Organic Letters

Hexafluoroisopropanol-Promoted Disulfidation and Diselenation of Alkyne, Alkene, and Allene

Chiyu Wei, Ying He, Jin Wang, Xiaohan Ye, Lukasz Wojtas, and Xiaodong Shi*

Cite This: https://dx.doi.org/10.1021/acs.orglett.0c01834 Read Online

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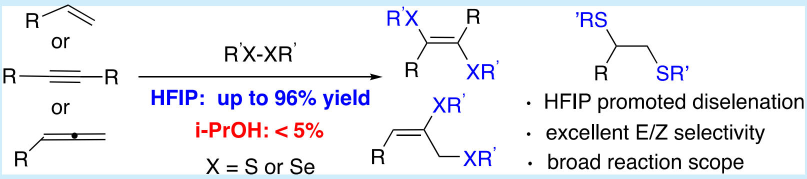
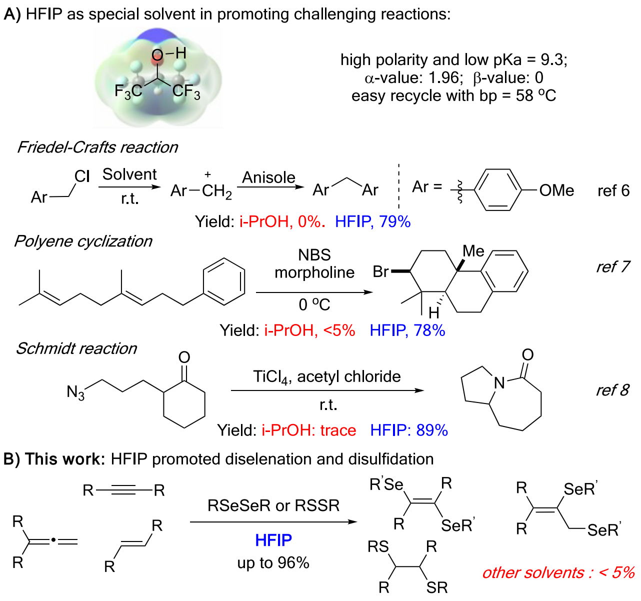


ABSTRACT: Hexafluoroisopropanol (HFIP)-promoted disulfidation and diselenation of C−C unsaturated bonds is reported. Reactions of unactivated alkyne, alkene, and allene, respectively, with disulfides or diselenides in HFIP led to desired products in good to excellent yields (up to 96%). In contrast, other solvents, such as isopropanol and dichloroethane, could not promote the same reaction. This method revealed an example of HFIP-promoted transformations under the mild conditions, which greatly highlighted the unique reactivity of this special solvent.

One basic mission of synthetic chemistry is to develop new methods for effective transformations, achieving a complex molecular scaffold from simple starting materials under mild conditions.[1] As one of the crucial reaction tuning factors, solvent could dramatically impact the reaction performance. With the discovery of ionic liquid and fluorinated solvents, unexpected results were obtained, greatly enriching the synthetic toolbox.[2] Fluorinated compounds have received tremendous attention in chemical, medicinal, and material research.[3] The high electronegativity and good hydrophobicity of the fluorine atom give fluorinated compounds unique chemical properties. Recently, HFIP has emerged as a unique polar organic solvent in promoting chemical reactions.[4] With two acidic protons, low nucleophilicity, and high ionizing power, HFIP has been used as an alternative solvent in substituting Lewis acid or Brønsted acid in promoting chemical transformations through H-bond networks under very mild conditions. In addition, HFIP is a promising solvent in industrial processing, owing to its recyclability and low cost.[5] Some representative HFIP-promoted transformations, including the Friedel−Crafts reaction,[6] polyene cyclization,[7] and Schmidt reaction,[8] are shown in Scheme 1A. In comparison with other solvents which failed to produce the desired products, HFIP significantly promoted the reaction to obtain good to excellent yields. These results greatly highlighted the unique reactivity of HFIP in facilitating organic transformations under mild conditions.

Recently, we reported thioallylation and diselenation of alkynes under gold redox catalysis.[9] Based on the solvent screening, we observed that HFIP could greatly promote the reaction of even more challenging unactivated internal alkyne without gold catalysts. Herein, we report this interesting solvent effect of HFIP on diselenation and disulfidation of the C−C unsaturated bond. Good to excellent yields were

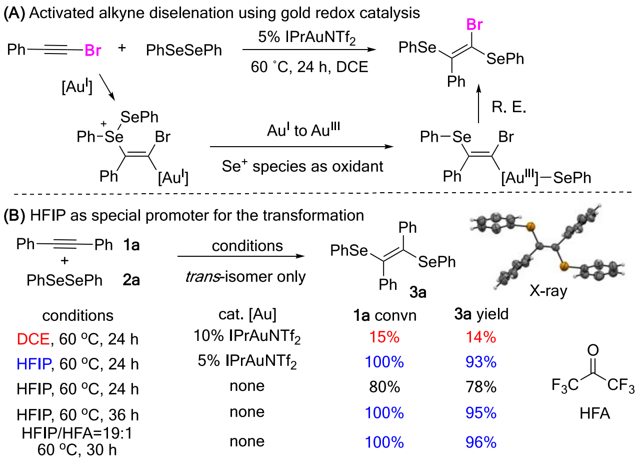
Scheme 1. HFIP-Promoted Challenging Transformations



Received: May 31, 2020

achieved with broad reaction substrate scope under metal-free conditions (Scheme 1B), which demonstrated the unique reactivity of the HFIP solvent in promoting challenging organic transformations.

Disulfidation[10] and diselenation[11] of C−C unsaturated bonds are the basic organic transformations with many chemical and medicinal applications. Great progress has been made regarding these transformations by using transition metal promoters/catalysts, Lewis acid, and photocatalysts to achieve the desired products. Our interest in this reaction was originated from our continuous efforts in gold-catalyzed alkyne activation.[12] As shown in Figure 1A, we recently reported the diselenation of alkynes through a gold(I)−gold(III) redox catalytic cycle, giving diselenation products in excellent yields with high stereoselectivity (trans-addition only). However, this transformation did not work well on simple internal alkynes, even with an increased catalyst loading (10% [Au]) at 60 °C.

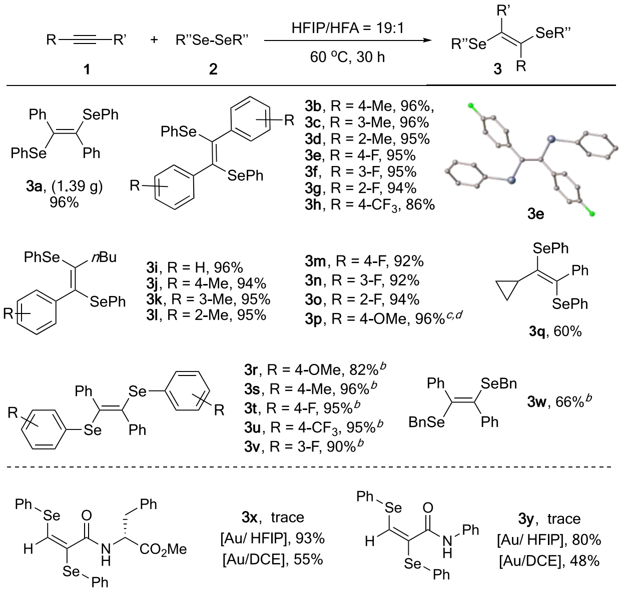


conditions	cat. [Au]	**1a** convn	**3a** yield
DCE, 60 °C, 24 h	10% IPrAuNTf$_2$	15%	14%
HFIP, 60 °C, 24 h	5% IPrAuNTf$_2$	100%	93%
HFIP, 60 °C, 24 h	none	80%	78%
HFIP, 60 °C, 36 h	none	100%	95%
HFIP/HFA=19:1 60 °C, 30 h	none	100%	96%

Figure 1. HFIP-promoted internal alkyne diselenation.

As shown in Figure 1B, treating internal alkyne **1a** with diselenide **2a** under previous gold catalytic conditions (DCE, 60 °C, 24 h) gave diselenation product **3a** in poor conversion (15%) and low yield (14%) associated with completed gold catalyst decomposition. Interestingly, switching the solvent to HFIP greatly enhanced the reaction performance, giving **3a** in nearly quantitative yield (93%). A control reaction using HFIP as solvent without gold catalyst was then carried out, achieving **3a** in 78% yield with 80% conversion of **1a** in 24 h. Extending the reaction time to 36 h gave a full conversion of **1a**, and the desired product **3a** was obtained in excellent yield (95%). HFIP is necessary to be a solvent (0.2 M). When applying a mixture of DCE/HFIP (9:1), only trace amount of product was obtained. In addition, applying hexafluoroacetone (HFA) as additive could accelerate the reaction with comparable yield in 30 h (for detailed screening of the optimal ratio of HFIP/HFA, see Table S1). Other solvents, including iPrOH, DMSO, DMF, CH_3CN, acetone, chlorobenzene, and nitromethane, could not promote this reaction, resulting in less than 5% yield in all cases. Lowering the temperature to 40 °C led to a decrease of conversion due to slower reaction kinetics. The detailed screening conditions are summarized in the Supporting Information. Notably, only *trans*-isomer **3a** was observed under these metal-free conditions, suggesting a concerted bimolecular *trans*-addition of diselenides to the plausible selenium cation intermediate formed from alkyne addition toward HFIP-activated diselenides. Encouraged by these results, we evaluated the reaction scope of this metal-free HFIP promoted transformations. The results are summarized in Scheme 2.

Scheme 2. HFIP-Promoted Alkyne Diselenation[a]



[a]Reaction conditions: alkyne (0.4 mmol) and diselenide (0.2 mmol) were added into HFIP/HFA = 19:1 (1 mL), and the mixture was stirred at 60 °C for 24 h. Isolated yield. [b]3 days. [c]Under room temperature. [d]12 h.

Various substituted alkynes were evaluated in this HFIP "boosted" transformation. First, several diaryl acetylenes with different substituents on the aromatic rings were tested. Both electron-donating group (EDG) and electron-withdrawing group (EWG) substituted aromatic rings worked well in this reaction, giving the desired products (**3a**−**3h**) in excellent yields. Second, aliphatic alkynes (**3i**−**3q**) gave similar results with the desired products obtained mostly in excellent yields. The cyclopropyl-substituted alkyne (**3q**) gave no ring-opening product, ruling out the potential radical mechanism. Third, a series of diselenides were also employed for this transformation (**3r**−**3v**), giving the expected products in good to excellent yields. A lower yield was observed with dibenzylselenide **3w** due to the slow reaction rate. The terminal alkynes (**3x**, **3y**) did not work under these HFIP conditions. After switching the conditions to Au catalysis, significantly improved yields were obtained. Notably, when HFIP was used as solvent instead of DCE, the reaction performance could be improved to 93% and 80%, respectively. No racemization of the chiral center on the amino acid after reaction highlighted the mild conditions as well as the great promoting effects of HFIP solvent in gold redox catalysis (for chiral HPLC characterization, see section VI in the Supporting Information).

Although the detailed mechanism of this HFIP-promoting effect remains to be explored, one plausible reason behind this "boosting effect" is that HFIP activates RSeSeR to form a selenium species as a good leaving group through an H-bond network.[4j] Therefore, under a gold catalytic pathway, this HFIP boosting effect could help the oxidation of gold(I) to gold(III)with the activated selenium, which is likely the turnover-limiting step. Similarly, in the nonmetal catalytic conditions, diselenides are good electrophiles that form an equilibrium with alkyne addition. This hypothesis also explained the reason why carbonyl-conjugated alkynes (**3x**, **3y**), as bad nucleophiles, did not work under nonmetal catalytic conditions. The cation-stabilizing effect of HFIP would also help the activation of unsaturated substrates under HFIP/Au conditions.[5] Overall, through the formation of H-bond, HFIP could greatly assist diselenides activation and increase the reactivity of this diselenation. Based on this hypothesis, we wondered if this activation mode could be applied to similar transformations using alkene and allene as the substrate. To confirm this idea, reactions of diselenides with alkenes and allenes were performed.

As shown in Figure 2A, styrene failed to give the diselenation product with the formation of complex reaction mixtures without diselenation product. With allene substrates, lower yields were observed. Monitoring the reactions with NMR revealed the decomposition of products over time under the reaction conditions. The addition of gold catalyst in the allene case could give a faster reaction. As a result, improved yields were observed (Figure 2A).

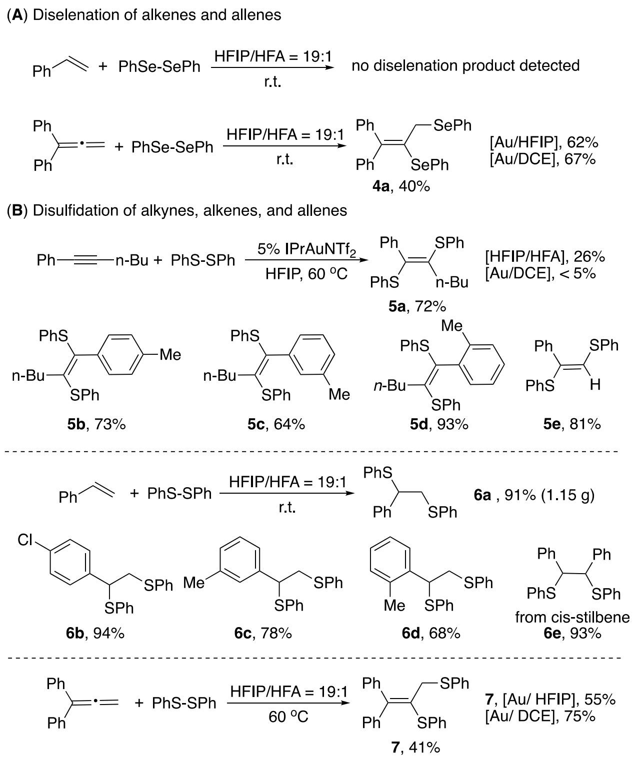


Figure 2. Disulfidation and diselenation reactions.

Considering that HFIP might provide a similar H-bond activation effect toward disulfide (RSSR), we started out our investigations on disulfidation with various unsaturated C−C bonds under Au/HFIP conditions. For alkyne substrates (Figure 2B), HFIP could not promote this transformation, giving the desired disulfide products in low yield. In comparison, the combination of gold and HFIP afforded the desired products in good yield (72%), while DCE solvent gave only a trace amount of product. Different internal and terminal alkynes were tested with desired products observed in good to excellent yields (**5a**−**5e**). Aliphatic substituted alkynes, both internal and terminal, give almost no products under either HFIP or HFIP/Au catalysis conditions (for the detailed scope for aliphatic alkynes, see Table S3). Overall, this result greatly highlighted the boosting effect of HFIP solvent applied in gold catalysis, which was critical for alkyne activations.

Furthermore, reactions of disulfide with alkene under the HFIP conditions gave the desired product **6a** in 91% yield with gram-scale synthesis. Some representative alkenes were tested with the desired disulfidation products obtained in good to excellent yields (**6b**−**6e**). Notably, the 1,2-disubstituted alkene, *cis*-stilbene, accomplished the reaction in excellent yield (**6e**, 93%), while the *trans*-stilbene did not work under these conditions. Notably, other solvents, such isopropanol and DCE, gave almost no reaction (<5%). Allene was also tested under the HFIP conditions, with or without gold catalyst presented. Moderate yield was observed using nonmetal conditions. With gold catalyst present, no significantly improved yield was observed in HFIP compared with DCE solvents due to the decomposition of product in HFIP. Nevertheless, HFIP presented a solvent-promoting effect over other solvents, suggesting this boosting effect could also be applied in C−C unsaturated bonds in the disulfidation reaction under mild conditions.

In conclusion, the diselenation and disulfidation of C−C unsaturated bonds were reported using HFIP as the promoting solvent. Compared with other common organic solvents, HFIP greatly enhanced the reaction performance by activating the diselenide and disulfide through H-bond networks. The desired products were achieved under mild conditions with good to excellent yield and high stereoselectivity. These results offered another prospective use of HFIP as the solvent to boost the reactivity of substrates and achieve challenging transformations.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c01834.

> Experimental details, NMR spectra, and details of the experiments (PDF)

Accession Codes

CCDC 2006688−2006689 contain 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 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.

■ AUTHOR INFORMATION

Corresponding Author

Xiaodong Shi − *Department of Chemistry, University of South Florida, Tampa, Florida 33620, United States*; orcid.org/0000-0002-3189-1315; Email: xmshi@usf.edu

Authors

Chiyu Wei − *Department of Chemistry, University of South Florida, Tampa, Florida 33620, United States*
Ying He − *Department of Chemistry, University of South Florida, Tampa, Florida 33620, United States*
Jin Wang − *College of Chemistry, Chemical Engineering and Material Science, Shandong Normal University, Jinan, Shandong 250014, China*

Xiaohan Ye − *Department of Chemistry, University of South Florida, Tampa, Florida 33620, United States*
Lukasz Wojtas − *Department of Chemistry, University of South Florida, Tampa, Florida 33620, United States*

Complete contact information is available at:
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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

We are grateful to the NSF (CHE-1665122) and NIH (1R01GM120240-01) for financial support.

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