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Some 2,6-bis(dimethylamino)phenyl–mercury(II) and –boron complexes

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Abstract

As a continuation of our studies on the 2,6-bis(dimethylamino)phenyl ligand (R^-), we report on the synthesis and characterisation, including the X-ray structures, of HgR_2 and BCl(Ph)R. © 2002 Elsevier Science Ltd. All rights reserved.

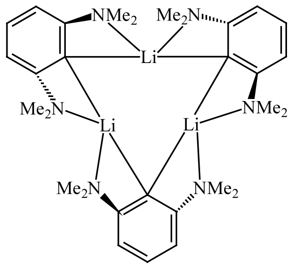
Keywords: Arylmetal complexes; Mercury(II); Boron; The 2,6-bis(dimethylamino)phenyl ligand

1. Introduction

In 1997, we drew attention to the potential of the 2,6-bis(dimethylamino)phenyl ligand $^-C_6H_3(NMe_2)_2$-2,6 ($\equiv R^-$); it was suggested that R^- might find a useful role in organometallic chemistry by virtue of facile on/off co-ordination of a pendant NMe_2 group [1], especially in catalytic systems. The ligand R^- differs from the more widely studied $^-C_6H_3(CH_2NMe_2)_2$-2,6 ($\equiv R'$) [2], in that in their metal complexes intramolecular $N \rightarrow M$ ligation is largely strain-free for (M–R′)-, but not (M–R)-, containing derivatives. This is clearly illustrated when comparing solid state and solution structures of Sn(Cl)R′ (**I**) [3], SnR_2 (**II**) [1] and Sn(Cl)R (**III**) [1,4]. In the crystalline complexes the angle α at the *ipso*-carbon is close to the sp^2 value in **I** [3], but in **II** it has an average value of 104° [1], while in **III** it is 112.1(4)° [4]. Furthermore, whereas in toluene-d_8 solution, ^1H NMR VT spectra showed that in **II** and **III** there was a rapid exchange process:

$$2\text{-}Me_2N \rightarrow Sn \rightleftharpoons 6\text{-}Me_2N \rightarrow Sn$$

at ambient temperature [1] (as also proved to be the rule for the Group 14 metal(II) complexes listed in Table 1), this was not the case for **I** [3].



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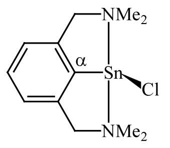

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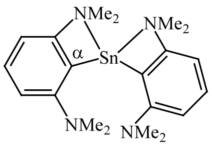

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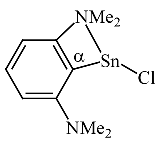

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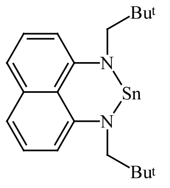

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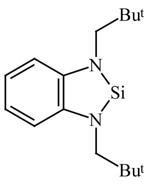

V

The conjugate acid RH of the ligand R^- was first prepared in 1970 from 1,3-diaminobenzene and trimethyl phosphate [5]. Almost 20 years later, LiR (**1**)

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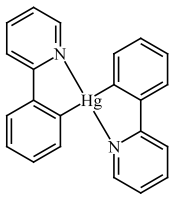
was obtained by direct lithiation of RH and shown to have a trinuclear structure in the crystal; this was retained in benzene or toluene solution, but in THF there was a monomer ⇆ trimer equilibrium [2].



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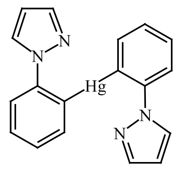

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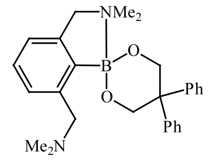

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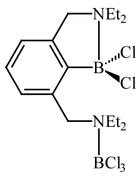


IX

We have used the ligand R^- to prepare the 12 crystalline germanium(II), tin(II) and lead(II) complexes listed in Table 1 [1,4,6–8]. For **1** [2] and eight others X-ray data are available; the principal point of interest in the present context and highlighted in Table 1 is that each R^- ligand has a single N···M intramolecular close contact in every case except in $GeR_2(BH_3)$ [6]. The germanium atom in the latter compound is four-coordinate, whereas in $SnR_2(BH_3)$ the tin atom is five-coordinate. In both, the MR_2 moiety behaves as a Lewis acid, as is also the case when SnX_2 (**IV**) [7] rather than BH_3 [6] is the electron pair acceptor. By contrast, with SnR_2 or $Sn[N(SiMe_3)_2]R$ as reagent, treatment with the thermally stable silylene **V** [abbreviated as Si(NN)] led to its product of insertion into the Sn–R or Sn–$N(SiMe_3)_2$ bond, respectively [8].

Table 1
Previously reported metal (M) crystalline complexes containing the 2,6-bis(dimethylamino)phenyl ligand (R^-)

Complex	Coordination number of M	Reference
$[Li(\mu\text{-}R)]_3$ (**1**)	2+2N	[2]
GeR_2	2+2N	[1]
SnR_2 (**II**)	2+2N	[1]
PbR_2	2+2N	[1]
$GeR_2(BH_3)$	3+1N	[6]
$SnR_2(BH_3)$	3+2N	[6]
Sn(Cl)R	2+1N	[1,4]
$Sn\{N(SiMe_3)_2\}R$	[a]	[1]
$Sn\{CH(SiMe_3)_2\}R$	[a]	[1]
$GeR_2(SnX_2)$ [b]	[a]	[7]
$SnR_2(SnX_2)$ [b]	3+2N	[7]
Sn{Si(NN)R}R [c]	2+2N	[8]
$Sn\{Si(NN)N(SiMe_3)_2\}R$ [c]	[a]	[8]

[a] Not determined.

[b] SnX_2 has the structure **IV**.

[c] Si(NN) is an abbreviation for **V**.

2. Experimental

All manipulations were carried out in flame-dried glassware under argon, using standard Schlenk techniques. Solvents were distilled from drying agents and degassed. Elemental analyses were carried out by Medac Ltd, Uxbridge. Melting points were determined in sealed capillaries under an argon atmosphere on an electrothermal apparatus and are uncorrected. The NMR spectra were recorded in benzene-d_6 at 298 K using a Bruker DPX 300 (^{1}H, 300.1; ^{13}C, 75.4 MHz) or AMX 500 (^{11}B, 160.4; ^{199}Hg, 89.1 MHz) instrument; the solvent resonances were used as internal references for ^{1}H and ^{13}C, while $^{11}BF_3(OEt_2)$ and $^{199}HgMe_2$ were the external references for the ^{11}B and ^{199}Hg NMR spectra, respectively. Electron-impact mass spectra (70 eV) were recorded using a Kratos MS 80 RF instrument. The compound $[Li(\mu\text{-}R)]_3$ (**1**) was prepared as described in the literature [2]. The other chemicals used were commercial samples (Aldrich).

2.1. Preparation of HgR_2 (**2**)

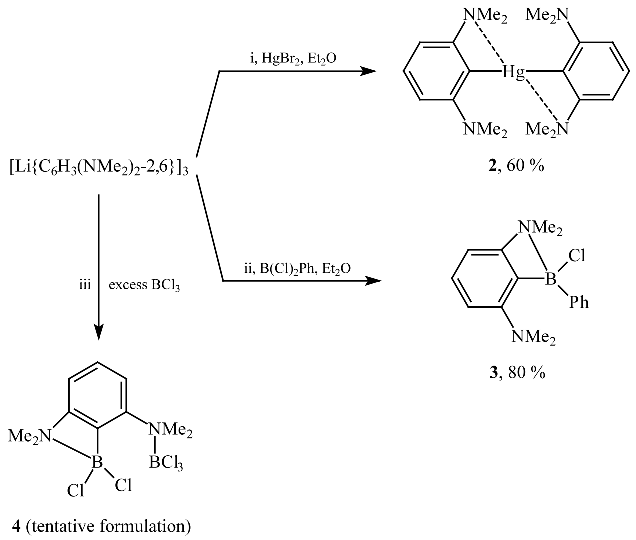
Solid $HgBr_2$ (0.63 g, 1.75 mmol) was added in small portions to a solution of LiR (0.60 g, 3.53 mmol) in Et_2O (30 cm^3) at room temperature. The reaction mixture was stirred for 20 h; the volatiles were then removed in vacuo. The residue was extracted with pentane (20 cm^3), the filtrate was concentrated in vacuo and stored at − 25 °C, yielding compound **2** (0.55 g, 60%), as colourless plate-shaped crystals. *Anal*. Found: C, 45.6; H, 5.71; N, 10.5. Required for $C_{20}H_{30}HgN_4$: C, 45.6; H, 5.74; N, 10.6%. M.p. 63.0–64.0 °C. Mass spectrum [m/z (%)]: 528 (35, $[M+1]^+$), 163 (100, $[R]^+$), 147 (59, $[R\text{–}Me-1]^+$), 120 (24, $[R\text{–}NMe_2+1]^+$), 77 (19, $[C_6H_5]^+$). ^{1}H NMR: δ 2.71 (s, 12H, NMe_2), 6.92 (d, 2H, m-H in R), 7.23 (t, 1H, p-H in R). $^{13}C\{^1H\}$ NMR: δ 45.90 (s, NMe_2), 113.78 (s, m-C in R), 129.24 (s, p-C in R), 160.45 (s, o-C in R), 161.32 (s, *ipso*-C in R). $^{199}Hg\{^1H\}$ NMR: δ − 642.0 (s).

2.2. Preparation of BCl(Ph)R (**3**)

Dichloro(phenyl)borane (1.25 cm^3, 9.63 mmol) was added dropwise at − 78 °C to a stirred solution of LiR (1.61 g, 9.46 mmol) in Et_2O (75 cm^3). The resulting

Table 2
Crystal data and structure refinement for HgR_2 (**2**) and BCl(Ph)R (**3**) [$R = C_6H_3(NMe_2)_2$-2,6]

	2	**3**
Formula	$C_{20}H_{30}HgN_4$	$C_{16}H_{20}BClN_2$
M	527.1	286.6
T (K)	173(2)	173(2)
Crystal system	orthorhombic	monoclinic
Space group	*Pbca* (no. 61)	$P2_1/n$ (no. 14)
a (Å)	13.311(6)	9.213(2)
b (Å)	19.253(9)	12.193(4)
c (Å)	8.012(3)	14.389(4)
β (°)	90	108.60(2)
U (Å^3)	2053(2)	1531.9(7)
Z	4	4
Total reflections	1803	3885
Independent reflections	1803	3676 [$R_{\text{int}} = 0.08$]
Reflections with $I > 2\sigma(I)$	953	2037
Data/restraints/parameters	1803/0/115	3676/0/181
R_1 [$I > 2\sigma(I)$]	0.033	0.066
wR_2 (all data)	0.094	0.167



Scheme 1.

viscous white suspension was allowed to warm to room temperature, stirred for 4 h and filtered. The filtrate was concentrated in vacuo and stored at −25 °C, then filtered; the residue was washed with pentane (2 × 10 cm^3) and dried in vacuo, yielding compound **3** (2.18 g, 80%) as a cream solid. *Anal.* Found: C, 66.8; H, 6.97; N, 9.63. Required for $C_{16}H_{20}BClN_2$: C, 67.1; H, 7.03; N, 9.77%. M.p. 104–106 °C. Mass spectrum [m/z (%)]: 286 (100, $[M-1]^+$), 251 (32, $[M\text{–}Cl]^+$), 209 (73, $[M\text{–}Ph-1]^+$), 163 (49, $[R]^+$), 147 (13, $[R\text{–}Me-1]^+$). ^{1}H NMR: δ 2.42 (s, 12H, NMe_2), 6.23 (d, 2H, *m*-H in R), 7.12–7.28 (m, 4H, R/Ph), 7.83 (d, 2H, *m*-H in Ph). $^{11}B\{^1H\}$ NMR: δ 14.7 (br s, $w_{1/2} = 200$ Hz). $^{13}C\{^1H\}$ NMR: δ 43.85 (s, NMe_2), 106.33 (s, *m*-C in R), 126.76 (s, R/Ph), 127.77 (s, R/Ph), 128.26 (s, R/Ph), 130.86 (s, R/Ph), 134.61 (s, *ipso*-C in Ph), 144.21 (s, *ipso*-C in R), 153.70 (s, *o*-C in R).

*2.3. Crystal data and refinement details for **2** and **3***

Suitable single crystals of HgR_2 (**2**) and BCl(Ph)R (**3**) were obtained from a concentrated diethyl ether (**2**) or THF–hexane (**3**) solution at −25 °C. Data were collected on an Enraf–Nonius CAD4 diffractometer using monochromated Mo Kα radiation, λ 0.71073 Å, with the crystals under a stream of cold nitrogen gas. Intensities were measured by an $\omega - 2\theta$ scan. Corrections were made for absorption using psi-scan measurements. The programs used for structure solutions and refinement (full-matrix on all F^2) were SHELXS-97 [9] and SHELXL-97 [10], respectively. Further details are in Table 2.

3. Results and discussion

The objectives of the present study were to prepare 2,6-bis(dimethylamino)phenyl–mercury(II) and –boron compounds.

The mercury(II) aryl HgR_2 (**2**) was sought as a potentially useful R^- or $R^\bullet$ transfer agent. A particular target was $^\bullet SnR_3$; crystalline, mononuclear tin(III) compounds are unknown, although earlier researches had revealed that in toluene solution $^\bullet Sn[CH(SiMe_3)_2]_3$ [11] and nitrogen analogues such as $^\bullet Sn[N(SiMe_3)_2]_3$ [11,12] were indefinitely stable at ambient temperature. In the event, there was no reaction between SnR_2 [1] and HgR_2. [Alternative strategies, based on $Sn(Br)R_3$ as a precursor, failed since this tin(IV) bromide proved to be inaccessible]. An added interest in HgR_2 was as a possible source of RHg–HgR {such mercury(I) organic compounds are unknown, an exception being XHg–HgX, $X = Si[Si(Me)_2SiMe_3]_3$ [13]}; but this also proved to be disappointing, as attempted reduction of **2** invariably yielded elemental mercury.

The compound $B(Cl)R_2$ appeared to be an attractive source of the crystalline $^\bullet BR_2$ radical or cation $[BR_2]^+$, both of which would be highly novel. However, we were unable unambiguously to displace a second chloride ion from BCl_3 using an excess of LiR, perhaps for steric reasons. An alternative strategy led us to the less hindered compound BCl(Ph)R (**3**).

Treatment of $HgBr_2$ with 2 equiv. of LiR in diethyl ether at ambient temperature yielded (step i of Scheme 1) HgR_2 (**2**) in 60% yield after crystallisation from pentane. The reaction between $B(Cl)_2Ph$ and an equivalent portion of LiR in diethyl ether at −78 °C afforded (step ii of Scheme 1) crystalline BCl(Ph)R (**3**) in high yield.

Each of the crystalline colourless **2** or cream **3** compounds was characterised by satisfactory elemental analysis, melting point (63–64 °C, **2**; 104–106 °C, **3**), multinuclear NMR and mass (molecular ions ± 1 mass units) spectra, and single crystal X-ray diffraction (seebelow). The mercury compound **2** was light-sensitive both in the solid state and in solution, a feature well documented for compounds containing Hg(II)–C bonds [14]. Substantial decomposition was significant in benzene solution only after approximately 3 weeks at ambient temperature.

The ^{199}Hg{^{1}H} NMR spectrum of **2** in benzene-d_6 showed a singlet at δ − 642. This value is at higher frequency than the recorded values for $HgPh_2$, which range from δ − 808 (acetone-d_6) to − 742 (CD_2Cl_2) [15]; but is close to the δ − 689 (in $CDCl_3$) reported for **VI**, for which intramolecular N···Hg interaction was inferred [16]. A further comparison is available with compound **VII**, having δ − 778 in $CDCl_3$, in which such a close N···Hg contact was excluded [17].

The ^{11}B{^{1}H} NMR spectrum of BCl(Ph)R (**3**) in benzene-d_6 showed a singlet at δ 14.7, consistent with the solution of **3** containing a single tetrahedrally co-ordinated boron compound [18] and having a strong N → B bond [19]. This chemical shift is close to the δ 14.4 for **VIII** [19].

Attempts were made to reduce compound **3** with potassium or potassium–graphite in order to obtain either the radical •B(Ph)R or its boron–boron bonded dimer. Although in each experiment the colour of the reaction mixture changed from colourless to fluorescent green, the ^{1}H NMR spectra showed only signals attributable to **3**. Likewise, efforts to generate the salts [B(Ph)R][BX_4], by treating **3** with $NaBPh_4$ or [NEt_3H][$B(C_6F_5)_4$], were unsuccessful; no evidence was found for a reaction having taken place.

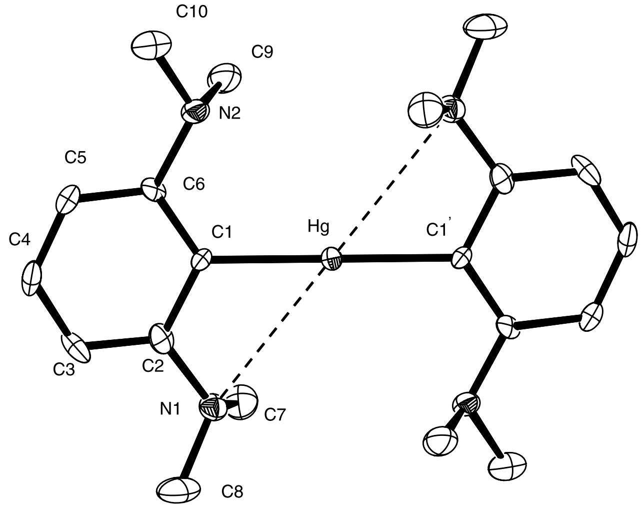


Fig. 1. The X-ray crystal structure of complex **2**.

Table 3
Selected bond distances (Å) and angles (°) for **2**

Bond distances		Bond angles	
Hg–C(1)	2.081(8)	C(1)–Hg–C(1′)	180
Hg···N(1)	3.077(8)	C(1)–C(2)–N(1)	116.7(7)
Hg···N(2)	3.247(8)	C(1)–C(6)–N(2)	117.6(7)
		Hg–C(1)–C(2)	117.0(5)
		Hg–C(1)–C(6)	123.3(6)
		Hg–C(1)···C(4)	168.7(6)

Symmetry element ′ is −*x*, −*y*, −*z*.

Treatment of BCl_3 with 2 equiv. of LiR in diethyl ether or toluene gave a white, sparingly soluble powder and unreacted LiR. This suggests that it was not possible to introduce two R^- ligands onto the boron atom, which may have a steric explanation. When an excess of boron trichloride was added to a solution of LiR, a complicated reaction mixture was obtained, from which a compound having somewhat greater solubility was eventually isolated. The ^{11}B{^{1}H} NMR spectrum showed two signals of approximately equal intensity at δ 11.4 and 17.5. A plausible formulation would be that shown in **4** (Scheme 1), by analogy with the crystallographically authenticated compound **IX**, for which ^{11}B spectral data were not recorded [20].

3.1. The molecular structure of HgR_2 (**2**)

The molecular structure of crystalline HgR_2 (**2**) is illustrated in Fig. 1; selected bond distances and bond angles are in Table 3.

Crystalline **2** is a centrosymmetric monomer, with a linear C(1)–Hg–C(1)′ vector. This structure is thus unexceptional. The Hg···N(1) and Hg···N(2) contacts of 3.077(8) and 3.247(8) Å are somewhat longer than those found in $Hg(C_6H_4CH_2NMe_2\text{-}2)_2$ (**X**) [2.89(1) Å] [21] or the 2-pyridylphenyl complex **VI** [2.798(7) Å] [16], but still fall well within the sum of the van der Waals radii for nitrogen (1.55 Å) [22] and a reasonable value of 1.73 Å for mercury [23]. Hence we suggest that there is a single weak Hg···N bonding interaction in **2** involving each of the R^- ligands. This is consistent with (i) the slight deviation from the sp^2 value in the angles C(1)–C(2)–N(1) and C(1)–C(6)–N(2) and (ii) magnitudes of the Hg–C(1)–C(2/6) angles which demonstrate that the fragment incorporating N(1) is slightly bent towards the mercury atom. The Hg–C(1) bond length of 2.081(8) Å in **2** is close to the 2.085(7) Å in $HgPh_2$ [24], the 2.10(2) Å in **X** [20] and the 2.098(8) Å in **VI** [16]. The mercury atom in **2** is 0.41 Å out of the C_6 aromatic mean plane, a situation similar to that in $HgPh_2$ in which, however, the deviation is only 0.1 Å [24]. The Hg–C(1)···C(4) angle in **2** is 168.7(6)°, the molecule being slightly bent at the C(1) atom. The phenyl planes are parallel to one another.

3.2. The molecular structure of BCl(Ph)R (**3**)

The molecular structure of crystalline BCl(Ph)R (**3**) is illustrated in Fig. 2; selected bond distances and bond angles are in Table 4.

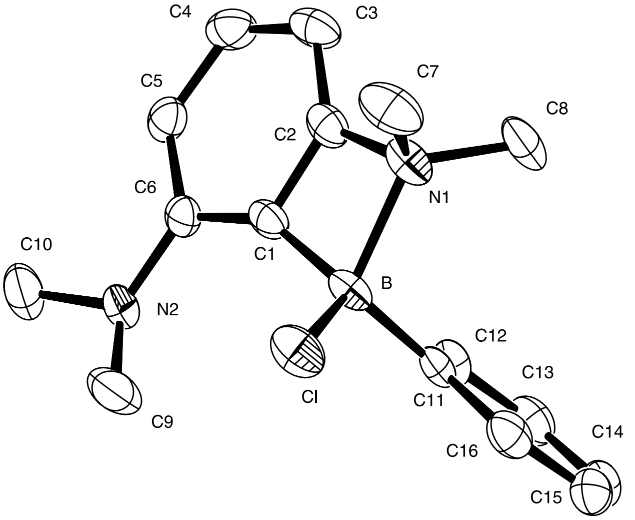


Fig. 2. The X-ray crystal structure of complex **3**.

Table 4
Selected bond distances (Å) and angles (°) for **3**

Bond distances		Bond angles	
B–C(1)	1.599(5)	C(1)–C(2)–N(1)	101.1(3)
B–C(11)	1.591(5)	B–C(1)–C(2)	92.4(3)
B–Cl	1.865(4)	C(1)–B–N(1)	82.3(2)
B···N(1)	1.740(4)	B–N(1)–C(2)	83.5(2)

Crystalline **3** is a monomer, with the boron atom in a four-coordinate, distorted tetrahedral environment. This distortion is evidently caused by the chelating R^- ligand, as reflected in the significantly narrower than the sp^2 value of the endocyclic angles at C(1) and C(2). The latter is also much narrower than in, for example, SnR_2 (**II**), 113.6(4)° [1], which is attributed to the relative sizes of the boron and tin atoms, $B \ll Sn$. The B–N(1)–C(2)–C(1) ring is planar, the sum of the endocyclic bond angles being 359.3°.

The B–Cl bond in **3**, 1.865(4) Å, is significantly longer than in the three-coordinate boron chloride $B(C_6F_5)_2Cl$, 1.746(5) Å [25], but is similar to those in the four-coordinated chloroboranes $B(Cl)Ph_2(THF)$, 1.893(2) Å [26], **IX** (av. 1.86 Å) [27], and the pyridine adduct of 1-chloro-1-borafluorene **XI**, 1.898(2) Å [28].

The mean B–C distance in **3** of 1.59 Å is slightly longer than in $B(C_6F_5)_2Cl$, 1.56 Å [25], but similar to that in the four-coordinated boron chlorides **IX**, 1.60 Å [27], or **XI**, 1.61 Å [28]. The B···N(1) distance in **3** is significantly longer than in $B(Br)_2(\kappa^2\text{-}C,N\text{–}C_6H_4CH_2\text{-}NMe_2\text{-}2)$, 1.625(1) Å [29] or **IX**, 1.649(5) Å [27]. Evidently the methylene group in the latter two complexes facilitates the $R'_2N \rightarrow B$ coordination ($R' = Me$ or Et), whereas this effect in **3** is subject to greater strain. The B···N separation in the spiro compound $B(C_6H_4CH_2\text{-}NMe_2\text{-}2)\{\kappa^2\text{-}O,O'\text{–}OCH_2C(Ph)_2O\}$ of 1.754(4) Å [30] is similar to that in **3**.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Centre, CCDC Nos. 170829 for **2** and **3**. Copies of this information may be obtained free of charge from The Director, CCDC 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-366-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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