Supporting Information

Borylation of Primary and Secondary Alkyl Bromides Catalyzed by Cu$_2$O Nanoparticles

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Part 1. General Information

All reactions were carried out in an oven-dried flask under a pure and dry argon atmosphere. Cu$_2$O nanoparticles were synthesized in our laboratory. Generally, alkyl iodides and alkyl bromides were purchased from commercial sources (Aldrich, Acros, Alfa Aesar, Fluka, Lancaster). Octadecylamine (ODA) was purchased from Aladdin and 1-octadecene (ODE) was purchased from Acros and Cu(OAc)$_2$·H$_2$O was purchased from Adamas. CuO nanoparticles (40 nm) were purchased from Aladdin. The solvents (MeOH, EtOH, DMF, THF, t-BuOH) and inorganic bases (LiOMe, t-BuONa, NaOH, K$_2$CO$_3$, K$_3$PO$_4$) were purchased from Aladdin and without further purification. Analytical thin layer chromatography (TLC) was performed using Merck silica gel GF254 plates. Flash column chromatography was performed with silica gel (200-300 mesh). $^1$H-NMR, $^{13}$C-NMR spectra were recorded on a Bruker Avance 300 spectrometer or Agilent VNMRS600 at ambient temperature in CDCl$_3$. Data for $^1$H-NMR are reported as follows: chemical shift (δ ppm), multiplicity, integration, and coupling constant (Hz). Data for $^{13}$C-NMR are reported in terms of chemical shift (δ ppm), multiplicity, and coupling constant (Hz). Gas chromatographic (GC) analyses were performed on a Shimadzu GC-2014 Series GC System. The samples were characterized by powder X-ray diffraction (XRD) on a D/Max2500V X-ray diffractometer with monochromatized Cu Ka (λ = 1.5406 Å) incident radiation. XRD patterns were recorded from 10° to 90° (2θ) with a scanning rate of 6°/min. The morphologies of the samples were analyzed by JEM-2100F high-resolution transmission electron microscope. AAS was analyzed by Perkin Eime AA800. XPS was recorded on a Thermo ESCALAB250.

Part 2. Experimental Section

a. General procedure for the synthesis of Cu$_2$O NPs/CB

Firstly, 4.04 g of ODA was added in a capped vial at 80 °C and stirred for 5 min. 2 mL of ODE was introduced to molten ODA. After stirring for 10 min, 200 mg of Cu(OAc)$_2$·H$_2$O was added to the mixture and stirred for 15 min at 100 °C to ensure complete dissolution. Secondly, the mixture was heated at 200 °C for 25 min, then 260 mg carbon black was added in to the mixture. At the same time, the temperature began to decrease to 70 °C under ambient temperature. The final product was centrifugalized and washed with ethanol and chloroform under 70 °C for three times to ensure the removal of the impurities, then dried in a vacuum oven at room temperature for 12 h.

b. General procedures for the optimization of the reaction conditions. (Table 1)

General Procedure A: A 25 mL oven-dried Schlenk tube was charged with Cu$_2$O NPs/CB (5 mg), B$_2$Pin$_2$ (0.375 mmol), base. The tube was evacuated and filled with argon (this procedure was repeated three times). Then solvent (1.5 mL) was added with a syringe under a counter flow of argon. Next, (2-bromoethyl)benzene (0.25 mmol) was added with a syringe under a counter flow of argon. The resulting reaction mixture was stirred at room temperature for 12 h to 24 h. The reaction mixture was then diluted with Et$_2$O, filtered through silica gel with copious washings (Et$_2$O), biphenyl (38.5 mg, 0.25 mmol) was added as internal standard. The yield was determined by GC.

c. Experimental Procedures for the reaction of alkyl bromides with B$_2$pin$_2$.

(Scheme 2 & Scheme 3)
**General Procedure B.** A 25 mL oven-dried Schlenk tube was charged with Cu$_2$O NPs/CB (5.0 mg), B$_2$Pin$_2$ (0.375 mmol), MeOLi (0.5 mmol) and alkyl bromide or alkyl iodide (0.25 mmol) (if solid). The tube was evacuated and filled with argon (this procedure was repeated three times). Then EtOH (1.5 mL) was added with a syringe under a counter flow of argon. Next, alkyl bromide or alkyl iodide (0.25 mmol) (if liquid) was added with a syringe under a counter flow of argon. The resulting reaction mixture was stirred at room temperature for 12 h. The reaction mixture was then diluted with Et$_2$O, filtered through silica gel with copious washings (Et$_2$O or EtOAc), concentrated, and purified by column chromatography.

**Part 3. Characterization of the products**

1. Compound name: 4,4,5,5-Tetramethyl-2-phenethyl-1,3,2-dioxaborolane (3a).[1]

   Following general procedure B, colorless liquid. $^1$H NMR (600 MHz, CDCl$_3$) δ 7.26 (t, $J = 7.5$ Hz, 2H), 7.22 (d, $J = 7.0$ Hz, 2H), 7.15 (t, $J = 7.2$ Hz, 1H), 2.79-2.70 (m, 2H), 1.22 (s, 12H), 1.17-1.12 (m, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) δ 144.39, 128.16, 127.98, 125.47, 83.08, 29.94, 24.79.

   This compound is known.

2. Compound name: 4,4,5,5-Tetramethyl-2-(3-phenylpropyl)-1,3,2-dioxaborolane (3b).[1]

   Following general procedure B, colorless liquid. $^1$H NMR (600 MHz, CDCl$_3$) δ 7.27 (t, $J = 7.5$ Hz, 2H), 7.17 (dd, $J = 13.7$, 7.1 Hz, 3H), 2.66-2.58 (m, 2H), 1.80-1.70 (m, 2H), 1.25 (s, 12H), 0.84 (t, $J = 7.9$ Hz, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) δ 142.68, 128.54, 128.15, 125.55, 82.92, 38.59, 26.11, 24.83.

   This compound is known.

3. Compound name: 2-Butyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3c).[2]

   Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) δ 1.37 (dd, $J = 15.3$, 7.8 Hz, 2H), 1.30 (dd, $J = 15.0$, 7.3 Hz, 2H), 1.23 (s, 12H), 0.87 (t, $J = 7.2$ Hz, 3H), 0.76 (t, $J = 7.8$ Hz, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) δ 81.80, 28.68, 25.19, 24.39, 23.79, 12.86.
This compound is known.

4. Compound name: 4,4,5,5-Tetramethyl-2-octyl-1,3,2-dioxaborolane (3d).\[7\]

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.32 (dd, $J = 14.1$, 6.9 Hz, 2H), 1.25-1.18 (m, 10H), 1.17 (s, 12H), 0.80 (t, $J = 6.9$ Hz, 3H), 0.69 (t, $J = 7.8$ Hz, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.79, 31.43, 30.88, 28.69, 28.24, 28.36, 23.80, 22.99, 21.66, 13.09. This compound is known.

5. Compound name: 2-Isobutyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3e).\[9\]

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.58 (s, 1H), 1.26 (s, 12H), 1.11 (d, $J = 41.0$ Hz, 2H), 0.86 (dt, $J = 14.9$, 6.2 Hz, 6H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 30.91, 28.68, 21.68, 13.11. This compound is known.

6. Compound name: 4,4,5,5-Tetramethyl-2-pentyl-1,3,2-dioxaborolane (3f).\[5\]

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.39 (dt, $J = 14.7$, 7.5 Hz, 2H), 1.30-1.25 (m, 4H), 1.23 (s, 12H), 0.86 (t, $J = 6.9$ Hz, 3H), 0.75 (t, $J = 7.9$ Hz, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.80, 33.69, 28.69, 23.79, 22.67, 21.42, 13.04. This compound is known.

7. Compound name: 2-(2-Ethylhexyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3g).\[3\]
Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 3.49 (t, $J = 6.8$ Hz, 2H), 1.78-1.69 (m, 2H), 1.43-1.34 (m, 4H), 1.29 (dd, $J = 15.3$, 8.3 Hz, 2H), 1.21 (s, 12H), 0.74 (t, $J = 7.7$ Hz, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.88, 44.13, 31.52, 30.52, 25.62, 23.80, 22.77. This compound is known.

8. Compound name: Ethyl 5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) pentanoate (3h).\[4\]

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 4.09 (q, $J = 7.1$ Hz, 2H), 2.27 (t, $J = 7.6$ Hz, 2H), 1.64-1.57 (m, 2H), 1.45-1.38 (m, 2H), 1.23 (d, $J = 10.6$ Hz, 15H), 0.77 (t, $J = 7.8$ Hz, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 173.83, 82.93, 60.09, 34.20, 27.55, 24.77, 23.60, 14.21. This compound is known.

9. Compound name: 2-(2-(1,3-Dioxan-2-yl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3i).\[1\]

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 4.44 (t, $J = 5.1$ Hz, 1H), 4.05 (dd, $J = 11.6$, 4.9 Hz, 2H), 3.71 (t, $J = 12.1$ Hz, 2H), 2.08 – 1.98 (m, 1H), 1.68 (dd, $J = 12.9$, 7.6 Hz, 2H), 1.28 (d, $J = 14.3$ Hz, 1H), 1.20 (s, 12H), 0.79 (t, $J = 7.7$ Hz, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 103.10, 82.87, 66.74, 29.46, 25.80, 24.73. This compound is known.

10. Compound name: 4,4,5,5-Tetramethyl-2-(3-phenoxypropyl)-1,3,2-dioxaborolane (3j).\[3\]

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.26 (t, $J = 7.7$ Hz, 2H), 6.90 (t, $J = 10.0$ Hz, 3H), 3.93 (t, $J = 6.7$ Hz, 2H), 1.92-1.88 (m, 2H), 1.24 (s, 12H), 0.92 (t, $J$
This compound is known.

11. Compound name: 3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl) propanenitrile (3k).

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 2.41-2.37 (m, 2H), 1.24 (s, 12H), 1.16 (t, $J = 7.8$ Hz, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 123.65, 86.59, 32.32, 27.41, 14.54.

This compound is known.

12. Compound name: 2-Benzyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3l).

Following general procedure B, colorless liquid. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.23 (t, $J = 7.4$ Hz, 2H), 7.18 (d, $J = 7.7$ Hz, 2H), 7.11 (t, $J = 6.8$ Hz, 1H), 2.29 (s, 2H), 1.23 (s, 12H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 137.61, 127.95, 127.21, 123.78, 82.38, 23.69.

This compound is known.

13. Compound name: 2-(4-Bromophenethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3m).

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.36 (d, $J = 8.4$ Hz, 2H), 7.08 (d, $J = 8.4$ Hz, 2H), 2.71-2.67 (m, 2H), 1.21 (s, 12H), 1.13-1.09 (m, 2H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 143.32, 131.17, 129.80, 119.16, 83.19, 29.38, 24.80, 19.16.

This compound is known.

14. Compound name: 1,3-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propane (3n).
Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.54-1.48 (m, 2H), 1.21 (s, 24H), 0.79 (t, $J$ = 7.9 Hz, 4H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 85.41, 27.45, 21.19. This compound is known.

15. Compound name: 2-Cycloheptyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5a).$^{[3]}$

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.70-1.64 (m, 2H), 1.60 (dd, $J$ = 14.1, 7.0 Hz, 2H), 1.54-1.45 (m, 2H), 1.45-1.33 (m, 6H), 1.17 (s, 12H), 1.04-0.97 (m, 1H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.72, 28.57, 27.94, 27.31, 23.70. This compound is known.

16. Compound name: 2-Cyclohexyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5b). $^{[3]}$

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.60 (dd, $J$ = 23.1, 16.7 Hz, 4H), 1.40-1.24 (m, 6H), 1.22 (s, 12H), 0.97 (t, $J$ = 10.0 Hz, 1H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.71, 26.94, 26.12, 25.74, 23.73. This compound is known.

17. Compound name: 2-Cyclopentyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5c). $^{[3]}$

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.71-1.64 (m, 2H), 1.56-1.48 (m, 2H), 1.40 (ddd, $J$ = 23.8, 13.5, 7.2 Hz, 4H), 1.17 (s, 12H), 1.13-1.08 (m, 1H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.75, 28.69, 27.49, 25.81, 23.71.
This compound is known.

18. Compound name: 2-Cyclobutyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5d).

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 2.03-1.84 (m, 6H), 1.55 (s, 1H), 1.19 (s, 12H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.86, 28.68, 23.72, 22.85, 21.63. HRMS calcd for C$_{10}$H$_{19}$BO$_2$: 182.1478; found: 182.1476.

This compound is new.


Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 2.26 (s, 1H), 2.20 (s, 1H), 1.49 (d, $J$ = 22.9 Hz, 3H), 1.32 (t, $J$ = 9.9 Hz, 2H), 1.23 (d, $J$ = 2.0 Hz, 3H), 1.20 (s, 12H), 0.87-0.84 (m, 1H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.75, 37.71, 37.11, 35.64, 31.22, 31.18, 28.28, 23.69.

This compound is known.


Following general procedure B, white soild. $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 1.98 (s, 2H), 1.74 (dd, $J$ = 26.0, 13.7 Hz, 7H), 1.67-1.60 (m, 4H), 1.29 (s, 1H), 1.18 (s, 13H), 0.83-0.76 (m, 1H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 81.74, 38.32, 36.72, 35.25, 28.32, 27.24, 27.11, 23.80.

This compound is known.

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) δ 7.17 (t, $J = 6.9$ Hz, 2H), 7.12 (d, $J = 7.2$ Hz, 2H), 7.07 (t, $J = 7.2$ Hz, 1H), 2.73 (dd, $J = 13.6$, 7.5 Hz, 1H), 2.47 (dd, $J = 13.6$, 8.4 Hz, 1H), 1.30 (dd, $J = 15.3$, 7.6 Hz, 1H), 1.11 (d, $J = 6.9$ Hz, 12H), 0.89 (d, $J = 7.4$ Hz, 3H); $^{13}$C NMR (151 MHz, CDCl$_3$) δ 141.25, 127.85, 126.96, 124.50, 81.95, 37.91, 28.68, 23.65, 14.15.

This compound is known.

22. Compound name: 3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl) butanenitrile (5h).

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) δ 2.43 (dd, $J = 16.8$, 5.8 Hz, 1H), 2.30 (dd, $J = 16.9$, 8.4 Hz, 1H), 1.37 (dt, $J = 21.2$, 10.5 Hz, 1H), 1.23 (s, 12H), 1.13 (d, $J = 7.5$ Hz, 3H); $^{13}$C NMR (151 MHz, CDCl$_3$) δ 122.51, 86.54, 32.32, 27.36, 27.31, 23.05, 17.54. HRMS calcd for C$_{10}$H$_{18}$BNO$_2$: 195.1431; found: 195.1432.

This compound is new.

23. Compound name: 5,5-Dimethyl-2-phenethyl-1,3,2-dioxaborinane (7a).

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) δ 7.26 (d, $J = 6.4$ Hz, 2H), 7.21 (d, $J = 7.4$ Hz, 2H), 7.14 (t, $J = 7.2$ Hz, 1H), 3.59 (s, 4H), 2.75-2.66 (m, 2H), 1.11-1.03 (m, 2H), 0.92 (s, 6H); $^{13}$C NMR (151 MHz, CDCl$_3$) δ 145.00, 128.15, 127.97, 125.31, 72.00, 31.63, 30.07, 29.71, 21.82. HRMS calcd for C$_{13}$H$_{19}$BO$_2$: 218.1478; found: 218.1475.

This compound is new.

24. Compound name: 5,5-Dimethyl-2-(1-phenylpropan-2-yl)-1,3,2-dioxaborinane (7b).

Following general procedure B, colorless oil. $^1$H NMR (600 MHz, CDCl$_3$) δ 7.23 (d, $J = 7.4$ Hz, 2H), 7.17 (d, $J = 6.8$ Hz, 2H), 7.14 (t, $J = 7.3$ Hz, 1H), 3.56 (s, 4H), 2.81 (dd, $J = 13.5$, 6.9 Hz, 1H), 2.45 (dd, $J = 13.5$, 8.7 Hz, 1H), 1.24-1.19 (m, 1H), 0.91 (d, $J = 7.4$ Hz, 3H), 0.89 (s, 6H); $^{13}$C NMR (151 MHz, CDCl$_3$) δ 142.93, 128.86, 127.97, 125.35, 71.95, 39.07, 31.59, 29.71, 21.77,
15.34. HRMS calcd for C_{14}H_{21}BO_{2}: 232.1635; found: 232.1632. This compound is new.

25. Compound name: 3-(5,5-Dimethyl-1,3,2-dioxaborinan-2-yl) butanenitrile (7c).

Following general procedure B, colorless oil. \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 3.59 (s, 4H), 2.41 (dd, \(J = 16.8, 5.9\) Hz, 1H), 2.25 (dd, \(J = 16.8, 8.3\) Hz, 1H), 1.10 (d, \(J = 7.5\) Hz, 3H), 0.94 (s, 6H); \(^{13}\)C NMR (151 MHz, CDCl\(_3\)) \(\delta\) 120.63, 72.15, 31.70, 21.75, 20.39, 15.25. HRMS calcd for C_{9}H_{18}BO_{2}: 181.1274; found: 181.1272. This compound is new.
Part 4. References


Part 5. Copies of $^1$H-NMR and $^{13}$C-NMR spectra

4,4,5,5-Tetramethyl-2-phenethyl-1,3,2-dioxaborolane (3a)
4,4,5,5-Tetramethyl-2-(3-phenylpropyl)-1,3,2-dioxaborolane (3b)
2-Butyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3c)
4,4,5,5-Tetramethyl-2-octyl-1,3,2-dioxaborolane (3d)
2-Isobutyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3e)
4,4,5,5-Tetramethyl-2-pentyl-1,3,2-dioxaborolane (3f)

2-(2-Ethylhexyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3g)
Ethyl 5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) pentanoate (3h)
2-(2-(1,3-Dioxan-2-yl)ethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3i)
4,4,5,5-Tetramethyl-2-(3-phenoxypropyl)-1,3,2-dioxaborolane (3j)
3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)propanenitrile (3k)
2-Benzyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3l)
2-(4-Bromophenethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (3m)
1,3-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)propane (3n)
2-Cycloheptyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5a)
2-Cyclohexyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5b)
2-Cyclopentyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5c)
2-Cyclobutyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5d)
2-((1S,4S)-Bicyclo[2.2.1]heptan-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5e)
2-Adamantyl-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5f)
4,4,5,5-Tetramethyl-2-(1-phenylpropan-2-yl)-1,3,2-dioxaborolane (5g)
3-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl) butanenitrile (5h)
5,5-Dimethyl-2-phenethyl-1,3,2-dioxaborinane (7a)
5,5-Dimethyl-2-(1-phenylpropan-2-yl)-1,3,2-dioxaborinane (7b)
3-(5,5-Dimethyl-1,3,2-dioxaborinan-2-yl) butanenitrile (7c)