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Aryl-Substituted Sulfonium Betaines: Preparation and Use in the Epoxidation of Aldehydes

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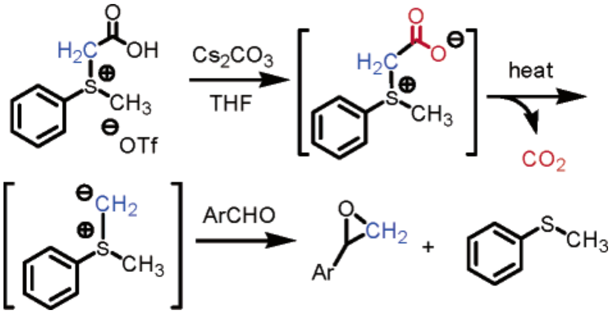
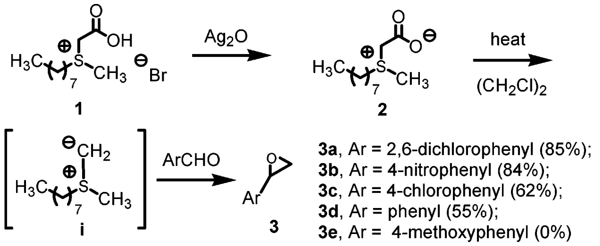


Thermally induced decarboxylation of carboxymethylsulfonium betaines results in formation of the corresponding sulfur ylides in situ. Decarboxylation rates for a range of arylcarboxymethylsulfonium betaine salts have been determined using NMR spectroscopy, and the efficiency of ylide generation and trapping has been evaluated via methylidene transfer to a range of aldehydes to form epoxides.

Sulfur ylides as nucleophilic alkylidene transfer agents represent an extremely useful tool for the introduction of functionality into organic molecules.[1] Sulfur ylides react with a large variety of π-acceptors, such as aldehydes, ketones, aziridines, and electron-deficient olefins, resulting in the direct assembly of small ring carbo- and heterocyclic building blocks, such as epoxides, aziridines, and cyclopropanes.[2,3] In our search for alternative mild protocols for sulfur ylide generation, which might be capable of sustaining catalytic methylidene transfers, we were intrigued by the possibility of the decarboxylation of carboxymethyl-substituted sulfonium salts. Earlier we reported on a novel protocol for the preparation of oxiranes using simple carboxymethylsulfonium betaines[4] (Scheme 1).

SCHEME 1



The key reagent **2** was prepared from the corresponding sulfonium bromide **1** using the procedure of Ratts and Yao.[5] This particular salt was the third of a series of carboxymethylsulfonium betaines.[4] Earlier generation betaines based on dimethyl sulfide and tetrahydrothiophene were rejected on the grounds of poor solubility in most organic solvents. Using betaine **2**, the highest levels of conversion were observed when using electron-deficient aryl aldehydes. Lower levels of conversion were observed with electron-rich or neutral systems. This is a generally observed trend seen for similar reactions carried out using the more conventional S-ylide protocols. However, the disappointingly low yields seen in our system with the less electrophilic (electron-rich) aldehydes are largely attributed to the instability of the S-ylide under the prevailing reaction conditions (temperatures ~60 °C), where side reactions predominate in the absence of an efficient ylide quencher. This is consistent with the fact that unstabilized sulfur ylides are known to have short half-lives in solution,[1a,6] and that high yields of oxirane can be obtained from less reactive carbonyl acceptors (e.g., benzophenone) using S-ylide protocols operating at or close to room temperature.[7] Therefore, if we could identify more facile betaine systems that decarboxylate at lower temperatures, which yield more stable S-ylides, this should increase the intrinsic ylide half-life and provide for cleaner and more selective epoxidation reactions.

We now wish to report on a set of next generation betaine reagents that have been designed to both probe the kinetics of the decarboxylation process and mediate more efficient and cleaner epoxidation conditions.

In the current work, we have sought to improve on the earlier generation compounds by synthesizing inherently less stable betaines that decarboxylate under milder conditions. One approach was to alter the substituents bound to the sulfonium center in order to facilitate ylide formation. Switching from octyl to aryl functionality added stability of the sulfur ylide and a more facile rate of decarboxylation was expected.[8] This, in turn, would hopefully translate to more efficient methylidene transfer in the trapping of carbonyl compounds. Our approach to the preparation of a fourth generation sulfonium betaine salt **4** is presented in Scheme 2.

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(6) Trost, B. M.; Bogdanowicz, M. J. *J. Am. Chem. Soc.* **1973**, *95*, 5298.

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10.1021/jo061370u CCC: $33.50

SCHEME 2

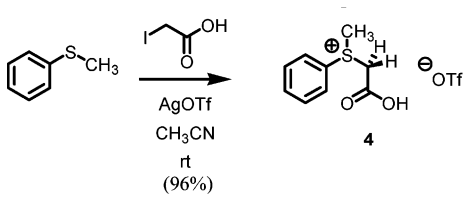
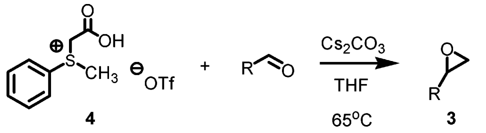
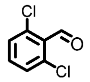
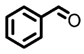
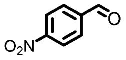
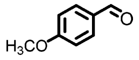
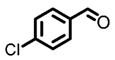
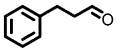


TABLE 1. Carboxymethylsulfonium Betaine 4 Promoted Oxirane Formation



entry	carbonyl derivative	**3**, %[a]	entry	carbonyl derivative	**3**, %[a]
1, **a**		94	4, **d**		77
2, **b**		98	5, **e**		25
3, **c**		75	6		48

[a] Isolated yields of distilled homogeneous spectroscopically pure products are reported.

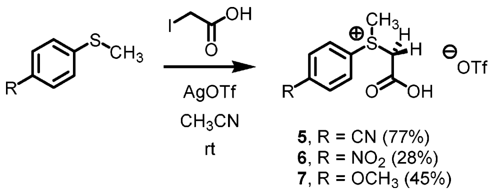
Starting from commercially available thioanisole, sulfonium salt **4** was prepared in one step using iodoacetic acid and silver triflate in acetonitrile. Following filtration of silver iodide, the desired compound could be precipitated either by addition of MTBE to give phenylmethylcarboxymethylsulfonium triflate as a white crystalline solid in 73% yield or by using as slightly modified procedure (Experimental Section), triturated at room temperature using MTBE (96% yield).

For comparative purposes, we chose to test our new betaine salt **4** against the same group of carbonyl derivatives used in our initial studies on the octylmethyl betaine.[4] These ranged from electron-deficient aryl aldehydes to less reactive ketone substrates. Attempts to isolate the carboxymethylsulfonium betaine from sulfonium salt **4** were unsuccessful. We therefore elected to use the sulfonium salt and generate the sulfonium betaine in situ using cesium carbonate as base. Except for this single modification, all other reaction conditions were kept the same. A total of six carbonyl derivatives were surveyed. The data from the study are presented in Table 1.

Table 1 reveals some interesting trends. All reactions reported here and in our initial communication were clean as judged by GC and NMR analysis of the crude reaction mixture. That is, although incomplete reactions were observed, analysis of the crude reaction mixtures revealed no byproducts other than desired product and unreacted starting material. In line with our earlier report, electron-deficient aryl aldehydes (entries 1−3) gave the best yields with the same downward trend toward the more electron-rich substrates (entries 4−6).

A comparison of the two protocols reveals several additional interesting trends. An improvement in overall conversion of aldehyde functionality to oxirane was observed in every case. While moderate improvements were observed for 2,6-dichlorobenzaldehyde (9%, entry 1), 4-nitrobenzaldehyde (15%, entry 2), and 4-chlorobenzaldehyde (13%, entry 3) compared with betaine **2**, significant improvements were observed for benzaldehyde (22%, entry 4) and 4-methoxybenzaldehyde (25%, entry 6). Reaction of 4-methoxybenzaldehyde to yield the desired oxirane in 25% yield is a significant improvement compared with the third generation betaine **2**, which gave no detectable yield of product.

SCHEME 3



5, R = CN (77%)
6, R = NO_2 (28%)
7, R = OCH_3 (45%)

Encouraged by the results using sulfonium salt **4**, we prepared and tested three additional systems (Scheme 3). Multigram quantities of sulfonium salts **5**−**7** were prepared via the exact protocol employed earlier for the preparation of compound **4** and involved treatment of commercially available thioanisole derivatives with acetonitrile solutions of iodoacetic acid and silver triflate.

We had initially reasoned that by changing the electronic environment of the sulfonium center a beneficial change in the overall kinetics of decarboxylation, ylide generation, and trapping would be achieved. For this study, we elected to use as trapping agent, both electron-rich 4-methoxybenzaldehyde and electron-deficient 2,6-dichlorobenzaldehyde. To our surprise, we found that all three sulfonium salts were ineffective at improving the levels of conversion from aldehyde to oxirane when compared to the results obtained with sulfonium salt **4**. Varying the amount of sulfonium salt, reaction times, rates of addition, and temperature all met with limited success.

Our study did, however, provide valuable kinetic information on the rate of decarboxylation of the sulfonium salts themselves.[9] Using NMR and focusing on the disappearance of the methylene protons ($R_2S^+CH_2COOH$) as a function of time, we obtained half-lives for sulfonium betaines **5**−**7**. The integration of the methylene peak was referenced to a solution of hexamethyldisilazane in CCl_4 contained within a sealed glass tube, as internal standard. The data representing the disappearance of the methylene signal were obtained on timed intervals ranging from every minute to every five minutes. The plots of the natural log of the concentration as seen over time provided us with the half-lives for each system. Results for this study are presented in Table 2.

S-Ylide epoxidation protocols found in the literature typically operate near or below room temperature. The results for our decarboxylation approach (Table 1) show similar conversions while operating at much warmer reaction conditions. The half-lives reported in Table 2 show that adjusting the electronic environment about the sulfur atom has a significant effect on the rate of decarboxylation of the betaine. Use of strong electron-deficient groups resulted in half-lives for sulfonium salts **5** and **6** in less than 1.5 min (entries 2 and 3). When switching to an electron-rich aryl substituent at position 4, the half-life of sulfonium salt (**7**) exceeded 1 h (entry 4). While higher rates of decarboxylation could be achieved compared with those of betaine salt **4**, use of modified sulfonium salts **5**−**7** offered no

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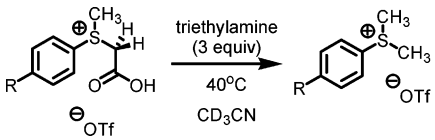
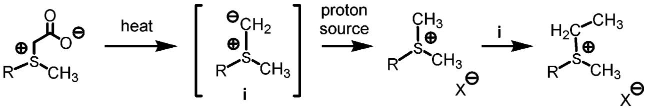
[9] For a detailed account of the decarboxylation study, please refer to the Supporting Information.

TABLE 2. Half-Lives in Decarboxylation Rates of Sulfonium Salts 4−7



entry	sulfonium salt	$t_{1/2}$ (min)
1	**4** (R = H)	13.3
2	**5** (R = CN)	1.3
3	**6** (R = NO_2)	1.2
4	**7** (R = OCH_3)	85.6

SCHEME 4



improvement in epoxidation efficiency. In fact, we were unable to detect more than traces of epoxide from reaction of either **5** or **6** with even the electron-deficient aldehydes. It is interesting that reaction of sulfonium salt **4** with 2,6-dichlorobenzaldehyde yielded the desired oxirane in only 12% when operating at room temperature. Warming to 65 °C, the level of conversion increased to 94%. To explain the above results, we propose that, at high temperatures, the decarboxylation rate is high providing relatively high levels of ylide that can be trapped by carbonyl compounds, particularly, the more electrophilic (electron-deficient) ones. As the electrophilicity of the carbonyl compound decreases, the forward rate of epoxidation slows down and fails to compete effectively with ylide fragmentation and other side reactions. Conversely, when the rate of decarboxylation is increased through electronic manipulation (aryl group substituents), the rate of epoxidation also slows down as the ylide (now more stable) is less reactive.

In terms of possible side reactions, unstabilized sulfur ylides are known to react with sulfonium ions (powerful methylating agents) to form new sulfonium salts.[10] One likely side reaction in our system would be initial protonation of sulfur ylide **i** to form a dimethylsulfonium species which, in turn, alkylates another ylide molecule to form mixtures of dimethyl or ethyl methyl sulfonium derivatives (Scheme 4).[11]

Research in our group continues in the areas of (1) understanding the kinetics of the reaction, (2) increasing the scope of substrate using modified sulfonium salts, and (3) the preparation and use of a chiral, nonracemic sulfide promoter. The results from these studies will be reported in due course.

In conclusion, methylidene transfer to aldehyde functionality using decarboxylation of arylcarboxymethylsulfonium betaines has been achieved. An improvement in the levels of epoxide conversion and isolated yield was observed upon replacement of the octyl substituent on the sulfur atom with phenyl. The levels were significant especially when working with electron-rich or neutral aryl aldehydes. Electronic tuning of the sulfonium center did not offer an improvement in the levels of epoxide conversion despite a significant acceleration in the rates of decarboxylation.

Experimental Section

Representative Procedure for the Preparation of Sulfonium Salts. A 250 mL round-bottomed flask (RBF) equipped with a stir bar, septum, and drying tube was charged with 5.6 g of iodoacetic acid (30.0 mmol, 1.5 equiv). The solid was dissolved using acetonitrile (30 mL). Once in solution, 22.1 mmol of sulfide (1.0 equiv) was added in one portion at room temperature. In a separate flask, 7.7 g of silver triflate (30 mmol, 1.5 equiv) was dissolved in 10.0 mL of acetonitrile. The silver triflate solution was added dropwise at room temperature via syringe to the solution of sulfide and iodoacetic acid. Precipitation was observed immediately. The suspension was monitored by TLC until completion of reaction, approximately 48 h. The silver salts were removed via filtration using a medium-sized glass funnel and filter paper (#2 Whatman). The solution was concentrated in vacuo without external heat. The resulting solid was triturated with MTBE (100 mL), filtered, and dried under vacuum. Precipitation of the sulfonium salts could as well be affected by addition of an ethereal solvent to the crude reaction mixture.[12]

(Carboxymethyl)methylphenylsulfonium Trifluoromethanesulfonate (4). From the combination of thioanisole (5.23 mL, 44.5 mmol), iodoacetic acid (5.5 g, 29.6 mmol) in 4.0 mL of acetonitrile, and silver triflate (8.0 g, 31.1 mmol) in 5.0 mL of acetonitrile, 7.2 g of (carboxymethyl)methylphenylsulfonium trifluoromethanesulfonate (22.2 mmol, 96% yield) was obtained after precipitation and isolation of solid by filtration. Formula weight: 332.32 g/mol. Mp: 124−125 °C. ^1H NMR (300 MHz, D_2O): δ 7.92−7.65 (m, 5H), 4.79 (br s), 4.64 (d, J = 16.0 Hz, 2H), 4.55 (d, J = 10.7 Hz, 2H), 3.23 (s, 3H). ^{13}C NMR (75.5 MHz, D_2O): δ 169.8, 137.3, 133.6, 132.6, 125.9, 122.2 (q, J = 317.0 Hz (CF_3)), 51.8 (CD_2COOD), 28.8. IR (neat (ATR corrected)): 3039 (m), 2991 (m), 2941 (m), 1732 (s) cm^{-1}. GC/MS (70 eV, LREI, HP-5): M^+ (−OTf, −carboxymethyl) 124, (t_R 5.49 min). GC (AT-5): t_R 5.27 min (thioanisole). HRMS (M^+ − OTf) calcd 183.04810; found 183.0474.

Carboxymethyl-4-cyanophenylmethylsulfonium Trifluoromethanesulfonate (5). From the combination of 4-cyanothioanisole (6.02 g, 40.3 mmol), iodoacetic acid (5.0 g, 26.9 mmol) in 2.5 mL of acetonitrile, and silver triflate (7.25 g, 28.2 mmol) in 5.0 mL of acetonitrile, 7.4 g of carboxymethyl-4-cyanophenylmethylsulfonium trifluoromethanesulfonate (20.7 mmol, 77% yield) was obtained after precipitation and isolation of solid by filtration. M_w: 357.33 g/mol. Mp: 136−138 °C. ^1H NMR (300 MHz, D_2O): δ 8.09−8.04 (m, 4H), 4.79 (br s), 4.68−4.46 (m, 2H), 3.25 (s, 3H). ^{13}C NMR (75.5 MHz, D_2O): δ 167.1, 134.7, 131.0, 130.2, 129.6, 126.1, 119.8 (q, J = 317.0 Hz (CF_3)), 117.7, 117.3, 49.6 (CD_2COOD), 28.3, 26.0. IR (neat (ATR corrected)): 2240 (m), 2953 (m), 3043 (m), 3093 (m) cm^{-1}. GC/MS (70 eV, LREI, HP-5): M^+ (−OTf, −carboxymethyl) 149, (t_R 10.51 min). GC (AT-5): t_R 9.69 min ((4-cyanophenyl)methyl sulfide). HRMS (M^+ − OTf) calcd 208.0432; found 208.0441.

Carboxymethyl-4-nitrophenylmethylsulfonium Trifluoromethanesulfonate (6). From the combination of methyl-4-nitrophenyl sulfide (6.82 g, 40.3 mmol), iodoacetic acid (5.0 g, 26.9 mmol) in 2.5 mL of acetonitrile, and silver triflate (7.25 g, 28.2 mmol) in 5.0 mL of acetonitrile, 4.2 g of carboxymethyl-4-nitrophenylmethylsulfonium trifluoromethanesulfonate (11.1 mmol, 28% yield) was obtained after precipitation and isolation of solid by

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(10) Corey, E. J.; Jautelat, M.; Oppolzer, W. *Tetrahedron Lett.* **1967**, 2325.

(11) (a) For formation of alkyldimethylsulfonium salts via the decarboxylation of carboxymethylbetaine functionality, see: Burness, D. *J. Org. Chem.* **1959**, *24*, 849. (b) For a discussion on ylide methylation, see: Gololobov, Y. G.; Nesmeyanov, A. N.; Lysenko, V. P.; Boldeskul, I. E. *Tetrahedron* **1987**, *43*, 2609

(12) Attempts to force precipitation of additional sulfonium salt from the filtrate a second time resulted in impure mixtures of the desired sulfonium salt. Multigram quantities of each system were obtained with the first filtration. Recrystallization of the impure salts requiring elevated temperatures was not considered.

filtration. M_w: 377.31 g/mol. Mp: 105−109 °C. ^{1}H NMR (300 MHz, D_2O): δ 8.48 (d, $J = 9.1$ Hz, 2H), 8.19 (d, $J = 9.4$ Hz, 2H), 4.79 (br s), 4.65 (d, $J = 15.7$ Hz, 1H), 4.54 (d, $J = 15.7$ Hz, 1H), 3.28 (s, 3H). ^{13}C NMR (75.5 MHz, D_2O): δ 167.4, 151.1, 131.8, 131.4, 125.8, 119.8 (q, $J = 317.0$ Hz (CF_3)), 50.3 (CD_2COOD), 28.4, 26.2. IR (neat (ATR corrected)): 1753 (s), 2928 (m), 2949 (m), 2978 (m), 3007 (m), 3038 (m), 3106 (m) cm^{-1}. GC/MS (70 eV, LREI, HP-5): M^+ (−OTf, −carboxymethyl) 169, (t_R 12.08 min). GC (AT-5): t_R 11.20 min (methyl(4-nitrophenyl)sulfide (major)). HRMS (M^+ − OTf) calcd 228.0331; found 228.0323.

Carboxymethyl-4-methoxyphenylmethylsulfonium Trifluoromethanesulfonate (7). From the combination of 4-methoxythioanisole (5.6 mL, 40.3 mmol), iodoacetic acid (5.0 g, 26.9 mmol) in 2.5 mL of acetonitrile, and silver triflate (7.25 g, 28.2 mmol) in 5.0 mL of acetonitrile, 4.4 g of carboxymethyl-4-methoxyphenylmethylsulfonium trifluoromethanesulfonate (12.1 mmol, 45% yield) was obtained after precipitation and isolation of solid by filtration. M_w: 362.34 g/mol. Mp: 110−112 °C. ^{1}H NMR (300 MHz, D_2O): δ 7.83 (d, $J = 9.1$ Hz, 2H), 7.14 (d, $J = 9.1$ Hz, 2H), 4.79 (br s), 3.82 (s, 3H), 3.19 (s, 3H). ^{13}C NMR (75.5 MHz, D_2O): δ 167.3, 164.2, 132.4, 131.6, 119.8 (q, $J = 317.0$ Hz (CF_3)), 116.7, 113.5, 113.1, 56.0, 49.6 (CD_2COOD), 29.1, 26.9. IR (neat (ATR corrected)): 1731 (s), 2903 (m), 2938 (m), 2992 (m) cm^{-1}. GC/MS (70 eV, LREI, HP-5): M^+ (−OTf, −carboxymethyl) 154 (t_R 8.68 min). GC (AT-5): t_R 8.27 min ((4-methoxyphenyl)methyl sulfide (major)). HRMS (M^+ − OTf) calcd 213.0585; found 213.0579.

Representative Procedure for Methylidene Transfer Reactions. An oven-dried round-bottomed flask was equipped with a stir bar, septum, and drying tube. To this system were added aldehyde (1.0 equiv), Cs_2CO_3 (2.0 equiv), and THF (5.0 mL). The system was externally heated to ~65 °C (sand bath) at which time a solution of sulfonium salt (2.0 equiv) in THF (5.0 mL) was added via syringe pump over a period of 5.0 h. The warm reaction mixture was allowed to stir for an additional period of 2.0 h at which time the system was cooled to room temperature. The crude reaction mixture was filtered through a Celite column (5 mm × 20 mm) and analyzed by GC and if necessary GC/MS. The crude reaction mixture was next concentrated in vacuo and immediately purified by Kugelrohr distillation to afford pure carbocycle. The resulting distillate was again analyzed by GC, GC/MS, and ^{1}H NMR to determine the isolated yield.

2,6-Dichlorophenyloxirane (3a). From the combination of 2,6-dichlorobenzaldehyde (65.8 mg, 0.39 mmol), cesium carbonate (245 mg, 0.75 mmol), and (carboxymethyl)methylphenylsulfonium trifluoromethanesulfonate (250 mg, 0.75 mmol), a 94% conversion of aldehyde to oxirane was observed after purification by Kugelrohr distillation via GC analysis. Spectral data are in agreement with those previously reported.[13] GC/MS (70 eV, LREI, HP-5): M^+ 188 (t_R 9.98 min). GC (AT-5): t_R 9.40 min.

4-Nitrophenyloxirane (3b). From the combination of 4-nitrobenzaldehyde (56.8 mg, 0.38 mmol), cesium carbonate (245 mg, 0.75 mmol), and (carboxymethyl)methylphenylsulfonium trifluoromethanesulfonate (250 mg, 0.75 mmol), a 98% conversion of aldehyde to oxirane was observed after purification by Kugelrohr distillation via GC analysis. Spectral data are in agreement with those previously reported.[13] GC/MS (70 eV, LREI, HP-5): M^+ 165 (t_R 12.32 min). GC (AT-5): t_R 10.50 min.

4-Chlorophenyloxirane (3c). From the combination of 4-chlorobenzaldehyde (52.9 mg, 0.38 mmol), cesium carbonate (245 mg, 0.75 mmol), and (carboxymethyl)methylphenylsulfonium trifluoromethanesulfonate (250 mg, 0.75 mmol), a 75% conversion of aldehyde to oxirane was observed after purification by Kugelrohr distillation via GC analysis. Spectral data are in agreement with those previously reported.[13] GC/MS (70 eV, LREI, HP-5): M^+ 153 (t_R 8.75 min). GC (AT-5): t_R 7.33 min

Phenyloxirane (3d). From the combination of benzaldehyde (38.2 μL, 0.38 mmol), cesium carbonate (245 mg, 0.75 mmol), and (carboxymethyl)methylphenylsulfonium trifluoromethanesulfonate (250 mg, 0.75 mmol), a 77% conversion of aldehyde to oxirane was observed after purification by Kugelrohr distillation via GC analysis. Spectral data are in agreement with those previously reported.[13] GC/MS (70 eV, LREI, HP-5): M^+ 121 (t_R 9.349 min). GC (AT-5): t_R 5.24 min.

4-Methoxyphenyloxirane (3e). From the combination of 4-methoxybenzaldehyde (45.77 μL, 0.38 mmol), cesium carbonate (245 mg, 0.75 mmol), and (carboxymethyl)methylphenylsulfonium trifluoromethanesulfonate (250 mg, 0.75 mmol), a 25% conversion of aldehyde to oxirane was observed after purification by Kugelrohr distillation via GC analysis. Spectral data are in agreement with those previously reported.[13] GC/MS (70 eV, LREI, HP-5): M^+ 150 (t_R 9.592 min). GC (AT-5): t_R 7.86 min.

4-Phenyl-1,2-epoxybutane (3f). From the combination of hydrocinnamaldehyde (49.5 μL, 0.38 mmol), cesium carbonate (245 mg, 0.75 mmol), and (carboxymethyl)methylphenylsulfonium trifluoromethanesulfonate (250 mg, 0.75 mmol), a 48% conversion of aldehyde to oxirane was observed after purification by Kugelrohr distillation via GC analysis. Spectral data are in agreement with those previously reported.[13] GC/MS (70 eV, LREI, HP-5): M^+ 148 (t_R 8.82 min). GC (AT-5): t_R 7.50 min.

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Supporting Information Available: Additional experimental, kinetic, and spectral data of compounds **3**−**7**. This material is available free of charge via the Internet at http://pubs.acs.org.

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(13) Spectral data of distilled product matched that of previous reports. See: Forbes, D. C.; Standen, M. C.; Lewis, D. L. *Org. Lett.* **2003** *5*, 2283 and references therein.