (d, *J* = 28.0 Hz), 136.3, 126.8, 124.9, 121.9, 67.7 (d, *J*_{C-P} = 6.2 Hz), 32.3 (d, *J*_{C-P} = 6.3 Hz), 18.6, 13.4. ³¹P NMR (162 MHz, CDCl₃) δ 4.12.



diisobutyl benzo[d]thiazol-2-ylphosphonate (3ae)

Yield: 84%; yellow oil; ¹H NMR (400 MHz, CDCl₃) δ 8.16 (d, J = 7.9 Hz, 1H), 7.91 (d, J = 8.1 Hz, 1H), 7.51 - 7.39 (m, 2H), 4.01 - 3.87 (m, 4H), 1.94 (dp, J = 13.4, 6.7 Hz, 2H), 0.87 (dd, J = 6.7, 1.7 Hz, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 159.8 (d, J_{C-P} = 239.0 Hz), 154.5 (d, J = 29.0 Hz), 136.3, 127.0, 126.8, 124.9, 121.9, 73.7 (d, J_{C-P} = 6.6 Hz), 29.1 (d, J_{C-P} = 6.5 Hz), 18.6. ³¹P NMR (162 MHz, CDCl₃) δ 3.95. HRMS calcd for C₁₅H₂₂NO₃PS⁺ (M + H⁺): 328.1131, Found: 328.1133.

4. References

- 1 S. Degorce, F. H. Jung, C. S. Harris, P. Koza, J. Lecoq and A. Stevenin, *Tetrahedron Lett.*, 2011, **52**, 6719
- 2 J. Ying, J.-S. Wang, L. Yao, W. Lu and X.-F. Wu, Chem. Eur. J., 2020, 26, 14565.
- 3 X.-L. Chen, X. Li, L.-B. Qu, Y.-C. Tang, W.-P. Mai, D.-H. Wei, W.-Z. Bi, L.-K. Duan, K. Sun, J.-Y. Chen, D.-D. Ke and Y.-F. Zhao, *J. Org. Chem.*, 2014, 79, 8407.
- 4 C. Hou, Y. Ren, R. Lang, X. Hu, C. Xia and F. Li, Chem. Comm., 2012, 48, 5181.
- 5 J. Gong, L. Huang, Q. Deng, K. Jie, Y. Wang, S. Guo and H. Cai, Org. Chem. Front., 2017, 4, 1781.
- 6 S. Hore, A. Srivastava and R. P. Singh, J. Org. Chem., 2019, 84, 6868.
- 7 W. Lin, F. Su, H.-J. Zhang and T.-B. Wen, Eur. J. Org. Chem., 2017, 2017, 1757.

5. Copies of ¹H, ¹³C, and ³¹P NMR Spectra











3ba ¹H NMR









3ca¹H NMR











3ea ¹H NMR













3ga ¹H NMR













3ia ¹H NMR













3ka ¹H NMR



















3ma ³¹P NMR









30a ¹H NMR













3ac ¹H NMR













3ae ¹H NMR





