Crystal structure of diaqualutetium(III) succinate dihydrate, $\text{Lu}(\text{H}_2\text{O})_2(\text{C}_4\text{H}_4\text{O}_4)_{1.5} \cdot 2\text{H}_2\text{O}$, the first succinate of a trivalent cation

M. Fleck*

Universität Wien - Geozentrum, Institut für Mineralogie und Kristallographie, Althanstraße 14, A-1090 Wien, Austria

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Abstract

$\text{C}_6\text{H}_{14}\text{LuO}_{10}$, monoclinic, $P12_1/c1$ (No. 14), $a = 5.224(1) \text{ \AA}$, $b = 26.055(5) \text{ \AA}$, $c = 8.800(2) \text{ \AA}$, $\beta = 97.38(3)^\circ$, $V = 1187.9 \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.019$, $wR_{\text{ref}}(F^2) = 0.046$, $T = 293 \text{ K}$.

Source of material

Small needle-shaped crystals (up to a length of 0.5 mm) of $\text{Lu}(\text{H}_2\text{O})_2(\text{C}_4\text{H}_4\text{O}_4)_{1.5} \cdot 2\text{H}_2\text{O}$ were obtained by controlled evaporation from an aqueous solution of the reaction product of succinic acid and lutetium(III) oxide in water at 353 K.

Discussion

Several succinates and malates of metal cations have been synthesised and studied crystallographically hitherto ([1,2] and references cited therein). However, all these compounds are salts of monovalent or divalent cations.

The crystal structure of $\text{Lu}(\text{H}_2\text{O})_2(\text{C}_4\text{H}_4\text{O}_4)_{1.5} \cdot 2\text{H}_2\text{O}$ consists of isolated irregular LuO_8 -polyhedra (Lu—O distances of 2.191 Å to 2.399 Å). Two corners of each polyhedron are water molecules (OW1 and OW2), the remaining six corners are oxygen atoms of the carboxyl groups of three succinate molecules. It is noteworthy that there are two crystallographically different succinate molecules in $\text{Lu}(\text{H}_2\text{O})_2(\text{C}_4\text{H}_4\text{O}_4)_{1.5} \cdot 2\text{H}_2\text{O}$. One is located on a centre of symmetry and therefore oriented in perfect *trans*-conformation (torsion angle C1B—C2B—C2B*—C1B* of 180°), the other one has *gauche*-conformation (torsion angle C1A—C2A—C3A—C4A of $66.3(4)^\circ$) with site symmetry 1. These organic molecules connect the polyhedra to a three-dimensional framework. This framework is not packed equally dense in all directions. Along [100] there are channels framed by the succinate molecules. In these channels, two crystallographically different, non-coordinating water molecules (OW3 and OW4) are located. A system of hydrogen bonds connects the building units additionally.

The structure of $\text{Lu}(\text{H}_2\text{O})_2(\text{C}_4\text{H}_4\text{O}_4)_{1.5} \cdot 2\text{H}_2\text{O}$ is novel in two respects: the title compound is the first example of a succinate of a trivalent cation. Also, it is the only succinate that has organic molecules in both *trans*- and *gauche*-conformation (although there is one malate that also shows this feature, namely copperdihydrogen-L-dimalate, $\text{CuH}_2(\text{C}_4\text{H}_4\text{O}_5)_2$ [3]. Further syntheses of succinates and malates with trivalent cations will be attempted.

Table 1. Data collection and handling.

Crystal:	colourless needle, size $0.05 \times 0.08 \times 0.40 \text{ mm}$
Wavelength:	Mo K_α radiation ($0.71073 \text{ \AA}$)
μ :	83.52 cm^{-1}
Diffractometer, scan mode:	Nonius Kappa CCD, ϕ/ω
$2\theta_{\text{max}}$:	60.98°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	7084, 3622
$N(\text{param})_{\text{refined}}$:	204
Programs:	SHELXS-97 [4], SHELXL-97 [5], ATOMS [6]

* e-mail: michel.fleck@univie.ac.at

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(2A1)	4e	1.005(7)	0.144(2)	0.620(4)	0.035(9)
H(2A2)	4e	1.056(9)	0.202(2)	0.649(5)	0.06(1)
H(3A1)	4e	0.586(8)	0.165(2)	0.513(5)	0.04(1)
H(3A2)	4e	0.769(9)	0.194(2)	0.421(5)	0.06(1)
H(2B1)	4e	0.2238	0.5170	0.4500	0.05
H(2B2)	4e	0.0005	0.4889	0.3474	0.05
H(1W1)	4e	0.696(9)	0.402(2)	0.747(5)	0.05(1)

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(2W1)	4e	0.85(1)	0.388(2)	0.660(5)	0.06(2)
H(1W2)	4e	0.065(8)	0.299(2)	0.690(4)	0.04(1)
H(2W2)	4e	0.255(8)	0.290(2)	0.787(5)	0.05(1)
H(1W3)	4e	0.738(9)	0.477(2)	0.905(5)	0.06(1)
H(2W3)	4e	0.914(8)	0.439(2)	0.993(5)	0.05
H(1W4)	4e	0.415(9)	0.439(2)	0.050(5)	0.05
H(2W4)	4e	0.30(1)	0.440(2)	0.187(6)	0.07(2)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Lu	4e	0.40348(2)	0.340833(4)	0.52144(1)	0.0224(2)	0.0229(2)	0.0238(2)	0.00240(3)	0.00336(4)	0.00131(4)
O(11A)	4e	0.6000(4)	0.16646(9)	0.8076(2)	0.0249(9)	0.047(1)	0.032(1)	-0.0053(8)	0.0063(8)	-0.0025(9)
O(12A)	4e	1.0041(4)	0.1639(1)	0.9103(3)	0.033(1)	0.056(2)	0.031(1)	-0.001(1)	-0.0051(9)	-0.0022(9)
C(1A)	4e	0.8350(5)	0.1685(1)	0.7954(3)	0.025(1)	0.022(1)	0.029(1)	-0.0010(9)	0.0035(9)	-0.0002(9)
C(2A)	4e	0.9236(6)	0.1760(1)	0.6417(3)	0.032(1)	0.029(1)	0.033(1)	0.008(1)	0.012(1)	0.005(1)
C(3A)	4e	0.7100(7)	0.1899(1)	0.5159(3)	0.047(2)	0.028(1)	0.024(1)	0.009(1)	0.008(1)	-0.000(1)
C(4A)	4e	0.5868(5)	0.2414(1)	0.5330(3)	0.028(1)	0.026(1)	0.025(1)	0.0017(9)	0.0060(9)	0.0004(9)
O(41A)	4e	0.3731(4)	0.25162(8)	0.4539(2)	0.032(1)	0.030(1)	0.034(1)	0.0017(8)	-0.0038(8)	-0.0060(8)
O(42A)	4e	0.6977(4)	0.27586(7)	0.6198(2)	0.0262(9)	0.0256(9)	0.032(1)	0.0006(7)	0.0002(7)	-0.0011(7)
O(11B)	4e	0.1887(4)	0.41599(8)	0.6099(3)	0.033(1)	0.029(1)	0.045(1)	0.0050(8)	0.0123(9)	0.0020(8)
O(12B)	4e	0.3578(5)	0.42179(9)	0.3963(3)	0.056(2)	0.037(1)	0.054(1)	0.016(1)	0.025(1)	0.016(1)
C(1B)	4e	0.2197(6)	0.4404(1)	0.4895(4)	0.037(2)	0.028(1)	0.057(2)	0.007(1)	0.014(1)	0.010(1)
C(2B)	4e	0.090(1)	0.4911(2)	0.4508(6)	0.084(3)	0.043(2)	0.086(3)	0.034(2)	0.044(3)	0.027(2)
O(1W)	4e	0.7134(4)	0.38393(9)	0.6794(3)	0.028(1)	0.034(1)	0.042(1)	-0.0020(8)	0.0091(9)	-0.0101(9)
O(2W)	4e	0.1987(4)	0.30923(8)	0.7173(2)	0.025(1)	0.034(1)	0.029(1)	-0.0006(8)	0.0007(8)	0.0076(8)
O(3W)	4e	0.7512(7)	0.4460(1)	0.9379(4)	0.066(2)	0.047(2)	0.054(2)	-0.002(1)	0.007(1)	-0.005(1)
O(4W)	4e	0.2861(7)	0.4472(1)	0.0903(4)	0.073(2)	0.056(2)	0.053(2)	0.006(2)	0.015(2)	0.011(1)

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