

$0.59(4) \times 10^{-4}$ , final  $\Delta\rho$  excursions  $\leq 3.2 \text{ e } \text{\AA}^{-3}$ . Scattering factors from *International Tables for X-ray Crystallography* (1974). Programs used: see Lundgren (1982).

Atomic coordinates and equivalent isotropic temperature factors are given in Table 1.\* Selected bond distances, angles and torsion angles are presented in Table 2. The building elements of the structure, infinite double chains, are shown in Fig. 1.

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43102 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Related literature.** The crystal structures of  $\text{HgCl}_2 \cdot \text{C}_4\text{H}_8\text{S}$  (Brändén, 1964),  $\text{HgCl}_2 \cdot 2\text{C}_4\text{H}_8\text{S}$  and  $\text{HgBr}_2 \cdot 2\text{C}_4\text{H}_8\text{S}$  (Sandström & Persson, in preparation).

We thank Dipl. chem. Klaus Dreisch for synthesizing tetrahydroselenophene.

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## Tetraaquatrinitratoeuropium(III) Monohydrate

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**Abstract.**  $[\text{Eu}(\text{NO}_3)_3(\text{H}_2\text{O})_4] \cdot \text{H}_2\text{O}$ ,  $M_r = 428.05$ , triclinic,  $P\bar{1}$ ,  $a = 10.638(3)$ ,  $b = 9.568(3)$ ,  $c = 6.704(2) \text{ \AA}$ ,  $\alpha = 76.12(3)$ ,  $\beta = 84.68(2)$ ,  $\gamma = 63.72(2)^\circ$ ,  $V = 595.1(4) \text{ \AA}^3$ ,  $Z = 2$ ,  $D_x = 2.388 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$ ,  $\mu = 5.35 \text{ mm}^{-1}$ ,  $F(000) = 412$ ,  $T = 293 \text{ K}$ , final  $R = 0.079$  for 2521 observed reflections. Three bidentate nitrates and four coordinated water molecules produce a ten-coordinated Eu atom. The coordination polyhedron around Eu approximates a bicapped square antiprism. The crystal structure is built up by the H-bridge-bonded layers of  $[\text{Eu}(\text{NO}_3)_3(\text{H}_2\text{O})_4]$  clusters. Between these layers there are the extra water molecules. The structure is isomorphous with its yttrium analogue [Eriksson (1982). *Acta Chem. Scand. Ser. A*, **36**, 186–188].

**Experimental.** A crystal (Merck, art. 12461) approximating a sphere of diameter 0.2 mm sealed in Lindemann-glass capillary. Philips PW 1100 diffractometer, graphite-monochromated Mo  $K\alpha$  radiation. Accurate cell constants by least-squares fit for 21 well-distributed general reflections in  $\theta$  range 9–31°. Data collected by  $\theta$ - $2\theta$  scan method, 3433 reflections,

$0.0768 \leq \sin\theta \leq 0.7033$ ,  $h - 12 \rightarrow 12$ ,  $k - 13 \rightarrow 13$ ,  $l 0 \rightarrow 9$ , 2521 reflections taken as observed with  $I > 10\sigma(I)$ . Three check reflections every 2 h, max. and min. correction factors 1.063 and 0.988. Data were corrected for Lorentz and polarization effects and for a small spherical absorption effect ( $\mu R = 0.53$ ). Structure solved by Patterson and successive structure-factor and Fourier calculations. Full-matrix refinement.  $\sum w(\Delta F)^2$  minimized 18 heavy atoms (164 parameters). Final  $R = 0.079$ ,  $wR = 0.094$ . The large  $R$  value is the consequence of the poor quality of the crystal and the difference of the real shape of the crystal from the ideal sphere.  $S = 2.11$ ,  $w = [\sigma^2(F_o) + (2 \times 10^{-4})(F_o)^2]^{-1}$ . Extinction coefficient  $4.84 \times 10^{-6}$ . No H positions could be located. The final difference map revealed four peaks around the Eu atom forming an irregular rectangle at an average distance of 0.79(3) Å with a mean electron density of 4(1)  $\text{e } \text{\AA}^{-3}$ ,  $(\Delta/\sigma)_{\text{max}} = 0.27$ . Scattering factors from *International Tables for X-ray Crystallography* (1962). Enraf–Nonius SDP with local modifications adapted to a PDP 11/34 minicomputer (64K). Final coordinates are given in Table 1. Interatomic distances and selected bond angles are reported

in Table 2.\* Fig. 1 shows the O coordination around Eu and Fig. 2 a view along the *a* axis.

**Related literature.** The rare-earth nitrates usually exist as hexahydrates,  $\text{Ln}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  ( $\text{Ln} = \text{Y}, \text{La}, \text{Ce}, \text{Pr}, \text{Nd}$ ). X-ray studies have established that in lanthanum

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43050 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates and mean temperature factors for the non-H atoms with *e.s.d.*'s in parentheses

$B_{\text{eq}} = \frac{1}{3} \text{trace}(\mathbf{BG})$  where  $\mathbf{G}$  is the direct metric tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}(\text{\AA}^2)$
Eu	0.2053 (0)	0.1508 (1)	0.2534 (1)	1.34 (3)
N(1)	-0.0558 (9)	0.2167 (11)	0.0311 (16)	1.7 (6)
N(2)	0.3742 (11)	0.2881 (12)	-0.0217 (18)	2.2 (6)
N(3)	0.3560 (11)	-0.1881 (13)	0.4741 (19)	2.4 (7)
O(11)	-0.0113 (9)	0.1185 (10)	0.2046 (15)	2.2 (6)
O(12)	0.0213 (9)	0.2837 (10)	-0.0521 (14)	2.3 (6)
O(13)	-0.1638 (9)	0.2449 (12)	-0.0514 (18)	2.9 (7)
O(21)	0.2430 (10)	0.3627 (12)	-0.0085 (18)	3.2 (8)
O(22)	0.4316 (9)	0.1487 (10)	0.0889 (16)	2.5 (7)
O(23)	0.4423 (11)	0.3481 (14)	-0.1395 (22)	4.0 (9)
O(31)	0.2381 (9)	-0.1381 (10)	0.3951 (15)	2.1 (6)
O(32)	0.4071 (9)	-0.0834 (9)	0.4397 (16)	2.4 (6)
O(33)	0.4186 (12)	-0.3167 (12)	0.5801 (21)	4.4 (9)
O(1)	0.2794 (9)	-0.0067 (10)	-0.0192 (15)	2.1 (6)
O(2)	0.3056 (8)	0.2408 (10)	0.4738 (16)	2.4 (6)
O(3)	0.1054 (9)	0.1140 (10)	0.5934 (13)	1.7 (6)
O(4)	0.0286 (9)	0.4076 (10)	0.3014 (15)	2.6 (6)
O(5)	0.2193 (10)	0.5486 (11)	0.5002 (21)	3.2 (8)

and cerium compounds the central ions are 11-coordinated (Eriksson, Larsson, Niinistö & Valkonen, 1980; Milinski, Ribár & Satařić, 1980), whereas in the remaining lanthanoid and yttrium structures there is one water molecule less in the first coordination sphere resulting in 10-coordination (Fuller & Jacobson, 1976; Rogers, Taylor & Toogood, 1983; Ribár, Milinski, Budovalčev & Krstanović, 1980). A series of pentahydrates also exist, of which the structure of  $\text{Y}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  is known so far (Eriksson, 1982).

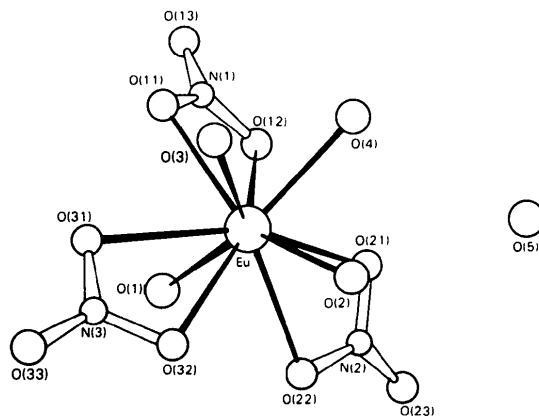


Fig. 1. O coordination around Eu forming a bicapped square antiprism.

Table 2. Bond lengths ( $\text{\AA}$ ) and selected bond angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

Eu—O(11)	2.52 (1)	N(1)—O(11)	1.28 (2)
Eu—O(12)	2.63 (1)	N(1)—O(12)	1.27 (2)
Eu—O(21)	2.50 (1)	N(1)—O(13)	1.21 (2)
Eu—O(22)	2.55 (1)	N(2)—O(21)	1.26 (2)
Eu—O(31)	2.58 (1)	N(2)—O(22)	1.26 (2)
Eu—O(32)	2.47 (1)	N(2)—O(23)	1.23 (2)
Eu—O(1)	2.50 (1)	N(3)—O(31)	1.25 (2)
Eu—O(2)	2.40 (1)	N(3)—O(32)	1.31 (2)
Eu—O(3)	2.44 (1)	N(3)—O(33)	1.19 (2)
Eu—O(4)	2.42 (1)		
O(11)—Eu—O(12)	49.5 (6)	O(31)—Eu—O(1)	68.1 (6)
O(11)—Eu—O(31)	67.8 (6)	O(31)—Eu—O(3)	67.5 (6)
O(11)—Eu—O(1)	78.0 (7)	O(32)—Eu—O(1)	82.2 (7)
O(11)—Eu—O(3)	73.7 (7)	O(32)—Eu—O(2)	72.0 (7)
O(11)—Eu—O(4)	80.7 (7)	O(32)—Eu—O(3)	79.7 (7)
O(12)—Eu—O(21)	67.0 (7)	O(2)—Eu—O(3)	72.3 (7)
O(12)—Eu—O(1)	68.6 (6)	O(2)—Eu—O(4)	72.5 (7)
O(12)—Eu—O(4)	70.3 (7)	O(3)—Eu—O(4)	71.4 (7)
O(21)—Eu—O(22)	50.4 (7)	O(11)—N(1)—O(12)	116 (2)
O(21)—Eu—O(1)	85.9 (7)	O(11)—N(1)—O(13)	123 (2)
O(21)—Eu—O(2)	81.1 (7)	O(12)—N(1)—O(13)	121 (2)
O(21)—Eu—O(4)	72.1 (7)	O(21)—N(2)—O(22)	117 (2)
O(22)—Eu—O(32)	70.8 (7)	O(21)—N(2)—O(23)	121 (3)
O(22)—Eu—O(1)	69.3 (7)	O(22)—N(2)—O(23)	122 (3)
O(22)—Eu—O(2)	72.8 (7)	O(31)—N(3)—O(32)	115 (2)
O(31)—Eu—O(32)	50.3 (6)	O(31)—N(3)—O(33)	125 (3)
		O(32)—N(3)—O(33)	120 (3)

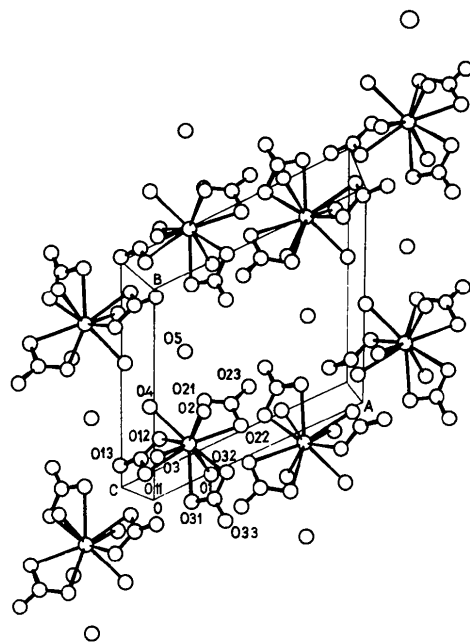


Fig. 2. Infinite layers of the H-bridge-bonded  $[\text{Eu}(\text{NO}_3)_3(\text{H}_2\text{O})_4]$  clusters separated by non-coordinated water molecules along the crystallographic *a* axis.

The authors express their thanks to Merck (Darmstadt) for supplying the crystals.

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## Structures of (+)-8,10- and (+)-9,10-Dibromocamphor\*

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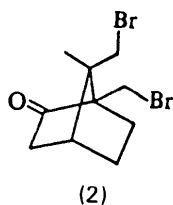
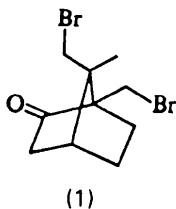
**Abstract.** C<sub>10</sub>H<sub>14</sub>Br<sub>2</sub>O, *M<sub>r</sub>* = 310.03, Mo K $\alpha$ ,  $\lambda_{\alpha_1}$  = 0.70930 Å, *T* = 295 K; 8,10 isomer (1): monoclinic, *P*2<sub>1</sub>, *a* = 7.438 (1), *b* = 7.293 (1), *c* = 10.177 (1) Å,  $\beta$  = 95.689 (5)°, *V* = 549.3 (1) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.874 (1) g cm<sup>-3</sup>,  $\mu$  = 73 cm<sup>-1</sup>, *F*(000) = 304, *R* = 0.051 for 891 reflections; 9,10 isomer (2): orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 10.607 (1), *b* = 10.610 (1), *c* = 9.8630 (3) Å, *V* = 1109.98 (12) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.855 (1) g cm<sup>-3</sup>,  $\mu$  = 72 cm<sup>-1</sup>, *F*(000) = 608, *R* = 0.047 for 805 reflections. The geometries and dimensions of the camphor ring systems are similar to those in related molecules; the C(1)–C(7)–C(4) bridgehead angles are 94.0 (7) and 92.8 (9)°, and the mean C–Br bond distances are 1.968 (6) and 1.960 (9) Å.

**Experimental.** Colourless crystals, dimensions 0.25 × 0.30 × 0.21 mm for 8,10-dibromo isomer (1) [0.48 × 0.48 × 0.28 mm for 9,10-dibromo isomer (2)], crystal faces {001}, ( $\bar{1}$ 10), (100), (101) [{100}, {011}]; Enraf–Nonius CAD-4F diffractometer; lattice parameters from 25 reflections with  $\theta$  = 20–26° [18–

24°]. Intensities for  $\theta \leq 30^\circ$  [25°]; *hkl*: 0 to 10, 0 to 10, –14 to 14 [0 to 13, 0 to 13, 0 to 12];  $\omega$ –2 $\theta$  scan,  $\omega$  scan width (0.65 + 0.35 tan $\theta$ )° at 1.3–10° min<sup>-1</sup>,

Table 1. Final positional (fractional, × 10<sup>4</sup>, Br × 10<sup>5</sup>) and equivalent isotropic thermal parameters (*U* × 10<sup>3</sup> Å<sup>2</sup>) with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub> *
8,10-Dibromocamphor				
Br(1)	9521 (16)	40000	103038 (9)	60
Br(2)	21598 (17)	36412 (29)	36724 (9)	72
O	2248 (11)	331 (11)	6044 (8)	56
C(1)	2994 (12)	3586 (14)	6515 (8)	33
C(2)	2906 (13)	1523 (16)	6761 (10)	36
C(3)	3833 (15)	1206 (16)	8144 (10)	38
C(4)	4418 (15)	3119 (15)	8571 (10)	85
C(5)	5970 (12)	3707 (19)	7761 (12)	54
C(6)	5018 (12)	3927 (19)	6365 (9)	45
C(7)	2822 (11)	4311 (14)	7928 (8)	29
C(8)	984 (12)	3861 (21)	8383 (7)	43
C(9)	3086 (17)	6387 (18)	8094 (11)	47
C(10)	1629 (14)	4292 (20)	5467 (8)	46
9,10-Dibromocamphor				
Br(1)	40245 (17)	33271 (17)	97060 (15)	68
Br(2)	25380 (20)	29256 (14)	30022 (13)	58
O	590 (9)	1241 (8)	4951 (12)	53
C(1)	2490 (14)	2231 (9)	5858 (10)	34
C(2)	1349 (13)	1337 (12)	5843 (17)	39
C(3)	1390 (13)	623 (14)	7163 (17)	46
C(4)	2564 (16)	1161 (11)	7853 (14)	46
C(5)	3710 (13)	667 (16)	7102 (20)	52
C(6)	3643 (12)	1320 (12)	5727 (16)	38
C(7)	2518 (16)	2574 (11)	7413 (12)	36
C(8)	1360 (13)	3289 (15)	7885 (17)	49
C(9)	3694 (13)	3301 (14)	7748 (13)	40
C(10)	2442 (14)	3362 (12)	4915 (11)	39



\* (1*R*,4*S*,7*R*)- and (1*R*,4*S*,7*S*)-1,7-bis(bromomethyl)-7-methylbicyclo[2.2.1]heptan-2-one.

\* *U*<sub>eq</sub> =  $\frac{1}{3}$  trace of diagonalized *U* tensor.