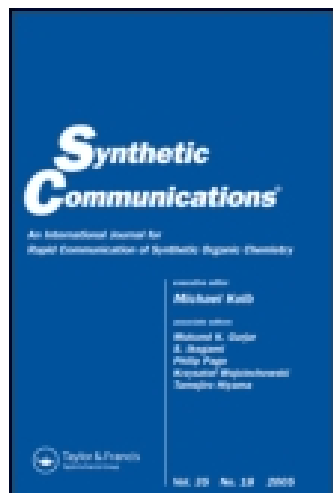




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SYNTHESIS OF 2-(5-(2-CHLOROPHENYL)-2-FUROYLAMIDO)-5-ARYLOXYMETHYL-1,3,4-THIADIAZOLES UNDER MICROWAVE IRRADIATION

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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.

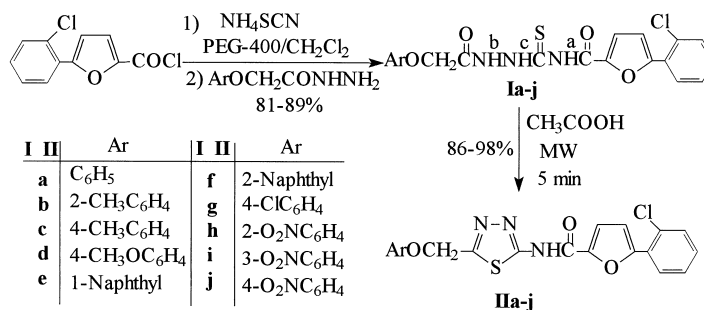
5-Aryl-2-furoic acid derivatives have been used as antibacterial agent,¹ local anesthesia,² analgesic³ and plant-growth regulator.^{4–5} Meanwhile, substituted 1,3,4-thiadiazoles have also attracted much attention due to their diverse biological activities, such as antimicrobial,^{6–11} antibacterial,¹² anesthetic,¹³ antithrombotic,¹⁴ anticonvulsant,¹⁵ cardiogenic,¹⁶ antihypertensive,¹⁷ antiinflammatory¹⁸ and antiulcer¹⁹ 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.

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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).



Scheme.

The characterization of compounds **Ia–j** and **IIa–j** is based on their IR (KBr), ^1H NMR and elemental analyses. The IR spectra exhibit a characteristic strong absorption at $1169\text{--}1210\text{ cm}^{-1}$ for compounds **Ia–j** attributable to the $\text{C}=\text{S}$ of the thio residue. The carbonyl absorption is observed at $1656\text{--}1705\text{ cm}^{-1}$ for **Ia–j** and $1659\text{--}1676\text{ cm}^{-1}$ for **IIa–j**. The ^1H NMR spectral data of **Ia–j** in d_6 -dimethylsulfoxide show peaks at $12.42\text{--}12.62$ (NH^a), $11.84\text{--}11.91$ (NH^b), $11.01\text{--}11.21$ (NH^c) and $4.63\text{--}4.91$ ppm (CH_2). In contrast, the ^1H NMR spectral data of **IIa–j** in the same deuterated solvent show signals at $13.17\text{--}13.45$ (NH), indicating a significant downfield shift (ca. 0.8 ppm) compared to the corresponding NH^a of **Ia–j**, and singlet at $5.40\text{--}5.56$ (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 ^1H NMR spectra on a FT-80A instrument



using $(\text{CD}_3)_2\text{SO}$ 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-furoylchloride²² and aryloxyacetic acid hydrazides²³ 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. ^1H 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, $\gamma \text{ cm}^{-1}$): 3276, 3151 (N-H), 1701, 1669 (C=O), 1210 (C=S). Anal. Calcd. for $\text{C}_{20}\text{H}_{16}\text{N}_3\text{O}_4\text{SCl}$: 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. ^1H 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, $\gamma \text{ cm}^{-1}$): 3308, 3226, 3119 (N-H), 1698, 1658 (C=O), 1197 (C=S). Anal. Calcd. for $\text{C}_{21}\text{H}_{18}\text{N}_3\text{O}_4\text{SCl}$: 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. ^1H 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, $\gamma \text{ cm}^{-1}$): 3281, 3213, 3124 (N-H), 1696, 1664 (C=O), 1172 (C=S). Anal. Calcd. for $\text{C}_{21}\text{H}_{18}\text{N}_3\text{O}_4\text{SCl}$: C, 56.82; H, 4.09; N, 9.47. Found: C, 57.03; H, 3.98; N, 9.54.

1-(4-methoxyphenyloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide

(Id): White solid. yield: 85%. m.p.: 189–190°C. ^1H NMR (80 MHz, DMSO-d_6) δ 12.54 (1H, s, NH), 11.89 (1H, s, NH), 11.08 (1H, s, NH), 7.01–



8.57 (10H, m, Ar-H and Fu-H), 4.68 (2H, s, CH₂), 3.43 (3H, s, CH₃). IR (KBr, γ cm⁻¹): 3351, 3276, 3160 (N-H), 1698, 1656 (C=O), 1188 (C=S). Anal. Calcd. for C₂₁H₁₈N₃O₅SCl: 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. ¹H NMR (80 MHz, DMSO-d₆) 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, CH₂). IR (KBr, γ cm⁻¹): 3350, 3271, 3180 (N-H), 1701, 1663 (C=O), 1175 (C=S). Anal. Calcd. for C₂₄H₁₈N₃O₄SCl: 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. ¹H NMR (80 MHz, DMSO-d₆) 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, CH₂). IR (KBr, γ cm⁻¹): 3346, 3268, 3180 (N-H), 1698, 1657 (C=O), 1178 (C=S). Anal. Calcd. for C₂₄H₁₈N₃O₄SCl: C, 60.06; H, 3.78; N, 8.76. Found: C, 60.28; H, 3.88; N, 8.63.

1-(4-chlorophenylloxyacetyl)-4-(5-(2-chlorophenyl)-2-furoyl)-thiosemicarbazide (Ig): White solid. yield: 89%. m.p.: 205–206°C. ¹H NMR (80 MHz, DMSO-d₆) 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, CH₂). IR (KBr, γ cm⁻¹): 3301, 3227, 3146 (N-H), 1692, 1668 (C=O), 1170 (C=S). Anal. Calcd. for C₂₀H₁₅N₃O₄SCl₂: 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. ¹H NMR (80 MHz, DMSO-d₆) 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, CH₂). IR (KBr, γ cm⁻¹): 3270, 3216 (N-H), 1705, 1667 (C=O), 1169 (C=S). Anal. Calcd. for C₂₀H₁₅N₄O₆SCl: 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. ¹H NMR (80 MHz, DMSO-d₆) 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, CH₂). IR (KBr, γ cm⁻¹): 3286, 3231 (N-H), 1701, 1672 (C=O), 1172 (C=S). Anal. Calcd. for C₂₀H₁₅N₄O₆SCl: 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. ¹H NMR (80 MHz, DMSO-d₆) δ 12.61 (1H, s, NH), 11.86 (1H, s, NH), 11.13 (1H, s, NH),



7.18–8.06 (10H, m, Ar-H and Fu-H), 4.89 (2H, s, CH₂). IR (KBr, γ cm⁻¹): 3277, 3208, (N-H), 1703, 1666 (C=O), 1174 (C=S). Anal. Calcd. for C₂₀H₁₅N₄O₆SCl: 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-H₂O. The physical and spectral data 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. ¹H NMR (80 MHz, DMSO-d₆) δ 13.21 (1H, s, NH), 7.01–8.40 (11H, m, Ar-H and Fu-H), 5.41 (2H, s, CH₂). IR (KBr, γ cm⁻¹): 3179 (N-H), 1667 (C=O), 1605, 1507, 1451, 1107 (C=N-N-C=S). Anal. Calcd. for C₂₀H₁₄N₃O₃SCl: 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. ¹H NMR (80 MHz, DMSO-d₆) δ 13.17 (1H, s, NH), 6.97–8.51 (10H, m, Ar-H and Fu-H), 5.44 (2H, s, CH₂), 2.26 (3H, s, CH₃). IR (KBr, γ cm⁻¹): 3186 (N-H), 1670 (C=O), 1602, 1510, 1446, 1101 (C=N-N-C=S). Anal. Calcd. for C₂₁H₁₆N₃O₃SCl: 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. ¹H NMR (80 MHz, DMSO-d₆) δ 13.36 (1H, s, NH), 6.99–8.53 (10H, m, Ar-H and Fu-H), 5.43 (2H, s, CH₂), 2.21 (3H, s, CH₃). IR (KBr, γ cm⁻¹): 3189 (N-H), 1669 (C=O), 1600, 1513, 1449, 1099 (C=N-N-C=S). Anal. Calcd. for C₂₁H₁₆N₃O₃SCl: 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-methoxyphenyloxymethyl)-1,3,4-thiadiazole (IId): White solid. yield: 96%. m.p.: 233–234°C. ¹H NMR (80 MHz, DMSO-d₆) δ 13.40 (1H, s, NH), 6.88–8.60 (10H, m, Ar-H and Fu-H), 5.40 (2H, s, CH₂), 3.40 (3H, s, CH₃). IR (KBr, γ cm⁻¹): 3163 (N-H), 1673 (C=O), 1608, 1503, 1442, 1105 (C=N-N-C=S). Anal. Calcd. for C₂₁H₁₆N₃O₄SCl: C, 57.08; H, 3.65; N, 9.51. Found: C, 56.86; H, 3.57; N, 9.70.



2-(5-(2-chlorophenyl)-2-furoylamido)-5-(1-naphthyloxymethyl)-1,3,4-thiadiazole (IIe): White solid. yield: 91%. m.p.: 248–249°C. ^1H 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, CH₂). IR (KBr, $\gamma\text{ cm}^{-1}$): 3184 (N-H), 1669 (C=O), 1601, 1515, 1481, 1107 (C=N-N-C=S). Anal. Calcd. for C₂₄H₁₆N₃O₃SCl: 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. ^1H 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, CH₂). IR (KBr, $\gamma\text{ cm}^{-1}$): 3188 (N-H), 1671 (C=O), 1603, 1516, 1476, 1104 (C=N-N-C=S). Anal. Calcd. for C₂₄H₁₆N₃O₃SCl: 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. ^1H 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, CH₂). IR (KBr, $\gamma\text{ cm}^{-1}$): 3190 (N-H), 1676 (C=O), 1600, 1509, 1457, 1110 (C=N-N-C=S). Anal. Calcd. for C₂₀H₁₃N₃O₃SCl₂: 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. ^1H 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, CH₂). IR (KBr, $\gamma\text{ cm}^{-1}$): 3220 (N-H), 1676 (C=O), 1609, 1511, 1483, 1102 (C=N-N-C=S). Anal. Calcd. for C₂₀H₁₃N₄O₅SCl: 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. ^1H 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, CH₂). IR (KBr, $\gamma\text{ cm}^{-1}$): 3196 (N-H), 1669 (C=O), 1602, 1507, 1479, 1109 (C=N-N-C=S). Anal. Calcd. for C₂₀H₁₃N₄O₅SCl: 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. ^1H 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, CH₂). IR (KBr, $\gamma\text{ cm}^{-1}$): 3211 (N-H), 1659 (C=O), 1604, 1514, 1473, 1106 (C=N-N-C=S). Anal. Calcd. for C₂₀H₁₃N₄O₅SCl: C, 52.58; H, 2.87; N, 12.26. Found: C, 52.50; H, 2.86; N, 12.30.



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