

335–338 °C dec (sealed tube);  $^1\text{H}$  NMR (90 MHz,  $\text{CDCl}_3$ )  $\delta$  1.93 (br s, 36 H), 2.87 (br s, 6 H);  $^{13}\text{C}$  NMR (22.5 MHz,  $\text{CDCl}_3$ )  $\delta$  28.4 (d), 37.5 (t), 39.2 (d), 39.4 (t), 106.9 (s), 130.1 (s); UV ( $\text{C}_6\text{H}_{12}$ )  $\lambda_{\text{max}}$  282<sub>sh</sub> (log  $\epsilon$  4.08), 298<sub>sh</sub> (4.41), 312 (4.58), 329 (4.50); HRMS  $m/z$  ( $M^+$ ) calcd for  $\text{C}_{33}\text{H}_{42}$  438.3284, found 438.3262. Anal. Calcd for  $\text{C}_{33}\text{H}_{42}$ : C, 90.35; H, 9.65. Found: C, 90.12; H, 9.77.

**Reaction of Tris(2-adamantylidene)cyclopropane (4) with TCNE.** A solution of 4 (0.0217 g, 0.0495 mmol) and TCNE (0.0769 g, 0.600 mmol) in dichloromethane (10 mL) was kept at room temperature for 4 h. Concentration under reduced pressure and chromatography over silica gel (column size: diameter 1 cm, length 20 cm) with benzene as eluent gave 7 as a colorless solid (0.0224 g, 81.3%): mp 261.5–263 °C dec;  $^1\text{H}$  NMR (270 MHz,  $\text{CDCl}_3$ )  $\delta$  1.71–2.10 (m, 32 H), 2.26 (s, 1 H), 2.30 (s, 1 H), 2.37 (s, 2 H), 2.67 (s, 3 H), 2.74 (s, 1 H), 3.15 (s, 2 H);  $^{13}\text{C}$  NMR (67.8 MHz,  $\text{CDCl}_3$ )  $\delta$  26.5 (d), 26.7 (d), 27.4 (d),  $^{14}$  27.6 (d),  $^{14}$  32.3 (t), 33.9 (d),  $^{14}$  34.2 (d),  $^{14}$  35.0 (t), 35.4 (d), 36.2 (t), 36.4 (d), 36.6 (t), 37.7 (t), 37.8 (t), 38.0 (t), 38.2 (t), 39.4 (t), 44.2 (s), 51.4 (s), 61.3 (s), 77.2 (s), 102.5 (s), 111.8 (s), 112.4 (s), 115.0 (s), 156.0 (s), 193.7 (s); UV ( $\text{C}_6\text{H}_{12}$ )  $\lambda_{\text{max}}$  256 nm (log  $\epsilon$  4.05); IR (KBr) 2244 (CN), 1967  $\text{cm}^{-1}$ ; MS  $m/z$  568 ( $M+2$ ) $^+$  (10), 567 ( $M+1$ ) $^+$  (42), 566 ( $M^+$ ) (96), 514 (48), 440 (35), 294 (100), 148 (40), 135 (37), 97 (35), 86 (79), 85

(14) One of these four signals is assumed to be due to two overlapping methine signals.

(75). Anal. Calcd for  $\text{C}_{39}\text{H}_{42}\text{N}_4$ : C, 82.65; H, 7.47%. Found: C, 82.45; H, 7.68.

**Cyclic Voltammetry.** Cyclic voltammograms were obtained by the use of a Hokuto-Denko HA104 potentiostat, an HB107A function generator, a Hitachi 057 X-Y recorder, and an air-tight three-electrode cell composed of platinum-wire working and counter electrodes and a silver-wire reference electrode. The sample solution was prepared with predistilled solvent directly transferred by vacuum distillation into the cell connected to a vacuum line. The cell was then sealed under vacuum and subjected to the measurement. The observed potential was corrected with reference to ferrocene ( $E_{1/2} + 0.374$  V vs SCE) added as an internal standard immediately after each measurement.

**Acknowledgment.** We are grateful to Professor M. Iyoda of Tokyo Metropolitan University for valuable suggestions and encouragement.

**Supplementary Material Available:** UV-visible spectra for charge-transfer bands of 2-TCNE, 3-TCNE, and 4-TCNE (1 page). 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.

## Additions and Corrections

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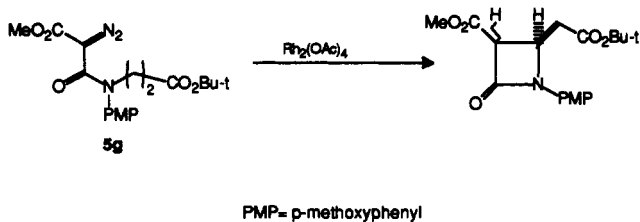
Scott C. Berk and Stephen L. Buchwald\*. An Air-Stable Catalyst System for the Conversion of Esters to Alcohols.

Page 3751. **Caution:** We have recently described an air-stable catalyst system for the conversion of esters to alcohols which uses a catalytic amount of  $\text{Ti}(\text{O}-i\text{-Pr})_4$  and  $\text{HSi}(\text{OEt})_3$  as the stoichiometric reductant. In our communication (*J. Org. Chem.* 1992, 57, 3751), we warned that, in the absence of substrate and under an inert atmosphere,  $\text{HSi}(\text{OEt})_3$  is disproportionated by  $\text{Ti}(\text{O}-i\text{-Pr})_4$  to form  $\text{SiH}_4$ , a pyrophoric gas. It has come to our attention that silane production may occur even in the presence of substrate, especially if the reaction is run on a large scale or a large excess of silane is used. A user of the procedure has informed us that he attempted to perform the reduction of methyl 11-bromoundecanoate (25 g, 90 mmol) using  $\text{HSi}(\text{OEt})_3$  (51.4 g, 313 mmol, 3.5 equiv). He first added the reagents to the reaction vessel, which was flushed with nitrogen, and the reaction was vented to an oil bubbler. After heating the reaction mixture to 50 °C, he reports that an exothermic reaction started and the temperature rose to 75 °C. The reaction mixture was then cooled to 40 °C, and on removing the cooling bath, the temperature of the reaction mixture rapidly rose to about 90 °C. During this time, an extremely pyrophoric gas (probably  $\text{SiH}_4$ ) was given off, which resulted in several fires and an explosion. At MIT, we have run this reaction on scales of up to 50 mmol, but

without event when run under an atmosphere of dry air, which we surmise may quench any  $\text{SiH}_4$  as soon as (or before) it is formed. When the reaction is run under an inert atmosphere, the  $\text{SiH}_4$  can build up, especially after the reduction reaction is complete, leading to fires when the mixture is finally exposed to air. We strongly urge those who are contemplating the use of this procedure to be alert to the possibility of  $\text{SiH}_4$  formation and possible exotherms and to take suitable precautions.

Andrew G. H. Wee,\* Baosheng Liu, and Lin Zhang. Dirhodium Tetraacetate Catalyzed Carbon-Hydrogen Insertion Reaction in N-Substituted  $\alpha$ -Carbomethoxy- $\alpha$ -diazoacetanilides and Structural Analogues. Substituent and Conformational Effects.

Page 4406, column 2, structure 5g. The brackets for the methylene unit were inadvertently omitted. The corrected structure is shown below



Page 4407, Chart I. The correct drawing for the carbonyl ylide formed from 5f is shown below.