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Ts₂O mediated deoxygenative C2dithiocarbamation of guinoline N-oxides with CS₂ and amines†

Long-Yong Xie, * Chu Liu, * Si-Yu Wang, * Zhong-Ying Tian and Sha Peng*

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A general, efficient and practical protocol for Ts₂O promoted deoxygenative dithiocarbamation of quinoline N-oxides with in situ generated dithiocarbamic acids from CS₂ and amines is reported. The reaction proceeded well under transition-metal free conditions to obtain a variety of novel quinolinedithiocarbamate compounds with wide functional group tolerance and good to high yields.

Introduction

Dithiocarbamates are a significant class of sulfur-containing organic compounds with diverse biological properties, including anticancer, antibacterial, antioxidation and insecticidal activities.1 Apart from their essential role in cancer treatment,2 rubber industry,3 agricultural chemistry4 and polymer chemistry,⁵ they also serve as versatile intermediates in organic synthesis.6 Among the various dithiocarbamates, aryl dithiocarbamates have obtained extensive attention due to their intriguing biological behaviors and synthetic applications. Traditionally, aryl dithiocarbamates are primarily synthesized through reactions involving isothiocyanates with thiophenols⁷ or dithiochloroformates with amines.8 However, these methods are associated with complex and laborious procedures, the use of hazardous and toxic reagents, and relatively low yields.

In the past few decades, the cross-coupling reaction has emerged as a prominent method for synthesizing aryl dithiocarbamates.9 Especially, the three-component cross-coupling reactions based on aryne, 10 aryl halides, 11 aryl diazonium fluoroborates,12 dibenzothiophenium salts13 or diaryliodonium triflates14 with CS2 and amines to access aryl dithiocarbamates have made great achievements. However, these protocols require the pre-functionalization of the aromatic substrates, and some reactions cannot avoid the use of transition metals and elevated temperature. Most importantly, these developed methods mainly focus on the synthesis of various phenyldithiocarbamates. Very limited examples are reported on the synthesis of heterocyclic dithiocarbamates via direct C-H functionalization of heterocycles despite that these heterocyclic

indole-dithiocarbamates, 16 triazole-dithiocarbamates, 17 indolizine-dithiocarbamates,18 benzimidazole-dithiocarbamates,19 aminosugar-dithiocarbamates20 and quinazolinone-dithiocarbamates,21 exhibit exceptional bioactivities. In 2006, Tang et al.22 firstly disclosed a C-H thiolation strategy for the incorporation of dithiocarbamates into imidazoheterocycles using I₂ and FeF3 as co-catalysts (Scheme 1a). Subsequently, In 2018, Halimehjani and co-workers23 developed a practical and molecular iodine mediated C-H sulfenylation method to introduce dithiocarbamate groups into indole units via the three-component reaction involving indoles, carbon disulfide and amines (Scheme 1b). Most recently, Nagula Shankaraiah et al.²⁴ reported a one-pot, microwave-assisted, copper-catalyzed dithiocarbamation of imidazo[1,2-a]pyridines with carbon disulfide and amines (Scheme 1c). To our knowledge, the corporation of dithiocarbamate groups into quinoline skeletons via C-H functionalization strategy has not been previously

dithiocarbamates, such as chromone-dithiocarbamates, 15

Scheme 1 Representative examples on C-H dithiocarbamation of heterocycles.

Ts₂O (1.5 equiv)

College of Chemistry and Bioengineering, Hunan University of Science and Engineering, 425100. longyongxie@yeah.net; Yongzhou, China. E-mail: ps807055147@126.com

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[‡] These authors contributed equally.

documented. Given the distinct biological properties of quinolines and dithiocarbamates along with their widespread application in pharmaceutical and pesticide industry,²⁵ the development of efficient approaches to simultaneously incorporate quinoline units and the dithiocarbamate groups to a single molecular structure is highly desirable and remains persistent challenges.

In the past few years, great achievements have been made on the C–H functionalization of quinoline *N*-oxides with various nucleophiles for the synthesis of 2-substituted quinolines.²⁶ These nucleophiles mainly include alcohols,²⁷ amines,²⁸ thiols,²⁹ phenols,³⁰ 1,3-dicarbonyl compounds,³¹ RSO₂Na³² and AgSCF₃.^{16b,33} However, the utilization of dithiocarbamic acids derived from the reaction of CS₂ and amines as nucleophiles to construct quinoline-dithiocarbamates has never been reported yet. With our continuing research interest in C–H functionalizations of quinoline *N*-oxides,³⁴ we herein report a convenient and practical protocol for the synthesis of quinoline-dithiocarbamates *via* Ts₂O promoted deoxygenative C2–H dithiocarbamation of quinoline *N*-oxides under mild and transition-metal free conditions (Scheme 1d).

Results and discussion

Initially, quinoline *N*-oxide (1a), carbon disulfide and diethylamine (2a) were selected as model substrates to evaluate this three-component reaction, as shown in Table 1. Performing the

Table 1 Optimization of the reaction conditions^a

Entry	Activating agent	Solvent	Yield ^b (%)
1	TsCl	DCM	11
2	MsCl	DCM	16
3	Ts ₂ O	DCM	84
4	Ms_2O	DCM	0
5	Tf_2O	DCM	57
6	BzCl	DCM	53
7	Ac_2O	DCM	0
8	PyBroP	DCM	0
9	Ts_2O	CH_3CN	34
10	Ts_2O	THF	26
11	Ts_2O	DCE	82
12	Ts_2O	Acetone	62
13	Ts_2O	DMF	78
14	Ts_2O	EtOAc	31
15	Ts_2O	DMSO	0
16	Ts_2O	H_2O	43
17	None	DCM	0

^a Conditions: 1a (0.1 mmol, 1 equiv.), 2a (0.15 mmol, 1.5 equiv.), activating agent (0.15 mmol, 1.5 equiv.), solvent (1 mL), r.t., 0.5 h, air atmosphere. ^b Yield of 3aa was determined by ¹H NMR. PyBroP: bromotripyrrolidinophosphonium hexafluorophosphate.

model reaction at room temperature for 0.5 h using p-toluenesulfonyl chloride as an electrophilic activating reagent and DCM as a solvent, 11% NMR yield of 3aa was detected (entry 1). Subsequently, some other electrophilic activating reagents including methyl sulfonyl chloride (entry 2), p-toluene sulfonic anhydride (entry 3), methyl sulfonic anhydride (entry 4), trifluoromethanesulfonic anhydride (entry 5), benzoyl chloride (entry 6), acetic anhydride (entry 7) and PyBrop (entry 8) were examined and the results revealed that p-toluenesulfonic anhydride gave the best 84% yield of 3aa. However, other investigated activating agents could only afford the target product in moderate to low yields, among which acetic anhydride, mesulfonic anhydride and PyBroP did not give the desired product likely due to their lower electrophilic activities compared to Ts₂O. Furthermore, the impact of different solvents on the reaction was explored, including CH3CN (entry 9), THF (entry 10), DCE (entry 11), acetone (entry 12), DMF (entry 13), EtOAc (entry 14), DMSO (entry 15) and water (entry 16). In contrast to DCM, other solvents failed to give better vields of 3aa and solvent DMSO was found not suitable for the present transformation. Besides, the absence of any activating reagent led to no detection of product 3aa, highlighting the crucial role of the activating agent in this process (entry 17).

After obtaining the optimized reaction conditions (Table 1, entry 3), the substrate scope and limitations of the present reaction were examined with respect to both quinoline N-oxides and amines, as shown in Table 2. Various quinoline N-oxides substituted with electron-donating, electron-withdrawing or sterically hindered groups at different positions on the quinoline rings, all reacted well to afford the expected products in moderate to good yields (3aa-3ra). Notably, functional groups such as methyl (3ba, 3fa, 3ga, 3ma and 3oa), methoxy (3ha), aryl (3ea and 3pa), fluoride (3ia), chloride (3ca and 3ja), bromide (3da and 3ka), trifluoromethyl (3na) and ester (3la) groups were found to be compatible with the reaction. Furthermore, disubstituted quinoline N-oxides also served as suitable substrates, providing products 3qa and 3ra in 72 and 64% respectively. Some other nitrogen-containing compounds were then investigated, of which 1H-pyrrolo[2,3-b] pyridine 7-oxide (1s) gave the corresponding product 3sa in 78% yield. In contrast to quinoline N-oxides, some substituted pyridine N-oxides as potential reaction substrates can also give acceptable results (3ta-3va), while isoquinoline N-oxide (1w), quinoxaline 1-oxide (1x), 4-nitroquinoline N-oxide (1y), 3-cyanoquinoline N-oxide (1z), pyridine N-oxide, and 4-cyanopyridine N-oxide failed to give the corresponding products (3wa-3za, 4aa and 4ba), with most of the starting substrates being recovered in the case of pyridine and 4-cyanopyridine N-oxides.

The scope of the three-component coupling reaction between quinoline *N*-oxide (1a), CS₂ and different amines was then investigated, as revealed in Table 3. Chain dialkylamines reacted well under standard conditions, yielding the corresponding products in 62–83% yields (3ab–3af). Furthermore, some cyclic amines including pyrrolidine (2g), piperidines (2h and 2i), morpholine (2j) and azocane (2k) all reacted readily and gave the desired products (3ag–3ak) in good to excellent yields. However, several primary amines such as butan-1-amine (2l), 2-

Table 2 Reaction scope^a

^a Reaction conditions: 1 (0.3 mmol), 2a (0.45 mmol), CS₂ (0.45 mmol), Ts₂O (0.45 mmol), DCM (3 mL), r.t., air, 0.5 h, isolated yields based on 1.

aminoethanol (**2m**), 4-aminobutanoic acid (**2n**), diisopropylamine (**2o**), aniline (**2p**) and *N*-methylaniline (**2q**) failed to deliver the expected products (**3al–3aq**). Notably, *N*-butylquinolin-2-amine was obtained in a 74% isolated yield under standard conditions *via* the direct deoxygenative C2–H amination reaction of quinoline *N*-oxide (**1a**) with butan-1-amine (**2l**).

To better demonstrate the practicability of the three-component reaction, a gram-scale experiment involving 1a (5 mmol, 0.7253 g), CS_2 and diethylamine (2a) was conducted under standard conditions. As expected, the desired product 3aa was obtained in 79% isolated yield (Scheme 2a). To further show the synthetic utility of the present method, cloquintocetmexyl, an effective herbicide 4c', was oxidized by m-CPBA to afford substrate 4c, which reacted smoothly with CS_2 and diethylamine under standard conditions and gave product 4ca in 74% isolated yield (Scheme 2b).

To gain further insight into the three-component reaction mechanism, a series of control experiments were conducted, as depicted in Scheme 3. Initially, quinoline 1a', carbon disulfide and diethylamine were combined under standard conditions, yet no formation of 3aa was observed (Scheme 3a). This result shows that quinoline *N*-oxide is crucial for this reaction and the

Table 3 Reaction scope^a

^a Reaction conditions: **1a** (0.3 mmol), **2** (0.45 mmol), CS₂ (0.45 mmol), Ts₂O (0.45 mmol), DCM (3 mL), r.t., air, 0.5 h, isolated yields based on **1a**.

Scheme 2 Gram-scale experiment and late stage functionalization.

possibility of quinoline as an intermediate during the transformation could be ruled out. Furthermore, two common free radical inhibitors, TEMPO and BHT were added to the reaction system under standard conditions, the yields of 3aa were not significantly affected, suggesting that free radicals might not be involved in the reaction (Scheme 3b). In addition, a reaction between 1a and 4g resulted in the formation of product 3ag in 85% yield, indicating that the *in situ* generation of dithiocarbamic acid from the reaction of CS₂ and amine could potentially serve as reaction intermediate (Scheme 3c). However, when compound 4b was utilized instead of CS₂ and amine, and its reaction with 1a under standard conditions, no product 3ab was detected (Scheme 3d), effectively ruling out tetraalkylthiuram disulfide as a reaction intermediate.

Based on the control experiment stated above and relevant literature reports, 26a,29,32c a possible reaction mechanism was illustrated in Scheme 4. Firstly, quinoline *N*-oxide **1a** was activated by *p*-toluenesulfonic anhydride to produce the activated intermediate **IM-1**, which is further regioselectively nucleophilic

Scheme 3 Control experiments.

Scheme 4 Possible mechanism.

attacked by dithiocarbamic acid 2' generated *in situ* from carbon disulfide and amine 2 to form another intermediate **IM-2**. Subsequently, the intermediate **IM-2** underwent rearomatization to yield the target product 3, along with the release of 4-methylbenzenesulfonate anion in the current basic system.

Conclusions

In summary, we have established the first example of Ts₂O promoted three-component reaction involving quinoline *N*-oxides, CS₂ and amines to introduce dithiocarbamates onto the quinoline skeleton at ambient temperature. The *in situ* generated dithiocarbamates from CS₂ and amines act as nucleophiles, attracting the C2 position of quinoline *N*-oxides towards diverse quinoline-dithiocarbamates in satisfactory yields with broad functional group tolerance. The present method may provide a powerful tool for the screening of potential bioactive quinoline molecules bearing dithiocarbamate frameworks.

Conflicts of interest

There are no conflicts to declare.

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