

in 12 mL of absolute ethanol was added 2.1 mL of concentrated HCl. The mixture was cooled to 0–5 °C and then treated with a cold solution of sodium nitrite (0.8 g, 11.6 mM) in water (11 mL), over a 5-min period, with the reaction temperature maintained at 0–5 °C. The resulting mixture was stirred for a further 20 min. The cold diazotized solution was then added dropwise to a cold mixture of Na<sub>2</sub>CO<sub>3</sub> (1.5 g, 14.2 mM) and dimethylamine (30% aqueous solution, 3 mL) in 12 mL of H<sub>2</sub>O. The resulting solution was stirred for 30 min and then acidified with concentrated HCl to pH 4.5–5.0. After 20 min, a light colored solid separated from the solution and was collected by filtration to give 250 mg (26%) of 4. The filtrate was thoroughly extracted with chloroform. The chloroform layer was dried (MgSO<sub>4</sub>) and concentrated to give 450 mg (47%) of 4 in a total yield of 73%. All attempts to recrystallize 4 failed; however, it appeared to be pure enough to use in the next step: mp 169–171 °C;<sup>7</sup> NMR (DMSO-*d*<sub>6</sub>) δ 7.63 (1 H, d, *J* = 8.4, H<sub>6</sub>), 7.52 (1 H, d, *J* = 8.4, H<sub>5</sub>), 3.58 (3 H, s, NCH<sub>3</sub>), 3.25 (3 H, s, NCH<sub>3</sub>), 2.59 (3 H, s, CH<sub>3</sub>); IR (KBr) 3480–3360, 2980, 1950, 1685, 1620, 1580, 1520, 1480, 1340, 1180, 1145, 1080, 900, 800, 780, 745, 660, cm<sup>-1</sup>; MS (CI, *i*-C<sub>4</sub>H<sub>10</sub>), *m/e* 209 (M + 1).

**3-(3,3-Diisopropyltriazen-1-yl)-2-methylpyridine-4-carboxylic Acid (9).** Compound 9 was prepared by the same method as 4 in 51% yield. It was obtained as an oil:<sup>7</sup> NMR (CDCl<sub>3</sub>) δ 7.98 (1 H, d, *J* = 8.2, H<sub>6</sub>), 7.78 (1 H, d, *J* = 8.4, H<sub>5</sub>), 5.22 (1 H, m), 4.11 (1 H, m), 2.65 (3 H, s, CH<sub>3</sub>), 1.40 (6 H, d, *J* = 6.8), 1.30 (6 H, d, *J* = 6.8); IR (neat) 3470–3365, 2980, 1950, 1690, 1620, 1570, 1180, 1080, 780 cm<sup>-1</sup>; MS (CI, *i*-C<sub>4</sub>H<sub>10</sub>), *m/e* 265 (M + 1).

(7) Satisfactory elemental analyses were not obtained, presumably owing to the rapid thermal decomposition of the triazene.

***N,N*-Dimethyl(2-acetyl-3-indolyl)acetamide (7).** (A) A suspension of 5 (320 mg, 1.56 mM) and 4-methylpyrano[3,4-*b*]indol-3-one (6)<sup>3</sup> (111 mg, 0.56 mM) in 22 mL of dry acetonitrile was refluxed for 40 h. The solvent was removed in vacuo, and the residue was chromatographed (CHCl<sub>3</sub>-MeOH) to give 53 mg (36%) of 7. Recrystallization from ethanol gave colorless crystals: mp 224–227 °C; NMR (CDCl<sub>3</sub>) δ 9.86 (1 H, br, s, NH), 7.57 (1 H, d, *J* = 8.2, H<sub>6</sub>), 7.26–7.08 (3 H, m), 4.15 (2 H, s, CH<sub>2</sub>CO), 3.28 (3 H, s, NCH<sub>3</sub>), 3.14 (3 H, s, NCH<sub>3</sub>), 1.88 (3 H, s, COCH<sub>3</sub>); IR (KBr) 3180, 1662, 1540, 1400, 1260, 1145, 745 cm<sup>-1</sup>; MS (CI, *i*-C<sub>4</sub>H<sub>10</sub>), *m/e* 245 (M + 1). Anal. Calcd for C<sub>14</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>: C, 68.85; H, 6.56; N, 11.48. Found: C, 68.78; H, 6.64; N, 11.39.

(B) A mixture of 4 (280 mg, 1.35 mM) and pyranindolone 6 (180 mg, 0.90 mM) in 20 mL of dry acetonitrile was refluxed for 50 h. Solvents were evaporated, and the dark residue was chromatographed to give 35 mg (35%) of 7, mp 223–226 °C, which was identical in all respects with the sample described above.

(C) A mixture of 6 (655 mg, 3.29 mM) in 100 mL of dry methanol, saturated with dimethylamine, was refluxed for 2 h and then allowed to cool to room temperature overnight. Methanol was removed in vacuo, and the residue was chromatographed (CHCl<sub>3</sub>-MeOH) to give 353 mg (44%) of the desired dimethyl amide, 7, mp 224–227 °C, which was identical in all respects with the sample described above.

(D) A mixture of 6 (90 mg, 0.45 mM) and 9 (250 mg, 0.95 mM) in 22 mL of dry acetonitrile was refluxed for 39 h. TLC indicated presence of the starting material only. Then 2–3 drops of trifluoroacetic acid were added, and reflux was continued for an additional 60 h. Workup as in the previous experiment (C) did not result in any identifiable product.

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## Additions and Corrections

Vol. 50, 1985

**Bryan P. Murphy and Thomas M. Schultz\*.** Synthesis and Physical Properties of 5,6-Dihydroxyindole.

Page 2791, Table I. Assignments for H<sub>2</sub> and H<sub>3</sub> should be reversed to read

H<sub>2</sub> 6.98 ppm  
H<sub>3</sub> 6.22 ppm

Vol. 53, 1988

**Francesco Fringuelli,\* Lucio Minuti, Lajos Radics, Aldo Taticchi,\* and Ernest Wenkert\*.** Diels-Alder Reactions of Cycloalkenones. 13. Reactions of 2-Cycloalkenones with (*E*)-1-Methoxy-1,3-butadiene.

Page 4607. Formula 4 should depict a C(8)–C(9) double bond.

Page 4609 (left column). Line 13 should read <sup>3</sup>J<sub>6,7A</sub> + <sup>3</sup>J<sub>6,7B</sub> = 16.0 Hz; lines 16 and 17 should read <sup>3</sup>J<sub>9A,10</sub> + <sup>3</sup>J<sub>9B,10</sub> = 6.5 Hz.

Page 4609 (right column). Lines 19 and 20 should read 5.76 (H-8), 5.93 (H-9); lines 43 and 44 should read 5.55 (H-8), 5.71 (H-12), 5.74 (H-9), 5.88 (H-13); lines 54 and 55 should read 5.44 (H-8), 5.72 (H-12), 5.74 (H-9), 5.92 (H-13).

Page 4610 (left column). Lines 30 and 31 should read 5.80 (H-8), 5.87 (H-9).

Page 4610 (right column). Lines 5–12 should read <sup>1</sup>H NMR δ 1.15 (H-11B), 1.20 (H-5B), 1.22 (H-9B), 1.24 (H-12B), 1.29 (H-7B), 1.31 (H-8B), 1.36 (H-13B), 1.37 (H-9A), 1.38 (H-7A), 1.42 (H-8A), 1.56 (H-11A), 1.71 (H-4B, H-12A), 1.84 (H-13A), 1.90 (H-4A), 2.11 (H-10), 2.31 (H-6), 2.34 (H-3B), 2.47 (H-5A), 2.49 (H-3A), 2.80 (H-14), 3.15 (OMe); <sup>13</sup>C NMR δ 19.6 (C-4), 20.1 (C-8), 24.2 (C-12), 25.3 (C-13), 26.1 (C-5), 26.2 (C-11), 28.5 (C-9), 30.6 (C-7), 32.2 (C-6), 35.9 (C-10), 40.8 (C-3), 57.2 (C-1, OMe), 89.5 (C-14), 217.6 (C-2).

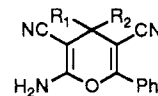
Vol. 54, 1989

**Vera M. Kolb,\* Joseph W. Stupar, Timothy E. Janota, and William L. Duax.** Abnormally High IR Frequencies for the Carbonyl Group of Semicarbazones of the Benzaldehyde and Acetophenone Series.

Page 2345. Column 1, line 13, should read as follows: The literature search revealed that no X-ray structures of semicarbazones of this series were reported, except that of benzaldehyde semicarbazone (Naik, D. V.; Palenik, G. J. *Acta Crystallogr.* 1974, B30, 2396–2401).

**Diego Armesto,\* William M. Horspool, Nazario Martin, Ana Ramos, and Carlos Seoane.** Synthesis of Cyclobutenes by the Novel Photochemical Ring Contraction of 4-Substituted 2-Amino-3,5-dicyano-6-phenyl-4H-pyrans.

Page 3069, column 2. The structure for compound 9 had the cyano groups omitted. The correct structure is given below.



- 9a: R<sup>1</sup>, R<sup>2</sup> = (CH<sub>2</sub>)<sub>4</sub>  
 b: R<sup>1</sup>, R<sup>2</sup> = (CH<sub>2</sub>)<sub>5</sub>  
 c: R<sup>1</sup> = R<sup>2</sup> = CH<sub>3</sub>  
 d: R<sup>1</sup> = (CH<sub>3</sub>)<sub>2</sub>CH, R<sup>2</sup> = H  
 e: R<sup>1</sup> = C<sub>6</sub>H<sub>5</sub>, R<sup>2</sup> = H  
 f: R<sup>1</sup> = *p*-CNC<sub>6</sub>H<sub>4</sub>, R<sup>2</sup> = H  
 g: R<sup>1</sup>, R<sup>2</sup> = (CH<sub>2</sub>)<sub>3</sub>  
 h: R<sup>1</sup> = R<sup>2</sup> = CN