Synthesis Md. M. Rahman et al. Paper

Thioesterification and Selenoesterification of Amides via Selective N–C Cleavage at Room Temperature: N–C(O) to S/Se–C(O) Interconversion

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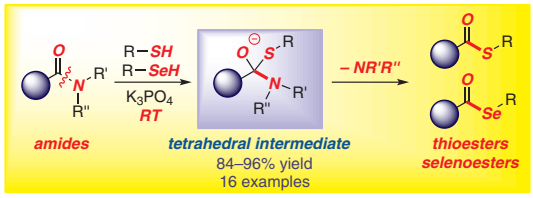

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Abstract The direct nucleophilic addition to amides represents an attractive methodology in organic synthesis that tackles amidic resonance by ground-state destabilization. This approach has been recently accomplished with carbon, nitrogen and oxygen nucleophiles. Herein, we report an exceedingly mild method for the direct thioesterification and selenoesterification of amides by selective N–C(O) bond cleavage in the absence of transition metals. Acyclic amides undergo N–C(O) to S/Se–C(O) interconversion to give the corresponding thioesters and selenoesters in excellent yields at room temperature via a tetrahedral intermediate pathway (cf. an acyl metal).

Key words amides, N–C activation, metal-free, thioesters, selenoesters, thioesterification, selenoesterification, tetrahedral intermediates

Amides represent an extremely important class of functional groups in chemistry.[1] As a result, the development of new methods for the direct interconversion of amides has been a field of intense study since the early days of organic synthesis.[2,3] The last five years have witnessed the establishment of powerful amide bond cross-coupling reactions by selective N–C(O) cleavage enabled by the amide bond twist and ground-state destabilization concept introduced by our group in 2015 (Figure 1A).[4–7] These reactions enable the use of amides in generic cross-coupling reactions of high synthetic value via acyl and aryl intermediates (after CO loss)[4b] using Pd, Ni, Rh, Co and Cr catalysis.[8] Of particular interest is the fact that acyl-type reactivity of amides is feasible in the absence of transition metals via a direct nucleophilic addition pathway involving tetrahedral intermediates (Figure 1B).[9] This mode of reactivity has been accomplished with carbon, nitrogen and oxygen nucleophiles,[10] and relies on the capacity of the amide bond to undergo ground-state destabilization to overcome amidic resonance (15–20 kcal/mol, $n_N \rightarrow \pi^*_{CO}$ conjugation).

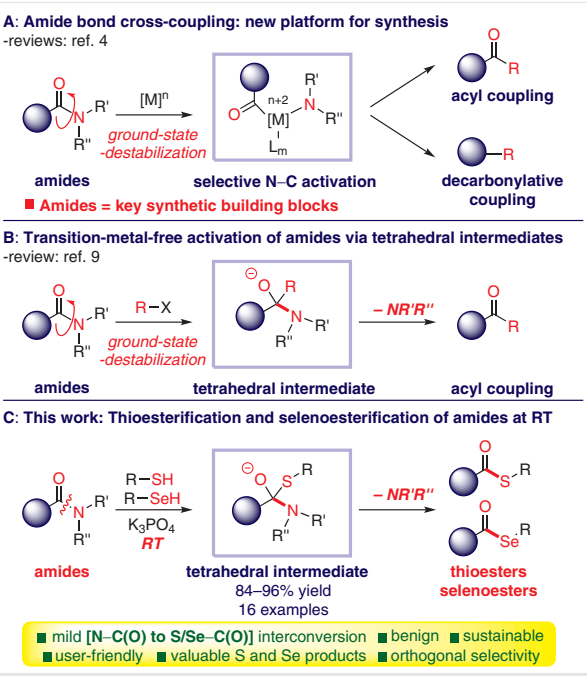

Figure 1 (A) Cross-coupling of amides. (B) Transition-metal-free activation of amides. (C) Thioesterification and selenoesterification of amides, N–C(O) to S/Se–C(O) interconversion at room temperature (this work).

In this article, we report an exceedingly mild method for direct thioesterification[11] and selenoesterification[12] of amides by selective N–C(O) bond cleavage in the absence of transition metals (Figure 1C). Acyclic amides undergo N–C(O) to S/Se–C(O) interconversion to give the corresponding thioesters and selenoesters in excellent yields at room temperature via a tetrahedral intermediate pathway (cf. an acyl metal). More broadly, this methodology enables rapid access to thioester and selenoester architectures from am-

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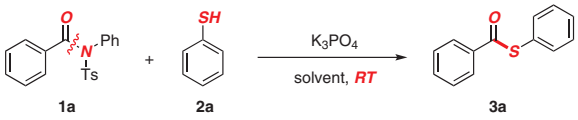
ides[13,14] and further highlights the advantages of this mild transition-metal-free tactic of amide bond functional group interconversion.

Transition-metal-free reactions of amides create new opportunities for organic synthesis by exploiting the tetrahedral intermediate avenue of ground-state destabilized amides. In this context, *N*-acyl-tosylamides [Ar = Ph, R = Ph, RE (resonance energy) = 9.7 kcal/mol; Winkler–Dunitz parameters, $\tau = 18.8°$; $\chi_N = 18.9°$][5a] have been established as broadly useful amides to achieve both (1) amide bond interconversion via direct metal insertion into the N–C(O) bond, and (2) amide bond interconversion via tetrahedral intermediates in the absence of transition metals.[4–9]

Encouraged by our results on transition-metal-free esterification and transamidation reactions of *N*-acyl-tosylamides,[9,10] we focused our efforts on identifying optimum conditions for mild, transition-metal-free thioesterification (Table 1). We were delighted to find that the reaction proceeded in excellent yield at room temperature using excess of K_3PO_4 as a base and close to a stoichiometric amount of thiophenol (Table 1, entry 1). Under these conditions, cleavage of the alternative amide bond was not observed, consistent with the high capacity of *N*-tosylamide to activate the acyclic amide bond. Furthermore, the amount of base could be decreased to 2.0 equivalents without deleterious effect (Table 1, entry 2); however, further decrease resulted in lower reaction efficiency (Table 1, entry 3). A brief solvent screen indicated THF to be the optimal solvent; however, reasonable efficiency was also observed in dioxane, CH_2Cl_2, toluene and MeCN (Table 1, entries 4–7). Finally, solvent concentration is an important parameter (Table 1, entries 8 and 9), and as expected in transition-metal-free interconversion of amides favors the acyl addition over the amide N-deprotection pathway.

With optimized conditions in hand, we next evaluated the scope of amides and thiols that participate in this mild N–C(O) to S–C(O) interconversion (Table 2). We were delighted to find that a broad range of amides was compatible with these mild conditions, including neutral (**3a**,**b**), electron-rich (**3c**), electron-deficient (**3d**), and sterically hindered (**3e**) amides. Pleasingly, the reaction was compatible with sensitive halide substituents such as bromo (**3f**), which would be problematic in transition-metal-catalyzed protocols. Furthermore, full chemoselectivity in the nucleophilic addition to the amide bond occurred in the presence of an alkyl ester (**3g**), indicating that N–Ts amides (RE = 9.7 kcal/mol) are the preferred addition site over methyl esters (C–O isomerization barrier, 12.8 kcal/mol). The reaction was also compatible with heterocycles (**3h**) and aliphatic amides (**3i**). Furthermore, we were pleased to find that electron-rich (**3j**) as well as more challenging electron-poor

Table 1 Optimization of the Thioesterification of Amides by Selective N–C(O) Bond Cleavage at Room Temperature[a]



1a 2a 3a

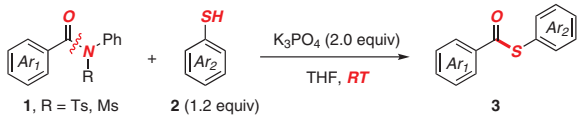
Entry	K_3PO_4 (equiv)	Solvent	Yield (%)[b]
1	3.0	THF	>98
2	2.0	THF	>98
3	1.5	THF	81
4	2.0	dioxane	86
5	2.0	CH_2Cl_2	91
6	2.0	toluene	95
7	2.0	MeCN	87
8[c]	2.0	THF	93
9[d]	2.0	THF	91

[a] Reaction conditions: amide (1.0 equiv), thiol (1.2 equiv), K_3PO_4 (1.5–3.0 equiv), solvent (0.25 M), 25 °C, 15 h.
[b] GC/^{1}H NMR yields.
[c] Concentration: 0.50 M.
[d] Concentration: 1.0 M.

benzenethiols (**3k**,**l**) underwent the desired addition in excellent yields. Moreover, this protocol could be applied to sterically hindered benzenethiols (**3m**), while less challenging aliphatic thiols (**3n**, *vide infra*) were also well tolerated. Finally, we were pleased to find that this protocol could be extended to N–Ms amides bearing an atom-economic *N*-mesyl activating group, without any decrease in the reaction efficiency (Table 2, entry 15). Note that *N*-alkyl amides, such as N–Me, are also amenable substrates. 2-Pyridylthiols have not been tested at this stage of reaction development. Future studies will address the use of disulfides under reductive conditions. Overall, this mild N–C(O) to S–C(O) interconversion method enables the preparation of a variety of valuable thioesters from amides. Given the plethora of metal-catalyzed and metal-free reactions of thioesters as well as the importance of thioesters in biochemical tagging, this mild process provides a useful alternative interconnecting amides with thioesters.[11,15]

To further demonstrate the synthetic potential of this approach, we applied this method to the synthesis of a selenoester using PhSeH via N–C(O) to Se–C(O) interconversion (Scheme 1). Since selenoesters represent an important class of acyl transfer reagents and radical precursors in organic synthesis, while recent studies have shown their utility in chemical ligation, our mild method permits the use of amides as selenoester precursors.[12,15]

Table 2 Substrate Scope of the Thioesterification of Amides by N–C Cleavage at Room Temperature[a]



1, R = Ts, Ms **2** (1.2 equiv) **3**

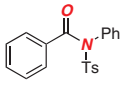
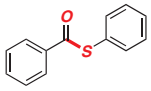
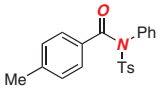
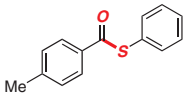
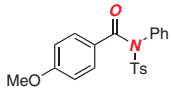
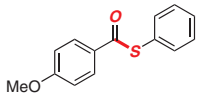
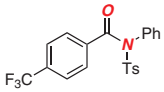
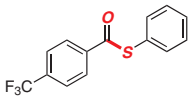
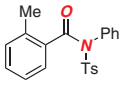
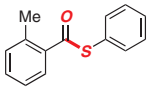
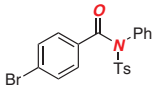
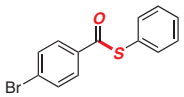
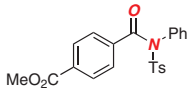
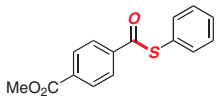
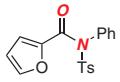
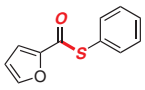
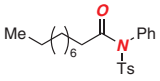
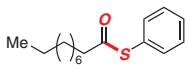
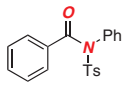
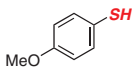
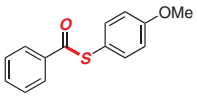
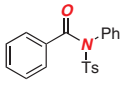
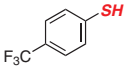
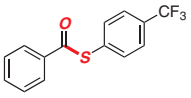
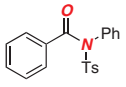
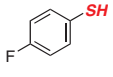
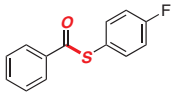
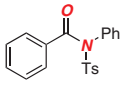
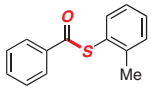
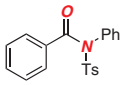
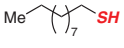
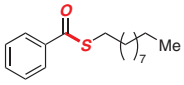
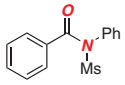
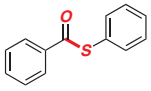
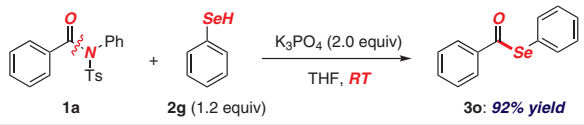
Entry	Amide (**1**)	Thiol (**2**)	Product (**3**)		Yield (%)[b]
1				**3a**	92
2				**3b**	93
3				**3c**	91
4				**3d**	94
5				**3e**	86
6				**3f**	90
7				**3g**	89
8				**3h**	88
9				**3i**	85
10				**3j**	94
11				**3k**	84
12				**3l**	86
13				**3m**	92

Table 2 (continued)

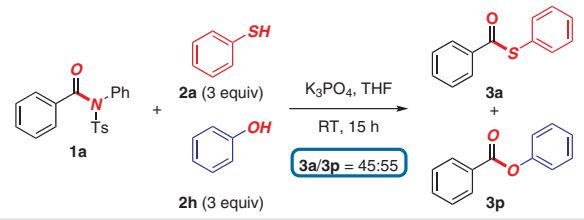
Entry	Amide (**1**)	Thiol (**2**)	Product (**3**)		Yield (%)[b]
14				**3n**	96
15				**3a**	92

[a] Reaction conditions: amide (1.0 equiv), thiol (1.2 equiv), K_3PO_4 (2.0 equiv), THF (0.25 M), 25 °C, 15 h.
[b] Yield of isolated product.

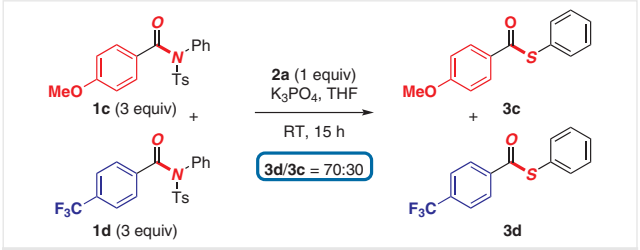
To gain preliminary insight on the reaction mechanism, we performed intermolecular competition experiments (Schemes 2–5). Most interestingly, intermolecular competition between thiophenol and phenol indicated similar reactivity (**3a**/**3p** = 45:55) (Scheme 2), which bodes well for future application of this [N–C(O) → S–C(O)] protocol in organic synthesis. Note that the ester is unreactive under the reaction conditions. Excess of reagents were used to stop the reaction at <30% conversion. Furthermore, electron-deficient amides are inherently more reactive (**3d**/**3c** = 70:30) (Scheme 3), which is consistent with the electrophilicity of the amide bond.



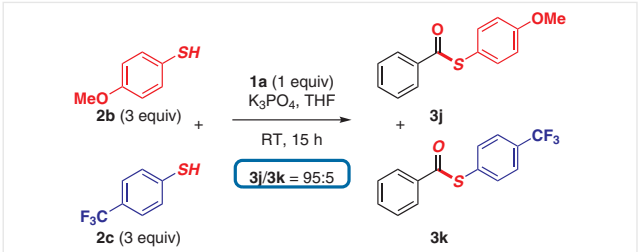
Scheme 1 Selenoesterification of an amide by N–C cleavage at room temperature



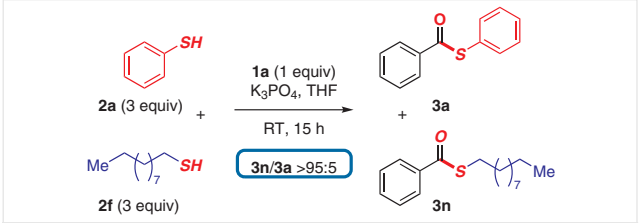
Scheme 2 Selectivity study on the thioesterification of amides at room temperature: thiophenol vs phenol



Scheme 3 Selectivity study on the thioesterification of amides at room temperature: electron-rich vs electron-deficient amides



Scheme 4 Selectivity study on the thioesterification of amides at room temperature: electron-rich vs electron-deficient thiols



Scheme 5 Selectivity study on the thioesterification of amides at room temperature: aromatic vs aliphatic thiol nucleophiles

Moreover, electron-rich thiophenols react preferentially (**3j**/**3k** = 95:5) (Scheme 4), while aliphatic thiols are significantly more reactive than aromatic thiols (**3n**/**3a** >95:5) (Scheme 5). Overall, these experiments are consistent with thiol nucleophilicity via a tetrahedral addition pathway.

In conclusion, we have reported a mild method for the direct thioesterification and selenoesterification of amides by selective N–C(O) bond cleavage. This protocol is highlighted by the absence of transition metals, operational simplicity and excellent reaction efficiency. It is worthwhile to point out that all amides used could be conveniently prepared from common secondary or primary amides.[4a–g] Our future studies will implement this transition-metal-free manifold in medicinal chemistry targets. More generally, this tactic accomplishes [N–C(O) → S/Se–C(O)] interconver-

sion to connect amides with thioesters and selenoesters. The tetrahedral intermediate pathway of ground-state destabilized amides is a serious alternative to acyl metals that should be considered as an alternative approach in all occasions when available.[4–9]

The starting materials reported in the manuscript have been previously described in the literature or are prepared by a previously reported method. All products have been previously reported, see the Supporting Information for details. All solvents were purchased at the highest commercial grade and used as received or after purification by passing through activated alumina columns or by distillation from Na/benzophenone. All solvents were deoxygenated prior to use. All other chemicals were purchased at the highest commercial grade and used as received. Reaction glassware was oven-dried at 140 °C for at least 24 h or flame-dried prior to use, allowed to cool under vacuum and purged with argon (three cycles). Flash chromatography was performed using SiliCycle silica gel (60 Å, 300 mesh). TLC analysis was carried out on Analtech glass plates coated with silica gel 60 F254 (0.2 mm thickness). The plates were visualized using a 254 nm ultraviolet lamp or aqueous potassium permanganate solution. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded in $CDCl_3$ on Bruker and Varian spectrometers at 500 MHz (^{1}H NMR), 125 MHz (^{13}C NMR) and 471 MHz (^{19}F NMR). All shifts are reported in parts per million (ppm) relative to the residual $CHCl_3$ resonances (7.27 and 77.2 ppm, ^{1}H NMR and ^{13}C NMR, respectively). All coupling constants (*J*) are reported in hertz (Hz); standard abbreviations are used for multiplicities. GC-MS chromatography was performed using an Agilent HP6890 GC System and an Agilent 5973A inert XL EI/CI MSD using helium as the carrier gas at a flow rate of 1 mL/min and an initial oven temperature of 50 °C. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra are given for all compounds in the Supporting Information for characterization purposes.

Thioesterification of Amides; General Procedure

An oven-dried reaction flask equipped with a stir bar was charged with an amide substrate (0.10 mmol, 1.0 equiv), thiol (0.12 mmol, 1.2 equiv) and K_3PO_4 (0.20 mmol, 2.0 equiv). THF (0.40 mL, 0.25 M) was added with vigorous stirring at room temperature, and the reaction mixture was stirred at room temperature for 15 h. The reaction mixture was then diluted with CH_2Cl_2 (3 mL), filtered and washed with water (3 mL). The aqueous layer was extracted with CH_2Cl_2 (2 × 3 mL) and the combined organic layers were dried, filtered, and concentrated. The sample was analyzed by ^{1}H NMR (500 MHz, $CDCl_3$) and GC-MS to obtain the conversion, yield and selectivity using an internal standard, and by comparison with authentic samples. Purification by chromatography on silica gel (EtOAc/hexanes) afforded the analytically pure product.

Selenoesterification of Amides; General Procedure

An oven-dried reaction flask equipped with a stir bar was charged with an amide substrate (0.10 mmol, 1.0 equiv), selenol (0.12 mmol, 1.2 equiv) and K_3PO_4 (0.20 mmol, 2.0 equiv). THF (0.40 mL, 0.25 M) was added with vigorous stirring at room temperature, and the reaction mixture was stirred at room temperature for 15 h. The reaction mixture was then diluted with CH_2Cl_2 (3 mL), filtered and washed with water (3 mL). The aqueous layer was extracted with CH_2Cl_2 (2 × 3 mL) and the combined organic layers were dried, filtered, and concentrated. The sample was analyzed by ^{1}H NMR (500 MHz, $CDCl_3$) and GC-MS to obtain the conversion, yield and selectivity using an internal standard, and by comparison with authentic samples. Purification by chromatography on silica gel (EtOAc/hexanes) afforded the analytically pure product.

S-Phenyl Benzothioate (3a)

White solid; yield: 19.7 mg (92%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.04 (d, *J* = 8.1 Hz, 2 H), 7.62 (t, *J* = 7.4 Hz, 1 H), 7.55–7.45 (m, 7 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 190.45, 137.00, 135.43, 133.98, 129.85, 129.59, 129.09, 127.82, 127.71.

S-Phenyl 4-Methylbenzothioate (3b)

White solid; yield: 21.2 mg (93%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.94 (d, *J* = 8.2 Hz, 2 H), 7.56–7.50 (m, 2 H), 7.48–7.43 (m, 3 H), 7.29 (d, *J* = 8.1 Hz, 2 H), 2.44 (s, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 190.02, 144.91, 135.46, 134.45, 129.75, 129.53, 127.91, 22.06.

S-Phenyl 4-Methoxybenzothioate (3c)

White solid; yield: 22.3 mg (91%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.04–7.99 (m, 2 H), 7.54–7.49 (m, 2 H), 7.48–7.43 (m, 3 H), 6.98–6.94 (m, 2 H), 3.89 (s, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 188.91, 164.34, 135.53, 130.05, 129.77, 129.70, 129.50, 128.01, 114.26, 55.89.

S-Phenyl 4-(Trifluoromethyl)benzothioate (3d)

White solid; yield: 26.6 mg (94%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.14 (d, *J* = 8.2 Hz, 2 H), 7.76 (d, *J* = 8.2 Hz, 2 H), 7.54–7.51 (m, 2 H), 7.50–7.47 (m, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 189.72, 139.79, 135.33, 135.27 (q, J_{C-F} = 32.9 Hz), 130.22, 129.76, 128.17, 126.91, 126.19 (q, J_{C-F} = 3.7 Hz), 123.84 (q, J_{C-F} = 273.3 Hz).

^{19}F NMR (471 MHz, $CDCl_3$): δ = –63.13.

S-Phenyl 2-Methylbenzothioate (3e)

Colorless oil; yield: 19.6 mg (86%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.95 (d, *J* = 7.7 Hz, 1 H), 7.54–7.51 (m, 2 H), 7.50–7.40 (m, 4 H), 7.34–7.26 (m, 2 H), 2.50 (s, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 192.50, 137.77, 137.13, 135.25, 132.34, 132.09, 129.80, 129.60, 128.97, 128.59, 126.20, 21.10.

S-Phenyl 4-Bromobenzothioate (3f)

White solid; yield: 26.4 mg (90%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.92–7.87 (m, 2 H), 7.66–7.62 (m, 2 H), 7.53–7.49 (m, 2 H), 7.48–7.46 (m, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 189.59, 135.77, 135.39, 132.41, 130.05, 129.68, 129.27, 129.09, 127.23.

Methyl 4-[(Phenylthio)carbonyl]benzoate (3g)

White solid; yield: 24.3 mg (89%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.15 (d, *J* = 8.5 Hz, 2 H), 8.08 (d, *J* = 8.5 Hz, 2 H), 7.54–7.50 (m, 2 H), 7.50–7.45 (m, 3 H), 3.97 (s, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 190.07, 166.42, 140.33, 135.35, 134.79, 130.33, 130.12, 129.73, 127.76, 127.19, 52.88.

S-Phenyl Furan-2-carbothioate (3h)

Colorless oil; yield: 17.9 mg (88%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.63 (d, *J* = 0.9 Hz, 1 H), 7.52–7.49 (m, 2 H), 7.47–7.43 (m, 3 H), 7.26 (dd, *J* = 3.6, 0.5 Hz, 1 H), 6.58 (dd, *J* = 3.5, 1.7 Hz, 1 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 178.95, 150.70, 146.79, 135.47, 129.98, 129.58, 126.53, 116.56, 112.75.

S-Phenyl Decanethioate (3i)

Colorless oil; yield: 22.5 mg (85%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.44–7.37 (m, 5 H), 2.65 (t, *J* = 7.5 Hz, 2 H), 1.74–1.68 (m, 2 H), 1.35–1.25 (m, 12 H), 0.89 (t, *J* = 6.9 Hz, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 197.91, 134.82, 129.61, 129.48, 128.35, 44.09, 32.19, 29.72, 29.60, 29.59, 29.31, 25.95, 23.01, 14.45.

S-(4-Methoxyphenyl) Benzothioate (3j)

White solid; yield: 23.1 mg (94%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.06–7.99 (m, 2 H), 7.60 (t, *J* = 7.4 Hz, 1 H), 7.48 (t, *J* = 7.7 Hz, 2 H), 7.42 (d, *J* = 8.8 Hz, 2 H), 6.99 (d, *J* = 8.8 Hz, 2 H), 3.85 (s, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 191.37, 161.15, 137.03, 136.97, 133.89, 129.05, 127.80, 118.25, 115.32, 55.73.

S-[4-(Trifluoromethyl)phenyl] Benzothioate (3k)

White solid; yield: 23.7 mg (84%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.03 (d, *J* = 7.4 Hz, 2 H), 7.71 (d, *J* = 8.2 Hz, 2 H), 7.67–7.62 (m, 3 H), 7.51 (t, *J* = 7.8 Hz, 2 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 189.24, 136.60, 135.56, 134.39, 132.54, 131.81 (q, J_{C-F} = 32.9 Hz), 129.24, 127.92, 126.34 (q, J_{C-F} = 3.7 Hz), 124.17 (q, J_{C-F} = 274.6 Hz).

^{19}F NMR (471 MHz, $CDCl_3$): δ = –62.85.

S-(4-Fluorophenyl) Benzothioate (3l)

White solid; yield: 20.1 mg (86%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.05–7.99 (m, 2 H), 7.62 (t, *J* = 7.4 Hz, 1 H), 7.52–7.47 (m, 4 H), 7.20–7.12 (m, 2 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 190.44, 164.00 (d, J_{C-F} = 250.6 Hz), 137.50 (d, J_{C-F} = 8.6 Hz), 136.77, 134.14, 129.15, 127.85, 122.98 (d, J_{C-F} = 3.5 Hz), 116.89 (d, J_{C-F} = 22.0 Hz).

^{19}F NMR (471 MHz, $CDCl_3$): δ = –111.09.

S-(*o*-Tolyl) Benzothioate (3m)

Colorless oil; yield: 21.0 mg (92%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.10–8.02 (m, 2 H), 7.62 (t, *J* = 7.4 Hz, 1 H), 7.52–7.48 (m, 3 H), 7.41–7.35 (m, 2 H), 7.30–7.26 (m, 1 H), 2.41 (s, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 189.96, 143.00, 137.16, 136.75, 133.90, 131.17, 130.56, 129.07, 127.89, 127.16, 127.01, 21.15.

S-Decyl Benzothioate (3n)

Yellow oil; yield: 26.6 mg (96%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.01–7.93 (m, 2 H), 7.55 (t, *J* = 7.4 Hz, 1 H), 7.44 (t, *J* = 7.7 Hz, 2 H), 3.07 (t, *J* = 7.4 Hz, 2 H), 1.71–1.64 (m, 2 H), 1.46–1.39 (m, 2 H), 1.35–1.24 (m, 12 H), 0.88 (t, *J* = 6.9 Hz, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 192.41, 137.63, 133.48, 128.85, 127.50, 32.22, 29.90, 29.88, 29.84, 29.63, 29.50, 29.39, 29.28, 23.01, 14.43.

Se-Phenyl Benzoselenoate (3o)

Yellow oil; yield: 24.0 mg (92%).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.98–7.91 (m, 2 H), 7.65–7.58 (m, 3 H), 7.50 (t, *J* = 7.8 Hz, 2 H), 7.46–7.41 (m, 3 H).

^{13}C NMR (126 MHz, $CDCl_3$): δ = 193.72, 138.89, 136.68, 134.23, 129.72, 129.41, 129.29, 127.69, 126.14.

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Supporting Information

Supporting information for this article is available online at https://doi.org/10.1055/s-0039-1690055.

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