Inorganica Chimica Acta, *82* (1984) 167–172

The Crystal and Molecular Structures of Potassium-μ-Oxalato-Di[tris-oxalato-lanthanate(III)]-14-Hydrates $K_8[Ox_3LnOxLnOx_3]\cdot 14H_2O$ [Ln = Tb, Dy, Er, Yb, Y]

I. A. KAHWA*, F. R. FRONCZEK and J. SELBIN**

Chemistry Department, Louisiana State University, Baton Rouge, La. 70803, U.S.A.

Received June 11, 1983

The crystal structures of the title complexes were determined at room temperature by single crystal X-ray diffraction. The complexes are monoclinic with space group C2/c and Z = 4. They exist as dinuclear species with two Ln^{3+} cations linked by a single oxalate bridge. The Ln^{3+} coordination polyhedron is a slightly distorted D_{2d} dodecahedron. The dimer has crystallographic C_2 symmetry with the bridging group normal to the twofold axis. Full structure determinations were carried out for Ln = Er and Y, while the structures with Ln = Tb, Dy, and Yb were determined to be isomorphous by determination of unit cell dimensions.

Introduction

Recently we reported the crystal structure of the trioxalatolanthanate complexes $K_3LnOx_3\cdot 3H_2O$ (Ln = Nd, Sm, Eu, Gd, Tb) [1]. In that report we described the procedure for the synthesis of crystalline oxalato complexes of heavy lanthanides with what appears to be a maximum number of possible oxalato ligands of 3.5 per Ln^{3+} cation ($K_8Ln_2Ox_7\cdot 14H_2O$). We have now determined the crystal and molecular structures of these complexes and have established that the compounds $K_8Ln_2Ox_7\cdot 14H_2O$, Ln = Tb, Dy, Er, Yb, Y are isomorphous and monoclinic with space group C2/c. Unlike the polymeric triclinic trioxalato complexes, the monoclinic complexes are dinuclear species with the lanthanide nuclei bridged by a single bis-bidentate oxalato ligand. The lanthanide coordination sphere is a slightly distorted dodecahedron made up of oxalate ligands only. The coordination number 8 is surprising in view of the many known oxalates in which Ln^{3+} (Ln = heavy lanthanide) are nonacoordinate, *e.g.* $H_5O_2^+$ $[ErOx_2\cdot H_2O]^-$ [2], $NH_4[YOx_2H_2O]\cdot H_2O$ [3], and $Ln_2Ox_3\cdot 10H_2O$ [4]. In the dioxalato Y complex the coordinated water molecule constitutes the shortest Y–O distance, indicating no instability associated with the YO_9 coordination polyhedron. A comparison of our results with data reported for other types of complexes revealed a useful (not unexpected) rule of thumb, namely that the lanthanide cations prefer lower coordination numbers if complexation results in an excess negative charge on the complex lanthanate anion. In this report we present the crystal data of $K_8Ln_2Ox_7\cdot 14H_2O$ complexes, full crystal structure determinations for the Er and Y complexes and the rationale for lower coordination numbers shown by Ln^{3+} in complex lanthanate anions. The probable reasons for differences in the complexes formed by the light and heavy lanthanides are also discussed.

Experimental

a) Synthesis of $K_8Ln_2Ox_7\cdot 14H_2O$

The complexes were synthesized as described elsewhere [1].

b) Data collection for $K_8[Er_2(Ox)_7]\cdot 14H_2O$

Intensity data were obtained from a pale pink crystal of dimensions 0.20 × 0.28 × 0.36 mm on an Enraf-Nonius CAD4 diffractometer equipped with MoKα radiation (λ = 0.71073 Å) and a graphite monochromator. Cell dimensions (Table I) and crystal orientation were determined from the angular settings of 25 reflections having $14^\circ \leqslant \theta \leqslant 15^\circ$. 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 0.39–10.0 deg min^{-1}. All data in one quadrant

---

*On study leave from the Chemistry Department, University of Dar-es-Salaam, P.O. Box 35061, Dar-es-Salaam, Tanzania.

**Author to whom correspondence should be addressed.

0020-1693/84/$3.00

TABLE I. Crystal Data for the Monoclinic Complexes $K_8[Ln_2Ox_7]\cdot 14H_2O$.

Ln	Tb	Dy	Er	Yb	Y
Formula wt:	1499.1	1506.2	1515.7	1527.3	1359.0
Space Group:	C2/c	C2/c	C2/c	C2/c	C2/c
a (A):	16.113(6)	16.078(5)	16.079(3)	16.062(7)	16.068(2)
b (A):	11.821(3)	11.785(3)	11.773(2)	11.726(5)	11.764(3)
c (A):	23.491(11)	23.453(8)	23.371(4)	23.301(7)	23.402(7)
β (•):	91.45(4)	91.25(3)	91.21(2)	90.94(3)	90.89(2)
V (A^3):	4473(5)	4443(4)	4423(3)	4388(5)	4423(3)
dc (g cm^{-3}):	2.226	2.252	2.276	2.312	2.041
Z:	4	4	4	4	4
μ (cm^{-1}):	40.33	42.35	46.90	51.02	34.97

having $1^\circ \leqslant \theta \leqslant 25^\circ$ and h + k even were measured in this fashion. No decline in the intensities of three periodically remeasured reflections nor movement of two periodically recentered reflections was noted. 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%. Equivalent data were averaged, yielding 3870 unique data, of which 3266 had $I > 3\sigma(I)$, and were used in the refinement.

Data collection for $K_8[Y_2(Ox)_7]\cdot 14H_2O$

The experimental procedure was identical to that for the Er compound. Differing parameters are: crystal size 0.24 × 0.24 × 0.44 mm, T = 27 °C, scan rates 0.35–10.0 deg min^{-1}, 3877 unique data, 2931 observed data, minimum relative transmission coefficient 86.23%.

Cell Dimensions for $K_8[Ln_2(Ox)_7]\cdot 14H_2O$

Unit cell dimensions for the isomorphous series, including the Tb, Dy, and Yb compounds, were obtained from single crystals with MoKα radiation on the CAD4 diffractometer. Setting angles for the same set of 25 reflections used for orienting the Er and Y 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, $K_8[Er_2(Ox)_7]\cdot 14H_2O$

Systematic absences hkl with k + k odd and hol with 1 odd limit possible space groups to C2/c and Cc. The centrosymmetric space group was assumed, and confirmed by successful refinement. Location of the Er 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 [5]. Nonhydrogen atoms were refined anisotropically; hydrogen atoms on four of the water molecules were located and included as fixed contributions with B = 5.0 Å^2. One of the K ions was found to be disordered into two equally-populated sites separated by 2.709 Å and related by the center at 0 ½ ½. One of the water molecules (O7W) was found to be disordered into two sites with approximate occupancies 0.79 (O7WA) and 0.21 (O7WB). The former was refined anisotropically, and the latter isotropically. Convergence was achieved with R = 0.035 (0.051 for all data), Rw = 0.040, and goodness of fit = 2.30 for 307 variables. The maximum residual electron density was 1.23 eÅ^{-3}.

Solution and Refinement, $K_8[Y_2(Ox)_7]\cdot 14H_2O$

As the Y compound is isomorphous with the Er compound, parameters from the latter were used as a starting model. Refinement was carried out in the same fashion as that for the Er compound with hydrogen atom positions adjusted by difference maps. At convergence, R = 0.037, Rw = 0.051, GOF = 1.632 and maximum residual density = 0.5 eÅ^{-3}.

Structure Description

Late lanthanides Tb, Dy, Er and Yb, as well as Y, form monoclinic hydrated complexes of formula $K_8[Ln_2(Ox)_7]\cdot 14H_2O$. These complexes exist as discrete dimers with a single oxalato bridge linking two octacoordinate metal centers. The lanthanide ions are coordinated by three bidentate oxalato ligands and one bridging oxalate in a slightly distorted D_{2d} dodecahedron (Fig. 1). The dimer has crystallographic C_2 symmetry, with the bridging group

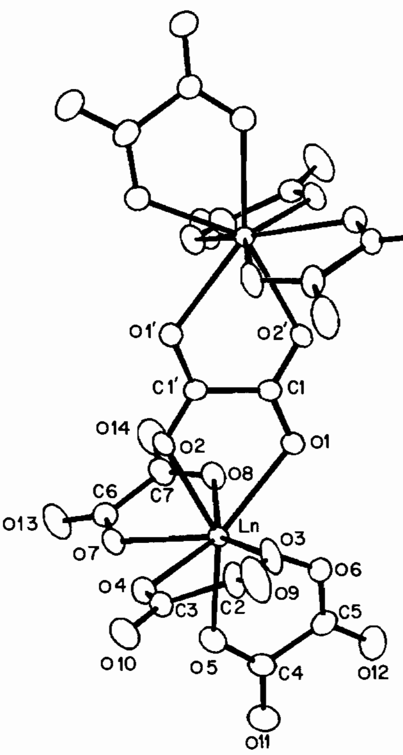

Fig. 1. ORTEP view of the $[Ln_2(Ox)_7]^{3-}$ ion viewed in a direction slightly oblique to the crystallographic twofold axis, illustrating atom numbering scheme. Parameters for Ln = Er were used.

TABLE II. Principal Interatomic Distances and Angles in the Coordination Polyhedron of the Lanthanide Ions.

a) distances (A)	Y	Er
(e.s.d.)	(0.003)	(0.002)
Ln-O1	2.386	2.396
Ln-O2	2.352	2.352
Ln-O3	2.339	2.342
Ln-O4	2.342	2.342
Ln-O5	2.299	2.315
Ln-O6	2.342	2.322
Ln-O7	2.351	2.337
Ln-O8	2.338	2.337
Average Ln-O	2.344	2.343
(b) Angles	Y	Er
O1-Ln-O2	68.49(9)	68.58(6)
O1-Ln-O3	75.73(11)	74.93(8)
O1-Ln-O4	131.12(11)	131.24(7)
O1-Ln-O5	141.46(11)	141.62(7)
O1-Ln-O6	73.70(10)	73.49(7)
O1-Ln-O7	130.43(10)	130.89(7)
O1-Ln-O8	73.75(11)	74.11(7)
O2-Ln-O3	87.22(12)	86.99(8)
O2-Ln-O4	77.56(10)	77.75(7)
O2-Ln-O5	149.62(11)	149.43(7)
O2-Ln-O6	142.19(10)	142.06(7)
O2-Ln-O7	77.72(10)	78.20(7)
O2-Ln-O8	86.33(12)	86.61(8)
O3-Ln-O4	68.33(11)	68.93(7)
O3-Ln-O5	103.47(13)	103.85(9)
O3-Ln-O6	83.24(11)	82.90(8)
O3-Ln-O7	138.62(10)	139.28(7)
O3-Ln-O8	149.06(11)	148.59(7)
O4-Ln-O5	80.19(11)	79.72(8)
O4-Ln-O6	130.71(11)	130.56(8)
O4-Ln-O7	70.84(10)	70.92(7)
O4-Ln-O8	138.83(11)	138.84(7)
O5-Ln-O6	68.02(11)	68.37(7)
O5-Ln-O7	75.59(11)	75.07(8)
O5-Ln-O8	97.23(12)	97.12(9)
O6-Ln-O7	130.54(11)	130.29(8)
O6-Ln-O8	83.51(11)	83.45(8)
O7-Ln-O8	68.79(10)	68.68(7)

TABLE III. Principal Interatomic Distances and Angles in the Oxalate Ligands.

Ligands 1	Y	Er
C1-C1	1.543(7)	1.531(5)
C1-O1	1.247(5)	1.254(3)
C1-O2	1.241(5)	1.252(3)
O1-C1-O2	126.3(4)	125.7(3)
O1-C1-C1	117.3(4)	117.3(3)
O2-C1-C1	116.4(5)	117.0(3)
Ligand 2		
C2-C3	1.564(6)	1.539(4)
C2-O3	1.263(5)	1.291(4)
C2-O9	1.228(6)	1.247(4)
C3-O4	1.264(5)	1.267(3)
C3-O10	1.224(5)	1.232(3)
O3-C2-O9	125.6(4)	122.4(3)
O3-C2-C3	115.4(4)	116.2(3)
O9-C2-C3	119.0(4)	121.3(3)
O4-C3-O10	126.4(4)	125.8(3)
O4-C3-C2	114.0(4)	115.1(3)
O10-C3-C2	119.6(4)	119.1(3)
Ligand 3		
C4-C5	1.536(7)	1.536(4)
C4-O5	1.279(5)	1.252(4)
C4-O11	1.227(6)	1.223(4)
C5-O6	1.251(6)	1.272(4)
C5-O12	1.241(5)	1.242(4)
O5-C4-O11	125.9(4)	126.2(3)
O5-C4-C5	114.3(4)	114.6(3)
O11-C4-C5	119.9(4)	119.1(3)
O6-C5-O12	126.8(5)	126.0(3)
O6-C5-C4	115.2(4)	115.4(3)
O12-C5-C4	118.0(4)	118.6(3)

(continued overleaf)

TABLE III. *(continued)*

Ligand 4		
C6–C7	1.555(6)	1.557(4)
C6–O7	1.258(5)	1.276(4)
C6–O13	1.241(5)	1.237(4)
C7–O8	1.266(6)	1.256(4)
C7–O14	1.220(6)	1.220(4)
O7–C6–O13	125.6(4)	125.4(3)
O7–C6–C7	115.7(4)	115.4(3)
O13–C6–C7	118.7(4)	119.2(3)
O8–C7–O14	125.5(4)	126.3(3)
O8–C7–C6	115.1(4)	114.7(3)
O14–C7–C6	119.4(4)	118.9(3)
Averages		
C–C (br)	1.543	1.531
C–C (nbr)	1.552	1.544
C–O (br)	1.245	1.253
C–O1 (nbr)	1.264	1.269
C–O2 (nbr)	1.230	1.234

normal to the twofold axis. Ln–O distances for the Er complex are in the range 2.315(2)–2.396(2) Å and average 2.343 Å; analogous values for the Y complex are 2.299(3)–2.386(3) Å and 2.344 Å (Table II). The distances fall in the range found for octacoordinated Y and Er.

Interatomic distances within the oxalato ligands are given in Table III. The C–C bond lengths in the Er complex range 1.531(5)–1.557(4) Å and average 1.538 Å; corresponding values for the Y complex are 1.536(7)–1.564(6) Å and 1.548 Å. The bridging oxalates interact more with the Ln^{3+} ions than the nonbridging ones, and have correspondingly shorter C–C bonds than the nonbridging ones as expected [6]. Averages for C–C distances are 1.543 Å for bridging ligands, 1.552 Å for nonbridging ligands for the Y complex, and respectively 1.531 and 1.544 Å for the Er complex. Averages for the C–O distances are 1.245(Y) and 1.253(Er) (bridging C–O), 1.264(Y) and 1.269(Er) (nonbridging coordinated C–O) and 1.230 Å(Y), and 1.234 Å(Er) for nonbridging uncoordinated C–O. These distances (C–O) agree closely with those found in planar and nonplanar carboxylate groups. Nonbridging oxalates are planar to within 0.13 Å, while the bridging oxalate is planar to within 0.02 Å.

Characterization of the K^+ ion coordination is complicated by the disorder in one of the cations (K5), in one of the water molecules (O7W), and by the high thermal motion and resulting uncertainty in the positions of most of the water molecules. Cation K1 has seven contacts of length less than 3.0 Å to oxalato oxygen atoms and a longer contact of 3.262(7) Å to water molecule O6W. Cation K2 is clearly octacoordinate, having four contacts of 3.0 Å or less to oxalato oxygen atoms and four similar contacts to water molecules. Cation K3 has six contacts less than 3.0 Å to oxalato O atoms and three contacts (2.94–3.32 Å) to water molecules. Cation K4, which lies on a twofold axis, had four contacts to the oxalato oxygen atoms and four to water molecules in the range 2.79–3.11 Å. The disordered cation position (K5) has two contacts to oxalato O atoms, four to ordered water molecules, and one to one of the disordered water molecule sites. There is also a distance of 2.64 Å between the two half-populated K5 positions of $K_8Y_2Ox_7 \cdot 14H_2O$, however, this distance is too short to allow them to be simultaneously occupied.

From the crystal data in Table I, it is apparent that lanthanides Tb, Dy, Er and Yb, as well as Y, form the oxalato dimeric structure. Ho and Tm were not studied. Proceeding across the series from Tb to Yb, the following trends are evident: decreasing unit cell volume, increasing density, decrease in each of the axial lengths, and a steady decrease in the β angle towards mutual orthogonality of the axes. Perhaps not unexpectedly, the Y compound does not fit well into the trends, having an identical cell volume to the Er compound, but a smaller β angle than any of the other compounds studied.

The Influence of the Ionic Potential of Ln^{3+} on the Maximum Number of Oxalates the Cation can accomodate and the Coordination Number of Ln^{3+}

This work and that covered elsewhere [1] have demonstrated that the maximum number of oxalates the lighter (larger) members of the lanthanide series are capable of accepting is 3 while the heavier members may accept up to 3.5 oxalates per Ln^{3+} cation [1]. It seems (not unexpectedly) that cations with a large ionic potential (q/r) have larger tolerance for large negative changes than those with a small q/r. The cutoff point in the series $LnOx_3^{3-}$ to $LnOx_{3.5}^{4-}$ is at Tb^{3+} with $q/r = 3.25$ ($r = 0.923$ Å in six-fold coordination [7] employed for comparative purposes) whereas the large but tetravalent cation Th^{4+} with ionic potential 4.0 may accept four and perhaps more oxalate ligands [8]. The ionic potentials of Th^{4+} and U^{4+} are large enough to support the large negative field afforded by the four ligands in $ThOx_4^{4-}$ and UOx_4^{4-}.

The preference for a lower coordination number by the smaller Ln^{3+} cations noted earlier appears to result from their larger q/r values which improves the tolerance of Ln^{3+} for excess negative charges. Thus, in complexes of Ln^{3+} with small coordination numbers the radius of Ln^{3+} is usually smaller than it is in complexes with large coordination numbers [7]. The ratio of Ox:Ln increases from 3.0 to 3.5, when the reduction in coordination number results in a sufficiently large q/r value to support excess negative charge of 4 on the resulting lanthanate complex

TABLE IV. Coordination Numbers in Some Lanthanide Compounds.

Complex compound	Negative charge on the complex lanthanate anion	CN	Ref.
$H_5O_2{}^+[Er(OxH \cdot Ox \cdot OH_2]^-$	1	9	2
$K_8[Er_2Ox_7] \cdot 14H_2O$	4	8	this work
$NH_4YOx_2 \cdot OH_2] \cdot H_2O$	1	9	3
$K_8[Y_2Ox_7] \cdot 14H_2O$	4	8	this work
$La[EDTAH \cdot (OH_2)_4] \cdot 3H_2O$	0	10	9
$KLa[EDTA \cdot (OH_2)_3] \cdot 5H_2O$	1	9	10
Ln_2O_3 (Ln = Light Ln)	0	7	11
$K[LnO_2]$ (Ln = Light Ln)	1	6	12
$LnPO_4$	0	8	13
$Na_3[Ln(PO_4)_2]$	3	6	14
$K_3[Ln_2(NO_3)_9]$ (Ln = Pr, Nd, Sm)	1.5	12	15
$[Ph_3EtP]_2[Ce(NO_3)_5]$	2	10	16
$LnCl_3$ (Ln = La–Gd)	0	9	19
(Ln = Tb, Dy)	0	8	19
$LnCl_6{}^{3-}$	3	6*	20
$LnBr_3$ (Ln = La–Pr)	0	9	19
(Ln = Nd–Eu)	0	8	19
$LnBr_6{}^{3-}$	3	6*	20
LnI (Ln = La–Nd)	0	8	19
$LnI_6{}^{3-}$	3	6*	21

*Spectroscopic evidence.

anion. Preference for low coordination numbers when the complex formation results in such excess negative charge is widespread among lanthanide compounds (see Table IV).

In fact large coordination numbers, *e.g.* 11 and 12, are found in complexes with neutral ligands such as neutral macrocycles with ethereal oxygens or nitrogen ligating atoms and/or monovalent bidentate ligands *e.g.* $NO_3{}^-$ [15, 17], which do not impose high excess negative charges on the lanthanide(ate) complex ion. In view of the foregoing, it seems to us that the distortions leading to an anomalously long Ln–O8 distance in the triclinic complexes [1] might be an expression of the need to expel the ninth atom from the coordination sphere of Ln^{3+}. The continued presence of the water molecule in the Ln^{3+} coordination sphere might be due to its convenient location on the corner of the prismatic triangle as well as to the neutrality of the H_2O.

Conclusion

Although steric factors frequently enjoy prominence when assessing the stability of a given coordination polyhedron [18] differences in the ionic potentials of Ln^{3+} seem also to play an important role. The Ln^{3+} cation will achieve the largest ionic potential possible to better accommodate an excess negative charge on the lanthanide. The formation of negatively charged lanthanate anions thus depends on the ability of Ln^{3+} to support the resulting negative field.

Acknowledgements

We thank the U.S.A. government for a Fulbright scholarship to I. A. Kahwa.

References

1 I. A. Kahwa, F. R. Fronczek and J. Selbin, *Inorganica Chimica Acta, 82* (1984) 161.
2 S. Hugo and B. D. George, *Inorg. Chem., 9,* 2112 (1970).
3 T. R. R. McDonald and J. M. Spink, *Acta Cryst., 23,* 944 (1967).
4 W. Ollendorff and F. Weigel, *Inorg. Nucl. Chem. Lett., 5,* 264 (1968).

5 B. A. Frenz and Y. Okaya, 'Enraf-Nonius Structure Determination Package', Delft, Holland, 1980.
6 I. A. Kahwa and A. M. Mulokozi, *J. Therm. Anal., 24,* 264 (1982).
7 R. D. Shannon and C. Prewitt, *Acta Cryst., B25,* 925 (1971).
8 M. N. Akhtah and A. J. Smith, *Acta Cryst., B31,* 1361 (1975).
9 J. L. Hoard, B. Lee and M. D. Lind, *J. Am. Chem. Soc., 87,* 1612 (1965).
10 M. D. Lind, B. Lee and J. L. Hoard, *J. Am. Chem. Soc., 87,* 1611 (1965).
11 L. Pauling, *Z. Krist., 69,* 415 (1928).
12 R. Clos, M. Devalette, P. Hagenmuller, R. Hoppe and F. Paletta; *Compt. Rend., 265C,* 801 (1967).
13 R. C. L. Mooney, *Acta Cryst., 3,* 337 (1950).
14 M. Vlasse, C. Parent, R. Salmon and G. L. Flem, 'The Rare Earths in Modern Science and Technology', Vol. 2, 1980, p. 195.
15 W. T. Carnall, S. Siegel, J. R. Ferraro, B. Tani and E. Gebert, *Inorg. Chem., 12,* 560 (1973).
16 A. R. Al-Karaghouli and J. S. Wood, *J. Chem. Soc. Chem. Comm.,* 135 (1970).
17 J. D. J. Backer-Dirks, C. J. Gray, F. A. Hart, M. B. Hursthouse and B. L. Schoop, *J. Chem Soc. Chem. Comm.,* 774 (1979).
18 S. P. Sinha, *Structure and Bonding, 25,* 70 (1976).
19 D. G. Karraker, *J. Chem. Ed., 47,* 424 (1970).
20 J. L. Ryan and C. K. Jorgenson, *J. Phys. Chem., 70,* 2845 (1966).
21 J. L. Ryan, *Inorg. Chem., 8,* 2053 (1969).

Appendix added at proof stage:

Coordinates for $K_8[Er_2(oxalate)_7]\cdot 14H_2O$.

Atom	x	y	z
Er	0.11120(2)	0.17221(3)	0.14464(1)
K1	−0.2692(1)	0.1703(2)	0.22454(9)
K2	0.1520(1)	0.3382(2)	−0.10137(8)
K3	0.0993(1)	0.0196(2)	0.39714(9)
K4	0.5	0.1305(3)	0.25
K5[a]	0.0202(3)	0.4152(5)	0.4631(2)
O1	−0.0287(3)	0.1810(5)	0.1777(2)
O2	−0.1078(3)	0.1820(5)	0.2549(2)
O3	0.0773(3)	0.3657(5)	0.1441(3)
O4	0.2281(3)	0.2839(4)	0.1668(2)
O5	0.1864(3)	0.1483(6)	0.0621(2)
O6	0.0251(3)	0.1691(5)	0.0636(2)
O7	0.2219(3)	0.0537(4)	0.1714(2)
O8	0.0677(3)	−0.0162(5)	0.1539(3)
O9	0.1156(4)	0.5381(5)	0.1733(3)
O10	0.2786(3)	0.4574(5)	0.1822(3)
O11	0.1852(4)	0.1282(6)	−0.0318(3)
O12	0.0158(4)	0.1364(7)	−0.0307(3)
O13	0.2657(4)	−0.1256(5)	0.1806(3)
O14	0.1004(4)	−0.1896(5)	0.1828(3)
O1W	0.4229(4)	0.2999(6)	0.1814(3)
O2W	0.4779(5)	0.1376(8)	0.4194(4)
O3W	0.4206(4)	0.9784(7)	0.1749(4)
O4W	0.1459(5)	0.4226(8)	0.0134(4)
O5W	0.3464(8)	0.3494(16)	0.4818(7)
O6W	0.2401(13)	0.1762(18)	0.4132(8)
O7WA[b]	0.1796(15)	0.3593(25)	0.4455(12)
O7WB[c]	0.078(4)	0.389(6)	0.451(3)
C1	−0.0391(4)	0.1817(6)	0.2307(3)
C2	0.1329(5)	0.4377(7)	0.1615(4)
C3	0.2215(4)	0.3908(6)	0.1708(3)
C4	0.1510(5)	0.1413(7)	0.0140(3)
C5	0.0558(5)	0.1492(7)	0.0149(3)
C6	0.2103(5)	−0.0533(7)	0.1744(3)
C7	0.1180(5)	−0.0924(7)	0.1694(4)

Estimated standard deviations in the least significant digits are shown in parentheses. [a]Population 1/2. [b]Population 0.79. [c]Population 0.21.

Coordinates for $K_8[Y_2(oxalate)_7]\cdot 14H_2O$.

Atom	x	y	z
Y	0.11099(3)	0.17084(4)	0.14463(2)
K1	−0.26867(7)	0.1690(1)	0.22407(6)
K2	0.15219(8)	0.3397(1)	−0.10153(6)
K3	0.09974(9)	0.0182(1)	0.39709(6)
K4	0.5	0.1301(2)	0.25
K5[a]	0.01866(21)	0.4179(3)	0.46393(16)
O1	−0.0285(2)	0.1786(3)	0.1779(1)
O2	−0.1072(2)	0.1802(3)	0.2550(1)
O3	0.0784(2)	0.3646(3)	0.1440(2)
O4	0.2275(2)	0.2829(3)	0.1669(2)
O5	0.1856(2)	0.1470(4)	0.0624(2)
O6	0.0247(2)	0.1674(3)	0.0631(1)
O7	0.2223(2)	0.0520(3)	0.1720(2)
O8	0.0673(2)	−0.0177(3)	0.1542(2)
O9	0.1165(2)	0.5362(3)	0.1734(2)
O10	0.2782(2)	0.4560(3)	0.1821(2)
O11	0.1848(3)	0.1297(4)	−0.0325(2)
O12	0.0150(3)	0.1355(4)	−0.0307(2)
O13	0.2660(2)	−0.1264(3)	0.1810(2)
O14	0.1009(3)	−0.1908(3)	0.1830(2)
O1W	0.4236(3)	0.2981(4)	0.1813(2)
O2W	0.4776(3)	0.1378(5)	0.4193(2)
O3W	0.4206(3)	0.9772(4)	0.1763(3)
O4W	0.1444(3)	0.4217(5)	0.0145(2)
O5W	0.3463(5)	0.3457(8)	0.4820(5)
O6W	0.2378(9)	0.1744(9)	0.4148(5)
O7WA[b]	0.1717(11)	0.3627(14)	0.4405(7)
O7WB[c]	0.0664(20)	0.3822(29)	0.4507(14)
C1	−0.0392(3)	0.1807(4)	0.2306(2)
C2	0.1309(3)	0.4360(5)	0.1626(3)
C3	0.2215(3)	0.3897(5)	0.1714(2)
C4	0.1498(3)	0.1403(5)	0.0134(2)
C5	0.0544(3)	0.1478(5)	0.0149(2)
C6	0.2108(3)	−0.0535(5)	0.1750(2)
C7	0.1185(3)	−0.0930(5)	0.1708(3)

Estimated standard deviations in the least significant digits are shown in parentheses. [a]Population 1/2. [b]Population 0.79. [c]Population 0.21.