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N-Heterocyclic carbene catalyzed *aza*-benzoin reaction for access to α-aminoketone molecules containing benzothiazole fragments

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ABSTRACT

An *N*-Heterocyclic carbene (NHC) catalyzed *aza*-benzoin reaction of benzothiazole-2-carboxaldehydes and *N*-sulfonylimines is reported for the first time. The target benzothiazole-containing aminoketone products bearing different substituents and substitution patterns can be obtained in good to excellent yields under mild conditions.

1. Introduction

Benzothiazole derivatives are interesting molecules with proven biological activities. Substituted benzothiazoles are frequently found as core structures in various human medicines and agrichemicals [1]. For instance, Ac-KQLR ketobenzothiazole (kbt) is a serine protease inhibitor and has potential application in the treatment of cancer [2]. Benthiavalicarb-isopropyl is a commercially available fungicide that has been widely used to prevent various plant diseases. Riluzole is currently the only commercial drug approved by the U.S. Food and Drug Administration (FDA) for the treatment of amyotrophic lateral sclerosis (ALS) (Fig. 1a). Therefore, the synthesis of benzothiazole derivatives with various functionalities has long been attractive.

N-Heterocyclic carbenes (NHCs) have proven to be efficient organic catalysts for the preparation of a variety of functional molecules [3]. (*Aza*)-benzoin reaction represents one of the most basic and significant transformations in the field of NHC organocatalysis [4]. *N*-sulfonylimines are versatile starting materials and can be used as both electrophiles and nucleophiles in organic transformations enabled by different reaction catalysts. However, to the best of our knowledge, *N*-sulfonylimines have never been used as reactant in NHC-catalyzed *aza*-benzoin reactions. Challenges might exist in the differentiation of the two electrophilic substrates by the NHC catalysts in the catalytic systems. For example, Hou [5], Chi [6] and others [7] have reported that *N*-sulfonylimines can be efficiently activated by the NHC catalysts and liberate free sulfonic anion to react with electrophiles (Fig. 1b, eq. 1). Ye [8] and others [9] have disclosed that the *N*-sulfonylimines can be used as electrophiles in the NHC-catalyzed Morita-Baylis-Hillman reactions and the [2 + 2] cycloaddition reactions with azoliumenolates (Fig. 1b, eq. 2).

Herein, we disclose an *aza*-benzoin reaction of the benzothiazole-2-carboxaldehydes with the *N*-sulfonylimines for access to α-aminoketone molecules containing benzothiazole fragments (Fig. 1c). Benzothiazole-2-carboxaldehydes are selectively activated by the NHC catalysts and behave as nucleophiles to react with the electrophilic *N*-sulfonylimines. α-Aminoketones bearing benzothiazole structures are afforded as the final products in good to excellent yields under mild conditions. It is worth noting that although imine molecules other than the *N*-sulfonylimines have been used in the aza-benzoin reactions with various carbaldehydes

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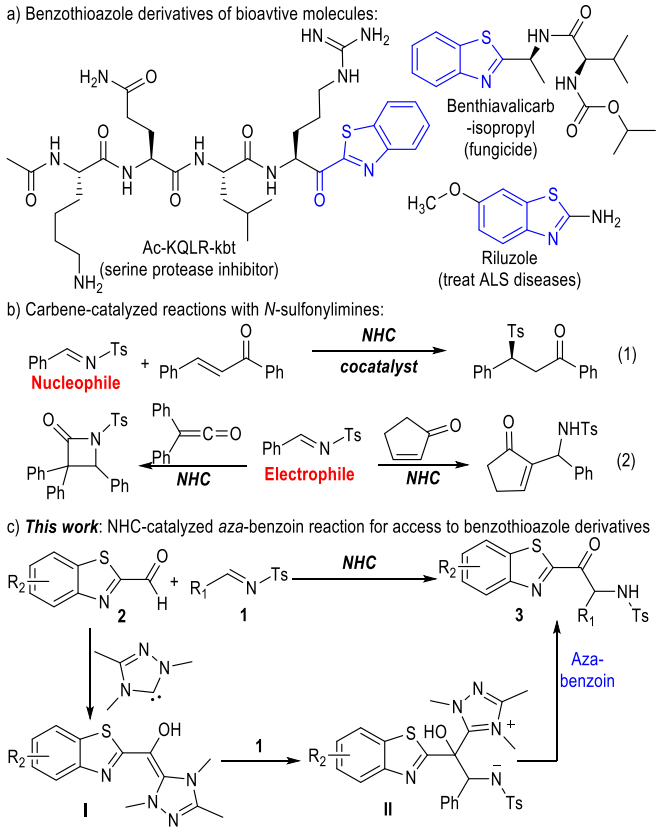
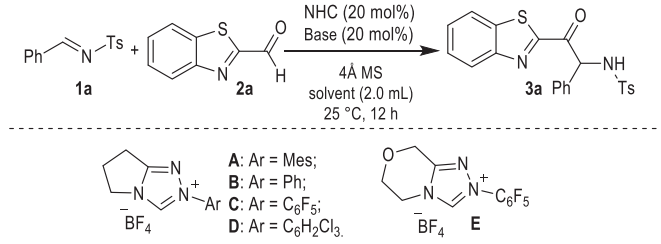


Fig. 1. Bioactive benzothiazole derivatives and NHC-catalyzed *Aza*-benzoin reaction.

[4b-4g,4j], the aza-benzoin reaction between *N*-sulfonylimines and aromatic carbaldehydes has not been disclosed.

2. Results and discussion



The *N*-sulfonylimine **1a** was chosen as the model substrate to react with the benzothiazole-2-carboxaldehyde **2a** to test the reaction condition (Table 1). The target product **3a** was not observed when using triazolium NHC pre-catalyst bearing electron-rich *N*-substituent (e.g., Table 1, entry 1, **A** [10]). To our delight, the triazolium NHC pre-catalysts **B** [11], **C** [12] and **D** [13] bearing electron-neutral or electron-deficient substituents could give the desired products in promising yields (entries 2 to 4). However, the yield of product **3a** dropped when using triazolium NHC pre-catalyst **E** [14] (entry 5). We then used catalyst **C** to evaluate the effect of base on our reaction (entries 6 to 8). The desired product **3a** could be afforded in 68% yield when using K_3PO_4 as the base (entry 8). Replacing THF with other solvents led to drops on the product yields (entries 9 to 11). Finally, the target product **3a** could be obtained in 83% yield when using an excess amount of

Table 1
Optimization of reaction conditions.[a]

Entry	NHC	Base	Solvent	Yield (%)[b]
1	**A**	Cs_2CO_3	THF	0
2	**B**	Cs_2CO_3	THF	60
3	**C**	Cs_2CO_3	THF	66
4	**D**	Cs_2CO_3	THF	65
5	**E**	Cs_2CO_3	THF	58
6	**C**	DBU	THF	32
7	**C**	Et_3N	THF	0
8	**C**	K_3PO_4	THF	68
9	**C**	K_3PO_4	EA	56
10	**C**	K_3PO_4	CH_2Cl_2	0
11	**C**	K_3PO_4	toluene	56
12[c]	**C**	K_3PO_4	THF	83

[a] General conditions (unless otherwise specified): **1a** (0.10 mmol), **2a** (0.10 mmol), NHC (0.02 mmol), base (0.02 mmol) and 4 Å MS (100 mg) in THF (2.0 mL) at 25 °C for 12 h.
[b] Isolated yield of **3a**.
[c] **1a** (0.10 mmol), **2a** (0.15 mmol), **C** (0.02 mmol), K_3PO_4 (0.02 mmol) and 4 Å MS (100 mg) in THF (2.0 mL) at 0 °C for 12 h.

benzothiazole-2-carboxaldehyde **2a** at 0 °C (entry 12).

Having got an optimal reaction condition at hand (Table 1, entry 12), we next examined the substrate scope of the *N*-sulfonylimines **1** (Scheme 1). Both electron-withdrawing and electron-donating groups could be installed on each position of the phenyl ring of the *N*-sulfonylimine **1a**, with the corresponding products afforded in good to excellent yields (Scheme 1, **3b** to **3m**). The phenyl group of the *N*-sulfonylimine **1a** could be switched to a heteroaromatic thiophenyl group, with the target product **3n** afforded in a good yield. Replacing the phenyl ring of the *N*-sulfonylimine **1a** with a *t*Bu group led to no formation of the desired product **3o**. The phenyl group of the *N*-sulfonylimine **1a** could also be replaced with a 1-napthyl or 2-napthyl group without much erosion on the product yields (**3p**, **3q**).

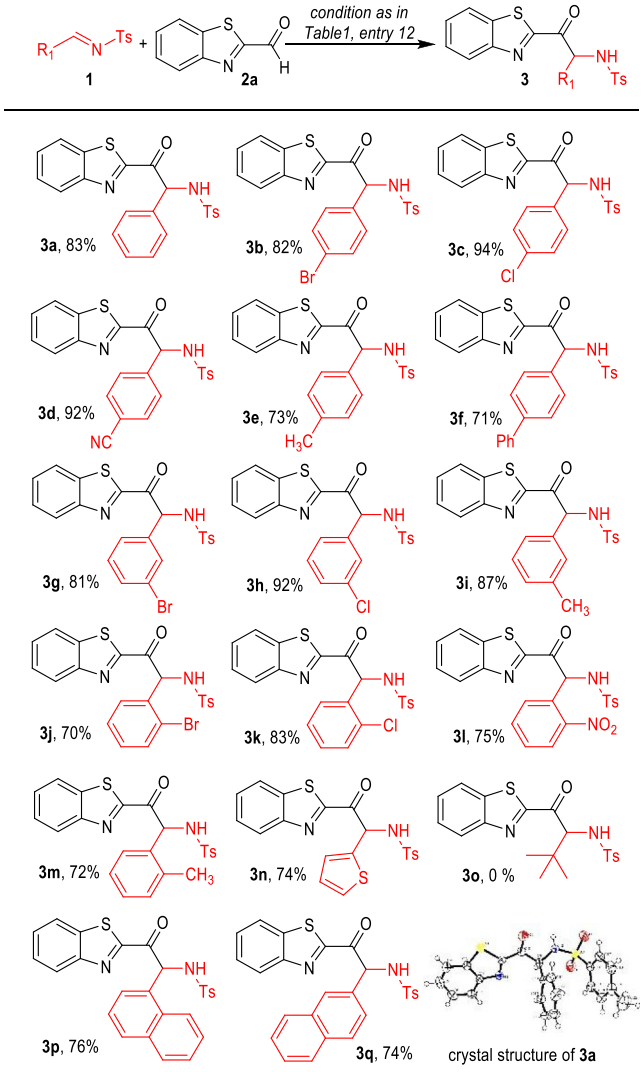
We then examined the scope of the benzothiazole-2-carboxaldehydes **2** (Scheme 2). Substituents with various electronic properties could be installed on the 4-position of the phenyl groups of the benzothiazole-2-carboxaldehyde **2a**, with the target products afforded in good to excellent yields (**3r** to **3u**). An electron-withdrawing Cl group was well tolerated on the 3-position of the phenyl ring of **2a**, and the desired product **3v** was given in a good yield. However, installing a 3-NO_2 group on the phenyl ring of **2a** led to no formation of the target product, with the aldehyde substrate remained unchanged. It is worth to note that no desired product was formed when the benzothiazole-2-carboxaldehyde was replaced by benzaldehydes, aliphatic carbaldehydes or cinnamaldehydes.

It is worth to note that our NHC-catalyzed *aza*-benzoin reaction can be carried out at gram scales (Fig. 2a). For example, the *aza*-benzoin reaction of **1a** and **2a** can be carried out at a 5.0-mmol scale and the desired product **3a** can be obtained in a 78% yield. The enantiomerically enriched α-aminoketone **3a** can be efficiently afforded under the catalysis of the chiral NHC **F**, although the er value was not satisfactory at this moment (Fig. 2b).

The carbonyl group of the α-aminoketone **3a** can react with a Grignard reagent and give the tertiary alcohol **4** in an excellent yield [15] (Fig. 3).

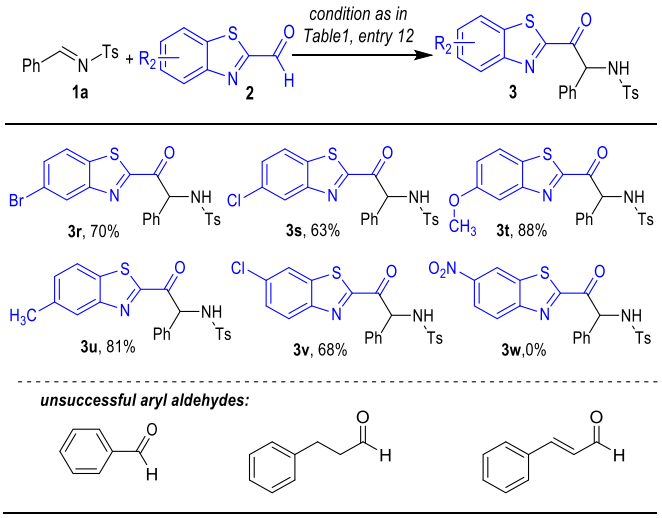
3. Conclusions

In summary, we have disclosed an NHC-catalyzed *aza*-benzoin reaction of the benzothiazole-2-carboxaldehydes and the *N*-sulfonylimines. α-Aminoketone products bearing different substituents and substitution patterns can be obtained in good to excellent



[a] Reaction conditions as stated in Table 1, entry **12**. Yields are isolated yields after purification via SiO_2 column chromatography.

Scheme 1. Scope of the *N*-sulfonylimines **1**.[a]



[a] Reaction conditions as stated in Table 1, entry **12**. Yields are isolated yields after purification via SiO_2 column chromatography.

Scheme 2. Scope of the benzothiazole-2-carboxaldehydes **2**.[a]

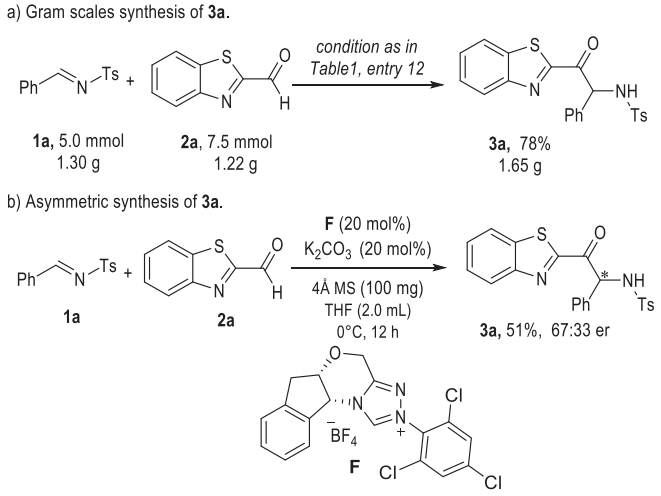


Fig. 2. Gram scale and asymmetric synthesis of **3a**.

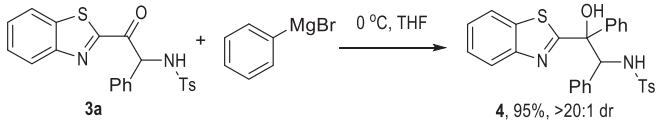


Fig. 3. Synthetic transformation of **3a**.

yields. Further studies on the bioactivities of the benzothiazole-containing α-aminoketones and their applications in agrichemical development are in progress in our laboratories.

4. Experimental section

4.1. General experimental information

Commercially available materials and dry solvents purchased from Energy Chemical and J&K were used as received. NMR spectra were measured on a Bruker ASCEND (AVANCE III HD 400 MHz) spectrometer. The chemical shift values were corrected to 2.50 ppm (^{1}H NMR) and 39.52 ppm (^{13}C NMR) for DMSO. ^{1}H NMR splitting patterns are designated as singlet (s), double (d), triplet (t), quartet (q), doublet of doublets (dd), multiplets (m), and etc. All first-order splitting patterns were assigned on the base of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). High resolution mass spectrometer analysis (HRMS) was performed on Thermo Fisher Q Exactive mass spectrometer. HPLC analyses were measured on Waters systems with Empower 3 system controller, Alliance 2695, and 2998 Diode Array Waters 2489 UV/V is detector. Chiralcel brand chiral columns from Daicel Chemical Industries were used with models OD-H in 4.6 × 250 mm size. The racemic products used to determine the er values were synthesized using racemic catalyst. Optical rotations were measured on an Insmark IP-digi Polarimeter in a 1 dm cuvette. The concentration (c) is given in g/100 mL. Melting Point (M.P.): Melting points were measured on an uncorrected Beijing Tech Instrument X-4 digital display micro melting point apparatus. Analytical thin-layer chromatography (TLC) was carried out on pre-coated silica gel plate (0.2 mm thickness). Visualization was performed using a UV lamp or $KMnO_4$

stain followed by heating.

4.2. General procedure for the preparation of substrates

The *N*-sulfonylimines [16] were prepared according to reported procedures and the benzothiazole-2-carboxaldehydes [17] were prepared according to reported procedures.

4.3. General procedure for the preparation of products (**3**)

To a 4 mL vial equipped with a magnetic stir bar was added NHC pre-catalyst **C** (0.02 mmol, 20 mol%, 7.3 mg), K_3PO_4 (0.02 mmol, 20 mol%, 4.2 mg), 4 Å MS (100.0 mg), substrates **1** (0.10 mmol) and substrates **2** (0.15 mmol), anhydrous THF (2.0 mL). The reaction mixture was allowed to stir for 12 h at 0 °C. After completion of the reaction, monitored by TLC, the mixture was concentrated under reduced pressure. The resulting crude residue was purified via column chromatography on silica gel (v/v/v: petroleum ether/CH_2Cl_2/EtOAc, from 10/2/1 to 10/2/2) to afford the desired product **3**.

4.3.1. N-(2-(benzo[d]thiazol-2-yl)-2-oxo-1-phenylethyl)-4-methylbenzenesulfonamide (**3a**)

White solid, 83% yield, 35 mg; m.p. 177–179 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.15 (d, J = 9.3 Hz, 1H), 8.31–8.22 (m, 2H), 7.71–7.59 (m, 4H), 7.40–7.37 (m, 2H), 7.30–7.21 (m, 3H), 7.17 (d, J = 8.1 Hz, 2H), 6.45 (d, J = 9.3 Hz, 1H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.4, 164.1, 153.1, 143.2, 138.3, 137.0, 134.9, 129.8, 129.3, 129.0, 128.9, 128.7, 128.2, 127.0, 126.0, 123.7, 61.1, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{17}N_2O_3S_2Na^+$: 445.0651, found: 445.0648.

4.3.2. N-(2-(benzo[d]thiazol-2-yl)-1-(4-bromophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3b**)

White solid, 82% yield, 42 mg; m.p. 174–175 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.15 (d, J = 9.4 Hz, 1H), 8.29–8.23 (m, 2H), 7.71–7.64 (m, 2H), 7.57–7.54 (m, 2H), 7.49–7.40 (m, 2H), 7.33–7.29 (m, 2H), 7.17–7.15 (m, 2H), 6.40 (d, J = 9.4 Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 189.7, 163.8, 153.1, 143.3, 138.2, 137.1, 134.3, 132.1, 130.8, 129.8, 129.1, 128.3, 127.1, 126.0, 123.8, 122.3, 60.7, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}BrN_2O_3S_2Na^+$: 522.9756, found: 522.9753.

4.3.3. N-(2-(benzo[d]thiazol-2-yl)-1-(4-chlorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3c**)

White solid, 94% yield, 43 mg; m.p. 182–183 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.21 (d, J = 9.4 Hz, 1H), 8.31–8.21 (m, 2H), 7.74–7.60 (m, 2H), 7.61 (d, J = 8.0 Hz, 2H), 7.43–7.40 (m, 2H), 7.34–7.32 (m, 2H), 7.18 (d, J = 8.0 Hz, 2H), 6.47 (d, J = 9.3 Hz, 1H), 2.27 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 189.8, 163.9, 153.1, 143.2, 138.2, 137.1, 133.9, 133.7, 130.5, 129.7, 129.2, 129.1, 128.2, 127.1, 126.0, 123.7, 60.6, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}ClN_2O_3S_2Na^+$: 479.0261, found: 479.0256.

4.3.4. N-(2-(benzo[d]thiazol-2-yl)-1-(4-cyanophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3d**)

White solid, 92% yield, 41 mg; m.p. 176–177 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.33 (d, J = 9.5 Hz, 1H), 8.32–8.22 (m, 2H), 7.77–7.65 (m, 4H), 7.61–7.58 (m, 4H), 7.16 (d, J = 8.1 Hz, 2H), 6.56 (d, J = 9.5 Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 189.3, 163.7, 153.0, 143.3, 140.3, 138.1, 137.2, 133.0, 129.8, 129.6, 129.2, 128.3, 127.1, 126.0, 123.8, 118.7, 111.6, 61.0, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{23}H_{17}N_3O_3S_2Na^+$: 470.0603, found: 470.0600.

4.3.5. N-(2-(benzo[d]thiazol-2-yl)-2-oxo-1-(p-tolyl)ethyl)-4-methylbenzenesulfonamide (**3e**)

White solid, 73% yield, 32 mg; m.p. 154–157 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.06 (d, J = 9.2 Hz, 1H), 8.29–8.27 (m, 1H), 8.24–8.21 (m, 1H), 7.70–7.62 (m, 2H), 7.60–7.58 (m, 2H), 7.24 (m, 2H), 7.18 (d, J = 8.1 Hz, 2H), 7.06 (d, J = 8.0 Hz, 2H), 6.38 (d, J = 9.2 Hz, 1H), 2.26 (s, 3H), 2.19 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.4, 164.2, 153.1, 143.1, 138.4, 137.0, 131.8, 129.8, 129.7, 129.0, 128.6, 128.2, 127.0, 125.9, 123.7, 61.0, 21.3, 21.0. HRMS (ESI, *m/z*) calcd. for $C_{23}H_{19}N_2O_3S_2Na^+$: 459.0808, found: 459.0810.

4.3.6. N-(1-([1,1′-biphenyl]-4-yl)-2-(benzo[d]thiazol-2-yl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3f**)

White solid, 71% yield, 35 mg; m.p. 195–196 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.19 (d, J = 9.3 Hz, 1H), 8.33–8.31 (m, 1H), 8.25–8.23 (m, 1H), 7.71–7.54 (m, 4H), 7.60–7.51 (m, 4H), 7.48–7.41 (m, 4H), 7.36–7.32 (m, 1H), 7.17 (d, J = 8.1 Hz, 2H), 6.52 (d, J = 9.3 Hz, 1H), 2.22 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.1, 164.1, 153.1, 143.1, 140.8, 139.8, 138.3, 137.1, 133.8, 129.7, 129.4, 129.3, 129.0, 128.2, 128.1, 127.6, 127.1, 127.1, 126.0, 123.8, 61.0, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{28}H_{21}N_2O_3S_2Na^+$: 521.0964, found: 521.0965.

4.3.7. N-(2-(benzo[d]thiazol-2-yl)-1-(3-bromophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3g**)

White solid, 81% yield, 41 mg; m.p. 169–170 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.17 (d, J = 9.5 Hz, 1H), 8.32–8.23 (m, 2H), 7.74–7.62 (m, 2H), 7.58–7.52 (m, 2H), 7.48 (m, 1H), 7.42–7.37 (m, 2H), 7.22–7.14 (m, 3H), 6.42 (d, J = 9.5 Hz, 1H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 189.6, 163.8, 153.1, 143.3, 138.1, 137.3, 137.2, 131.7, 131.3, 131.1, 129.7, 129.2, 128.3, 127.9, 127.0, 126.0, 123.8, 122.4, 60.6, 21.4. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}BrN_2O_3S_2Na^+$: 522.9756, found: 522.9750.

4.3.8. N-(2-(benzo[d]thiazol-2-yl)-1-(3-chlorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3h**)

White solid, 92% yield, 42 mg; m.p.151–153 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.19 (d, J = 9.6 Hz, 1H), 8.33–8.21 (m, 2H), 7.74–7.62 (m, 2H), 7.56 (m, 2H), 7.36 (m, 2H), 7.29–7.27 (m, 2H), 7.16 (d, J = 8.0 Hz, 2H), 6.45 (d, J = 9.5 Hz, 1H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 189.6, 163.8, 153.1, 143.3, 138.1, 137.2, 137.1, 133.8, 131.0, 129.7, 129.1, 128.8, 128.3, 127.5, 127.0, 126.0, 123.8, 60.7, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}ClN_2O_3S_2Na^+$: 479.0261, found: 479.0256.

4.3.9. N-(2-(benzo[d]thiazol-2-yl)-2-oxo-1-(m-tolyl)ethyl)-4-methylbenzenesulfonamide (**3i**)

White solid, 87% yield, 38 mg; m.p. 144–145 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.08 (d, J = 9.2 Hz, 1H), 8.33–8.26 (m, 1H), 8.26–8.19 (m, 1H), 7.72–7.58 (m, 4H), 7.18–7.13 (m, 5H), 7.03 (m, 1H), 6.41 (d, J = 9.2 Hz, 1H), 2.25 (s, 3H), 2.18 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.3, 164.2, 153.1, 143.1, 138.5, 138.3, 137.1, 134.7, 129.7, 129.5, 129.2, 129.1, 129.0, 128.2, 127.0, 126.0, 125.8, 123.7, 61.1, 21.3, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{23}H_{19}N_2O_3S_2Na^+$: 459.0808, found: 459.0815.

4.3.10. N-(2-(benzo[d]thiazol-2-yl)-1-(2-bromophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3j**)

White solid, 70% yield, 35 mg; m.p. 196–197 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.10 (d, J = 8.8 Hz, 1H), 8.23–8.21 (m, 1H), 8.09–8.06 (m, 1H), 7.68–7.58 (m, 5H), 7.44–7.42 (m, 1H), 7.38–7.34 (m, 1H), 7.25–7.21 (m, 1H), 7.18–7.16 (m, 2H), 6.78 (d, J = 8.7 Hz, 1H), 2.24 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 189.6, 163.7, 153.0, 143.0, 138.6, 137.0, 135.3, 133.5, 130.8, 130.3, 129.7, 128.9, 128.4, 128.1, 126.9, 125.9, 124.0, 123.7, 60.5, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}BrN_2O_3S_2Na^+$: 522.9756, found: 522.9753.

*4.3.11. N-(2-(benzo[d]thiazol-2-yl)-1-(2-chlorophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3k**)*

White solid, 83% yield, 37 mg; m.p. 193–194 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.12 (d, J = 8.8 Hz, 1H), 8.23–8.21 (m, 1H), 8.11–8.08 (m, 1H), 7.66–7.61 (m, 4H), 7.45–7.41 (m, 2H), 7.33–7.30 (m, 2H), 7.18 (d, J = 8.1 Hz, 2H), 6.82 (d, J = 8.8 Hz, 1H), 2.24 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 189.7, 163.7, 153.0, 143.0, 138.5, 137.0, 133.5, 133.3, 130.7, 130.2, 130.1, 129.7, 128.9, 128.1, 127.9, 126.9, 125.9, 123.7, 58.1, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}ClN_2O_3S_2Na^+$: 479.0261, found: 479.0257.

*4.3.12. N-(2-(benzo[d]thiazol-2-yl)-1-(2-nitrophenyl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3l**)*

White solid, 75% yield, 35 mg; m.p. 191–192 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.33 (d, J = 9.0 Hz, 1H), 8.22–8.20 (m, 1H), 8.05–8.00 (m, 1H), 7.95–7.93 (m, 1H), 7.81–7.74 (m, 2H), 7.69–7.56 (m, 5H), 7.13 (d, J = 8.0 Hz, 2H), 7.06 (d, J = 9.0 Hz, 1H), 2.21 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 187.8, 163.3, 152.8, 148.1, 143.0, 138.6, 136.9, 134.5, 131.5, 130.6, 130.2, 129.7, 128.9, 128.0, 126.8, 125.7, 125.5, 123.6, 57.5, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}N_3O_5S_2Na^+$: 490.0502, found: 490.0504.

*4.3.13. N-(2-(benzo[d]thiazol-2-yl)-2-oxo-1-(o-tolyl)ethyl)-4-methylbenzenesulfonamide (**3m**)*

White solid, 72% yield, 32 mg; m.p. 197–198 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.93 (d, J = 8.7 Hz, 1H), 8.24–8.14 (m, 2H), 7.66–7.60 (m, 4H), 7.23 (d, J = 8.1 Hz, 2H), 7.17–7.08 (m, 4H), 6.56 (d, J = 8.7 Hz, 1H), 2.45 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.7, 164.3, 153.0, 143.2, 138.4, 137.3, 136.8, 133.2, 131.4, 129.8, 129.1, 128.9, 128.2, 127.0, 126.8, 125.8, 123.7, 57.8, 21.4, 19.4. HRMS (ESI, *m/z*) calcd. for $C_{23}H_{19}N_2O_3S_2Na^+$: 459.0808, found: 459.0806.

*4.3.14. N-(2-(benzo[d]thiazol-2-yl)-2-oxo-1-(thiophen-3-yl)ethyl)-4-methylbenzenesulfonamide (**3n**)*

White solid, 74% yield, 32 mg; m.p. 170–172 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.11 (d, J = 9.5 Hz, 1H), 8.32–8.24 (m, 2H), 7.72–7.64 (m, 2H), 7.60 (m, 2H), 7.47–7.43 (m, 2H), 7.19 (d, J = 8.1 Hz, 2H), 7.06 (m, 1H), 6.54 (d, J = 9.5 Hz, 1H), 2.27 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 189.9, 164.0, 153.2, 143.2, 138.3, 137.1, 134.9, 129.8, 129.0, 128.2, 127.7, 127.4, 127.0, 126.0, 125.6, 123.8, 57.1, 21.4. HRMS (ESI, *m/z*) calcd. for $C_{20}H_{16}N_2O_3S_3Na^+$: 451.0215, found: 451.0215.

*4.3.15. N-(2-(benzo[d]thiazol-2-yl)-1-(naphthalen-1-yl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3p**)*

White solid, 76% yield, 36 mg; m.p. 189–190 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.09 (d, J = 8.9 Hz, 1H), 8.25–8.19 (m, 2H), 8.08–8.04 (m, 1H), 7.93–7.91 (m, 1H), 7.87 (d, J = 8.0 Hz, 1H), 7.63–7.54 (m, 6H), 7.43–7.35 (m, 2H), 7.19–7.14 (m, 3H), 2.27 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 190.6, 164.3, 153.0, 143.2, 138.1, 136.9, 134.1, 130.9, 130.8, 129.9, 129.7, 129.2, 128.9, 128.2, 127.3, 127.1, 126.9, 126.7, 125.8, 125.8, 123.7, 123.6, 57.3, 21.4. HRMS (ESI, *m/z*) calcd. for $C_{26}H_{19}N_2O_3S_2Na^+$: 495.0808, found: 495.0811.

*4.3.16. N-(2-(benzo[d]thiazol-2-yl)-1-(naphthalen-2-yl)-2-oxoethyl)-4-methylbenzenesulfonamide (**3q**)*

White solid, 74% yield, 35 mg; m.p. 191–192 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.23 (d, J = 9.1 Hz, 1H), 8.33–8.19 (m, 2H), 7.87–7.81 (m, 4H), 7.72–7.60 (m, 4H), 7.53–7.48 (m, 3H), 7.10 (d, J = 8.0 Hz, 2H), 6.62 (d, J = 9.1 Hz, 1H), 2.15 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.1, 164.1, 153.1, 143.1, 138.3, 137.1, 133.0, 132.9, 132.2, 129.7, 129.0, 129.0, 128.4, 128.2, 128.2, 127.9, 127.2, 127.0, 127.0, 126.0, 123.7, 61.4, 21.2. HRMS (ESI, *m/z*) calcd. for $C_{26}H_{19}N_2O_3S_2Na^+$: 495.0808, found: 495.0805.

*4.3.17. N-(2-(5-bromobenzo[d]thiazol-2-yl)-2-oxo-1-phenylethyl)-4-methylbenzenesulfonamide (**3r**)*

White solid, 70% yield, 35 mg; m.p. 193–194 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.15 (d, J = 9.3 Hz, 1H), 8.53 (d, J = 2.0 Hz, 1H), 8.20 (d, J = 8.7 Hz, 1H), 7.80 (m, 1H), 7.59–7.57 (m, 2H), 7.39–7.36 (m, 2H), 7.30–7.23 (m, 3H), 7.17 (d, J = 8.0 Hz, 2H), 6.40 (d, J = 9.2 Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.2, 165.8, 154.2, 143.2, 138.2, 136.2, 134.7, 131.7, 129.8, 129.3, 128.9, 128.7, 128.2, 127.0, 125.6, 121.0, 61.2, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}BrN_2O_3S_2Na^+$: 522.9756, found: 522.9754.

*4.3.18. N-(2-(5-chlorobenzo[d]thiazol-2-yl)-2-oxo-1-phenylethyl)-4-methylbenzenesulfonamide (**3s**)*

White solid, 63% yield, 29 mg; m.p. 191–192 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.16 (d, J = 9.3 Hz, 1H), 8.40 (d, J = 2.1 Hz, 1H), 8.26 (d, J = 8.7 Hz, 1H), 7.70 (m, 1H), 7.59–7.57 (m, 2H), 7.39–7.37 (m, 2H), 7.30–7.24 (m, 3H), 7.18 (d, J = 8.1 Hz, 2H), 6.41 (d, J = 9.3 Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.2, 166.1, 153.9, 143.2, 138.2, 135.8, 134.7, 133.0, 129.8, 129.3, 129.2, 128.9, 128.7, 127.0, 125.3, 125.2, 61.2, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}ClN_2O_3S_2Na^+$: 479.0261, found: 479.0259.

*4.3.19. N-(2-(5-methoxybenzo[d]thiazol-2-yl)-2-oxo-1-phenylethyl)-4-methylbenzenesulfonamide (**3t**)*

White solid, 88% yield, 35 mg; m.p. 151–152 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.1 (d, J = 9.4 Hz, 1H), 8.1 (d, J = 9.0 Hz, 1H), 7.8 (d, J = 2.5 Hz, 1H), 7.6–7.6 (m, 2H), 7.4–7.3 (m, 2H), 7.3–7.2 (m, 4H), 7.2 (d, J = 8.1 Hz, 2H), 6.4 (d, J = 9.4 Hz, 1H), 3.9 (s, 3H), 2.3 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 190.3, 164.9, 160.0, 154.7, 143.2, 138.3, 135.0, 129.8, 129.4, 129.3, 128.9, 128.6, 127.0, 124.0, 120.2, 107.0, 61.0, 56.2, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{23}H_{20}N_2O_4S_2Na^+$: 475.0757, found: 475.0756.

*4.3.20. 4-methyl-N-(2-(5-methylbenzo[d]thiazol-2-yl)-2-oxo-1-phenylethyl)benzenesulfonamide (**3u**)*

White solid, 81% yield, 35 mg; m.p. 171–172 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.13 (d, J = 9.3 Hz, 1H), 8.09–8.07 (m, 2H), 7.60 (d, J = 6.4 Hz, 2H), 7.47 (m, 1H), 7.39–7.37 (m, 2H), 7.29–7.23 (m, 3H), 7.17 (d, J = 8.1 Hz, 2H), 6.44 (d, J = 9.3 Hz, 1H), 2.49 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.3, 164.1, 153.5, 143.2, 138.3, 138.2, 134.9, 134.2, 130.8, 129.8, 129.2, 128.8, 128.6, 127.0, 125.5, 123.2, 61.1, 21.4, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{23}H_{19}N_2O_3S_2Na^+$: 459.0808, found: 459.0810.

*4.3.21. N-(2-(6-chlorobenzo[d]thiazol-2-yl)-2-oxo-1-phenylethyl)-4-methylbenzenesulfonamide (**3v**)*

White solid, 68% yield, 31 mg; m.p. 140–141 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.15 (d, J = 9.3 Hz, 1H), 8.40–8.39 (m, 1H), 8.29–8.27 (m, 1H), 7.72–7.69 (m, 1H), 7.59–7.57 (m, 2H), 7.39–7.36 (m, 2H), 7.30–7.24 (m, 3H), 7.17 (d, J = 8.0 Hz, 2H), 6.40 (d, J = 9.2 Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.3, 164.9, 151.8, 143.2, 138.4, 138.2, 134.7, 133.8, 129.8, 129.3, 128.9, 128.7, 127.2, 127.0, 123.4, 61.1, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{16}ClN_2O_3S_2Na^+$: 479.0261, found: 479.0260.

*4.4. General procedure for gram scale synthesis of **3a***

To a 50 mL flask equipped with a magnetic stir bar was added NHC pre-catalyst **C** (1.0 mmol, 365.0 mg), K_3PO_4 (1.0 mmol, 212.0 mg), 4 Å MS (1000.0 mg), substrate **1a** (5.0 mmol) and substrate **2a** (7.5 mmol). Freshly distilled anhydrous THF (20.0 mL) was added via syringe. The reaction mixture was allowed to stir for 12 h at 0 °C. After completion of the reaction, monitored by TLC, the mixture was concentrated under reduced pressure. The resulting crude residue was purified via column chromatography on silica gel

(v/v/v: petroleum ether/CH_2Cl_2/EtOA, from 10/2/1 to 10/2/2 petroleum ether/CH_2Cl_2/EtOAc) to afford the desired product **3a** (78% yield).

4.5. General procedure for asymmetric synthesis of *3a*

To a 4 mL vial equipped with a magnetic stir bar was added NHC pre-catalyst **F** (0.02 mmol, 20 mol %,7.3 mg), K_3PO_4 (0.02 mmol, 20 mol %, 4.2 mg), 4 Å MS (100.0 mg), substrate **1a** (0.10 mmol) and substrate **2a** (0.15 mmol), anhydrous THF (2.0 mL). The reaction mixture was allowed to stir for 12 h at 0 °C. After completion of the reaction, monitored by TLC, the mixture was concentrated under reduced pressure. The resulting crude residue was purified via column chromatography on silica gel (v/v/v: petroleum ether/CH_2Cl_2/EtOAc, from 10/2/1 to 10/2/2) to afford the desired product **3a**.

4.5.1. N-(2-(benzo[d]thiazol-2-yl)-2-oxo-1-phenylethyl)-4-methylbenzenesulfonamide (*3a*)

White solid, 51% yield, 22 mg; m.p. 177–179 °C, $[\alpha]^{25}D = 3.05$ (c = 1.0 in $CHCl_3$); ^1H NMR (400 MHz, DMSO-d_6) δ 9.15 (d, $J = 9.3$ Hz, 1H), 8.31–8.22 (m, 2H), 7.71–7.59 (m, 4H), 7.40–7.37 (m, 2H), 7.30–7.21 (m, 3H), 7.17 (d, $J = 8.1$ Hz, 2H), 6.45 (d, $J = 9.3$ Hz, 1H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 190.4, 164.1, 153.1, 143.2, 138.3, 137.0, 134.9, 129.8, 129.3, 129.0, 128.9, 128.7, 128.2, 127.0, 126.0, 123.7, 61.1, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{22}H_{17}N_2O_3S_2Na^+$: 445.0651, found: 445.0648. HPLC analysis: 67:33 er (OD-H column, 25 °C, hexane/*i*PrOH = 95/5, 0.5 mL/min, λ = 254 nm), Rt (major) = 83.2 min, Rt (minor) = 92.4 min.

4.6. General procedure for the preparation of product *4*

Product **3a** (0.15 g, 0.36 mmol) was added to a 100 mL round-bottom flask, and THF (20 mL) was then added. After cooling the solution to 0 °C, phenylmagnesium bromide (0.24 ml, 0.72 mmol) was added dropwise. The solution was allowed to warm to room temperature and stirred overnight. After completion of reaction as determined by TLC, the reaction was cooled to 0 °C and quenched with saturated NH_4Cl (35 mL). Upon separation of the two layers, the aqueous layer was extracted with CH_2Cl_2 (3 x 35 mL). The combined organic layers were then dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting crude oil was then purified by column chromatography using a 5%–15% EtOAc in petroleun ether, to afford alcohol **4** in 95% yield (0.17 g) as a white solid.

4.6.1. N-(2-(benzo[d]thiazol-2-yl)-2-hydroxy-1,2-diphenylethyl)-4-methylbenzenesulfonamide (*4*)

White solid, 95% yield, 169 mg; m.p. 194–195 °C; ^1H NMR (400 MHz, DMSO-d_6) δ 8.00–7.98 (m, 1H), 7.87–7.78 (m, 4H), 7.45–7.41 (m, 1H), 7.37–7.33 (m, 2H), 7.31–7.26 (m, 2H), 7.19–7.16 (m, 2H), 6.99–6.95 (m, 4H), 6.89–6.85 (m, 1H), 6.82–6.78 (m, 2H), 6.58 (s, 1H), 5.64 (d, $J = 10.1$ Hz, 1H), 2.22 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 177.7, 153.3, 142.7, 142.3, 138.7, 137.2, 134.8, 129.6, 129.3, 128.3, 127.8, 127.2, 126.9, 126.9, 126.6, 126.4, 125.2, 123.1, 122.4, 81.9, 64.6, 21.3. HRMS (ESI, *m/z*) calcd. for $C_{28}H_{23}N_2O_3S_2H^+$: 501.1301, found: 501.1297. Diastereoselective ratio were determined by ^1H NMR on the crude product mixture (dr > 20:1).

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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