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Bis(4-pyridylmethyl) hexanedioate

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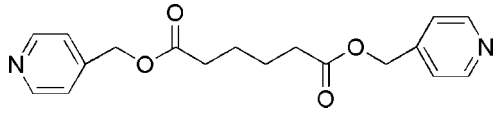
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 17.6.

The asymmetric unit of the title compound, $C_{18}H_{20}N_2O_4$, 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 C—H···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

$C_{18}H_{20}N_2O_4$
M_r = 328.36
Monoclinic, $P2_1/c$
a = 9.1489 (18) Å
b = 10.164 (2) Å
c = 8.9206 (18) Å
β = 102.11 (3)°
V = 811.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 113 (2) K
0.12 × 0.10 × 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_{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—H···A	D—H	H···A	D···A	D—H···A
C6—H6B···O2^{i}	0.97	2.56	3.3333 (17)	137

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

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: *ORTEPIII* (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). *ORTEPIII*. 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/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

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S1. 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-C9^{i} 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

S2. 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 titaniun 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 acetation at room temperature(m.p. 359 k).

S3. 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_{iso}(H) = 1.2 U_{eq}(C)].

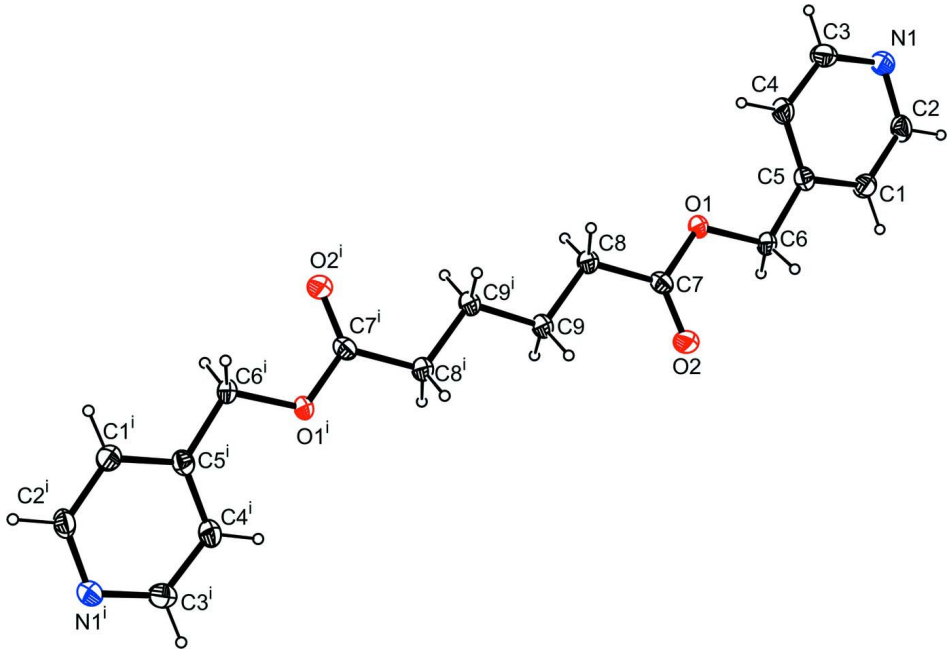

Figure 1

A view of the molecular structure of (I)with the atoms labelling scheme. Displacement ellopsoids 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*]

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Crystal data

$C_{18}H_{20}N_2O_4$
$M_r = 328.36$
Monoclinic, $P2_1/c$
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$a = 9.1489\ (18)$ Å
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$\beta = 102.11\ (3)°$
$V = 811.1\ (3)$ Å^3
$Z = 2$

$F(000) = 348$
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Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2434 reflections
$\theta = 2.3–27.9°$
$\mu = 0.10$ mm^{-1}
$T = 113$ K
Block, colorless
0.12 × 0.10 × 0.08 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Confocal monochromator
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 $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.3°$
$h = -12\rightarrow12$
$k = -13\rightarrow13$
$l = -11\rightarrow11$

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
0 restraints
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{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 (Å²)

	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 (Å, º)

O1—C7	1.3489 (14)	C4—H4	0.9300
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 code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*B*···O2^{ii}	0.97	2.56	3.3333 (17)	137

Symmetry code: (ii) *x*, −*y*+3/2, *z*−1/2.