

Note

Synthesis of novel oxaziridines derived from *trans*-4-aminocyclohexanol

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A series of novel oxaziridines **3a-f** have been synthesized from *trans*-4-aminocyclohexanol **1** which on condensation with aryl aldehydes yield aldimines **2a-f**. The imines on oxidation with *m*-chloroperbenzoic acid give oxaziridines.

In view of their practical and theoretical interest^{1,2} oxaziridines^{3,4} represent one of the class of reactive three membered heterocycles⁵, which since their discovery by Emmons remain the object of considerable study. Oxaziridine's remarkable configurational stability about nitrogen show many pairs of isolable isomers⁶. Oxaziridines are usually prepared by oxidation of imines with per acids and is presumably a non-stereoselective⁷ stepwise reaction contrary to the stereoselective epoxidation of alkenes.

We report herein the synthesis of novel oxaziridines **3a-f** (cf. Scheme I) from *trans*-4-aminocyclohexanol **1** followed by condensation with different aryl aldehydes to yield aldimines **2a-f** with *trans* stereochemistry. The aldimines **2a-f** having cycloalkyl substituent at the nitrogen atom gave only *E* isomers when oxidized with *m*-chloroperbenzoic acid since two bulky substituents make the *Z* form unfavourable. IR and ¹H NMR spectra of compounds **2a-f** showed the presence of CH=N at 1510-1575 cm⁻¹ and at δ 8.3-8.7 respectively. The formation of

compounds **3a-f** was evidenced by the absence of peak due to C=N and appearance of singlet at δ 4.0-4.2 due to oxaziridine ring proton. The compounds **2a-f** and **3a-f** were characterised on the basis of analytical and spectral data (cf. Table I).

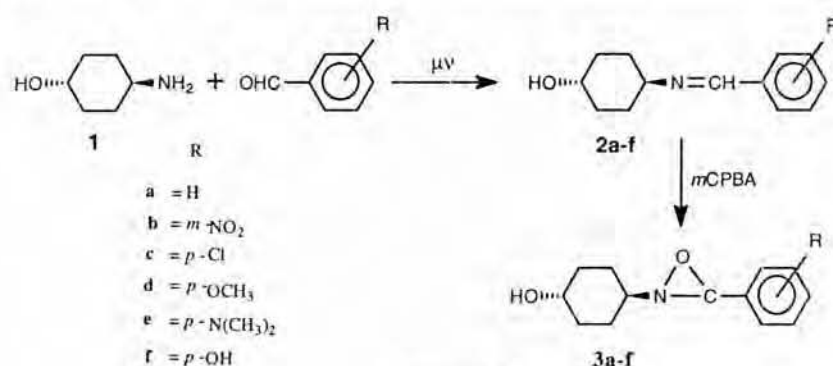
Experimental Section

Melting points were taken on a Thomas-Hoover melting point apparatus and are uncorrected. IR spectra were recorded on a Perkin-Elmer spectrophotometer model 599 using KBr discs; ¹H NMR on a Perkin-Elmer R-32 (90 MHz) instrument using TMS as internal standard. The purity of the compounds were checked on silica coated Al Plates (Merck).

trans-4-Aminocyclohexanol **1** was prepared according to the literature method⁸.

General procedure for synthesis of aldimines 2a-f. *trans*-4-aminocyclohexanol **1** (0.01 mole) and arylaldehyde (0.01 mole) were taken in ethanol (15 mL) in a 100 mL beaker. The mixture was subjected to microwave irradiation for 1.5-2.0 min at 2450 MHz. The reaction mixture was cooled and the solid separated was filtered, washed with water and recrystallized from ethanol.

Synthesis of 2-[4'-hydroxycyclohexane]-3-aryloxaziridines 3a-f. To a chilled solution of aldimines **2a-f** (0.01 mole) in DMF (20 mL), *m*-chloroperbenzoic acid (0.01 mole) was added slowly. The reaction mixture was stirred for 4-5 hr. The excess solvent was removed under reduced pressure. The residue was poured into ice cold water, and the solid separated was filtered, washed with aqueous NaHCO₃ to give oxaziridines **3a-f**.



Scheme I

Table I—Analytical and spectral data of compounds 2a-f and 3a-f

Compd	R	mp °C	Yield (%)	Found (Calcd)%			M ^r observed (expected)	¹ H NMR (CDCl ₃ + DMSO- <i>d</i> ₆) (δ, ppm)
				C	H	N		
2a	H	101-03	70	76.82 (76.84)	8.34 8.37	6.91 6.89)	202 (203)	0.90-2.20 (comp multi, 8H, C ₆ H ₈), 2.45-2.85 (m, 1H, CHOH), 3.5-3.75 (m, 1H, CHN) 4.5 (s, 1H, OH), 7.1-7.8 (m, 5H, Ar-H), 8.5 (s, 1H, N=CH)
2b	<i>m</i> -NO ₂	66-68	73	62.88 (62.90)	6.48 6.45	11.26 11.29)	248 (248)	1.0-2.2 (comp multi, 8H, C ₆ H ₈), 2.40-2.83 (m, 1H, CHOH), 3.4-3.74 (m, 1H, CHN), 4.5 (s, 1H, OH), 7.0-7.5 (m, 4H, Ar-H), 8.5 (s, 1H, N=CH)
2c	<i>p</i> -Cl	113-15	68	65.66 (65.68)	6.72 6.73	5.87 5.89)	238 (237.5)	0.88-2.1 (comp multi, 8H, C ₆ H ₈), 2.48-2.86 (m, 1H, CHOH), 3.5-3.77 (m, 1H, CHN), 4.5 (s, 1H, OH), 7.3-7.6 (m, 4H, Ar-H), 8.7 (s, 1H, N=CH)
2d	<i>p</i> -OCH ₃	106-08	65	72.12 (72.10)	8.12 8.15	6.04 6.00)	231 (233)	0.90-2.0 (comp multi, 8H, C ₆ H ₈), 2.48-2.85 (m, 1H, CHOH), 3.4-3.75 (m, 1H, CHN), 3.9 (s, 3H, OCH ₃), 4.6 (s, 1H, OH), 7.2-7.6 (m, 4H, Ar-H), 8.3 (s, 1H, N=CH)
2e	<i>p</i> -N(CH ₃) ₂	60-64	72	73.16 (73.17)	8.92 8.94	6.04 11.38)	245 (246)	1.0-2.0 (comp multi, 8H, C ₆ H ₈), 2.45-2.45 (m, 1H, CHOH), 3.5-3.73 (m, 1H, CHN), 4.5 (s, 1H, OH), 6.7 (s, 6H, N(CH ₃) ₂), 7.1-7.4 (m, 4H, Ar-H), 8.6 (s, 1H, N=CH)
2f	<i>p</i> -OH	>230	67	71.25 (71.23)	7.79 7.76	6.36 6.39)	220 (219)	0.90-2.2 (comp multi, 8H, C ₆ H ₈), 2.48-2.8 (m, 1H, CHOH), 3.5-3.76 (m, 1H, CHN), 4.6 (s, 1H, OH), 6.6 (s, 1H, Ar-OH), 7.0-7.4 (m, 4H, Ar-H), 8.5 (s, 1H, N=CH)
3a	H	163-65	60	71.21 (71.23)	7.73 7.79	6.41 6.39)	222 (219)	1.0-2.2 (comp multi, 8H, C ₆ H ₈), 2.43-2.81 (m, 1H, CHOH), 3.5-3.73 (m, 1H, CHN), 4.0 [s, 1H, CH (ring)], 4.4 (s, 1H, OH), 7.0-7.4 (m, 5H, Ar-H)
3b	<i>m</i> -NO ₂	115-17	62	59.00 (59.02)	6.08 6.06	10.63 10.60)	263 (264)	1.0-2.0 (comp multi, 8H, C ₆ H ₈), 2.45-2.87 (m, 1H, CHOH), 3.5-3.73 (m, 1H, CHN), 4.2 [s, 1H, CH (ring)], 4.6 (s, 1H, OH), 7.2-7.6 (m, 4H, Ar-H)
3c	<i>p</i> -Cl	142-44	58	61.55	6.33	5.50	252	0.90-2.2 (comp multi, 8H, C ₆ H ₈), 2.45-2.85 (m, 1H, CHOH), 3.4-3.75 (m, 1H, CHN), 4.0 [s, 1H, CH (ring)], 4.5 (s, 1H, OH), 7.1-7.6 (m, 4H, Ar-H)

(Contd.)

Table I—Analytical and spectral data of compounds 2a-f and 3a-f—(Contd.).

3d	<i>p</i> -OCH ₃	154-56	64	(61.53 67.49 67.46)	(6.30 7.65 7.63)	(5.52) 5.64 5.62)	(253.5) 249 (249)	0.90-2.0 (comp multi, 8H, C ₆ H ₈), 2.48-2.85 (m, 1H, CHOH), 3.4- 3.6 (m, 1H, CHN), 3.9 (s, 3H, OCH ₃), 4.2 [s, 1H, CH (ring)], 4.6 (s, 1H, OH), 7.2-7.5 (m, 4H, Ar-H)
3e	<i>p</i> -N(CH ₃) ₂	54-56	55	(68.73 68.70)	(8.42 8.39)	(10.64 10.68)	264 (262)	0.9-2.2 (comp multi, 8H, C ₆ H ₈), 2.48-2.83 (m, 1H, CHOH), 3.5- 3.73 (m, 1H, CHN), 4.2 [s, 1H, CH (ring)], 6.6 (s, 6H, N (CH ₃) ₂), 7.1-7.7 (m, 4H, Ar-H)
3f	<i>p</i> -OH	124-26	63	(66.35 66.38)	(7.25 7.23)	(5.99 5.95)	234 (235)	1.0-2.2 (comp multi, 8H, C ₆ H ₈), 2.48-2.85 (m, 1H, CHOH), 3.56- 3.73 (m, 1H, CHN), 4.0 [s, 1H, CH (ring)], 4.6 (s, 1H, OH), 6.6 [s, 1H, OH (Ar-OH)], 7.0-7.6 (m, 4H, Ar-H)

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