K and was unobservable at 183 K. These data are consistent with hindered rotation about the Bi–N bond at low temperature although we have not been able to obtain limiting spectra. In ref 3 we reported that the ^{1}H NMR spectrum of $[Bi\{N(SiMe_3)_2\}_3]$ (**4**) was also significantly broadened at 190 K. We have now obtained better data on this complex, which give an activation energy for Bi–N bond rotation of 41 (2) kJ mol^{-1} (see Experimental Section).

Experimental Section

Preparation. A solution of $Li[NMe_2]$ (1.20 g, 24.0 mmol) in THF (15 mL) was added to a stirred solution of $BiCl_3$ (2.47 g, 7.8 mmol) in THF (15 mL) cooled to −78 °C (dry ice/ethanol bath), which resulted in the appearance of a yellow coloration. The mixture was stirred for 1 h and allowed to warm to room temperature after which time all volatiles were removed by vacuum, resulting in a yellow-green oily residue. Extraction with hexane (20 mL) followed by filtration through Celite afforded a yellow solution. Removal of all volatiles by vacuum from this yellow filtrate and sublimation onto a cold finger at dry ice temperature afforded **1** as a yellow crystalline solid (1.66 g, 62%). The best conditions for sublimation are using a pressure of about 10^{-2} Torr and warming the flask to 30 °C. X-ray quality crystals were obtained in the same way although it is better to use smaller quantities of material and to crystallize them directly onto the sides of the flask. In contrast to the report of Ando et al.,[4] we did not find it necessary to reflux the reaction mixture, which factor may be responsible for the higher yields obtained. Moreover, lower temperature sublimation is preferable to a higher temperature distillation as a means of purifying **1**. Bismuth analysis for the sublimed material (by EDTA titration): Calcd for $C_6H_{18}N_3Bi$, 61.3; found, 60.4%.

We have observed that **1** is rather light sensitive, especially in bright sunlight, turning black on exposure, although it can be handled under normal laboratory conditions for short periods without noticeable decomposition. It is best stored in a freezer in the dark, under which conditions it is stable for months.

X-ray Crystallography. Crystal data for **1** at 240 K: crystal dimensions 0.62 × 0.27 × 0.19 mm, M_r = 170.60; orthorhombic, a = 7.646 (2), b = 15.114 (4), c = 8.726 (2) Å, V = 1008.3 Å^3; space group *Pnma*, Z = 4; D_c = 2.247 g cm^{-3}; μ (Mo Kα) = 17.38 mm^{-1}. Data were measured on a Stoe–Siemens diffractometer using Mo Kα radiation (λ = 0.71073 Å) (graphite monochromated), ω/θ scan mode, with $2\theta_{max}$ = 50°, *hkl* ranges 0–9, 0–18, and 0–10, respectively. Due to rapid linear decay of intensity, only one unique set of 926 reflections were used, of which 683 had $F > 4\sigma_c(F)$. Data were corrected for Lorentz/polarization effects and absorption (empirically; ratio of maximum:minimum transmission = 3.40), using DIFABS[12] after all non H atoms were found and refined isotropically. The structure was solved by direct methods[13] with atomic scattering factors taken from ref 14. Anisotropic thermal motion parameters for all non H atoms were refined to minimize $\sum w\Delta^2$, $\Delta = |F_o| - |F_c|$, $w^{-1} = \sigma^2(F) = \sigma_c^2(F) + 109 - 72G + 1324G^2 - 336S + 350S^2 - 652GS$ ($G = F_o/F_{max}$, $S = (\sin\theta)/(\sin\theta_{max})$).[15] H atoms were refined at idealized positions with C–H = 0.96 Å, H–C–H = 109.5°, and U(H) = $1.2U_{eq}$(C). For 50 refined parameters, R = 0.061, R' = 0.048, and goodness of fit = 1.26. An isotropic secondary extinction coefficient x was refined to 6 (3) × 10^{-7}, whereby $F'_c = F_c/(1 + xF_c^2/(\sin 2\theta))^{1/4}$. Largest features in final difference synthesis were next to the Bi atom.

NMR Data for 4 Obtained in Toluene-d_8. 500-MHz data (Bruker AMX 500): ^{1}H (293 K) 0.55, (190 K) 0.74, 0.53, T_c = 210 K, ΔG^* = 41.3 kJ mol^{-1}; $^{13}C\{^1H\}$ (293 K) 7.51, (190 K) 8.24, 6.13, T_c = 215 K, ΔG^* = 40.7 kJ mol^{-1}. 300-MHz data (Bruker WM 300): ^{1}H (297 K) 0.54, (185 K) 0.70, 0.54, T_c = 208 K, ΔG^* = 42.2 kJ mol^{-1}; $^{13}C\{^1H\}$ (290 K) 7.48, (195 K) 8.07, 6.00, T_c = 210 K, ΔG^* = 40.6 kJ mol^{-1}. ΔG^* was calculated from $\Delta G^* = RT_c(22.96 + (\ln T_c)/\Delta\nu)$. R = 8.314 J K^{-1} mol^{-1}.

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Registry No. 1, 57403-58-4; **4**, 76505-24-3; $Li[NMe_2]$, 3585-33-9; $BiCl_3$, 7787-60-2.

Supplementary Material Available: Tables of hydrogen positional parameters and anisotropic thermal parameters (1 page); a table of observed and calculated structure factors (3 pages). Ordering information is given on any current masthead page.

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Additions and Corrections

1991, Volume 30

Habib Nasri, Yaning Wang, Boi Hanh Huynh,* F. Ann Walker,* and W. Robert Scheidt*: Reactions of Bis(nitro)($\alpha,\alpha,\alpha,\alpha$-tetrakis(*o*-pivalamidophenyl)porphinato)iron(III) with Pyridine and Imidazole. EPR and Mössbauer Spectra and Molecular Structures of the Mixed-Ligand Species.

Page 1486. Footnote *b* of Table V is incorrectly printed. The footnote should have read as follows: $\Delta/\lambda = E_{xz} - E_{xy} - (1/2)V/\lambda = g_x/(g_z + g_y) + g_z/(g_y - g_x) - (1/2)V/\lambda$ = tetragonality of Blumberg and Peisach. Nonetheless, all values in the table were calculated correctly.—W. Robert Scheidt