charged with argon. THF and 2-methyletrahydrofuran were freshly distilled from sodium/benzophenone under Ar prior to use. Yields are reported as isolated chemical yields of purified materials based on the average of at least two runs.

Representative Nitrilation Procedure

The aryl bromide (2.5 mmol) and THF (6 mL) are added by syringe to a 25 mL round bottom flask under positive argon. The magnetically stirred solution is cooled to −94 °C (internal temperature) in a liquid N_2/hexanes slush bath, at which time fresh *tert*-butyllithium (3.0 mL, 1.7 M in hexanes, 2.0 equiv) is added slowly by syringe. The solution is maintained at −94 °C for 30 min. To a dried, 50 mL, three-neck round bottom flask is added tosyl cyanide (0.90 g, 5.0 mmol, 2.0 equiv) and dry THF (11 mL) under positive argon. The TsCN flask is magnetically stirred and cooled to an internal temperature of −94 °C. The Ph−Li solution is then transfered by cannula into the TsCN solution. Subsequently, the reaction mixture is allowed to come to room temperature, at which time the reaction is quenched with concentrated NH_4OH (3 mL) and the mixture stirred for 15 min. The mixture is then poured into 1 M NaOH (150 mL) and extracted with ether (3 × 50 mL), washed with brine (1 × 25 mL), and dried with $MgSO_4$. Products are purified and characterized as described below. Characterization of commercially available compounds by GC−MS, FT-IR, and NMR was routine. Pertinent characterizations for the remaining compounds are provided.

Each of the following compounds was purified as indicated by normal phase HPLC and subsequently found by analytical HPLC and GC to be greater than 98% pure: *m*-tolunitrile (**1b**), *o*-tolunitrile (**1c**), *o*-methoxybenzonitrile (**6c**), and 2,4,6-trimethylbenzonitrile (**2**) with 50% CH_2Cl_2:hexanes; *p*-tolunitrile (**1a**) with 15% EtOAc:hexanes; *m*-methoxybenzonitrile (**6b**) with 10% EtOAc:cyclohexane; *p*-methoxybenzonitrile (**6a**) with 15% EtOAc:cyclohexane; 4-(methylthio)benzonitrile (**7**) with CH_2Cl_2. 2,5-Di-*tert*-butylbenzonitrile (**4**) and 2,4,6-tri-*tert*-butylbenzonitrile (**5**) were purified by preparative reverse phase HPLC with 15% MeOH:water and subsequently found by analytical HPLC and GC to be greater than 98% pure. 2-cyanothiophene (**8**) was purified by flash chromatography with 15% EtOAc:hexanes and subsequently found by analytical HPLC and GC to be greater than 98% pure. 2-(3-cyanophenyl)-1,3-dioxlane (**3**) was purified by short path distillation 125 °C/0.5 mmHg or by normal phase HPLC in 15% EtOAc:cyclohexane with comparable efficieny and subsequently found by analytical HPLC and GC to be greater than 98% pure.

2,4,6-Trimethylbenzonitrile[17] (**2**): mp 50−52 °C; IR 2219 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.32 (s, 3H), 2.48 (s, 6H), 6.93 (br s, 2H).

2-(3-Cyanophenyl)-1,3-dioxolane[18] (**3**): bp 125 °C/0.5 mmHg. MS (relative intensity) *m*/*z* 176 (3), 175 ($[M]^+$ 33), 174 (100), 144 (18), 130 (42), 103 (23), 102 (26), 73 (61); ^1H NMR ($CDCl_3$) δ 4.10 (m, 4H), 5.83 (s, 1H), 7.50 (t, J = 7.8 Hz, 1H), 7.66 (td, J = 7.8, 1.5 Hz, 1H), 7.71 (td, J = 7.8, 1.5 Hz, 1H), 7.80 (t, J = 1.5 Hz, 1H). Anal. Calcd for $C_{10}H_9NO_2$: C, 68.56; H, 5.18; N, 8.00. Found: C, 68.42; H, 5.21; N, 7.94.

2,5-Di-*tert*-butylbenzonitrile[14a] (**4**): mp 62.5−63.5 °C. IR 2223 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.31 (s, 9H), 1.50 (s, 9H), 7.39 (d, J = 8.5 Hz, 1H), 7.51 (dd, J = 8.5, 2.3 Hz, 1H), 7.66 (d, J = 2.3 Hz, 1H).

2,4,6-Tri-*tert*-butylbenzonitrile[3b,14] (**5**): mp 147−148 °C. IR 2212 cm^{-1}; ^1H NMR ($CDCl_3$) δ 1.33 (s, 9H), 1.57 (s, 18H), 7.38 (s, 2H).

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(18) Takahashi, K; Tomita, S.; Murakami, H. *Jpn. Kokai Tokkyo Koho* **1993**, JP 05,230,045; *Chem. Abstr.* **1994**, *120*, 163952.

Additions and Corrections

Vol. 54, 1989

Philip Hughes* and Jon Clardy. Total Synthesis of 3(*S*)-Carboxy-4(*S*)-hydroxy-2,3,4,5-tetrahydropyridazine, an Unusual Amino Acid Constituent of Luzopeptin A.

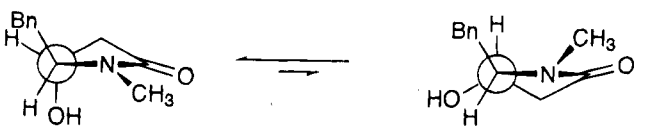
Page 3260. The optical rotation for the title compound **2** was incorrectly reported to be minus. The rotation of **2** should have been reported as $[\alpha]^{25} = +57.5°$ (c = 53 mg/mL, MeOH).

JO954004B

Vol. 59, 1994

Klemens Stratmann, David L. Burgoyne, Richard E. Moore,* Gregory M. L. Patterson, and Charles D. Smith. Hapalosin, a Cyanobacterial Cyclic Depsipeptide with Multidrug-Resistance Reversing Activity.

Page 7222. Since **5a** is the major conformer and **5b** is the minor conformer, the arrows in the equation should be reversed as follows.



5a **5b**

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