

Ate complexes of Ge(II) and Sn(II) with bidentate ligands $[\text{LiE}^{14}(\text{OCH}_2\text{CH}_2\text{NMe}_2)_3]_2$ ($\text{E}^{14} = \text{Ge}, \text{Sn}$): synthesis and structure [☆]

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Abstract

The reaction of equimolar quantities of $\text{LiOCH}_2\text{CH}_2\text{NMe}_2$ and $\text{E}^{14}(\text{OCH}_2\text{CH}_2\text{NMe}_2)_2$ ($\text{E}^{14} = \text{Ge}, \text{Sn}$) in ether yielded new *ate* complexes $[\text{LiE}^{14}(\text{OCH}_2\text{CH}_2\text{NMe}_2)_3]_2$ ($\text{E}^{14} = \text{Ge}$ (**1**), Sn (**2**)) with bidentate ligands. The compounds **1** and **2** are white crystalline substances which are highly soluble in THF and pyridine and very sensitive to the traces of oxygen and moisture. The structures of these compounds are studied by X-ray diffraction analysis. The *ate* complexes **1** and **2** are powerful nucleophiles and may be employed as ligands (neutral) in the coordination chemistry of the transition metals. The electronegative O-substituents at the divalent E^{14} atoms render them less oxidizable than alkyl- or aryl-substituted derivatives, and the bidentate ligands, owing to intramolecular donor–acceptor interactions, make them more thermodynamically stable compared to monodentate ligands.

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1. Introduction

The chemistry of $[\text{E}^{14}\text{R}_3]^{(-)}$ anionic trisubstituted metalate(II) of group 14 elements – Ge(II), Sn(II) and Pb(II) *ate*-complexes – containing amido- or oxo-ligands has attracted considerable attention in recent years [1–6]. The structural features of E^{14}R_3 fragment, reduced ability of the E^{14} atom to the oxidation due to the presence of electronegative N- or O-substituents and increased stability $\text{R}_3\text{E}^{14}\text{–M}$ (M = transition metal) bond to the homolytic cleavage allow to employ these

compounds in transition metals coordination chemistry as components of potential catalytic systems, as precursors for electronic and ceramic materials, etc. The metalated tin reagents of the general type $\text{R}_3\text{SnM}'$ ($\text{M}' = \text{alkali metal}$) are well established as versatile tools in organic synthesis [7,8]. However, their high reactivity, thermal lability and the difficulties of crystallization have inhibited further studies. In order to overcome these difficulties, the use of polydentate ligand systems which offer the stabilizing effect of chelation along with the shielding of a large sector in the coordination sphere of the low valent metal was thought to offer a route to systems of greater stability.

In a development of the concept of the “active ligand periphery”, Gade and co-workers have synthesized and studied by the X-ray diffraction analysis of the triamido-lithium–stannate with peripheral 2-anisyl groups. In this

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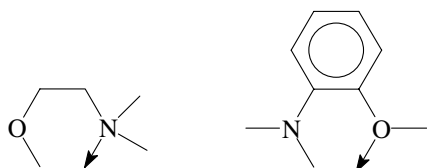
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polydentate amide the lithium cation is intramolecularly solvated by two of the three *ortho*-anisyl groups [4].

We have recently studied a series of sterically non-hindered monomeric compounds $\text{RE}^{14}\text{OCH}_2\text{CH}_2\text{NMe}_2$ ($\text{E}^{14} = \text{Ge}$, $\text{R} = \text{Cl}$, OCOMe , $\text{OCH}_2\text{CH}_2\text{NMe}_2$; $\text{E}^{14} = \text{Sn}$, $\text{R} = \text{OCH}_2\text{CH}_2\text{NMe}_2$) containing bidentate ligands [9,10]. Their high thermodynamical stability is due to the presence of intramolecular coordination bonds $\text{E}^{14} \leftarrow \text{N}$.

In this paper we report the synthesis of *ate* complexes $[\text{LiE}^{14}(\text{OCH}_2\text{CH}_2\text{NMe}_2)_3]_2$ ($\text{E}^{14} = \text{Ge}$ (**1**), Sn (**2**)) with bidentate ligands $\text{OCH}_2\text{CH}_2\text{NMe}_2$ and discussed the structural patterns in the solid-state and in solution as established by X-ray diffraction study and NMR spectroscopy.

It is very interesting to note that the β -dimethylaminoethoxy substituent is the “inverted” bidentate ligand with respect to the *ortho*-anisylamido substituent.



β -dimethylaminoethoxy group *ortho*-anisylamido group

2. Experimental

2.1. General procedures

All manipulations were carried out under purified argon atmosphere using standard schlenk and high-vacuum-line techniques. The solvents commercially available were purified by conventional methods, and distilled immediately prior to use. NMR spectra were recorded on Bruker AM-360 NMR spectrometer at 360.134 MHz (^1H), 90.555 MHz (^{13}C) and 111.92 MHz (^{119}Sn) in THF-d_8 . Chemical shifts are relative to SiMe_4 for H, C or indirectly referenced to TMS via the solvent signals, and relative to SnMe_4 for ^{119}Sn . Accuracy of coupling constant determination is ± 0.1 Hz, accuracy of chemical shift measurements is ± 0.01 ppm (^1H), ± 0.05 ppm (^{13}C) and ± 0.2 ppm (^{119}Sn).

2.2. *Bis[tris(dimethylaminoethoxy)lithiumgermanate]*, $[\text{LiGe}(\text{OCH}_2\text{CH}_2\text{NMe}_2)_3]_2$ (**1**)

To a solution of $\text{LiOCH}_2\text{CH}_2\text{NMe}_2$ [prepared from $\text{HOCH}_2\text{CH}_2\text{NMe}_2$ (0.37 g, 4.1 mmol) in 10 mL of Et_2O and 3.1 mL of halogen-free CH_3Li (in diethyl ether, 1.30 N, 4.1 mmol)] was added to a solution of bis(dimethylaminoethoxy)germanium [9] (0.99 g, 4.0 mmol) in 15 mL of Et_2O . The mixture was stirred for 1 h at room temperature. The filtered solution was reduced in volume to

5–10 mL and kept at -5 °C to give white crystals of **1** (0.76 g, 56%) which are suitable for X-ray analysis. ^1H NMR: δ 2.21 (s, 18H, Me_2N), 2.39 (t, 4H, CH_2N , $^3J_{\text{HH}} = 6.0$ Hz), 3.72 (t, 4H, CH_2N , $^3J_{\text{HH}} = 6.0$ Hz). ^{13}C NMR: δ 46.08 (Me_2N), 59.85 (CH_2N), 64.31 (CH_2O). Anal. Calc. for $\text{C}_{24}\text{H}_{60}\text{Ge}_2\text{Li}_2\text{N}_6\text{O}_6$: C, 41.91; H, 8.79; N, 12.22. Found: C, 41.83; H, 8.70; N, 12.48%.

2.3. *Bis[tris(dimethylaminoethoxy)lithium-stannate]*, $[\text{LiSn}(\text{OCH}_2\text{CH}_2\text{NMe}_2)_3]_2$ (**2**)

To a solution of $\text{LiOCH}_2\text{CH}_2\text{NMe}_2$ [prepared from $\text{HOCH}_2\text{CH}_2\text{NMe}_2$ (0.46 g, 5.1 mmol) in 10 mL of Et_2O and 3.9 mL of halogen-free CH_3Li (in diethyl ether, 1.30 N, 5.1 mmol)] was added a solution of bis(dimethylaminoethoxy)tin [9] (1.51 g, 5.1 mmol) in 5 mL of Et_2O . The mixture was stirred for 1 h at room temperature and then kept at -12 °C to give white crystals of **2** (1.38 g, 69%) which are suitable for X-ray analysis. Anal. Calc. for $\text{C}_{24}\text{H}_{60}\text{Li}_2\text{N}_6\text{O}_6\text{Sn}_2$: C, 36.96; H, 7.75; N, 10.77. Found: C, 36.73; H, 7.70; N, 10.68%. ^1H NMR: δ 2.22 (s, 18H,

Table 1
Crystallographic data for **1** and **2**

Compound	1	2
Empirical formula	$\text{C}_{24}\text{H}_{60}\text{Li}_2\text{N}_6\text{O}_6\text{Ge}_2$	$\text{C}_{24}\text{H}_{60}\text{Li}_2\text{N}_6\text{O}_6\text{Sn}_2$
Formula weight	687.84	780.04
Temperature (K)	200(2)	240(2)
Crystal size (mm)	$0.4 \times 0.3 \times 0.2$	$0.4 \times 0.3 \times 0.3$
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
<i>a</i> (Å)	9.088(3)	10.080(2)
<i>b</i> (Å)	10.665(3)	10.232(2)
<i>c</i> (Å)	11.022(3)	11.617(2)
α (°)	104.731(7)	67.433(4)
β (°)	102.451(6)	65.705(4)
γ (°)	110.405(6)	62.974(4)
<i>V</i> (Å ³)	912.1(4)	943.8(3)
<i>Z</i>	1	1
<i>D_c</i> (Mg m ⁻³)	1.252	1.372
<i>F</i> (000)	364	400
μ (mm ⁻¹)	1.687	1.362
θ range (°)	2.03–28.01	1.98–30.05
Index range	$-11 \leq h \leq 11$ $-14 \leq k \leq 14$ $-14 \leq l \leq 14$	$-14 \leq h \leq 14$ $-14 \leq k \leq 14$ $-16 \leq l \leq 16$
No. of reflections collected	9230	11253
No. of unique reflections	4302	5471
No. of reflections with $I > 2\sigma(I)$	2852	3899
<i>R</i> ₁ ; <i>wR</i> ₂ ($I > 2\sigma(I)$)	0.0519; 0.1105	0.0353; 0.0923
<i>R</i> ₁ ; <i>wR</i> ₂ (all data)	0.0818; 0.1202	0.0525; 0.1027
Data/restraints/parameters	4302/0/181	5471/0/208
GOF on <i>F</i> ²	0.922	1.022
Max shift/error	0.001	0.001
Largest difference peak and hole (e Å ⁻³)	0.863/−0.351	1.400/−0.954
Abs. cor. <i>T</i> _{max} ; <i>T</i> _{min}	0.729; 0.552	0.928; 0.786

Me₂N), 2.35 (t, 4H, CH₂N, ³J_{HH} = 5.5 Hz), 3.83 (t, br, 4H, CH₂N, ³J_{HH} = 5.5 Hz). ¹³C NMR: δ 45.55 (Me₂N), 60.98 (CH₂N), 65.14 (br, CH₂O). δ_{119 Sn} –285.1.

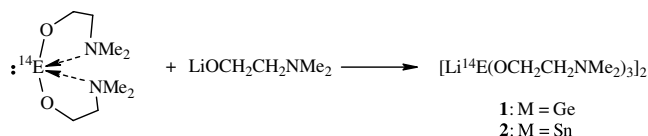
2.4. X-ray structure determination

Data were collected on a Bruker SMART CCD 1000 diffractometer and corrected for Lorentz and polarization effects and for absorption [11]. For details see Table 1. The structures were determined by direct methods and by full-matrix least squares refinement with anisotropic thermal parameters for non-hydrogen atoms. The two CH₂CH₂NMe₂-groups coordinated to Li atoms in **2** were disordered over two sites, with occupancies 0.7:0.3. The hydrogen atoms were placed in calculated positions and refined in riding model with fixed thermal parameters. All calculations were carried out by use of the SHELXTL PLUS (PC Version 5.0) program [12].

3. Results and discussion

3.1. Synthesis

Complexes **1** and **2** were prepared by the reaction of equimolar quantities of LiOCH₂CH₂NMe₂ and E¹⁴(OCH₂CH₂NMe₂)₂ (E¹⁴ = Ge, Sn) in ether.



Compounds **1** and **2** are white crystalline substances which are highly soluble in THF and pyridine and very sensitive to the traces of oxygen and moisture.

3.2. Solid-state structures of [LiE¹⁴(OCH₂CH₂NMe₂)₃]₂ (E¹⁴ = Ge (**1**), Sn (**2**))

Three types of interactions between the anionic cage and the solvated lithium cation are possible in lithium metallate(II) of the group 14 elements containing amido- or oxo-ligands:

- solvent-separated salt;
- the lithium ion is directly bound to the group 14 element, which is thought to be a primarily ionic metal–metal bond interaction;
- the lithium ion is attached to the nitrogen or oxygen atom in a bridging coordination mode.

The structures of **1** and **2** are found to be the c type of anion–cation association. The lithium cation in these compounds as well as in the triamido-lithium–stannate with peripheral 2-anisyl groups (see above) is intramolecularly solvated by one of the three β-dimethylami-

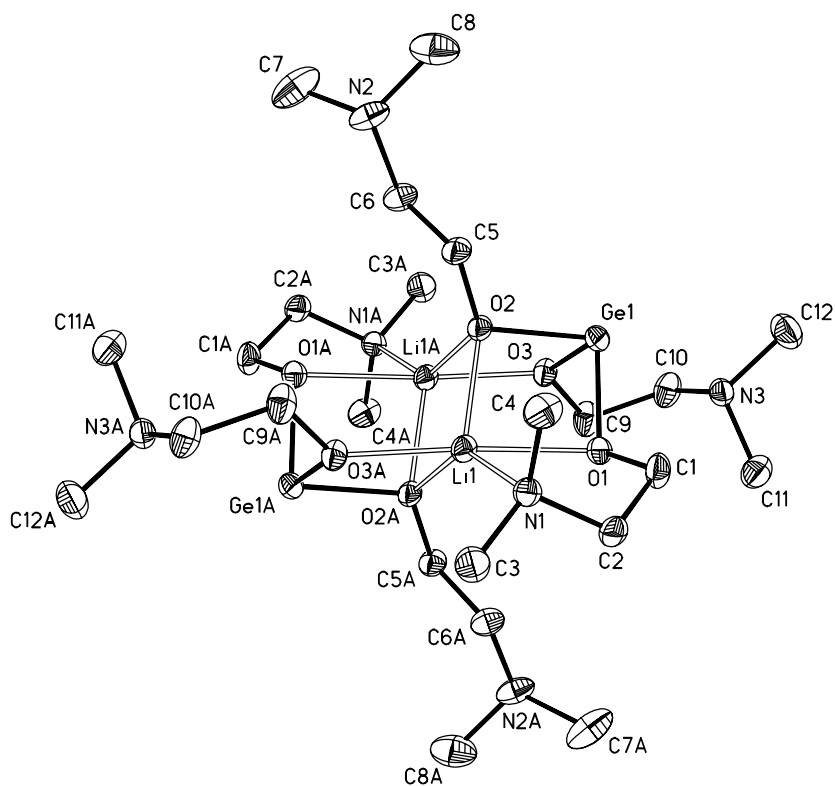


Fig. 1. The structure of compound **1** (50% probability ellipsoids). The labeling A denotes symmetrically equivalent atom with respect to inversion centre.

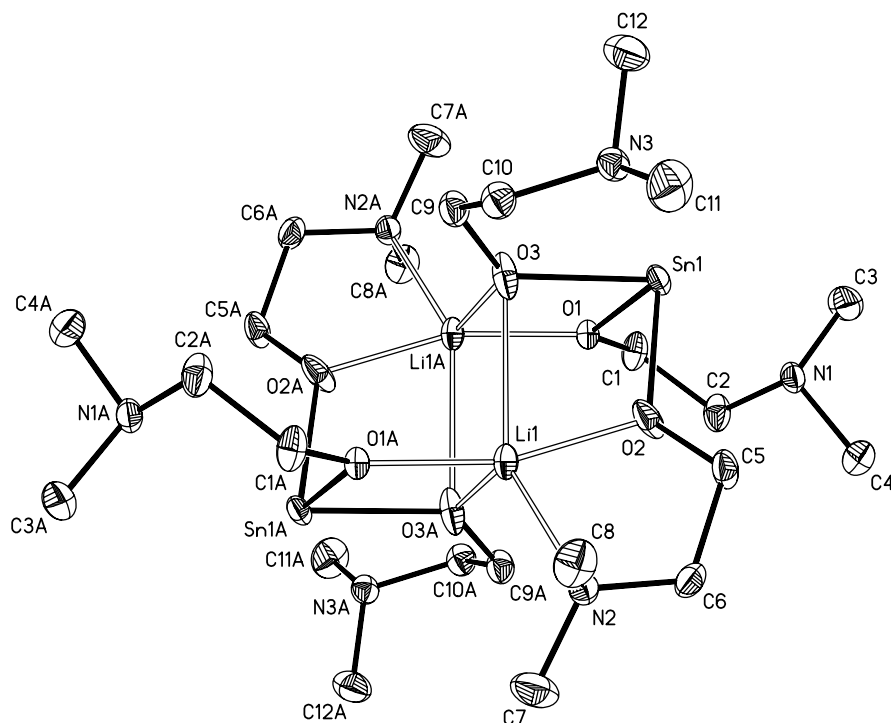


Fig. 2. The structure of compound **2** (50% probability ellipsoids). The labeling A denotes symmetrically equivalent atom with respect to inversion centre.

Table 2
Selected bond lengths (Å) and angles (°) for **1**

Ge(1)–O(1)	1.888(2)	O(3)–Li(1A)	2.033(6)
Ge(1)–O(2)	1.954(2)	N(1)–Li(1)	2.180(6)
Ge(1)–O(3)	1.876(2)	Ge(1)···Li(1)	3.010(6)
O(1)–Li(1)	2.193(6)	Ge(1)···Li(1A)	3.012(5)
O(2)–Li(1)	2.019(6)	Li(1)···Li(1A)	2.713(11)
O(2)–Li(1A) ^a	2.034(6)		
O(1)–Ge(1)–O(2)	88.09(10)	O(1)–Li(1)–O(3A)	176.2(3)
O(1)–Ge(1)–O(3)	92.88(10)	O(2)–Li(1)–O(2A)	96.0(2)
O(2)–Ge(1)–O(3)	83.18(9)	O(2)–Li(1)–O(3A)	104.4(2)
Ge(1)–O(1)–Li(1)	94.8(2)	O(2A)–Li(1)–O(3A)	77.4(2)
Ge(1)–O(2)–Li(1)	98.5(2)	O(1)–Li(1)–N(1)	80.3(2)
Ge(1)–O(2)–Li(1A)	98.1(2)	O(2)–Li(1)–N(1)	120.5(3)
Ge(1)–O(3)–Li(1A)	100.7(2)	O(2A)–Li(1)–N(1)	142.4(3)
O(1)–Li(1)–O(2)	78.6(2)	O(3A)–Li(1)–N(1)	100.0(2)
O(1)–Li(1)–O(2A)	100.0(2)	Li(1)–O(2)–Li(1A)	84.0(2)

^a The labeling A denotes symmetrically equivalent atom with respect to inversion centre.

noethoxy groups. The molecular structures of **1** and **2** with the atomic numbering schemes are shown in Figs. 1 and 2, with pertinent bond lengths and angles given in Tables 2 and 3.

The compounds **1** and **2** are centrosymmetric dimers. This molecular species is structurally similar to Sn₂Li₂(OBU')₆ (**3**) [13] and Pb₂Li₂(HFIP)₆ (HFIP = OCH(CF₃)₂) (**4**) [14]. Their common structural feature is a E¹⁴Li₂O₆ cage, which is built of two seco-norcube E¹⁴Li₂O₃ units, sharing a Li₂O₂ four-membered ring. A molecular inversion center lies at the center of this ring relating unprimed to primed atoms. Alkoxide groups of

this ring bridging to E¹⁴ atom are μ³ and the E¹⁴–μ–OR–Li bridges are μ². The E¹⁴ O₃-fragment is distorted trigonal pyramidal with stereochemically active E¹⁴ lone pair as is commonly observed for E^{14(II)}R₃ complexes of the VSEPR type AX₃E.

While the Sn–O (2.095(2), 2.055(2) and 2.087(2) Å in **2** and 2.084(3), 2.099(3) and 2.095(3) Å in **3**) and Pb–O (2.279(14), 2.268(14) and 2.291(11) Å in **4**) bond lengths are essentially invariant, the Ge–μ²–O (1.876(2) and 1.888(2) Å) and Ge–μ³–O (1.954(2) Å) distances in **1** are significantly different. As expected due to the difference of the atomic radii of Ge, Sn and Pb, the O–M–O angles

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

Sn(1)–O(1)	2.095(2)	O(3)–Li(1A)	1.926(5)
Sn(1)–O(2)	2.055(2)	N(2)–Li(1)	2.258(6)
Sn(1)–O(3)	2.087(2)	Sn(1)···Li(1)	3.313(6)
O(1)–Li(1A) ^a	1.968(5)	Sn(1)···Li(1A)	3.110(5)
O(2)–Li(1)	1.935(6)	Li(1)···Li(1A)	2.929(11)
O(3)–Li(1)	2.511(7)		
O(1)–Sn(1)–O(2)	91.66(11)	O(1A)–Li(1)–O(3A)	82.6(2)
O(1)–Sn(1)–O(3)	75.87(8)	O(2)–Li(1)–O(3)	73.0(2)
O(2)–Sn(1)–O(3)	80.86(14)	O(2)–Li(1)–O(3A)	113.5(3)
Sn(1)–O(1)–Li(1A)	99.9(2)	O(3)–Li(1)–O(3A)	98.5(2)
Sn(1)–O(2)–Li(1)	112.2(2)	O(1A)–Li(1)–N(2)	103.9(3)
Sn(1)–O(3)–Li(1)	91.7(2)	O(2)–Li(1)–N(2)	80.5(2)
Sn(1)–O(3)–Li(1A)	101.5(2)	O(3)–Li(1)–N(2)	143.9(2)
O(1A)–Li(1)–O(2)	160.2(3)	O(3A)–Li(1)–N(2)	114.5(3)
O(1A)–Li(1)–O(3)	93.9(2)	Li(1)–O(3)–Li(1A)	81.5(2)

^aThe labeling A denotes symmetrically equivalent atom with respect to inversion centre.

decrease from **1** [O–Ge–O: 83.18(9)°, 88.09(10)°, 92.88(10)°] to **3**, **2** [O–Sn–O: **3**: 79.7(1)°, 80.1(1)°, 97.6(1)°; **2**: 75.87(8)°, 80.86(14)°, 91.66(11)°] and **4** [O–Pb–O: 74.2(4)°, 74.8(3)°, 88.3(4)°]. These variations of the O–M–O angles are in relation with the expected decreasing hybridizations of the s and p orbitals when moving down a group in the periodic table for heavier main group elements [15–33].

The Li atoms in **1** and **2** have a distorted trigonal bipyramidal geometry with one amino group in equatorial position (Tables 2 and 3). The coordination site at Li atom occupied the amino group in **1** and **2** is free in **3** and **4**. The Li–N bond length in **1** (2.180(6) Å) is much shorter than the Li–N bond length in **2** (2.258(6) Å), but both the Li–N bonds are remarkably longer than those observed in most lithium amides [34]. The distortion of trigonal bipyramidal geometry of Li atom in **2** is more considerable than that in **1**. It is reflected in the difference of the equatorial Li–O bond lengths (2.019(6) and 2.034(6) Å in **1** and 1.926(5) and 2.511(7) Å in **2**) as well as in mean of the axial O–Li–O angle (176.2(3)° in **1** and 160.2(3)° in **2**). It should be noted that the distortion of pseudo trigonal bipyramidal geometry of Li atom in **4** is less considerable than that in **3**. The Li₂O₂ four-membered rings in **1** and **4**, compared to those in **2** and **3**, are nearly a perfect square.

The packing of molecules in crystals of **1** and **2** is very close to that in crystal of **3**.

4. Conclusion

The combination of thermodynamic stabilization of the trioxymetalates(II) **1** and **2** by the intramolecular coordination interactions of the donor atoms of the bidentate ligands, along with the well-defined orientation and high variability of the O-substituents, has established tripodal trioxymetalates with bidentate ligands

as a versatile new class of ligands in transition-metal chemistry. The electronegative O-substituents at the divalent E¹⁴ atoms render them less oxidizable than alkyl- or aryl-substituted derivatives.

The synthesis of transition metal complexes with metals such as rhodium or palladium suggests that the group 14 *ate* complexes may find applications in the development of novel homogeneous catalysts. This aspect of reactivity of the trioxygermanates(II) and -stannates(II) with bidentate ligands is the focus of our current studies.

5. Supplementary material

Crystallographic Data for **1** and **2** have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 215120 and 215121. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk).

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