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Hydroximato complexes of boron—the X-ray crystal structures of dicyclohexylammonium bis(benzohydroximato)borate chloroform solvate and tetra-*n*-propylammonium bis(benzohydroximato)borate

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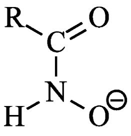
Abstract

Four new alkylammonium bis(benzohydroximato)borates have been prepared and characterized by elemental analysis, IR and ^{1}H NMR spectra. The crystal and molecular structures of dicycohexylammonium bis(benzohydroximato)borate chloroform solvate (**1**·CHCl$_3$) and tetra-*n*-propylammonium bis(benzohydroximato)borate (**3**) have been determined by X-ray diffraction methods. In both examples the bis(hydroximato)borate anions show the expected spirochelate structure with slightly distorted tetrahedral BO$_4$ coordination. The C–N as well as the C–O bonds within the chelate rings have considerable double bond character. Bond lengths and angles of the BONCO heterocyle are in good agreement with those observed in *N*-methylacetohydroxamatoboron complexes (Can. J. Chem. 55 (1977) 1, Can. J. Chem. 56 (1978) 1676). In the crystal structure of **1**·CHCl$_3$ the bis(benzoydroximato)borate anions are linked to the dicyclohexylammonium cations by strong N–H···O and N–H···N hydrogen bonds.

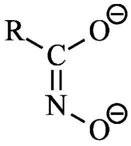
Keywords: Boron complexes; Benzohydroximate anion; Synthesis; Crystal structures

1. Introduction

Hydroxamic acids of the general composition RC(O)NHOH are well known as versatile chelating agents [3,4]. Their metal chelates are derived from the hydroxamate(1−) anion **a** or the hydroximate(2−) anion **b** as shown below:



a



b

Most of the well characterized metal complexes contain the hydroxamate(1−) anions **a**.

Several molecular boron chelates involving N-substituted hydoxamate(1−) anions have been described [1,2,5–11]. The crystal and molecular structures of difluoro-*N*-methylacetohydroxamatoboron [1], diphenyl-*N*-methylacetohydroxamatoboron [2] and diphenyl-*N*-(1-hydroxycyclohexyl)benzohydroxamatoboron [11] have been determined. Several anionic boron chelates involving the hydroximate(2−) anion have been described: dihydroxohydroximatoborates [12] and bis(hydroximato)borates [13–15]. So far, no structural informations are available concerning boron chelates with N-unsubstituted hydroxamic anions. We consider the structure investigation of the hydroximatoborates of some interest, in order to compare bonding parameters of the chelate with those of boron hydroxamates [1,2,11].

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2. Experimental

2.1. Instrumental

IR spectra were recorded on a BOMEM FT-IR model BM 100 spectrometer. ^{1}H NMR spectra were measured on a BRUKER AC250 instrument with TMS as internal standard. Elemental analyses (CHN) were determined with a FISONS EA-1108 equipment. Enraf–Nonius CAD4 diffractometers were used for the diffraction measurements. Routine FAB$^-$ mass spectra have been recorded on a TSQ spectrometer (Finnigan) with nitrobenzylalcohol as matrix.

2.2. Preparation of the boron complexes

The hydroximatoborates were prepared as described previously [14], with slight modifications. Solvents and commercially available chemicals (H_3BO_3, benzohydroxamic acid, di-*n*-butylamine, dicyclohexylamine, tetra-*n*-butylammonium bromide, tetra-*n*-propylammonium bromide and silver oxide) were used as purchased.

2.2.1. Dicyclohexylammonium bis(benzohydroximato)borate (*1*)

A mixture of 0.068 g H_3BO_3 (1.10 mmol), 0.302 g benzohydroxamic acid (2.20 mmol) and 0.200 g dicyclohexylamine (1.1 mmol) in 25 ml C_6H_6 was heated under reflux for 8 h, using a Dean-Stark trap for azeotropic elimination of water. After evaporation of the C_6H_6, the solid residue was dissolved in 15 ml CH_2Cl_2, 15 ml *n*-C_6H_{14} was added and the mixture was kept at −15 °C. After 48 h the crystals were filtered off and washed with *n*-C_6H_{14} yielding 0.474 g of **1** (yield: 91%). M.p. 226–229 °C. *Anal*. Found: C, 67.12; H, 6.93; N, 9.12. Calc. for $C_{26}H_{34}BN_3O_4$: C, 67.39; H, 7.40; N, 9.07%. NMR (25 °C): ^{1}H (250 MHz, $CDCl_3$): δ 1.02 (m, 6H, CH_2), 1.47 (m, 4H, CH_2), 1.56 (m, 6H, CH_2), 1.95 (m, 4H, CH_2), 3.10 (m, 2H, CH), 7.26 (m, 6H, C_6H_5), 7.78 (m, 4H, C_6H_5). FTIR (KBr): 3035m (ν NH), 2934s (ν CH_2), 2856s (ν CH_2), 1597ms (C_6H_5), 1562ms (C_6H_5), 1499m, 1447m (δ CH_2), 1388vs (ν OCN), 1125s (ν_{as} $^{10}BO_4$), 1085vs (ν_{as} $^{11}BO_4$), 987s, 942m (δ_{as} BO_4), 792m (ν ONC), 742m (ν ONC), 691m (C_6H_5) (IR data: vs, very strong; s, strong; ms, medium strong; m, medium).

2.2.2. Di-n-butylammonium bis(benzohydroximato)borate (*2*)

Compound **2** was prepared from 0.080 g H_3BO_3 (1.30 mmol), 0.357 g benzohydroxamic acid (2.60 mmol) and 0.170 g di-*n*-butylamine (1.31 mmol) as described above. Yield: 0.450 g (84%). M.p. 113–114 °C. *Anal*. Found: C, 64.06; H, 6.45; N, 10.28. Calc. for $C_{22}H_{30}BN_3O_4$: C, 64.24; H, 7.35; N, 10.22%. NMR (25 °C): ^{1}H (250 MHz, $CDCl_3$): δ 0.80 (t, 6H, CH_3), 1.3 (m, 4H, CH_2–CH_3), 1.79 (m, 4H, CH_2–CH_2–CH_2), 3.10 (t, 4H, N–CH_2) 7.49 (m, 6H, C_6H_5), 7.91 (m, 4H, C_6H_5). FTIR (KBr): 3122m (ν NH), 2956ms (ν CH_2/CH_3), 1594ms (C_6H_5), 1563s (C_6H_5), 1498m, 1470m, 1446m (δ CH_2), 1380s (ν OCN), 1124s (ν_{as} $^{10}BO_4$), 1086vs (ν_{as} $^{11}BO_4$), 993s, 942m (δ_{as} BO_4), 773m (ν ONC), 738m (ν ONC), 703m (C_6H_5), 690m (C_6H_5).

2.2.3. Tetra-n-propylammonium bis(benzohydroximato)borate (*3*)

A mixture of 0.031 g H_3BO_3 (0.50 mmol), 0.058 g Ag_2O (0.25 mmol), 0.137 g benzohydroxamic acid (1.00 mmol), 0.133 g tetra-*n*-propylammonium bromide (0.50 mmol) in 25 ml C_6H_6 was heated to reflux for 8 h as described above. The solid was filtered off and extracted with 15 ml CH_2Cl_2. After vacuum evaporation 0.201 g of **3** (yield 86%) were obtained as colorless crystals (m.p. 136 °C). *Anal*. Found: C, 66.41; H, 8.10; N, 8.88. Calc. for $C_{26}H_{38}BN_3O_4$: C, 66.81; H, 8.19; N, 8.99%. NMR (25 °C): ^{1}H (250 MHz, $CDCl_3$): δ 0.84 (t, 12H, CH_3), 1.50 (m, 8H, CH_2–CH_3), 3.03 (t, 8H, CH_2–N), 7.32 (m, 6H, C_6H_5), 7.86 (m, 4H, C_6H_5). FTIR (KBr): 2975ms (ν CH_2/CH_3), 2945m (ν CH_2/CH_3), 1592ms (C_6H_5), 1558ms (C_6H_5), 1488ms, 1461m, 1447m (δ CH_2), 1377vs (ν OCN/δ CH_3), 1114s (ν_{as} $^{10}BO_4$), 1078s (ν_{as} $^{11}BO_4$), 981s, 944m (δ_{as} BO_4), 780m (ν ONC), 736m (ν ONC), 701ms (C_6H_5), 674m. FAB$^-$ MS; *m/z*: 281.2, 162.1.

2.2.4. Tetra-n-butylammonium bis(benzohydroximato)borate (*4*)

Compound **4** has been prepared from 0.062 g H_3BO_3 (1.00 mmol), 0.274 g benzohydroxamic acid (2.00 mmol), 0.240 g tetra-*n*-butylammonium bromide (1.00 mmol) and 0.116 g Ag_2O (0.50 mmol) as described for **3**, yielding 0.470 g (90%). M.p. 165 °C. *Anal*. Found: C, 67.64; H, 8.79; N, 8.07. Calc. for $C_{30}H_{46}BN_3O_4$: C, 68.83; H, 8.86; N, 8.03%. NMR (25 °C): ^{1}H (250 MHz, $CDCl_3$): δ 0.84 (t, 12H, CH_3), 1.40 (m, 16H, CH_2–CH_3), 3.14 (t, 8H, N–CH_2), 7.32 (m, 6H, C_6H_5), 7.86 (m, 4H, C_6H_5). FTIR (KBr): 2966ms (ν CH_2/CH_3), 2929m (ν CH_2/CH_3), 1594m (C_6H_5), 1558m (C_6H_5), 1490m, 1382vs (ν OCN), 1118s (ν_{as} $^{10}BO_4$), 1083vs (ν_{as} $^{11}BO_4$), 981 s, 944m (δ_{as} BO_4), 770m (ν ONC), 737m (ν ONC), 695s (C_6H_5), 670m (C_6H_5).

2.3. X-ray crystal structure determinations

Crystals of the chloroform solvate $\mathbf{1}\cdot CHCl_3$ and of **3** suitable for structure analysis were obtained from $CHCl_3$–*n*-C_6H_{14} solutions (1:1) of the complexes. The cell constants were calculated from 25 reflexes with a wide range of 2θ. The program HELENA [16] was used for the data reduction. Both complexes crystallize monoclinic. The space groups were assigned using the criterion of choosing the setting which corresponds to the lowest monoclinic β angle. The structures were solved by direct methods with SHELXS-97 [17] and refined with SHELXL-97 [18]. A ψ-scans [16] absorption correction was performed and all non-hydrogen atoms were refined with anisotropic displacement parameters in both structures. The hydrogen atoms were found in the Fourier-map in **1**·$CHCl_3$ and calculated at idealized positions and refined using the riding model in **3**. Crystal data and more detailed information about the structure determinations are given in Table 1. Labeled diagrams of the structures of **1**·$CHCl_3$ and **3** are shown in Figs. 1 and 2, respectively. Selected bond lengths and angles are given in Table 2.

3. Results and discussion

3.1. Synthesis of the bis(benzohydroximato)borates

Bis(benzohydroximato)borates were obtained in high yield in simple one-step reactions according to:

$$H_3BO_3 + 2C_6H_5CONHOH + R_2NH \rightarrow [R_2NH_2][B(C_6H_5CONO)_2] + 3H_2O$$

for **1** and **2**, or:

$$2H_3BO_3 + 4C_6H_5CONHOH + 2[R_4N]Br + Ag_2O \rightarrow 2[R_4N][B(C_6H_5CONO)_2] + 2AgBr + 7H_2O$$

for **3** and **4**.

The use of silver oxide and a quaternary ammonium bromide, which resulted in the formation of insoluble silver bromide and easily removable water as byproducts, proved to be a favorable synthetic route for tetraalkylammonium bis(benzohydroximato)borates.

These quaternary ammonium cations acted efficiently, as expected due to their favorable size, as counter ions for the crystallographic study also in absence of the N–H···O and N–H···N hydrogen bonds between the anionic complex and the counter ion, normally present in the lower alkylammonium salts, as in **1**·$CHCl_3$. Good quality crystals of **3**, additionally

Table 1
Crystal data and structure refinement for **1** $CHCl_3$ and **3**

Compound	**1**·$CHCl_3$	**3**
Empirical formula	$C_{27}H_{35}BCl_3N_3O_4$	$C_{26}H_{38}BN_3O_4$
Formula weight	582.74	467.40
Temperature (°C)	−65(2)	20(2)
Wavelength (pm)	154.056	71.073
Radiation	Cu Kα	Mo Kα
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	Pn
Unit cell parameters		
a (pm)	1302.88(9)	1118.7(3)
b (pm)	1127.18(17)	967.64(8)
c (pm)	1987.55(10)	1278.8(3)
β (°)	91.484(8)	108.89(2)
V (pm^3)	2917.9(5) × 10^6	1309.7(5) × 10^6
Z	4	2
Absorption coefficient (mm^{-1})	3.146	0.079
Crystal size (mm)	0.5 × 0.4 × 0.1	0.40 × 0.20 × 0.20
Method	ω scans	ω scans
θ range (°)	5–65	3–25
Index ranges (h, k, l)	$-1 \rightarrow 15$, $0 \rightarrow 13$, $-23 \rightarrow 20$	$-13 \rightarrow 13$, $0 \rightarrow 11$, $-15 \rightarrow 15$
Collected reflections	5511	4606
Unique reflections	4843	4606
R_{int}	0.0930	0.0000
Observed data [$I > 2\sigma(I)$]	2774	2518
Absorption correction	ψ-scans [16]	ψ-scans [16]
Min/max transmission	0.76443/0.95645	0.84812/0.94824
Structure refinement	full-matrix least-squares on F^2	full-matrix least-squares on F^2
Weighting scheme	$w = 1/[\sigma 2(F_o^2)+(0.0810P)^2+1.8367P]$, $P = (F_o^2+2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2)+(0.1097P)^2+0.0139P]$, $P = (F_o^2+2F_c^2)/3$
Hydrogen treatment	Fourier-map	riding model
Refined parameters	483	308
Structure factors [$I > 2\sigma(I)$]	$R_1 = 0.0647$, $wR_2 = 0.1583$	$R_1 = 0.0758$, $wR_2 = 0.1712$
Goodness-of-fit, S	1.039	1.049
Extinction coefficient	not refined	0.056(7)
Absolute structure parameter	none	0.00001
Programs used	HELENA, PLATON [16], SHELXS-97 [17], SHELXL-97 [18]	HELENA, PLATON [16], SHELXS-97 [17], SHELXL-97 [18]

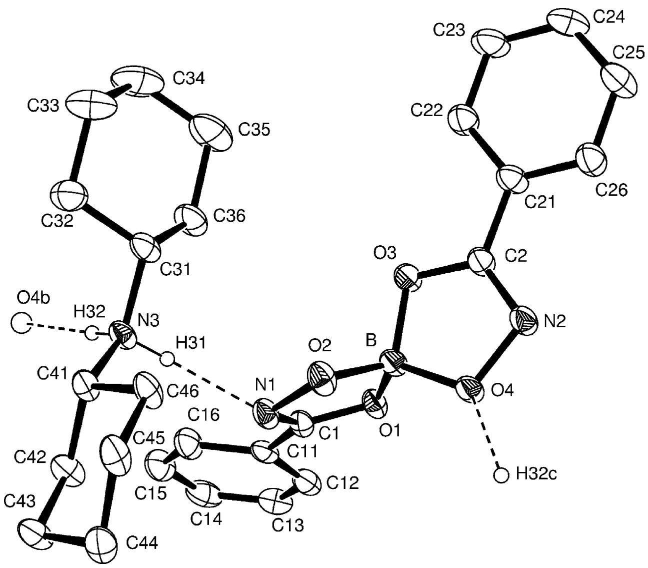


Fig. 1. ORTEP plot (30% probability) of **1** in **1**·$CHCl_3$. The H-atoms are omitted for clarity, except H(31) and H(32). The dashed lines indicate the H-bonds.

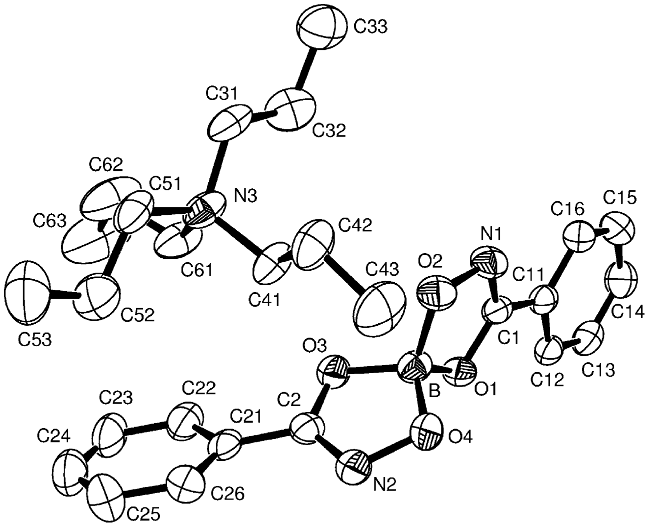


Fig. 2. ORTEP plot (30% probability) of **3**. The H-atoms are omitted for clarity.

without solvent of crystallization, could be obtained for this purpose.

3.2. Spectroscopic characterization

The infrared spectra of the presented compounds (KBr pellets) exhibit the expected characteristic features of the bis(hydroximato)borate moiety [14]. The strongest absorption at about 1080 cm^{-1} corresponds to ν_{as} $^{11}BO_4$, showing the component for ν_{as} $^{10}BO_4$ at about 1120 cm^{-1}. Another strong absorption band at approximately 985 cm^{-1} can be attributed to a deformation mode δ_{as} BO_4. The strong absorption band at about 1380 cm^{-1} most probably involves a stretching vibration of the OCN group (ν OCN) within the hydroximato chelate. The N–H stretching vibration of the dialkylammonium salts **1** and **2** appears as multicomponent absorption pattern in the range 3050–2400 cm^{-1}, characteristic for strong hydrogen bonds.

Table 2
Selected bond lengths (pm) and angles (°) for **1**·$CHCl_3$ and **3**

	1·$CHCl_3$	**3**
Bond lengths		
B–O(1)/B–O(3) [a]	147.0(6)/147.3(5)	146.7(8)/147.9(8)
B–O(2)/B–O(4) [a]	146.8(6)/147.2(5)	146.4(8)/146.9(8)
O(1)–C(1)/O(3)–C(2) [a]	132.6(4)/133.6(5)	133.4(6)/132.7(7)
C(1)–N(1)/C(2)–N(2) [a]	128.1(5)/128.9(5)	126.5(7)/126.9(7)
N(1)–O(2)/N(2)–O(4) [a]	142.0(4)/144.6(4)	141.6(6)/142.6(6)
C(1)–C(11)/C(2)–C(21) [a]	146.6(6)/147.2(5)	147.2(8)/147.6(8)
N(3)–H(31)···N(1)	286.7(6)	
N(3)–H(31)	88.0(6)	
H(31)···N(1)	199(6)	
(N3)–H(32)···O(4) [b]	283.6(4)	
N(3)–H(32)	88.0(5)	
H(32)···O(4) [b]	196(6)	
Bond angles		
O(1)–B–O(2)/O(3)–B–O(4) [a]	102.3(3)/101.9(3)	102.7(5)/101.6(5)
O(1)–B–O(3)	113.3(3)	113.9(5)
O(1)–B–O(4)	113.6(4)	112.8(4)
O(2)–B–O(3)	112.6(4)	112.5(4)
O(2)–B–O(4)	113.6(3)	113.8(5)
O(1)–C(1)–N(1)/ O(3)–C(2)–N(2) [a]	116.0(4)/117.7(3)	117.5(5)/118.5(5)
C(1)–N(1)–O(2)/ C(2)–N(2)–O(4) [a]	107.2(3)/104.7(3)	106.5(4)/105.1(4)
N(1)–O(2)–B/N(2)–O(4)–B [a]	107.0(3)/109.0(3)	107.8(4)/108.9(4)
O(1)–C(1)–C(11)/ O(3)–C(2)–C(21) [a]	120.3(4)/119.9(3)	118.7(4)/118.7(5)
N(1)–C(1)–C(11)/ N(2)–C(2)–C(21) [a]	123.7(4)/122.5(4)	123.8(5)/122.8(6)
N(3)–H(31)–N(1)	178.0(6)	
N(3)–H(32)–O(4) [b]	175.0(5)	

[a] Values for two non equivalent chelate rings, respectively.

[b] Symmetry operation: $-x+1$, $y+1/2$, $-z+1/2$.

The ^1H NMR spectra of the described hydroximatoborates in $CDCl_3$ show the expected patterns.

The FAB^- MS-Spectrum of **3** (nitrobenzylalcohol matrix) shows *m*/*z* peaks at 281.2 and 162.1, corresponding to $[B(C_6H_5CONO)_2]^-$ (281) and $[OB(C_6H_5CONO)]^-$ (162), respectively.

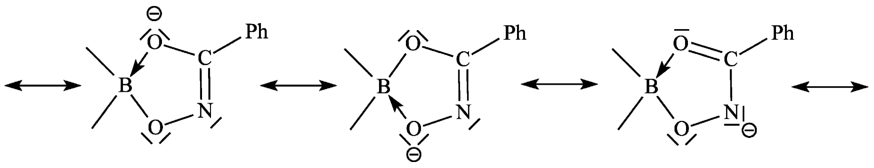
3.3. Crystal structures

The crystal structures show the expected spirochelate structure of the bis(benzohydroximato)borate anions with slightly distorted tetahedral BO_4 coordination. The B–O distances (mean value 147.0 pm) are in good agreement with the mean value of 146.8 pm for compounds containing the BO_4^- moiety [19]. The endocyclic O–B–O-angles lie between 101.6° and 102.7°, the exocyclic O–B–O-angles (112.5°–113.9°) are slightly larger compared to the ideal tetrahedral angle. The C–N bond lengths (126.5–128.9 pm) are close to the standard value of a double bond. The C–O bonds (132.6–133.6 pm) are considerably shortened with respect to a single bond [19]. The N–O bond lengths (141.6–144.6 pm) correspond to a single bond and are longer compared to the mean N–O distance of 139.4 pm in oximes [19].

The chelate rings are essentially planar with maximum atomic displacements from the mean plane between 0.80 and 6.1 pm. The phenyl groups are slightly distorted with respect to the adjacent chelate rings, showing torsion angles from 4.80° to 27.18°.

Bond lengths and angles of the BONCO heterocyles in **1** and **3** show only slight variations and are in fairly good agreement with those observed in difluoro-*N*-methylacetohydroxamatoboron (**5**) [1]. The B–O distances in **1** and **3** (mean value 147.0 pm) are slightly shorter compared to **5** (mean value 148.3 pm) while the N–O bonds are considerably longer (mean value 142.7 for **1** and **3**, and 134.2 pm for **5**).

The structural data indicate a considerable delocalization of π electrons in the boron chelates **1** and **3**, as well as in the hydroxamatoboron complexes [1,2]. We propose the following resonance structures having major contribution to the bonding hybrid in the benzohydroximatoborates:



Strong hydrogen bonds between the dicyclohexylammonium nitrogen N(3) and the atoms N(1) and O(4), from different complex molecules, occur in $1 \cdot CHCl_3$, forming a chain like arrangement, as shown in Fig. 1. The distances N(3)–H(31)···N(1) = 286.7(6) and N(3)–H(31)···O(4b) = 283.6(4) pm are considerably shorter compared to the mean values for these types of H-bond, 307 and 293 pm [20], respectively. The corresponding bond angles are N(3)–H(31)–N(1) = 178(6)° and N(3)–H(31)–O(4b) = 175(5)°.

4. Conclusions

The comparison of crystallographic data shows that the bond lengths among the chelate ring in hydroximate(2−)- and in N-substituted hydroxamate(1−) boron complexes do not differ significantly, suggesting that the presence of an additional negative charge in the hydroximate anion does not change substantially the electron density distribution in the chelate ring, as it could be expected. Thus, the bonds among the rings are not affected due to the presence of the lone electron pair at the hydroximate's nitrogen atom in the bis(benzohydroximato)borate complex. Also the involvement of chelate's atoms in strong hydrogen bonds between the complex and the counter ion do not promote any substantial changes in the ring's bonds in $1 \cdot CHCl_3$, in comparison with the values found in **3**.

5. Supplementary material

Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 178861 and 178862 for compounds $1 \cdot CHCl_3$ and **3**. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2, 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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