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o-Phenylenediamine as a New Catalyst in the Highly Regioselective Conversion of Epoxides to Halohydrins with Elemental Halogens

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Summary. The regioselective ring opening of epoxides using elemental iodine and bromine in the presence of *o*-phenylenediamine as a new catalyst affords vicinal iodo alcohols and bromo alcohols in high yields. The major advantages of this method are versatility, high regioselectivity, a cheap and commercially available catalyst, mild and neutral reaction conditions, and short reaction times. *Fourier* transform *Raman* spectroscopy was used to study the reaction of iodine with *o*-phenylenediamine. The results indicate that the complex $[(\mathrm{Diamine})\mathrm{I}]^+ \cdot \mathrm{I}_5^-$ is formed, and we suggest that the major nucleophile is the pentaiodide ion. This bulky nucleophile has a fundamental role in the high regioselectivity observed attacking the less sterically hindered epoxide carbon.

Keywords. Catalyst; Diamines; Epoxides; Halohydrin; FT *Raman* spectroscopy.

Introduction

Epoxides are useful substrates in organic synthesis due to their high reactivity [1]. Conversion of an epoxide to a vicinal halohydrin is a synthetically useful reaction, which is typically performed with hydrogen halides [2]. Unless there is a structural or a stereochemical bias, a couple of products is obtained due to the lack of the regioselectivity in the ring opening step [3]. Moreover, the harsh reaction conditions and the lack of chemoselectivity among various substituted epoxides limit the synthetic utility of this reaction in multistep synthesis and have prompted a search for more selective and milder procedures [2]. The ring opening of unsymmetrical substituted epoxides with $\mathrm{Li_2[NiBr_4]}$ [4], pyridine · HCl [5], haloborane reagents [6–9], $\mathrm{Br_2/P}Ph_3$ [10], $\mathrm{Ti(O\text{-}}i\text{-}Pr)_4$ [11], chlorosilane [12], $[n\text{-}Bu_4\mathrm{N}]\mathrm{Br/Mg(NO_3)_2}$

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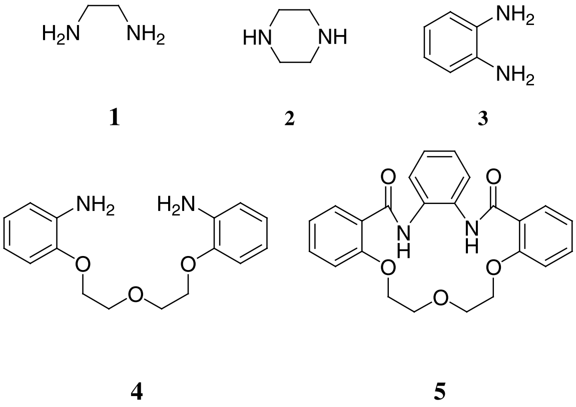
[13], *Lewis* acid metal halides [14–17] and *Me*$_3$SiBr [18], TiCl$_4$-Li*X* [14], $CeCl_3 \cdot 7H_2O$–NaI [19], SmI_2 [20], $POCl_3$ or PCl_3/*DMAP* [21], H*X* or Li*X*/β-cyclodextrin [22], and *Et*$_3$N $\cdot$ 3HF/MW [23] have been reported. However, these methods are not always fully satisfactory and suffer from disadvantages such as acidity, handling and *in situ* preparation of reagent, the noncatalytic nature of the reagents, lack of versatility, and relatively long reaction times [8, 11, 24–26]. It has been found that epoxides can be converted into iodohydrins and bromohydrins by means of elemental iodine and bromine [27], but this method has some severe limitations, such as low yield and lacking regioselectivity with long reaction times and formation of acetonide byproducts in addition to the expected iodoadduct. Furthermore, iodination does not occur using CH_2Cl_2, $CHCl_3$, C_6H_6, CH_3CN, and *THF* as solvents.

Recently, *Sharghi et al.* [28–33] solved these problems by catalytic ring opening of epoxides with elemental halogens. Some recently new synthesized macrocyclic diamides [28, 29], diamine podands [30], *Schiff*-base complexes of the first-row transition metals (II) [31], metalloporphyrins [32], and 2-phenyl-2-(2-pyridyl)imidazolidine [33] were applied successfully as catalysts for this ring opening reaction. Advantages of these procedures are: high regioselectivity, increase in chemical yield of halohydrin without any side product formation, short reaction times, and neutral and mild reaction conditions in inert solvents such as CH_2Cl_2.

In this study, we wish to report the results of the reactions of epoxides with elemental iodine and bromine in the presence of a catalytic amount of the inexpensive and readily available *o*-phenylenediamine.

Results and Discussion

Aromatic diamines are valuable building blocks for the preparation of polyamides [34], macrocyclic diamides [35], and azacrowns [36]. Because of these properties they are inexpensive and commercially available. The diamines **1**–**3**, which are commercially available, were used as catalysts (Scheme 1). The results of the

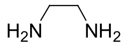
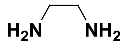
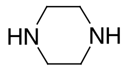
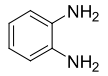
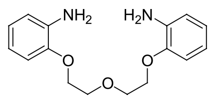
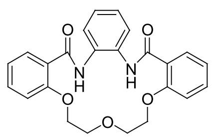


1 **2** **3**

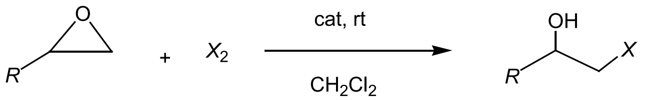
4 **5**

Scheme 1

Table 1. Reaction of styrene oxide (1 mmol) with elemental iodine (1 mmol) in the presence of representative catalysts in CH_2Cl_2 at 25°C

Entry	Catalyst (0.1 mmol)	Conditions	Time/h	Yield/%[a]	Ref.
1	–	I_2, rt/acetone	2	83	[27]
2	–	I_2, excess/CH_2Cl_2	–	0	[27]
3	–	LiI, *Ac*OH, *THF*, rt	1.3	87	[24a]
4		I_2, rt/CH_2Cl_2	1	50	
5		I_2, rt/CH_2Cl_2	24	80	
6		I_2, rt/CH_2Cl_2	1	40	
7		I_2, rt/CH_2Cl_2	1	>98	
8		I_2, rt/CH_2Cl_2	0.75	>95	[30]
9		I_2, rt/CH_2Cl_2	24	90	

[a] Relative yield according to GC



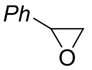
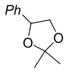
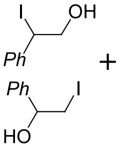
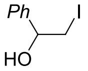
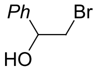
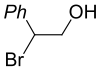
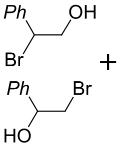
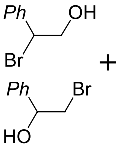
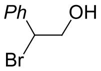
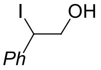
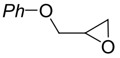
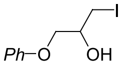
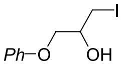
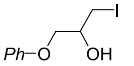
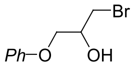
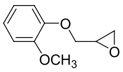
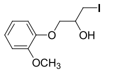
Scheme 2

reactions of styrene oxide with elemental iodine in the presence of **1**–**3**, the above catalysts, are summarized in Table 1 and are compared with the corresponding results obtained in recently applied podand catalyst **4** [30] and the macrocyclic diamide **5** (Scheme 2). In each case, cleavage of the epoxide ring occurs and upon thiosulfate workup the corresponding iodohydrin was obtained. In comparison, the cleavage behavior of styrene oxide with elemental iodine in the absence of catalyst is given in entries 1–3. As shown in Table 1, yields of iodination with this methodology are quite good and catalyst **3** is the most effective one (Table 1, entry 7). However, iodination of styrene oxide with an excess of elemental iodine in the absence of catalyst did not occur even under reflux and extension of reaction time to several days, and unreacted styrene oxide was completely recovered.

Diamine **3** in comparison with the corresponding synthetic macrocyclic diamide **5** [28, 35a] and the diamino podand **4** [30, 35b] is the best selection because it's inexpensive and readily available with high conversion yield in short reaction times. The catalyst was easily recovered and could be reused several times.

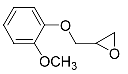
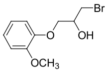
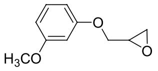
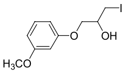
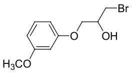
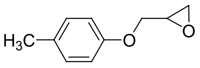
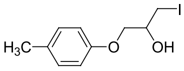
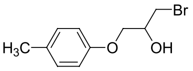
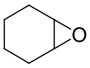
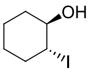
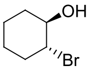
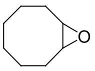
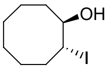
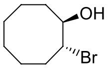
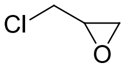
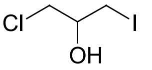
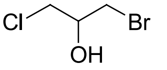
The results obtained with some representative epoxides in the presence of *o*-phenylenediamine (**3**) as the catalyst are summarized in Table 2 and are compared

Table 2. Reaction of epoxides with elemental bromine and iodine in the presence of *o*-phenylenediamine (**3**) as the catalyst

Entry	Epoxide	Catalyst	Mol %	Reaction conditions	Time/h	Yield/%[a]	Product(s)	Ref.
1		–	–	I_2, acetone, rt	2	83		[27]
2	"	–	–	I_2 excess/ CH_2Cl_2, reflux	–	–	No reaction	[27]
3	"	–	–	LiI, *Ac*OH, *THF*, rt	1.3	87 (2:1)		[24a]
4	"	**3**	10	I_2, rt, CH_2Cl_2	0.25	96		
5	"	**3**	10	Br_2, rt, CH_2Cl_2	Immed.	95		
6	"	–	–	Br_2, rt, CH_2Cl_2	1	31		[27]
7	"	–	–	$n\text{-}Bu_4N^+Br^-$/ $Mg(NO_3)_2$, $CHCl_3$, rt	5	78 (1:5)		[13]
8	"	–	–	$(Me_2N)_2BBr$/ CH_2Cl_2, N_2, atm, rt	12	75 (4.5:1)		[8]
9	"	–	–	HBr, rt, $CHCl_3$	0.25	>99		[25]
10	"	–	–	HI, rt, $CHCl_3$	0.25	>99		[25]
11		**4**	10	I_2, rt, CH_2Cl_2	10	90		[30]
12	"	**3**	10	I_2, rt, CH_2Cl_2	8	90		
13	"	**3**	20	I_2, rt, CH_2Cl_2	4	93		
14	"	**3**	20	Br_2, rt, CH_2Cl_2	1.5	95		
15		**3**	20	I_2, rt, CH_2Cl_2	3	92		

(*continued*)

Table 2 (*continued*)

Entry	Epoxide	Catalyst	Mol %	Reaction conditions	Time/h	Yield/%[a]	Product(s)	Ref.
16		**3**	20	Br_2, rt, CH_2Cl_2	1.5	94		
17		**3**	20	I_2, rt, CH_2Cl_2	4.5	88		
18	"	**3**	20	Br_2, rt, CH_2Cl_2	2	92		
19		**3**	20	I_2, rt, CH_2Cl_2	4	85		
20	"	**3**	20	Br_2, rt, CH_2Cl_2	2	88		
21		**3**	10	I_2, rt, CH_2Cl_2	1	94		
22	"	**3**	10	Br_2, rt, CH_2Cl_2	0.5	96		
23		**3**	20	I_2, rt, CH_2Cl_2	5	75		
24	"	**3**	20	Br_2, rt, CH_2Cl_2	2.5	80		
25		**3**	10	I_2, rt, CH_2Cl_2	3	82		
26	"	**3**	10	Br_2, rt, CH_2Cl_2	2	87		

[a] Isolated yield

with the corresponding results obtained in the reaction of the same epoxides in the absence of catalyst (Table 2, entries 1, 2, and 6). On the other hand, some other methods for conversion of epoxides to the corresponding halohydrins are given in Table 2, entries 3 and 7–10 for comparison.

When epoxides were allowed to react in the presence of **3**, increases in yield and regioselectivities were observed in all of the reactions studied. However, bromination is faster than iodination cleavage. Generally, the optimum amounts of the catalysts were found to be 0.1 mol per 1 mol of epoxide and halogen. Thus in the case of the phenoxy epoxide (Entry 12), when the reaction was carried out using 0.1 equivalent of catalyst at 25°C for 8 h, the corresponding iodohydrin was obtained in 90% yield. However, the use of a bigger amount of catalyst (0.2 equivalent) provided a shorter reaction time of 4 h to affording the iodohydrin in 93% yield. So, in the cases of entries 13–20, 23, and 24 reactions were reacted in the presence of 0.2 mol of the catalyst.

In addition, other factors can have a pronounced effect on the observed ratio of halohydrin isomers and overall yield *e.g.*, rate and order in which the reagents are combined. For example, if iodine is added to the epoxide before the catalyst, two isomeric iodohydrins are produced. However, if the epoxide is added to the catalyst, and then iodine is added over a period of time (45 min), only one isomer is formed. In this case, the rapid addition of iodine reduced the regioselectivity.

A comparison of the reaction of the same epoxide with elemental iodine in the presence of **4** as the catalyst indicated that an increase in steric hindrance at the catalyst structure results in a general decrease in the rate of halohydrin formation (long reaction times, Table 2, entry 11).

Recent studies of a halogenative cleavage of epoxides with elemental halogens showed that in the absence of a catalyst, iodination does not occur in CH_2Cl_2, $CHCl_3$, C_6H_6, and CH_3CN as solvents, while it occurred in acetone solution with some limitations [27]. Whereas in the presence of a catalyst halogenations proceed [28–33] most cleanly in CH_2Cl_2, $CHCl_3$, C_6H_6, and CH_3CN solutions, whereas, those done in *THF* and acetone led to a lower yield of halohydrins. Now, our observations showed that iodine solutions in CH_2Cl_2, $CHCl_3$, and C_6H_6 cannot produce a polyiodide ion, whereas in acetone concomitantly with the disappearing of iodine bands a strong absorption band is observed at 364 nm corresponding to I_3^- or I_5^- and the iodine bands have vanished. The intensity of this band in *THF* and CH_3CN decreased in this order: acetone > *THF* > CH_3CN, presumably due to the complex formation between iodine and electron pair donor ligands [37, 38]. So, we suggest that the useful solvents for the catalytic cleavage of epoxide rings by elemental halogens are those without electron pair donors, such as CH_2Cl_2, $CHCl_3$, and C_6H_6, whereas for non-catalytic cleavage acetone is a properly selected solvent.

The variation in yield and rate of cleaving epoxides by elemental iodine or bromine in the presence of a catalyst can be satisfactory rationalized in terms of the suggested mechanism [28–33]. In support of this mechanism, the UV spectra of iodine, catalyst **3**, and complex formation between iodine and catalyst **3**, and complex formation of iodine with all used catalysts in dichloromethane solution at 25°C are shown in Figs. 1 and 2. As can be seen from Fig. 1, none of the initial

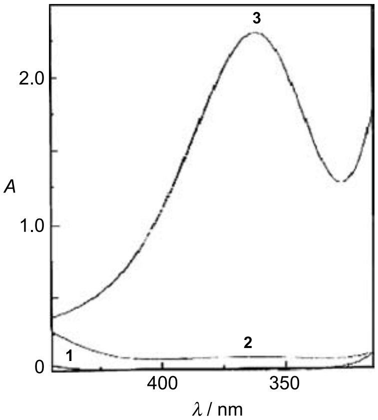


Fig. 1. Absorption spectra of: (1) *o*-phenylenediamine **3**; (2) iodine; (3) complex of *o*-phenylenediamine **3** with iodine in dichloromethane solution

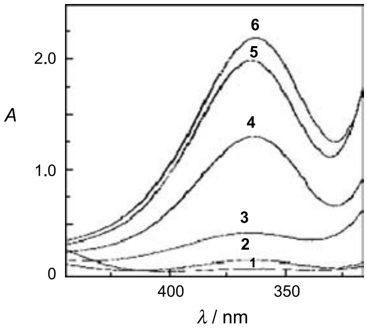


Fig. 2. Absorption spectra of iodine (1) and complexes between iodine and the used catalysts: macrocyclic diamide **5** (2); piperazine **2** (3); ethylene diamine **1** (4); diamino podand **4** (5); *o*-phenylenediamine **3** (6)

reactants show any measurable absorption in the 315–445 nm region, whereas the addition of diamine **3** to iodine results in a strong absorption band at 362 nm, presumably due to the complex formation of iodine with diamine **3**. It should be noted that the band at 362 nm is characteristic for the formation of polyiodide ions such as I_3^- and I_5^-, in the process of complex formation between iodine and electron-pair-donating nitrogen atoms [37, 38]. It is well known that both ions absorb in the same region around 360 and 290 nm with little deviation in their absorptions [37c, 37e].

As shown in Fig. 2, in all cases a characteristic band at 362 nm was observed, which is specific for the formation of polyiodide ion. The intensity of this band, as evidence for the ease of formation of polyiodide ion, decreased in the order **3**>**4**>**1**≫**2**>**5**. In the cases of **3** and **4** this band appeared immediately and corroborated the much faster complexation of iodine with these catalysts.

Photometric titrations of **3** by iodine, monitoring the absorbance at 360 nm, showed that until one equivalent of iodine is consumed, no absorption is detected. However, with addition of an excess of iodine an absorption band developed at 360 nm that is characteristic for the formation of polyiodide ions, such as I_3^- and I_5^-. The intensity of this band increased until three equivalents were consumed. While the molar ratio of iodine:diamine was increased up to 4:1, a new absorption band developed at 505 nm that is characteristic for excess of iodine. Accordingly, the polyiodides complex might be identified as $[(\text{Diamine})\text{I}]^+ \cdot \text{I}_5^-$. The proposed $[(\text{Diamine})\text{I}]^+$ type of the cation is analogous to $[(\textit{HMTA})\text{I}]^+$ reported by *Bowmaker et al.* [39] in the reaction of iodine with hexamethylenetetramine (*HMTA*) and also to $[(\textit{TACTD})\text{I}]^+$ and $[(\textit{TODACOD})\text{I}]^+$ reported by *Nour et al.* [38], to be formed in the reaction of iodine with the cyclic polyamine 1,4,8,11-tetraazacyclotetradecane (*TACTD*) and 1,4,10,13-tetraoxa-7,16-diazacyclooctadecane (*TODACOD*). On the other hand, the formation of I_5^- ions has been reported [40] to be formed in the reactions of iodine with trimesic acid (*TMA*) and $(CH_3)_4NI$.

On cooling, the mixture deposited a brown solid complex and the structure of this complex was studied. The *Raman* and the far-IR spectra of the diamine-iodine complex in the solid phase are shown in Figs. 3 and 4. According to the earliest mentioned mechanism [28–33], we initially concentrated on the spectral features of the triiodide systems [38–42], since we believed that these display the

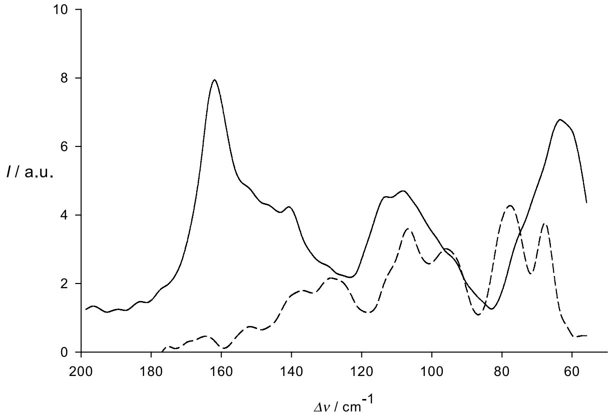


Fig. 3. The *Raman* spectra of $[(\mathrm{Diamine})\mathrm{I}]^+ \cdot \mathrm{I}_5^-$ (—) and **3** (- - -)

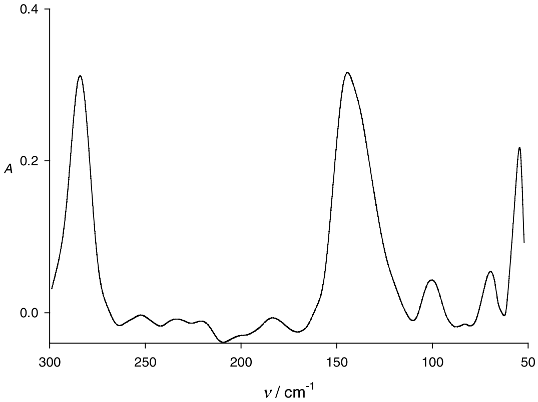


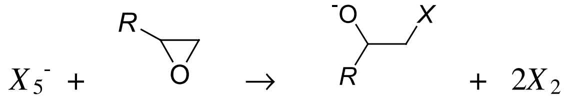
Fig. 4. The far-IR spectra of $[(\mathrm{Diamine})\mathrm{I}]^+ \cdot \mathrm{I}_5^-$

fundamental role in the course of the reaction. The *Raman* spectra of the triiodide systems exhibit a sharp band at $110\,\mathrm{cm}^{-1}$ and additional weak bands in the region of 130–$170\,\mathrm{cm}^{-1}$, whereas the other polyiodides, such as I_5^-, I_7^-, and I_{11}^-, produce an increase in intensity of the band at about $170\,\mathrm{cm}^{-1}$ relative to the band at $110\,\mathrm{cm}^{-1}$. This band also shifts to higher wavenumbers until it reaches that of free iodine (approx. $180\,\mathrm{cm}^{-1}$). Solid I_2 is known [40] to show a *Raman* active stretching frequency near $190\,\mathrm{cm}^{-1}$ which was not observed in our spectrum. The comparison of the *Raman* results with reported data [38b, 41, 42] (Table 3) on the pentaiodide systems strongly suggests the identity of the major chromophore with the pentaiodide (I_5^-) anion. The strong scattering band at $162\,\mathrm{cm}^{-1}$ was assigned to a fundamental normal mode of vibration in I_5^-. Thus we suggest that the major nucleophile in the course of the reaction is I_5^-, and this bulky nucleophile has a fundamental role in the high regioselectivity observed attacking on the less sterically hindered epoxide carbon.

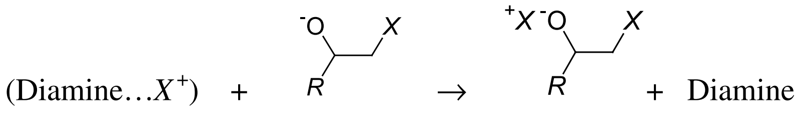
Accordingly, it is supported that the halogenative cleavage of epoxides occurs by the following four-step mechanism: The first step involves the formation of a 1:3 or 2:3 molecular complex between diamine and elemental halogen, in which

Table 3. Fundamental stretching vibrations for some pentaiodide ions

Entry	Pentaiodide complexes Ref.	*Raman*, $\bar{\nu}/\text{cm}^{-1}$	Far-IR, $\bar{\nu}/\text{cm}^{-1}$
1	$[(\text{Diamine})\text{I}]^+ \cdot \text{I}_5^-$ (present work)	162 vs, 154 sh, 141 m, 97 sh, 63 s	183 w, 160 sh, 144 vs, 100 mw
2	$[Et_3\text{S}]^+\text{I}_5^-$ [41]	~170 s, ~154 sh, ~110 w	160 sh, 145 s, 85 m
3	$[(\textit{TODACOD})\text{I}]^+ \cdot \text{I}_5^-$ [38b]	164, 137 m, 110, 87	
4	$[(\text{Mn}(\textit{modtc})_3]\text{I}^- \cdot 2\text{I}_2$ [42]	165 sh, 143 s	



Scheme 3



Scheme 4

the pentahalide ion (X_5^-) exists as an intimiate ion pair: Diamine + $3X_2 \rightarrow$ (Diamine...X^+)X_5^- or 2Diamine + $3X_2 \rightarrow$ (2Diamine...X^+)X_5^-. In the second step this complex is further decomposed to release X_5^- ion into solution as: (Diamine...X^+)$X_5^- \rightarrow$ (Diamine...X^+) + X_5^-.

Thus, in this way molecular halogen is converted to a nucleophilic halogen species in the presence of a suitable diamine. In the third step this ion participates in the ring opening reaction of epoxides (Scheme 3). Finally, the catalyst is regenerated in step 4 (Scheme 4). These steps occur continuously until all of the epoxides and halogen are consumed and after workup, the catalyst can be recovered easily.

The decrease in regioselectivity that results by merely reversing the order of mixing of epoxide and halogen, namely the slow addition of epoxide to bromine or fast addition of bromine to epoxide before the catalyst was added can readily be understood from this model. When the initial epoxide was introduced (in the absence or presence of catalyst) it would encounter an excess amount of bromine; electrophilic attack by bromine can then occur, giving the more stable carbonium ion-like transition state, and bromine anions will attack the more substituted carbon. On the other hand, slow addition of bromine to the mixture of catalyst and epoxide fosters the four-step mechanism presented above in which all of the elemental bromine is converted to Br_5^- by the catalyst and it then attacks the less substituted carbon selectively.

In conclusion, we have found that inexpensive and readily available **3** can catalyze the regioselective ring opening of epoxides by elemental iodine and bromine under neutral conditions with a variety of sensitive functional groups, as well as that it is a convenient procedure, which makes this synthetic technique highly useful. Resonance *Raman* spectroscopy indicated that the complex $[(\text{Diamine})I]^+ \cdot I_5^-$ is formed. Accordingly, we suggest that the major nucleophile in the course of the reaction is the pentaiodide ion, I_5^-. This bulky nucleophile plays a fundamental role in the high regioselectivity observed. It is due to an attack on the less sterically hindered epoxide carbon.

Experimental

All materials and solvents were obtained from Merck (Germany) and Fluka (Switzerland). Melting points were determined in open capillary tubes in an Electrothermal IA 9000 melting point apparatus. ^{1}H NMR spectra were recorded on a Bruker-80 MHz instrument using *TMS* as an internal standard. UV-Vis spectra were obtained with a JASCO spectrometer. IR spectra were recorded on a Shimadzu-IR 470 spectrometer. The far-IR spectrum in the region 650–50 cm^{-1} was obtained using a Thermo Nicolet NEXUS 870 FT-IR spectrometer equipped with a *DTGS*/polyethylene detector and a solid substrate beam splitter. The spectrum was collected with a resolution of 4 cm^{-1} by coadding the results of 125 scans. The *Raman* spectrum was recorded employing a 180° back-scattering geometry and a Bomem MB-154 *Fourier* transform *Raman* spectrometer. It was equipped with a ZnSe beam splitter and a TE cooled InGaAs detector. *Rayleigh* filtration was afforded by a set of two holographic technology filters. The spectrum was collected with a resolution of 4 cm^{-1} by coadding the results of 1000 scans. The purity determination of the substrates and reaction monitoring was accomplished by TLC on silica gel polygram SILG/UV 254 plates or GLC on a Shimadzu GC-10A instrument with a flame ionization detector using a column of 15% carbowax 20 M chromosorb W acid-washed 60–80 mesh. Column chromatography was carried out on short columns of silica gel 60 (230–400 mesh) in glass columns (2–3 cm diameter) using 15–30 g silica gel per 1 g of crude mixture. Compounds **4** and **5** were obtained according to Ref. [35].

General Procedure for Halogenative Cleavage of Epoxides

Epoxide (1 mmol) in 5 cm^3 of CH_2Cl_2 was added to a stirred solution of 0.1–0.2 mmol of *o*-phenylenediamine (**3**) in 5 cm^3 of CH_2Cl_2 at room temperature. Next, a solution of 1 mmol of elemental halogen in 10 cm^3 of CH_2Cl_2 was added dropwise during 45 min. The progress of reaction was monitored by TLC and GLC. After complete disappearance of the starting material, the reaction mixture was washed with 2×10 cm^3 of 10% aqueous $Na_2S_2O_3$ and 2×10 cm^3 of H_2O. The aqueous layer was further extracted with 2×10 cm^3 of CH_2Cl_2. The combined organic layer was dried (anh. $MgSO_4$) and the solvent was evaporated. The crude product was purified by chromatography on a column of silica gel. The halohydrins obtained throughout this procedure were identified by comparison with authentic samples prepared according to literature procedures [17, 22, 24a, 28].

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