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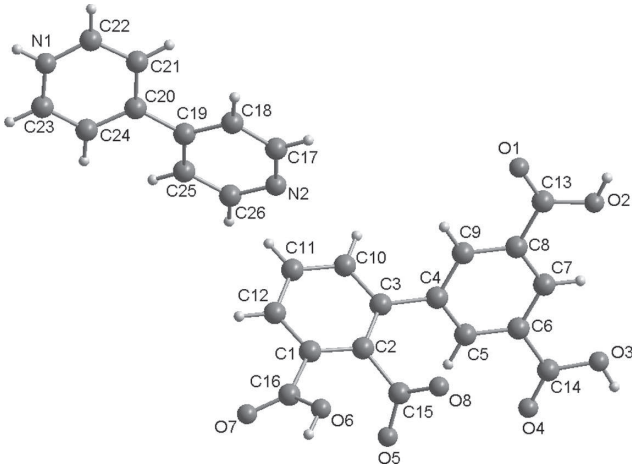
Li-Ping Yao and Yan Zhao*

Crystal structure of 4,4′-bipyridin-1-ium 3,3′,5′-tricarboxy-[1,1′-biphenyl]-2-carboxylate, ($C_{26}H_{18}N_2O_8$)



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Abstract

$C_{26}H_{18}N_2O_8$, triclinic, $P\bar{1}$ (No. 2), $a = 10.101(5)$ Å, $b = 10.589(6)$ Å, $c = 10.705(6)$ Å, $\alpha = 93.183(8)°$, $\beta = 108.614(8)°$, $\gamma = 97.323(8)°$, $V = 1070.5(10)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0399$, $wR_{ref}(F^2) = 0.1027$, $T = 296(2)$ K.

CCDC no.: 1434967

Source of material

A mixture of biphenyl-2,3,3′,5′-tetracarboxylic acid (0.1 mmol, 33.0 g), 4,4′-bipyridine (0.1 mmol, 31.2 mg), KOH (0.1 mmol, 5.6 mg) and H_2O (8 mL) were placed in a Teflon lined stainless steel vessel, heated to 413 K for four days, and then cooled to room temperature over 24 h. Colourless block-shaped crystals of the title compound were obtained.

***Corresponding author: Yan Zhao,** College of Physics and Electronic Information, Luoyang Normal University, Luoyang 471022, Henan Province, P. R. China, e-mail: zhumei471022@126.com
Li-Ping Yao: College of Physics and Electronic Information, Luoyang Normal University, Luoyang 471022, Henan Province, P. R. China

Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.28×0.35×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.14 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω scans
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5700, 3924
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3396
$N(param)_{refined}$:	328
Programs:	SHELXS-97 (SHELDRICK, 1990)

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.1774	1.0429	−0.1471	0.074
H(2)	2*i*	0.0639	0.7031	1.1961	0.067
H(3)	2*i*	0.4611	0.297	1.3125	0.091
H(6)	2*i*	−0.0985	0.0443	0.402	0.064
H(5)	2*i*	0.3625	0.327	0.8901	0.042
H(7)	2*i*	0.2829	0.5091	1.1891	0.042
H(9)	2*i*	0.1153	0.5978	0.82	0.042
H(10)	2*i*	0.2609	0.6128	0.6585	0.054
H(11)	2*i*	0.2122	0.5821	0.4337	0.059
H(12)	2*i*	0.1164	0.3827	0.3224	0.051
H(17)	2*i*	0.3179	0.8574	0.5753	0.057
H(18)	2*i*	0.2334	0.9404	0.3797	0.05
H(21)	2*i*	0.2085	1.0606	0.2235	0.057
H(22)	2*i*	0.1162	1.1253	0.0145	0.069
H(23)	2*i*	0.3236	0.9038	−0.1278	0.072
H(24)	2*i*	0.4181	0.8331	0.0736	0.057
H(25)	2*i*	0.5418	0.8178	0.2737	0.066
H(26)	2*i*	0.6137	0.7351	0.4717	0.075

Discussion

Over the past few decades, supramolecular chemistry depending upon hydrogen bonding and intermolecular weak interactions have been a field of rapid growth due to potential application such as catalysis, magnetism, semiconduction, nonlinear optical and biological activity [1–5]. 4,4′-bipyridine and its derivatives, which are important building modules

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	0.2130(2)	1.0180(2)	−0.0700(2)	0.080(1)	0.055(1)	0.0347(8)	−0.0036(9)	0.0022(8)	0.0149(7)
N(2)	2*i*	0.4729(2)	0.7876(2)	0.5422(2)	0.0479(9)	0.0605(9)	0.0408(8)	0.0166(7)	0.0090(7)	0.0189(7)
O(1)	2*i*	0.0330(1)	0.7208(1)	0.9791(1)	0.0648(8)	0.0562(8)	0.0499(7)	0.0340(7)	0.0221(6)	0.0161(6)
O(2)	2*i*	0.1157(1)	0.6562(1)	1.1789(1)	0.0517(7)	0.0474(7)	0.0394(6)	0.0190(6)	0.0176(5)	0.0029(5)
O(3)	2*i*	0.4194(2)	0.3480(2)	1.2658(1)	0.080(1)	0.077(1)	0.0351(7)	0.0457(8)	0.0160(7)	0.0191(6)
O(4)	2*i*	0.4919(1)	0.2539(1)	1.1154(1)	0.0599(8)	0.0594(8)	0.0515(8)	0.0341(7)	0.0174(6)	0.0121(6)
O(5)	2*i*	0.1885(1)	0.1097(1)	0.6810(1)	0.0402(6)	0.0375(6)	0.0390(6)	0.0145(5)	0.0124(5)	0.0048(5)
O(6)	2*i*	−0.0663(1)	0.1143(1)	0.4449(1)	0.0435(7)	0.0444(7)	0.0416(6)	0.0057(5)	0.0169(5)	−0.0066(5)
O(7)	2*i*	0.0549(2)	0.1314(2)	0.3079(2)	0.117(1)	0.073(1)	0.064(1)	−0.0102(9)	0.062(1)	−0.0207(8)
O(8)	2*i*	0.0429(1)	0.1944(1)	0.7626(1)	0.0517(7)	0.0410(6)	0.0371(6)	0.0130(5)	0.0245(5)	0.0084(5)
C(1)	2*i*	0.1048(2)	0.2952(2)	0.4816(2)	0.0338(8)	0.0430(9)	0.0299(8)	0.0129(7)	0.0124(6)	0.0065(6)
C(2)	2*i*	0.1386(2)	0.3111(1)	0.6187(1)	0.0320(8)	0.0377(8)	0.0279(7)	0.0128(6)	0.0119(6)	0.0063(6)
C(3)	2*i*	0.1986(2)	0.4313(2)	0.6857(2)	0.0364(8)	0.0387(8)	0.0306(8)	0.0086(7)	0.0103(6)	0.0071(6)
C(4)	2*i*	0.2318(2)	0.4560(1)	0.8307(2)	0.0351(8)	0.0331(8)	0.0314(8)	0.0036(6)	0.0095(6)	0.0037(6)
C(5)	2*i*	0.3178(2)	0.3877(2)	0.9204(2)	0.0364(8)	0.0348(8)	0.0339(8)	0.0090(6)	0.0114(6)	0.0027(6)
C(6)	2*i*	0.3385(2)	0.4080(2)	1.0538(2)	0.0339(8)	0.0344(8)	0.0328(8)	0.0069(6)	0.0078(6)	0.0041(6)
C(7)	2*i*	0.2713(2)	0.4968(2)	1.0993(2)	0.0363(8)	0.0370(8)	0.0306(8)	0.0061(7)	0.0091(6)	0.0030(6)
C(8)	2*i*	0.1870(2)	0.5670(1)	1.0114(2)	0.0336(8)	0.0296(7)	0.0350(8)	0.0047(6)	0.0095(6)	0.0028(6)
C(9)	2*i*	0.1697(2)	0.5479(1)	0.8786(2)	0.0362(8)	0.0332(8)	0.0336(8)	0.0074(6)	0.0077(6)	0.0071(6)
C(10)	2*i*	0.2232(2)	0.5317(2)	0.6142(2)	0.053(1)	0.0400(9)	0.0396(9)	0.0023(8)	0.0114(8)	0.0086(7)
C(11)	2*i*	0.1932(2)	0.5139(2)	0.4797(2)	0.056(1)	0.051(1)	0.0388(9)	0.0023(8)	0.0137(8)	0.0189(8)
C(12)	2*i*	0.1353(2)	0.3956(2)	0.4135(2)	0.0439(9)	0.057(1)	0.0297(8)	0.0106(8)	0.0123(7)	0.0128(7)
C(13)	2*i*	0.1040(2)	0.6563(2)	1.0536(2)	0.0363(8)	0.0333(8)	0.0388(8)	0.0051(6)	0.0123(7)	0.0034(6)
C(14)	2*i*	0.4258(2)	0.3287(2)	1.1468(2)	0.0392(9)	0.0433(9)	0.0351(9)	0.0108(7)	0.0083(7)	0.0064(7)
C(15)	2*i*	0.1186(2)	0.1973(1)	0.6921(1)	0.0340(8)	0.0345(8)	0.0242(7)	0.0087(6)	0.0061(6)	0.0011(6)
C(16)	2*i*	0.0311(2)	0.1723(2)	0.4031(2)	0.0460(9)	0.0468(9)	0.0280(8)	0.0146(7)	0.0153(7)	0.0044(7)
C(17)	2*i*	0.3632(2)	0.8483(2)	0.5128(2)	0.050(1)	0.060(1)	0.0399(9)	0.0163(9)	0.0199(8)	0.0144(8)
C(18)	2*i*	0.3119(2)	0.8986(2)	0.3958(2)	0.0400(9)	0.051(1)	0.0397(9)	0.0188(7)	0.0152(7)	0.0114(7)
C(19)	2*i*	0.3766(2)	0.8875(2)	0.3017(2)	0.0319(8)	0.0374(8)	0.0335(8)	0.0080(6)	0.0096(6)	0.0075(6)
C(20)	2*i*	0.3218(2)	0.9366(2)	0.1717(2)	0.0348(8)	0.0373(8)	0.0360(8)	0.0059(7)	0.0097(7)	0.0088(6)
C(21)	2*i*	0.2322(2)	1.0265(2)	0.1529(2)	0.051(1)	0.050(1)	0.047(1)	0.0206(8)	0.0153(8)	0.0153(8)
C(22)	2*i*	0.1778(2)	1.0654(2)	0.0285(2)	0.052(1)	0.047(1)	0.067(1)	0.0131(9)	0.005(1)	0.024(1)
C(23)	2*i*	0.3002(3)	0.9343(2)	−0.0557(2)	0.087(2)	0.054(1)	0.038(1)	0.009(1)	0.019(1)	0.0092(8)
C(24)	2*i*	0.3560(2)	0.8924(2)	0.0637(2)	0.058(1)	0.048(1)	0.0402(9)	0.0116(8)	0.0200(8)	0.0081(8)
C(25)	2*i*	0.4927(2)	0.8262(2)	0.3331(2)	0.049(1)	0.081(1)	0.048(1)	0.034(1)	0.0240(9)	0.023(1)
C(26)	2*i*	0.5356(2)	0.7777(2)	0.4527(2)	0.051(1)	0.089(2)	0.057(1)	0.040(1)	0.0172(9)	0.030(1)

of N-containing heterocyclic ring compounds, may provide supramolecular recognition sites for N–H$\cdots$O, C–H$\cdots$O, C–H$\cdots\pi$ and π-π interactions to construct supramolecular architectures. On account its various coordination modes, multi-carboxylate ligands are extremely popular as efficient linkers in the construction of supramolecular compounds. [1,1′-biphenyl]-2,3,3′,5′-tetracarboxylic acid (bta) as unsymmetrically carboxylate possess several coordination sites with differing donor abilities. Its carboxylate group can also be used as hydrogen-bond acceptor. To our knowledge, the discrete subunits or low-dimensional entities have been linked highdimensional supramolecular compounds through hydrogen bonds, $\pi\cdots\pi$ stacking and host-guest interactions [6–8].

The asymmetric unit of the title structure contains one 4,4′-bipyridin-1-ium monocation and one 3,3′,5′-tricarboxy-[1,1′biphenyl]-2-carboxylate monoanion. All bond lengths are in the expected ranges. There are intermolecular hydrogen bonds d(O(2)—H(2)$\cdots$O(8)#2) = 2.5769(18) Å, d(O(6)—H(6)$\cdots$O(5)#4) = 2.6084(19) Å, d(O(3)—H(3)$\cdots$N(2)#3) = 2.601(2) Å, d(N(1)—H(1)$\cdots$O(8)#1) = 2.979(2) Å, d(N(1)—H(1)$\cdots$O(5)#1) = 2.835(2) Å connecting anions and cations to an infinite three dimensional architecture (Symmetry codes: #1 x,y+1,z−1; #2 −x,−y+1,−z+2; #3 −x+1,−y+1,−z+2; #4 −x,−y,−z+1).

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