

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Bis(4-pyridylmethyl) hexanedioate

Jin-Hui Yang,^a Jian-Min Zhang,^a Yan-Xue Chen,^{b*} Jian-Zhi Diao^a and Zheng Peng^a

^aSchool of Materials Science and Engineering, Shijiazhuang Railway Institute, Shijiazhuang 050043, People's Republic of China, and ^bSchool of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: chenyanxue8010@yahoo.com.cn

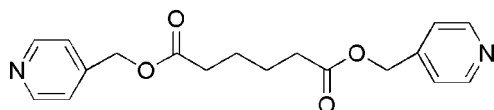
Received 8 May 2008; accepted 13 May 2008

Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 17.6.

The asymmetric unit of the title compound, $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_4$, contains one half-molecule. The molecule lies on an inversion centre and is roughly planar, the chains between the two pyridine rings being only slightly twisted, with torsion angles ranging from 170.9 (1) to 177.2 (1)°. Weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds result in the formation of a three-dimensional network.

Related literature

For related literature, see: Banfi *et al.* (2002); Magden & Basel (1984).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_4$
 $M_r = 328.36$

Monoclinic, $P2_1/c$
 $a = 9.1489$ (18) Å

$b = 10.164$ (2) Å
 $c = 8.9206$ (18) Å
 $\beta = 102.11$ (3)°
 $V = 811.1$ (3) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 113$ (2) K
 $0.12 \times 0.10 \times 0.08$ mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.979$, $T_{\max} = 0.988$

9823 measured reflections
1918 independent reflections
1288 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.106$
 $S = 0.98$
1918 reflections

109 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.34$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C6}-\text{H6B}\cdots\text{O2}^i$ | 0.97 | 2.56 | 3.3333 (17) | 137 |

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-III* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2349).

References

- Banfi, S., Carlucci, L., Caruso, E., Ciani, G. & Proserpio, D. (2002). *J. Chem. Soc. Dalton Trans.* pp. 2714–2721.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP-III*. Report ORNL-6895. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Magden, A. & Basel, E. (1984). United States Patent US 4 461 898.
Rigaku (2005). *CrystalClear*. Rigaku/MS, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2008). E64, o1100 [doi:10.1107/S1600536808014414]

Bis(4-pyridylmethyl) hexanedioate

J.-H. Yang, J.-M. Zhang, Y.-X. Chen, J.-Z. Diao and Z. Peng

Comment

Hexanedioic acid dipyridin-4-ylmethyl ester is a very important intermediate in the synthesis of cephalosporins (Magden & Basel, 1984). Also, it can be used as a ligand designed for the self-assembly of coordination frameworks and architectures (Banfi *et al.*, 2002);

The title compound is arranged around an inversion centre located in the middle of the C9-C9ⁱ bond [symmetry code:(i) 1-x, 1-y, 1-z] (Fig. 1). The molecule is roughly planar, the chains between the two pyridyl rings being only slightly twisted with torsion angles ranging from 170.9 (1) to 177.2 (1)° .

Weak intermolecular C—H···O hydrogen bonds (Table 1) result in the formation of a three dimensionnal network

Experimental

4-pyridinemethanol (9.82 g, 0.09 mol) and dimethyl adipate (5.22 g, 0.03 mol) were stirred with 200 ml n-octane at 343 k, then titanium tetraisopropoxide (0.2 g) was added. The mixture was heated to 399 k, the methanol was distilled off. After stirring at this temperature for 4 h, the reaction finished. The solvent was evaporated under reduced pressure. The product was purified by chromatography on silica. Crystals of hexanedioic acid dipyridin-4-ylmethyl ester were obtained by slow evaporation of a solution of ethyl acetate at room temperature (m.p. 359 k).

Refinement

H atoms were positioned geometrically and refined as riding on their parent atoms [C—H distances are 0.93 Å (aromatic) and 0.97 Å (methylene) with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$].

Figures

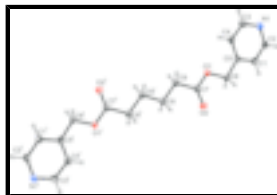


Fig. 1. A view of the molecular structure of (I) with the atoms labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (i) 1-x, 1-y, 1-z]

Bis(4-pyridylmethyl) hexanedioate

Crystal data

C₁₈H₂₀N₂O₄

$F_{000} = 348$

supplementary materials

$M_r = 328.36$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.1489$ (18) Å

$b = 10.164$ (2) Å

$c = 8.9206$ (18) Å

$\beta = 102.11$ (3)°

$V = 811.1$ (3) Å³

$Z = 2$

$D_x = 1.345$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2434 reflections

$\theta = 2.3$ – 27.9 °

$\mu = 0.10$ mm⁻¹

$T = 113$ (2) K

Block, colorless

$0.12 \times 0.10 \times 0.08$ mm

Data collection

Rigaku Saturn
diffractometer

Radiation source: rotating anode

Monochromator: confocal

$T = 113$ (2) K

ω scans

Absorption correction: multi-scan
(CrystalClear; Rigaku, 2005)

$T_{\min} = 0.979$, $T_{\max} = 0.988$

9823 measured reflections

1918 independent reflections

1288 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.060$

$\theta_{\text{max}} = 27.9$ °

$\theta_{\text{min}} = 2.3$ °

$h = -12 \rightarrow 12$

$k = -13 \rightarrow 13$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.107$

$S = 0.98$

1918 reflections

109 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.34$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -

factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|---------------|----------------------------------|
| O1 | 0.70599 (9) | 0.51214 (8) | 0.11415 (9) | 0.0198 (2) |
| O2 | 0.64123 (11) | 0.69241 (9) | 0.22847 (10) | 0.0307 (3) |
| N1 | 0.90646 (13) | 0.38939 (10) | -0.35175 (12) | 0.0234 (3) |
| C1 | 0.89598 (14) | 0.58417 (13) | -0.20251 (14) | 0.0219 (3) |
| H1 | 0.9218 | 0.6722 | -0.1854 | 0.026* |
| C2 | 0.93751 (15) | 0.51700 (13) | -0.32073 (15) | 0.0226 (3) |
| H2 | 0.9901 | 0.5625 | -0.3828 | 0.027* |
| C3 | 0.83041 (15) | 0.32799 (13) | -0.25920 (15) | 0.0250 (3) |
| H3 | 0.8082 | 0.2394 | -0.2773 | 0.030* |
| C4 | 0.78264 (14) | 0.38780 (13) | -0.13832 (14) | 0.0221 (3) |
| H4 | 0.7297 | 0.3404 | -0.0781 | 0.026* |
| C5 | 0.81535 (14) | 0.51996 (12) | -0.10895 (13) | 0.0184 (3) |
| C6 | 0.76640 (15) | 0.59800 (12) | 0.01395 (14) | 0.0206 (3) |
| H6A | 0.8509 | 0.6460 | 0.0727 | 0.025* |
| H6B | 0.6910 | 0.6614 | -0.0323 | 0.025* |
| C7 | 0.64740 (13) | 0.57452 (13) | 0.22139 (13) | 0.0188 (3) |
| C8 | 0.59372 (14) | 0.47965 (12) | 0.32611 (14) | 0.0200 (3) |
| H8A | 0.6751 | 0.4212 | 0.3707 | 0.024* |
| H8B | 0.5141 | 0.4264 | 0.2669 | 0.024* |
| C9 | 0.53658 (14) | 0.54786 (11) | 0.45402 (14) | 0.0196 (3) |
| H9A | 0.6193 | 0.5912 | 0.5219 | 0.023* |
| H9B | 0.4647 | 0.6148 | 0.4102 | 0.023* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|------------|------------|------------|-------------|------------|-------------|
| O1 | 0.0269 (5) | 0.0179 (5) | 0.0184 (5) | 0.0006 (4) | 0.0133 (4) | 0.0013 (4) |
| O2 | 0.0492 (7) | 0.0193 (5) | 0.0301 (6) | -0.0007 (4) | 0.0230 (5) | -0.0018 (4) |
| N1 | 0.0283 (6) | 0.0223 (6) | 0.0218 (6) | 0.0034 (5) | 0.0102 (5) | -0.0005 (5) |
| C1 | 0.0242 (7) | 0.0193 (7) | 0.0236 (7) | 0.0012 (5) | 0.0081 (6) | 0.0020 (6) |
| C2 | 0.0244 (7) | 0.0263 (7) | 0.0197 (7) | 0.0016 (6) | 0.0106 (6) | 0.0040 (6) |
| C3 | 0.0288 (8) | 0.0211 (7) | 0.0263 (8) | -0.0004 (6) | 0.0089 (6) | -0.0040 (6) |
| C4 | 0.0235 (7) | 0.0249 (7) | 0.0199 (7) | -0.0010 (5) | 0.0093 (6) | 0.0018 (6) |
| C5 | 0.0189 (6) | 0.0216 (7) | 0.0145 (6) | 0.0016 (5) | 0.0033 (5) | 0.0012 (5) |
| C6 | 0.0283 (7) | 0.0175 (7) | 0.0191 (7) | -0.0018 (5) | 0.0122 (6) | 0.0023 (5) |
| C7 | 0.0199 (7) | 0.0208 (7) | 0.0168 (7) | 0.0002 (5) | 0.0062 (5) | -0.0028 (6) |
| C8 | 0.0240 (7) | 0.0183 (6) | 0.0202 (7) | -0.0005 (5) | 0.0106 (5) | 0.0010 (5) |
| C9 | 0.0220 (7) | 0.0204 (7) | 0.0187 (7) | -0.0001 (5) | 0.0098 (6) | 0.0005 (6) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-------|-------------|-------|--------|
| O1—C7 | 1.3489 (14) | C4—H4 | 0.9300 |
|-------|-------------|-------|--------|

supplementary materials

| | | | |
|-------------|--------------|-------------------------|-------------|
| O1—C6 | 1.4398 (14) | C5—C6 | 1.4959 (17) |
| O2—C7 | 1.2019 (15) | C6—H6A | 0.9700 |
| N1—C3 | 1.3402 (16) | C6—H6B | 0.9700 |
| N1—C2 | 1.3441 (16) | C7—C8 | 1.4954 (17) |
| C1—C2 | 1.3751 (17) | C8—C9 | 1.5186 (17) |
| C1—C5 | 1.3874 (16) | C8—H8A | 0.9700 |
| C1—H1 | 0.9300 | C8—H8B | 0.9700 |
| C2—H2 | 0.9300 | C9—C9 ⁱ | 1.516 (2) |
| C3—C4 | 1.3860 (17) | C9—H9A | 0.9700 |
| C3—H3 | 0.9300 | C9—H9B | 0.9700 |
| C4—C5 | 1.3891 (18) | | |
| C7—O1—C6 | 114.63 (10) | C5—C6—H6A | 109.6 |
| C3—N1—C2 | 115.92 (11) | O1—C6—H6B | 109.6 |
| C2—C1—C5 | 119.68 (12) | C5—C6—H6B | 109.6 |
| C2—C1—H1 | 120.2 | H6A—C6—H6B | 108.1 |
| C5—C1—H1 | 120.2 | O2—C7—O1 | 122.40 (11) |
| N1—C2—C1 | 123.82 (12) | O2—C7—C8 | 125.79 (11) |
| N1—C2—H2 | 118.1 | O1—C7—C8 | 111.80 (11) |
| C1—C2—H2 | 118.1 | C7—C8—C9 | 112.64 (10) |
| N1—C3—C4 | 124.35 (12) | C7—C8—H8A | 109.1 |
| N1—C3—H3 | 117.8 | C9—C8—H8A | 109.1 |
| C4—C3—H3 | 117.8 | C7—C8—H8B | 109.1 |
| C3—C4—C5 | 118.70 (11) | C9—C8—H8B | 109.1 |
| C3—C4—H4 | 120.6 | H8A—C8—H8B | 107.8 |
| C5—C4—H4 | 120.6 | C9 ⁱ —C9—C8 | 111.98 (12) |
| C1—C5—C4 | 117.52 (11) | C9 ⁱ —C9—H9A | 109.2 |
| C1—C5—C6 | 118.04 (11) | C8—C9—H9A | 109.2 |
| C4—C5—C6 | 124.42 (11) | C9 ⁱ —C9—H9B | 109.2 |
| O1—C6—C5 | 110.27 (10) | C8—C9—H9B | 109.2 |
| O1—C6—H6A | 109.6 | H9A—C9—H9B | 107.9 |
| C1—C5—C6—O1 | -170.85 (11) | O1—C7—C8—C9 | 176.27 (10) |
| C6—O1—C7—C8 | -177.17 (10) | | |

Symmetry codes: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|-------|-------------|-------------|---------------|
| C6—H6B \cdots O2 ⁱⁱ | 0.97 | 2.56 | 3.3333 (17) | 137 |

Symmetry codes: (ii) $x, -y+3/2, z-1/2$.

Fig. 1

