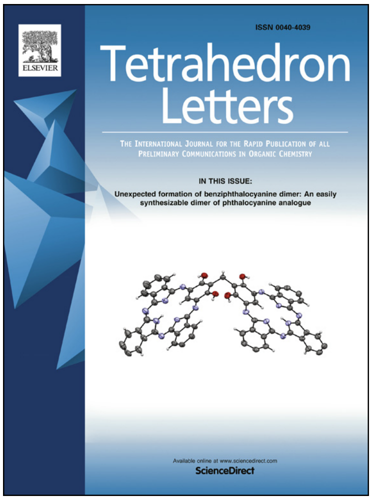

# An oxidovanadium(IV) complex having a perrhenato ligand: An efficient catalyst for aerobic oxidation reactions of benzylic and propargylic alcohols

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## Graphical Abstract

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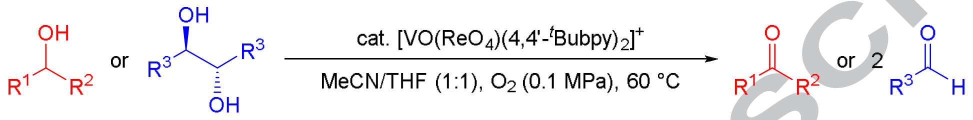

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# An oxidovanadium(IV) complex having a perrhenato ligand: An efficient catalyst for aerobic oxidation reactions of benzylic and propargylic alcohols

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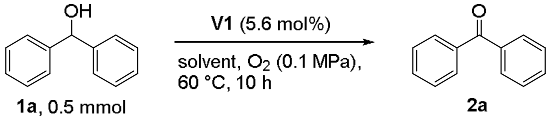
## ABSTRACT

An oxidovanadium(IV) complex having a perrhenato ligand [VO($ReO_4$)(4,4'-$^t$Bubpy)$_2$][0.25$SO_4$·0.5$ReO_4$] (4,4'-$^t$Bubpy = 4,4'-di-*tert*-butyl-2,2'-bipyridine) efficiently catalyzes not only dehydrogenative oxidation of benzylic and propargylic mono-alcohols but also oxidative C–C bond cleavage of *meso*-1,2-diaryl-1,2-ethanediols under atmospheric molecular oxygen, affording the corresponding carbonyl compounds in good yield.

Oxidation reactions of alcohols to the corresponding carbonyl compounds are among the most important transformations in synthetic organic chemistry. In particular, increasing attentions have been paid towards the transition-metal-catalyzed aerobic oxidation reactions due to both economic and environmental reasons.[1] Oxidovanadium complexes are known to act as catalysts for various aerobic alcohol oxidation reactions, including not only dehydrogenative oxidation of mono-alcohols but also oxidative C–C bond cleavage of 1,2-diols.[2] However, oxidovanadium complexes with the ability to effectively catalyze both dehydrogenative oxidation and oxidative cleavage reactions are still limited so far.

Herein, we report the synthesis and catalytic activities of an oxidovanadium(IV) complex having a perrhenato ligand [VO($ReO_4$)(4,4'-$^t$Bubpy)$_2$][0.25$SO_4$·0.5$ReO_4$] (**V1**; 4,4'-$^t$Bubpy = 4,4'-di-*tert*-butyl-2,2'-bipyridine). It is interesting to note that **V1** catalyzes dehydrogenative oxidation of secondary benzylic mono-alcohols to the corresponding ketones under $O_2$ (balloon) or atmospheric air more effectively than the structurally related oxidovanadium(IV) complex [VO(OH)(4,4'-$^t$Bubpy)$_2$][$BF_4$] (**V2**)[3] and other commercially available vanadium compounds (vide infra). In addition, oxidative cleavage of *meso*-1,2-diaryl-1,2-ethanediols proceeded selectively to afford the corresponding benzaldehydes under similar reaction conditions with **V1** as a catalyst.

**Table 1.** Effects of the solvents on oxidation of **1a** catalyzed by **V1** under $O_2$



| Entry | Solvent | Conv. (%)[a] | Yield of **2a** (%)[a] | Select. (%)[b] |
|---|---|---|---|---|
| 1 | MeCN | 48 | 30 | 63 |
| 2 | MeCN/THF (1:1) | 100 | 94 (97)[c] | 94 |
| 3 | THF | 56 | 42 | 75 |
| 4 | MeOH/THF (1:1) | 96 | 39 | 41 |
| 5 | EtOH/THF (1:1) | 96 | 18 | 19 |
| 6 | MeCN/2-MeTHF (1:1) | 64 | 39 | 61 |
| 7 | MeCN/1,4-dioxane (1:1) | 52 | 36 | 69 |

[a] Determined by $^1$H NMR using 1,2-diphenylethane as an internal standard. [b] Select. = (**2a** (mol)/**1a** consumed (mol))×100. [c] Isolated yield.

When oxidovanadium(IV) sulfate ($VOSO_4$) was allowed to react with potassium perrhenate ($KReO_4$) and 4,4'-$^t$Bubpy in $H_2O$/MeOH at room temperature, the reaction mixture turned into a green suspension. The precipitates were collected, washed with $H_2O$ and $Et_2O$, and dried in vacuo to afford **V1** as a green powder (29% yield). Positive fast atom bombardment mass spectrometry (FAB-MS) analysis of **V1** showed *m/z* (relative intensity) 854.6 (100, $M^+$) and 852.6 (68, $M^+$) attributable to the cationic complex

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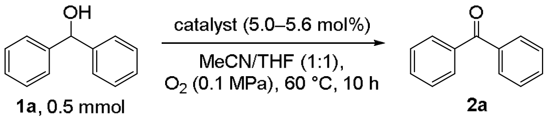
**Table 2.** Effects of catalysts on oxidation of **1a** under $O_2$



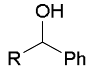
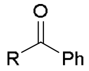
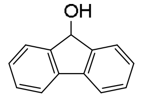
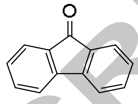
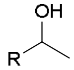
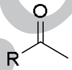
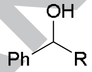
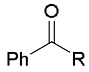
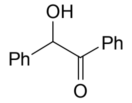
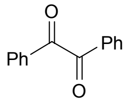
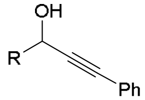
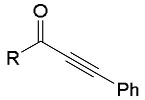
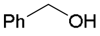
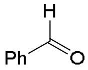
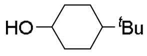
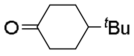
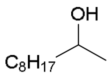
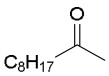
**1a**, 0.5 mmol

**2a**

| Entry | Catalyst | Conv. (%)[a] | Yield of **2a** (%)[a] | Select. (%)[b] |
|---|---|---|---|---|
| 1 | **V1** | 100 | 94 (97)[c] | 94 |
| 2 | **V2** | 21 | 5 | 24 |
| 3 | $VOSO_4$ | 68 | 32 | 47 |
| 4 | $KReO_4$[d] | 8 | 0 | 0 |
| 5 | [VO(acac)$_2$] | 29 | 14 | 48 |
| 6 | [$VOCl_3$] | 84 | 18 | 21 |
| 7 | [VO(O$^i$Pr)$_3$] | 44 | trace | – |
| 8 | $V_2O_5$ | 12 | 0 | 0 |
| 9 | $NH_4ReO_4$ | 2 | 0 | 0 |

[a] Determined by $^1$H NMR using 1,2-diphenylethane as an internal standard. [b] Select. = (**2a** (mol)/**1a** consumed (mol))×100. [c] Isolated yield. [d] 10 mol%.

**Table 3.** Oxidation of various benzylic mono-alcohols catalyzed by **V1** under $O_2$[a]

| Entry | Substrate | Product | Time (h) | Yield (%)[b] |
|---|---|---|---|---|
| |  |  | | |
| 1 | $R = C_6H_5$ (**1a**) | **2a** | 10 | 94 (97)[c] |
| 2 | R = 4-MeOC$_6$H$_4$ (**1b**) | **2b** | 10 | 88 (83)[c] |
| 3 | R = 4-ClC$_6$H$_4$ (**1c**) | **2c** | 10 | 88 (90)[c] |
| 4 | R = 4-NO$_2$C$_6$H$_4$ (**1d**) | **2d** | 10 | 48 |
| 5 | <br>**1e** | <br>**2e** | 10 | 93[d] |
| |  |  | | |
| 6 | $R = C_6H_5$ (**1f**) | **2f** | 96 | 76[d] |
| 7 | R = 4-MeOC$_6$H$_4$ (**1g**) | **2g** | 96 | 62[d] |
| 8 | R = 4-ClC$_6$H$_4$ (**1h**) | **2h** | 96 | 80[d] |
| |  |  | | |
| 9 | R = $^i$Pr (**1i**) | **2i** | 96 | 55 |
| 10 | R = $^c$Hex (**1j**) | **2j** | 96 | 68 |
| 11 | <br>**1k** | <br>**2k** | 1 | 83[d] |
| |  |  | | |
| 12 | $R = C_6H_5$ (**1l**) | **2l** | 1 | 88[d] |
| 13 | R = $CH_3$ (**1m**) | **2m** | 30 | 88[d] |
| 14 | <br>**1n** | <br>**2n** | 48 | 84 |
| 15 | <br>**1o** | <br>**2o** | 10 | n.d.[e] |
| 16 | <br>**1p** | <br>**2p** | 24 | n.d.[e] |

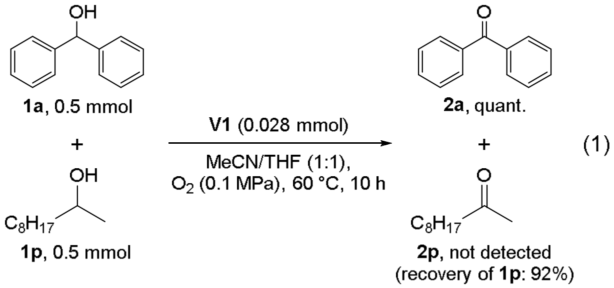
[a] Oxidation reactions were carried out using substrate (0.5 mmol), **V1** (0.028 mmol), and 3.0 mL of MeCN/THF (1:1) in a glass vessel equipped with $O_2$ balloon (0.1 MPa) at 60 °C for appropriate times. [b] Determined by $^1$H NMR using 1,2-diphenylethane as an internal standard. [c] Isolated yield. [d] Determined by $^1$H NMR using 1,3,5-trimethoxybenzene as an internal standard. [e] n.d. = not detected.

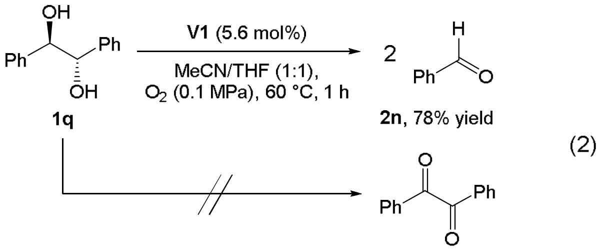
[VO(ReO$_4$)(4,4'-$^t$Bubpy)$_2$]$^+$ (calcd *m/z*: 854.3 (100%), 852.3 (59.7%)), the peak pattern of which is in good agreement with the isotopic distribution of this cation.[4,5]

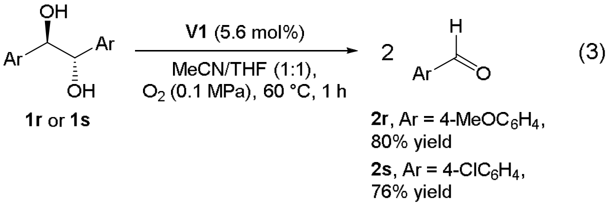
To investigate the catalytic activity of **V1** in an alcohol oxidation with $O_2$, diphenylmethanol (**1a**) was chosen as a model substrate. Oxidation of **1a** was performed in a glass vessel equipped with an $O_2$ balloon in the presence of 5.6 mol% of **V1** at 60 °C for 10 h in various solvents, and the results are summarized in Table 1. In MeCN as the solvent, benzophenone (**2a**) was formed in 30% yield (entry 1), whereas the yield of **2a** dramatically increased to 94% by using a mixed MeCN/THF (1:1) solvent (entry 2).[6] In THF (entry 3) and other mixed solvents (entries 4–7), **2a** was obtained in low to moderate (18–42%) yield.

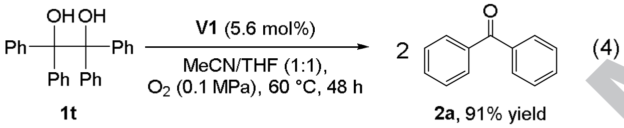
Under similar reaction conditions shown in Table 1, entry 2, oxidation of **1a** was examined using different catalysts (Table 2). When the oxidovanadium(IV) complex **V2**[3] which is structurally related to **V1** was employed as the catalyst, **2a** was formed in 5% yield (entry 2). $VOSO_4$ and $KReO_4$, the starting materials of **V1**, were not effective as catalysts (entries 3 and 4). Commercially available vanadium and perrhenate compounds such as [VO(acac)$_2$] (acac = acetylacetonato), [$VOCl_3$], [VO(O$^i$Pr)$_3$], $V_2O_5$, and $NH_4ReO_4$ also failed to exhibit appreciable catalytic activities and selectivities (entries 5–9).

Oxidation of various mono-alcohols catalyzed by **V1** was examined under the optimized reaction conditions (Table 1, entry 2), and the results are summarized in Table 3. Diarylmethanols **1b** and **1c** bearing methoxy or chloro substituents could be oxidized efficiently to the corresponding ketones **2b** and **2c** in high yield (entries 2 and 3), whereas the reaction with α-phenyl-4-nitrobenzenemethanol (**1d**) gave 4-nitrobenzophenone (**2d**) in 48% yield (entry 4). The oxidation of 9-fluorenol (**1e**) proceeded smoothly to afford 9-fluorenone (**2e**) in 93% yield (entry 5). 1-Phenylethanol (**1f**) and its derivatives **1g** and **1h** were oxidized slowly to the corresponding acetophenones **2f**–**2h** in moderate to good (62–80%) yield (entries 6–8). The oxidation of α-isopropylbenzenemethanol (**1i**) and α-cyclohexylbenzene-methanol (**1j**) proceeded more slowly than that of **1f** (entries 9 and 10), where steric hindrance of substrates is assumed to influence the reactivity of **V1**. In contrast, benzoin (**1k**) was qui-







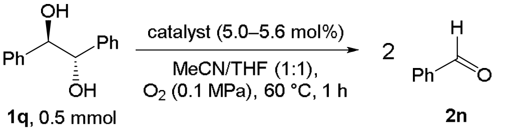


ckly converted into benzil (**2k**) in 83% yield (entry 11). The oxidation of 1,3-diphenylpropargyl alcohol (**1l**) also proceeded smoothly to form 1,3-diphenylpropynone (**2l**) in 88% yield, whereas the methyl analog of **1l** (**1m**) required a longer reaction time (30 h) to afford the corresponding propargylic ketone **2m**. Benzyl alcohol (**1n**) was oxidized selectively to benzaldehyde (**2n**) in 84% yield (entry 14). Aliphatic alcohols **1o** and **1p** were inert to oxidation (entries 15 and 16), which motivated us to examine the chemoselective oxidation reactions of two different alcohols. The reaction with a mixture of **1a** and **1p** resulted in the formation of **2a** in quantitative yield, while the aliphatic ketone **2p** was not detected (Eq. 1).

In addition, we expected that *meso*-1,2-diphenyl-1,2-ethanediol (**1q**) would be oxidized to benzil (**2k**) in the present catalytic system. The reaction with **1q**, however, afforded benzaldehyde (**2n**), the oxidative C–C bond cleavage[1b,2p–r] product, selectively in 78% yield (Eq. 2). *meso*-1,2-Diphenyl-1,2-ethanediols **1r** and **1s** bearing methoxy and chloro substituents were also oxidized to the corresponding aldehydes **2r** and **2s** in good yield, respectively (Eq. 3). The oxidative cleavage of benzopinacol (**1t**) proceeded more slowly than that of **1q**–**1s** to afford benzophenone (**2a**) in 91% yield, probably due to the steric congestion (Eq. 4).

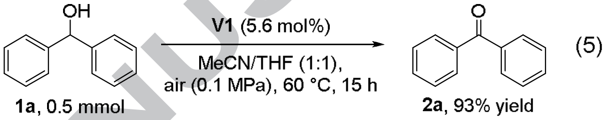
By using **1q** as a model substrate, we further investigated the catalytic activities of several vanadium complexes in the oxidative cleavage of 1,2-diols as shown in Table 4. In the case of **V2**, **2n** was obtained in 51% yield (entry 2). Among the commercially available vanadium compounds employed, [VOCl$_3$] exhibited similar catalytic activity to that of **V1** (entries 3–7).

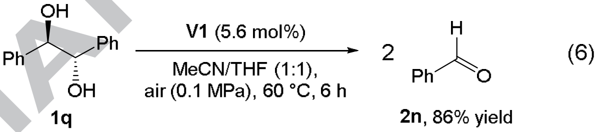
**Table 4.** Effects of vanadium catalysts on oxidative cleavage of **1q** under O$_2$

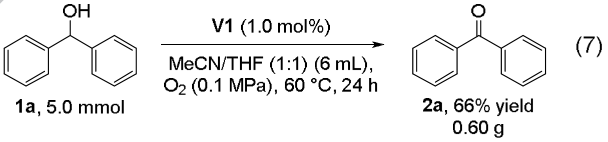


| Entry | Catalyst | Conv. (%)[a] | Yield of **2n** (%)[a] | Select. (%)[b] |
|---|---|---|---|---|
| 1 | **V1** | 100 | 78 | 78 |
| 2 | **V2** | 84 | 51 | 61 |
| 3 | VOSO$_4$ | 98 | 39 | 40 |
| 4 | [VO(acac)$_2$] | 88 | 58 | 66 |
| 5 | [VOCl$_3$] | 100 | 83 | 83 |
| 6 | [VO(O$^i$Pr)$_3$] | 81 | 56 | 69 |
| 7 | V$_2$O$_5$ | 14 | trace | – |

[a] Determined by $^1$H NMR using 1,3,5-trimethoxybenzene as an internal standard. [b] Select. = (**2n** (mol)/**1q** consumed (mol))×100.







Although some oxidovanadium complexes [VO(acac)$_2$], [VOCl$_3$], and [VO(O$^i$Pr)$_3$] were found to exhibit moderate to good catalytic activities toward the oxidative cleavage, these complexes were not effective as catalysts for the dehydrogenative oxidation of mono-alcohol **1a** (Tables 2 and 4). It is interesting to note that the present results with **V1** provide a rare example of the vanadium catalyst that is highly effective in both dehydrogenative alcohol oxidation and oxidative cleavage of 1,2-diols with O$_2$.

Furthermore, the oxidations of **1a** and **1q** catalyzed by **V1** were examined using ambient air as the oxidant. To our delight, the corresponding oxidation products **2a** and **2n** were obtained in 93% and 86% yield, respectively, by elongating the reaction time (Eqs. 5 and 6).

Finally, gram-scale oxidation reaction of **1a** with O$_2$ was carried out by reducing the catalyst loading (1.0 mol% of **V1**) to demonstrate the utility of the present catalytic system. As expected, **2a** was isolated in 66% yield (Eq. 7).

In conclusion, we have successfully revealed that the newly synthesized oxidovanadium(IV) complex having a perrhenato ligand **V1**[8] exhibits efficient catalytic activities towards the aerobic oxidation reactions of secondary benzylic mono-alcohols and *meso*-1,2-diaryl-1,2-ethanediols.[9] Further studies to elucidate the effect of a perrhenato ligand and detailed reaction mechanisms together with oxidations of other organic substrates using the present catalytic system are now in progress.

## Acknowledgments

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6. Although we must await further studies to clarify the role of THF in the present catalytic system, we inferred that THF promotes the oxidation of the vanadium(IV) complex **V1** by $O_2$[7] to form the vanadium(V) complex which often acts as active species for the aerobic oxidation of alcohols.[2]
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8. Synthesis of $[VO(ReO_4)(4,4'\text{-}^t\text{Bubpy})_2][0.25SO_4\cdot 0.5ReO_4]$ (**V1**): To an aqueous solution of $VOSO_4\cdot 5H_2O$ (50.7 mg, 0.20 mmol) and $KReO_4$ (232.0 mg, 0.80 mmol) was added a MeOH solution of 4,4'-di-*tert*-butyl-2,2'-bipyridine (107.3 mg, 0.40 mmol), and the resulting green suspension was stirred at room temperature for 10 min. The green precipitates were collected, washed with $H_2O$ and $Et_2O$, and dried in vacuo to afford **V1** as a green powder (57.5 mg, 29% yield). IR (KBr, $cm^{-1}$) 2964, 1617, 1411, 979, 912; Anal. Calcd for $C_{36}H_{48}N_4O_8Re_{1.5}S_{0.25}V$: C, 43.11; H, 4.82; N, 5.59. Found: C, 43.05; H, 4.84; N, 5.55%.
9. General procedure: **V1** (27.6 mg, 0.028 mmol) and substrate (0.5 mmol) were placed in a 20 mL schlenk flask, and then the mixed MeCN/THF (1:1) solvent (3 mL) was added. The mixture was stirred at 60 °C for an appropriate time under $O_2$ (balloon) or atmospheric air. After the reaction was completed, $Et_2O$ was added to the reaction mixture. The resulting suspension was filtered and rinsed with $Et_2O$. The combined filtrate was concentrated in vacuo. The yield of the oxidation products was confirmed by $^1H$ NMR spectroscopy using 1,2-diphenylethane or 1,3,5-trimethoxybenzene as the internal standard. Purification of **2a**–**2c** was performed by silica gel chromatography using hexane and EtOAc as the eluent to afford the analytically pure ketones. The isolated products were identified by comparing their $^1H$ NMR spectra with those of authentic samples.

# An oxidovanadium(IV) complex having a perrhenato ligand: An efficient catalyst for aerobic oxidation reactions of benzylic and propargylic alcohols

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## *Highlights*

An oxidovanadium(IV) complex having a perrhenato ligand (**V1**) has been synthesized.

**V1** exhibits high catalytic activities for alcohol oxidation reactions with $O_2$.

Dehydrogenative oxidation and oxidative cleavage reaction of alcohols are described.

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