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δ-Sultone Formation Through Rh-Catalyzed C–H Insertion

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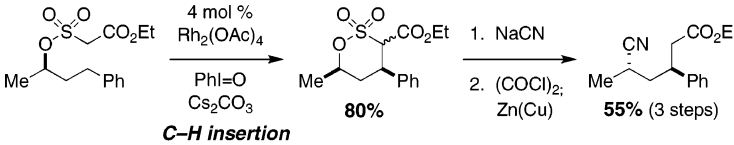
ABSTRACT



Rhodium-catalyzed reactions of sulfonate ester derivatives are biased strongly toward 1,6-insertion and thus offer a general method for assembling δ-sultones. Two protocols for staging this cyclization reaction are described, which capitalize on the unique ability of either diazo or iodonium ylide intermediates to form Rh-carbene species. The value of these heterocycles for fine chemicals synthesis is demonstrated in both reductive and oxidative reactions that make possible excision of the –SO_3– moiety.

Metal-catalyzed activation of diazo compounds has evolved as an exceptionally powerful tool for the formation of C−C bonds.[1] Such methods are unique in their ability to functionalize unactivated C−H centers under mild conditions on substrates bearing a high degree of attendant functionality. The application of these types of chemistries can lead to unconventional and inventive strategies to problems in complex molecule synthesis.[2] In this context, intramolecular diazocarbonyl C−H insertion reactions are most commonly employed, and typically occur with beneficial levels of chemo- and regioselectivity.[1,3] A hallmark of such processes is the overwhelming bias toward five-membered-ring formation. Although substrate conformation, local electronic effects, and catalyst ligand architecture may override this intrinsic preference, there is no general paradigm that governs the application of such controlling elements.[2b,4−6] Accordingly, an alternative tactic that would inherently favor six-membered-ring formation could potentially create opportunities to employ C−H insertion in ways not currently possible with diazocarbonyl derivatives.

Within the context of studies related to Rh-catalyzed intramolecular C−H amination, our laboratory has revealed that six-membered oxathiazinane heterocycles are produced from sulfamate starting materials through exclusive γ-C−H insertion.[7] The marked contrast of these results vis-à-vis five-membered-ring formation observed with carbamate, urea, and guanidine starting materials intimated that a similar relation-

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[1] Reviews: (a) Doyle, M. P.; McKervey, M. A.; Ye, T. *Modern Catalytic Methods for Organic Synthesis with Diazo Compounds*; Wiley-Interscience: New York, 1998. (b) Davies, H. M. L.; Beckwith, R. E. J. *Chem. Rev.* **2003**, *103*, 2861−2903. (c) Merlic, C. A.; Zechman, A. L. *Synthesis* **2003**, 1137−1156. (d) Doyle, M. P. In *Modern Rhodium-Catalyzed Organic Reactions*; Evans, P. A., Ed.; Wiley-VCH: Weinheim, Germany, 2005; pp 341−355. (e) Taber, D. F.; Joshi, P. V. In *Modern Rhodium-Catalyzed Organic Reactions*; Evans, P. A., Ed.; Wiley-VCH: Weinheim, Germany, 2005; pp 357−377. (f) Davies, H. M. L. *Angew. Chem., Int. Ed.* **2006**, *45*, 6422−6425.

[2] For recent examples, see: (a) Kurosawa, W.; Kan, T.; Fukuyama, T. *J. Am. Chem. Soc.* **2003**, *125*, 8112−8113. (b) Hinman, A.; Du Bois, J. *J. Am. Chem. Soc.* **2003**, *125*, 11510−11511. (c) Davies, H. M. L.; Walji, A. M. *Angew. Chem., Int. Ed.* **2005**, *44*, 1733−1735. (d) Taber, D. F.; Frankowski, K. J. *J. Org. Chem.* **2005**, *70*, 6417−6421. (e) Reisman, S. E.; Ready, J. M.; Hasuoka, A.; Smith, C. J.; Wood, J. L. *J. Am. Chem. Soc.* **2006**, *128*, 1448−1449. (f) Davies, H. M. L.; Dai, X.; Long, M. S. *J. Am. Chem. Soc.* **2006**, *128*, 2485−2490. (g) Tambar, U. K.; Ebner, D. C.; Stoltz, B. M. *J. Am. Chem. Soc.* **2006**, *128*, 11752−11753.

[3] (a) Taber, D. F.; Ruckle, R. E. *J. Am. Chem. Soc.* **1986**, *108*, 7686−7693. (b) Lee, E.; Jung, K. W.; Kim, Y. S. *Tetrahedron Lett.* **1990**, *31*, 1023−1026. (c) Doyle, M. P.; Westrum, L. J.; Wolthuis, W. N. E.; See, M. M.; Boone, W. P.; Bagheri, V.; Pearson, M. M. *J. Am. Chem. Soc.* **1993**, *115*, 958−964. (d) Padwa, A.; Austin, D. J. *Angew. Chem., Int. Ed.* **1994**, *33*, 1797−1815. (e) Wang, P.; Adams, J. *J. Am. Chem. Soc.* **1994**, *116*, 3296−3305.

[4] (a) Cane, D. E.; Thomas, P. J. *J. Am. Chem. Soc.* **1984**, *106*, 5295−5303. (b) Aburel, P. S.; Romming, C.; Undheim, K. *J. Chem. Soc., Perkin Trans. 1* **2001**, 1024−1029.

[5] Lee, E.; Choi, I.; Song, S. Y. *J. Chem. Soc., Chem. Commun.* **1995**, 321−322.

[6] (a) Doyle, M. P.; Dyatkin, A. B. *J. Org. Chem.* **1995**, *60*, 3035−3038. (b) Doyle, M. P.; Dyatkin, A. V.; Ene, D. G. *J. Am. Chem. Soc.* **1996**, *118*, 8837−8846.

[7] (a) Espino, C. G.; Wehn, P. M.; Chow, J.; Du Bois, J. *J. Am. Chem. Soc.* **2001**, *123*, 6935−6936. (b) Wehn, P. M.; Lee, J.; Du Bois, J. *Org. Lett.* **2003**, *5*, 4823−4826.

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ship might exist between C−H insertion reactions of diazosulfonyl and diazocarbonyls compounds (Figure 1).[8] Interestingly, the former class of substrates has received almost no attention in the literature.[9,10] Following a recent, analogous report by Novikov, we now disclose our own findings that (1) make available substituted six-membered-ring δ-sultones from diazosulfonates through C−H insertion and (2) enable the subsequent manipulation of these novel heterocycles to value-added materials. To further augment this chemistry, we have also delineated a protocol that obviates preparation of diazo intermediates, using instead in situ-generated aryliodonium ylides. This latter work borrows from earlier reports by Dauban and Dodd, Müller, and Charette, and nicely parallels studies in C−H amination.[11−13] As such, selective methods for δ-sultone generation and the subsequent modification of these heterocycles should open new pathways for addressing problems in fine chemicals synthesis.

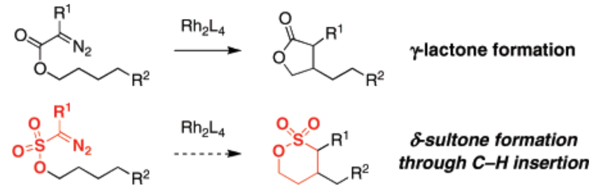


Figure 1. Substrate design alters positional selectivity.

At the onset of our studies, we were aware of only a single report that described the application of diazosulfonate esters for metal-catalyzed alkene cyclopropanation.[9,14] While attempts to prepare diazo derivatives from alkyl mesylates met with varying degrees of success, high-yielding conditions for the generation of diazosulfonates bearing an α-ester group were considerably more fruitful. These studies and those of Novikov have shown that substrates such as **3** may be readily prepared from ethyl chlorosulfonylacetate **2** followed by diazo transfer ($MeSO_3N_3$, DBU, Figure 2).[15−17] Such conditions provide reliable access from both 1° and 2° alcohols to the desired diazosulfonates (Table 1).[18]

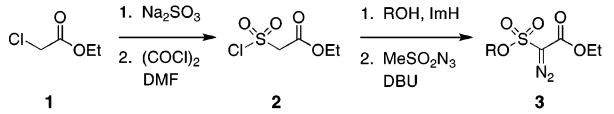
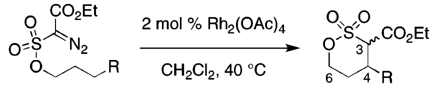
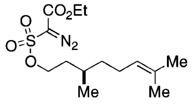
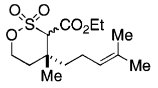
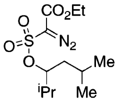
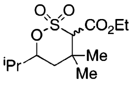
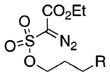
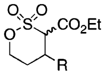
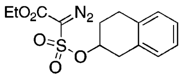
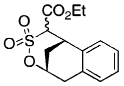
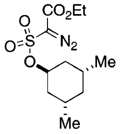
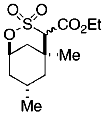
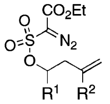
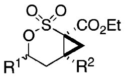


Figure 2. Preparative method for diazosulfonate substrates.

Table 1. Intramolecular C−H Insertion of Diazosulfonates



entry	substrate	product[a]		% yield[b]
1				**84[c]**
2				**94**
3			R = Ph R = ⁱPr	**85[d]** **64[d]**
4				**57[d,e]**
5				**60[f]**
6			R¹ = H, R² = Ph R¹ = ⁱPr, R² = H	**68** **77[g]**

[a] Reactions were conducted by the dropwise addition of a diazosulfonate solution to a refluxing suspension of 2 mol % of $Rh_2(OAc)_4$ in CH_2Cl_2. [b] Product yields are combined for the two diastereomers. Diastereomer ratios with respect to the C3 center range from 1−3:1. [c] Reaction performed with 2 mol % of $Rh_2(esp)_2$. [d] Reaction performed with added powdered 3 Å molecular sieves. [e] Reaction performed with 2 mol % of $Rh_2(O_2CCPh_3)_4$. [f] Reaction performed with 2 mol % of $Rh_2(O_2CC_3F_7)_4$ in CCl_4. [g] Product formed as a ∼3:1 mixture of diastereomers; reported yield is of the mixture.

Refluxing a CH_2Cl_2 solution of diazosulfonate with 2 mol % of $Rh_2(OAc)_4$ leads to exclusive formation of the corresponding six-membered δ-sultones in the majority of substrates we have examined (Table 1).[19] Of the different catalysts tested, $Rh_2(OAc)_4$ was generally superior.[20] As inferred from the collective data, 3°, benzylic, and 2° C−H

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(8) (a) Espino, C. G.; Du Bois, J. *Angew. Chem., Int. Ed.* **2001**, *40*, 598−600. (b) Lee, M.; Mulcahy, J. V.; Espino, C. G.; Du Bois, J. *Org. Lett.* **2006**, *8*, 1073−1076.

(9) Ye, T.; Zhou, C. *New J. Chem.* **2005**, *29*, 1159−1163.

(10) John, J. P.; Novikov, A. V. *Org. Lett.* **2007**, *9*, 61−63.

(11) Use of iodonium ylides as carbene precursors was originally advanced by Varvoglis and Moriarty, see: (a) Hatjiarapoglou, L.; Varvoglis, A.; Alcock, N. W.; Pike, G. A. *J. Chem. Soc., Perkin Trans. 1* **1988**, 2839−2846. (b) Moriarty, R. M.; Prakash, O.; Vaid, R. K.; Zhao, L. *J. Am. Chem. Soc.* **1989**, *111*, 6443−6444. For an excellent review on this subject, see: (c) Müller, P. *Acc. Chem. Res.* **2004**, *37*, 243−251.

(12) For leading references, see: (a) Dauban, P.; Saniere, L.; Tarrade, A.; Dodd, R. H. *J. Am. Chem. Soc.* **2001**, *123*, 7707−7708. (b) Müller, P.; Ghanem, A. *Org. Lett.* **2004**, *6*, 4347−4350. (c) Moreau, B.; Charette, A. *J. Am. Chem. Soc.* **2005**, *127*, 18014−18015.

(13) Espino, C. G.; Du Bois, J. In *Modern Rhodium-Catalyzed Organic Reactions*; Evans, P. A., Ed.; Wiley-VCH: Weinheim, Germany, 2005; pp 379−416.

(14) Thermal reactions of diazosulfonates to generate alkene products have been reported, see: Berkessel, A.; Voges, M. *Chem. Ber.* **1989**, *122*, 1147−1151.

(15) Sulfonyl chloride **2** was prepared following a modification of a procedure outlined by: Oliver, J. E.; DeMilo, A. B. *Synthesis* **1975**, 321−322. See the Supporting Information for details.

(16) Imidazole may be replaced with pyridine in the sulfonylation reaction. Stronger bases such as Et_3N were considerably less effective.

(17) Use of *p*-acetamidobenzenesulfonyl azide gave poor yield of the diazo compound and, instead, a significant amount of the alcohol, ROH, was generated.

(18) Purified diazosulfonate derivatives are generally stable for weeks if stored at −20 °C. In two cases (entries 2 and 4), decomposition was observed when these materials were left to stand overnight at 23 °C.

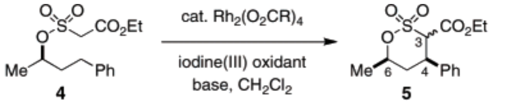
bonds are all susceptible to functionalization. Qualitative reactivity trends are, in fact, analogous to those of diazocarbonyl compounds.[1] In all cases, δ-sultone products were obtained as epimeric mixtures at the α-ester carbon center. The bias for diazosulfonates to form six-membered-ring products is noted in entries 3−5, the latter two reactions yielding novel bridging bicyclic structures.[21] Such transannular insertion events are less common with diazocarbonyl derivatives, and may prove serviceable for installing angular groups at fused carbon centers in complex polycyclic natural products.[22]

When unsaturated sulfonate starting materials are used, alkene cyclopropanation is quite efficient and no product of allylic C−H insertion is obtained (entry 6). In addition, the *cis*-fused cyclopropane is generated exclusively. We attribute the strong bias of the sulfonate group toward the six-membered products in both σ-C−H and π-bond functionalization reactions to the close accord between acyclic sulfonate and δ-sultone C−S−O bond angles (102−103°).[23,24] Insertion to form the five-membered γ-sultone would force a C−S−O angle deformation of more than 5°.[25] Interestingly, distortions in bond length are almost nil (<0.03 Å) between the acyclic and 6-membered cyclic forms.

The utility of diazo compounds notwithstanding, their preparation with sulfonyl azide reagents and their hazard potential raise safety concerns. Aryliodonium ylides can function as surrogates to diazo species and may be prepared in situ from common hypervalent iodine oxidants.[11,12,26] When such species are generated in the presence of a metal catalyst and an alkene, cyclopropanation is smoothly effected. The iodonium ylide conditions thus offer salient advantages over traditional diazo chemistry. On the basis of prior art, sulfonate esters appeared well-suited as substrates for iodonium ylide chemistry because of the ease of generation and stability of the α-enolate anion. We were unaware, however, of any report demonstrating one-pot iodonium ylide formation and metal-catalyzed C−H bond insertion employing sulfonate esters or related starting materials (e.g., 1,3-dicarbonyl derivatives).[27]

Initial attempts to induce the cyclization of sulfonate **4** employed the commercial oxidant $PhI(OAc)_2$ and catalytic $Rh_2(OAc)_4$ (entry 1, Table 2). Although sultone **5** was generated under these conditions, ~30% of the starting material was left unreacted along with a decomposition product, 4-phenyl-2-butanol (20%). Switching to PhI═O resulted in a dramatic improvement in the reaction outcome, as **5** was produced exclusively and could be isolated in 80% yield (entry 2). This protocol has proven most effective, with both 3 Å molecular sieves and Cs_2CO_3 being necessary for high product conversion. Formation of **5** was depressed in the absence of sieves or when alternate inorganic bases were employed. Additionally, it appears that the sparing solubility of both $Rh_2(OAc)_4$ and PhI═O in CH_2Cl_2 is an important factor for the success of this process. Circumstantial support for this conclusion is based on the poor performance of $PhI(OAc)_2$ and of $Rh_2(oct)_4$ and $Rh_2(esp)_2$ as catalysts for the reaction, both of which are completely soluble in CH_2Cl_2 (entries 7 and 8).[28] The use of benzene as solvent, however, did not improve the reaction (entry 9).

Table 2. In Situ Iodonium Ylide Formation and C−H Insertion



entry	catalyst[a]	oxidant	base	additive	% product[b]
1	4 mol% **A**	$PhI(OAc)_2$	Cs_2CO_3	3 Å MS	**50**
2	4 mol% **A**	PhI=O	Cs_2CO_3	3 Å MS	**100(80)**[c]
3	2 mol% **A**	PhI=O	Cs_2CO_3	3 Å MS	**75**
4	4 mol% **A**	PhI=O	Cs_2CO_3	–	**30**[d]
5	4 mol% **A**	PhI=O	Na_2CO_3	3 Å MS	**5**
6	4 mol% **A**	PhI=O	K_2CO_3	3 Å MS	**10**
7	4 mol% **B**	PhI=O	Cs_2CO_3	3 Å MS	**45**
8	4 mol% **C**	PhI=O	Cs_2CO_3	3 Å MS	**20**
9	4 mol% **A**	PhI=O	Cs_2CO_3	3 Å MS	**30**[e]

[a] **A** = $Rh_2(OAc)_4$; **B** = $Rh_2(oct)_4$; **C** = $Rh_2(esp)_2$. Reactions performed at 25 °C with 1.3 equiv of oxidant, 3.0 equiv of base, and 100 mg of powdered 3 Å molecular sieves. [b] Product conversion based on ^1H NMR integration. [c] Isolated yield in parentheses; product obtained as a 6:1 mixture of epimers at C3 and >20:1 cis/trans selectivity at C4/C6. [d] 4-Phenyl-2-butanol accounted for most of the mass balance in this reaction. [e] Reaction performed in C_6H_6.

Cyclization of sulfonate esters with PhI═O, Cs_2CO_3, and catalytic $Rh_2(OAc)_2$ is functional with a range of 2° alcohol-derived materials (Table 3). Highest yields of δ-sultones are obtained for substrates bearing 3° and benzylic C−H bonds. The success of these reactions is apparently due to a combination of effects involving Thorpe−Ingold-type preorganization and the established electronic preference of Rh-carbenoids for 3° and benzylic C−H centers.[1,29] In substrates lacking these structural elements, product yields are dimin-

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(19) The addition of powdered 3 Å molecular sieves was found to improve sultone yields in a few instances, see the Supporting Information for details.

(20) Other Rh catalysts examined included $Rh_2(oct)_4$, $Rh_2(OPiv)_4$, $Rh_2(O_2CCPh_3)_4$, $Rh_2(O_2CCF_3)_4$, $Rh_2(NHCOCF_3)_4$, and $Rh_2(cap)_4$.

(21) In entry 4, the five-membered-ring product formed through benzylic C−H insertion was also obtained (42% yield).

(22) For select examples of transannular C−H insertions with diazoketones, see: (a) Ghatak, U. R.; Chakrabarty, S. *J. Am. Chem. Soc.* **1972**, *94*, 4756−4758. (b) Spero, D. M.; Adams, J. *Tetrahedron Lett.* **1992**, *33*, 1143−1146. (c) West, F. G.; Eberlein, T. H.; Tester, R. W. *J. Chem. Soc., Perkin Trans. 1* **1993**, 2857−2859. (d) White, J. D.; Hrnciar, P.; Stappenbeck, F. *J. Org. Chem.* **1999**, *64*, 7871−7884. (e) Wardrop, D. J.; Velter, A. I.; Forslund, R. E. *Org. Lett.* **2001**, *3*, 2261−2264.

(23) A typical γ-sultone C−S−O bond angle is ≤98°, see: (a) Billodeaux, D. R.; Owens, C. V.; Sayes, C. M.; Soper, S. A.; Fronczek, F. R. *Acta Cryst.* **1999**, *C55*, 2126−2129. (b) Muir, K. W.; Rodger, C. S.; Morris, D. G.; Ryder, K. S. *Acta Cryst.* **1998**, *C54*, 1546−1548.

(24) For select X-ray data of acyclic sulfonates and δ-sultones, see: (a) Ferguson, G.; Schwan, A. L.; Kalin, M. L.; Snelgrove, J. L. *Acta Crystallogr.* **1997**, *C53*, IUC9700009. (b) Horger, R.; Marsch, M.; Geyer, A.; Harms, K. *Acta Crystallogr.* **2005**, *E61*, o3447−o3448.

(25) We have used a similar argument to explain the strong bias for oxathiazinane dioxide formation in amination reactions of sulfamate esters, see ref 7a.

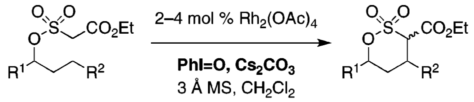
(26) (a) Ghanem, A.; Lacrampe, F.; Schurig, V. *Helv. Chim. Acta* **2005**, *88*, 216−239. (b) Bonge, H. T.; Hansen, T. *Synlett* **2007**, 55−58.

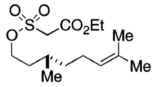
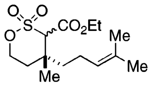
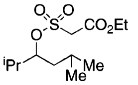
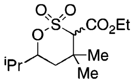
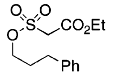
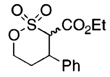
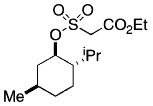
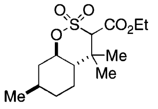
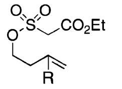
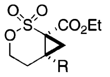
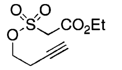
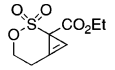
(27) Müller has demonstrated Rh-catalyzed C−H insertions of isolated iodonium ylides: Müller, P.; Fernandez, D. *Helv. Chim. Acta* **1995**, *78*, 947−958.

(28) $Rh_2(esp)_2$ = $Rh_2(\alpha,\alpha,\alpha',\alpha'$-tetramethyl-1,3-benzenedipropionate$)_2$, see: Espino, C. G.; Fiori, K. W.; Kim, M.; Du Bois, J. *J. Am. Chem. Soc.* **2004**, *126*, 15378−15379. Interestingly, syringe-pump addition of $Rh_2(oct)_4$ over a 1 h period to a suspension of **4**, PhI═O, Cs_2CO_3, and 3 Å MS, gave results comparable to entry 7. The unique effectiveness of $Rh_2(OAc)_4$ as a catalyst for this process is absent a clear explanation at this time.

(29) Jung, M. E.; Piizzi, G. *Chem. Rev.* **2005**, *105*, 1735−1766.

Table 3. Rh-Catalyzed C−H Insertion of Sulfonate Esters

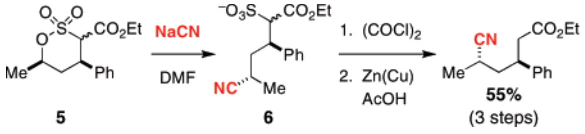


entry	substrate	product[a]	% yield[b]
1			52
2			93
3			27
4			97
5			R = H 82 R = Ph 82
6			91

[a] Reactions were conducted by addition of iodosobenzene (1.2 equiv), powdered 3 Å molecular sieves, Cs_2CO_3 (3 equiv), and 2−4 mol % of $Rh_2(OAc)_4$ to a solution of substrate in CH_2Cl_2. [b] Product yields are reported for the mixture of C3-epimers. Diastereomer ratios with respect to the α-ester carbon range from 1−6:1.

ished when compared to analogous experiments conducted with diazo compounds (cf. entries 1 and 3, Tables 1 and 3). Both base-promoted decomposition of the starting sulfonate and the adventitious consumption of PhI=O are observed in reactions that underperform. Nevertheless, the success of this method for certain substrate types and its facile, single-step operation make it a worthy compliment to the diazo chemistry. Further investigations will continue to advance this technology as a broadly applicable tool for synthesis.

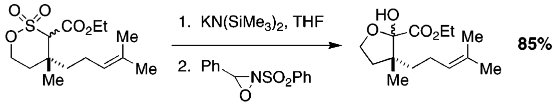
The chemistry of δ-sultones as synthetic intermediates en route to complex molecules has not been fully exploited.[30] As with their smaller-ring counterparts, it is possible to displace the C−O bond with nucleophiles of varying strength (eq 1). In our hands, NaCN, NaN_3, NaOAc, and thiolates



5 6 (1)

are all found to react with 1° and 2° alcohol-derived sultones to give the corresponding sulfonate salts. Direct extrusion of SO_3 (or SO_4^{2-}) from these ring-opened products, however, proved unfeasible under a variety of conditions investigated.[31] Presumably, it is for this reason that sultones have enjoyed only sparing use as electrophilic reagents. Accordingly, we have developed a two-step protocol to effect the removal of the SO_3^- moiety from the acyclic products (e.g., **6**). Initial conversion of the sulfonate salt to the sulfonyl chloride with use of oxalyl chloride is clean and quantitative. Subsequent treatment of the sulfonyl chloride with Zn(Cu) couple in THF/AcOH affords the desired ester product.[32] Although this procedure requires two steps, the overall sequence is high yielding and does not necessitate isolation of the intermediate sulfonyl chloride. This protocol should help to expand the potential applicability of C−H insertion methods for sultone assembly.

Following literature reports, removal of the sulfonyl linkage was also examined under oxidative conditions.[30b,33] Treatment of the potassium enolate derived from sultone **7** with Davis oxaziridine in THF furnishes lactol **8** in 85% yield (eq 2).[34] This transformation presumably involves *O*-atom transfer followed by C−S bond cleavage. The intermediate α-keto ester is then trapped internally by the pendant alcohol. Sultone ring-opening employing this method is easily executed, extremely efficient, and well-suited for application in complex synthesis.



7 **8** (2)

Chemoselective generation of δ-sultones through Rh-catalyzed C−H insertion makes available a novel class of heterocycles, which have the potential to serve as valuable intermediates in synthesis. Sulfonate ester starting materials are conveniently prepared from 1° and 2° alcohols and may be subjected to one of two sets of conditions for processing to the desired sultone. With the ability to modify the cyclized products through S_N2 displacement and enolate α-oxidation reactions, new strategies for employing Rh-catalyzed C−H functionalization in complex synthesis may be unveiled.

Acknowledgment. We are grateful to the National Institutes of Health, Camille and Henry Dreyfus Foundation, the A. P. Sloan Foundation, Amgen, Boehringer-Ingelheim, GlaxoSmithKline, Merck, and Pfizer for generous support of this work.

Supporting Information Available: General experimental protocols and characterization data for all new compounds. This material is available free of charge via the Internet at http://pubs.acs.org.

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[30] For reviews on sultone synthesis and reactivity, see: (a) Roberts, D. W.; Williams, D. L. *Tetrahedron* **1987**, *43*, 1027−1062. (b) Metz, P. *J. Prakt. Chem.* **1998**, *340*, 1−10.

[31] Transformations of this type have almost no precedent in the literature. Attempts to extrude SO_3 upon heating with or without acid gave no reaction.

[32] Use of Zn dust alone resulted in conversion to the corresponding sulfinic acid, which proved resistant toward desulfinylation.

[33] For related transformations, see: (a) Little, R. D.; Myong, S. O. *Tetrahedron Lett.* **1980**, *21*, 3339−3342 (b) Hwu, J. R. *J. Org. Chem.* **1983**, *48*, 4432−4433. (c) Williams, D. R.; Robinson, L. A.; Amato, G. S.; Osterhout, M. H. *J. Org. Chem.* **1992**, *57*, 3740−3744. (d) Metz, P.; Fleischer, M.; Fröhlich, R. *Tetrahedron* **1995**, *51*, 711−732. (e) Doye, S.; Hotopp, T.; Wartchow, R.; Winterfeldt, E. *Chem. Eur. J.* **1998**, *4*, 1480−1488.

[34] Chen, B.-C.; Zhou, P.; Davis, F. A.; Ciganek, E. *Org. React.* **2003**, *62*, 1−356.