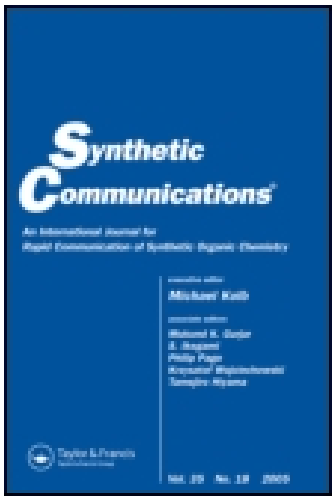

Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

SYNTHESIS OF 2-(5-(2-CHLOROPHENYL)-2-FUROYLAMIDO)-5-ARYLOXYMETHYL-1,3,4-THIADIAZOLES UNDER MICROWAVE IRRADIATION

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Published online: 16 Aug 2006.

To cite this article: Zheng Li , Xicun Wang & Yuxia Da (2001) SYNTHESIS OF 2-(5-(2-CHLOROPHENYL)-2-FUROYLAMIDO)-5-ARYLOXYMETHYL-1,3,4-THIADIAZOLES UNDER MICROWAVE IRRADIATION, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 31:12, 1829-1836, DOI: 10.1081/SCC-100104332

To link to this article: http://dx.doi.org/10.1081/SCC-100104332

SYNTHETIC COMMUNICATIONS, 31(12), 1829–1836 (2001)

SYNTHESIS OF 2-(5-(2-CHLOROPHENYL)-2-FUROYLAMIDO)-5-ARYLOXYMETHYL-1,3,4-THIADIAZOLES UNDER MICROWAVE IRRADIATION

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ABSTRACT

2-(5-(2-chlorophenyl)-2-furoylamido)-5-aryloxymethyl-1,3,4-thiadiazoles **(IIa–j)** are synthesized under microwave irradiation via the cyclization of 1-aryloxyacetyl-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazides **(Ia–j)** in the presence of glacial acetic acid.

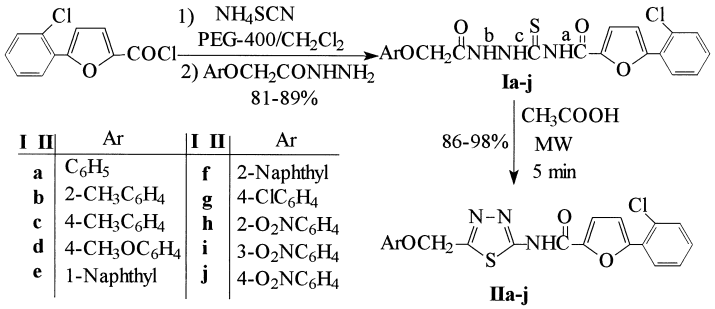
5-Aryl-2-furoic acid derivatives have been used as antibacterial agent,[1] local anesthesia,[2] analgesic[3] and plant-growth regulator.[4–5] Meanwhile, substituted 1,3,4-thiadiazoles have also attracted much attention due to their diverse biological activities, such as antimicrobial,[6–11] antibacterial,[12] anesthetic,[13] antithrombotic,[14] anticonvulsant,[15] cardiotonic,[16] antihypertensive,[17] antiinflammatory[18] and antiulcer[19] activity.

Keeping in view the above facts, we report herein the preparation of a new series of compounds bearing both 1,3,4-thiadiazole and 5-aryl-2-furoyl moiety, with the objective of obtaining new biologically active compounds.

* Corresponding author.

Although the substituted 1,3,4-thiadiazoles can be prepared from the substituted thiosemicarbazides by conventional method,[20,21] the reaction yield is often not high and the reaction time is always very long. This paper we introduce a convenient and efficient microwave method.

Reaction of 5-(2-chlorophenyl)-2-furoyl chloride with ammonium thiocyanate catalyzed by polyethylene glycol-400 (PEG-400) at room temperature gives 5-(2-chlorophenyl)-2-furoylisothiocyanate, which on treatment with aryloxyacetic acid hydrazides *in situ* at room temperature affords 1-aryloxyacetyl-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazides (**Ia–j**) in excellent yields. Compounds (**Ia–j**) on exposure to microwave irradiation in the presence of glacial acetic acid result in the formation of 2-(5-(2-chlorophenyl)-2-furoylamido)-5-aryloxymethyl-1,3,4-thiadiazoles (**IIa–j**) (Scheme).



I	II	Ar	I	II	Ar
a		C_6H_5	f		2-Naphthyl
b		2-$CH_3C_6H_4$	g		4-ClC_6H_4
c		4-$CH_3C_6H_4$	h		2-$O_2NC_6H_4$
d		4-$CH_3OC_6H_4$	i		3-$O_2NC_6H_4$
e		1-Naphthyl	j		4-$O_2NC_6H_4$

Scheme.

The characterization of compounds **Ia–j** and **IIa–j** is based on their IR (KBr), ^{1}H NMR and elemental analyses. The IR spectra exhibit a characteristic strong absorption at 1169–1210 cm^{-1} for compounds **Ia–j** attributable to the C=S of the thio residue. The carbonyl absorption is observed at 1656–1705 cm^{-1} for **Ia–j** and 1659–1676 cm^{-1} for **IIa–j**. The ^{1}H NMR spectral data of **Ia–j** in d_6-dimethylsulfoxide show peaks at 12.42–12.62 (NH^a), 11.84–11.91 (NH^b), 11.01–11.21 (NH^c) and 4.63–4.91 ppm (CH_2). In contrast, the ^{1}H NMR spectral data of **IIa–j** in the same deuterated solvent show signals at 13.17–13.45 (NH), indicating a significant downfield shift (ca. 0.8 ppm) compared to the corresponding NH^a of **Ia–j**, and singlet at 5.40–5.56 (CH_2). All found of C, H, N of **Ia–j** and **IIa–j** are good agreement with the calculated.

EXPERIMENTAL

IR spectra were recorded using KBr pellets on an Alpha Centauri FTIR spectrophotometer and ^{1}H NMR spectra on a FT-80A instrument

using $(CD_3)_2SO$ as solvent and Me_4Si as internal standard. Elemental analyses were performed on a Carlo-Erba 1106 Elemental Analysis instrument. Melting points were observed in an open capillary tube and uncorrected. 5-(2-chlorophenyl)-2-furoylchloride[22] and aryloxyacetic acid hydrazides[23] were prepared according to literature procedures. Ammonium thiocyanate, glacial acetic acid and PEG-400 were commercially available and used as received.

General Procedure for Preparation of Ia–j

A suspension of 5-(2-chlorophenyl)-2-furoyl chloride (0.36 g, 1.5 mmol), ammonium thiocyanate (0.20 g, 2.6 mmol) and PEG-400 (0.02 g, 0.05 mmol) in 40 mL of methylene chloride was stirred for 1 h at room temperature, then aryloxyacetic acid hydrazide (1.45 mmol) was added. The mixture was stirred for another 0.5 h, and a precipitate was formed. The resulting mixture was filtered and washed with water to remove inorganic salts. The residue was recrystallized from DMF-EtOH-H_2O to yield I as crystals. The physical and spectral results are shown below.

1-phenyloxyacetyl-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ia): white solid. yield: 83%. m.p.: 195–196°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.44 (1H, s, NH), 11.89 (1H, s, NH), 11.14 (1H, s, NH), 7.34–8.26 (11H, m, Ar-H and Fu-H), 4.91 (2H, s, CH_2). IR (KBr, $\gamma\,cm^{-1}$): 3276, 3151 (N-H), 1701, 1669 (C=O), 1210 (C=S). Anal. Calcd. for $C_{20}H_{16}N_3O_4SCl$: C, 55.88; H, 3.75; N, 9.77. Found: C, 56.01; H, 3.61; N, 9.89.

1-(2-tolyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ib): white solid. yield: 82%. m.p.: 209–210°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.42 (1H, s, NH), 11.85 (1H, s, NH), 11.08 (1H, s, NH), 6.86–8.53 (10H, m, Ar-H and Fu-H), 4.63 (2H, s, CH_2), 2.30 (3H, s, CH_3). IR (KBr, $\gamma\,cm^{-1}$): 3308, 3226, 3119 (N-H), 1698, 1658 (C=O), 1197 (C=S). Anal. Calcd. for $C_{21}H_{18}N_3O_4SCl$: C, 56.82; H, 4.09; N, 9.47. Found: C, 56.96; H, 4.01; N, 9.23.

1-(4-tolyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ic): white solid. yield: 81%. m.p.: 187–188°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.51 (1H, s, NH), 11.87 (1H, s, NH), 11.01 (1H, s, NH), 6.94–8.60 (10H, m, Ar-H and Fu-H), 4.65 (2H, s, CH_2), 2.26 (3H, s, CH_3). IR (KBr, $\gamma\,cm^{-1}$): 3281, 3213, 3124 (N-H), 1696, 1664 (C=O), 1172 (C=S). Anal. Calcd. for $C_{21}H_{18}N_3O_4SCl$: C, 56.82; H, 4.09; N, 9.47. Found: C, 57.03; H, 3.98; N, 9.54.

1-(4-methoxylphenyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Id): White solid. yield: 85%. m.p.: 189–190°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.54 (1H, s, NH), 11.89 (1H, s, NH), 11.08 (1H, s, NH), 7.01–

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8.57 (10H, m, Ar-H and Fu-H), 4.68 (2H, s, CH_2), 3.43 (3H, s, CH_3). IR (KBr, $\gamma\,cm^{-1}$): 3351, 3276, 3160 (N-H), 1698, 1656 (C=O), 1188 (C=S). Anal. Calcd. for $C_{21}H_{18}N_3O_5SCl$: C, 54.84; H, 3.94; N, 9.14. Found: C, 54.63; H, 4.02; N, 9.28.

1-(1-naphthyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ie): White solid. yield: 83%. m.p.: 210–211°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.51 (1H, s, NH), 11.84 (1H, s, NH), 11.21 (1H, s, NH), 7.30–8.27 (13H, m, Ar-H and Fu-H), 4.82 (4H, s, CH_2). IR (KBr, $\gamma\,cm^{-1}$): 3350, 3271, 3180 (N-H), 1701, 1663 (C=O), 1175 (C=S). Anal. Calcd. for $C_{24}H_{18}N_3O_4SCl$: C, 60.06; H, 3.78; N, 8.76. Found: C, 60.36; H, 3.82; N, 8.59.

1-(2-naphthyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (If): White solid. yield: 85%. m.p.: 203–204°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.49 (1H, s, NH), 11.86 (1H, s, NH), 11.09 (1H, s, NH), 7.21–8.34 (13H, m, Ar-H and Fu-H), 4.81 (4H, s, CH_2). IR (KBr, $\gamma\,cm^{-1}$): 3346, 3268, 3180 (N-H), 1698, 1657 (C=O), 1178 (C=S). Anal. Calcd. for $C_{24}H_{18}N_3O_4SCl$: C, 60.06; H, 3.78; N, 8.76. Found: C, 60.28; H, 3.88; N, 8.63.

1-(4-chlorophenyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ig): White solid. yield: 89%. m.p.: 205–206°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.48 (1H, s, NH), 11.91 (1H, s, NH), 11.12 (1H, s, NH), 6.98–8.12 (10H, m, Ar-H and Fu-H), 4.83 (2H, s, CH_2). IR (KBr, $\gamma\,cm^{-1}$): 3301, 3227, 3146 (N-H), 1692, 1668 (C=O), 1170 (C=S). Anal. Calcd. for $C_{20}H_{15}N_3O_4SCl_2$: C, 51.73; H, 3.26; N, 9.05. Found: C, 51.99; H, 3.15; N, 8.89.

1-(2-nitrophenyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ih): White solid. yield: 84%. m.p.: 209–210°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.62 (1H, s, NH), 11.90 (1H, s, NH), 11.13 (1H, s, NH), 7.21–8.11 (10H, m, Ar-H and Fu-H), 4.87 (2H, s, CH_2). IR (KBr, $\gamma\,cm^{-1}$): 3270, 3216 (N-H), 1705, 1667 (C=O), 1169 (C=S). Anal. Calcd. for $C_{20}H_{15}N_4O_6SCl$: C, 50.59; H, 3.18; N, 11.80. Found: C, 50.86; H, 3.21; N, 11.70.

1-(3-nitrophenyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ii): White solid. yield: 81%. m.p.: 211–212°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.60 (1H, s, NH), 11.87 (1H, s, NH), 11.11 (1H, s, NH), 7.20–8.11 (10H, m, Ar-H and Fu-H), 4.89 (2H, s, CH_2). IR (KBr, $\gamma\,cm^{-1}$): 3286, 3231 (N-H), 1701, 1672 (C=O), 1172 (C=S). Anal. Calcd. for $C_{20}H_{15}N_4O_6SCl$: C, 50.59; H, 3.18; N, 11.80. Found: C, 50.76; H, 3.11; N, 11.67.

1-(4-nitrophenyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ij): White solid. yield: 87%. m.p.: 206–207°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 12.61 (1H, s, NH), 11.86 (1H, s, NH), 11.13 (1H, s, NH),

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7.18–8.06 (10H, m, Ar-H and Fu-H), 4.89 (2H, s, CH_2). IR (KBr, $\gamma\,cm^{-1}$): 3277, 3208, (N-H), 1703, 1666 (C=O), 1174 (C=S). Anal. Calcd. for $C_{20}H_{15}N_4O_6SCl$: C, 50.59; H, 3.18; N, 11.80. Found: C, 50.46; H, 3.08; N, 11.72.

General Procedure for the Preparation of Compounds IIa–j

A mixture of compound **I** (0.5 mol) and glacial acetic acid (5 mL) was irradiated in a microwave oven at 375 W for 5 minutes, the completion of the reaction was monitored by TLC. The excess of glacial acetic acid was removed by evaporation, the residue was poured into ice (100 g), and the precipitate was collected by filtration and washed with water (3×20 mL). The product was obtained as white crystals after recrystallization from DMF-EtOH-H_2O. The physical and spectral date of compounds **IIa–j** are shown below.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-phenyloxymethyl-1,3,4-thiadiazole (IIa): White solid. yield: 94%. m.p.: 227–228°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.21 (1H, s, NH), 7.01–8.40 (11H, m, Ar-H and Fu-H), 5.41 (2H, s, CH_2). IR (KBr, $\gamma\,cm^{-1}$): 3179 (N-H), 1667 (C=O), 1605, 1507, 1451, 1107 (C=N-N-C=S). Anal. Calcd. for $C_{20}H_{14}N_3O_3SCl$: C, 58.32; H, 3.43; N, 10.20. Found: C, 58.51; H, 3.32; N, 10.47.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-(2-tolyloxymethyl)-1,3,4-thiadiazole (IIb): White solid. yield: 92%. m.p.: 238–239°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.17 (1H, s, NH), 6.97–8.51 (10H, m, Ar-H and Fu-H), 5.44 (2H, s, CH_2), 2.26 (3H, s, CH_3). IR (KBr, $\gamma\,cm^{-1}$): 3186 (N-H), 1670 (C=O), 1602, 1510, 1446, 1101 (C=N-N-C=S). Anal. Calcd. for $C_{21}H_{16}N_3O_3SCl$: C, 59.22; H, 3.79; N, 9.87. Found: C, 58.97; H, 3.63; N, 10.03.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-(4-tolyloxymethyl)-1,3,4-thiadiazole (IIc): White solid. yield: 97%. m.p.: 221–222°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.36 (1H, s, NH), 6.99–8.53 (10H, m, Ar-H and Fu-H), 5.43 (2H, s, CH_2), 2.21 (3H, s, CH_3) IR (KBr, $\gamma\,cm^{-1}$): 3189 (N-H), 1669 (C=O), 1600, 1513, 1449, 1099 (C=N-N-C=S). Anal. Calcd. for $C_{21}H_{16}N_3O_3SCl$: C, 59.22; H, 3.79; N, 9.87. Found: C, 59.44; H, 3.96; N, 9.69.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-(4-methoxylphenyloxymethyl)-1,3,4-thiadiazole (IId): White solid. yield: 96%. m.p.: 233–234°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.40 (1H, s, NH), 6.88–8.60 (10H, m, Ar-H and Fu-H), 5.40 (2H, s, CH_2), 3.40 (3H, s, CH_3) IR (KBr, $\gamma\,cm^{-1}$): 3163 (N-H), 1673 (C=O), 1608, 1503, 1442, 1105 (C=N-N-C=S). Anal. Calcd. for $C_{21}H_{16}N_3O_4SCl$: C, 57.08; H, 3.65; N, 9.51. Found: C, 56.86; H, 3.57; N, 9.70.

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2-(5-(2-chlorophenyl)-2-furoylamido)-5-(1-naphthyloxymethyl)-1,3,4-thiadiazole (IIe): White solid. yield: 91%. m.p.: 248–249°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.38 (1H, s, NH), 6.93–8.49 (13H, m, Ar-H and Fu-H), 5.42 (2H, s, CH_2). IR (KBr, γ cm^{-1}): 3184 (N-H), 1669 (C=O), 1601, 1515, 1481, 1107 (C=N-N-C=S). Anal. Calcd. for $C_{24}H_{16}N_3O_3SCl$: C, 62.40; H, 3.49; N, 9.10. Found: C, 62.15; H, 3.28; N, 9.29.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-(2-naphthyloxymethyl)-1,3,4-thiadiazole (IIf): White solid. yield: 98%. m.p.: 240–241°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.41 (1H, s, NH), 7.02–8.51 (13H, m, Ar-H and Fu-H), 5.41 (2H, s, CH_2). IR (KBr, γ cm^{-1}): 3188 (N-H), 1671 (C=O), 1603, 1516, 1476, 1104 (C=N-N-C=S). Anal. Calcd. for $C_{24}H_{16}N_3O_3SCl$: C, 62.40; H, 3.49; N, 9.10. Found: C, 62.56; H, 3.40; N, 9.33.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-(4-chlorophenyloxymethyl)-1,3,4-thiadiazole (IIg): White solid. yield: 96%. m.p.: 244–245°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.45 (1H, s, NH), 6.79–8.61 (10H, m, Ar-H and Fu-H), 5.46 (2H, s, CH_2). IR (KBr, γ cm^{-1}): 3190 (N-H), 1676 (C=O), 1600, 1509, 1457, 1110 (C=N-N-C=S). Anal. Calcd. for $C_{20}H_{13}N_3O_3SCl_2$: C, 53.82; H, 2.94; N, 9.41. Found: C, 54.02; H, 3.13; N, 9.26.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-(2-nitrophenyloxymethyl)-1,3,4-thiadiazole (IIh): White solid. yield: 97%. m.p.: 245–246°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.26 (1H, s, NH), 7.03–8.52 (10H, m, Ar-H and Fu-H), 5.51 (2H, s, CH_2). IR (KBr, γ cm^{-1}): 3220 (N-H), 1676 (C=O), 1609, 1511, 1483, 1102 (C=N-N-C=S). Anal. Calcd. for $C_{20}H_{13}N_4O_5SCl$: C, 52.58; H, 2.87; N, 12.26. Found: C, 52.63; H, 2.92; N, 12.40.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-(3-nitrophenyloxymethyl)-1,3,4-thiadiazole (IIi): White solid. yield: 96%. m.p.: 274–275°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.37 (1H, s, NH), 7.11–8.46 (10H, m, Ar-H and Fu-H), 5.48 (2H, s, CH_2). IR (KBr, γ cm^{-1}): 3196 (N-H), 1669 (C=O), 1602, 1507, 1479, 1109 (C=N-N-C=S). Anal. Calcd. for $C_{20}H_{13}N_4O_5SCl$: C, 52.58; H, 2.87; N, 12.26. Found: C, 52.42; H, 2.79; N, 12.11.

2-(5-(2-chlorophenyl)-2-furoylamido)-5-(4-nitrophenyloxymethyl)-1,3,4-thiadiazole (IIj): White solid. yield: 86%. m.p.: 245–256°C. ^{1}H NMR (80 MHz, DMSO-d_6) δ 13.29 (1H, s, NH), 7.06–8.51 (10H, m, Ar-H and Fu-H), 5.56 (2H, s, CH_2). IR (KBr, γ cm^{-1}): 3211 (N-H), 1659 (C=O), 1604, 1514, 1473, 1106 (C=N-N-C=S). Anal. Calcd. for $C_{20}H_{13}N_4O_5SCl$: C, 52.58; H, 2.87; N, 12.26. Found: C, 52.50; H, 2.86; N, 12.30.

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ACKNOWLEDGMENT

The authors thank the Natural Science Foundation of Gansu Province for the financial support of this work.

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Received in the USA August 24, 2000

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