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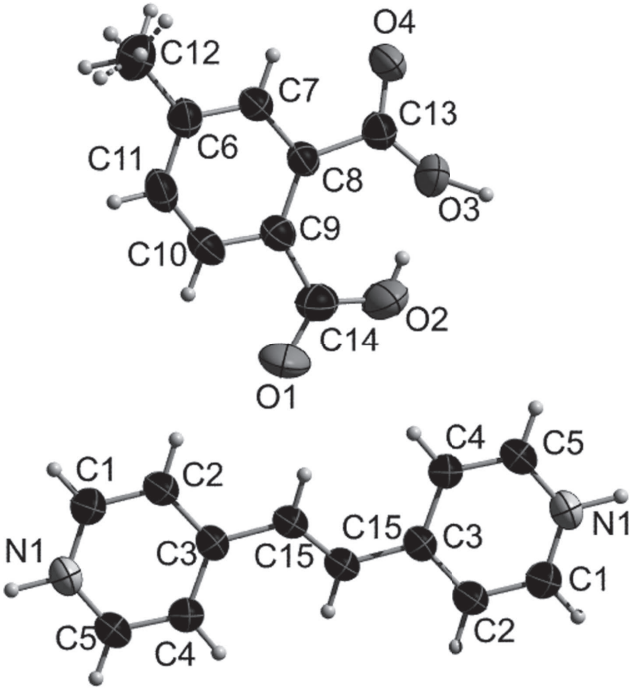
Liu Jing and Han Min-Le*

Crystal structure of trans-1,2-bis(pyridinium-4-yl) ethylene–2-carboxy-4-methylbenzoate (1/2), $C_{30}H_{26}N_2O_8$



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Abstract

$C_{30}H_{26}N_2O_8$, monoclinic, $C2/c$ (no. 15), $a = 17.567(2)$ Å, $b = 13.1375(17)$ Å, $c = 13.4212(17)$ Å, $\beta = 124.08(1)°$, $V = 2565.3(6)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0379$, $wR_{ref}(F^2) = 0.0839$, $T = 296$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Han Min-Le,** College of Chemistry and Chemical Engineering, LuoYang Normal University, Luoyang, Henan 471022, P. R. China, e-mail: minle_han@163.com
Liu Jing: College of Chemistry and Chemical Engineering, LuoYang Normal University, Luoyang, Henan 471022, P. R. China

Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.28×0.30×0.39 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.04 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω scans
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9611, 2391
$N(param)_{refined}$:	200
Programs:	SHELXS-97 [13]

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

Atom	Site	x	y	z	U_{iso}
H(2)	8*f*	0.1904	0.3412	0.7729	0.127
H(3)	8*f*	−0.11(1)	0.85(2)	0.38(2)	1.3(2)
H(1)	8*f*	0.3520	0.7249	0.1323	0.070
H(2A)	8*f*	0.4444	0.7051	0.3366	0.066
H(4)	8*f*	0.4036	0.4041	0.2902	0.058
H(5)	8*f*	0.3149	0.4306	0.0866	0.060
H(6)	8*f*	0.21(1)	0.89(1)	0.57(1)	0.9(1)
H(7)	8*f*	0.1791	0.6723	0.6769	0.056
H(8)	8*f*	0.06(2)	0.64(2)	0.30(3)	1.5(3)
H(10)	8*f*	0.0637	0.4036	0.4153	0.069
H(11)	8*f*	0.0580	0.5631	0.3457	0.071
H(12A)	8*f*	0.1148	0.7909	0.5104	0.110
H(12B)	8*f*	0.0581	0.7492	0.3783	0.110
H(12C)	8*f*	0.1657	0.7518	0.4523	0.110
H(12D)	8*f*	0.1110	0.7370	0.3836	0.110
H(12E)	8*f*	0.1676	0.7787	0.5157	0.110
H(12F)	8*f*	0.0600	0.7762	0.4417	0.110
H(15)	8*f*	0.5295	0.5982	0.5135	0.056
H(1D)	8*f*	0.288(1)	0.588(2)	0.002(2)	0.094(7)

Experimental detail

A mixture of H_2mph (4-methyl-phthalic acid, 0.2 mmol, 36 mg), bpe (1,2- bi(4-pyridyl)ethene, 0.1 mmol, 18 mg), and H_2O 10 mL was placed in a Teflon-lined stainless steel vessel, heated to 393 K for 3 days, and then cooled to room temperature over 24 h. Colourless block crystals of the title compound were obtained.

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)	8*f*	0.3271(1)	0.5794(1)	0.0935(1)	0.0551(9)	0.0520(9)	0.0330(8)	0.0002(7)	0.0194(7)	−0.0011(7)
O(1)	8*f*	0.0874(1)	0.2649(1)	0.5345(1)	0.109(1)	0.0520(9)	0.083(1)	−0.0214(8)	0.028(1)	−0.0219(8)
O(2)	8*f*	0.1609(1)	0.2947(1)	0.7260(1)	0.126(1)	0.0464(8)	0.076(1)	−0.0029(8)	0.053(1)	0.0045(7)
O(3)	8*f*	0.2392(1)	0.4355(1)	0.8619(1)	0.101(1)	0.0573(8)	0.0443(7)	0.0160(8)	0.0321(8)	0.0104(6)
O(4)	8*f*	0.2225(1)	0.60091(9)	0.8588(1)	0.099(1)	0.0518(8)	0.0373(7)	−0.0085(7)	0.0288(7)	−0.0062(6)
C(1)	8*f*	0.3638(1)	0.6600(1)	0.1655(2)	0.075(1)	0.045(1)	0.044(1)	−0.0021(9)	0.026(1)	0.0018(8)
C(2)	8*f*	0.4184(1)	0.6483(1)	0.2874(2)	0.071(1)	0.043(1)	0.039(1)	−0.0071(9)	0.0233(9)	−0.0047(8)
C(3)	8*f*	0.4354(1)	0.5520(1)	0.3383(1)	0.0453(9)	0.0431(9)	0.0345(9)	−0.0015(7)	0.0202(8)	−0.0031(7)
C(4)	8*f*	0.3946(1)	0.4698(1)	0.2599(1)	0.057(1)	0.0390(9)	0.0412(9)	−0.0001(8)	0.0223(9)	−0.0009(8)
C(5)	8*f*	0.3414(1)	0.4859(1)	0.1385(1)	0.056(1)	0.047(1)	0.039(1)	−0.0027(8)	0.0201(9)	−0.0077(8)
C(6)	8*f*	0.1174(1)	0.6363(1)	0.5034(2)	0.052(1)	0.053(1)	0.040(1)	−0.0010(9)	0.0221(9)	0.0028(8)
C(7)	8*f*	0.1561(1)	0.6174(1)	0.6241(1)	0.051(1)	0.044(1)	0.0369(9)	−0.0050(8)	0.0207(8)	−0.0046(7)
C(8)	8*f*	0.1623(1)	0.5209(1)	0.6706(1)	0.0398(9)	0.0441(9)	0.0344(9)	−0.0034(7)	0.0178(7)	−0.0024(7)
C(9)	8*f*	0.1275(1)	0.4370(1)	0.5910(1)	0.0425(9)	0.0451(9)	0.043(1)	−0.0042(8)	0.0201(8)	−0.0052(8)
C(10)	8*f*	0.0881(1)	0.4573(1)	0.4697(2)	0.056(1)	0.059(1)	0.042(1)	−0.0091(9)	0.0177(9)	−0.0151(9)
C(11)	8*f*	0.0840(1)	0.5535(2)	0.4273(2)	0.060(1)	0.070(1)	0.0323(9)	−0.003(1)	0.0166(9)	0.0000(9)
C(12)	8*f*	0.1137(2)	0.7416(2)	0.4569(2)	0.092(2)	0.064(1)	0.059(1)	0.001(1)	0.039(1)	0.015(1)
C(13)	8*f*	0.2109(1)	0.5184(1)	0.8059(2)	0.054(1)	0.050(1)	0.0384(9)	−0.0029(8)	0.0230(9)	0.0006(9)
C(14)	8*f*	0.1244(1)	0.3258(1)	0.6166(2)	0.059(1)	0.048(1)	0.066(1)	−0.0047(9)	0.030(1)	−0.005(1)
C(15)	8*f*	0.4953(1)	0.5415(1)	0.4697(1)	0.053(1)	0.0402(9)	0.0373(9)	−0.0049(8)	0.0196(8)	−0.0062(7)

Discussion

In the last two decades, the design and synthesis of supramolecular polymeric networks, especially those constructed by hydrogen bonding and intermolecular weak interactions have been a field of rapid growth due to their special physical properties and potential application in functional materials [1–6]. Most of these materials are constructed by organic bridging ligand. Bi(pyridyl) ligands, such as 1,2-bi(4-pyridyl)ethene (bpe), as one kinds of the extensively studied neutral ligand, is often used for the construction of various supramolecular architectures by means of coordination and hydrogen bonds due to its two potential binding sites. They do not only coordinate to metal ions but also act as the hydrogen bonding acceptor or hydrogen bonding donor when one or two N atoms are protonated [7–9]. On the other hand, multicarboxylate ligands, such as 1,2-benzenedicarboxylic acid (phthalic acid, H_2ph) or its derivatives, such as 4-methyl-phthalic acid (H_2mph) has two carboxyl groups which may be completely, partially deprotonated or not deprotonated and act as hydrogen-bond acceptor or hydrogen-bond donor, depending upon the numbers of deprotonated carboxyl groups [10–12]. Taking into account of these, we report the molecular assemblies of bpe and H_2mph in order to understand the physical-chemical character of the compound in future. In the title co-crystal structure, the asymmetric unit comprises half of H_2bpe molecule, and one Hmph molecule (Figure). Hydrogen bonding and aromatic π···π stacking interactions play an important role in the structure. Two Hmph and one H_2bpe are linked by hydrogen bonding N–H···O with a N···O distances of 2.628(2) Å to form a three-component unit. Hydrogen bonding between the H_2bpe CH and carboxylic groups oxygen atoms of Hmph with C···O distances of 3.158(3), 3.220(3), and 3.341(3) Å, respectively, also may exist. There are three kinds of aromatic π···π interactions: (a) π···π interactions between the benzene ring and the pyridine ring (The angle between the benzene ring plane and pyridine ring plane are 16°. The distances of Cg···Cg are 3.99 Å; Cg···Cg are distances between ring centroids.); (b) π···π interactions between the benzene ring and the benzene ring (The angle between the two rings are 0°. The distances of Cg···Cg are 3.87 Å.); (c) π···π interactions between the pyridine ring and the pyridine ring (The angle between the two rings are 9.55°. The distances of Cg···Cg are 3.76 Å). An extended 3D supramolecular architecture with π-π interactions and hydrogen bonds contributes to the stability of the structure.

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