Shi-Jun Chen, Wei Tian, Yan-Ni Hui and Mei Zhu*

Crystal structure of 4,4'-di(1*H*-imidazol-1-yl)-1,1'-biphenyl-1-ium 5,3',5'-tricarboxy-[1,10-biphenyl]-2-carboxylate, C₂₅H₁₇N₂O₈

<https://doi.org/10.1515/ncrs-2020-0237>

Received May 17, 2020; accepted May 28, 2020; available online June 18, 2020

Abstract

C₂₅H₁₇N₂O₈, triclinic, *P* $\bar{1}$ (no. 2), $a = 9.6002(5)$ Å, $b = 10.4192(5)$ Å, $c = 11.0226(5)$ Å, $\alpha = 93.412(4)^\circ$, $\beta = 98.094(4)^\circ$, $\gamma = 98.787(4)^\circ$, $V = 1075.04(9)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0547$, $wR_{\text{ref}}(F^2) = 0.1426$, $T = 293(2)$ K.

CCDC no.: 2006273

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.43 × 0.35 × 0.29 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	xcalibur, ω
θ_{max} , completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11742, 3995, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3271
$N(\text{param})_{\text{refined}}$:	323
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

*Corresponding author: Mei Zhu, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, Henan 471934, P.R. China, e-mail: zhumei471022@126.com. <https://orcid.org/0000-0002-7525-1934>

Shi-Jun Chen: College of Chemistry and Chemical Engineering, Xi'an SHIYOU University, Xi'an 710065, Shaanxi Province, P.R. China

Wei Tian: Oil and Gas Technology Research Institute of Changqing Oilfield Company, CNPC, Xi'an Shaanxi 710021, China

Yan-Ni Hui: The Seventh Oil Production Plant, Changqing Oilfield Co. of Petrochina, Xi'an 710200, Shaxi Province, P.R. China

Source of material

A mixture of 4,4'-di(1*H*-imidazol-1-yl)-1,1'-biphenyl (0.1 mmol, 0.052 g) and biphenyl-2,5,3',5'-tetracarboxylic acid (H4btc) (0.1 mmol, 0.0330 g), was dissolved in 10 mL H₂O. Then the solution was heated in a 23 mL Teflon-lined autoclave under

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
N1	0.5311(2)	0.7217(2)	0.88468(19)	0.0356(5)
N2	0.6703(2)	0.84588(18)	0.78875(17)	0.0311(5)
O1	0.4383(2)	0.72017(17)	0.4025(2)	0.0593(6)
H1	0.4855	0.6990	0.3504	0.089*
O2	0.3716(2)	0.50632(17)	0.40205(17)	0.0508(5)
O3	0.3221(3)	1.02544(17)	0.69027(19)	0.0579(6)
H3	0.3226	1.0991	0.7213	0.087*
O4	0.2026(2)	0.96708(17)	0.84229(18)	0.0568(6)
O5	−0.28987(18)	0.46306(17)	0.87376(18)	0.0429(5)
H5	−0.3753	0.4507	0.8786	0.064*
O6	−0.30274(19)	0.2621(2)	0.9322(2)	0.0588(6)
O7	0.43473(17)	0.46186(15)	0.86832(16)	0.0366(4)
O8	0.37580(18)	0.27656(15)	0.75173(17)	0.0398(5)
C1	0.5525(3)	0.8422(2)	0.9452(2)	0.0409(6)
H1B	0.5139	0.8657	1.0144	0.049*
C2	0.6395(3)	0.9206(2)	0.8865(2)	0.0404(6)
H2	0.6729	1.0089	0.9075	0.049*
C3	0.6030(3)	0.7245(2)	0.7912(2)	0.0347(6)
H3A	0.6062	0.6540	0.7364	0.042*
C4	0.7634(3)	0.8896(2)	0.7034(2)	0.0322(5)
C5	0.8051(4)	1.0195(3)	0.6969(3)	0.0647(10)
H5A	0.7702	1.0797	0.7453	0.078*
C6	0.8988(4)	1.0615(3)	0.6187(3)	0.0662(10)
H6	0.9286	1.1506	0.6172	0.079*
C7	0.9500(3)	0.9770(2)	0.5426(2)	0.0343(6)
C8	0.9028(4)	0.8475(3)	0.5493(3)	0.0728(12)
H8	0.9333	0.7869	0.4979	0.087*
C9	0.8115(4)	0.8031(3)	0.6295(4)	0.0741(12)
H9	0.7831	0.7141	0.6328	0.089*
C10	0.3752(3)	0.6153(2)	0.4454(2)	0.0338(6)
C11	0.3079(2)	0.6473(2)	0.5554(2)	0.0291(5)
C12	0.2445(2)	0.5468(2)	0.6162(2)	0.0294(5)
H12	0.2435	0.4609	0.5875	0.035*
C13	0.1822(2)	0.5729(2)	0.7201(2)	0.0265(5)
C14	0.1836(2)	0.7019(2)	0.7605(2)	0.0291(5)
H14	0.1401	0.7208	0.8282	0.035*
C15	0.2489(2)	0.8031(2)	0.7013(2)	0.0291(5)
C16	0.3115(3)	0.7759(2)	0.5990(2)	0.0306(5)
H16	0.3560	0.8435	0.5595	0.037*
C17	0.1167(2)	0.4656(2)	0.7881(2)	0.0261(5)
C18	0.1911(2)	0.3673(2)	0.8296(2)	0.0255(5)
C19	0.1263(2)	0.2683(2)	0.8915(2)	0.0327(5)
H19	0.1758	0.2023	0.9174	0.039*
C20	−0.0115(3)	0.2673(2)	0.9150(2)	0.0338(6)
H20	−0.0539	0.2016	0.9578	0.041*
C21	−0.0863(2)	0.3636(2)	0.8752(2)	0.0296(5)
C22	−0.0225(2)	0.4623(2)	0.8119(2)	0.0294(5)
H22	−0.0734	0.5270	0.7851	0.035*
C23	0.3451(2)	0.3691(2)	0.8145(2)	0.0254(5)
C24	−0.2367(2)	0.3569(2)	0.8973(2)	0.0329(5)
C25	0.2537(3)	0.9395(2)	0.7527(2)	0.0360(6)
H1A	0.480(3)	0.643(3)	0.902(3)	0.066(10)*

autogenous pressure at 403 K for 4 days. After cooling to room temperature crystals formed.

Experimental details

Coordinates of hydrogen atoms were positioned using geometric constraints. Their *U*_{iso} values were set to 1.2*U*_{eq} of the parent atoms.

Comment

In the construction of CPs, multidentate organic ligands play a vital role since the structural integrity of these ligands in most cases remains unaltered throughout the mixed-ligands strategy incorporating N-donor colinkers. In most situations, N-containing ligands are electrically neutral, while carboxylate ligands are negatively charged. Among organic aromatic multicarboxylates like the biphenyl-2,5,3',5'-tetracarboxylate anion can yield predetermined networks and have been widely utilized to construct MOFs [4–9]. On the other hand, 4,4'-di(1*H*-imidazol-1-yl)-1,1'-biphenyl serves as bridging ligand to coordinate metal ions [10, 11].

In the title complex, there are one biphenyl-2,5,3',5'-tetracarboxyl anion and one monoprotonated 4,4'-di(1*H*-imidazol-1-yl)-1,1'-biphenyl in the asymmetric unit (see the figure).

References

- Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.35.15. Agilent Technologies UK Ltd, Oxford, UK (2011).
- Sheldrick, G. M.: SHELXT – Integrated space-group and crystal-structure determination. *Acta Crystallogr. A* **71** (2015) 3–8.
- Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr. C* **71** (2015) 3–8.
- Yao, L. P.; Zhao, Y.; Zhang, R.: Crystal structure of aqua (5, 5'-dicarboxy-(1,10-biphenyl)-2,30-dicarboxylato-κO) bis(1,10-phenanthroline-κ²N,N')cadmium monohydrate. *Z. Kristallogr. NCS* **231** (2016) 1017–1019.
- Chang, X. H.; Zhao, Y.; Han, M. L.; Ma, L.-F.; Wang, L. Y.: Five Cd(II) coordination polymers based on 2,3',5,5'-biphenyltetracarboxylic acid and N-donor coligands: syntheses, structures and fluorescent properties. *CrystEngComm* **16** (2014) 6417–6424.
- Li, S. H.; Han, M. L.; Liu, G. Z.; Ma, L. F.; Wang, L. Y.: Guest-induced single-crystal-to-single-crystal transformations of a new 4-connected 3D cadmium(II) metal-organic framework. *RSC Adv.* **5** (2015) 17588–17591.
- Yao, L. P.; Zhao, Y.: Crystal structure of 4,4'-bipyridin-1-ium 3,3', 5'-tricarboxy-[1,10-biphenyl]-2-carboxylate. *Z. Kristallogr. NCS* **231** (2016) 153–155.
- Li, S. H.; Zang, J.; Guo, J. B.: Synthesis and crystal structure of a new three-dimensional Ho(III) coordination polymer based on 5'-(4-carboxyphenyl)-[1,1':3',1''-terphenyl]-4, 4''-dicarboxylic acid. *Inorg. Nano-Met. Chem.* **12** (2017) 1746–1749.

9. Li, S. H.; Zhao, Y.: Synthesis and crystal structure of a new three-dimensional Co(II) coordination polymer based on 3,3', 4,5'-biphenyl tetracarboxylic acid and 4,4'-bipyridine. *Inorg. Nano-Met. Chem.* **47** (2017) 256–259.
10. Chen, S. J.; Li, S. H.; Wang, W. H.; Long, Y. F.; Zhang, W. L.; Wang, T.; Xu, Y. L.: Crystal structure of poly[aqua- μ_2 -4,4'-bis (pyrid-4-yl)biphenyl- $\kappa^2 N:N'$]- μ_4 -4'-methyl-[1,1'-biphenyl]-3, 5-dicarboxylato- $\kappa^4 O:O':O'':O'''$) manganese(II)]. *Z. Kristallogr. NCS* **234** (2019) 373–375.
11. Song, W.; Li, S. H.; Xu, X. L.: Crystal structure of 4-(4'-(pyridin-4-yl)-[1,1'-biphenyl]-4-yl)pyridin-1-ium *catena*-poly[{5-carboxy-4'-methyl-[1,1'-biphenyl]-3-carboxylato- $\kappa^2 O,O'$ -(κ_2 -4'-methyl-[1,1'-biphenyl]-3,5-dicarboxylato- $\kappa^4 O:O':O'':O'''$)lead(II)]. *Z. Kristallogr. NCS* **233** (2018) 51–53.