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Chemistry

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# Aniline-terephthalaldehyde resin *p*-toluenesulfonate (ATRT) as a highly efficient and reusable catalyst for alcoholysis, hydrolysis, and acetolysis of epoxides

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#### ABSTRACT

Alcoholysis, hydrolysis, and acetolysis of epoxides were carried out in the presence of a catalytic amount of aniline-terephthalaldehyde resin *p*-toluenesulfonate (ATRT) to give the corresponding  $\beta$ -substituted alcohols in good yields. Alcoholysis and hydrolysis of epoxides catalyzed by ATRT proceeded faster than those by pyridinium *p*-toluenesulfonate (PPTS).

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Acetolysis; alcoholysis; aniline resins; epoxides; hydrolysis; solid acids

#### GRAPHICAL ABSTRACT



# Introduction

Ring-opening reactions of epoxides with numerous nucleophiles are of great importance in synthetic organic chemistry because of the usefulness in the preparation of various synthetic intermediates. Treatments of epoxides with alcohols,<sup>[1-8]</sup> water,<sup>[9-15]</sup> and acetic acid<sup>[9-15]</sup> afford  $\beta$ -alkoxy alcohols, diols, and  $\beta$ -acetoxy alcohols, respectively. These transformations are very frequently carried out by using Lewis acids,<sup>[16-18]</sup> Brønsted acids,<sup>[19-21]</sup> and basic reagents. However, the use of some acidic catalysts often encounters serious problems such as polymerization of substrates, low activity, and inconvenient handling of catalysts. Therefore, the development of highly active and mildly acidic catalysts for ring

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<sup>()</sup> Supplemental data (Full experimental detail, IR spectra, and <sup>1</sup>H and <sup>13</sup>C NMR spectra) can be accessed on the publisher's website.

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Figure 1. Plausible structure of ATRT.

opening of epoxides is still required. Among them, mildly acidic solid acids provide the convenient workup procedure and the reuse of the catalyts.<sup>[22-24]</sup> As for commercially available polymers, a few examples such as poly(4-vinylpyridinium *p*-toluenesulfonate) [polyPPTS]<sup>[25]</sup> and poly(4-vinylpyridinium chloride) [polyPCL]<sup>[26]</sup> are known. However, these polymers are expensive.

We have recently reported that aniline-terephthalaldehyde resin *p*-toluenesulfonate  $(ATRT)^{[27]}$  (Fig. 1) and more active diphenylamine-terephthalaldehyde resin *p*-toluenesulfonate  $[DTRT (H)]^{[28]}$  with high sulfonate group contents are useful acidic polymer catalysts for tetrahydropyranylation of alcohols, deprotection of tetrahydropyranyl (THP) ethers and *tert*-butyldimethylsilyl (TBDMS) ethers, and acetalization of carbonyl compounds.<sup>[8–10]</sup> ATRT possessed higher activities than pyridinium *p*-toluenesulfonate (PPTS) for the deprotection of THP ethers and TBDMS ethers and the dimethylacetalization of carbonyl compounds.<sup>[27,29]</sup> In this article, we report that ATRT can act as a highly efficient catalyst for alcoholysis, hydrolysis, and acetolysis of epoxides under mild conditions. Furthermore, the activities of ATRT for ring opening reactions of epoxides are compared with those of PPTS.

#### **Results and discussion**

First, we examined the catalytic activity of various mildly acidic polymers for ethanolysis of styrene oxide (1). The results are shown in Table 1. We also prepared ATRT (H) with high sulfonate group content by the treatment of ATRT with *p*-toluenesulfonic acid (TsOH) at 90 °C for 8 h in water, but the increase in the yield of 2-ethoxy-2-phenylethanol (10) was not observed (entries 1 and 2). When DTRT (H) was employed, the yield of 10 was quite low (2%) (entry 3). Elemental analysis indicated that nominal composition of the recovered DTRT (H) was  $C_{19.32}H_{16.58}N_{1.00}O_{2.87}S_{0.28}$  [DTRT (H):  $C_{17.38}H_{15.43}N_{1.00}O_{2.02}S_{0.34}$ ], suggesting the decomposition of the catalyst. The proportion of the *p*-toluenesulfonate salt per aniline unit decreased from 34% to 28%. Furthermore, the increase in the proportion of carbon, hydrogen, and oxygen atoms was observed. However, the structure of decomposed DTRT (H) is not clear at the present time. Activity of polyPPTS was comparable to that of ATRT (entries 1 and 4).

	Ph	Catalyst EtOH Ph	
	1	10	
Entry	Catalyst	Acid density (mmol $g^{-1}$ )	Yield (%) <sup>b</sup>
1	ATRT	1.01	60
2	ATRT (H)	1.55	55
3	DTRT (H)	1.17	2
4	PolyPPTS	3.5	56
5	PolyPCL	6.5	17

Table 1. Ethanolysis of 1 catalyzed by mildly acidic polymers.<sup>a</sup>

 $^a\mathrm{Epoxide}$  1 (5.0 mmol), catalyst (1 mol%), EtOH (10 mL), room temp, 45 min.  $^b\mathrm{Isolated}$  yield.

As shown in Table 2, various epoxides 1-8 reacted with several alcohols catalyzed by ATRT to give the corresponding  $\beta$ -alkoxy alcohols 9-30 in good yields. Only 1 mol% of the catalyst was sufficient to complete the reactions except the reaction of cyclohexene oxide (2) with allyl alcohol (entry 9). Alcoholysis of 1 with MeOH proceeded very quickly at room temperature within 7 min to give 9 in 99% yield with the exclusive attack at the benzylic position. The reactions of 1 with the other alcohols such as EtOH, *n*PrOH, *i*PrOH,

**Table 2.** Alcoholysis of epoxides catalyzed by ATRT.<sup>a</sup>

	R	$R^2 \xrightarrow{0} R^4 \xrightarrow{\text{cat.}} R^4 \xrightarrow{1} R^4$	roh R <sup>2</sup>	$R^{1}$ $R^{3}$	
		1-8		9-30	
Entry	Epoxide	ROH	Time (h)	Product (Yield <sup>b</sup> )	
1 2 3 4 <sup>c</sup> 5 6 7 8 9 <sup>c,d</sup>	Ph 1	MeOH EtOH nPrOH iPrOH H <sub>2</sub> C=CHCH <sub>2</sub> OH HC=CCH <sub>2</sub> OH MeOH EtOH H <sub>2</sub> C=CHCH <sub>2</sub> OH	7 min 1.5 h 15 h 3 h 1 h 10 min 96 h 137 h	Ph OR OH	9 (99%) 10 (97%) 11 (92%) 12 (80%) 13 (96%) 14 (99%) 15 (86%) 16 (80%) 17 (80%)
11		MeOH	4 h		19 (51%) 20 (41%)
12	iPr-0_0	MeOH	12 h		21 (69%)
				iPr O OH	22 (16%)

(Continued)

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Table 2.	Continued.				
Entry	Epoxide	ROH	Time (h)	Product (Yield <sup>b</sup> )	
13	Ph-0-50	MeOH	32 h	OH Ph <sup>O</sup> OMe	23 (95%)
14		MeOH	24 h		24 (69%)
	P			оме он	25 (16%)
15	Ph COOEt	MeOH	48 h	Ph COOEt OH	26 (90%)
16 17 18 19	Ph 8	$\begin{array}{c} MeOH\\ EtOH\\ H_2C{=}CHCH_2OH\\ H_2C{\equiv}CCH_2OH \end{array}$	1.5 h 15 h 15 h 2 h	Ph OR OH	27 (98%) 28 (99%) 29 (96%) 30 (90%)

<sup>a</sup>Epoxide (5.0 mmol), ATRT (50 mg, 1 mol%), alcohol (10 mL), room temp.

<sup>b</sup>lsolated yield.

<sup>c</sup>The reaction was conducted at 40 °C.

<sup>d</sup>2 mol% of ATRT was used.

allyl alcohol, and 2-propyn-1-ol proceeded more slowly (entries 2-6). Treatment of 2 with MeOH proceeded very quickly at room temperature within 10 min to afford 15 in 86% yield. Alcoholysis of 2 with EtOH, allyl alcohol, and 2-propyn-1-ol proceeded much more slowly compared with those of 1 (entries 2, 5, 6, and 8-10). Only trans isomers were obtained in the reactions of 2. The reactions of 4-7, which contain deactivating groups such as an ethereal linkage and an ester function, proceeded more slowly than that of 3

Entry	Epoxides	Time	Products (yield <sup>b</sup> )
1	1 + 3	7 min	9 (98%)
			<b>19</b> (11%) <b>20</b> (12%)
2	1 + 5	7 min	<b>9</b> (99%)
			23 (0%)
3	1 + 6	7 min	<b>9</b> (99%)
			24 (5%) 25 (1%)
4	1 + 7	7 min	<b>9</b> (99%)
			26 (0%)
5	2 + 3	10 min	15 (84%)
			<b>19</b> (4%) <b>20</b> (4%)
6	2 + 5	10 min	15 (86%)
			23 (0%)
7	2 + 6	10 min	15 (81%)
			24 (3%) 25 (trace)
8	2 + 7	10 min	15 (86%)
			26 (0%)
9 <sup>c</sup>	1 + 2	1 h	14 (91%)
			18 (9%)

Table 3. Competitive alcoholysis of epoxides catalyzed by ATRT.<sup>a</sup>

<sup>a</sup>Epoxides (5.0 mmol), ATRT (50 mg, 1 mol%), MeOH (10 mL), room temp.

<sup>b</sup>Isolated yield.

<sup>c</sup>2-Propyn-1-ol was used instead of MeOH.

	$R^2 \rightarrow R^4 \leftarrow R^4$	$\frac{\text{at. ATRT}}{\text{H}_{2}\text{O}}$ $R^{2} \xrightarrow{\text{O}}_{\text{P1}} R^{4}$	rat. ATRT AcO OH AcOH $R^2 \rightarrow R^4$
	31-37	- K K 1-7	R' R° 38-44
Entry	Epoxide	Time (h)	Product (yield <sup>c</sup> )
1 <sup><i>a</i></sup>	Ph 1	1	OH Ph OH 31 (86%)
2 <sup><i>a</i></sup>		1	OH 
3 <sup><i>a,d,e</i></sup>	₩ <sub>7</sub> 3	24	ОН () 7 ОН 33 (89%)
4 <sup><i>a</i></sup>	iPr 0 0	36	0H 0H 0H 34 (82%)
5 <sup>a</sup>	Ph-0_0	97	Ph <sup>-0</sup> OH 35 (80%)
6 <sup><i>a</i></sup>		48	он оон <b>36</b> (91%)
7 <sup>a,d,e,f</sup>	Phr COOEt	87	OH Ph OH OH 37 (86%)
8 <sup>b</sup>	Ph 1	0.5	OAc Ph OH 38 (87%)
9 <sup>6</sup>		1	OAc 
10 <sup>6</sup>	₩ <sub>7</sub> 3	60	$ \overset{OH}{}_{7} \overset{OAc}{40} \overset{+}{}_{40} \overset{OAc}{}_{7} \overset{+}{}_{7} \overset{OH}{}_{41} \overset{(43\%)}{} $
11 <sup>b,d</sup>	iPr <sup>0</sup> 4	92	OH iPr OAc 42 (80%)
12 <sup>b,d</sup>	Ph-0-05	163	OH OAc 43 (80%)
13 <sup>b</sup>	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	48	OH OAc 44 (80%)

**Table 4.** Hydrolysis<sup>*a*</sup> and acetolysis<sup>*b*</sup> of epoxides catalyzed by ATRT.

<sup>a</sup>Epoxide (2.0 mmol), ATRT (100 mg, 5 mol%), MeCN-H<sub>2</sub>O (1:1) (4 mL), room temp. <sup>b</sup>Epoxide (2.0 mmol), ATRT (100 mg, 5 mol%), acetic acid (4 mL), room temp. <sup>c</sup>Isolated yield. <sup>d</sup>10 mol% of ATRT was used. <sup>e</sup>MeCN-H<sub>2</sub>O (2:1) was used.

<sup>f</sup>The reaction was conducted at 40 °C.

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			Yields (%) <sup>a</sup>		
Entry	1st	2nd	3rd	4th	5th
1 <sup><i>b</i></sup>	92	90	92	89	88
2 <sup>c</sup>	87	87	84	82	83
3 <sup>d</sup>	85	88	90	87	87

#### Table 5. Recycle experiments for the reactions of epoxide 1.

<sup>*a*</sup>lsolated yields.

<sup>b</sup>Epoxide 1 (5.0 mmol), ATRT (50 mg, 1 mol%), EtOH (10 mL), room temp, 1.5 h.

<sup>c</sup>Epoxide 1 (2.0 mmol), ATRT (100 mg, 5 mol%), MeCN-H<sub>2</sub>O (1:1) (4 mL), room temp, 1 h.

<sup>d</sup>Epoxide 1 (2.0 mmol), ATRT (100 mg, 5 mol%), acetic acid (4 mL), room temp, 0.5 h.

possessing an alkyl chain. Ring-opening reactions of 3, 4, and 6 provided regioisomers. The reactions of 4-6 possessing ethereal linkage with MeOH underwent with the preferential attack of MeOH at the terminal positions. Various substituents such as an isopropoxy group, a phenoxy group, a double bond, and an ester function were tolerated during the reactions (entries 12-15).

Table 3 summarizes the results for the competitive alcoholysis of epoxides. Selective ring opening of epoxide 1 in the presence of epoxide 3 possessing an alkyl chain, epoxide 5 or 6

Table 6.	Reactions	of	epoxides	catalyze	d b	y ATRT	and	PPTS.
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(a) Alcoholysis of epoxides. <sup>a</sup>					
		ATRT	PPTS		
Entry	Epoxide	Time product (yield)	Time product (yield)		
1 <sup>b</sup>	1	1.5 h <b>10</b> (97%)	4 h <b>10</b> (95%)		
2 <sup>c</sup>	1	1.5 h <b>14</b> (99%)	4.5 h 14 (97%)		
3 <sup>d</sup>	6	21 h <b>24</b> (68%)	157 h <b>24</b> (66%)		
		25 (19%)	25 (16%)		
4	8	1.5 h <b>27</b> (98%)	7 h 27 (91%)		
<sup>a</sup> Enoxide (5.0 mm	ol) catalyst (1 mol%) MeOH	(10 ml) room temp			

ixide (5.0 mmol), catalyst (1 mol%), MeOH (10 mL), room temp.

<sup>b</sup>EtOH was used instead of MeOH.

<sup>c</sup>2-Propyn-1-ol was used instead of MeOH.

<sup>d</sup>2 mol% of catalyst was used.

(b) Hydrolysis of epoxides.<sup>a</sup>

		ATRT	PPTS
Entry	Epoxide	Time product (yield)	Time product (yield)
1	1	1 h <b>31</b> (86%)	3 h <b>31</b> (84%)
2	2	1 h <b>32</b> (98%)	3 h <b>32</b> (91%)
3 <sup><i>b</i>,<i>c</i></sup>	3	24 h 33 (89%)	240 h 33 (83%)
4 <sup>b</sup>	6	24 h <b>36</b> (90%)	96 h <b>36</b> (87%)

<sup>a</sup>Epoxide (2.0 mmol), catalyst (5 mol%), MeCN-H<sub>2</sub>O (1:1) (4 mL), room temp. <sup>b</sup>10 mol% of catalyst was used.

<sup>c</sup>MeCN-H<sub>2</sub>O (2:1) was used.

(c) Acetolysis of epoxides. <sup>a</sup>					
		ATRT	PPTS		
Entry	Epoxide	Time product (yield)	Time product (yield)		
1	1	2 h <b>38</b> (87%) <sup>d</sup>	2 h <b>38</b> (88%)		
2	2	5 h <b>39</b> (80%)	5 h <b>39</b> (80%)		
3 <sup>6</sup>	4	92 h <b>42</b> (83%)	76 h <b>42</b> (85%)		
4 <sup>c</sup>	6	48 h 44 (83%)	40 h 44 (84%)		

<sup>a</sup>Epoxide (2.0 mmol), catalyst (1 mol%), acetic acid (4 mL), room temp.

<sup>b</sup>10 mol% of catalyst was used.

<sup>c</sup>5 mol% of catalyst was used.

<sup>d</sup>Compound 38 was obtained in 48% yield in the absence of ATRT.

possessing an alkoxy group, or epoxide 7 possessing an ester function was observed to give **9** in good yield (entries 1–4). Similarly, epoxide **2** reacted preferentially in the presence of epoxide **3**, **5**, **6**, or 7 to give **15**, respectively (entries 5–8). Selective ring opening of **1** with 2-propyn-1-ol in the presence of **2** led to the production of **14** in good yield (entry 9). These results were similar to those observed in the other acid catalysts such as electrogenarated acid.<sup>[4,10]</sup>

Table 4 shows the results for hydrolysis and acetolysis of various epoxides using 5–10 mol% of ATRT. ATRT works efficiently to give the corresponding diols **31–37** in good yields by the reactions of **1–7** in MeCN-H<sub>2</sub>O. Treatments of **1–6** in acetic acid catalyzed by 5–10 mol% of ATRT resulted in the formation of  $\beta$ -acetoxy alcohols **38–44** in good yields. Regioselective formation of  $\beta$ -acetoxy alcohols was observed in every case except epoxide **3**. The other regioi-somers were not detected for the acetolysis of epoxides **1**, **2**, and **4–6**.

In order to check the reusability of the catalyst, ATRT was removed by filtration and employed for the next experiment. ATRT was recycled five times without the loss of activity for alcoholysis, hydrolysis, and acetolysis of **1** (Table 5).

It is interesting to note that alcoholysis and hydrolysis of various epoxides promoted by ATRT proceeded faster than those by PPTS (Tables 6a and 6b). The higher activity of ATRT might be attributed to the hydrophobic effect of polymers.<sup>[30–33]</sup> Acetolysis of epoxides catalyzed by ATRT proceeded with almost the same rate or somewhat slower than those by PPTS (Table 6c). In these cases, the reactions were partially catalyzed by acetic acid because the acetolysis of 1 in the absence of ATRT afforded **38** in 44% yield (entry 1). PPTS is widely used as a mild reagent for many transformations such as tetra-hydropyranylation of alcohols,<sup>[34]</sup> deprotection of THP ethers<sup>[34]</sup> and silyl ethers,<sup>[35]</sup> and alcoholysis of epoxides.<sup>[36,37]</sup> However, it is somewhat difficult to handle in air because PPTS is hygroscopic. ATRT will be useful for these transformations because of its reusable and nonhygroscopic properties.

#### Conclusion

We have demonstrated that ATRT is an efficient solid acid catalyst for ring opening reactions of epoxides with alcohols, water, and acetic acid. Alcoholysis and hydrolysis of epoxides catalyzed by ATRT were faster than those by PPTS. It is worth pointing out that (1) ATRT can be synthesized by only one step (heating a mixture of aniline, terephthalaldehyde, and TsOH in EtOH followed by grinding; see the Supporting Information), (2) the workup procedure is remarkably simple, and (3) ATRT is nonhygroscopic and easy to handle.

#### **Experimental**

# General procedure for alcoholysis of epoxides catalyzed by ATRT

ATRT (50 mg, 1.0 mol%) was added to a solution of **1** (600 mg, 5.0 mmol) in dry EtOH (10 mL). The mixture was stirred at room temperature for 1.5 h. The catalyst was collected and washed with EtOH. Evaporation of the filtrate gave a residue, which was purified by column chromatography on silica gel using hexane–acetone (3:1) as eluent to afford **10**<sup>[38]</sup> (803 mg, 97%).

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# General procedure for hydrolysis of epoxides catalyzed by ATRT

ATRT (100 mg, 5.0 mol%) was added to a solution of **1** (240 mg, 2.0 mmol) in MeCN-H<sub>2</sub>O (1:1) (4 mL). The mixture was stirred at room temperature for 1 h. The catalyst was collected and washed with MeCN. Evaporation of the filtrate gave a residue, which was purified by column chromatography on silica gel using hexane-acetone (2:1) as eluent to afford **31**<sup>[38]</sup> (237 mg, 86%).

#### General procedure for acetolysis of epoxides catalyzed by ATRT

ATRT (100 mg, 5.0 mol%) was added to a solution of **1** (240 mg, 2.0 mmol) in acetic acid (4 mL). The mixture was stirred at room temperature for 0.5 h. The catalyst was collected and washed with  $Et_2O$ . Evaporation of the filtrate gave a residue which was purified by column chromatography on silica gel using hexane-acetone (2:1) as eluent to afford **38**<sup>[38]</sup> (313 mg, 87%).

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