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Environmentally green synthesis of thioformamide derivatives

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Abstract: Reactions of isocyanides with thioacids in water proceeded smoothly at room temperature and in neutral conditions to afford thioformylamide and thioformamide derivatives in high yields. The reaction proceeded smoothly and cleanly under mild conditions and no side reactions were observed.

Key words: Isocyanide, thioacid, thioformylamide, thioformamide, water

1. Introduction

Recently, multicomponent reactions (MCRs) have become an important tool in modern primary synthetic chemistry as these reactions expand the efficiency by combining several operational steps without any isolation of intermediates or changes in the conditions.[1−10] Therefore, this principle is very efficient in terms of time as well as resources.[11] Among the known MCRs, the most valuable reactions are those based on isocyanides. Isocyanide-based multicomponent reactions (abbreviated to IMCRs by Ugi and Dömling)[11−14] due to their synthetic potential, their inherent atom efficiency, convergent nature, ease of implementation, and molecular diversity have attracted much attention. Therefore, because of these advantages they offer they are a valuable tool in the field of combinatorial chemistry.[12−14] The reaction of carboxylic acids with isocyanides forms the basis of the Passerini and Ugi reactions, which are much admired in combinatorial chemistry.[15−17]

The amides and their thio analogues are a significant constituent of many biologically active compounds.[18] Thioamides are used as isosteric replacements for amides.[19] In recent years, thioamides and their derivatives because of their utility as synthons in organic chemistry, for example, the synthesis of a variety of heterocycles such as thiazoline or thiazole derivatives, betaines, mesoionic rhodanine, and other heterocyclic compounds, have received great attention.[20−23] In the future, reactions of thioamides will play an important role in the development of polypeptides and protein chemistry. Therefore, the development of easy synthetic methods toward thioamides creates an extensive area of research in organic synthesis.[24] The most important method for the preparation of thioamides involves constitution of the parent amide followed by thionation.[25,26]

Recently, we established a one-pot method for the preparation of organic compounds.[27−30] As part of our ongoing program to develop efficient and robust methods for the synthesis of heteroatom-containing compounds,[31−37] we wish to report the preparation of thioformylamide derivatives **3a–d** and thioformamide derivatives **4e–g** by a 2-component condensation reaction of thioacid **1** and isocyanide **2** in water with excellent yields (Scheme 1).

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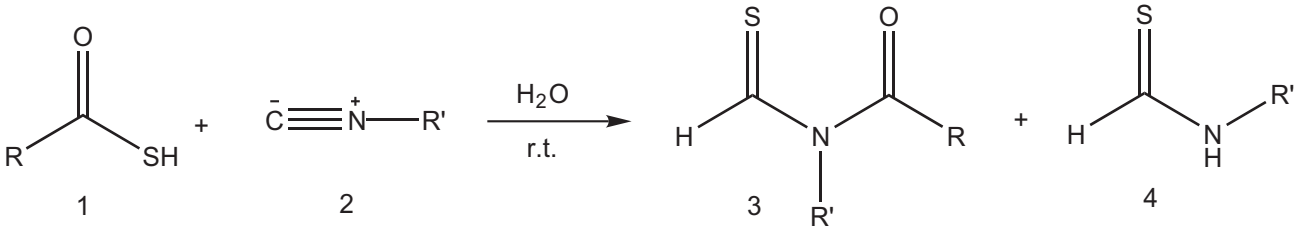


Scheme 1. Two-component synthesis of thioformylamide and thioformamide derivatives (see Tables 1 and 2).

2. Experimental

The starting materials and solvents were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. The methods used to follow the reactions were TLC and NMR. TLC and NMR indicated that there was no side product. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were measured on a Jasco 6300 FTIR spectrometer. ^{1}H and ^{13}C NMR spectra ($CDCl_3$) were recorded on a BRUKER DRX-250 AVANCE spectrometer at 250.0 and 62.5 MHz, respectively. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a FINNIGAN-MATT 8430 mass spectrometer operating at an ionization potential of 70 eV. Preparative layer chromatography (PLC) plates were prepared from Merck silica gel (F_{254}) powder.

2.1. General procedure for the preparation of compounds 3 and 4

A mixture of isocyanide (1 mmol) and thioacid (1 mmol) in H_2O (5 mL) was stirred at room temperature for 24 h. The solvent was removed under reduced pressure, and the viscous residue was purified by preparative layer chromatography (PLC) (silica gel (F_{254}) powder; petroleum ether-ethyl acetate (4:1)). The characterization data of the compounds are given below.

N^1-cyclohexyl-N^1-thioformylbenzamide (3a)

Yellow powder, mp 55–57 °C, (yield: 87%). IR (neat): υ = 3003, 2939, 1700, 1599, 1418, 1325, 1223, 789, 698 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm): 1.24–2.30 (m, 10H, cyclohexane), 5.14–5.24 (m, 1H, cyclohexane), 7.45–7.60 (m, 5H, arom), 10.00 (s, 1H, HC=S). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm): 25.31, 26.12, 28.61 ($5CH_2$, cyclohexane), 57.97 (CH, cyclohexane), 129.01, 129.62, 132.93 (5CH, arom), 133.74 (C, arom), 173.49 (C=O), 195.47 (C=S). MS, m/z (%): 247 (41), 142 (100), 105 (95), 77 (58), 55 (16). Analysis of $C_{14}H_{17}NOS$ (247.36). (% calculation/found): C: 67.98/67.93, H: 6.93/6.98, N: 5.66/5.61.

N^1-benzyl-N^1-thioformylbenzamide (3b)

Yellow oil, (yield: 89%). IR (neat): υ = 3061, 2934, 1671, 1598, 1580, 1447, 1207, 903, 773, 686 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm): 5.57 (s, 2H, CH_2), 7.30–7.81 (m, 10H, arom), 10.27 (s, 1H, HC=S). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm): 47.26 (CH_2), 127.73, 128.47, 128.57, 128.95, 129.02, 132.53 (10CH, arom), 132.73, 135.70 (2C, arom), 173.90 (C=O), 195.87 (C=S). Analysis of $C_{15}H_{13}NOS$ (255.33). (% calculation/found): C: 70.56/70.61, H: 5.13/5.08, N: 5.49/5.54.

N^1-cyclohexyl-N^1-thioformylacetamide (3c)

Yellow oil, (yield: 86%). IR (neat): υ = 2931, 1697, 1535, 1450, 1285, 970 cm^{-1}. ^{1}H NMR (250 MHz,

$CDCl_3$) δ (ppm): 1.34–2.12 and 2.22–2.34 (m, 10H, $5CH_2$ of cyclohexane), 2.16 (s, 3H, CH_3), 4.40–4.70 (m, 1H, cyclohexane), 9.33 (s, 1H, HC=S). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm): 24.50, 25.36, 30.98 ($5CH_2$, cyclohexane), 31.37 (CH_3), 51.64 (CH, cyclohexane), 187.38 (C=O), 207.23 (C=S). Analysis of $C_9H_{15}NOS$ (185.29). (% calculation/found): C: 58.34/58.39, H: 8.16/8.21, N: 7.56/7.51.

N^1-(1,1,3,3-tetramethylbutyl)-N^1-thioformylbenzamide (3d)

Yellow oil, (yield: 86%). IR (neat): v = 3063, 2953, 1736, 1691, 1562, 1447, 1369, 1200, 772, 686 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm): 0.98 (s, 9H, $3CH_3$), 1.30 (s, 6H, $2CH_3$), 1.60 (s, 2H, CH_2), 7.30–8.06 (m, 5H, arom), 9.20 (s, 1H, HC=S). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm): 29.71, 31.47 ($5CH_3$), 55.05 (CH_2), 31.64, 59.48 (2C), 128.46, 128.93, 134.51 (5CH, arom), 136.95 (C, arom), 186.81 (C=O), 207.14 (C=S). Analysis of $C_{16}H_{23}NOS$ (277.42). (% calculation/found): C: 69.27/69.33, H: 8.36/8.42, N: 5.05/5.11.

N-benzylthioformamide (4e)

Yellow oil, (yield: 87%). IR (neat): v = 3277 (NH), 3059, 2934, 2855, 1698, 1625, 1540, 1385, 1236, 696 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm): 4.86 (s, 2H, CH_2), 7.35–7.52 (m, 5H, arom), 7.57 (s, 1H, NH), 9.49 (s, 1H, HC=S). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm): 47.60 (CH_2), 128.27, 128.37, 129.00 (5CH, arom), 127.54 (C, arom), 188.85 (C=S). Analysis of C_8H_9NS (151.23). (% calculation/found): C: 63.54/63.62, H: 6.00/5.92, N: 9.26/9.34.

N-(tert-butyl)thioformamide (4f)

Yellow powder, mp 68–70 °C, (yield: 84%). IR (neat): v = 2922, 1732, 1652, 1568, 1370, 739 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm): 1.36 (s, 9H, $3CH_3$), 8.43 (s, 1H, NH), 9.27 (d, 1H, J = 15.5 Hz, HC=S). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm): 29.77 ($3CH_3$), 56.02 (C), 187.36 (C=S). Analysis of $C_5H_{11}NS$ (117.21). (% calculation/found): C: 51.23/51.29, H: 9.46/9.40, N: 11.95/11.89.

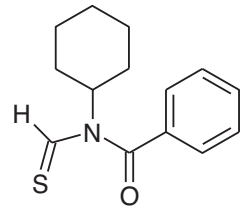
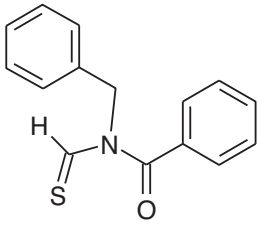
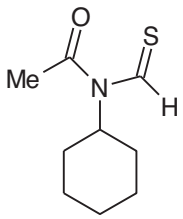
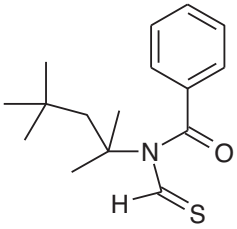
N-(1,1,3,3-tetramethylbutyl)thioformamide (4g)

Yellow oil, (yield: 88%). IR (neat): v = 2955, 1567, 1469, 1380, 944 cm^{-1}. ^{1}H NMR (250 MHz, $CDCl_3$) δ (ppm): 0.98 (s, 9H, $3CH_3$), 1.39 (s, 6H, $2CH_3$), 1.59 (s, 2H, CH_2), 8.35 (s, 1H, NH), 9.19 (d, 1H, J = 15.5 Hz, HC=S). ^{13}C NMR (62.5 MHz, $CDCl_3$) δ (ppm): 29.76, 31.49 ($5CH_3$), 55.14 (CH_2), 31.66, 59.46 (2C), 186.93 (C=S). Analysis of $C_9H_{19}NS$ (173.32). (% calculation/found): C: 62.37/61.30, H: 11.05/11.12, N: 8.08/8.15.

3. Results and discussion

The thioacid derivatives **1** with isocyanides **2** in H_2O reacted together in a 1:1 ratio at room temperature to produce thioformylamide derivatives **3a–d** and thioformamide derivatives **4e–h** (Scheme 1; Tables 1 and 2). Thiobenzoic acid with cyclohexyl isocyanide, benzyl isocyanide, 1,1,3,3-tetramethylbutyl isocyanide, and thioacetic acid with cyclohexyl isocyanide produced thioformylamide derivatives **3a–d.** These reactions proceeded smoothly and cleanly under mild conditions along with a small amount of thioformamide **4**. Reactions of thiobenzoic acid with tert-butyl isocyanide, and thioacetic acid with benzyl isocyanide, 1,1,3,3-tetramethylbutyl isocyanide, and tertbutyl isocyanide produced thioformamide derivatives **4e–g.**[38,39] These reactions also proceeded smoothly and clearly under mild conditions and no side reactions were observed. In comparison with other methods reported previously, the important advantage of the reported method in this paper is to use water as an available, cheap, nontoxic, and environmentally green solvent at ambient temperature, without using any kind of reagent.

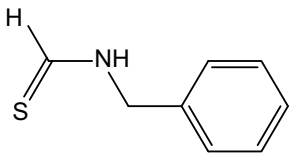
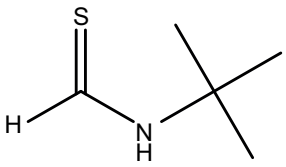
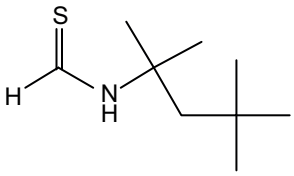
Table 1. Synthesis of thioformylamide derivatives **3a–d** from thioacid **1** and isocyanide **2** in H_2O (see Scheme 1).

3	R	R'	Product	Yield (%)[a]
a	Ph	Cyclohexyl		87
b	Ph	Benzyl		89
c	CH_3	Cyclohexyl		86
d	Ph	1,1,3,3-tetramethylbtyl		86

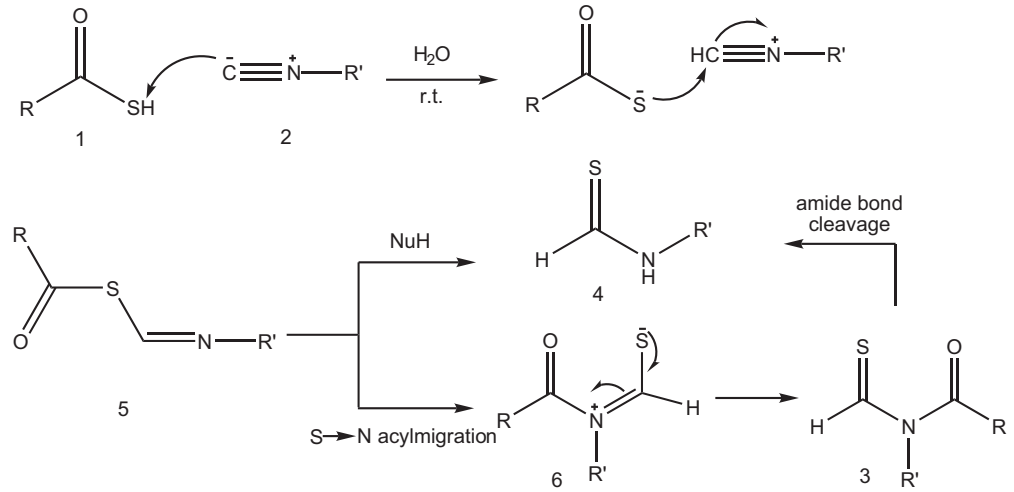
[a]Isolated yields.

A possible mechanism for the present reactions is shown in Scheme 2. On the basis of the chemistry of isocyanides with acids,[40] it is reasonable to assume that the first step may involve protonation of the isocyanide by the thioacid to produce S-acylimine **5**, which may undergo intramolecular attack by nitrogen to thionyl carbon (S→N acylmigration) to form adduct **6**. Then this intermediate affords the thioformylamide derivatives **3.** S-acylimine **5** may undergo nucleophilic attack by H_2O to form adduct **4** or hydrolysis of **3** might happen and form adduct **4**.

Table 2. Synthesis of thioformamide derivatives **4e–g** from thioacid **1** and isocyanide **2** in H_2O (see Scheme 1).

4	R'	Product	Yield (%)[a]
e	Benzyl		87
f	*Tert-butyl*		84
g	1,1,3,3-tetramethylbutyl		88

[a]Isolated yields.



Scheme 2. Proposed mechanism for the formation of thioformylamide and thioformamide derivatives.

4. Conclusions

The reported method offers a mild, simple, and efficient route for the preparation of thioformylamide and thioformamid derivatives. Its ease of work-up, high yields, and fairly mild reaction conditions make it a useful addition to modern synthetic methodologies.

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