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Syntheses and crystal structures of four-coordinate aryloxo neodymium complexes

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Abstract

Two four-coordinate complexes of neodymium with 2,6-di-*tert*-butyl-4-methylphenolate have been synthesized and their single-crystal structures determined. Anhydrous $NdCl_3$ reacted with ArONa in a 1:3 molar ratio in THF at room temperature to yield the neutral four-coordinate complex $[(ArO)_3Nd(THF)]\cdot MePh$ (**1**), which crystallized as a solvate, in which three aryloxide ligands and one THF ligand are coordinated to neodymium to form a slightly distorted tetrahedron. The reaction of $NdCl_3$ with 4 equiv. of ArONa in THF gave $[(ArO)_4Nd][Na(THF)_6]$ (**2**). In this anion–cation pair compound, the neodymium and sodium atoms exhibit distorted tetrahedral and distorted octahedral coordination geometry, respectively. The average Nd–O(Ar) distance is 2.176 and 2.230 Å in complex **1** and **2**, respectively. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Synthesis; Crystal structure; Aryloxide; Neodymium; Anion–cation pair compound

1. Introduction

Lanthanide alkoxides and aryloxides have found a variety of applications as homogeneous catalysts for organic reactions [1] and precursors for organolanthanide syntheses [2] and high purity oxide materials [3]. The syntheses, structures and reactivities of alkoxo- and aryloxolanthanide complexes have recently attracted a great deal of attention [4–6]. Investigation into the chemistry of lanthanide complexes containing aryloxo ligands has produced complexes exhibiting a variety of structural types [7–9]. The coordination number and degree of oligomerization of the products can be controlled by varying the bulky substituents on the arene ring in the reaction of europium ingot with substituted phenols [9,10]. These facts are consistent with general expectations. From metathesis reactions, the neutral monomeric or dimeric complexes can be obtained when the bulky substituted phenoxides are used as ligands [11–13]. However, for a less bulky ligand, such as 2,6-diisopropyl phenoxide, anionic ate complexes containing alkali metals are obtained via bridging interactions to form one-dimensional chains, two-dimensional sheets or extended three-dimensional structures [14,15]. Recently, homoleptic anionic aryloxide–lanthanoid complexes have also been obtained by Clark and Deacon et al. using substituted phenoxides as ligands [16–18].

Previous studies with the 2,6-di-*tert*-butyl-4-methyl phenoxo ligand, O-C_6H_2-2,6-*tert*-Bu-4-Me, (OAr in this paper) have shown that it gives low-coordinate neutral monomeric complexes with scandium and samarium, $Sc(ArO)_3$ and $[Sm(ArO)_3(THF)]\cdot THF$, from the reaction of $LnCl_3$ with NaOAr [11,19]. To understand more about this reaction with the other lanthanide elements, we have studied the reaction of anhydrous $NdCl_3$ with 3 and 4 equiv. of NaOAr in THF, and the desired complex $[Nd(ArO)_3(THF)]\cdot MePh$ (**1**) and unusual compound $[Nd(ArO)_4][Na(THF)_6]$ (**2**) were synthesized. Single-crystal structure studies revealed that the latter is a disconnected anion–cation pair compound with the sodium well separated from the lanthanoid coordination sphere.

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2. Experimental

All manipulations were performed under argon using the Schlenk technique. THF was distilled from sodium benzophenone ketyl before use. Anhydrous $NdCl_3$ was prepared according to the literature method [20]. NaOAr was prepared from the reaction of ArOH and Na in THF. Metal analyses were carried out using complexometric titration [21]. IR spectra were recorded on a Nicolet-550 FTIR spectrometer as KBr pellets. ^1H and ^{13}C NMR spectra were recorded on a Bruker AM-300 spectrometer in C_6D_6.

2.1. Synthesis of *[Nd(ArO)$_3$(THF)]·MePh* (**1**)

A solution of NaOAr (15.7 cm^3, 13.59 mmol) in THF was slowly added to a suspension of $NdCl_3$ (1.135 g, 4.53 mmol) in 20 cm^3 THF at room temperature. After being stirred for 72 h, the NaCl was separated from the reaction mixture by centrifugation. The solvent was removed in vacuum and toluene was added to extract the product. The dissolved portion was removed by centrifugation. The filtrate was concentrated and cooled at −20°C for crystallization. Blue single crystals suitable for X-ray determination were obtained over a few days (yield 3.15 g, 72%). Found: Nd, 14.83. Calc. for $[Nd(ArO)_3(THF)]\cdot(MePh)$: Nd, 14.92%. IR absorptions (cm^{-1}): 2957(s), 2918(w), 2873(w), 1634(s), 1434(s), 1396(m), 1362(m), 1231(m), 1151(s), 1065(m), 1027(w), 864(s), 771(s), 460(m).

Table 1
Crystal data and experimental parameters

Compound	$[Nd(OAr)_3(THF)]\cdot$ MePh (**1**)	$[Nd(OAr)_4]$-$[Na(THF)_6]$ (**2**)
Formula	$C_{56}H_{85}NdO_4$	$C_{84}H_{140}O_{10}NaNd$
Molecular weight	966.48	1477.26
Crystal size (mm)	0.20 × 0.20 × 0.30	0.20 × 0.25 × 0.30
Crystal system	monoclinic	monoclinic
Crystal color and habit	blue, prism	yellow, prism
Space group	*Cc* (No. 9)	$P2_1/c$ (No. 14)
Unit cell dimensions		
a (Å)	25.907(1)	14.414(2)
b (Å)	10.601(1)	15.119(2)
c (Å)	20.160(1)	39.686(4)
β (°)	103.38(1)	97.28(2)
V (Å^3)	5386.5(6)	8579(4)
D_{calc} (g cm^{-3})	1.192	1.144
Z	4	4
μ (mm^{-1})	1.005	0.661
$F(000)$	2052	3180
Temperature (K)	293(2)	293
Scan type	ω	ω
θ_{max} (°)	24.99	22.46
Number of unique reflections	3827	9139
Number of reflections with $I \geq 2.0\sigma(I)$	3819	6039
Number of parameters refined	550	637
R	0.0397	0.0640
R_w	0.1023	0.0840
Goodness-of-fit	0.965	2.93

2.2. Synthesis of *[Nd(ArO)$_4$][Na(THF)$_6$]* (**2**)

A solution of NaOAr (25.0 cm^3, 21.56 mmol) in THF was slowly added to a suspension of $NdCl_3$ (1.352 g, 5.39 mmol) in 25 cm^3 THF at room temperature. After stirring for 72 h, the precipitate was removed from the reaction mixture by centrifugation. The solvent was removed in vacuum and toluene was added. The precipitate was separated and the solution concentrated, and then cooled at −20°C. The yellow crystals were obtained after several weeks (4.05 g, 51%). Found: Nd, 9.62. Calc. for $[Nd(ArO)_4][Na(THF)_6]$: 9.75%. IR absorptions (cm^{-1}): 2962(s), 2925(m), 2876(w), 1637(s), 1437(s), 1225(s), 1159(s), 864(m), 771(m), 507(s). ^1H NMR (C_6D_6): δ 7.16(8H, Ph), 2.35(12H, Me), 1.62 (48H, THF), −1.11(72H, Bu^t). ^{13}C NMR: 123.3(PhO), 88.7(PhO), 84.2(PhO), 74.9 (PhO), −9.3(THF), −19.8(Me), −26.8(THF), −28.9(Me), −29.4(Bu^t), −29.6(Bu^t).

2.3. Crystallography

Single crystals suitable for X-ray measurements were sealed in thin-walled glass capillaries under argon. Intensity data were collected in the variable ω scan mode on a Rigaku AFC 7R four-circle diffractometer using Mo Kα radiation ($\lambda = 0.71073$ Å) at 293 K. Crystallographic data are listed in Table 1.

The crystal structures of complexes **1** and **2** were determined by direct methods which yielded the positions of all non-hydrogen atoms. All the non-hydrogen atoms were refined anisotropically. Hydrogen atoms were all generated geometrically (C–H bond lengths fixed at 0.96 Å), assigned appropriate isotropic thermal parameters and allowed to ride on their parent carbon atoms. All the H atoms were held stationary and were included in the structure factor calculation in the final stage of full-matrix least-squares refinement.

3. Results and discussion

3.1. Syntheses of the complexes

The metathesis reaction between anhydrous $NdCl_3$ and NaOAr in the molar ratio 1:3 in THF at room temperature, followed by crystallization from toluene, yielded the neutral four-coordinate neodymium com-

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

*$[Nd(OAr)_3(THF)]\cdot MePh$ (**1**)*			
Nd(1)–O(1)	2.173(5)	Nd(1)–O(2)	2.190(5)
Nd(1)–O(3)	2.164(5)	Nd(1)–O(4)	2.444(6)
O(1)–C(1)	1.367(8)	O(2)–C(16)	1.356(8)
O(3)–C(31)	1.370(9)	O(4)–C(46)	1.450(12)
O(4)–C(49)	1.452(11)		
O(1)–Nd(1)–O(2)	115.5(2)	O(1)–Nd(1)–O(3)	119.7(2)
O(1)–Nd(1)–O(4)	100.8(2)	O(2)–Nd(1)–O(3)	116.5(2)
O(2)–Nd(1)–O(4)	107.7(2)	O(3)–Nd(1)–O(4)	90.7(2)
Nd(1)–O(1)–C(1)	162.0(5)	Nd(1)–O(2)–C(16)	147.3(4)
Nd(1)–O(3)–C(31)	155.9(5)	Nd(1)–O(4)–C(46)	120.2(5)
Nd(1)–O(4)–C(49)	130.5(5)		
*$[Nd(OAr)_4][Na(THF)_6]$ (**2**)*			
Nd–O(1)	2.202(6)	Nd–O(2)	2.256(7)
Nd–O(3)	2.247(7)	Nd–O(4)	2.217(7)
Na–O(5)	2.33(2)	Na–O(6)	2.49(2)
Na–O(7)	2.36(1)	Na–O(8)	2.32(2)
Na–O(9)	2.33(2)	Na–O(10)	2.36(1)
O(1)–C(1)	1.36(1)	O(2)–C(16)	1.34(1)
O(3)–C(31)	1.35(1)	O(4)–C(46)	1.36(1)
O(1)–Nd–O(2)	108.2(2)	O(1)–Nd–O(3)	112.7(2)
O(1)–Nd–O(4)	104.3(2)	O(2)–Nd–O(3)	110.8(2)
O(2)–Nd–O(4)	111.5(3)	O(3)–Nd–O(4)	109.1(2)
O(5)–Na–O(6)	88.7(7)	O(5)–Na–O(7)	89.8(8)
O(5)–Na–O(8)	173.0(9)	O(5)–Na–O(9)	90.4(8)
O(5)–Na–O(10)	93.7(7)	O(6)–Na–O(7)	86.7(6)
O(6)–Na–O(8)	88.8(7)	O(6)–Na–O(9)	173.6(7)
O(6)–Na–O(10)	97.4(6)	O(7)–Na–O(8)	83.6(7)
O(7)–Na–O(9)	87.0(5)	O(7)–Na–O(10)	174.7(5)
O(8)–Na–O(9)	91.4(9)	O(8)–Na–O(10)	93.1(7)
O(9)–Na–O(10)	89.0(5)		

plex $[Nd(ArO)_3(THF)]\cdot MePh$ in high yield, as shown in Eq. (1).

$$NdCl_3 + 3NaOAr \xrightarrow{THF} (ArO)_3Nd(THF) + 3NaCl \qquad (1)$$

This reaction is similar to the reaction of NaOAr with anhydrous $SmCl_3$ [19], but different from that of 2,6-diisopropylphenoxide which reacted with $NdCl_3$ in THF to produce $K[Nd(OC_6H_3\text{-}Pr^i_2\text{-}2,6)_4]$ instead of $Nd(OC_6H_3\text{–}Pr^i_2\text{-}2,6)_3$ [14]. Subsequent studies have shown that $LnCl_3$ reacted with 4 equiv. of potassium 2,6-diisopropylphenoxide in THF, followed by crystallization from toluene, to form the 'ate' complex $K[Ln(OC_6H_3\text{–}Pr^i_2\text{-}2,6)_4]$ in high yield as shown in Eq. (2) [15].

$$\begin{aligned} &LnCl_3 + 4K\text{–}OC_6H_3\text{–}Pr^i_2\text{-}2,6 \underset{2.\ Toluene}{\overset{1.\ THF}{\rightarrow}} \\ &\quad K[Ln(OC_6H_3\text{–}Pr^i_2\text{-}2,6)_4 + 3KCL \qquad (2) \end{aligned}$$

The reaction of anhydrous $NdCl_3$ with 4 equiv. of NaOAr in THF at room temperature, did not give the 'ate' complex $Na[Nd(OAr)_4]$, but rather produced the unusual compound $[Nd(ArO)_4][Na(THF)_6]$ as illustrated in Eq. (3).

$$NdCl_3 + 4NaOAR \xrightarrow{THF} [(ArO)_4Nd][Na(THF)_6] + 3NaCl \qquad (3)$$

^1H, ^{13}C NMR and IR studies show that there are not only phenolate ligand signals, but also THF signals. A single-crystal study reveals (see below) that compound **2** contains a discrete homoleptic aryloxoneodymium anion with the alkali metal cation well separated from the neodymium coordination sphere. To our knowledge, only three analogous complexes $[Na(diglyme)_2]$-$[Nd(Odpp)_4]$, [Odpp = 2,6-diphenylphenolate, diglyme = bis(2-methoxyethyl) ether], $[Na(diglyme)_2]$-$[Er(Odpp)_4]$ and $[Na(DME)_2][Nd(Odpp)_4]$ (DME = 1,2-dimethoxyethane) have been recently reported in the literature [18]. Generally, unidentate coordinating or non-coordinating solvents were considered disadvantageous for ion pair complex formation. Watkin et al. reported that $Sm(OC_6H_3\text{–}Pr^i_2\text{-}2,6)_3(THF)_2$ reacted with one equiv. of LiC_5Me_5 in THF to form the lithium ate complex, which however reacted with an excess of TMEDA (tetramethylethylenediamine) to form a charge–separated salt [22]. In our case, the disconnected anion–cation pair compound can be crystallized from mixed THF–toluene solvent. This difference may be attributed to the large size of the substituents on the arene ring.

3.2. Structures

The molecular structures of compound **1** and **2** were determined by single-crystal studies. The selected bond lengths and angles are listed in Table 2.

3.2.1. Crystal structure of $[Nd(OAr)_3(THF)]\cdot MePh$ (1)

The molecular structure of $[Nd(OAr)_3(THF)]\cdot MePh$ is shown in Fig. 1. The compound is isostructural with $[Sm(OAr)_3(THF)]\cdot THF$ and the toluene solvent molecule replaces the THF molecule in the molecular packing [19]. The complex is a four-coordinate monomer with three aryloxo oxygen atoms and one THF oxygen atom around the neodymium forming a distorted tetrahedron. The coordination geometry around the metal is different from that in $[Nd(Odpp)_3(THF)]$ which has a distorted trigonal bipyramidal array by forming an intramolecular π-bond [23]. Interestingly, in $[Nd(Odpp)_3(DME)]$ [24], $[Nd(Odpp)_3(THF)_2]$, $[Nd(Odpp)_3(THF)]$ and $Nd(Odpp)_3$ [23], all of these compounds show distorted trigonal bipyramidal stereochemistry and the neodymium atoms are five- or seven-coordinate. Few examples of four-coordinate neodymium complexes are known. Evans and co-workers reported the structure of

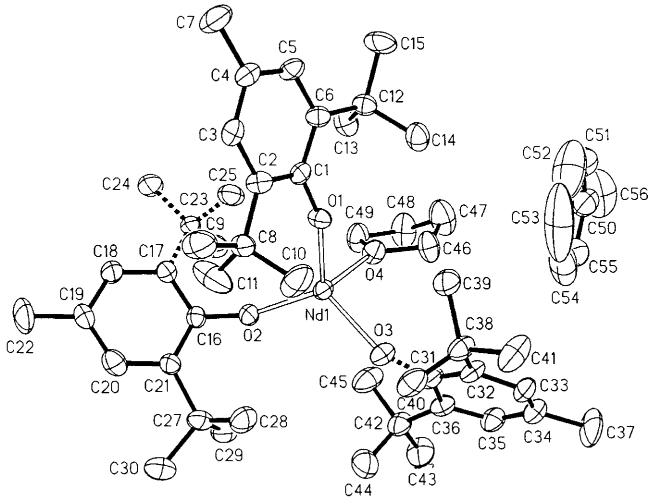

Fig. 1. Molecular structure of [Nd(ArO)$_3$(THF)]·(MePh).

a four-coordinate neodymium amide complex; however, to achieve steric saturation of the metal center, an agostic interaction between the α carbon and neodymium was observed [25]. The current difference in the coordination number can be attributed to the more bulky ArO ligand as compared to the Odpp ligand and amide.

The three aryloxo oxygen atoms around neodymium show a nearly trigonal planar array with Σ O–Nd–O = 351.7°; this value is comparable with those in Nd(Odpp)$_3$(THF) (358.7°) and Nd(Odpp)$_3$ (353.2°) [23]. The Nd–O(Ar) distances range from 2.164(5) to 2.190(5) Å, giving the average Nd–O(Ar) distance 2.176 Å. This value is a little shorter than those in [Nd(Odpp)$_3$(DME)] (2.191 Å) [24], [Nd(Odpp)$_3$(THF)$_2$] (2.190 Å), [Nd(Odpp)$_3$(THF)] (2.193 Å) [23], K[Nd(OC$_6$H$_3$–Pr$^i_2$-2,6) (2.211 Å) [15] and comparable with that in Nd(Odpp)$_3$ (2.169 Å) [23]. Subtraction of the estimated radius (0.86 Å) [26] for four-coordinate Nd^{3+} from Nd–O(Ar) gives 1.31 Å, which is similar to those for [Sm(OAr)$_3$(THF)]·THF (1.31 Å) [19], and Sc(OAr)$_3$ (1.30 Å) [8], but a little longer than those for other aryloxo lanthanide complexes [Yb(Odpp)$_3$-(THF)$_2$] (1.27 Å) [27] [La(Odpp)$_3$(THF)$_2$] (1.28 Å) [28], and Yb(OC$_6$H$_2$Bu$^t_3$-2,4,6)$_3$(THF) (1.27 Å) [29]. The Nd–O(THF) distance is 2.444(6) Å, and a similar subtraction from this value gives 1.58 Å. This is much higher than the usual value 1.34 Å for organolanthanoid complexes [30], but still within the range for the complexes of bulky lanthanoid aryloxides [19,27,29].

3.2.2. Crystal structure of [Nd(OAr)$_4$][Na(THF)$_6$] (2)

The molecular structure of **2** is comprised of a discrete [Na(THF)$_6$]$^+$ cation and a [Nd(ArO)$_4$]$^-$ anion (Fig. 2).

In the anion, the neodymium metal is coordinated to four oxygen atoms from aryloxide ligands in a pseudo-tetrahedral fashion. The stereochemistry around the neodymium atom is similar to that in K[Nd(OC$_6$H$_3$–Pr$^i_2$-2,6)[15], [Na(diglyme)$_2$][Nd(Odpp)$_4$] and [Na(diglyme)$_2$][Er(Odpp)$_4$] [18]. The Nd–O distances range from 2.202(6) to 2.256(7) Å, giving the average distance of 2.230 Å, which is somewhat longer than the average Nd–O(Ar) distance (2.176 Å) observed for complex **1**, although both of them have similar stereochemical arrangement around the central metal. This difference can be attributed to steric repulsion, as an additional ArO ligand replacing the THF ligand increases the steric repulsion among the ligands. It is also longer than that in the analogous aryloxide complex [Na(diglyme)$_2$][Nd(Odpp)$_4$] [18], which is consistent with the fact that ArO is more bulky than Odpp. The Nd–O(Ar) distances in complex **2** are similar to those in K[Nd(OC$_6$H$_3$Pr$^i_2$-2,6)$_4$] (average = 2.21 Å) [14] and Na[Nd(Odpp)$_4$] (2.20 Å) [17,18], but slightly longer than those in [Nd(Odpp)$_3$](THF) (2.193 Å) and Nd(Odpp)$_3$ (2.169 Å) [23]. The Nd–O distance is also longer than the Er–O(Ar) distance in

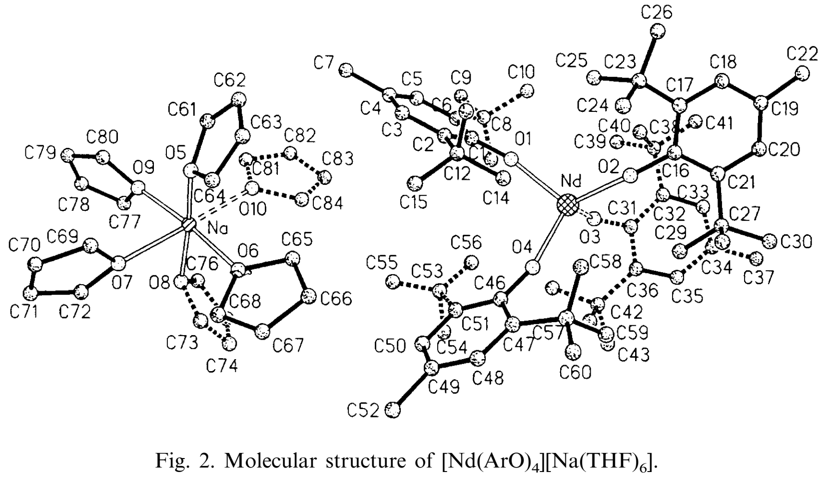

Fig. 2. Molecular structure of [Nd(ArO)$_4$][Na(THF)$_6$].

$[Na(diglyme)_2][Er(Odpp)_4]$ even if the differences in ionic radii are considered [18]. This Nd–O(Ar) distance is very similar to $\langle$Nd–O$_{br}\rangle$ (2.211 Å) and apparently longer than $\langle$Nd–O$_{ter}\rangle$ (2.122 Å) of $Nd_2(O\text{-}2,3\text{-}Pr_2^iC_6H_3)_6$ [7]. The C–O distances of the phenolate ligands range from 1.34(1) to 1.36(1) Å, and are apparently shorter than the single bond length, reflecting substantial delocalization from oxygen into the aromatic rings. The average C–O bond length is 1.35 Å, and is similar to that in complex **1** (1.36 Å), $Yb(OC_6H_2Bu_3^t\text{-}2,4,6)_3(THF)$ (1.38 Å) [29], $[Yb(Odpp)_3(THF)_2]$ (1.33 Å) [27], and $[La(Odpp)_3(THF)_2]$ (1.31 Å) [28].

The six O–Nd–O angles range from 104.3(2) to 112.7(2)°, which is comparable with those angles in $[Na(diglyme)_2][Nd(Odpp)_4]$ [18]. But there are apparently differences from those in $K[(Nd(O\text{-}2,6\text{-}Pr_2^iC_6H_3)_4]$ [14], in which one of the six O–Nd–O angles (95.29°) deviates significantly from that of an ideal tetrahedron because of the interaction between the potassium atom and two aryloxide ligands. The Nd–O–C angles range from 144.6(7) to 167.9(7)°.

In the cation $[Na(THF)_6]^+$, the sodium ion is solvated and octahedrally bonded to the oxygen atoms of six THF molecules. The bond lengths of Na–O range from 2.32(2) to 2.49(2) Å and the angles for the two closest oxygen atoms to the sodium range from 83.6(7) to 97.4(6)°. These are slightly different from the values 2.380(5)–2.434(4), 2.340(10)–2.4410(10) Å and 88.2(2)–92.8(2), 88.8(4)–91.2(4)° in $[Na(THF)_6][(C_9H_7)_3Nd(\mu\text{-}Cl)Nd(C_9H_7)_3]$ [31] and $[Na(THF)_6][Cp_3Lu(\mu\text{-}H)LuCp_3]$ [32], respectively. Actually, in the last two compounds, the cations are centrosymmetrical, but in complex **2**, the O(5)–Na–O(8), O(6)–Na–O(9) and O(7)–Na–O(10) angles are 173.0(9), 173.6(7) and 174.7(5)°, respectively. These values show the cation is a distorted octahedron.

4. Supplementary data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 136299 for compound **1** and CCDC No. 136300 for compound **2**. 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:http://www.ccdc.cam.ac.uk).

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