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ORIGINAL PAPER

Green hydrothiolation of dialkyl azodicarboxylates

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Abstract
Hydrothiolations are the most useful and atom-economical pathway for the generation of sulfur–carbon and sulfur–nitrogen bonds. In particular, the preparation of sulfenylhydrazines-1,2-dicarboxylates by sulfur functionalization of nitrogen–nitrogen double bonds has received great interest in organic synthesis of compounds of unique biological properties. Previous protocols have drawbacks such as long reaction times and the use of toxic solvents. Here, we describe the preparation of sulfenylhydrazines-1,2-dicarboxylates by hydrothiolation of dialkyl azodicarboxylates under neat conditions. This new eco-friendly methodology afforded the products in up to 99% yield, in only 5 min at room temperature. A possible reaction mechanism is proposed.

Keywords Neat conditions · Atom-economical · Organosulfur · Hydrothiolation · Sulfenylhydrazines

Introduction

Catalytic hydrothiolation processes have emerged as the most useful and atom-economical pathway for the generation of new sulfur–carbon bonds (Beletskaya and Ananikov 2011; Degtyareva et al. 2019; Dondoni and Marra 2014; Sinha and Equbal 2019). In this regard, the most traditional protocols usually involve the monohydrothiolation of alkenes and alkynes via Markovnikov (Giuseppe et al. 2012; Malyshev et al. 2006; Yang et al. 2009; Palacios et al. 2016) or anti-Markovnikov type addition through metal-catalyzed reactions (Liao et al. 2013; Modem et al. 2016; Rocha et al. 2017; Sarma et al. 2012). Remarkably, hydrothiolation reactions can also provide the corresponding products with high selectivity achieved by agro-waste extracts (Godoi et al. 2019) as well as under metal-free conditions (Chun et al. 2016; Rosa et al. 2017).

On the other hand, the development of new methodologies for the generation of new sulfur–nitrogen bonds has gained special attention and several transformations have been notably reported (Terent've et al. 2018; Yang et al. 2019). Among them, sulfur functionalization of nitrogen–nitrogen double bonds has received great interest in organic synthesis (Wen et al. 2015; Zhou and Xu 2016; Xu et al. 2018), since it allows the preparation of target molecules with unique synthetic and biological properties (Kamal et al. 2007; Li et al. 2013). Indeed, sulfenylhydrazines-1,2-dicarboxylates have shown versatile synthetic applications (Huang and Hu 2007), playing an important role as intermediate in the preparation of disulfides via thiol dimerization (Mukaiyama and Takahashi 1968) and likewise in the cyclization of olefinic carboxylic acids (An et al. 2018).

Due to sulfenylhydrazines-1,2-dicarboxylates relevance, these organosulfur compounds have been efficiently prepared since the pioneer work described by Linke (Fig. 1, Eq. 1) (Linke et al. 1973). More recently, Xu and co-workers reported a convenient method for the synthesis of sulfenylhydrazines-1,2-dicarboxylates under catalyst-free conditions (Fig. 1, Eq. 2) (Zhou et al. 2017). Although the previous protocols represent good advances on this research field,

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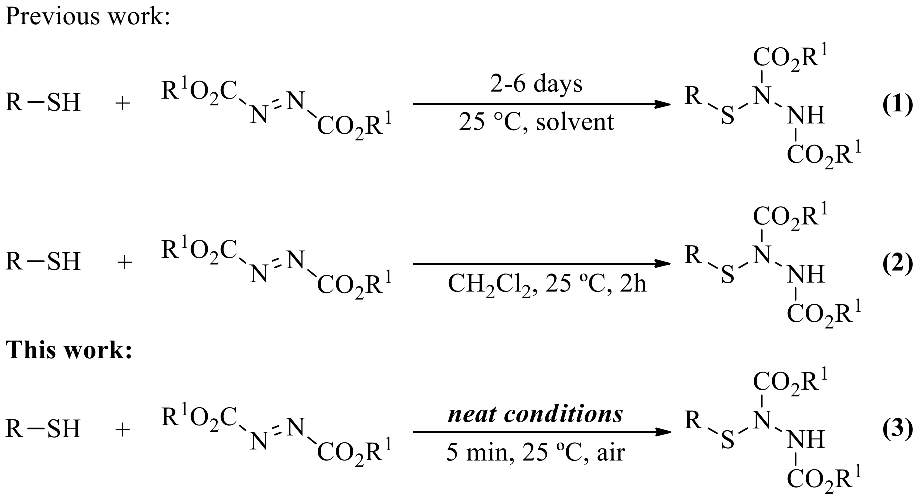


Fig. 1 Hydrothiolation of dialkyl azodicarboxylates

they still have some drawbacks, since long reaction times as well as toxic solvents are generally required. Moreover, the vast majority of the protocols for the monohydrothiolation processes have employed exclusively alkenes and/or alkynes as substrates. Therefore, the development of a mild and eco-friendly approach for the synthesis of sulfenylhydrazines-1,2-dicarboxylates is highly desirable.

In this context, neat reactions medium have become a particularly valuable and environmental benign alternative in organic synthesis, avoiding the use of harmful and carcinogenic solvents (Sarkar et al. 2016). In this regard, the development of new methods for the preparation of organosulfur compounds employing neat conditions has recently received considerable attention (Joshi et al. 2019; Mondal and Saha 2019). Despite the well-recognized versatility of neat transformations, the preparation of sulfenylhydrazines-1,2-dicarboxylates through a hydrothiolation reaction associated with neat conditions has not been reported to date. Thus, according to our interest into straightforward and eco-friendly methods, herein we have disclosed a rapid and suitable methodology for the synthesis of sulfenylhydrazines-1,2-dicarboxylates (Fig. 1, Eq. 3).

Experimental

General

Hydrogen nuclear magnetic resonance spectra (^{1}H NMR) were obtained at 400 MHz. Spectra were recorded in deuterated chloroform ($CDCl_3$) solutions. Data are reported as follows: chemical shift (δ), multiplicity, coupling constant (J) in Hertz and integrated intensity. Carbon-13 nuclear magnetic resonance spectra (^{13}C NMR) were obtained at 100 MHz. Spectra were recorded in $CDCl_3$ solutions. Chemical shifts are reported in ppm relative to the tetramethylsilane (TMS) for ^{1}H NMR and to the solvent for ^{13}C NMR. Electrospray ionization quadrupole time-of-flight mass spectrometry (ESI-Q-TOFMS) measurements performed with a micrOTOF Q-II (Bruker Daltonics) mass spectrometer equipped with an automatic syringe pump from KD Scientific for sample injection. The ESI-QTOF mass spectrometer was running at 4.5 kV at a desolvation temperature of 180 °C. The mass spectrometer was operating in the positive ion mode. Thin layer chromatography (TLC) performed using Merck Silica Gel GF254, 0.25 mm thickness. Generally, the reactions were monitored by TLC until disappearance of starting material. For visualization, TLC plates were either placed under ultraviolet light or stained with iodine vapor. The solvents were used without further purification.

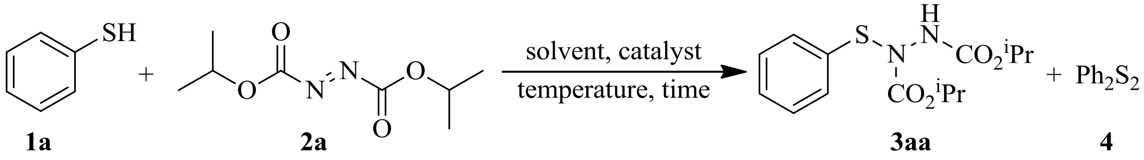
General procedure for the preparation of sulfenylhydrazines-1,2-dicarboxylates

Thiol **1** and dialkyl azodicarboxylate **2** were placed into a round bottom flask. The reaction was carried out under room temperature for 5 min. After the completion, the reaction system was then extracted with ethyl acetate and water (3 × 5 mL). The organic phase was dried over $MgSO_4$ and filtered, and the solvent was removed under reduced pressure. Purification by flash chromatography with a mixture of hexane/ethyl acetate (80:20) afforded the desired product.

Results and discussion

In order to establish the best reaction conditions, we employed diisopropyl azodicarboxylate and benzenethiol as standard substrates (Table 1). Firstly, the effect of the solvent on the reaction system has been evaluated (entries 1–4).

Table 1 Optimization of the reaction conditions



Entry	Solvent	Time (min)	Temperature (°C)	Catalyst (mol%)	Yield **3aa** (%)[a]	Yield **4** (%)[a]
1	H_2O	5	25	–	65	–
2	WEB	5	25	–	41	–
3	WEPAB	5	25	–	33	–
4	WERSA	5	25	–	59	–
5	–	**5**	**25**	–	**78**	–
6	–	8	25	–	72	–
7	–	2	25	–	52	–
8	–	5	0	–	55	–
9	–	5	50	–	55	42
10	–	5	100	–	59	38
11	–	5	25	nano-CuO (20)	78	–
12	–	5	25	nano-Fe_3O_4 (20)	44	–
13	–	5	25	NH_2SO_3H (20)	61	–
14[b]	–	5	25	–	59	–

Benzenethiol (0.5 mmol), diisopropyl azodicarboxylate (0.6 mmol), catalyst, solvent

[a] Yields for isolated products

[b] Under ultrasound irradiation

When the reaction was carried out using water as a solvent, the desired product was obtained in 65% yield (entry 1).

Next, we carried out some experiments employing well-known eco-friendly solvents such Water Extract of Banana peel ash (WEB), Water Extract of Papaya Bark (WEPAB) and Water Extract of Rice Straw Ash (WERSA). However, no improvement in the yield value of **3aa** was observed in the presence of these agro-waste aqueous extracts (entries 2–4). On the other hand, when the reaction has proceeded in the absence of any solvent the expected product was achieved in 78% yield (entry 5). Consequently, further experiments were conducted under neat conditions.

The influence of the time and temperature has been investigated in detail (entries 6–10). No significant change on the reaction yield was observed on increasing the time to 8 min (entry 6). However, a substantial decrease on the yield was observed by decreasing the reaction time from 5 to 2 min (entry 7). Similarly, the reaction showed to be very sensitive to any change on the temperature (entries 8–10).

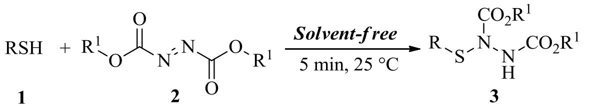
Furthermore, the formation of a significant amount of diphenyl disulfide **4** as a by-product was observed at higher temperatures (entries 9 and 10). It most probably explained due the high ability of azodicarboxylates to convert mercaptans into respective disulfides, accordingly to previous reports (Mukaiyama and Takahashi 1968).

Subsequently, we evaluated the influence of green catalysts on the reaction (entries 11–13). However, no improvement in the reaction yield was achieved in the presence of any catalyst. Finally, we performed an additional experiment employing ultrasound irradiation (entry 14). However, the conventional heating proved to be a better option when compared to this kind of energy source. Thus, the best reaction condition was found by carrying the reaction at 25 °C for 5 min in the absence of solvent, affording the desired product in 78% yield.

Having established the best reaction set, we have studied the scope and limitations of the methodology (Table 2). Initially, diisopropyl azodicarboxylate was treated with different aromatic thiols under the optimized conditions (entries 1–5). In terms of electronic effects, benzenethiol derivatives containing *para*-donating groups were more reactive than their withdrawing analogues, under the same conditions (entries 2–4). In particular, when *p*-methyl benzenethiol was used, the product **3ba** was achieved with 85% yield (entry 2).

We also attempted to employed aliphatic thiols under the same reaction conditions (entries 6–9). In this regard, benzyl mercaptan proves to be a suitable sulfur source, furnishing the respective product **3fa** in 62% yield (entry 6). Similarly, when 1-dodecanethiol was treated with diisopropyl azodicarboxylate, the desired product **3ga** was obtained in good yield (entry 7).

Table 2 Synthesis of several sulfenylhydrazines-1,2-dicarboxylates



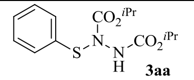
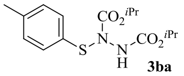
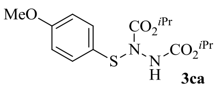
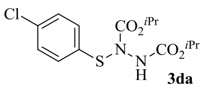
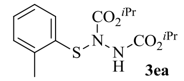
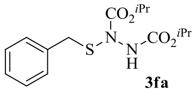
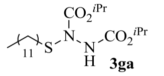
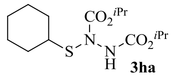
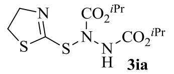
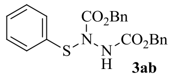
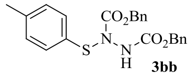
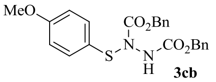
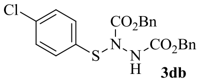
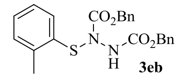
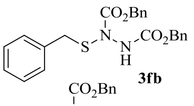
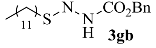
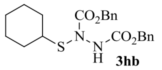
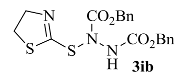
Entry	R	R^1	Product 3	Yield (%) [a]
1	Ph	i-C_3H_7	 **3aa**	78
2	p-MeC_6H_4	i-C_3H_7	 **3ba**	85
3	p-$MeOC_6H_4$	i-C_3H_7	 **3ca**	76
4	p-ClC_6H_4	i-C_3H_7	 **3da**	36
5	o-MeC_6H_4	i-C_3H_7	 **3ea**	60
6	Bn	i-C_3H_7	 **3fa**	62
7	n-$C_{12}H_{25}$	i-C_3H_7	 **3ga**	62
8	n-C_6H_{11}	i-C_3H_7	 **3ha**	52
9	n-C_3H_4NS	i-C_3H_7	 **3ia**	–
10	Ph	Bn	 **3ab**	78
11	p-MeC_6H_4	Bn	 **3bb**	54
12	p-$MeOC_6H_4$	Bn	 **3cb**	99
13	p-ClC_6H_4	Bn	 **3db**	69
14	o-MeC_6H_4	Bn	 **3eb**	70
15	Bn	Bn	 **3fb**	42
16	n-$C_{12}H_{25}$	Bn	 **3gb**	99
17	n-C_6H_{11}	Bn	 **3hb**	76
18	n-C_3H_4NS	Bn	 **3ib**	–

Table 2 (continued)

Reaction conditions: thiol (0.5 mmol), dialkyl azodicarboxylate (0.6 mmol), 5 min at 25 °C

[a] Yields for isolated products

Next, we evaluated whether the methodology could tolerate the modification in the R^1 group from azodicarboxylate. For this purpose, dibenzyl azodicarboxylate was reacted with a wide range of thiols (entries 10–18). Generally, dibenzyl azodicarboxylate reacts very smoothly with aromatic thiols containing both electron donating and electron withdrawing substituents attached to the *para* position of the aromatic ring, affording the corresponding products in moderate to excellent yields (entries 10–13). For instance, when *p*-methoxybenzenethiol was employed as a sulfur source, the product **3cb** was isolated with 99% yield (entry 12). In terms of steric effect, the reaction also well tolerated a methyl substituent at *ortho* position of the aromatic ring, affording the respective sulfenylhydrazine **3eb** in 70% yield (entry 14). Moreover, dibenzyl azodicarboxylate reacted very smoothly with aliphatic thiols, affording the expected products **3fb** and **3gb** in 42% and 99% yields, respectively (entries 15 and 16).

To get insights into the mechanisms of the reaction, a series of control experiments were also performed (Fig. 2). At first, we observed a decreased in the value of **3aa** when the reaction was carried out under inert atmosphere. Next, carrying the experiments under dark conditions, it has indicated that the reaction course is dependent of air and light combination. Further experiments were carried out employing different radical scavengers. However, no product formation was observed in the presence of radical inhibitors such as 2,2,6,6-tetramethyl-1-piperidinyloxyl (TEMPO) and 2,6-di-tert-butyl-4-methylphenol (BHT), even at extended reaction time. According to these experiments, the reaction most probably takes place through a radical pathway.

Based on our observations and in accordance with previous reports (Chun et al. 2016; Nakajima et al. 2019; Ryu et al. 2013; Zalesskiy et al. 2016), a plausible reaction pathway has been proposed for the hydrothiolation of dialkyl azodicarboxylates (Fig. 2). We believe that initially a thiyl

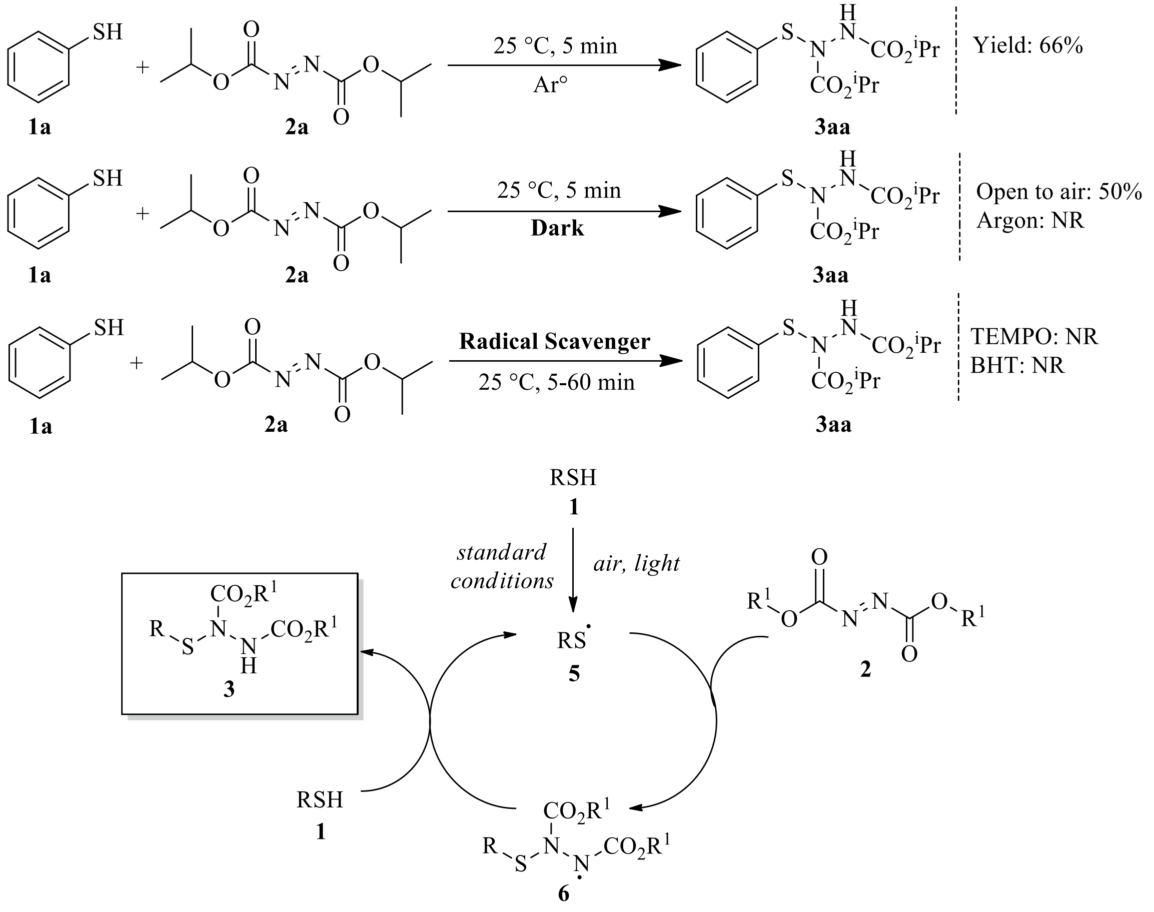


Fig. 2 Control experiments and plausible mechanism

radical **5** might be generated in the reaction media assisted by light and air oxygen (Bhat et al. 2015; Li et al. 2019). Subsequently, this specie would be smoothly converted into intermediate **6** through the reaction with dialkyl azodicarboxylate. Finally, this radical would react with another equivalent of thiol to form the corresponding product and concomitantly regeneration of the thiyl radical, completing the reaction pathway.

Conclusion

In summary, we have successfully developed an environmentally benign and efficient protocol for the preparation of sulfenylhydrazines-1,2-dicarboxylates, at room temperature, under ambient atmosphere (open flask) and solvent-free conditions. Generally, the synthetic method showed to be tolerant to aryl and alkyl thiols bearing electron donating and withdrawing groups affording the desired organosulfur compounds in high yields by spending very short reaction time via hydrothiolation of nitrogen–nitrogen double bonds. We believe that the chemistry described herein represents a new tendency for the sulfenylation of double bonds. Studies regarding the hydrothiolation of other organic compounds are still under investigation in our laboratory.

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