

# Dihydronium bis(2,6-diaminopyridinium) tris(pyridine-2,6-dicarboxylato- $\kappa^3O,O',N$ )-dysprosate(III) dihydrate

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## Key indicators

Single-crystal X-ray study  
T = 145 K  
Mean  $\sigma(C-C)$  = 0.006 Å  
R factor = 0.041  
wR factor = 0.101  
Data-to-parameter ratio = 17.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The reaction of dysprosium(III) chloride with  $[H_2pyda]^{2+}$ - $[pydc]^{2-}$  (pyda is 2,6-diaminopyridine and  $H_2pydc$  is 2,6-pyridinedicarboxylic acid) in an aqueous solution leads to the formation of the title  $Dy^{III}$  complex,  $(H_5O_2)(C_5H_8N_3)_2 \cdot [Dy(C_7H_3NO_4)_3] \cdot 2H_2O$ . The Dy ion lies on a twofold rotation axis and is nine-coordinated with a distorted tricapped trigonal-prismatic geometry. The dihydronium cation  $(H_5O_2)^+$  is located on a twofold axis, the longer O—H bond distance being 1.225 Å.

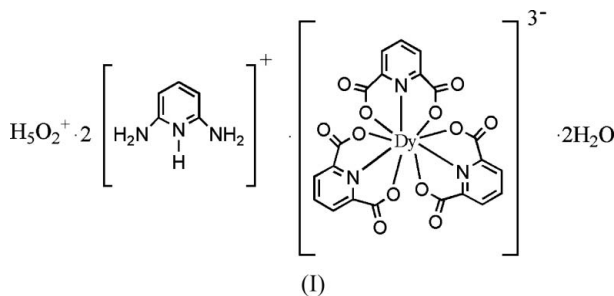
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## Comment

We have recently become interested in metal complexes with a pyridine-containing ligand system (Rafizadeh *et al.*, 2004). We present here the structure of the title compound, (I), which includes a pyridinedicarboxylate (pydc) anion and a diaminopyridinium (pyda) cation.



The structure of (I) comprises  $Dy^{III}$  complex anions, free pyda cations, dihydronium cations  $(H_5O_2)^+$  and uncoordinated water molecules (Fig. 1). The Dy ion lies on a twofold rotation axis and the nine-coordinate  $Dy^{III}$  complex assumes a distorted tricapped trigonal-prismatic geometry, formed by three pydc dianions. This is comparable to the situation found in the Yb analogue (Rafizadeh *et al.*, 2004). The Dy—O and Dy—N bond distances (Table 1) agree with those found in previously reported structures (Fernandes *et al.*, 2001).

Atom H1W atom of the dihydronium cation lies on a twofold axis. The O1W—H1W bond distance of 1.225 Å and O1W—H1W—O1W<sup>ii</sup> bond angle of 170° [symmetry code: (ii)  $-x - \frac{1}{2}, y, -z + \frac{1}{2}$ ] are comparable to the values found in the Yb analogue (1.218 and 1.221 Å and 170°; Rafizadeh *et al.*, 2004) and in the Gd<sup>III</sup> analogue [1.216 Å and 164°; Rafizadeh *et al.*, 2005]. These also agree with the values deduced from *ab initio* computations (1.2 Å and 173.5°; Ojamae *et al.*, 1995).

## Experimental

To a solution of  $[H_2pyda][pydc]$  (0.5 g, 1.8 mmol) in water (100 ml) was added a solution of  $DyCl_3 \cdot 6H_2O$  (0.226 g, 0.6 mmol) in water

(20 ml) and the resulting pale-yellow solution was stirred for 30 min. This solution was left to evaporate slowly at room temperature, affording single crystals of (I) after one week; yield 73.4%, m.p. 536–539 K.

#### Crystal data

(H<sub>5</sub>O<sub>2</sub>)(C<sub>5</sub>H<sub>8</sub>N<sub>3</sub>)<sub>2</sub>-  
[Dy(C<sub>7</sub>H<sub>3</sub>NO<sub>4</sub>)<sub>3</sub>]·2H<sub>2</sub>O  
*M<sub>r</sub>* = 951.17  
Monoclinic, *P*<sub>2</sub>/*n*  
*a* = 9.654 (2) Å  
*b* = 10.579 (2) Å  
*c* = 17.972 (4) Å  
*β* = 94.483 (4)°  
*V* = 1830.0 (7) Å<sup>3</sup>  
*Z* = 2

*D<sub>x</sub>* = 1.726 Mg m<sup>-3</sup>  
Mo *Kα* radiation  
Cell parameters from 1024  
reflections  
*θ* = 3–30°  
*μ* = 2.13 mm<sup>-1</sup>  
*T* = 145 (2) K  
Prism, yellow  
0.30 × 0.25 × 0.20 mm

#### Data collection

Bruker SMART 1000 CCD area-  
detector diffractometer  
*φ* and *ω* scans  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1998)  
*T<sub>min</sub>* = 0.406, *T<sub>max</sub>* = 0.653  
18268 measured reflections

4508 independent reflections  
3876 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.095  
*θ<sub>max</sub>* = 28.5°  
*h* = -12 → 12  
*k* = -13 → 13  
*l* = -23 → 23

#### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.041  
*wR* (*F*<sup>2</sup>) = 0.101  
*S* = 1.03  
4508 reflections  
259 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 1.585P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
(Δ/σ)<sub>max</sub> < 0.001  
Δρ<sub>max</sub> = 2.75 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -1.14 e Å<sup>-3</sup>

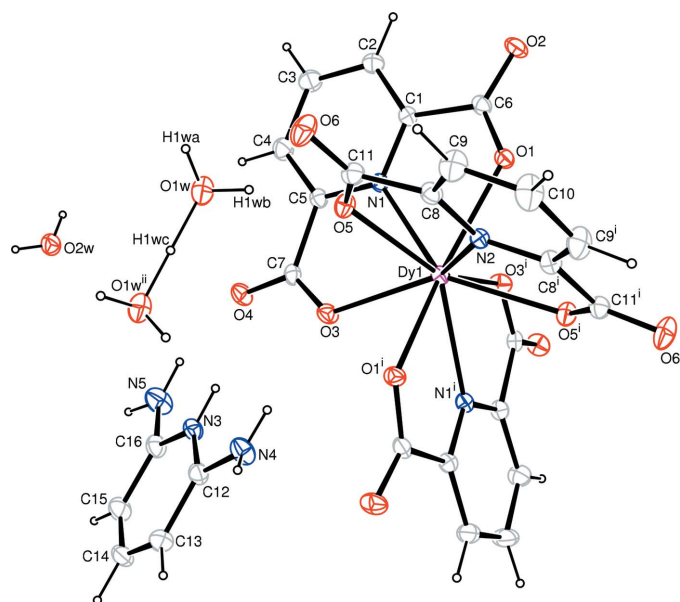
**Table 1**

Selected bond lengths (Å).

Dy1—O1	2.389 (3)	Dy1—N1	2.495 (3)
Dy1—O3	2.424 (3)	Dy1—N2	2.492 (4)
Dy1—O5	2.418 (3)		

H atoms bonded to O and N atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(carrier). Other H atoms were positioned geometrically with C—H = 0.95 Å and refined in a riding-model approximation, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C). The maximum residual electron density peak is 1.12 Å<sup>-1</sup> from Dy1.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTPlus* (Bruker, 1998); data reduction: *SAINTPlus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to



**Figure 1**

The molecular structure of (I), with 30% probability displacement ellipsoids [symmetry codes: (i)  $-x + \frac{1}{2}, y, -z + \frac{1}{2}$ ; (ii)  $-x - \frac{1}{2}, y, -z + \frac{1}{2}$ ].

refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL*.

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#### References

- Bruker (1998). *SMART* (Version 5.059) and *SAINTPlus* (Version 6.01). Bruker AXS Inc., Madison, Wisconsin, USA.
- Fernandes, A., Jaud, J., Dexpert-Ghys, J. & Brouca-Cabarrecq, C. (2001). *Polyhedron*, **20**, 2385–2391.
- Ojamae, L., Shavitt, I. & Singer, S. J. (1995). *Int. J. Quantum Chem*, **29**, 657–660.
- Rafizadeh, M., Ranjbar, M. & Amani, V. (2004). *Acta Cryst. E* **60**, m479–m481.
- Rafizadeh, M., Ranjbar, M. & Amani, V. (2005). *Anal. Sci.* **21**, 113–114.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1998). *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.