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Lithium (*S*)-1-[2′-(Methoxymethyl)pyrrolidin-1′-yl]-3,5-dimethylboratabenzene. An Enantiopure Lithium Salt Possessing a Novel Polymeric Structure[1]

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Reaction of (*S*)-2-(methoxymethyl)pyrrolidine (SMPH) with the 1-chloro-3,5-dimethyldihydroborinines **3a**,**b** in the presence of NEt_3 afforded the SMP-substituted dihydroborinine isomers **4a**−**c** (95% yield). Metalation of **4a**−**c** with $LiN(SiMe_3)_2$ produced the salt lithium (*S*)-1-[2′-(methoxymethyl)pyrrolidin-1′-yl]-3,5-dimethylboratabenzene [Li(**1**)] (92%). Treatment of Li(**1**) with $[(C_5Me_5)Fe(NCMe)_3]PF_6$ gave the enantiopure ferrocene analogue (C_5Me_5)Fe(**1**) (98%). Crystallization of Li(**1**) from THP afforded colorless rods of [Li(**1**)$(THP)_{0.5}]_\infty$ (≡**2**). In the crystal structure, **2** consists of infinite chains with alternating bis(boratabenzene)lithiate sandwich units and tetrahedral LiN_2O_2 centers. Whereas the *B*-exocyclic amino and the pendant ether functionalities from the SMP moieties act as donating coordination sites to the metal, the THP serves solely as a space-filling molecule.

Introduction

The chemistry of facial π-ligands bearing pendant donor substituents is of interest, both from the point of view of reactivity and from the point of view of structure.[2,3] In most cases, the additional donor functionalities act as auxiliary chelating sites to metal centers, exerting characteristic steric and electronic effects on the coordination spheres. In the field of alkali metal cyclopentadienides, structurally characterized compounds with side-chain donors are still quite rare,[4] presumably because of their high reactivity.

Boratabenzene ions[5,6] are heterocyclic 6π-electron ligands that are closely related to cyclopentadienides. In this paper, we report the synthesis and characterization of lithium (*S*)-1-[2′-(methoxymethyl)pyrrolidin-1′-yl]-3,5-dimethylboratabenzene [Li(**1**)], a novel boratabenzene possessing two characteristic features. It carries a pendant sidearm which, as will be seen below, may act as a chelating κ^2N,O-ligand. Furthermore, it is enantiomerically pure because of its chiral *B*-exocyclic pyrrolidinyl substituent. At present very few chiral and enantiopure boratabenzenes,[7] neutral borabenzene Lewis base adducts,[7b,8] and complexes of these systems[8,9] are known.

Results and Discussion

Our synthetic approach is outlined in Scheme 1. We chose the readily accessible isomeric mixture of the 1-chloro-3,5-dimethyldihydroborinines **3a**,**b**[10] as a source of the borinine fragment. Compounds **3a**,**b** react with (*S*)-2-(methoxymethyl)pyrrolidine (SMPH)[11] in the presence of NEt_3 as base to give an isomeric mixture of the SMP-substituted dihydroborinines **4a**−**c** in 95% yield. Subsequent metalation of this mixture with $LiN(SiMe_3)_2$ produced the solvent-free lithium (*S*)-1-[2′-(methoxymethyl)pyrrolidin-1′-yl]boratabenzene [Li(**1**)] as an extremely oxygen- and moisture-sensitive white powder.

Slow diffusion of hexane into Li(**1**) in THP at 0 °C afforded [Li(**1**)$(THP)_{0.5}]_\infty$ (≡**2**) as colorless rods. The empirical formula of **2** was established by means of NMR spectroscopy and later confirmed by an X-ray single-crystal structure determination. In the solid state, compound **2** adopts the chiral orthorhombic space group $P222_1$ (no. 17) with $Z = 16$ on the basis of the formula Li(**1**)$(THP)_{0.5}$. It displays a novel polymeric structure which consists of infinite chains with alternating bis(boratabenzene)lithiate sandwich units and tetrahedral LiN_2O_2 centers. Although half of an equivalent

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Scheme 1

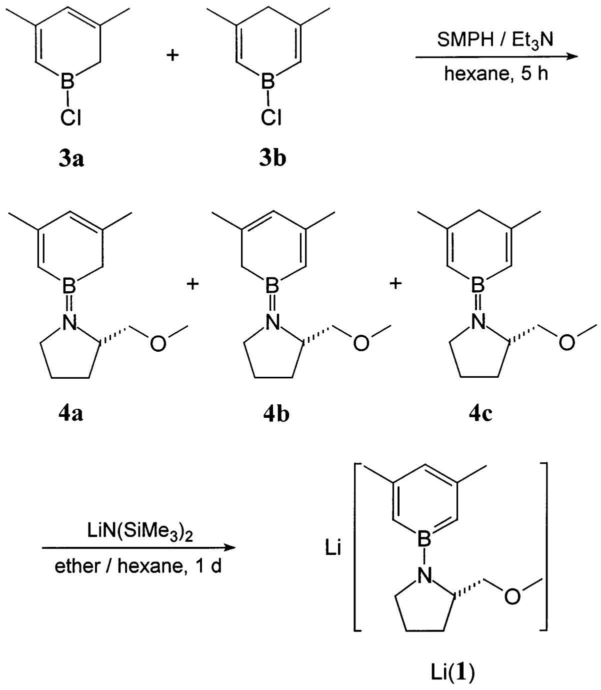

Table 1. Selected Bond Distances and Bond Angles for 2

(a) Bond Distances (Å)			
Li1−B1	2.524(8)	Li4−B3	2.489(8)
Li1−C11	2.451(12)	Li4−C51	2.434(11)
Li1−C12	2.384(12)	Li4−C52	2.375(12)
Li1−C13	2.303(7)	Li4−C53	2.325(8)
Li1−C14	2.326(11)	Li4−C54	2.345(10)
Li1−C15	2.414(11)	Li4−C55	2.368(10)
Li2−B2	2.455(11)	Li5−B2	2.460(10)
Li2−C31	2.455(11)	Li5−C71	2.300(10)
Li2−C32	2.511(8)	Li5−C72	2.329(12)
Li2−C33	2.462(12)	Li5−C73	2.391(11)
Li2−C34	2.347(13)	Li5−C74	2.465(8)
Li2−C35	2.315(7)	Li5−C75	2.495(11)
Li3−N1	2.152(12)	Li6−N3	2.194(14)
Li3−N2	2.154(11)	Li6−N4	2.202(14)
Li3−O1	1.980(11)	Li6−O3	1.901(13)
Li3−O2	1.989(11)	Li6−O4	1.963(14)
B1−N1	1.473(9)	B3−N3	1.505(9)
B2−N2	1.491(10)	B4−N4	1.502(9)
(b) Bond Angles (deg)			
N1−Li3−N2	120.4(5)	N3−Li6−N4	119.0(6)
N1−Li3−O1	83.9(4)	N3−Li6−O3	84.8(5)
N1−Li3−O2	141.1(6)	N3−Li6−O4	138.1(7)
N2−Li3−O1	133.4(6)	N4−Li6−O3	134.1(7)
N2−Li3−O2	84.0(4)	N4−Li6−O4	82.6(5)
O1−Li3−O2	101.5(5)	O3−Li6−O4	106.0(6)

of THP per Li(**1**) moiety is present in the structure, the solvent acts solely as a space-filling molecule and does not coordinate to the lithium atom as one might have expected. Selected bond parameters are listed in Table 1, and a representative structural fragment is illustrated in Figure 1.

The structure of **2** exhibits several remarkable features. The unit cell contains four independent Li(**1**) moieties which are grouped into two pairs, each forming a one-dimensional polymeric chain. The two asymmetric chains run across the unit cell along the longest lattice axis *c* [$c = 36.90(2)$ Å; cf. $a = 11.664(2)$ and $b = 15.176(2)$ Å]. The asymmetric unit contains four lithium atoms (Li1, Li2, Li4, and Li5) in special positions, i.e., on 2-fold rotational axes, and two lithium atoms (Li3 and Li6) in general positions. The former type of lithium atom is sandwiched by two η^6-boratabenzene rings, which are related to one another by a crystallographic 2-fold axis. The latter type of lithium atom is surrounded by two nitrogen and two oxygen atoms from the SMP fragments; the resulting tetrahedral LiN_2O_2 centers serve as a link between two neighboring lithium sandwich units.

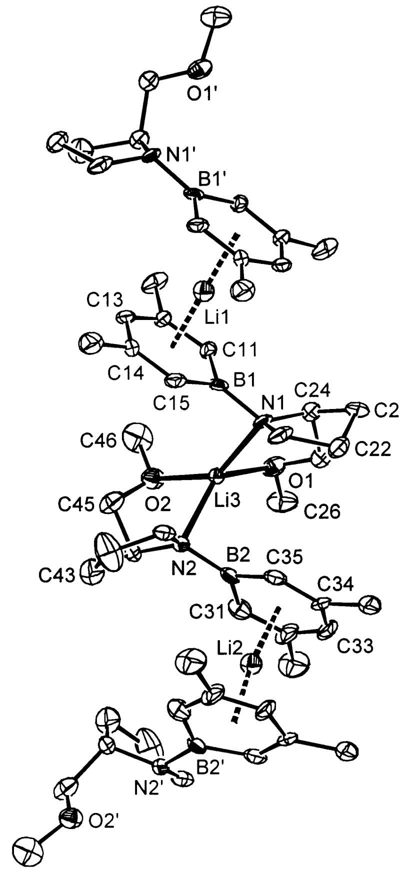

Figure 1. Representative fragment of one of the two polymeric chains in the structure of **2** (Platon plot[12] with displacement ellipsoids at the 30% probability level). The lithium atoms Li1 and Li2 are on 2-fold axes. The other independent chain is very similar and is not presented.

This type of polymeric structure is unprecedented in boratabenzene chemistry[5] and also in the related cyclopentadienide chemistry.[13] Prior to this work, four structural types of boratabenzene salts were known. The year 1993 saw the first structure in this field, that of $Li(TMPDA)(C_5H_5BNMe_2)$,[14] which is built up of contact ion pairs stabilized by additional neutral donors; a number of examples of this type have since become known.[7,15] The ammonium salt $NMe_3Ph(C_5H_5BMe)$ possesses an AB-type ionic structure with “naked” boratabenzene ions,[16] and $[Li(THF)_4][Li(C_5H_5BH)_2]$[17] con-

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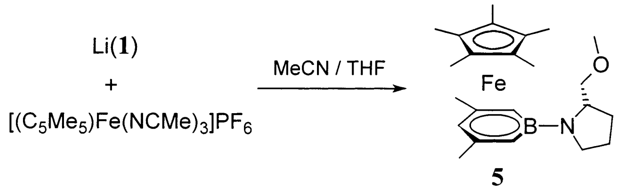
Scheme 2



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tains a bis(boratabenzene)lithiate anion, which is a close analogue of the lithocenates with a $[LiCp_2]^-$ anion.[13,18] The recently described $Na(OEt_2)[(E)\text{-}C_5H_5BCH{=}CHPh]$ has a zigzag chain structure with each boratabenzene ion bridging two sodium ions.[19]

Rather soft bonding geometries are found in the sandwich fragments of **2**. The distances from the lithium atoms to the boratabenzene ring centroids range from 1.92 to 1.95 Å, slightly shorter than those of simpler lithium sandwich compounds $[Li(C_5H_5)_2]^-$ (2.01 Å)[18a] and $[Li(C_5H_5BH)_2]^-$ (1.98 Å).[17] While the parent lithocene possesses a typical staggered parallel conformation in the solid state, the sandwich units in **2** are slightly bent, with four symmetrically independent interplanar angles ranging from 0.9° to 10.5°. Moreover, the rotational positions within pairs of boratabenzene ligands vary from an approximate trans-conformation to an almost perpendicular fashion. This flexibility reflects the highly ionic nature of the interactions between the lithium atom and the boratabenzene ligands.

An aminoboratabenzene serving simultaneously as a facially bonded ligand[5] as well as a σ-donor[6c,20] has not been observed previously. However, an analogous bifunctional π/σ-bonding mode has also been found in the lithium aminoborole compound $[Li_2(TMEDA)(C_4H_4BNEt_2)]_2$.[21] In general, considerable π-interaction exists between the exocyclic amino group and the borinine ring for aminoboratabenzene complexes.[6c,14,22] The B−N bond distances in **2** [ranging from 1.473(9) to 1.505(9) Å] are longer than those in aminoboratabenzenes [e.g., 1.448(2) Å in $Li(TMPDA)(C_5H_5BNMe_2)$[14]]. This comparison demonstrates that the σ-coordination of the amino group stabilizes the nitrogen lone pair and consequently reduces the π-interaction in the π/σ-bonded aminoboratabenezene.

The salt Li(**1**) can be utilized to introduce the boratabenzene ligand with central chirality to metal complexes. For instance, reaction of Li(**1**) with $[(C_5Me_5)Fe(NCMe)_3]PF_6$[23] in acetonitrile/THF (1:1) afforded asymmetric iron(II) sandwich complex $(C_5Me_5)Fe(\mathbf{1})$ (≡**5**) as a deep red powder in 98% yield (Scheme 2).

In summary, we have reported here a chiral and enantiopure lithium boratabenzene of a novel polymeric structural type. It is the first time the π/σ-coordination of an aminoboratabenzene species is observed. We note that derivatives of the 3,5-dimethylbora- and 3,5-dimethylboratabenzene series are easily accessible.[15a] Therefore, the salt Li(**1**) may offer interesting perspectives for future developments of chiral and enantiopure boratabenzene derivatives.

Experimental Section

General Procedures. Reactions were carried out under an atmosphere of nitrogen by means of conventional Schlenk techniques. Hexane was distilled from Na/K alloy; Et_2O, THF, and THP were distilled from sodium benzophenone ketyl; acetonitrile was dried from the activated aluminum oxide and degassed by condensation under vacuum prior to use. Kieselguhr was heated under high vacuum at 300 °C for 12 h and stored under nitrogen. Elemental analyses were performed at the Analystische Laboratorien, D-51779 Lindlar, Germany.

Mass spectra were recorded on a Finnigan MAT-95 at a nominal electron energy of 70 eV. NMR spectra were recorded on a Varian Unity 500 (^1H, 500 MHz; ^{13}C, 125.7 MHz; ^{11}B, 160.4 MHz) spectrometer. Chemical shifts are referenced to TMS for ^1H and ^{13}C and to $BF_3{\cdot}Et_2O$ for ^{11}B. Assignments of NMR signals are based on $^1H/^1H$-COSY, HMQC, and NOE techniques.

Synthesis of (*S*)-1-[2′-(Methoxymethyl)pyrrolidin-1′-yl]-3,5-dimethyldihydroborinines 4a−c. A mixture of (*S*)-2-(methoxymethyl)pyrrolidine[11] (3.58 g, 28.8 mmol) and triethylamine in hexane (20 mL) was added dropwise to 1-chloro-3,5-dimethyldihydroborines **3a**,**b**[10] (4.04 g, 28.8 mmol) in hexane (60 mL) at 0 °C. A white precipitate of $NHEt_3Cl$ formed immediately. After the reaction mixture was stirred at ambient temperature for 5 h, the solid was removed by filtration and washed with hexane (3 × 20 mL). The solvent was completely evaporated from the combined filtrate under reduced pressure. The residue was heated to 90 °C under high vacuum (10^{-3} mbar), and dihydroborinines **4a**−**c** (6.02 g, 95%) were collected by condensation as a colorless liquid, moderately sensitive to air and moisture.

Selected Data for 4a−c. ^1H NMR (500 MHz, $CDCl_3$): δ 5.78, 5.77 (m, 4-H, **4a**,**b**), 5,72, 5.68 (s, br, 6-H, **4a**,**b**), 5.92, 5.90 (m, 2-/6-H, **4c**). ^{11}B NMR (160 MHz, $CDCl_3$, $BF_3{\cdot}Et_2O$ external): δ 39.7 (**4a**,**b**), 32.5 (**4c**). MS (70 eV): m/z (I_{rel}) 219 (6, M^+), 174 (100, $M^+ - CH_2OMe$). Anal. Calcd for $C_{13}H_{22}BNO$: C, 71.25; H, 10.12; N, 6.39. Found: C, 71.05; H, 10.29; N, 6.54.

Synthesis of Lithium (*S*)-1-[2′-(Methoxymethyl)pyrrolidin-1′-yl]-3,5-dimethylboratabenzene Li(1). A solution of lithium bis(trimethylsilyl)amide (1.0 M, 27.2 mL, 27.2 mmol) in hexane was added dropwise to the dihydroborinine mixture **4a**−**c** (5.95 g, 27.2 mmol) in ether (20 mL) at −78 °C. The reaction mixture was allowed to warm to ambient temperature, and stirring was continued for 24 h to give a white suspension. The precipitate was collected by filtration, washed with hexane (3 × 30 mL), and dried in a high vacuum overnight to afford Li(**1**) (5.62 g, 92%) as an air- and moisture-sensitive colorless powder. Slow diffusion of hexane (2 mL) into Li(**1**) (0.33 g) in THP (4 mL) at 0 °C afforded the compound $[Li(\mathbf{1})(THP)_{0.5}]_\infty$ (**2**) as colorless rods. The ratio of the THP to Li(**1**) in **2** was established by NMR spectroscopy and X-ray diffraction methods.

Data for Li(1). ^1H NMR (500 MHz, THF-d_8): δ 5.21 (s, 1H, 4-H), 5.17 (s, 2H, 2-/6-H), 3.70 ("sext", $J \approx 4.3$ Hz, 1H, $NCHCH_2$), 3.40 (dd, $^2J = 8.9$ Hz, $^3J = 4.8$ Hz, 1H, CH_2OMe), 3.29 (s, 3H, OMe), 3.14 (m, 2H, NCH_2CH_2), 3.10 (dd, $^2J = 8.9$ Hz, $^3J = 8.5$ Hz, 1H, CH_2OMe), 2.03 (s, 6H, 3-/5-Me), 1.68−1.74 (m, 4H, $CH_2CH_2CH_2CH$). ^{13}C NMR (126 MHz, THF-d_8): δ 142.6 (C-3,5) 108.9 (br, C-2,6), 104.1 (C-4), 76.8 (CH_2OMe), 58.7 (OMe), 58.6 ($NCHCH_2$), 49.2 (NCH_2CH_2), 30.7 ($NCHCH_2$), 25.8 (3-/5-Me), 25.5 (NCH_2CH_2). ^{11}B NMR (160 MHz, THF-d_8,

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$BF_3 \cdot Et_2O$ external): δ 32.8. Anal. Calcd for $C_{13}H_{21}BLiNO$: C, 69.38; H, 9.41; N, 6.22. Found: C, 69.12; H, 9.33; N, 6.34.

Synthesis of {(*S*)-1-[2′-(Methoxymethyl)pyrrolidin-1′-yl]-3,5-dimethylboratabenzene}(pentamethyl-cyclopentadienyl)iron (5). A solution of $[(C_5Me_5)Fe(NCMe)_3]PF_6$[23] (1.54 g, 3.35 mmol) in acetonitrile (10 mL) was added dropwise to Li(**1**) (0.756 g, 3.36 mmol) in THF (10 mL) at −10 °C. The reaction mixture turned red immediately. After the system was stirred for 2 h at ambient temperature, the solvent was pumped off under vacuum. Hexane (40 mL) was added to the residue to form a fine white precipitate of LiCl. The solid was collected by filtration, with the help of a frit covered by a layer of Kieselguhr (4 mL), and washed with hexane until the eluate was colorless. The solvent was completely removed from the combined filtrate under high vacuum to give **5** (1.35 g, 98%) as a viscous deep red oil which solidified at ambient temperature after a week.

Data for 5. ^1H NMR (500 MHz, C_6D_6): δ 4.24 (m, 1H, NC*H*CH$_2$), 4.22 (s, 1H, 4-H), 3.73 (dd, $^2J = 9.3$, $^3J = 3.6$ Hz, 1H, C*H*$_2$OMe), 3.49 (m, 1H, NC*H*$_2$CH$_2$), 3.40 (m, 1H, NC*H*$_2$CH$_2$), 3.20 (s, 3H, OMe), 3.14 (s, 1H, 2- or 6-H), 3.06 (dd, $^2J = 9.3$, $^3J = 9.0$ Hz, 1H, C*H*$_2$OMe), 3.03 (s, 1H, 2- or 6-H), 2.32 (m, 1H, NCHC*H*$_2$), 2.15 (m, 1H, NCHC*H*$_2$), 1.94 (m, 1H, NCHC*H*$_2$), 1.91 (s, 3H, 3- or 5-Me), 1.90 (s, 3H, 3- or 5-Me), 1.88 (m, 1H, NCHC*H*$_2$), 1.56 (s, 15H, C_5Me_5). ^{13}C NMR (126 MHz, C_6D_6): δ 102.8 (C-3 or C-5), 102.7 (C-3 or C-5), 81.1 (*C*$_5$Me$_5$), 75.7 (*C*H$_2$OMe), 75.5 (C-4), 67.6, 67.0 (br, C-2,6), 58.7 (OMe), 58.4 (N*C*HCH$_2$), 48.8 (N*C*H$_2$CH$_2$), 30.6 (NCH*C*H$_2$), 25.0 (NCH$_2$*C*H$_2$), 22.9, 22.7 (3-/5-Me), 10.3 (C_5Me_5). ^{11}B NMR (160 MHz, C_6D_6, $BF_3 \cdot Et_2O$ external): δ 18.8. MS (EI, 70 eV): *m*/*z* (I_{rel}) 409 (25, M^+), 364 (89, $M^+ - CH_2OMe$), 295 (7, $M^+ - C_6H_{12}NO$), 174 (51, $C_{11}H_{17}BN^+$), 136 (100, $C_5HMe_5{}^+$), 119 (41, $C_8H_{12}B^+$), 105 (32, $C_7H_{10}B^+$). Anal. Calcd for $C_{23}H_{36}BNOFe$: C, 67.51; H, 8.87; N, 3.42. Found: C, 67.49; H, 8.82; N, 3.43.

Crystal Structure Determination of 2. The data collection was performed on an ENRAF-Nonius CAD4 diffractometer equipped with a graphite monochromator. Crystal data, data collection parameters, and convergence results are listed in Table 2. The structure was solved by direct methods[24] and refined on reflection intensities (F^2) with the SHELXL-97 program.[25] One THP molecule suffers from a partial disorder problem. Non-hydrogen atoms were assigned with anisotropic parameters except that the lithium and the disordered atoms were treated isotropically. In the structural model, most hydrogen atoms were refined on positional parameters, while those from the THP molecules were included as riding with fixed displacement parameters [C−H = 0.98 Å, $U_{iso}(H) = 1.3U_{eq}(C)$].[26]

Table 2. Crystal Data, Data Collection Parameters, and Convergence Results for 2

empircal formula	$C_{13}H_{21}BLiNO(C_5H_{10}O)_{0.5}$
fw	268.64
cryst syst	orthorhombic
space group (no.)	$P222_1$ (17)
radiation (λ, Å)	Cu Kα (1.54184)
a, Å	11.664(2)
b, Å	15.176(2)
c, Å	36.90(2)
cell volume, Å^3	6532(5)
Z	16
d_{calcd}, g cm^{-3}	1.09
F(000)	2336
μ, cm^{-1}	4.83
θ range, deg	3.1−69.9
temperature, K	200
scan mode	$\omega-2\theta$
cryst dimens, mm^3	0.8 × 0.4 × 0.3
no. of reflns	14 859
no. of indep reflns	12 400
no. of indep obsd reflns	4135 [$I > 2\sigma(I)$]
no. of variables	753
R_1,[a] obsd (all data)	0.099 (0.268)
wR_2,[b] obsd (all data)	0.193 (0.243)
GOF	0.895
max resd density, e Å^{-3}	0.28

[a] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $wR_2 = [\sum w(F_o{}^2 - F_c{}^2)^2/\sum w(F_o{}^2)^2]^{1/2}$, where $w = 1/[\sigma^2(F_o{}^2) + (0.0711P)^2]$ and $P = [\max(F_o{}^2,0) + 2F_c{}^2]/3$.

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Supporting Information Available: Tables of bond distances and angles, anisotropic displacement parameters, and positional coordinates for **2**.[26] This material is available free of charge via the Internet at http://pubs.acs.org.

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(24) Sheldrick, G. M. *SHELXS-97*, Program for Structure Solution; University of Göttingen: Göttingen, Germany, 1997.

(25) Sheldrick, G. M. *SHELXL-97*, Program for Structure Refinement; University of Göttingen: Göttingen, Germany, 1997.

(26) Further details of the crystal structure determinations are available from the Cambridge Crystallographic Data Center, on quoting the depository numbers CCDC-162684 for **2**.