Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry. This journal is © The Royal Society of Chemistry 2020

Supplementary Information

An Efficient Approach for the Synthesis of New (±)-Coixspirolactams

Vinicius Rodrigues do Nascimento^a, Melissa Lieko Souza Suenaga^a and Leandro Helgueira Andrade^{*a}

^a Institute of Chemistry, University of São Paulo, São Paulo, Brazil. 05508-000.

Table of Contents

| 1. | General Procedure for the Synthesis of the 2-(hydroxymethyl)-N-Alkyl-N-arylacrylamides S3 |
|----|--|
| | 1.1 Synthesis of acryloyl chloride |
| | S3 |
| | 1.2 General procedure for synthesis of acrylamides 2a-i |
| | S3 |
| | 1.3 General procedure for synthesis of N-methyl-N-arylacrylamides 3a-i |
| | S6 |
| | 1.4 General procedure for synthesis of <i>N</i> -benzyl- <i>N</i> -arylacrylamides 3j-r |
| | 1.5 General procedure for synthesis of 2-(hydroxymethyl)-N-alkyl-N-arylacrylamides 4a-r |
| | S10 |
| | |
| 2. | Coixspirolactams: Copies of ¹ H and ¹³ C NMR Spectra |

S17

1. General Procedure for the Synthesis of the 2-(hydroxymethyl)-*N*-alkyl-*N*-arylacrylamide



1.1 Synthesis of acryloyl chloride¹



A two-neck round-bottom flask (50 mL) equipped with a reflux condenser and a magnetic stirrer bar was charged with 4.35 mL of thionyl chloride (60 mmol) and 4.15 mL of acrylic acid (60 mmol) under nitrogen atmosphere. A solution of 10 mg of hydroquinone in 462 μ L *N-N*-dimethylformamide was added to the reaction dropwise. The resulting mixture was heated at 40 °C for 30 minutes. Finally, acryloyl chloride was removed by distillation at 77 °C (2.95 mL, 60%) as a colorless liquid.

1.2 General procedure for synthesis of acrylamides 2a-i²



R₁ = H, *p*-Me, *p*-F,*m*-F, *o*-F, *p*-CF₃, *p*-Cl, *p*-OMe, *o*-Ph

A round-bottom flask (250 mL) containing a magnetic stirrer bar was charged with aniline **1a–i** (20 mmol), triethylamine (1.05 eq, 2.92 mL) and anhydrous ethyl acetate (80 mL) under nitrogen atmosphere. Acryloyl chloride (freshly prepared; 1.5 eq, 3.25 mL) was added dropwise with stirring at 0 °C. The reaction mixture was stirred for 2 h at room temperature. Then, it was quenched with 20 mL of deionized water. The organic phase was washed with HCl 2 M aqueous solution (3 x 10 mL), H₂O (2 x 10 mL) and brine (10 mL). The organic phase was collected and dried over anhydrous MgSO₄ and the solvent was removed by reduced pressure. The crude product was used in the next step without further purification.



N-phenylacrylamide (2a): light salmon solid, 81% yield. MS-EI: m/z(%) 147(M+, 38), 93(100).



N-(4-fluorophenyl)acrylamide (2b): white solid, 92% yield. MS-EI: m/z(%) 165(M+, 34), 111(100).



N-(2-fluorophenyl)acrylamide (2c): pale yellow solid, 92% yield. MS-EI: m/z(%) 165(M+, 27), 111(100).



N-(3-fluorophenyl)acrylamide (2d): pale brown solid, 99% yield. MS-EI: m/z(%) 165(M+, 19), 55(100).



N-(4-chlorophenyl)acrylamide (2e): white solid, 87% yield. MS-EI: m/z(%) 181(M+, 12), 55(100).



N-(4-(trifluoromethyl)phenyl)acrylamide (2f): pale yellow solid, 90% yield. MS-EI: m/z(%) 215(M+, 21), 55(100).

H₃CO 、╷╷╷╱

N-(4-methoxyphenyl)acrylamide (2g): white solid, 92% yield. MS-EI: m/z(%) 177(M+, 50), 123(100).



N-(*p*-tolyl)acrylamide (2h): white solid, 90% yield. MS-EI: m/z(%) 161(M+, 31), 107(100).



N-([1,1'-biphenyl]-2-yl)acrylamide (2i): light salmon solid, 99% yield. MS-EI: m/z(%) 223(M+, 44), 169(100).

1.3 General procedure for synthesis of *N*-methyl-*N*-arylacrylamides 3a-i³



R₁ = H, *p*-Me, *p*-F,*m*-F, *o*-F, *p*-CF₃, *p*-CI, *p*-OMe, *o*-Ph

A two-neck round-bottom flask (100 mL) containing a magnetic stirrer bar was charged with acrylamide **2a–i** (20 mmol) and anhydrous THF (20 mL) under nitrogen atmosphere. Sodium hydride 60% (1.1 eq, 0.88 g) was added in portions with stirring at 0 °C. The mixture was stirred for an additional 15 min. Then, iodomethane (1.1 eq, 1.37 mL) was added dropwise at 0 °C, and the mixture was stirred at room temperature for 1 h. Deionized water (20 mL) was slowly added to quench the reaction. THF was removed by reduced pressure. Ethyl acetate (50 mL) was added to the crude mixture and transferred to a separatory funnel. The organic phase was washed with HCl 2 M aqueous solution (30 mL), H₂O (20 mL) and brine (10 mL). The organic phase was collected and dried over anhydrous MgSO₄ and the solvent was removed by reduced pressure. The crude material was purified by column chromatography on silica gel (1:1 hexane/ethyl acetate).

N-methyl-*N*-phenylacrylamide (3a): orange solid, 96% yield. MS-EI: m/z(%) 161 (M+, 37), 107(100).

,Å∕∕

N-methyl-*N*-(*p*-tolyl)acrylamide (3b): white solid, 64% yield. MS-EI: m/z(%) 175 (M+, 37), 121(100)



N-(4-fluorophenyl)-*N*-methylacrylamide (3c): crystalline solid, 50% yield. MS-EI: m/z(%) 179 (M+, 27), 55(100).



N-(2-fluorophenyl)-*N*-methylacrylamide (3d): yellow oil, 80% yield. MS-EI: m/z(%) 179 (M+, 12), 125(100).

Ĵ,ľ∕

N-(3-fluorophenyl)-*N*-methylacrylamide (3e): white solid, 64% yield. MS-EI: m/z(%) 179 (M+, 13), 55(100).

._NĹ

N-(4-chlorophenyl)-*N*-methylacrylamide (3f): white solid, 87% yield. MS-EI: m/z(%) 195 (M+, 12), 55(100).

C, i

N-methyl-*N*-(4-(trifluoromethyl)phenyl)acrylamide (3g): colorless oil, 63% yield. MS-EI: m/z(%) 229 (M+, 16), 55(100).



N-(4-methoxyphenyl)-*N*-methylacrylamide (3h): pale yellow solid, 65% yield. MS-EI: m/z(%) 191 (M+, 51), 68(100).



N-([1,1'-biphenyl]-2-yl)-*N*-methylacrylamide (3i): off-white solid, 61% yield. MS-EI: m/z(%) 237(M+, 40), 68(100).

1.4 General procedure for synthesis of N-benzyl-N-phenylacrylamides 3j-r³



R₁ = H, *p*-Me, *p*-F,*m*-F, *o*-F, *p*-CF₃, *p*-Cl, *p*-OMe, *o*-Ph

The benzylation reaction was carried out using the same protocol described for methylation. However, benzylation required 1 h at 50 °C. The crude material was purified by column chromatography on silica gel (7:3 hexane/ethyl acetate).



N-benzyl-*N*-phenylacrylamide (3j): white solid, 22% yield. MS-EI: m/z(%) 237 (M+, 23), 91(100).



N-benzyl-*N*-(*p*-tolyl)acrylamide (3k): white solid, 70% yield. MS-EI: m/z(%) 251 (M+, 25), 91(100).



N-benzyl-*N*-(4-fluorophenyl)acrylamide (31): white solid, 89% yield. MS-EI: m/z(%) 255 (M+, 26), 91(100).



N-benzyl-*N*-(3-fluorophenyl)acrylamide (3m): off-white solid, 54% yield. MS-EI: m/z(%) 255 (M+, 12), 91(100).



N-benzyl-*N*-(2-fluorophenyl)acrylamide (3n): light salmon solid, 52% yield. MS-EI: m/z(%) 255 (M+, 15), 91(100).



N-benzyl-*N*-(4-chlorophenyl)acrylamide (30): white solid, 63% yield. MS-EI: m/z(%) 271 (M+, 11), 91(100).



N-benzyl-*N*-(4-(trifluoromethyl)phenyl)acrylamide (3p): white solid, 58% yield. MS-EI: m/z(%) 305 (M+, 31), 91(100).



N-benzyl-*N*-(4-methoxyphenyl)acrylamide (3q): orange solid, 72% yield. MS-EI: m/z(%) 267 (M+, 52), 91(100).



N-([1,1'-biphenyl]-2-yl)-*N*-benzylacrylamide (3r): off-white solid, 63% yield. MS-EI: m/z(%) 313(M+, 25), 91(100).





 $\mathbf{R}_1 = H$, *p*-Me, *p*-F,*m*-F, *o*-F, *p*-CF₃, *p*-Cl, *p*-OMe, *o*-Ph $\mathbf{R}_2 = Me$, Bn

A two-neck round-bottom flask (25 mL) equipped with a reflux condenser and a magnetic stirrer bar was charged with paraformaldehyde (3.0 g, 5 eq), DABCO (2.24 g, 1 eq), phenol (0.47 g, 0.25 eq) and a mixture of *t*-butanol and water (7 mL, 3:7). The mixture was stirred at 55 °C until dissolution is complete. *N*-phenylacrylamides (**3a–r**, 20 mmol) were added and stirred at 55 °C for 24 h. Then, the organic solvent was removed by reduced pressure and the residue was extracted with chloroform (3 x 20 mL). The organic phase was collected and dried over anhydrous MgSO₄ and the solvent was removed by reduced pressure. The crude material was purified by column chromatography on silica gel (1:1 or 3:7 hexane/ethyl acetate).



2-(hydroxymethyl)-N-methyl-N-phenylacrylamide (4a): White solid, 52% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.38–7.29 (m, 3H), 7.18 (d, *J* = 6.0 Hz, 2H), 5.30 (s, 1H), 4.97 (s, 1H), 4.23 - 4.17 (m, 2H), 3.38 (s, 3H). ¹³**C NMR** (75 MHz, CDCl₃) δ 170.30, 143.06, 141.46, 129.35, 127.16, 126.73, 120.06, 64.43, 37.84.



2-(hydroxymethyl)-N-methyl-N-(p-tolyl)acrylamide (4b): White solid, 53% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.15 (d, *J* = 9.0 Hz, 2H), 7.06 (d, *J* = 9.0 Hz, 2H), 5.29 (s, 1H), 4.99 (s, 1H), 4.21 - 4.13 (m, 2H), 3.34 (s, 3H), 2.34 (s, 3H). ¹³**C NMR** (75 MHz, CDCl₃) δ 170.35, 143.24, 141.77, 137.10, 129.94, 126.52, 119.79, 68.51, 64.40, 37.86.



N-(4-fluorophenyl)-2-(hydroxymethyl)-*N*-methylacrylamide (4c): Colorless oil, 78% yield. ¹H NMR (300 MHz, CDCl₃) δ 7.21–7.15 (m, 2H), 7.07–6.01 (m, 2H), 5.31 (s, 1H), 4.95 (s, 1H), 4.21 (s, 2H), 3.35 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 172.83, 161.30(d, $J_{C,F}$ = 246.75), 141.37, 128.45 (d, $J_{C,F}$ = 8.25), 119.90, 116.34 (d, $J_{C,F}$ = 8.25 Hz), 116.04 (d, $J_{C,F}$ = 7.5 Hz), 64.40, 37.98.



N-(2-fluorophenyl)-2-(hydroxymethyl)-*N*-methylacrylamide (4d): White solid, actual mass 0.9587g, 30% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.31–7.21 (m, 2H), 7.17–7.09 (m, 2H), 5.30 (s, 1H), 5.00 (s, 1H), 4.24 (s, 2H), 3.32 (s, 3H). ¹³**C NMR** (75 MHz, CDCl₃) δ 170.73, 157.38 (d, $J_{C,F}$ = 248.25), 142.56, 132.10, 129.22 (d, $J_{C,F}$ = 7.50 Hz), 129.06, 124.85 (d, $J_{C,F}$ = 3.75 Hz), 119.11, 116.71 (d, $J_{C,F}$ = 20.25 Hz), 64.16, 37.00.



N-(3-fluorophenyl)-2-(hydroxymethyl)-*N*-methylacrylamide (4e): Colorless oil, 51% yield. ¹H NMR (300 MHz, CDCl₃) δ 7.38–7.24 (m, 1H), 7.04–6.90 (m, 3H), 5.33 (s, 1H), 4.99 (s, 1H), 4.26–4.21 (m, 2H), 3.37 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 170.18, 162.77 (d, *J*_{C,F} = 246.75 Hz), 145.87 (d, *J*_{C,F} = 9.75 Hz), 142.89, 130.45 (d, *J*_{C,F} = 9.2 Hz), 122.50 (d, *J*_{C,F} = 3.0 Hz), 120.13, 114.29 (d, *J*_{C,F} = 7.5 Hz), 114.00 (d, *J*_{C,F} = 4.5 Hz), 64.30, 37.76.



N-(4-chlorophenyl)-2-(hydroxymethyl)-*N*-methylacrylamide (4f): Colorless oil, 99% yield. ¹H NMR (300 MHz, CDCl₃) δ 7.36–7.24 (m, 2H), 7.20–7.10 (m, 2H), 5.32 (s, 1H), 5.0–4.95 (m, 1H), 4.25 - 4.21 (s, 2H), 3.36 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 170.14, 142.88, 132.75, 129.52, 128.00, 121.06, 120.18, 64.36, 37.80.



2-(hydroxymethyl)-*N***-(4-methoxyphenyl)**-*N***-methylacrylamide (4g):** Pale yellow solid, 58% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.13–7.05 (m, 2H), 6.85 (d, *J* = 9.0 Hz, 2H), 5.29 (s, 1H), 5.00– 5.95 (m, 1H), 4.20–4.11 (m, 2H), 3.78 (s, 3H), 3.33 (s, 3H). ¹³**C NMR** (75 MHz, CDCl₃) δ 170.41, 143.28, 141.64, 128.02, 127.90, 114.48, 114.39, 68.64, 64.46, 55.43.



2-(hydroxymethyl)-*N***-methyl**-*N***-(4-(trifluoromethyl)phenyl)acrylamide (4h):** White solid, 36% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.62 (d, J = 9.0 Hz, 2H), 7.34 (d, J = 6.0 Hz, 2H), 5.34 (s, 1H), 4.95 (s, 1H), 4.31 - 4.23 (m, 2H), 3.41 (s, 3H). ¹³**C NMR** (75 MHz, CDCl₃) δ 170.16, 147.58,

142.76, 126.85 (q, $J_{C,F}$ = 3.75 Hz) 126.46 (q, $J_{C,F}$ = 3.75 Hz), 123.70 (q, $J_{C,F}$ = 270.75 Hz), 121.35, 120.47, 64.23, 37.66.



N-([1,1'-biphenyl]-2-yl)-2-(hydroxymethyl)-*N*-methylacrylamide (4i): White solid, 75% yield. ¹H NMR (300 MHz, CDCl₃) δ 7.46–7.21 (m, 9H), 5.24 (s, 1H), 4.77 (s, 1H), 3.50–3.41 (m, 2H), 3.32 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 169.85, 141.96, 138.74, 138.63, 131.38, 128.85 (2C), 128.65, 128.27, 127.94, 127.84, 127.75, 120.85, 64.20, 38.43.



N-benzyl-2-(hydroxymethyl)-N-phenylacrylamide (4j): White solid, 65% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.35–7.20 (m, 8H), 7.05–7.00 (m, 2H), 5.30 (s, 1H), 5.00 (s, 3H), 4.21 (s, 2H). ¹³**C NMR** (75 MHz, CDCl₃) δ 169.66, 143.04, 142.95, 137.07, 129.17, 128.46, 128.37, 127.65, 127.42, 127.34, 120.27, 64.66, 53.39.



N-benzyl-2-(hydroxymethyl)-N-(p-tolyl)acrylamide (4k): White solid, 58% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.33–7.19 (m, 5H), 7.05 (d, *J* = 6.0 Hz, 2H), 6.89 (d, *J* = 9.0 Hz, 2H), 5.28 (s, 1H), 5.02 (s, 1H), 4.96 (s, 2H), 4.21–4.15 (m, 2H), 2.30 (s, 3H). ¹³**C NMR** (75 MHz, CDCl₃) δ 170.20, 143.29, 140.25, 137.26, 137.18, 129.78, 128.43 (2C), 127.45, 127.36, 119.90, 64.56, 53.40, 21.00.



N-benzyl-*N*-(4-fluorophenyl)-2-(hydroxymethyl)acrylamide (4l): White solid, 73% yield. ¹H NMR (300 MHz, CDCl₃) δ 7.29–7.19 (m, 5H), 7.01–6.91 (m, 4H), 5.29 (s, 1H), 4.97 (m, 3H), 4.24–4.16 (m, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 170.06, 161.42(d, $J_{C,F}$ = 246 Hz), 141.17, 138.77, 136.80, 129.46 (d, $J_{C,F}$ = 8.25 Hz), 128.55-128.46 (m, 2C), 127.57, 119.89, 116.07(d, $J_{C,F}$ = 22.5 Hz), 64.50, 53.39.



N-benzyl-N-(2-fluorophenyl)-2-hydroxyacrylamide (4m): White solid, 34% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.33–7.15 (m, 6H), 7.07–6.95 (m, 3H), 5.30 (s, 2H), 5.05 (s, 1H), 4.69 (s, 1H), 4.42–4.07 (m, 2H).¹³**C NMR** (75 MHz, CDCl₃) δ 170.58, 157.57 (d, $J_{C,F}$ = 247.50), 142.71, 136.52, 129.80, 129.29 (d, $J_{C,F}$ = 7.50 Hz), 128.60, 128.40 (2C), 127.56, 124.55 (d, $J_{C,F}$ = 4.50 Hz), 119.12, 116.55 (d, $J_{C,F}$ = 20.25 Hz), 64.19, 52.45.



N-benzyl-*N*-(3-fluorophenyl)-2-(hydroxymethyl)acrylamide (4n): Colorless oil, 34% yield. ¹H NMR (300 MHz, CDCl₃) δ 7.31–7.19 (m, 6H), 6.98–6.88 (m, 1H), 6.88–6.75 (m, 2H), 5.33 (s, 1H), 5.04 (s, 1H), 5.00 (s, 2H), 4.29–4.20 (m, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 170.00, 162.63 (d, $J_{C,F}$ = 246.75 Hz), 144.45 (d, $J_{C,F}$ = 9.75 Hz), 143.02, 136.74, 130.23 (d, $J_{C,F}$ = 9.0 Hz), 128.57, 128.23, 127.58, 123.45 (d, $J_{C,F}$ = 2.25 Hz), 120.16, 114.94 (d, $J_{C,F}$ = 22.5 Hz), 114.37 (d, $J_{C,F}$ = 21.0 Hz), 64.40, 53.28.



N-benzyl-*N*-(4-chlorophenyl)-2-(hydroxymethyl)acrylamide (40): Crystalline solid, 56% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.32–7.16 (m, 7H), 6.97 (d, *J* = 9.0 Hz, 2H), 5.31 (s, 1H), 4.99– 4.95 (m, 3H), 4.29–4.19 (m, 2H). ¹³**C NMR** (75 MHz, CDCl₃) δ 169.99, 143.07, 141.41, 136.73, 133.08, 129.36, 128.97, 128.58, 128.36, 127.59, 120.13, 64.41, 53.27.



N-benzyl-2-(hydroxymethyl)-*N*-(4-methoxyphenyl)acrylamide (4p): White solid, 46% yield. ¹H NMR (300 MHz, CDCl₃) δ 7.35–7.17 (m, 6H), 6.96–6.88 (m, 2H), 6.76 (d, *J* = 9.0 Hz, 2H), 5.29 (s, 1H), 4.97 (s, 2H), 4.23–4.13 (m, 2H), 3.78 (s, 3H). ¹³C NMR (75 MHz, CDCl₃) δ 170.27, 143.48, 141.80, 137.12, 135.46, 129.03, 128.89, 128.53, 128.43, 114.26, 114.16, 68.62, 64.51, 55.33.



N-benzyl-2-(hydroxymethyl)-*N*-(4-(trifluoromethyl)phenyl)acrylamide (4q): Crystalline solid, 75% yield.

¹**H NMR** (300 MHz, CDCl₃) δ 7.53 (d, *J* = 6.0 Hz, 2H), 7.30–7.18 (m, 7H), 5.33 (s, 1H), 5.03 (s, 2H), 4.99 (s, 1H), 4.27 (s, 2H). ¹³**C NMR** (75 MHz, CDCl₃) δ 170.02, 146.23, 142.91, 136.62, 128.65, 128.12, 127.72, 127.69 (m), 127.66, 127.26 (q, *J*_{C,F} = 211.00 Hz) 126.31 (q, *J*_{C,F} = 3.75 Hz), 120.53, 64.31, 53.22.



N-([1,1'-biphenyl]-2-yl)-*N*-benzyl-3-hydroxy-2-oxopropanamide (4r): White solid, 17% yield. ¹H NMR (300 MHz, CDCl₃) δ 7.54–7.14 (m, 13H), 7.08-7.00 (m, 1H), 5.58 (d, *J* = 15.0 Hz, 1H), 5.30 (s, 1H), 4.89 (s, 1H), 4.12 (d, *J* = 15.0 Hz, 1H), 3.61 - 3.36 (m, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 169.85, 142.24, 140.88, 138.65, 138.48, 137.26, 131.52, 129.01, 128.86, 128.55, 128.46 (2C), 128.32, 128.01, 127.76, 127.50, 121.26, 64.38, 53.62.



Figure S2. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound **6a**.



Figure S4. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound **6b**.





Figure S8. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound 6d.









Figure S14. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound **6g**.



S25



S26



S27



S28





Figure S26. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound 6m.



Figure S28. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound **6n**.



S32



S33



Figure S34. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound **6q**.



Figure S36. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound **6r**.



Figure S38. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound 6s.



S37



S38



Figure S44. ¹³C NMR spectrum (75 MHz, CDCl₃) of the compound **7b**.







Figure S48. ¹³C NMR spectrum (75 MHz, CD₃CN) of the compound 7d.



Figure S49. ¹H NMR spectrum (300 MHz, CD₃CN) of the compound 7e.





Figure S50. ¹³C NMR spectrum (75 MHz, CD₃CN) of the compound 7e.







Figure S56. ¹³C NMR spectrum (75 MHz, CD₃CN) of the compound 7h.



S46



References

¹ A-X. Zheng, C-B. Gong, W-J. Zhang, Q. Tang, H-R. Huang, C-F. Chow and Q. Tang, *Mol. Cat.* 2017, **442**, 115–125.

- ² L. Ackermam, A. V. Lygin and N. Hofmann, Org. Lett. 2011, **13**, 3278–3281.
- ³ C-W. Chan, P-Y. Lee and W-Y. Yu, *Tetrahedron Letters*, 2015, **56**, 2559–2563.
- ⁴ X. Mu, T. Wu, H.-Y. Wang, Y.-L. Guo and G. Liu, J. Am. Chem. Soc. 2012, **134**, 878–881.