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Fe-catalyzed three-component carboazidation of alkenes with alkanes and trimethylsilyl azide

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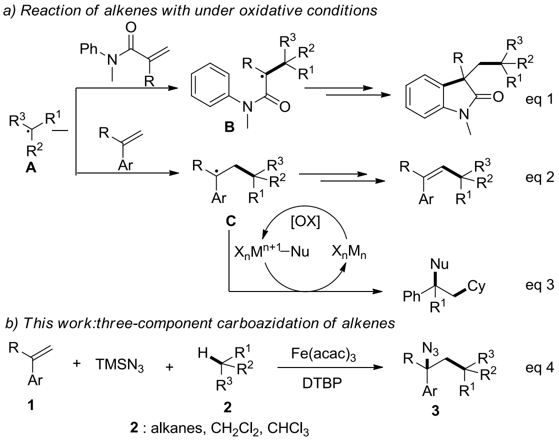
Wei-Yu Li, Chuan-Shuo Wu, Wang Zhou, Luo Yang*

Reported herein is the novel iron-catalyzed, DTBP-mediated carboazidation of alkenes using cycloalkanes, CH_2Cl_2, $CHCl_3$ and CCl_4 as alkylating reagents to generate electrophilic or nucleophilic alkyl radicals. Mechanistic studies suggested that the reaction proceeded via addition of alkyl radicals to alkenes followed by iron-mediated ligand transfer process. The reaction is unique as it is applicable not only to diversely functionalized electron rich alkenes, but also to electron-poor olefins to provide chain extended azides and γ-azido chloroalkanes in good to high yields.

Difunctionalization of unactivated alkenes producing value-added chemicals from easily available hydrocarbons has attracted much recent attention.[1] Among them, azide transfer reaction is of particular interest as it generates organic azides of significant synthetic importance. In this context, diazidation,[2] aminoazidation,[3] oxyazidation,[4] azidocyanation,[5] haloazidation[6] and carboazidation[7-10] of alkenes using different azide sources (NaN_3, $TMSN_3$ or azido hypovalent iodine) have been realized.[11] Most of these reactions are initiated by addition of the in situ generated azide radical to the double bonds. The carboazidation pioneered by Renaud and co-workers is however different as it is initiated by the addition of alkyl radicals to the olefins followed by azidation.[7] Alkyl halides,[7,8c] and trifluoromethyl hypervalent iodine reagents[8a,8b] are generally used as precursors of alkyl radical. More recently, oxidative carboazidation of alkenes using acetonitrile[9] as alkyl radical donors have been developed by J.P. Zhu *et.al*. Note that alkyl radical used in these studies are all electrophilic, therefore their reaction with alkenes is polarity matched.[12]



Scheme 1. Alkanes as alkylating reagent and carboazidation of alkenes

Oxidative generation of alkyl radicals from alkanes has been known for many years.[13] However, there is a recent resurgence of interest in this area.[14] In addition to alkylation of aromatic/heteroaromatic compounds[15] and amines/amides,[16] reaction of these alkyl radicals with alkenes have also been reported recently. The electron-poor olefins are generally preferred substrates to match the nucleophilicity of the alkyl radical **A**. The resulting electrophilic radical adduct **B** underwent rapid intramolecular homolytic aromatic substitution (HAS) with the tethered aromatic ring to afford cyclic compounds (eq 1, Scheme 1).[17] On the other hand, vinylation of alkanes by electron rich alkenes under oxidative conditions have also been reported.[18] In these cases, 1,1-diaryl substituted ethylenes or a stronger oxidant [e.g. $Cu(OTf)_2$] has to be used to most probably accelerate the oxidation of benzyl radical to carbenium intermediate (eq 2, Scheme 1). Although it was concluded in an authoritative review that the reaction with alkyl radical is synthetically significant only when the olefinic double bond is conjugated with electron-withdrawing groups, owing to the nucleophilic character of the alkyl radical,[12] we hypothesized that if the adduct radical **C** could be intercepted intermolecularly by a kinetically competent redox ligand transfer process, then a three-component reaction could be developed even if the initiation radical addition step was polarity mismatched (eq 3). We report herein an Fe-catalyzed DTBP-mediated carboazidation of unactivated

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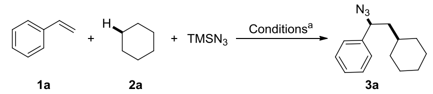
[a] *Address here. Key Laboratory for Green Organic Synthesis and Application of Hunan Province, College of Chemistry, Xiangtan University, Hunan, 411105, PR China,*
E-mail: yangluo@xtu.edu.cn

[b] † W.-Y. Li and C.-S. Wu contributed equally to this work.

Electronic Supplementary Information (ESI) available: experimental details, mechanistic studies and characterization of products. See DOI: 10.1039/x0xx00000x alkenes with $TMSN_3$ and alkanes (eq 4, Scheme 1). Dichloromethane, chloroform and carbon tetrachloride could also take part in the reaction as precursors of chlorinated alkyl radicals to afford interesting γ-azido chloroalkanes. Importantly, the reaction is applicable not only to styrene and its derivatives, known to be prone to undergo facial radical polymerization, but also to electron-poor olefins.

We began our reaction optimization using styrene (**1a**), cyclohexane (**2a**, BDE of C-H: 99.5 kcal/mol) and $TMSN_3$ as the model substrates (Table 1). In the presence of di-*tert*-butyl peroxide (DTBP), the reaction afforded the desired carboazidation product **3a** in only 7% yield accompanied by styrene oxide and benzaldehyde (*c* 0.2, entry 1). Adding $FeCl_3$ (10 mol%) to the reaction mixture raised the yield of **3a** to 29% (entry 2). Subsequent survey of different iron and copper salts (entries 3-8) indicated that $Fe(acac)_3$ was the most effective one to provide **3a** in 53% yield. Performing the reaction at lower concentration (*c* 0.13 M) further increased the reaction efficiency (entry 9). Replacing DTBP by *tert*-butyl hydroperoxide (TBHP), benzoyl peroxide (BPO) and dicumylperoxide (DCP) under otherwise identical conditions furnished **3a** in diminished yield (entries 10-12). Potassium persulfate ($K_2S_2O_8$) was an ineffective oxidant for the desired transformation (entry 13). Importantly, decreasing the loading of $Fe(acac)_3$ from 10 to 0.5 mol% afforded **3a** in similar yield (entries 17 *vs* 14). Overall, optimum conditions consisted of performing the reaction of **1a**, with $TMSN_3$ in cyclohexane (*c* 0.13 M) at 100 °C in the presence of a catalytic amount of $Fe(acac)_3$ (0.5 mol%). Under these conditions, the three-component adduct **3a** was isolated in 74% yield.

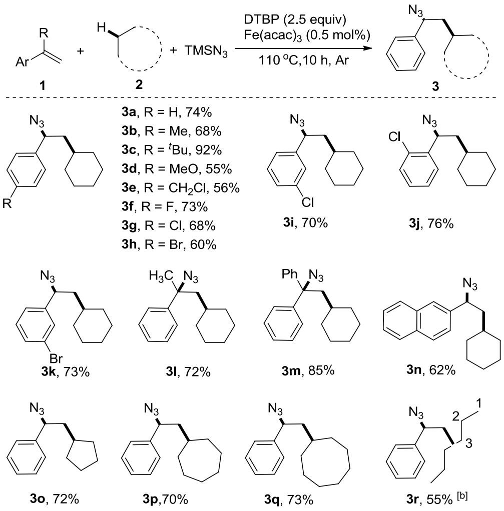
Table 1. Optimization of reaction conditions. [a]



entry	Cat. (mol%)	Peroxide (equiv)	**2a** (mL)	Yield (%)[b]
1	-	DTBP (3.0)	1.0	7
2	$FeCl_3$ (10)	DTBP (3.0)	1.0	29
3	$FeCl_2$ (10)	DTBP (3.0)	1.0	< 2
4	$Fe(acac)_3$ (10)	DTBP (3.0)	1.0	53
5	$Fe(acac)_2$ (10)	DTBP (3.0)	1.0	42
6	$Fe(OAc)_2{\cdot}4H_2O$(10)	DTBP (3.0)	1.0	n.d.
7	CuCl (10)	DTBP (3.0)	1.0	n.d.
8	$Cu(OTf)_2$ (10)	DTBP (3.0)	1.0	n.d.
9	$Fe(acac)_3$ (10)	DTBP (3.0)	1.5	68
10	$Fe(acac)_3$ (10)	TBHP (3.0)	1.5	17
11	$Fe(acac)_3$ (10)	DCP (3.0)	1.5	37
12	$Fe(acac)_3$ (10)	BPO (3.0)	1.5	35
13	$Fe(acac)_3$ (10)	$K_2S_2O_8$ (3.0)	1.5	n.d.
14	$Fe(acac)_3$ (10)	DTBP (2.5)	1.5	73
15	$Fe(acac)_3$ (5)	DTBP (2.5)	1.5	72
16	$Fe(acac)_3$ (1)	DTBP (2.5)	1.5	72
17	**$Fe(acac)_3$ (0.5)**	**DTBP (2.5)**	**1.5**	**74**
18	$Fe(acac)_3$ (0.25)	DTBP (2.5)	1.5	69

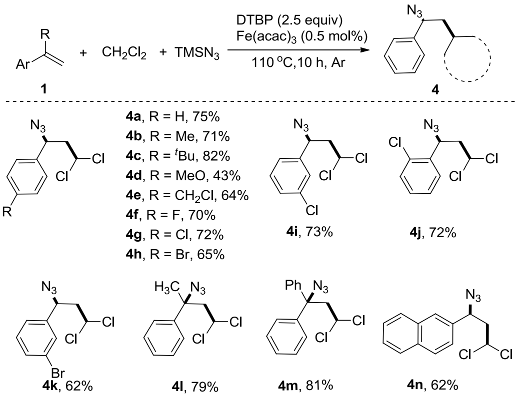
[a] Reaction conditions: styrene (**1a**, 0.2 mmol, 1.0 equiv), cyclohexane (**2a**), $TMSN_3$ (0.5 mmol, 2.5 equiv), oxidant (equiv), catalyst (mol%), 110 °C, 10 h, under Ar. [b] yield of isolated product.

The generality of this azidoalkylation of alkenes were next investigated. As shown in Scheme 2, styrenes bearing an electron donating (Me, *t*Bu, MeO) or withdrawing groups (F, Cl, Br) at the *para, meta and ortho* position of the phenyl ring were transformed smoothly to the desired alkylated azides (**3a**-**3j**) in good to high yields. Note that the benzyl chloride function remained untouched under these conditions (**3e**). The α-methylstyrene, 1,1-diphenylethylene and 2-vinyl naphthalene took part in the reaction to provide the corresponding three-component adducts (**3l**-**3n**). Cyclohexene was tried under the optimized conditions but was not reactive and no similar carboazidation product could be detected, which implied that the substrate of this reaction limited to terminal alkenes. Other cyclic alkanes such as cyclopentane (**2b**, BDE of C-H: 95.6±1.0 kcal/mol), cycloheptane (**2c**, BDE of C-H: 94.0 kcal/mol) and cyclooctane (**2d**, BDE of C-H: 95.7 kcal/mol) participated in the reaction to deliver the corresponding adducts (**3o**-**3r**) in good yields. As expected, when linear alkane such as *n*-hexane (**2e**) was used in this cascade reaction, a mixture of regioisomers was obtained.



Scheme 2. [a] Reaction conditions: alkene (**1**, 0.2 mmol), alkane (**2**, 1.5 mL), $TMSN_3$ (0.5 mmol, 2.5 equiv), DTBP (0.5 mmol, 2.5 equiv), $Fe(acac)_3$ (0.5 mol %), 110 °C, 10 h, under Ar. [b] a mixture of C1, C2 and C3 regioisomers.

The use of CH_2Cl_2 (BDE of C-H: 97.3±1 kcal/mol) as the alkylating agent of the above carboazidation reaction was next examined. Gratefully, simply heating a CH_2Cl_2 solution of alkenes (*c* 0.13 M) and $TMSN_3$ in the presence of DTBP (2.5 equiv) and $Fe(acac)_3$ (0.005 equiv) afforded the γ-azido dichloroalkanes in good to excellent yields (Scheme 3). Reaction turned out to be generally applicable to a diverse set of styrenes with different electronic properties (**4a**-**4k**). 1,1-Disubstituted ethylenes and 2-vinylnaphthalene were converted to the corresponding γ-azido chloroalkanes (**4l**-**4n**) in good yields.



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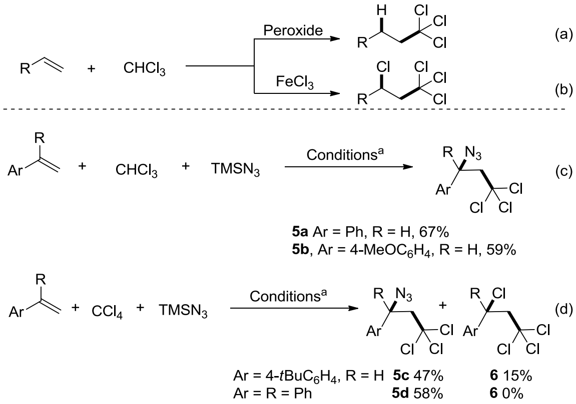
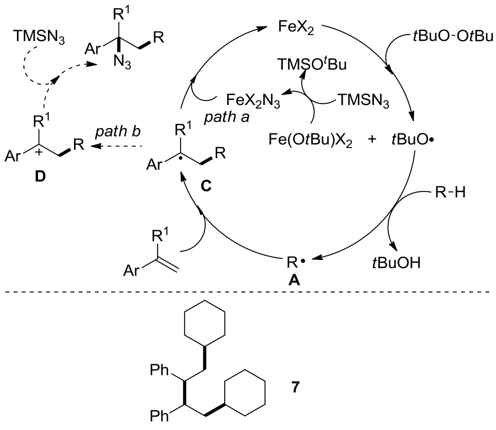
Scheme 3 [a] Reaction conditions: alkene (0.2 mmol), CH_2Cl_2 (1.5 mL), $TMSN_3$ (0.5 mmol, 2.5 equiv), DTBP (0.5 mmol, 2.5 equiv), $Fe(acac)_3$ (0.5 mol %), 110 °C, 10 h, under Ar.

Reaction of alkenes with chloroform (BDE of C-H: 93.8±0.6 kcal/mol) in the presence of peroxide is known to afford 1,1,1-trichloroalkanes (Kharasch reaction, Scheme 4a).[19] Minisci[20] and Asscher[21] demonstrated that in the presence of an iron salt, the same reaction can be diverted to afford the 1,1,3-trichloroalkanes. Notwithstanding these precedents, the reaction of styrene with $TMSN_3$ in $CHCl_3$ under our standard conditions furnished the 1,1,1-trichloro-3-azido-3-phenylpropane (**5a**) in 67% yield indicating that the reaction went through a different reaction manifold in the presence of both peroxide and iron salt. The 4-methoxystyrene was similarly converted to **5b** in good yield (Scheme 4c). Interestingly, carbon tetrachloride also took part in the reaction to afford **5c** in 47% yield together with 1,1,1,3-tetrachloroalkane **6** (15%).[22] When diphenylethylene was used, alkyl azide **5d** was formed exclusively. These results seems to indicate that azido-transfer to the benzylic radical was faster than the competitive Cl-transfer process under our conditions.



Scheme 4. Azidoalkylation of alkenes using $CHCl_3$ and CCl_4 as alkyl sources. [a] alkene (0.2 mmol), $CHCl_3$ or CCl_4 (1.5 mL), $TMSN_3$ (0.5 mmol, 2.5 equiv), DTBP (0.5 mmol, 2.5 equiv), $Fe(acac)_3$ (0.5 mol %), 110 °C, under Ar.

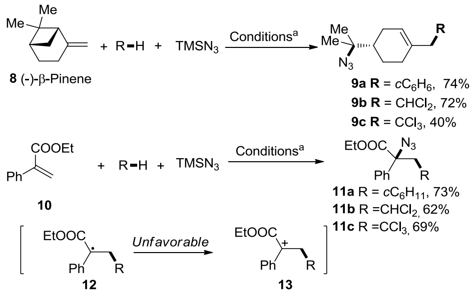
A possible reaction pathway is depicted in Scheme 5. Reduction of DTBP by in situ generated Fe(II) salt would produce *tert*-butoxy radical and $Fe(OtBu)X_2$. Intermolecular hydrogen abstraction from alkane by *t*BuO• (BDE of O-H: 106.3±0.7 kcal/mol) would generate alkyl radical **A** which, upon addition to alkene, would provide the radical **C**. On the other hand, reaction of Fe(III) salt with $TMSN_3$ would produce the $Fe(III)N_3$ salt, which would then transfer the azido radical to **C** leading to the three-component adduct with concurrent regeneration of the Fe(II) salt (path a). Several experimental observations were in line with the proposed reaction pathway. Firstly, reaction of **1a** with $TMSN_3$ in cyclohexane was completely inhibited in the presence of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) and the formation of 1-(cyclohexyloxy)-2,2,6,6-tetramethylpiperidine was observed (see ESI). Secondly, dimer **7**, resulting most probably from the dimerization of benzyl radical **C**, was isolated in some of our initial experiments due to the inefficient azido transfer process. The formation of Kharasch's adduct **6** (Scheme 4b) was also in accord with the presence of intermediate **C**. The fact that the reaction of (-)-β-pinene with cyclohexane, CH_2Cl_2 and $CHCl_3$ afforded the ring opened products **9a**-**9c** (Scheme 6) supported further the proposed radical mechanism.



Scheme 5. Proposed mechanism for the cascade reaction.

Oxidation of the benzylic radical followed by trapping of the resulting carbocation **D** by azide could also account for the formation of the three-component adduct (path b). To gain further mechanistic insights, azidoalkylation of ethyl phenylacrylate (**10**) was examined. As is shown in Scheme 6b, compound **10** was converted under standard conditions to the adducts **11a**-**11c** in good yields. Since it is reasonable to assume that oxidation of benzyl radical **12** to benzyl cation **13** would be an energetically unfavourable process due to the presence of the adjacent electron-withdrawing ester group, the azidation might proceed via Fe-mediated azide transfer process (path a, Scheme 5). We note that compounds **11** are interesting precursor of 2-substituted phenylglycine derivatives.

It is known that azide radical can be generated in the presence of peroxide and iron salt. Azide radical, being moderately electrophilic, should be more reactive than nucleophilic alkyl radicals towards the electron rich styrenes, leading to diazidation product.[20] This is however not the case and we think that iron might also coordinate to double bond modifying therefore its electronic properties.



Scheme 6. Examples with mechanistic implication. Conditions (See foot note of schemes 2-4).

The azides can be subjected to further chemical manipulations as depicted in Scheme 7. The Pd/C-catalyzed hydrogenation of azide **4a** provided the chlorinated benzyl amine **4** that is armed for the further functionalization at both the C1 and C3 positions. The CuI-catalyzed "Click" reaction of **3a** with phenylacetylene produced triazole **15** in 84% yield without event.

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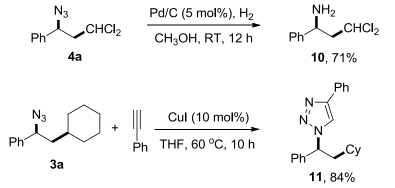

Scheme 7. Transformation of the azido function.

Conclusions

We have developed a novel Fe-catalyzed, DTBP-mediated carboazidation of alkenes using cycloalkanes, CH_2Cl_2, $CHCl_3$ and CCl_4 as alkylating reagents. The reaction provided chain elongated benzyl azides and γ-azido chloroalkanes in good to high yields. The reaction is unique since it is applicable not only to diversely substituted styrenes, known to be prone to polymerization, but also to electron-poor olefins.

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Abstract for the Contents Pages

Fe-catalyzed three-component carboazidation of alkenes with alkanes and trimethylsilyl azide

Wei-Yu Li, Chuan-Shuo Wu, Wang Zhou, Luo Yang*

Text:

Iron out the difference *Carboazidation of alkenes using cycloalkanes, CH_2Cl_2, $CHCl_3$ and CCl_4 as alkylating reagents proceeded smoothly in the presence of $TMSN_3$, DTBP and a catalytic amount of $Fe(acac)_3$ to afford the chain extended alkyl azides and γ-azido chloroalkanes in good to high yields.*

Graphic: