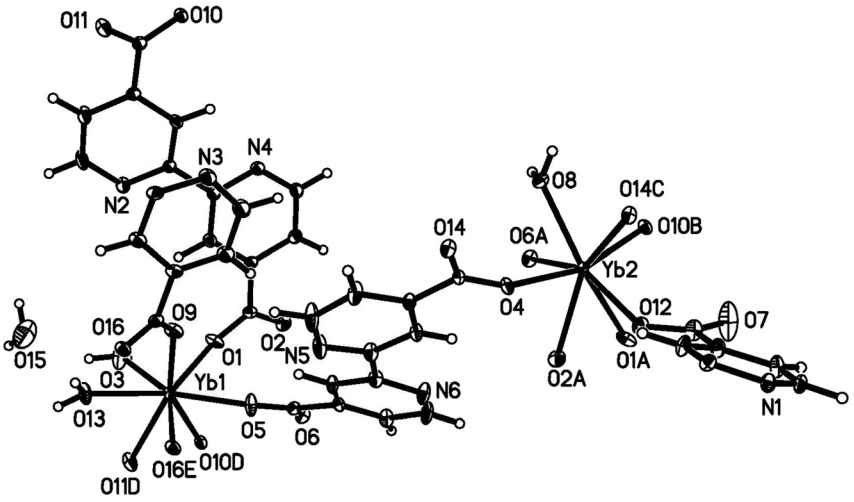
Zheng Cheng, Xiao-Yu Zhang*, Xu Liu and Xiao-rui Yang

Crystal structure of poly[hexaqua-pentakis(μ$_4$-2,2′-bipyridine-4,4′-dicarboxylato-κ^4*O:O′:O″:O‴*)-(μ$_2$-2,2′-bipyridine-4,4′-dicarboxylato-κ^2*O:O*) tetraytterbium(III)] hydrate, $C_{36}H_{26}N_6O_{16}Yb_2$



https://doi.org/10.1515/ncrs-2024-0278
Received July 1, 2024; accepted October 18, 2024;
published online October 31, 2024

Abstract

$C_{36}H_{26}N_6O_{16}Yb_2$, triclinic, $P\bar{1}$ (no. 2), a = 11.3387(3) Å, b = 12.1820(4) Å, c = 14.1433(4) Å, α = 95.090(2)°, β = 103.517(3)°, γ = 101.762(2)°, V = 1840.41(10) Å^3, Z = 2, $R_{gt}(F)$ = 0.0282, $wR_{ref}(F^2)$ = 0.0576, T = 150 K.

CCDC no.: 2366863

A part of 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.

***Corresponding author: Xiao-Yu Zhang**, College of Food and Bioengineering, Henan University of Science and Technology, Luoyang, Henan 471022, P.R. China, E-mail: zhangxiaoyu7909@163.com. https://orcid.org/0000-0001-6078-9611

Zheng Cheng, Xu Liu and Xiao-rui Yang, College of Food and Bioengineering, Henan University of Science and Technology, Luoyang, Henan 471022, P.R. China

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.19 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	5.14 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	28.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	26,819, 7,969, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 6,913
N(param)$_{refined}$:	547
Programs:	CrysAlis^{PRO},[1] Olex2,[2] SHELX[34]

1 Source of materials

A mixture of $Yb(NO_3)_3{\cdot}5H_2O$ (0.0452 g, 0.1 mmol) and 2,2′-bipyridine-4,4′-dicarboxylic acid (0.0366 g, 0.15 mmol) was dissolved in 8 mL of deionized water. The mixture was sealed in a 25 mL Teflon-lined steel autoclave after ultrasound treatment for 15 min and heated at 140 °C for 72 h. The mixture was cooled to room temperature at a rate of 3 °C/h, and colorless block crystals were isolated by filtration, washed with distilled water and dried in air.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Yb1	0.39387 (2)	0.83407 (2)	0.36524 (2)	0.01324 (5)
Yb2	−0.20611 (2)	0.36514 (2)	0.88971 (2)	0.01646 (5)
O1	−0.3433 (3)	0.3780 (2)	0.7482 (2)	0.0297 (7)
O2	−0.3998 (3)	0.3477 (2)	0.5874 (2)	0.0280 (7)
O3	−0.3981 (3)	0.3573 (3)	0.9160 (2)	0.0500 (10)
H3A	−0.407438	0.360905	0.974050	0.075*
H3B	−0.460853	0.307063	0.882111	0.075*
O4	0.4402 (2)	0.8629 (2)	0.52661 (19)	0.0212 (6)
O5	−0.1004 (3)	0.3635 (3)	0.7723 (2)	0.0306 (7)
O6	−0.1967 (2)	0.2439 (2)	0.6343 (2)	0.0217 (6)
O7	0.5991 (3)	0.7861 (4)	0.2037 (3)	0.0704 (14)
O8	0.2921 (2)	0.9808 (2)	0.3956 (2)	0.0222 (6)
H8A	0.213803	0.956274	0.372306	0.033*
H8B	0.305293	1.046184	0.382026	0.033*
O9	−0.1422 (3)	0.5525 (2)	0.8976 (2)	0.0289 (7)
O10	−0.2969 (2)	1.1659 (2)	0.80136 (19)	0.0201 (6)
O11	−0.2384 (3)	1.1979 (2)	0.9623 (2)	0.0314 (7)
O12	0.5827 (2)	0.8078 (2)	0.3563 (2)	0.0259 (7)
O13	−0.1737 (3)	0.4302 (3)	1.0591 (2)	0.0321 (7)
H13A	−0.125488	0.491814	1.090910	0.048*
H13B	−0.200348	0.395944	1.102569	0.048*
O14	0.4892 (2)	0.9989 (2)	0.6544 (2)	0.0217 (6)
O16	−0.0015 (2)	0.6250 (2)	1.0399 (2)	0.0238 (7)
N1	1.0404 (3)	0.9518 (3)	0.3897 (2)	0.0215 (8)
N2	−0.3752 (4)	0.7737 (3)	0.8986 (3)	0.0327 (9)
N3	−0.0002 (4)	0.9642 (3)	0.8731 (3)	0.0337 (9)
N4	−0.3424 (3)	0.7689 (3)	0.6514 (2)	0.0224 (8)
N5	0.2319 (3)	0.6854 (3)	0.7716 (3)	0.0345 (10)
N6	0.1716 (3)	0.4843 (3)	0.5539 (3)	0.0316 (9)
C1	−0.2823 (4)	1.1295 (3)	0.8844 (3)	0.0201 (9)
C2	−0.0168 (5)	0.8719 (4)	0.8087 (4)	0.0412 (13)
H2	−0.011184	0.881718	0.743844	0.049*
C3	0.8388 (4)	0.9229 (4)	0.4214 (3)	0.0250 (10)
H3	0.788922	0.933519	0.465330	0.030*
C4	−0.3690 (3)	0.4139 (3)	0.6663 (3)	0.0197 (9)
C5	−0.3486 (4)	0.6952 (4)	0.5742 (3)	0.0247 (10)
H5	−0.345743	0.723275	0.514028	0.030*
C6	−0.1112 (3)	0.3245 (3)	0.6851 (3)	0.0184 (9)
C7	−0.3589 (4)	0.5803 (3)	0.5756 (3)	0.0228 (9)
H7	−0.362887	0.531481	0.518112	0.027*
C8	0.4348 (3)	0.9008 (3)	0.6105 (3)	0.0148 (8)
C9	−0.3451 (4)	0.8125 (3)	0.8192 (3)	0.0211 (9)
C10	−0.0644 (4)	0.6331 (3)	0.9560 (3)	0.0209 (9)
C11	−0.3499 (4)	0.7266 (3)	0.7352 (3)	0.0201 (9)
C12	−0.0464 (4)	0.7471 (3)	0.9246 (3)	0.0213 (9)
C13	0.9671 (4)	0.9676 (3)	0.4507 (3)	0.0214 (9)
C14	−0.0120 (3)	0.3773 (3)	0.6378 (3)	0.0171 (9)
C15	0.3568 (4)	0.8646 (4)	0.7585 (3)	0.0312 (11)
H15	0.396871	0.940439	0.787395	0.037*
C16	−0.3632 (3)	0.5380 (3)	0.6629 (3)	0.0184 (9)
C17	0.0952 (4)	0.3833 (4)	0.5130 (3)	0.0341 (12)
H17	0.106008	0.347787	0.454043	0.041*
C18	−0.3146 (4)	1.0059 (3)	0.8899 (3)	0.0220 (9)
C19	0.0019 (4)	0.3277 (4)	0.5512 (3)	0.0243 (10)
H19	−0.051118	0.257218	0.518428	0.029*
C20	−0.0415 (4)	0.7635 (4)	0.8310 (3)	0.0313 (11)
H20	−0.055159	0.700516	0.781866	0.038*
C21	0.2925 (5)	0.7922 (4)	0.8090 (4)	0.0445 (14)
H21	0.291247	0.819810	0.873665	0.053*
C22	0.6434 (4)	0.8145 (4)	0.2926 (3)	0.0298 (11)
C23	−0.0086 (4)	0.9477 (3)	0.9647 (3)	0.0232 (10)
C24	0.3621 (3)	0.8248 (3)	0.6651 (3)	0.0198 (9)
C25	0.1550 (4)	0.5325 (3)	0.6371 (3)	0.0238 (10)
C26	0.2997 (3)	0.7140 (3)	0.6249 (3)	0.0196 (9)
H26	0.302121	0.683691	0.561295	0.024*
C27	0.0657 (4)	0.4815 (3)	0.6815 (3)	0.0222 (9)
H27	0.057700	0.517563	0.741300	0.027*
C28	−0.0294 (4)	0.8422 (3)	0.9930 (3)	0.0257 (10)
H28	−0.032131	0.834101	1.058708	0.031*
C29	0.8589 (4)	0.8491 (4)	0.2653 (3)	0.0269 (10)
H29	0.824196	0.809806	0.200431	0.032*
C30	−0.3593 (3)	0.6132 (3)	0.7434 (3)	0.0214 (9)
H30	−0.363202	0.586950	0.804156	0.026*
C31	0.9860 (4)	0.8942 (4)	0.3004 (3)	0.0244 (10)
H31	1.037667	0.883407	0.257989	0.029*
C32	−0.3136 (4)	0.9278 (3)	0.8138 (3)	0.0211 (9)
H32	−0.291314	0.952754	0.757207	0.025*
C33	−0.3721 (5)	0.8510 (4)	0.9732 (3)	0.0432 (13)
H33	−0.391467	0.824542	1.030194	0.052*
C34	0.7838 (4)	0.8632 (4)	0.3285 (3)	0.0236 (10)
C35	−0.3431 (5)	0.9651 (4)	0.9726 (3)	0.0380 (12)
H35	−0.342295	1.016006	1.027856	0.046*
C36	0.2339 (4)	0.6487 (4)	0.6796 (3)	0.0242 (10)
O15	−0.4598 (5)	0.4209 (4)	1.0785 (4)	0.0941 (17)
H15A	−0.510080	0.370121	1.096450	0.141*
H15B	−0.495080	0.476151	1.074100	0.141*

2 Experimental details

CrysAlisPro 1.171.39.46 (Rigaku Oxford Diffraction, 2018).[1] Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Using Olex2,[2] the structure was solved with the ShelXT[3] structure solution program and refined with the ShelXL[4] refinement package.

The carbon bound hydrogen atoms were placed in calculated positions and refined using a riding model on attached atoms.

3 Comment

In recent years, ytterbium(III) metal-organic frameworks (Yb–MOFs) have attracted considerable attentions due to their structural diversity and potential applications in various fields such as adsorption, catalysis, detection,

degradation, etc.[5–8]. Nitrogen-containing carboxylic acids are widely used as ligands in the synthesis of Yb–MOFs.[5,8,9] Among these ligands, 2,2′-bipyridine-4,4′-dicarboxylic acid (H_2BPDC) has garnered considerable interest due to its potential as a bridging ligand and abundant coordination modes.[10] In this study, a new Yb–MOF was constructed using H_2BPDC as ligand and water as solvent.

The asymmetric unit contains two Yb(III) ions, three deprotonated $BPDC^{2-}$ ligands (with two different coordination modes), three coordinated water molecules, and one lattice water (see figure). Yb1 and Yb2 are both 8-coordinated. Yb1 coordinates with six oxygen atoms from five deprotonated $BPDC^{2-}$ ligands (O1; O5; O9; O10D, O11D; O16E; D: $x, -1 + y, z$; *E*: $-x, 1 - y, 2 - z$), and two oxygen atoms from coordinated water molecules (O3, O13). Yb2 coordinates with seven oxygen atoms from six deprotonated $BPDC^{2-}$ ligands (O1A, O2A; O4; O6A; O10B; O12; O14C; A: $-x, 1 - y, 1 - z$; *B*: $-x, 2 - y, 1 - z$; *C*: $1 - x, 2 - y, 1 - z$), and one oxygen atom from a water molecule (O8). The bond distances of Yb–O range from 2.198(3) to 2.795(3) Å, which are in agreement with distances found in similar Yb compound.[8,9] Yb1 and Yb2 are linked by a bridging $BPDC^{2-}$ ligand, and the title compound was connected into a three-dimensional MOF structure through alternating bridging ligands and Yb(III) centers.[11] The crystal structure of the title compound is further stablized by multiple hydrogen bonds.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the National Natural Science Foundation of China (No. 22371110), the Program for Science & Technology Innovation Research Team in Universities of Henan Province, China (No. 21IRTSTHN004).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Oxford Diffraction Ltd, CrysAlis^{PRO}, Rigaku Oxford Diffraction, Version 1.171.39.6a, England, 2018.
2. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. OLEX2: A Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Cryst.* **2009**, *42*, 339–341.
3. Sheldrick, G. M. *SHELXT* – Integrated Space-Group and Crystal-Structure Determination. *Acta Crystallogr.* **2015**, *A71*, 3–8.
4. Sheldrick, G. M. Crystal Structure Refinement with *SHELXL*. *Acta Crystallogr.* **2015**, *C71*, 3–8.
5. Fan, W.; Cheng, Y.; Zhao, H.; Yang, S.; Wang, L.; Zheng, L.; Cao, Q. e.; Fan, W.; Cheng, Y.; Zhao, H.; Yang, S.; Wang, L.; Zheng, L.; Cao, Q. A Turn-On NIR Fluorescence Sensor for Gossypol Based on Yb-Based Metal-Organic Framework. *Talanta* **2022**, *238*, 123030.
6. Xie, Y.; Sun, G.; Mandl, G. A.; Maurizio, S. L.; Chen, J.; Capobianco, J. A.; Sun, L. Upconversion Luminescence through Cooperative and Energy–Transfer Mechanisms in Yb^{3+}–Metal–Organic Frameworks. *Angew. Chem.* **2023**, *135*, e202216269.
7. Li, Q.-L.; Wang, X.; Chen, X.-F.; Wang, M.-L.; Zhao, R.-S. In Situ Hydrothermal Growth of Ytterbium-Based Metal–Organic Framework on Stainless Steel Wire for Solid-phase Microextraction of Polycyclic Aromatic Hydrocarbons from Environmental Samples. *J. Chromatogr. A* **2015**, *1415*, 11–19.
8. Hashemzadeh, A.; Amini, M. M.; Khavasi, H. R.; Ng, S. W. Ligand Preferences in Ytterbium Ions Complexation with Carboxylate-Based Metal-Organic Frameworks. *J. Coord. Chem.* **2017**, *70*, 3217–3232.
9. Dang, S.; Min, X.; Yang, W.; Yi, F. Y.; You, H.; Sun, Z. M. Lanthanide Metal–Organic Frameworks Showing Luminescence in the Visible and Near–Infrared Regions with Potential for Acetone Sensing. *Chem. Eur. J.* **2013**, *19*, 17172–17179.
10. Vitiu, B. A.; Croitor, L.; Siminel, A.; Koropchanu, E.; Bourosh, P. A Novel 2D Zinc (II) Coordination Polymer Based on 2, 2′-Bipyridine-4, 4′-dicarboxylic Acid: Synthesis, Crystal Structure and Photoluminescence Property. *Chem. J. Mold.* **2018**, *13*, 30–35.
11. Fan, K.; Bao, S.-S.; Huo, R.; Huang, X.-D.; Liu, Y.-J.; Yu, Z.-W.; Kurmoo, M.; Zheng, L.-M. Luminescent Ir(iii)–Ln(iii) Coordination Polymers Showing Slow Magnetization Relaxation. *Inorg. Chem. Front.* **2020**, *7*, 4580–4592.