for 24 h with stirring. The mixture was then cooled to room temperature, 10 mL of ether was added, and after the mixture was stirred for another 5 min, the inorganic residue was separated by filtration through Celite. This residue was washed several times with ether, after which the filtrates were combined, dried over anhydrous magnesium sulfate, and evaporated to yield 0.37 g (2.1 mmol, 84%) of crude di-*n*-butyl sulfone, mp 35–44 °C. Recrystallization from petroleum ether (bp 30–60 °C) gave 0.34 g (1.9 mmol, 76%) of purified di-*n*-butyl sulfone [mp 42–44 °C (lit.[8] mp 43–44 °C)] which exhibited no melting point depression when mixed with an authentic sample of this compound.

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Registry No. 2-Butanol, 78-92-2; 2-nonanol, 628-99-9; 2-decanol, 1120-06-5; 5-decanol, 5205-34-5; cyclohexanol, 108-93-0; 3-methylcyclohexanol, 591-23-1; norborneol, 1632-68-4; menthol, 1490-04-6; borneol, 507-70-0; benzhydrol, 91-01-0; 1-octen-3-ol, 3391-86-4; 1-phenyl-1-buten-3-ol, 17488-65-2; 1-phenyl-1-penten-3-ol, 34862-94-7; 1-phenyl-1-hexen-3-ol, 22596-38-9; 1-phenyl-1-butyn-3-ol, 5876-76-6; 1-decanol, 112-30-1; benzyl alcohol, 100-51-6; benzaldehyde, 100-52-7; di-*n*-butyl sulfide, 544-40-1.

(8) Barnard, D. *J. Chem. Soc.* **1957**, 4547.

Pyrido[1,2-*a*]azepines. A Correction

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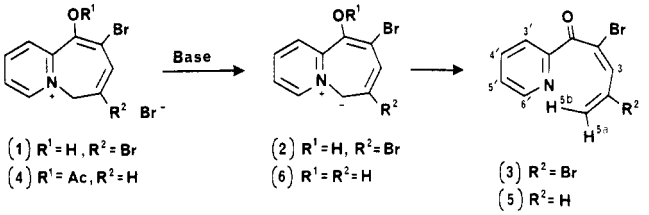
The only example of a pyrido[1,2-*a*]azepine in the literature is that reported by one of us,[1] as formed when the salt **1** was treated with aqueous base, and given the formula **2**. A renewed interest in pyridoazepines prompted us to



repeat the preparation of compound **2** and submit it to ^{13}C NMR and to the higher resolving power of 100-MHz Fourier transform ^1H NMR. The ^1H NMR spectrum now reveals that the signals at δ 5.5, 5.79, and 6.92 (assigned to protons 6, 8, and the *O*H in formula **2**) are in fact a doublet, a quartet, and a doublet and that the coupling constants are 1.8 and 1.2 Hz. More crucially, the ^{13}C NMR spectrum of the compound previously formulated as **2** shows signals at δ ($CDCl_3$) 121.2 (s, C4)[3], 123.2 (s, C2), 123.6 (t, C5), 123.7 (d, C3′), 127.5 (d, C5′), 134.3 (d, C3), 137.0 (d, C4′), 149.5 (d, C6′), 151.2 (s, C2′), and 185 (s, Cl). The most notable signals are at δ 123.6 triplet in off resonance, and a carbonyl signal at δ 185. Our original IR determination in Nujol showed no strong band higher than 1620 cm^{-1}; a determination on the chloroform extract after basification of the salt **1** shows bands at 1690, 1620, and 1610 cm^{-1}. The three new pieces of evidence establish the structure of the compound as a pyridoylbutadiene **3**. Further, treatment of compound **4** (also reported in our earlier paper[1]) with cold aqueous bases gave a red, unstable compound (characterized as its picrate) whose spectral characteristics establish it as the pyridoylbutadiene **5**. Apart from the signals due to the pyridine protons, the free base **5** showed signals at δ 5.25 (1 H, d of d, J = 9 and 2 Hz, H5a), 5.4 (1 H, d of d, J = 15 and 2 Hz, H5b), 5.9–6.7 (1 H, m, H4), 7.05 (1 H, d, J = 10 Hz, H3). We have established previously that salt **1** is bicyclic, and the ^1H NMR (Me_2SO-d_6) of the salt **4** confirmed its bicyclic nature, with signals at δ 5.1 (2 H, broad, unresolved at room temperature, d of d at −40 °C, H_6), 6.1 (1 H, d of t, H7), and 6.9 (1 H, d, J = 9.5 Hz, H8). We thus assume that the pyrido[1,2-*a*]azepines **2** and **6** have only transient existence, being unstable relative to the pyridoylbutadienes **3** and **5**; we cannot rule out the possibility that traces of the pyridoazepines are present (though undetected by NMR), since the deep red color associated with compounds **3** and **5** is hard to explain on the basis of a pyridoylbutadiene chromophore. Attempts to generate the pyridoazepine **6** by treatment of salt **4** with nonnucleophilic bases in nonprotonating solvents gave red colors, but no NMR spectra could be obtained even after prolonged accumulation.

Experimental Section[2]

1-Bromo-1-(2-pyridoyl)-1,3-butadiene (5). A solution of the salt **4** in the minimum of water was treated with a few drops of a saturated sodium bicarbonate solution (sodium carbonate and pyridine were also used successfully). A deep red color was produced, and extraction with dichloromethane gave, after drying and evaporation, a red oily solid. The ^1H NMR spectrum showed this to be almost pure diene **5**, characterized as its **picrate**: mp 152–154 °C; ^1H NMR ($CDCl_3$) δ 5.25 (1 H, d of d, J = 9 and 2 Hz), 5.4 (1 H, d of d, J = 15 and 2 Hz), 5.9–6.7 (1 H, m), 7.05 (1 H, d, J = 10 Hz), 7.4 (1 H, m), 7.9 (2 H, m), 8.7 (1 H, d of d, J = 4 and 1 Hz). Anal. Calcd for $C_{16}H_{11}BrN_4O_8 \cdot C_2H_5OH$: C, 42.4; H, 3.3; N, 10.9. Found: C, 42.5; H, 3.0; N, 11.15.

Registry No. **1**, 1532-74-7; **3**, 81625-42-5; **4**, 1532-75-8; **5**, 81625-43-6; **5** picrate, 81625-44-7.

(1) A. Fozard and G. Jones, *J. Org. Chem.*, **30**, 1523 (1965).

(2) NMR spectra were determined on a JEOL FX100 FT spectrometer.

(3) Off-resonance multiplicities are given in parentheses.

Synthesis of Allolaurinterol

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Metalation of phenolic methoxymethyl ethers is an effective means of controlling the regiochemistry of substitution in complex systems and affords a convenient entry into a class of marine sesquiterpenes from *Aplysia* species and *Laurencia* species such as allolaurinterol **1**,[1] laurinterol **2**,[2] aplysin **3**,[3] and laurene **4**.[4] These com-

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