Supporting Information

Chemo- and regioselective synthesis of polysubstituted 2-aminothiophenes by the cyclization of gem-dibromo or gem-dichloroalkenes with β-keto tertiary thioamides

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1. General information

All reagents were of analytical grade, and obtained from commercial suppliers and used without further purification. DMSO and other solvents were dried by standard method prior to use. Melting points were measured in an open capillary using Büchi melting point B-540 apparatus and are uncorrected. $^1$H NMR and $^{13}$C NMR spectra were recorded on a 400 spectrometer (400 MHz for $^1$H and 100 MHz for $^{13}$C NMR, respectively) using TMS as internal standard. The GC and GC-MS were recorded on HP 5973 MSD with 6890 GC. High resolution mass spectra (HRMS) were recorded under electron impact conditions using a MicroMass GCT CA 055 instrument and recorded on a MicroMass LCTTM spectrometer. Silica gel (300–400 mesh size) was used for column chromatography. TLC analysis of reaction mixtures was performed using silica gel plates.

2. The gem-dibromoalkenes (1a–l), gem-dichloroalkenes (1m–r) and β-keto tertiary thioamides (2a–g) used in this reaction

(1) The gem-dibromoalkenes (1a–l) used in this reaction

![Dibromoalkenes](image1)

The gem-dibromoalkenes (1a–l) were prepared according to the reported procedure (J. Liu, F. Dai, Z. Yang, S. Wang, K. Xie, A. Wang, X. Chen and Z. Tan, *Tetrahedron Lett.*, 2012, 53, 5678–5683).

(2) The gem-dichloroalkenes (1m–r) used in this reaction

![Dichloroalkenes](image2)

The gem-dichloroalkenes (1m–r) were prepared according to the reported procedure (S. G. Newman, C. S. Bryan, D. Perez and M. Lautens, *Synthesis*, 2011, 342).
(3) The β-keto tertiary thioamides (2a–g) used in this reaction


3. General procedure for the synthesis of compounds 3

A 25 mL of dried round-bottom flask was charged with 1,1-dihaloalkene 1 (1.0 mmol), β-ketothioamide 2 (1.2 mmol), K$_2$CO$_3$ (2.5 mmol, 345.0 mg), and DMSO (5 mL) under argon atmosphere. The mixture was stirred at 120 °C for 4 h (monitored by TLC). After the reaction completed, the reaction mixture was quenched with H$_2$O (20 mL) and extracted with EtOAc (3×10 mL). The combined organic layer was washed with brine (2×10 mL), dried over Na$_2$SO$_4$, and concentrated under reduced pressure. The crude residue was then purified by column chromatography on silica gel to afford the pure target compounds 3.

4. Characterization data of compounds 3

(4-(4-Chlorophenyl)-2-(piperidin-1-yl)thiophen-3-yl)(phenyl)methanone (3aa=3ma): Yellow solid; yield: 90% (3aa), 82% (3ma); mp: 97.0–97.9 °C. $^1$H NMR (400 MHz, CDCl$_3$): δ 7.85 (d, $J = 7.6$ Hz, 2H), 7.53–7.49 (m, 1H), 7.41–7.37 (m, 2H), 7.20–7.15 (m, 4H), 6.70 (s, 1H), 2.91 (t, $J = 5.0$ Hz, 4H), 1.35–1.27 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 194.0, 162.9, 141.0, 137.9, 135.2, 133.0, 129.9, 129.6, 128.3, 128.1, 124.9, 113.1, 55.7, 25.3, 23.5. HRMS (EI) calcd for C$_{22}$H$_{20}$ClN$_2$O$_2$ [M]+: 381.0952, found: 381.0953.
4-(4-Benzoyl-5-(piperidin-1-yl)thiophen-3-yl)benzonitrile (3ba): Yellow solid; yield: 92%; mp: 94.8–95.6 °C. 

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.84 (d, $J = 7.6$ Hz, 2H), 7.55–7.49 (m, 3H), 7.42–7.38 (m, 2H), 7.32 (d, $J = 8.0$ Hz, 2H), 6.76 (s, 1H), 2.92 (t, $J = 5.2$ Hz, 4H), 1.28–1.26 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 193.5, 163.6, 141.4, 140.5, 137.7, 133.1, 132.0, 129.9, 129.0, 128.2, 124.2, 118.9, 114.3, 110.6, 55.6, 25.3, 23.4. HRMS (EI) calcd for C$_{23}$H$_{20}$N$_2$O$_2$S [M$^+$]: 372.1296, found: 372.1298.

(4-([1,1′-Biphenyl]-4-yl)-2-(piperidin-1-yl)thiophen-3-yl)(phenyl)methanone (3ca=3oa): Yellow solid; yield: 88% (3ca), 78% (3oa); mp: 135.6–136.2 °C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.92–7.90 (m, 2H), 7.57–7.47 (m, 5H), 7.43–7.39 (m, 4H), 7.35–7.30 (m, 3H), 6.79 (s, 1H), 2.95 (t, $J = 5.2$ Hz, 4H), 1.37–1.31 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.2, 162.7, 141.8, 140.8, 139.8, 138.1, 135.7, 132.8, 130.0, 128.7, 128.1, 127.2, 127.0, 126.9, 125.4, 113.1, 55.7, 25.4, 23.5. HRMS (EI) calcd for C$_{28}$H$_{25}$NOS [M$^+$]: 424.1683, found: 424.1685.

Phenyl(4-phenyl-2-(piperidin-1-yl)thiophen-3-yl)methanone (3da=3pa): Yellow oil; yield: 80% (3da), 72% (3pa). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.84 (d, $J = 7.2$ Hz, 2H), 7.49–7.45 (m, 1H), 7.38–7.34 (m, 2H), 7.24–7.16 (m, 5H), 6.71 (s, 1H), 2.91 (t, $J = 4.8$ Hz, 4H), 1.34–1.29 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.2, 162.6, 142.2, 138.1, 136.7, 132.7, 129.9, 128.3, 128.2, 128.1, 127.0, 125.4, 112.9, 55.7, 25.4, 23.5. HRMS (EI) calcd for C$_{22}$H$_{21}$NOS [M$^+$]: 347.1347, found: 347.1346.
Phenyl(2-(piperidin-1-yl)-4-(p-tolyl)thiophen-3-yl)methanone (3ea=3qa): Yellow solid; yield: 80% (3ea), 80% (3qa); mp: 85.0–86.0 °C. $^1$H NMR (400 MHz, CDCl$_3$): δ 7.89 (d, $J = 7.6$ Hz, 2H), 7.53–7.50 (m, 1H), 7.42–7.38 (m, 2H), 7.15 (d, $J = 7.6$ Hz, 2H), 7.04 (d, $J = 8.0$ Hz, 2H), 6.72 (s, 1H), 2.93 (t, $J = 4.8$ Hz, 4H), 2.29 (s, 3H), 1.34–1.29 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 194.3, 162.3, 142.1, 138.1, 136.7, 133.8, 132.7, 129.9, 128.9, 128.2, 128.0, 125.6, 112.6, 55.7, 25.4, 23.5, 21.2. HRMS (EI) calcd for C$_{23}$H$_{23}$NOS [M]$^+$: 361.1506, found: 361.1504.

(4-(4-Methoxyphenyl)-2-(piperidin-1-yl)thiophen-3-yl)(phenyl)methanone (3fa=3ra): Yellow solid; yield: 75% (3fa), 50% (3ra); mp: 68.5–69.3 °C. $^1$H NMR (400 MHz, CDCl$_3$): δ 7.87 (d, $J = 7.2$ Hz, 2H), 7.52–7.49 (m, 1H), 7.41–7.37 (m, 2H), 7.18 (d, $J = 8.8$ Hz, 2H), 6.77 (d, $J = 8.8$ Hz, 2H), 6.68 (s, 1H), 3.75 (s, 3H), 2.92 (t, $J = 5.0$ Hz, 4H), 1.31–1.28 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 194.4, 162.3, 158.7, 141.7, 138.1, 132.7, 129.9, 129.4, 129.3, 128.1, 125.5, 113.6, 112.2, 55.7, 55.2, 25.4, 23.5. HRMS (EI) calcd for C$_{23}$H$_{23}$NO$_2$S [M]$^+$: 377.1451, found: 377.1452.

Phenyl(5'-(piperidin-1-yl)-[2,3'-bithiophen]-4'-yl)methanone (3ga): Yellow oil; yield: 85%. $^1$H NMR (400 MHz, CDCl$_3$): δ 7.87 (d, $J = 7.2$ Hz, 2H), 7.53–7.49 (m, 1H), 7.41–7.38 (m, 2H), 7.12–7.10 (m, 1H), 6.92–6.91 (m, 1H), 6.87–6.85 (m, 2H), 2.93 (t, $J = 4.8$ Hz, 4H), 1.35–1.31 (m, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 194.4, 161.8, 138.0, 137.9, 133.7, 132.9, 129.8, 128.1, 127.2, 125.7, 125.2, 124.6, 113.4, 55.6, 25.4, 23.5. HRMS (EI) calcd for C$_{20}$H$_{19}$NOS$_2$ [M]$^+$: 353.0912, found: 353.0910.
(E)-Phenyl(2-(piperidin-1-yl)-4-styrylthiophen-3-yl)methanone (3ha): Yellow oil; yield: 88%. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.86 (d, $J = 7.6$ Hz, 2H), 7.52–7.49 (m, 1H), 7.42–7.37 (m, 4H), 7.27–7.23 (m, 2H), 7.18–7.15 (m, 1H), 7.11 (d, $J = 16.4$ Hz, 1H), 6.91 (d, $J = 16.0$ Hz, 1H), 6.90 (s, 1H), 2.85 (t, $J = 5.2$ Hz, 4H), 1.27–1.26 (m, 2H), 1.17–1.12 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 193.8, 163.8, 138.8, 138.2, 137.5, 132.7, 130.0, 129.6, 128.6, 128.1, 127.5, 126.5, 123.6, 122.8, 109.7, 55.7, 25.2, 23.4. HRMS (EI) calcd for C$_{24}$H$_{23}$NOS [M]$^+$: 373.1500, found: 373.1501.

(4-(4-Chlorophenyl)-2-(dimethylamino)thiophen-3-yl)(phenyl)methanone (3ad=3md): Yellow oil; yield: 86% (3ad), 75% (3md). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.76–7.74 (m, 2H), 7.44–7.40 (m, 1H), 7.31–7.27 (m, 2H), 7.10 (s, 4H), 6.51 (s, 1H), 2.80 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.3, 162.4, 141.3, 138.6, 135.5, 132.8, 132.7, 129.8, 129.6, 128.2, 120.7, 109.8, 45.6. HRMS (EI) calcd for C$_{19}$H$_{16}$ClNOS [M]$^+$: 341.0639, found: 341.0641.

(4-([1,1'-Biphenyl]-4-yl)-2-morpholinothiophen-3-yl)(phenyl)methanone (3cc=3oc): Yellow solid; yield: 88% (3cc), 76% (3oc); mp: 134.5–135.0 °C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.84–7.82 (m, 2H), 7.52–7.43 (m, 5H), 7.40–7.34 (m, 4H), 7.31–7.29 (m, 3H), 6.87 (s, 1H), 3.46 (t, $J = 4.8$ Hz, 4H), 2.98 (t, $J = 4.6$ Hz, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 194.3, 160.6, 141.6, 140.6, 139.9, 138.0, 135.2, 133.0, 129.8, 128.7, 128.6, 128.2, 127.3, 127.2, 127.0, 126.9, 114.1, 66.4, 54.4. HRMS (EI) calcd for C$_{27}$H$_{23}$NO$_2$S [M]$^+$: 425.1450, found: 425.1452.
(4-((1,1′-Biphenyl)-4-yl)-2-(dimethylamino)thiophen-3-yl)(phenyl)methanone (3cd=3od): Yellow solid; yield: 90% (3cd), 81% (3od); mp: 127.1–128.0 °C. $^1$H NMR (400 MHz, CDCl$_3$): δ 7.79–7.77 (m, 2H), 7.48–7.46 (m, 2H), 7.37–7.34 (m, 5H), 7.28–7.22 (m, 5H), 6.57 (s, 1H), 2.79 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 194.6, 162.2, 142.1, 140.7, 139.6, 138.8, 136.0, 132.7, 129.9, 128.7, 128.2, 127.2, 127.0, 126.8, 121.4, 109.9, 45.7. HRMS (EI) calcd for C$_{25}$H$_{21}$NOS [M]+: 383.1344, found: 383.1346.

(4-(4-(Methylthio)phenyl)-2-morpholinothiophen-3-yl)(phenyl)methanone (3ic): Yellow oil; yield: 78%. $^1$H NMR (400 MHz, CDCl$_3$): δ 7.81–7.79 (m, 2H), 7.50–7.48 (m, 1H), 7.39–7.35 (m, 2H), 7.17–7.08 (m, 4H), 6.81 (s, 1H), 3.46 (t, $J$ = 4.6 Hz, 4H), 2.96 (t, $J$ = 4.6 Hz, 4H), 2.41 (s, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 194.3, 160.5, 141.4, 137.9, 137.5, 133.3, 129.7, 128.6, 128.2, 127.1, 126.4, 113.8, 66.4, 54.4, 15.7. HRMS (EI) calcd for C$_{22}$H$_{21}$NO$_2$S$_2$ [M]+: 395.1014, found: 395.1015.

(4-(3-Bromophenyl)-2-(pyrrolidin-1-yl)thiophen-3-yl)(phenyl)methanone (3jb): Yellow oil; yield: 86%. $^1$H NMR (400 MHz, CDCl$_3$): δ 7.65–7.63 (m, 2H), 7.30–7.26 (m, 2H), 7.19–7.16 (m, 2H), 7.12–7.09 (m, 1H), 7.01–6.99 (m, 1H), 6.89–6.85 (m, 1H), 6.26 (s, 1H), 3.19 (t, $J$ = 6.6 Hz, 4H), 1.98–1.94 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 192.8, 159.7, 141.3, 139.8, 139.6, 132.0, 131.4, 129.7, 129.5, 129.2, 127.8, 127.0, 121.8, 114.8, 106.2, 53.3, 26.1. HRMS (EI) calcd for C$_{21}$H$_{18}$BrNOS [M]+: 413.0272, found: 413.0279.
Benzo[d][1,3]dioxol-5-yl(4-(3-bromophenyl)-2-(piperidin-1-yl)thiophen-3-yl)methanone (3je): Yellow oil; yield: 82%. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.43–7.41 (m, 2H), 7.35–7.34 (m, 1H), 7.31–7.29 (m, 1H), 7.11–7.03 (m, 2H), 6.76 (d, $J = 8.4$ Hz, 1H), 6.71 (s, 1H), 6.01 (s, 2H), 2.94 (s, 4H), 1.40 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 192.3, 161.8, 151.7, 147.9, 140.3, 138.7, 132.7, 131.1, 130.0, 129.6, 126.9, 126.8, 124.9, 122.2, 113.3, 109.2, 107.7, 101.8, 55.5, 25.5, 23.6. HRMS (EI) calcd for C$_{23}$H$_{20}$BrNO$_3$S $[M]^+$: 471.0327, found: 471.0332.

(4-(Naphthalen-2-yl)-2-(pyrrolidin-1-yl)thiophen-3-yl)(phenyl)methanone (3kb): Yellow solid; yield: 78%; mp: 109.6–110.6 °C. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.70–7.61 (m, 5H), 7.47 (d, $J = 8.4$ Hz, 1H), 7.37–7.30 (m, 2H), 7.24–7.21 (m, 1H), 7.11–7.01 (m, 3H), 6.35 (s, 1H), 3.19 (t, $J = 6.6$ Hz, 4H), 1.95–1.92 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 193.4, 159.4, 143.0, 139.8, 135.2, 133.0, 132.1, 131.9, 129.8, 127.8, 127.7, 127.5, 127.3, 127.0, 126.8, 125.9, 125.5, 115.3, 106.2, 53.3, 26.1. HRMS (EI) calcd for C$_{25}$H$_{21}$NOS $[M]^+$: 383.1344, found: 383.1346.

(5-Bromo-5'-(dimethylamino)-[2,3'-bithiophen]-4'-yl)(3,4,5-trimethoxyphenyl)methanone (3lf): Yellow oil; yield: 88%. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.08 (s, 2H), 6.75 (d, $J = 4.0$ Hz, 1H), 6.62 (s, 1H), 6.52 (d, $J = 3.6$ Hz, 1H), 3.89 (s, 3H), 3.83 (s, 6H), 2.82 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 193.1, 161.2, 152.8, 142.6, 139.7, 133.5, 133.4, 130.1, 126.0, 119.9, 110.9, 110.4, 107.3, 60.9, 56.3, 45.5. HRMS (EI) calcd for C$_{20}$H$_{20}$BrNO$_3$S$_2$ $[M]^+$: 482.9997, found: 483.0000.
(5-Bromo-5'-morpholino-[2,3'-bithiophen]-4'-yl)(furan-2-yl)methanone (3lg): Yellow oil; yield: 80%. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.57 (d, $J = 4.0$ Hz, 1H), 7.00 (d, $J = 3.6$ Hz, 1H), 6.86 (s, 1H), 6.83 (d, $J = 4.0$ Hz, 1H), 6.67 (d, $J = 3.6$ Hz, 1H), 6.48 (dd, $J = 3.2$, 1.6 Hz, 1H), 3.65 (t, $J = 4.6$ Hz, 4H), 3.05–3.03 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 180.9, 160.7, 153.1, 147.2, 139.1, 132.6, 130.3, 125.9, 125.3, 120.2, 114.3, 112.4, 111.3, 66.5, 54.4. HRMS (EI) calcd for C$_{17}$H$_{14}$BrNO$_3$S$_2$ [M]$^+$: 424.9578, found: 424.9577.

4-(4-Benzoyl-5-(dimethylamino)thiophen-3-yl)benzonitrile (3nd): Yellow oil; yield: 88%. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.74 (d, $J = 8.1$ Hz, 2H), 7.42–7.40 (m, 3H), 7.31–7.25 (m, 4H), 6.56 (s, 1H), 2.82 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 193.7, 163.0, 141.7, 140.8, 138.6, 133.0, 131.8, 129.7, 128.9, 128.3, 119.7, 118.9, 110.8, 110.4, 45.5. HRMS (EI) calcd for C$_{20}$H$_{16}$N$_2$OS [M]$^+$: 332.0436, found: 332.0431.
5. $^1$H, $^{13}$C NMR and HRMS (EI) spectra of compounds 3

$^1$H NMR spectrum of 3aa (3ma)

$^{13}$C NMR spectrum of 3aa (3ma)
HRMS (EI) of 3aa (3ma)

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\text{TCP MS EI} \\
\end{array} \]

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350.0957 \\
330.1220 \\
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195.0215 \\
172.0479 \\
158.0190 \\
117.0096 \\
91.0544 \\
77.0389 \\
51.0337 \\
0 \\
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$^1$H NMR spectrum of 3ba

![Chemical structure of 3ba](image)

![NMR spectrum of 3aa](image)
$^{13}$C NMR spectrum of 3ba

HRMS (EI) of 3ba
$^1$H NMR spectrum of 3ca (3oa)

$^{13}$C NMR spectrum of 3ca (3oa)
HRMS (EI) of 3ca (3oa)

\[ \text{Waters GCT Premier} \]

\[ \begin{array}{c}
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\text{Cm (550-13+519)} \\
\end{array} \]

\[ \text{m/z} \]

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106.0385 \\
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234.0505 \\
263.0553 \\
278.1029 \\
318.1303 \\
339.0594 \\
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\[ \text{HH} \] NMR spectrum of 3da (3pa)

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\text{3.91} \\
\text{2.92} \\
\text{2.36} \\
\text{1.35} \\
\end{array} \]

\[ \text{Ph} \]
$^{13}$C NMR spectrum of 3da (3pa)

HRMS (EI) of 3da (3pa)
$^1$H NMR spectrum of 3ea (3qa)

$^{13}$C NMR spectrum of 3ea (3qa)
HRMS (EI) of 3ea (3qa)

\[ S \]

$^1$H NMR spectrum of 3fa (3ra)
$^{13}$C NMR spectrum of 3fa (3ra)

HRMS (EI) of 3fa (3ra)
$^1$H NMR spectrum of 3ga

$^{13}$C NMR spectrum of 3ga
HRMS (EI) of 3ga

$^1$H NMR spectrum of 3ha
$^{13}$C NMR spectrum of 3ha

HRMS (EI) of 3ha
$^1$H NMR spectrum of 3ad (3md)

$^{13}$C NMR spectrum of 3ad (3md)
HRMS (EI) of 3ad (3md)

1H NMR spectrum of 3cc (3oc)
$^{13}$C NMR spectrum of 3cc (3oc)

HRMS (EI) of 3cc (3oc)
$^1$H NMR spectrum of 3cd (3od)

$^{13}$C NMR spectrum of 3cd (3od)
HRMS (EI) of 3cd (3od)

$^1$H NMR spectrum of 3ic
$^{13}$C NMR spectrum of 3ic

[Image of $^{13}$C NMR spectrum]

HRMS (EI) of 3ic

[Image of HRMS (EI) spectrum]
$^1$H NMR spectrum of 3jb

$^{13}$C NMR spectrum of 3jb
HRMS (EI) of 3jb

^1H NMR spectrum of 3je
$^{13}$C NMR spectrum of 3je

HRMS (EI) of 3je
$^1$H NMR spectrum of 3kb

$^{13}$C NMR spectrum of 3kb
HRMS (EI) of 3kb

\(^1\)H NMR spectrum of 3lf
$^{13}$C NMR spectrum of 3lf

HRMS (EI) of 3lf
$^1$H NMR spectrum of 3lg

$^{13}$C NMR spectrum of 3lg
HRMS (EI) of 3lg

$^1$H NMR spectrum of 3nd
\(^{13}\)C NMR spectrum of 3nd
6. X-ray crystal structure of compound 3ba

![Figure S1. X-ray crystal structure of 3ba.](image)

Single crystals of 3ba (yellowish prisms) suitable for diffraction study were obtained by slow diffusion of hexane into a dichloromethane solution of compound 3ba. X-ray crystallographic data for compound 3ba (CCDC 1434924) has been deposited in the Cambridge Crystallographic Data Center. The data can be obtained free of charge via [http://www.ccdc.ac.ck./data_request/cif](http://www.ccdc.ac.ck./data_request/cif). The X-ray crystallography data of 3ba are listed below.

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<tr>
<td>Unit cell dimensions</td>
<td>a = 7.7469(8) Å, b = 17.4348(19) Å, c = 14.3624(16) Å</td>
</tr>
<tr>
<td></td>
<td>a= 90°, b= 92.609(2)°, g = 90°</td>
</tr>
<tr>
<td>Volume</td>
<td>1937.9(4) Å³</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
</tr>
<tr>
<td>Density (calculated)</td>
<td>1.277 Mg/m³</td>
</tr>
<tr>
<td>Absorption coefficient</td>
<td>0.182 mm⁻¹</td>
</tr>
<tr>
<td>F(000)</td>
<td>784</td>
</tr>
<tr>
<td>Crystal size</td>
<td>0.220 x 0.170 x 0.130 mm³</td>
</tr>
<tr>
<td>Theta range for data collection</td>
<td>1.838 to 25.998°</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-9&lt;=h&lt;=9, -21&lt;=k&lt;=20, -17&lt;=l&lt;=9</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>11485</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>3825 [R(int) = 0.0353]</td>
</tr>
<tr>
<td>Completeness to theta</td>
<td>25.242°</td>
</tr>
<tr>
<td>Absorption correction</td>
<td>Semi-empirical from equivalents</td>
</tr>
<tr>
<td></td>
<td>S37</td>
</tr>
<tr>
<td>Description</td>
<td>Value</td>
</tr>
<tr>
<td>--------------------------------------------------</td>
<td>--------------------------------</td>
</tr>
<tr>
<td>Max. and min. transmission</td>
<td>0.7457 and 0.6382</td>
</tr>
<tr>
<td>Refinement method</td>
<td>Full-matrix least-squares on $F^2$</td>
</tr>
<tr>
<td>Data / restraints / parameters</td>
<td>3825 / 0 / 244</td>
</tr>
<tr>
<td>Goodness-of-fit on $F^2$</td>
<td>1.044</td>
</tr>
<tr>
<td>Final R indices [$I &gt; 2\sigma(I)$]</td>
<td>$R_1 = 0.0540$, $wR_2 = 0.1297$</td>
</tr>
<tr>
<td>R indices (all data)</td>
<td>$R_1 = 0.0731$, $wR_2 = 0.1401$</td>
</tr>
<tr>
<td>Extinction coefficient</td>
<td>n/a</td>
</tr>
<tr>
<td>Largest diff. peak and hole</td>
<td>0.246 and -0.191 eÅ$^{-3}$</td>
</tr>
</tbody>
</table>