

are observed (Hathaway, 1987a). In general, a trigonally distorted  $\text{CuCl}_2\text{L}_2\text{Cl}'$  chromophore is involved, with a long Cu—Cl bridging distance ( $\approx 2.75 \text{ \AA}$ ) and a non-linear Cu—Cl—Cu angle of  $114\text{--}145^\circ$ . Table 3 gives some crystallographic data on copper complexes involving single bridge ( $\text{Cl}'$ ) chains. It may be noted that the present complex contains a trigonally distorted  $\text{CuN}_2\text{OCICl}'$  chromophore. A perusal of Table 3 shows that the axial Cu—Cl distance is shortest in the present complex. It has been reported (Willett & Geiser, 1984) that for  $\text{Cu}^{\text{II}}$  halides the bonding nature of the bridging halide atoms affects the  $\text{Cu}^{\text{II}}$  geometry. Both  $\text{Cu}^{\text{II}}$  coordination geometry and the parameters involving the bridging atom are important in the formation of Cu—X—Cu linear chains. It is interesting to note (Table 3) that even though the Cu—Cl—Cu angle in the present complex is equal to that found in complex (3), the former is more linear as is evident from the Cu—Cu—Cu angle. The Cl—Cu—Cl angles for these two complexes are  $99.55(3)$  and  $89.5(1)^\circ$ , respectively. This shows that the angle made by Cl atoms at Cu is one of the factors responsible for linearity in the present system. The Cu—Cu—Cu angle in the present complex is very close to that in complex (1), the best linear chain observed so far among these complexes.

The chain structure is further stabilized by hydrogen bonds, van der Waals and stacking interactions. A pyridine ring of dipyam is involved in stacking interactions with the pyridine ring of the glide-related dipyam moiety with a separation of  $3.50 \text{ \AA}$  (Fig. 2). The possible hydrogen bonds and van der Waals contact distances ( $< 3.5 \text{ \AA}$ ) have been deposited.\*

\* See deposition footnote.

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## Structure of the Dehydrated Octaacetate of Dineodymium(III) and Cobalt(II)

BY AMPARO GONZALEZ AND AURELIO BELTRAN

*UIBCM, Department de Quimica Inorgànica, Universitat de Valencia, Dr. Moliner 50, 46100 Burjassot, Valencia, Spain*

AND ARMEL LE BAIL

*Laboratoire des fluorures, URA CNRS 449, Université du Maine, 72017 Le Mans CEDEX, France*

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**Abstract.** Octakis( $\mu$ -acetato- $O:O'$ )-decaaquacobalt-(II)dineodymium(III),  $[\text{CoNd}_2(\mu\text{-C}_2\text{H}_3\text{O}_2)_8(\text{H}_2\text{O})_{10}]$ ,  $M_r = 999.92$ , triclinic,  $P\bar{1}$ ,  $a = 8.243(2)$ ,  $b =$

$10.368(3)$ ,  $c = 11.497(3) \text{ \AA}$ ,  $\alpha = 111.64(1)$ ,  $\beta = 107.94(1)$ ,  $\gamma = 93.45(1)^\circ$ ,  $V = 851(1) \text{ \AA}^3$ ,  $Z = 1$ ,  $D_x = 1.95 \text{ Mg m}^{-3}$ ,  $\text{Mo } K\alpha$ ,  $\lambda = 0.71069 \text{ \AA}$ ,  $\mu =$

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3.58 mm<sup>-1</sup>,  $F(000) = 495$ , room temperature,  $R = 0.024$  for 6789 unique reflections. Crystal growth was by slow evaporation from an aqueous solution of the simple acetates. Chains are built up from distorted [NdO<sub>7</sub>(OH<sub>2</sub>)<sub>2</sub>] tricapped trigonal prisms and almost regular (CoO<sub>6</sub>) octahedra (O atoms from acetate bidentate bridging groups). The [NdO<sub>7</sub>(OH<sub>2</sub>)<sub>2</sub>] units share an edge (two O atoms from two acetate groups). The resulting dimeric entities are linked one to another through one (CoO<sub>6</sub>) octahedron, which shares one corner with each prism. This gives an (NdNdCo) sequence in the chains, which are interconnected by rather weak hydrogen bonds involving the six lattice water molecules.

**Introduction.** The catalytic properties of perovskites and related mixed oxides are highly dependent on the synthetic procedure. The preparation of mixed oxides at low temperatures could be advantageously carried out by using as precursor a single phase in which the reactants are present in the required stoichiometry (Rao & Gopalakrishnan, 1986). Although it has not been discussed in detail, some lanthanide ions form anionic carboxylate complexes (Mehrotra & Bohra, 1983) which, after the incorporation of a suitable counterion, might be true solid precursors of the respective mixed oxides. This is the case for the acetato complex of formula Nd<sub>2</sub>Co(CH<sub>3</sub>COO)<sub>8</sub>·10H<sub>2</sub>O, whose crystal structure we report here.

**Experimental.** Well formed crystals of Nd<sub>2</sub>Co(CH<sub>3</sub>COO)<sub>8</sub>·10H<sub>2</sub>O were separated by filtration (after slow evaporation in air) of the pink mother liquor from a slightly acidic aqueous solution containing equimolar amounts of the single acetates. Prismatic purple crystal 0.19 × 0.08 × 0.25 mm. Data collected on a Siemens AED2 four-circle diffractometer,  $\omega$ -2 $\theta$  step-scan mode in 36 steps of  $\Delta\omega = 0.035^\circ$ . Time per step  $T_{\min} = 1$  s,  $T_{\max} = 4$  s. Profile-fitting data analysis (Clegg, 1981); isotropic linewidth  $w = (0.86 + 0.02\tan\theta)^\circ$ . Aperture  $D = 4.0$  mm. Lattice constants based on 40 reflections at  $2\theta \approx 30^\circ$ . Absorption correction by the Gauss method  $A_{\max} = 0.77$ ,  $A_{\min} = 0.53$ . Intensity measurement of one independent set to  $2\theta_{\max} = 70^\circ$  within the range  $-13 \leq h \leq 13$ ,  $-16 \leq k \leq 16$ ,  $0 \leq l \leq 18$ . Standard reflections  $\bar{3}43$ ,  $0\bar{5}5$ ,  $\bar{3}24$ , intensity variation 1.0%. 7505 reflections measured, 6789 independent reflections used for the refinements [ $|F| > 6\sigma(|F|)$ ]. Structure solved from Patterson interpretation with *SHELX76* program (Sheldrick, 1976).  $F$  magnitudes used in least-squares refinements: 274 parameters refined; mean  $\Delta/\sigma = 0.03$ , maximum  $\Delta/\sigma = 0.14$ ; secondary-extinction factor  $x = 4.0(2) \times 10^{-7}$ ; atomic scattering factors for H, C, O, Co and Nd atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV); calculations with *SHELX76* (Sheldrick,

Table 1. Atomic coordinates and equivalent isotropic temperature factors ( $\text{\AA}^2$ ) with e.s.d.'s in parentheses

$$B_{\text{eq}} = \frac{1}{3} \sum_i \sum_j b_{ij}(\mathbf{a}, \mathbf{a}) \text{ (Hamilton, 1959).}$$

	x	y	z	$B_{\text{eq}}$
Nd	0.08652 (1)	0.91889 (1)	0.33204 (1)	1.65 (1)
Co	0.0	0.0	0.0	1.62 (2)
O(1)	0.1104 (2)	0.1320 (2)	0.5666 (2)	2.6 (1)
O(2)	0.2765 (3)	0.1607 (2)	0.4591 (2)	3.1 (1)
O(3)	0.1692 (2)	0.9534 (2)	0.1531 (1)	2.1 (1)
O(4)	0.3817 (2)	0.9169 (2)	0.2987 (2)	3.0 (1)
O(5)	0.9338 (2)	0.1611 (2)	0.1408 (2)	2.5 (1)
O(6)	0.8954 (2)	0.0779 (2)	0.2862 (2)	2.6 (1)
O(7)	0.7959 (2)	0.8361 (2)	0.9563 (2)	2.4 (1)
O(8)	0.8594 (3)	0.7728 (2)	0.1286 (2)	3.3 (1)
Ow(1)	0.2875 (2)	0.8905 (2)	0.5313 (2)	2.8 (1)
Ow(2)	0.3925 (3)	0.2934 (2)	0.3264 (2)	3.4 (1)
Ow(3)	0.1131 (3)	0.6666 (2)	0.2612 (3)	4.6 (2)
Ow(4)	0.0941 (4)	0.6002 (3)	0.9368 (3)	4.9 (2)
Ow(5)	0.3018 (4)	0.5562 (3)	0.4230 (3)	5.4 (2)
C(1)	0.3266 (2)	0.9366 (2)	0.1939 (2)	2.1 (1)
C(2)	0.7705 (2)	0.7568 (2)	0.0125 (2)	2.1 (1)
C(3)	0.2321 (3)	0.2062 (2)	0.5599 (2)	2.4 (1)
C(4)	0.1002 (3)	0.8257 (2)	0.7565 (2)	2.2 (1)
C(5)	0.4411 (3)	0.9424 (4)	0.1169 (3)	3.7 (2)
C(6)	0.6243 (4)	0.6297 (3)	0.9338 (3)	3.8 (2)
C(7)	0.3274 (5)	0.3442 (3)	0.6732 (3)	4.6 (2)
C(8)	0.8637 (7)	0.3150 (3)	0.3215 (4)	5.1 (3)

1976) on a MicroVAX II computer. A Patterson interpretation gave a solution with two metallic atoms. Then refinements of atomic positions and isotropic thermal motion, followed by difference Fourier maps, led to the localization of all the non-H atoms ( $R = 0.086$ ). The residual decreased to  $R = 0.026$  when the anisotropic thermal parameters were refined. Positions of all H atoms were then found from a difference Fourier map calculated from data limited to  $\sin\theta = 0.4$ . Geometrical constraints were applied to CH<sub>3</sub> and H<sub>2</sub>O groups ( $d_{\text{C-H}} = 1.08$ ,  $d_{\text{O-H}} = 1.00$  \AA) and two thermal parameters were refined for the C-bonded and the O-bonded H atoms respectively ( $R = 0.024$ ,  $wR = 0.029$ ). Maximum and minimum heights in the final difference  $F$  map were 0.88 and  $-0.53 e \text{\AA}^{-3}$ ;  $w = 1.00/[\sigma^2(|F_o|) + 0.00082F_o^2]$ .

**Discussion.** The final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1,\* and selected bond distances and angles are in Table 2. The molecular geometry and atom-labelling scheme are shown in Fig. 1. The coordination polyhedron around Nd atoms is similar to those found in simple lanthanide acetates (Sadikov, Kukina & Porai-Koshits, 1967; Aslanov, Abdul'minev, Porai-Koshits & Ivanov, 1972) and can be described as a distorted tricapped trigonal prism defined by seven O atoms (from acetate groups) and two water molecules [Ow(1), Ow(3)]. Co atoms are in nearly regular octahedra. The molecular

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54018 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Selected bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

Co octahedron									
Co	O(5)	O(5)	O(3)	O(3)	O(7)	O(7)			
O(5)	2.089 (1)	4.179 (0)	2.950 (2)	2.996 (2)	2.786 (1)	3.150 (3)			
O(5)	180.0 (0.0)	2.089 (1)	2.996 (2)	2.950 (2)	3.150 (3)	2.786 (1)			
O(3)	89.1 (0.2)	90.9 (0.1)	2.115 (1)	4.230 (2)	3.049 (2)	2.933 (2)			
O(3)	90.9 (0.1)	89.1 (0.2)	180.0 (0.1)	2.115 (1)	2.933 (2)	3.049 (2)			
O(7)	83.0 (0.2)	97.0 (0.2)	92.2 (0.1)	87.8 (0.1)	2.116 (1)	4.231 (3)			
O(7)	97.0 (0.2)	83.0 (0.2)	87.8 (0.1)	92.2 (0.1)	180.0 (0.2)	2.116 (1)			
Nd tricapped trigonal prism									
Nd	O(1)	O(1)	O(2)	O(3)	O(4)	O(6)	O(8)	Ow(1)	Ow(3)
O(1)	2.406 (2)	2.782 (4)	4.131 (3)	4.787 (1)	4.829 (1)	3.218 (2)	3.193 (3)	3.085 (2)	3.367 (2)
O(1)	65.4 (0.2)	2.724 (1)	2.181 (2)	4.615 (2)	4.387 (1)	2.979 (3)	4.726 (1)	2.936 (2)	4.814 (1)
O(2)	114.4 (0.1)	49.1 (0.2)	2.508 (1)	3.171 (3)	2.891 (4)	3.021 (3)	4.632 (1)	3.201 (3)	4.682 (2)
O(3)	155.8 (0.1)	124.5 (0.1)	78.8 (0.2)	2.490 (2)	2.181 (2)	3.182 (1)	2.950 (2)	4.454 (2)	3.676 (2)
O(4)	151.3 (0.1)	111.6 (0.1)	69.3 (0.2)	50.9 (0.1)	2.578 (1)	4.417 (2)	4.060 (2)	3.102 (2)	3.118 (3)
O(6)	83.4 (0.1)	70.3 (0.2)	75.4 (0.2)	80.5 (0.2)	123.6 (0.1)	2.433 (2)	2.951 (4)	4.620 (1)	4.671 (4)
O(8)	83.9 (0.2)	136.0 (0.1)	143.4 (0.1)	74.7 (0.2)	110.2 (0.1)	75.8 (0.2)	2.370 (1)	4.542 (2)	2.731 (3)
Ow(1)	77.7 (0.1)	68.1 (0.2)	79.3 (0.2)	126.0 (0.1)	75.1 (0.1)	138.4 (0.1)	137.1 (0.1)	2.509 (2)	2.938 (5)
Ow(3)	87.1 (0.1)	135.2 (0.1)	139.5 (0.1)	95.3 (0.1)	76.0 (0.2)	143.8 (0.2)	68.4 (0.2)	72.1 (0.2)	2.482 (2)
Acetate groups									
Nd—Co <i>anti-syn</i> + monatomic bridge					Nd—Co <i>syn-syn</i> bridge				
C(1)—O(3)	1.283 (3)				C(2)—O(7)	1.262 (2)			
C(1)—O(4)	1.247 (4)				C(2)—O(8)	1.252 (4)			
O(3)—O(4)	2.181 (1)				O(7)—O(8)	2.235 (2)			
O(3)—C(1)—O(4)	119.1 (3)				O(7)—C(2)—O(8)	125.5 (2)			
Nd—Nd <i>syn-syn</i> + monatomic bridge					Nd—Co <i>syn-syn</i> bridge				
C(3)—O(1)	1.266 (3)				C(4)—O(5)	1.257 (4)			
C(3)—O(2)	1.256 (6)				C(4)—O(6)	1.269 (2)			
O(1)—O(2)	2.181 (1)				O(5)—O(6)	2.234 (1)			
O(1)—C(3)—O(2)	119.7 (3)				O(5)—C(4)—O(6)	124.4 (3)			
C(1)—C(5)	1.491 (4)				C(2)—C(6)	1.505 (6)			
C(3)—C(7)	1.490 (7)				C(4)—C(8)	1.508 (8)			
Non-coordinated water molecules									
Ow(2)—O(2)	2.725 (5)	Ow(4)—Ow(3)	2.853 (2)						
Ow(2)—O(7)	2.863 (3)	Ow(4)—O(5)	2.925 (6)						
Ow(2)—Ow(5)	2.782 (4)	Ow(5)—Ow(3)	2.682 (7)						

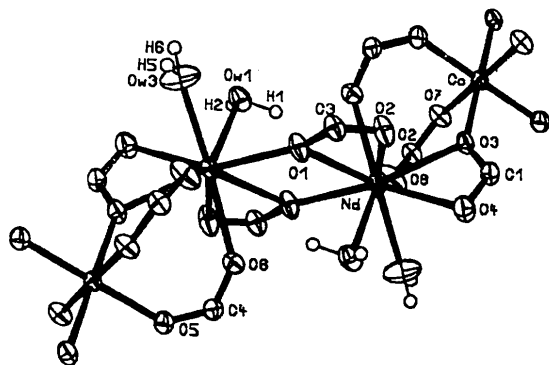


Fig. 1. ORTEP drawing (Johnson, 1965) of Nd<sub>2</sub>Co(CH<sub>3</sub>COO)<sub>8</sub>.10H<sub>2</sub>O with the atom-labelling scheme (atoms not labelled may be identified by using the inversion centres either on the Co atoms or relating the two Nd atoms) and thermal ellipsoids (scaled to include 50% probability). Lattice water molecules and CH<sub>3</sub> units from acetate groups have been omitted for clarity; thermal parameters of the H atoms have been divided by ten.

unit, which involves one Co and two Nd atoms, contains a centrosymmetric dimer with two acetate groups behaving as both *syn-syn* and monatomic (*i.e.* 3<sub>2</sub>-*s*; Porai-Koshits, 1980) bridges between the Nd atoms. In each moiety, neighbouring Nd and Co atoms are linked through three acetate groups (two

acting as *syn-syn* or *s-2-s* bridges and one as both *anti-syn* and monatomic or a 3<sub>2</sub>-*a* bridge). In this way, each cobalt octahedron shares two corners [2 × O(3)] with two different trigonal prisms and the adjacent trigonal prisms are linked by one edge [2 × O(1)]. This results in infinite chains of metallic polyhedra (NdNdCo) along the *c* axis in which the cohesion is furnished by the four independent acetate groups. These chains are interconnected *via* hydrogen bonding through three non-coordinated water molecules [Ow(2), Ow(4), Ow(5)] which are some of the five water molecules that are crystallographically independent. Although there are no crystal data for any hydrates of neodymium triacetate (Vadura & Kvapil, 1971), there exists a great similarity between the dimeric core of the title Nd—Co complex and that found in Ln(CH<sub>3</sub>COO)<sub>3</sub>.4H<sub>2</sub>O (Ln = Sm—Lu) (Aslanov, Abdul'minev, Porai-Koshits & Ivanov, 1972; Vadura & Kvapil, 1971). Even though these differ slightly (mainly in the nature of the metal-to-metal acetate bridges) from that described for Ce(CH<sub>3</sub>COO)<sub>3</sub>.0.7H<sub>2</sub>O (Sadikov, Kukina & Porai-Koshits, 1967), our present results seem to confirm the stabilization in solution of dinuclear lanthanide anionic acetate complexes. Given the bridging versatility of the carboxylate groups, the presence of a suitable cation can yield polymetallic derivatives.

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## Structures of $(\text{PPh}_4)_2[\text{Pd}(\text{Te}_4)_2]\cdot\text{DMF}$ and $(\text{Ph}_2\text{PC}_2\text{H}_4\text{PPh}_2)\text{Pt}(\mu\text{-Te})_2\text{Pt}(\text{Ph}_2\text{PC}_2\text{H}_4\text{PPh}_2)\cdot 2\text{DMF}$ . $^{125}\text{Te}$ Gamma Ray Resonance of $(\text{PPh}_4)_2[\text{Pd}(\text{Te}_4)_2]\cdot\text{DMF}$

BY HANS WOLKERS AND KURT DEHNICKE\*

*Fachbereich Chemie der Universität Marburg, Hans Meerwein-Straße, D-3550 Marburg, Germany*

DIETER FENSKE

*Institut für Anorganische Chemie der Universität Karlsruhe, Engesserstraße, D-7500 Karlsruhe, Germany*

AND AIRAT KHASANOV AND STEFAN S. HAFNER

*Institut für Mineralogie der Universität Marburg, Hans Meerwein-Straße, D-3550 Marburg, Germany*

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**Abstract.** Bis(tetraphenylphosphonium) octatelluro-palladate-*N,N*-dimethylformamide (1/1),  $[\text{P}(\text{C}_6\text{H}_5)_4]_2[\text{Pd}(\text{Te}_4)_2]\cdot\text{C}_3\text{H}_7\text{NO}$  (I),  $M_r = 1879.11$ , orthorhombic, *Pbca*,  $a = 16.320$  (8),  $b = 23.409$  (11),  $c = 29.606$  (13) Å,  $V = 11310.7$  (10) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 2.20$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 41.5$  cm<sup>-1</sup>,  $F(000) = 6880$ ,  $T = 293$  K,  $R = 0.049$  for 6931 observed unique reflections. The structure consists of  $\text{PPh}_4^+$  cations and  $[\text{Pd}(\text{Te}_4)_2]^{2-}$  anions with nearly square-planar coordination of palladium with two chelated  $\text{Te}_4^{2-}$  ligands.  $^{125}\text{Te}$  gamma resonance yields a large quadrupole splitting indicative of a significantly asymmetric population of the  $5p$  orbitals similar to that observed for transition metal ditellurides.  $\text{Di}[1,2\text{-bis}(\text{diphenylphosphino})\text{ethane-}P,P']\text{-di-}\mu\text{-tellurio-diplatinum-dimethylformamide}$  (1/2),  $\text{Pt}_2\text{Te}_2[(\text{C}_6\text{H}_5)_2\text{PC}_2\text{H}_4\text{P}(\text{C}_6\text{H}_5)_2]_2\cdot 2\text{C}_3\text{H}_7\text{NO}$  (II),  $M_r = 1588.40$ , triclinic,  $P\bar{1}$ ,  $a = 9.474$  (6),  $b = 12.342$  (8),  $c = 14.222$  (9) Å,  $\alpha = 114.41$  (3),  $\beta = 97.44$  (3),  $\gamma = 100.85$  (3)°,  $V = 1446.7$  (8) Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.78$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu =$

$56.9$  cm<sup>-1</sup>,  $F(000) = 760$ ,  $T = 200$  K,  $R = 0.048$  for 5212 observed unique reflections. The complex has a centrosymmetric molecular structure with a planar  $\text{Pt}_2\text{Te}_2$  ring, and two chelating (tetraphenyldiphosphino)ethane ligands.

**Introduction.** Recently, the interest in polytellurium and polyselenium complexes has increased significantly, mainly because of their expected semiconducting properties (Böttcher, 1988; Ansari & Ibers, 1990; Kanatzidis, 1990). In this paper, the syntheses of two different polytellurium complexes, their crystal structures, and some data obtained from  $^{125}\text{Te}$  gamma ray resonance are reported. Adams, Wolfe, Eichhorn & Haushalter (1989) had obtained a triclinic form (space group  $P\bar{1}$ ) of solvate-free  $(\text{PPh}_4)_2[\text{Pd}(\text{Te}_4)_2]$  by reacting  $\text{PdCl}_2$  with  $\text{K}_2\text{Te}_4$  in dimethylformamide (DMF) in the presence of  $\text{PPh}_4\text{Br}$  dissolved in ethylenediamine. Subsequently, Kanatzidis (1990) replaced ethylenediamine by ether, otherwise using a similar procedure. His result was orthorhombic  $(\text{PPh}_4)_2[\text{Pd}(\text{Te}_4)_2]$  with the same space group as  $(\text{PPh}_4)_2[\text{Pd}(\text{Te}_4)_2]\cdot\text{DMF}$  (I) of this study.

\* To whom correspondence should be addressed.