

# Efficient and highly-selective cycloaddition of epoxides with carbonyl compound over Wells–Dawson type heteropolyacids

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## Abstract

Wells–Dawson type Heteropolyacids ( $H_{6+n}P_2Mo_{18-n}V_nO_{62}$ ) ( $n = 0–2, 4$ ) have proved to be efficient heterogeneous catalysts for the reaction of epoxides with carbonyl compounds to form 1,3-dioxolane derivatives. Especially, when  $H_7P_2Mo_{17}VO_{62}$  was used to catalyze the reaction between acetone and epoxypropane, the selectivity to 1,3-dioxolane reached to 93.3% with nearly complete conversion of epoxide and  $1.1 \times 10^4 h^{-1}$  of TOF.

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**Keywords:** Cycloaddition; Wells–Dawson type molybdoxovanadophosphoricacids; Epoxides; Carbonyl compound; 1,3-Dioxolane

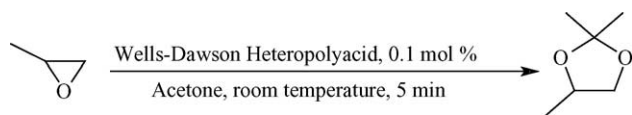
## 1. Introduction

Epoxides are important intermediates in organic synthesis [1] and their reactions with different nucleophiles under both nonsolvolytic [1,2] and solvolytic conditions have been the subject of extensive studies [3]. Direct conversion of epoxides into 1,3-dioxolanes with carbonyl compounds instead of adding water to form a diol with subsequent elimination is a subject of interest due to the importance of 1,3-dioxolanes as widely used protecting groups for diols and particularly for carbohydrates and steroid chemistry [4]. In addition, compounds with 1,3-dioxolanes structure or moiety are of interest in the manufacture of pharmaceuticals and fragrances. 1,3-Dioxolanes were usually prepared from oxiranes with carbonyl compounds in the presence of

Lewis or Brønsted acid catalysts [5]. The use of  $BF_3 \cdot OEt_2$ ,  $SnCl_4$ ,  $TiCl_4$  [6], KSF clay [7],  $HBF_4$  [8] and some other Lewis acids has been investigated for this transformation. Among these Lewis acids,  $BF_3 \cdot OEt_2$  has been successfully used for conversion of different types of carbonyl compounds to their corresponding 1,3-dioxolane derivatives with only ethylene and propylene oxides [6], whereas  $SnCl_4$  and  $TiCl_4$  gave little or no acetal [5].  $TiO(TFA)_2$ ,  $TiCl_3(OTf)$  [9],  $RuCl_3$  [10] were also very effective catalysts for this reaction. The achieved yields were comparable to those observed with  $BF_3$ . Anhydrous copper sulfate [11] was also shown to be a suitable catalyst for the reaction of acetone and epoxides, resulting in convenient rates and good yields [11]. Noble metal catalysts such as  $[Cp^*Ir(NCMe)_3]^{2+}$  [12] and  $CH_3ReO_3$  [13] and *N*-benzylpyridium hexafluoroantimonates [14] can also catalyze the title reaction. Some solid materials, zeolites (ZSM-5, Y and ultra stabilized HUSY)K10 montmorillonite [5], can also be used to produce

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Scheme 1.

the dioxolanes derivatives from epoxides and carbonyl compounds.

Heteropoly compounds have many advantages as catalysts that make them economically and environmentally attractive both in academic and industrial chemistry. Heteropoly compounds are effective catalysts for various reactions and have high capability in practical uses, because their redox and acidic properties can be controlled at the atomic/molecular levels by changing the constituent elements as per the needs of the chemical process [15]. Keggin-type heteropolyacids have been investigated for conversion of epoxides with acetone to give 1,3-dioxolanes, but polymerization and isomerization of epoxides greatly inhibit the formation of 1,3-dioxolanes [13]. Recently, Habibi [16] and Mirkhani [17] have reported the conversions of epoxides to 1,3-dioxolanes in the presence of  $K_5CoW_{12}O_{40} \cdot 3H_2O$  and  $(NH_4)_8[CeW_{10}O_{36}] \cdot 20H_2O$ , respectively. Although the substrate could also be completely consumed over  $(NH_4)_8[CeW_{10}O_{36}] \cdot 20H_2O$ , the prolonged reaction time (1.5–5 h) or/and the high temperature (reflux conditions) are needed. Therefore, it is necessary to develop a more effective and convenient catalytic system for this reaction. Here, we first report a novel and extremely efficient method for conversion of epoxides to their corresponding 1,3-dioxolanes using Wells–Dawson heteropoly acids as catalysts (Scheme 1).

## 2. Experimental

### 2.1. Materials and reagents

All reagents used were highest purity commercial products (99%), and applied to the reaction without further purification. Keggin-type heteropolyacids, such as 12-tungstophosphoric acid ( $H_3PW_{12}O_{40} \cdot 25H_2O$ ), 12-tungstosilicic acid ( $H_4SiW_{12}O_{40} \cdot 26H_2O$ ) and 12-molybdophosphoric acid ( $H_3PMo_{12}O_{40} \cdot 28H_2O$ ), were purchased from Beijing Chemical Reagent Co. with CP. Before use, these reagents were dried at 60 °C for 48 h.

### 2.2. Preparation of catalysts

The catalyst  $H_6P_2W_{18}O_{62}$  was prepared according to previous method [18].  $H_7P_2W_{17}VO_{62}$  was prepared from an aqueous solution of  $\alpha_2$ - $K_7P_2W_{17}VO_{62}$  [19].  $H_6P_2Mo_{18}O_{62}$  was prepared according to the report of Wu [20].  $H_{6+n}P_2Mo_{18-n}V_nO_{62}$  ( $n = 1, 2, 4$ ) was synthesized according to Ref. [21].  $H_4PMo_{11}VO_{40}$  were prepared according to Ref. [22]. The structure of the prepared catalysts was confirmed by

FTIR, UV–vis, thermal gravimetric analysis (TGA) and elemental analysis, and the results were all satisfactory. Purities of all prepared heteropolyacid catalysts are >98% according to the above-mentioned analysis.

### 2.3. Catalytic reactions

#### 2.3.1. Reaction procedure

All reactions were carried out in a magnetically stirred flask equipped with a dropping funnel and a condenser. The heteropolyacid 0.1 mol% based on the oxirane was used. The oxirane was added to a mixture of the catalyst and the carbonyl compound through a dropping funnel within 5 min at room temperature.

#### 2.3.2. Products analysis

Quantitative and qualitative analysis of organic components were conducted with a HP6890/5973 GC/MS and a Techcomp GC-7890 II equipped with an FID detector. The concentration of reactant and product was directly given by the system of GC chemstation according to the area of each chromatograph peak. The GC yield was obtained in a Perkin-Elmer AutoSystem XL GC equipped with an FFAP (50 m × 0.25 mm column) using chlorobenzene as internal standard.

## 3. Results and discussion

### 3.1. Effect of catalysts on the cycloaddition of epoxides with carbonyl compound

Table 1 shows the results of the cycloaddition reaction of 1,2-epoxypropane with acetone over various types of heteropolyacids. The selectivity to 2,2,4-trimethyl-1,3-dioxolane was strongly dependent on the kinds of the HPA catalysts while the conversions of 1,2-epoxypropane were not sensitive and are all higher than 95% in the presence of catalysts (Entries 1–9). No reaction was observed in the absence of catalyst (Entry 10). Compared with Wells–Dawson type heteropolyacids ( $H_{6+n}P_2Mo_{18-n}V_nO_{62}$ ) ( $n = 0–2, 4$ ) were preferable to Keggin type heteropolyacids, which were less selective to 2,2,4-trimethyl-1,3-dioxolane. Especially, when  $H_7P_2Mo_{17}VO_{62}$  was used, the selectivity was as high as 93.3% with a nearly complete conversion of 1,2-epoxypropane. It is worthy to be noted that the TOF of  $H_{6+n}P_2Mo_{18-n}V_nO_{62}$  ( $n = 0–2, 4$ ) in this reaction was all greater than  $1.0 \times 10^4 h^{-1}$ . The ring-opening oligomerization products of 1,2-epoxypropane were also detected by GC/MS analyses. Interestingly, 2-ethyl-4-methyl-1,3-dioxolane (less than 1% in entry 2), which is derived from the cycloaddition reaction between acetaldehyde and 1,2-epoxypropane, was also detected although its amount was negligible. It demonstrates the isomerization of 1,2-epoxypropane to acetaldehyde was also possible under acidic conditions.

Table 1  
Reaction of 1,2-epoxypropane and acetone over various catalysts<sup>a</sup>

| Entry           | Catalysts  | Conversion (%) <sup>b</sup> | Selectivity (%) <sup>c</sup> | Yield (%) <sup>d</sup> | TOF/h <sup>e</sup> (10 <sup>4</sup> ) |
|-----------------|--|-----------------------------|------------------------------|------------------------|---------------------------------------|
| 1               | H <sub>6</sub> P <sub>2</sub> Mo <sub>18</sub> O <sub>62</sub>                 | 99.3                        | 92.1                         | 91.5                   | 1.1                                   |
| 2               | H <sub>7</sub> P <sub>2</sub> Mo <sub>17</sub> VO <sub>62</sub>                | 99.9                        | 93.3                         | 93.2                   | 1.1                                   |
| 3               | H <sub>8</sub> P <sub>2</sub> Mo <sub>16</sub> V <sub>2</sub> O <sub>62</sub>  | 99.2                        | 91.4                         | 90.7                   | 1.1                                   |
| 4               | H <sub>10</sub> P <sub>2</sub> Mo <sub>14</sub> V <sub>4</sub> O <sub>62</sub> | 99.4                        | 90.9                         | 90.4                   | 1.1                                   |
| 5               | H <sub>6</sub> P <sub>2</sub> W <sub>18</sub> O <sub>62</sub>                  | 99.0                        | 74.5                         | 73.8                   | 0.9                                   |
| 6               | H <sub>7</sub> P <sub>2</sub> W <sub>17</sub> VO <sub>62</sub>                 | 99.3                        | 85.7                         | 85.1                   | 1.0                                   |
| 7               | H <sub>3</sub> PMo <sub>12</sub> O <sub>40</sub>                               | 98.8                        | 19.8                         | 19.6                   | 0.2                                   |
| 8               | H <sub>4</sub> PMo <sub>11</sub> VO <sub>40</sub>                              | 97.3                        | 25.9                         | 25.2                   | 0.3                                   |
| 9               | H <sub>3</sub> PW <sub>12</sub> O <sub>40</sub>                                | 95.7                        | 21.7                         | 20.8                   | 0.2                                   |
| 10              | No catalyst  | 0.0                         | 0.0                          | 0.0                    | 0.0                                   |
| 11 <sup>f</sup> | H <sub>7</sub> P <sub>2</sub> Mo <sub>17</sub> VO <sub>62</sub>                | 99.0                        | 90.4                         | 89.5                   | 1.0                                   |

<sup>a</sup> 50 mmol 1,2-epoxypropane; 100 mmol acetone; room temperature; 0.05 mmol catalyst.

<sup>b</sup> Conversion of 1,2-epoxypropane.

<sup>c</sup> Selectivity to 2,2,4-trimethyl-1,3-dioxolane.

<sup>d</sup> GC yield.



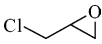

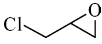



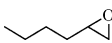
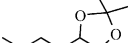






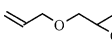
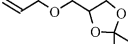
<sup>e</sup> TOF = mol product/mol catalyst h.

<sup>f</sup> Reused for the fourth time.

Environmental and economical consideration prompts an urgent need to redesign commercially important processes. In this context, heterogeneous catalysts play a dramatic role [23]. The use of heterogeneous catalysts in different areas of the organic synthesis has now reached significant levels, not only for the possibility to perform environmentally benign synthesis, but also for the good yields, accompanied by excellent selectivity that can be achieved. In fact, the advantages of these catalysts are: (i) the easy

workup (separation of the catalyst simply by filtration); (ii) the high level of purity of the products; (iii) and the possibility of recycling catalysts, as well as avoiding the use of noxious substances [17]. In this case, we have found that H<sub>7</sub>P<sub>2</sub>Mo<sub>17</sub>VO<sub>62</sub> is immiscible with reaction mixture and could be reused four times without visible loss of the activity in the reaction between 1,2-epoxypropane and acetone (Table 1, Entry 11), simply by filtering the catalyst, washing with acetone, drying and immediately reusing. Therefore,

Table 2  
Reaction of epoxides and acetone over H<sub>7</sub>P<sub>2</sub>Mo<sub>17</sub>VO<sub>62</sub><sup>a</sup>

| Entry          | Epoxides  | Conversion (%) <sup>b</sup> | Products  | Selectivity (%) <sup>c</sup> | Yield (%) <sup>d</sup> | TOF/h <sup>e</sup> (10 <sup>4</sup> ) |
|----------------|---|-----------------------------|---|------------------------------|------------------------|---------------------------------------|
| 1              |  | 99.9                        |  | 93.3                         | 93.2                   | 1.1184                                |
| 2              |  | 99.6                        |  | 96.1                         | 95.7                   | 1.1                                   |
| 3 <sup>f</sup> |  | –                           |  | –                            | 75                     | <0.0014                               |
| 4              |  | 99.2                        |  | 92.7                         | 92.0                   | 1.1                                   |
| 5              |  | 99.5                        |  | 90.4                         | 90.0                   | 1.0                                   |
| 6              |  | 99.7                        |  | 92.6                         | 92.3                   | 1.1                                   |
| 7              |  | 99.0                        |  | 91.3                         | 90.4                   | 1.1                                   |
| 8              |  | 99.8                        |  | 94.0                         | 93.8                   | 1.1                                   |
| 9              |  | 99.4                        |  | 95.6                         | 95.0                   | 1.1                                   |

<sup>a</sup> 50 mmol epoxides; 0.05 mmol catalyst; 100 mmol acetone; 5 min; room temperature.

<sup>b</sup> Conversion of 1,2-epoxides.

<sup>c</sup> Selectivity to 1,3-dioxolanes.

<sup>d</sup> GC yield.

<sup>e</sup> TOF = mol product/mol catalyst h.

<sup>f</sup> Reported in literature [16] using K<sub>5</sub>CoW<sub>12</sub>O<sub>40</sub>·3H<sub>2</sub>O as catalyst, reaction conditions: reflux for 420 min, catalyst/epoxide: 1/100.

this catalyst can be used as a stable and recyclable catalyst.

### 3.2. Reactions of various epoxides with acetone catalyzed by $H_7P_2Mo_{17}VO_{62}$


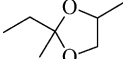
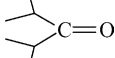
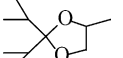
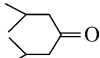
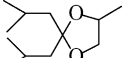

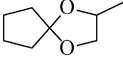

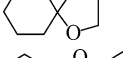

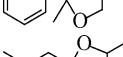
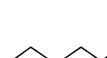
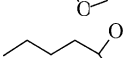
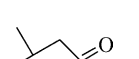
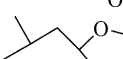
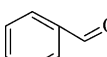
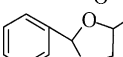
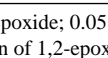
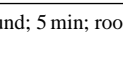
The results of cycloadditions of different epoxides with acetone over  $H_7P_2Mo_{17}VO_{62}$  are shown in Table 2. This catalyst proved to be very active toward all examined epoxides (Entries 1, 2, 4–9), catalyst loads as low as 0.1 mol% leading to high epoxide conversion within 5 min at room temperature. For the purpose of comparison, a reported result was also listed in Table 2 (Entry 3). In our  $H_7P_2Mo_{17}VO_{62}$  catalytic system, epichlorohydrin could be completely converted to the corresponding desired product within 5 min whereas the same reaction has to be carried out at reflux conditions for 7 h over  $K_5CoW_{12}O_{40} \cdot 3H_2O$  with a slight inferior yield [16]. Although, at worst, the selectivity to 1,3-dioxolane was 90.4% (Entry 5), it presents a potential advantage over conventional catalytic systems. Using  $H_7P_2Mo_{17}VO_{62}$  as catalyst, we found that 2,2,4-trimethyl-1,3-dioxolane was formed with a TOF of  $1.1 \times 10^4$  which is much greater than that of conventional catalytic systems. It clearly indicates the high efficiency of this catalyst. Therefore, we have introduced Wells–Dawson type heteropolyacids ( $H_{6+n}P_2Mo_{18-n}V_nO_{62}$ )

( $n=0-2, 4$ ), especially  $H_7P_2Mo_{17}VO_{62}$ , as more suitable, nonhygroscopic, nontoxic, available, stable, reusable and heterogeneous catalysts for the title reactions. In addition, the advantages, such as mild reaction conditions, ease of preparation, simple filtration at the end of reaction and high yields, give a new synthetic application of  $H_7P_2Mo_{17}VO_{62}$  in organic synthesis.

### 3.3. Reactions of different carbonyl compounds with 1,2-epoxypropane over $H_7P_2Mo_{17}VO_{62}$

Table 3 shows the results of  $H_7P_2Mo_{17}VO_{62}$  catalyzed reactions between 1,2-epoxypropane and ketones or aldehydes. All the ketones with the exception of diisopropyl ketone, which gave the corresponding 1,3-dioxolane with 85.2% epoxide conversion, reacted with 1,2-epoxypropane in almost quantitative epoxide conversion and good selectivity to the 1,3-dioxolanes at room temperature within 5 min. The poor reactivity of diisopropyl ketone may be caused by its steric hindrance. When using aliphatic aldehydes as substrate, the epoxide conversions are all satisfactory. However, the selectivity of 1,3-dioxolanes slightly declined with formations of a few unidentified by-products. When benzaldehyde was used as substrate, the corresponding 1,3-dioxolane was formed in 98.9% epoxide conversion and 96.3% selectivity.

Table 3  
Reaction of 1,2-epoxypropane with different carbonyls over  $H_7P_2Mo_{17}VO_{62}$ <sup>a</sup>

| Entry | Carbonyl compound   | Conversion (%) <sup>b</sup> | Products  | Selectivity (%) <sup>c</sup> | Yield (%) <sup>d</sup> | TOF/h <sup>e</sup> ( $10^4$ ) |
|-------|---|-----------------------------|---|------------------------------|------------------------|-------------------------------|
| 1     |  | 97.1                        |  | 97.6                         | 94.8                   | 1.1                           |
| 2     |  | 85.2                        |  | 90.7                         | 77.3                   | 0.9                           |
| 3     |  | 97.6                        |  | 92.1                         | 89.9                   | 1.1                           |
| 4     |  | 98.4                        |  | 94.2                         | 92.7                   | 1.1                           |
| 5     |  | 99.0                        |  | 94.4                         | 93.5                   | 1.1                           |
| 6     |  | 98.9                        |  | 93.3                         | 92.3                   | 1.1                           |
| 7     |  | 99.6                        |  | 81.2                         | 80.9                   | 1.0                           |
| 8     |  | 97.3                        |  | 95.0                         | 92.4                   | 1.1                           |
| 9     |  | 98.4                        |  | 90.4                         | 92.5                   | 1.1                           |
| 10    |  | 98.9                        |  | 96.3                         | 95.2                   | 1.1                           |

<sup>a</sup> 50 mmol epoxide; 0.05 mmol catalyst; 100 mmol carbonyl compound; 5 min; room temperature.

<sup>b</sup> Conversion of 1,2-epoxypropane.

<sup>c</sup> Selectivity to 1,3-dioxolane.

<sup>d</sup> GC yield.

<sup>e</sup> TOF = mol product/mol catalyst h.

In view of the foregoing,  $\text{H}_7\text{P}_2\text{Mo}_{17}\text{VO}_{62}$  has a good generality as catalyst for the reactions of epoxides with carbonyl compounds. It is worthy to be noted that TOF of this catalyst in Table 3 was all greater than  $0.9 \times 10^4$ , indicating the effectiveness of our catalytic system.

#### 4. Conclusion

In conclusion, Wells–Dawson type heteropoly acids ( $\text{H}_{6+n}\text{P}_2\text{Mo}_{18-n}\text{V}_n\text{O}_{62}$ ) ( $n=0-2, 4$ ) proved to be effective catalysts for the reactions between epoxides and aldehydes or ketones to afford 1,3-dioxolanes. Various epoxides could be efficiently converted to 1,3-dioxolanes over  $\text{H}_7\text{P}_2\text{Mo}_{17}\text{VO}_{62}$  catalyst within 5 min. Short reaction time, simple work up, mild reaction conditions and reliable reusability are the features obtained in the Wells–Dawson type heteropoly acids catalytic systems.

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