



SUPPLEMENTARY MATERIAL TO

Antibacterial and antifungal properties of guanyldiazones

VLADIMIR AJDAČIĆ^{1#}, JELENA LAZIĆ^{1#}, MARIJA MOJIĆEVIĆ², SANDRA ŠEGAN³,
JASMINA NIKODINOVIC-RUNIC^{2**} and IGOR M. OPSENICA^{1***}

¹Faculty of Chemistry, University of Belgrade, Studentski trg 16, P. O. Box 51, 11158, Belgrade, Serbia, ²Institute of Molecular Genetics and Genetic Engineering, University of Belgrade, Vojvode Stepe 444a, 11000 Belgrade, Serbia and ³Institute of Chemistry, Technology, and Metallurgy, University of Belgrade, Njegoševa 12, 11000 Belgrade, Serbia

J. Serb. Chem. Soc. 82 (6) (2017) 641–649

HPLC ANALYSIS OF PURITY

The compounds were analyzed for purity (HPLC) using a Agilent 1200 HPLC system equipped with Quat pump (G1311B), injector (G1329B) 1260 ALS, TCC 1260 (G1316A) and detector 1260 DAD VL+ (G1315C). HPLC analyses were performed in diverse systems:

Method A. Zorbax Eclipse Plus C18 4.6 mm×150 mm, 1.8 μm, S.N. USWKY01594 was used as the stationary phase. The eluent was made from the following solvents: 0.2 % formic acid in water (A) and acetonitrile (B). The flow rate was 0.5 mL min⁻¹. The analyses were performed at the UV maximum of the compounds to maximize selectivity. The compounds were dissolved in methanol, the final concentrations were ≈1 mg mL⁻¹.

Compounds **22–24** were eluted using the gradient protocol: 0–0.5 min, 95 % A; 0.5–3 min, 95 % A→5 % A; 3–13 min, 5 % A; 13–14 min, 5 % A→95 % A; 14–16 min, 95 % A.

Method B. Zorbax Eclipse Plus C18 4.6 mm×150 mm, 1.8 μm, S.N. USWKY01594 was used as the stationary phase. The eluents were made from the following solvents: 0.2 % formic acid in water (A) and methanol (B). The flow rate was 0.5 mL min⁻¹. The analyses were performed at the UV max of the compounds to maximize the selectivity. The compounds were dissolved in methanol, the final concentrations were ≈1 mg mL⁻¹.

Compounds **22–24** were eluted using the gradient protocol: 0–0.5 min, 95 % A; 0.5–3 min, 95 % A→5 % A; 3–13 min, 5 % A; 13–14 min, 5 % A→95 % A; 14–16 min 95 % A.

Method C. Zorbax Eclipse Plus C18 2.1 mm×100 mm, 1.8 μm, was used as the stationary phase. The eluent was made from the following solvents: 0.2 % formic acid in water (A) and acetonitrile (B). The analyses were performed at the UV max of the compounds to maximize selectivity. Compounds were dissolved in methanol. The final concentrations were ≈1 mg mL⁻¹. The flow rate was 0.2 mL min⁻¹.

Compounds **18–21** and **25–27** were eluted using the gradient protocol: 0–0.5 min 95 % A, 0.5–3 min 95 % A→5 % A, 3–13 min 5 % A, 13–14 min 5 % A→95 % A, 14–16 min 95 % A.

*** Corresponding authors. E-mail: (*)jasmina.nikodinovic@imgge.bg.ac.rs;

(**)igorop@chem.bg.ac.rs

Method D. Zorbax Eclipse Plus C18 2.1 mm×100 mm, 1.8 μm, was used as the stationary phase. Eluent was made from the following solvents: 0.2 % formic acid in water (A) and methanol (B). The analyses were performed at the UV max of the compounds to maximize selectivity. Compounds were dissolved in methanol. The final concentrations were ≈1 mg mL⁻¹. The flow rate was 0.2 mL min⁻¹.

Compounds **18–21** and **25–27** were eluted using the gradient protocol: 0–0.5 min 95 % A, 0.5–3 min 95 % A→5 % A, 3–13 min 5 % A, 13–14 min 5 % A→95 % A, 14–16 min 95 % A.

ANALYTICAL AND SPECTRAL DATA OF THE COMPOUNDS

5-(4-Methylphenyl)furan-2-carbaldehyde (4).¹ Orange amorphous powder; m.p.: 49–51 °C; IR (ATR, cm⁻¹): 3308w, 3128w, 3026w, 2915w, 2859w, 2824w, 1657s, 1607m, 1528m, 1482m, 1416w, 1387w, 1291w, 1255m, 1203w, 1118w, 1028m, 964w, 921w, 822w; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.63 (1H, s), 7.72 (2H, d, *J* = 8.2 Hz), 7.31 (1H, d, *J* = 3.7 Hz), 7.27 (1H, s), 7.23 (1H, s), 6.79 (1H, d, *J* = 3.7 Hz), 2.39 (3H, s); GC/MS (*m/z* (%)): 186.0 ([M]⁺, 100), 129.0 (70).

5-(4-Bromophenyl)furan-2-carbaldehyde (5).² Orange amorphous powder; m.p.: 151–152 °C; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.66 (1H, s), 7.72–7.65 (2H, m), 7.61–7.55 (2H, m), 7.31 (1H, d, *J* = 3.7 Hz), 6.84 (1H, d, *J* = 3.7 Hz); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 177.22, 158.21, 152.17, 132.20, 127.88, 126.69, 123.93, 123.39, 108.04; GC/MS (*m/z* (%)): 251.9 ([M]⁺, 100), 192.9 (40).

5-(4-Fluorophenyl)furan-2-carbaldehyde (6).³ Yellow solid; m.p.: 70–71 °C; IR (ATR, cm⁻¹): 3315m, 3135m, 3103m, 2918m, 2850m, 1666s, 1602s, 1567m, 1482s, 1420s, 1392m, 1356m, 1304w, 1286m, 1254m, 1227s, 1157m, 1102m, 1065w, 1024m, 966m, 922m, 889w, 834m, 813s; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.65 (1H, s), 7.86–7.82 (1H, m), 7.81–7.77 (1H, m), 7.32 (1H, d, *J* = 3.8 Hz), 7.20–7.09 (2H, m), 6.79 (1H, d, *J* = 3.7 Hz); GC/MS (*m/z* (%)): 190.0 ([M]⁺, 100).

5-(4-Methoxyphenyl)furan-2-carbaldehyde (7).³ Orange oil; IR (ATR, cm⁻¹): 3318w, 3214.8w, 3118w, 3004w, 1937m, 2838m, 2733w, 2552w, 1733w, 1668s, 1609s, 1530wm, 1481s, 1389m, 1296m, 1254s, 1177m, 1114m, 1065wm, 1026m, 967m, 921w, 835m; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.60 (1H, s), 7.77 (2H, d, *J* = 9.0 Hz), 7.31 (1H, d, *J* = 4.3 Hz), 6.96 (2H, d, *J* = 9.0 Hz), 6.72 (1H, d, *J* = 3.7 Hz), 3.86 (3H, s); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 176.85, 160.88, 159.81, 151.59, 126.96, 124.19, 114.40, 106.28, 95.75, 55.33; GC/MS (*m/z* (%)): 202.0 ([M]⁺, 100), 187.0 (40), 145.0 (40).

4-Bromo-5-phenylthiophene-2-carbaldehyde (10).⁴ Yellow solid; m.p.: 57–60 °C; IR (ATR, cm⁻¹): 3310w, 3082w, 3053w, 3026w, 2845w, 1678s, 1645s, 1519w, 1449m, 1430m, 1394w, 1309w, 1226m, 1122w, 1031w, 997w, 966w, 915w, 842w, 755w; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.86 (1H, s), 7.72 (1H, s), 7.70–7.67 (2H, m), 7.50–7.45 (3H, m); ¹³C-NMR (125 MHz, CDCl₃,

δ / ppm): 181.79, 148.06, 141.34, 139.82, 131.82, 129.68, 129.01, 128.80, 108.78; GC/MS (m/z): 267.9 [M]⁺.

4-Bromo-5-phenyl-2-furaldehyde (11).⁵ Dark oil; IR (ATR, cm⁻¹): 3341w, 3132w, 2834w, 1682s, 1566w, 1521w, 1476m, 1284w, 1140w; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.64 (1H, s), 8.15–8.05 (2H, m), 7.52–7.41 (3H, m), 7.34 (1H, s); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 176.97, 153.96, 150.46, 130.04, 128.74, 128.17, 126.76, 125.72, 98.23; GC/MS (m/z): 249.9 [M]⁺.

4,5-Diphenylthiophene-2-carbaldehyde (12). Yellow oil; IR (ATR, cm⁻¹): 2919m, 2851m, 1734w, 1657s, 1542w, 1452w, 1427m, 1253w, 1165m, 1106w, 1071w; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.92 (1H, s), 7.80 (1H, s), 7.34–7.29 (8H, m), 7.28–7.25 (2H, m); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 182.83, 148.74, 141.41, 139.55, 139.08, 135.16, 133.10, 129.19, 128.97, 128.87, 128.72, 128.64, 127.65; GC/MS (m/z (%)): 264.0 ([M]⁺, 100), 235.0 (50).

4-(4-Fluorophenyl)-5-phenylthiophene-2-carbaldehyde (13). Yellow oil; IR (ATR, cm⁻¹): 3318w, 3058w, 2926w, 2819w, 1670s, 1604w, 1543w, 1507m, 1434m, 1259w, 1226m, 1175m, 1159w, 1109w, 1072w; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.91 (1H, s), 7.76 (1H, s), 7.37–7.28 (5H, m), 7.25–7.20 (2H, m), 7.04–7.97 (2H, m); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 182.72, 162.27 (*d*, *J* = 245.5 Hz), 148.69, 141.46, 138.72, 138.42, 132.89, 131.15 (*d*, *J* = 2.7 Hz), 130.63 (*d*, *J* = 7.1 Hz), 129.15, 128.97, 128.81, 115.66 (*d*, *J* = 21.7 Hz); GC/MS (m/z (%)): 282.0 ([M]⁺, 100), 253.0 (30).

2-(4-Bromo-5-phenyl-2-furyl)-1,3-dioxolane (14). Yellow oil; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.98–7.94 (2H, m), 7.44–7.39 (2H, m), 7.35–7.31 (1H, m), 6.57 (1H, s), 5.96 (1H, s), 4.17–4.09 (2H, m), 4.07–3.95 (2H, m); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 150.46, 149.25, 129.41, 128.42, 128.23, 125.72, 114.51, 97.43, 96.12, 65.18.

4-Fluoro-5-phenyl-2-furaldehyde (15). Yellow solid; IR (ATR, cm⁻¹): 3188w, 3115w, 3067w, 2847w, 1683s, 1607m, 1528m, 1435m, 1314m, 1164w, 1132w, 964w; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 9.61 (1H, s), 7.88–7.85 (2H, m), 7.50–7.46 (2H, m), 7.43–7.38 (1H, m), 7.20–7.19 (1H, m); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 177.60, 149.49 (*d*, *J* = 255.5 Hz), 147.46 (*d*, *J* = 6.4 Hz), 142.68, 142.52, 129.50, 129.00, 127.20 (*d*, *J* = 4.5 Hz), 125.05 (*d*, *J* = 5.4 Hz); GC/MS (m/z): 190.0 [M]⁺.

(4-Nitro-5-phenyl-2-thienyl)methylene diacetate (16). Yellow oil; IR (ATR, cm⁻¹): 3108w, 3063w, 3025w, 2937w, 1769s, 1558m, 1528s, 1505m, 1372m, 1338m, 1225s, 1193s, 1129m, 1070w, 1006m; ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.81 (1H, s), 7.78 (1H, s), 7.50–7.42 (5H, m), 2.17 (6H, s); ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 168.24, 146.35, 141.73, 136.14, 129.93, 129.89, 129.54, 128.50, 124.20, 85.35, 20.61; (+)ESI-HRMS (m/z): [M+Na]⁺ 358.03576 (error: 0.5 ppm).

4-Nitro-5-phenylthiophene-2-carbaldehyde (17). GC/MS (m/z): 233.0 [M]⁺.

(2E)-2-*l*-[5-(4-methylphenyl)furan-2-yl]methylidene}hydrazinecarboximidamide hydrochloride (**18**). Yellow solid; m.p.: 84–87 °C; IR (ATR, cm⁻¹): 3573w, 3433m, 3281s, 3217s, 3111s, 3001m, 1682s, 1635s, 1602s, 1528m, 1492m, 1426m, 1372w, 1333w, 1296w, 1268w, 1194w, 1139m, 1027m, 966w, 937m, 822w; ¹H-NMR (500 MHz, CD₃OD, δ / ppm): 8.00 (1H, s), 7.68 (2H, d, *J* = 8.2 Hz), 7.24 (2H, d, *J* = 8.0 Hz), 7.00 (1H, d, *J* = 3.5 Hz), 6.86 (1H, d, *J* = 3.5 Hz), 2.36 (3H, s); ¹³C-NMR (125 MHz, CD₃OD, δ / ppm): 158.31, 156.92, 149.11, 139.95, 138.73, 130.58, 128.45, 125.50, 118.46, 108.01, 21.35; (+)ESI-HRMS (*m/z*): [M+H]⁺ 243.12366 (error: -1.53 ppm). The compound was >95 % pure based on HPLC purity analysis.

(2E)-2-*l*-[5-(4-Methoxyphenyl)furan-2-yl]methylidene}hydrazinecarboximidamide hydrochloride (**19**). Yellow solid; m.p.: 174–177 °C; IR (ATR, cm⁻¹): 3405s, 3167m, 2932m, 2863m, 1676s, 1635s, 1494s, 1441m, 1294m, 1251m, 1175m, 1114w, 1020m, 967w, 923w, 831m; ¹H-NMR (500 MHz, CD₃OD, δ / ppm): 7.97 (1H, s), 7.71 (2H, d, *J* = 8.6 Hz), 6.96 – 6.95 (3H, m), 6.75 (1H, d, *J* = 3.3 Hz), 3.80 (3H, s); ¹³C-NMR (125 MHz, D₂O, δ / ppm): 161.43, 158.08, 156.65, 148.54, 138.52, 126.86, 123.72, 118.48, 115.16, 106.90, 55.62; (+)ESI-HRMS (*m/z*): [M+H]⁺ 259.11951 (error: 2.15 ppm). The compound was >95 % pure based on HPLC purity analysis.

(2E)-2-*l*-[5-(4-Fluorophenyl)furan-2-yl]methylidene}hydrazinecarboximidamide hydrochloride (**20**). Orange solid; m.p.: 197–199 °C; IR (ATR, cm⁻¹): 3060m, 2998m, 2927m, 2857m, 2775m, 1668s, 1615s, 1531s, 1484s, 1445s, 1334m, 1304m, 1270m, 1214s, 1158s, 1136s, 1019m, 924m, 836m; ¹H-NMR (500 MHz, CD₃OD, δ / ppm): 8.01 (1H, s), 7.85–7.82 (2H, m), 7.18–7.15 (2H, m), 7.02 (1H, d, *J* = 3.6 Hz), 6.91 (1H, d, *J* = 3.6 Hz); ¹³C-NMR (125 MHz, D₂O, δ / ppm): 164.25 (d, *J* = 246.3 Hz), 157.01, 156.96, 149.53, 138.58, 127.70 (d, *J* = 3.5 Hz), 127.61 (d, *J* = 8.1 Hz), 118.33, 116.88 (d, *J* = 21.6 Hz), 108.53; (+)ESI-HRMS (*m/z*): [M+H]⁺ 307.01822 (error: -2.22 ppm). The compound was >95 % pure based on HPLC purity analysis.

(2E)-2-*l*-[5-(4-Bromophenyl)furan-2-yl]methylidene}hydrazinecarboximidamide hydrochloride (**21**). Orange solid; m.p.: 99–101 °C; IR (ATR, cm⁻¹): 3590m, 3320s, 1689s, 1638s, 1476m, 1406w, 1338w, 1272w, 1206w, 1155m, 1073w, 1032w, 1007w, 972w, 827w; ¹H-NMR (500 MHz, CD₃OD, δ / ppm): 8.00 (1H, s), 7.74–7.72 (2H, m), 7.59–7.57 (2H, m), 7.03 (1H, d, *J* = 3.6 Hz), 6.98 (1H, d, *J* = 3.6 Hz); ¹³C-NMR (125 MHz, D₂O, δ / ppm): 156.98, 156.73, 149.85, 138.49, 133.15, 130.21, 127.13, 123.34, 118.22, 109.39; (+)ESI-HRMS (*m/z*): [M+H]⁺ 307.01822 (error: -2.22 ppm). The compound was >95 % pure based on HPLC purity analysis.

(2E)-2-*l*-(4-Fluoro-5-phenyl-furan-2-yl)methylidene}hydrazinecarboximidamide hydrochloride (**22**). Yellow solid; m.p.: 97–101 °C; IR (ATR, cm⁻¹): 3408s, 1694w, 1631m, 1493w, 1432w, 1168w; ¹H-NMR (500 MHz, CD₃OD, δ / ppm):

7.95 (1H, *s*), 7.79–7.76 (2H, *m*), 7.48–7.44 (2H, *m*), 7.37–7.32 (1H, *m*), 7.08 (1H, *s*); ^{13}C -NMR (125 MHz, CD_3OD , δ / ppm): 157.06, 151.29 (*d*, $J = 252.0$ Hz), 146.69, 146.62, 138.26 (*d*, $J = 2.7$ Hz), 130.07, 129.47, 129.26, 125.13 (*d*, $J = 4.5$ Hz), 107.69 (*d*, $J = 20.7$ Hz); (+)ESI-HRMS (m/z): $[\text{M}+\text{H}]^+$ 247.09872 (error: -0.98 ppm). The compound was $>95\%$ pure based on HPLC purity analysis.

(2E)-2-[(4-Bromo-5-phenyl-2-thienyl)methylidene]hydrazinecarboximidamide hydrochloride (**23**). Yellow solid; m.p.: 186–190 °C; IR (ATR, cm^{-1}): 3391s, 2508s, 1679m, 1623s, 1530w, 1456w, 1300w, 1248w, 1149w; ^1H -NMR (500 MHz, CD_3OD , δ / ppm): 8.24 (1H, *s*), 7.70–7.65 (2H, *m*), 7.50–7.41 (4H, *m*); ^{13}C -NMR (125 MHz, CD_3OD , δ / ppm): 157.05, 143.04, 142.92, 138.40, 136.44, 133.77, 130.34, 130.11, 129.98, 108.91; (+)ESI-HRMS (m/z): $[\text{M}+\text{H}]^+$ 322.99557 (error: -1.50 ppm). The compound was $>95\%$ pure based on HPLC purity analysis.

(2E)-2-[(4-Bromo-5-phenyl-2-furyl)methylidene]hydrazinecarboximidamide hydrochloride (**24**). Yellow solid; m.p.: 99–102 °C; IR (ATR, cm^{-1}): 3361s, 3168s, 2878m, 1682s, 1629s, 1479w, 1445w, 1340w, 1249w, 1153w, 1073w, 1024w, 981w, 955w, 926w, 813w; ^1H -NMR (500 MHz, CD_3OD , δ / ppm): 8.07–8.04 (2H, *m*), 7.99 (1H, *s*), 7.51–7.46 (2H, *m*), 7.45–7.40 (1H, *m*), 7.16 (1H, *s*); ^{13}C -NMR (125 MHz, CD_3OD , δ / ppm): 157.06, 152.32, 149.22, 137.71, 130.25, 129.79, 127.15, 127.68, 120.65, 98.98; (+)ESI-HRMS (m/z): $[\text{M} + \text{H}]^+$ 307.01797 (error: -3.03 ppm). The compound was $>95\%$ pure based on HPLC purity analysis.

(2E)-2-[(4-Nitro-5-phenyl-2-thienyl)methylidene]hydrazinecarboximidamide hydrochloride (**25**). Yellow solid; m.p.: 198–202 °C; IR (ATR, cm^{-1}): 3391s, 3275s, 1691s, 1662s, 1621s, 1543m, 1521m, 1398w, 1331m, 1157w, 1015w; ^1H -NMR (500 MHz, CD_3OD , δ / ppm): 8.29 (1H, *s*), 7.99 (1H, *s*), 7.55–7.45 (5H, *m*); ^{13}C -NMR (125 MHz, CD_3OD , δ / ppm): 156.95, 148.49, 143.99, 142.18, 137.08, 131.69, 131.15, 130.56, 129.98, 128.38; (+)ESI-HRMS (m/z): $[\text{M}+\text{H}]^+$ 209.07008 (error: -1.85 ppm). The compound was $>95\%$ pure based on HPLC purity analysis.

(2E)-2-[(4,5-Diphenyl-2-thienyl)methylidene]hydrazinecarboximidamide hydrochloride (**26**). Yellow solid; m.p.: 208–210 °C; IR (ATR, cm^{-1}): 3271s, 3169s, 2324m, 1680s, 1620s, 1258m, 1492m, 1423m, 1314m, 1272m, 1233m, 1195m, 1106m, 1071m, 1012m; ^1H -NMR (500 MHz, CD_3OD , δ / ppm): 8.31 (1H, *s*), 7.50 (1H, *s*), 7.32–7.23 (10H, *m*); ^{13}C -NMR (125 MHz, CD_3OD , δ / ppm): 156.81, 143.96, 143.57, 140.25, 137.34, 137.03, 135.74, 135.00, 130.23, 130.04, 129.73, 129.60, 129.37, 128.48; (+)ESI-HRMS (m/z): $[\text{M}+\text{H}]^+$ 321.11597 (error: -2.73 ppm). The compound was $>95\%$ pure based on HPLC purity analysis.

(2E)-2-[[4-(4-Fluorophenyl)-5-phenyl-2-thienyl]methylidene]hydrazinecarboximidamide hydrochloride (**27**). Yellow solid; m.p.: 102–108 °C; IR (ATR,

cm⁻¹): 3158s, 1674s, 1622s, 1508s, 1435m, 1255m, 1193m, 1157m; ¹H-NMR (500 MHz, CD₃OD, δ / ppm): 8.29 (1H, s), 7.50 (1H, s), 7.31–7.23 (7H, m), 7.05–7.00 (2H, m); ¹³C-NMR (125 MHz, CD₃OD, δ / ppm): 163.61 (*d*, *J* = 243.6 Hz), 156.84, 143.87, 143.65, 139.11, 137.47, 135.54, 134.85, 133.24 (*d*, *J* = 3.6 Hz), 131.94 (*d*, *J* = 8.1 Hz), 130.25, 129.84, 129.50, 116.38 (*d*, *J* = 21.6 Hz); (+)ESI-HRMS (*m/z*): [M+H]⁺ 339.10657 (error: -2.52 ppm). The compound was >95 % pure based on HPLC purity analysis.

REFERENCES

1. T. Hosoya, H. Aoyama, T. Ikemoto, Y. Kihara, T. Hiramatsu, M. Endo, M. Suzuki, *Bioorg. Med. Chem.* **11** (2003) 663
2. J. C. Bussolari, D. C. Reborn, *Org. Lett.* **1** (1991) 965
3. M. Oliveira, I. Serrano, *Frontiers in Antimicrobial Agents: The Challenging of Antibiotic Resistance in the Development of New Therapeutics*, Bentham Science Publ., Sharjah, UAE, 2015
4. V. Ajdačić, S. Stepanović, M. Zlatović, M. Gruden, I. M. Opsenica, *Synthesis* **48** (2016) 4423
5. K. L. Milkiewicz, D. J. Parks, T. Lu, *Tetrahedron Lett.* **44** (2013) 4257.