Catalytic selective oxidation of benzyl alcohols to aldehydes with rhenium complexes

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Abstract

The system (^{n}Bu$_4$N)ReO$_4$ **5**/PhIO/CH$_2$Cl$_2$, $T = 298$ K catalyses effectively and with total selectivity the anaerobic oxidation of a range of primary substituted benzyl alcohols (*o*-, *m*-, *p*-X-C$_6$H$_4$-CH$_2$OH, X = H, Me, MeO, Cl, NO$_2$, CF$_3$) to the corresponding aldehydes; in contrast, it is unreactive towards secondary benzyl and aliphatic (primary and secondary) alcohols. This may prove of interest in synthetic organic transformations, when several alcoholic functionalities are present in the same molecule.
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1. Introduction

The selective oxidation of primary and secondary alcohols to the corresponding aldehydes and ketones plays a central role in Organic Chemistry, hence a wide variety of methods have been developed. Stoichiometric reactions include Swern oxidation [1], Dess–Martin oxidation [2] and various metal oxidants [3], whereas catalytic reactions involve transition metal complexes in combination with co-oxidants used as sacrificial reagents (NaIO$_4$, NaOCl, ^{t}BuOOH, *N*-oxides, *S*-oxides, etc.) [4–7]. Despite progress on the catalytic oxidation of alcohols with various metal complexes and the use of environmentally friendly systems [8], there are still severe limitations with respect to the chemo-, regio- and stereoselectivity of this reaction.

Mechanistic studies demonstrate that metal mediated alcohol oxidation can proceed via abstraction of a hydrogen atom by a metal oxo- or peroxo-unit, or by means of a concerted β-hydrogen elimination pathway [9].

We have previously reported [10,11] that the redox couple [Re$_3$(μ-Cl)$_3$Cl$_9$]$^{3-}$ (Re$_3$$^{9+}$) **2**/[Re$_3$(μ-Cl)$_3Cl_7$(H$_2$O)$_2$]$^{2-}$ (Re$_3$$^{8+}$) **3**, deriving from dissolution of the polymeric triangulo rhenium chloride [Re$_3$(μ-Cl)$_3$Cl$_6$]$_n$ **1**, which features a diamagnetic Re$_3$$^{9+}$ core (D_{3h}, (a')^2(a'')^2(e')^4(e'')^4) and has three Re=Re bonds, in HCl (3–12 M)/Hg efficiently catalyses dioxygen activation and reduction to water in aqueous (HCl) or biphasic media (HCl/CH$_2$Cl$_2$) at $T = 298$ K.

In view that (a) metal oxo- or peroxo-species formed from the reaction of dioxygen with metal complexes can produce catalytically active oxidants [12,13] and (b) alkoxide clusters containing the Re$_3$$^{9+}$ core can undergo facile β-hydrogen elimination at a terminal alkoxide ligand forming a metal hydride complex and the carbonyl compound [14,15], we have examined the possibility of aerobically oxidizing alcohols with the above mentioned system.

Indeed, this was found to convert selectively, but inefficiently benzyl alcohol to benzaldehyde, but its further exploitation in organic media led to the development of a unique catalytic system (^{n}Bu$_4$N)ReO$_4$ **5**/PhIO/CH$_2$Cl$_2$, $T = 298$ K, which efficiently and selectively oxidizes a range of primary substituted benzyl alcohols to the corresponding

aldehydes, while it is unreactive towards secondary benzyl and aliphatic (primary and secondary) alcohols.

2. Experimental

2.1. General considerations

All operations were performed under a pure argon or nitrogen atmosphere, using Schlenk and Syringe techniques on an inert gas/vacuum manifold, unless otherwise stated. Diethyl ether and C_6H_6 were distilled over Na/Ph_2CO in an inert atmosphere. MeCN, CH_2Cl_2, hexane and 1,2-$C_6H_4Cl_2$ were distilled over CaH_2 by fractional distillation (column Vigreux). Petroleum ether 40–60 (PE) and Et_2O were distilled over Na/benzophenone. Solvents were degassed by three freeze-pump-thaw cycles. $PhCH_2OH$, Ph_2CHOH, Na_2SO_4, PhCHO and (NBA)Cl were purified and dried according to literature methods [16]. All chemicals and solvents were purchased from Aldrich, rhenium chloride, **1**, from Alfa Aesar, decahydronaphthalene (*cis* and *trans* mixture of isomers) from Riedel de Haën and 1,2-$C_6H_4Cl_2$ from Fluka. Nerol, geraniol and farnesol were kindly offered by Assoc. Prof. V. Roussis (isolated from natural products). **2**(TPP)$_3$ [11], **3**(TPP)$_2$ [11], **5** [17], 1-[4-(hydroxymethyl)phenyl]-1-ethanol [18] and PhIO [19] were prepared according to the cited literature methods.

^{1}H and ^{13}C NMR spectra were obtained on a Varian 300 Unity Plus spectrometer. IR spectra were recorded on a Perkin-Elmer 883 spectrometer. UV–vis spectra were obtained on a Hitachi U-2000 or a Varian Cary 3E spectrophotometer. GC–MS experiments were performed on a Varian Star 3400CX GC coupled with a Varian Saturn 2000 MS. The GC and MS parameters are shown in Table 1. The analysis of the samples (a) and recycling of the solvent and the co-oxidant (b) was performed as follows:

(a) Decahydronaphthalene (internal standard for quantitation) was added to the mixture at the end of the reaction and the solvent was removed under vacuum. The organic compounds were extracted with hexane or diethyl ether, depending on their solubility, and were dried over Na_2SO_4. All complexes are insoluble in both solvents.

Table 1
GC and MS parameters

Column	30 m × 0.25 mm ID, 0.25 μm, CP-Sil 8 CB LB/MS column with a 2 m fused Silica precolumn
Injection	1 μL, 50:1 split
Flow	1 mL/min-He, constant flow
Oven	50 °C/hold 3.00 min, 7 °C/min to 150 °C, 50 °C/min to 280 °C, 280 °C/hold 2.12 min, 5 °C/min to 300 °C, 300 °C/hold 4.00 min
Injector	280 °C
Transfer line	280 °C
MSD scan range	10–600 amu

(b) At the end of the reaction the solvent was removed and collected in a cold trap under vacuum. The crude residue, which remains after extracting the organic compounds (see above), can be reused in combination with PhIO for further catalytic reactions. PhI was separated from the aldehyde (RCHO) and the unreacted alcohol (RCH$_2$OH) and was converted to PhIO, according to literature [19,20] (e.g. when R = Ph, the solvent containing the organic products was removed under vacuum and the mixture was separated by column chromatography (silica gel 60, eluent PE (PhI); PE/Et$_2$O = 6/1 (PhCHO); PE/Et$_2$O = 1/1 (PhCH$_2$OH)).

2.2. Catalytic oxidation of PhCH$_2$OH with (a) **2**/HCl 3 M/Hg/O$_2$ and (b) **2**/HCl 3 M/Hg/CH$_2$Cl$_2$/O$_2$ (P_{O_2} = 1 atm, T = 298 K)

(a) Complex **1** (>99.9%, 10.0 mg, 11.4 μmol) was dissolved in HCl (3 M, 5.0 mL) and treated with excess Hg (10 mg, 0.050 mmol). The mixture was stirred vigorously under Ar for 2 h and the colour changed from deep red to deep purple. Then, PhCH$_2$OH was added (59 μL, 62 mg, 0.057 mmol) and dioxygen (O$_2$) was passed through the solutions for 15 min. The reaction mixture was left under stirring for 24 h at ambient temperature and pressure.

(b) The same procedure as above has been employed except that after the 2 h steering, a solution of (TPP)Cl (98%, 8.7 mg, 22.8 μmol) in CH$_2$Cl$_2$ (5.0 mL) was added as phase transfer reagent.

2.3. Catalytic oxidation of substituted benzyl alcohols with **2**(TPP)$_3$/PhIO/CH$_2$Cl$_2$

A typical oxidation of alcohols mediated by compound **2**(TPP)$_3$ was performed as follows: compound **2**(TPP)$_3$ (10.0 mg, 5.0 μmol) was dissolved under anaerobic conditions in CH$_2$Cl$_2$ (15.0 mL) followed by addition of PhIO (55.0 mg, 250 μmol) and PhCH$_2$OH (26 μL, 27 mg, 250 μmol). The mixture was stirred for 24 or 48 h under Ar at T = 298 K.

2.4. Catalytic oxidation of PhCH$_2$OH with **5**/O$_2$ (P_{O_2} = 3 atm, T = 298 K)/CH$_2$Cl$_2$

The experiment was performed as described above (Section 2.2(b)), with compound **5** instead of **2**(TPP)$_3$ (**5** (5.0 mg, 10 μmol), PhCH$_2$OH (52 μL, 54 mg, 500 μmol), O$_2$ (P_{O_2} = 3 atm, 34 mmol), decalin (77.1 μL, 69.1 mg, 500 μmol)).

2.5. Catalytic oxidation of alcohols with **5**/PhIO/solvent

The procedure described above (Section 2.3) was also followed with compound **5** instead of compound **2**(TPP)$_3$ (**5** (10 μmol), PhIO (500 μmol), alcohol (500 μmol)) and in some cases, with solvents other than CH$_2$Cl$_2$. The mixture

was stirred for the time stated in Tables 2–5. The organic products of the non-aromatic alcohols oxidations were also analysed by ^{1}H and ^{13}C NMR in CDCl$_3$.

The same procedure was followed with the other co-oxidants.

2.6. Reaction of alkenes with 5/PhIO/CH$_2$Cl$_2$

The reaction was performed as described above, with alkenes instead of alcohols (**5** (10 μmol), PhIO (500 μmol), alkene (500 μmol)). The organic products were also analysed by ^{1}H and ^{13}C NMR in CDCl$_3$.

In all cases blank experiments have been carried out.

3. Results and discussion

3.1. Oxidations with the redox couple 2/3

Aerobic oxidation of benzyl alcohol (phenylmethanol, PhCH$_2$OH) with the redox couple **2/3** in HCl 3 M/Hg (**2**/PhCH$_2$OH = 1/50) or in a biphasic system (HCl 3 M/Hg/CH$_2$Cl$_2$/Ph$_4$PCl (TPPCl), **2**/(TPP)Cl/PhCH$_2$OH = 1/2/50)[1] produces benzaldehyde (benzenecarbaldehyde, PhCHO) in respectable yields (30 and 28%, respectively, t = 24 h, T = 298 K). No reaction occurred in the absence of **2**, **3** or in the presence of Hg and Hg$_2$Cl$_2$.

Oxidation of alcohols in organic solvents (CH$_2$Cl$_2$) has been carried out using **2**(TPP)$_3$ and molecular oxygen (P_{O_2} = 3 atm, T = 298 K) or iodosylbenzene (oxo(phenyl)-λ^3-iodane, PhIO) [21–23] as co-oxidants. When dioxygen is employed, the yields remain low, whereas the use of PhIO as co-oxidant leads to total chemoselectivity and nearly quantitative yields of PhCHO under certain conditions (**2**(TPP)$_3$/PhIO/PhCH$_2$OH = 1/50/50, CH$_2$Cl$_2$, 48 h, 96% PhCHO). At high molar ratios of PhIO and alcohol versus catalyst longer reaction times are required and traces of other products were observed (<0.1% of benzyl benzenecarboxylate).

During the course of the reaction colour changes were observed, along with the formation of a yellowish insoluble precipitate. At molar ratios of **2**(TPP)$_3$/PhIO/PhCH$_2$OH = 1/10/50 the initial colour of the solution changed from red to green-brown approximately within 1 h, whereas at higher PhIO/PhCH$_2$OH ratios fast decolourisation occurred upon mixing the reagents. Monitoring the reaction by visible spectroscopy, λ_{max} (nm) 517 ($5a_2'' \rightarrow 7e''$, $\pi \rightarrow \pi^*$) and 778 ($6e'' \rightarrow 9e'$, $\pi \rightarrow \sigma^*$), indicated the gradual disruption of the trimetal core [24]. Filtration and purification of the insoluble precipitate left behind a white solid, proved

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[1] Addition of CH$_2$Cl$_2$ containing (TPP)Cl into a solution of **3** in HCl 3 M/Hg in a ratio **3**/(TPP)Cl = 1/2 results in the formation of complex **3**(TPP)$_2$, which is transferred in the organic layer of the biphasic system. Air oxidation of **3**(TPP)$_2$ produces "**2**(TPP)$_2$(H$_3$O$^+$)", which is partially soluble in water and can be re-reduced in the aqueous layer to the parent Re$_3^{8+}$ state.

Table 2
Oxidation of PhCH$_2$OH with **5**/co-oxidant at T = 298 K

Entry	Co-oxidant	Molar ratio **5**/co-oxidant/PhCH$_2$OH	t (h)	PhCHO (blank[a]) (%)
1	O$_2$[b]	1/3400[c]/50	24	14 (0)
2	H$_2$O$_2$[d]	1/50/50	24	23 (0)
3	H$_2$O$_2$[d]	1/50/50	48	32 (0)
4	H$_2$O$_2$[d,e]	1/50/50	24	10 (0)
5	H$_2$O$_2$[d,e]	1/50/50	48	14 (0)
6	Me$_3$COOCMe$_3$[b]	1/50/50	24	33 (24)
7	PhI(OCOMe)$_2$[b]	1/50/50	48	33 (24)
8	PhI(OCOMe)$_2$[b,f]	1/50/50	24	30 (18)
9	PhI(OCOMe)$_2$[b,f]	1/50/50	48	90 (24)
10	PhIO[b]	1/50/50	24	82 (19)

[a] The reaction was performed without **5**.
[b] In CH$_2$Cl$_2$.
[c] P_{O_2} = 3 atm.
[d] In MeCN.
[e] H$_2$O$_2$ was added dropwise.
[f] In the air.

Table 3
Oxidation of PhCH$_2$OH with **5**/PhIO in CH$_2$Cl$_2$ at T = 298 K

Entry	Molar ratio **5**/PhIO/PhCH$_2$OH	t (h)	PhCHO (blank[a]) (%)
1	1/50/50	12	30
2	1/50/50	24	82 (19)
3	1/50/50	48	98
4	1/200/200	12	34
5	1/200/200	48	86
6	1/1000/1000	72	88

[a] The reaction was performed without **5**.

to be by analysis, IR spectroscopy, ν_{max} (cm^{-1}) 914 (Re=O), and X-ray crystallography the (TPP)ReO$_4$ **4** salt [11].

PhIO oxidizes PhCH$_2$OH to PhCHO in non-catalytic reactions (Table 2, entry 10). It can form adducts with transition metal complexes (L$_n$M(OIPh)), where the metal acts as a Lewis acid activator for PhIO [25], or can act as an oxygen atom transfer reagent resulting in formation of metal-oxo-species [26,27]. In both cases, those compounds are active and are considered responsible for the efficient oxidation of alkanes, alkenes and various organic substrates [25–27]. In the presence of ruthenium catalysts, such as RuCl$_2$(P(C$_6$H$_5$)$_3$)$_2$, Ru$_3$(CO)$_{12}$, RuCl$_3$·H$_2$O or ruthenocene

Table 4
Oxidation of PhCH$_2$OH with **5**/PhIO[a] in various solvents

Entry	Solvent	t (h)	PhCHO (blank[b]) (%)
1	1,2-C$_6$H$_4$Cl$_2$[c]	1	9
2	1,2-C$_6$H$_4$Cl$_2$[c]	2	31
3	1,2-C$_6$H$_4$Cl$_2$[c]	3	44
4	1,2-C$_6$H$_4$Cl$_2$[c]	4	69
5	1,2-C$_6$H$_4$Cl$_2$[c]	5	88
6	1,2-C$_6$H$_4$Cl$_2$[c]	6	98 (36)
7	C$_6$H$_6$[c]	2	70
8	C$_6$H$_6$[c]	4	100 (39)
9	CH$_2$Cl$_2$ + MeCN 10%[d]	24	64 (19)

[a] Molar ratio **5**/PhIO/PhCH$_2$OH = 1/50/50.
[b] The reaction was performed without **5**.
[c] Reflux.
[d] T = 298 K.

Table 5
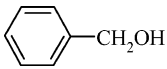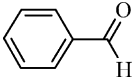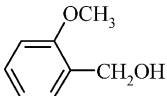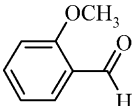Oxidation of alcohols with **5**/PhIO[a] in CH_2Cl_2 at $T = 298$ K
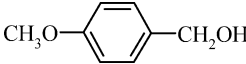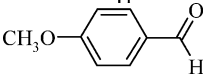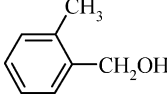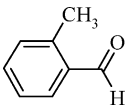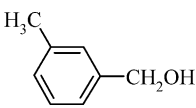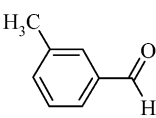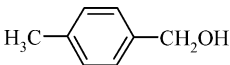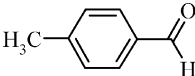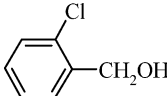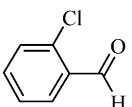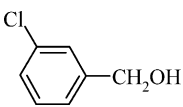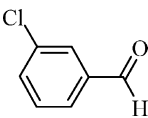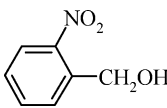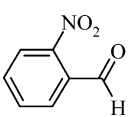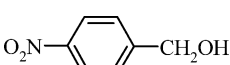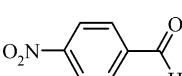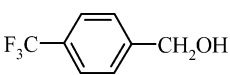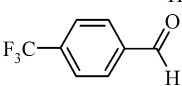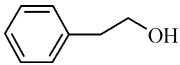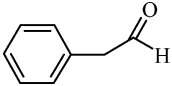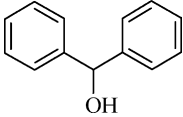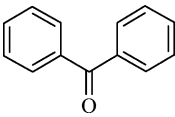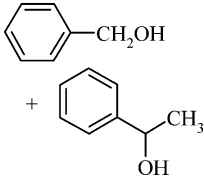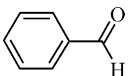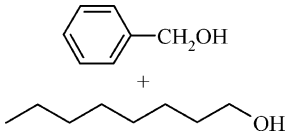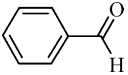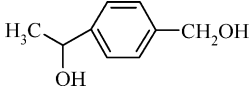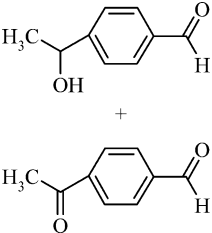
Entry	Alcohol	Carbonyl compound	t (h)	Yield (blank[b]) (%)	TON[c]
1			24	82 (19)	32
2			24 44	22 (18) 40 (30)	2.0 5.0
3			24	98 (22)	38
4			24	90 (15)	38
5			24	85 (35)	25
6			24	92 (75)	8.5
7			24	89 (27)	31
8			24	91 (75)	8.0
9			24 44	20 (11) 33 (13)	4.5 10
10			24	47 (15)	16
11			24	86 (18)	34
12			24	42 (12)	15
13			24	99 (15)	42
14			72	90	

Table 5 (*Continued*)

Entry	Alcohol	Carbonyl compound	*t* (h)	Yield (blank[b]) (%)	TON[c]
15			72	100	
16			24	40 (19)	
				7 (2)	

[a] Molar ratio **5**/PhIO/alcohol = 1/50/50.

[b] The reaction was performed without **5**.

[c] Calculated after subtraction of the non-catalysed yield.

[28], PhIO oxidizes a variety of alcohols to carbonyl compounds and carboxylic acids, whereas with ytterbium(III) salts [29], such as $Yb(NO_3)_3$, aldehydes or ketones are selectively produced at 80 °C in 1,2-dichloroethane solutions.

Compound **4** does not oxidize $PhCH_2OH$ to PhCHO in CH_2Cl_2, but becomes effective in combination with PhIO (**4**/PhIO/$PhCH_2OH$ = 1/50/50, CH_2Cl_2, 24 h, 80% PhCHO). The corresponding (NBA)ReO$_4$ **5** was further used in catalytic tests.

3.2. Catalytic oxidations with 5

Combination of **5** with various co-oxidants, such as O_2, H_2O_2, $Me_3COOCMe_3$, PhI(OCOMe), PhIO, and reaction with $PhCH_2OH$ results in formation of PhCHO (Table 2). Among the co-oxidants used, the best results were obtained with PhIO (entry 10, TON = 31.5 h^{-1}). The yields were increased compared to the system **2**(TPP)$_3$/PhIO and PhCHO was the only product.

The results from optimising the reaction conditions are shown in Tables 3 and 4. Longer reaction times are required when the reaction is scaled up, presumably due to the low solubility of the PhI=O in organic solvents because of its polymeric nature ([–(Ph)IO–]$_n$). The reaction worked well for ratios up to 1/1000/1000 and the residue remained active and could be reused several times (>10 in our hands). In aromatic solvents the rate of the reaction decreases, but becomes quantitative within a few hours under reflux (Table 4, entries 6 and 8). The use of MeCN as co-solvent lowers the yield (entry 9).

Addition of molecular sieves (3 Å, powder) to trap the water formed during alcohol oxidation had no effect on the reaction.

Therefore, the typical catalytic system which was used for the oxidation of a range of alcohols consists of **5** (1 eq), PhIO (50 eq), and alcohol (50 eq) at $T = 298$ K.

Representative results are presented in Table 5. Primary substituted benzyl alcohols were oxidized nearly quantitatively with total selectivity. *Para*- or *ortho*-substituents with electron releasing or withdrawing properties do not induce any change on the selectivity of the reaction, except in the case of o-$CH_3C_6H_4CH_2OH$ (entry 4) and p-$CH_3C_6H_4CH_2OH$ (entry 6), where the corresponding esters (7%) and traces of $CH_3C_6H_4CH_2I$ and $CH_3C_6H_4CH_2Cl$ (yield < 10^{-3}%) were detected at the end of the reaction. In the absence of **5** the yield of the ester was 5% in both cases. Introduction of –NO_2 groups lowers the yield (entries 9 and 10). Secondary benzyl alcohols stabilized by hyperconjugation (1-phenyl-1-ethanol, 2,2-dimethyl-1-phenyl-1-propanol), were not oxidized. Primary or secondary aliphatic alcohols (1-pentanol, 1-octanol, 2-octanol), also were not oxidized. When mixtures of $PhCH_2OH$ and 1-phenyl-1-ethanol or 1-octanol were used, only $PhCH_2OH$ was oxidized, although longer reaction times were required (entries 14 and 15). 1-[4-(Hydroxymethyl)phenyl]-1-ethanol was oxidized to 4-(1-hydroxyethyl)benzaldehyde and 4-acetylbenzaldehyde (entry 16). Allylic, homoallylic and poly-unsaturated alcohols (2-propen-1-ol, 3-buten-1-ol, (2*E*)-3,7-dimethyl-2,6-octadien-1-ol (geraniol), (2*Z*)-3,7-dimethyl-2,6-octadien-1-ol (nerol) and (2*E*,6*E*)-3,7,11-trimethyl-2,6,10-dodecatrien-1-ol (farnesol)) show reactivity similar to the substituted benzyl alcohols, but no selectivity (isomerisation and oxidation to aldehydes, acids, esters). This result is consistent with earlier reports that **5** in the presence of 4-methylbenzenesulfonic acid in CH_2Cl_2 [30–31] or MeReO$_3$ (MTO) by itself [32] catalyse the metathesis and dehydration of allylic alcohols.

Oxidation of alkenes with the same system (molar ratio **5**/PhIO/alkene = 1/50/50, CH_2Cl_2, $t = 24$ h, $T = 298$ K) was not effective. 2,3-Diphenyloxirane (10%) and benzil (10%) were the major products from the oxidation of 1-[(*Z*)-2-phenylethenyl]benzene (*cis*-stilbene). 1-Cyclohexene was oxidized to 2-cyclohexen-1-ol (10%)

and 7-oxabicyclo[4.1.0]heptane (2%) in low yields. In the absence of **5** no reaction occurred.

The described system is added to the arsenal of the relevant MO_4^{n-}/co-oxidant congeners (M = Mo [33], W [33], $n = 2$, M = Ru [6] $n = 1$), from which distinctly differs in terms of reactivity and selectivity. It also differs from the organometallic MTO/H_2O_2 [34] and MTO/H_2O_2/HBr [35] systems, which oxidize primary and secondary alcohols, but lack selectivity, as they produce mixtures of aldehydes and acids [35]. Addition of 2,2,6,6-tetramethyl-1-piperidinyloxy, free radical (TEMPO) to the latter system is required to achieve selectivity [36]. Recently, the system $CuBr_2$/2,2′-bipy/^{t}BuOK/TEMPO/MeCN/H_2O has been reported to catalyse aerobically the mild and selective oxidation of primary alcohols (benzylic and aliphatic) to aldehydes [37].

4. Conclusions

We report the first example of application of the ReO_4^- anion for the effective catalytic oxidation of a range of primary benzyl alcohols to the corresponding aldehydes having the following positive advantages: (a) excellent yields and selectivity towards primary benzylic hydroxy functionalities, which may be useful in fine product synthesis; (b) ease of operation; (c) repeated use of the catalyst without loss of activity; (d) use of a commercially available or easily prepared and safely stored derivative of I(III) than the usually employed explosive reagents of I(V) and (e) facile product work-up and recycling of the solvent and the co-oxidant, which makes the system environmentally conscious.

Further investigations aiming at improving the rate of the oxidation and providing mechanistic insight are in progress.

Acknowledgements

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