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Wells–Dawson type molybdovanadophosphoric heteropolyacids catalyzed Prins cyclization of alkenes with paraformaldehyde under mild conditions—a facile and efficient method to 1,3-dioxane derivatives

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Abstract

Wells–Dawson type molybdovanadophosphoric heteropolyacids ($H_{6+n}P_2Mo_{18-n}V_nO_{62}$) ($n = 1$–2, 4) have proved to be effective catalysts for Prins cyclization between alkenes and paraformaldehyde to produce corresponding 1,3-dioxane derivatives. The effects of solvents, catalysts, reaction-time, reaction-temperature and the ratio of olefin to catalyst were investigated systematically using styrene and paraformaldehyde as model substrates. Compared with conventional catalytic systems, the high-reaction rate, mild conditions, and improved selectivity are the features obtained in our systems. A reasonable mechanism was also proposed.

Keywords: Prins cyclization; Wells–Dawson type molybdovanadophosphoric heteropolyacids; Styrene; Paraformaldehyde; 1,3-Dioxane

1. Introduction

The acid-catalyzed condensation of olefins with aldehydes, known as the Prins reaction, is an important carbon–carbon bond forming reaction in organic synthesis [1]. The major products of classical Prins reaction are normally 1,3-diols, 1,3-dioxanes, unsaturated alcohols [2]. In addition, the Prins cyclization of an olefin with formaldehyde has been considered as one of the most straightforward method for the synthesis of 1,3-dioxanes, which are widely used in organic synthesis as solvents or intermediates [3]. Although Lewis acids as well as mineral acids can promote this transformation, most of them cause the corrosion of reaction equipment, use toxic reagents, and also require prolonged reaction-time and high-temperature reaction conditions resulting in low to moderate selectivity of products due to the polymerization of starting materials [4–6]. Recently, Yadav et al. reported $InBr_3$ catalyzed Prins cyclization of styrenes with paraformaldehyde in dialkylimidazolium-based ionic liquids [7].

Heteropolyacids (HPAs) have many advantages that make them economically and environmentally attractive in both academic and industrial significance; they are useful acids and oxidation catalysts in various reactions since its catalytic features can be varied at a molecular level [8–10]. Furthermore, Wells–Dawson type heteropolyacids possess super-acidity and a remarkable stability both in solution and in the solid state [11]. Many typical acid-catalyzed reactions, including tetrahydropyranylation of alcohols [12], acetolization, ketonilzation [13] and etherification [14] are all effective in the presence of a suitable Wells–Dawson type heteropolyacid catalyst. Prins cyclization of styrene with formaldehyde have been examined using Keggin-type HPAs as catalysts in aqueous solution, however, the conversion of styrene was only 29% at best with a difficulty of catalyst recycling due to the use of water as solvent (which has low volatility) [15]. To our best knowledge, there are no examples of the use of Wells–Dawson type heteropolyacids as

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catalysts for this type of Prins cyclization. Here, we wish to report an efficient catalytic Prins cyclization of olefins with paraformaldehyde to produce 1,3-dioxane derivatives using Wells–Dawson type molybdovanadophosphoric heteropolyacids as catalysts.

2. Experimental

2.1. Materials and reagents

All reagents used were highest purity commercial products (99%), and applied to the reaction without further purification. Some Keggin-type heteropolyacids, such as 12-tungstophoric 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 of commercially available purity and dried at 60 °C for 48 h prior to use. 1-Butyl-3-methylimidazolium hexefluorophosphate ionic liquid ([BMIm]PF_6) was prepared according to the procedures reported in the literature [16]. The purity of [BMIm]PF_6 was characterized with FTIR and NMR, and satisfactory results were obtained.

2.2. Preparation of catalysts

The Wells–Dawson type heteropolyacid $H_6P_2W_{18}O_{62}\cdot 24H_2O$ was prepared by the Drechsel method [17] from an aqueous solution of $\alpha/\beta K_6P_2W_{18}O_{62}$ salt. This Dawson-type salt was prepared according to [18]. $H_7P_2W_{16}V_2O_{62}$ was prepared from an aqueous solution of α_2-K_7 $P_2W_{16}V_2O_{62}$ salt according to the method described by Pope and co-workers [19]. $H_6P_2Mo_{18}O_{62}\cdot 39H_2O$ was prepared according to the technique reported by Wu [20]. $H_{6+n}P_2Mo_{18-n}V_nO_{62}$ ($n = 1$–2, 4) was synthesized according to [21]. The prepared heteropolyacids were characterized with FTIR and element analysis, and the results were all satisfactory.

2.3. Catalytic reactions

2.3.1. Reaction procedure

All Prins cyclization reactions were carried out in a magnetically stirred flask equipped with a small side tube for convenience of periodical sampling through a syringe. Each catalyst 0.5 mol% was added to a mixture of solvent, olefin and paraformaldehyde in the reactor. This reaction mixture is then stirred at desired temperature for the appropriate time.

2.3.2. Products analysis

Qualitative and quantitative analyses were conducted with a HP 6890/5973 GC/MS and a HP 1790 GC 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 chromatograph peak. The GC yield was obtained in 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. Effects of solvents on the Prins cyclization of styrene with paraformaldehyde

Effects of different solvents on the Prins cyclization of styrene with paraformaldehyde were investigated using the Wells–Dawson type heteropoly acid ($H_8P_2Mo_{16}V_2O_{62}$) as a catalyst, and the results are presented in Table 1. All the nonpolar organic solvents except toluene, which gave 78.8% conversion and 94.3% of selectivity to 4-phenyl-1,3-dioxane, were less suitable than polar chlorine-containing organic solvents (entries 1–7). In DMF, DMAc and CH_3CN, both the conversion and selectivity are rather poor (entries 8–10). The dipole moment of these solvents is also listed in Table 1. However, it is difficult to offer an explanation for the different activity only by the solvent dipole moment. It is well known that pK_a of the same HPAs depends on the solvent. This may be one of the reasons for the difference. In light of above discussion, 1,2-dichloroethane proved to be a suitable solvent for the Prins cyclization of styrene with paraformaldehyde using $H_8P_2Mo_{16}V_2O_{62}$ as a catalyst. (entry 4).

Table 1
Effects of solvents on the reaction of styrene with paraformaldehyde over $H_8P_2Mo_{16}V_2O_{62}$[a]

Entry	Solvents	Dipole moment (*D*)	Conversion[b] (%)	Selectivity[c] (%)
1	CH_2Cl_2	1.60	12.4	95.6
2	$CHCl_3$	1.15	11.5	58.0
3	CCl_4	0	8.7	70.5
4	$ClCH_2CH_2Cl$	1.86	99.9	98.7
5	Toluene	0.43	78.8	94.3
6	Cyclohexane	0	1.5	61.4
7	THF	1.73	1.3	44.2
8	DMF	3.82	10.2	57.1
9	DMAc	3.79	8.4	49.7
10	CH_3CN	3.45	2.7	61.3
11[d]	[BMIm]PF_6	NA[i]	5.1	90.0
12[e]	[BMIm]PF_6	NA	91[f]	–
13	H_2O	1.82	25.2	99.9
14[g]	–	1.82	4.8	68.5
15[h]	–	1.82	29.0	–

[a] Solvent: 5 ml; styrene: 2 mmol (0.23 ml); paraformaldehyde: 0.12 g; reaction-time: 25 min; catalyst: 0.03 mmol; reaction-temperature: 25 °C.

[b] Conversion of styrene.

[c] Selectivity to 4-phenyl-1,3-dioxane.

[d] Seacted for 6 h.

[e] Literature [7], using $InBr_3$ as catalyst.

[f] Separation yield.

[g] Formaldehyde aqueous solution (37 wt.%) as substrate.

[h] Literature [15], using $H_4PW_{12}O_{40}$ as catalyst.

[i] Not available.

Recently, the environmentally benign solvents, ionic liquids, have been used as reaction media for the same reaction combined with a green Lewis acid, $InBr_3$ (the selected result is presented in Table 1, entry 12). Due to the inherent non-volatility of ionic liquids as well as its potential convenience for product separation, we have also examined the feasibility of ionic liquid $[BMIm]PF_6$ as a solvent using $H_8P_2Mo_{16}V_2O_{62}$ as a catalyst. As observed in Table 1 (entry 11), selectivity to 4-phenyl-1,3-dioxane proved to be quite high reaching nearly 90%, but conversion was only 5% even after 6 h. When H_2O was used solvent, the conversion of styrene was 25.1% with an exclusive selectivity to 4-phenyl-1,3-dioxane (entry 13). This may be related with poor kinetic transportation beside the solvent effect, because the substrates were floated in the top of the aqueous phase wherein the most of catalyst was present. From the industrial point of view, aqueous solution of formaldehyde is a very attractive substrate, and its feasibility was investigated by Izumi and his co-workers using $H_4PW_{12}O_{40}$ as a catalyst [13] (a selected result is listed in Table 1, entry 15). However, over $H_8P_2Mo_{16}V_2O_{62}$, it seems to be inadvisable when paraformaldehyde was replaced with aqueous solution of formaldehyde because the conversion of styrene only reached 4.8% (entry 14).

3.2. Effect of catalysts on the Prins cyclization of styrene with paraformaldehyde

Subsequently, various heteropolyacids were evaluated in the Prins cyclization of styrene with paraformaldehyde at 25 °C in 1,2-dichloroethane, and the results are listed in Table 2. In this transformation, Wells–Dawson type molybdophosphoric and molybdovanadophoshoric heteropolyacids ($H_{6+n}P_2Mo_{18-n}V_nO_{62}$) ($n = 1$–2, 4) showed higher catalytic activity than Wells–Dawson type tungstophoric and tungstovanadophoshoric heteropolyacids and Keggin-type heteropolyacids (entries 1–4). Especially when $H_8P_2Mo_{16}V_2O_{62}$ was used as a catalyst, the conversion of styrene reached 99.9% with almost exclusive selectivity to 4-phenyl-1,3-dioxane within 25 min. Although it is difficult to offer an explanation for the activity difference between Wells–Dawson and Keggin-type heteropolyacids, it is certain that there is a complex relationship between the activity and structure of polyanions. It is well known that Dawson-type heteropolyacids are amorphous in homogeneous solution due to the ellipsidal shape of the polyanion, whereas Keggin-type polyanions, having spherical shape, are crystalline (bcc structure). As a result, the former is easy to adsorb polar molecules, such as formaldehyde, and forms active pseudoliquid that allows the reactions to take place at the surface and solid bulk of the heteropoly-anion, but the latter are much less active [22]. Further, by changing the constituent elements of polyanion (both hetero and addenda atoms), the acid strength of HPAs as well as its catalytic activity is able to vary in a wide range [22]. In the present reaction, $H_8P_2Mo_{16}V_2O_{62}$ represents a higher activity than others examined in Table 2. It further illustrates the crucial effect of constituent element on the activity of heteropolyacids. This point deserves further investigation.

In the absence of catalyst, the reaction did not yield any product even after a prolonged reaction-time (entry 10). It is worth noting that the result obtained over $H_8P_2Mo_{16}V_2O_{62}$ is much better than those hitherto reported for Prins cyclization [3–7]. Beside the desired 4-phenyl-1,3-dioxane, 3-phenylpropionaldehyde and an unidentified product were also detected as by-products in this reaction. The molecular weight of the unidentified by-product was 238 by GC–MS analysis, and thus, it could be derived from the reaction of one formaldehyde molecule (MW = 30) with two styrene molecules (MW = 104).

Table 2
Reaction of styrene with paraformaldehyde over different heteropoly acids in 1,2-dichloroethane under room temperature[a]

Entry	Catalysts	Conversion[b] (%)	Selectivity[c] (%)
1	$H_6P_2Mo_{18}O_{62}$	95.8	97.1
2	$H_7P_2Mo_{17}VO_{62}$	87.5	97.9
3	$H_8P_2Mo_{16}V_2O_{62}$	99.9	98.7
4	$H_{10}P_2Mo_{14}V_4O_{62}$	99.1	93.4
5	$H_6P_2W_{18}O_{62}$	11.6	89.0
6	$H_7P_2W_{16}V_2O_{62}$	6.9	82.4
7	$H_3PMo_{12}O_{40}$	63.4	96.3
8	$H_3PW_{12}O_{40}$	65.7	97.6
9	$H_4SiW_{12}O_{40}$	8.8	72.6
10[d]	–	0.0	0.0

[a] 1,2-Dichloroethane: 5 ml; styrene: 2 mmol (0.23 ml); paraformaldehyde: 0.12 g; reaction-time: 25 min; catalyst: 0.03 mmol; reaction-temperature: 25 °C.

[b] Conversion of styrene.

[c] Selectivity to 4-phenyl-1,3-dioxane.

[d] The absence of catalyst.

3.3. Effect of catalyst amount on the Prins cyclization of styrene with paraformaldehyde over $H_8P_2Mo_{16}V_2O_{62}$ catalyst

Fig. 1 shows the effect of catalyst amount on the Prins cyclization of styrene with paraformaldehyde using $H_8P_2Mo_{16}V_2O_{62}$ as a catalyst in 1,2-dichloroethane. A significant increase of the styrene conversion was observed when the catalyst amount was increased from 0.004 to 0.01 mmol, and to finish this reaction 0.01 mmol of the catalyst was enough. When the catalyst over 0.03 mmol was used, the conversion of styrene levelled off. For selectivity to 4-phenyl-1,3-dioxane, the increase in catalyst amount was profitable for its improvement until 0.03 mmol. Further increase could result in a slight decrease in the selectivity. The reason is currently under investigation, but it is most likely related to the stability of 4-phenyl-1,3-dioxane at a high acid concentration. Ultimately, for the sake of completion of the reaction and improvement of its selectivity, the suitable catalyst amount should be 0.03 mmol.

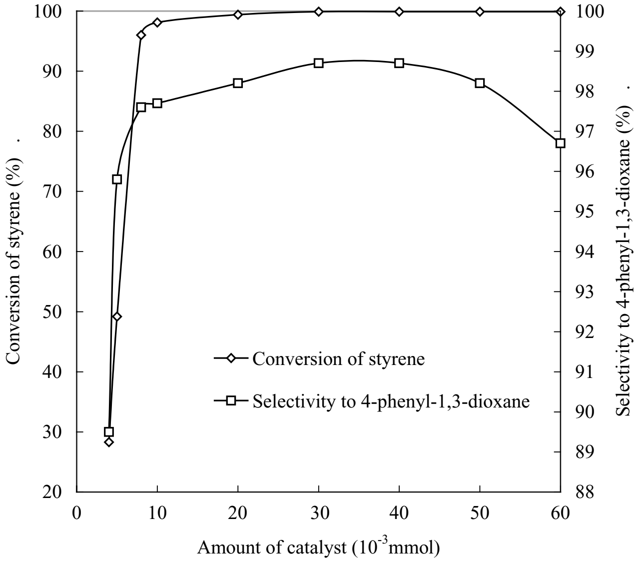


Fig. 1. Effect of catalyst amount on the Prins cyclization of styrene with paraformaldehyde. 1,2-Dichloroethane: 5 ml; styrene: 2 mmol (0.23 ml); paraformaldehyde: 0.12 g; reaction-time: 25 min; reaction-temperature: 25 °C.

3.4. *Effect of reaction-time on the Prins cyclization of styrene with paraformaldehyde*

Fig. 2 illustrates the effect of reaction-time on the title reaction over $H_8P_2Mo_{16}V_2O_{62}$. It can be seen that the reaction was very expeditious at the beginning and a 92.9% conversion was obtained in the first 15 min. When reaction-time was extended, the conversion of styrene was further increased and nearly reached 100% after 30 min. To our surprise, the selectivity to 4-phenyl-1,3-dioxane was rather poor at the beginning of reaction, and it markedly increased with the increase of reaction-time within the first 15 min as illustrated in Fig. 2. GC–MS analysis of reaction products revealed that the generation of 3-phenylpropionaldehyde disturbed the selectivity of this reaction. To make it clear, a profile of 3-phenylpropionaldehyde versus reaction-time was also given in Fig. 2. At the beginning of reaction, the amount of 3-phenylpropionaldehyde was about 13% (reacted for 5 min), then it gradually decreased, and finally, 3-phenylpropionaldehyde almost disappeared (reacted for 30 min). It is well known that 3-phenylpropionaldehyde is the intermediate for the Prins cyclization of styrene and formaldehyde to 4-phenyl-1,3-dioxane in conventional systems [7]. On the other hand, the mechanism of homogeneous catalysis by heteropolyacids is in principle similar to the mechanism of catalysis by solution of inorganic acids [23]. Therefore, we can conjecture that 3-phenylpropionaldehyde could also be the intermediate in the $H_8P_2Mo_{16}V_2O_{62}$ catalyzed Prins cyclization of styrene and paraformaldehyde, and the possible mechanism of this reaction is schematically shown in Fig. 3. From this hypothesis, the poor selectivity observed in the beginning of the reaction could be reasonable because the formation of 3-phenylpropionaldehyde may be predominant. As the reaction proceeded, 3-phenylpropionaldehyde was gradually consumed and as a result, the selectivity to desired product was improved. However, another one of the by-products (MW = 238, the same as described in Section 3.2) was gradually increased after 15 min, and thus, the selectivity to 4-phenyl-1,3-dioxane was slightly disturbed, although its magnitude is unconspicuous. From the above discussion, a suitable reaction-time could be 25 min for the $H_8P_2Mo_{16}V_2O_{62}$ catalyzed Prins cyclization between styrene and paraformaldehyde.

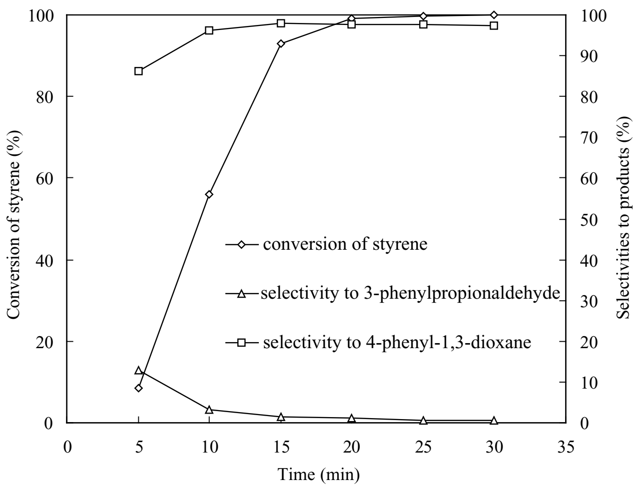


Fig. 2. Effect of reaction-time on the reaction of styrene with paraformaldehyde over $H_8P_2Mo_{16}V_2O_{62}$. 1,2-Dichloroethane: 5 ml; styrene: 2 mmol (0.23 ml); paraformaldehyde: 0.12 g; catalyst: 0.03 mmol; reaction-temperature: 25 °C.

3.5. *Influence of the temperature*

Fig. 4 illustrates the effect of reaction-temperature on the reaction of styrene with paraformaldehyde. It can be seen that the conversion of styrene was increased with elevation of reaction-temperature. The selectivity to 4-phenyl-1,3-dioxane was the highest at 25 °C and decreased as the reaction-temperature increased or decreased from 25 °C. It should be pointed out that the ability to run this Prins cyclization at ambient temperature represents a substantial

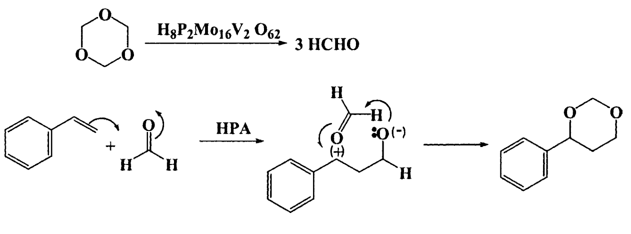


Fig. 3. Mechanism of the Prins cyclization of styrene with paraformaldehyde over $H_8P_2Mo_{16}V_2O_{62}$. 1,2-Dichloroethane: 5 ml; styrene: 2 mmol (0.23 ml); paraformaldehyde: 0.12 g; catalyst: 0.03 mmol; reaction-time: 25 min.

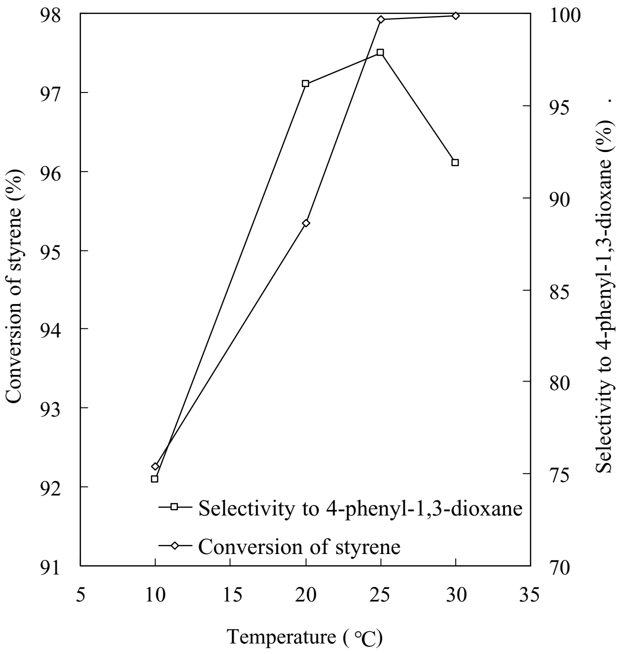


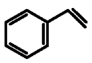
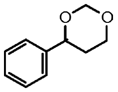
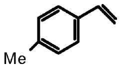
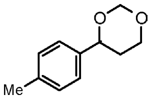
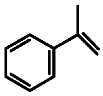
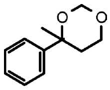
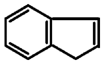
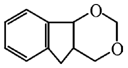
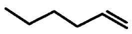
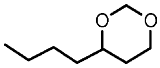
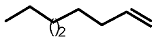
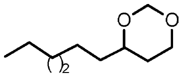
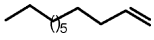
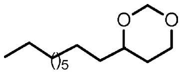
Fig. 4. Effect of reaction-time on the reaction of styrene with paraformaldehyde over $H_8P_2Mo_{16}V_2O_{62}$. 1,2-Dichloroethane: 5 ml; styrene: 2 mmol (0.23 ml); paraformaldehyde: 0.12 g; catalyst: 0.03 mmol; reaction-time: 25 min.

improvement over the previous methods [3,6,15], wherein a temperature >40°C (in sealed tubes) were required to effect the reaction.

3.6. *Reactions of different olefins with paraformaldehyde catalyzed by $H_8P_2Mo_{16}V_2O_{62}$ in 1,2-dichloroethane*

Having a set of optimized conditions for $H_8P_2Mo_{16}V_2O_{62}$ catalyzed Prins cyclization with styrene and paraformaldehyde in hand, we next explored the usefulness of this catalytic system and various olefins were used as electron donors to react with paraformaldehyde. As shown in Table 3, various styrene derivatives and aliphatic linear olefins all reacted well. In the reaction of styrene, yield reported are based on GC analysis using chlorobenzene as an internal standard; but yields by weight were also evaluated and were quantitatively the same as the GC data. It indicated that our GC analysis is reliable and that the GC yield was comparable with those in the literature. In contrast to previously reported catalytic systems [7], enhanced reaction rates and high-selectivity are the features obtained in our system. For example, the treatment of 4-methylstyrene and paraformaldehyde with $H_8P_2Mo_{16}V_2O_{62}$ in 1,2-dichloroethane for 25 min affords the corresponding 1,3-dioxane in 91.5% of 4-methylstyrene conversion and 96.8% of selectivity, whereas the same

Table 3
Efficiency of $H_8P_2Mo_{16}V_2O_{62}$ catalyzed synthesis of 1,3-dioxane derivatives in 1,2-dichloroethane[a]

Entry	Alkene	Product[b]	Reaction-time (min)	Conversion of alkene (%)	Selectivity to 1,3-dioxane derivatives
1			25	99.9	98.7
2	 Me	 Me	25	91.5	96.8
3			25	93.0	31.2
4			60	95.7	95.5
5			150	99.7	84.6[c]
6			150	99.6	45.0[c]
7			150	75.8	27.3[c]

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[a] 1,2-Dichloroethane: 5 ml; olefins: 2 mmol; paraformaldehyde: 0.12 g; catalyst: 0.03 mmol; reaction-time: 25 min.

[b] All products were characterized by mass spectroscopy.

[c] Reactions were carried out at 80 °C.

reaction has to take 210 min to achieve a satisfactory yield in $InBr_3$/[BMIm]PF_6. α-Methylstyrene, which tends to oligomerize under acidic conditions [24], was also successfully used as an electron donor in this type reaction (entry 3). In terms of aliphatic linear olefins, which are considered to be unresponsive in this type reaction, the analogous Prins cyclizations also proceeded well at elevated temperature and prolonged time using $H_8P_2Mo_{16}V_2O_{62}$ as a catalyst (entries 5–7). It indicated that the scope of the substrates over this catalytic system is generally wide.

4. Conclusion

Wells–Dawson type heteropolyacid $H_8P_2Mo_{16}V_2O_{62}$ was used for the first time as a catalyst in the Prins cyclization of various olefins and paraformaldehyde. Effects of various parameters, including solvents, reaction-time, reaction-temperature and the molar ratio of substrate to catalyst, were investigated systematically using styrene as a substrate. The suitable reaction conditions are 1,2-dichloroethane as solvent, 30 min, 25 °C and 200:3 molar ratio of olefin to $H_8P_2Mo_{16}V_2O_{62}$. Under these optimized conditions, styrene could be completely converted with an exclusive selectivity to 4-phenyl-1,3-dioxane. Various aliphatic linear olefins, which are considered to be inactive, also reacted well, indicating the wide scope of the substrates over this catalytic system. Moreover, the experimental procedure is quite simple and convenient, and the reaction conditions are amenable to scale-up.

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