Regioselective synthesis of *vic*-halo alcohols and symmetrical or unsymmetrical *vic*-dihalides from epoxides using triphenylphosphine – *N*-halo imides

Nasser Iranpoor, Habib Firouzabadi, Roya Azadi, and Farzaneh Ebrahimzadeh

Abstract: A simple, novel, and highly regioselective cleavage of epoxides into vicinal halo alcohols and symmetrical or unsymmetrical dihalides is described using different stoichiometries of triphenylphosphine (PPh_3) and *N*-halo succinimide (NXS) or *N*-halo saccharine (NXSac).

Key words: *N*-halo succinimide (NXS), *N*-halo saccharine (NXSac), triphenylphosphine (PPh_3), epoxide, *vic*-halo alcohol, symmetrical dihalide, unsymmetrical dihalide.

Résumé : On a effectué un nouveau clivage simple et hautement régiosélectif d'époxydes qui conduit à des halogénoalcools et des dihalogénures symétriques et non symétriques vicinaux et qui a été réalisé en utilisant des stoechiométries diverses de triphénylphosphine, de *N*-halogénosuccinimide (NXS) ou de *N*-halogénosaccharine (NXSac).

Mots clés : *N*-halogénosuccinimide (NXS), *N*-halogénosaccharine (NXSac), triphénylphosphine (PPh_3), époxyde, halogénoalcool vicinal, dihalogénure symétrique, dihalogénure non symétrique.

[Traduit par la Rédaction]

Introduction

Epoxides are suitable starting materials for the preparation of different functional groups (1). Owing to the importance of vicinal halo alcohols in organic synthesis (2, 3), diverse methods have been developed for their preparation from epoxides. The conventional reagents for epoxide ring opening to halo alcohols are hydrogen halides and hypohalite–water (4). However, the disadvantages of these procedures are an intolerance to acid-sensitive moieties and by-product formation (5). A great effort has been made in the last few years to find milder procedures for converting epoxides into halo alcohols. For example, silyl halides can be added to epoxides to give *O*-silyl protected halo alcohols (6). Other methods include the use of a halogen and triphenylphosphine (PPh_3) (7), or disubstituted borane halogenides (8), β-bromo bis(dimethylamino)borane (9), monochloroborane – dimethyl sulfide (10), $Li_nM_nX_n$ (M = Ni, Cu, Ti) (11), and MX_n (12). Also, different metal salts (13–17) in the presence of halide ions, TMSCl, and phosphaferrocene as the catalyst (18); lithium halides in the presence of silica gel or Amberlyst 15 (19, 20); a combination of elemental halogen and 2-phenyl-2-(2-pyridyl)imidazolidine (PPI) (21), phenyl hydrazine (22), or crown ethers (23); PPh_3–CBr_4 (24), and acyl methylimidazolium halide ([AcMIm]X) (25) are among the reagents used for this transformation. Although there are many methods reported for the conversion of epoxides into vicinal halo alcohols, their conversion to symmetrical or unsymmetrical *vic*-dihalides is rarely found in the literature. The use of reagents such as PPh_3–CCl_4 (26) and PPh_3–X_2 (X = Cl, Br) (27) is reported for the preparation of symmetrical *vic*-dihalides and PPh_3–X_2 (X = Cl, Br) followed by using HCl–THF for the preparation of unsymmetrical *vic*-dihalides (27).

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Results and discussion

Recently, we reported a method for the preparation of *vic*-halo alcohols or symmetrical or unsymmetrical dihalides (28) from epoxides using a combination of PPh_3–DDQ–R_4NX (X = Cl, Br, I). In conjunction with this research and in continuation of our previous work on the use of PPh_3 – *N*-halo succinimide (NXS) (29), we now introduce a new, mild, regioselective, and simpler method for the efficient conversion of epoxides to *vic*-halo alcohols or symmetrical or unsymmetrical *vic*-dihalides. In this method, epoxides in the presence of PPh_3–NXS or PPh_3 – *N*-halo saccharine (NXSac) are converted to *vic*-halo alcohols or symmetrical or unsymmetrical *vic*-dihalides depending on the molar ratio and the type of halo imide used (Scheme 1).

We took oxiranylmethyl phenyl ether (Table 1, entry 1) as an example and optimized the reaction conditions for its conversion to *vic*-bromo alcohol. It was observed that the mixture of oxiranylmethyl phenyl ether – PPh_3 – NBS or NBSac with the molar ratio of 1:1.2:1.2 in 1% aqueous acetonitrile at room temperature is suitable for the quantita-

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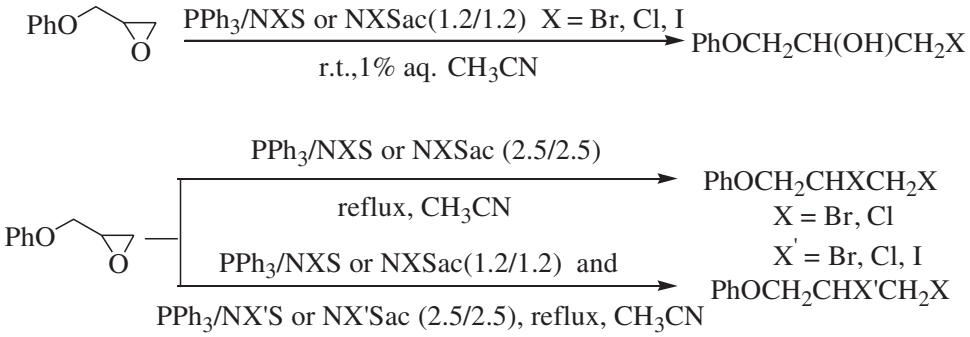
Scheme 1.



tive conversion of oxiranylmethyl phenyl ether to 1-bromo-3-phenoxy-2-propanol. Aqueous acetonitrile is required to hydrolyze the intermediate to the corresponding vicinal halohydrine. Similar reaction conditions were then applied for the conversion of structurally different epoxides to their corresponding *vic*-halo alcohols. The results are shown in Table 1.

Under these reaction conditions, the ethereal bonds, ester groups, carbon–carbon double bond, and activated aromatic ring do not interfere with the formation of halo alcohols and remain intact. Electronic and steric factors control the orientation of the attack of halide ion. In the case of styrene oxide, owing to electronic factors, the attack of the halide ion occurs mainly at the more substituted position. However, in other epoxides that bear alkyl or electron-withdrawing groups, owing to the combination of steric and electronic factors, the attack occurs at the less hindered site of the ring and 1-halo-2 alcohols are produced as the major product (Table 1).

We also investigated the possibility of converting epoxides to symmetrical vicinal dihalides using PPh_3–NXS or PPh_3–NXSac (X = Cl, Br). For this purpose, oxiranylmethyl phenyl ether (Table 2, entries 1 and 2) was treated as a model compound with the PPh_3–NBS or PPh_3–NBSac reagent system using the optimized molar ratio of 2.5:2.5 in refluxing acetonitrile, and the corresponding dibromide was obtained in high yield. The use of NCS or NCSac in this method under the same reaction conditions as used for the preparation of dibromide offers the possibility of producing *vic*-dichlorides. We extended this procedure to other epoxides to obtain symmetrical *vic*-dihalides. The results of this investigation are shown in Table 2.

However, the preparation of *vic*-diiodides by this method was not successful. This result is in accordance with the literature (30), which indicates that alkene formation is favoured owing to the ease of elimination of molecular iodine. In all the reactions of epoxides with PPh_3–NIS or PPh_3–NISac (2.5:2.5), alkene formation was the only pathway. For example, the addition of oxiranylmethyl phenyl ether to the mixture of PPh_3–NBS or PPh_3–NBSac (2.5:2.5) in refluxing acetonitrile produced allyl phenyl ether as the only product after 0.5 h. Therefore, the use of a PPh_3–NIS or PPh_3–NISac mixture offers a new mixed reagent system for the deoxygenation of epoxides into their corresponding alkenes.

To overcome the problems of using highly acidic conditions for the conversion of epoxides to *vic*-dihalides (27), and to introduce a mild and novel method for this transformation, we applied our method to epoxides. For this transformation, the successive use of two *N*-halo imides having different halide ions produces the desired unsymmetrical *vic*-dihalide in high yield. To optimize the reaction conditions, oxiranylmethyl phenyl ether was added to a mixture of PPh_3–NCS or PPh_3–NCSac with the molar ratio of 1.2:1.2 in acetonitrile at room temperature. When all the epoxide was consumed, the mixture was poured into another mixture containing PPh_3–NBS or PPh_3–NBSac (2.5:2.5) in acetonitrile and refluxed for 8 h. After completion of the reaction, 2-bromo-1-chloro-3-phenoxypropane was obtained in 85% yield (Table 3, entry 2). Since this attempt was successful, we applied it to the preparation of unsymmetrical *vic*-dihalides from other epoxides. By using the proper halide anion in each step, we could easily control the formation of *vic*-dihalides with high regioselectivity. The results obtained for this study are tabulated in Table 3. In these reactions, as we expected, in the first step, the first halide ion attacks the epoxide ring from the same side of the formation of *vic*-halo alcohols. In the second step, the resulting halo alcohol reacts with the mixture of PPh_3–NXS or PPh_3–NXSac and the second halide anion displaces the hydroxyl group to produce the desired unsymmetrical dihalide. It was observed that when one of the nucleophiles was iodide, because of the possibility of the elimination of IX (X = Cl, Br), alkene formation was a competing pathway and lowered the yield of *vic*-dihalides considerably (Table 3, entries 3 and 6).

Although the exact mechanism of these reactions is not clear, it is suggested that, at first, a positively charged adduct (**I**) (31) is formed from the reaction of PPh_3 with NBS (Scheme 2). This positively charged adduct interacts with the oxygen atom of the epoxide and produces (**II**). When *R*-(–)-styrene oxide, the formation of the corresponding β-bromohydrin, which did not show any optical rotation, accounts for the formation of the racemic product through the intermediacy of a benzylic carbocation. Nucleophile attack of the halide ion on (**II**) produces the intermediate (**III**). This intermediate in the presence of water could produce triphenylphosphine oxide, succinimide, and the corresponding *vic*-halo alcohol. Similarly, the attack of another halide ion on this intermediate produces the corresponding vicinal symmetrical or unsymmetrical dihalide.

As shown in Table 3, entries 1 and 4, when the first nucleophile used was bromide, a mixture of two regioisomers was obtained. It is assumed that, in these cases,

Table 1. Conversion of epoxides to *vic*-halo alcohols with PPh_3–NXS or PPh_3–NXSac (X = Br, C1, I) in 1% aqueous acetonitrile at room temperature.

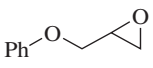
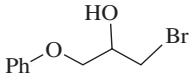
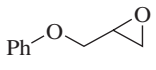
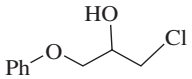
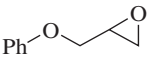
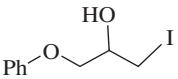
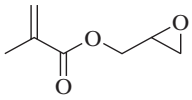
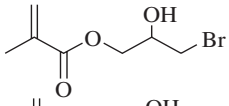
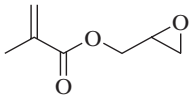
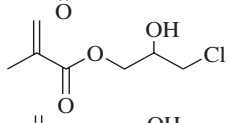
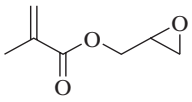
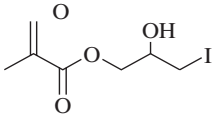
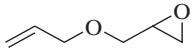
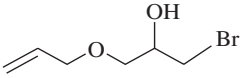
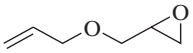
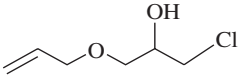
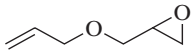
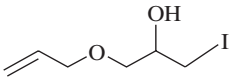
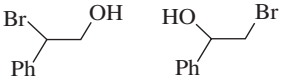
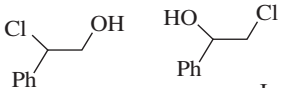
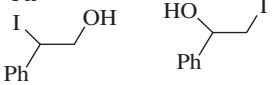
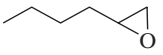
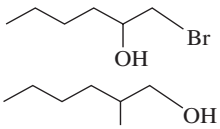
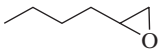
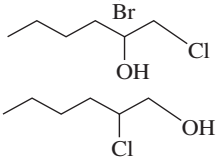
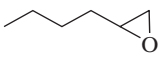
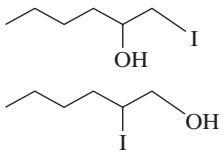
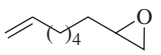
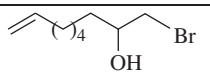
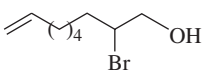
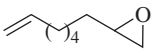
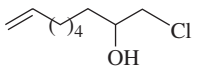
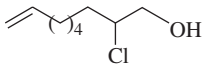
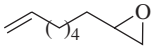
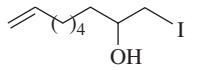
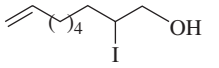
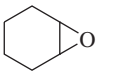
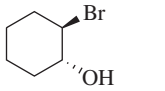
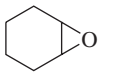
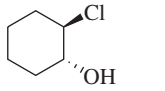
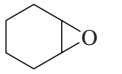
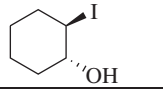
Entry	Substrate	Product [a]		Time (min)	Yield (%)[b]
1			NBS	40	93
			NBSac	8	95
2			NCS	6	88
			NCSac	15	92
3			NIS	5	90
			NISac	7	85
4			NBS	20	82
			NBSac	3	90
5			NCS	3	95
			NCSac	8	85
6			NIS	5	80
			NISac	3	86
7			NBS	3	96
			NBSac	3	85
8			NCS	10	82
			NCSac	6	93
9			NIS	3	90
			NISac	10	88
10			NBS	30	80(16)[c]
			NBSac	3	90(8)[c]
11			NCS	3	78(18)[c]
			NCSac	5	85(8)[c]
12			NIS	8	80(15)[c]
			NISac	5	78(10)[c]
13			NBS	3	65(15)[c]
			NBSac	3	70(16)[c]
14			NCS	8	75(20)[c]
			NCSac	3	66(20)[c]
15			NIS	3	65(15)[c]
			NISac	10	76(15)[c]

Table 1 *(concluded)*.

Entry	Substrate	Product [a]		Time (min)	Yield (%)[b]
16			NBS	3	69(15)[c]
			NBSac	5	75(20)[c]
17			NCS	3	70(18)[c]
			NCSac	10	75(10)[c]
18			NIS	5	65(25)[c]
			NISac	10	70(20)[c]
19			NBS	10	95
			NBSac	8	90
20			NCS	3	87
			NCSac	10	92
21			NIS	3	90
			NISac	5	90

[a]All the products are known compounds (16, 20, 21, 24, 28) and are identified by their physical or spectral data.
[b]Isolated yield.
[c]Yield % in parentheses shows the formation of the other regioisomer.

the intermediate (**a**) can release $OPPh_3$ through the anchimeric assistance of bromide to form the bromonium ion (**b**). An attack of the second nucleophile to either side of (**b**) can produce a mixture of two regioisomers (Scheme 3).

In summary, the present investigation has demonstrated that the use of a PPh_3–NXS or PPh_3–NXSac system offers a simple, novel, and convenient method for the selective conversion of epoxides to either their corresponding *vic*-halo alcohols or symmetrical or unsymmetrical *vic*-dihalides.[3]

Experimental section

All the solvents and reagents were purchased from Fluka (Switzerland) or Merck (Germany) Chemical Companies. The products were purified by column or thick layer chromatography techniques. FT-IR spectra were recorded on a Shimadzu DR-8001 spectrometer. NMR spectra were recorded on a Brucker Avance DPX 250 MHz instrument. Mass spectra were recorded on a Shimadzu GC–MS-QP 1000PX.

Typical procedure for the conversion of oxiranylmethyl phenyl ether to 1-bromo-3-phenoxy-2-propanol by PPh_3–NBS

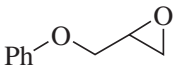
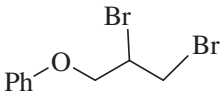
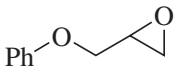
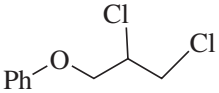
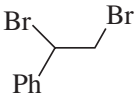
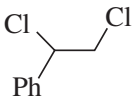
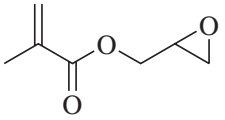
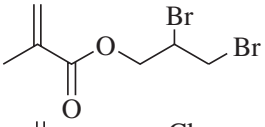
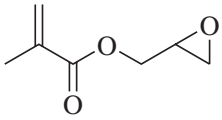
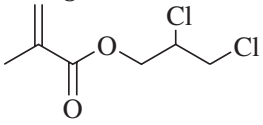
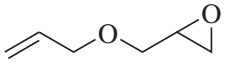
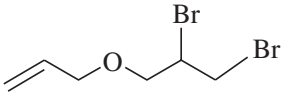
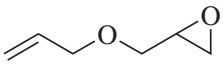
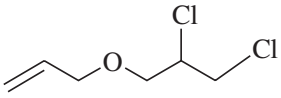
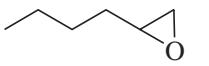
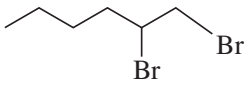
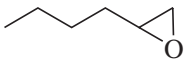
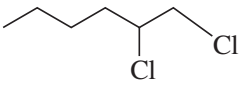
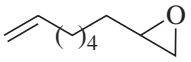
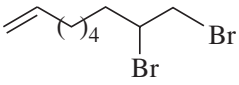
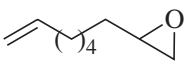
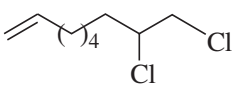
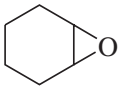
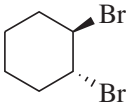
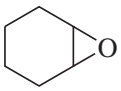
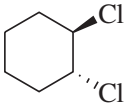
Oxiranylmethyl phenyl ether (0.15 g, 1 mmol) was added to a flask containing PPh_3 (0.314 g, 1.2 mmol) and NBS (0.213 g, 1.2 mmol) in 1% aq. CH_3CN (5 mL) at room temperature. TLC monitoring showed the completion of the reaction after 40 min. (This reaction with 1.2 mmol of NBSac takes 8 min, Table 1, entry 1.) After evaporation of the solvent, column chromatography of the crude mixture on silica gel using *n*-hexane – ethyl acetate afforded 1-bromo-3-phenoxy-2-propanol as a yellow oil in 93% yield. ^{1}H NMR ($CDCl_3$, 250 MHz, ppm) δ: 2.6 (d, 1H, J = 5 Hz), 3.6–3.7 (m, 2H), 4.07–4.10 (m, 2H), 4.15–4.25 (m, 1H), 6.90–7.10 (m, 3H), 7.3–7.6 (m, 1H). ^{13}C NMR ($CDCl_3$, 62.9 MHz, ppm) δ: 35.0, 69.5, 70.3, 114.5, 121.4, 129.6, 158.1. Mass spectra *m/e* (%): 232 ([M + 2], 33.1), 230 ([M], 35.6), 151 ([M – Br], 20.3).

Typical procedure for the conversion of oxiranylmethyl phenyl ether to 1,2-dibromo-3-phenoxy propane by PPh_3–NBS

PPh_3 (0.654 g, 2.5 mmol) was added to a flask containing NBS (0.443 g, 2.5 mmol) in refluxing acetonitrile. Then oxiranylmethyl phenyl ether (0.15 g, 1 mmol) was added. TLC or GC analysis showed the completion of the conversion of oxiranylmethyl phenyl ether to 1,2-dibromo-3-phenoxypropane after 3 h. After completion of the reaction, the solvent was evaporated. 1,2-Dibromo-3-phenoxy propane was obtained in 88% yield after column chromatography of the crude mixture using *n*-hexane – ethyl acetate (5:1) as eluent. ^{1}H NMR ($CDCl_3$, 250 MHz, ppm) δ: 3.8–3.9 (m,

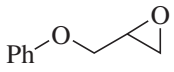
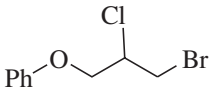
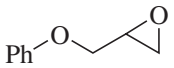
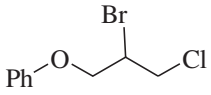
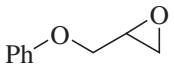
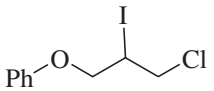
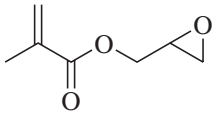
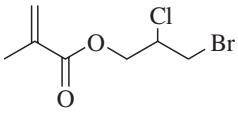
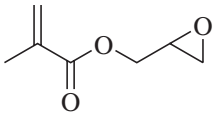
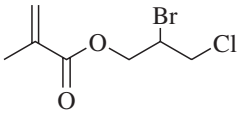
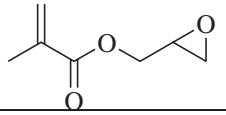
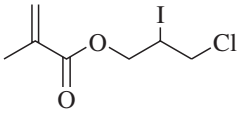
[3] Supplementary data for this article are available on the journal Web site (http://canjchem.nrc.ca) or may be purchased from the Depository of Unpublished Data, Document Delivery, CISTI, National Research Council Canada, Building M-55, 1200 Montreal Road, Ottawa, ON K1A 0R6, Canada. DUD 4096. For more information on obtaining material refer to http://cisti-icist.nrc-cnrc.gc.ca/irm/unpub_e.shtml.

Table 2. Conversion of epoxides to *vic*-dihalides with PPh_3–NXS or PPh_3–NXSac (X = Br, C1) in acetonitrile under reflux conditions.

Entry	Substrate	Product[a]	Time (h)	Yield (%)[b]
1			3.5	88
2			3	90
3			3	80
4			2	87
5			3.5	89
6			2	79
7			5.5	82
8			5	75
9			5	80
10			4	78
11			3	80
12			2	85
13			4.5	83[c]
14			4	88

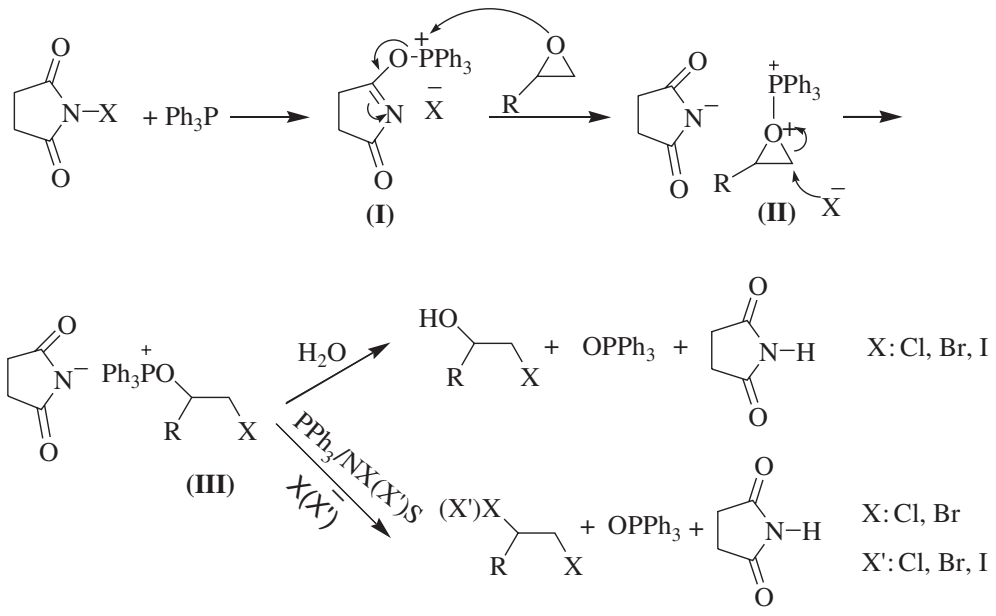
[a]All the products are known compounds (28) and are identified by their physical or spectral data.
[b]Isolated yield.
[c]The stereochemistry of product was determined by the comparison of its ^{1}H NMR spectrum with the literature (32).

Table 3. Conversion of epoxides to unsymmetrical *vic*-dihalides with PPh_3–NXS of different halides.

Entry	Substrate	Product[a]	Time (h)[b]	Yield (%)[c]
1			5	65[d]
2			8	85
3			8.5	52
4			7	68[d]
5			9	90
6			10	44

[a]All the products are known compounds (28) and are identified by their physical or spectral data.
[b]These reactions were performed in the presence of PPh_3–NXSac with similar results.
[c]Isolated yield.
[d]The other isomeric product was also formed in 25% yield.

Scheme 2.

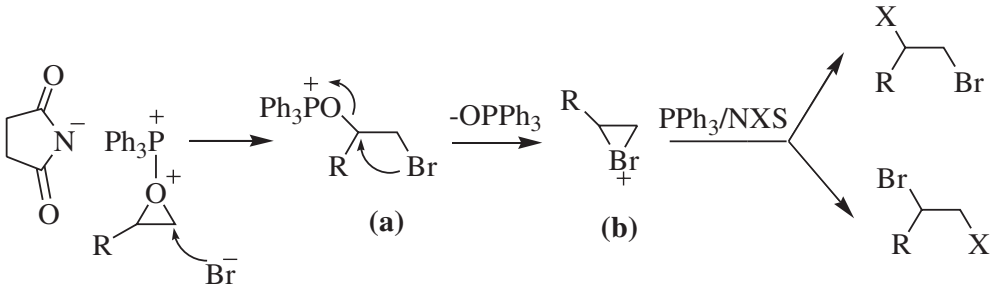


2H), 4.3–4.4 (m, 3H), 6.9–7.0 (m, 3H), 7.2–7.32 (m, 2H). ^{13}C NMR ($CDCl_3$, 62.9 MHz, ppm) δ: 31.7, 46.7, 68.0, 113.8, 120.6, 128.6, 156.9. Mass spectra *m/e* (%): 296 ([M + 4], 16.2), 294 ([M + 2], 41.7), 292 ([M], 14.1), 203 ([M + 4 – PhO], 3.2), 201 ([M + 2 – PhO], 8.7), 199 ([M – PhO], 5.3).

Typical procedure for the conversion of oxiranylmethyl phenyl ether to 2-bromo-1-chloro-3-phenoxypropane

PPh_3 (0.314 g, 1.2 mmol) was added to a flask containing of NCS (0.159 g, 1.2 mmol) in CH_3CN at room temperature. Then oxiranylmethyl phenyl ether (0.15 g, 1 mmol) was

Scheme 3.



gradually added to the reaction mixture over a period of 1.5 h. TLC or GC analysis showed the completion of the conversion of oxiranylmethyl phenyl ether to 1-chloro-3-phenoxy-2-propanol. Then the reaction mixture was transferred to another flask containing a solution of PPh_3 (0.654 g, 2.5 mmol) and NBS (0.443 g, 2.5 mmol) in CH_3CN (5 mL) and refluxed for 8 h to produce 2-bromo-1-chloro-3-phenoxypropane. After completion of the reaction, the solvent was evaporated. 2-Bromo-1-chloro-3-phenoxypropane was obtained in 85% yield after column chromatography of the crude mixture using *n*-hexane – ethyl acetate (5:1) as eluent. ^{1}H NMR ($CDCl_3$, 250 MHz, ppm) δ: 3.9–4.0 (m, 2H), 4.25–4.4 (m, 3H), 6.8–7.1 (m, 3H), 7.2–7.3 (m, 2H). ^{13}C NMR ($CDCl_3$, 62.9 MHz, ppm) δ: 45.0, 48.1, 68.3, 114.8, 121.6, 129.6, 157.9. Mass spectra *m/e* (%): 252 ([M + 4], 8.7), 250 ([M + 2], 29.3), 248 ([M], 24), 171 ([M + 2 – ^{79}Br] and [M + 4 – ^{81}Br], 7.2), 169 ([M + 2 – ^{81}Br] and [M – ^{79}Br], 12.9).

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