13.9, 7.1 Hz), 4.44 (s, 2 H), 3.95−3.85 (m, 1 H), 3.60−3.50 (m, 1 H), 1.85−1.42 (m, 6 H); MS(EI) *m/z* 396 (M^+, 1.3%), 171 (53%), 141 (62%), 128 (63%), 77 (100%), HRMS (EI) calcd for $C_{23}H_{24}O_4S$: 396.1396, found 396.1395.

Acknowledgment. We thank the National Science Council of Republic of China for financial support of this work.

Supporting Information Available: ^{1}H NMR spectra of compounds **1**, **8**, and **9** (3 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

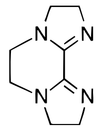
JO970513X

Additions and Corrections

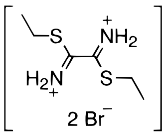
Vol. 61, 1996

Gary R. Weisman* and David P. Reed. A New Synthesis of Cyclen (1,4,7,10-Tetraazacyclododecane).

Pages 5186−5187. Subsequent to publication, we discovered that the reported reaction giving bis-amidine cyclen precursor **2**, while viable, does *not* involve the intermediacy of bis-thioimido ester salt **3**. Isolation and solution NMR studies have shown that dithiooxamide is not detectably alkylated by bromoethane under the reaction conditions employed by us and originally described by Wang and Bauman for the synthesis of 2,2′-bi-2-imidazoline (Wang, J. C.; Bauman, J. E., Jr. *Inorg. Chem.* **1965**, *4*, 1613). In fact, dithiooxamide and triethylenetetraamine cleanly react in the absence of EtBr to give **2**. The sulfur-containing gaseous byproduct of the reaction is therefore H_2S, *not ethanethiol* as reported by us and by Wang and Bauman. The reaction is analogous to the direct reaction of dithiooxamide and ethylenediamine to give 2,2′-biimidazoline, originally reported by Forssell (Forssell, G. *Chem. Ber.* **1891**, *24*, 1846).



2



3

As a consequence of these observations, we have developed a modified preparation of **2**, which should be substituted for the preparation originally reported. The revised procedure (below) is experimentally simpler, less expensive, and gives **2** in higher yield (81%). The overall yield of the two-step synthesis is improved to 67%.

2,3,5,6,8,9-Hexahydrodiimidazo[1,2-*a*:2′,1′-*c*]pyrazine (2). [**Caution:** Hydrogen sulfide (toxic) is evolved. This procedure must be carried out in an efficient hood with provisions for H_2S trapping]. A three-neck flask equipped with a fritted gas dispersion tube (initially closed) and reflux condenser was charged with dithiooxamide (3.26 g, 27.1 mmol). The N_2 manifold exit line was routed through two fritted gas washing bottles charged with 20% aqueous NaOH to trap H_2S. (An additional trap consisting of a tube packed with acid gas absorbing carbon granules may be used as a further precaution if desired.) A solution of triethylenetetraamine (3.97 g, 27.1 mmol) in absolute EtOH (33 mL) was added in one portion, the heterogeneous mixture was refluxed under N_2 for 4 h, and the reaction mixture was cooled to rt. Residual H_2S and NH_3 were purged from the solution by entrainment with N_2, which was bubbled through the reaction mixture (fritted gas dispersion tube) for 2 h. Most EtOH was removed by simple distillation (water aspirator) in the hood, $CHCl_3$ (100 mL) was added, and the solution was concentrated by rotary evaporation. Solid residue was taken up in boiling toluene (100 mL) and filtered through a glass wool plug (short-stemmed funnel). Insoluble material was washed with additional boiling toluene (75 mL), and the combined filtrates were concentrated to afford 4.34 g (98%) of crude yellow product (mp 144−146 °C). Sublimation (95 °C, 0.05 Torr) gave 3.59 g (81%) of white solid product; mp 149−150 °C (>98% by NMR; suitable for conversion to **1**). Resublimation gave material having mp 150−151 °C. Characterization as originally reported. (Note: **2** is unstable toward hydrolysis and should be stored in a desiccator.)

JO974002M

Clifton K.-F. Shen, Kuo-Ming Chien, Chiun-Gung Juo, Guor-Rong Her, and Tien-Yau Luh*. Chiral Bisazafulleroids.

Page 9242. The correct address is Republic of China, not People's Republic of China.

JO9740047