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COMMUNICATION

Switchable synthesis of sulfinylated and sulfonylated indoles and benzofurans from *o*-aminophenyl/*o*-hydroxyphenyl propargyl alcohols and β -sulfinyl estersReceived 00th January 20xx,
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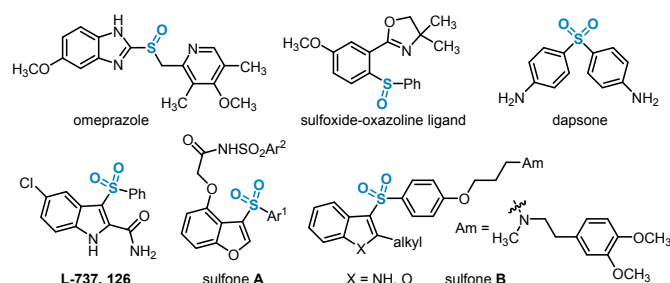
Ming Bian,^a Cheng-Mei Luo,^a Li-Na Gao,^a Tian-Yu Zhang,^a Yu-Ning Gao,^a Cun-Feng Song,^a Hui-Yu Chen,^{*a} and Zhen-Jiang Liu^{*a}

The cascade reaction of *o*-aminophenyl/*o*-hydroxyphenyl propargyl alcohols with β -sulfinyl esters was explored under basic conditions. *o*-Aminophenyl propargyl alcohols were converted to 3-sulfinyl indoles using KOH, while 3-sulfonyl indoles were obtained using Cs₂CO₃ and CuI. *o*-Hydroxyphenyl propargyl alcohols were only compatible with the sulfonylation process using Cs₂CO₃ and CuI, delivering 3-sulfonyl benzofurans.

Sulfones and sulfoxides are fundamental sulfur-containing building blocks¹ and ubiquitous motifs in chiral catalysts or ligands,² natural products,³ materials,⁴ agrichemicals,⁵ and bioactive molecules (e.g., omeprazole and dapsone).⁶ Indolyl and benzofuryl sulfones and sulfoxides are particularly valuable due to their bioactive cores, as exemplified by HIV-1 reverse transcriptase inhibitor L-737,126,⁷ EP3 antagonist benzofuryl sulfone **A**,⁸ and calcium antagonist sulfone **B** (Scheme 1).⁹ In general, they are prepared by direct oxidation of the corresponding thioether,¹⁰ Friedel–Crafts-type reaction of indole or benzofuran with sulfinyl or sulfonyl derivatives,¹¹ benzoheterocycle formation from sulfone/sulfoxide precursors,¹² or cascade strategies combining cyclization and sulfinyl/sulfonyl incorporation.¹³ Although some of these methods are highly effective and appealing, most of them still suffer from several drawbacks such as the usage of odorous thiols, stoichiometric oxidants, noble metal catalysts, starting materials with limited availability, or narrow substrate scope. Thus, developing efficient and novel routes to access these scaffolds from readily available substrates are still in demand.

o-Hydroxyphenyl/*o*-aminophenyl propargyl alcohols/amines serve as excellent precursors to *o*-alkynyl quinone methides (*o*-AQMs)/*aza-o*-AQMs, enabling efficient construction of

functionalized benzofurans and indoles. These compounds can react with nucleophilic sulfinyl or sulfonyl sources to afford 3-sulfinyl or 3-sulfonyl derivatives. While numerous methods for synthesizing 3-sulfonyl indoles/benzofurans have been reported based on *o*-hydroxyphenyl/*o*-aminophenyl propargyl alcohols/amines using various sulfonyl sources (TosMIC, ^{13c}, ^{13g} sodium sulfinate, ^{13a}, ^{13b}, ^{13f} arylsulfonyl hydrazides^{13e}) under Ag(I)-, Cu(I)-, or metal-free conditions, no nucleophilic sulfinyl reagent has been successfully employed in such cascade reactions to date.



Scheme 1 Representative indolyl and benzofuryl sulfoxides and sulfones.

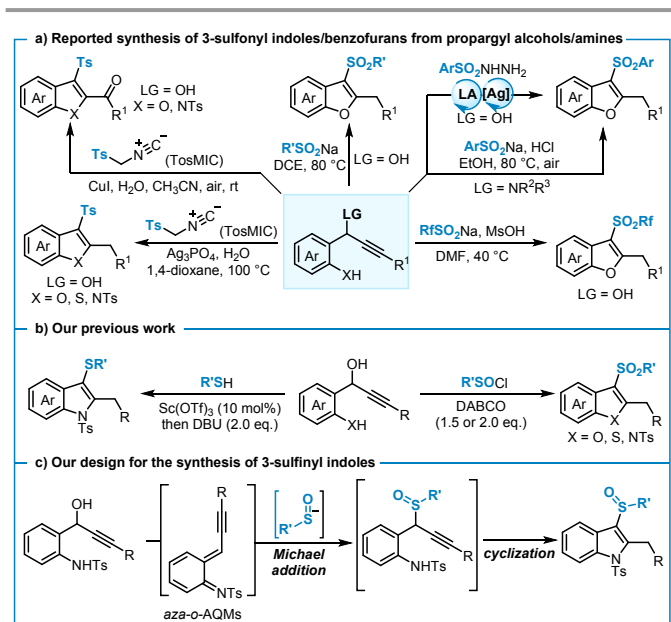
Based on our ongoing research in developing cascade reactions of propargyl alcohols to construct functionalized indoles and benzofurans,¹⁴ and inspired by our recent success in synthesizing 3-sulfinyl ^{14a} and 3-sulfonyl ^{14b} benzoheterocycles, we sought to expand this strategy to access 3-sulfinyl benzoheterocycles. β -Sulfinyl esters emerged as promising candidates, having been widely explored as a masked sulfinyl nucleophile pioneered by Perrio and co-workers.¹⁵ The highly reactive sulfenate anion (RSO⁻) generated *in situ* from β -sulfinyl esters via base-induced *retro*-Michael fragmentation, have demonstrated versatility in S–C¹⁶ or S–N¹⁷ bond formation reactions. Moreover, β -sulfinyl esters are also efficient precursors of sulfinyl¹⁸ and sulfonyl¹⁹ species. We hypothesized that *in situ* generated sulfenate anions could similarly react with

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COMMUNICATION

o-hydroxyphenyl/*o*-aminophenyl propargyl alcohols to afford 3-sulfinyl benzofurans/indoles.



Scheme 2 Methods for synthesis of 3-sulfonyl/sulfinyl benzofurans and indoles.

Delightfully, initial screening using *o*-aminophenyl propargyl alcohol **1a** and β -sulfinyl ester **2a** with KOH/4Å MS (molecular sieves) in THF at room temperature afforded the desired sulfoxide **3a** (31%) along with sulfone **4a** (8%) (entry 1, Table 1). Solvent evaluation identified toluene as optimal, improving the yield of **3a** to 42% (entries 2-4). The molecular sieves proved crucial, as their omission dramatically reduced yields (entry 5). Elevated temperature (50 °C) significantly enhanced the yield of **3a** to 85% (entry 6), while reducing reagent stoichiometry proved detrimental (entries 7-8). Further temperature increases favored **4a** formation (entries 9-11). Base screening revealed that alternative bases (Cs_2CO_3 , Et_3N) preferentially yielded **4a** (entries 12-13), prompting separate optimization for sulfone production. At 80 °C, exclusive **4a** formation was achieved in 65% yield (entry 14). Introducing CuI (5 mol%)^{19b} instead of molecular sieves further improved the yield of **4a** to 84% (entry 15), allowing reagent reduction (2 equiv. of **2a**, 4 equiv. of base, Table 1, entry 16). Other Cu(I) salts also performed well (Table 1, entries 17-19). Other sulfinyl anion precursors were evaluated but proved less efficient, affording the target products in diminished yields. (See Supporting Information)

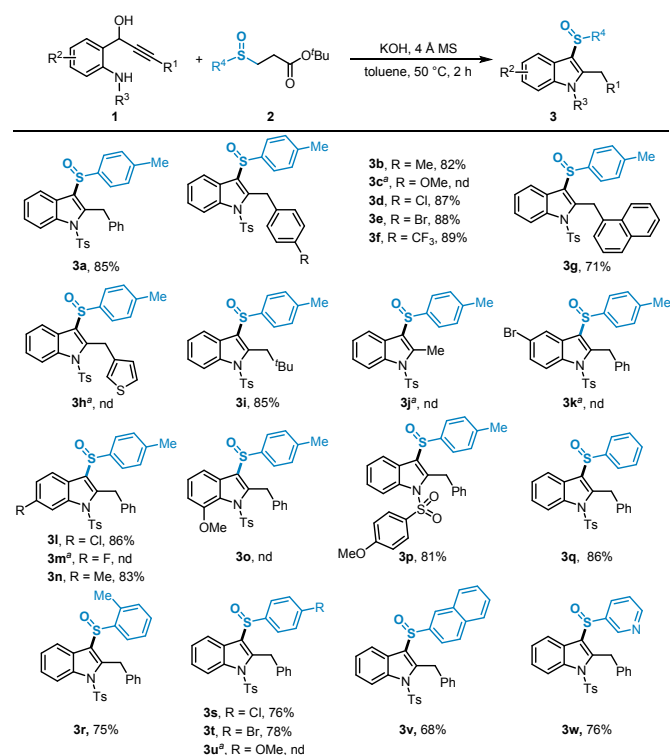
Table 1 Optimization of reaction conditions.^a

entry	base	additives	solvent	temp (°C)	t (h)	yield (%) ^b	
						3a	4a
1	KOH	4 Å MS (20 mg)	THF	rt	5	31	8
2	KOH	4 Å MS (20 mg)	CH_3CN	rt	5	16	7
3	KOH	4 Å MS (20 mg)	DMF	rt	5	nd	nd
4	KOH	4 Å MS (20 mg)	toluene	rt	5	42	9

5	KOH	/	toluene	rt	5	trace	10
6	KOH	4 Å MS (20 mg)	toluene	50	2	85	13
7 ^c	KOH	4 Å MS (20 mg)	toluene	50	2	46	10
8 ^d	KOH	4 Å MS (20 mg)	toluene	50	2	69	18
9	KOH	4 Å MS (20 mg)	toluene	60	2	56	19
10	KOH	4 Å MS (20 mg)	toluene	80	2	45	33
11	KOH	4 Å MS (20 mg)	toluene	100	2	15	35
12	Cs_2CO_3	4 Å MS (20 mg)	toluene	50	2	27	42
13	Et_3N	4 Å MS (20 mg)	toluene	50	2	19	33
14	Cs_2CO_3	4 Å MS (20 mg)	toluene	80	2	nd	65
15	Cs_2CO_3	CuI (5 mol%)	toluene	80	2	nd	84
16 ^{cde}	Cs_2CO_3	CuI (5 mol%)	toluene	80	2	nd	86
17 ^{cde}	Cs_2CO_3	CuBr (5 mol%)	toluene	80	2	nd	80
18 ^{cde}	Cs_2CO_3	CuCl (5 mol%)	toluene	80	2	nd	82
19 ^{cde}	Cs_2CO_3	$\text{Cu}(\text{OTf})_2$ (5 mol%)	toluene	80	2	nd	78

^a Reaction conditions: **1a** (0.15 mmol), 4 Å MS, and base (0.9 mmol) were stirred in solvent (1 mL) at room temperature for 15 min under Ar or N_2 , then **2a** (0.45 mmol in 1 mL of solvent) was added dropwise, then warm to indicated temperature and stirred. ^b isolated yield; ^c **2a** (0.3 mmol) was used; ^d base (0.6 mmol) was used; ^e **1a**, **2a**, base, and Cu(I) salt in solvent (1 mL) were stirred under N_2 . nd: not detected.

With these optimized conditions established, we achieved a switchable synthesis of both 3-sulfinyl and 3-sulfonyl indoles from propargyl alcohol **1a** and β -sulfinyl ester **2a**. The optimal conditions (entries 6 and 16, Table 1) were subsequently employed to explore the substrate scope.

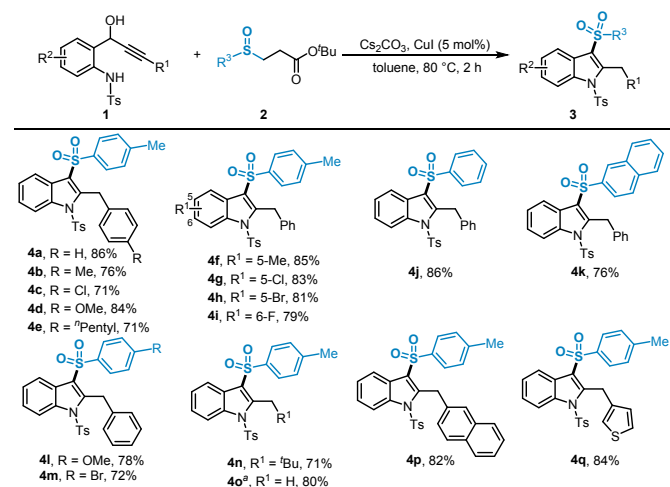


Scheme 3 Synthesis of 3-sulfinyl indoles. Reaction conditions: **1** (0.15 mmol), KOH (0.9 mmol), 4 Å MS (20 mg), **2** (0.45 mmol), toluene (1 mL + 1 mL), 50 °C; yields of the isolated products are given, nd = not detected. ^aSulfone was isolated, yield for **3c**: **4d** (74%), for **3h**: **4g** (69%), for **3j**: **4o** (%), for **3k**: **4h** (67%), for **3m**: **4i** (68%), for **3u**: **4l** (74%).

We first explored the substrate scope for 3-sulfinyl indole synthesis using various *o*-aminophenyl propargyl alcohols and β -sulfinyl esters (Scheme 3). The reaction tolerated diverse substituents on the alkyne terminal, including electron-donating (Me) and -withdrawing (Cl, Br, CF_3) groups on phenyl rings, 2-naphthyl, as well as *tert*-butyl, yielding products **3b**, **3d-3g** and **3i** efficiently. Substituted aniline rings (Cl, Me) also

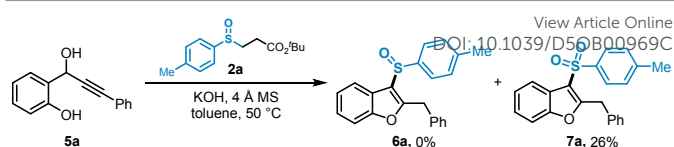
reacted smoothly, affording **3l** (86%) and **3n** (83%). Whereas for substrates bearing 4-OMe substituted phenyl (**3c**), thienyl (**3h**) and trimethylsilyl (**3j**) groups at R¹, 5-Br (**3k**), and 6-F (**3m**) on the aniline ring, only the corresponding sulfones were isolated. For trimethylsilyl-substrate, the trimethylsilyl group was cleaved in situ. 7-OMe (**3o**) gave a complex mixture under the standard conditions. The *N-p*-methoxyphenyl sulfonyl derivative gave **3p** in 81% yield. Various β -sulfinyl esters performed well under the standard conditions, including phenyl, *o*-tolyl, *p*-chlorophenyl, *p*-bromophenyl, and 2-naphthyl variants (**3q-3t**, **3v**, 68-86%). Notably, even 3-pyridyl sulfinyl ester was compatible, delivering **3s** in 76% yield. *p*-Methoxyphenyl β -sulfinyl ester (**3u**) only gave the sulfone product. The observed selectivity difference may be attributed to the varying reactivity of the in situ-generated *aza-o*-QMs toward the weakly nucleophilic sulfinyl anion.

Next, representative *o*-aminophenyl propargyl alcohols and β -sulfinyl esters were tested for the synthesis of 3-sulfonyl indoles, and they all afforded the desired sulfonyl indoles in good yields (Scheme 4). Generally, phenyls, alkyl, 2-naphthyl, and 2-thienyl at R¹ (**4a-4e**, **4n**, **4p-4q**) were well tolerated, while trimethylsilyl group at R¹ (**4o**) cleaved in situ. Substrates bearing methyl and halogen on the aniline ring gave the corresponding sulfones in 79-85% yields (**4f-4i**). Various β -sulfinyl esters underwent the desired reaction smoothly and provided the sulfones **4j-4m** in 72-86% yields.



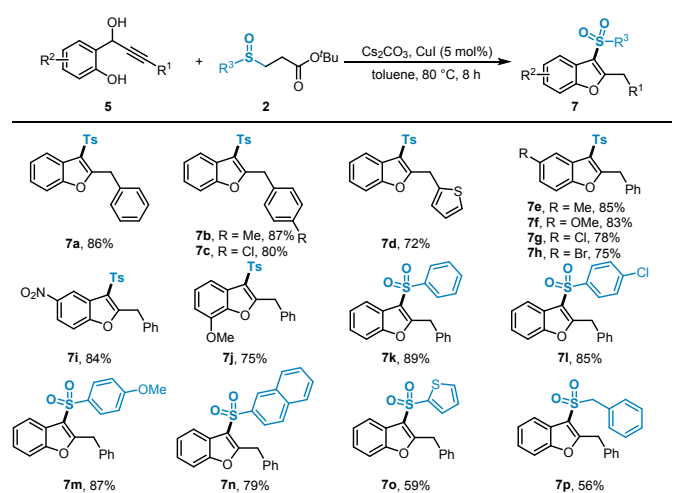
Scheme 4 Synthesis of 3-sulfonyl indoles. Reaction conditions: **1** (0.3 mmol), **2** (0.6 mmol), Cs₂CO₃ (1.2 mmol), CuI (0.015 mmol), toluene (2 mL), 80 °C; yields of the isolated products are given. ^oR¹ = TMS in **1**, and the TMS group was cleaved in situ.

We next examined the reactivity of *o*-hydroxyphenyl propargyl alcohol **5a** under the optimized-indole conditions. Surprisingly, only sulfone **7a** was obtained (26% yield) under the standard sulfonylation conditions (Scheme 5), with no detectable formation of sulfoxide **6a** despite extensive optimization (Table S1). In contrast, **5a** proved highly compatible with sulfonylation conditions, delivering the corresponding product in 86% yield (Scheme 6).



Scheme 5 Attempt to synthesize of 3-sulfonyl benzofuran.

The substrate scope for 3-sulfonyl benzofuran synthesis was extensively explored (Scheme 6). Both electron-donating (Me) and -withdrawing (Cl) groups on the phenyl ring (R¹) were well tolerated, affording **7b** and **7c** in >80% yields. Thienyl-substituted propargyl alcohol also reacted efficiently to give **7d** (72%). Various phenol ring substituents - including methyl (**7e**), methoxy (**7f**, **7j**), chloro (**7g**), bromo (**7h**), and nitro (**7i**) groups - all participated successfully, yielding the corresponding products in 75-85% yields. The reaction showed broad compatibility with different β -sulfinyl esters, with phenyl, naphthyl, and thienyl variants producing **7k-7o** in 59-89% yields, while the benzyl derivative gave **7p** in 56% yield.



Scheme 6 Synthesis of 3-sulfonyl benzofurans. Reaction conditions: **5** (0.3 mmol), **2** (0.6 mmol), Cs₂CO₃ (1.2 mmol), CuI (0.015 mmol), toluene (2 mL), 80 °C; yields of the isolated products are given.

To elucidate the mechanistic details, we conducted a series of control experiments. These studies underscored the critical role of sulfinyl anion stability in these transformations. In our reaction system, complete conversion of β -sulfinyl ester **2a** to diaryl disulfide **8** was achieved within approximately 4 hours. This transformation proceeded to completion with comparable efficiency when using either strong (KOH) or relatively mild base (Cs₂CO₃) (**Fig. 1**). Notably, the process was significantly accelerated in the presence of catalytic CuI (reaction 2 vs reactions 1 and 3), a finding consistent with the results reported by Zeng,¹⁸ Yang,^{19a} and Zhao^{19b}. These results suggest that *aza-o*-QMs might exhibit markedly higher reactivity toward the weakly nucleophilic sulfinyl anion prior to its disproportionation, compared to conventional *o*-QMs, rationalizing the distinct reactivity patterns observed between *o*-aminophenyl and *o*-hydroxyphenyl propargyl alcohols.

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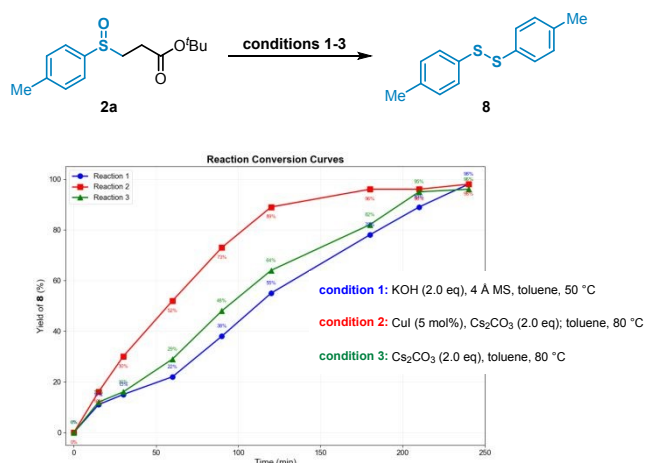
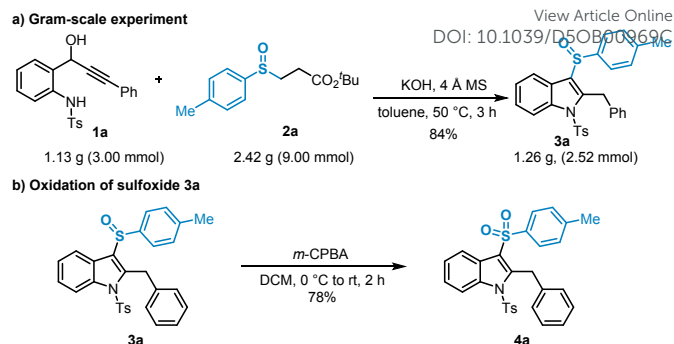
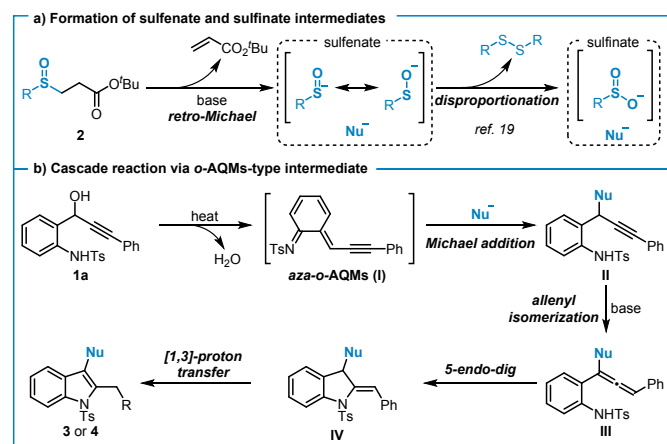


Fig. 1 Control experiments.

Based on the experimental evidence and literature precedents,¹⁹ we proposed a plausible mechanism shown in Scheme 7. The reaction might initiate through base-mediated *retro*-Michael fragmentation of β -sulfinyl ester to generate a sulfenate anion (sulfinyl anion), which may undergo disproportionation to form disulfide and sulfinate species (Scheme 7a).¹⁹ Concurrently, dehydration of *o*-aminophenyl propargyl alcohol **1a** might produce an *aza-o*-AQM (I). This electrophilic intermediate may undergo conjugate addition with either sulfenate or sulfinate nucleophiles, yielding adduct II. Subsequent base-promoted alkyne-allene isomerization, followed by 5-*endo*-dig cyclization and 1,3-proton transfer, ultimately affords the 3-sulfinyl (**3a**) or 3-sulfonyl (**4a**) indole products.^{13f}



Scheme 7 Proposed reaction mechanisms.

Further utility of this methodology was demonstrated by gram-scale synthesis of **3a**, which retained a very good yield under the standard conditions (Scheme 8a). Additionally, oxidation of **3a** by *m*-chloroperbenzoic acid (*m*-CPBA) provided **4a** in 78% yield (Scheme 8b).

Scheme 8 Scaled-up preparation of **3a** and oxidation of **3a**.

In conclusion, we have established a switchable synthetic protocol for the divergent preparation of 3-sulfinyl and 3-sulfonyl indoles from *o*-aminophenyl propargyl alcohols and β -sulfinyl esters. Intriguingly, *o*-hydroxyphenyl propargyl alcohols displayed fundamentally different reactivity patterns, failing to produce 3-sulfinyl benzofurans even under optimized conditions and only participating in sulfonylation reactions. The reaction likely proceeds through Michael addition of sulfenate or sulfinate nucleophiles to *o*-AQMs type intermediate generated *in situ*, followed by alkyne-allene isomerization, cyclization and 1,3-proton transfer sequence.

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Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the ESI.†

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Data availability statements

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The data supporting this article have been included as part of the Supplementary Information.