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Oxoammonium Salt-Mediated Vicinal Oxyazidation of Alkenes with NaN_3: Access to β-Aminooxy Azides

Fei Chen,[a,*] Yu-Ting Tang,[a] Xin-Ru Li,[a] Yan-Yan Duan,[a] Chao-Xing Chen,[a] and Yang Zheng[a,*]

[a] Henan Provincial Engineering and Technology Research Center for Precise Synthesis of Fluorine-Containing Drugs, College of Chemistry and Chemical Engineering, Anyang Normal University, Anyang, 455000, People's Republic of China
E-mail: chenf2019@aynu.edu.cn; zhengy2019@aynu.edu.cn

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Abstract: An approach to the vicinal oxyazidation of alkenes has been achieved under mild and transition metal-free conditions. This method utilizes NaN_3 as the azidation agent and 2,2,6,6-tetramethylpiperidine-1-oxoammonium tetrafluoroborate ($TEMPO^+BF_4^-$) as the single-electron oxidant as well as the oxygen source. By using this protocol, various β-aminooxy azides were synthesized and several complex bioactive molecules were functionalized.

Keywords: vicinal oxyazidation; alkenes; TEMPO; β-aminooxy azides; single electron transfer

Organic azides as a versatile reagent have been widely applicated in synthetic chemistry, biochemistry and materials science.[1] In particular, β-oxy azides not only exists in biologically active molecules but also can be transformed into other important compounds that contain two vicinal nitrogen and oxygen atoms.[2] For example, zidovudine is a prodrug used in the treatment of HIV/AIDS.[2a] Azidocillin is an anti-inflammatory drug.[2b] Salbutamol is an important bronchodilator.[2c] Bupropion is an antidepressant (Figure 1).[2d]

Thus, the efficient synthesis of β-oxy azides has attracted much attention from organic and pharmaceutical chemists.[3–5] In the past few years, a series of metal-catalyzed and metal-free methods on oxyazidation of alkenes have been developed.[4,5] For instance, Mn-catalyzed hydroxyazidation,[4e] Cu-catalyzed alkoxyazidation[4f]/oxo-azidation[4g] and Ag-catalyzed azidotrifluoromethoxylation[4h] of olefins were individually reported by several different research groups (Scheme 1a). Moreover, Studer[5a] et al. demonstrated a novel TEMPONa mediated azidooxygenation of alkenes with Zhdankin reagent. Lu's,[5d] Wei's[5f] and Kashyap's[5g,h] groups independently disclosed visible-light-promoted oxyazidation of olefins. Lin[5e] and co-workers developed a convenient electrochemical azidooxygenation of alkenes (Scheme 1b). Despite these elegant works toward this end, however, the progress is far from meeting the need of synthesis, and the exploration of an efficient and low-cost approach to this structurally important β-oxy azides is still in demand.

As a continuation of our research on the difunctionation of alkenes,[6] herein we wish to present a new and efficient method for the oxyazidation of various alkenes by using commercially available NaN_3 as the azidation reagent and easily prepared $TEMPO^+BF_4^-$ as the oxidant as well as the oxygen source (Scheme 1c).[7] This strategy not only provides a practical way for the convenient synthesis of biologically important β-aminooxy azides but also can be applied to late-stage modification of bioactive molecule derivatives.

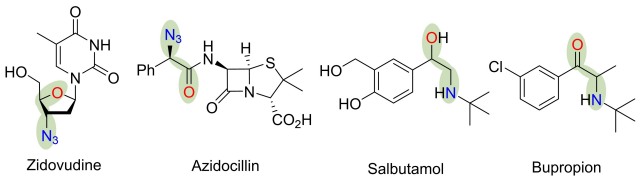
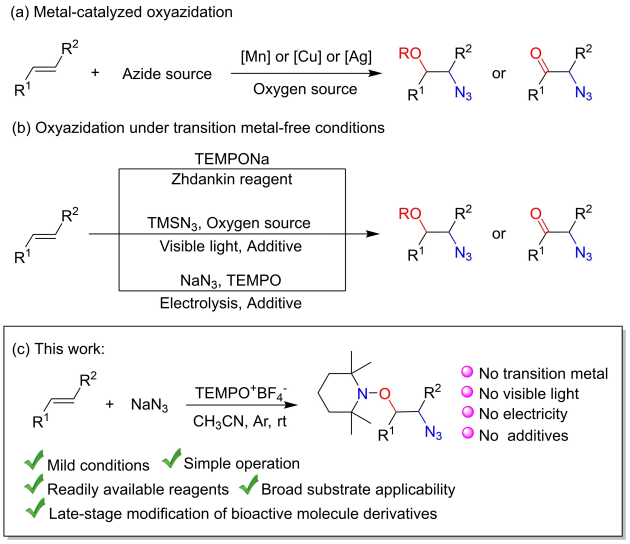


Figure 1. Bioactive molecules containing β-oxy azide/1,2-amino alcohol/α-amino ketone moiety.

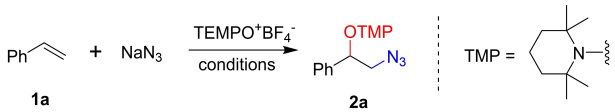
We commenced our study by subjecting styrene (**1a**) (1.0 equiv.) to NaN_3 (1.0 equiv.) and $TEMPO^+BF_4^-$ (1.0 equiv.) in CH_3CN under an argon atmosphere at room temperature for 24 h. To our delight,



Scheme 1. Representative oxyazidation of alkenes and our work using $TEMPO^+BF_4^-$.

the desired product 1-(2-azido-1-phenylethoxy)-2,2,6,6-tetramethylpiperidine (**2a**) was generated in 79% yield (Table 1, entry 1). When the usage amounts of NaN_3 and $TEMPO^+BF_4^-$ were both increased to 1.1 equivalent, the yield of **2a** was improved to 89% (Table 1, entry 2). However, further increasing the application amounts of NaN_3 and $TEMPO^+BF_4^-$ did not give a better result (Table 1, entry 3). Amazingly, **2a** was still obtained in 85% yield under air atmosphere (Table 1, entry 4). Besides CH_3CN, several other solvents, such as THF, PhCl, DMSO, DMF, and DCM were also investigated, but no better yield was obtained (Table 1, entries 5–9). In addition, other azidation agent and other oxidant were also examined, but the outcomes received under these conditions were not satisfying (Table 1, entries 10 and 11).

Table 1. Optimization of the reaction conditions.[a]



1a 2a

Entry	NaN_3	$TEMPO^+BF_4^-$	Solvent	Yield (%)[b]
1	1.0 equiv.	1.0 equiv.	CH_3CN	79
2	1.1 equiv.	1.1 equiv.	CH_3CN	89
3	1.5 equiv.	1.5 equiv.	CH_3CN	89
4[c]	1.1 equiv.	1.1 equiv.	CH_3CN	85
5	1.1 equiv.	1.1 equiv.	THF	49
6	1.1 equiv.	1.1 equiv.	PhCl	63
7	1.1 equiv.	1.1 equiv.	DMSO	0
8	1.1 equiv.	1.1 equiv.	DMF	28
9	1.1 equiv.	1.1 equiv.	DCM	47
10[d]	1.1 equiv.	1.1 equiv.	CH_3CN	15
11[e]	1.1 equiv.	2.2 equiv.	CH_3CN	0

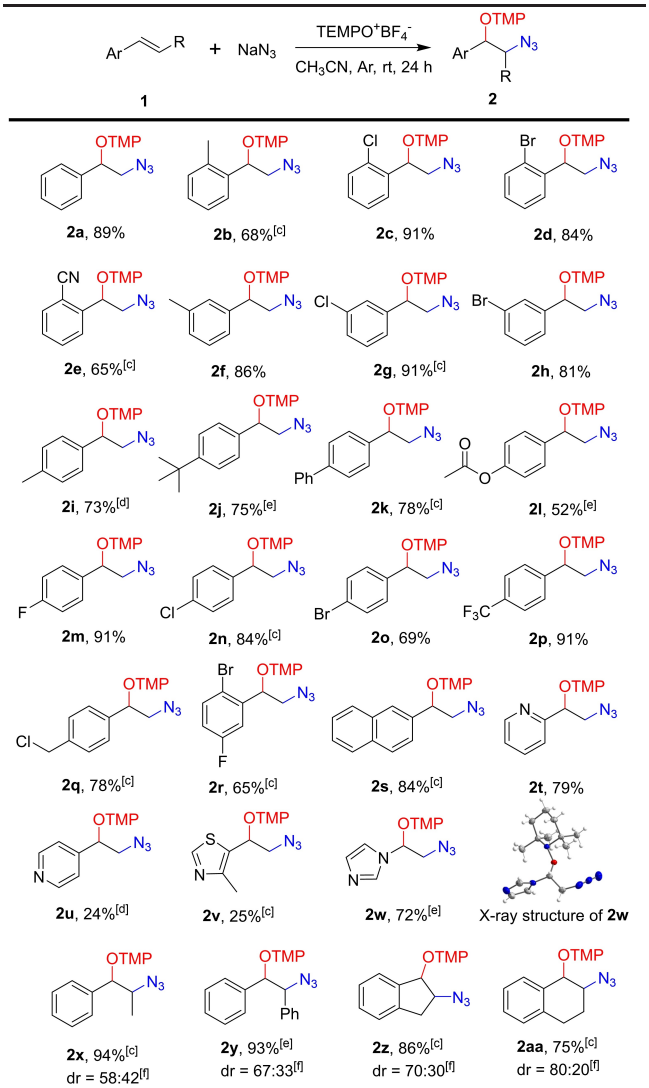
[a] Unless otherwise specified, all reactions were carried out by stirring a mixture of **1a** (0.3 mmol, 1.0 equiv.), NaN_3 and $TEMPO^+BF_4^-$ in 2 mL of solvent under argon atmosphere (1 atm) at room temperature for 24 h.
[b] Isolated yield.
[c] Under air atmosphere (1 atm).
[d] $TMSN_3$ was used instead of NaN_3.
[e] TEMPO was used instead of $TEMPO^+BF_4^-$.

With the optimized conditions in hand (Table 1, entry 2), the scope of styrenes was surveyed (Table 2). First, ortho/meta/para-substituted styrenes with a variety of electronic properties were well compatible in the reaction, delivering the desired products **2a–q** in good to excellent yields. Polysubstituted styrene also participated well in the procedure, affording the expected product **2r** in 65% yield. Reaction of 2-vinylnaphthalene with NaN_3 and $TEMPO^+BF_4^-$ yielded the product **2s** in 84% yield. The heterocyclic substrates such as 2-vinylpyridine, 4-vinylpyridine, 4-methyl-5-vinylthiazole, and 1-vinyl-1*H*-imidazole were also good candidates for this process, providing the corresponding oxyazidation products **2t–w** in 24% to 79% yields. The structure of **2w** was confirmed by a single-crystal X-ray diffraction study.[8] Significantly, this strategy was also suitable to 1,2-disubstituted substrates, as exhibited in the cases of **2x–aa**.

Having successfully achieved the oxyazidation of styrenes, we next shifted our attention to explore the applicability of this new approach to aliphatic alkenes (Table 3). Gratifyingly, both simple and various functionalized terminal alkenes participated smoothly in the reaction, delivering the corresponding oxyazidation products **2ab–ag** in 23–38% yields with starting material being recovered in some cases (66% of **1ad**, 64% of **1ae** and 32% of **1af**). Unfortunately, pent-4-en-1-ol was not applicable for this method and only unidentifiable complex mixtures were observed. Significantly, internal alkenes such as cyclopentene, cyclohexene, and (Z)-cyclooctene were very suitable for this strategy, providing the desired products **2ai–ak** in good to excellent yields. When buta-1,3-dien-1-ylbenzene was used as the substrate, the reaction did not yield the expected oxyazidation product; instead, compound **2al′** was acquired in a yield of 23%.[9] In addition, phenylacetylene was also investigated, but only unidentifiable complex mixtures were obtained.

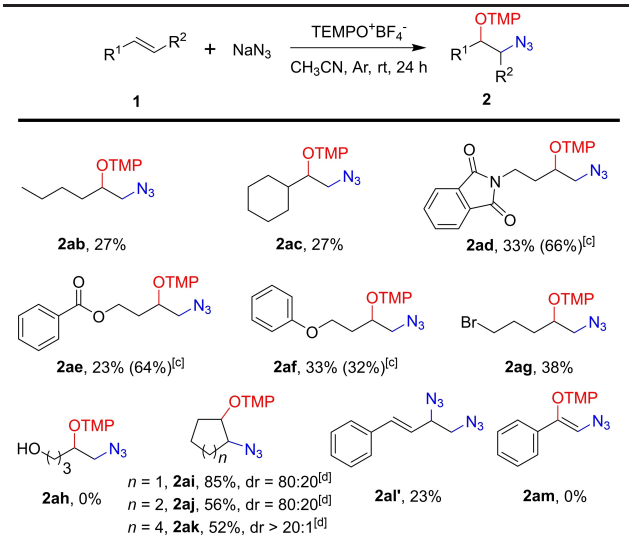
To demonstrate the synthetic potentiality of this strategy, this reaction was applied to the late-stage modification of complex molecules derived from natural products and drugs (Figure 2). For example, when estrone and deoxycholic acid derivatives were used as substrates, the desired oxyazidation product **2an** and **2ao** were gained in good yields. Moreover, styrenes derived from drugs such as aspirin, ibupro-

Table 2. Oxyazidation of styrenes.[a,b]



[a] Reaction conditions: **1** (0.3 mmol, 1.0 equiv.), NaN_3 (1.1 equiv.) and $TEMPO^+BF_4^-$ (1.1 equiv.) in CH_3CN (2 mL) at room temperature under argon atmosphere (1 atm) for 24 h.
[b] Isolated yields.
[c] NaN_3 (1.7 equiv.) and $TEMPO^+BF_4^-$ (1.7 equiv.) were used.
[d] NaN_3 (1.3 equiv.) and $TEMPO^+BF_4^-$ (1.3 equiv.) were used.
[e] NaN_3 (2.1 equiv.) and $TEMPO^+BF_4^-$ (2.1 equiv.) were used.
[f] The ratio of diastereomers was determined by ^1H NMR spectroscopy.

Table 3. Oxyazidation of aliphatic alkenes.[a,b]



[a] Reaction conditions: **1** (0.3 mmol, 1.0 equiv.), NaN_3 (1.8 equiv.) and $TEMPO^+BF_4^-$ (1.8 equiv.) in CH_3CN (2 mL) at room temperature under argon atmosphere (1 atm) for 24 h.
[b] Isolated yields.
[c] The yield of recovered substrate is listed in brackets.
[d] The ratio of diastereomers was determined by ^1H NMR spectroscopy.

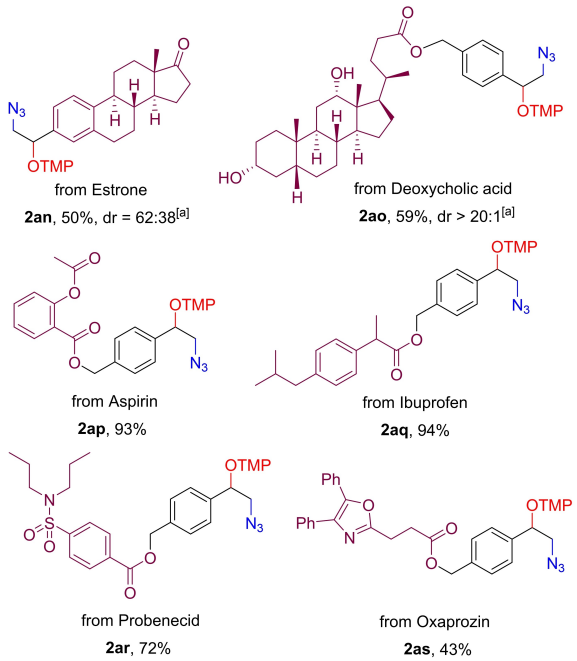
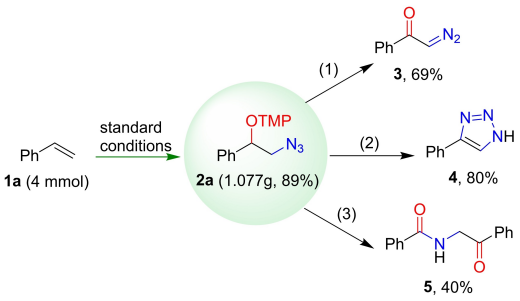


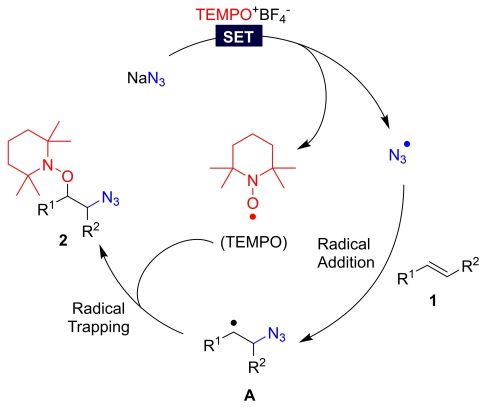
Figure 2. Oxyazidation of natural product derivatives and drug derivatives.[a] The ratio of diastereomers was determined by ^1H NMR spectroscopy.

fen, probenecid, and oxaprozin were also applicable for this protocol, affording the expected products **2 ap**–**as** in moderate to excellent yields.

To further examine the practicability of this protocol in the organic synthesis chemistry, a gram-scale reaction of styrene **1 a** was performed under the standard conditions. Cheerfully, the reaction still worked smoothly and the product **2 a** was obtained in 89% yield (Scheme 2). Moreover, *β*-oxy azides are functional synthetic building blocks in organic synthesis. Follow-up conversions of *β*-oxy azides



Scheme 2. Gram-scale reaction and follow-up transformations of **2 a**. For the reaction conditions in details, see Supporting Information.

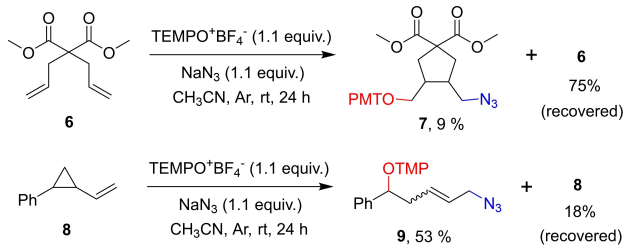


Scheme 4. Proposed mechanism.

can supply multifarious important N/O-containing compounds that are potentially bioactive molecules or significant organic synthetic precursors. Transformations of product **2 a** via click reaction, Staudinger reaction, reduction, and oxidation to afford corresponding triazole, amine, alcohol, and α-azidoketone have previously been described in the literature.[5a] Notably, the α-azidoketone skeleton is very useful in the synthesis of pyrroles,[10] imidazoles,[11] isoquinoline,[12] and other azacyclic compounds.[13] Furthermore, as illustrated in Scheme 2, some important synthetic intermediates such as α-diazoketone **3**, 4-phenyl-1*H*-1,2,3-triazole **4**, and *N*-(2-oxo-2-phenylethyl)benzamide **5** can be obtained separately as well through corresponding subsequent transformations of **2 a**.

To gain insights into the reaction mechanism, the control experiments were conducted as shown in Scheme 3. When dimethyl 2,2-diallylmalonate was subjected to the standard conditions, the cyclic product **7** was obtained in 9% yield, with 75% of **6** being recovered. When (2-vinylcyclopropyl)benzene was applied to the same conditions, the ring opening product **9** was gained in 53% yield along with recovered substrate **8** in 18% yield. These results demonstrated clearly that the oxyazidation reaction is triggered by the azide radical.

Based on the above results and previous literatures,[5e,14] a free radical mechanism is suggested to rationalize the oxoammonium salt-mediated vicinal oxyazidation process. As shown in Scheme 4, NaN_3 is firstly oxidized to the azide radical by $TEMPO^+BF_4^-$ via single electron transfer (SET). At the same time, $TEMPO^+BF_4^-$ is reduced to TEMPO. Then, azide radical adds to alkene **1** to give the carbon-centered radical **A**. Finally, **A** is trapped immediately by TEMPO, yielding the oxyazidation product **2**.



Scheme 3. Mechanistic investigations.

In summary, a protocol for the vicinal oxyazidation of alkenes has been developed. This method employs NaN_3 as the azide source and readily accessible $TEMPO^+BF_4^-$ as the oxidant as well as the oxygen donor. Consequently, this protocol has the advantages of transition metal free, broad substrate applicability, mild conditions, simple operation, and late-stage functionalization of bioactive molecules. By using this new method, various alkenes can be very easily converted into β-aminooxy azides. We hope that the reaction depicted herein will find applications in the synthesis of important bioactive compounds which contain vicinal nitrogen and oxygen atoms. Further studies of the oxoammonium salt-mediated reactions are in progress in our laboratory.

Experimental Section

General experimental procedure for synthesis of products 2

Substrates **1** (0.30 mmol, 1.0 equiv.), NaN_3 (1.1–2.2 equiv.), $TEMPO^+BF_4^-$ (1.1–2.2 equiv.) and MeCN (2 mL) were added to a 25 mL round bottom flask. The mixture was stired at room temperature under argon atmosphere (1 atm) for 24 h. Upon completion of the reaction, the solvent was then removed under vacuo. The residue was purified with chromatography column on silica gel (gradient eluent of EtOAc/petroleum ether: 1/100 to 1/1) to give the corresponding product **2**.

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Adv. Synth. Catal. **2021**, *363*, 1 – 7

F. Chen*, Y.-T. Tang, X.-R. Li, Y.-Y. Duan, C.-X. Chen, Y. Zheng*