

The Crystal Structure and Coordination Geometry of Potassium-Catena- μ -Oxalato-Bis-Oxalato Aquo Lanthanate(III) Dihydrates, $K_3[Ln(Ox)_3(OH_2)] \cdot 2H_2O$, (Ln = Nd, Sm, Eu, Gd, Tb)

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The crystal structures of the title compounds with Ln = Nd and Gd were determined at room temperature by single crystal X-ray diffraction methods. The compounds are triclinic with space group $P\bar{1}$ and $Z = 2$. The Ln^{3+} cations are nonacoordinate in a distorted tricapped trigonal prismatic environment formed by 4 oxalates and a water molecule. The oxalates coordinate to Ln^{3+} via 5-membered rings; two of these 4 oxalate ligands serve as bridging ligands to coordinate to neighboring Ln^{3+} leading to an infinite chain of $[Ox(LnOx_2 \cdot H_2O)]_n \cdots$ units. Compounds with Ln = Sm, Eu, and Tb are isomorphous with this structure as determined by their cell dimensions.

Introduction

Dioxalato complexes of trivalent lanthanides, $M LnOx_2 \cdot nH_2O$, where $M = NH_4^+$, [1, 2] and $H_5O_2^+$ [3] are well known and characterized. No other oxalato complexes of trivalent lanthanides have been reported in spite of the fact that other oxalato metallates of the type MOx_3^{3-} , $M = Al, Cr$ [4] Rh [5] and carboxylato lanthanates of the type: LnL_3^{3-} , e.g. $Na_3LnL_3 \cdot NaClO_4 \cdot 6H_2O$, $L = OOCCH_2 \cdot OCH_2COO$ [6] and $Na_3Ln(dip)_3 \cdot nH_2O$, dip = dipicolinate anion [7], are well known. Mixed oxalates are versatile starting materials for the fabrication of well-defined multicomponent oxides which have found wide industrial application because of their important optical, magnetic and electrical properties; e.g. $MTiO_3$, an important ferroelectric and ion conductor [8], is made by the thermodecomposition of $BaTiOx_3 \cdot H_2O$ [9]. The inclusion of rare

earth atoms in the mixed oxide system has improved and extended its use to semiconductor, capacitor and electrooptic systems [8]. The use of mixed oxide systems containing lanthanide atoms in industrial catalysis [10, 11] and laser technology [12] is well known.

For these reasons we undertook the synthesis of oxalato lanthanates in search of new or starting materials for the fabrication of lanthanide based systems showing characteristics of current technological interest. We have now prepared complexes of the type $K_3LnOx_3 \cdot nH_2O$ ($n = 6$ for Ln = La, $n = 5.5$ for Ln = Ce, Pr, and $n = 3$, for Ln = Nd-Tb) and $K_8Ln_2Ox_7 \cdot 14H_2O$ (Ln = Tb-Yb, Y). We report here the crystal structures of the complexes $K_3LnOx_3 \cdot 3H_2O$, specifically where Ln = Nd, Sm, Eu, Gd and Tb.

Experimental

Preparation of the Complexes

Lanthanide sesquioxalates $Ln_2Ox_3 \cdot nH_2O$ prepared according to established procedures were suspended in about 10 ml of boiling water; solid potassium oxalate was then added slowly, allowing each aliquot to dissolve before adding another one, until a clear solution was obtained. The solutions were allowed to cool to 45–55 °C and then evaporation was allowed to proceed to near dryness. Excess potassium oxalate was then extracted as a dilute solution with a 0.5 M potassium oxalate solution. Use of this solution was necessary as the complexes were found to decompose to the dioxalato lanthanates when extraction was attempted with more dilute solutions. The crystals were digested in the potassium oxalate extract for 2 days at 45–55 °C, after which they were filtered and air dried for 15 minutes. The crystalline precipitates obtained were found to have the general stoichiometry: $K_3LnOx_3 \cdot nH_2O$

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TABLE I. Crystal Data for the Triclinic Complexes $K_3[Ln(Ox)_3(OH_2)] \cdot 2H_2O$.

Ln:	Nd	Sm	Eu	Gd	Tb
Formula wt.:	579.6	585.8	587.4	592.7	594.3
Space group:	P1	P1	P1	P1	P1
<i>a</i> (Å)	9.416(2)	9.392(3)	9.383(1)	9.374(4)	9.361(5)
<i>b</i> (Å)	8.502(4)	8.488(1)	8.484(1)	8.474(4)	8.475(2)
<i>c</i> (Å)	9.799(3)	9.767(2)	9.751(1)	9.739(2)	9.716(3)
α (°)	88.91(3)	88.89(1)	88.86(1)	88.34(3)	88.79(2)
β (°)	82.17(3)	81.85(2)	81.76(1)	81.62(2)	81.54(3)
γ (°)	96.28(3)	96.46(2)	96.55(1)	96.58(4)	96.67(3)
<i>V</i> (Å ³)	772.0(7)	765.4(5)	762.7(2)	759.8(7)	756.7(8)
<i>d_c</i> (g cm ⁻³)	2.493	2.542	2.558	2.590	2.608
<i>Z</i>	2	2	2	2	2
μ (cm ⁻¹)	42.63	47.43	50.14	52.86	56.30

where $n = 6$ for Ln = La, $n = 5.5$ for Ln = Ce, Pr, and $n = 3$ for Ln = Nd–Tb.

On the other hand the heavy lanthanides and yttrium formed very soluble complexes. Therefore the solutions formed as above were cooled to room temperature and the crystalline materials obtained were recrystallized from dilute solutions of potassium oxalate. These materials have the stoichiometry: $K_8Ln_2Ox_7 \cdot 14H_2O$: Ln = Tb–Yb, Y.

In attempts to establish the maximum number of oxalate ligands which can be put on the lanthanide cations, the colored ions Nd³⁺ and Er³⁺ were used since they are easily identified by their color. In this case complexation was carried out in a saturated solution of potassium oxalate and the crystals were isolated from the solid mixtures with the aid of a microscope. The materials do not co-precipitate with potassium oxalate, but form well defined crystals which were found to be identical with those obtained by the procedure described above.

X-ray Data Collection for $K_3[Nd(Ox)_3OH_2] \cdot 2H_2O$

Intensity data were obtained from a pale lavender crystal of dimensions $0.20 \times 0.28 \times 0.40$ mm on an Enraf-Nonius CAD4 diffractometer equipped with MoK α radiation ($\lambda = 0.71073$ Å) and a graphite monochromator. Cell dimensions (Table I) and crystal orientation were determined from the angular settings of 25 reflections having $13^\circ < \theta < 14^\circ$. Data were collected at 25 °C by the ω - 2θ scan method employing scans of variable rate designed to yield $I \cong 50 \sigma(I)$ for all significant reflections. Scan rates, determined in a 10 deg. min⁻¹ prescan, varied from 0.50–10.0 deg min⁻¹. All data in one hemisphere having $1^\circ \leq \theta \leq 25^\circ$ were measured in this fashion. No decline in the intensities of three periodically remeasured reflections nor movement of two periodically recentered reflections was noted. Of 2709 unique data, 2529 had $I > 3\sigma(I)$, and were

used in the refinement. Data reduction included corrections for background, Lorentz, polarization and absorption effects. The absorption correction was based upon ψ scans of reflections near $\chi = 90^\circ$; the minimum relative transmission coefficient was 77.09%.

X-ray Data Collection for $K_3[Gd(Ox)_3OH_2] \cdot 2H_2O$

The experimental procedure was identical to that for the Nd compound. Differing parameters are: crystal size $0.16 \times 0.24 \times 0.44$ mm, scan rates 0.63–10.0 deg min⁻¹, 2666 unique data, 2512 observed data, minimum relative transmission coefficient 78.87%.

Cell Dimensions for $K_3[Ln(Ox)_3OH_2] \cdot 2H_2O$

Unit cell dimensions for the isomorphous series, including the Sm, Eu, and Tb compounds were obtained from single crystals with MoK α radiation on the CAD4 diffractometer. Setting angles for the same set of 25 reflections used for orienting the Nd and Gd crystals were used in a least squares procedure. Measurements at $\pm 2\theta$ were made. Crystal data derived from these measurements are given in Table I.

Structure Solution and Refinement, $K_3[Nd(Ox)_3OH_2] \cdot 2H_2O$

The centrosymmetric space group was assumed, and confirmed by successful refinement. Location of the Nd atom from the Patterson map led to the full structure by standard Fourier methods. Refinement was carried out by full matrix least squares based upon F with statistical weights, using the Enraf Nonius SDP programs [13]. Nonhydrogen atoms were refined anisotropically; hydrogen atoms were located from difference maps and included as fixed contributions with isotropic $B = 5.0$ Å². Convergence was achieved with $R = 0.036$ (0.040 for all

TABLE II. Triclinic Complexes: $K_3LnOx_3 \cdot 3H_2O$; Principal Interatomic Distances and Bond Angles in the Lanthanide Coordination Polyhedron.

	Nd (e.s.d. = 0.003 Å)	Gd (e.s.d. = 0.002 Å)
(a) distances		
Ln–O1	2.525	2.464
Ln–O2	2.474	2.429
Ln–O3	2.484	2.444
Ln–O4	2.492	2.450
Ln–O5	2.450	2.404
Ln–O6	2.463	2.416
Ln–O7	2.447	2.392
Ln–O8	2.536	2.504
Ln–O13(H ₂ O)	2.572	2.517
Average Ln–O	2.494	2.447
(b) angles		
O1–Ln–O2	65.74(8)	67.27(7)
O1–Ln–O3	75.17(9)	74.72(8)
O1–Ln–O4	70.24(9)	70.54(8)
O1–Ln–O5	128.89(9)	129.23(8)
O1–Ln–O6	151.47(9)	150.06(8)
O1–Ln–O7	85.00(9)	85.66(8)
O1–Ln–O8	67.15(9)	67.42(8)
O1–Ln–O13	131.94(8)	132.46(8)
O2–Ln–O3	130.89(8)	132.58(8)
O2–Ln–O4	74.05(8)	74.87(8)
O2–Ln–O5	70.77(8)	69.90(8)
O2–Ln–O6	136.47(9)	136.62(8)
O2–Ln–O7	131.35(8)	132.13(7)
O2–Ln–O8	68.62(8)	68.23(7)
O2–Ln–O13	91.01(9)	90.12(8)
O3–Ln–O4	65.31(8)	66.29(7)
O3–Ln–O5	118.91(9)	111.95(8)
O3–Ln–O6	76.33(9)	75.37(8)
O3–Ln–O7	69.79(9)	68.99(8)
O3–Ln–O8	121.73(9)	121.26(8)
O3–Ln–O13	138.01(9)	131.18(8)
O4–Ln–O5	73.16(9)	73.10(8)
O4–Ln–O6	96.46(9)	96.07(8)
O4–Ln–O7	132.73(9)	133.33(8)
O4–Ln–O8	131.74(9)	131.91(8)
O4–Ln–O13	145.44(9)	145.14(8)
O5–Ln–O6	65.87(9)	66.90(8)
O5–Ln–O7	145.70(9)	144.71(8)
O5–Ln–O8	119.30(9)	118.76(8)
O5–Ln–O13	72.51(9)	72.20(8)
O6–Ln–O7	86.50(9)	84.97(8)
O6–Ln–O8	131.69(9)	131.96(8)
O6–Ln–O13	72.88(9)	73.31(8)
O7–Ln–O8	64.31(8)	65.03(7)
O7–Ln–O13	80.41(9)	79.80(8)
O8–Ln–O13	65.23(9)	65.38(8)

data), $R_w = 0.047$, and goodness of fit = 1.617 for 266 variables. The maximum residual electron density was $0.66 \text{ e}\text{\AA}^{-3}$.

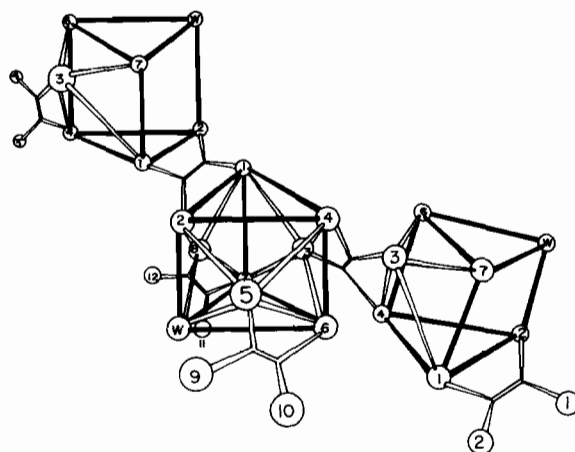


Fig. 1. A portion of the structure of $K_3[Ln(Ox)_3(OH_2)] \cdot 2H_2O$, showing all of the nine coordinated oxygens (1–8 from Ox and W from OH_2) which comprise the tricapped trigonal prism, and portions of the two neighboring oxalato bridged trigonal prisms.

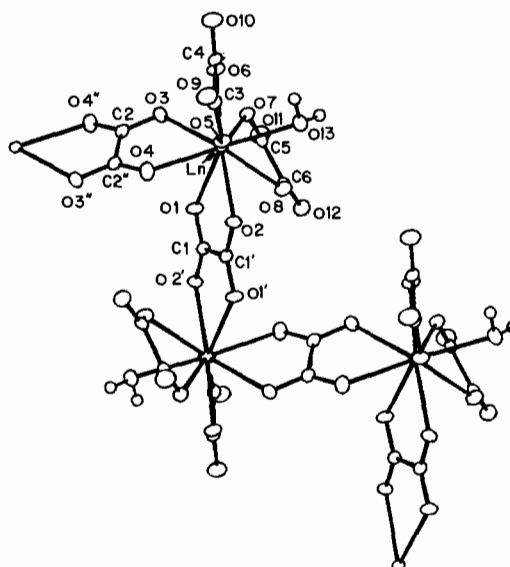


Fig. 2. ORTEP drawing of a portion of the polymeric structure of $[Ln(Ox)_3(OH_2)]^{3-}$, with atom numbering scheme. Parameters for Ln = Gd were used.

Solution and Refinement, $K_3[Gd(Ox)_3OH_2] \cdot 2H_2O$

As the Gd compound is isomorphous with the Nd compound, parameters from the latter were used as a starting model. Refinement was carried out in the same fashion as that for the Nd compound, with hydrogen atom positions adjusted by difference maps. At convergence, $R = 0.021$ (0.024 for all data), $R_w = 0.033$, $GOF = 1.184$, and maximum residual electron density = $0.69 \text{ e}\text{\AA}^{-3}$.

TABLE III. Distances and Angles in the Oxalate Ligands.

	Nd	Gd
Ligand 1: C1-C1	1.575(4)	1.567(6)
C1-O1	1.238(4)	1.253(4)
C1-O2	1.246(4)	1.247(4)
O1-C1-O2	125.5(3)	125.5(3)
O1-C1-C1	116.8(3)	116.2(4)
O2-C1-C1	117.8(3)	118.2(3)
Ligand 2: C2-C2	1.539(4)	1.556(6)
C2-O3	1.258(4)	1.261(4)
C2-O4	1.254(4)	1.241(4)
O3-C2-O4	125.7(3)	126.8(3)
O3-C2-C2	117.0(3)	115.4(4)
O4-C2-C2	117.3(3)	117.8(4)
Ligand 3: C3-C4	1.558(4)	1.564(5)
C3-O5	1.273(4)	1.272(4)
C3-O9	1.232(5)	1.226(4)
C4-O6	1.257(5)	1.266(4)
C4-O1	1.246(5)	1.230(4)
O5-C3-O9	125.8(4)	125.9(3)
O5-C3-C4	115.6(4)	114.9(3)
O9-C3-C4	118.6(4)	119.3(3)
O6-C4-O10	127.4(4)	127.1(3)
O6-C4-C3	116.3(9)	115.7(3)
O10-C4-C3	116.2(4)	117.1(3)
Ligand 4: C5-C6	1.574(4)	1.567(5)
C5-O7	1.269(5)	1.269(4)
C5-O11	1.226(5)	1.234(4)
C6-O8	1.267(5)	1.259(4)
C6-O12	1.225(5)	1.226(4)
O7-C5-O11	125.8(4)	125.7(3)
O7-C5-C6	115.7(4)	115.6(3)
O11-C5-C6	118.5(4)	118.7(3)
O8-C6-O12	126.1(4)	127.2(3)
O8-C6-C5	114.6(4)	114.2(3)
O12-C6-C5	119.3(4)	118.6(3)
Averages: C-C	1.562	1.564
C-O(bg)*	1.249	1.251
C-O _i (nbg)*	1.267	1.267
C-O _{ii} (nbg)*	1.235	1.229

*bg = bridging, nbg = non-bridging.

Structure Description

Lanthanides Nd through Tb form triclinic hydrated complex salts with the formula $K_3Ln(Ox)_3 \cdot 3H_2O$. The lanthanide atoms are nonacoordinate, with a geometry best described as a distorted tricapped trigonal prism, with the nine donor atoms composed of two bidentate oxalato ligands, two bridging oxalato ligands, and a coordinated water molecule. The water molecule occupies one of the prism corners, three of the oxalates span corner-to-cap positions, and one of the bridging oxalates spans the edge of a trigonal face (Figs. 1, 2). The bridging oxalates lie on centers of symmetry at $\frac{1}{2} \frac{1}{2} 0$ and $\frac{1}{2} 00$, leading to infinite zigzag chains of coordination centers

TABLE IV. Principal Distances in the Coordination Polyhedra of the Potassium Ions.

	Nd	Gd
K1-O2	2.868(3)	2.854(2)
K1-O4	2.913(3)	2.941(2)
K1-O5	2.828(3)	2.817(3)
K1-O9	2.786(3)	2.792(3)
K1-O10	3.179(3)	3.233(3)
K1-O12	2.824(3)	2.817(3)
K1-O14	2.736(3)	2.715(3)
K1-O15	2.797(3)	2.757(3)
K2-O3	2.867(3)	2.879(2)
K2-O6	3.015(3)	2.962(2)
K2-O7	2.830(3)	2.803(2)
K2-O7	2.851(3)	2.853(2)
K2-O9	2.738(3)	2.727(3)
K2-O10	3.100(3)	3.158(3)
K2-O11	3.105(3)	3.068(3)
K2-O12	2.966(3)	2.969(3)
K3-O2	3.002(3)	2.968(2)
K3-O6	2.938(3)	2.941(2)
K3-O8	2.790(3)	2.773(2)
K3-O10	2.867(3)	2.839(3)
K3-O11	2.718(3)	2.708(3)
K3-O13	2.971(3)	2.958(3)
K3-O1	2.861(3)	2.851(3)

running parallel to the b axis of the crystal. The distance Ln-O8 is the longest next to Ln-O13 and this makes the cap at the O8 apex unequal to the other two. The plane occupied by the equatorial oxygens is tilted so that O8 is above O3 and O5. Ln-O (oxalate) distances (Table II) in the Nd complex are in the range 2.447(3)–2.536(3) Å and average 2.484 Å; the Nd-O (water) distance is 2.572(3) Å. Corresponding values for the Gd compound are 2.392(2)–2.504(2) Å, average 2.438 Å, and Gd-O (water) 2.517(2) Å, reflecting the smaller ionic radius of Gd³⁺. The average Ln-O distances are in good agreement with those found in other structures in which Ln³⁺ is nonacoordinate, e.g. Nd-O = 2.50 Å in Nd₂Ox₃·10.5H₂O [14] and Nd₂(mal)₃·6H₂O (mal = malonate), [15] and Gd-O = 2.44 Å in Na₃Gd(O(CH₂COO)₂)₃·NaClO₄·6H₂O [16]. The interatomic distances within the oxalato ligands, listed in Table III, are longer than those found in Nd₂Ox₃·10.5H₂O [14]. The C-C distances are in the range 1.539(4)–1.575(4) Å and average 1.562 Å (Nd) and 1.564 Å (Gd) in contrast to the range 1.47–1.59 Å and an average of 1.54(4) Å found in Nd₂Ox₃·10.5H₂O [14]. The normal oxalates have a rigid extensive bridging structure involving all oxalate ligands while the complexes studied here have modest bridging. The thermodynamic consequences of bridging differences are discussed

elsewhere, [16]. The C—O distances fall into three distinct classes: bridging C—O, non-bridging coordinated C—O and non-bridging uncoordinated C—O (Table III). Non-bridging oxalates are planar to within 0.09 Å; bridging oxalates to within 0.005 Å. As expected the non-bridging oxalates coordinate to Ln^{3+} via their most negatively charged oxygens. An accumulation of negative charge on Ln^{3+} occurs, and the magnitude of this seems to have a great influence on the coordination numbers of Ln^{3+} and the maximum number of anions the Ln^{3+} can accommodate [17].

The three K^+ ions form contacts less than 3.3 Å with various oxygen atoms of the structures, as tabulated in Table IV. Two are octacoordinate and the third (K3) is heptacoordinate. K1 has six contacts to oxalate oxygen atoms and two to uncoordinated (to Ln^{3+}) water molecules. K2 has eight contacts to oxalate oxygen atoms as well as a longer distance (3.427(3) Å to Gd) to the coordinated water molecule. K3 has five contacts to oxalate oxygens, one to the coordinated water molecule, and one to an uncoordinated water molecule.

Group Trends

As is evident from the data presented in Table I, lanthanides from Nd to Tb (with the possible but unlikely exception of Pm, which was not studied) all have this chain structure. The effects of the lanthanide contraction appear in the smooth decrease in unit cell volume and resulting increase in calculated density. In addition to the decreasing trends in axial lengths, the trends in interaxial angles are those of decreasing α , decreasing β , and increasing γ in proceeding from Nd to Tb. These trends are upset somewhat by the b and α values of Tb.

Preliminary results on the structure determination of the compound $\text{K}_3\text{PrOx}_3 \cdot 5.5\text{H}_2\text{O}$ indicate that the complex has a different structure. The break in the

lanthanide series is thus found at Pr—Nd and Tb. No other break is found in the crystal properties of the complexes of the heavy lanthanides [17].

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Appendix added at proof stage:

Coordinates for $\text{K}_3\text{Nd}(\text{oxalate})_3 \cdot 3\text{H}_2\text{O}$.

Atom	x	y	z
Nd	0.35566(2)	0.22705(2)	0.22081(2)
K1	0.1414(1)	0.2885(1)	0.8873(1)
K2	0.3172(1)	0.0458(1)	0.6133(1)
K3	0.7823(1)	0.3425(1)	0.7010(1)
O1	0.5816(4)	0.3499(4)	0.0711(3)
O2	0.3180(3)	0.4502(3)	0.0690(3)
O3	0.5067(4)	0.0065(4)	0.1771(3)
O4	0.3645(3)	0.1215(4)	-0.0153(3)
O5	0.1071(3)	0.1928(3)	0.1689(3)
O6	0.2054(4)	-0.0028(4)	0.3414(3)
O7	0.5119(4)	0.2118(4)	0.3996(3)

(continued)

Atom	x	y	z
O8	0.4556(3)	0.4903(4)	0.3067(3)
O9	-0.0961(4)	0.0296(4)	0.1760(4)
O10	-0.0102(4)	-0.1496(4)	0.3734(4)
O11	0.7302(4)	0.3147(4)	0.4359(4)
O12	0.6661(4)	0.6071(4)	0.3519(3)
O13	0.1951(4)	0.3332(4)	0.4242(3)
O14	0.9641(4)	0.4714(4)	0.2435(4)
O15	0.8426(4)	0.2240(4)	0.9597(4)
C1	0.5766(4)	0.4713(5)	0.0009(4)

(continued overleaf)

(continued)

Atom	x	y	z
C2	0.5410(5)	-0.0329(5)	0.0550(4)
C3	0.0223(5)	0.0699(5)	0.2125(4)
C4	0.0769(5)	-0.0368(5)	0.3208(5)
C5	0.6115(5)	0.3250(5)	0.4006(4)
C6	0.5767(5)	0.4915(5)	0.3497(4)
H131	0.1133	0.2773	0.4707
H132	0.2285	0.3594	0.5000
H141	1.0000	0.3887	0.1934
H142	0.8848	0.4434	0.3047
H151	0.8555	0.1387	1.0273
H152	0.7695	0.2773	1.0000

Estimated standard deviations in the least significant digits are shown in parentheses.

Coordinates for $K_3Gd(oxalate)_3 \cdot 3H_2O$.

Atom	x	y	z
Gd	0.35762(1)	0.22748(2)	0.21755(1)
K1	0.14156(9)	0.2887(1)	0.88675(8)
K2	0.31730(9)	0.0435(1)	0.61061(9)
K3	0.78057(10)	0.3434(1)	0.70243(9)
O1	0.5807(3)	0.3462(3)	0.0683(3)
O2	0.3165(2)	0.4476(3)	0.0703(2)

(continued)

Atom	x	y	z
O3	0.5084(3)	0.0112(3)	0.1772(2)
O4	0.3645(3)	0.1216(3)	-0.0152(3)
O5	0.1118(3)	0.1939(3)	0.1694(3)
O6	0.2128(3)	-0.0013(3)	0.3389(3)
O7	0.5082(3)	0.2115(3)	0.3944(2)
O8	0.4569(3)	0.4898(3)	0.3003(3)
O9	-0.0929(3)	0.0304(3)	0.1781(3)
O10	-0.0035(3)	-0.1488(3)	0.3775(3)
O11	0.7257(3)	0.3143(3)	0.4382(3)
O12	0.6644(3)	0.6078(3)	0.3554(3)
O13	0.1995(3)	0.3306(3)	0.4192(2)
O14	0.9625(3)	0.4730(3)	0.2445(3)
O15	0.8448(3)	0.2236(4)	0.9593(3)
C1	0.5766(3)	0.4712(4)	-0.0013(3)
C2	0.5429(4)	-0.0323(4)	0.0550(3)
C3	0.0258(4)	0.0704(4)	0.2136(4)
C4	0.0815(4)	-0.0361(4)	0.3227(4)
C5	0.6082(4)	0.3250(4)	0.3981(3)
C6	0.5756(4)	0.4918(4)	0.3466(3)
H131	0.1387	0.2500	0.4434
H132	0.2500	0.3594	0.5000
H141	1.0000	0.3887	0.2207
H142	0.9160	0.4160	0.3047
H151	0.8613	0.1680	1.0293
H152	0.7793	0.2793	1.0293

Estimated standard deviations in the least significant digits are shown in parentheses.