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Rearrangement of epoxides to carbonyl compounds in the presence of reusable acidic zeolite catalysts under mild conditions

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Rearrangement of 4-substituted styrene oxides with acidic zeolite catalysts in the presence of polar solvents at room temperature gives the corresponding 4-substituted phenylacetaldehydes in high yields. The procedure has been applied successfully to the rearrangement of disubstituted epoxides. The zeolite can be easily recovered, regenerated by heating and reused.

KEY WORDS: rearrangement; epoxide; 4-substituted styrene oxides; zeolites; carbonyl compounds.

1. Introduction

Substituted epoxides are versatile compounds for organic syntheses due to their ready availability and easy transformation to a wide range of organic compounds [1–4]. Several catalytic systems have been developed for the rearrangement of epoxides to carbonyl compounds. These include Brφnsted or Lewis acids [5–8], including $BF_3 \cdot Et_2O$ [9–11], lithium salts [12], $MgBr_2$ [13,14], $Pd(OAc)_2$ in the presence of phosphine ligands [15], $InCl_3$ [16], $IrCl_3 \cdot nH_2O$ [17], $Er(OTf)_3$ [18], $Sn(OTf)_2$ [19] and hydrated $BiOClO_4$ [20]. Unfortunately, most such catalytic reagents create a number of environmental disadvantages, such as the production of large quantities of toxic or corrosive waste resulting from the use of stoichiometric quantities. Moreover, the rearrangement reactions mostly need to be carried out at high temperatures for long reaction times.

Major effort has therefore been made to find environmentally friendly processes for the rearrangement of epoxides. The use of solid catalysts can overcome many of the limitations associated with the use of solution phase Lewis or Brφnsted acid catalysts. The nature and proportions of the products are dependent largely on the types of catalyst used. In particular, various types of zeolites, such as HY, HZSM-5 and offretite, effect the rearrangement of styrene oxide, at 95 °C in toluene, to give a good yield of phenylacetaldehyde [21–24]. Also, rearrangements of different epoxides have been reported over acidic zeolites at 200–300 °C in the gas phase [25].

We have shown that solid catalysts [26] can have advantages in alkylation [27], acylation [28,29], methanesulfonylation [30], bromination [31], chlorination [32] and nitration [33,34] of aromatic compounds. Therefore, we decided to investigate the scope and limitations of rearrangement reactions of epoxides and to try to develop a simple process, involving mild conditions. We have already given a preliminary account of some of the results [35]. We now report the full details of this work.

2. Experimental

2.1. Materials

Chemicals were purchased from Aldrich Chemical Company and used without further purification. Commercial zeolites were purchased from Aldrich Chemical Company or provided as gifts from Zeolyst International. All zeolite catalysts were freshly calcined at 400 °C for a minimum of 4 h prior to use.

2.2. Analysis and characterisation of the products

^1H and ^{13}C NMR spectra were recorded on an AV400 Bruker spectrometer operating at 400 MHz for ^1H and 100 MHz for ^{13}C measurement.

Product mixtures from the rearrangement reactions of epoxides (Schemes 1–5) were subjected to gas chromatography on a Philips PU 4400 gas chromatograph, fitted with an HP-1 (100% dimethylpolysiloxane; 30 m, 0.32 mm ID) column. The product from rearrangement of 1,2-epoxyoctane (**7**) was quantified by the use of a DB-225 (50% cyanopropylmethylsilicone, 50% methylphenylsilicone; 30 m, 0.25 mm ID) column. The GC conditions used for analysis were: 100 °C for 1 min, ramped to 160 °C at 15 °C/min and held for 1 min. The injection temperature was 200 °C and the detection temperature 250 °C. Hexadecane was used as an internal standard.

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Styrene oxide (**1a**) is a commercial material and was used without further purification. 4-Substituted styrene oxides **1b–f**, *trans*-stilbene oxide (**3a**), *cis*-stilbene oxide (**3b**), 1,1-diphenylethene oxide (**6**) and 1,2-epoxyoctane (**7**) were prepared according to literature procedures [36–40].

The structures of products **2**, **4**, **5** and **8**, obtained from the rearrangement reactions of the corresponding epoxides **1**, **3**, **6** and **7**, were confirmed by their ^1H and ^{13}C NMR spectra, and were identical to those reported in the literature [41–51].

2.3. *Typical experimental procedure for the rearrangement of 4-substituted styrene oxides **1** in the presence of acidic zeolite catalysts*

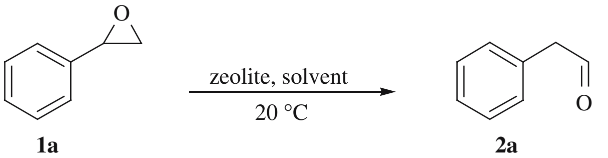
Quantities are recorded in the footnotes to the appropriate tables or text. All reactions were carried out under a nitrogen atmosphere in a 100 ml two necked round bottomed flask equipped with a water condenser and a magnetic stirrer. In a typical experiment, epoxide **1** (8.3 mmol) was directly added to a mixture of freshly calcined zeolite catalyst and chloroform (10 ml) and the mixture was stirred at 20 °C for the appropriate reaction time. Samples (1 ml) were withdrawn periodically, hexadecane (0.2 g) was added as internal standard, and the aliquot was filtered to remove the zeolite before analysis by gas chromatography. When the reaction was complete, the bulk sample was filtered and the catalyst was washed with $CHCl_3$ (3×30 ml), then the solvent was removed under reduced pressure. ^1H NMR spectroscopy was used to identify and quantify the product **2** [41–46].

2.4. *Experimental procedure for the rearrangement of trans-stilbene oxide (**3a**), cis-stilbene oxide (**3b**) and 1,1-diphenylethene oxide (**6**) in the presence of acidic zeolite catalysts*

The procedure was identical to that used for the rearrangement of 4-substituted styrene oxides **1**, except that **3a**, **3b** or **6** (1.0 g, 5.1 mmol) was used in $CHCl_3$ (10 ml) at 20 °C under nitrogen. The reaction mixture was worked up and the product was identified and quantified by GC and NMR [47–50].

2.5. *Experimental procedure for the rearrangement of 1,2-epoxyoctane (**7**) in the presence of acidic zeolite catalysts*

A mixture of **7** (1.0 g, 7.0 mmol) and zeolite catalyst (0.5 g) in $CHCl_3$ (10 ml) was heated at the appropriate temperature for the appropriate reaction time under a nitrogen atmosphere. The reaction mixture was worked up and the product was identified and quantified by GC and NMR [51].



Scheme 1. Catalytic rearrangement of styrene oxide (**1a**).

3. Results and discussions

Initially, we investigated the rearrangement of styrene oxide (**1a**) to phenylacetaldehyde (**2a**) (Scheme 1), over the zeolite HZSM-5 (Si/Al = 75) in toluene as solvent at 95 °C, as described in the literature [21]. The yield of phenylacetaldehyde (**2a**) was 77%. Although the yield was good, and the GC trace showed no other products, the yield was much less than the quantitative yield reported in the literature. Further investigation revealed that the remaining styrene oxide was converted into polymeric material that did not show up in the gas chromatographic analysis.

In order to test the effect of the solvent in this process, several reactions of styrene oxide (**1a**) were carried out over zeolite HZSM-5, under identical conditions except for the nature of the solvent. The reactions were conducted at 20 °C in order to allow differences in rates of reaction to be revealed. The results obtained are recorded in Table 1.

The results indicated that the yield of **2a** obtained was high after just 30 min when the reaction was carried out in diethyl ether (entry 6), acetone (entry 10), chloroform (entry 11) or dichloromethane (entry 12). However, the yield was low in methanol (entries 8 and 9), even after 4 h, presumably due to the weakening of the acidity of the catalytic sites by hydrogen bonding to methanol.

Table 1
Influence of solvent on the rearrangement of styrene oxide (**1a**) according to Scheme 1 in the presence of HZSM-5[a]

Entry	Solvent	Reaction time (h)	Conversion (%)[b]	Yield (%) of **2a**[c]
1	Toluene	0.5	28	26
2	Toluene	2	47	37
3	Toluene	5	71	59
4	Hexane	0.5	16	15
5	Hexane	5	82	78
6	Et_2O	0.5	94	88
7	Et_2O	1	97	85
8	MeOH	0.5	17	16
9	MeOH	4	49	47
10	MeCOMe	0.5	100	77
11	$CHCl_3$	0.5	100	79
12	CH_2Cl_2	0.5	70	66
13	CH_2Cl_2	2	96	84

[a] HZSM-5 (Si/Al = 75; 0.5 g) and **1a** (8.3 mmol) in solvent (10 ml) at 20 °C under N_2 for the indicated reaction time.
[b] Decrease in amount of **1a** by quantitative GC.
[c] Calculated from GC and ^1H NMR. Numbers expressed as percentages have been rounded to the nearest whole number.

Also, the reaction was slow in toluene (entries 1–3) and the yield of **2a** obtained was only 59% even after a long reaction time (5 h). Using hexane as solvent (entries 4 and 5) the yield of **2a** increased from 15 to 78% by increasing the reaction time from 0.5 to 5 h. As a result of these findings, chloroform was used as the solvent for further studies.

Several reactions were conducted using HZSM-5 zeolite samples with different Si/Al ratios. The results obtained are recorded in Table 2. As the results show, the yield of phenylacetaldehyde (**2a**) was uniformly high (77–79%) when HZSM-5 zeolites with Si/Al ratios of 40, 62.5 and 75 were used. Even with higher or lower Si/Al ratios the yields were still reasonable (73–74%). In further studies, therefore, zeolites HZSM-5 with Si/Al ratios between 40 and 75 could be used interchangeably, according to amounts available.

For comparison, a number of different acidic zeolites (0.5 g for 8.3 mmol of **1a**) were then screened for efficacy in the rearrangement of styrene oxide (**1a**) (Scheme 1) using various reaction times. The results obtained are recorded in Table 3.

Table 2
Influence of the HZSM-5 zeolite Si/Al ratios on the rearrangement of styrene oxide (**1a**) according to Scheme 1[a]

Entry	HZSM-5 (Si/Al)	Yield (%) of **2a**[b]
1	12.5	74
2	40	78
3	62.5	77
4	75	79
5	500	73

[a] HZSM-5 (0.5 g) and **1a** (8.3 mmol) in $CHCl_3$ (10 ml) at 20 °C under N_2 for 30 min.

[b] Calculated from GC and ^1H NMR. Numbers expressed as percentages have been rounded to the nearest whole number.

Table 3
Influence of acidic zeolite catalysts on the rearrangement of styrene oxide (**1a**) according to Scheme 1[a]

Entry	Zeolite (Si/Al)	Reaction time (h)	Conversion (%)[b]	Yield (%) of **2a**[c]
1	HZSM-5 (75)	0.25	98	88
2	HZSM-5 (75)	0.50	100	79
3	HM (10)	0.25	40	34
4	HM (10)	0.50	69	61
5	HM (10)	2	94	86
6	HY (30)	0.25	84	72
7	HY (30)	0.50	100	88
8	Hβ (12.5)	0.25	92	79
9	Hβ (12.5)	0.50	100	81
10	HX	6	20	14
11	HA	6	< 10	< 10

[a] Catalyst (0.5 g) and **1a** (8.3 mmol) in $CHCl_3$ (10 ml) at 20 °C under N_2 for the indicated reaction time.

[b] Decrease in amount of **1a** by quantitative GC.

[c] Calculated from GC and ^1H NMR. Numbers expressed as percentages have been rounded to the nearest whole number.

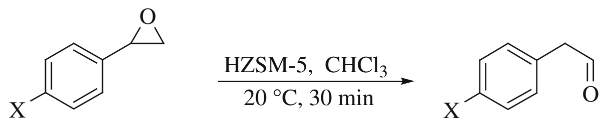
The results indicated that all medium and large pore zeolites can accommodate the reaction and that quite strongly acidic sites are better for effective catalysis. It was found, for example, that HY (entries 6 and 7) was more effective than HX (entry 10). The reaction was almost complete after 15 min over zeolites HZSM-5 (entry 1) and Hβ (entry 8), almost as fast over zeolite HY (entries 6 and 7), but significantly slower over zeolite HM (entries 3–5). However, all four of these zeolites gave high yields eventually (2 h in the case of HM – entry 5).

Another study was conducted using various quantities of the zeolite catalysts. It was found that increasing the amount of catalyst from 0.5 to 1.0 g (per 8.3 mmol of **1a**) brought decreases in the yield of **2a**. The reduction in yield may be due to increased formation of a polymeric material, which could be trapped in the catalyst or does not show in the GC analysis. A series of experiments was therefore conducted using a smaller quantity of the zeolite catalysts (0.2 g for 8.3 mmol of **1a**). The results obtained are recorded in Table 4. It is clear from the results that reducing the amount of catalyst from 0.5 to 0.2 g can still give a good yield of **2a**, but the reactions are slower and longer reaction times are required in order for them to go to completion. The fastest reaction was that involving HZSM-5 (Si/Al = 75), for which the conversion was 94% after 30 min (entry 2).

The reaction over zeolite HZSM-5 (Si/Al = 40) in chloroform was applied to a range of 4-substituted styrene oxides **1a–f** (Scheme 2). The yields obtained of the corresponding 4-substituted phenylacetaldehydes **2a–f** are recorded in Table 5.

The results indicated that the reaction was successful for a range of 4-substituted styrene oxides **1a–f** with electron-donating or electron-withdrawing groups. Somewhat higher yields were obtained with electron-withdrawing groups. The reaction has been proven to be fast, efficient and general under mild conditions, which render it attractive for organic syntheses.

Samples of zeolite HZSM-5 that were recovered from the reactions depicted in Schemes 1, 2 were regenerated by calcination at 400 °C and reused in rearrangement reactions identical to those from which the samples were recovered (rearrangement of **1a**). For comparison, a



1a-f **2a-f**

Scheme 2. Catalytic rearrangement of 4-substituted styrene oxides **1a–f**.

Table 4

Influence of acidic zeolite catalysts on the rearrangement of styrene oxide (**1a**) according to Scheme 1[a]

Entry	Zeolite (Si/Al)	Reaction time (h)	Conversion (%)[b]	Yield (%) of **2a**[c]
1	HZSM-5 (75)	0.25	85	82
2	HZSM-5 (75)	0.50	94	85
3	HM (10)	0.50	19	16
4	HM (10)	4	92	84
5	HY (30)	0.50	62	51
5	HY (30)	2	78	74
6	Hβ (12.5)	0.50	77	67
6	Hβ (12.5)	2	100	79

[a] Catalyst (0.2 g) and **1a** (8.3 mmol) in $CHCl_3$ (10 ml) at 20 °C under N_2 for the indicated reaction time.

[b] Decrease in amount of **1a** by quantitative GC.

[c] Calculated from GC and ^1H NMR. Numbers expressed as percentages have been rounded to the nearest whole number.

Table 5

Rearrangement of 4-substituted styrene oxides **1** in the presence of HZSM-5 according to Scheme 2[a]

Entry	X	Conversion (%)[b]	Yield (%) of **2**[c]
1	H	100	79
2	Me	100	73
3	OMe	100	76[d]
4	Cl	100	92
5	Br	100	75
6	NO_2	100	92

[a] HZSM-5 (Si/Al: 40, 0.5 g) and **1** (8.3 mmol) in $CHCl_3$ (10 ml) at 20 °C under N_2 for 30 min.

[b] Decrease in amount of **1** by quantitative GC.

[c] Calculated from GC using hexadecane as an internal standard.

[d] After 1 h.

series of experiments was conducted under similar reaction conditions for the rearrangement of **1a** in the presence of recovered non-calcined HZSM-5 samples. The yields obtained of phenylacetaldehyde (**2a**) in both series are recorded in Table 6.

As the results in Table 6 show, the HZSM-5 zeolite catalyst could be recycled and reused for at least six times with only modest reduction in the yield of **2a**, irrespective of whether the zeolite was calcined between runs or not. However, it is likely that calcinations would become necessary after a number of runs.

Our attention was next turned to the rearrangement of 1,2-disubstituted epoxides such as those from *trans*- and *cis*-stilbenes, and of 1,1-disubstituted epoxides such as 1,1-diphenylethene oxide. Treatment of *trans*-stilbene oxide (**3a**) and *cis*-stilbene oxide (**3b**) with $BF_3 \cdot Et_2O$ have been reported to give diphenylacetaldehyde (**4**) as the only product [14]. Initially, we attempted the rearrangement of *trans*-stilbene oxide (**3a**) over zeolite catalysts in chloroform at 20 °C (Scheme 3). On the assumption that the medium-size pores of HZSM-5 might be more reluctant to accommodate the larger epoxide than those of the large pore zeolites, Hβ (Si/Al = 12.5; 0.5 g) was initially screened for efficacy in the rearrangement of **3a** (1.0 g) in chloroform. The major products of the rearrangement were diphenylacetaldehyde (**4**) and α-phenylacetophenone (**5**), but with the aldehyde **4** predominating.

Table 6

Recovery and re-use of HZSM-5 zeolite in the rearrangement of styrene oxide (**1a**) according to Scheme 1[a]

Number of zeolite uses (run)	Yield (%) of **2a**[b]	
	Calcined HZSM-5	Non-calcined HZSM-5
1	79	79
2	69	68
3	65	62
4	62	60
5	62	56
6	62	47

[a] HZSM-5 (Si/Al: 40, 0.5 g) and **1a** (8.3 mmol) in $CHCl_3$ (10 ml) at 20 °C under N_2 for 30 min.

[b] Calculated from GC and ^1H NMR. Numbers expressed as percentages have been rounded to the nearest whole number.

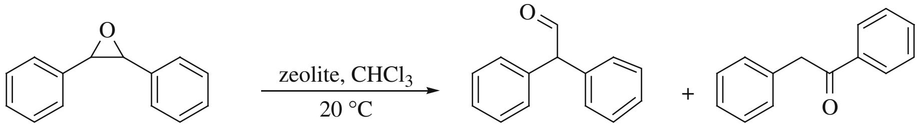
Table 7

Influence of acidic zeolite catalysts on the rearrangement of *trans*-stilbene oxide (**3a**) according to Scheme 3[a]

Entry	Zeolite (Si/Al)	Reaction time (h)	Yield (%)[b]	
			4	**5**
1	Hβ (12.5)	0.25	90	5
2	HY (30)	0.25	90	6
3	H-M (10)	4	91	3
4	HZSM-5 (75)	4	60	5
5	HZSM-5 (75)	6	69	6
6	HZSM-5 (75)	8	80	7

[a] Catalyst (0.5 g) and **3a** (1.0 g) in $CHCl_3$ (10 ml) at 20 °C under N_2 for the indicated reaction time.

[b] Calculated from ^1H NMR. Numbers expressed as percentages have been rounded to the nearest whole number.



3a,b **4** **5**

Scheme 3. Catalytic rearrangement of *trans*- and *cis*-stilbene oxides **3a** and **3b**.

Table 8
Influence of reaction time on the rearrangement of *trans*-stilbene oxide (**3a**) in the presence of acidic zeolite according to Scheme 3[a]

Entry	Zeolite (Si/Al)	Reaction time (h)	Yield (%)[b]	
			4	**5**
1	H*β* (12.5)	0.25	91	5
4	HY (30)	0.25	49	3
5	HY (30)	0.50	57	4
6	HY (30)	1	67	4
7	HY (30)	4	90	5
8	HM (10)	0.25	13	0
9	HM (10)	4	62	1
10	HM (10)	8	90	2
11	HM (10)	16	93	2
12	HZSM-5 (75)	8	58	4

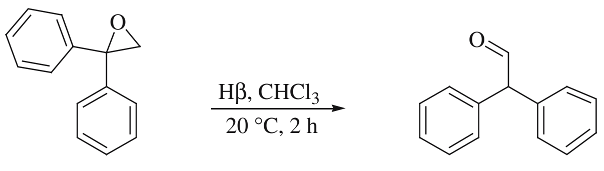
[a] Zeolite (0.2 g) and **3a** (1.0 g) in $CHCl_3$ (10 ml) at 20 °C under N_2 for the indicated reaction time.
[b] Calculated from ^1H NMR. Numbers expressed as percentages have been rounded to the nearest whole number.

The yields of **4** and **5** were 90 and 5%, respectively, after 15 min, but 86 and 7% (entry 7) when the reaction time was 4 h. The general trend on increasing the reaction time was a small decrease in the yield of **4**, and an even smaller increase in the yield of **5**, with possibly some formation of polymeric materials according to the NMR spectra.

In view of the success with H*β*, various zeolite catalysts were used for the rearrangement of **3a** (Table 7). As a result, we found that H*β* (entry 1) and HY (entry 2) were the most active solids, while HZSM-5 (entries 4–6) was the least active, reinforcing the view that *trans*-stilbene oxide (**3a**) might not fit easily into the channels of zeolite HZSM-5. In addition, we found that HM (entry 3) was essentially the most selective solid for the production of **4** over **5**, but the reaction was slower than those over H*β* or HY.

To determine if there was any benefit to be gained by reducing the quantity of zeolite catalyst, the quantity of zeolite was reduced to 0.2 g for 1.0 g of **3a**. The results obtained are given in Table 8. It was found that when the quantity of zeolite catalyst was reduced to 0.2 g, the yields of **4** and **5** were higher still, but the reaction was slow in most cases. The yield and selectivity were excellent over zeolite HM (Si/Al = 10) if the reaction was left for the appropriately long time (entry 10).

Similarly, rearrangement of *cis*-stilbene oxide (**3b**) over H*β* (Si/Al = 12.5) in chloroform at room temperature gave a mixture of **4** and **5** (Scheme 3) with the aldehyde **4** predominating. The yields of **4** and **5** were 28 and 3%, respectively, when the reaction time was 15 min, 80 and 9%, respectively, after 2 h and 86 and 9%, respectively, after 4 h.



6 **4** (86%)

Scheme 4. Catalytic rearrangement of 1,1-diphenylethene oxide (**6**).

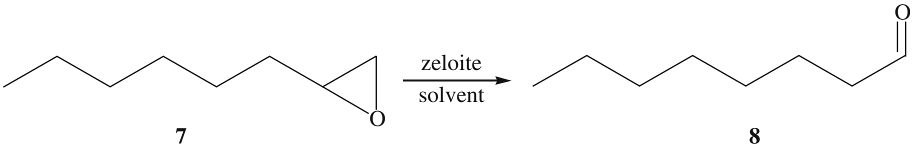
However, the rearrangement of 1,1-diphenylethene oxide (**6**; 1 g) over H*β* (Si/Al = 12.5; 0.5 g) in chloroform (10 ml) at 20 °C for 2 h gave only the corresponding aldehyde **4**, in 86% yield (Scheme 4).

Finally, in order to assess the usefulness of the rearrangement for aliphatic epoxides, rearrangement of 1,2-epoxyoctane (**7**) over various zeolite catalysts in chloroform was attempted (Scheme 5). However, the rearrangement of **7** in chloroform at 20 °C for 4 h gave octanal (**8**) in only ~1% yield in the case of H*β* (Si/Al = 12.5), HY (Si/Al = 30) or HM (Si/Al = 10).

The yield of **8** was ~2% when HZSM-5 (Si/Al = 75) was used as the catalyst. Even when the rearrangement of **7** was carried out over HZSM-5 for 24 h the yield of **8** was only 4%. When the temperature over 24 h was increased to 50 °C, the yield of **8** increased to 9%. When the reaction was carried out under reflux conditions for 24 h the maximum yield of **8** was still only 14%. The NMR and GC indicated that the majority of the reaction mixture was the starting material **7**.

4. Conclusion

The work reports a procedure that demonstrates a useful application of acidic zeolite catalysts for the efficient and selective synthesis of carbonyl compounds in high yields from the rearrangement of epoxides under mild conditions. Furthermore, heating easily regenerates the zeolite, which can be reused up to six times to give results similar to those obtained with a fresh sample of the catalyst.



7 **8**

Scheme 5. Catalytic rearrangement of 1,2-epoxyoctane (**7**).

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