One-pot, Three-component Synthesis of Fully Substituted 1,3,4-Oxadiazole

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Supplementary Information

One-pot, Three-component Synthesis of Fully Substituted 1,3,4-Oxadiazole Derivatives from (*N*-isocyanoimino)triphenylphosphorane, Aromatic Carboxylic acids and (1*R*)-(-)-Campherchinon

Ali Ramazani,* Fatemeh Zeinali Nasrabadi, Behnaz Abdian, and Morteza Rouhani*

Chemistry Department, Zanjan University, P.O. Box 45195-313, Zanjan, Iran. *E-mail: aliramazani@gmail.com [†]Young Researchers Club, Zanjan Branch, Islamic Azad University, Zanjan, Iran Received October 28, 2011, Accepted November 29, 2011



Scheme 1. Three-component synthesis of sterically congested 2,5-disubstituted 1,3,4-oxadiazoles 4 (see Table 1).

	ArCOOH	Product	Yield ^a (%)
1	C ₆ H ₅ COOH		90
2	4-ClC ₆ H ₄ COOH		88
3	4-MeOC ₆ H ₄ COOH		87
4	4- BrCH ₂ C ₆ H ₄ COOH		88
5	3,4-diMeC ₆ H ₄ COOH	CH ₃ 4e CH ₃	86
6	4- t-BuC ₆ H ₄ COOH	4f H ₃ C CH ₃	87

Table 1. Synthesis of sterically congested 1,3,4-oxadiazole derivatives 4a-o from (1R)-(-)-campherchinon 2 and (N-isocyanimino)-triphenylphosphorane 3 in the presence of carboxylic acid 1 (See Scheme 1)

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Table	1.	Continued
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	ArCOOH	Product	Yield ^a (%)
7	4-MeC ₆ H ₄ COOH	HILL OF CH3	86
8	4- BrC ₆ H ₄ COOH		87
9	4- FC ₆ H ₄ COOH		85
10	3-MeC ₆ H ₄ COOH		89
11	C ₁₀ H ₇ COOH		86
12	3-ClC ₆ H ₄ COOH		88
13	2-MeC ₆ H ₄ COOH	OH N N CH3	87
14	3-PhOC ₆ H ₄ COOH		86
15	3,5-diMeOC₀H₃COOH		88

3-Hydroxyl-1,7,7-trimethyl-3-(5-phenyl)-1,3,4-oxadiazol-2-yl]bicyclo[2.2.1]heptan-2-one (4a). White powder, yield: 90%, mp 173-175°. IR (KBr): 3283 (OH), 2961, 2928, 1762, 1606, 1548, 1485, 1088, 785, 690 cm⁻¹; ¹H NMR & 7.50-8.03 (m, 5H, CH_{arom}), 3.65 (s, 1H, OH), 2.52 (s, 1H, CH), 1.68-1.91 (m, 4H, 2CH₂), 1.05 (s, 3H, CH₃), 1.16 (s, 6H, 2CH₃). ¹³C NMR & 213.00 (C=O), 166.76, 165.54 (2C=N), 131.95, 129.01, 127.10 (5CH), 123.21 (C), 77.65 (C-OH), 58.29 (C), 52.79 (CH), 46.22 (C), 27.96, 23.29 (2CH₂), 21.92, 20.42, 9.45 (3CH₃). MS *m/z* (%) 312 (M⁺, 48), 269 (12), 241 (20), 202 (92), 187 (100),147 (28), 105 (40), 83 (44), 77 (48), 55 (56), 41 (32). Anal. Calcd for C₁₈H₂₀N₂O₃ (312.15): C 69.21, H 6.45, N 8.97. Found: C 69.15, H 6.51, N 9.03.

3-[5-(4-Chlorophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (4b). White powder, yield: 88%, mp 127-129°. IR (KBr): 3354 (OH), 2960, 2928, 1762, 1606, 1547, 1458, 1100, 843 cm⁻¹; ¹H NMR δ 7.95 (d, ³*J*_{HH} = 6.5 Hz, 2H, CH_{arom}), 7.45 (d, ³*J*_{HH} = 6.5 Hz, 2H, CH_{arom}), 3.75 (s, 1H, OH), 2.51 (m, 1H, CH), 1.73-1.91 (m, 4H, 2CH₂), 1.04 (s, 3H, CH₃), 1.16 (s, 6H, 2CH₃). ¹³C NMR δ 213.01 (C=O), 165.74, 164.24 (2C=N), 138.30 (C), 129.40, 128.37 (4CH), 121.88 (C), 77.60 (C-OH), 58.29 (C), 52.75 (CH), 46.23 (C), 27.93, 23.31 (2CH₂), 9.43, 20.42, 21.93 (3CH₃). MS *m/z* (%) 346 (M⁺, 32), 249 (20), 236 (48), 221 (64), 207 (32), 167 (28), 156 (36), 149 (96), 139 (100), 111 (56), 95 (48), 83 (68), 69 (56), 55 (72), 41 (64). Anal. Calcd for C₁₈H₁₉ClN₂O₃ (346.11): C 62.34, H 5.52, N 8.08. Found: C 62.39, H 5.47, N 8.03.

3-Hydroxyl-3-[5-(4-methoxyphenyl)-1,3,4-oxadiazol-2yl]-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (4c). White powder, yield 87%, mp 177-179°. IR (KBr): 3275 (OH), 2963, 2934, 1765, 1616, 1503, 1455, 1084, 837 cm⁻¹; ¹H NMR δ 7.92 (d, ${}^{3}J_{\rm HH} = 8.75$ Hz, 2H, CH_{arom}), 6.94 (d, ${}^{3}J_{\rm HH} =$ 8.75 Hz, 2H, CHarom), 4.00 (s, 1H, OH), 3.85 (s, 3H, OCH₃), 2.46 (m, 1H, CH), 1.60-1.94 (m, 4H, 2CH₂), 1.03 (s, 3H, CH₃), 1.04 (s, 3H, CH₃), 1.16 (s, 3H, CH₃). ¹³C NMR δ 213.08 (C=O), 165.18, 165.07 (2C=N), 162.41 (C), 128.87, 114.40 (4CH), 115.93 (C), 76.63 (C-OH), 55.47 (OCH₃), 58.52 (C), 52.83 (CH), 46.18 (C), 27.97, 23.29 (2CH₂), 21.92, 20.44, 9.45 (3CH₃). MS *m/z* (%) 342 (M⁺, 40), 293 (16), 271 (16), 245 (24), 232 (36), 217 (100), 203 (48), 176 (36), 149 (56), 133 (84), 83 (24), 69 (28), 55 (32), 41 (20). Anal. Calcd for C₁₉H₂₂N₂O₄ (342.16): C 66.65, H 6.48, N 8.18. Found: C 66.60, H 6.43, N 8.123.

3-{5-[4-(Bromomethyl)phenyl]-1,3,4-oxadiazol-2-yl}-3-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (4d). White powder, yield 88%, mp 117-119°. IR (KBr): 3274 (OH), 2960, 2928, 1764, 1616, 1554, 1417, 1087, 858 cm⁻¹; ¹H NMR δ 7.99 (d, ³*J*_{HH} = 7.2 Hz, 2H, CH_{arom}), 7.50 (d, ³*J*_{HH} = 7.2 Hz, 2H, CH_{arom}), 7.50 (d, ³*J*_{HH} = 7.2 Hz, 2H, CH_{arom}), 4.50 (s, 2H, CH₂), 3.93 (s, 1H, OH), 2.51(m, 1H, CH), 1.73-1.85 (m, 4H, 2CH₂), 1.04 (s, 3H, CH₃), 1.17 (s, 6H, 2CH₃). ¹³C NMR δ 213.21 (C=O), 165.83, 164.85 (2C=N), 141.72 (2C), 129.68, 127.52 (4CH), 123.32 (C), 76.65 (C-OH), 58.28 (C), 52.79 (CH), 46.22 (C), 32.15 (CH₂Br), 27.95, 23.28 (2CH₂), 21.93, 20.42, 9.44 (3CH₃). MS *m/z* (%) 404 (M⁺, 20), 296 (24), 279 (56), 214 (24), 159

(60),116 (100), 83 (48), 69 (36), 55 (80), 41 (56). Anal. Calcd for $C_{19}H_{21}BrN_2O_3$ (404.07): C 56.31, H 5.22, N 6.91. Found: C 56.36, H 5.17, N 6.86.

3-[5-(3,4-Dimethylphenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (4e). White powder, yield 86%, mp 152-154°. IR (KBr): 3285 (OH), 2943, 2928, 1764, 1615, 1551, 1489, 1088, 856, 726 cm⁻¹; ¹H NMR δ 7.21-7.80 (m, 3H, CH_{arom}), 3.57 (s, 1H, OH), 2.48 (m, 1H, CH), 2.31 (s, 6H, 2CH₃), 1.62-1.96 (m, 4H, 2CH₂), 1.05 (s, 3H, CH₃), 1.16 (s, 6H, 2CH₃). ¹³C NMR δ 213.00 (C=O), 166.34, 165.20 (2C=N), 141.26, 137.51 (2C), 130.22, 128.05, 124.62 (3CH), 120.93 (C), 76.65 (C-OH), 58.26 (C), 52.80 (CH), 46.20 (C), 27.95, 23.30 (2CH₂), 19.94, 19.62 (2CH₃), 21.91, 20.44, 9.45 (3CH₃). MS *m/z* (%) 340 (M⁺, 68), 269 (24), 243 (48), 230 (28), 215 (88), 201 (92), 175 (24), 149 (36), 133 (100), 116 (32), 105 (44), 69 (40), 55 (40), 41 (44). Anal. Calcd for C₂₀H₂₄N₂O₃ (340.18): C 70.56, H 7.11, N 8.23. Found: C 70.50, H 7.17, N 8.17.

3-{5-[4-(Tert-butyl)phenyl]-1,3,4-oxadiazol-2-yl}-3-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (4f). Colorless Oil, yield 87%, IR (KBr): 3408 (OH), 2963, 2871, 1762, 1615, 1500, 1458, 1112, 843 cm⁻¹; ¹H NMR δ 7.95 (d, ³*J*_{HH}= 8.0 Hz, 2H, CH_{arom}), 7.49 (d, ³*J*_{HH} = 8.0 Hz, 2H, CH_{arom}), 3.51 (s, 1H, OH), 2.47 (m, 1H, CH), 1.62-1.90 (m, 4H, 2CH₂), 1.34 (s, 9H, 3CH₃), 1.04 (s, 3H, CH₃), 1.17 (s, 6H, 2CH₃). ¹³C NMR δ 213.11 (C=O), 165.37 (2C=N), 155.62 (C), 126.95, 125.97 (4CH), 120.61(C), 76.66 (C-OH), 58.27 (C), 52.86 (CH), 46.20 (C), 35.06 (C), 31.07 (3CH₃), 27.98, 23.24 (2CH₂), 21.92, 20.43, 9.45 (3CH₃). Anal. Calcd for C₂₂H₂₈N₂O₃ (368.21): C 71.71, H 7.66, N 7.60. Found: C 71.76, H 7.61, N 7.65.

3-Hydroxyl-1,7,7-trimethyl-3-[5-(4-methylphenyl)-1,3,4oxadiazol-2-yl]bicyclo[2.2.1]heptan-2-one (4g). White powder, yield 86%, mp 136-138°. IR (KBr): 3281 (OH), 2955, 2929, 1766, 1615, 1548, 1499, 1088, 823 cm⁻¹; ¹H NMR δ 7.89 (d, ³*J*_{HH} = 6.25 Hz, 2H, CH_{arom}), 7.26 (d, ³*J*_{HH} = 6.25 Hz, 2H, CH_{arom}), 3.73 (s, 1H, OH), 2.50 (s, 1H, CH), 2.40 (s, 3H, CH₃), 1.67-1.95 (m, 4H, 2CH₂), 1.04 (s, 3H, CH₃), 1.16 (s, 6H, 2CH₃). ¹³C NMR δ 213.11 (C=O), 165.34 (2C=N), 142.50 (C), 129.67, 127.05 (4CH), 120.66 (C), 76.66 (C-OH), 58.27(C), 52.84 (CH), 46.20 (C), 27.97, 23.28 (2CH₂), 21.92, 21.62, 20.43, 9.45 (4CH₃). Anal. Calcd for C₁₉H₂₂N₂O₃ (326.16): C 69.92, H 6.79, N 8.58. Found: C 69.86, H 6.73, N 8.52.

3-[5-(4-Bromophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (**4h**). White powder, yield 87%, mp 153-155°. IR (KBr): 3354 (OH), 2959, 2925, 1762, 1603, 1542, 1486, 1087, 838 cm⁻¹; ¹H NMR δ 7.89 (d, ³*J*_{HH} = 8.5 Hz, 2H, CH_{arom}), 7.62 (d, ³*J*_{HH} = 8.5 Hz, 2H, CH_{arom}), 3.78 (s, 1H, OH), 2.47 (m, 1H, CH), 1.63-1.97 (m, 4H, 2CH₂), 1.05 (s, 3H, CH₃), 1.16 (s, 6H, 2CH₃). ¹³C NMR δ 210.00 (C=O), 165.73, 153.50 (2C=N), 132.38, 128.49 (4CH), 127.86, 122.21 (2C), 76.23 (C-OH), 58.28 (C), 52.73 (CH), 46.23 (C), 27.95, 23.32 (2CH₂), 21.92, 20.43, 9.43 (3CH₃). Anal. Calcd for C₁₈H₁₉BrN₂O₃ (390.06): C 55.26, H 4.89, N 7.16. Found: C 55.31, H 4.94, N 7.11.

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3-[5-(4-Fluorophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (4i). Colorless Oil, yield 85%, IR (KBr): 3416 (OH), 2964, 2928, 1760, 1611, 1500, 1417, 1237, 845 cm⁻¹; ¹H NMR & 7.15-8.07(m, 4H, CH_{arom}), 3.55 (s, 1H, OH), 2.51 (m, 1H, CH), 1.74-1.87 (m, 4H, 2CH₂), 1.05 (s, 3H, CH₃), 1.16 (s, 6H, 2CH₃). ¹³C NMR & 212.96 (C=O), 166.75, 165.26 (2C=N), 161.50 (C, d, ${}^{3}J_{CF}$ = 503.3 Hz), 129.44 (2CH, d, ${}^{3}J_{CF}$ = 8.8 Hz), 119.87 (C, d, ${}^{4}J_{CF}$ = 4.4 Hz), 116.40 (2CH, d, ${}^{2}J_{CF}$ = 22.6 Hz), 76.23 (C-OH), 58.28 (C), 52.72 (CH), 46.23 (2C), 27.92, 23.32 (2CH₂), 21.91, 20.43, 9.43 (3CH₃). Anal. Calcd for C₁₈H₁₉FN₂O₃ (330.14): C 65.44, H 5.80, N 8.48. Found: C 65.39, H 5.74, N 8.53.

3-Hydroxyl-1,7,7-trimethyl-3-[5-(3-methylphenyl)-1,3,4oxadiazol-2-yl]bicyclo[2.2.1]heptan-2-one (4j). White powder, yield 89%, mp 134-136°. IR (KBr): 3282 (OH), 2959, 2927, 1762, 1597, 1557, 1456, 1089, 819, 723, 688 cm⁻¹; ¹H NMR δ 7.33-7.83 (m, 4H, CH_{arom}), 3.95 (s, 1H, OH), 2.53 (m, 1H, CH), 2.39 (s, 3H, CH₃), 1.77-1.85 (m, 4H, 2CH₂), 1.04 (s, 3H, CH₃), 1.05 (s, 3H, CH₃), 1.16 (s, 3H, CH₃). ¹³C NMR δ 213.04 (C=O), 165.50 (2C=N), 138.89 (C), 132.72, 128.88, 127.59, 124.26 (4CH), 123.30 (C), 76.66 (C-OH), 58.28 (C), 52.84 (CH), 46.20 (C), 27.97, 23.28 (2CH₂), 21.25 (CH₃), 21.93, 20.43, 9.45 (3CH₃). Anal. Calcd for C₁₉H₂₂N₂O₃ (326.16): C 69.92, H 6.79, N 8.58. Found: C 69.87, H 6.85, N 8.64.

3-Hydroxyl-1,7,7-trimethyl-3-[5-(1-naphthyl)-1,3,4-oxadiazol-2-yl]bicyclo[2.2.1]heptan-2-one (4k). White powder, yield 86%, mp 135-137°. IR (KBr): 3426 (OH), 2925, 2872, 1760, 1579, 1536, 1456, 1108, 857, 774 cm⁻¹; ¹H NMR δ 7.51-9.16 (m, 7H, CH_{arom}), 3.62 (s, 1H, OH), 2.57 (m, 1H, CH), 1.66-1.96 (m, 4H, 2CH₂), 1.08 (s, 3H, CH₃), 1.20 (s, 6H, 2CH₃). ¹³C NMR δ 213.34 (C=O), 165.21 (2C=N), 133.86, 130.00, 128.30 (3C), 132.84, 128.74, 128.66, 128.22, 126.71, 126.04, 124.78 (7CH), 76.43 (C-OH), 58.33 (C), 52.87 (CH), 46.28 (C), 28.03, 23.36 (2CH₂), 21.95, 20.45, 9.47 (3CH₃). Anal. Calcd for C₂₂H₂₂N₂O₃ (362.16): C 72.91, H 6.12, N 7.73. Found: C 72.85, H 6.17, N 7.78.

3-[5-(3-Chlorophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (4l). White powder, yield 88%, mp 146-148°. IR (KBr): 3272 (OH), 2962, 2928, 1764, 1581, 1547, 1444, 1092, 806, 779, 680 cm⁻¹; ¹H NMR δ 7.43-8.01 (m, 4H, CH_{arom}), 3.15 (s, 1H, OH), 2.52 (m, 1H, CH), 1.70-1.95 (m, 4H, 2CH₂), 1.05 (s, 3H, CH₃), 1.07 (s, 3H, CH₃), 1.17 (s, 3H, CH₃). ¹³C NMR δ 213.12 (C=O), 165.34, 161.20 (2C=N), 135.31 (C), 132.02, 130.22, 127.06, 125.19 (4CH), 124.13 (C), 73.65 (C-OH), 58.46 (C), 52.82 (CH), 46.24 (C), 27.94, 23.31 (2CH₂), 21.92, 20.42, 9.43 (3CH₃). Anal. Calcd for $C_{18}H_{18}CIN_{3}O$ (327.81): C 65.95, H 5.53, N 12.82. Found: C 65.83, H 5.49, N 12.77.

3-Hydroxyl-1,7,7-trimethyl-3-[5-(2-methylphenyl)-1,3,4oxadiazol-2-yl]bicyclo[2.2.1]heptan-2-one (4m). White powder, yield 87%, mp 110-112°. IR (KBr): 3417 (OH), 2957, 2925, 1763, 1600, 1542, 1455, 1109, 772, 723 cm⁻¹; ¹H NMR δ 7.05-7.91 (m, 4H, CH_{arom}), 3.80 (s, 1H, OH), 2.64 (s, 3H, CH₃), 2.48 (m, 1H, CH), 1.69-1.97 (m, 4H, 2CH₂), 1.04 (s, 3H, CH₃), 1.05 (s, 3H, CH₃), 1.16 (s, 3H, CH₃). ¹³C NMR δ 213.95 (C=O), 168.45, 166.35 (2C=N), 138.51 (C), 131.72, 131.44, 129.17, 126.13 (4CH), 122.53 (C), 93.15 (C-OH), 58.31 (C), 52.87 (CH), 46.26 (C), 28.05, 23.25 (2CH₂), 19.24 (CH₃), 21.94, 20.41, 9.44 (3CH₃). Anal. Calcd for C₁₉H₂₂N₂O₃ (326.16): C 69.92, H 6.79, N 8.58. Found: C 69.86, H 6.73, N 8.64.

3-Hydroxyl-1,7,7-trimethyl-3-[5-(3-phenoxyphenyl)-1,3,4-oxadiazol-2-yl]bicyclo[2.2.1]heptan-2-one (4n). White powder, yield 86%, mp 105-107°. IR (KBr): 3159 (OH), 2950, 2927, 1762, 1596, 1551, 1448, 1098, 894, 759, 688 cm⁻¹; ¹H NMR δ 7.01-7.77 (m, 9H, CH_{arom}), 3.62 (s, 1H, OH), 2.49 (m, 1H, CH), 1.68-1.96 (m, 4H, 2CH₂), 1.04 (s, 3H, CH₃), 1.15 (s, 6H, 2CH₃). ¹³C NMR δ 205.96 (C=O), 165.97, 157.98 (2C=N), 156.45, 154.60 (2C), 130.53, 129.98, 124.98, 124.01, 121.75, 119.23, 117.07 (9CH), 122.11 (C), 76.45 (C-OH), 58.26 (C), 52.88 (CH), 46.21 (C), 27.90, 23.31 (2CH₂), 21.92, 20.42, 9.44 (3CH₃). Anal. Calcd for C₂₄H₂₄N₂O₄ (404.17): C 71.27, H 5.98, N 6.93. Found: C 71.33, H 5.92, N 6.87.

3-[5-(3,5-Dimethoxyphenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxy-1,7,7-trimethylbicyclo[2.2.1]heptan-2-one (40). Yellow Oil, yield: 88%. IR (KBr): 3465 (OH), 2961, 2873, 1762, 1601, 1557, 1456, 1159, 884 cm⁻¹; ¹H NMR & 6.58-7.19 (m, 3H, CH_{arom}), 3.82 (s, 6H, OCH₃), 3.70 (s, 1H, OH), 2.51 (m, 1H, CH), 1.67-1.90 (m, 4H, 2CH₂), 1.03 (s, 3H, CH₃), 1.04 (s, 3H, CH₃), 1.16 (s, 3H, CH₃). ¹³C NMR & 213.87 (C=O), 166.67, 161.07 (2C=N), 161.07, 124.86 (3C), 104.18, 104.62 (3CH), 76.45 (C-OH), 58.28 (C), 55.64 (2OCH₃), 52.78 (CH), 46.19 (C), 27.94, 23.29 (2CH₂), 21.92, 20.43, 9.44 (3CH₃). Anal. Calcd for C₂₀H₂₄N₂O₅ (372.17): C 64.50, H 6.50, N 7.52. Found: C 64.45, H 6.55, N 7.57.



Figure 1. ¹H NMR spectrum of 4a.



Figure 2. ¹³C NMR spectrum of 4a.



Figure 3. IR spectrum of 4a.

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Figure 5. ¹H NMR spectrum of 4b.







Figure 7. IR spectrum of 4a.







Figure 10. ¹³C NMR spectrum of 4c.



Figure 11. IR spectrum of 4c.



Figure 12. Mass spectrum of 4c.



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Figure 13. ¹H NMR spectrum of 4d.



Figure 14. ¹³C NMR spectrum of 4d.



Figure 15. IR spectrum of 4d.



Figure 16. Mass spectrum of 4d.



Figure 17. ¹H NMR spectrum of 4e.













Figure 19. IR spectrum of 4e.





Figure 21. ¹H NMR spectrum of 4f.



*L*ε.291 —



Figure 22. ¹³C NMR spectrum of 4f.

l kini kanang ing pangang pang

180

200



Figure 23. IR spectrum of 4f.





30

Figure 25. ¹³C NMR spectrum of 4g.



Figure 25. ¹³C NMR spectrum of 4g.



Figure 26. IR spectrum of 4g.



Figure 27. ¹H NMR spectrum of 4h.







Figure 29. IR spectrum of 4h.



Figure 30. ¹H NMR spectrum of 4i.







Figure 31. ¹³C NMR spectrum of 4i.



Figure 32. IR spectrum of 4i.



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Figure 33. ¹H NMR spectrum of 4j.



Figure 33. ¹H NMR spectrum of 4j.



Figure 34. ¹³C NMR spectrum of 4j.



Figure 35. IR spectrum of 4j.



Figure 36. ¹H NMR spectrum of 4k.



Figure 37. ¹³C NMR spectrum of 4k.



Figure 37. ¹³C NMR spectrum of 4k.



Figure 38. IR spectrum of 4k.



Figure 39. ¹H NMR spectrum of 41.



Figure 40. ¹³C NMR spectrum of 4l.



Figure 40. ¹³C NMR spectrum of 4l.



Figure 41. IR spectrum of 4l.



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Figure 42. ¹H NMR spectrum of 4m.



Figure 43. ¹³C NMR spectrum of 4m.



Figure 43. ¹³C NMR spectrum of 4m.



Figure 44. IR spectrum of 4m.



Figure 45. ¹H NMR spectrum of 4n.







Figure 46. ¹³C NMR spectrum of 4n.



Figure 47. IR spectrum of 4n.









Figure 50. IR spectrum of 40.