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(Alkylamido)gallate Complexes of the Group 1 and 2 Elements

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Summary: The reaction of 2 equiv of $LiNH(t\text{-}Bu)$ *with* $[Cy_2GaCl]_2$ *(Cy = cyclohexyl) produces the gallate complex* $Li(THF)_2(\mu\text{-}NH(t\text{-}Bu))_2GaCy_2$ *(1). The reaction of 2 equiv of* $Li(3,5\text{-}Me_2Pz)$ *(Pz = pyrazolyl) with* $[Cy_2GaCl]_2$ *produces the gallate species* $MgCl(THF)_2(\mu\text{-}3,5\text{-}Me_2Pz)_2GaCy_2$ *(2). An X-ray structural study shows that the amide ligands for 1 and 2 bridge from the Ga atom to* Li^+ *and* $MgCl^+$*, respectively.*

As an extension of our work on group 13 amides, we recently reported the synthesis and structural characterization of the cationic species $[Me_2Ga(NH_2(t\text{-}Bu))_2]Br$.[1] To our knowledge this is the first instance that the cationic R_2Ga^+ fragment had been structurally characterized. In comparison to the case for neutral compounds, very little is known about ionic gallium amide and adduct species. Storr and co-workers have described anionic bis(alkyl)-bis(pyrazolyl)gallium compounds which were used to prepare gallium-containing transition-metal compounds, several of which were structurally characterized.[2] The gallate species used to prepare these transition-metal complexes were generally not isolated but rather used in situ in reactions with transition-metal halides.

We describe here two compounds in which formally anionic gallium fragments are bound to Li^+ ($Li(THF)_2(\mu\text{-}NH(t\text{-}Bu))_2GaCy_2$ (1)) and $MgCl^+$ ($MgCl(THF)_2(\mu\text{-}3,5\text{-}Me_2Pz)_2GaCy_2$ (2)) via bridging amide groups.

Results and Discussion

Synthesis and Structure of $Li(THF)_2(\mu\text{-}NH(t\text{-}Bu))_2GaCy_2$ (1). The reaction of 2 equiv of $LiNH(t\text{-}Bu)$ with Cy_2GaCl in THF results in a colorless solution from which the gallate species $Li(THF)_2(\mu\text{-}NH(t\text{-}Bu))_2GaCy_2$ (1) may be isolated in 64% yield. Alternatively, 1 can be prepared by the addition of 1 equiv of $LiNH(t\text{-}Bu)$ to $[Cy_2GaNH(t\text{-}Bu)]_2$.[3] The ^1H NMR spectrum in C_6D_6 confirms the presence of two THF molecules (δ 3.46 (m) and 2.28 (m)). The cyclohexyl units appear as four distinct multiplets in the range 0.82–2.01 ppm, while the two *tert*-butylamide resonances appear at 1.31 (s) and 1.27 (s) ppm. The equivalence of the two THF resonances indicates symmetrical bonding or fluxionality in solution, although there is no crystallographic symmetry in the solid state.

The solid-state structure of 1 was determined by X-ray crystallography. Molecules of 1 crystallize in the orthorhombic space group $P2_12_12_1$ with 8 molecules per unit cell. Figure 1 shows the molecular geometry and atom-numbering scheme. Positional parameters and selected bond lengths and angles are given in Tables II and IV.

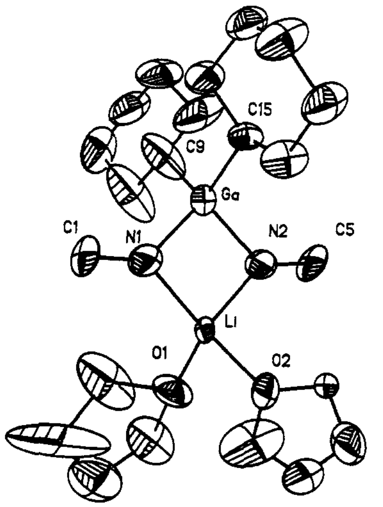

Figure 1. Molecular structure and atom-numbering scheme for 1. The *tert*-butyl methyls have been omitted for clarity.

The structure of 1 consists of discrete monomeric units in which the two amido groups are bonded to the lithium atom (N1–Li = 2.04 (2) Å and N2–Li = 1.97 (2) Å), which is further coordinated to two THF groups (O1–Li = 1.94 (3) Å and O2–Li = 2.19 (3) Å). The symmetry around Li is distorted tetrahedral. A similar coordination environment for Li is observed in $Li(THF)_2(\mu\text{-}Cl)_2GaCl(C(SiMe_3Ph)_3)\cdot THF$.[4] The two Ga–N bond lengths are 1.99 (2) Å. This is somewhat shorter than the Ga–N bond lengths found in the dimers $[Me_2Ga(\mu\text{-}NH(t\text{-}Bu))]_2$ (Ga–N = 2.011 (4) and 2.012 (4) Å)[5] and $[t\text{-}Bu_2Ga(\mu\text{-}NHPh)]_2$ (Ga–N = 2.103 (9) and 2.018 (9) Å).[6] This difference may be caused by a reduction in electronic repulsion between the nitrogen lone pairs, now bonded to Li^+, and the anionic gallium atom. The Ga–C bond lengths are typical for alkylgallium complexes in general (Ga–C9 = 2.12 (3) Å and Ga–C15 = 2.03 (2) Å). The $Ga\text{–}(N)_2\text{–}Li$ four-membered ring is planar within experimental error with N–Ga–N and N–Li–N angles of 89.1 (5) and 86.3 (8)°, respectively. These values may be compared to those found in $Li(THF)_2(\mu\text{-}Cl)_2GaCl(C(SiMe_3Ph)_3)\cdot THF$, wherein the $Ga\text{–}Cl_2\text{–}Li$ four-membered ring is also planar with the Cl1–Ga–Cl2 and Cl1–Li–Cl2 bond angles being 95.1 (1) and 88 (1)°, respectively.[4]

Synthesis and Structure of $MgCl(THF)_2(\mu\text{-}3,5\text{-}Me_2Pz)_2GaCy_2$ (2). The treatment of $GaCl_3$ with 2 equiv of CyMgCl followed by the in situ addition of 2 equiv of $Li(3,5\text{-}Me_2Pz)$ produces compound 2 ($MgCl(THF)_2(\mu\text{-}3,5\text{-}Me_2Pz)_2GaCy_2$) in almost quantitative yield. The ^1H NMR

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Table I. Crystallographic Data for Compounds 1 and 2

	1	2
formula	$C_{28}H_{50}LiN_2O_2Ga$	$C_{30}H_{44}N_4O_2MgClGa$
fw	523.38	622.18
cryst syst	orthorhombic	triclinic
space group	$P2_12_12_1$	$P\bar{1}$
a, Å	17.074 (2)	9.075 (2)
b, Å	21.632 (1)	10.974 (3)
c, Å	17.661 (2)	17.607 (8)
α, deg	90	105.48 (3)
β, deg	90	92.38 (3)
γ, deg	90	93.06 (2)
V, Å^3	6523 (1)	1685 (15)
Z	8	1
density	1.070	0.616
μ(Mo Kα), cm^{-1}	8.64	4.69
cryst dimens, mm	0.10 × 0.08 × 0.08	0.12 × 0.10 × 0.10
scan width, deg	0.8 = 0.35 tan θ	0.8 = 0.35 tan θ
2θ range, deg	2–50	2–50
no. of rflns measd	4965	5338
no. of rflns obsd	1927	3956
no. of params	306	352
R	0.0806	0.0597
R_w	0.0877	0.0690

Table II. Fractional Coordinates and Equivalent Isotropic Thermal Parameters[a] for the Non-Hydrogen Atoms of 1

atom	*x*	*y*	*z*	*U*, Å^2
Gal	0.0003 (2)	0.17073 (8)	0.12963 (9)	0.0676 (6)
Li	−0.011 (2)	0.3045 (8)	0.1222 (13)	0.043 (6)
N1	−0.0536 (12)	0.2384 (7)	0.1850 (11)	0.111 (10)
C1	−0.122 (2)	0.237 (2)	0.2373 (14)	0.13 (2)
C2	−0.202 (2)	0.2488 (14)	0.190 (2)	0.19 (2)
C3	−0.139 (2)	0.176 (2)	0.2721 (14)	0.17 (2)
C4	−0.125 (2)	0.301 (2)	0.2785 (15)	0.19 (2)
N2	0.0522 (13)	0.2313 (8)	0.0609 (10)	0.123 (11)
C5	0.1234 (14)	0.2276 (14)	0.0147 (13)	0.096 (11)
C6	0.142 (2)	0.1644 (14)	−0.0196 (15)	0.18 (2)
C7	0.1803 (15)	0.239 (2)	0.072 (2)	0.206 (14)
C8	0.124 (3)	0.275 (2)	−0.0373 (12)	0.40 (4)
C9	0.063 (2)	0.1157 (14)	0.208 (2)	0.17 (2)
C10	0.101 (2)	0.067 (2)	0.171 (2)	0.20 (3)
C11	0.133 (2)	0.0144 (15)	0.233 (2)	0.19 (2)
C12	0.181 (2)	0.044 (2)	0.287 (2)	0.18 (2)
C13	0.145 (2)	0.099 (2)	0.3187 (15)	0.18 (2)
C14	0.102 (2)	0.146 (2)	0.264 (2)	0.20 (2)
C15	−0.0645 (10)	0.1169 (9)	0.0598 (11)	0.068 (7)
C16	−0.107 (2)	0.0653 (11)	0.097 (2)	0.148 (14)
C17	−0.148 (2)	0.0172 (14)	0.0402 (13)	0.14 (2)
C18	−0.194 (2)	0.0485 (14)	−0.016 (2)	0.18 (2)
C19	−0.154 (2)	0.103 (2)	−0.0541 (13)	0.91 (2)
C20	−0.122 (2)	0.1506 (10)	0.0041 (14)	0.107 (12)
O1	−0.0604 (9)	0.3624 (7)	0.0535 (9)	0.107 (7)
C21	−0.100 (4)	0.343 (3)	−0.0106 (14)	0.53 (6)
C22	−0.124 (3)	0.411 (2)	−0.040 (2)	0.23 (3)
C23	−0.109 (5)	0.451 (2)	0.006 (3)	0.49 (7)
C24	−0.120 (3)	0.423 (2)	0.072 (3)	0.32 (3)
O2	0.0731 (12)	0.3625 (9)	0.1833 (11)	0.146 (10)
C25	0.093 (3)	0.416 (2)	0.166 (3)	0.40 (3)
C26	0.138 (3)	0.453 (2)	0.208 (2)	0.25 (3)
C27	0.150 (2)	0.407 (2)	0.273 (2)	0.21 (3)
C28	0.102 (3)	0.351 (2)	0.252 (2)	0.30 (4)

[a] For anisotropic atoms, the *U* value is U_{eq}, calculated as $U_{eq} = {}^1/{}_3\sum_i\sum_j U_{ij}a_i^*a_j^*A_{ij}$, where A_{ij} is the dot product of the *i*th and *j*th direct-space unit cell vectors.

spectrum indicates the presence of two equivalent THF molecules (δ 3.71 (m) and 2.36 (m) ppm). The pyrazolyl groups appear as two singlets at 5.86 and 2.22 ppm, while the cyclohexyl resonances give a series of multiplets in the range 1.94–1.00 ppm.

Table III. Fractional Coordinates and Equivalent Isotropic Thermal Parameters[a] for the Non-Hydrogen Atoms of 2

atom	*x*	*y*	*z*	*U*, Å^2
Ga	0.47269 (9)	−0.35935 (7)	0.78014 (5)	0.0523 (3)
Mg	0.5672 (3)	−0.0813 (2)	0.69730 (13)	0.0590 (9)
Cl	0.6690 (4)	0.0883 (2)	0.6584 (2)	0.121 (2)
N1	0.5685 (6)	−0.3652 (5)	0.6785 (3)	0.054 (2)
N2	0.5939 (6)	−0.2695 (5)	0.6436 (3)	0.055 (2)
C1	0.6591 (9)	−0.3182 (8)	0.5755 (4)	0.069 (3)
C2	0.7043 (12)	−0.2357 (9)	0.5230 (5)	0.102 (5)
C3	0.6759 (9)	−0.4455 (8)	0.5664 (5)	0.074 (4)
C4	0.6171 (8)	−0.4730 (7)	0.6316 (4)	0.064 (3)
C5	0.6046 (11)	−0.5980 (7)	0.6529 (5)	0.090 (4)
N3	0.42353 (6)	−0.1776 (5)	0.8262 (3)	0.050 (2)
N4	0.4531 (6)	−0.0752 (5)	0.7973 (3)	0.054 (2)
C6	0.4084 (8)	0.0267 (6)	0.8504 (4)	0.063 (3)
C7	0.4227 (12)	0.1577 (7)	0.8364 (6)	0.100 (5)
C8	0.3509 (8)	−0.0092 (7)	0.9133 (4)	0.065 (3)
C9	0.3632 (8)	−0.1385 (7)	0.8966 (4)	0.058 (3)
C10	0.3161 (10)	−0.2260 (8)	0.9451 (5)	0.078 (4)
C11	0.6374 (8)	−0.3980 (7)	0.8480 (4)	0.066 (3)
C12	0.7086 (10)	−0.2799 (8)	0.9075 (5)	0.095 (4)
C13	0.8469 (11)	−0.3124 (11)	0.9509 (7)	0.116 (5)
C14	0.8040 (15)	−0.420 (2)	0.9884 (7)	0.135 (7)
C15	0.7428 (13)	−0.5350 (12)	0.9291 (7)	0.120 (6)
C16	0.6021 (11)	−0.5063 (9)	0.8844 (6)	0.102 (5)
C17	0.2704 (9)	−0.4517 (7)	0.7522 (5)	0.079 (3)
C18	0.2526 (10)	−0.5729 (9)	0.7786 (6)	0.104 (4)
C19	0.0916 (14)	−0.6332 (12)	0.7610 (9)	0.141 (6)
C20	0.0411 (14)	−0.6482 (13)	0.6799 (10)	0.152 (7)
C21	0.0496 (13)	−0.5273 (13)	0.6572 (9)	0.149 (6)
C22	0.2142 (12)	−0.4627 (12)	0.6723 (7)	0.135 (6)
O1	0.3682 (7)	−0.0876 (6)	0.6266 (3)	0.089 (3)
C23	0.302 (2)	0.0201 (13)	0.6140 (12)	0.262 (11)
C24	0.1614 (15)	−0.0222 (14)	0.5686 (9)	0.156 (7)
C25	0.1597 (12)	−0.1602 (13)	0.5420 (7)	0.134 (6)
C26	0.295 (2)	−0.1869 (12)	0.5750 (12)	0.239 (9)
O2	0.7670 (6)	−0.0811 (5)	0.7697 (3)	0.087 (2)
C27	0.8068 (13)	0.0275 (10)	0.8377 (7)	0.137 (6)
C28	0.9666 (15)	0.019 (2)	0.8539 (11)	0.202 (8)
C29	1.0151 (13)	−0.096 (2)	0.8000 (9)	0.206 (9)
C30	0.8953 (11)	−0.1430 (13)	0.7384 (7)	0.132 (6)

[a] For anisotropic atoms, the *U* value is U_{eq}, calculated as $U_{eq} = {}^1/{}_3\sum_i\sum_j U_{ij}a_i^*a_j^*A_{ij}$, where A_{ij} is the dot product of the *i*th and *j*th direct-space unit cell vectors.

Table IV. Selected Bond Lengths (Å) and Angles (deg) for 1

Ga–N1	1.99 (2)	N1–Li	1.95 (3)
Ga–N2	1.99 (2)	N2–Li	2.20 (3)
Ga–C9	2.12 (3)	O1–Li	1.94 (3)
Ga–C15	2.03 (2)	O2–Li	2.19 (3)
N1–Ga–N2	91.2 (7)	C9–Ga–C15	111 (1)
N1–Ga–C9	109 (1)	N1–Ga–C15	118.1 (8)
N2–Ga–C9	123 (1)	N1–Li–N2	86 (1)
N2–Ga–C15	105.5 (8)	O1–Li–O2	103 (1)

An X-ray diffraction study was performed on 2. Molecules of 2 crystallize in the triclinic space group $P\bar{1}$ with one molecule per unit cell. Figure 2 shows the molecular geometry and atom-numbering scheme. Positional parameters and selected bond lengths and angles are given in Tables III and V. The structure of 2 consists of two pyrazolyl groups bridging the Ga and Mg atoms through adjacent pyrazolyl nitrogens. To the best of our knowledge, this is the first instance where an (alkylpyrazolyl)gallate bonded to a group 2 element has been structurally characterized.

The Ga–N bond lengths in 2 are 2.009 (6) and 2.020 (5) Å, which are similar to those found in 1 (1.99 (2) Å). In this case, however, a reduction in electron repulsion between Ga and N may be the result of resonance stabilization of the N lone pair into the Pz ring. It is interesting to note that these values are also similar to those found for Ga–N bonds in neutral dialkylgallium pyrazolyl complexes (1.98–2.00 Å).[7] The Ga–C bond lengths for 2 are standard, falling in the range 2.03–2.00 Å.

The coordination environment around Mg is trigonal-bipyramidal, with the nitrogen atoms of the gallate

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[7] Rendle, D. F.; Storr, A.; Trotter, J. *Can. J. Chem.* **1975**, *53*, 2930.

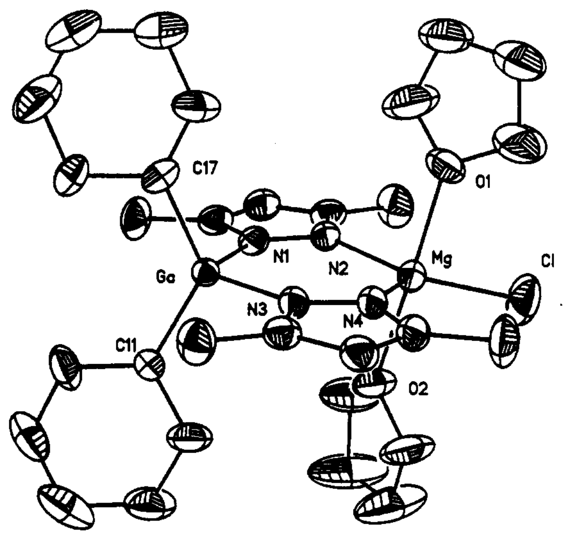

Figure 2. Molecular structure and atom-numbering scheme for **2**.

Table V. Selected Bond Lengths (Å) and Angles (deg) for 2

Ga–N1	2.009 (6)	Mg–N2	2.061 (5)
Ga–N3	2.020 (5)	Mg–N4	2.067 (6)
Ga–C11	2.007 (8)	Mg–O1	2.138 (6)
Ga–C17	2.025 (8)	Mg–O2	2.172 (6)
Mg–Cl	2.310 (4)		
N1–Ga–N3	106.4 (2)	Cl–Mg–N2	125.4 (2)
N1–Ga–C11	102.9 (3)	Cl–Mg–N4	127.3 (2)
N3–Ga–C11	108.1 (2)	Cl–Mg–O1	91.8 (2)
N3–Ga–C17	103.1 (3)	N4–Mg–O1	92.5 (2)
C11–Ga–C17	127.5 (3)	N4–Mg–O2	86.7 (2)
C17–Ga–N1	107.5 (3)	O1–Mg–O2	178.1 (3)

occupying equatorial positions. This is a somewhat rare geometry for Mg. Some early structurally characterized examples are $MgBr_6O\cdot 4Et_2O$[8] and $CH_3MgBr\cdot 3C_4H_8O$.[9] An additional point of interest is that the Ga–(N-N)$_2$–Mg six-membered ring is planar within experimental error. Planar Ga–(N-N)$_2$–M ring systems are common in transition-metal pyrazolylgallate complexes in which the pyrazolyl is unsubstituted. One example is $Ga(CH_3)_2(Pz)_2Mn(CO)_3PzH$.[10] However, when the pyrazolyl ligand has substituents bulkier than H (e.g. 3,5-dimethylpyrazole) the Ga–(N-N)$_2$–Mg ring frequently adopts a boat conformation in order to reduce steric repulsions.[11] It may be that steric interactions do not play a significant role in the structure of **2**.

Experimental Section

General Considerations. All reactions were performed under oxygen-free nitrogen using standard Schlenk-line techniques in conjunction with an inert-atmosphere drybox. Toluene and diethyl ether were distilled from sodium/benzophenone under nitrogen before use. Neat *tert*-butylamine was dried over molecular sieves and then distilled from calcium hydride before use. The pyrazole 2,6-Me_2Pz was dried in vacuo at 60 °C (12 h). Instruments: IR, Perkin-Elmer 1330, Digilab FTS-40; NMR, GE QE-300, (^1H, 300.17 MHz); mass spectrum, EI and CI, Bell and Howell 21-491; elemental analyses, Perkin-Elmer 2400 analyzer. IR spectra were run as Nujol mulls using KBr plates. NMR spectra were recorded in C_6D_6 solution at ambient temperatures, unless otherwise stated, and are in units of ppm referenced to Me_4Si. All NMR solvents were dried over 4-Å molecular sieves prior to use. Melting points were obtained in sealed capillaries under argon (1 atm) and are uncorrected. Elemental analyses (C, H, N) were performed on a Perkin-Elmer 2400 analyzer. $[Cy_2GaCl]_2$ was prepared from the reaction of CyMgCl and $GaCl_3$ according to the literature method.[12]

Preparation of 1. A solution of LiNH(*t*-Bu) (0.29 g, 4.00 mmol) was added dropwise to a solution (−78 °C) of $[Cy_2GaCl]_2$ (0.54 g, 1.00 mmol) in THF (30 mL). The reaction mixture was warmed to 25 °C and then stirred (12 h). After the volatile materials were removed in vacuo, the residue was extracted with toluene (100 mL) and the resulting solution filtered. The filtrate was evaporated to dryness, resulting in a white microcrystalline solid, which was extracted with Et_2O (20 mL). Crystals suitable for X-ray crystallography were grown from an Et_2O solution, which was stored at −20 °C for 1 week. Data for **1**: mp 101–103 °C dec; yield 0.52 g, 64%; IR (Nujol mull) 3187 w, 1660 m, 1345 m, 1296 w, 1223 m, 802 s, 650 m cm^{-1}; ^1H NMR (C_6D_6) δ 3.46, 2.28 (m, m, 8 H, THF), 1.31, 1.27 (s, s, 18 H, N-*t*-Bu), 2.01, 1.79, 1.58, 0.82 (all m, 22 H, Ga–Cy). Anal. Calcd: C, 64.26; H, 9.63; N, 5.35. Found: C, 64.81; H, 9.79; N, 5.23.

Preparation of 2. A solution of Li(3,5-Me_2Pz) (3.58 g, 37.26 mmol) was added dropwise to a solution (−78 °C) of $[Cy_2GaCl]_2$, prepared in situ from CyMgCl (37.26 mmol, 17.49 mL, 2.13 M) and $GaCl_3$ (3.28 g, 18.63 mmol) in Et_2O/THF (1:1, 100 mL). The reaction mixture was warmed to 25 °C and then stirred (12 h). After the volatile materials were removed in vacuo, the residue was extracted with toluene (100 mL), and the resulting solution was filtered. The filtrate was evaporated to dryness, resulting in a white microcrystalline solid which was extracted with Et_2O (20 mL). Crystals suitable for X-ray crystallography were grown from an Et_2O solution which was stored at −20 °C for 1 week. Data for **2**: mp 101–103 °C dec; yield 10.65 g, 92%; ^1H NMR (C_6D_6) δ 5.86 (s, 2 H, Pz H), 3.71, 2.36 (m, m, 8 H, THF), 2.22 (s, 12 H, Pz–CH_3), 1.94–1.00 (m, 22 H, Ga–Cy); ^{13}C NMR (C_6D_6) δ 145.15 (s, Pz), 105.34 (s, THF), 31.68, 29.40, 28.30, 27.00 (s, Cy), 12.14 (s, Pz–CH_3). Anal. Calcd: C, 57.91; H, 7.13; N, 9.01. Found: C, 58.03; H, 7.25; N, 8.89.

Crystallography. X-ray structural details are given in Table I. Crystals were mounted in thin-walled glass capillaries and sealed under argon. Unit cell parameters were obtained by centering 25 reflections having 2θ values between 20 and 26°. Data were collected on an Enraf-Nonius CAD-4 diffractometer at 2θ = 2–50° using graphite-monochromated Mo Kα radiation. Intensity data were recorded in a manner as described previously.[13] All calculations were performed on a Microvax 3100 computer using the SHELX software package.[14] For each structure the data were corrected for Lorentz and polarization effects. For both structures all the non-hydrogen atoms were refined anisotropically. For **2** a ψ scan of 4 reflections having χ values between 80 and 90° showed an average transmission of 68.10%, and thus, an absorption correction was applied.

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Supplementary Material Available: X-ray crystal data for **1** and **2**, including tables of thermal parameters, bond lengths, and bond angles (8 pages). Ordering information is given on any current masthead page.

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