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TMSCl-Catalyzed Electrophilic Thiocyano Oxyfunctionalization of Alkenes Using *N*-Thiocyano-dibenzenesulfonimide

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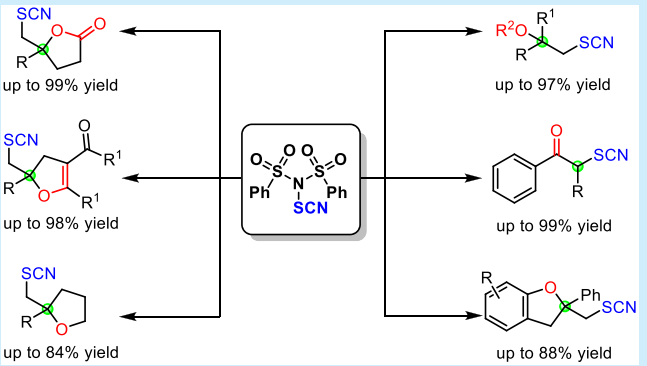
Supporting Information

ABSTRACT: Numerous electrophilic thiocyano oxyfunctionalization reactions of alkenes have been achieved using *N*-thiocyano-dibenzenesulfonimide, which is a new electrophilic thiocyanation reagent and could be easily prepared in two steps from dibenzenesulfonimide. This approach provides efficient, simple, and modular methods for the formation of SCN-containing heterocycles such as lactones, tetrahydrofurans, dihydrofurans, and dihydrobenzofurans in moderate to excellent yields. Meanwhile, diverse oxa-quaternary centers were rapidly constructed. Additionally, this protocol is free of transition metals and features broad substrate toleraance and mild reaction conditions.



Organosulfur compounds widely exist in biologically active natural products, drug molecules, and functional materials.[1] Among them, thiocyanates not only are present in bioactive compounds such as HDAC inhibitor psammaplin B and fasicularin exhibiting cytotoxic activity but also are versatile and common building blocks due to their easy access to trifluoromethylthiol compounds, thioethers, thiotetrazole, etc. (Figure 1).[2,3] Consequently, tremendous effort has been

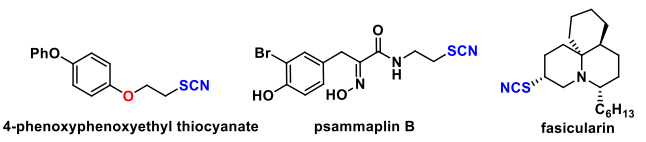


Figure 1. Representative examples of SCN-containing bioactive compounds.

dedicated to developing practical, simple, and highly efficient methods for the introduction of the SCN group into organic motifs.[4,5] Traditionally, cyanations of thiols or thioethers are indirect and limited routes for the construction of SCN-containing compounds.[6] Recently, some radical thiocyano functionalizations of alkenes have been described, which offer a direct and attractive method to thiocyanates.[7] For example, the Guo group reported a radical thiocyanooxygenation of alkenes for the synthesis of SCN-containing heterocycles using $K_2S_2O_8$ as the oxidant.[7a,b] The Bolm group documented a photocatalytic radical thiocyano functionalization of vinyl arenes using *N*-SCN sulfoximines as the thiocyano source.[7e] Despite these advances, radical thiocyano functionalizations of alkenes are still limited in substrate scope and have the disadvantage of using an excess amount of oxidant. For instance, radical thiocyano lactonization of 2-(1-phenylvinyl)benzoic acid and its derivatives proceeded smoothly and gave excellent yields. However, hardly any reaction could be observed using 4-phenylpent-4-enoic acid **2a** as the substrate under radical conditions (Scheme 1a).[7b] Thus, the development of complementary and new approaches to the synthesis of SCN-containing compounds is still highly desirable.

Electrophilic functionalization of alkenes has proven to be an efficient, widely applicable, and remarkable method for the preparation of functionalized compounds. During the past several years, electrophilic halogenation,[8] trifluoromethylthiolation,[9] and sulfenylation[10] of alkenes have been widely investigated. To the best of our knowledge, electrophilic thiocyano functionalization of alkenes has not been achieved, although some electrophilic thiocyanations of arenes and *β*-keto carbonyl compounds have been reported by the Chen group and others.[11] Herein, we introduce our preliminary results for a series of electrophilic thiocyano oxyfunctionalization reactions of alkenes using a new thiocyanation reagent, which is part of our ongoing study of the efficient synthesis of organosulfur compounds (Scheme 1b).[12]

Our investigation began with the synthesis of *N*-thiocyano-dibenzenesulfonimide **1e**. As shown in Figure 2, some electrophilic thiocyanation reagents have been developed; for example, *N*-thiocyanatosaccharin (**1d**) was reported by the Yin and Chen groups in 2018.[11d] Compared with electrophilic SCF_3 reagents,[13] thiocyanation reagents are relatively scarce. In

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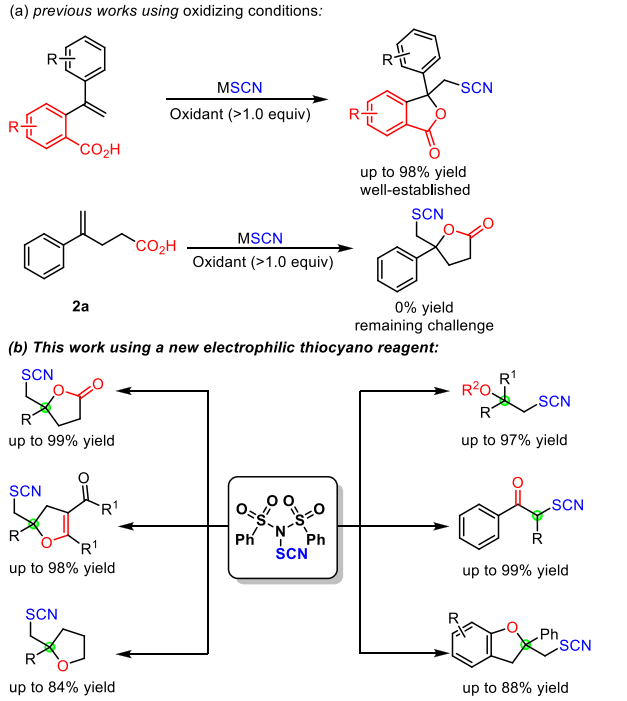
Org. Lett. XXXX, XXX, XXX−XXX

Scheme 1. Design of Electrophilic Thiocyano Oxyfunctionalization of Alkenes



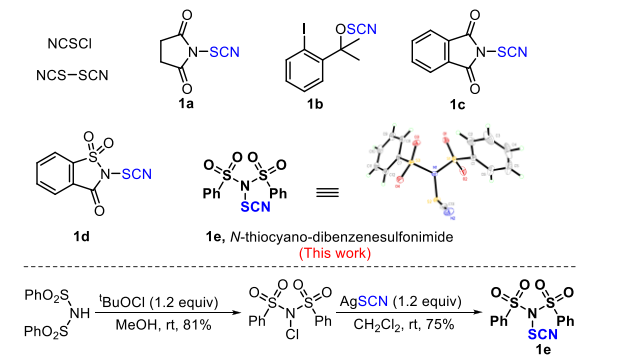
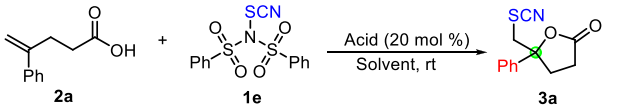


Figure 2. Preparation of *N*-thiocyano-dibenzenesulfonimide.

light of the strong electrophilicity of *N*-SCF_3-dibenzenesulfonimide developed by Shen and Zhao,[9f,13] we synthesized *N*-thiocyano-dibenzenesulfonimide and examined its reactivity. To our delight, **1e** was easily prepared in two steps from commercially available dibenzenesulfonimide in 61% total yield. *N*-Chloro-dibenzenesulfonimide was prepared according to previous procedures,[13] which was subsequently performed with AgSCN (1.2 equiv) in CH_2Cl_2 to produce **1e** as a white solid in 75% yield. It should be noted that the configuration of **1e** was confirmed by X-ray crystallography.[14]

Using **1e** developed above, we first selected 4-phenylpent-4-enoic acid **2a** as a model substrate to evaluate thiocyano lactonization (Table 1). To our delight, **2a** was facilely converted to desired product **3a** in 91% yield using acetyl chloride as an acid catalyst in dichloromethane and at room temperature (entry 1). Meanwhile, we also tested some other solvents such as acetonitrile, toluene, and dioxane, but no better results were obtained (entries 2–4, respectively). Next, the use of trimethylchlorosilane instead of acetyl chloride further improved the yield to 98% (entry 5). It should be noted that a Brønsted acid also can catalyze this reaction.

Table 1. Optimization of Conditions[a]



entry	solvent	acid	yield[b] (%)
1	CH_2Cl_2	AcCl	91
2	MeCN	AcCl	85
3	toluene	AcCl	not determined
4	dioxane	AcCl	not determined
5	CH_2Cl_2	TMSCl	98
6	CH_2Cl_2	MsOH	87
7[c]	CH_2Cl_2	TMSCl	97
8[d]	CH_2Cl_2	–	87

[a]Reaction conditions: **2a** (0.2 mmol), **1e** (0.24 mmol), acid (0.04 mmol) in solvent (2 mL) stirred at 25 °C for 10–20 min under Ar. [b]Isolated yield. [c]TEMPO (0.2 mmol) was added. [d]The reaction time is 5 h.

Using methanesulfonic acid gave an 87% yield (entry 6). Next, we added radical scavenger TEMPO to the system, and the desired product was produced without a decrease in yield, which could exclude the radical process (entry 7). Finally, the reaction was conducted without an acid catalyst. It was found that the lactonization process is relatively slow (5 h) and delivered the desired product in 87% yield (entry 8).

With the optimal reaction conditions in hand (Table 1, entry 5), the scope was tested with various olefinic acids (Scheme 2). In general, the desired SCN-containing lactones were obtained in moderate to excellent yields. Compared to the model substrate (**2a**), the methyl group at the *meta* or *para* position of the phenyl ring did not have an evident influence on the yield (**3c** or **3d**, respectively), whereas the *ortho* substitution obviously decreased the yield maybe due to steric hindrance (**3b**). The electronic effect of substituents was also investigated. It was found that substrates with electron-poor or electron-rich aryl rings gave relatively lower yields (**3h** and **3i**). It was found that multisubstituted phenyl acids were also suitable for this reaction producing the desired lactone products in good yields (**3j** and **3k**). It should be noted that thiocyanations of arene and alkene were simultaneously achieved using 2,4-dimethoxy-substituted substrate **2k**, affording product **3k** in 83% yield using 2.0 equiv of **1e**. Furthermore, the heteroaromatic ring and phenylacetylene substrates also performed well and gave the corresponding products in moderate yields (**3l** and **3m**). To our satisfaction, unbiased alkyl-substituted alkene substrates **2n** and terminal alkene **2o** were incorporated well and delivered products **3n** and **3o** in 85% and 84% yields, respectively. It was found that 2-(1-phenylvinyl)benzoic acid and its derivatives were also tolerable in this transformation, delivering the corresponding products in nearly quantitative yield (**3p** and **3q**). Subsequently, 5-phenylhex-5-enoic acid **2r** was also subjected to the reaction to produce SCN-containing *δ*-valerolactone **3r** in 83% yield using AcCl as the catalyst.

Internal alkene acids were also tested. Desired cyclization product **3s** was obtained in 78% yield using (*E*)-5-phenylpent-4-enoic acid **1s** as a substrate, whereas desired product **3t** was delivered in only 23% yield using trisubstituted alkene acid **1t**. To demonstrate the practicability of this method, a gram-scale reaction of **2a** was performed under the standard conditions and desired product **3a** was readily obtained in 84% yield.

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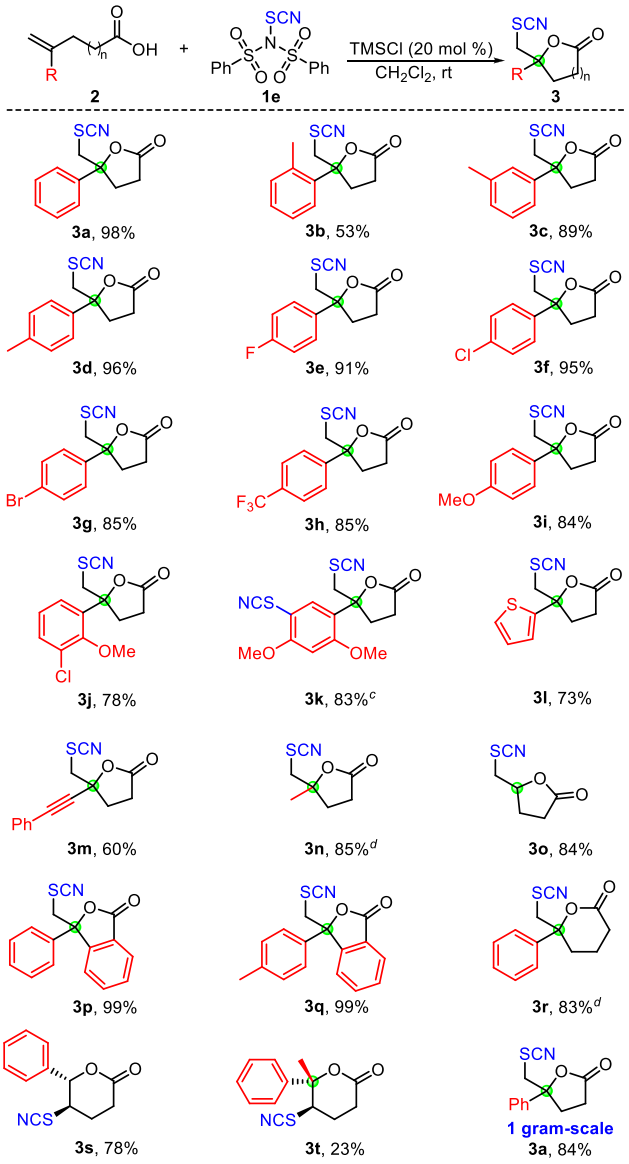
Scheme 2. Scope of Olefinic Acids[a,b]

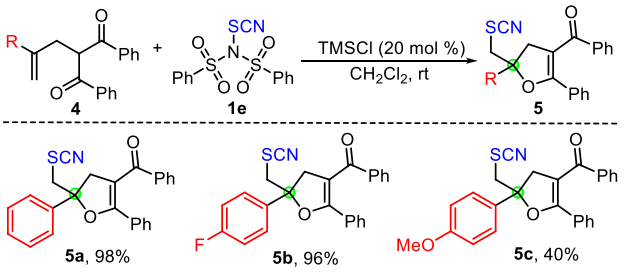


[a]Reaction conditions: olefinic acid **2** (0.2 mmol), **1e** (0.24 mmol), TMSCl (0.04 mmol) in CH_2Cl_2 (2 mL) stirred at 25 °C for 10−20 min under Ar. [b]Isolated yield. [c]With 0.4 mmol of **1e** added. [d]AcCl was used instead of TMSCl.

After establishing the lactonization of alkenes using an acid group as a nucleophile, we wondered whether other groups such as ketones and alcohols can be used as nucleophiles. Considering compounds containing the dihydrofuran framework also exhibit many valuable biological activities, some olefinic 1,3-diketone compounds were subjected to the standard conditions as the substrates, which can furnish SCN-containing dihydrofurans (Scheme 3). To our delight, the reaction proceeded smoothly, forming the desired dihydrofuran products in moderate to excellent yields.

There is no example of thiocyano etherification of alkene alcohols, to the best of our knowledge. Inspired by the results presented above, we sought to develop electrophilic thiocyano etherification of alkene alcohols for the synthesis of SCN-containing tetrahydrofurans. It was found that acetonitrile is a suitable solvent for this transformation. As shown in Scheme 4,

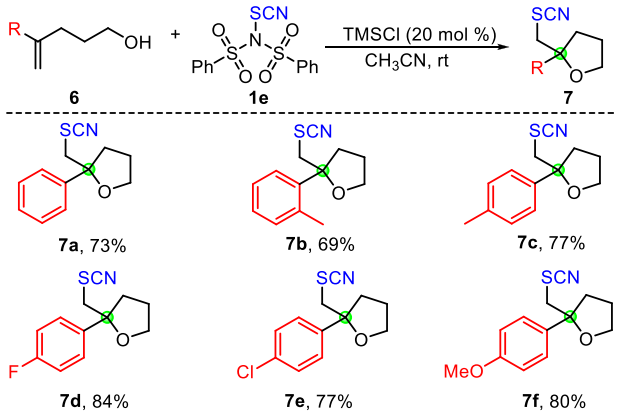
Scheme 3. Scope of Olefinic 1,3-Diketone Compounds[a,b]



[a]Reaction conditions: olefinic 1,3-diketone compound **4** (0.2 mmol), **1e** (0.24 mmol), TMSCl (0.04 mmol) in CH_2Cl_2 (2 mL) stirred at 25 °C for 10−20 min under Ar. [b]Isolated yield.

the substrates with a methyl group at different positions as well as different electronic substituents at the *para* position gave the desired products in good yields.

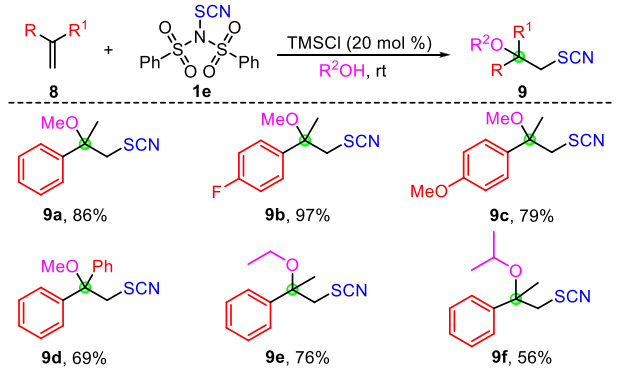
Scheme 4. Scope of Alkene Alcohols[a,b]



[a]Reaction conditions: alkene alcohol **6** (0.2 mmol), **1e** (0.24 mmol), TMSCl (0.04 mmol) in CH_3CN (2 mL) stirred at 25 °C for 10−20 min under Ar. [b]Isolated yield.

To further examine the general utility of the reagent, intermolecular thiocyano oxygenation of alkenes was also investigated (Scheme 5). First, prop-1-en-2-ylbenzene **8a** was

Scheme 5. Scope of Intermolecular Thiocyano Oxygenation of Alkenes[a,b]



[a]Reaction conditions: alkene **8** (0.2 mmol), **1e** (0.24 mmol), TMSCl (0.04 mmol) in R^2OH (2 mL) stirred at 25 °C for 10−20 min under Ar. [b]Isolated yield.

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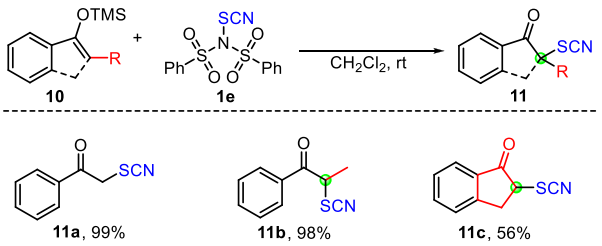
Org. Lett. XXXX, XXX, XXX−XXX

chosen as a model substrate with **1e**, and methanol was chosen as the solvent and nucleophile at room temperature. Fortunately, desired product **9a** was obtained in 86% yield. Subsequently, a number of different 1,1-disubstituted alkenes were tested using methanol as the solvent and nucleophile, which always furnished the products in good to excellent yields. As expected, the yield obviously decreased when the more sterically hindering isopropanol was used.

We also carried out the reaction with silyl enol ethers for the construction of α-SCN ketones. To our satisfaction, the linear substrates reacted very well, producing the products in quantitative yield. However, cyclic silyl enol ether **10c** gave an only 56% yield (Scheme 6). It is worth mentioning that an acid initiator is not integral in this system.

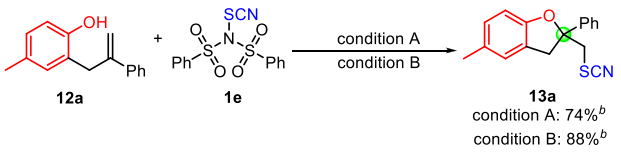
Scheme 6. Scope of Silyl Enol Ethers[a,b]



[a]Reaction conditions: alkene **8** (0.2 mmol), **1e** (0.24 mmol), in CH_2Cl_2 (2 mL) stirred at 25 °C for 10−20 min under Ar. [b]Isolated yield.

To further extend the application range of **1e** and synthesize diverse SCN-containing heterocycles, we studied the cyclization of olefinic phenols with **1e**, which could enable the rapid preparation of useful dihydrobenzofuran frameworks (Scheme 7). Considering that electrophilic thiocyanation may occur at

Scheme 7. Preliminary Studies of Cyclization of Olefinic Phenol[a,b]

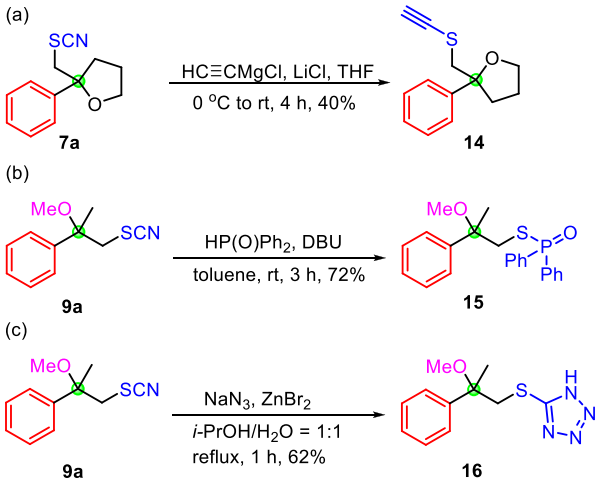


[a]Reaction condition A: **12a** (0.2 mmol), **1e** (0.24 mmol), TMSCl (0.04 mmol), in CH_2Cl_2 (2 mL) stirred at 25 °C for 10−20 min under Ar. Reaction condition B: **12a** (0.2 mmol), **1e** (0.24 mmol), AcCl (0.04 mmol), in CH_3CN (2 mL) stirred at 25 °C for 10−20 min under Ar. [b]Isolated yield.

the *para* position of phenol, compound **12a** with a 4-Me-phenol motif was used as a model substrate. It was found that both TMSCl and AcCl can be used as acid initiators, and product **13a** was obtained in a higher yield (88%) using AcCl as the acid promoter.

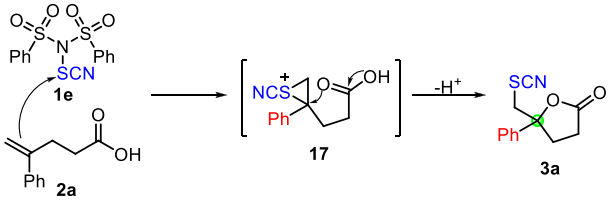
Ultimately, the synthetic utilities of products **7a** and **9a** have been achieved. As indicated in Scheme 8, treatment of **7a** with acetylenylmagnesium chloride and LiCl in THF delivered thioalkyne **14** in moderate yield. Next, nucleophilic substitution of diphenylphosphine oxide on compound **9a** gave broad potential phosphonothioate **15** in 72% yield. Additionally, [3+2] cycloaddition of **9a** with sodium azide was also performed, affording desired thiotetrazole **16** in 62% yield.

Scheme 8. Synthetic Applications



A plausible mechanistic pathway for the thiocyano lactonization is proposed in Scheme 9. In the presence of an

Scheme 9. Proposed Mechanism for Electrophilic Thiocyano Lactonization of Alkene Acid



acid catalyst, a thiiranium ion intermediate **17** is easily formed. Subsequently, intermediate **17** is attacked by a nucleophilic carboxylic acid group to generate desired lactone **3**. Due to the strong electrophilicity of **1d**, the thiiranium ion intermediate could be formed with activated alkenes.

In summary, *N*-thiocyano-dibenzenesulfonimide, a new electrophilic thiocyanation reagent, has been designed and readily prepared. A variety of practical and efficient electrophilic thiocyano oxyfunctionalizations of alkenes have been successfully developed by using *N*-thiocyano-dibenzenesulfonimide. These transformations are valuable because various SCN-containing compounds were obtained in moderate to excellent yields and an oxa-quaternary center was efficiently constructed under simple and mild conditions. Further studies of the development of asymmetric electrophilic thiocyanations of alkenes are underway.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b01706.

Experimental details, X-ray crystallography structure of compound **1e**, analytical data for new compounds, and NMR spectra of new compounds (PDF)

Accession Codes

CCDC 1909782 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing

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Org. Lett. XXXX, XXX, XXX−XXX

data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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(14) CCDC 1909782 (**1e**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

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