Synthesis, Characterization, and Crystal Structures of Novel Coordination Polymers of Praseodymium(III) and Neodymium(III) Containing Dialkyl Phosphate

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Received November 13, 2008, Accepted December 15, 2008

Key Words: Neodymium(III), Praseodymium(III), Diethyl phosphate, Dimethyl phosphate, Dimethyl sulfoxide

Metal complexes incorporating various types of phosphate moieties are being examined in order to explore the metalbinding properties of such ligands and to elucidate the importance of these interactions in biological systems. Mononuclear,[1-2] binuclear,[2-16] trinuclear,[17,18] tetranuclear[19-23] and polymeric[24] complexes have been reported in which the phosphate moieties adopt monodentate, chelating, and bridging binding modes. These examples include heterometallic complexes.[25] Moreover, in addition to phosphate and phosphate ester complexes; complexes incorporating nucleic acid fragments, ATP, polyphosphates, and polyphosphate esters have been structurally characterized.[26-29] Recently, we have reported the synthesis and crystal structure of $[La(\mu\text{-DMP})_2(\mu\text{-NO}_3)(DMSO)]_n$,[30] $[UO_2(\mu\text{-DEP})_2(DMSO)]_n$,[31] $[UO_2(\mu\text{-DMP})_2(DMSO)]_n$,[32] $[Ce_2(\mu\text{-DEP})_6(TEP)]_n$,[33] $\{[Mn_2(\mu\text{-DMP})_3(\mu\text{-DMSO})_2(DMSO)(H_2O)]\text{-}NO_3{\cdot}H_2O\}_n$,[34] $[Cu_2(\mu\text{-DMP})_4(\mu\text{-DMSO})]_n$[35] and $[Mn_2(\mu_3\text{-DMP})_2\text{-}(\mu\text{-DMP})_2]_n$,[36] (DMP is dimethyl phosphate, DEP is diethyl phosphate, TEP is triethyl phosphate and DMSO is dimethyl sulfoxide). In these complexes, DMP and DEP act as an O-atom donor ligand, forming coordination polymers in the solid state. Also, we reported the synthesis and crystal structures of $[Ni(H_2O)_6](DMP)_2$ as an ionic complex.[37] In this complex, DMP is not bonded to the metal, and acts as a independent counter-ion. We have also reported the synthesis and crystal structures of $[Fe_{16}(\mu_3\text{-O})_8(\mu_3\text{-OH})_4(\mu\text{-OH})_4(\mu\text{-DMP})_{12}(\mu\text{-OAc})_{12}\text{-}(DMSO)_4]{\cdot}2DMSO{\cdot}1.5H_2O$,[38] within which an macromolecule constellation consisting of sixteen iron ion connected by twelve bridging dimethyl phosphate, twelve bridging acetate, eight μ_3-oxo, four μ_3-OH, four μ-OH and four dimethyl sulfoxide ligands. We now report on the synthesis, characterization and crystal structure of novel polymer of praseodymium(III) and neodymium(III) complexes.

Experimental Section

All chemicals were purchased from Merck and Aldrich. Infrared spectra (4000-250 cm^{-1}) of solid samples were taken as 1% dispersion in KBr pellets using a Shimadzu-470 spectrometer. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were acquired on a Bruker AC-300 MHz spectrometer at ambient temperature in D_2O. Melting points were is uncorrected and were obtained by a Kofler Heizbank Rechart type 7841 melting point apparatus.

Synthesis of $[Nd(\mu\text{-DEP})_3]_n$ (1). Triethyl phosphate (3.31g, 3.1 mL, 18 mmol) was added to a solution of $NdCl_3$ (0.93 g, 3.6 mmol) in ethanol (40 mL) and DMSO (5mL) and the resulting pale violet solution was stirred at 40 °C for 30 minute. Then, it was left to evaporate slowly at room temperature. After two months, colorless prismatic crystals of **1** were isolated (yield 1.62 g, 74.56%, m.p < 300 °C). IR (KBr, cm^{-1}): 2984s, 2940m, 2912m (νCH), 1446w, 1399m, 1372w, 1197s, 1181s, 1168s, 1117s, 1093s, 1066s, 1044s (νC-O-P), 977s, 825s, 778m (νP-O), 576m, 541m, 504m, 409w, 388m, 332w (νNd-O). ^{1}H NMR (D_2O): 1.52 (bs, $\underline{CH_3}\text{-}CH_2$) and 4.71 (bs, $CH_3\text{-}\underline{CH_2}$) ppm. ^{13}C NMR ($D_2O$): 16.0 (d, J_{cp} = 6.3Hz) and 63.3(d, J_{cp} = 4.6Hz) ppm. ^{31}P NMR (D_2O): 40.2 (s) ppm. Elemental analysis (%) calcd. for $C_{12}H_{30}NdO_{12}P_3$ (603.51g/mol): C, 23.86; H, 4.97. Found. C, 23.71; H, 4.89.

Synthesis of $[Pr(\mu\text{-DMP})_2(\mu\text{-NO}_3)(DMSO)]_n$ (2). Trimethyl phosphate (2.55g, 2.1 mL, 18 mmol) was added to a solution of $Pr(NO_3)_3{\cdot}5H_2O$ (1.53 g, 3.6 mmol) in ethanol (40 mL) and DMSO (5 mL) and the resulting pale green solution was stirred at 40 °C for 30 minute. Then, it was left to evaporate slowly at room temperature. After three months, pale green needle crystals of **2** were isolated (yield 1.48 g, 77.40%, m.p < 300 °C). IR (KBr, cm^{-1}): 3013s, 2992s, 2964s, 2933m, 2860s (νCH), 1482s, 1465s, 1443s, 1406s, 1361s, 1340s, 1311s, 1243s, 1160s, 1136s, 1081s, 1020s (νC-O-P), 995s, υ(S=O), 965s, 946s, 849s, 838s (ν P-O), 820s, 785s, 778s, 755s, 730m, 683w, 604m, 576m, 534s, 490s, 412s, 402s, 368m, 323w (νPr-O-P) and (νPr-O-S), 286m (νPr-O-N). ^{1}H NMR (D_2O): 2.88 (s, CH_3) and 5.69 (d, J_{Hp} = 10.5Hz, OMe) ppm. ^{13}C NMR (D_2O): 38.9 (s, CH_3) and 55.7 (s, OCH_3) ppm. ^{31}P NMR (D_2O): 41.0 (s) ppm.

Elemental analysis (%) calcd. for $C_6H_{18}NO_{12}P_2PrS$ (531.12g/mol): C, 13.55; H, 3.39; N, 2.64. Found. C, 13.43; H, 3.31; N, 2.56.

X-ray Structure Analysis. The X-ray diffraction measurements were made on a Bruker APEX II CCD diffractometer for **1** and Bruker SMART 1000 CCD diffractometer for **2** (Mo-Kα radiation, graphite monochromator, λ = 0.71073 Å). The structures of **1** and **2** were solved by SHELXTL ver. 5.1.[39] The non-hydrogen atoms were refined anisotropically by a full-matrix least-squares method based on F^2 using SHELXTL ver. 5.1 for **1** and **2**.[39]

Results and Discussion

1 can be obtained by the reaction of $NdCl_3$ with TEP in EtOH and DMSO as colorless prismatic crystals according to Equation (1).

$$\mathrm{NdCl_3} + 5n\ \mathrm{OP(OC_2H_5)_3} \xrightarrow[\text{-3n } \mathrm{C_2H_5Cl}]{\text{EtOH, DMSO}} \{\mathrm{Nd}[\mu\text{-}\mathrm{O_2P(OC_2H_5)_2}]_3\}_n \qquad (1)$$

2 can be obtained by the reaction of $Pr(NO_3)_3 \cdot 5H_2O$ with TEP in EtOH and DMSO as pale green needle crystals according to Equation (2).

$$\mathrm{Pr(NO_3)_3 \cdot 5H_2O} + 5n\ \mathrm{OP(OCH_3)_3} \xrightarrow[\text{-2n } \mathrm{CH_3NO_3}]{\text{EtOH, DMSO}} \{\mathrm{Pr}[\mu\text{-}\mathrm{O_2P(OCH_3)_2}]_2(\mu\text{-}\mathrm{NO_3})(\mathrm{DMSO})\}_n \qquad (2)$$

Both complexes are air-stable. IR spectra of **1** and **2** show a very sharp absorption peak for the CH stretching vibrations in the range of ca 3013 to 2860 cm^{-1}. Strong phosphate stretches at 1197 and 1168 cm^{-1} for **1** and 1243 and 1136 cm^{-1} for **2**. Five strong sharp bands at 1181, 1093, 1066, 1054, and 1044 cm^{-1} for **1** and 1189, 1160, 1036, 1081 and 1020 cm^{-1} for **2** are due to C-O of P-O-C species. Bands at 825 and 778 cm^{-1} for **1** and 849 and 838 cm^{-1} for **2** corresponds to ν(P-O) of P-O-C phosphate ester fragment. S=O stretching vibration for complex **2** is seen at 995 cm^{-1}. Other Pr(III) DMSO complexes reported in the literature have shown υ(S=O) in the 992-998cm^{-1} region.[40] Several bands in the 576-323 cm^{-1} spectral region are assigned to the ν(P-O-Nd), ν(P-O-Pr) and ν(S=O-Pr) stretching vibrations for complex **1** and **2**.[40,41] The Pr-ONO$_2$ stretching vibration for complex **2** is seen at 286 cm^{-1}.[40] Our experimental interpretations of the individual vibrational bands confirm the results of the previous findings on the infrared spectrum of solid NaDMP[42] and other ab initio computational calculations such as normal coordinate analysis of DMP.[43]

Table 1. Crystallographic and structure refinements data for complexes **1** and **2**

	Complex **1**	Complex **2**
Formula	$C_{12}H_{30}NdO_{12}P_3$	$C_6H_{18}NO_{12}P_2PrS$
Formula weight	603.51	531.12
Temperature /K	100(2)	120(2)
Crystal system, Space group	Triclinic, $P\bar{1}$	Monoclinic, $P2_1/c$
Crystal size /mm^3	0.22×0.12×0.09	0.55×0.08×0.06
a / Å	10.2503(9)	17.0016(11)
b / Å	11.2599(9)	9.9709(7)
c / Å	20.6739(17)	10.1173(7)
α / °	100.677(2)	90
β / °	90.619(2)	97.0510(10)
γ / °	92.061(2)	90
Volume / Å^3	2342.9(3)	1702.1(2)
Z, Density (calc.) / g cm^{-1}	2, 1.711	4, 2.073
θ ranges for data collection	1.84 - 27.88	2.37 - 29.00
F(000)	1212	1048
Absorption coefficient mm^{-1}	2.472	3.229
Index ranges	$-13 \le h \le 13$	$-23 \le h \le 22$
	$-14 \le k \le 14$	$-13 \le k \le 13$
	$-27 \le l \le 27$	$-13 \le l \le 13$
Data collected	26425	18172
Unique data (R_{int})	11174, (0.0250)	4500, (0.0380)
Final R_1, wR_2	0.0254, 0.0620	0.0390, 0.0831
Goodness of fit on F^2 (S)	1.027	1.040
Largest diff peak and hole /e Å^{-3}	1.784, -1.163	3.117, -1.065

The ^1H NMR spectrum of **1** exhibited two broad singlets for CH_3 and CH_2 (1.51 and 4.69) protons. The ^{13}C NMR spectrum of **1** showed two doublets for CH_3 and CH_2 (16.2 and 63.3) in agreement with the suggested structure. The phosphorous groups are homotopic and show a singlet (40.2) in the ^{31}P NMR spectrum. The ^1H NMR spectrum of **2** exhibited one doublet for CH_3O (5.69) and one singlet for CH_3 (2.88) protons. The ^{13}C NMR spectrum of **2** showed two singlets for CH_3 and CH_3O (38.9 and 55.7) in agreement with the suggested structure. The phosphorous groups are homotopic and show a singlet (41.0) in the ^{31}P NMR spectrum.

Crystallographic data for **1** and **2** are given in Table 1 and selected bond lengths and angles are presented in Table 2 and 3 for **1** and **2** respectively. The structure of **1** consists of a [Nd-(μ-DEP)$_3$] fragment and a repetition unit in this one-dimen-

Table 2. Selected bond lengths/Å and angle/° of **1**

Nd(1)-O(24)#1	2.3118(18)	O(13)-Nd(1)-O(5)	91.94(7)
Nd(1)-O(9)	2.3157(17)	O(24)#1-Nd(1)-O(1)	90.25(7)
Nd(1)-O(20)#1	2.3342(18)	O(24)#1-Nd(1)-O(5)	91.11(7)
Nd(1)-O(13)	2.3424(18)	O(9)-Nd(1)-O(5)	89.91(6)
Nd(1)-O(5)	2.3486(18)	O(20)#1-Nd(1)-O(5)	178.58(6)
Nd(1)-O(1)	2.3564(18)	O(9)-Nd(1)-O(1)	88.70(6)
P(1)-O(1)	1.494(3)	O(20)#1-Nd(1)-O(1)	93.21(7)
P(1)-O(2)	1.496(3)	O(13)-Nd(1)-O(1)	178.60(6)
P(1)-O(4)	1.585(4)	O(5)-Nd(1)-O(1)	86.67(7)
P(1)-O(3)	1.586(3)	O(1)-P(1)-O(2)	115.28(11)
O(24)#1-Nd(1)-O(9)	178.50(7)	O(1)-P(1)-O(4)	107.15(11)
O(24)#1-Nd(1)-O(20)#1	87.47(7)	O(2)-P(1)-O(4)	111.27(11)
O(24)#1-Nd(1)-O(13)	89.57(7)	O(1)-P(1)-O(3)	110.45(11)
O(9)-Nd(1)-O(13)	91.50(6)	O(2)-P(1)-O(3)	109.71(11)
O(20)#1-Nd(1)-O(13)	88.17(6)	O(4)-P(1)-O(3)	102.16(11)

Symmetry codes: #1 = x+1,y,z

Table 3. Selected bond lengths/Å and angle/° of **2**

Pr(1)-O(1)	2.362(3)	O(5)-Pr(1)-O(9)	91.75(12)
Pr(1)-O(5)	2.376(3)	O(2)#1-Pr(1)-O(9)	139.63(11)
Pr(1)-O(2)#1	2.378(3)	O(6)#2-Pr(1)-O(9)	98.12(12)
Pr(1)-O(6)#2	2.400(3)	O(1)-Pr(1)-O(10)	73.70(11)
Pr(1)-O(9)	2.458(3)	O(5)-Pr(1)-O(10)	114.72(11)
Pr(1)-O(10)	2.645(3)	O(2)#1-Pr(1)-O(10)	142.39(10)
Pr(1)-O(10)#1	2.692(3)	O(9)-Pr(1)-O(10)	73.30(10)
Pr(1)-O(12)#1	2.757(3)	O(1)-Pr(1)-O(10)#1	139.51(11)
Pr(1)-O(11)	2.789(4)	O(5)-Pr(1)-O(10)#1	68.85(10)
P(1)-O(1)	1.494(3)	O(1)-Pr(1)-O(11)	74.30(11)
P(1)-O(2)	1.496(3)	O(5)-Pr(1)-O(11)	67.79(11)
P(1)-O(4)	1.585(4)	O(9)-Pr(1)-O(11)	71.41(11)
P(1)-O(3)	1.586(3)	O(10)-Pr(1)-O(11)	47.04(10)
O(1)-Pr(1)-O(5)	87.47(12)	O(1)-P(1)-O(2)	118.31(19)
O(1)-Pr(1)-O(2)#1	77.13(11)	O(1)-P(1)-O(4)	110.3(2)
O(5)-Pr(1)-O(2)#1	86.83(12)	O(2)-P(1)-O(4)	105.5(2)
O(1)-Pr(1)-O(6)#2	83.97(12)	O(1)-P(1)-O(3)	105.86(19)
O(5)-Pr(1)-O(6)#2	170.06(12)	O(2)-P(1)-O(3)	110.60(19)
O(1)-Pr(1)-O(9)	143.16(12)	O(4)-P(1)-O(3)	105.7(2)

Symmetry codes: #1 = x,-y+3/2, z-1/2 #2= x,-y+3/2,z+1/2 sional polymer. As is clear from an ORTEP view of the compound, Fig. 1(a), the repeation unit of this linear polymer consists of 6-coordinated neodymium atoms, which are linked together *via* six O-P-O bridges from phosphate esters.

The phosphate groups in $[Nd(\mu\text{-DEP})_3]_n$ are arranged as an abnormal distorted tetrahedron. The angles of O-P-O and EtO-P-OEt are unequal. The angle of the EtO(3)-P(1)-O(4)Et moiety is 102.16(11)°, whereas that of O(1)-P(1)-O(2) is 115.28(11)°. According to a survey on similar polymeric $[Ce_2(\mu\text{-DEP})_6(TEP)]_n$ complexe, an acceptable reason is simultaneous z-in and z-out distortions for the DEP. In addition, the conformation of the DEP anion depends on the C-O-P-O angles. In this complex,[33] we observed the gt (gauche, trans) conformation for the DEP base on the dihedral angles. At this complex, for example, the dihedral angles of one of the DEP ligands are: C(1)-O(3)-P(1)-O(4) = -177.4(2)° and C(3)-O(4)-P(1)-O(3) = -75.5(2)°. The unit cell packing diagram of the complex **1** is shown in Figure 1(b).

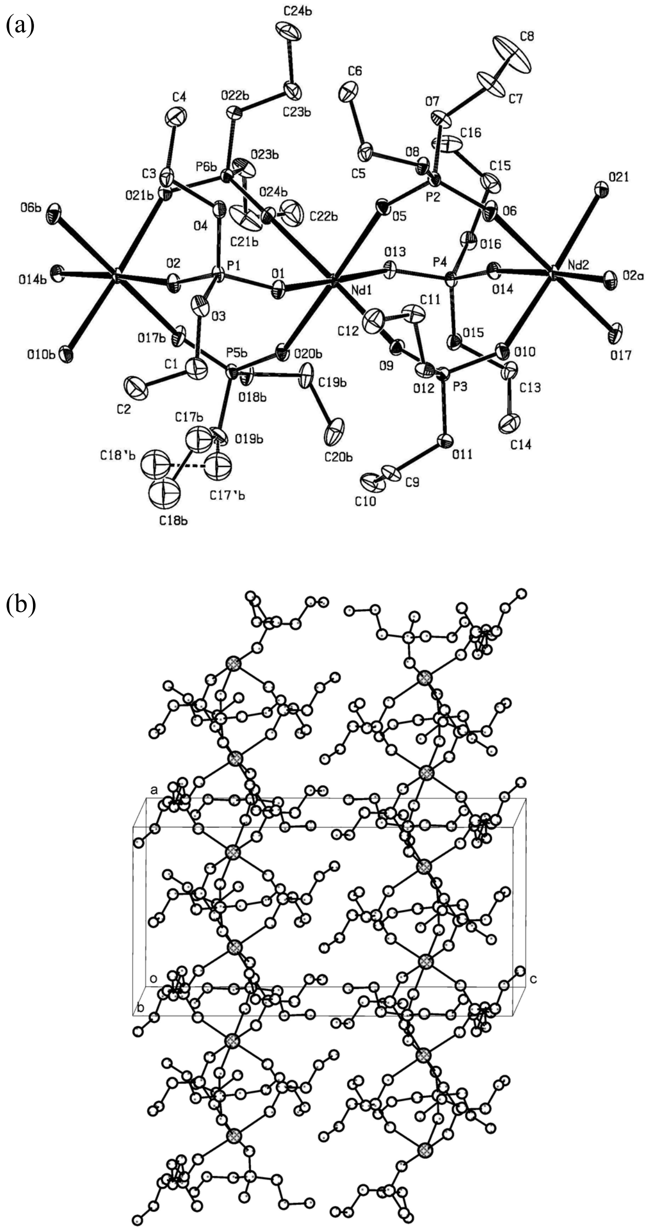

Figure 1. Graphical representation of **1** (40 % probability for the thermal ellipsoids) (a) and the unit cell-packing diagram of **1** (b). Hydrogen atoms are omitted for clarity.

The structure of **2** consists of a $[Pr(\mu\text{-DMP})_2(\mu\text{-NO}_3)(DMSO)]$ fragment and a repetition unit in this one-dimensional polymer. An ORTEP plot (Fig. 2(a)) shows that the complex consists of one praseodymium, which is linked to other fragments *via* four O-P-O bridges of four phosphate esters; also, one DMSO and two nitrate are linked to each praseodymium *via* its oxygen atom during chelating and bridging. There is a 4-membered circle created by the cooperation of two oxygen atoms of nitrate ion (O11, O12) and a central Pr atom. In addition, a third oxygen atom (O10) of nitrate also forms a bridge between two neighbor Pr atoms. Figure 2(a) shows the polymeric structure of the title complex, which the (O-P-O) moieties of the phosphate form a double bridge between two praseodymium. The DMSO molecule appears as a monodentate ligand on every single Pr atom.

The phosphate groups, $[Pr(\mu\text{-DMP})_2(\mu\text{-NO}_3)(DMSO)]_n$, are arranged as an abnormal distorted tetrahedron. The angles of O(1)-P(1)-O(2) and MeO(3)-P(1)-O(4)Me are unequal. The angle of the MeO(3)-P(1)-O(4)Me moiety is 105.7(2)°, whereas that of O(1)-P(1)-O(2) is 118.31(19)°. According to a survey on similar polymeric La complex[30] an acceptable reason is simultaneous z-in and z-out distortions for the DMP. In addition, the conformation of the DMP anion depends on the C-O-P-O torsion angles. In this complex, we expectedly observed the gg (gauche, gauche) conformation for the DMP base on the dihedral angles. At this complex, for example, the

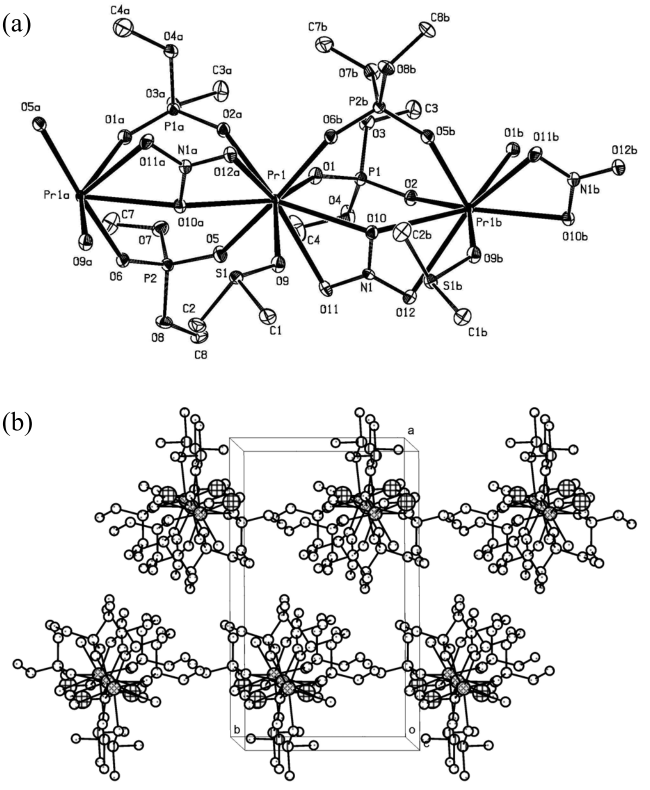

Figure 2. Graphical representation of **2** (40 % probability for the thermal ellipsoids) (a) and the unit cell-packing diagram of **2** (b). Hydrogen atoms are omitted for clarity.

torsion angles of one of the DMP ligands are: C(3)-O(3)-P(1)-O(4) = -76.0(4)° and C(4)-O(4)-P(1)-O(3) = -95.8(5)°. Each Pr center occupies an irregular distorted tricapped trigonal prismatic geometry with the DMSO ligand in the vertical position. The angle of S(1)-O(9)-Pr(1) is 128.70(19)°. The large mentioned angle may belong to a steric hindrances induced by the methyl groups of DMSO, and lone pairs on the oxygen of DMSO with the Pr atom environment. According to experimental geometry data for DMSO, the bond angles are CSC = 96.6° and CSO = 106.5°. At this complex the bond angles of the DMSO ligand are: C(1)-S(1)-C(2): 99.5(2)°, O(9)-S(1)-C(1): 105.1(2)° and O(9)-S(1)-C(2): 105.3(2)°. Therefore, we observed that they have almost not changed. It is clear from Figure 2(a) that, a Pr ion is connected to adjacent Pr by bridging of four DMP and two nitrate ions. These infinite chains are packed along the *b*-axis to create the channels to which O-Pr bond of DMSO molecules are perpendicular. The unit cell packing diagram of the complex **2** is shown in Figure 2(b).

Supplementary Materials. CCDC 693501 and 693502 contain the supplementary crystallographic data for **1** and **2** respectively. These data can be obtained free of charge *via* *http://www.ccdc.cam.ac.uk/conts/retrieving.html*, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223 336 033; or e-mail: *deposit@ccdc.cam.ac.uk*.

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