Organic LETTERS

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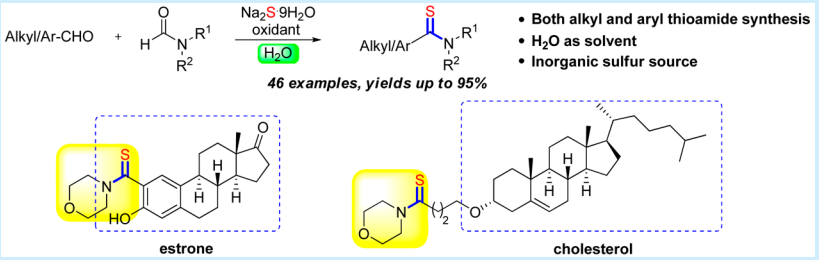
Aqueous Compatible Protocol to Both Alkyl and Aryl Thioamide Synthesis

Jianpeng Wei,[†] Yiming Li,[†] and Xuefeng Jiang*[,†,‡]

[†]Shanghai Key Laboratory of Green Chemistry and Chemical Process, Department of Chemistry, East China Normal University, Shanghai 200062, P. R. China

[‡]Beijing National Laboratory for Molecular Sciences (BNLMS), Beijing 100190, P. R. China

Supporting Information



ABSTRACT: An efficient aqueous synthesis of thioamides through aldehydes, sodium sulfide, and *N*-substituted formamides has been developed. Both alkyl and aryl aldehydes are amenable to this protocol. *N*-Substituted formamides are essential for this transformation. Readily available inorganic salt (sodium sulfide) serves as the sulfur source in water, which makes this method much more practical and efficient. Furthermore, the late-stage modification of bioactive molecules and derivatives through this protocol has been established.

Thioamides are prevalent organic motifs found in vital biological and pharmaceutical molecules, such as closthioamide,[1a] hydroxymethyl thiolactam cyclothialidine,[1b] and *N*-cyclohexylethyl-ETAsV,[1c] etc. (Figure 1). Meanwhile, as

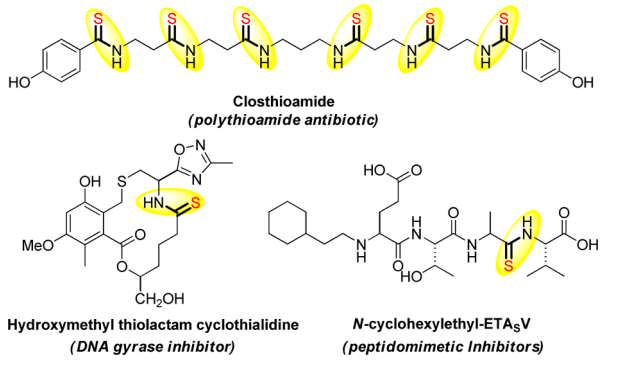
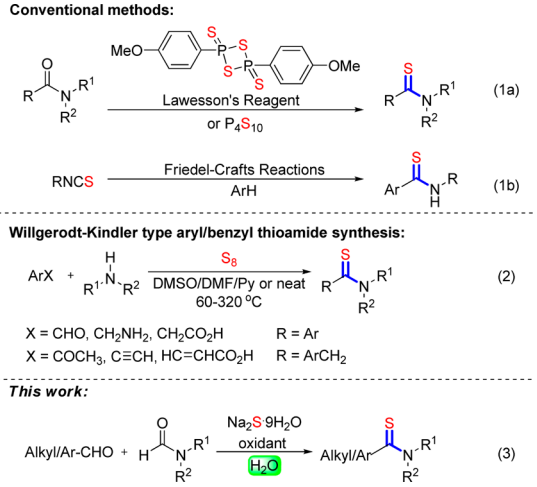


Figure 1. Representative bioactive thioamide scaffolds.

significant building blocks,[2] they are widely applied to construction of many important sulfur-containing heterocycles,[3] such as thiazolins,[3a,e] thiazolinones,[3b] thiazoles,[3c−e] tetrazoles,[3f] etc. Conventionally, Lawesson's reagent and its analogues are applied to the synthesis of thioamides (Scheme 1, eq 1a).[4] Similarly, sulfur−phosphorus-type reagents are also used to afford the thioamides through carboxylic acids[5a] or nitriles.[5b,c] Practically, isothiocyanates are employed for preparing thioamides thesis efficiently under Friedel−Crafts conditions[6]

Scheme 1. Synthetic Methods for Thioamide



(Scheme 1, eq 1b). Moreover, Willgerodt−Kindler reaction[7] is another alternative means to reach thioamides, starting from aryl aldehydes or aryl alkyl ketones. However, only aryl/benzyl thioamides can be afforded even under harsh conditions.[7c] Recently, several excellent approaches have been exploited by the Nguyen,[8a,d] Singh,[8e] and Jiang[8f] groups (Scheme 1, eq 2) that improve the approaches for the synthesis of thioamides by

Received: December 14, 2015

DOI: 10.1021/acs.orglett.5b03541
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altering different types of starting materials. Therefore, the establishment of an effective, practical, and green strategy,[9] particularly for alkyl thioamides formation, remains highly desirable. Based on the development of sulfur atom transfer reactions in our laboratory,[10] we herein report a new aqueous three-component synthesis of thioamides involving alkyl and aryl aldehydes, sodium sulfide ($Na_2S{\cdot}9H_2O$), and *N*-substituted formamides (Scheme 1, eq 3).

We commenced our study by investigating 3-phenylpropanal and *N*-formylmorpholine in the presence of sodium sulfide. With the assistance of benzoyl peroxide (BPO), the desired product was found in 33% yield (Table 1, entry 1). Then, cosolvents were

Table 1. Optimization of Reaction Conditions

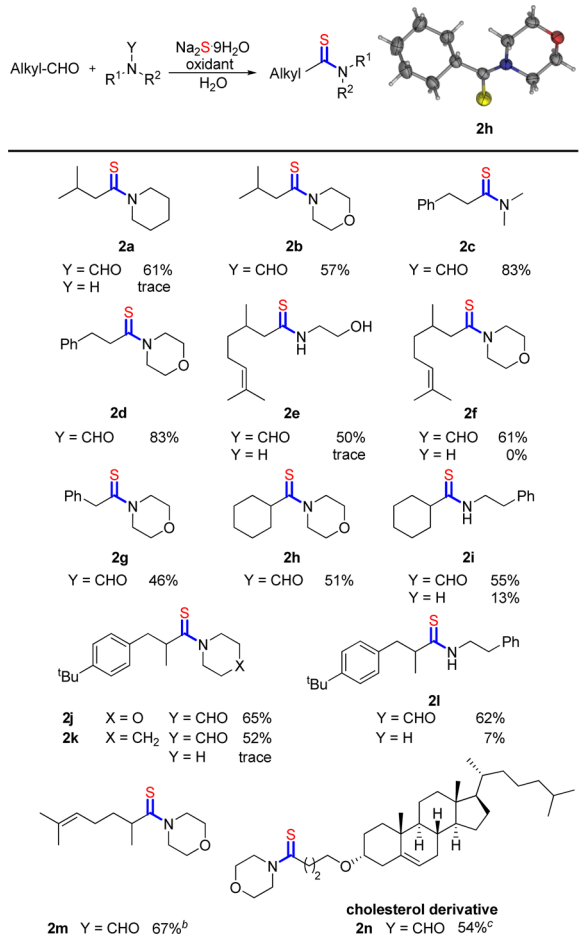
entry	$Na_2S\ 9H_2O$ (equiv)	oxidant (equiv)	additive (equiv)	temp (°C)	yield (%)[a]
1	7	BPO (5)	/	100	33
2	7	BPO (5)	DMSO (5)	100	30
3	7	BPO (5)	1,4-dioxane (5)	100	28
4	7	BPO (5)	NMP (5)	100	37
5	7	BPO (5)	Py (5)	100	42
6	3.5	BPO (2.5)	Py (5)	100	56
7	3.5	BPO (2.5)	Py (5)	100	43[b]
8	3.5	1,4-BQ(2.5)	Py (5)	100	trace
9	3.5	oxone (2)	Py (5)	100	trace
10	3.5	$K_2S_2O_8$ (2)	Py (5)	100	74
11	***3.5***	***$K_2S_2O_8$ (1.8)***	***Py (5)***	***100***	***83***
12	3.5	$K_2S_2O_8$ (1.5)	Py (5)	100	66
13	3.5	$K_2S_2O_8$ (1.8)	Py (5)	60	48
14	3.5	$K_2S_2O_8$ (1.8)	Py (5)	100	trace[c]

[a]Isolated yields. [b]*N*-Formylmorpholine (2 equiv) was used. [c]Morpholine was used instead of *N*-formylmorpholine. BPO = benzoyl peroxide; DMSO = dimethyl sulfoxide; NMP = 1-methylpyrrolidin-2-one; BQ = benzoquinone.

tested to improve the solubility, in which pyridine performed the best in 42% yield (Table 1, entry 2−5). When the amounts of $Na_2S{\cdot}9H_2O$ and BPO were reduced, the yield increased to 56% (Table 1, entry 6). There was no higher transformation when the amount of *N*-formylmorpholine (Table 1, entry 7) was reduced. Different oxidants were estimated (Table 1, entry 8−10), in which potassium persulfate ($K_2S_2O_8$) was found to be the best choice giving 74% yield. The yield was further promoted to 83% by adjusting the amount of $K_2S_2O_8$ to 1.8 equiv (Table 1, entry 11, conditions A). Remarkably, only a trace amount of product could be detected when morpholine was used instead of *N*-formylmorpholine (entry 14).

The readily oxidized alkyl aldehydes, which are big challenges in thioamide synthesis, were widely investigated under conditions A (Table 2). Different formamides and corresponding amines were surveyed under the same conditions. As shown in Table 2, a wide array of primary aldehydes worked well with *N*-substituted formamides to afford the corresponding thioamides in moderate to excellent yields (**2a**−**g**). Notably, a sensitive hydroxyl group could be tolerated in this transformation (**2e**). Moreover, the secondary aldehydes worked commendably under these conditions (**2h**−**m**). The structure was further confirmed by X-ray analysis of **2h**.[11] It is worth noting that naturally occurring aldehydes and cholesterol derivative with carbon−carbon double bonds could realize the late-stage modification[12] through this transformation, such as citronellal (**2e**, **2f**), lily aldehyde (**2j**, **2k**, **2l**), melonal (**2m**), and cholesterol derivative (**2n**), which offered a convenient method for drugs and bioactive

Table 2. Scope of Alkyl Thioamides[a]



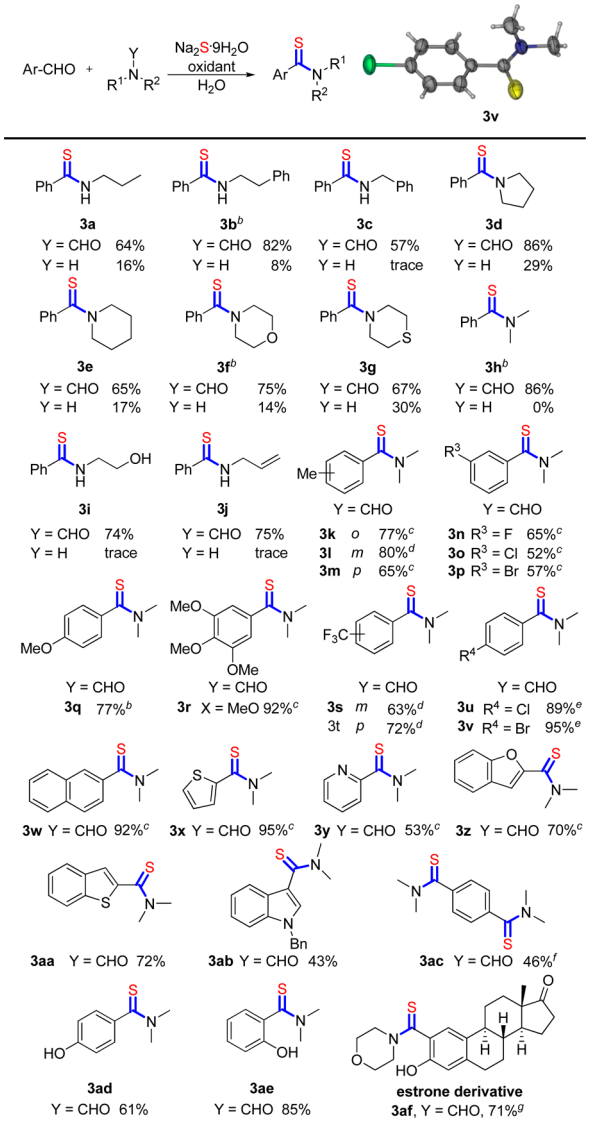
[a]Conditions A: alkyl aldehyde (0.3 mmol), $Na_2S{\cdot}9H_2O$ (1.05 mmol), $K_2S_2O_8$ (0.54 mmol), *N*-substituted formamide (1.5 mmol), pyridine (1.5 mmol), H_2O (0.5 M), 100 °C, isolated yields. [b]60 °C. [c]H_2O/glycol = 1/1 (0.3 M).

molecule modification. These results show the excellent substrate compatibility between alkyl aldehydes and formamides.

After the generalities of alkyl aldehydes were studied, aryl aldehydes were further investigated (Table 3). In general, aryl aldehydes could be assembled with both primary formamides (**3a**−**c**) and secondary formamides (**3d**−**h**) to afford the desired products in moderate to excellent yields. Delightedly, the formamides containing sensitive hydroxyl (**3i**) and allylic groups (**3j**) could afford the thioamides in 74% and 75% yields. Meanwhile, aryl aldehydes substituted with electron-rich, -neutral, and -deficient groups (**3k**−**v**) all could afford thioamides in 52%−95% yields. The structure was further confirmed by X-ray crystallographic analysis of **3v**.[13] Thioamide with a strong electron-rich group could be obtained as well in excellent yield through this transformation (**3r**). Moreover, this method could also be applied to condensed groups, and heterocycles, such as naphthalene (**3w**), thiophene (**3x**), pyridine (**3y**), benzofuran (**3z**), benzothiophene (**3aa**), and *N*-benzylindole (**3ab**) could be afforded in excellent yields. Remarkably, bis(carbothioamide) (**3ac**) could be obtained through double thioamidation. 4-Hydroxybenzaldehyde (**3ad**), salicylaldehyde (**3ae**), and estrone derivative (**3af**) containing a free phenolic hydroxyl group could afford the desired thioamides

DOI: 10.1021/acs.orglett.5b03541
Org. Lett. XXXX, XXX, XXX−XXX

Table 3. Scope of Aryl Thioamides[a]

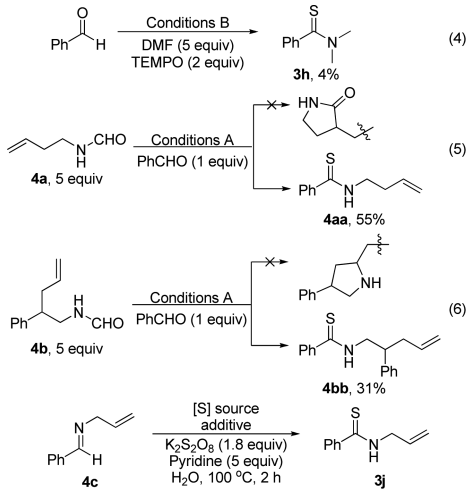


[a]Conditions A, isolated yields. [b]Conditions B: aldehyde (0.5 mmol), $Na_2S·9H_2O$ (1.75 mmol), BPO (1.25 mmol), DMF (2.5 mmol), H_2O (1 M), 100 °C. [c]Conditions B in 60 °C. [d]Aldehyde (0.5 mmol), $Na_2S·9H_2O$ (3.5 mmol), BPO (2.5 mmol), DMF (5 mmol), H_2O (0.5 M), 60 °C. [e]Aldehyde (0.5 mmol),$Na_2S·9H_2O$ (3.5 mmol), BPO (2.5 mmol), DMF (5 mmol), H_2O (0.5 M), 100 °C. [f]Aldehyde (0.3 mmol), $Na_2S·9H_2O$ (2.1 mmol), $K_2S_2O_8$ (1.08 mmol), DMF (3 mmol), pyridine (3 mmol), H_2O (0.3 M), 100 °C. [g]H_2O/glycol = 1/1 (0.3 M).

in excellent yields as well. These results show great substrate compatibility.

In order to gain mechanistic insight into this protocol, radical trapping reagent TEMPO was introduced to the reaction system, in which the formation of desired product **3h** was sharply suppressed (Scheme 2, eq 4). Furthermore, radical clock reactions were examined through designed **4a** and **4b** with benzaldehyde under the standard conditions.[14] However, thioamides (**4aa** and **4bb**) were obtained instead of cyclization products, which excluded the radical pathway to a large extent (Scheme 2, eq 5 and 6). Imine intermediate was another possibility,[8c] which was formed from aldehyde and amine. Then *N*-benzylideneprop-2-en-1-amine (**4c**) was subjected to the standard conditions with only 10% of product formation

Scheme 2. Control Experiments

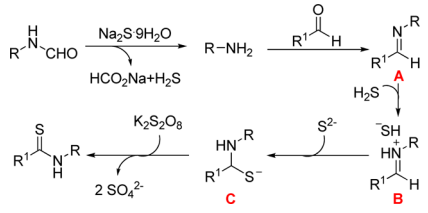


entry	[S] source (equiv)	additive (equiv)	yield of **3j** (%)
1	$Na_2S·9H_2O$ (3.5)	/	10
2	$Na_2S·9H_2O$ (3.5)	$HCO_2Na·2H_2O$ (5)	11
3	$Na_2S·9H_2O$ (3.5)	$NaH_2PO_4·2H_2O$ (3.5)	71
4	$Et_3N·H_2S$ (3.5)	/	trace
5	$Et_3N·H_2S$ (3.5) $Na_2S·9H_2O$ (3.5)	/	72

(Scheme 2, entry 1). Sodium formate was further added, affording the similar result (Scheme 2, entry 2). But when sodium dihydrogen phosphate was added to the system, **3j** was obtained in 71% yield (Scheme 2, entry 3), indicating that hydrogen sulfide generated from sodium sulfide assisting the transformation. To furtherly verify the hypothesis, triethylammonium hydrogen sulfide was examined in the system (Scheme 2, entry 4 and 5), which demonstrated that imine was activated by hydrogen sulfide and then attacked by sodium sulfide.

On the basis of the above results, the possible mechanism is proposed in Scheme 3. Amine release was achieved with the help

Scheme 3. Proposed Mechanism



of sodium sulfide (SI, part III), accompanied by sodium formate and hydrogen sulfide.[15] Imine **A** was formed with aldehyde[16] and then activated by hydrogen sulfide, forming iminium hydrogen sulfide **B**. Sulfide **B** was attacked by sulfur anion, and intermediate **C** was immediately formed, which was further oxidized by potassium persulfate to afford the desired thioamide.

In summary, we have developed an efficient aqueous method for the synthesis of thioamides involving aldehydes, sodium sulfide, and *N*-substituted formamides. Both alkyl and aryl aldehydes are amenable to this protocol with great functional group toleration. *N*-Substituted formamides are superior to the corresponding amines due to the slow release of hydrogen sulfide through the hydrolysis of formamide by sodium sulfide. Readily available inorganic salt (sodium sulfide) serves as sulfur source in water, which makes it more practical and efficient. Further studies on synthetic applications are ongoing in our laboratory.

DOI: 10.1021/acs.orglett.5b03541
Org. Lett. XXXX, XXX, XXX−XXX

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.5b03541.

Experimental procedure, NMR spectra, and X-ray and analytical data for all new compounds (PDF)
X-ray data for **2h** (CIF)
X-ray data for **3v** (CIF)

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: xfjiang@chem.ecnu.edu.cn.

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

Financial support was provided by NBRPC (973 Program, 2015CB856600), NSFC (21472050, 21272075), NCET (120178), DFMEC (20130076110023), Fok Ying Tung Education Foundation (141011), the program for Shanghai Rising Star (15QA1401800), Professor of Special Appointment (Eastern Scholar) at Shanghai Institutions of Higher Learning, and the Changjiang Scholar and Innovative Research Team in University.

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DOI: 10.1021/acs.orglett.5b03541
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