

(CDCl₃) δ 8.90–7.20 (m, 10 H, aromatic and 2-pyridine H), 7.11 (s, 1 H, NH); mass spectrum *m/z* (relative intensity) (EI) *M*⁺ 249 (100).

B: From 1,5-Di-2-pyridylpentane-1,3,5-trione (4). The procedure described in the following text for the preparation of 4 is a significant improvement over that reported¹¹ in the literature. A solution of acetone (1.8 mL, 25 mmol) and ethyl 2-pyridine-carboxylate (10.1 mL, 75 mmol) in dry THF (50 mL) was added dropwise to a refluxing suspension of sodium hydride (95%, 1.9 g, 75 mmol) in dry THF (50 mL) in a nitrogen atmosphere over 4 h. After an additional reflux (2 h), the THF was removed in vacuo and the remaining orange paste carefully treated with water (100 mL). The resultant orange solution was filtered through Celite and the pH of the filtrate adjusted to pH 7 by the dropwise addition of 5% acetic acid. The resultant yellow solid was collected, washed with water, and dried. After recrystallization from 95% ethanol, the triketone was obtained as small, yellow needles: 5.4 g (80%), mp 105 °C (lit.¹⁰ mp 105 °C); IR (KBr) ν_{CO} 1611, 1560 cm⁻¹; mass spectrum *m/z* (relative intensity) (CI) (*M* + 1) 269 (100). Conversion of this triketone into the pyridone 3 was carried out using the method described in the literature.¹¹

4'-[[[(Trifluoromethyl)sulfonyl]oxy]-2,2':6',2''-terpyridine (5). A solution of 2,6-di-2-pyridyl-4(1*H*)-pyridone (498 mg, 2 mmol) in dry pyridine (5 mL) was treated slowly at 0 °C with trifluoromethanesulfonic anhydride (594 mg, 2 mmol). The resulting mixture was stirred at 0 °C (30 min), allowed to warm to room temperature (25 °C), and kept at this temperature for 48 h. It was then poured into ice-water (50 g) and stirred for 0.5 h. The light-brown solid was separated, washed with cold water (50 mL), and, after drying, dissolved in hexane (15 mL), and the insoluble portion was filtered off. Concentration of the mother liquor to ca. 5 mL and cooling gave colorless, irregular prisms of the triflate: 0.53 g (70%); mp 108 °C; IR (KBr) 1575, 1435–1405, 1245–1200, 1135, 950 cm⁻¹; ¹H NMR (CDCl₃) δ 7.3–8.8 (m, 10 H,

aromatic protons); mass spectrum (CI) *m/z* (relative intensity) (*M* + 1)⁺ 382 (100), 250 (30), 234 (16). Anal. Calcd for C₁₆H₁₀N₃O₃SF₃: C, 50.39; H, 2.64; N, 11.02. Found: C, 50.30; H, 2.47; N, 10.91.

Preparation of 4'-Vinyl-2,2':6',2''-terpyridine (6). A mixture of 4'-[[[(trifluoromethyl)sulfonyl]oxy]-2,2':6',2''-terpyridine (1.91 g, 5 mmol), vinyltributyltin (2.22 g, 7 mmol), NEt₃ (3 mL, 22 mmol), and bis(triphenylphosphine)palladium dichloride (100 mg, 0.14 mmol) in DMF (15 mL) was stirred at 90 °C for 4 h under dry nitrogen. The reaction mixture was then diluted with ice-water (100 mL), stirred for 1 h, and filtered. The light-yellow solid was washed several times with water and dried. The crude product was dissolved in diethyl ether (100 mL) and the insoluble material removed by filtration. Evaporation of the ether under reduced pressure resulted in a pale-yellow solid that was chromatographed on neutral alumina using a 9:1 hexane/ethyl acetate mixture as the eluting solvent. Removal of the solvent under reduced pressure yielded 4'-vinylterpyridine as colorless, irregular prisms: 1.1 g (86%), mp 90–91 °C (lit.¹ mp 89–91 °C); IR (KBr) 1600–1550, 1465, 1380, 990, 929, 790, 745 cm⁻¹; ¹H NMR (CDCl₃; 200 MHz) δ 8.74–7.27 (m, 10 H, aromatic), 6.89 (dd, 1 H, H_b, *J*_{H_b-H_a} = 10.9 Hz, *J*_{H_b-H_c} = 17.6 Hz), 6.22 (d, 1 H, H_a, *J*_{H_a-H_b} = 17.6 Hz), 5.56 (d, 1 H, H_c, *J*_{H_a-H_b} = 10.9 Hz); mass spectrum (CI) *m/z* (relative intensity) (*M* + 1)⁺ 260 (100). Anal. Calcd for C₁₇H₁₃N₃: C, 78.73; H, 5.06; N, 16.21. Found: C, 78.02; H, 5.15; N, 15.57.

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Additions and Corrections

Vol. 54, 1989

Mouhsine Lourak, Régis Vanderesse, Yves Fort, and Paul Caubère*. Activation of Reducing Agents. Sodium Hydride Containing Complex Reducing Agents. 32. NiCRAL's as Very Efficient Agents in Promoting Cross-Coupling of Aryl Halides.

Page 4847, column 1, paragraph 7. *t*-AmONa (20 mmol) should read *t*-AmOH (20 mmol).

Vol. 55, 1990

A. Srikrishna* and P. Hemamalini. Radical Cyclization Strategies to Bridged Systems. Synthesis of Bicyclo[3.2.1]octan-3-ones from (*S*)-Carvone.

Page 4884, Table I, column 6 ([α]_D), should read as follows:

endo	exo
-51.8	-26.0
-23.9	-10.7
-39.1	-10.7
-30.7	-12.4
-32.5	-12.7
-14.1	-5.6
-24.6	-0.3

Vol. 56, 1991

Jeffrey C. Bottaro, Paul E. Penwell, and Robert J. Schmitt*. Improved Synthesis of Cubane-1,2,4,7-tetracarboxylic Acid.

Page 1306, columns 1 and 2, compounds 1 and 2 in Scheme I and Table I should be drawn as follows:

