Stereoselective Synthesis of *N*-Hydroperoxyalkyloxaziridines: A New Class of Heterocyclic Hydroperoxides

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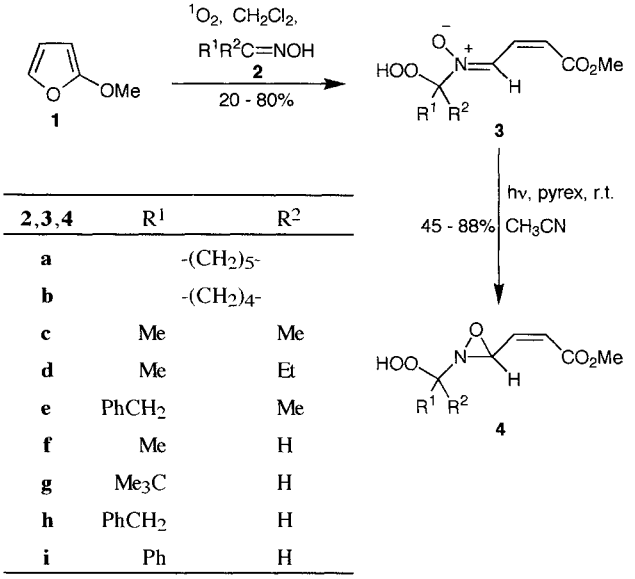
Irradiation of hydroperoxynitrones **3**, which can be easily prepared by dye-sensitized photooxygenation of 2-methoxyfuran **(1)** in the presence of oximes **2**, leads to the highly functionalized *trans*-oxaziridines **4**. The reaction proceeds smoothly at room temperature in fairly good yields.

Oxaziridines are among the most versatile intermediates in organic synthesis as the inherent strain of the ring and the relatively weak N–O bond make them unusually reactive.[1] Indeed, they can react as both aminating and oxygenating reagents with nucleophiles, undergo a number of addition and cycloaddition reactions with heterocumulenes and rearrange to nitrones or amides.[1,2] In addition to these transformations one particular property of oxaziridines is the non-inverting nature of the nitrogen pyramid.[1,2] Although oxaziridines are generally obtained by the action of a peracid on a combination of a carbonyl compound and an amine, either as a Schiff base or a simple mixture,[2] sometimes photoisomerization of nitrones is also used and, by this method, oxaziridines bearing alkyl and aryl substituents or condensed with five-, six-, and seven-membered rings as well as spirooxaziridines have been prepared.[3] Here we extend the photochemical approach to the synthesis of functionalized oxaziridines.

Recently, we developed an efficient route for hydroperoxynitrones **3**, by dye-sensitized photooxygenation of 2-methoxyfuran **(1)** in the presence of oximes **2**.[4] In the course of our work in this field, we obtained very poor yields of *N*-hydroperoxyalkyloxaziridine **4a** by chance, through the prolonged irradiation of the photooxygenation mixture in the presence of cyclohexanone oxime.[4] Bearing this in mind, as well as the fact that compound **4a** was the first example of an oxaziridine functionalized with a hydroperoxy group, we planned a synthetic scheme in which the oximes **2** lead to *N*-hydroperoxyalkyloxaziridines **4** via hydroperoxynitrones **3** (Scheme). Table 1 reports the physical and spectral data as well as the yields of the previously unreported nitrones **3b,d,e,g,h**. The isomerization of nitrones **3** to *trans*-oxaziridines **4** takes 10–30 minutes at room temperature upon irradiation (500 W high pressure mercury lamp, Pyrex filter) of a 0.02 M solution of the nitrone in dry acetonitrile. Silica gel chromatography affords the *trans*-oxaziridines **4** in fairly good yields (Table 2). Stereochemical assignment of the *trans*-configuration was based on NOE experiments carried out on **4c** and, for the other oxaziridines, by comparison of the chemical shifts of the oxaziridinyl proton (δ = 5.38–5.95) with that of **4c** (δ = 5.78).[5] The reaction is highly stereoselective. All the oxaziridines **4** prepared have the same stereochemistry as the initial nitrones. This is in accordance with the predictions of the orbital symmetry rules[7] and with the configurational stability at the oxaziridine nitrogen.[8] Therefore, the formation of diastereomers for oxaziridines **4d–i** is evidently due to the presence of the additional stereogenic center on the hydroperoxy substituted carbon.



2,3,4	R^1	R^2
a	-$(CH_2)_5$-	
b	-$(CH_2)_4$-	
c	Me	Me
d	Me	Et
e	$PhCH_2$	Me
f	Me	H
g	Me_3C	H
h	$PhCH_2$	H
i	Ph	H

Scheme

A limitation of the synthetic method is that it does not permit the preparation of oxaziridines **4** bearing both alkyl and aryl groups or two aryl groups on the hydroperoxy substituted carbon since the initial nitrones cannot be isolated.[4,9]

In conclusion, the reaction represents a stereoselective synthesis of a new class of functionalized oxaziridines. The synthetic versatility of these compounds as well as the presence of both oxaziridine and hydroperoxy functions renders the route particularly attractive. Moreover, through the use of a chiral auxiliary, this methodology should permit the preparation of optically active compounds.[10] This possibility is currently under investigation in our laboratory.

Irradiations were performed using a 500 W high pressure mercury lamp (Helios Italquartz). Melting points were determined using a Gallenkamp melting point apparatus and are uncorrected. The IR spectra were obtained on a Perkin Elmer 1760X-FT spectrophotometer using $CHCl_3$ as solvent. ^1H and ^{13}C NMR spectra were recorded at 270 MHz and 67.9 MHz, respectively, on a Bruker AC-270 spectrometer using $CDCl_3$ as solvent and TMS as internal standard. Elemental analyses were obtained using a Carlo Erba EA 1108-Elemental Analyzer. CH_2Cl_2 and MeCN used for the reactions were anhydrous. Silica gel (0.063–0.2 mm Macherey-Nagel) and light petroleum (bp 40–60 °C) were used for column chromatogra-

Table 1. New Hydroperoxynitrones **3** Prepared

Product[a]	Yield[b] (%)	mp[c] (°C)	IR ($CHCl_3$) ν (cm^{-1})	^1H NMR ($CDCl_3$/TMS) δ, J (Hz)	^{13}C NMR ($CDCl_3$/TMS) δ
3b	76	75–77	3529, 3157, 1713, 1606, 1091	1.65–2.52 [m, 8H, $(CH_2)_4$], 3.77 (s, 3H, OCH_3), 6.08 (d, J = 11.8, 1H, CH=C*H*CO), 7.34 (dd, J = 11.8, 10.2, 1H, C*H*=CHCO), 9.05 (d, J = 10.2, 1H, CH=N^+), 10.10 (br s, 1H, OOH)	24.7 (t, 2 × CH_2), 35.0 (t, 2 × CH_2), 51.7 (q, OCH_3), 114.3 (s, C–OOH), 123.4 (d, CH=*C*HCO), 131.5 and 131.7 (2 × d, *C*H=CHCO and CH=N^+), 166.3 (s, CO_2)
3d	70	72–73	3531, 3160, 1713, 1605, 1077	0.96 (t, J = 7.3, 3H, C*H*$_3$CH$_2$), 1.75 (s, 3H, CH_3), 2.05 (q, J = 7.3, 2H, CH_2), 3.77 (s, 3H, OCH_3), 6.08 (dd, J = 11.5, 1.2, 1H, CH=C*H*CO), 7.34 (dd, J = 11.5, 10.2, 1H, C*H*=CHCO), 8.98 (dd, J = 10.2, 1.2, 1H, CH=N^+), 10.80 (br s, 1H, OOH)	7.6 (q, *C*H$_3$CH$_2$), 20.0 (q, CH_3), 30.0 (t, CH_2), 51.7 (q, OCH_3), 106.9 (s, C–OOH), 123.3 (d, CH=*C*HCO), 131.0 and 131.5 (2 × d, *C*H=CHCO and CH=N^+), 166.4 (s, CO_2)
3e	60	83–84	3525, 3160, 1713, 1605, 1075	1.69 (s, 3H, CH_3), 3.30 (s, 2H, CH_2), 3.69 (s, 3H, OCH_3), 6.00 (dd, J = 11.5, 1.1, 1H, CH=C*H*CO), 7.23 (s) and 7.26 (dd, J = 11.5, 10.2) (together 6H, ArH and C*H*=CHCO), 8.81 (dd, J = 10.2, 1.1, 1H, CH=N^+), 10.93 (br s, 1H, OOH)	20.2 (q, CH_3), 42.5 (t, CH_2), 51.7 (q, OCH_3), 106.5 (s, C–OOH), 123.5 (d, CH=*C*HCO), 127.2 and 128.2 (2 × d, CH-2,6 and CH-3,5 of Ar), 130.5 (d) and 131.4 (two overlapping d) (*C*H=CHCO, CH=N^+ and CH-4 of Ar), 133.9 (s, C-1 of Ar), 166.2 (s, CO_2)
3g	24	oil	3525, 3404, 1714, 1605, 1079	1.07 [s, 9H, $C(CH_3)_3$], 3.77 (s, 3H, OCH_3), 5.05 (s, 1H, C*H*–OOH), 6.07 (dd, J = 11.2, 1.3, 1H, CH=C*H*CO), 7.36 (dd, J = 11.2, 10.2, 1H, C*H*=CHCO), 8.65 (dd, J = 10.2, 1.3, 1H, CH=N^+), 11.10 (br s, 1H, OOH)	25.7 (q, 3 × CH_3), 35.4 [s, *C*$(CH_3)_3$], 51.8 (q, OCH_3), 110.6 (d, CH–OOH), 123.6 (d, CH=*C*HCO), 130.6 and 133.4 (2 × d, *C*H=CHCO and CH=N^+), 166.2 (s, CO_2)
3h	66	oil	3517, 3055, 1718, 1607, 1080	3.00 (dd, J = 14.3, 6.7) and 3.20 (dd, J = 14.3, 5.7) (together 2H, CH_2), 3.68 (s, 3H, OCH_3), 5.44 (dd, J = 6.7, 5.7, 1H, C*H*–OOH), 6.00 (d, J = 11.8, 1H, CH=C*H*CO), 7.20–7.40 (m, 6H, ArH and C*H*=CHCO), 8.52 (d, J = 10.2, 1H, CH=N^+), 9.72 (br s, 1H, OOH)	36.8 (t, CH_2), 51.7 (q, OCH_3), 105.1 (d, CH–OOH), 124.6 (d, CH=*C*HCO), 127.3, 128.6 and 129.3 (3 × d, CH of Ar), 130.1 (d) and 133.8 (overlapping d and s) (*C*H=CHCO, CH=N^+ and C-1 of Ar), 165.8 (s, CO_2)

[a] Satisfactory microanalyses obtained: C ± 0.16, H ± 0.20, N ± 0.15.
[b] Yield of isolated product.
[c] Recrystallization solvent: hexane/Et_2O.

Table 2. *N*-Hydroperoxyalkyloxaziridines **4** Prepared[a]

Product[b]	Yield[c] (%)	IR ($CHCl_3$) ν (cm^{-1})	^1H NMR ($CDCl_3$/TMS) δ, J (Hz)	^{13}C NMR ($CDCl_3$/TMS) δ
4a[4]	85	–	–	–
4b	88	3525, 3442, 1724, 1655, 1441	1.72–2.08 (m, 7H) and 2.24–2.42 (m, 1H) [$(CH_2)_4$], 3.80 (s, 3H, OCH_3), 5.76 (d, J = 7.6, 1H, oxaziridinyl), 5.90 (dd, J = 11.5, 7.6, 1H, C*H*=CHCO), 6.23 (d, J = 11.5, 1H, CH=C*H*CO), 9.43 (br s, 1H, OOH)	24.5, 25.0, 32.7 and 34.4 (4 × t, 4 × CH_2), 51.9 (q, OCH_3), 69.4 (d, oxaziridinyl), 107.6 (s, C–OOH), 127.8 (d, CH=*C*HCO), 142.5 (d, *C*H=CHCO), 165.7 (s, CO_2)
4c	65	3533, 3444, 1724, 1654, 1441	1.38 (s, 3H, CH_3), 1.62 (s, 3H, CH_3), 3.80 (s, 3H, OCH_3), 5.78 (d, J = 7.7, 1H, oxaziridinyl), 5.87 (dd, J = 11.2, 7.7, 1H, C*H*=CHCO), 6.23 (d, J = 11.2, 1H, CH=C*H*CO), 8.90 (br s, 1H, OOH)	20.4 (q, CH_3), 22.2 (q, CH_3), 51.9 (q, OCH_3), 69.2 (d, oxaziridinyl), 95.9 (s, C–OOH), 127.7 (d, CH=*C*HCO), 142.5 (d, *C*H=CHCO), 165.7 (s, CO_2)
4d[d]	54	3528, 3439, 1724, 1654, 1441	0.98 (t, J = 7.6) and 1.04 (t, J = 7.4) (together 6H, 2 × C*H*$_3$CH$_2$), 1.29 (s, 3H, CH_3), 1.56 (s, 3H, CH_3), 1.64–1.92 (m, 3H) and 2.00–2.20 (m, 1H) (2 × CH_2), 3.80 (s, 6H, 2 × OCH_3), 5.72–5.95 (m, 4H, 2 × oxaziridinyl and 2 × C*H*=CHCO), 6.23 (d, J = 11.1, 2H, 2 × CH=C*H*CO), 9.28 (br s, 2H, 2 × OOH)	7.7 (q, *C*H$_3$CH$_2$), 7.8 (q, *C*H$_3$CH$_2$), 16.8 (q, CH_3), 19.4 (q, CH_3), 27.8 (t, CH_2), 28.1 (t, CH_2), 51.9 (q, 2 × OCH_3), 68.9 (d, oxaziridinyl), 69.1 (d, oxaziridinyl), 97.5 (s, C–OOH), 98.1 (s, C–OOH), 127.7 (d, 2 × CH=*C*HCO), 142.5 (d, *C*H=CHCO), 142.6 (d, *C*H=CHCO), 165.7 (s, 2 × CO_2)

Table 2. (continued)

Product[b]	Yield[c] (%)	IR ($CHCl_3$) ν (cm^{-1})	^1H NMR ($CDCl_3$/TMS) δ, J (Hz)	^{13}C NMR ($CDCl_3$/TMS) δ
4e[d]	87	3518, 3434, 1724, 1662, 1441	1.18 (s, 3H, CH_3), 1.50 (s, 3H, CH_3), 2.93 (d, J = 14.3, 1H), 3.11 (d, J = 14.3, 2H) and 3.43 (d, J = 14.3, 1H) (2 × CH_2), 3.74 (s) and 3.75 (s) (together 6H, 2 × OCH_3), 5.75–5.94 (m, 4H, 2 × oxaziridinyl and 2 × C*H*=CHCO), 6.11–6.26 (m, 2H, 2 × CH=C*H*CO), 7.20–7.40 (m, 10H, 2 × ArH), 9.22 (br s, 1H, OOH), 9.40 (br s, 1H, OOH)	17.3 (q, CH_3), 20.3 (q, CH_3), 40.8 (t, CH_2), 41.1 (t, CH_2), 51.8 (q, 2 × OCH_3), 69.3 (d, 2 × oxaziridinyl), 97.1 (s, C–OOH), 98.0 (s, C–OOH), 126.6 and 126.7 (2 × d, CH-4 of two Ar), 127.8 and 128.0 [2 × d, 2 × CH=*C*HCO and CH-2,6 (or CH-3,5) of two Ar], 130.6 and 130.7 [2 × d, CH-2,6 (or CH-3,5) of two Ar], 134.3 and 135.2 (2 × s, C-1 of two Ar), 142.1 (d, *C*H=CHCO), 142.4 (d, *C*H=CHCO), 165.4 (s, CO_2), 165.5 (s, CO_2)
4f	76[e]	*apolar isomer:* 3527, 3409, 1719, 1661, 1442	*apolar isomer:* 1.40 (d, J = 6.3, 3H, CH_3), 3.80 (s, 3H, OCH_3), 4.61 (q, J = 6.3, 1H, CH), 5.59 (d, J = 6.8, 1H, oxaziridinyl), 5.96 (dd, J = 11.6, 6.8, 1H, C*H*=CHCO), 6.24 (d, J = 11.6, 1H, CH=C*H*CO), 10.25 (br s, 1H, OOH)	*polar isomer:* 15.9 (q, CH_3), 51.9 (q, OCH_3), 72.3 (d, oxaziridinyl), 97.7 (d, CH–OOH), 127.4 (d, CH=*C*HCO), 141.9 (d, *C*H=CHCO), 165.8 (s, CO_2)
		polar isomer: 3521, 3412, 1723, 1663, 1442	*polar isomer:* 1.42 (d, J = 6.3, 3H, CH_3), 3.80 (s, 3H, OCH_3), 4.29 (q, J = 6.3, 1H, CH), 5.48 (d, J = 7.8, 1H, oxaziridinyl), 5.88 (dd, J = 11.6, 7.8, 1H, C*H*=CHCO), 6.26 (d, J = 11.6, 1H, CH=C*H*CO), 9.59 (br s, 1H, OOH)	*polar isomer:* 14.8 (q, CH_3), 52.0 (q, OCH_3), 69.7 (d, oxaziridinyl), 99.3 (d, CH–OOH), 128.0 (d, CH=*C*HCO), 142.4 (d, *C*H=CHCO), 165.9 (s, CO_2)
4g	60[e]	*apolar isomer:* 3525, 3405, 1724, 1655, 1442	*apolar isomer:* 1.05 [s, 9H, $C(CH_3)_3$], 3.81 (s, 3H, OCH_3), 4.17 (s, 1H, CH), 5.78 (d, J = 7.3, 1H, oxaziridinyl), 5.92 (dd, J = 11.7, 7.3, 1H, C*H*=CHCO), 6.25 (d, J = 11.7, 1H, CH=C*H*CO), 10.40 (br s, 1H, OOH)	*apolar isomer:* 25.8 (q, 3 × CH_3), 35.9 [s, *C*$(CH_3)_3$], 52.2 (q, OCH_3), 70.3 (d, oxaziridinyl), 106.9 (d, CH–OOH), 127.5 (d, CH=*C*HCO), 142.7 (d, *C*H=CHCO), 166.2 (s, CO_2)
		polar isomer: 3514, 3432, 1724, 1656, 1441	*polar isomer:* 1.05 [s, 9H, $C(CH_3)_3$], 3.82 (s, 3H, OCH_3), 3.84 (s, 1H, CH), 5.60 (d, J = 8.3, 1H, oxaziridinyl), 5.82 (dd, J = 11.7, 8.3, 1H, C*H*=CHCO), 6.27 (d, J = 11.7, 1H, CH=C*H*CO), 8.87 (br s, 1H, OOH)	*polar isomer:* 26.0 (q, 3 × CH_3), 36.0 [s, *C*$(CH_3)_3$], 51.9 (q, OCH_3), 71.2 (d, oxaziridinyl), 108.6 (d, CH–OOH), 128.6 (d, CH=*C*HCO), 142.2 (d, *C*H=CHCO), 165.7 (s, CO_2)
4h	65[e]	*apolar isomer:* 3525, 3407, 1723, 1666, 1442	*apolar isomer:* 3.02 (dd, J = 15.0, 7.3) and 3.09 (dd, J = 15.0, 4.4) (together 2H, CH_2), 3.78 (s, 3H, OCH_3), 4.63 (dd, J = 7.3, 4.4, 1H, CH), 5.61 (d, J = 6.8, 1H, oxaziridinyl), 5.93 (dd, J = 11.7, 6.8, 1H, C*H*=CHCO), 6.87 (d, J = 11.7, 1H, CH=C*H*CO), 7.20–7.35 (m, 5H, ArH), 10.29 (br s, 1H, OOH)	*apolar isomer:* 37.4 (t, CH_2), 52.2 (q, OCH_3), 72.1 (d, oxaziridinyl), 101.6 (d, CH–OOH), 126.9 (d, CH-4 of Ar), 127.4 (d, CH=*C*HCO), 128.5 and 129.6 (2 × d, CH-2,6, and CH-3,5 of Ar), 135.2 (s, C-1 of Ar), 141.9 (d, *C*H=CHCO), 166.0 (s, CO_2)
		polar isomer: 3516, 3407, 1724, 1660, 1441	*polar isomer:* 3.08 (d, J = 6.3, 2H, CH_2), 3.75 (s, 3H, OCH_3), 4.39 (t, J = 6.3, 1H, CH), 5.38 (d, J = 7.8, 1H, oxaziridinyl), 5.82 (dd, J = 11.7, 7.8, 1H, C*H*=CHCO), 6.22 (d, J = 11.7, 1H, CH=C*H*CO), 7.20–7.30 (m, 5H, ArH), 9.40 (br s, 1H, OOH)	*polar isomer:* 36.2 (t, CH_2), 51.9 (q, OCH_3), 70.4 (d, oxaziridinyl), 102.9 (d, CH–OOH), 126.9 (d, CH-4 of Ar), 128.1 (d, CH=*C*HCO), 128.6 and 129.5 (2 × d, CH-2,6 and CH-3,5 of Ar), 135.1 (s, C-1 of Ar), 141.9 (d, *C*H=CHCO), 165.5 (s, CO_2)
4i	45[e]	*apolar isomer:* 3524, 3397, 1723, 1659, 1442	*apolar isomer:* 3.81 (s, 3H, OCH_3), 5.34 (s, 1H, CH), 5.79 (d, J = 6.8, 1H, oxaziridinyl), 5.99 (dd, J = 11.7, 6.8, 1H, C*H*=CHCO), 6.25 (d, J = 11.7, 1H, CH=C*H*CO), 7.30–7.60 (m, 5H, ArH), 10.39 (br s, 1H, OOH)	*apolar isomer:* 52.2 (q, OCH_3), 73.0 (d, oxaziridinyl), 102.0 (d, CH–OOH), 127.6 (d, CH=*C*HCO), 127.8 and 128.8 (2 × d, CH-2,6 and CH-3,5 of Ar), 129.8 (d, CH-4 of Ar), 133.4 (s, C-1 of Ar), 141.7 (d, *C*H=CHCO), 166.0 (s, CO_2)
		polar isomer: 3516, 3416, 1724, 1661, 1442	*polar isomer:* 3.65 (s, 3H, OCH_3), 4.99 (s, 1H, CH), 5.65 (d, J = 7.8, 1H, oxaziridinyl), 5.82 (dd, J = 11.7, 7.8, 1H, C*H*=CHCO), 6.16 (d, J = 11.7, 1H, CH=C*H*CO), 7.30–7.50 (m, 5H, ArH), 9.20 (br s, 1H, OOH)	*polar isomer:* 51.8 (q, OCH_3), 69.8 (d, oxaziridinyl), 104.1 (d, CH–OOH), 127.8 (d, CH=*C*HCO), 128.5 and 130.2 (2 × d, CH-2,6 and CH-3,5 of Ar), 128.8 (d, CH-4 of Ar), 133.3 (s, C-1 of Ar), 141.4 (d, *C*H=CHCO), 165.3 (s, CO_2)

[a] All products are oils except **4a**: mp 45–47 °C (Lit.[4] mp 45–47 °C).
[b] Satisfactory microanalyses obtained: C ± 0.14, H ± 0.18, N ± 0.09.
[c] Yield of isolated product.
[d] Isolated as a ca. 1:1 diastereomeric mixture.
[e] The yield corresponds to a ca. 1:1 diastereomeric mixture.

phy. 2-Methoxyfuran (Aldrich) and tetraphenylporphyrin (TPP) (Fluka) were used without purification. The oximes were either purchased commercially or prepared according to conventional methods.

Caution! Since organic peroxides are potentially hazardous compounds, they must be handled with care. No particular difficulties were experienced in handling any of the new peroxides reported in this work.

Hydroperoxynitrones 3; General Procedure:
Hydroperoxynitrones **3** were obtained, as previously reported,[4] by TPP (3.6×10^{-4} mmol) sensitized photooxygenation of 2-methoxyfuran (**1**; 98 mg, 1 mmol) in CH_2Cl_2 (50 mL) at −20 °C in the presence of oximes **2** (5 mmol). When the reaction was complete (90 min, ^1H NMR) the solvent was removed on a rotary evaporator at r.t. and the residue was chromatographed on a short silica gel column. Elution with light petroleum/Et_2O (8:2, 1:1) gave the unreacted oximes **2** and the hydroperoxynitrones **3**, respectively. The yields, as well as the physical, analytical and spectroscopic data for the new nitrones **3b,d,e,g,h**, are reported in Table 1. The nitrones **3a,c,f,i** have been described.[4]

***N*-Hydroperoxyalkyloxaziridines 4; General Procedure:**
A solution of the hydroperoxynitrone **3** (0.5 mmol) in MeCN (25 mL) was irradiated at r.t. with a 500 W high pressure mercury lamp in a 50 mL Pyrex flask for 30 min (20 min for **3e** and **h**, 10 min for **3i**). After completion of the reaction, the solvent was evaporated and the oil residue chromatographed on a silica gel column using light petroleum/Et_2O (4:1) as eluent. Table 2 reports the yields of the pure *N*-hydroperoxyalkyloxaziridines **4a–c** and those of **4d–i** as mixtures of diastereomers. In contrast to oxaziridines **4d,e**, the isomers of the oxaziridines **4f–i** were separated by chromatography (Table 2).

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