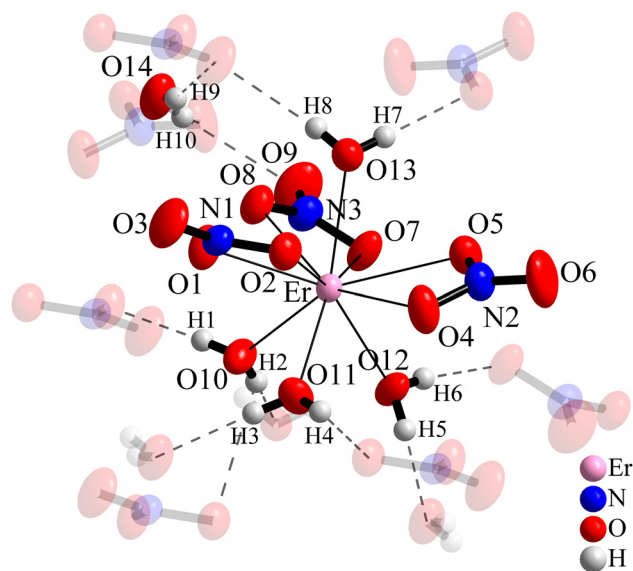


Wilhelm Klein*

Crystal structure of tetraaqua-tris(nitrato- κ^2O,O') erbium(III) monohydrate, $\text{Er}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, $\text{H}_{10}\text{ErN}_3\text{O}_{14}$

**Table 1:** Data collection and handling.

Crystal:	Pink fragment
Size:	0.30 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	7.41 mm ⁻¹
Diffractometer, scan mode:	STOE StadiVari, ω
θ_{max} , completeness:	30.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	21,486, 3337, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3078
$N(\text{param})_{\text{refined}}$:	204
Programs:	Stoe [1], SHELX [2, 3], Diamond [4]

From concentrated solutions pink single crystals up to 1 mm length were grown at room temperature within two weeks. For the X-ray data collection crystals have been immersed into perfluoroalkylether acts as glue on a glass tip during the measurement.

<https://doi.org/10.1515/ncrs-2021-0484>

Received December 22, 2021; accepted January 18, 2022;

published online January 31, 2022

Abstract

$\text{H}_{10}\text{ErN}_3\text{O}_{14}$, triclinic, $P\bar{1}$ (no. 2), $a = 6.5913(4)$ Å, $b = 9.5211(5)$ Å, $c = 10.4936(6)$ Å, $\alpha = 63.742(4)^\circ$, $\beta = 84.551(5)^\circ$, $\gamma = 76.038(5)^\circ$, $V = 573.09(6)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0141$, $wR_{\text{ref}}(F^2) = 0.0318$, $T = 223$ K.

CCDC no.: 2109106

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound $\text{Er}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ was prepared by dissolving Er_2O_3 (Fluka, 99.9%) in hot aqueous nitric acid.

*Corresponding author: Wilhelm Klein, Fakultät für Chemie, Technische Universität München, Lichtenbergstr. 4, 85747 Garching, Germany, E-mail: wilhelm.klein@tum.de. <https://orcid.org/0000-0002-6351-9921>

Experimental details

The H atoms have been located from the difference Fourier map and refined with unrestrained atomic coordinates and isotropic displacement parameters.

Comment

Anhydrous rare earth nitrates crystallize poorly because their thermal stability is usually too low to crystallize from melt. However, from aqueous solutions crystals of these compounds grow easily as hydrates, and there are numerous structural determinations of their hydrates [5]. While the highest amounts of water are found in hexahydrates which are structurally characterized for yttrium and the lanthanides La–Dy [5] as well as Tm [6], for some of the heavier rare earth elements, namely Ho [7] and Yb [8], at most the pentahydrates are known as maximally hydrated compounds so far [7, 8]. Also the structure of the pentahydrate of $\text{Er}(\text{NO}_3)_3$ has been investigated, but no structural data other than lattice parameters have been published [8].

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Er	0.24376 (2)	0.34916 (2)	0.29471 (2)	0.01301 (4)
N1	0.4680 (3)	0.2812 (2)	0.55523 (19)	0.0173 (3)
O1	0.2944 (3)	0.3817 (2)	0.50813 (18)	0.0233 (3)
O2	0.5523 (3)	0.21277 (19)	0.47857 (17)	0.0219 (3)
O3	0.5474 (3)	0.2555 (2)	0.6665 (2)	0.0319 (4)
N2	0.5196 (3)	0.2135 (2)	0.1248 (2)	0.0223 (4)
O4	0.5009 (3)	0.1406 (2)	0.25847 (18)	0.0275 (4)
O5	0.4057 (3)	0.35638 (19)	0.06676 (17)	0.0237 (3)
O6	0.6372 (4)	0.1552 (3)	0.0564 (2)	0.0393 (5)
N3	0.0243 (3)	0.6851 (2)	0.1465 (2)	0.0207 (4)
O7	0.0612 (3)	0.58090 (19)	0.09570 (18)	0.0232 (3)
O8	0.1048 (3)	0.6376 (2)	0.26695 (18)	0.0229 (3)
O9	-0.0811 (3)	0.8196 (2)	0.0812 (2)	0.0372 (5)
O10	-0.0912 (3)	0.3861 (2)	0.3939 (2)	0.0219 (3)
H1	-0.147 (6)	0.456 (5)	0.421 (4)	0.040 (10)*
H2	-0.178 (6)	0.387 (5)	0.352 (4)	0.042 (10)*
O11	0.1964 (3)	0.1000 (2)	0.46836 (18)	0.0220 (3)
H3	0.134 (6)	0.087 (4)	0.544 (4)	0.040 (10)*
H4	0.272 (6)	0.014 (4)	0.476 (4)	0.035 (9)*
O12	0.0252 (3)	0.2641 (2)	0.19424 (18)	0.0223 (3)
H5	0.027 (5)	0.162 (4)	0.221 (4)	0.036 (9)*
H6	0.008 (6)	0.312 (5)	0.112 (4)	0.047 (11)*
O13	0.5142 (3)	0.5014 (2)	0.22275 (18)	0.0203 (3)
H7	0.539 (6)	0.540 (5)	0.141 (4)	0.047 (11)*
H8	0.496 (5)	0.573 (4)	0.243 (4)	0.031 (9)*
O14	0.0041 (4)	0.9543 (2)	0.2792 (2)	0.0311 (4)
H9	0.097 (9)	0.890 (7)	0.290 (6)	0.083 (18)*
H10	-0.065 (7)	0.958 (5)	0.224 (5)	0.048 (12)*

The crystal structure of $\text{Er}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ was determined from single crystal data, the compound crystallizes in the $\text{Y}(\text{NO}_3)_3(\text{H}_2\text{O})_5$ structure type [9, 10] and is composed of $[\text{Er}(\text{NO}_3)_3(\text{H}_2\text{O})_4]$ complexes and free water molecules. All crystallographically independent nitrate anions are planar and have significantly different N–O bond lengths. The O atoms coordinated to the Er atom form much longer N–O bonds (1.259(2)–1.286(2) \AA) than the non-coordinating O atoms (1.210(3)–1.224(2) \AA). The Er atom is coordinated by 10 O atoms from three nitrate anions and four water molecules. All nitrate anions in the complex act as bidentate ligands. They are circularly coordinated around the Er atom and divide the water ligands into two groups, so that three lie on one and one on the other side of the nitrate ligands. The nitrate ions are slightly inclined to the main axis of the complex as defined by the Er–O13 bond (18.2–

24.3°) and to each other (28.7–39.9°), and are slightly asymmetrically coordinated with a shorter (2.4077(16)–2.4551(16) \AA) and a longer bond (2.5043(17)–2.6076(16) \AA) to the Er atom. The shortest Er–O bonds are formed to the O10–O12 molecules (2.3345(16)–2.3651(17) \AA), while the isolated ligand O13 is a little further away (2.4230(17) \AA). The positional and isotropic displacement parameters of the H atoms were refined freely, the O–H bond lengths were found to be between 0.73(6) and 0.88(4) \AA and the H–O–H angles between 102(4)° and 110(4)°. The molecular units are interconnected via relatively strong hydrogen bonds, as shown in the figure (note: probability level for non-H atom ellipsoids is 80%). More precisely, nine of the 10 independent H atoms form hydrogen bonds shorter than 2.38 \AA with O–H–O angles above 154°.

Author contributions: The author has accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: None declared.

Conflict of interest statement: The author declares no conflicts of interest regarding this article.

References

1. Stoe & Cie GmbH. X-Area (version 1.76); Stoe & Cie GmbH: Darmstadt, Germany, 2017.
2. Sheldrick G. M. A short history of SHELX. *Acta Crystallogr.* 2008, *A64*, 112–122.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Brandenburg K. DIAMOND. Visual Crystal Structure Information System (version 3.2i); Crystal Impact: Bonn, Germany, 2012.
5. Wickleder M. S. Inorganic lanthanide compounds with complex anions. *Chem. Rev.* 2002, *102*, 2011–2087.
6. Klein W. Crystal structures of the penta- and hexahydrate of thulium nitrate. *Acta Crystallogr.* 2020, *E76*, 1863–1867.
7. Rincke C., Schmidt H., Voigt W. Rebuttal of the existence of solid rare earth bicarbonates and the crystal structure of holmium nitrate pentahydrate. *Z. Anorg. Allg. Chem.* 2017, *643*, 437–442.
8. Junk P. C., Kepert D. L., Skelton B. W., White A. H. Structural systematics of rare earth complexes. XIII ('maximally') hydrated (heavy) rare earth nitrates. *Aust. J. Chem.* 1999, *52*, 497–505.
9. Eriksson B. Crystal and molecular structure of tetraaquatrinatoyttrium(III) hydrate. *Acta Chem. Scand., Ser. A* 1982, *36*, 186–188.
10. Klein W. Redetermination of the crystal structure of yttrium(III) nitrate(V) pentahydrate, $\text{Y}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, $\text{H}_{10}\text{N}_3\text{O}_{14}\text{Y}$. *Z. Kristallogr. NCS* 2020, *235*, 801–802.