

Yu-Mei Dai,^{a,b*} Hui-Ying Shen^a
and Jin-Feng Huang^a

^aCollege of Chemistry and Materials, Fujian Normal University, Fuzhou 350007, People's Republic of China, and ^bThe State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, The Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China

Correspondence e-mail: dym@ms.fjirsm.ac.cn

Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}—\text{C}) = 0.002\text{ \AA}$

R factor = 0.043

wR factor = 0.120

Data-to-parameter ratio = 12.7

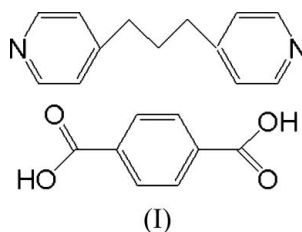
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Terephthalic acid–1,3-di-4-pyridylpropane (1/1)

The title compound, $\text{C}_{13}\text{H}_{14}\text{N}_2 \cdot \text{C}_8\text{H}_6\text{O}_4$, was obtained by the hydrothermal reaction of CuCl with 1,2,4,5-benzenetetracarboxylic acid and 1,3-bis(4-pyridyl)propane. The 1,3-bis(4-pyridyl)propane molecule lies on a crystallographic twofold axis, and terephthalic acid is on an inversion centre. In the crystal structure, strong intermolecular $\text{O}—\text{H} \cdots \text{N}$ hydrogen bonds between the components lead to the formation of zigzag chains extending along $[10\bar{1}]$. The chains are linked through intermolecular $\text{C}—\text{H} \cdots \text{O}$ hydrogen bonds.

Comment

The construction of inorganic coordination polymeric complexes has developed rapidly in recent years, owing to their interesting molecular topologies and crystal-packing motifs (Hagman *et al.*, 1999; Yaghi *et al.*, 1998) along with potential applications as functional materials (Evans *et al.*, 1999; Fujita *et al.*, 1994). 1,2,4,5-Benzenetetracarboxylic acid (H_4btc) attracted our attention for the process of constructing coordination polymers. During the process, a hydrothermal decarboxylation reaction was observed and the H_4btc ligand was converted *in situ* into terephthalic acid by loss of two carboxyl groups. We report here the synthesis and crystal structure of the title compound, (I).



The asymmetric unit contains one half-molecule each of terephthalic acid and 1,3-bis(4-pyridyl)propane; the 1,3-bis(4-pyridyl)propane molecule lies on a crystallographic twofold axis, and terephthalic acid is on an inversion centre (Fig. 1). In the crystal structure, $\text{O}—\text{H} \cdots \text{N}$ hydrogen bonds between carboxylic acid groups and N atoms of the pyridine rings link the molecules into zigzag chains along $[10\bar{1}]$ (Table 1). Intermolecular $\text{C}—\text{H} \cdots \text{O}$ hydrogen bonds link the chains into a three-dimensional network (Fig. 2).

Experimental

CuCl (0.10 g, 1 mmol), H_4btc (0.127 g, 0.5 mmol) and KOH (0.06 g, 1 mmol) were mixed in water (15 ml) and heated at 433 K for 3 d in a sealed 25 ml Teflon-lined stainless steel vessel under autogenous pressure. Cooling to room temperature at 5 K h^{-1} produced yellow

Received 12 September 2005

Accepted 20 September 2005

Online 28 September 2005

block crystals, which were collected, washed with water and dried in air.

Crystal data

$C_{13}H_{14}N_2 \cdot C_8H_6O_4$
 $M_r = 364.38$
 Monoclinic, $C2/c$
 $a = 23.270$ (2) Å
 $b = 4.7000$ (2) Å
 $c = 18.9600$ (19) Å
 $\beta = 119.440$ (3)°
 $V = 1805.9$ (3) Å³
 $Z = 4$

$D_x = 1.340$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 1874 reflections
 $\theta = 3.5$ – 27.5°
 $\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K
 Prism, yellow
 $0.40 \times 0.30 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.967$, $T_{\max} = 0.981$
 6545 measured reflections

2063 independent reflections
 1811 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 27.5^\circ$
 $h = -30 \rightarrow 30$
 $k = -5 \rightarrow 6$
 $l = -13 \rightarrow 24$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.120$
 $S = 1.09$
 2063 reflections
 163 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.7554P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.25$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1 \cdots N1$	1.06 (2)	1.53 (2)	2.5782 (14)	171 (2)
$C5-H5A \cdots O1^i$	0.96 (2)	2.49 (2)	3.2162 (16)	132 (1)
$C8-H8A \cdots O2^{ii}$	0.95 (2)	2.35 (2)	3.2268 (19)	155 (1)

Symmetry codes: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x, -y + 1, -z$.

H atoms were located in a difference Fourier map and both positional and isotropic displacement parameters were refined. The C–H range is 0.946 (16)–1.002 (16) Å and the O–H distance is 1.06 (2) Å.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1994); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Siemens, 1994); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

This work was financially supported by the Foundation of the Ministry of Education of Fujian Province (grant No. JB03131).

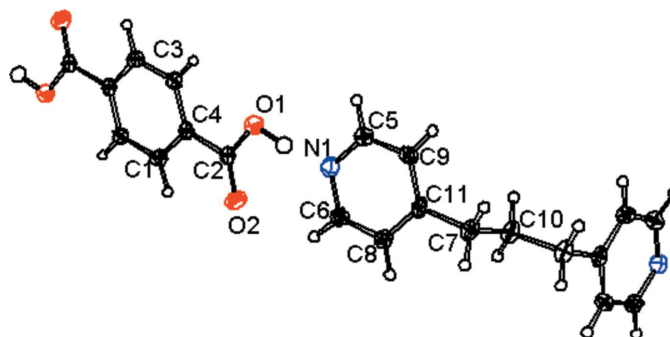


Figure 1

The structure of (I). Displacement ellipsoids are drawn at the 40% probability level. Unlabelled atoms in the terephthalic acid and 1,3-bis(4-pyridyl)propane molecules are related to labelled atoms in the same molecule by $(\frac{1}{2} - x, -\frac{1}{2} - y, -z)$ and $(-x, y, \frac{1}{2} - z)$, respectively.

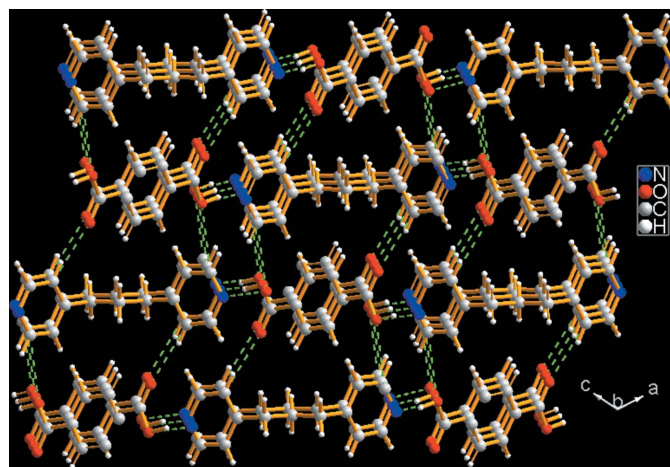


Figure 2

The crystal packing of (I), showing the three-dimensional hydrogen-bonded (dashed lines) network.

References

- Evans, O. R., Xiong, R., Wang, Z., Wong, G. K. & Lin, W. (1999). *Angew. Chem. Int. Ed.* **38**, 536–538.
- Fujita, M., Kwon, Y. J., Washizu, S. & Ogura, K. (1994). *J. Am. Chem. Soc.* **116**, 1151–1152.
- Hagman, P. J., Hagman, D. & Zubieta, J. (1999). *Angew. Chem. Int. Ed.* **38**, 2638–2684.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Siemens (1994). *SAINT and SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Siemens (1996). *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Yaghi, O. M., Li, H., Davis, C., Richardson, D. & Groy, T. L. (1998). *Acc. Chem. Res.* **31**, 474–484.