Absorption correction: empirical ψ scans (*XSCANS*; Siemens, 1994)
$T_{min} = 0.378$, $T_{max} = 0.525$
4743 measured reflections
3843 independent reflections
3514 reflections with $I > 2\sigma(I)$
$h = -1 \rightarrow 9$
$k = -13 \rightarrow 14$
$l = -14 \rightarrow 14$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.085$
$S = 1.071$
3843 reflections
293 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.1566P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.280$ e Å^{-3}
$\Delta\rho_{min} = -0.471$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Cu—O1	1.9654 (14)	Cu—O4	2.690 (2)
Cu—O3	1.9669 (14)	O1—C11	1.267 (2)
Cu—N1	1.9742 (15)	O2—C11	1.239 (3)
Cu—N3	1.995 (2)	O3—C13	1.279 (2)
Cu—O2	2.4824 (15)	O4—C13	1.233 (2)
O1—Cu—O3	89.15 (6)	N1—Cu—O2	96.90 (6)
O1—Cu—N1	154.42 (6)	N3—Cu—O2	106.41 (6)
O3—Cu—N1	93.99 (6)	O1—Cu—O4	114.89 (6)
O1—Cu—N3	94.46 (6)	O3—Cu—O4	53.50 (5)
O3—Cu—N3	156.92 (6)	N1—Cu—O4	86.84 (6)
N1—Cu—N3	92.50 (6)	N3—Cu—O4	104.90 (5)
O1—Cu—O2	57.53 (5)	O2—Cu—O4	148.25 (6)
O3—Cu—O2	94.76 (6)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O5—H1O5···O2	0.78 (3)	2.01 (3)	2.778 (3)	169 (3)
O5—H2O5···O3^{i}	0.76 (3)	2.11 (3)	2.845 (3)	164 (4)
O6—H1O6···O5^{ii}	0.76 (4)	2.04 (4)	2.798 (4)	174 (3)
O6—H2O6···O4	0.80 (3)	2.02 (3)	2.801 (3)	167 (3)
N2—H1N2···O6^{iii}	0.77 (2)	2.02 (2)	2.784 (3)	174 (2)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $x, y, z-1$; (iii) $-x, -y, -z$.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93* and *PARST* (Nardelli, 1995).

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Metal Alkoxycarboxylate Complexes. I. Poly[aquabis(methoxyacetato)(nitrato)-neodymium(III)]

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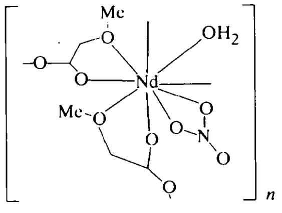
Abstract

The title compound, $[Nd(O_2CCH_2OCH_3)_2(NO_3)(H_2O)]_n$, contains nine-coordinate Nd atoms in which the carboxylate ligands chelate to one metal atom and each bridges to a second metal atom, resulting in the formation of pleated sheets. The Nd—O distances range from 2.375 (4) to 2.629 (4) Å.

Comment

The discovery that lanthanide metal salts of 2-[2-(2-methoxyethoxy)ethoxy]acetate (MEEA) are room-temperature liquids (Apblett, Long *et al.*, 1994; Apblett *et al.*, 1995) has prompted an investigation of the structural chemistry of simpler alkoxycarboxylates so that the resulting structural and spectroscopic characterizations of these solid complexes can be used towards a greater understanding of the behavior of the MEEA salts. In addition, methoxyacetate complexes have been shown to be useful ceramic precursors for materials such as barium titanate (Apblett, Georgieva & Raygoza-Maceda, 1994). In our initial attempts to prepare [$Nd(O_2CCH_2OCH_3)_3$] from sodium methoxyacetate and neodymium(III) nitrate, the mixed nitrate–carboxylate complex of empirical formula [$Nd(O_2CH_2OCH_3)_2(NO_3)(H_2O)$], (1), was the crystalline product isolated.



(1)

As shown in Fig. 1, the Nd atom is nine-coordinate, with the coordination sphere consisting of two methoxyacetate ligands chelating *via* one carboxylate oxygen and the ether oxygen, an asymmetrically chelating nitrate ion, a water molecule and the second carboxylate oxygen (O4 and O6″) of the methoxyacetate ligands on two adjacent metals. This leads to the formation of pleated sheets running approximately parallel to **b**. Previous examples of methoxyacetate complexes whose structures are known involve both chelation as observed here and monodentate coordination *via* one carboxyl oxygen (Prout *et al.*, 1968; Prout, Allison & Rossotti, 1971; Prout, Barrow & Rossotti, 1971; Prout, Walker & Rossotti, 1971). The greater complexity of the present structure may, in part, be the result of the larger coordination number available to neodymium but is not limited to this particular example (Mague *et al.*, 1998). The various Nd—O distances compare favorably with those for their respective ligands obtained from the Cambridge Structural Database (Allen & Kennard, 1993), with the exception of those involving the ether O atoms (O2 and O3), which are significantly shorter than the average found (*ca* 2.68 Å). Hydrogen bonding occurs between the water molecule and a carboxylate oxygen on each of the two methoxyacetate ligands chelated to adjacent metals.

Experimental

The title compound was prepared by treating methoxyacetic acid (2.72 g, 30.2 mmol) with sodium carbonate (1.59 g, 15.0 mmol) in water (20 ml). Following cessation of carbon dioxide evolution, [$Nd(NO_3)_3(H_2O)_6$] (4.38 g, 10.0 mmol) was added and the solution stirred until all solids had dissolved. Addition of absolute ethanol to the cloud point and cooling

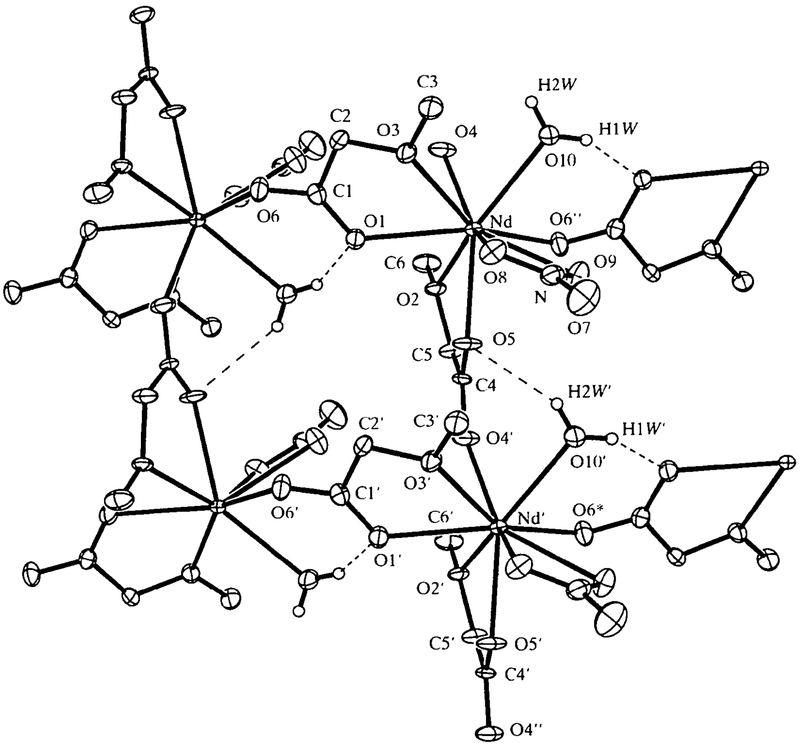


Fig. 1. View of a portion of the pleated-sheet structure adopted by (1). H atoms bonded to carbon have been omitted for clarity. The atoms at the extreme right of the drawing are additional Nd atoms with only part of their coordination spheres shown.

at 253 K for several months afforded a mass of purple cube-shaped crystals.

Crystal data

$[Nd(C_3H_5O_3)_2(NO_3)(H_2O)]$
$M_r = 402.41$
Monoclinic
$P2_1/c$
$a = 6.2457\,(11)$ Å
$b = 11.9757\,(9)$ Å
$c = 17.1228\,(14)$ Å
$\beta = 90.081\,(2)°$
$V = 1280.7\,(3)$ Å^3
$Z = 4$
$D_x = 2.087$ Mg m^{-3}
$D_m = 2.1$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 23 reflections
$\theta = 12.23–17.76°$
$\mu = 4.098$ mm^{-1}
$T = 293\,(2)$ K
Block
$0.33 \times 0.33 \times 0.23$ mm
Light purple

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: by integration
$T_{\min} = 0.412$, $T_{\max} = 0.483$
2465 measured reflections
2245 independent reflections
1823 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.021$
$\theta_{\max} = 25°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 14$
$l = -20 \rightarrow 20$
2 standard reflections
frequency: 120 min
intensity decay: −5.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.088$
$S = 1.222$
2245 reflections
165 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0540P)^2 + 1.1239P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 1.781$ e Å^{-3}
$\Delta\rho_{\min} = -0.738$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O10—H1W···O1$^{\rm i}$	0.87	2.00	2.816 (7)	154
O10—H2W···O5$^{\rm ii}$	0.77	2.49	3.069 (7)	134

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $x - 1, y, z$.

The water H atoms were placed in the locations obtained from the difference map, while those attached to carbon were placed in calculated positions. All were included as riding contributions with fixed isotropic displacement parameters 20% larger than those of the attached atoms. The largest features in the final difference map are 0.78–0.97 Å from the Nd atom, but there is no obvious source.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *XCAD*4 (Harms & Wocadlo, 1987). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Siemens, 1994). Software used to prepare material for publication: *SHELXTL-Plus*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1460). Services for accessing these data are described at the back of the journal.

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Dichloro(tetrahydrofuran-*O*)[tris(1-pyrazolyl-N^2)borato]vanadium(III)

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Abstract

The title compound, $[HB(pz)_3]VCl_2(thf)$ or $[VCl_2(C_9H_{10}BN_6)(C_4H_8O)]$ (pz = pyrazolyl, thf = tetrahydrofuran), is an analogue of the unknown η^5-$CpVCl_2$ (Cp =

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