thiobisamines are more sensitive to the presence of water than the thiobisamine from morpholine.

Experimental Section

Preparation of 1 from Bromine, Thiosulfate, and an Amine. With piperidine or morpholine, a solution of 3.2 g (20 mmol) of bromine in 10 mL of methylene chloride (or hexane) was added slowly at 5–10 °C to a stirred suspension of 2.48 g (10 mmol) of sodium thiosulfate pentahydrate in a solution containing 75 mmol of the amine in 50 mL of CH_2Cl_2 (or hexane). In the case of diethylamine or dimethylamine, where the product thiobisamines are liquids rather than solids, the reaction was carried out using the same procedure, but on a larger scale: 16.0 g (100 mmol) of bromine in 50 mL of solvent; 12.4 g (50 mmol) of $Na_2S_2O_3{\cdot}5H_2O$; and ~350 mmol of amine in 500 mL of solvent. The reaction mixture was allowed to warm to room temperature, stirred for several hours, and filtered (removing the sodium sulfate and amine hydrobromide that had separated), and the organic solvent was evaporated under reduced pressure to leave crude thiobisamine. This material was then purified as outlined below.

4,4′-Thiobismorpholine (1a). Recrystallization of the residue from methanol gave 1.7 g (83%) of **1a**, mp 126–127 °C (lit.[13] mp 125–126 °C); ^{1}H NMR ($CDCl_3$) δ 3.64 (m, 8 H), 3.28 (m, 8 H).

Thiobispiperidine (1b). Recrystallization from methanol gave 1.25 g (62%) of **1b**, mp 75–76 °C (lit.[5b] mp 74 °C, lit.[14] mp 76–77 °C); ^{1}H NMR ($CDCl_3$) δ 3.26 (m, 8 H), 1.53 (m, 12 H).

Thiobis(diethylamine) (1c). Distillation of the residue under reduced pressure gave 5.32 g (61%) of **1c** as a colorless liquid, bp 80–81 °C (11 mm) (lit.[5a] bp 87–87.5 °C (19 mm), lit.[15] bp 39–40 °C (4 mm); ^{1}H NMR ($CDCl_3$) δ 3.06 (q, J = 7 Hz, 8 H), 1.14 (t, J = 7 Hz, 12 H).

Thiobis(dimethylamine) (1d). Distillation of the residue under reduced pressure gave 4.02 g (67%) of **1d** (bp 58 °C (55 mm)) as a colorless liquid that crystallized on cooling to a low-melting solid, mp 20–23 °C (lit.[13] mp 20 °C, lit.[14] mp 22–23 °C); ^{1}H NMR ($CDCl_3$) δ 3.01 (s).

Reaction of Morpholine with Sodium Tetrathionate. In Methylene Chloride. Sodium tetrathionate dihydrate (3.06 g, 10 mmol) was added to a solution of 4.36 g (50 mmol) of morpholine in 50 mL of CH_2Cl_2, and the suspension was stirred for 6 h at room temperature. The mixture was filtered, and the CH_2Cl_2 was removed under reduced pressure. To the residue, which was a colorless oil, was added 15 mL of pentane, and the 4,4′-thiobismorpholine (**1a**) that precipitated was filtered off and washed with a little pentane to give 0.23 g (11%) of **1a**, mp 126 °C.

In Water–Methylene Chloride. Sodium tetrathionate (3.06 g, 10 mmol) in 20 mL of water and 3.48 g (40 mmol) of morpholine in 20 mL of CH_2Cl_2 were stirred for 6 h at room temperature. The organic layer was dried, and the solvent was evaporated. Recrystallization of the residue from methanol gave 1.53 g (75%) of **1a**, mp 127 °C.

Formation of 1a Using Sodium Periodate as the Oxidizing Agent. A solution of sodium periodate (4.26 g, 20 mmol) in 10 mL of water was added slowly to a stirred mixture of 2.48 g (10 mmol) of sodium thiosulfate pentahydrate, 0.2 g (0.54 mmol) of tetrabutylammonium iodide in 15 mL of water, and 2.0 g (23 mmol) of morpholine in 30 mL of CH_2Cl_2. The reaction mixture was stirred for 8 h at room temperature. The mixture was then filtered, and the CH_2Cl_2 layer was dried (Na_2SO_4) and evaporated under reduced pressure to give a crystalline residue that was washed with a little pentane (to remove excess morpholine) and then recrystallized from methanol giving 0.96 g (47%) of **1a**, mp 124–126 °C.

Registry No. **1a**, 5038-11-9; **1b**, 25116-80-7; **1c**, 3768-61-4; **1d**, 2129-20-6; Et_2NH, 109-89-7; Me_2NH, 124-40-3; morpholine, 110-91-8; piperidine, 110-89-4; bromine, 7726-95-6; sodium thiosulfate, 7772-98-7; sodium tetrathionate, 10101-86-7; sodium periodate, 7790-28-5.

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Additions and Corrections

Vol. 56, 1991

Eietsu Hasegawa,* Kenyuki Ishiyama, Takaaki Horaguchi, and Takahachi Shimizu. Exploratory Study on Photoinduced Single Electron Transfer Reactions of α,β-Epoxy Ketones with Amines.

Page 1632, Scheme I. ICS should be ISC.

Page 1632, ref 16a. ΔG values should be −13 to −14 kcal/mol.

Page 1632, ref 17. The scan rate of cyclic voltammetry should be 200 mV/s.

Page 1634, ref 28. The last sentence should read "When excess TEA is present in the reaction solution, $BDMAP^{\bullet+}$ could be reduced to BDMAP by SET from TEA although this process is somewhat endothermic."

James A. Marshall* and William J. DuBay. A Novel Synthesis of Furans by Base-Catalyzed Isomerization of Alkynyloxiranes.

Page 1685. A highly relevant paper by Arens and co-workers (Rompers, J. A.; Hoff, S.; Montijn, P. P.; Brandsma, L.; Arens, J. R. *Recl. Trav. Chim. Pays-Bas* **1969**, *88*, 1445) describing the base-catalyzed conversion of 2-methoxy-2,3,4-pentatrienols to furans should be included with ref 3. We thank Professor R. L. Danheiser (MIT) for calling this paper to our attention.