

mixture was then poured into a saturated NH_4Cl solution (100 mL). The mixture was filtered on a pad of Celite and extracted with CH_2Cl_2 (3×100 mL). The combined organic phases were dried over Na_2SO_4 , and the solvent was removed under reduced pressure. The crude product (5.35 g) was dissolved in methanol (66 mL). To this solution were added concentrated ammonium hydroxide (22 wt %, 5 mL) and copper(II) acetate monohydrate (0.57 g, 2.85 mmol). Oxygen was bubbled through the yellow solution so obtained until a persistent deep blue color was observed. The solvent was then removed under reduced pressure and the crude product redissolved in CHCl_3 (100 mL). This solution was washed with a saturated NaHCO_3 solution (50 mL) and dried over Na_2SO_4 and the solvent removed under reduced pressure. The crude product (5.15 g) was dissolved in 50 mL of anhydrous THF and treated with PhMgBr (same quantity and conditions as above). After workup (see above) 6 g of crude product was obtained. It was oxidized by O_2 /copper acetate (same quantities, conditions, and workup as above), furnishing 7 g of brown viscous oil. Purification by column chromatography on silica gel (hexane/EtOAc, 98:2) provided nitroxide **3** (1.17 g, 24% yield from **2**, 93% ee), mp 123.5–129 °C. Two recrystallizations from hexane gave 0.71 g of **3** with ee = 99.8%: mp 132.5–133 °C; $[\alpha]_D^{21} -170.5^\circ$ (*c* 1.04, EtOAc); IR (KBr) 1601, 1496, 1444, 1416, 1372, 1269, 1063 cm^{-1} ; MS (DCI, NH_3 + isobutane) *m/z* 266 (100), 284 (16); UV-vis (0.32×10^{-3} M in CH_2Cl_2) 241 nm ($\epsilon = 2500$), 423 nm ($\epsilon = 4$); ESR (1.3×10^{-3} M in toluene) *g* = 2.0066, $a_N = 13.3$ G. Anal. Calcd for $\text{C}_{18}\text{H}_{20}\text{NO}$: C, 81.17; H, 7.56; N, 6.00. Found: C, 81.18; H, 7.72; N, 5.86.

(2S,5S)-2,5-Dimethyl-2,5-diphenylpyrrolidine (4). A mixture of 0.532 g (2 mmol) of nitroxide **3** (99.8% ee), 12 mL of water, 3 mL of concentrated hydrochloric acid, and 0.94 g (14 mmol) of zinc powder were refluxed under vigorous stirring until the yellow color of the nitroxide had disappeared (1 h). After cooling, the reaction mixture was made alkaline (pH > 12) with concentrated NaOH (30 wt %) and then extracted with Et_2O . The combined extracts were dried over Na_2SO_4 ; the solvent was removed under reduced pressure to afford 0.502 g of pure **4** as a colorless oil: bp 115 °C (0.5 mmHg). **4·HCl**: mp 165–168 °C; IR (neat) cm^{-1} 3083, 3058, 3024, 2968, 1445, 1094, 1027; ^1H NMR δ (ppm) 1.34 (6H, s), 1.73 (1H, bs), 2.13–2.27 (4H, m), 7.19–7.62 (10H, m); ^{13}C NMR δ (ppm) 32.3, 39.8, 65.4, 125.3, 125.9, 151.3. Anal. Calcd for $\text{C}_{18}\text{H}_{21}\text{N}$: C, 86.01; H, 8.41; N, 5.57. Found: C, 86.12; H, 8.50; N, 5.58.

Reoxidation of Amine 4 into Nitroxide 3. Amine **4** (25.1 mg, 0.1 mmol) was dissolved in a mixture of 1 mL of EtOH^{20} and 0.3 mL of water. To this solution were added, under stirring, 106 mg (1 mmol) of Na_2CO_3 followed by 361 mg of Oxone (0.46 mmol) in portions of ca. 50 mg every 10 min. At the end of the addition, the mixture was stirred for another 5 h. The solid was then filtered off and washed with ethanol. The combined filtrate and washings were evaporated at reduced pressure, and the residue was chromatographed on silica gel (hexane/EtOAc, 98:2), affording 19.7 mg of nitroxide **3** (74%), ee = 99.8%.¹⁶

JO971812P

Additions and Corrections

Vol. 60, 1995

Narasimhachari Narayanan, Lucjan Strekowski, Malgorzata Lipowska, and Gabor Patonay*. A New Method for the Synthesis of Heptamethine Cyanine Dyes: Synthesis of New Near Infrared Fluorescent Labels.

Page 2391. L. Strekowski and M. Lipowska should be included as authors for this paper.

Page 2395, Acknowledgment. This work was supported by a grant from LI-COR, Inc., Lincoln, NE.

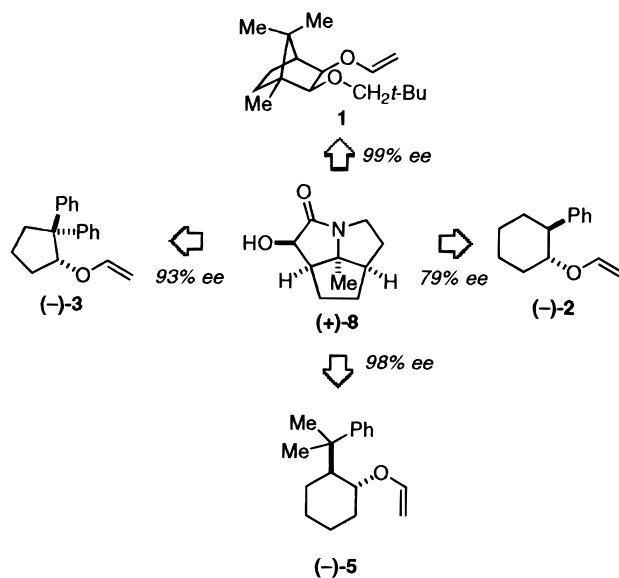
JO9740220

Vol. 61, 1996

Scott E. Denmark* and Atli Thorarensen. Tandem [4 + 2]/[3 + 2] Cycloadditions of Nitroalkenes. 10. *trans*-2-(1-Methyl-1-Phenylethyl)cyclohexanol as a New Auxiliary.

Page 6728, Figure 2. Structure **8** in Figure 2 is correctly depicted as the (1*R*) isomer for the intended correlations. However, the (1*R*) isomer is dextrorotatory and the sign of rotation shown for **8** is in error, the correct label is (+)-**8**.

JO974005Z



Leo Paquette* and Jingsung Tae. Stereocontrolled Preparation of Spirocyclic Ethers by Intramolecular Trapping of Oxonium Ions with Allylsilanes.

Page 7860. The pioneering investigations by Denmark on the stereochemistry and mechanism of allylmethyl-acetal additions were inadvertently not cited. The relevant references are as follows.

- (1) Denmark, S. E.; Willson, T. M. *J. Am. Chem. Soc.* **1989**, *111*, 3475.
 (2) Denmark, S. E.; Willson, T. M. In *Selectivities in Lewis Acid Promoted Reactions*; Schnizer, D., Ed.; Kluwer Academic Publishers: 1989; pp 247–263.

JO974032P

Vol. 62, 1997

N. André Sasaki,* Michael Dockner, Angèle Chiaroni, Claude Riche, and Pierre Potier. A Novel Stereodivergent Synthesis of Optically Pure *cis*- and *trans*-3-Substituted Proline Derivatives.

Page 766, column 1, lines 6–11, should read “While **5a** exhibits a multiplet centered at 5.70 ppm which is attributed to one of the allylic protons, its counterpart of **5b** appears somewhat downfield centered at 6.08 ppm suggesting *cis* relationship between the allyl and the hydroxymethyl groups.”

JO9740249

Shikegi Matsunaga, Toshiyuki Wakimoto, and Nobuhiro Fusetani*. Isolation of Four New Calyculins from the Marine Sponge *Discodermia calyx*.

Page 2640. Structural formulas for compounds **1–5** were inadvertently drawn in an enantiomeric form.

JO9740251

Neville P. Pavri and Mark L. Trudell*. An Efficient Method for the Synthesis of 3-Arylpyrroles.

Page 2649. The fourth sentence of the last paragraph should read “A recent report describes the synthesis of **4a** from benzonitrile in four steps and 37% overall yield⁵...”.

JO9740150

Dennis P. Arnold* and David A. James. Dimers and Model Monomers of Nickel(II) Octaethylporphyrin Substituted by Conjugated Groups Comprising Combinations of Triple Bonds with Double Bonds and Arenes. 1. Synthesis and Electronic Spectra.

Page 3468, column 1. The electronic spectral data for compound **25** were incorrect. The data should read as follows: $\text{vis } \lambda_{\text{max}} 408 \text{ nm } (\epsilon 153\,000), 442 \text{ sh } (100\,000), 530 (24\,400), 567 (30\,000), 602 \text{ sh } (21\,000)$. The spectrum displayed in Figure 6 is correct.

JO974026T

Raymond J. Cvetovich,* Chris H. Senanayake, Joseph S. Amato, Lisa M. DiMichele, Timothy J. Bill, Ji Liu, Sheo B. Singh, Robert D. Larsen, R. F. Shuman, Thomas R. Verhoeven, and Edward J. J. Grabowski. Practical Syntheses of 13-*O*-[(2-Methoxyethoxy)methyl]-22,23-dihydroavermectin B₁ Aglycon [Dimedectin Isopropanol, MK-324] and 13-*epi-O*-(Methoxymethyl)-22,23-Dihydroavermectin B₁ Aglycon [L-694,554], Flea Active Ivermectin Analogues.

Page 3989. Due to an oversight, two names (Ji Liu and Sheo B. Singh) were not included in the list of authors for this paper, for which they are fully deserving.

JO974017K

N. A. J. M. Sommerdijk, P. J. J. A. Buynsters, H. Akdemir, D. G. Geurts, R. J. M. Nolte,* and Binne Zwanenburg*. Aziridines as Synthons for Chiral Amide-Containing Surfactants.

Page 4958–9. The names of compounds **8b**, **8c**, **9b**, and **9c** should read as follows: disodium (2*S*)-3-butanoyl-2-(dodecanoylamino)propan-1-yl phosphate (**8b**), disodium (2*S*)-3-butanoyl-2-(octadecanoylamino)propan-1-yl phosphate (**8c**), disodium (2*R*)-3-butanoyl-1-(dodecanoylamino)propan-2-yl phosphate (**9b**), and disodium (2*R*)-3-butanoyl-1-(octadecanoylamino)propan-2-yl phosphate (**9c**).

JO974027L

Robert B. Grossman* and Melissa A. Varner. Selective Monoalkylation of Diethyl Malonate, Ethyl Cyanoacetate, and Malonitrile Using a Masking Group for the Second Acidic Hydrogen.

Page 5235. A relevant reference (Padgett, H. C.; Csendes, I. G.; Rapoport, H. *J. Org. Chem.* **1979**, *44*, 3492) was inadvertently omitted. The reference describes the use of triethyl methanetricarboxylate as a diethyl malonate surrogate in the alkylation of 1,2-dibromoethane and 1,4-dibromobutane. One ethoxycarbonyl group could be removed from the alkylation product using various acidic or basic conditions. Thanks to Prof. Rapoport for bringing this work to our attention.

JO974023G