

ound **29**: mp 196–198 °C (lit. mp 197–198 °C);<sup>17</sup> <sup>1</sup>H NMR (acetone-*d*<sub>6</sub>) δ 9.73 (1 H, br s, NH), 8.22, 7.98 (2 H each, d, *J* = 8.8, Ar H's), 7.89, 7.40 (2 H each, d, *J* = 8.3, Ts H's), 2.39 (3 H, s, Ts-Me), 2.32 (3 H, s, Me); <sup>13</sup>C NMR (acetone-*d*<sub>6</sub>) δ 149.0 (C=N), 144.8, 144.5, 137.3, 129.8, 130.3, 128.8, 128.0, 124.2 (Ar C's), 21.4 (Ts-Me), 13.9 (Me).

**Acetophenone (*p*-Tolylsulfonyl)hydrazone (30) and [α-(*p*-Tolylsulfonyl)ethyl]benzene (31).** These two compounds were obtained in 4:1 ratio (**30/31**) from acetophenone (*p*-tolylsulfonyl)hydrazine (**26**).<sup>8</sup> They were separated and purified by flash chromatography (20% EtOAc/hexane). The total yield was 90%. Compound **30** (white crystal, *R*<sub>f</sub> = 0.64 developed in 50% EtOAc/hexane): mp 134–136 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.94 (2 H, d, *J* = 8.3, Ts H's), 7.87 (1 H, br s, NH), 7.85–7.62 (2 H, m, Ar H's), 7.36–7.21 (5 H, m, Ts and Ar H's), 2.43 (3 H, s, Ts-Me), 2.15 (3 H, s, Me); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 152.6 (C=N), 144.2, 137.3, 135.4, 129.6, 128.3, 128.1, 126.3 (Ar C's), 21.6 (Ts-Me), 13.5 (Me); high-resolution FAB-MS calcd for C<sub>15</sub>H<sub>17</sub>N<sub>2</sub>O<sub>2</sub>S (M + H)<sup>+</sup> 289.1011, found 289.1002. Anal. Calcd for C<sub>15</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>S: C, 62.48; H, 5.59; N, 5.09; S, 11.12. Found: C, 62.55; H, 5.67; N, 5.05; S, 11.19. Compound **31** (pale yellow solid, *R*<sub>f</sub> = 0.78 developed in 50% EtOAc/hexane): <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.40 (2 H, d, *J* = 8.3, Ts H's), 7.27–7.10 (7 H, m, Ar H's), 4.20 (1 H, q, *J* = 7.2, CHS), 2.38 (3 H, s, Ts-Me), 1.74 (3 H, d, *J* = 7.2, Me); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 144.5, 142.0, 134.0–127.7 (Ar C's), 66.1 (C-S), 21.6 (Ts-Me), 14.1 (Me); high-resolution FAB-MS calcd for C<sub>15</sub>H<sub>17</sub>O<sub>2</sub>S (M + H)<sup>+</sup> 261.0949, found 261.0933. Anal. Calcd for C<sub>15</sub>H<sub>17</sub>O<sub>2</sub>S: C, 68.93; H, 6.56; S, 12.27. Found: C, 68.83; H, 6.55; S, 12.34.

**4'-Methoxyacetophenone (*p*-Tolylsulfonyl)hydrazone (32) and 1-[α-(Tolylsulfonyl)ethyl]-4-methoxybenzene (33).** These two compounds were obtained in 3:2 ratio (**32/33**) from 4'-methoxyacetophenone (*p*-tolylsulfonyl)hydrazine (**27**).<sup>8</sup> They were separated and purified by flash chromatography (10–20% EtOAc/hexane). The total yield was 81%. Compound **32** (*R*<sub>f</sub> = 0.57 developed in 50% EtOAc/hexane): mp 168–170 °C (lit. mp 169–171 °C);<sup>17</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.91, 7.30 (2 H each, d, *J* = 8.3, Ts H's), 7.59, 6.83 (2 H each, d, *J* = 8.9, Ar H's), 3.80 (3 H, s, OMe), 2.40 (3 H, s, Ts-Me), 2.10 (3 H, s, Me); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 160.8 (C=N), 152.6, 144.1, 135.5, 129.8, 129.6, 128.2, 127.8, 113.7 (Ar C's), 55.3 (OMe), 21.6 (Ts-Me), 13.3 (Me). Anal. Calcd for C<sub>16</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub>S: C, 60.36; H, 5.70; N, 8.80; S, 10.07. Found: C, 60.30;

(17) Hutchins, R. O.; Milewski, C. A.; Maryanoff, B. E. *J. Am. Chem. Soc.* **1973**, *95*, 3662.

H, 5.77; N, 8.72; S, 10.12. Compound **33** (yellow solid, *R*<sub>f</sub> = 0.73 developed in 50% EtOAc/hexane): <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.41, 7.18 (2 H each, d, *J* = 8.2, Ts H's), 7.05, 6.76 (2 H each, d, *J* = 8.7, Ar H's), 4.15 (1 H, q, *J* = 7.1, CHS), 3.77 (3 H, s, OMe), 2.38 (3 H, s, Ts-Me), 1.69 (3 H, d, *J* = 7.1, Me); <sup>13</sup>C NMR (CDCl<sub>3</sub>) δ 159.9, 144.4, 134.0, 130.6, 129.3, 129.2, 125.7, 113.8 (Ar C's), 65.4 (C-S), 55.3 (OMe), 21.6 (Ts-Me), 14.2 (Me); high-resolution FAB-MS calcd for C<sub>16</sub>H<sub>19</sub>O<sub>3</sub>S (M + H)<sup>+</sup> 291.1055, found 291.1046. Anal. Calcd for C<sub>16</sub>H<sub>18</sub>O<sub>3</sub>S: C, 66.18; H, 6.25; S, 11.04. Found: C, 66.11; H, 6.31; S, 11.09.

**1-[α-(Tolylsulfonyl)ethyl]-4-aminobenzene (34) and 1-(α-iodoethyl)-4-aminobenzene (35).** These two compounds were obtained in 4:1 ratio (**34/35**) from 4'-aminoacetophenone (*p*-tolylsulfonyl)hydrazine (**28**).<sup>8</sup> They were separated and purified by flash chromatography (30% ether/hexane) followed by preparative TLC (50% ether/hexane). The total yield was 56%. Compound **34** (*R*<sub>f</sub> = 0.58 developed in ether): <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 7.43, 7.18 (2 H each, d, *J* = 8.3, Ts H's), 6.90, 6.53 (2 H each, d, *J* = 8.5, Ar H's), 4.10 (1 H, q, *J* = 7.2, CHS), 3.69 (1 H, br s, NH), 2.39 (3 H, s, Ts-Me), 1.68 (3 H, d, *J* = 7.2, Me); high-resolution FAB-MS calcd for C<sub>15</sub>H<sub>18</sub>NO<sub>2</sub>S (M + H)<sup>+</sup> 276.1058, found 276.1046. Anal. Calcd for C<sub>15</sub>H<sub>17</sub>NO<sub>2</sub>S: C, 65.43; H, 6.22; N, 5.09; S, 11.64. Found: C, 65.35; H, 6.29; N, 5.15; S, 11.52. Compound **35** (*R*<sub>f</sub> = 0.87 developed in ether): <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 6.89, 6.26 (2 H each, d, *J* = 8.5, Ar H's), 4.29 (1 H, q, *J* = 6.7, CHI), 1.38 (3 H, d, *J* = 6.7, Me); high-resolution FAB-MS calcd for C<sub>8</sub>H<sub>11</sub>NI (M + H)<sup>+</sup> 247.9938, found 247.9928.

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**Registry No.** *galacto*-1, 122948-60-1; *gluco*-1, 122948-75-8; **2**, 4049-59-6; **3**, 122948-61-2; **4**, 113668-66-9; **5**, 113668-65-8; **6**, 122948-62-3; **7**, 122948-63-4; **8**, 122948-64-5; **9**, 122948-65-6; **10**, 122948-66-7; **11**, 122948-67-8; **21**, 1146-49-2; **22**, 91011-12-0; **23**, 67963-06-8; **24**, 51751-71-4; **25**, 122948-68-9; **26**, 60565-67-5; **27**, 122948-69-0; **28**, 122948-70-3; **29**, 41780-82-9; **30**, 4545-21-5; **31**, 24422-77-3; **32**, 32117-52-5; **33**, 122948-71-4; **34**, 122948-72-5; **35**, 122948-73-6; **36**, 56750-58-4; **37**, 67381-20-8; **38**, 122948-74-7; *N*-iodosuccinimide, 516-12-1; tosylhydrazine, 1576-35-8.

## Additions and Corrections

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**Harry H. Wasserman,\* Vincent M. Rotello, David R. Williams, and John W. Benbow.** Synthesis of the "Tricarbonyl" Region of FK-506 through an Amidophosphorane.

Page 2785, column 1. Reference 9 was inadvertently omitted during make-up of the printed version:

(9) Our use of BSA represents a modification of a procedure reported earlier: Cooke, M.; Burman, P. *J. Org. Chem.* **1982**, *47*, 4955. Cooke, M. *J. Org. Chem.* **1982**, *47*, 4963. In the coupling of ylide **3** with acid chloride **4** (outlined in Scheme II) the reaction failed to give the desired keto ylide carboxylate **5** in the absence of BSA.