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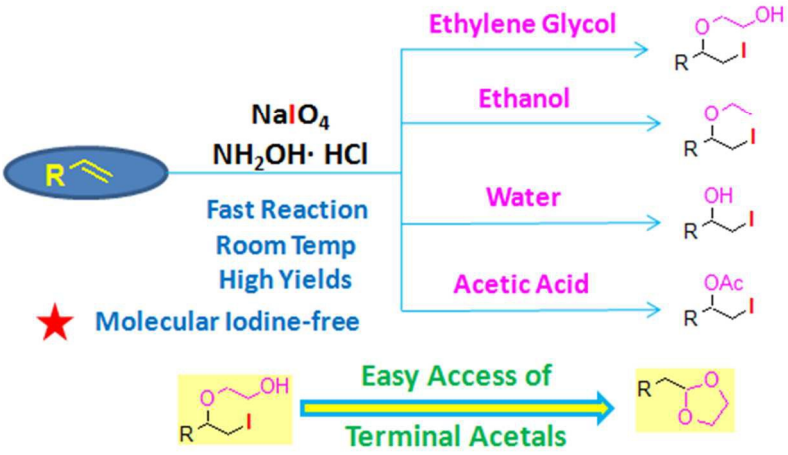

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ARTICLE TYPE

Combination of $NH_2OH·HCl$ and $NaIO_4$: an effective reagent for molecular iodine-free regioselective 1,2-difunctionalization of olefins and easy access of terminal acetals†

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We have demonstrated a new application of our oxidizing reagent, combination of $NH_2OH·HCl$ and $NaIO_4$, in the first generalized regioselective 1,2-difunctionalization of olefins. It is a general method for the preparation of *β*-iodo-*β′*-hydroxy ethers, *β*-iodo ethers, *β*-iodohydrin, and *β*-iodo acetoxy compounds using different reaction media. The reactions are highly regioselective; always afford Markovnikov's type addition products. The methodology is also applicable for the easy access of terminal acetals. Molecular iodine-free synthesis, room temperature reaction conditions, high yields, use of less expensive reagents, mild reaction conditions, broad applicability of nucleophiles, and applicable for gram-scale synthesis are the notable advantages of this present protocol.

Introduction

Reaction of alkenes mainly difunctionalization is a finest approach adopted in organic synthesis. It has been widely studied and utilized in various techniques for functional groups interconversions.[1] Among them the halohydrin, *β*-iodo ether and *β*-iodo acetoxy compounds play a crucial role in the field of drug scaffolds, synthetic organic chemistry, medicinal and industrial chemistry as well as material sciences.[2] They are also the key intermediates in the synthesis of several halogenated marine natural products.[2] The vicinal dihaloalkanes are formed by the electrophilic halogenation of alkenes.[3] When the halogenation of the alkene is carried out in a nucleophilic solvent such as water, alcohols, carboxylic acids, nitriles, etc., difunctionalized products like halohydrins, *β*-haloethers, *β*-haloesters, etc. are obtained. This process is known as 'cohalogenation' and this is very important strategy to provide useful products for diverse synthetic applications.[4]

The formation of halohydrins from alkenes is a well-established method.[5] On the other hand, halohydrins are also useful intermediates in epoxide synthesis in both laboratory and industrial scales.[6] The formation of chlorohydrins and bromohydrins by the reaction of alkenes and dilute aqueous solutions of the halogens undergoes smoothly[7] but the formation of iodohydrins is not so smooth because of the reversibility of the addition of iodine to the double bond. An iodide ion scavenger such as $AgNO_3$, HgO,[8] $CuO·HBF_4$[9] or an oxidizing agent[10] is essential for the formation of iodohydrins. The ring-opening of epoxides by hydrogen halides is also the most common procedure for the preparation of halohydrins. Hydrogen halides and hypohalite-water are the conventional reagents for epoxide ring opening to halohydrins.[11] The main disadvantage of the methodologies for synthesis of halohydrins is to synthesize the epoxide first by employing the traditional methodologies like the reaction of alkenes with peracids/bases,[12] O_2 or H_2O_2 using a metal-based catalytic system[13] or zeolites,[14] H_2O_2/auxiliaries (nitriles, carbodiimides, etc.).[15] Few methodologies have also been reported for the preparation of halohydrin compounds using mild reaction conditions with limited nucleophiles.[16]

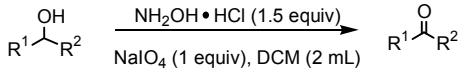
β-Iodo ethers are important intermediates for stereoselective radical reactions[17] as well as synthesis of *E*- or *Z*-alkenes with good to moderate diastereoselectivity.[18] A number of methodologies for the synthesis of this important framework has been developed by various groups. Among these the most important approaches are the reactions of alkenes with $I(py)_2BF_4$,[19] excess amount of iodine,[20] diacetoxyiodine(I) complexes,[21] I_2/clays,[22] I_2/ultrasound,[23] *N*-haloimides,[24] *N*-halosaccharin,[25] *N*-iodosuccinimide/alcohols,[26] triiodoisocyanuric acid[27] and IBX-I_2.[28]

Regardless of their efficiency and reliability, most of these methods suffer from one or more of these disadvantages such as using expensive reagents and catalysts, long reaction times, requirement of inert atmosphere, harsh reaction conditions and mostly use of molecular iodine as iodo source. Although molecular iodine is a versatile reagent in organic synthesis; it is highly corrosive, toxic, and sublimable, making its use somewhat unattractive.[29] Again, it is important to note that all these methods[8-28] are not general for the preparation of halohydrin, *β*-iodo ether and *β*-iodo acetoxy compounds using the same reaction conditions; varying the nucleophilic medium like water, alcohol, carboxylic acid etc. respectively. Therefore, finding a general and efficient methodology for the synthesis of iodohydrins and *β*-iodo ethers in terms of using basic chemicals

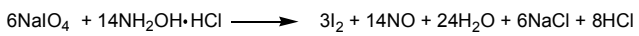
as starting materials, increasing efficiency, operational simplicity, mild reaction conditions, and economic practicability is highly desirable.

In continuation of our research in organic synthesis[30] herein, we report a mild and efficient approach for the regioselective synthesis of various *β*-iodo-*β′*-hydroxy ethers, *β*-iodo ethers, iodohydrins, and *β*-iodo acetoxy compounds from alkenes using the combination of $NaIO_4$ and $NH_2OH{\cdot}HCl$ at room temperature within a short reaction time (Scheme 1, b). Recently we have reported a mild and efficient approach for oxidation of alcohols to corresponding carbonyl compounds (Scheme 1, a).[31] Based on this report we can suggest that the *in situ* generated iodine undergoes the addition to the double bond to form iodonium ion which in presence of nucleophilic solvents like alcohols, water, carboxylic acids etc. might afford the corresponding *β*-iodo-*β′*-hydroxy ether, *β*-iodo ethers, iodohydrins, and *β*-iodo acetoxy compounds.

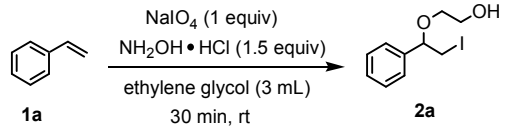
(a) Our previous approach:



Proposed equation for *in situ* generation of iodine:



(b) This work:

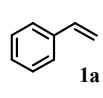
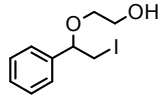
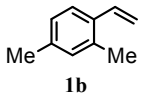
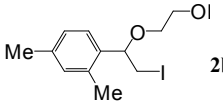
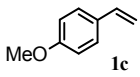
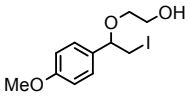
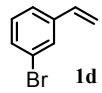
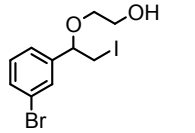
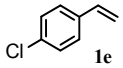
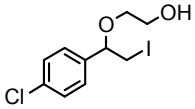
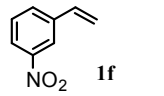
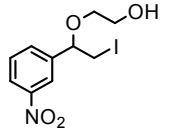
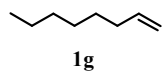
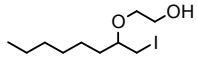
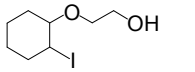
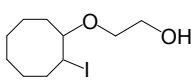
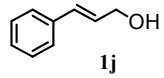
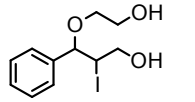
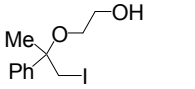
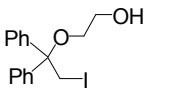


Scheme 1 Reaction of styrene with ethylene glycol to synthesize *β*-iodo-*β′*-hydroxy ether.

Results and discussion

During our initial study, readily available styrene **1a** was taken as a model substrate using $NaIO_4$ (2 equiv) and $NH_2OH{\cdot}HCl$ (4 equiv) as reagent in ethylene glycol solvent. The reaction proceeded smoothly at room temperature and the product 2-(2-iodo-1-phenylethoxy)ethanol (**2a**) was isolated in 86% yield within 30 min. Encouraged by this result our attention was focused on the optimization of the reagents ratios. First of all, we used 1:1 proportion of $NaIO_4$ and $NH_2OH.HCl$ and 68% of desired product (**2a**) was observed. By increasing the proportion of $NH_2OH.HCl$ from 1 to 1.5, the desired product (**2a**) was increased to 87% yield. The maximum amount of yield was obtained by using 1:1.5 ratios of $NaIO_4$ and $NH_2OH.HCl$ respectively. Further increasing the amount of both the reagents the yield of the product did not improve significantly.

Table 1 Substrate scopes to synthesize *β*-iodo-*β′*-hydroxy ethers[a]

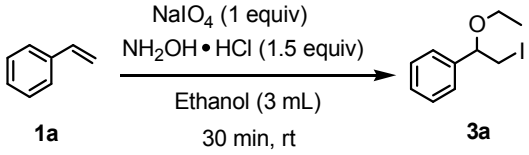
Entry	Substrates (**1**)	Products (**2**)		Yields (%)[b]
1	 **1a**		**2a**	87, 80[c]
2	 **1b**		**2b**	83
3	 **1c**		**2c**	81
4	 **1d**		**2d**	87
5	 **1e**		**2e**	86
6	 **1f**		**2f**	81
7	 **1g**		**2g**	80
8	 **1h**		**2h**	82[d]
9	 **1i**		**2i**	80[d]
10	 **1j**		**2j**	85
11	 **1k**		**2k**	82
12	 **1l**		**2l**	81

[a] All reactions were performed on a 1 mmol scale in presence of $NaIO_4$ (1 mmol) and $NH_2OH{\cdot}HCl$ (1.5 mmol) in 3 mL of ethylene glycol at room temperature for 30 min. [b] Isolated yields. [c] styrene **1a** (10 mmol), $NaIO_4$ (10 mmol), $NH_2OH.HCl$ (15 mmol) in 30 mL of ethylene glycol at room temperature for 30 min. [d] *cis* product.

After optimizing the reaction conditions the scope and limitations of this reaction were investigated (Table 1). Our attention was focused on the use of different olefinic systems to prove the general applicability of the reaction conditions. It was observed that electron-rich and electron-deficient styrenes reacted efficiently with ethylene glycol to afford the desired products with good yields under the present reaction conditions. The styrene containing an electron donating Me & OMe group on the aromatic ring showed good efficiency (**2b** & **2c**). The bromo- and chloro-substituted styrenes gave the corresponding **2d** and **2e** in 87% and 86% yields respectively without forming any

dehalogenated products. Other electron withdrawing substituent NO_2 group on styrene moiety afforded the desired product with satisfactory yield (**2f**). In addition, aliphatic olefinic systems were also found to afford the desired products with high yields (**2g-2i**). Our present protocol is also effective for cinnamyl alcohol to produce the corresponding *β*-iodo-*β′*-hydroxy ether (**2j**). We are pleased to notice that *α*-methyl styrene and 1,1-diphenylethylene both gave the desired products (**2k** & **2l**) with good yields under the stated reaction conditions. However, sodium 4-vinyl benzenesulfonate, β-methyl-β-nitrostyrene, and cholesterol did not give the corresponding iodoethers under the present reaction conditions. This methodology is also applicable on a gram-scale synthesis. We have successfully prepared the iodoether **2a** in 80% yield by the reaction of styrene (**1a**, 10 mmol) in ethylene glycol. In general, all the reactions were clean and *β*-iodo-*β′*-hydroxy ethers were found to be furnished regioselectively in all cases.

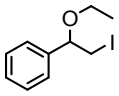
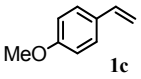
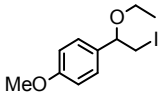
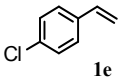
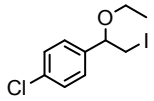
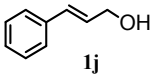
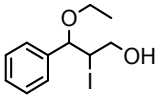
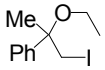
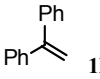
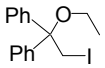
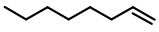
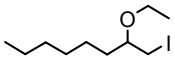
Next, we explored our present methodology using ethanol as other nucleophilic solvent to react with olefinic systems to synthesize various *β*-iodo ethers (Scheme 2). To our delight the corresponding *β*-iodo ethers (**3**) were obtained regioselectively in good yields; the results are summarized in Table 2.



Scheme 2 Synthesis of *β*-iodo ether using ethanol.

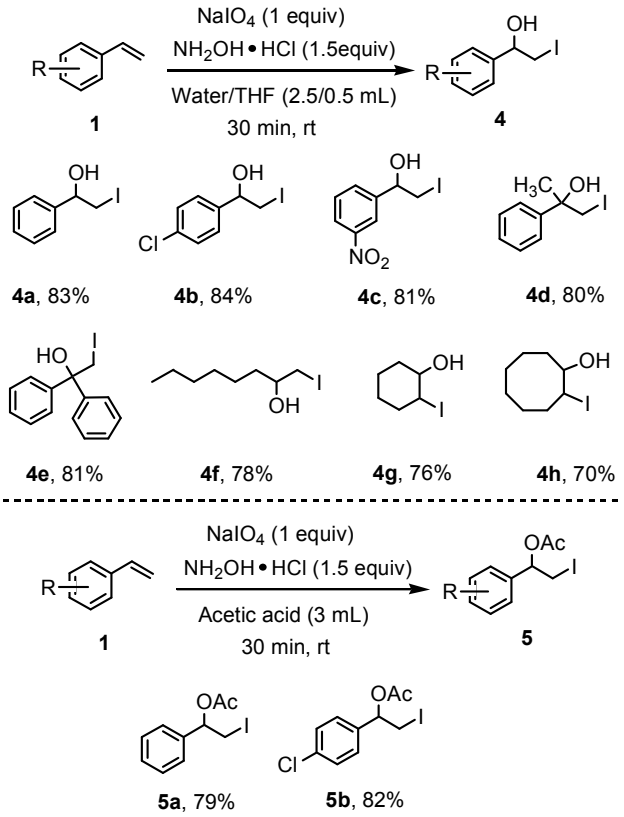
Simple styrene reacted well to give the desired *β*-iodo ether with high yield (**3a**). Styrenes substituted by electron donating OMe group (**3b**) as well as electron withdrawing halogen group (**3c**) underwent smooth reactions which highlighted the wide scope of this reaction. Meanwhile, the effect of alcoholic group in the olefinic system also investigated. Cinnamyl alcohol can also afford the desired product with excellent yield (**3d**). *α*-Methyl styrene and 1,1-diphenylethylene also nicely participated in the reaction, yielding the corresponding products **3e** and **3f** in 83% and 80% yields respectively. Moreover, aliphatic alkene such as 1-octene also afforded the desired product (**3g**) with good yield which also proves the general applicability of this present protocol.

Table 2 Substrate scopes using ethanol[a]

Entry	Substrates (**1**)	Products (**3**)		Yields[b] (%)
1	 **1a**		**3a**	86
2	 **1c**		**3b**	81
3	 **1e**		**3c**	83
4	 **1j**		**3d**	85
5	 **1k**		**3e**	83
6	 **1l**		**3f**	80
7	 **1g**		**3g**	78

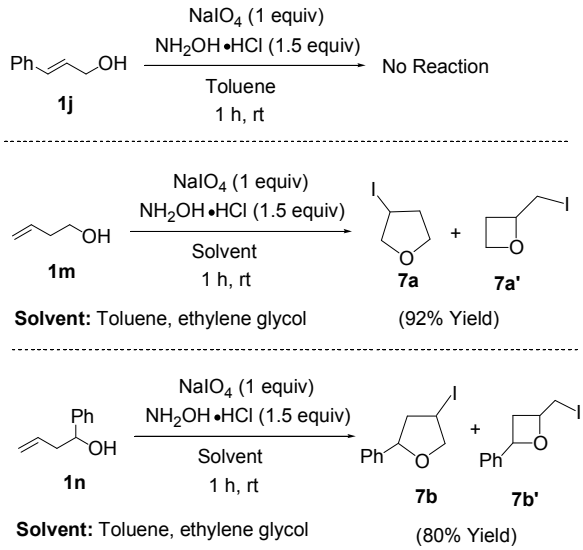
[a] All reactions were performed on a 1 mmol scale in presence of $NaIO_4$ (1 mmol) and $NH_2OH{\cdot}HCl$ (1.5 mmol) in 3 mL of ethanol at room temperature for 30 min. [b] Isolated yields.

General applicability of the methodology has further been established by using the different solvents (nucleophiles) for the synthesis of iodohydrin and *β*-iodoacetoxy compounds (Scheme 3). We have successfully used water and acetic acid to synthesize iodohydrin (**4**) and *β*-iodoacetoxy compounds (**5**) with good yields which increases the scope of this transformation. It is worthy to mention that a little amount of THF was added to water as solvent to synthesize the iodohydrins. The styrene, substituted with chloro- and nitro-substituent smoothly afforded the desired products (**4a**, **4b**, **4c**, **5a** & **5b**). We are delighted to inform that *α*-methyl styrene and 1,1-diphenylethylene both reacted well to give the corresponding iodohydrins (**4d** & **4e**). Aliphatic olefins such as 1-octene, cyclohexene and cyclooctene can also afford the desired iodohydrins (**4f-4h**) with good yields. However, β-methyl-β-nitrostyrene did not give the corresponding iodohydrin under this present reaction conditions.



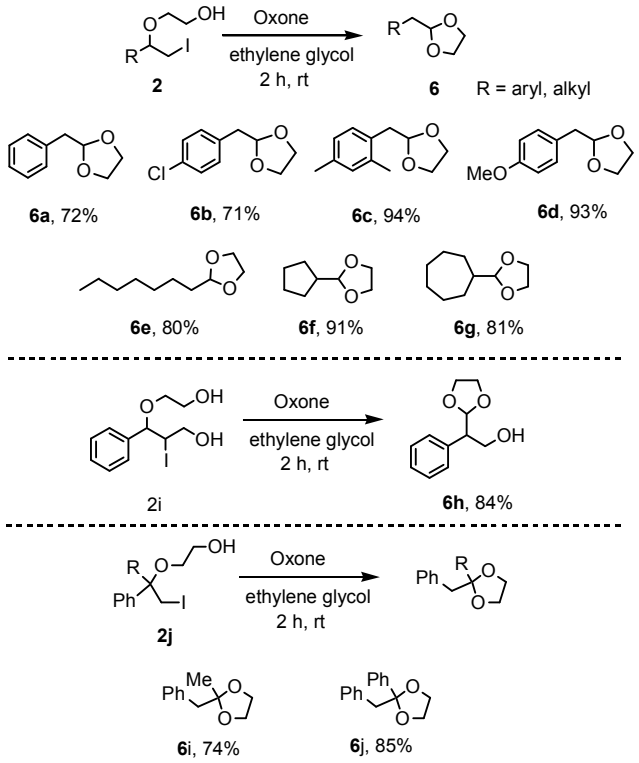
Scheme 3 Synthesis of iodohydrin and *β*-iodoacetoxy compounds.

Next we have examined the possibility of intramolecular cyclization reaction under the present reaction conditions (Scheme 4). We have found that cinnamyl alcohol (**1j**) gave no reaction either oxidation[31] to aldehyde or the intramolecular reaction by nucleophilic attack of alcoholic oxygen when the reaction has been carried out in non coordinating solvent like toluene. But it is worthy to mention that other homoallylic alcohols like **1m** and **1n** gave the cyclization products (**7a**, **7a′**, **7b** and **7b′**) in presence of toluene or ethylene glycol. We have got inseparable mixture of products with good yields.



Scheme 4 Additional experiments on intramolecular cyclization reaction.

Preparation of acetals at the terminal position of alkenes instead of aldehydes as substrate is a demanding task. Very few methods are available in literature using palladium,[32] iron[33] and a couple of nonmetal catalyzed methods.[34] Recently, Narender *et al.* reported a metal-free approach for the synthesis of terminal acetals by tandem oxidative rearrangement of olefins using oxone as an oxidant in the presence of iodine.[35] It is noteworthy to mention that our synthesized compound **2** is the key intermediate for synthesizing the terminal acetals. After successive reaction with *β*-iodo-*β′*-hydroxy ethers (**2**) using oxone as oxidant, various terminal acetals (**6a-6j**) were successfully synthesized by employing the reported method (Scheme 5).[35]



Scheme 5 Synthesis of terminal acetals from *β*-iodo-*β′*-hydroxy ethers.

Conclusions

In summary, we have developed a simple and general method for the synthesis of *β*-iodo-*β′*-hydroxy ether, *β*-iodo ether, *β*-iodo hydrin, and *β*-iodo acetoxy compounds using the combination of $NH_2OH{\cdot}HCl$ and $NaIO_4$ as iodine source at room temperature within a short reaction time using different solvents which act as nucleophiles. Furthermore, the *β*-iodo-*β′*-hydroxy ethers have been converted to terminal acetals using the reported method by using oxone as oxidant. The preparation of terminal acetals using *β*-iodo-*β′*-hydroxy ether is the very important functionalization of alkene as we can functionalize the germinal position by non-Wacker reaction. The advantages of this present protocol are: (i) molecular iodine-free synthesis, (ii) room temperature and mild reaction conditions, (iii) short reaction time, (iv) high yields, (v) use of less expensive reagents, (vi) applicable for broad solvent (nucleophile) systems, and (vii) applicable for gram-scale synthesis. These advantages render this protocol facile and suitable to create a diversified library of *β*-iodo-*β′*-hydroxy ether,

β-iodo ether, *β*-iodo hydrin, and *β*-iodo acetoxy compounds.

Experimental Section

General experimental methods

^{1}H NMR spectra were determined on a Bruker 400 (400 MHz) spectrometer as solutions in $CDCl_3$. Chemical shifts are expressed in parts per million (δ) and are referenced to tetramethylsilane (TMS) as internal standard and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet) and coupling constants *J* were given in Hz. ^{13}C NMR spectra were recorded at 100 MHz in $CDCl_3$ solution. TLC was done on silica gel coated glass slide (Merck, Silica gel G for TLC). Silica gel (60-120 mesh, SRL, India) was used for column chromatography. Petroleum ether refers to the fraction boiling in the range of 60-80 ºC unless otherwise mentioned. Melting points were determined on a glass disk with an electric hot plate and are uncorrected. All solvents were dried and distilled before use. Commercially available substrates were freshly distilled before the reaction. Solvents, reagents and chemicals were purchased from Aldrich, Fluka, Merck, SRL, Spectrochem and Process Chemicals. All reactions involving moisture sensitive reactants were executed using oven dried glassware.

Typical procedure for the synthesis of compound 2

A mixture of alkene (1 mmol), $NaIO_4$ (1 mmol, 213 mg) in 3 mL of ethylene glycol was taken in a round bottomed flask at room temperature and then $NH_2OH{\cdot}HCl$ (1.5 mmol, 104 mg) was added by portion for 5 min. The reaction mixture was stirred for 30 min at room temperature. After completion (TLC), the reaction mixture was diluted with a 1:1 mixture of water/ethyl acetate (10 mL) and washed with 10% (w/v) $Na_2S_2O_3$ (3x5 mL) followed by brine solution (1x10 mL). Then the combined organic layer was dried over anhydrous Na_2SO_4. Evaporation of solvent furnished the crude product which was subjected to column chromatography using ethyl acetate-petroleum ether (1:15) as eluent to obtain the analytically pure product.

Typical procedure for the synthesis of compound 2a on gram-scale

A mixture of styrene **1a** (10 mmol, 1.04 g), $NaIO_4$ (10 mmol, 2.13 g) in 30 mL of ethylene glycol was taken in a round bottomed flask at room temperature and then $NH_2OH{\cdot}HCl$ (15 mmol, 1.04 g) was added by portion for 5-10 min. The reaction mixture was stirred for 30 min at room temperature. After completion (TLC), the reaction mixture was diluted with a 1:1 mixture of water/ethyl acetate (50 mL) and washed with 10% (w/v) $Na_2S_2O_3$ (3x20 mL) followed by brine solution (1x30 mL). Then the combined organic layer was dried over anhydrous Na_2SO_4. Evaporation of solvent furnished the crude product which was subjected to column chromatography using ethyl acetate-petroleum ether (1:15) as eluent to obtain the analytically pure product.

2-(2-Iodo-1-phenylethoxy)ethanol (2a). 254 mg, yield 87% (2.34 g, 80% yield for 10 mmol), yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.28-7.22 (m, 5H), 4.39-4.36 (m, 1H), 3.66 (s, 2H), 3.48-3.24 (m, 4H), 2.47 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 139.7, 128.8, 128.6, 126.4, 82.3, 70.7, 61.7, 11.0. Anal. calcd for $C_{10}H_{13}IO_2$: C, 41.12; H, 4.49%; Found: C, 41.08; H, 4.46%.

2-(1-(2,4-dimethylphenyl)-2-iodoethoxy)ethanol (2b). 275 mg, yield 83%, yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18 (d, *J* = 7.6 Hz,1H), 6.95 (d, *J* = 8 Hz, 1H), 6.89 (s, 1H), 4.58 (t, *J* = 6.4 Hz,1H), 3.66 - 3.64 (m, 2H), 3.47-3.43 (m, 1H), 3.33 - 3.28 (m, 1H), 3.20 (d, *J* = 6.8 Hz, 2H), 2.47 (br, 1H), 2.21 (s, 6H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 137.9, 135.2, 134.7, 131.6, 127.3, 125.5, 79.1, 70.7, 61.8, 21.1, 19.0, 10.0 Anal. calcd for $C_{12}H_{17}IO_2$: C, 45.02; H, 5.35 %; Found: C, 44.98; H, 5.30%.

2-(2-Iodo-1-(4-methoxyphenyl)ethoxy)ethanol (2c). 261 mg, yield 81%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.16 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.4 Hz, 2H), 4.36-4.33 (m, 1H), 3.74 (s, 3H), 3.71-3.66 (m, 2H), 3.49-3.44 (m, 1H), 3.37-3.21(m, 3H), 2.25 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.9, 131.7, 127.7, 114.3, 82.0, 70.6, 61.9, 55.4, 11.3. Anal. calcd for $C_{11}H_{15}IO_3$: C, 41.01; H, 4.69%; Found: C, 40.95; H, 4.67%.

2-(1-(3-Bromophenyl)-2-iodoethoxy)ethanol (2d). 323 mg, yield 87%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.47-7.44 (m, 2H), 7.25-7.21 (m, 2H), 4.43-4.40 (m,1H), 3.76-3.74 (m, 2H), 3.57-3.52 (m, 1H), 3.47-3.42 (m, 1H), 3.37-3.29 (m, 2H), 2.59 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 142.1, 131.7, 130.4, 129.5, 125.1, 122.9, 81.6, 71.0, 61.7, 10.3. Anal. calcd for $C_{10}H_{12}BrIO_2$: C, 32.37; H, 3.26%; Found: C, 32.32; H, 3.21%.

2-(1-(4-Chlorophenyl)-2-iodoethoxy)ethanol (2e). 281 mg, yield 86%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.37-7.34 (m, 2H), 7.28-7.26 (m, 2H), 4.46-4.43 (m, 1H), 3.76 (t, *J* = 4.4 Hz, 2H), 3.57-3.53 (m, 1H), 3.48-3.43 (m, 1H), 3.39-3.30 (m, 2H), 2.43 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.3, 134.4, 129.1, 127.9, 81.6, 70.9, 61.8, 10.4. Anal. calcd for $C_{10}H_{12}ClIO_2$: C, 36.78; H, 3.70%; Found: C, 36.73; H, 3.62.%

2-(2-Iodo-1-(3-nitrophenyl)ethoxy)ethanol (2f). 273 mg, yield 81%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.23-8.21 (m, 2H), 7.71 (d, *J* = 8 Hz, 1H), 7.62-7.57 (m, 1H), 4.60-4.57 (m, 1H), 3.81 (s, 2H), 3.62-3.51 (m, 2H), 3.44-3.37 (m, 2H), 2.32 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.7, 142.2, 132.6, 130.0, 123.7, 121.7, 81.2, 71.3, 61.9, 9.7. Anal. calcd for $C_{10}H_{12}INO_4$: C, 35.63; H, 3.59; N, 4.15%; Found: C, 35.56; H, 3.53; N, 4.11%.

2-(1-Iodooctan-2-yloxy)ethanol (2g). 240 mg, yield 80%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.77-3.56 (m, 4H), 3.36-3.31 (m, 1H), 3.28-3.20 (m, 1H), 2.23 (br, 1H), 1.81-1.73 (m, 1H), 1.65-1.55 (m, 2H), 1.40-1.30 (m, 8H), 0.92-0.85 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 78.9, 70.6, 62.1, 34.7, 31.8, 29.3, 25.3, 22.7, 14.2, 10.6. Anal. calcd for $C_{10}H_{21}IO_2$: C, 40.01; H, 7.05%; Found: C, 39.98; H, 7.01%.

2-(2-Iodocyclohexyloxy)ethanol (2h). 221 mg, yield 82%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.01-3.95 (m, 1H), 3.72-3.67 (m, 3H), 3.48-3.44 (m, 1H), 3.30-3.24 (m, 1H), 2.41-2.36 (m, 1H), 2.11-2.07 (m, 1H), 2.01-1.89 (m, 1H), 1.80-1.76 (m, 1H), 1.50-1.46 (m, 1H), 1.31-1.22 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 83.3, 70.5, 62.0, 38.7, 36.5, 31.8, 27.9, 24.1. Anal. calcd for $C_8H_{15}IO_2$: C, 35.57; H, 5.60%; Found C, 35.53; H, 5.56%.

2-(2-Iodocyclooctyloxy)ethanol (2i). 238 mg, yield 80%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.33-4.28 (m, 1H), 3.69-3.62 (m, 4H), 3.37-3.33 (m, 1H), 2.11-1.56 (m, 9H), 1.32-1.25 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 87.0, 70.5, 61.9, 42.8, 33.2, 30.6, 27.2, 26.7, 25.8, 25.3. Anal. calcd for $C_{10}H_{19}IO_2$: C, 40.28; H, 6.42%; Found: C, 40.22; H, 6.34%.

3-(2-Hydroxyethoxy)-2-iodo-3-phenylpropan-1-ol (2j). 274 mg, yield 85%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40-7.31 (m, 5H), 4.69 (d, J = 7.6 Hz, 1H), 4.39-4.35 (m, 1H), 4.19-4.11 (m, 1H), 3.93-3.81 (m, 1H), 3.74 (s, 2H), 3.60-3.29 (m, 2H), 3.18 (br, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 138.9, 128.7, 128.6, 127.6, 85.1, 70.9, 65.8, 61.7, 39.7. Anal. calcd for $C_{11}H_{15}IO_3$: C, 41.01; H, 4.69%; Found: C, 40.97; H, 4.63%.

2-(1-Iodo-2-phenylpropan-2-yloxy)ethanol (2k). 251 mg, yield 82%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.44-7.32 (m, 5H), 3.80-3.74 (m, 2H), 3.57 (d, J = 10.8 Hz,1H), 3.49-3.40 (m, 2H), 3.33-3.29 (m, 1H), 2.37 (br, 1H), 1.76 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 141.4, 128.7, 128.0, 126.2, 76.5, 64.3, 62.2, 24.5, 20.3. Anal. calcd for $C_{11}H_{15}IO_2$: C, 43.16; H, 4.94%; Found: C, 43.12; H, 4.91%.

2-(2-Iodo-1,1-diphenylethoxy)ethanol (2l). 298 mg, yield 81%, pale yellow solid, mp 70-72 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40-7.25 (m, 10H), 4.18 (s, 2H), 3.84 (t, J = 4.4 Hz, 2H), 3.32 (t, J = 4.4 Hz, 2H), 2.21 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 142.9, 128.3, 127.6, 127.1, 80.2, 64.0, 62.3, 16.8. Anal. calcd for $C_{16}H_{17}IO_2$: C, 52.19; H, 4.65%; Found: C, 52.13; H, 4.61%.

Typical procedure for the synthesis of compound 3

A mixture of alkene (1 mmol), $NaIO_4$ (1 mmol, 213 mg) in 3 mL of ethanol was taken in a round bottomed flask at room temperature and then $NH_2OH{\cdot}HCl$ (1.5 mmol, 104 mg) was added by portion for 5 min. The reaction mixture was stirred for 30 min at room temperature. After completion (TLC), the reaction mixture was diluted with a 1:1 mixture of water/ethyl acetate (10 mL) and washed with 10% (w/v) $Na_2S_2O_3$ (3x5 mL) followed by brine solution (1x10 mL). Then the combined organic layer was dried over anhydrous Na_2SO_4. Evaporation of solvent furnished the crude product which was subjected to column chromatography using ethyl acetate-petroleum ether (1:15) as eluent to obtain the analytically pure product.

1-(1-Ethoxy-2-iodoethyl)benzene (3a).[16g] 237 mg, yield 86%, orange liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42-7.35 (m, 5H), 4.46-4.43 (m, 1H), 3.52-3.44 (m, 2H), 3.41-3.34 (m, 2H), 1.26 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 140.6, 128.7, 128.3, 126.5, 81.9, 65.1, 15.2, 11.0.

1-(1-Ethoxy-2-iodoethyl)-4-methoxybenzene (3b). 248 mg, yield 81%, yellowish orange liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.27-7.23 (m, 2H), 6.91-6.88 (m, 2H), 4.38-4.34 (m, 1H), 3.81 (s, 3H), 3.46-3.26 (m, 4H), 1.21 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.8, 159.7, 132.7, 128.1, 127.8, 114.1, 81.5, 64.9, 64.8, 55.4, 15.3, 15.28, 11.4. Anal. calcd for $C_{11}H_{15}IO_2$: C, 43.16; H, 4.94%; Found: C, 43.12; H, 4.91%.

1-Chloro-4-(1-ethoxy-2-iodoethyl)benzene (3c). 257 mg, yield 83%, orange liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.33 (d, J = 8.4 Hz, 2H), 7.26 (d, J = 8.4 Hz, 2H), 4.38-4.35 (m, 1H), 3.45-3.40 (m, 2H), 3.34-3.26 (m, 2H), 1.21 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 139.2, 134.1, 128.9, 128.0, 81.2, 65.3, 15.3, 10.5. Anal. calcd for $C_{10}H_{12}ClIO$: C, 38.67; H, 3.89%; Found: C, 38.61; H, 3.82%.

3-Ethoxy-2-iodo-3-phenylpropan-1-ol (3d). 260 mg, yield 85%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.31 (m, 5H), 4.62 (d, J = 7.2 Hz, 1H), 4.35-4.31 (m, 1H), 3.99-3.95 (m, 1H), 3.84-3.80 (m, 1H), 3.43-3.38 (m, 2H), 3.11 (br, 1H), 1.18 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 139.4, 128.6, 128.5, 127.5, 86.4, 66.5, 65.6, 39.1, 15.3. Anal. calcd for $C_{11}H_{15}IO_2$: C, 43.16; H, 4.94%; Found: C, 43.13; H, 4.90%.

1-(2-Ethoxy-1-iodopropan-2-yl)benzene (3e).[20] 241 mg, yield 83%, orange liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42-7.25 (m, 5H), 3.53-3.45 (m, 2H), 3.36-3.32 (m, 1H), 3.24-3.18 (m, 1H), 1.70 (s, 1H), 1.23-1.19 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 142.4, 128.5, 127.7, 126.3, 76.7, 58.9, 24.8, 20.0, 15.7.

1-Ethoxy-2-iodo-1,1-diphenylethane (3f). 262 mg, yield 80%, pale orange solid, mp 67-68 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38-7.18 (m, 10H), 4.10 (s, 2H), 3.22 (q, J = 7.2 Hz, 2H), 1.25 (t, J = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 143.5, 128.5, 128.1, 127.3, 127.1, 127.0, 126.2, 80.2, 57.9, 16.8, 15.4. Anal. calcd for $C_{16}H_{17}IO$: C, 54.56; H, 4.86%; Found: C, 54.51; H, 4.82%.

2-Ethoxy-1-iodooctane (3g). 221 mg, yield 78%, pale yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.59-3.36 (m, 2H), 3.20-3.18 (m, 2H), 3.12-3.07 (m, 1H), 1.56-1.46 (m, 3H), 1.27-1.13 (m, 10H), 0.83-0.80 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 78.5, 65.1, 34.8, 31.9, 29.3, 25.4, 22.7, 15.6, 14.2, 10.6. Anal. calcd for $C_{10}H_{21}IO$: C, 42.26; H, 7.45%; Found: C, 42.18; H, 7.36%.

Typical procedure for the synthesis of compound 4

A mixture of alkene (1 mmol), $NaIO_4$ (1 mmol, 213 mg) in a mixture of 2.5 mL of water and 0.5 mL of THF was taken in a round bottomed flask at room temperature and then $NH_2OH{\cdot}HCl$ (1.5 mmol, 104 mg) was added by portion for 5 min. The reaction mixture was stirred for 30 min at room temperature. After completion (TLC), the reaction mixture was diluted with a 1:1 mixture of water/ethyl acetate (10 mL) and washed with 10% (w/v) $Na_2S_2O_3$ (3x5 mL) followed by brine solution (1x10 mL). Then the combined organic layer was dried over anhydrous Na_2SO_4. Evaporation of solvent furnished the crude product which was subjected to column chromatography using ethyl acetate-petroleum ether (1:15) as eluent to obtain the analytically pure product.

2-Iodo-1-phenylethanol (4a).[16g] 206 mg, yield 83%, pale yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40-7.26 (m, 5H), 4.85-4.82 (m, 1H), 3.51-3.48 (m, 1H), 3.43-3.38 (m, 1H), 2.52 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 141.3, 128.8, 128.5, 125.9, 74.1, 15.4.

1-(4-Chlorophenyl)-2-iodoethanol (4b). 237 mg, yield 84%, pale yellow solid, mp 67-69 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.29 (m, 4H), 4.81-4.78 (m, 1H), 3.48-3.44 (m, 1H), 3.38-3.33 (m, 1H), 2.61 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 139.7, 134.2, 129.0, 127.3, 73.4, 15.1. Anal. calcd for C_8H_8ClIO: C, 34.01; H, 2.85%; Found: C, 33.94; H, 2.78%.

2-Iodo-1-(3-nitrophenyl)ethanol (4c). 237 mg, yield 81%, pale orange liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.28-8.22 (m, 2H), 7.73 (d, J = 8 Hz, 1H), 7.60 (t, J = 8 Hz,1H), 5.15 (q, J = 5.2 Hz, 1H), 3.87-3.83 (m, 1H), 3.71 (t, J = 10.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.5, 141.3, 133.5, 130.0, 124.1, 122.5, 59.7, 8.9. Anal. calcd for $C_8H_8INO_3$: C, 32.79; H, 2.75; N, 4.78%; Found: C, 32.72; H, 2.67; N, 4.70%.

1-iodo-2phenylpropan-2-ol (4d). 218 mg, yield 80%, pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46-7.20 (m, 5H), 3.70 - 3.61 (m, 2H), 2.34 (br, 1H), 1.73 (s, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.4, 128.6 (2C), 127.6 (2C), 124.9, 72.8, 29.1, 24.3 Anal. calcd for $C_9H_{11}IO$: C, 41.24; H, 4.34 %; Found: C, 41.26; H, 4.30%.

2-iodo-1,1-diphenylethanol (4e). 270 mg, yield 81%, deep yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, J = 7.6 Hz, 4H), 7.42-7.33 (m, 6H), 4.07 (s, 2H), 2.98 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5 (2C), 128.4 (4C), 127.7 (4C), 126.2 (2C), 76.7, 22.5. Anal. calcd for $C_{14}H_{13}IO$: C, 51.87; H, 4.04%; Found: C, 51.83; H, 4.08%.

1-iodooctan-2-ol (4f). 208 mg, yield 78%, pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.52 - 3.50 (m, 1H), 3.41- 3.38 (m, 1H), 3.25-3.22 (m, 1H), 1.57 - 1.25 (m, 11H), 0.88 (t, J = 5.2 Hz, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 71.2, 36.8, 31.8, 29.3, 25.8, 22.7, 16.9, 14.2. Anal. calcd for $C_8H_{17}IO$: C, 37.51; H, 6.69%; Found: C, 37.49; H, 6.72%.

2-iodocyclohexanol (4g). 180 mg, yield 76%, orange gummy mass; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.05-4.00 (m, 1H) 3.70-3.60 (m, 1H), 2.11-1.20 (m, 9H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 76.0, 43.4, 38.6, 33.6, 28.0, 24.4. Anal. calcd for $C_6H_{11}IO$: C, 31.88; H,4.90%; Found: C, 31.82; H, 4.96%.

2-iodocyclooctanol (4h). 184 mg, yield 70%, gummy brown liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.42-4.38 (m, 1H), 4.03-3.98 (m, 1H), 2.21-1.90 (m, 6H), 1.65-1.44 (m, 7H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 78.4, 50.5, 34.4, 32.5, 27.0, 26.0, 25.7, 25.5. Anal. calcd for $C_8H_{15}IO$: C, 37.81; H, 5.95%; Found: C, 37.83; H, 5.91%.

Typical procedure for the synthesis of compound 5

A mixture of alkene (1 mmol), $NaIO_4$ (1 mmol, 213 mg) in 3 mL of acetic acid was taken in a round bottomed flask at room temperature and then $NH_2OH{\cdot}HCl$ (1.5 mmol, 104 mg) was added by portion for 5 min. The reaction mixture was stirred for 30 min at room temperature. After completion (TLC), the reaction mixture was diluted with a 1:1 mixture of water/ethyl acetate (10 mL) and washed with 10% (w/v) $Na_2S_2O_3$ (3x5 mL) followed by brine solution (1x10 mL). Then the combined organic layer was dried over anhydrous Na_2SO_4. Evaporation of solvent furnished the crude product which was subjected to column chromatography using ethyl acetate-petroleum ether (1:15) as eluent to obtain the analytically pure product.

2-Iodo-1-phenylethyl acetate (5a).[16c] 229 mg, yield 79%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38-7.34 (m, 5H), 5.98-5.86 (m, 1H), 3.82-3.70 (m, 1H), 3.48-3.45 (m, 1H), 2.14-2.13 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.1, 169.9, 138.6, 137.3, 129.0, 128.9, 128.8, 126.8, 126.6, 75.3, 75.2, 46.6, 21.2, 21.1, 7.9.

1-(4-Chlorophenyl)-2-iodoethyl acetate (5b). 266 mg, yield 82%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.36-7.26 (m, 4H), 5.93-5.81 (m, 1H), 3.76-3.70 (m, 1H), 3.45-3.42 (m, 1H), 2.13-2.13 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 169.9, 169.8, 137.0, 135.8, 134.9, 134.7, 129.0, 128.2, 128.0, 74.5, 74.4, 46.3, 21.1, 21.0, 7.4. Anal. calcd for $C_{10}H_{10}ClIO_2$: C, 37.01; H, 3.11%; Found: C, 36.97; H, 3.05%.

Typical procedure for the synthesis of compound 6[35]

Oxone (0.75 mmol) was slowly added to compound **2** (1 mmol) in 2 mL of ethylene glycol in a round bottomed flask and the reaction mixture was stirred at room temperature for 2 h. After completion (TLC), the reaction mixture was diluted with a 1:1 mixture of water/DCM (10 mL) and washed with 10% (w/v) $Na_2S_2O_3$ (2x5 mL) followed by brine solution (1x10 mL). Then the combined organic layer was dried over anhydrous Na_2SO_4. Evaporation of solvent furnished the crude product which was subjected to column chromatography using ethyl acetate-petroleum ether (1:15) as eluent to obtain the analytically pure product.

2-Benzyl-1,3-dioxolane (6a).[35] 116 mg, yield 71%, red liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.35-7.21 (m, 5H), 5.07 (t, J = 4.8 Hz, 1H), 3.99-3.82 (m, 4H), 2.97 (d, J = 4.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.3, 129.8, 128.5, 126.7, 104.8, 65.1, 40.9.

2-(4-Chlorobenzyl)-1,3-dioxolane (6b).[35] 158 mg, yield 80%, white solid, mp 38-40 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.28-7.18 (m, 4H), 5.04 (t, J = 4.8 Hz, 1H), 3.94-3.82 (m, 4H), 2.93 (d, J = 4.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 134.6, 132.6, 131.2, 128.5, 104.4, 65.2, 40.2.

2-(2,4-Dimethylbenzyl)-1,3-dioxolane (6c). 180 mg, yield 94%, pale orange liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.39 (d, J = 7.5 Hz, 1H), 7.24-7.22 (m, 2H), 5.30 (t, J = 5 Hz, 1H), 4.21(t, J = 6.5 Hz, 2H), 4.08 (t, J = 6.5 Hz, 2H), 3.22 (d, J = 5 Hz, 2H), 2.59 (s, 3H), 2.55 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.4, 136.0, 131.3, 130.8, 130.1, 126.4, 104.3 (2C), 64.7, 37.3, 20.8, 19.6. Anal. calcd for $C_{12}H_{16}O_2$: C, 74.97; H, 8.39%; Found: C, 74.95; H, 8.41%.

2-(4-Methoxybenzyl)-1,3-dioxolane (6d).[35] 180 mg, yield 93%, pale yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.12-7.10 (m, 2H), 6.78 – 6.75 (m, 2H), 4.94 (t, J = 4.8 Hz, 1H) 3.87-3.73 (m, 4H), 3.70 (s, 3H) 2.83 (d, J = 4.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100

MHz): δ 158.4, 130.7 (2C), 128.3, 113.9 (2C), 104.9 (2C), 65.0, 55.3, 39.9.

2-Heptyl-1,3-dioxolane (6e). 138 mg, yield 80%, pale yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.77 (t, *J* = 4.8 Hz, 1H), 3.93-3.75 (m, 4H), 1.57 ((t, *J* = 4.8 Hz, 2H), 1.35-1.20 (m, 10H), 0.80 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 104.8, 64.9 (2C), 34.0, 31.8, 29.6, 29.3, 24.2, 22.7, 14.2. Anal. calcd for $C_{10}H_{20}O_2$: C, 69.72; H, 11.7%; Found: C, 69.69; H, 11.69%.

2-Cyclopentyl-1,3-dioxolane (6f).[35] 130 mg, yield 91%, yellow gummy mass; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.63 (d, *J* = 5.6 Hz, 1H), 3.91- 3.76 (m, 4H), 2.07-1.99 (m, 1H), 1.71- 1.33 (m, 8H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 107.9. 65.1 (2C), 43.1, 27.7 (2C), 25.9 (2C).

2-Cycloheptyl-1,3-dioxolane (6g). 138 mg, yield 81%, pale yellow gummy mass; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.60 (d, *J* = 4.4 Hz, 1H), 3.89-3.75 (m, 4H), 2.11-2.07 (m, 1H), 1.67-1.51 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 108.1, 65.1 (2C), 43.2, 28.8 (2C), 26.9 (2C), 26.4 (2C). Anal. calcd for $C_{10}H_{18}O_2$: C, 70.55; H, 10.66%; Found: C, 70.60; H, 10.69%.

2-(1,3-Dioxolan-2-yl)-2-phenylethanol (6h).[35] 162 mg, yield 84%, pale yellow gummy mass; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.31-7.21 (m. 5H), 5.11 (d, *J* = 5.2 Hz, 1H), 4.08-4.03(m, 2H), 3.95-3.75 (m, 4H), 3.10-3.06 (m, 1H), 2.56 (br, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 137.7, 128.5 (2C), 127.2, 106.3, 64.6 (2C), 63.6, 51.4.

2-Benzyl-2-methyl-1,3-dioxolane (6i). 121 mg, yield 68%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.28-7.22 (m, 5H), 3.92-3.89 (m, 2H), 3.77-3.73 (m, 2H), 2.92 (s, 2H), 1.31 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 137.0, 130.6, 128.1, 126.5, 109.9, 64.9, 45.5, 24.4. Anal. calcd for $C_{11}H_{14}O_2$: C, 74.13; H, 7.92%; Found: C, 74.07; H, 7.86%.

2-Benzyl-2-phenyl-1,3-dioxolane (6j). 215 mg, yield 85%, yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.31-7.03 (m, 10 H), 3.76-3.63 (m, 4H), 3.09 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 142.5, 136.0, 130.9 (2C), 128.0, 127.9, 127.7, 126.4 (2C), 125.9 (2C), 110.0 (2C), 64.8 (2C), 47.1. Anal. calcd for $C_{17}H_{18}O_2$: C, 79.97; H, 6.71%; Found: C, 79.91; H, 6.73%.

Typical procedure for the synthesis of compound 7

A mixture of alkene (1 mmol), $NaIO_4$ (1 mmol, 213 mg) in 3 mL of toluene was taken in a round bottomed flask at room temperature and then $NH_2OH{\cdot}HCl$ (1.5 mmol, 104 mg) was added by portion for 5 min. The reaction mixture was stirred for 1 h at room temperature. After completion (TLC), the reaction mixture was diluted with a 1:1 mixture of water/ethyl acetate (10 mL) and washed with 10% (w/v) $Na_2S_2O_3$ (3x5 mL) followed by brine solution (1x10 mL). Then the combined organic layer was dried over anhydrous Na_2SO_4. Evaporation of solvent furnished the crude product which was subjected to column chromatography using ethyl acetate-petroleum ether (1:15) as eluent to obtain the product.

3-Iodo-tetrahydrofuran (7a) & 2-(Iodomethyl)oxetane (7a′). Inseparable mixture, 182 mg, yield 92%, colorless liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 4.78-4.41 (m, 1H), 4.24-4.18 (m, 1H), 4.11-4.07 (m, 1H), 3.92-3.83 (m, 4H), 3.79-3.76 (m, 1H), 3.66-3.62 (m, 1H), 3.53-3.48 (m, 1H), 2.42-2.23 (m, 2H), 2.02-1.87 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 62.0, 59.4, 57.9, 50.5, 40.0, 39.1, 29.3, 11.6.

4-Iodo-2-phenyl-tetrahydrofuran (7b) & 2-(Iodometyl)-4-phenyloxetane (7b′). Inseparable mixture, 219 mg, yield 80%, pale yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.42-7.37 (m, 10H), 5.05-4.94 (m, 2H), 4.68-4.61 (m, 1H), 4.43-4.37 (m, 1H), 4.13-4.09 (m, 1H), 3.89-3.84 (m, 1H), 3.67-3.63 (m, 1H), 3.50-3.46 (m, 1H), 2.50-2.43 (m, 1H), 2.35-2.28 (m, 1H), 2.06-1.90 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 144.0, 129.0, 128.8, 128.6, 128.1, 125.9, 125.8, 74.0, 71.3, 58.2, 50.8, 47.3, 46.4, 30.5, 11.5.

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