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Synthesis of 1,3-Bis[(3-aryl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzenes

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Synthesis of 1,3-Bis[(3-aryl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzenes

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*1,3-bis[(3-aryl)-s-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzenes 2 were synthesized in high yields by the reaction of 3-aryl 4-amino-5-mercapto-1,2,4-triazole 1 with *m*-phthalic acid.*

Keywords 1,3-Bis[(3-aryl)-s-triazolo-[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzenes; 2,4-triazole; 3-aryl-4-amino-5-mercapto-1; synthesis

S-triazolo[3,4-*b*]-[1,3,4]thiadiazole derivatives were found to possess significant biological activities such as antinflammatory, antiviral, antifungal, antineoplastic, and antidepressant effects^{1–4} and electrochemical properties.⁵ They are highly important heterocycles and have been used in the research and development of agrochemicals and in the pharmaceutical chemistry. Most derivatives that have been reported only contain one s-triazolo[3,4-*b*]-[1,3,4]thiadiazole in one molecule.^{6–11} As part of our current studies on the synthesis of the biologically active s-triazolo[3,4-*b*]-[1,3,4]thiadiazole derivatives, we now report an efficient synthesis of some novel fused heterocyclic compounds combining two s-triazolo[3,4-*b*]-[1,3,4]thiadiazole rings in one framework from 3-aryl 4-amino-5-mercapto-1,2,4-triazoles with the hope to obtain compounds of better biological activities.

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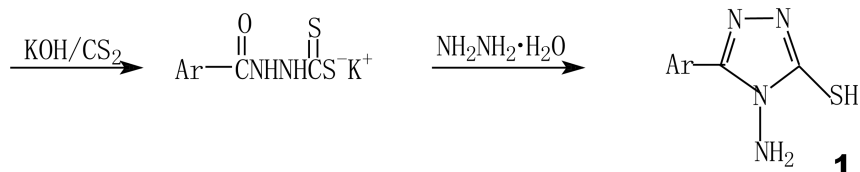
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RESULTS AND DISCUSSION

The aroylhydrazides were prepared by the esterification and hydrazinolysis of corresponding aromatic carboxylic acids (Scheme 1). The reaction of aroylhydrazides with CS₂/KOH in absolute ethanol gave potassium aroyldithiocarbazates, and then the hydrazinolysis of potassium aroyldithiocarbazates with hydrazine hydrate afforded 3-aryl-4-amino-5-mercapto-1,2,4-triazoles **1** (Scheme 2).¹²

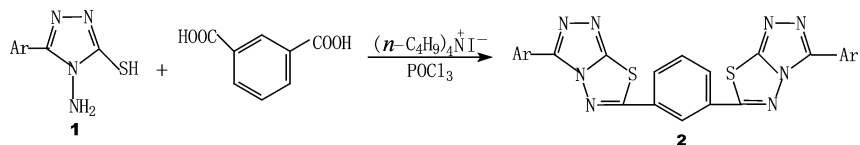


SCHEME 1



SCHEME 2

The synthesis of **2** was accomplished in one step with good yields by condensing 3-aryl-4-amino-5-mercapto-1,2,4-triazoles **1** with *m*-phthalic acid in the presence of POCl₃ and the phase transfer catalyst tetrabutylammonium iodide (Scheme 3, Table I).



SCHEME 3

The structures of all of compounds **2** was established on the basis of elemental analysis and spectral data. The IR spectral data of compound **2** showed bands at 1600–1630 cm⁻¹, 1235–1260 cm⁻¹, and 700 cm⁻¹, due to –C=N, N=N=C, and –C–S–C, respectively. The ¹H NMR spectra of **2** exhibited multiple signals in the δ 7.00–8.70 range accounting for hydrogens of the aryl group. With compound **2g**, as an example, multiple signals appeared in the range of δ 8.59–8.44, δ 8.30–8.24, δ 8.07–8.05, and δ 7.59–7.56, which accounted for the 12 hydrogens of the aryl group. A single signal at δ 2.57 accounted for 6 hydrogens of 2 methyl group. The EI-MS for compounds **2**, except for **2b**, **2c**, and **2d**, exhibited strong

TABLE I Preparation of 1,3-Bis[(3-aryl)-s-triazolo[3,4-b]-[1,3,4]thiadiazole-6-yl]benzenes **2** from 3-aryl-4-amino-5-mercapto-1,2,4-triazoles **1**

Entry	Ar	Condition	Yield (%) ^a	m.p. (°)
2a	Ph	115–120°C/11 h	76	>300
2b	2-Cl-Ph	115–120°C/12 h	62	>300
2c	3-Cl-Ph	115–120°C/13 h	60	>300
2d	4-Cl-Ph	115–120°C/11 h	65	>300
2e	2-CH ₃ -Ph	115–120°C/14 h	56	>300
2f	3-CH ₃ -Ph	115–120°C/13 h	61	>300
2g	4-CH ₃ -Ph	115–120°C/14 h	70	>300
2h	3-Br-Ph	115–120°C/12 h	63	>300
2i	4-Br-Ph	115–120°C/11 h	68	>300
2j	2-I-Ph	115–120°C/11 h	60	>300
2k	3-I-Ph	115–120°C/11 h	67	>300
2l	4-I-Ph	115–120°C/10 h	70	>300
2m	4-OCH ₃ -Ph	115–120°C/15 h	66	>300
2n	4-Pyridyl	115–120°C/12 h	53	>300
2o	3-Pyridyl	115–120°C/13 h	51	>300
2p	2-Furyl	115–120°C/10 h	45	>300

^aPurified yields of **2a–2p** based on *m*-ophthalmic acid.

molecular ion peaks, including some base peaks, for example, **2g** and **2o**. Compounds **2b**, **2c**, and **2d** showed molecular ion peaks corresponding to $[M^+ + 1]$ at m/z 548 and $[M^+ - 1]$ m/z 546.

EXPERIMENTAL

Melting points were determined on an X₄ melting point apparatus and were uncorrected. The IR spectra were recorded on a Nicolet Nexus 470 FT-IR spectrophotometer using KBr discs in the range 4000–400 cm⁻¹. ¹H NMR spectra were recorded on a Varian Mercury-Plus 400 NMR spectrometer in CF₃COOD or pyridine-*d*₅ solution using TMS as an internal reference. MS spectra were recorded on a Finnigan Trace GC-MS spectrometer. Elemental analyses were taken on a Perkin-Elmer-2400-CHN elemental analysis instrument.

General Procedure for the Preparation of 3-Aryl-4-amino-5-mercapto-1,2,4-triazole **1**¹²

A mixture of potassium aroyldithiocarbazate (30 mmol) and hydrazine hydrate (80%, 36 mL) was refluxed with stirring for 4–5 h at 120°C. The color of the reaction mixture changed to green, and a homogeneous

solution resulted. The acidification with HCl (3 mol/L) resulted in the precipitation of a white solid. The product was filtered, washed with cold water, and recrystallized (ethanol) to give the pure products **1**.

3-*p*-Methylphenyl-4-amino-5-mercapto-1,2,4-triazole (**1 g**)

Needle crystals, yield 55%, m.p. 217–218°. ¹H NMR (DMSO-*d*₆, 400 MHz) δ 12.7 (s, 1H, S-H), 7.51–8.24 (m, 4H, Ar-H), 5.63 (s, 2H, NH₂), 2.54 (s, 3H, CH₃); IR (cm⁻¹): 3270, 1621, 1600, 1548, 1251. Elemental anal. calcd. for C₉H₁₀N₄S: C, 52.41; H, 4.89; N, 27.16. Found: C, 52.30; H, 4.97; N, 27.03.

General Procedure for the Preparation of 1,3-bis[(3-aryl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzenes **2**

A mixture of compound 3-aryl-4-amino-5-mercapto-1,2,4-triazole (2.2 mmol), *m*-phthalic acid (1.0 mmol), the phase transfer catalyst tetrabutylammonium iodide (0.5 mmol), and POCl₃ (7 mL) was stirred for 6 h at 55–60°C, and then refluxed for 11–15 h at 115–120°C. Excess POCl₃ was removed under reduced pressure. The concentrated mass was cooled and poured into crushed ice and neutralized with potassium carbonate. The separated solid was filtered, washed with water and ethanol, and then dried. The crude material was recrystallized (ethanol-pyridine), giving the pure products **2**.

1,3-Bis[(3-phenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (**2a**)

Yellow powder, ¹H NMR (CF₃COOD, 400 MHz) δ 8.52–8.42 (m, 5H, Ar-H), 8.19–8.11 (m, 4H, Ar-H), 7.83–7.58 (m, 5H, Ar-H); IR (KBr, cm⁻¹): 1614, 1253, 702; MS (*m/z*): 478 (M⁺, 2%), 321 (2%), 303 (3%), 146 (86%), 128 (100%). elemental anal. calcd. for C₂₄H₁₄N₈S₂: C, 60.24; H, 2.95; N, 23.42. Found: C, 60.15; H, 2.88; N, 23.61.

1,3-Bis[(3-*o*-chlorophenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (**2b**)

Yellow powder, ¹H NMR (CF₃COOD, 400 MHz) 8.53–8.47 (m, 4H, Ar-H), 8.31–8.26 (m, 3H, Ar-H), 8.20–8.14 (m, 2H, Ar-H), 7.83–7.51 (m, 3H, Ar-H); IR (KBr, cm⁻¹): 1623, 1251, 701; MS (*m/z*): 548 (M⁺+1, 54%), 546 (M⁺-1, 100%), 356 (1%), 357 (22%), 146 (53%). Elemental anal. calcd. for C₂₄H₁₂N₈S₂Cl₂: C, 52.66; H, 2.21; N, 20.47. Found: C, 52.81; H, 2.13; N, 20.56.

1,3-Bis[(3-*m*-chlorophenyl)-S-triazolo[3,4-*b*]-[1,3,4]-thiadiazole-6-yl]benzene(2c)

Pale yellow powder, ^1H NMR (CF_3COOD , 400 MHz) δ 8.71–8.46 (m, 3H, Ar-H), 8.38–8.24 (m, 2H, Ar-H), 8.17–8.10 (m, 4H, Ar-H), 7.81–7.52 (m, 3H, Ar-H); IR (KBr, cm^{-1}): 1609, 1243, 700; MS (m/z): 548 ($\text{M}^+ + 1$, 65%), 546 ($\text{M}^+ - 1$, 78%), 356 (4%), 357 (12%), 146 (100%). Elemental anal. calcd. for $\text{C}_{24}\text{H}_{12}\text{N}_8\text{S}_2\text{Cl}_2$: C, 52.66; H, 2.21; N, 20.47. Found: C, 52.51; H, 2.30; N, 20.67.

1,3-Bis[(3-*p*-chlorophenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl] benzene (2d)

Yellow powder, ^1H NMR (CF_3COOD , 400 MHz) δ 8.64–8.34 (m, 7H, Ar-H), 8.05–7.71 (m, 5H, Ar-H); IR (KBr, cm^{-1}): 1627, 1254, 701; MS (m/z): 548 ($\text{M}^+ + 1$, 71%), 546 ($\text{M}^+ - 1$, 100%), 356(3%), 357 (16%), 146 (45%). Elemental anal. calcd. for $\text{C}_{24}\text{H}_{12}\text{N}_8\text{S}_2\text{Cl}_2$: C, 52.66; H, 2.21; N, 20.47. Found: C, 52.78; H, 2.30; N, 20.41.

1, 3-Bis[(3-*o*-methylphenyl)-S-triazolo[3, 4-*b*]-[1,3,4]-thiadiazole-6-yl]benzene(2e)

Pale yellow powder, ^1H NMR (CF_3COOD , 400 MHz) δ 8.46–8.41 (m, 3H, Ar-H), 8.35–8.29 (m, 2H, Ar-H), 8.12–8.05 (m, 4H, Ar-H), 7.57–7.49 (m, 3H, Ar-H), 2.56 (s, 6H, 2CH_3); IR (KBr, cm^{-1}): 1627, 1261, 695; MS (m/z): 506 (M^+ , 94%), 335 (9%), 317 (32%), 146 (21%), 117 (100%). Elemental anal. calcd. for $\text{C}_{26}\text{H}_{18}\text{N}_8\text{S}_2$: C, 61.64; H, 3.58; N, 22.12. Found: C, 61.76; H, 3.60; N, 22.25.

1,3-Bis[(3-*m*-methylphenyl)-S-triazolo[3,4-*b*]-[1,3,4]-thiadiazole-6-yl]benzene (2f)

Pale yellow powder, ^1H NMR (CF_3COOD , 400 MHz) δ 8.41–8.35 (m, 5H, Ar-H), 8.29–8.01 (m, 4H, Ar-H), 7.51–7.42 (m, 3H, Ar-H), 2.55 (s, 6H, 2CH_3); IR (KBr, cm^{-1}): 1621, 1252, 705; MS (m/z): 506 (M^+ , 100%), 335 (12%), 317 (52%), 146 (13%), 145 (67%), 117 (41%). Elemental anal. calcd. for $\text{C}_{26}\text{H}_{18}\text{N}_8\text{S}_2$: C, 61.64; H, 3.58; N, 22.12. Found: C, 61.52; H, 3.49; N, 22.27.

1,3-Bis[(3-*p*-methylphenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (2g)

Yellow powder, ^1H NMR (CF_3COOD , 400 MHz) δ 8.59–8.44 (m, 3H, Ar-H), 8.30–8.24 (m, 4H, Ar-H), 8.07–8.05 (m, 1H, Ar-H), 7.59–7.56 (m, 4H,

Ar-H), 2.57 (s, 6H, 2CH₃); IR (KBr, cm⁻¹): 1615, 1252, 701; MS (*m/z*): 506 (M⁺, 100%), 335 (12%), 317 (28%), 146 (32%), 145 (51%), 117 (32%), 116 (91%). Elemental anal. calcd. for C₂₆H₁₈N₈S₂: C, 61.64; H, 3.58; N, 22.12. Found: C, 61.52; H, 3.69; N, 22.07.

1,3-Bis[(3-*m*-bromophenyl)-S-triazolo[3,4-*b*]-[1,3,4]-thiadiazole-6-yl]benzene (2h)

Yellow powder, ¹H NMR (Pyridine-*d*₅, 400 MHz) δ 8.52–8.46 (m, 3H, Ar-H), 8.37–8.30 (m, 3H, Ar-H), 8.12–8.07 (m, 2H, Ar-H), 7.57–7.52 (m, 4H, Ar-H); IR (KBr, cm⁻¹): 1621, 1263, 704; MS (*m/z*): 636 (M⁺, 46%), 555 (8%), 399 (26), 381 (23%), 146 (100%), 101 (43%). Elemental anal. calcd. for C₂₄H₁₂N₈S₂Br₂: C, 45.31; H, 1.90; N, 17.61. Found: C, 45.42; H, 2.11; N, 17.49.

1,3-Bis[(3-*p*-bromophenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (2i)

Yellow powder, ¹H NMR (Pyridine-*d*₅, 400 MHz) δ 8.48–8.41 (m, 4H, Ar-H), 8.31–8.25 (m, 3H, Ar-H), 8.09–8.04 (m, 2H, Ar-H), 7.54–7.50 (m, 3H, Ar-H); IR (KBr, cm⁻¹): 1614, 1254, 700; MS (*m/z*): 636 (M⁺, 55%), 555 (7%), 399 (15), 381 (37%), 146 (63%), 145 (100%), 101 (61%). Elemental anal. calcd. for C₂₄H₁₂N₈S₂Br₂: C, 45.31; H, 1.90; N, 17.61. Found: C, 45.42; H, 2.11; N, 17.49.

1,3-Bis[(3-*o*-iodophenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (2j)

Yellow powder, ¹H NMR (CF₃COOD, 400MHz) δ 8.71–8.68 (m, 4H, Ar-H), 8.42–8.34 (m, 2H, Ar-H), 8.21–8.09 (m, 3H, Ar-H), 7.50–7.42 (m, 3H, Ar-H); IR (KBr, cm⁻¹): 1615, 1258, 703; MS (*m/z*): 730 (M⁺, 52%), 604 (2%), 447 (13), 429 (51%), 276 (100%), 229 (26%), 146 (38%), 145 (63%), 101 (71%). Elemental anal. calcd. for C₂₄H₁₂N₈S₂I₂: C, 39.47; H, 1.66; N, 15.34. Found: C, 39.59; H, 1.58; N, 15.42.

1,3-Bis[(3-*m*-iodophenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (2k)

Pale yellow powder, ¹H NMR (CF₃COOD, 400 MHz) δ 8.76–8.72 (m, 3H, Ar-H), 8.35–8.45 (m, 4H, Ar-H), 8.15–8.07 (m, 3H, Ar-H), 7.49–7.45 (m, 2H, Ar-H); IR (KBr, cm⁻¹): 1610, 1239, 710; MS (*m/z*): 730 (M⁺, 44%), 604 (4%), 447 (10), 429 (55%), 276 (100%), 229 (41%), 146 (50%), 145

(56%), 101 (84%). Elemental anal. calcd. for $C_{24}H_{12}N_8S_2I_2$: C, 39.47; H, 1.66; N, 15.34. Found: C, 39.59; H, 1.58; N, 15.42.

1,3-Bis[(3-*p*-iodophenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (2l)

Pale yellow powder, 1H NMR (CF_3COOD , 400 MHz) δ 8.45–8.42 (m, 2H, Ar-H), 8.13–7.81 (m, 10H, Ar-H); IR (KBr, cm^{-1}): 1620, 1247, 700; MS (m/z): 730 (M^+ , 88%), 604 (4%), 447 (19), 429 (85%), 302 (20%), 229 (39%), 146 (41%), 145 (100%), 101 (61%). Elemental anal. calcd. for $C_{24}H_{12}N_8S_2I_2$: C, 39.47; H, 1.66; N, 15.34. Found: C, 39.38; H, 1.52; N, 15.27.

1,4-Bis[(3-*p*-methoxyphenyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (2m)

Pale yellow powder, 1H NMR (CF_3COOD , 400 MHz) 8.56–8.42 (m, 4H, Ar-H), 8.31–8.25 (m, 3H, Ar-H), 8.11–8.04 (m, 3H, Ar-H), 7.42–7.40 (m, 2H, Ar-H), 3.89 (s, 6H, $2OCH_3$); IR (KBr, cm^{-1}): 1614, 1254, 700; MS (m/z): 538 (M^+ , 99%), 351 (3%), 333 (32), 146 (30%), 145 (32%), 132 (100%), 101 (22%). Elemental anal. calcd. for $C_{26}H_{18}N_8O_2S_2$: C, 57.98; H, 3.37; N, 20.80. Found: C, 57.83; H, 3.42; N, 20.73.

1,3-Bis[(3-4'-pyridyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (2n)

Pale yellow powder, 1H NMR (Pyridine- d_5 , 400 MHz) 8.51–8.38 (m, 3H, Ar-H), 8.33–8.24 (m, 4H, Ar-H), 8.17–8.00 (m, 2H, Ar-H), 7.38–7.26 (m, 3H, Ar-H); IR (KBr, cm^{-1}): 1626, 1242, 707; MS (m/z): 480 (M^+ , 100%), 322 (26), 304 (10), 146 (23%), 145 (51%), 104 (11%). Elemental anal. calcd. for $C_{22}H_{12}N_{10}S_2$: C, 54.99; H, 2.52; N, 29.15. Found: C, 54.81; H, 2.42; N, 29.26.

1,3-Bis[(3-3'-pyridyl)-S-triazolo[3,4-*b*]-[1,3,4]thiadiazole-6-yl]benzene (2o)

Yellow powder, 1H NMR (Pyridine- d_5 , 400 MHz) 8.57–8.43 (m, 4H, Ar-H), 8.35–8.22 (m, 3H, Ar-H), 8.14–8.01 (m, 3H, Ar-H), 7.31–7.25 (m, 2H, Ar-H); IR (KBr, cm^{-1}): 1614, 1254, 700; MS (m/z): 480 (M^+ , 100%), 324 (7%), 322 (42), 304 (12), 146 (27%), 145 (40%), 104 (18%). Elemental anal. calcd. for $C_{22}H_{12}N_{10}S_2$: C, 54.99; H, 2.52; N, 29.15. Found: C, 54.83; H, 2.69; N, 29.06.

1,3-Bis[(3-2'-furyl)-S-triazolo[3, 4-b]-[1,3,4]thiadiazole-6-yl]-benzene (2p)

Pale yellow powder, ^1H NMR (Pyridine- d_5 , 400 MHz) 8.45–8.34 (m, 2H, Ar-H), 8.27–8.16 (m, 2H, Ar-H), 7.12–6.37 (m, 6H, furyl-H); IR (KBr, cm^{-1}): 1614, 1254, 700; MS (m/z): 458 (M^+ , 48%), 311 (9%), 293 (64), 146 (32%), 145 (100%), 93 (27%). Elemental anal. calcd. for $\text{C}_{20}\text{H}_{10}\text{N}_8\text{O}_2\text{S}_2$: C, 52.39; H, 2.20; N, 24.40. Found: C, 52.48; H, 2.11; N, 24.31.

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