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Site-Specific Oxidation of $(sp^3)C-C(sp^3)/H$ Bonds by $NaNO_2/HCl$

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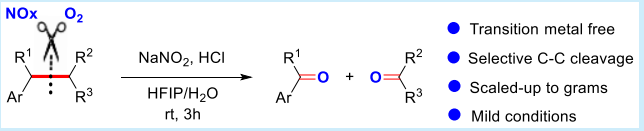
ABSTRACT: A site-specific oxidation of $(sp^3)C-C(sp^3)$ and $(sp^3)C-H$ bonds in aryl alkanes by the use of $NaNO_2/HCl$ was explored. The method is chemical-oxidant-free, transition-metal-free, uses water as the solvent, and proceeds under mild conditions, making it valuable and attractive to synthetic organic chemistry.



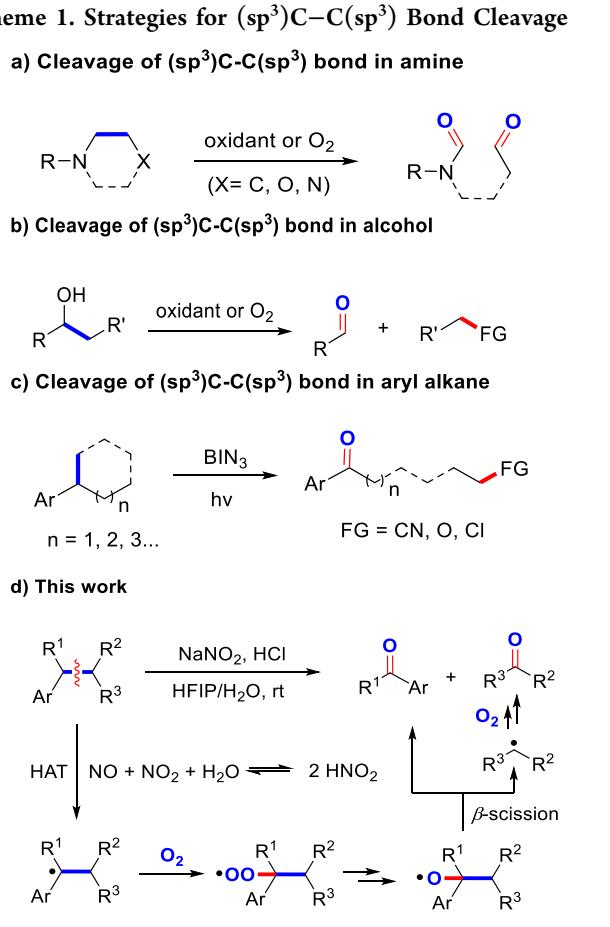
Exploration of highly efficient strategies for C−C bond conversion would benefit biomass transformation, polymer degradation, material recycling, etc.[1] In the past decades, considerable developments in functionalizing relatively active C−C bonds have been made.[2] However, effective methods for cleavage of inert $(sp^3)C-C(sp^3)$ bonds have remained undeveloped to date.

Recently, a few elegant strategies for selective cleavage of specific $(sp^3)C-C(sp^3)$ bonds in amines,[3] alcohols,[4] and aryl alkanes[5] have been achieved (Scheme 1a−c). Nevertheless, most of these systems suffer from excess chemical oxidants, limited substrate scope, or hash conditions.

Inspired by previous reports and our efforts on C−C bond conversions,[6] we continued to explore more efficient systems. On the basis of our previous studies of the chemistry of nitric oxide (NO) and nitrogen dioxide (NO_2),[7] we hypothesized that these gaseous radicals might serve as primary radicals that further initiate oxidative C−C bond cleavage (Scheme 1d). Decomposition of nitrous acid would liberate NO and NO_2, which would selectively abstract a hydrogen atom from the aryl alkane to give a relatively stable benzyl radical. This would capture molecular O_2 to generate a peroxide radical. Hydrogen atom transfer followed by homolysis of the peroxide radical would produce an alkoxyl radical. Subsequently, β-fragmentation of alkoxyl radical gives rise to the aryl ketone and an alkyl radical, which would react with molecular oxygen to afford another ketone. Fortunately, we realized this assumption and discovered a simple protocol to activate $(sp^3)C-C(sp^3)$ bonds attached to arenes (Scheme 1d).

To test the hypothesis and modify the conditions, we used 1-butoxy-4-isopropylbenzene (**1a**) as a model substrate (Table 1). We found that the efficiency of this reaction heavily depended on the solvent and the amounts of $NaNO_2$ and HCl. Initially, the desired ketone **1b** and a byproduct **1b′** were obtained by the use of 3 equiv of $NaNO_2$ and 5 equiv of HCl in hexafluoroisopropanol (HFIP) at 40 °C (entry 1). Through careful variation of the acid, solvent, and temperature, we got the product in 70% yield (entries 2−11), and the nitrated product was decreased less than 10%. As expected, nitrated

Scheme 1. Strategies for $(sp^3)C-C(sp^3)$ Bond Cleavage



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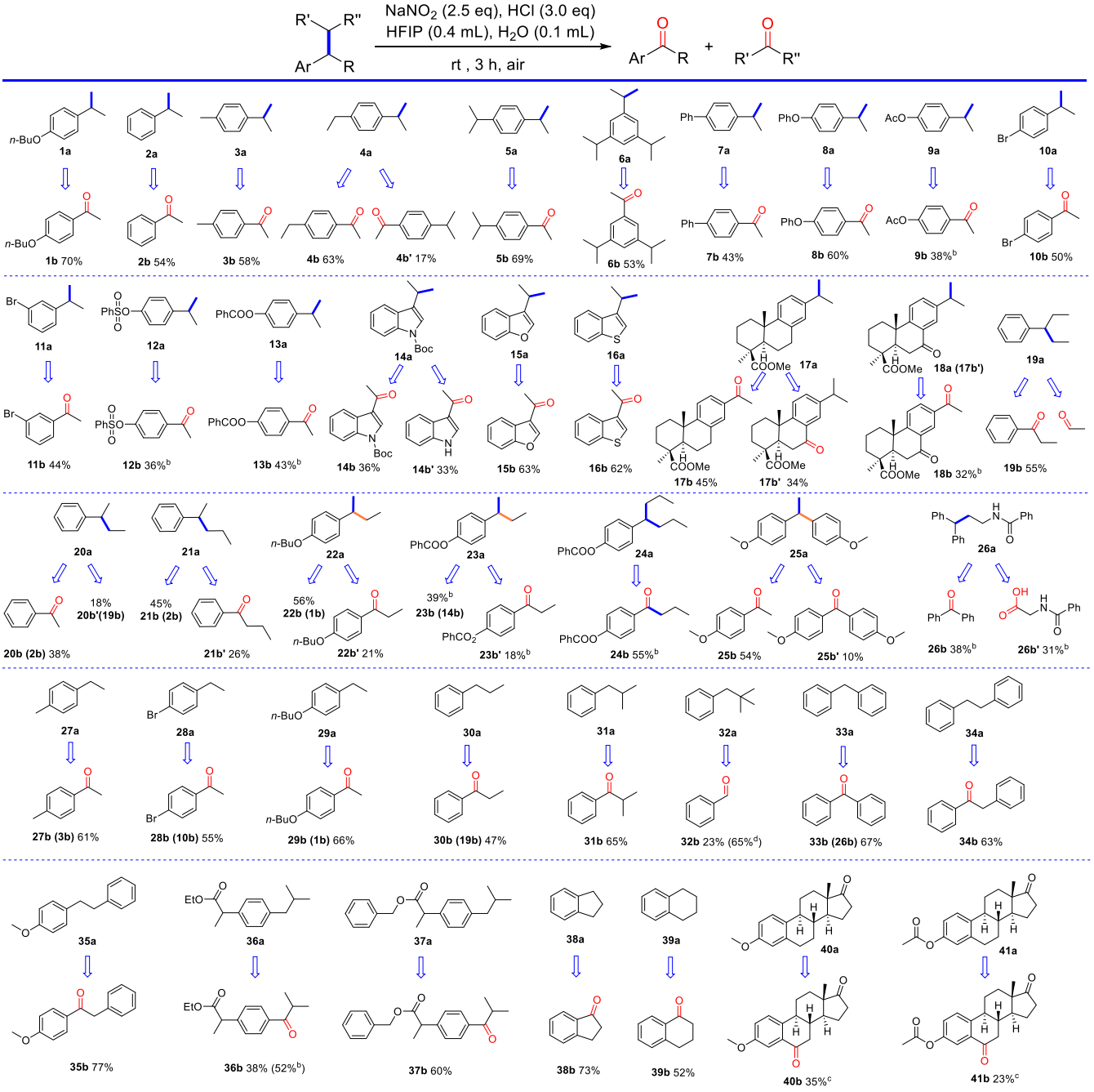
Scheme 2. Substrate Scope[a]



[a]Reaction conditions: 0.4 mmol of substrate, 2.5 equiv of $NaNO_2$, 3.0 equiv of HCl, 4:1 HFIP/H_2O (0.5 mL), room temperature, 3 h. Isolated yields are shown. [b]60 °C, 6 h. [c]4:1 HFIP/H_2O (1.0 mL), 60 °C, 12 h. [d]^{1}H NMR yield.

compound **1b′** became the major product when the reaction was conducted under an atmosphere of N_2 (entry 12).

With the optimized conditions in hand, we began to examine the substrate scope (Scheme 2). We found that a wide range of aryl alkanes are compatible with this system. First, aryl alkanes with a tertiary benzylic carbon gave the corresponding (sp^3)C−C(sp^3) bond cleavage products in moderate to good yields (**1a**−**26a**). It is interesting that 1-ethyl-4-isopropylbenzene (**4a**) afforded 1-(4-ethylphenyl)ethan-1-one (**4b**) and 1-(4-isopropylphenyl)ethan-1-one (**4b′**) in 63% and 17% yield, respectively. In addition, alkyl heteroarenes such as alkyl indole, benzofuran and benzothiophene were also effective substrates (**14a**−**16a**). Furthermore, it is noteworthy that some complex natural products were also amenable to this reaction. For example, methyl dehydroabietate (**17a**) gave rise to a C−C cleavage product (**17b**) as well as a methylene oxidation product (**17b′**). In order to evaluate an electron-poor substrate, we put **17b′** (**18a**) into the system, and the desired product **18b** was isolated in 32% yield. To confirm the other products derived from the released alkyl group, a GC−MS detection experiment for **19a** was carried out. As expected, propiophenone (**19b**) and acetaldehyde were observed as the major products in this reaction (see the Supporting Information for more details). Next, an array of aryl alkanes

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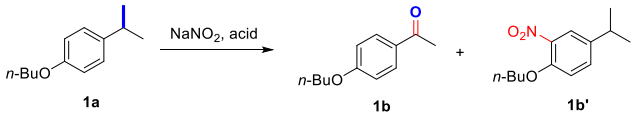
Table 1. Optimization of the Reaction Conditions[a]



entry	reagents (equiv), atmosphere, temperature	solvent (mL)	RSM[b] (%)	yield of **1b** (%)[c]	yield of **1b′** (%)[c]
1	$NaNO_2$ (3.0), HCl (5.0), air, 40 °C	HFIP (0.5)	0	48	15
2	$NaNO_2$ (3.0), HCO_2H (5.0), air, 40 °C	HFIP (0.5)	45	19	12
3	$NaNO_2$ (3.0), TFA (5.0), air, 40 °C	HFIP (0.5)	0	37	23
4	$NaNO_2$ (3.0), HCl (5.0), air, 40 °C	CH_3CN (0.5)	53	17	12
5	$NaNO_2$ (3.0), HCl (5.0), air, 40 °C	DCM (0.5)	32	13	31
6	$NaNO_2$ (3.0), HCl (5.0), air, 40 °C	TFE (0.5)	37	25	12
7	$NaNO_2$ (3.0), HCl (5.0), air, 40 °C	1:1 HFIP/H_2O (0.5)	10	37	35
8	$NaNO_2$ (3.0), HCl (5.0), air, 40 °C	4:1 HFIP/H_2O (0.5)	0	59	15
9	$NaNO_2$ (1.0), HCl (2.0), air, 40 °C	4:1 HFIP/H_2O (0.5)	27	33	21
10	$NaNO_2$ (2.5), HCl (3.0), air, 40 °C	4:1 HFIP/H_2O (0.5)	0	67	<10
11	$NaNO_2$ (2.5), HCl (3.0), air, 23 °C	4:1 HFIP/H_2O (0.5)	0	70	<10
12	$NaNO_2$ (2.5), HCl (3.0), N_2, 23 °C	4:1 HFIP/H_2O (0.5)	16	<10	64

[a]Reaction conditions: 0.4 mmol of substrate, 3 h. [b]RSM denotes recovered starting material. [c]Isolated yields.

were examined to demonstrate the selectivity in the C−C bond cleavage (**20a**−**26a**). We discovered that the cleavage of the C−C bond always occurred to liberate a longer alkyl chain (**20a**−**23a**), which should be due to the stability of the released alkyl radical. 4,4′-(Ethane-1,1-diyl)bis(methoxybenzene) (**25a**) generated 1-(4-methoxyphenyl)ethan-1-one (**25b**) and bis(4-methoxyphenyl)methanone (**25b′**) in 54% and 10% yield, respectively, indicating that the aryl radical should be more stable than the methyl radical but less stable than the long-chain alkyl radical (**26a**).

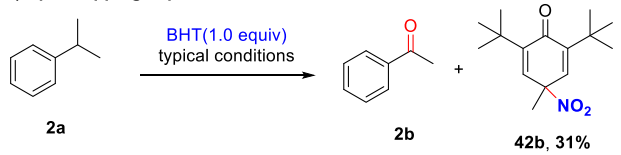
Next, a series of aryl alkanes with a benzylic methylene group were examined (**27a**−**41a**), and the corresponding aryl ketones were obtained smoothly. It is worth noting that benzaldehyde was isolated as the major product when neopentylbenzene (**32a**) was used as the substrate. This result might reveal two interesting issues. One is that the four-membered peroxide ring should be ruled out as the critical intermediate for the C−C bond cleavage since it could not be formed in this case. The other is that C−C bond cleavage should occur preferentially over methylene oxidation if the leaving alkyl radical is stable. Both linear and cyclic aryl alkanes were compatible with this system. Especially, complex natural products (**40a** and **41a**) also gave the corresponding products under the reaction conditions. However, strongly electron-deficient aryl alkanes were not compatible with this method.

Finally, we carried out several experiments to verify the proposed mechanism (Scheme 3). As shown in Scheme 3a, when 2,6-di-*tert*-butyl-4-methylphenol (BHT) was added to the reaction, **42b** was isolated in 31% yield, which indicated that NO_2 radical should be involved in this system. When 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) was used as an alternative spin trapping agent, radical adduct **43b** was isolated in 16% yield, which suggested that a benzylic-carbon-centered radical should be generated in this process. Besides, the desired product **2b** was not observed in either reaction. These results support the possible mechanism given in Scheme 1d.

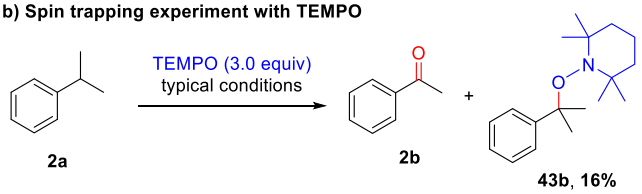
In summary, we developed a $NaNO_2$/HCl promoted oxidation of $(sp^3)C-C(sp^3)$ and $(sp^3)C-H$ bonds in aryl alkanes. This process is transition-metal-free and chemical-oxidant-free and proceeds under mild conditions, which makes it attractive to synthetic organic chemistry.

Scheme 3. Mechanistic Studies

a) Spin trapping experiment with BHT



b) Spin trapping experiment with TEMPO



■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.1c01303.

Experimental procedures, mechanistic studies, and characterization and spectral data (PDF)

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Notes

The authors declare no competing financial interest.

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