Mononuclear Tris(aminopyridinato)zirconium Alkyl, Aryl, and Alkynyl Complexes

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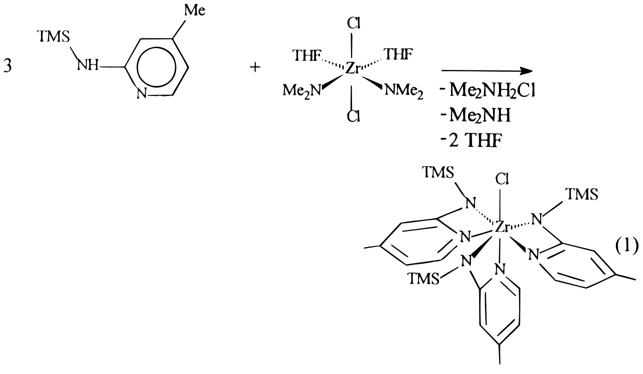
*Summary: The reaction of 3 equiv of 4-methyl-2-((trimethylsilyl)amino)pyridine (TMS-AP-H) with $(Me_2N)_2ZrCl_2(THF)_2$ or the in situ lithiation of TMS-AP-H followed by addition of $ZrCl_4$ affords $(TMS\text{-}AP)_3Zr{-}Cl$ (**1**) in high yield. The X-ray analysis of **1** reveals a mononuclear zirconium complex coordinated by six nitrogen atoms with an overall 3-fold molecule symmetry. The η^2-coordinated aminopyridinato ligands arrange in a propeller-like fashion. The reactions of **1** with MeLi, phenyllithium, and (phenylethynyl)lithium afford the corresponding σ-alkyl, -aryl, and -alkynyl complexes ($(TMS\text{-}AP)_3Zr{-}Me$ (**2**), $(TMS\text{-}AP)_3Zr{-}Ph$ (**3**), and $(TMS\text{-}AP)_3Zr{-}C{\equiv}CPh$ (**4**). X-ray diffraction studies of **4** establish its monomeric structure, a long Zr−C bond distance, and a sterically shielded alkynyl ligand.*

Introduction

Amido-based tripod ligand systems [1−3] that form stable early transition metal complexes have gained a lot of attention. The ligands in these complexes shield a large sector of the coordination sphere and thus provide a "reactive pocket" for metal-supported activation and transformation reactions. Such complexes have been shown to activate small molecules like dinitrogen,[4] to give rise to unusual transformation reactions,[5] and to provide access to stable polar metal−metal bonds.[6] With regard to their catalytic applications, we are currently examining the chemistry of early transition metal complexes that are reactive due to strained η^2-coordinated aminopyridinato ligands.[7] We expect the 4-methyl-2-((trimethylsilyl)amino)pyridinato (TMS-AP) ligand with a "maximal steric angle" of 144° [8] to gives rise to a propeller-like C_3 symmetric complex fragment containing a reactive pocket similar to those observed for tripod chelating ligands. Herein is reported the synthesis and structure of the first mononuclear zirconium complex that contains aminopyridinato ligands:[9] $(TMS\text{-}AP)_3Zr{-}Cl$ and its σ-alkyl, -aryl, and -alkynyl derivatives.

Results and Discussion

The reaction of 3 equiv of TMS-AP-H with mixed chloro(dialkylamido)zirconium complexes like $(Me_2N)_2ZrCl_2(THF)_2$ (easily accessible by reacting $Zr(NMe_2)_4$ with $ZrCl_4$ in the presence of THF[10]) affords the colorless crystalline compound **1** (eq 1). A small amount Me_2NH_2Cl is observed as a byproduct. ^1H and ^{13}C NMR spectra are indicative of an overall C_3 molecular symmetry, and elemental analysis is consistent with the formula $(TMS\text{-}AP)_3Zr{-}Cl$.



(**1**)

The common synthetic procedure of ligand lithiation followed by adding of zirconium chloride leads to **1** in a high-yield reaction (eq 2). This observation is in contrast to synthetic protocols of titanium aminopyridinato complexes,[7] where only very low yields could be obtained.

⊗ Abstract published in *Advance ACS Abstracts,* December 15, 1995.

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[8] The "maximal steric angle" was defined as the angle between the outermost H atoms of the pyridine ring, the metal at which the aminopyridinato ligand coordinates, and the outermost H atom of the substituent at the amido nitrogen in relation to the cone angle approach, which is widely used to evaluate the bulkiness of ligands. See also: Brown, T. L.; Lee, K. J. *Coord. Chem. Rev.* **1993**, *128*, 89.

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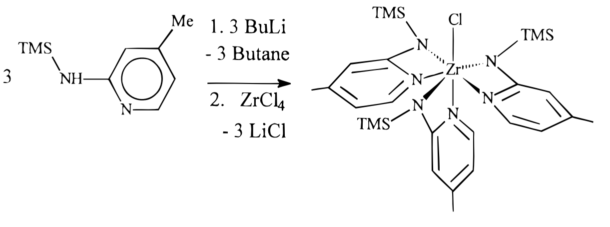

(2)

(**1**)

Suitable crystals for X-ray diffraction studies of **1** can be obtained by slowly cooling a saturated ether solution to −30 °C. The single-crystal X-ray structure analysis of **1** establishes its monomeric structure as shown in Figure 1 including principal bond distances and angles. Some important crystallographic features are listed in Table 1. **1** crystallizes in the quite uncommon cubic lattice system and is oriented along a 3-fold crystallographic axis. The Zr−Cl vector coincides with this axis, and the aminopyridinato ligands arrange in a propeller-like fashion around it. This kind of C_3 symmetric mononuclear zirconium complex coordinated by six nitrogen atoms constitutes a new structural type of zirconium complexes.[11] The $N_{pyridine}-C-N_{amido}$ angle (110.8(3)°) reflects the strained bonding situation. The Zr−N2 distance (2.324(2) Å) is well in the range of such $Zr-N_{pyridine}$ bonds (2.39(1) Å).[12] The Zr−N1 bond length (2.209(2) Å) is approximately 0.1 Å shorter than the averaged $Zr-N_{amido}$ distance (2.11(1) Å)[13] and indicates a weak amido bond. The nitrogen atoms have an almost planar geometry, as has been found in virtually all structurally characterized transition metal amido complexes.[14] The interest in the tris(aminopyridinato)zirconium moiety is focused on the stabilization of σ-alkyl, -aryl, and -alkynyl complexes in order to study the stability of the zirconium carbon bonds with regard to metal-supported C−C coupling reactions which might lead to catalytic applications. The reactions of **1** with MeLi, phenyllithium, and (phenylethynyl)lithium afford the corresponding organometallic complexes, $(TMS\text{-}AP)_3Zr-Me$ (**2**), $(TMS\text{-}AP)_3Zr-Ph$ (**3**), and $(TMS\text{-}AP)_3Zr-C\equiv CPh$ (**4**), as shown in Scheme 1. ^1H and ^{13}C NMR spectra are in accordance with the formulations, and elemental analyses are consistent. An X-ray crystal structure analysis of **4** was carried out in order to evaluate the steric protection of the aminopyridinato ligand core with respect to the Zr−C and the C≡C triple bond. Suitable crystals for X-ray diffraction studies of **4** could be obtained by slowly cooling a saturated ether solution to −30 °C. A perspective ORTEP drawing of the molecular structure of **4** is shown in Figure 2 including key bond distances and angles. Crystallographic details are listed in Table 1. The molecular structure of **4** is characterized by a monomeric propeller-like ligand core which is largely in accordance with the bond lengths and angles observed for **1**. **4** is the first monoalkynylzirconium complexes,[15] characterized by X-ray analysis, which has an amido ligand core. The $Zr-C_{alkynyl}$ distance (2.281(7)Å) is slightly longer than the averaged $Zr-C_{sp}$ bond (2.237(13)Å)[16] length and indicates a weak Zr σ-alkynyl bond. The molecular structure of **4** verifies that the sterically demanding trimethylsilyl substituents of the ligand core do protect the metal−carbon bond.

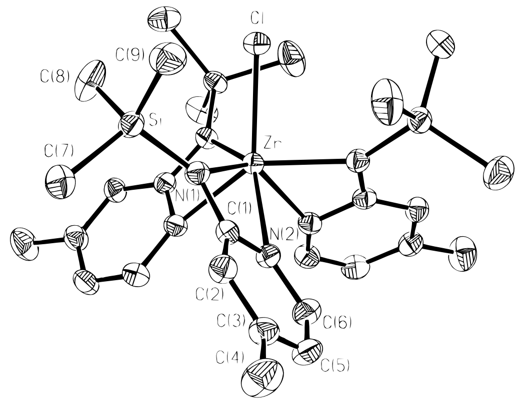

Figure 1. Structural representation of **1**. Hydrogen atoms are omitted for clarity. The thermal ellipsoids correspond to 40% probability. Selected bond distances and angles: Zr−N(1) 2.209(2), Zr−N(2) 2.324(2), Zr−Cl 2.5160(14); N(1)−Zr−N(2) 59.59(9), N(1)−Zr−Cl 86.25(6), N(2)−Zr−Cl 131.22(6), C(6)−N(2)−C(1) 120.2(3), N(2)−C(1)−N(1) 110.8(3).

Table 1. Crystallographic Details of the X-ray Crystal Structure Analysis of 1 and 4

compd	**1**	**4**
cryst system	cubic	monoclinic
space group	$Pa\bar{3}$	$P2_1/n$ (No. 14)
a, Å	19.423(2)	11.914(2)
b, Å		20.364(2)
c, Å		17.419(2)
β, deg		100.48 (1)
V, Å^3	7327.4(1.3)	4102.4(9)
Z	24	4
cryst size, mm	0.4 × 0.4 × 0.2	0.4 × 0.3 × 0.3
fw	663.36	730.30
ρ_{calcd}, g cm^{-3}	1.203	1.167
μ, cm^{-1} (Mo Kα)	4.92	3.80
$F(000)$	2779	1536
T, K	173	293
θ range, deg	$2.3 < \theta < 23.2$	$2.3 < \theta < 25.0$
scan type	$\omega/2\theta$	$\omega/2\theta$
no. of reflcns	5058	7659
no. of unique reflcns	1741	7284
no. of obsd reflcns ($I > 2\sigma(I)$)	1358	4706
no. of params	115	351
wR^2 (all data)	0.093	0.214
R value ($I > 2\sigma(I)$)	0.032	0.073

Several conclusions can be drawn from this study. First, aminopyridinato ligands can stabilize a new type of monomolecular zirconium complexes due to the strained η^2 binding mode. Second, the tris(aminopyridinato)zirconium moiety forms stable alkyl, aryl, and alkynyl complexes. Third, the sterically demanding TMS groups of the ligand protect the metal−carbon σ-bonds by a propeller-like arrangement. We are continuing to explore the potential utility of aminopyridinato ligands for organometallic chemistry and extend our studies to other early transition metals.

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(12) Sample mean and standard deviation of 18 $Zr-N_{py}$ bonds distances by using QUEST and VISTA.[19]

(13) Sample mean and standard deviation of 66 $Zr-N_{amido}$ bond distances by using QUEST and VISTA.[19]

(14) Lappert, M. F.; Power, P. P.; Sanger, A. R.; Srivastava, R. C. *Metal and Metalloid Amides*; Ellis Norwood Ltd.: Chichester, England, 1980.

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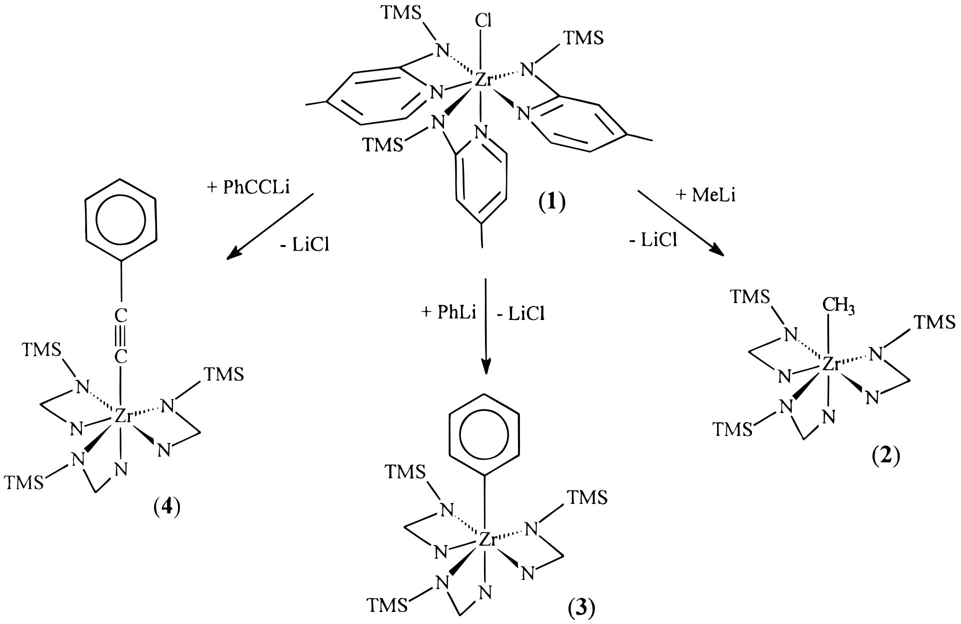
(16) Sample mean and standard deviation of 6 $Zr-C_{sp}$ bond distances by using QUEST and VISTA.[19]

Scheme 1. Synthesis of Tris(aminopyridinato)zirconium σ-Alkyl, -Aryl, and -Alkynyl Complexes



Experimental Section

Materials and Procedures. 4-Methyl-2-((trimethylsilyl)-amino)pyridine[7] was prepared according to a previously published procedure. All other reagents were obtained commercially and used as supplied. All manipulations of air-sensitive materials were performed with rigorous exclusion of oxygen and moisture in dried Schlenk-type glassware on a dual manifold Schlenk line, interfaced to a high-vacuum line, or in an argon-filled Vacuum Atmospheres glovebox (mBraun labmaster 130) with a high-capacity recirculator (<1.5 ppm O_2). Solvents (Aldrich) and NMR solvents (Cambridge Isotope Laboratories all 99 atom % D) were freshly distilled from sodium tetraethylaluminate.

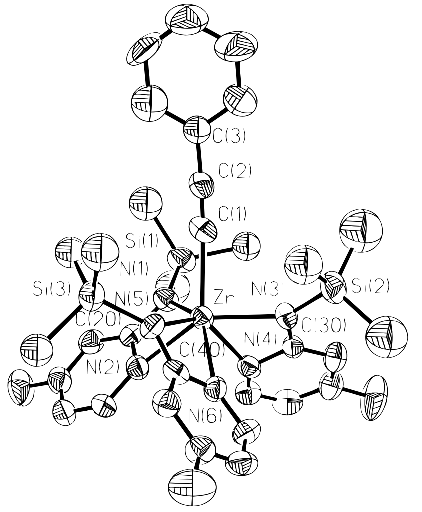


Figure 2. Perspective ORTEP drawing of the molecular structure of **4**. All non- hydrogen atoms are represented by thermal ellipsoids drawn to encompass 40% probability. Selected bond distances and angles: C(1)−C(2) 1.184(9), C(1)−Zr 2.281(7), C(2)−C(3) 1.453(9), C(20)−N(1) 1.356-(8), C(20)−N(2) 1.359(7), C(24)−N(2) 1.338(8), C(30)−N(3) 1.354(8), C(30)−N(4) 1.368(8), C(34)−N(4) 1.340(8), C(40)−N(5) 1.351(8), C(40)−N(6) 1.354(8), C(40)−C(41) 1.424(9), C(44)−N(6) 1.335(7), N(1)−Si(1) 1.730(5), N(1)−Zr 2.210-(5), N(2)−Zr 2.318(5), N(3)−Zr 2.198(5), N(4)−Zr 2.299(5), N(5)−Zr 2.202(5), N(6)−Zr 2.314(5); C(2)−C(1)−Zr 170.2-(7), C(1)−C(2)−C(3) 176.9(8), N(1)−C(20)−N(2) 110.0(5), N(3)−C(30)−N(4) 110.1(5), N(5)−C(40)−N(6) 110.9(5), C(30)−N(3)−Si(2) 128.1(4),), N(3)−Zr−N(5) 121.8(2), N(3)−Zr−N(1) 118.5(2), N(5)−Zr−N(1) 119.0(2), N(3)−Zr−N(4) 59.4(2), N(5)−Zr−N(4) 138.9(2), N(1)−Zr−N(4) 80.7(2), N(3)−Zr−N(6) 81.4(2), N(5)−Zr−N(6) 59.1(2), N(1)−Zr−N(6) 141.0(2), N(4)−Zr−N(6) 82.6(2), N(3)−Zr−N(2) 139.9-(2), N(5)−Zr−N(2) 80.2(2), N(1)−Zr−N(2) 58.7(2), N(4)−Zr−N(2) 81.7(2), N(6)−Zr−N(2) 84.1(2).

Physical Measurements. NMR spectra were recorded on a Bruker ARX 400 instrument with variable-temperature unit. ^{1}H and ^{13}C chemical shifts are referenced to the solvent resonances and reported relative to TMS. ^{29}Si chemical shifts are reported relative to TMS. Melting points were determined in sealed capillaries on a Büchi 535 apparatus. Elemental analysis were performed with a Leco CHNS-932 elemental analyzer. X-ray diffraction data were collected on a CAD4 MACH3 diffractometer using graphite-monochromated Mo Kα radiation. The crystals were mounted in a cold nitrogen stream or sealed inside a capillary. An absorption correction was carried out by a ψ-scan. The structure was solved by direct methods (SHELXS-86)[17] and refined by full-matrix least-squares techniques against F^2 (SHELXL-93).[18] XP (Siemens Analytical X-ray Instruments, Inc.) was used for structure representations.

(TMS-AP)$_3$Zr−Cl (1). To a solution of 4-methyl-2-((trimethylsilyl)amino)pyridine (6.56 mL, 22.4 mmol) in ether (35 mL) was added slowly via syringe a 1.6 M hexane solution of MeLi (14 mL, 22.4 mmol) at −70 °C. The mixture was allowed to warm to room temperature over a period of 20 min, $ZrCl_4$ (1.74 g, 7.46 mmol) was added over a period of 20 min, and the mixture was stirred for a further 5 h. A white solid precipitated. The solution was filtered, and the volume was reduced under vacuum to approximately 10 mL. Cooling to −30 °C overnight afforded a colorless crystalline material. Yield: 4.11 g, 6.18 mmol, 83%. ^{1}H NMR (303 K, C_6D_6) (δ): 7.26 (d, J = 5.6 Hz, 1H, H-6); 6.24 (s, 1 H, H-3); 5.86 (d, J = 5.6 Hz, 1 H, H-5); 1.76 (s, 3 H, Me); 0.43 (s, 9 H, $SiMe_3$). ^{13}C NMR (303 K, C_6D_6) (δ): 168.1 (C-2); 152.0 (C-4); 142.1 (C-6); 112.2 (C-3); 111.4 (C-5), 21.7 (Me); 1.2 ($SiMe_3$). Anal. Calcd

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for $C_{27}H_{45}ClN_6Si_3Zr$: C, 48.79; H, 6.82; N, 12.64. Found: C, 49.07; H, 6.42; N, 12.65.

((TMS-AP)$_3$Zr−Me (2). To a solution of **1** (0.33 g, 0.50 mmol) in ether (30 mL) was added slowly via syringe a 1.6 M hexane solution of MeLi (310 μL, 0.50 mmol) at −70 °C. The mixture was allowed to warm to room temperature over a period of 20 min and stirred for another 3 h. A white solid precipitated. The solution was filtered, and the volume was reduced under vacuum to approximately 10 mL. Cooling to −30 °C afforded overnight a colorless crystalline material. Yield: 0.206 g, 0.32 mmol, 64%. ^{1}H NMR (303 K, C_6D_6) (δ): 7.38 (d, J = 5.6 Hz, 3 H, H-6); 6.26 (s, 3 H, H-3); 5.85 (d, J = 5.6 Hz, 3 H, H-5); 1.76 (s, 9 H, Me); 0.93 (s, 3 H, Me-Zr); 0.38 (s, 27 H, $SiMe_3$). ^{13}C NMR (303 K, C_6D_6) (δ): 168.9 (C-2); 151.4 (C-4); 142.8 (C-6); 112.0 (C-3), (111.5 C-5), 47.6 (Me-Zr); 21.6 (Me); 1.1 ($SiMe_3$). Anal. Calcd for $C_{28}H_{48}N_6Si_3Zr$: C, 52.21; H, 7.51; N, 13.05. Found: C, 52.19; H, 7.60; N, 13.06.

(TMS-AP)$_3$Zr−Ph (3). To a solution of **1** (0.41 g, 0.62 mmol) in ether (30 mL) was added slowly via syringe a 1.8 M hexane solution of phenyllithium (350 μL, 0.63 mmol) at −70 °C. The color changed to light yellow immediately. The mixture was allowed to warm to room temperature over a period of 30 min and stirred for another 18 h. A white solid precipitated. The solution was filtered, and the volume was reduced under vacuum to approximately 10 mL. Cooling to −30 °C afforded overnight a yellow crystalline material. Yield: 0.30 g, 0.42 mmol, 68%. ^{1}H NMR (303 K, C_6D_6) (δ): 7.97 (d, J = 7.9 Hz, 2 H, *o*-Ph); 7.64 (d, J = 5.7 Hz, 3 H, H-6); 7.30 (tr, 2 H, *m*-Ph); 7.20 (m, 1 H, *p*-Ph); 6.30 (s, 3 H, H-3); 5.92 (d, J = 5.7 Hz, 3 H, H-5); 1.76 (s, 9 H, Me); 0.20 (s, 27 H, $SiMe_3$). ^{13}C NMR (303 K, C_6D_6) (δ): 190.5 (*ipso*-Ph); 171.3 (C-2); 151.7 (C-4); 143.7 (C-6); 135.9 (*o*-Ph); 126.7 (*m*-Ph); 126.0 (*p*-Ph); 112.7 (C-3); 111.4 (C-5), 21.6 (Me); 1.3 ($SiMe_3$). Anal. Calcd for $C_{33}H_{50}N_6Si_3$ Zr: C 56.12; H, 7.14; N, 11.94. Found: C, 56.13; H,7.33; N, 11.94.

(TMS-AP)$_3$Zr−C≡CPh (4). To a solution of phenylacetylene (109 μL, 1.00 mmol) in ether (30 mL) was added slowly via syringe a 1.6 M hexane solution of MeLi (0.62 mL, 1.00 mmol) at −70 °C. The mixture was allowed to warm to room temperature and was stirred over a period of 1 h. **1** (0.66 g, 1.00 mmol) was added slowly, and the mixture was stirred for a further 18 h. A white solid precipitated. The solution was filtered, and the volume was reduced under vacuum to approximately 10 mL. Cooling to −30 °C afforded over night a colorless crystalline material. Yield: 0.49 g, 0.67 mmol, 67%. ^{1}H NMR (303 K, C_6D_6) (δ): 7.75 (d, J = 8.7 Hz, 2 H, *o*-Ph); 7.35 (d, J = 5.4 Hz, 3H, H-6); 7.11 (tr, 2 H, *m*-Ph); 6.98 (tr, 1 H, *p*-Ph); 6.26 (s, 3 H, H-3); 5.89 (d, J = 5.4 Hz, 3 H, H-5); 1.77 (s, 9 H, Me); 0.51 (s, 27 H, $SiMe_3$). ^{13}C NMR (303 K, C_6D_6) (δ): 168.2 (C-2); 151.8 (C-4); 142.8 (C-6); 111.9, 111.6 (C-3, C-5), 21.3 (Me); 0.9 ($SiMe_3$); 152.5, 103.9 (C≡C); 131.4, 128.5, 126.3 (*o*-, *m*-, *p*-Ph); 127.3 (*ipso-Ph*). IR (Nujol): 2078 (C≡C) cm^{-1}. Anal. Calcd for $C_{35}H_{50}N_6Si_3Zr$: C, 57.56; H, 6.90; N, 11.51. Found: C, 57.28; H, 6.80; N, 11.52.

Acknowledgment. We thank Prof. U. Rosenthal for generous support and helpful discussions. Financial support by the Fonds der Chemischen Industrie and the Max-Planck-Gesellschaft is gratefully acknowledged.

Supporting Information Available: Tables of crystal data, positional parameters, full bond distances and angles, and thermal parameters (16 pages). Ordering information is given on any current masthead page.

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