Complex **26a**: yellow oil; MS, *m/e* (relative intensity) 466 (83%), 365 (15), 336 (33), 326 (21), 253 (28), 222 (6), 57 (100); HRMS calcd for $C_{26}H_{39}CoSi_2$ 466.1922, found 466.1925. Anal. ($C_{26}H_{39}CoSi_2$) C, H. Complex **26b**: yellow oil; MS, *m/e* (relative intensity) 466 (100%), 368 (18), 336 (37), 326 (28), 222 (7), 253 (41); HRMS calcd for $C_{26}H_{39}CoSi_2$ 466.1922, found 466.1920. Anal. ($C_{26}H_{39}CoSi_2$) C, H.

Cocyclization of (Trimethylsilyl)ethyne with 1-(Triethylsilyl)-3-phenyl-1-butyne. A mixture of $CpCo(CO)_2$ (0.90 g, 5 mmol), (trimethylsilyl)ethyne (0.49 g, 5 mmol), and octane (15 mL) was added (0.26 mL h^{-1}) to a boiling mixture of 1-(triethylsilyl)-3-phenyl-1-butyne in octane (30 mL). Workup as in the synthesis of **26** gave **27a,b** (1.82 g, 78%) separable by HPLC to give first **27a**: yellow oil; MS, *m/e* (relative intensity) 466 (100%), 368 (19), 336 (61), 326 (29), 253 (32), 222 (12); HRMS calcd for $C_{26}H_{39}CoSi_2$ 466.1922, found 466.1916. Anal. ($C_{26}H_{39}CoSi_2$) C, H. Subsequently **27b**: yellow oil; MS, *m/e* (relative intensity) 466 (100%), 368 (17), 336 (33), 326 (27), 253 (29), 222 (4); HRMS calcd for $C_{26}H_{39}CoSi_2$ 444.1922, found 466.1920.

Cocyclization of 1-(Triethylsilyl)-2-(trimethylsilyl)ethyne and 3-Phenyl-1-butyne. $CpCo(CO)_2$ (0.90 g, 5 mmol), the bis(silyl)ethyne[34] (30 mL), and 3-phenyl-1-butyne (0.65 g, 5 mmol) were cocyclized as in the above preparation of **26** and **27**. A similar workup and HPLC separation gave **26a,b** and **27a,b** the identity of which was ascertained by coinjection on HPLC with authentic samples, isolation, and spectroscopic comparison.

Acknowledgment. This work was jointly supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division, and the Assistant Secretary for Fossil Energy, Office of Coal Research, Liquefaction Division, of the U.S. Department of Energy under Contract No. W-7405-ENG-48 through the Pittsburgh Energy Technology Center, Pittsburgh, PA, and in part by NSF (CHE 79-03954 and 82-00049). K.P.C.V. was a Camille and Henry Dreyfus Teacher-Scholar (1978–1983). G.V. was a NATO Postdoctoral Fellow, 1980–1981. M.J.W. was an SRC-NATO Postdoctoral Fellow, 1978–1980. A generous gift of btmse from Silar Laboratories is gratefully acknowledged.

Registry No. **2a**, 78736-24-0; **2b**, 78821-50-8; **3a**, 90412-53-6; **3b**, 90458-27-8; **4a**, 78736-25-1; **4b**, 78821-51-9; **5a**, 78207-58-6; **5b**, 78247-00-4; **6**, 2028-63-9; **7**, 17869-76-0; 8, 18857-02-8; **9**, 4544-28-9; **10**, 90412-54-7; 11, 90412-55-8; **12**, 90458-30-3; **14**, 78341-15-8; **17a**, 90412-56-9; **17b**, 90458-28-9; **18a**, 90412-57-0; **18b**, 90458-29-0; **20**, 65018-52-2; **21**, 90412-58-1; **22**, 90432-12-5; **23**, 90412-59-2; **24**, 90412-60-5; **26a**, 78736-26-2; **26b**, 79026-86-1; **27a**, 78736-27-3; **27b**, 79026-87-2; btmse, 14630-40-1; 1-(trimethylsilyl)-3-phenylbut-1-yne, 28129-04-6; (triethylsilyl)ethyne-$^{13}C_2$, 68285-45-0; ethyne-$^{13}C_2$, 35121-31-4; 3-phenyl-1-butyne, 4544-28-9; 3-phenyl-1-pentyne, 69153-22-6; methyl propynoate, 922-67-8; (triethylsilyl)ethyne, 1777-03-3; (trimethylsilyl)ethyne, 1066-54-2; 1-(triethylsilyl)-3-phenyl-1-butyne, 90412-52-5; 1-(triethylsilyl)-2-(trimethylsilyl)ethyne, 23183-97-3.

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A Novel Type of Cyclopentadienyl Bridge—Structure of (μ-C_5H_5)(μ-$N(SiMe_3)_2$)(ZnC_5H_5)$_2$

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The compound $Cp_3Zn_2N(SiMe_3)_2$ was prepared from Cp_2Zn and $Zn[N(SiMe_3)_2]_2$ and characterized by X-ray diffraction analysis. It crystallizes in the monoclinic system of space group $P2_1/c$ with four molecules in a cell of dimensions a = 15.447 (3) Å, b = 11.049 (1) Å, c = 14.503 (3) Å, and β = 108.35 (2)°. The structure refinement, with anisotropic thermal parameters for the non-hydrogen atoms, converged at $R_F = 0.045$ ($R_{wF} = 0.049$) for 336 parameters and 3424 observed reflections with $I > 2.5\sigma(I)$. The molecule consists of two zinc atoms connected by a bridging cyclopentadienyl group and a bridging amide group; in addition, each zinc atom carries a terminal Cp group. This is the first example of a compound containing a Cp group that bridges between nonbonded metal atoms located on the same side of the ring plane. The Cp–Zn arrangements suggest that both σ- and π-type interactions contribute to the Cp–Zn bonds. In solution, intramolecular exchange of Cp groups occurs at elevated temperature, presumably via opening of the Cp bridge.

Introduction

Electron-deficient cyclopentadienyl compounds of the main-group metals are usually associated via bridging cyclopentadienyl groups. In these compounds, two metal atoms are located on different sides of the ring plane. The bridging mode can be symmetrical, as in CpNa·TMEDA[1] or Cp_2Pb[2] (Figure 1a), or asymmetrical, as in $CpGaMe_2$[3] or CpZnMe[4] (Figure 1b). Cyclopentadienyl groups coordinating simultaneously to two[5] or even three[6] transition-metal atoms located on the same side of the ring plane are known; in these cases, the metal atoms are invariably linked by direct metal–metal bonds as well. In this paper, we wish to report on the structure of a zinc compound

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Figure 1. Symmetric (**1a**) and asymmetric (**1b**) bridging modes of cyclopentadienyl groups.

containing a cyclopentadienyl group that bridges between two nonbonded zinc atoms located on the same side of the ring plane.

Experimental Section

General Remarks. All manipulations were carried out in an atmosphere of dry, oxygen-free nitrogen. All solvents were dried and distilled under nitrogen prior to use. NMR spectra were recorded on Varian EM-390 and CFT-20 and Bruker WP-200 spectrometers. $Zn[N(SiMe_3)_2]_2$[7] and Cp_2Zn[8] were prepared by literature methods. Et_2Zn was purchased from Alfa-Ventron GmbH and distilled in vacuo prior to use.

$Cp_3Zn_2N(SiMe_3)_2$. To a suspension of 5.1 g of Cp_2Zn in 50 mL of benzene was added 3.28 g of $Zn[N(SiMe_3)_2]_2$, and the stirred suspension was warmed to 60 °C. After 5 min, the solution was cooled to room temperature, and the slight excess of Cp_2Zn was removed by centrifugation. The solvent was removed in vacuo; the residue was washed once with cold pentane (10 mL, −55 °C) and dried in vacuo, giving 7.8 g of colorless, microcrystalline $Cp_3Zn_2N(SiMe_3)_2$. Crystals, suitable for X-ray diffraction analysis, were obtained by slow crystallization from pentane. NMR (C_6D_6, δ in ppm relative to internal Me_4Si): ^1H, 6.50 (s, terminal Cp), 5.84 (s, μ-Cp), 0.20 (s, Me_3Si); $^{13}C\{^1H\}$ 111.3 (terminal Cp), 108.8 (μ-Cp), 5.3 (Me_3Si).

$EtZnN(SiMe_3)_2$. A 3.85-g sample of $Zn[N(SiMe_3)_2]_2$ and 1.23 g of Et_2Zn were dissolved in 30 mL of benzene, and the solvent was removed in vacuo. The residue was washed once with 6 mL of pentane at −55 °C and dried in vacuo to give 4.5 g of colorless $EtZnN(SiMe_3)_2$. The compound was extremely soluble in all organic solvents, including aliphatic hydrocarbons. The degree of association was determined by cryometry in benzene as 1.93. NMR (as above): ^1H, 1.42 (t, CH_3(Et)), 0.53 (q, CH_2(Et)), 0.25 (s, Me_3Si); $^{13}C\{^1H\}$, 12.5 (CH_3(Et)), 6.5 (CH_2(Et)), 5.6 (Me_3Si).

Data Collection and Structure Determination for $Cp_3Zn_2N(SiMe_2)_2$. A colorless crystal suitable for data collection was sealed under nitrogen in a Lindemann capillary and transferred to an Enraf-Nonius CAD4F diffractometer. Unit cell dimensions and standard deviations were determined in the usual way[9] from the setting angles of 20 carefully centered reflections. The space group was determined as $P2_1/c$, and the intensities of one quadrant of reflection data were collected in the $\omega/2\theta$ scan mode with use of Zr-filtered Mo Kα radiation. The intensities of two reference reflections were measured regularly during the data collection. The data were corrected for Lorentz and polarization effects and variations in the control reflections in the previously described way.[9] Psi scans for some close to axial reflections showed variations in intensity of less than 15% about the mean. In view of the objectives of this study, absorption correction was not considered necessary. Details of the data collection and structure determination are summarized in Table I.

The structure was solved by standard Patterson and Fourier techniques and refined by blocked full-matrix least-squares techniques. All atomic positions were refined. For all non-hydrogen atoms, anisotropic thermal parameters were refined. The hydrogen atoms were divided in two groups (the Me_3Si and the Cp protons), each of which was assigned one variable overall isotropic thermal parameter. Weights were introduced in the final stages of the refinement. Two reflections (100 and −1,1,1) were excluded because they apparently suffered from extinction. The final difference Fourier showed some residual peaks of ca. 0.5 e/Å^3 at ca 1 Å from the zinc atoms as the highest features.

Table I. Crystal Data and Details of the Structure Determination

Crystal Data	
formula	$C_{21}H_{33}NSi_2Zn_2$
mol wt	486.42
cryst system	monoclinic-*b*
space group	No. 14, $P2_1/c$
a, Å	15.447 (3)
b, Å	11.049 (1)
c, Å	14.503 (3)
β, deg	108.35 (2)
V, Å^3	2349.5 (8)
Z	4
D_{calcd}, g/cm^3	1.375
F_{000}	1016
μ(Mo Kα), cm^{-1}	22.04
cryst size, mm	0.45 × 0.45 × 0.35
Data Collection	
θ_{min}, θ_{max}, deg	0.1, 27.5
radiatn	Mo Kα (Zr-filtered), 0.710 69 Å
$\omega/2\theta$ scan, deg	$0.60 + 0.35 \tan \theta$
max. time/refl, min	1.5
horizontal and vertical aperture, mm	3, 3
ref refl	602, $6\bar{1}\bar{3}$
total data	5585
total unique	4440
obsd data ($I > 2.5\sigma(I)$)	3424
Refinement	
no. of refined parameters	336
weighing scheme	$w^{-1} = (\sigma^2(F) + 0.003F^2)/0.56$
final $R_F = \Sigma \lvert F_o - \lvert F_c \rvert \rvert / \Sigma F_o$	0.045
final $R_{wF} = [\Sigma w(F_o - \lvert F_c \rvert)^2 / \Sigma w F_o^2]^{1/2}$	0.049
rms deviation of refl of unit wt	1.46

Neutral atom scattering factors were taken from ref 10 and corrected for anomalous dispersion.[11] All calculations were carried out on the Cyber 175 of the University of Utrecht computer center. Programs used include SHELX-76 (structure refinement[12]), PLATO (geometry analysis[13]), and ORTEP (plotting program[14]).

Results and Discussion

In our laboratory, Cp_2Zn is usually prepared by the procedure of Lorberth.[8]

$$Zn[N(SiMe_3)_2]_2 + 2CpH \xrightarrow{Et_2O} Cp_2Zn + 2(Me_3Si)_2NH$$

This reaction is somewhat capricious: the reaction rate is variable, and sometimes an induction period is observed. The Cp_2Zn from one particular preparation was found to contain a large amount of $(Me_3Si)_2N$ groups, and a compound of the stoichiometry $Cp_3Zn_2N(SiMe_3)_2$ could be extracted from it with pentane, in which Cp_2Zn is insoluble. $Cp_3Zn_2N(SiMe_3)_2$ (**1**) is more conveniently prepared by combining appropriate amounts of Cp_2Zn and $Zn[N(SiMe_3)_2]_2$ in warm benzene.

The structure of **1** consists of discrete molecular units. The molecule contains two zinc atoms that are connected by a bridging cyclopentadienyl group and a bridging amide group; in addition, each zinc atom carries a terminal Cp group. ORTEP drawings of two orientations of the molecule

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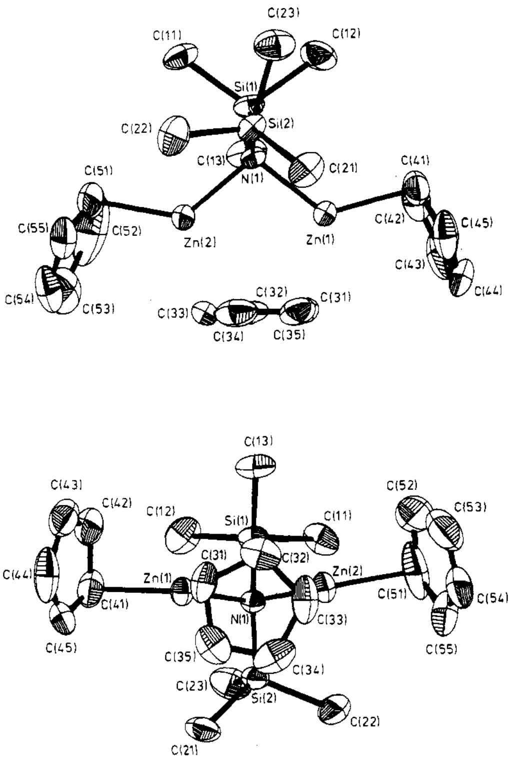

Figure 2. ORTEP drawings of two orientations of the $Cp_3Zn_2N(SiMe_3)_2$ molecule, showing 40% probability thermal ellipsoids. Hydrogen atoms have been omitted for clarity.

Table II. Fractional Atomic Coordinates of the Non-Hydrogen Atoms of $Cp_3Zn_2N(SiMe_3)_2$

	x/a	y/b	z/c
Zn(1)	0.35916 (3)	−0.02007 (5)	0.20237 (4)
Zn(2)	0.18479 (4)	0.12554 (5)	0.18762 (4)
N(1)	0.2501 (2)	−0.0272 (3)	0.2490 (2)
Si(1)	0.28604 (9)	−0.0016 (1)	0.37591 (8)
Si(2)	0.18163 (8)	−0.1554 (1)	0.20076 (9)
C(11)	0.1892 (4)	0.0074 (5)	0.4247 (4)
C(12)	0.3653 (5)	−0.1217 (7)	0.4438 (4)
C(13)	0.3487 (4)	0.1445 (5)	0.3994 (4)
C(21)	0.2172 (4)	−0.2253 (6)	0.1027 (4)
C(22)	0.0600 (4)	−0.1110 (7)	0.1495 (5)
C(23)	0.1900 (5)	−0.2765 (6)	0.2921 (5)
C(31)	0.3600 (4)	0.1188 (6)	0.0993 (4)
C(32)	0.3113 (5)	0.2121 (5)	0.1218 (4)
C(33)	0.2205 (4)	0.1962 (6)	0.0670 (5)
C(34)	0.2151 (5)	0.0939 (8)	0.0099 (4)
C(35)	0.3008 (5)	0.0488 (6)	0.0300 (4)
C(41)	0.4703 (4)	−0.1338 (6)	0.2447 (5)
C(42)	0.5219 (5)	−0.0425 (7)	0.2983 (6)
C(43)	0.5721 (5)	0.0038 (6)	0.2486 (8)
C(44)	0.5568 (6)	−0.060 (1)	0.1630 (7)
C(45)	0.4934 (5)	−0.1479 (7)	0.1624 (6)
C(51)	0.0790 (8)	0.218 (1)	0.2156 (7)
C(52)	0.1300 (7)	0.322 (2)	0.243 (1)
C(53)	0.0935 (8)	0.3983 (8)	0.1528 (8)
C(54)	0.0303 (6)	0.3366 (8)	0.0914 (7)
C(55)	0.0180 (5)	0.2330 (9)	0.1236 (7)

are shown in Figure 2. Fractional atomic coordinates are given in Table II, selected bond distances in Table III, and bond angles in Table IV. The large Zn–Zn separation of 3.087 (1) Å precludes any direct Zn–Zn bonding. The Zn–N distances resemble those found in $[MeZnNPh_2]_2$.[15] For every Cp–Zn interaction, there is one short Zn–C distance of 2.0–2.1 Å representing a normal Zn–C single bond; in addition, however, an intermediate distance of about 2.4–2.6 Å to another carbon atom of the same ring is found. This is also true for the Cp–Zn arrangements in crystalline Cp_2Zn[16] and CpZnMe.[4] Some relevant data for 1 and Cp_2Zn are summarized in Table V. The intermediate distances suggest that both σ- and π-type interactions contribute to the Cp–Zn bonds; i.e., the bonding is not purely η^1. A schematic description of the bonding starts with two sp^2-hybridized zinc atoms, each forming one Zn-N σ-bond and two Zn–C σ-bonds. π-Donation from the Cp π orbitals to the remaining empty Zn p orbitals results in coordination saturation of the zinc atoms. Obviously, it is not essential for both zinc atoms to be on the same side of the ring plane: the chain structure of Cp_2Zn, which has Cp groups bridging between Zn atoms on opposite sides of the ring plane, can be rationalized in an analogous fashion.

Table III. Selected Bond Distances (Å)

Zn(1)-N(1)	2.005 (3)	Zn(1)-C(31)	2.146 (6)
Zn(2)-N(1)	2.024 (3)	Zn(1)-C(35)	2.495 (6)
Si(1)-N(1)	1.769 (3)	Zn(1)-C(41)	2.059 (6)
Si(2)-N(1)	1.775 (3)	Zn(1)-C(42)	2.473 (7)
Si(1)-C(11)	1.847 (6)	Zn(2)-C(33)	2.140 (7)
Si(1)-C(12)	1.864 (7)	Zn(2)-C(32)	2.611 (7)
Si(1)-C(13)	1.859 (6)	Zn(2)-C(51)	2.07 (1)
Si(2)-C(21)	1.848 (6)	Zn(2)-C(52)	2.55 (2)
Si(2)-C(22)	1.856 (6)		
Si(2)-C(23)	1.858 (7)		

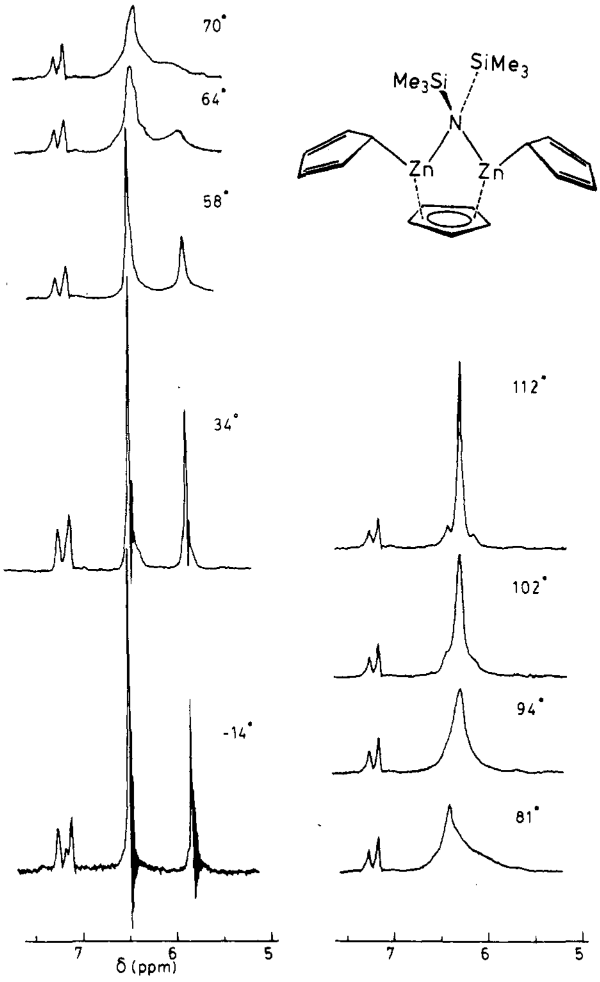

Figure 3. Temperature dependence of the ^1H NMR spectrum of $Cp_3Zn_2N(SiMe_3)_2$ in toluene-d_8.

The ^1H NMR spectrum of 1 at room temperature shows two singlets (in the ratio 2:1) for the two kinds of Cp

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Table IV. Selected Bond Angles (deg)

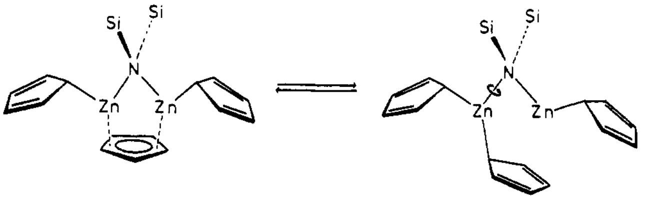
Zn(1)-N(1)-Zn(2)	100.1 (1)	N(1)-Si(1)-C(13)	107.7 (2)	C(21)-Si(2)-C(23)	106.0 (3)
Zn(1)-N(1)-Si(1)	108.8 (1)	C(11)-Si(1)-C(12)	108.2 (3)	C(22)-Si(2)-C(23)	108.1 (3)
Zn(1)-N(1)-Si(2)	111.0 (2)	C(11)-Si(1)-C(13)	108.7 (3)	N(1)-Zn(1)-C(31)	116.9 (2)
Zn(2)-N(1)-Si(1)	105.9 (2)	C(12)-Si(1)-C(13)	107.7 (3)	N(1)-Zn(1)-C(41)	125.9 (2)
Zn(2)-N(1)-Si(2)	109.9 (1)	N(1)-Si(2)-C(21)	110.6 (2)	C(31)-Zn(1)-C(41)	117.2 (3)
Si(1)-N(1)-Si(2)	119.4 (2)	N(1)-Si(2)-C(22)	110.5 (3)	N(1)-Zn(2)-C(33)	116.3 (2)
N(1)-Si(1)-C(11)	112.3 (2)	N(1)-Si(2)-C(23)	113.3 (3)	N(1)-Zn(2)-C(51)	129.7 (3)
N(1)-Si(1)-C(12)	112.0 (2)	C(21)-Si(2)-C(22)	108.1 (3)	C(33)-Zn(2)-C(51)	113.7 (3)

Table V. Short and Intermediate Zn–C Distances (Å) in $Cp_3Zn_2N(SiMe_3)_2$ and Cp_2Zn (Standard Deviations 0.01 and 0.05 Å, Respectively)

compd	M–C		
	short	intermed	long
(a) Terminal Cp Groups			
Cp_2Zn[16]	2.08	2.33	2.84
	2.04	2.48	2.68
$Cp_3Zn_2N(SiMe_3)_2$	2.06	2.47	2.72
	2.07	2.55	2.72
(b) Bridging Cp Groups			
Cp_2Zn[16] [a]	2.04	2.41	2.73
	2.06	2.46	2.84
$Cp_3Zn_2N(SiMe_3)_2$	2.15	2.50	2.82
	2.14	2.61	2.78

[a] Distances involving the ordered bridging Cp group in Cp_2Zn.

groups. At higher temperatures, however, the two singlets broaden and coalesce until at about 120 °C only one singlet is left (Figure 3). This dynamic behaviour closely parallels that found for the intramolecular exchange of methyl groups in $(\mu\text{-Me})(\mu\text{-NPh}_2)(AlMe_2)_2$.[17] The most probable explanation is an intramolecular exchange by opening of the Cp bridge, followed by rotation around a Zn–N bond and reformation of the bridge (eq 1). From the observed coalescence temperature, an activation energy of about 17 kcal/mol is estimated.



(1)

Most zinc amides are associated via bridging nitrogen atoms. $Zn[N(SiMe_3)_2]_2$, however, is a monomer. Of course, the bis(trimethylsilyl)amino group is a particularly weak donor because of the presence of N–Si p_π–d_π bonding. Nevertheless, it is still capable of bridging between zinc atoms: the compound $EtZnN(SiMe_3)_2$, prepared from Et_2Zn and $Zn[N(SiMe_3)_2]_2$, was found to be a dimer in benzene. The fact that $Zn[N(SiMe_3)_2]_2$ is not associated must probably be ascribed to the bulkiness of the $N(SiMe_3)_2$ groups.

Most organozinc amides correspond to the simple stoichiometry $RZnNR'_2$. This is also true for the compound $EtZnN(SiMe_3)_2$ mentioned above. However, the only compound that could be obtained from Cp_2Zn and $Zn[N(SiMe_3)_2]_2$ was **1**. This implies that the hypothetical compound $CpZnN(SiMe_3)_2$ (**2**) is unstable with respect to disproportionation into **1** and $Zn[N(SiMe_3)_2]_2$. It may be that the difference in size between an ethyl group and a cyclopentadienyl group is large enough to make dimerization of **2** impossible on steric grounds. In that case, the disproportionation of **2** would be favorable because it would increase the average coordination number of the zinc.

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Registry No. $Cp_3Zn_2N(SiMe_3)_2$, 90030-15-2; $EtZnN(SiMe_3)_2$, 90030-16-3; $Zn[N(SiMe_3)_2]_2$, 11077-31-9; Et_2Zn, 557-20-0.

Supplementary Material Available: Tables of structure factor amplitudes, all positional and thermal parameters, and bond distances and angles (25 pages). Ordering information is given on any current masthead page.

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