OL | Organic Letters

Letter

Cite This: Org. Lett. 2019, 21, 7674−7678

pubs.acs.org/OrgLett

Copper-Catalyzed Phosphonylation/Trifluoromethylation of *N*-*p*-NO_2-Benzoylacrylamides Coupled with Dearomatization and Denitration

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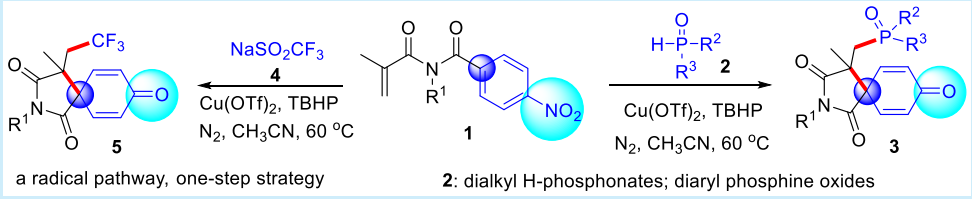
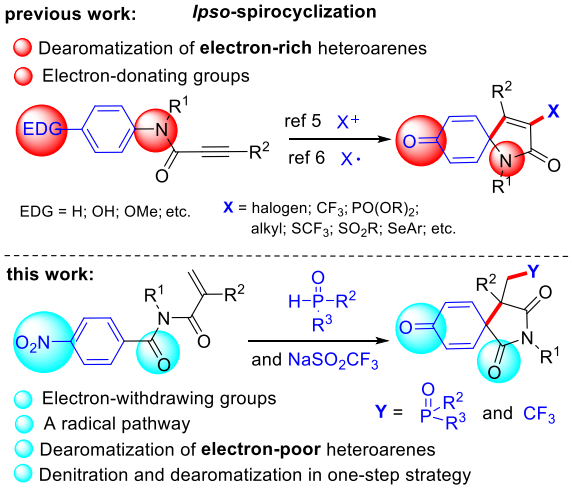
Supporting Information



ABSTRACT: A novel and efficient copper-*tert*-butyl hydroperoxide mediated intramolecular spirocyclization of *N*-*p*-NO_2-benzoylacrylamides through a cascade radical addition−*ipso*-cyclization−dearomatization−denitration process has been developed, affording a convenient and powerful tool for the preparation of valuable phosphonated or trifluoromethylated azaspiro[4.5]decadientriones under mild conditions in good yields.

Spirocyclic compounds are widely found in many bioactive compounds and functional molecules, among which azaspiro[4.5]decenones are privileged structural motifs occurring in various biologically active natural compounds.[1] Not surprisingly, their preparation has been the focus of recent research, and a few synthetic methods have emerged.[2] On the other hand, the development of efficient methods from readily accessible aromatic derivatives as the starting materials has therefore recently attracted considerable attention.[3] Dearomatization reactions provide a powerful and straightforward strategy to synthesize alicyclic frameworks from aromatic compounds.[4] Because of the current interest in this class of synthetically important azaspiro[4.5]decane derivatives and the valuable dearomatization reactions, not surprisingly, many efforts have been devoted to the efficient synthesis of such spirocyclic compounds through intramolecular *ipso*-cyclization of *N*-arylpropiolamide derivatives (Scheme 1). In the past decades intramolecular electrophilic halogenation−*ipso*-cyclization has emerged as an efficient way to access functionalized spirocyclic compounds.[5] Meanwhile, many studies were focused on dearomatization of phenols and related derivatives through a radical-participated tandem reaction, which provided a direct approach to the highly valuable azaspiroenones and could be applied to complex total syntheses (Scheme 1).[6] Despite the success of these radical and electrophilic *ipso*-cyclization of *N*-arylpropiolamides procedures for the azaspiro[4.5]decane derivatives, the range of starting materials is limited to *ipso*-cyclization and dearomatization of electron-rich heteroarenes, and a general method for the synthesis of azaspiro[4.5]decanes from electron-poor heteroarenes would be appealing.

Scheme 1. Route to Azaspiro[4.5]decenones



Received: August 25, 2019
Published: September 9, 2019

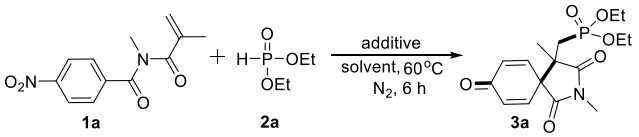
DOI: 10.1021/acs.orglett.9b03034
Org. Lett. 2019, 21, 7674−7678

Organophosphorus compounds have a wide range of applications in organic synthesis, medicinal chemistry, and materials industries, which have drawn much attention from the synthetic community. The incorporation of phosphonated functional groups into organic molecules has been widely recognized as a general strategy in pharmaceutical research and drug development.[7] We speculated that, if both an $(RO)_2P(O)$ group and an azaspiro[4.5]decane structural motif can be simultaneously introduced into novel organic compounds, efficient synthesis of phosphonyl azaspiro[4.5]decanes might be expected. Further it would provide an opportunity to introduce a phosphonyl group into the original compounds to adjust their bioactivities. To the best of our knowledge, the example of phosphonated azaspiro[4.5]decane via *ipso*-cyclization and dearomatization of *p*-nitrobenzamide *has not yet been reported* (Scheme 1).

As a continuation of our endeavor to develop step-economical heterocyclic phosphonates formations,[8] we reported a direct synthesis of phosphonoisoquinolinediones via radical phosphonylation−cyclization of *N*-methacryloyl-*N*-methylbenzamides with P(O)H compounds in 2015.[8e] Herein, we describe a tandem preparation of phosphonated or trifluoromethylated azaspiro[4.5]deca-6,9-diene-1,3,8-triones through a cascade radical addition−*ipso*-cyclization−dearomatization−denitration process.

At the outset of our studies, we chose *N*-methacryloyl-*N*-methyl-4-nitrobenzamide (**1a**) and diethyl *H*-phosphonate (**2a**) as the model substrates to optimize the reaction conditions (Table 1). It is well-known that many salts such as copper, silver, and manganese can convert $R_2P(O)H$ to the corresponding phosphonyl radical, which promotes the development of phosphorus-centered radical chemistry.[9] Initially, manganese and silver catalysts were tested, but all gave a trace amount of **3a** (entries 1 and 2). Gratifyingly, the combination of $Cu(OAc)_2$ and TBHP (*tert*-butyl hydroperoxide, 70% aqueous solution) gave **3a** in 72% yield (entry 3). Subsequently, various copper salts were further tested, which implied that $Cu(OTf)_2$ was more effective to furnish the desired product **3a** in 85% yield within 6 h (entries 4−6).

Table 1. Optimization of Reaction Conditions[a]



entry	additive (equiv)	solvent	yield (%)
1	$Mn(OAc)_3{\cdot}2H_2O$ (3)	CH_3COOH	trace
2	$AgNO_3$ (0.05) + $Mg(NO_3)_2$ (0.5)	CH_3CN	trace
3	$Cu(OAc)_2$ (0.1) + TBHP (3)	CH_3CN	72
4	$CuCl_2$ (0.1) + TBHP (3)	CH_3CN	trace
5	CuCl (0.1) + TBHP (3)	CH_3CN	trace
6	**$Cu(OTf)_2$ (0.1) + TBHP (3)**	**CH_3CN**	**85**
7	$Cu(OTf)_2$ (0.1) + TBHP (3)	CH_3CN	85,[b] 88[c]
8	$Cu(OTf)_2$ (0.05) + TBHP (3)	CH_3CN	80
9[d]	$Cu(OTf)_2$ (0.1) + TBHP (3)	CH_3CN	77
10	$Cu(OTf)_2$ (0.1)	CH_3CN	trace
11	TBHP (3)	CH_3CN	65
12	$Cu(OTf)_2$ (0.1) + DTBP (3)	CH_3CN	12

[a]Isolated yield. [b]12 h. [c]TBHP (5 M in decane), anhydrous acetonitrile, 6 h. [d]Open to air.

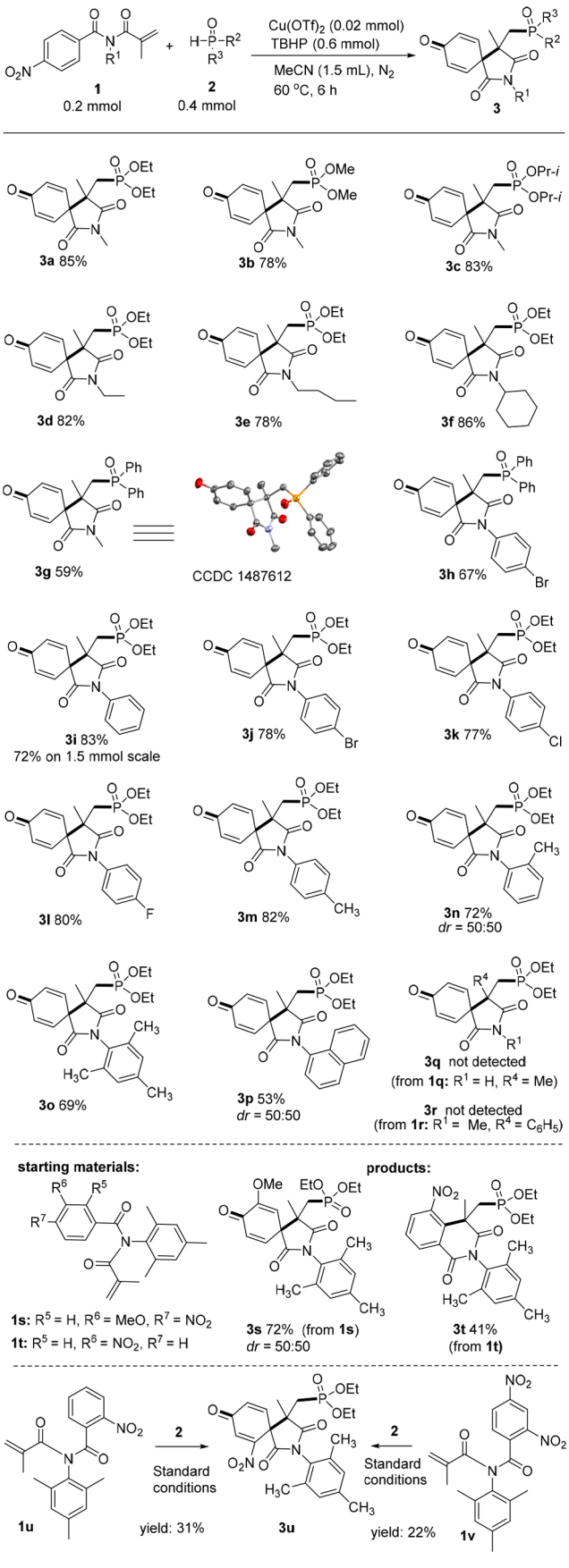
Prolonging the reaction time up to 12 h gave the same yield (entry 7). Conducting the reaction under anhydrous conditions (*tert*-butyl hydroperoxide, 5 M in decane, anhydrous acetonitrile) furnished **3a** in 88% yield (entry 7). When the loading of $Cu(OTf)_2$ was reduced to 5 mol %, the reaction also afforded **3a** with a slightly lower yield (80%, entry 8), which demonstrates the high catalytic efficiency of this $Cu(OTf)_2$−TBHP system for the generation of phosphonyl radical and the *ipso*-cyclization−dearomatization−denitration reaction. The yield dropped to 77% when the reaction was conducted under aerobic conditions (entry 9). Without TBHP, copper salt was unable to perform this reaction (entry 10). Although TBHP alone could work, it gave a much lower yield (entry 11). A poor yield was obtained with DTBP (di-*tert*-butyl peroxide) instead of TBHP (entry 12). After a series of investigations, we established an efficient route to formation of phosphonated azaspiro[4.5]deca-6,9-diene-1,3,8-triones. The optimal conditions are **1a** (0.2 mmol), **2a** (0.4 mmol), $Cu(OTf)_2$ (0.02 mmol), TBHP (0.6 mmol, 70% aqueous solution), and MeCN (1.5 mL) at 60 °C for 6 h under a nitrogen atmosphere (Table 1, entry 6).

With the optimal conditions in hand, the substrate scope of this reaction was studied (Scheme 2). The reaction could proceed well by using diverse dialkyl H-phosphonates to afford the desired products **3a**−**3f** and **3i**−**3u** in good yields. Importantly, $Ph_2P(O)H$ was also tested for this reaction to produce the corresponding triarylphosphine oxides **3g** and **3h** in 59% and 67% yields, respectively. Moreover, the structure of **3g** was confirmed by X-ray crystal structure analysis (CCDC 1487612). Furthermore, we explored the effect of substituents at the nitrogen atom. Substrates with ethyl, *n*-butyl, and cyclohexyl substituents afforded the corresponding compounds **3d**, **3e**, and **3f** in good yields (82%, 78%, 86%). Substrates with an aryl group afford the desired products **3h**−**3p** and **3s**−**3u**. When *N*-substituent R^1 was a phenyl group, the desired product **3i** was obtained in 83% yield. The nature of the substituents on the benzene ring was next scrutinized. The products **3j**−**3l** bearing bromo, chloro, and fluoro groups were generated in high yields (78%, 77%, 80%). The steric hindrance at the nitrogen atom has a significant influence on the formation of phosphonated azaspiro[4.5]deca-6,9-diene-1,3,8-triones (**3m**−**3o**). For example, with methyl substituted on benzene R^1, such as *para*- and *ortho*-methyl groups, these compounds reacted efficiently to give the desired products in good yields (**3m**, 82%; **3n**, 72%, 50:50 dr). In the presence of the mesityl group, the reaction led to the desired **3o** in 69% yield. When naphthalene was severed as the substituent, the expected product **3p** was obtained in acceptable yield (53%, 50:50 dr). Unfortunately, in the case of the free hydrogen at the nitrogen, it failed to deliver the corresponding product **3q**. *N*-Methyl-*N*-(4-nitrophenyl) cinnamamide (**1r**) was also examined; no desired product **3r** was detected by ^{31}P NMR. When 3-MeO-4-NO_2-benzoylacrylamide (**1s**) was used as starting material, the desired dearomatization product **3s** was obtained in 72% yield. The reaction of 3-NO_2-benzoylacrylamide (**1t**) under the standard conditions gave only the normal coupling product **3t** in 41% yield; no dearomatization product was isolated.[10] Both 2-NO_2-benzoylacrylamide (**1u**) and 2,4-di-NO_2-benzoylacrylamide (**1v**) under the standard conditions gave the same dearomatization product **3u** in slightly lower yields.

The trifluoromethyl group plays a privileged role in pharmaceuticals and agrochemicals because introducing this

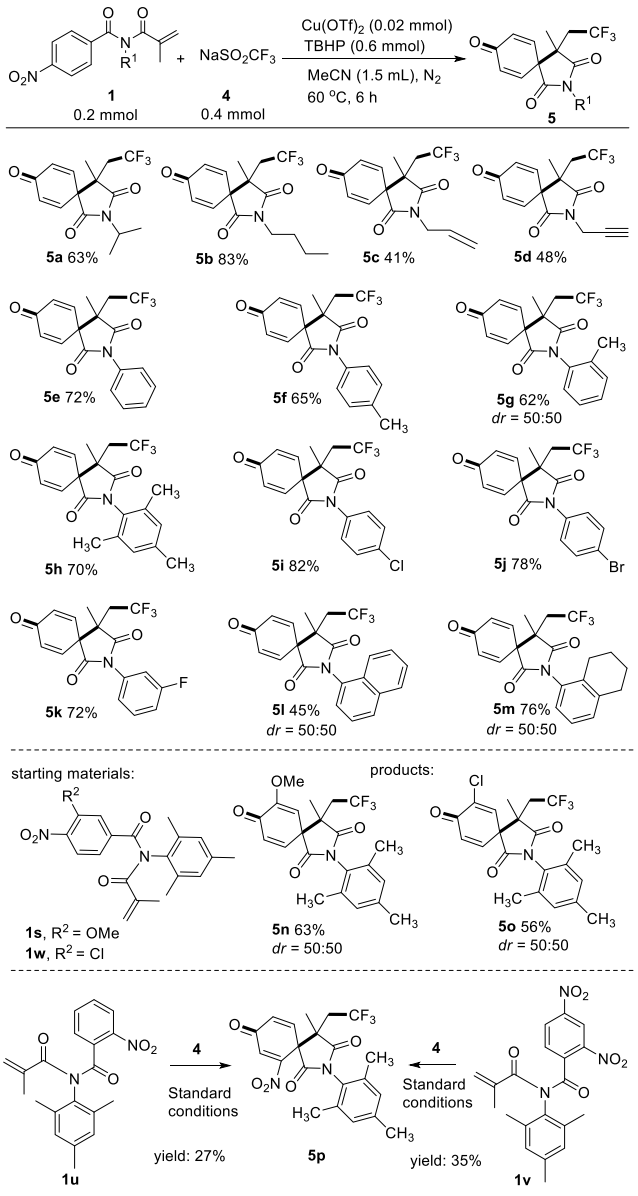
DOI: 10.1021/acs.orglett.9b03034
Org. Lett. 2019, 21, 7674−7678

Scheme 2. Reaction Scope Study



group into organic molecules tends to improve their chemical and metabolic stability and increase their lipophilicity, bioavailability, and protein-binding affinity. Consequently, the development of efficient, versatile methods for introducing a trifluoromethyl group has attracted considerable attention.[11] Encouraged by the findings described above, we continued to explore the synthesis of trifluoromethylated azaspiro[4.5]decadientriones (Scheme 3). Eventually, the trifluoromethylated

Scheme 3. Dearomative Trifluoromethylation of Nitrobenzamide Derivatives



product **5a** was obtained in 63% yield by using sodium trifluoromethanesulfinate as the source of trifluoromethyl radical under the same conditions. As for the substrate with *N*-*n*-butyl **5b**, the yield was improved to 83%. Interestingly, even for the substituents with unsaturated moiety allyl and propargyl at the nitrogen, the desired products were also isolated in acceptable yields without damaging the double and triple bonds (**5c**, 41%; **5d**, 48%). In the cases of benzene and toluene as the substituents, the expected products were obtained in good yields (**5e**, 72%; **5f**, 65%). Consistent with

DOI: 10.1021/acs.orglett.9b03034
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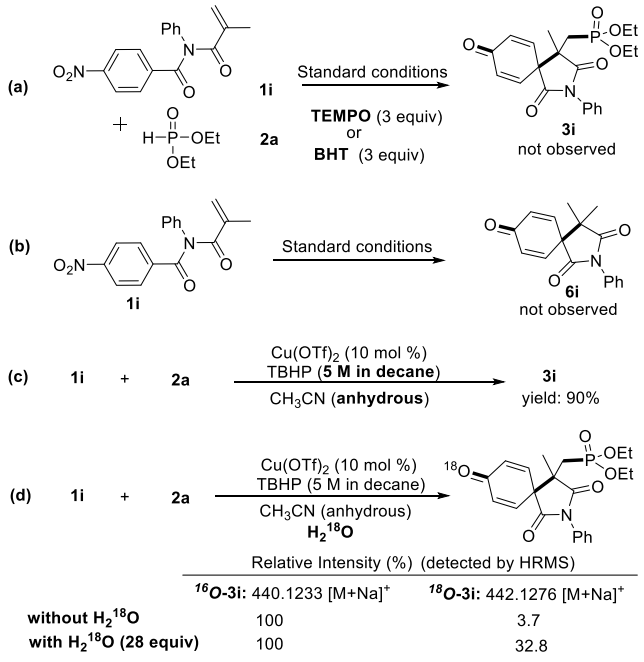
3n, the same starting material led to the trifluoromethylated product **5g** as a mixture of diastereoisomers (62%, 50:50 dr). Concerning substitutions on the benzene ring, the mesityl group (**5h**; 70%) and *para*-fluoro, *para*-bromo (**5i**, 82%; **5j**, 78%), *meta*-fluoro (**5k**, 72%) gave the desired products. Increasing the steric hindrance at the substituent still furnished **5l** and **5m** in moderate to good yields (**5l**, 45%, 50:50 dr; **5m**, 76%, 50:50 dr). When 3-MeO-4-NO_2-benzoylacrylamide **1s** and 3-Cl-4-NO_2-benzoylacrylamide **1w** were used as starting materials, the dearomatization product **5n** and **5o** were obtained in moderate yields. Both 2-NO_2-benzoylacrylamide (**1u**) and 2,4-di-NO_2-benzoylacrylamide (**1v**) under the standard conditions gave the same dearomatization product **5p** with high conversions but low yields; moreover, product **5p** was unstable and decomposed during the column chromatography separation.

In order to gain insight into the mechanism of this reaction, some preliminary studies were carried out (Scheme 4). No

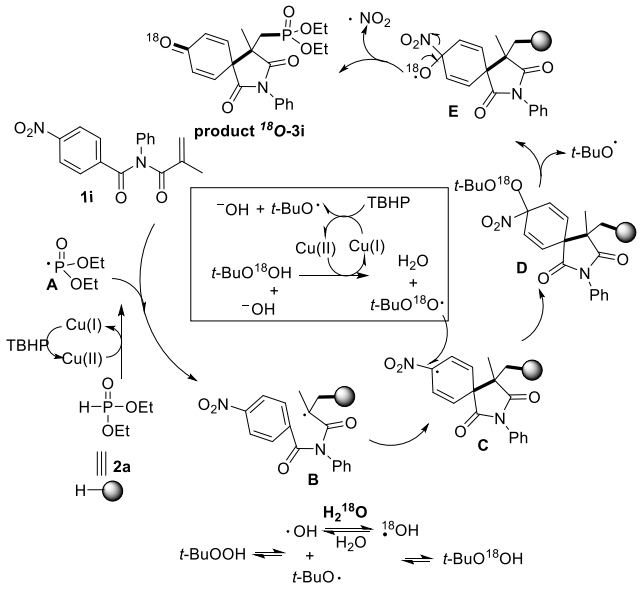
Scheme 4. Mechanistic Investigation



desired product **3i** was obtained when 3.0 equiv of the radical inhibitor 2,2,6,6-tetramethylpiperidine oxide (TEMPO) or butylated hydroxytoluene (BHT) was added to this system under standard conditions (Scheme 4a), thus suggesting that the radical processes might be involved. When **1i** was investigated under the standard conditions without **2a**, the *ipso*-cyclization product **6i** was not detected (Scheme 4b). When the transformation was carried out under anhydrous conditions, the desired product **3i** was obtained in 90% yield, thus implying that H_2O is not the key starting material (Scheme 4c). Then, an ^{18}O-labeling experiment was performed to explore the source of the oxygen atom of the newly formed carbonyl (Scheme 4d). Without $H_2{}^{18}O$, the ^{18}O-**3i** was detected by HRMS and its relative intensity was 3.7%. When 28 equiv of $H_2{}^{18}O$ were added into the reaction, the ^{18}O-labeled product was detected by HRMS with the relative intensity of 32.8% (Scheme 4d). There is a dramatic increase in terms of the amount of ^{18}O-labeled product when $H_2{}^{18}O$ was added (for details, see the Supporting Information). In light of these results, we propose that the oxygen atom of the newly formed carbonyl mainly comes from TBHP, with an exchange of oxygen atom with $H_2{}^{18}O$.

On the basis of this study and previous reports,[9] a plausible mechanism was proposed (Scheme 5). First, the interaction of

Scheme 5. A Tentative Mechanistic Pathway



H-phosphonate **2a** with TBHP and Cu(II) generates phosphonyl radical **A**. Subsequently, the phosphorus-centered radical interacts with the activated alkene **1i** to give a new C−P bond and an alkyl radical intermediate **B**. The alkyl radical immediately undergoes thermodynamically controlled 5-exo cyclization onto the nitrobenzene ring to furnish spirocyclic intermediate **C**, which could be coupled with *t*-Bu$O^{18}O$· to afford **D**. Then, homolysis of the peroxide oxygen−oxygen bond forms free radical **E**. Finally, denitration of **E** affords ^{18}O-**3i** or **3i**. On the other hand, hydrogen abstractions from molecules by free radicals have been studied extensively ($H^{18}OH + H^{16}O· \rightarrow H^{18}O· + H_2{}^{16}O$).[12] This isotope exchange reaction promotes the conversion of *t*-BuOOH into *t*-Bu$O^{18}OH$.

In summary, we have developed a highly efficient protocol for the synthesis of various phosphonated and trifluoromethylated azaspiro[4.5]decanes by copper-catalyzed cascade difunctionalization of alkenes through a cascade radical addition−*ipso*-cyclization−dearomatization−denitration process. Moreover, the use of an inexpensive copper catalyst, using readily available TBHP means that this facile protocol will be attractive for academia and industry. Given that a wide range of substrates can be utilized for the cascade annulation−dearomatization, this simple protocol may provide a general approach to phosphonoylated and trifluoromethylated azaspiro[4.5]dienone frameworks of importance in medicinal chemistry and synthetic chemistry.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b03034.

DOI: 10.1021/acs.orglett.9b03034
Org. Lett. 2019, 21, 7674−7678

Copies of ^{1}H NMR and ^{13}C NMR spectra of compounds **3a−3p**, **3s−3u**, **5a−5p**; single-crystal X-ray spectrum of compound **3g** (PDF)

Accession Codes

CCDC 1487612 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 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.

ACKNOWLEDGMENTS

We thank NSFC (21772163, 21778042) and NFFTBS (J1310024). We are grateful to Prof. Dr. Guillaume Vincent from Univ. Paris Sud for generous support on this work.

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DOI: 10.1021/acs.orglett.9b03034
Org. Lett. 2019, 21, 7674−7678