Catalyst-free reductive cyclization of bis(2-aminophenyl) disulfide with CO$_2$ in the presence of BH$_3$NH$_3$ to synthesize 2-unsubstituted benzothiazole derivatives†

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An efficient and catalyst-free methodology for the reductive cyclization of various disulfides using BH$_3$NH$_3$ as a reductant and CO$_2$ as a C1 resource was developed. The desired 2-unsubstituted benzothiazole derivatives were obtained in good to excellent yields. Moreover, mechanism investigation demonstrated that BH$_3$NH$_3$ played an important role in the formation of benzothiazole. As a reducing agent, BH$_3$NH$_3$ reduced CO$_2$ and cleaved the S–S bond of the disulfide efficiently. In addition, the N–H bond of the amino group was also activated by BH$_3$NH$_3$. To the best of our knowledge, this is an unprecedented catalyst-free protocol for the synthesis of 2-unsubstituted benzothiazole from bis(2-aminophenyl) disulfide and CO$_2$.

1 Introduction

CO$_2$ is a low-cost, sustainable, and abundant C1 resource which has been widely employed in the synthesis of value-added chemicals.$^{1-3}$ Among the various reactions reported for the transformations of CO$_2$, reductive functionalization of amines with CO$_2$ in the presence of a reducing reagent has attracted extensive attention.$^{4-7}$ Significantly, new bonds such as C–N, C–O, C–C or C–S bonds were formed in the process of reductive functionalization by reducing CO$_2$ with different functionalization reagents.$^{8-12}$ Among these, formations of C–N and C–S bonds are important transformations in organic synthesis.

One of the common compounds containing both C–N and C–S bonds is benzothiazole, a bicyclic compound in which a thiazole ring is fused with a benzene ring. It is an important synthetic intermediate and an important privileged scaffold of many natural compounds and/or bioactive compounds.$^{13}$ Benzothiazole is used in the synthesis of pharmaceutical compounds such as ethoxazolamide, riluzole and florapronol (Fig. 1). In particular, 2-unsubstituted benzothiazole is the core structure that can be used to synthesize a variety of valuable 2-substituted benzothiazoles.

There are many methods to prepare 2-unsubstituted benzothiazole derivatives.$^{14}$ It is worth noting that various raw materials have been utilized via different reaction routes in the synthesis of 2-unsubstituted benzothiazole. Among these raw materials, 2-aminothiophenol has attracted extensive attention of researchers. The most commonly used method is the condensation reaction of 2-aminothiophenol with formic acid, formamide,$^{15}$ or formaldehyde$^{17}$ in the presence of a catalyst. Recently, the synthesis of 2-unsubstituted benzothiazole using CO$_2$ as the carbon source has been reported, in the presence of various reductants and catalysts (Scheme 1, route a). With H$_2$ as the reductant, 2-unsubstituted benzothiazole could be obtained from the reaction of CO$_2$ and 2-aminothiophenol catalyzed by CoF$_2$/PP$_3$/CsF.$^{18}$ It should be noted that both H$_2$ and CO$_2$ need to be activated. Therefore, high temperatures and high pressures are needed in the reaction. With hydroisilanes as the reductant, 2-unsubstituted benzothiazole can be obtained from the reaction of CO$_2$ and 2-aminothiophenol catalyzed by 1,5-diazabicyclo[4.3.0]non-5-ene (DBN)$^{19}$, 1,5,7-triazabicyclo[4.4.0]
unsubstituted benzothiazole compounds.

Part of our ongoing work, we investigated BH$_3$NH$_3$ as a reducing reagent, which could reduce CO$_2$ and the S-de and CO$_2$, in which C-N and C-S bonds were constructed simultaneously. This simple and effective method could be applied in the synthesis of many 2-unsubstituted benzothiazole compounds.

2 Results and discussion

The model reaction of bis(2-aminophenyl) disulfide (1a, 0.5 mmol) and CO$_2$ to synthesize benzothiazole (2a) was initially explored. First, the reaction was carried out in NMP at 1 MPa of CO$_2$, with 3 equiv. of BH$_3$NH$_3$ at 100 °C in 24 h, and the desired product 2a was obtained in 32% yield (Table 1, entry 1). Notably, a large amount of disulfide was recovered, which demonstrated that the S-S bond of disulfide was not easily cleaved under this reaction condition and the raw material was not completely consumed. To improve the yield of target product, the reaction temperature was increased from 100 °C to 120 °C, which increased the yield of 2a from 32% to 85% (Table 1, entries 1–3). These experimental results indicated that higher temperature is beneficial to the cleavage of S-S bond. However, as the reaction temperature was increased to 140 °C, the yield of target product decreased to 46% (Table 1, entries 4–5), possibly because the higher reaction temperature decreased the solubility of CO$_2$ and further reduced CO$_2$ into methylation by-products. Consequently, the reaction temperature would be 120 °C in subsequent experiments.

To further improve the yield of 2a, the amount of BH$_3$NH$_3$ and the pressure of CO$_2$ were optimized (Table 1, entries 3, 6–9). Increasing the amount of BH$_3$NH$_3$ reduced the yield of the target product, which demonstrated that the amount of BH$_3$NH$_3$ was critical to the reaction. The excessive BH$_3$NH$_3$ might further reduce CO$_2$ into methylated by-products. When the amount of BH$_3$NH$_3$ was 2.5 equiv. of 1a, the yield of 2a reached 93% (Table 1, entry 6). However, when the amount of BH$_3$NH$_3$ was further reduced, the yield of 2a was also reduced (Table 1, entries 7 and 8), suggesting that the amount of reducing agent is insufficient. Therefore, 2.5 equivalents of reducing agent were used in the subsequent reactions of 1a and CO$_2$. Furthermore, the effect of CO$_2$ pressure on the reaction yield was also examined (Table 1, entries 6, 10–12). Only trace amount of 2a was found at 0.1 MPa CO$_2$ (Table 1, entry 10), indicating that higher concentration of CO$_2$ was needed in the reaction of 1a and CO$_2$. Increasing the pressure of CO$_2$ to 1 MPa led to a higher yield of 2a (Table 1, entry 6). When the CO$_2$ pressure was further increased to 2 MPa, the yield of 2a decreased significantly (Table 1, entry 12). These experimental results indicated that BH$_3$NH$_3$ is consumed by excess CO$_2$, resulting in insufficient amounts of BH$_3$NH$_3$ to break the S-S bond. Therefore, the balanced optimization of the reaction temperature, the amount of BH$_3$NH$_3$ and CO$_2$ pressure was critical for the high-yield formation of 2a from 1a and CO$_2$.

The effect of solvent on the reaction yield was also investigated. When weakly polar solvents such as 1,4-dioxane, CH$_3$CN and THF were used (Table 1, entries 13–15), 2a was obtained in lower yields. When DMSO was used (Table 1, entry 16), a lower yield of 2a was obtained, likely due to the oxidizing property of DMSO to convert thiol into disulfide. When DMF was used (Table 1, entries 17 and 18), 2a was obtained in 80% yield. However, 2a could also be obtained in a yield of 75% in the absence of CO$_2$, because DMF can serve as the C1 source to produce the target product in the presence of BH$_3$NH$_3$. Water was not suitable for the reaction system (Table 1, entry 19), because the formed carbonic acid promoted the hydrolysis of BH$_3$NH$_3$. To our delight, an excellent yield of 2a was obtained in NMP; therefore, NMP was chosen as the solvent in the following experiments. Finally, the reaction time was also
investigated (Table 1, entries 6, 20–22), and the highest yield was obtained in 24 h. Therefore, 1 equiv. of 1a reacted with 1 MPa of CO₂ in the presence of 2.5 equiv. BH₃NH₃ in 120 °C for 24 h to give the highest yield of the target product. With the optimized reaction conditions in hand, the reaction scope was investigated with a range of differently substituted bis(2-aminophenyl) disulfide (Table 2). We found that all the substrates reacted with CO₂ and BH₃NH₃ to produce the corresponding target products in good to excellent yields. The reaction of 1a with CO₂ and BH₃NH₃ provided 2a in an isolated yield of 93% under the optimized reaction conditions (Table 2, entry 1). The substrates with electron-donating groups like methyl and methoxy displayed higher reactivity, and the corresponding products were obtained in excellent yields (86–95%, Table 2, entries 2–4). The substrates with electron-withdrawing groups including –F, –Cl, –Br and –CF₃ displayed good reactivity, and the corresponding products were obtained in good yields (63–85%, Table 2, entries 5–10). Furthermore, bis(2-aminophenyl) disulfides bearing functional groups at the C4 position showed higher reactivity than those at the C6 position, (Table 2, entries 2 vs. 3; entries 6 vs. 8) which presumably contributed to the steric effects of substituent groups. When the substituent group was –SO₂CH₃, a strong electron-withdrawing group, corresponding 6-(methylsulfonyl)-benzothiazole was obtained in a yield of 55% (Table 2, entry 11). In addition, naphtho[2,3]-thiazole could be obtained in 66% yield from the corresponding disulfide (Table 2, entry 12). To the best of our knowledge, this is the first example of successfully synthesizing 6-(methylsulfonyl)-benzothiazole and naphtho[2,3]-thiazole using CO₂ as the carbon source.

To verify the applicability of the developed methodology, the reductive cyclization of disulfide with CO₂ to synthesize benzothiazole was carried out on the gram scale (Scheme 2). The desired product was obtained in 80% isolated yield, demonstrating that this methodology can be used in gram-scale synthesis.

To explore the reaction mechanism, several control experiments were performed under the optimized conditions. As shown in Scheme 3, no desired product was detected in the absence of BH₃NH₃ or CO₂, which indicated that both BH₃NH₃ and CO₂ were indispensable (Scheme 3). When BH₃NH₃ and CO₂ were mixed in NMP, it was interesting to find that the formate borohydride species BH₃COOH was formed, which was confirmed by ¹H NMR (8.31 ppm, 8.04 ppm), ¹³C NMR (166.38 ppm), and ²⁷B NMR (19.39 ppm) (Fig. S1†) analysis. The experimental results are similar with the data reported in the literature.¹⁴,⁴₅–⁴⁷

### Table 1  Optimization of the reaction conditions

| Entry | BH₃NH₃ (mmol) | Solvent (mL) | P CO₂ (MPa) | T (°C) | t (h) | Yield[^b] [%] |
|-------|-------------|-------------|------------|------|-----|--------------|
| 1     | 1.5         | NMP         | 1          | 100  | 24  | 32           |
| 2     | 1.5         | NMP         | 1          | 110  | 24  | 68           |
| 3     | 1.5         | NMP         | 1          | 120  | 24  | 85           |
| 4     | 1.5         | NMP         | 1          | 130  | 24  | 75           |
| 5     | 1.5         | NMP         | 1          | 140  | 24  | 46           |
| 6     | 1.25        | NMP         | 1          | 120  | 24  | 93           |
| 7     | 1           | NMP         | 1          | 120  | 24  | 68           |
| 8     | 0.5         | NMP         | 1          | 120  | 24  | 30           |
| 9     | 2           | NMP         | 1          | 120  | 24  | 70           |
| 10    | 1.25        | NMP         | 0.1        | 120  | 24  | Trace        |
| 11    | 1.25        | NMP         | 0.5        | 120  | 24  | 46           |
| 12    | 1.25        | NMP         | 2          | 120  | 24  | 62           |
| 13    | 1.25        | 1,4-Dioxane | 1          | 120  | 24  | 13%          |
| 14    | 1.25        | CH₃CN       | 1          | 120  | 24  | 25%          |
| 15    | 1.25        | THF         | 1          | 120  | 24  | 18%          |
| 16    | 1.25        | DMSO        | 1          | 120  | 24  | 26%          |
| 17    | 1.25        | DMF         | 1          | 120  | 24  | 80%          |
| 18    | 1.25        | DMF         | 0          | 120  | 24  | 75%          |
| 19    | 1.25        | H₂O         | 1          | 120  | 24  | Trace        |
| 20    | 1.25        | NMP         | 1          | 120  | 18  | 83           |
| 21    | 1.25        | NMP         | 1          | 120  | 12  | 60           |
| 22    | 1.25        | NMP         | 1          | 120  | 6   | 25           |

[^a] Reaction conditions: bis(2-aminophenyl) disulfide (0.1242 g, 0.5 mmol), solvent (1 mL).
[^b] Isolated yield.
[^c] No CO₂.
finding suggested that BH$_3$·(OCOH)$_n$NH$_3$ (I) was an intermediate for the synthesis of benzothiazole.

Furthermore, $^1$H NMR analysis was employed to identify the cleavage of S–S bond of disulfide by BH$_3$NH$_3$ during the reaction.
process (Fig. 2). As illustrated in Fig. 2c, a peak pattern similar to that of 2-aminothiophenol appeared when disulfide reacted with BH$_3$NH$_2$ under an inert atmosphere at 120 °C in deuterated DMF (Fig. 2c red rectangle). However, the aromatic hydrogen shifted to upfield in 2-aminothiophenol after reduced by BH$_3$NH$_2$, which might be resulted from the coordination of boron atom of BH$_3$NH$_2$ with the nitrogen atom of 2-aminothiophenol. In addition, the $^1$H NMR spectra of reaction mixture (Fig. 2e) showed that the –NH$_2$ group hydrogen in 2-aminothiophenol formed from disulfide reduction shifted downfield from 4.87 to 5.19 (Fig. 2e blue rectangle), indicating that the N–H bond of 2-aminothiophenol was activated. These results demonstrated that BH$_3$NH$_2$ reduced disulfide to 2-aminothiophenol and activated the N–H bond of amino group, which increased the nucleophilicity of the amino group in 2-aminothiophenol.

Based on our experimental results and previous reports, a possible reductive cyclization reaction mechanism was proposed (Scheme 4). Firstly, BH$_3$NH$_2$ reacts with CO$_2$ to produce the intermediate BH$_3$–(OCOH)$_n$NH$_2$ (I). Meanwhile, the S–S bond of the disulfide is cleaved by BH$_3$NH$_2$ to form complex (II) of 2-aminothiophenol with BH$_3$. Subsequently, the nucleophilic N atom of 2-aminothiophenol attacks the carbon atom of BH$_3$–(OCOH)$_n$NH$_2$ (I) to form intermediate III. Finally, intermediate IV is formed through the intramolecular nucleophilic cyclization of intermediate III, followed by dehydration to yield the target product benzothiazole.

3 Experimental

3.1 General information

All reagents and solvents were purchased from commercial suppliers and used without further purification. All reactions were monitored by TLC with GF254 silica gel coated plates. Purification of reaction products were carried out by chromatography using silica gel (200–300 mesh). The $^1$H, $^{13}$C and $^{11}$B NMR spectra were recorded on an Agilent 500 MHz DD2 spectrometer at 500 MHz ($^1$H), 126 MHz ($^{13}$C) and 160 MHz ($^{11}$B) in $d_6$-DMSO, or CDCl$_3$ or D$_2$O using tetramethylsilane (TMS) or solvent residue as internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. Melting points were measured with SGC X-4 microscopic melting point meter and were uncorrected. Molecular weights were obtained using Shimadzu LCMS-2020 (ESI) instrument.

3.2 General procedure for the synthesis of benzothiazoles (2a–2l)

A stainless-steel autoclave reactor equipped with a magnetic stirrer was charged with bis(2-amino phenyl) disulfide (0.5 mmol), BH$_3$NH$_2$ (1.25 mmol) and NMP (1 mL). Then the stainless-steel autoclave was sealed and pressurized with 1 MPa of CO$_2$ after air was exchanged by CO$_2$ at ambient temperature. And then it was heated and stirred at 120 °C for 24 h. When the reaction was completed, it was cooled down to room temperature and the excessive CO$_2$ was released slowly. Subsequently, the reaction mixture solution was quenched by brine water and extracted with ethyl acetate three times. The combined organic layers were dried over anhydrous MgSO$_4$ and evaporated under reduced pressure. The desired products were obtained in good to excellent yields after purification by column chromatography on silica gel using petroleum ether/ethyl acetate as the eluent. All the desired products were identified through comparisons with the corresponding $^1$H NMR, $^{13}$C NMR data reported in the literature.

3.3 Mechanistic study

3.3.1 Reaction of BH$_3$NH$_2$ and CO$_2$ in NMP. A stainless-steel autoclave reactor equipped with a magnetic stirrer was charged with BH$_3$NH$_2$ (1 mmol) and NMP (1 mL). The reactor was pressurized with 1 MPa of CO$_2$ at ambient temperature, and then was heated and stirred at 120 °C for 24 h. After the reaction was completed, the reactor was cooled to room temperature and the excessive CO$_2$ was vented discreetly. Subsequently, 0.1 mL of the reaction mixture solution was quenched by brine water and extracted with ethyl acetate three times. The combined organic layers were dried over anhydrous MgSO$_4$ and evaporated under reduced pressure. The desired products were obtained in good to excellent yields after purification by column chromatography on silica gel using petroleum ether/ethyl acetate as the eluent.

3.3.2 Reaction of disulfide and BH$_3$NH$_2$ in deuterated DMF. The disulfide (0.05 mmol) was dissolved with 0.5 mL deuterated DMF in an NMR tube and detected by $^1$H NMR. The $^1$H NMR spectra of 1a in deuterated DMF was obtained (Fig. S2†). Then, the disulfide (0.05 mmol) and BH$_3$NH$_2$ (0.05 mmol) were dissolved with 0.5 mL deuterated DMF in an NMR tube under the inert atmosphere and detected by $^1$H NMR. The $^1$H NMR spectra of the mixture of 1a and BH$_3$NH$_2$ in deuterated DMF were obtained (Fig. S3†). Subsequently, the mixture solution was then heated to 120 °C for an indicated time, and then the reaction solution was detected by $^1$H NMR at 0.5 h, 1 h, 2 h and 5 h. The corresponding $^1$H NMR are measured and shown in Fig. S4†. Finally, the 2-aminothiophenol was dissolved with 0.5 mL deuterated DMF in an NMR tube and detected by $^1$H NMR. The $^1$H NMR spectra of 2-aminothiophenol was obtained (Fig. S5†). Additionally, the stacked $^1$H NMR spectra of Fig. S2–S5† were shown in Fig. S6†.
3.4 Characterization (NMR) of the products

3.4.1 Benzothiazole (2a). Isolated as a yellow oil (PE/EA = 3 : 1, 0.126 g, 93%); 1H NMR (500 MHz, chloroform-d) δ 9.00 (s, 1H), 2.47 (d, J = 8.1 Hz, 1H), 7.74 (d, J = 8.1 Hz, 1H), 12.54 (s, 1H), 12.87 (s, 1H), 152.20, 151.52, 131.91, 131.73, 127.12, 124.33, 121.49. MS (ESI): m/z calcd for C14H13NS [M + H]+: 213.08, found 213.06.

3.4.2 4-Methyl-benzothiazole (2b). Isolated as a yellow oil (PE/EA = 6 : 1, 0.128 g, 86%); 1H NMR (500 MHz, chloroform-d) δ 8.98 (s, 1H), 2.47 (d, J = 8.1 Hz, 1H), 7.74 (d, J = 8.1 Hz, 1H), 12.54 (s, 1H), 12.87 (s, 1H), 152.20, 151.52, 131.91, 131.73, 127.12, 124.33, 121.49. MS (ESI): m/z calcd for C14H13NS [M + H]+: 213.08, found 213.06.

3.4.3 6-Methyl-benzothiazole (2c). Isolated as a yellow oil (PE/EA = 6 : 1, 0.141 g, 95%); 1H NMR (500 MHz, chloroform-d) δ 8.98 (s, 1H), 2.47 (d, J = 8.1 Hz, 1H), 7.74 (d, J = 8.1 Hz, 1H), 12.54 (s, 1H), 12.87 (s, 1H), 152.20, 151.52, 131.91, 131.73, 127.12, 124.33, 121.49. MS (ESI): m/z calcd for C14H13NS [M + H]+: 213.08, found 213.06.

3.4.4 6-Fluoro-benzothiazole (2d). Isolated as a yellow solid (DCM/EA = 10 : 1, 0.096 g, 63%); 1H NMR (500 MHz, chloroform-d) δ 9.00 (s, 1H), 2.47 (d, J = 8.1 Hz, 1H), 7.74 (d, J = 8.1 Hz, 1H), 12.54 (s, 1H), 12.87 (s, 1H), 152.20, 151.52, 131.91, 131.73, 127.12, 124.33, 121.49. MS (ESI): m/z calcd for C14H13NS [M + H]+: 213.08, found 213.06.

3.4.5 4-Chloro-benzothiazole (2e). Isolated as a yellow oil (DCM/EA = 10 : 1, 0.119 g, 70%); 1H NMR (500 MHz, chloroform-d) δ 9.08 (s, 1H), 7.39 (t, J = 8.1 Hz, 1H), 2.47 (d, J = 8.1 Hz, 1H), 7.74 (d, J = 8.1 Hz, 1H), 7.47 (d, J = 8.1 Hz, 1H), 12.54 (s, 1H), 12.87 (s, 1H), 152.20, 151.52, 131.91, 131.73, 127.12, 124.33, 121.49. MS (ESI): m/z calcd for C14H13NS [M + H]+: 213.08, found 213.06.

3.4.6 4-Chloro-benzothiazole (2f). Isolated as a yellow solid (DCM/EA = 10 : 1, 0.119 g, 70%); 1H NMR (500 MHz, chloroform-d) δ 9.08 (s, 1H), 7.39 (t, J = 8.1 Hz, 1H), 2.47 (d, J = 8.1 Hz, 1H), 7.74 (d, J = 8.1 Hz, 1H), 12.54 (s, 1H), 12.87 (s, 1H), 152.20, 151.52, 131.91, 131.73, 127.12, 124.33, 121.49. MS (ESI): m/z calcd for C14H13NS [M + H]+: 213.08, found 213.06.
the utilization of CO2 in the synthesis of benzothiazole group. This simple and green route provides a new method for synthesizing 2-unsubstituted benzothiazole derivatives by the reductive cyclization of bis(2-aminophenyl) disulfide with CO2 was realized, which could be used to prepare a variety of 2-unsubstituted benzothiazole derivatives in good to excellent yields. The reaction mechanism investigation demonstrated that BH3NH3 plays an important role in the formation of benzothiazole, as an efficient reductant to reduce CO2, to cleave the S-S bond of disulfide and to activate the N-H bond of amino group. This simple and green route provides a new method for the utilization of CO2 in the synthesis of benzothiazole derivatives.

4 Conclusions
In conclusion, a green, catalyst-free and efficient approach to synthesize 2-unsubstituted benzothiazole derivatives by the reductive cyclization of bis(2-aminophenyl) disulfide with CO2 was realized, which could be used to prepare a variety of 2-unsubstituted benzothiazole derivatives in good to excellent yields. The reaction mechanism investigation demonstrated that BH3NH3 plays an important role in the formation of benzothiazole, as an efficient reductant to reduce CO2, to cleave the S-S bond of disulfide and to activate the N-H bond of amino group. This simple and green route provides a new method for the utilization of CO2 in the synthesis of benzothiazole derivatives.

Conflicts of interest
The authors declare no conflict of interest.

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