Sodium dithionite-promoted synthesis of 2-arylbenzothiazoles by reaction of 2,2'-disulfanediyldianiline with aldehydes in water

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Sodium dithionite-promoted synthesis of 2-arylbenzothiazoles by reaction of 2,2'-disulfanediyldianiline with aldehydes in the presence of sodium dodecyl sulfate in water has been developed. Possible reaction pathways are discussed and the effects of different promoter on the reaction are investigated. The important features of this methodology are simple work-up, environmentally benign, high yields, metal-free, and inexpensive reagents.

Keywords: 2-arylbenzothiazoles, 2,2'-disulfanediyldianiline, sodium dithionite

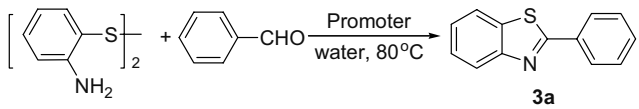
Benzothiazole is an important class of heterocycles with a wide range of pharmacological and biological activities.[1,2] Various approaches towards the synthesis of these compounds have already been explored (Scheme 1).[3-14]

One of the most practically and widely used routes for the synthesis of these compounds is the direct condensation of the 2-aminothiophenol with aldehydes.[15-20] However, this method usually suffers from limitation such as the use of foul-smelling 2-aminothiophenol. Recently, Westwell *et al.*[21,22] and Zheng *et al.*[23] reported that *p*-TsOH-catalysed synthesis of 2-arylbenzothiazoles by reaction of disulfides with aldehydes in the presence of PPh_3 in toluene under reflux conditions. Although the modifications on this reaction have been reported,[21-23] some limitations remain: for example, the use of hazardous organic solvents, toxic catalysts, long reaction times as well as moderate yields. From a synthetic point of view, the development of an improved, facile procedure using less expensive and more sustainable catalysts has remained highly desirable. As a result of our interest in developing novel synthetic routes for the formation of carbon–carbon and carbon–heteroatom bond,[24-32] we now report a new and simple sodium dithionite-promoted synthesis of 2-arylbenzothiazole by reaction of 2,2'-disulfanediyldianiline with aldehydes in water.

The model reaction of 2,2'-disulfanediyldianiline with benzaldehyde in water was conducted to screen the optimal reaction conditions and the results are listed in Table 1. Initially, the effect of promoter was tested. Among all the promoter screened, ($NaHSO_3$, $Na_2S_2O_3{\cdot}4H_2O$, $Na_2S_2O_4$, Ph_3P, and $NaHSO_2{\cdot}CHO{\cdot}2H_2O$), $Na_2S_2O_4$ afforded good yield (Table 1, 74%, entry 6).

Encouraged by this promising result, we further optimised the reaction conditions. To improve the yields, various phase transfer catalyses (PTC) were investigated (Table 2). It was found that TMAB, NH_4Cl and NH_4PF_6 can only achieve **3a** in low yields (Table 2, entries 1 and 7–8). However, it was satisfying to find that the reaction could reach to completion and afforded **3a** in 93% yield when the combination of sodium dodecyl sulfate (SDS) and $Na_2S_2O_4$ was employed (Table 2, entry 5). Moreover, we found that the yield was not significantly affected by adding different amount of SDS. Just 10 mol% of SDS was sufficient, excessive amount of catalyst did not increase the yield remarkably (Table 2, entry 5).

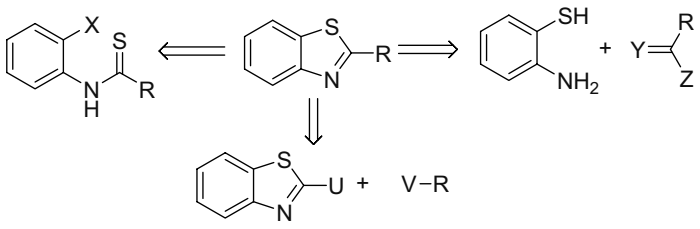
Table 1 Effect of promoters on the synthesis of 2-phenylbenzothiazole[a]



3a

Entry	Promoter/equiv	Time/h	Yield/%[b]
1	$NaHSO_3$ (2)	4	10
2	$Na_2S_2O_3{\cdot}4H_2O$ (2)	4	<5
3	$Na_2S_2O_4$ (2)	4	63
4	Ph_3P (2)	4	30
5	$NaHSO_2{\cdot}CHO{\cdot}2H_2O$ (2)	4	57
6	$Na_2S_2O_4$ (2)	6	74 (70[c], 42[d])
7	$Na_2S_2O_4$ (1)	6	60

[a]Reaction conditions: 2,2'-disulfanediyldianiline (0.5 mmol), benzaldehyde (1.1 mmol), H_2O (5 mL), 80 °C.
[b]Isolated yield.
[c]The reaction was carried out at 100 °C.
[d]The reaction was carried out at 50 °C.



X=H, Cl, Br, I, SMe; Y=O, S, Se; Z=H, OH, OR, NH_2, Cl

U=H, $SnBu_3$, $SnMe_3$, SMe; V=COOH, Br, I, ZnBr

Scheme 1 Strategies for the synthesis of 2-arylbenzothiazoles.

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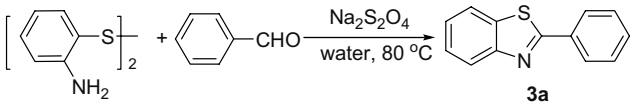
Table 2 Effect of PTC on the synthesis of 2-phenylbenzothiazole[a]



Entry	PTC/mol%	Yield/%[b]
1	TMAB[c] (10)	30
2	TBAF[d] (10)	70
3	CTAB[e] (10)	79
4	Sodium dodecyl sulfonate (10)	85
5	Sodium dodecyl sulfate (10)	93 (94[f], 94[g])
6	NH_4Cl (10)	21
7	NH_4PF_6 (10)	17

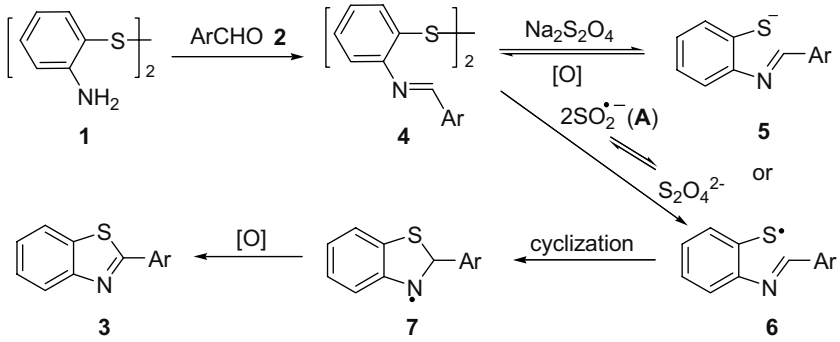
[a]Reaction conditions: 2,2'-disulfanediyldianiline (0.5 mmol), benzaldehyde (1.1 mmol), $Na_2S_2O_4$ (2 equiv), 10 mol% SDS, H_2O (5 mL), 80 °C, 6 h.
[b]Isolated yield.
[c]TMAB: Tetrmethyl ammonium bromide.
[d]TBAF: Tetrabutyl ammonium fluoride.
[e]CTAB: Cetyltrimethyl ammonium bromide.
[f]With 20 mol% SDS.
[g]With 50 mol% SDS.

In light of these results, subsequent studies were carried out under the following optimised conditions, that is, with 2 equiv. $Na_2S_2O_4$, 10 mol% SDS in water at 80 °C.

Next, we studied the scope of this reaction (Table 3). As expected, this reaction proceeded smoothly and the desired products were obtained in good to excellent yields. A series of aldehydes (**2a**–**o**) with either electron-donating or electron-withdrawing groups attaching to aromatic ring were investigated. The substitution groups on the aromatic ring had no obvious effect on the yield. The substituents on the *ortho* and opposite position have no obvious difference (Table 3, entries 2, 3, 12 and 13). Moreover, we also examined reaction of aromatic heterocyclic aldehydes such as **2n** and **2o** with 2,2'-disulfanediyldianiline, and the desired products of **3n**, **3o** were obtained in 88% and 90% yields, respectively (Table 3, entries 14 and 15). However, 4-(dimethylamino)benzaldehyde **2h** and methyl 4-formylbenzoate **2k** gave moderate yield (Table 3, entry 8 and 11).

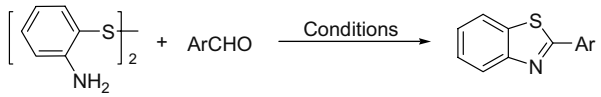
According to the above observations, a tentative mechanism for the formation of 2-arylbenzothiazoles was proposed based on the previous proposed mechanism in Scheme 2.[33-36] The first step may involve the formation of Schiff bases **4** by the reaction of 2,2'-disulfanediyldianiline with aldehydes. $Na_2S_2O_4$ can be readily decomposed into radical anion intermediates (**A**). Intermediate (**A**) then reacts with **4** to generate two intermediates anion **5** or radical **6**. Thus, intermediate **6** could be converted to intermediate **7** by intramoleculer nucleophilic attack of the sulfur to imine carbon. Subsequently, 2-arylbenzothiazoles **3** could be formed by oxidation of **7**.

In summary, a new protocol to synthesise 2-arylbenzothiazole derivatives has been developed. The present protocol enjoys simple work-up, is metal-free and has mild reaction



Scheme 2 A tentative mechanism for the formation of 2-arylbenzothiazoles.

Table 3 Sodium dithionite-promoted synthesis of 2-arylbenzothiazoles[a]



Entry	Ar	Time/h	Product	Yield/%[b]
1	C_6H_5 (**2a**)	6	**3a**	93
2	4-MeC_6H_4 (**2b**)	6.5	**3b**	90
3	4-$MeOC_6H_4$ (**2c**)	6	**3c**	92
4	4-FC_6H_4 (**2d**)	7	**3d**	85
5	4-ClC_6H_4 (**2e**)	7	**3e**	83
6	4-BrC_6H_4 (**2f**)	7	**3f**	82
7	3,4-$OCH_2OC_6H_3$ (**2g**)	7.5	**3g**	88
8	4-$N(CH_3)_2C_6H_4$ (**2h**)	7	**3h**	73
9	1-Naphthalenyl (**2i**)	8	**3i**	94
10	4-$CF_3C_6H_4$ (**2j**)	10	**3j**	89
11	4-$MeO_2CC_6H_4$ (**2k**)	10	**3k**	76
12	2-MeC_6H_4 (**2**)	8	**3l**	80
13	2-$MeOC_6H_4$ (**2m**)	8	**3m**	81
14	2-Pyridyl (**2n**)	7	**3n**	88
15	2-Furyl (**2o**)	7	**3o**	90

[a]Reaction conditions: 2,2'-disulfanediyldianiline (0.5 mmol), benzaldehyde (1.1 mmol), $Na_2S_2O_4$ (2 equiv), 10 mol% SDS, H_2O (5 mL), 80 °C.
[b]Isolated yield.

conditions. Compared to previous reported methodologies, the present protocol demonstrates simple work-up, high yields, uses metal-free and inexpensive reagents, avoids the use of hazardous solvent and does not require extra oxidants. Studies on the extension of this protocol are ongoing in our laboratory.

Experimental

Chemicals were purchased and used without further purification. All the melting points were uncorrected. ^{1}H NMR and ^{13}C NMR spectra were measured on a FT-Bruker AT-300 spectrometer (^{1}H: 300 MHz, ^{13}C: 75 MHz), using $CDCl_3$ as the solvent with tetramethylsilane (TMS) as an internal standard at room temperature. Chemical shifts are given in δ relative to TMS, the coupling constants *J* are given in Hz. Column chromatography was performed using EM Silica gel 60 (300-400 mesh). All known products were identified by comparison with authentic samples.

General synthetic procedure for synthesis of 2-arylbenzothiazoles of **3a–o**

Sodium dodecyl sulfate (SDS, 14.4 mg, 10 mol%) $Na_2S_2O_4$ (174 mg, 1 mmol) and H_2O (5 mL) was added to a mixture of benzaldehyde (1.1 mmol) and 2,2'-disulfanediyldianiline (124 mg, 0.5 mmol). The mixture was heated at 80 °C. The reaction was monitored by TLC. After completion of the reaction, the product was extracted with ethyl acetate (3 × 10 mL), the organic layer washed with brine (3 × 10 mL), then dried over Na_2SO_4 and concentrated. The product was separated and purified by column chromatography on silica gel (300–400 mesh) using an ethyl acetate/petrol mixture as the eluent to afford a pure product. When necessary, the products are purified by recrystallising with 95% ethanol.

2-Phenylbenzothiazole (**3a**): White solid, m.p. 111–112 °C (Lit.[37] 113–114 °C); ^{1}H NMR: δ 8.10–8.12 (m, 3H, ArH), 7.91(d, *J* = 7.7 Hz, 1H, ArH), 7.48–7.53 (m, 4H, ArH), 7.40 (d, *J* = 7.7 Hz, 1H, ArH). ^{13}C NMR: δ 168.1, 154.1, 135.0, 133.6, 131.0, 129.1, 127.6, 126.3, 125.2, 123.2, 121.6

2-(4-Methylphenyl)-benzothiazole (**3b**): Yellow crystal, m.p. 85–86 °C (Lit.[37] 84–85 °C); ^{1}H NMR: δ 8.05 (d, *J* = 8.0 Hz, 1H, ArH), 7.97 (d, *J* = 8.0 Hz, 2H, ArH), 7.86 (d, *J* = 8.0 Hz, 1H, ArH), 7.44–7.46 (m, 1H, ArH), 7.34–7.37 (m, 1H, ArH), 7.27 (d, *J* = 8.0 Hz, 1H, ArH), 2.40 (s, 3H, CH_3). ^{13}C NMR: δ 168.2, 154.2, 141.4, 135.0, 131.0, 129.7, 127.5, 126.2, 125.0, 123.0, 121.5, 21.5.

2-(4-Methoxyphenyl)benzothiazole (**3c**): Yellow crystal, m.p. 120–121 °C (Lit.[38] 119–121 °C); ^{1}H NMR: δ 8.04–8.07 (m, 3H, ArH), 7.90 (m, 1H, ArH), 7.48 (d, *J* = 7.3 Hz, 1H, ArH), 7.37 (d, *J* = 7.3 Hz, 1H, ArH), 7.01–7.04 (m, 2H, ArH), 3.90 (s, 3H, OCH_3). ^{13}C NMR: δ 168.0, 162.1, 154.4, 135.1, 129.3, 126.6, 126.4, 125.0, 123.0, 121.7, 114.5, 55.6.

2-(4-Fluorophenyl)benzothiazole (**3d**): White crystal, m.p. 98–100 °C (Lit.[38] 98–99 °C); ^{1}H NMR: δ 8.03–8.07 (m, 3H, ArH), 7.85 (d, *J* = 8.0 Hz, 1H, ArH), 7.47 (d, *J* = 7.7 Hz, 1H, ArH), 7.35 (d, *J* = 7.7 Hz, 1H, ArH), 7.15 (t, *J* 8.0 Hz, 1H, ArH). ^{13}C NMR: δ 166.9, 163.0 (d, $^1J_{C\text{-}F}$ = 250.3 Hz), 154.3, 135.2, 130.1, 129.7 (d, $^3J_{C\text{-}F}$ = 7.0 Hz), 129.6, 125.4, 123.4 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 121.7, 116.3 (d, $^2J_{C\text{-}F}$ = 22.0 Hz).

2-(4-Chlorophenyl)benzothiazole (**3e**): Yellow crystal, m.p. 113–114 °C (Lit.[39] 113 °C); ^{1}H NMR: δ 7.87–8.06 (m, 4H, ArH), 7.35–7.51(m, 4H, ArH). ^{13}C NMR: δ 166.8, 154.3, 137.2, 135.3, 132.3, 129.5, 128.9, 126.7, 125.6, 123.5, 121.8.

2-(4-Bromophenyl)benzothiazole (**3f**): Yellow crystal, m.p. 131–132 °C (Lit.[33] 130–131 °C); ^{1}H NMR: δ 7.37–8.08(m, 8H, ArH). ^{13}C NMR: δ 166.7, 154.1, 135.0, 132.5, 132.2, 128.9, 126.5, 125.5, 125.4, 123.3, 121.6.

2-(Benzo[1, 3]dioxol-5-yl)benzothiazole (**3g**): Yellow crystal, m.p. 125–127 °C (Lit.[37] 127–128 °C); ^{1}H NMR: δ 8.04 (d, *J* = 8.1 Hz, 1H, ArH), 7.90 (d, *J* = 8.1 Hz, 1H, ArH), 7.60–7.63 (m, 2H, ArH), 7.46–7.49 (m, 1H, ArH), 7.38–7.41 (m, 1H, ArH), 7.00 (d, *J* = 7.8 Hz, 1H, ArH), 6.08 (s, 2H, OCH_2O). ^{13}C NMR: δ 167.5, 154.1, 150.1, 148.4, 134.9, 128.0, 126.2, 124.9, 122.9, 122.5, 121.5, 108.6, 107.5, 101.7.

2-(4-N,N-dimethylaminophenyl)benzothiazole (**3h**): Brown crystal, m.p. 173–175 °C (Lit.[37] 176–178 °C); ^{1}H NMR: δ 7.98 (dd, *J* = 7.0 Hz, *J* = 2.0 Hz, 3H, ArH), 7.83–7.86 (m, 1H, ArH), 7.42–7.45(m, 1H, ArH), 7.3– 7.33 (m, 1H, ArH), 6.74 (dd, *J* = 2.0 Hz, *J* = 7.0 Hz, 2H, ArH), 3.05(s, 6H, $N(CH_3)_2$). ^{13}C NMR: δ 168.8, 154.4, 152.1, 134.5, 128.8, 125.9, 124.1, 122.2, 121.3, 111.6, 40.1.

2-(Naphthalen-1-yl)benzothiazole (**3i**): White crystal, m.p. 125–127 °C (Lit.[37] 126 °C); ^{1}H NMR: δ 8.97 (d, *J* = 8.3 Hz, 1H, ArH), 8.23 (d, *J* = 7.8 Hz, 1H, ArH), 7.95–8.03 (m, 4H, ArH), 7.58–7.63 (m, 4H, ArH), 7.48–7.56 (m, 1H, ArH). ^{13}C NMR: δ 167.7, 154.2, 135.5, 134.1, 131.1, 130.9, 130.7, 129.5, 128.5, 127.7, 126.6, 126.3, 126.0, 125.3, 125.0, 123.6, 121.5.

2-(4-(Trifluoromethyl) phenyl) benzothiazole (**3j**)[40]: Yellow crystal, m.p. 160–162 °C; ^{1}H NMR: δ 8.08–8.18 (m, 3H, ArH), 7.90(d, *J* = 8.1 Hz, 1H, ArH), 7.70 (d, *J* = 8.1 Hz, 2H, ArH), 7.39–7.7.54 (m, 2H, ArH). ^{13}C NMR: δ 165.9, 153.9, 136.6, 135.1, 132.3(q, $^2J_{C\text{-}F}$ = 32.8 Hz, *C*-CF_3), 127.7, 126.6, 125.8(q, $^3J_{C\text{-}F}$ = 3.8 Hz, *C*H–C–CF_3), 125.7, 124.6(q, $^1J_{C\text{-}F}$ = 270.1 Hz, CF_3), 123.5, 121.7.

*2-(4-Methoxycarbonylphenyl) benzothiazol*e (**3k**): White crystal, m.p. 166–167 °C (Lit.[41] 166 °C); ^{1}H NMR: δ 8.07–8.12 (m, 5H, ArH), 7.87–7.89 (m, 1H, ArH), 7.39–7.50 (m, 2H, ArH), 3.94 (s, 3H, OCH_3). ^{13}C NMR: δ 166.4, 166.3, 150.4, 137.3, 135.2, 131.9, 130.1, 127.3, 126.5, 125.6, 123.5, 121.6, 52.25.

2-(2-Methylphenyl)-benzothiazole (**3l**): White crystal, m.p. 53–54 °C (Lit.[42] 53–54 °C); ^{1}H NMR: δ8.1(d, *J* = 8.0 Hz, 1H, ArH), 7.90 (d, *J* = 8.0 Hz, 1H, ArH), 7.75 (d, *J* = 7.6 Hz, 1H, ArH), 7.50 (d, *J* = 7.6 Hz, 1H, ArH), 7.26–7.41 (m, 4H, ArH), 2.65 (s, 3H, CH_3). ^{13}C NMR: δ 163.1, 157.2, 152.1, 136.1, 131.7, 129.5, 125.8, 124.5, 122.7, 122.2, 121.2, 121.1, 111.6, 55.6.

2-(2-Methoxyphenyl)-benzothiazole (**3m**):[42] White crystal, m.p. 120–122° C; ^{1}H NMR: δ 7.03–8.58 (m, 8H, ArH), 4.03 (s, 3H, OCH_3). ^{13}C NMR: δ 162.8, 156.9, 151.9, 135.8, 131.4, 129.2, 125.6, 124.3, 122.5, 120.9, 120.8, 111.4, 55.4.

2-(Pyridin-2-yl) benzothiazole (**3n**): White crystal, m.p. 136–137 °C (Lit.[37] 136–137 °C); ^{1}H NMR: δ 8.64–8.65 (m, 1H, ArH), 8.32–8.35 (m, 1H, ArH), 8.05–8.08 (m, 1H, ArH), 7.91–7.93 (m, 1H, ArH), 7.76–7.79(m, 1H, ArH), 7.32–7.47 (m, 2H, ArH). ^{13}C NMR: δ 169.5, 154.5, 151.6, 149.8, 137.1, 136.3, 126.4, 125.8, 125.4, 123.8, 122.2, 120.9.

2-(Furan-2-yl)benzothiazole (**3o**): Yellow crystal, m.p. 102–104 °C (Lit.[37] 103 °C); ^{1}H NMR: δ 8.05 (d, *J* = 8.1 Hz, 1H, ArH), 7.90 (d, *J* = 8.1 Hz, 1H, ArH), 7.60 (s, 1H, ArH), 7.46–7.7.51 (m, 1H, ArH), 7.35–7.40 (m, 1H, ArH), 7.18–7.19 (m, 1H, ArH), 6.59–6.60 (m, 1H, ArH). ^{13}C NMR: δ 157.7, 153.9, 140.9, 144.9, 134.5, 126.6, 125.3, 123.3, 121.7, 112.7, 111.6.

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