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# Efficient regio- and stereoselective ring opening of epoxides with alcohols, acetic acid and water catalyzed by ammonium decatungstocerate(IV)

Valiollah Mirkhani,[a,*] Shahram Tangestaninejad,[a] Bahram Yadollahi[b] and Ladan Alipanah[a]

[a]*Chemistry Department, Isfahan University, Isfahan 81746-73441, Iran*
[b]*Institute for Advanced Studies in Basic Sciences(IASBS), Gava Zang, Zanjan 45138-79368, Iran*

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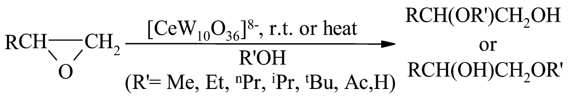
**Abstract**—Epoxides can be cleaved in a regio- and stereoselective manner under neutral conditions with alcohols and acetic acid in the presence of catalytic amounts of decatungstocerate(IV) ion, ($[CeW_{10}O_{36}]^{8-}$), affording the corresponding β-alkoxy and β-acetoxy alcohols in high yields. In water, ring opening of epoxides occurs with this catalyst to produce the corresponding diols in good yields.

## 1. Introduction

Epoxides and β-alkoxy alcohols are important intermediates in organic synthesis.[1] Reactions of epoxides frequently constitute crucial process in the transformation of aliphatic compounds due to high regio- and stereoselectivity and specificity.[2] Epoxide ring-opening reactions to give β-substituted alcohols with carbon- and heteroatomic nucleophiles, rearrangement reactions providing carbonyl compounds, and isomerization reactions leading to allylic alcohols are useful tools in organic synthesis and most of these reactions generally proceed under basic or acidic conditions. Several reagents have been reported in the literature that can promote ring opening of epoxides. Commerically available Woelm 200 neutral alumina[3] has been applied as a mild and heterogeneous reagent in the presence of alcohols and acetic acid. The conversion of cyclohexene oxide to *trans*-1,2-diol has been reported by the action of water in the presence of Nafion-H,[4] a perfluorinated sulfonic acid resine catalyst. Other reagents such as organotin phosphate condensate,[5] lithium and magnesium perchlorate,[6] tin tetrachloride,[7] diethyl ether–boron trifluoride (1/1),[8] trialkylaluminium,[9] iron(III) chloride,[10] iron(III) chloride supported on silica-gel,[11] dehydrated alumina,[12] lanthanoid complexes,[13] bismuth(III) chloride[14] and iron(III) trifluoroacetate[15] appear to be versatile promoters for heteroatomic nucleophilic ring opening of epoxides. According to the procedures reported so far, the efficiency of these promoters is still unsatisfactory, because they need significant amounts of the promoters in order to attain a high yield of the desired products except for reactions at elevated temperature. There have been some reports on regio- and stereoselective ring-opening reactions of oxaziridines and epoxides promoted by metalloporphyrin and metal Schiff base complexes, which act as Lewis acids.[16–22] Recently, some one-electron transfer catalysts, such as 2,3-dichloro-5,6-dicyano-*p*-benzoquinone (DDQ) and tetracyanoethylene (TCNE) have also been reported for the alcoholysis, acetolysis and thiolysis of epoxides.[23–25]

Heteropolyoxometalates as oxidatively stable inorganic porphyrins have received much attention in the preceding two decades.[26,27] The versatility of these catalysts has also been demonstrated by applications in various oxidation reactions. However, there has been very little exploitation of their catalytic reactivity in nucleophilic ring-opening of epoxides.[28]

In this paper, we wish to report that $(NH_4)_8[CeW_{10}O_{36}]\cdot 20H_2O$ can act as a mild and highly effective heterogeneous catalyst for the efficient regio- and stereoselective ring-opening of epoxides with various nucleophiles such as primary, secondary or tertiary alcohols, water and acetic acid (Scheme 1).



**Scheme 1.**

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*Keywords*: catalysis; ring opening; epoxides; alcohols; hydrolysis.

[*] Corresponding author. Tel.: +98-311-7932713; fax: +98-311-6689732; e-mail: mirkhani@sci.ui.ac.ir

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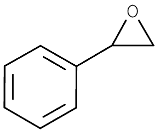
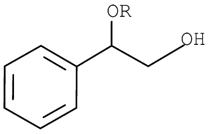
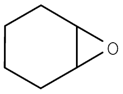
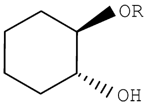
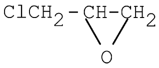
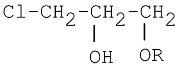
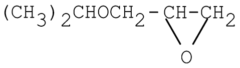
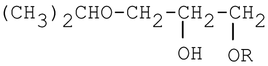
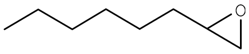
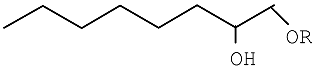
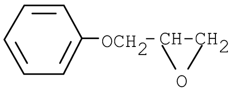
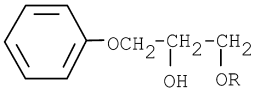
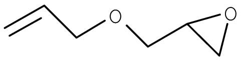
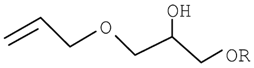
**Table 1**. Alcoholysis of various epoxides catalyzed by decatangestocerate(IV)[a]

| Entry | Epoxide | R-OH/condition | Time (min) | Product/yield[b,c] | Bp (°C/Torr) or $n_D^{25}$ | |
|---|---|---|---|---|---|---|
| | | | | | Found | Reported[10,11,33,34] |
| |  | | |  | | |
| 1 | | $R{=}CH_3$/RT | 5 | 97 | 74–76/0.7 | 75/0.7 |
| 2 | | $R{=}C_2H_5$/reflux | 10 | 95 | 1.5146 | 1.5143 |
| 3 | | R=*n*-Pr/reflux | 10 | 93 | 1.5109 | 1.5106 |
| 4 | | R=*i*-Pr/reflux | 15 | 87 | 1.5074 | 1.5075 |
| 5 | | R=*t*-Bu/reflux | 60 | 80 | 1.5129 | 1.5130 |
| |  | | |  | | |
| 6 | | $R{=}CH_3$/R. T. | 10 | 97 | 108–111/65 | 107–111/65 |
| 7 | | $R{=}C_2H_5$/reflux | 10 | 94 | 82–85/15 | 80–85/15 |
| 8 | | R=*n*-Pr/reflux | 30 | 90 | 94–97/14 | 95–97/14 |
| 9 | | R=*i*-Pr/reflux | 60 | 83 | 90–93/18 | 89–93/18 |
| 10 | | R=*t*-Bu/reflux | 60 | 64 | 90–92/15 | 86–91/14 |
| |  | | |  $\mathrm{Cl{-}CH_2{-}\underset{OH}{CH_2}{-}\underset{OR}{CH_2}}$ | | |
| 11 | | $R{=}CH_3$/reflux | 60 | 86 | 98–100/9 | 105/10 |
| 12 | | $R{=}C_2H_5$/reflux | 60 | 89 | 103–106/8 | 108–110/10 |
| 13 | | R=*n*-Pr/reflux | 60 | 95 | 1.4382 | 1.4391 |
| 14 | | R=*i*-Pr/reflux | 60 | 93 | 107–110/10 | 108–110/10 |
| 15 | | R=*t*-Bu/reflux | 60 | 97 | 1.4436 | 1.4435 |
| |  | | |  $\mathrm{(CH_3)_2CHO{-}CH_2{-}\underset{OH}{CH_2}{-}\underset{OR}{CH_2}}$ | | |
| 16 | | $R{=}CH_3$/reflux | 10 | 97 | 202–204 | 200–203 |
| 17 | | $R{=}C_2H_5$/reflux | 30 | 91 | 207–209 | 209 |
| 18 | | R=*n*-Pr/reflux | 30 | 90 | 1.4290 | 1.4294 |
| 19 | | R=*i*-Pr/reflux | 30 | 86 | 1.4199 | 1.4196 |
| 20 | | R=*t*-Bu/reflux | 30 | 85 | 1.4050 | 1.4055 |
| |  | | |  | | |
| 21 | | $R{=}CH_3$/reflux | 15 | 95 | 85–87/6 | 84–86/6 |
| 22 | | $R{=}C_2H_5$/reflux | 15 | 94 | 127–130/11 | 129–131/11 |
| 23 | | R=*n*-Pr/reflux | 30 | 89 | 1.4315 | 1.4318 |
| 24 | | R=*i*-Pr/reflux | 60 | 85 | 1.4326 | 1.4327 |
| 25 | | R=*t*-Bu/reflux | 60 | 82 | 1.4410 | 1.4413 |
| |  | | |  | | |
| 26 | | $R{=}CH_3$/reflux | 10 | 95 | 120/123/3 | 121–123/3 |
| 27 | | $R{=}C_2H_5$/reflux | 15 | 93 | 1.5147 | 1.5146 |
| 28 | | R=*n*-Pr/reflux | 15 | 96 | 1.5043 | 1.5042 |
| 29 | | R=*i*-Pr/reflux | 15 | 88 | 1.4974 | 1.4972 |
| 30 | | R=*t*-Bu/reflux | 15 | 85 | 1.4951 | 1.4950 |
| |  | | |  | | |
| 31 | | $R{=}CH_3$/reflux | 10 | 97 | 1.4462 | 1.4460 |
| 32 | | $R{=}C_2H_5$/reflux | 30 | 89 | 1.4370 | 1.4375 |
| 33 | | R=*n*-Pr/reflux | 30 | 89 | 1.4384 | 1.4380 |
| 34 | | R=*i*-Pr/reflux | 30 | 88 | 1.4366 | 1.4362 |
| 35 | | R=*t*-Bu/reflux | 30 | 85 | 1.4584 | 1.4580 |

[a] Catalyst 0.04 M amount.

[b] All products were identified by comparison of their physical and spectral data with those of authentic samples.

[c] Yields refer to isolated products.

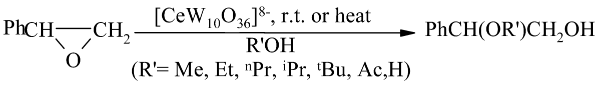
## 2. Results and discussion

At first, we examined the methanolysis of styrene oxide using catalytic amounts of various polyoxometallate complexes (0.04 M) at room temperature. Although the reaction without a catalyst gave no product, it proceeded catalytically upon addition of polyoxometallate complexes. Among the catalysts tested, $[CeW_{10}O_{36}]^{8-}$ was found to be highly active for the reaction. The catalytic activity of the catalysts appear to be in the following order: $(NH_4)_8[CeW_{10}O_{36}]\cdot 20H_2O$ (95%)$\gg$$(NH_4)_8[CeMo_{12}O_{42}]\cdot 8H_2O$ (33%)>$\alpha$-$K_7PW_9Mo_2O_{39}$ (28%)>$\alpha$-$K_4PFeW_9Mo_2O_{39}\cdot 3H_2O$ (20%)>$\alpha$-$K_5PCoW_9Mo_2O_{39}\cdot 3H_2O$ (11%)>$\alpha_2$-$K_7P_2W_{17}O_{62}(Mn^{2+}, H_2O)\cdot 12H_2O$ (5%)>$\beta$-$Na_8HPW_9O_{34}$ (3%). Even though the reaction mechanism is interesting, at the present time it is obscure to us, and we have not been able to assign any reasonable mechanism in our studies.

### 2.1. Alcoholysis of epoxides catalyzed by $[CeW_{10}O_{36}]^{8-}$

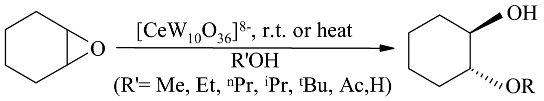
In the presence of $[CeW_{10}O_{36}]^{8-}$, the alcoholysis of different classes of epoxides was performed with primary, secondary and tertiary alcohols. Table 1 gives the results obtained for the reactions of some representative epoxides with catalytic amounts of $[CeW_{10}O_{36}]^{8-}$.

Reactions of styrene oxide as an active epoxide were carried out at room temperature in methanol and reflux in ethanol, 1-propanol, 2-propanol and 2-methyl-2-propanol in the presence 0.04 M amount of $[CeW_{10}O_{36}]^{8-}$, which afforded 2-alkoxy-2-phenyl ethanol in 80–97% isolated yields within 5–60 min with no formation of other regioisomers (Table 1, entries 1–5) (Scheme 2).



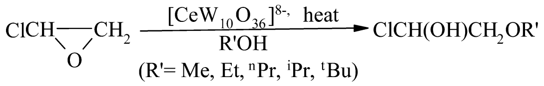
Scheme 2.

The reactions of cyclohexene oxide were slightly slower than those of styrene oxide. Although the reactions of primary and secondary alcohols were nearly complete in about 10–60 min in the presence of 0.04 M amount of the catalyst at room temperature or reflux (Table 1, entries 6–9), in 2-methyl-2-propanol a decrease in the reaction rate was observed and *trans*-2-*t*-butoxycyclohexanol was obtained after 75 min in only 64% yield (Scheme 3). In all of the reactions involving cyclohexene oxide only trans products were obtained.



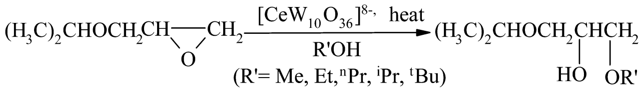
Scheme 3.

In order to see the effect of polar electron-withdrawing groups adjacent to the epoxide's ring, the reactions of (chloromethyl)oxirane were studied (Scheme 4). As expected, the reaction rates were considerably decreased. All of these reactions were performed under reflux conditions (Table 1, entries 11–15). In all of these reactions only one regioisomer was obtained.



Scheme 4.

For isopropyl oxiranylmethyl ether, which has a deactivating group containing an ethereal linkage, all of the reactions were performed under reflux conditions (Table 1, entries 16–20). Cleavage of ethereal linkage was not observed in these reactions (Scheme 5).

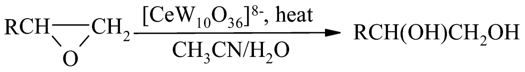


Scheme 5.

Alcoholysis of other epoxides, such as oxiranylmethylphenyl ether, allyl oxiranylmethyl ether and 1,2-epoxyoctane, were performed under refluxing conditions (Table 1, entries 21–35). Our results showed that the etheral linkage and the double bond remain intact during the course of the alcoholysis reaction of the epoxide ring. For these unsymmetrical epoxides, the alkoxy group is incorporated preferentially at the less-hindered epoxide carbon atom.

### 2.2. Hydrolysis and acetolysis of epoxides catalyzed by $[CeW_{10}O_{36}]^{8-}$

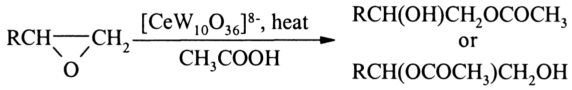
Epoxides were converted to the corresponding diols as the sole product in the presence of 0.04 M amount of $[CeW_{10}O_{36}]^{8-}$ in an acetonitrile/water mixture (Table 2) (Scheme 6). All of these reactions were performed under reflux conditions.



Scheme 6.

Different classes of epoxides were converted to their corresponding $\beta$-acetoxy alcohols in the presence of 0.04 M amount of $[CeW_{10}O_{36}]^{8-}$ as a catalyst in acetic acid (Table 2).

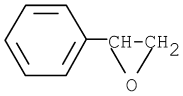
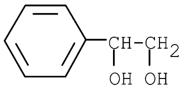
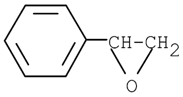
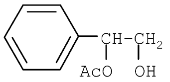
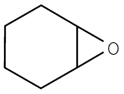
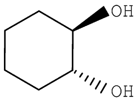
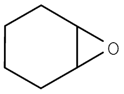
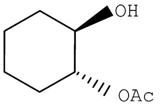
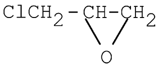
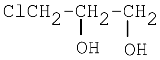
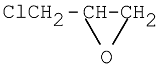
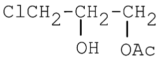
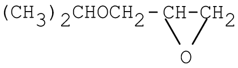
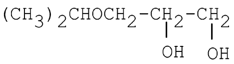
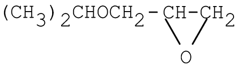
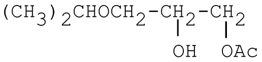
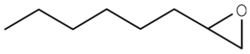
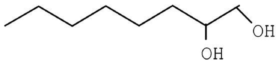
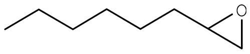
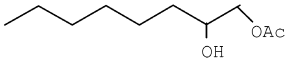
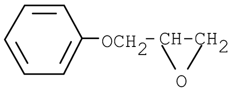
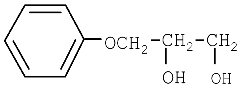
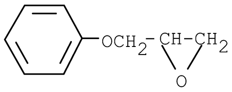
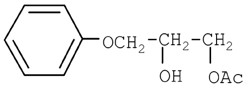
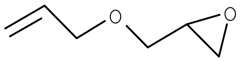
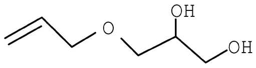
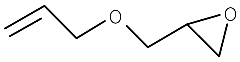
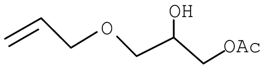
The reactions of epoxides require reflux conditions for complete conversion to their corresponding $\beta$-acetoxy alcohols. As shown in Table 2, a highly regioselective formation of $\beta$-acetoxy alcohols was observed in all cases involving unsymmetrical epoxides (Scheme 7).



Scheme 7.

In addition, we have found that $[CeW_{10}O_{36}]^{8-}$ can be reused several times without loss of activity simply by filtering the catalyst, washing it with acetone, and drying. The yield of 2-methyl-2-phenylethanol starting from styreneoxide promoted by the catalyst recovered four times remained 97%; even after eight reuses, the yield was still about 92%.

**Table 2**. Hydrolysis and acetolysis of various epoxides catalyzed by decatangestocerate(IV) under reflux[a]

| Entry | Epoxide | Solvent | Time (min) | Product[b] | Yield[c] | Mp, bp (°C/Torr) or $n_D^{25}$ | |
|---|---|---|---|---|---|---|---|
| | | | | | | Found | Reported[10,11,33,34] |
| 1 |  | $H_2O/CH_3CN$ | 60 |  | 87 | 65–68 | 65–67 |
| 2 |  | $CH_3CO_2H$ | 30 |  | 58 | 163–165/15 | 163–170/15 |
| 3 |  | $H_2O/CH_3CN$ | 60 |  | 97 | 104–105 | 105 |
| 4 |  | $CH_3CO_2H$ | 30 |  | 97 | 1.5416 | 1.5418 |
| 5 |  | $H_2O/CH_3CN$ | 120 |  | 80 | 119–122/18 | 115–120/18 |
| 6 |  | $CH_3CO_2H$ | 60 |  | 89 | 1.4753 | 1.4755 |
| 7 |  | $H_2O/CH_3CN$ | 60 |  | 90 | 150–152/9 | 150–152/9 |
| 8 |  | $CH_3CO_2H$ | 30 |  | 97 | 1.4186 | 1.4182 |
| 9 |  | $H_2O/CH_3CN$ | 60 |  | 87 | 130–134/10 | 131–134/10 |
| 10 |  | $CH_3CO_2H$ | 30 |  | 93 | 1.4160 | 1.4162 |
| 11 |  | $H_2O/CH_3CN$ | 60 |  | 97 | 200–202/22 | 145–148/0.6 |
| 12 |  | $CH_3CO_2H$ | 60 |  | 89 | 174–177/10 | 175/10 |
| 13 |  | $H_2O/CH_3CN$ | 60 |  | 82 | 1.5580 | 1.5584 |
| 14 |  | $CH_3CO_2H$ | 30 |  | 90 | 1.4379 | 1.4380 |

[a] Catalyst 0.04 M amount.

[b] All products were identified by comparison of their physical and spectral data with those of authentic samples.

[c] Yields refer to isolated products.

## 3. Conclusion

In comparison with the results reported for some other reagents, such as $FeCl_3$,[10] $FeCl_3/SiO_2$,[11] ceric ammonium nitrate (CAN),[23] $BiCl_3$[14] and lanthanoide triflates[29,30] the use of $[CeW_{10}O_{36}]^{8-}$ is more suitable. The use of anhydrous $FeCl_3$ suffers from the highly hygroscopic nature of the reagent. $FeCl_3/SiO_2$ has the disadvantages of bulk use of the reagent, and requires a large amount of the solvent. In the cases of CAN and $BiCl_3$, in many reactions, large amounts of the catalysts (up to 0.1–0.8 M ratio) have been used to complete the reaction. It is worth mentioning that lanthanoide triflates are very expensive reagents. Therefore, we have introduced $[CeW_{10}O_{36}]^{8-}$ as a nonhygroscopic, non-toxic, available, stable, reusable and heterogeneous catalyst for the reactions of epoxides with alcohols, acetic acid and water. In addition, the advantages such as mild, high regio- and stereoselectivity, ease of preparation, simple filtration at the end of reaction, cheapness of the catalyst, high yields and short reaction times, give a new synthetic application for $[CeW_{10}O_{36}]^{8-}$ in organic synthesis.

## 4. Experimental

### 4.1. General

Products were characterized by comparison of their physical data with those of known samples. All yields refer to isolated products. The ammonium decatungstocerate(IV) icosohydrate was prepared according to the literature.[31,32] All catalysts were characterized by UV–Vis and IR spectroscopy and elemental analysis. Infrared spectra were recorded on a Philips PU-9716 or Shimadzu IR-435 spectrophotometer. $^1$H NMR spectra were recorded on a Bruker AW 80 (80 MHz) spectrometer. All analyses were performed on a Shimadzu GC-16A instrument with a flame-ionization detector using silicon D.C.-200 or carbowax 20-M columns. Mass spectra were recorded on a Shimadzu GCMS-QP 1000 EX. A microanalysis was carried out by the Research Institute of Petroleum Industry. Chemicals were purchased from Fluka and Merck chemical companies. The chemical purities of all epoxides were checked by gas chromatography and confirmed to be higher than 98%.

### 4.2. Catalytic reactions of styrene oxide with different polyoxometallates in methanol

A mixture of styrene oxide (120 mg, 1 mmol) and polyoxometallate complex (0.04 mmol) in methanol (5 mL) was stirred at room temperature. The progress of the reaction was monitored by GLC. After completion of the reaction, the suspension was filtered and washed with ether. Evaporation of the solvent, followed by chromatography on silica-gel, afforded pure 2-methoxy-2-phenylethanol as a colorless liquid.

### 4.3. Alcoholysis of epoxides; General procedure

To a solution of epoxide (1 mmol) in the appropriate alcohol (5 mL), $(NH_4)_8[CeW_{10}O_{36}]\cdot 20H_2O$ (122.3 mg, 0.04 mmol) was added and the mixture stirred at room temperature or under reflux. The reaction was monitored by gas chromatography. After completion of the reaction, the suspension was filtered and washed with ether. The eluate was concentrated under reduced pressure and chromatographed on a silica-gel column to give a pure product in 64–97% yields (Table 1).

### 4.4. Acetolysis of epoxides; General procedure

A mixture of epoxides (1 mmol) in acetic acid (5 mL) and $(NH_4)_8$ $[CeW_{10}O_{36}]\cdot 20H_2O$ (122.3 mg, 0.04 mmol) was stirred under reflux for 30–60 min. The solvent was evaporated, ether was added and the mixture filtered. The filtrate was washed with 5% aqueous sodium bicarbonate and then with water and dried ($MgSO_4$). Evaporation of the solvent followed by column chromatography on silica-gel gave the product in 89–97% yields (Table 2).

### 4.5. Hydrolysis of epoxides; General procedure

To a solution of epoxide (1 mmol) in an equal mixture of $CH_3CN/H_2O$ (5 mL) was added to $(NH_4)_8[CeW_{10}O_{36}]\cdot 20H_2O$ (122.3 mg, 0.04 mmol). The mixture was stirred for a specified time under reflux condition according to Table 2. The progress of the reaction was monitored by GLC. After completion of the reaction, the mixture was filtered and washed with ether. The eluate was concentrated under reduced pressure and chromatographed on a silica-gel column to give a pure product in 80–97% yields.

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