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Synthesis of borylindenides; structure of $[Li(N,N',N''\text{-}Me_3\text{-}1,3,5\text{-}C_3H_6N_3)][1\text{-}C_9H_6\{B(NMe_2)_2\}]$

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Abstract

Borylation of lithium indenide (LiInd) affords indenylboranes. With $BCl(NMe_2)_2$ the primary allylic 1-indenyl derivative $IndB(NMe_2)_2$ (**1**) readily rearranges to the more stable vinylic bis(dimethylamino)(3-indenyl)borane **1**′. With $BCl(OCMe_2)_2$ the (1-indenyl)-1,3,2-dioxaborolane $IndB(OCMe_2)_2$ (**2**) is obtained. Both indenylboranes **1**′ and **2** readily undergo metalation when treated with lithium amides such as LiTMP or LDA to give 1-borylindenides $Li[C_9H_6B(NMe_2)_2]$ [Li(**3**)] and $Li[C_9H_6B(OCMe_2)_2]$ [Li(**5**)] in high yields. In the systems **1**′/LiInd and **2**/LiInd metalation equilibria are established with equlibrium constants $K \approx 1$ (**1’**/LiInd) and $K \approx 1.4$ (**2**/LiInd). The structure of the solvate $[Li(TMHT)][1\text{-}C_9H_6\{B(NMe_2)_2\}]$ (**4**) (TMHT = $N,N',N''\text{-}Me_3\text{-}1,3,5\text{-}C_3H_6N_3$) has been confirmed by X-ray diffraction. © 1998 Elsevier Science S.A. All rights reserved.

Keywords: Boron; Borylindene; Borylindenide; Crystal structure

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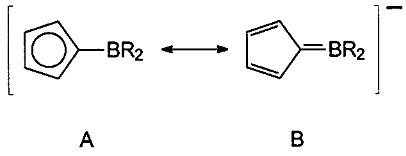
1. Introduction

In a previous paper we have developed the synthesis and the structural chemistry of alkali metal borylcyclopentadienides [1]. The bonding situation in these systems can be described by resonance formulae **A** and **B**. Formula **A** stresses the presence of an aromatic six-electron system while formula **B** emphasizes the perturbation of the cyclopentadienide ion by a boryl substituent. The interaction between the boryl group and the cyclopentadienyl ring is seen as a shortening of the Cl–B bond length and a bond lengths alternation in the ring. These effects are weak for a $B(NMe_2)_2$ group and strong for the 1,3,2-dioxaborolanyl group $B(OCMe_2)_2$ as well as for dialkylboryl groups BR_2 (R = Me, ^iPr). The strongly electron-accepting boryl groups $B(OCMe_2)_2$ and BR_2 produce an overall stabilization for the boryl substituted cyclopentadienide ions while this is not the case for the bis(dialkylamino)boryl group [2].



We have tried to extend this work to boryl-substituted indenes and indenides, and here we report our first results. We note that the chemistry of indenylboranes has not been studied in much detail [3]. Alkali metal borylindenides are unknown, but should be of interest as potential building blocks in organometallic chemistry.

2. Results and discussion

2.1. Indenylboranes

In principle, indenylboranes can exist as five constitu-

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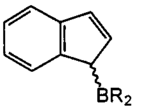
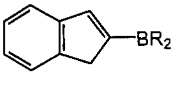
tional isomers. Three of these are indene derivatives with boryl substituents in the 1- (**C**), 2- (**D**), and 3-position (**E**), and the remaining two isomers are isoindenes with boryl substituents in the 1- (**F**) and 2-position (**G**). In the present paper we shall encounter indenylboranes of types **C** and **E**.



C



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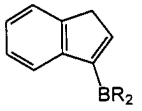

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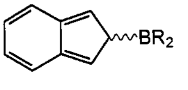
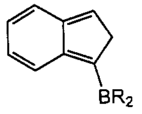


F

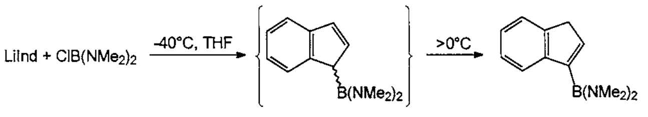


G

When lithium indenide (LiInd) in THF is treated with chlorobis(dimethylamino)borane $BCl(NMe_2)_2$ a smooth borylation takes place at −40°C. Monitoring the reaction mixture by ^1H and ^{13}C NMR spectroscopy demonstrates the primary formation of the allylic isomer **1** (the 1-indenyl derivative, type **C**) as the kinetic product. At temperature above 0°C isomer **1** rearranges to give the more stable vinylic isomer **1**′ (the 3-indenyl derivative, type **E**) (Scheme 1).

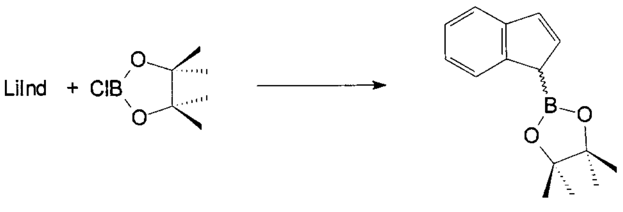
The analogous reaction of LiInd in hexane with the pinacol-derived 1,3,2-dioxaborolane $BCl(OCMe_2)_2$ [4] gave the allylic borane **2**. Note that THF cannot be used as solvent because ether cleavage is induced by $BCl(OCMe_2)_2$ to a considerable extent. In the case of **2**, isomerization to a vinylic isomer **2**′ is not observed during distillative workup (Scheme 2).

The difference in the behavior of **1** and **2** is remarkable. 1,5-sigmatropic hydrogen shifts have a much higher activation energy for indenes than for cyclopentadienes because they require isoindene intermediates. These are high-energy species because of the loss of aromatic stabilization [5]. Base catalyzed 1,3-hydrogen shifts are a second mode of rearrangement of indenes [6]; typical bases in this context are tertiary amines. Our observations suggest that **1** can isomerize spontaneously to give **1**′ because of the presence of the basic $B(NMe_2)_2$ group. On the other hand, **2** has no basic substituent and hence is unable to rearrange to **2**′ under mild conditions although the unknown isomer **2**′ is presumed to be more stable.



1 **1**′

Scheme 1.



2

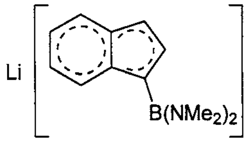
Scheme 2.

2.2. Metalation of the boranes *1*′ and *2*

There is now a considerable body of knowledge on the metalation of unsaturated organoboranes [7]. In most structural situations organoboranes react with bases under quaternization at boron. This quaternization is often irreversible and often may be followed by detrimental B–C bond breaking.

Three situations have been found which favor metalation. The first of these is relatively high C–H acidity of the unsaturated organoborane [2,8] as in the metalation of 1-phenylbora-3,5-cyclohexadiene [8]. Quaternization can be suppressed by double steric hindrance, that is by steric hindrance at the boron center and the use of a bulky base [9]. Finally, quaternization can be made less favorable and hence reversible by resonance stabilization of the boron center and the use of weaker bases. The metalation reactions of bis(dialkylamino)organoboranes with lithium amides are pertinent examples. Two such reactions have been reported in the literature, the metalation of pentadienylboranes such as $CH_2{=}CHCH{=}CHCH_2B(NR_2)_2$ (R = Me, Et) [10] and the metalation of cyclopentadienylboranes $C_5H_5B(NMe_2)_2$ [2]. Often combinations of the three situations are operative.

We now find that the 3-indenyl compound **1**′ can readily be metalated with more or less bulky bases such as lithium 2,2,6,6-tetramethylpiperidide (LiTMP) or LDA in THF to produce the borylindenide Li(**3**) in high yields. With *N*,*N*′,*N*″-Me_3-1,3,5-$C_3H_6N_3$ (TMHT) as auxiliary base a solvate [Li(TMHT)](**3**) (**4**) is obtained. Crystals of **4** suitable for crystallographic characterization were obtained in this way.



Li(3)

2 may be metalated similiar to **1**′ with bases such as LiTMP or LDA in THF. However, the metalation can also be achieved with lithium bis(trimethylsilyl)amide in hexane. This synthetic pathway is of advantage due to a more simple work up and a higher yield.

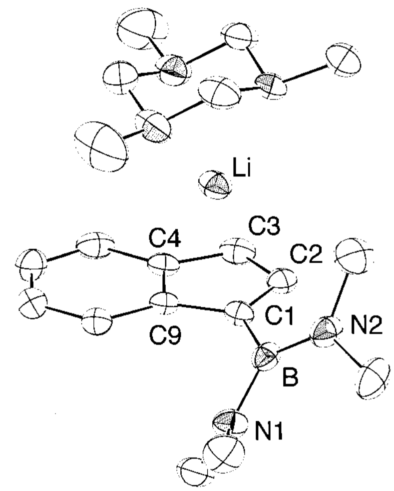
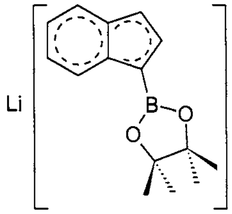


Fig. 1. Molecular structure of **4**. Thermal ellipsoids are shown at 50% probability. The hydrogen atoms have been omitted for clarity.



Li(5)

The metalation gives information about the relative stabilities of LiInd/Li(**3**) and LiInd/Li(**5**). To assess the relative stability of **1′**, we have studied the equilibrium of this borane with LiInd in THF in NMR tube experiments at ambient temperature. Spectra were measured after 1, 2 and 6 h after preparation of the sample. The equilibrium was established in less than 1 h and the equilibrium constant is $K \approx 1$ at ambient temperature. An analogous equilibrium is observed in the system LiInd/**2** with an estimated equilibrium constant of $K \approx 1.4$.

$$\mathbf{1'} + \text{LiInd} \rightleftharpoons \text{Li}(\mathbf{3}) + \text{Indene}$$

$$\mathbf{2} + \text{LiInd} \rightleftharpoons \text{Li}(\mathbf{5}) + \text{Indene}$$

Table 1
Selected bond distances (in pm)

Li–N3	225.5(4)	Li–C2	226.5(4)
Li–N4	228.0(4)	Li–C3	222.6(4)
Li–N5	213.7(4)	Li–C4	226.1(4)
Li–C1	236.3(4)	Li–C9	233.7(4)
C1–C2	142.0(3)	C4–C9	143.5(3)
C1–C9	144.0(3)	C5–C6	136.9(3)
C1–B	155.5(3)	C6–C7	139.9(3)
C2–C3	139.4(3)	C7–C8	136.5(3)
C3–C4	141.9(3)	C8–C9	141.3(3)
C4–C5	141.3(3)		
N1–C10	145.6(3)	N2–C12	145.3(3)
N1–C11	145.2(3)	N2–C13	144.9(3)
N1–B	143.6(3)	N2–B	143.0(3)

Table 2
Selected bond angles (in degrees)

C2–C1–C9	104.6(2)	C3–C4–C9	107.3(2)
C2–C1–B	128.4(2)	C1–C9–C4	109.1(2)
C9–C1–B	126.7(2)	N1–B–C1	119.0(3)
C1–C2–C3	111.5(3)	N2–B–C1	122.4(3)
C2–C3–C4	107.5(2)		

2.3. Structure of the solvate 4

The main question deals with the extend of interaction between the indenide and the attached aminoboryl group. Two structural aspects may help finding an answer; the bond distance between boron and the carbon of the ring and the intraring C–C distances in the five-membered ring (borylindenide vs borataindene; see formulae **A** and **B**) (Fig. 1, Table 1).

The B–C1 bond length amounts to 155.5 pm for **4**. We notice that this value is close to the same bond length in Na(THP)[$C_5H_4B(NMe_2)_2$] (154.5 pm) [2]. The typical B(sp^2)–C(sp^2) single bond length is 158 pm (see BPh_3 and $BMes_3$) [5], while the typical B(sp^2)–C(sp^2) double bond distance in a borataethene ion (see Li(H_2C=BMes)$_2$) [6] is close to 144 pm. We may conclude from these data that the π interactions in **3**, as in

Table 3
Atomic coordinates of non-hydrogen atoms

Atom	*x*	*y*	*z*	B_{eq}
N1	0.5790(3)	0.5580(2)	0.7242(2)	3.53(5)
N2	0.3215(3)	0.4879(2)	0.6065(2)	3.80(5)
N3	−0.0313(2)	0.1274(2)	0.6727(2)	3.49(5)
N4	0.0790(3)	−0.0008(2)	0.7791(2)	4.39(6)
N5	0.0892(3)	0.2313(2)	0.8451(2)	4.52(6)
C1	0.4501(3)	0.3077(2)	0.6860(2)	2.67(5)
C2	0.4166(3)	0.1827(2)	0.6075(2)	3.05(6)
C3	0.4428(3)	0.0736(2)	0.6502(2)	3.18(6)
C4	0.4955(3)	0.1266(2)	0.7601(2)	2.62(5)
C5	0.5343(3)	0.0665(2)	0.8441(2)	3.48(6)
C6	0.5789(3)	0.1468(3)	0.9447(2)	4.03(6)
C7	0.5857(3)	0.2877(3)	0.9658(2)	3.77(6)
C8	0.5464(3)	0.3498(2)	0.8874(2)	3.00(6)
C9	0.5012(3)	0.2711(2)	0.7822(2)	2.34(5)
C10	0.7422(3)	0.5297(3)	0.7485(2)	4.29(7)
C11	0.5678(4)	0.7015(3)	0.7590(2)	5.14(8)
C12	0.1643(4)	0.4049(3)	0.5726(3)	5.73(8)
C13	0.3371(4)	0.5989(3)	0.5554(2)	5.01(7)
C20	−0.0534(3)	−0.0025(3)	0.6992(2)	4.21(7)
C21	0.0708(4)	0.1020(3)	0.8738(2)	5.31(8)
C22	−0.0446(3)	0.2338(3)	0.7668(2)	4.73(8)
C23	−0.1505(3)	0.1292(4)	0.5831(2)	6.07(9)
C24	0.0799(5)	−0.1345(3)	0.8019(3)	8.2(1)
C25	0.1053(5)	0.3464(4)	0.9380(3)	9.0(1)
B	0.4483(3)	0.4536(3)	0.6714(2)	2.93(6)
Li	0.2371(5)	0.1604(4)	0.7244(3)	3.5(1)

The anisotropic thermal parameters are given in the form of their isotropic equivalents: $(4/3)[a_{11}^{2}+b^{2}\beta_{22}+c^{2}\beta_{33}+ab(\cos\gamma)\beta_{12}+ac(\cos\beta)\beta_{13}+bc(\cos\alpha)\beta_{23}]$; in 10^4 pm^2.

$Na(THP)[C_5H_4B(NMe_2)_2]$, are rather less important. Considering the intraring C–C distances we find lengthened distances for C1–C2/C1–C9 (142.0/144.0 pm) and C3–C4 (141.9 pm) and a shortened C2–C3 (139.4 pm) distance. Considering the standard deviations there is a weak but significant tendency of bond length alternation as observed on the 3σ level (Table 2).

The dihedral angle between the indenide ring and the boryl function amounts to 42° while this value in $Na(THP)[C_5H_4B(NMe_2)_2]$ is 29°. This difference can be understood as a consequence of the presence of the six-membered ring in the α-position to C1 of **3**.

Summarizing the experimental data of the structure there is clear evidence for a pertubation of the five-membered ring by the aminoboryl group (Table 3).

3. Conclusion

Work currently in progress shows that the methods developed here can be used to produce borylindenides with several boryl substituents or with further functionalities. Thus, the borylindenides can be used to synthesize sandwich type complexes and open the route to a new organometallic chemistry.

4. Experimental

Reactions were carried out under an atmosphere of nitrogen by means of conventional Schlenk techniques. Pentane was distilled from Na/K alloy, hexane from potassium, and THF from sodium benzophenone ketyl. NMR spectra were used to judge the purity of the new compounds.

NMR spectra were recorded on a Varian Unity 500 spectrometer (^{1}H, 500 MHz; ^{13}C, 125.7 MHz; ^{11}B, 160.4 MHz) and on a Varian EM 390A (^{11}B, 27.8 MHz). Mass spectra was recorded on a Finnigan MAT-95 spectrometer.

*4.1. Bis(dimethylamino)(3-indenyl)borane (**1′**)*

$BCl(NMe_2)_2$ (29.30 g, 0.22 mol) was added dropwise to a stirred solution of lithium indenide (LiInd) [11] (25.64 g, 0.21 mol, 0.95 equivalents) in THF (300 ml) at − 78°C. A color change from pink to orange was taken to indicate the equivalence point. After the solution had warmed up to ambient temperature, the solvent was removed in a vacuum. Pentane was then added. The lithium chloride formed was filtered off and carefully washed with several portions of pentane. After removal of the solvent, the residue was warmed up under vacuum (10^{-6} bar) and **1′** was collected by condensation (35.92 g, 80%) as colorless solid; mp 38.5°C.

MS (EI, 70 eV): m/z ($I_{rel.}$) = 214 (100, M^+), 171 (57, M^+—$MeNCH_2$), 116 (45, $C_9H_8^+$). Anal. Found: C, 72.48; H, 9.00; N, 12.70. $C_{13}H_{19}BN_2$ calc.: C, 72.92; H, 8.94; N, 13.08%.

^{1}H NMR ($CDCl_3$): $\delta = 2.72$ (s, 2 NMe_2), 3.44 (m, CH_2), 6.52 (t, $^{3}J_{12} = 1.7$ Hz, 2-H), C_6H_4: 7.17 (tm, 1H), 7.27 (tm, 1H), 7.29 (dm, 1H, 4-H), 7.49 (dm, 1H, 7-H); assignments of 4-H and 7-H on the basis of NOE spectra. ^{13}C NMR ($CDCl_3$): $\delta = 40.23$ (CH_2), 40.83 (NMe), 139.48 (C-2), C-3 not observed, C_6H_4: 122.11, 123.31, 123.73, 125.96, 144.38,148.94. ^{11}B NMR (28 MHz, $CDCl_3$): $\delta = 32$.

*4.2. (1-Indenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (**2**)*

$BCl(OCMe_2)_2$ [2] (6.75 g, 41.59 mmol) was added dropwise to a stirred suspension of LiInd (4.82 g, 41.59 mmol) in hexane (150 ml) at − 78°C. The equivalence point was seen as a color change from white to light yellow. After the suspension had warmed up to ambient temperature, the LiCl formed was filtered off and washed with several portions of hexane. The solvent was then removed in a vacuum to leave **2** (9.10 g, 91%) as a white powder; mp 50.5°C.

MS (EI, 70 eV): m/z ($I_{rel.}$) = 242 (38, M^+), 116 (100, $C_9H_8^+$). Anal. Found: C, 74.02; H, 7.93. $C_{15}H_{19}BO_2$ calc.: C, 74.41; H, 7.91%.

^{1}H NMR ($CDCl_3$): $\delta = 1.20$ (s, 2 Me), 1.22 (s, 2 Me), 3.48 (s br, 1-H), 6.68 (dd, 2-H), 6.93 (ddd, 3-H), $^{3}J_{23} = 5.4$, $^{3}\partial_{12} = 2.0$, $^{4}\partial_{13} = 2.3$, $^{4}J_{34} = 0.7$ Hz, C_6H_4: 7.18 (td, 1H), 7.25 (tm, 1H), 7.43 (dm, 1H), 7.54 (dm, 1H). ^{13}C NMR ($CDCl_3$): $\delta = 24.65$ (Me), 24.86 (Me), 83.84 (CO), C-1 not observed, 131.12 and 135.79 (C-2 and C-3), C_6H_4: 121.11, 123.69, 124.39, 125.73, 144.61 (only one signal for C-3a and C-7a). ^{11}B NMR (28 MHz, $CDCl_3$): $\delta = 32$.

*4.3. Lithium [1-{bis(dimethylamino)boryl}indenide] (Li(**3**))*

Compound **1** (2.09 g, 9.78 mmol) in THF (20 ml) was added dropwise to a stirred solution of LiTMP [12] (1.38 g, 9.35 mmol in THF (60 ml) at − 78°C within 5 min. After the solution had warmed up to ambient temperature, the solid was transferred onto a frit and washed twice with hexane. Drying in a vacuum gave Li(**3**) (1.44 g, 70%) as a white powder, highly sensitive to air and humidity. LDA is also suitable as base.

^{1}H NMR (THF-d_8): $\delta = 2.78$ (s, 2 NMe_2), 6.00 (dd, 3-H), 6.70 (d, 2-H), $^{3}J_{23} = 3.4$, $^{4}J_{34} = 0.7$ Hz, C_6H_4: 6.43 (tm, 1H), 6.49 (tm, 1H), 7.24 (tm, 2H). ^{13}C NMR (THF-d_8): $\delta = 41.99$ (Me), C-1 not observed, 95.06 (C-3), 114.22, 114.62, 119.19, 122.12, 127.13, 132.87, 135.87. ^{11}B (28 MHz, THF-d_8): $\delta = 32$.

*4.4. The N,N′,N″-trimethyl-hexahydro-1,3,5-triazine (TMHT) solvate [Li(TMHT)][$C_9H_6B(NMe_2)_2$] (**4**)*

Salt Li(**3**) (0.26 g, 1.19 mmol) was dissolved in ether (10 ml). Dropwise addition of TMHT (0.18 g, 1.39 mmol) and cooling to − 30°C gave **4** as large colorless crystals. Anal. Found: C, 65.53; H, 9.48; N, 19.40. $C_{19}H_{33}BLiN_5$ Calc.: C,65.15; H, 9.78; N, 19.99%.

*4.5. Lithium [1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)indenide (Li(**5**))*

Compound **2** (2.90 g, 11.93 mmol) in hexane (30 ml) was added dropwise to a stirred solution of LiN($SiMe_3$)$_2$ [13] (2.01 g, 11.93 mmol) in hexane (40 ml) at − 78°C. After the solution had warmed up to ambient temperature, the salt was transferred onto a frit and washed twice with hexane. Drying in a vacuum gave Li(**5**) (2.48 g, 85%) as a white powder, highly sensitive to air and humidity. LiTMP, LDA and LiInd can also be used as base.

^{1}H NMR (THF-d_8): $\delta = 1.25$ (s, 2 CMe_2), 6.01 (dd, 3-H), 6.96 (d, 2-H), $^3J_{23} = 3.5$, $^4J_{34} = 0.6$ Hz, C_6H_4: 6.49 (1m, 1H), 6.54 (tm, 1H), 7.27 (dm, 1H), 7.82 (dm, 1H). ^{13}C NMR (THF-d_8): $\delta = 25.78$ (Me), 80.97 (CO), C-1 not observed, 98.50 (C-3), 115.14, 115.52, 119.10, 122.22, 126.98, 134.19, 138.43. ^{11}B NMR (160 MHz, THF-d_8): $\delta = 30.5$.

*4.6. X-ray structure determination of **4***

$C_{19}H_{33}BLiN_5$ molecular weight 349.3 g mol^{-1}, triclinic, $\bar{P}$(No. 2), $a = 838.4(5)$, $b = 1025.6(3)$, $c = 1306.2(2)$ pm, $\alpha = 103.67(2)$, $\beta = 94.95(4)$, $\gamma = 96.91(4)°$, $Z = 2$, $V = 1.076(1)$ nm^3, $d_{calc} = 1.078$ g cm^{-3}, (Mo–$K_\alpha = 0.6017$ cm^{-1}, $F(000) = 380.0$. Diffraction data were collected with an ENRAF-Nonius CAD4-diffractometer at 203 K, Mo–K_α radiation (graphite monochromator, $\lambda = 71.07$ pm), colorless crystal (needle), crystal size 0.3 × 0.4 × 0.8 mm, ω-2Θ scans ($3 < \Theta < 28°$). All data were corrected for Lorentz and polarization effects, using the SDP system of programs [14]. Of the 6128 data collected, 2736 were unique with intensities $I > 1\ \sigma(I)$ and only those were used in the structure solution and refinement. The structure was solved by direct methods (SHELXS-86) [15] and difference fourier maps. The hydrogen atoms were refined isotropically, except the ones in the triazane ligand. Those hydrogen atoms were treated as riding with isotropic thermal parameters of $B_H = 1.3 \cdot B_C$. Convergence was obtained for 307 parameters with the agreement factors $R = 0.058$ and $R_w = 0.071$, using statistical weights $w = 1/\sigma^2(F_0)$. The final difference fourier map showed a maximum of residual electron density of $0.253 \cdot 10^{-6}$ e pm^{-3} [16].

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