Notes

Chemistry of Oxaziridines. 9.[1] Synthesis of 2-Sulfonyl- and 2-Sulfamyloxaziridines Using Potassium Peroxymonosulfate (Oxone)

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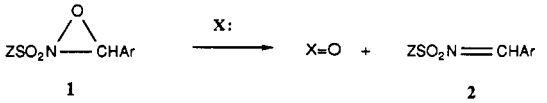
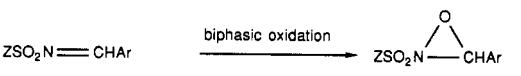
In recent years 2-sulfonyl- and 2-sulfamyloxaziridines, **1a** and **1b**, respectively, have received considerable attention as selective aprotic oxidizing reagents for a large variety of substrates. These reagents oxidize sulfides (selenides) to sulfoxides (selenoxides) without overoxidation.[2,3] They selectively oxidize thiones to sulfines[4] and epoxidize alkenes.[5] The aprotic nature of these reagents has made possible the facile syntheses of alcohols and phenols by hydroxylation of organometallic reagents,[6] the oxidation of enolates to α-hydroxy carbonyl compounds,[7] and the synthesis of extremely acid-sensitive species such as α-siloxy epoxides[8] and sulfenic acids.[9] Optically active 2-sulfonyl- and 2-sulfamyloxaziridines **1a,b** afford the highest stereoselectivities reported for the oxidation of nonfunctionalized sulfides (up to 91% ee)[1] and alkenes (up to 61% ee).[5c] The asymmetric oxidation of prochiral enolates to optically active α-hydroxy carbonyl compounds (up to 95% ee) by (+)- and (−)-(camphorylsulfonyl)oxaziridines has recently been reported.[10]



1 2

a) Z = alkyl, aryl b) Z = R_2N

Both 2-sulfonyl- and 2-sulfamyloxaziridines **1a,b** are prepared in good to excellent yields (80–90%) by biphasic, basic oxidation of the corresponding sulfon- and sulfamimines **2** using *m*-chloroperbenzoic acid (*m*-CPBA).[1,11] Complete oxidation generally takes from 1 to 12 h and requires a phase-transfer catalyst. A mechanism involving a Baeyer–Villiger-type oxidation of the electron-deficient C–N bond in **2** by the peroxyacid anion has been proposed.[11] *m*-Chloroperbenzoic acid is expensive, and large-scale oxidations are sometimes contaminated with bis(*m*-chlorobenzoyl) peroxide, which complicates product purification.[12] For these reasons an oxidizing reagent that would avoid these difficulties was sought. Improved methodology for the general synthesis of 2-sulfonyl- and 2-sulfamyloxaziridines **1a,b** using buffered potassium peroxymonosulfate (Oxone) is described.



2 1

Oxone ($2KHSO_5 \cdot KHSO_4 \cdot K_2SO_4$) is an inexpensive and stable oxidizing reagent that is commercially available. This reagent has been used in the oxidation of sulfides to sulfoxides and sulfones.[13] Buffered Oxone has been used in the generation of dioxirane intermediates,[14] in our synthesis of the novel tricyclic optically active (camphorylsulfonyl)oxaziridine,[10a] and more recently in the epoxidation of α,β-unsaturated acids.[15]

In general, replacement of *m*-CPBA by buffered Oxone in toluene results in increased yields, a significant reduction of the reaction time, and easier purification of the oxaziridine. Furthermore, a phase-transfer catalyst is no longer necessary. These results are summarized in Table I.

Oxone buffered with $KHCO_3$ (pH ca. 7.5) gives nearly quantitative yields of 2-sulfonyloxaziridines **1a** within 2 h (entries 1, 3, and 8). Significantly when Oxone is buffered with K_2CO_3 (pH ca. 9) the time for complete oxidation is reduced to 15 min (entries 2, 4, 5, 7, and 9). Apparently a higher concentration of the peroxymonosulfate anion (SO_5^{2-}), necessary for oxidation of **2**, is present at the higher pH.

By contrast, oxidation of sulfamimines **2b** to 2-sulfamyloxaziridines **1b** is more difficult (compare entries 1–9 with 10–22). Even at the higher pH the reaction time required for complete oxidation varies from 30 min to 24 h (entries 12, 16, and 22). Nevertheless the time for oxidation of these sulfamimines **2b** with *m*-CPBA is 12–18 h.[1] The notable exception is the pentafluorophenyl sulfamylimine **2b** (Ar = C_6F_5, entries 18–22) where *m*-CPBA gives a better yield in a shorter time period.[1] We speculate

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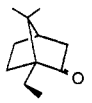
Table I. Biphasic Preparation of 2-Sulfonyl- and 2-Sulfamyloxaziridines 1a,b in Toluene Using Buffered Oxone

entry	oxaziridine Z =	oxaziridine Ar =	Oxone, equiv (buffer)	time (h)	% isoltd yield (ratio of *RR*:*SS*)[a]	ref
1	Ph	Ph	1.2 ($KHCO_3$)	2	>95	11
2			1.2 (K_2CO_3)	15 min	>95	
3	Ph	2-NO_2Ph	1.2 ($KHCO_3$)	2	>95	17
4			1.2 (K_2CO_3)	15 min	>95	
5	Ph	3-NO_2Ph	1.2 (K_2CO_3)	15	>95	11
6	Ph	4-NO_2Ph	1.2 (K_2CO_3)	15 min	>95	11
7	Ph	2-Cl, 5-NO_2Ph	1.2 (K_2CO_3)	15 min	>95	1
8		2-Cl, 5-NO_2Ph	2.0 ($KHCO_3$)	2	90 (50:50)	15
9			3.0 (K_2CO_3)	15 min	95 (54:46)	
10	$(PhCH_2)_2N$	2-Cl, 5-NO_2Ph	2.0 ($KHCO_3$)	2	no reaction	1
11			6.0 (K_2CO_3) $CHCl_3$	2	70 (30)[b]	
12			6.0 (K_2CO_3) $CHCl_3$	6	90	
13			6.0 (K_2CO_3) $CHCl_3$[c]	2	complex mixture[d]	
14		3-NO_2Ph	2.0 ($KHCO_3$)	2	10	1
15			6.0 ($KHCO_3$)[c]	12	>95 (53:47)	
16			3.0 (K_2CO_3)	30 min	93 (53:47)	
17		2-Cl, 5-NO_2Ph	3.0 (K_2CO_3)	30 min	93 (50:50)	1
18		C_6F_5	6.0 ($KHCO_3$)	24	10	1
19			6.0 ($KHCO_3$)[c] $CHCl_3$	24	40 (49:51)	
20			6.0 (K_2CO_3) $CHCl_3$	24	no reaction	
21			6.0 (K_2CO_3)[c] $CHCl_3$	6	complex mixture[d]	
22			6.0 (K_2CO_3) $CHCl_3$	24	76 (53:47)[e]	

[a] Ratio determined by NMR. [b] Yield of starting imine as determined by NMR. [c] 20% by weight 18-crown-6 added. [d] Hydrolysis to sulfamide (ZSO_2NH_2) detected by NMR. [e] Sulfamylimine, 18%, was detected by NMR.

that these rate differences may reflect reduced reactivity of the C–N bond caused by the sterically demanding and more lipophilic sulfamyl group in **2b** compared to **2a**. Increased lipophilicity in **2b** presumably reduces the concentration of the sulfamimine at the water–toluene interface where oxidation is thought to take place. Consistent with these speculations is the fact that the sulfamimine most difficult to oxidize under these conditions is the one containing the highly lipophilic pentafluorophenyl group (entries 18–22). Furthermore, the rates of oxidation are improved by using a more polar solvent such as $CHCl_3$ (entries 11–13 and 19–22). In one case addition of 18-crown-6 ether gives improved yields (compare entry 15) but in others results in hydrolysis of the sulfamimines **2b** (entries 13 and 21).

In summary, improved methodology for the preparation of 2-sulfonyl- and 2-sulfamyloxaziridines **1a,b** is described using buffered Oxone in place of *m*-CPBA. This modification results in faster rates of oxaziridine formation and easier product purification. The only disadvantage in using Oxone in place of *m*-CPBA is the slow rate of oxidation for the pentafluorophenyl sulfamimine (entry 22), and the diastereoisomer ratio for the oxidation of (+)-*N*-(2-chloro-5-nitrobenzylidene)-*d*-camphorsulfonamide changes from 65:35 (*m*-CPBA) to 54:46 (entries 8 and 9).[16]

Experimental Section

Melting points were determined on a Mel-Temp apparatus and are uncorrected. ^1H NMR spectra were measured on a JOEL FX 90Q (90 MHz) NMR spectrometer using Me_4Si as the internal reference. Sulfonimines **2a**[11,17] and sulfamimines **2b**[1] were prepared as previously described. Potassium peroxymonosulfate (Oxone) was purchased from Aldrich. Solvents were reagent grade and used without additional purification.

General Procedure for Oxidation of Sulfonimines 2a. In a 250-mL three-necked Morton flask, equipped with a mechanical stirrer and a 50-mL addition funnel, were placed 8.2 mmol of the appropriate sulfonimines **2a** in 80 mL of toluene and 6.8 g of $KHCO_3$ or 9.5 g K_2CO_3 (3.5 equiv based on potassium peroxymonosulfate) in 50 mL of water. The reaction was stirred vigorously and a solution of 6.0 g (9.8 mmol) of Oxone (19.6 mmol of potassium peroxymonosulfate) in 50 mL of water was added dropwise over 15 min. The reaction mixture was stirred until all of the sulfonimine had been consumed. The reaction progress was determined by removing 1-mL aliquots from the organic solvent, evaporating it in vacuo below 40 °C, and determining the ratio of sulfonimine (δ 8.7–9.2) to oxaziridine (δ 5.4–6.0) by NMR. When the reaction was complete the aqueous layer was separated and washed once with 25 mL of toluene. The combined toluene extracts were washed with 25 mL of aqueous 10% sodium sulfite and dried over anhydrous $MgSO_4$, and the solvent was evaporated on an efficient rotary evaporator keeping the bath temperature below 40 °C. The resulting viscous oil was triturated with a few milliliters of *n*-pentane to afford the oxaziridines **1a** analytically pure as determined by melting point and NMR.

General Procedure for Oxidation of Sulfamimines 2b. In a 50-mL two-necked Morton flask, equipped with a magnetic stirring bar and a dropping funnel, were placed 1.0 mmol of the appropriate sulfamimines **2b** in 5 mL of toluene or $CHCl_3$ and 4.2 g of $KHCO_3$ or 5.8 g of K_2CO_3 (3.5 equiv based on potassium peroxymonosulfate) in 10 mL of water. The reaction was stirred vigorously and a solution of 3.7 g (6 mmol) of Oxone in 15 mL of water was added dropwise over 2 min. Stirring of the reaction mixture was continued until the sulfamimine had been oxidized. The progress of the oxidation was monitored by TLC (silica gel) eluting with methylene chloride by observing the absence of sulfamylimine hydrolysis products and the formation of the oxaziridine **1**.[18] When the reaction was complete, the mixture was quenched by addition of approximately 1.0 g of sodium metabisulfite. After being stirred for a few minutes the aqueous layer was separated and washed once with 10 mL of toluene. The

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(18) Sulfon- and sulfamimines **2** are hydrolyzed on silica gel to the corresponding sulfonamides and sulfamides and aldehydes. 2-Sulfamyloxaziridines **1b** are stable to chromatography[9] while 2-sulfonyloxaziridines **1a** are hydrolyzed.[16]

combined toluene extracts were dried over anhydrous $MgSO_4$ and the solvent was evaporated on an efficient rotary evaporator keeping the bath temperature below 40 °C. The resulting viscous oil was washed with a few milliliters of *n*-pentane to afford the sulfamyloxaziridines **1b**. The purity and diastereomer ratios were determined by NMR and melting point.

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Note added in proof: We have observed that Oxone that has been exposed to moisture for several months gives reduced reactivity in the oxidations described here.

Registry No. (±)-**1a** (Z = Ph, Ar = Ph), 113548-13-3; (±)-**1a** (Z = Ph, Ar = 2-NO_2Ph), 113548-14-4; (±)-**1a** (Z = Ph, Ar = 3-NO_2Ph), 113548-15-5; (±)-**1a** (Z = Ph, Ar = 4-NO_2Ph), 113548-16-6; (±)-**1a** (Z = Ph, Ar = 2-Cl, 5-NO_2Ph), 113625-69-7; (*R*,*R*)-**1a** (Z = 10-camphoryl, Ar = 2-Cl,5-NO_2Ph), 81369-89-3; (*S*,*S*)-**1a** (Z = 10-camphoryl, Ar = 2-Cl,5-NO_2Ph), 81310-08-9; (±)-**1b** (Z = $(PhCH_2)_2N$, Ar = 2-Cl,5-NO_2Ph), 113625-70-0; (R,-*R*)-**1b** (Z = (*S*)-$PhCH(CH_3)PhCH_2N$, Ar = 3-NO_2Ph), 108167-38-0; (*S*,S)-**1b** (Z = (*S*)-$PhCH(CH_3)PhCH_2N$, Ar = 3-NO_2Ph), 108266-24-6; (*R*,*R*)-**1b** (Z = (*S*)-$PhCH(CH_3)PhCH_2N$, Ar = 2-Cl,5-NO_2Ph), 89616-61-5; (*S*,*S*)-**1b** (Z = (*S*)-$PhCH(CH_3)PhCH_2N$, Ar = 2-Cl,5-NO_2Ph), 89556-80-9; (*R*,*R*)-**1b** (Z = (*S*)-$PhCH(CH_3)PhCH_2N$, Ar = C_6F_5), 108167-42-6; (*S*,*S*)-**1b** (Z = (*S*)-$PhCH(CH_3)PhCH_2N$, Ar = C_6F_5), 108391-91-9; **2a** (Z = Ph, Ar = Ph), 13909-34-7; **2a** (Z = Ph, Ar = 2-NO_2Ph), 113567-60-5; **2a** (Z = Ph, Ar = 3-NO_2Ph), 52962-76-2; **2a** (Z = Ph, Ar = 4-NO_2Ph), 36176-89-3; **2a** (Z = Ph, Ar = 2-Cl,5-NO_2Ph), 108167-37-9; **2b** (Z = 10-camphoryl, Ar = 2-Cl,5-NO_2Ph), 82679-82-1; **2b** (Z = $(PhCH_2)_2N$, Ar = 2-Cl,5-NO_2Ph), 108167-36-8; **2b** (*Z* = (*S*)-$PhCH(CH_3)PhCH_2N$, Ar = 2-Cl,5-NO_2Ph), 89556-76-3; **2b** (Z = (*S*)-$PhCH(CH_3)PhCH_2N$, Ar = C_6F_5), 108167-35-7; Oxone, 37222-66-5.

Conversion of Aucubin to a Useful Corey Lactone Analogue for the Synthesis of 11-Methyl PGA_2

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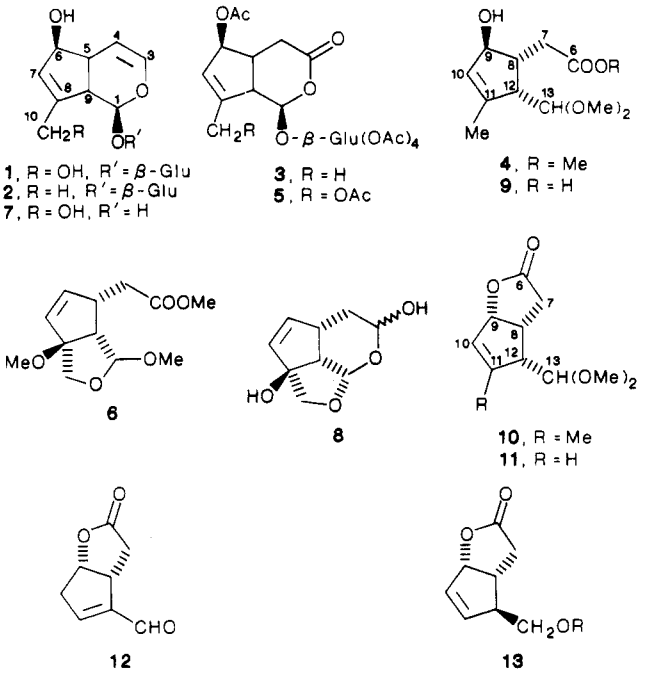
We have recently been interested in the synthesis of bioactive compounds or their intermediates from readily accessible iridoid glucosides. In particular the preparation of cyclopentenoid 1,5-dialdehydes,[1,2] of a Corey lactone analogue,[3] and, more recently, of a new 11-deoxy-11-β-methoxy-11α-(hydroxymethyl)-12-*epi*-$PGF_{2\alpha}$ methyl ester[4] from aucubin (**1**) (Chart I) have already been described.

Herein we wish to report the synthesis of bicyclic γ-lactone **10** (3-oxo-6-α-(dimethoxymethyl)-7-methyl-*cis*-2-oxabicyclo[3.3.0]7-octene),[5] a useful intermediate for 11-methyl PG's syntheses.

Our approach to **10** started from aucubin (**1**), the most diffuse and abundant iridoid glucoside[6] (20 g of **1** were

Chart I



obtained from 1 kg of fresh leaves of the common shrub *Aucuba japonica*) and represents a new route to 11-methyl PG analogues, which in some cases showed high activity.[7] In particular, 11-deoxy-11α,16,16-trimethyl PGE_2[8] (Trimoprostil), synthetized by Hoffmann La Roche Co., as well as a recent Syntex example,[9] have been successfully tested as antiulcer drugs.

Results and Discussion

Aucubin (**1**) was subjected for a short period (15 min) to a Birch reduction (Li/NH_3) at a low temperature (−90 °C). After the usual workup and chromatographic purification 10-monodeoxyaucubin (**2**) was isolated in good yield (83%) besides 6,10-dideoxyaucubin (12%).

According to the Berkowitz procedure,[10,11] the penta-*O*-acetyl derivative of **2** was transformed into the well-known acetyl lactone **3**[11] (85% overall yield from **2**), previously utilized as the 7,8-dihydro derivative for synthesis of 11-deoxy-11-methyl PG intermediates.[11]

The aim of our strategy was, on the contrary, that of retaining the Δ^7 double bond of **3** to take advantage of the reactivity of the allylic 6-OH for closure of the bicyclo γ-lactone system (Corey lactone).

Therefore, the lactone **3** was subjected to acidic methanolysis, affording, after the usual workup and final

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