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4-Dimethylaminopyridine-catalyzed synthesis of isothiocyanates from amines and carbon disulfide

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ABSTRACT

Isothiocyanates were synthesized by reactions between primary amines and CS_2 in the presence of 4-dimethylaminopyridine as a catalyst and *tert*-butyl hydroperoxide as an oxidant. Various aryl, benzyl, alkyl, and hydroxyl amines were transformed into the corresponding isothiocyanates in 41–82% yields.

Introduction

Isothiocyanates are an important class of heteroallenic compounds because they have a broad range of bioactivities (Figure 1). For example phenyl, allyl, benzyl, and phenethyl isothiocyanates isolated from Brassicales show antibiotic and antitumor activities [1–3]. Sulforaphane [4], which can be isolated from cruciferous vegetables, exhibits anticancer activity. In addition, isothiocyanates are versatile building blocks for the synthesis of chiral thiourea catalysts [5–8] and various heterocycles [9–10]. Therefore, the development of methods for the synthesis of isothiocyanates has attracted considerable attention.

Isothiocyanates have traditionally been synthesized from amines, tertiary alcohols, thioureas, amides, nitrile oxides, and aldoximes [11]. Of these precursors, amines are used the most frequently, owing to their versatility and ready accessibility. There are two general strategies for the synthesis of isothiocyanates from amines. The first involves direct thiocarbonylation of amines with a thiocarbonyl transfer reagent [12–16] such as thiophosgene, di(2-pyridyl) thiocarbamate, or $(Me_4N)SCF_3$. Although this strategy can provide isothiocyanates in a single step, the transfer reagents are either toxic or not commercially available, and the transformation usually requires anhydrous conditions. Recently, thiocarbonyl fluoride [17] derived from CF_3SiMe_3, elemental sulfur, and KF has also been reported for the synthesis of isothiocyanates under air. In the second strategy (Scheme 1), the primary amine is first converted to the corresponding dithiocarbamate salt by reaction with CS_2 in the presence of excess base, and then the dithiocarbamate salt undergoes desulfurization to afford the isothiocyanate. $Na_2S_2O_8$ [18], H_2O_2 [19], I_2 [20–21], $Fe(NO_3)_3$ [22], and various other reagents [23–31] have been used for this purpose. However, these two strategies require excess base, and therefore the development of new methods would be desirable.

Results and discussion

Recently, we reported a catalytic desulfurization strategy for the synthesis of guanidines [32–33], and we envisioned that a similar strategy could be used for the synthesis of isothiocyanates with catalytic base. In fact, in 1997 Tajima and Li reported the synthesis of hydroxyl isothiocyanates from hydroxy primary amines and CS_2 in the presence of catalytic NEt_3 [34], but similar transformations of other amines require excess base [19].

We began our work on the development of a catalytic method for the synthesis of isothiocyanates by exploring reactions of CS_2 and 4-methoxyaniline (**1a**) as a model substrate (Table 1). Initially, we used I_2 as an additive and 70% aqueous *tert*-butyl hydroperoxide (TBHP) as an oxidant at rt in methanol and found that only 25% of the desired isothiocyanate (**2a**) was produced (entry 1).

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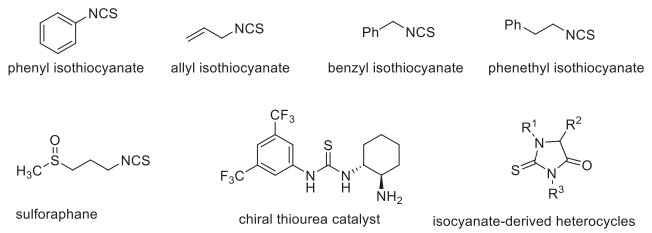
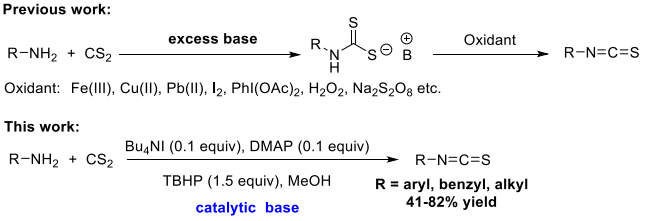
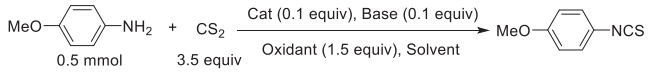


Fig. 1. Representative isothiocyanates and related compounds.



Scheme 1. Synthesis of isothiocyanates from amines and CS_2.

Table 1
Optimization of reaction conditions.[a]



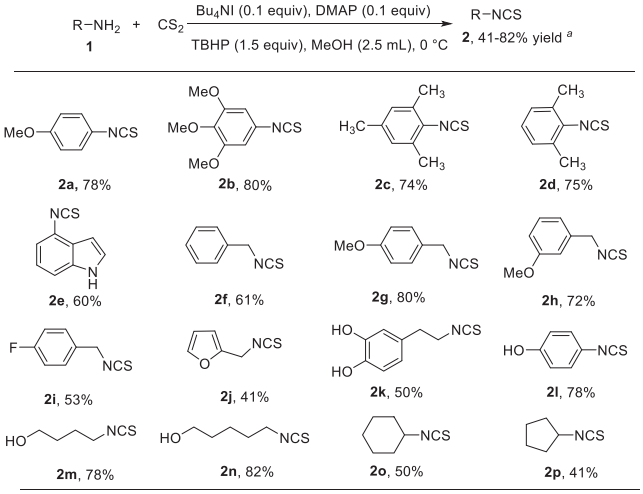
Entry	Solvent	t (°C)	Additive	Base	Oxidant	Yield
1	MeOH	25	I_2	None	TBHP	25
2	MeOH	25	Bu_4NI	None	TBHP	27
3	MeOH	25	Bu_4NI	DMAP[b]	TBHP	65
4	MeOH	25	Bu_4NI	NEt_3	TBHP	60
5	DMF	25	Bu_4NI	DIPEA	TBHP	58
6	MeOH	25	Bu_4NI	DMAP	H_2O_2	45
7	MeOH	25	Bu_4NI	DMAP	$Na_2S_2O_8$	56
8	MeOH	25	Bu_4NI	DMAP	NaClO	48
9	MeOH	0	Bu_4NI	DMAP	TBHP	73
10[c]	MeOH	0	Bu_4NI	DMAP	TBHP	78

[a] Reaction conditions: the catalyst and then the oxidant were added to a solution of the amine (0.5 mmol) in the solvent (5 mL.
[b] DMAP = 4-dimethylaminopyridine, DIPEA = N,N-diisopropylamine.
[c] The amount of MeOH was decreased to 2.5 mL.

Changing the additive to Bu_4NI (entry 2) or an iodine reagent such as *N*-iodosuccinimide or phenyliodine(III) diacetate (not shown) had no obvious effect on the yield. However, when 0.1 equiv of *N*,*N*-dimethylaminopyridine (DMAP) was present in a reaction mixture containing Bu_4NI, the desired isothiocyanate was obtained in 65% yield (entry 3). Careful screening of several other bases, solvents and oxidants (entries 4–8) revealed that the combination of DMAP and TBHP was optimal. In addition, the yield of the isothiocyanate could be improved to 78% by decreasing the reaction temperature to 0 °C and increasing the concentration of the reactants (entries 9 and 10).

With the optimal conditions in hand (Table 1, entry 10), we carried out reactions of CS_2 with various primary anilines, benzyl amines, hydroxyl amines, and alkyl amines (Table 2). Most of the tested substrates afforded the corresponding isothiocyanates, and the yields were moderate to good (41–82%). The reaction worked well with electron-rich anilines (**2a** and **2b**) and sterically hindered anilines (**2c** and **2d**). In contrast, electron-deficient amines gave thioureas as the major products, and only traces of the desired isothiocyanates were detected (not shown). We surmised that this outcome was probably due to reaction of the product isothiocyanates with the amine substrates rather than with CS_2; that is, the unchanged starting amines reacted preferentially with the generated product isothiocyanates but not with CS_2.

Table 2
Catalytic synthesis of isothiocyanates from various primary amines.



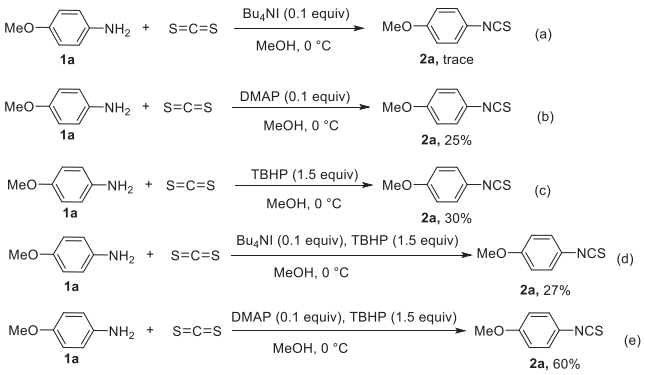
[a]Reaction conditions: to a solution of amine (0.5 mmol) in MeOH (2.5 mL) at 0 °C was added Bu_4NI (16 mg), 4-dimethylaminopyridine (DMAP, 6 mg), CS_2 (133 mg), and 70% aqueous *tert*-butyl hydroperoxide (TBHP, 96 mg) in that order.

To elucidate the reaction mechanism, we conducted a series of control experiments (Scheme 2). We started with reactions of **1a** and CS_2 in MeOH in the presence of Bu_4NI, DMAP, or TBHP alone. The reaction in the presence of Bu_4NI alone gave only a trace of isothiocyanate **2a** (Scheme 2a), and reactions in the presence of DMAP or TBHP alone gave the isothiocyanate in 25% or 30% yield, respectively, after 6 h (Scheme 2b,c). When a combination of Bu_4NI and TBHP or a combination of DMAP and TBHP was used, **2a** was isolated in 27% or 60% yield, respectively (Scheme 2d,e). These experiments demonstrate that Bu_4NI, DMAP, and TBHP were essential for the transformation. In a radical capture experiment conducted with 2.0 equiv of TEMPO (2,2,6,6-tetramethyl-1-piperidinyloxy), none of

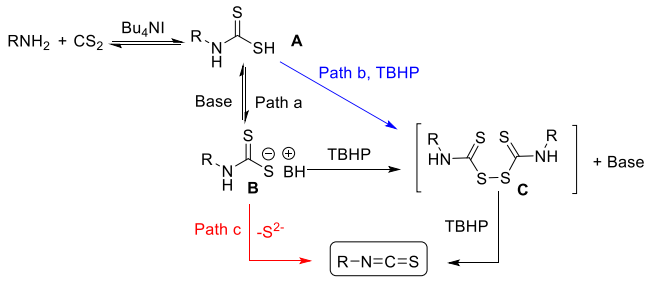
the TEMPO capture product was detected, and the yield of the isothiocyanate was 73% (results not shown). This finding suggests that the reaction did not proceed via a radical pathway [35].

On the basis of our control experiments and previously reported results for desulfurization of dithiocarbamate salts [19,36], we propose the reaction pathway outlined in Scheme 3. First,

addition of the amine to CS_2 gives dithiocarbamate acid **A**, which reacts with the base to give dithiocarbamate salt **B**. Salt **B** either can be oxidized by TBHP [37] to give the isothiocyanate via intermediate **C** (path b) or can undergo elimination of S^{2-} to form the isothiocyanate directly [36] (path c). Intermediate **C** can also be obtained by direct oxidation of intermediate **A** (path a).



Scheme 2. Control experiments.



Scheme 3. Proposed mechanism.

Meanwhile, TBHP may oxidize S^{2-} to elemental sulfur [32] or [SO_x-H] [37]. Considering that Bu_4NI was involved in the reaction and that in the absence of Bu_4NI, the reaction afforded the desired isothiocyanate in only 60% yield (Scheme 2e), we surmised that Bu_4NI facilitated addition of the amine to CS_2.

Conclusion

In conclusion, we have developed a protocol for DMAP-catalyzed synthesis of isothiocyanates from amines and CS_2. Various aryl, benzyl, alkyl, and hydroxyl primary amines were transformed into the corresponding isothiocyanates in 41–82% yields, and the mechanism was explored by means of control experiments. Although the yields achieved with this protocol were moderate, we believe our findings will lead to the development of more-efficient methods for catalytic synthesis of isothiocyanates.

Experimental section

General information

Unless otherwise noted, all reagents were used without further purification. Flash column chromatographies were performed on Qingdao silica gel (200–300 mesh). ^{1}H, ^{13}C spectra were measured on a NMR instrument (500 MHz for ^{1}H NMR; 126 MHz for ^{13}C NMR). Chemical shifts of ^{1}H NMR spectra were recorded relative to internal standard (TMS δ 0.00). Chemical shifts of ^{13}C NMR spectra were recorded relative to solvent resonance ($CDCl_3$: δ 77.0).

General procedure for the synthesis of isothiocyanates

To a solution of amine (0.5 mmol) in MeOH (2.5 mL) at 0 °C was added Bu_4NI (18 mg, 0.05 mmol), DMAP (6 mg, 0.05 mmol) sequentially. The solution was stirred at 0 °C for 10 min. After that, 70% aq. TBHP (96 mg, 0.75 mmol), CS_2 (133 mg, 1.75 mmol) was added. The solution was stirred at 0 °C for 6–18 h. Upon consumption of the amine, 50 mL EtOAc was added, the organic phase was washed with H_2O (10 mL) and dried with Na_2SO_4. After concentration of the organic phase under reduced pressure, the residue was purified by column chromatography on silica gel using EtOAc/PE = 1:20 to give the desired product.

1-isothiocyanato-4-methoxybenzene (**2a**) [18].

Colorless oil; 64 mg, 78% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16 (d, J = 8.9 Hz, 2H), 6.85 (d, J = 8.9 Hz, 2H), 3.80 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 158.62, 134.29, 126.90, 123.76, 114.84, 55.54.

5-isothiocyanato-1,2,3-trimethoxybenzene(**2b**) [38].

Brown solid; 90 mg, 80% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.46 (s, 2H), 3.84 (s, 6H), 3.83 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 153.66, 137.89, 135.04, 126.60, 103.40, 60.99, 56.30.

2-isothiocyanato-1,3,5-trimethylbenzene(**2c**) [18].

White solid; 66 mg, 74% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.84 (s, 2H), 2.32 (s, 6H), 2.26 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 136.96, 135.19, 134.79, 128.69, 126.93, 21.01, 18.51.

2-isothiocyanato-1,3-dimethylbenzene (**2d**) [39].

Colorless oil; 62 mg, 75% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16–6.88 (m, 3H), 2.37 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 135.68, 135.06, 129.63, 127.97, 126.88, 18.62.

4-isothiocyanato-1H-indole (**2e**) [18].

Yellow oil; 52 mg, 60% yield; ^{1}H NMR (500 MHz, Chloroform-d) δ 8.33 (brs, 1H), 7.32 (d, J = 8.1 Hz, 1H), 7.28–7.23 (m, 1H), 7.11 (t, J = 7.9 Hz, 1H), 7.04–6.90 (m, 1H), 6.75–6.66 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 136.65, 136.15, 125.37, 125.09, 122.90, 122.19, 116.61, 110.81, 100.30.

(isothiocyanatomethyl)benzene(**2f**) [18].

Colorless oil; 46 mg, 61% yield;^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46–7.28 (m, 5H), 4.69 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 134.27, 132.67, 128.92, 128.32, 126.80, 48.68.

1-isothiocyanato-4-(methoxymethyl)benzene(**2g**) [18].

Colorless oil; 72 mg, 80% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16 (d, J = 8.9 Hz, 2H), 6.85 (d, J = 8.9 Hz, 2H), 4.61 (s, 2H), 3.80 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 159.63, 132.28, 128.31, 126.34, 114.31, 55.29, 48.26.

1-(isothiocyanatomethyl)-4-methoxybenzene(**2h**) [40].

Colorless oil; 65 mg, 72% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.21 (m, 1H), 7.02–6.75 (m, 3H), 4.66 (s, 2H), 3.81 (s, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 160.11, 135.80, 132.80, 130.03, 119.01, 113.88, 112.50, 55.33, 48.64.

1-fluoro-4-(isothiocyanatomethyl)benzene(**2i**) [41].

Colorless oil; 44 mg, 53% yield;^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.27 (m, 2H), 7.12–7.00 (m, 2H), 4.68 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ162.63 (d, J = 247.5 Hz), 133.28, 130.17 (d, J = 3.3 Hz), 128.72 (d, J = 8.3 Hz), 115.93 (d, J = 22.0 Hz), 48.11.

2-(isothiocyanatomethyl)furan(**2j**) [18].

Colorless oil; 29 mg, 41% yield;^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 (dd, J = 1.8, 0.9 Hz, 1H), 6.35 (dd, J = 9.0, 1.4 Hz, 2H), 4.64 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.38, 143.18, 135.31, 110.60, 108.65, 41.92.

4-(2-isothiocyanatoethyl)benzene-1,2-diol(**2k**) [42].

Colorless oil; 49 mg, 50% yield;^{1}H NMR (500 MHz, $CDCl_3$) δ 6.82 (d, J = 8.1 Hz, 1H), 6.74 (d, J = 2.1 Hz, 1H), 6.64 (dd, J = 8.1, 2.1 Hz, 1H), 5.84–5.67 (brs, 2H), 3.65 (t, J = 6.8 Hz, 2H), 2.85 (t, J = 6.8 Hz, 2H).^{13}C NMR (126 MHz, $CDCl_3$) δ 143.77, 142.69, 130.69, 130.10, 121.38, 115.99, 115.75, 46.55, 35.81.

4-isothiocyanatophenol(**2l**) [18].

Colorless oil; 59 mg, 78% yield;^{1}H NMR (500 MHz, $CDCl_3$) δ 7.10 (d, J = 8.8 Hz, 2H), 6.79 (d, J = 8.8 Hz, 2H), 6.00 (s, 1H).^{13}C NMR (126 MHz, $CDCl_3$) δ 154.76, 134.00, 127.18, 123.75, 116.40.

4-isothiocyanatobutan-1-ol (**2m**) [43].

Colorless oil; 51 mg, 78% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.69 (t, J = 5.8 Hz, 2H), 3.58 (t, J = 6.5 Hz, 2H), 1.91–1.76 (m, 2H), 1.74–1.64 (m, 2H).^{13}C NMR (126 MHz, $CDCl_3$) δ 130.30, 61.79, 44.99, 29.51, 26.65.

5-isothiocyanatopentan-1-ol (**2n**) [19].

Colorless solid; 60 mg, 82% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.66 (t, J = 6.4 Hz, 2H), 3.54 (t, J = 6.6 Hz, 2H), 1.85 (brs, 1H), 1.79–1.70 (m, 2H), 1.68–1.57 (m, 2H), 1.56–1.44 (m, 2H).^{13}C NMR (126 MHz, $CDCl_3$) δ 130.01, 62.29, 44.97, 31.70, 29.69, 22.87.

Isothiocyanatocyclohexane (**2o**) [18].

Colorless oil; 35 mg, 50% yield; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.87–3.49 (m, 1H), 2.04–1.86 (m, 2H), 1.77–1.61 (m, 4H), 1.54–1.45 (m, 1H), 1.44–1.32 (m, 3H).^{13}C NMR (126 MHz, $CDCl_3$) δ 130.18, 55.46, 33.24, 25.07, 23.24.

isothiocyanatocyclopentane (**2p**) [18].

Colorless oil; 26 mg, 41% yield; ^{1} ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.60–3.68 (m, 1H), 2.07–1.74 (m, 6H), 1.73–1.58 (m, 2H) [13].

C NMR (126 MHz, $CDCl_3$) δ 129.80, 57.70, 34.35, 23.21.

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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