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Propane-1,3-diyl bis(pyridine-3-carboxylate)

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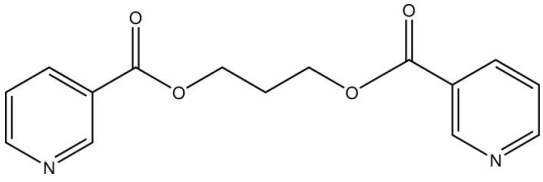
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.010 Å; R factor = 0.109; wR factor = 0.276; data-to-parameter ratio = 13.4.

The title compound, $C_{15}H_{14}N_2O_4$, has a *trans–gauche* [O/C/C/C–O/C/C/C] (TG) conformation. The angle between the planes of aromatic rings is 76.4 (3)°. The crystal structure is stabilized by van der Waals interactions and C—H···O hydrogen bonds. The crystal used was a non-merohedral twin with a fractional contribution of the minor component of 0.443 (5).

Related literature

For conformation definitions, see: Carlucci *et al.* (2002). For applications of crystalline nanoporous coordination polymers, see Matsuda *et al.* (2005); Wu *et al.* (2005); Xiang *et al.* (2005).



Experimental

Crystal data

$C_{15}H_{14}N_2O_4$
M_r = 286.28
Triclinic, $P\overline{1}$
a = 4.4797 (11) Å
b = 10.911 (3) Å
c = 14.842 (4) Å
α = 104.41 (2)°
β = 95.90 (2)°
γ = 100.90 (2)°
V = 681.3 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 173 K
0.22 × 0.14 × 0.07 mm

Data collection

Stoe IPDS II two-circle diffractometer
8629 measured reflections
2558 independent reflections
1382 reflections with $I > 2\sigma(I)$
R_{int} = 0.119

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.109
$wR(F^2)$ = 0.276
S = 1.49
2558 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···O4^{i}	0.99	2.49	3.341 (8)	144
C16—H16···O2^{ii}	0.95	2.45	3.198 (10)	136
C24—H24···O2^{iii}	0.95	2.45	3.218 (10)	138

Symmetry codes: (i) $-x+3, -y+1, -z+1$; (ii) $-x+1, -y, -z$; (iii) $-x+1, -y, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2324).

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supporting information

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Propane-1,3-diyl bis(pyridine-3-carboxylate)

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S1. Comment

In the past decade, crystalline nanoporous coordination polymers have been extensively studied for their potential applications in magnetism (Xiang *et al.*, 2005), catalysis (Wu *et al.*, 2005) and gas adsorption or separation (Matsuda *et al.*, 2005). The propanediyl group in the crystal structure can adopt four possible conformations: trans-trans (TT), trans-gauche (TG), gauche-gauche (GG), gauche-gauche′ (GG′) (Carlucci *et al.*, 2002). The propanediyl group in the title compound has a *trans-gauche* (TG) [O1/C2/C3/C4 - O3/C4/C3/C2] conformation (Fig. 1). The angle between the planes of aromatics rings is 76.4 (3)°. The crystal structure is stabilized by van der Waals interactions and C—H···O hydrogen bonds (Table 1). To the best of our knowledge coordination polymer with this ligand still remain unknown.

S2. Experimental

Nicotinic acid (15 g, 0.122 mol) was stirred in $SOCl_2$ (40 ml) in the presence of DMF (0.6 ml) at 60 °C for 12 h. Excess thionyl chloride was removed in vacuo. Dried propanediol (4.3 ml, 0.061 mol) was added. After the evolution of hydrogen chloride ended, the mixture was heated at 150 °C for 2 h. The mixture was dissolved in water, and NH_4OH solution was added. After filtration, recrystallization in ethyl acetate gave colorless crystal. Yield 11.53 g (80 %). Analysis calculated for $C_{15}H_{14}N_2O_4$: C:62.9 , H:4.89 , N:9.68 ; found: C: 62.25, H: 4.68, N:9.52 . IR (KBr, cm^{-1}): (C═O) 1715 s, (C═C) 1591 m, (Ar C—C, C═N) 1429 s,(C—O) 1277 m.

S3. Refinement

The crystal turned out to be a non-merohedral twin (twin law: -1 0 0/0 -0.476 -0.740/ 0 -1 0.478) with a fractional contribution of the minor component of 0.443 (5). H atoms were placed in idealized positions and treated as riding atoms with C—H distances in the range 0.95-0.99 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The material was difficult to obtain in a suitable crystalline form.

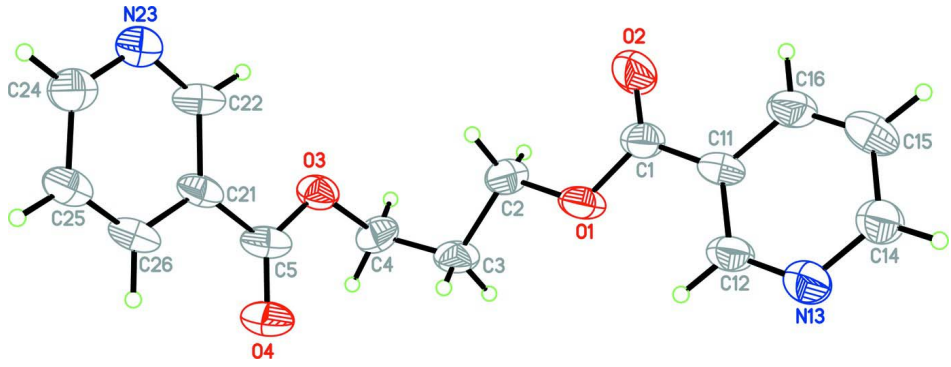

Figure 1

A view of the molecular structure with the atom-numbering scheme. Displacemenent ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

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Crystal data

$C_{15}H_{14}N_2O_4$
$M_r = 286.28$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.4797\ (11)$ Å
$b = 10.911\ (3)$ Å
$c = 14.842\ (4)$ Å
$\alpha = 104.41\ (2)°$
$\beta = 95.90\ (2)°$
$\gamma = 100.90\ (2)°$
$V = 681.3\ (3)$ Å^3
$Z = 2$
$F(000) = 300$
$D_x = 1.395$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3924 reflections
$\theta = 3.9–25.8°$
$\mu = 0.10$ mm^{-1}
$T = 173$ K
Plate, colourless
0.22 × 0.14 × 0.07 mm

Data collection

Stoe IPDS II two-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
8629 measured reflections
2558 independent reflections
1382 reflections with $I > 2\sigma(I)$
$R_{int} = 0.119$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.8°$
$h = -5\rightarrow5$
$k = -13\rightarrow12$
$l = -12\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.109$
$wR(F^2) = 0.276$
$S = 1.49$
2558 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.090P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ 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	0.8187 (10)	0.2723 (4)	0.2445 (4)	0.0458 (12)
O2	0.6697 (13)	0.0749 (5)	0.1430 (4)	0.0637 (16)
O3	1.1653 (9)	0.2272 (4)	0.5109 (3)	0.0405 (11)
O4	1.2061 (11)	0.4252 (4)	0.6081 (4)	0.0540 (14)
C1	0.6560 (14)	0.1865 (6)	0.1644 (5)	0.0434 (17)
C2	1.0002 (15)	0.2161 (6)	0.3054 (5)	0.0434 (16)
H2A	1.1296	0.1648	0.2688	0.052*
H2B	0.8629	0.1587	0.3334	0.052*
C3	1.2023 (14)	0.3317 (6)	0.3827 (5)	0.0448 (17)
H3A	1.3515	0.3838	0.3543	0.054*
H3B	1.0718	0.3881	0.4135	0.054*
C4	1.3748 (14)	0.2826 (7)	0.4557 (5)	0.0445 (17)
H4A	1.4795	0.2160	0.4234	0.053*
H4B	1.5339	0.3554	0.4981	0.053*
C5	1.0994 (13)	0.3111 (6)	0.5848 (5)	0.0439 (17)
C11	0.4701 (14)	0.2473 (6)	0.1081 (5)	0.0420 (16)
C12	0.4516 (15)	0.3777 (6)	0.1359 (5)	0.0451 (17)
H12	0.5646	0.4308	0.1948	0.054*
N13	0.2835 (15)	0.4308 (6)	0.0838 (5)	0.0568 (17)
C14	0.1172 (16)	0.3544 (7)	0.0009 (6)	0.0538 (19)
H14	−0.0070	0.3904	−0.0367	0.065*
C15	0.1253 (17)	0.2229 (7)	−0.0306 (6)	0.055 (2)
H15	0.0121	0.1712	−0.0899	0.066*
C16	0.2940 (17)	0.1699 (7)	0.0235 (6)	0.0516 (19)
H16	0.2923	0.0800	0.0037	0.062*
C21	0.8829 (13)	0.2446 (6)	0.6378 (5)	0.0386 (15)
C22	0.7613 (14)	0.1126 (6)	0.6107 (6)	0.0472 (18)
H22	0.8192	0.0626	0.5560	0.057*
N23	0.5693 (13)	0.0501 (6)	0.6553 (4)	0.0505 (16)
C24	0.4879 (17)	0.1254 (7)	0.7329 (6)	0.0526 (19)
H24	0.3486	0.0843	0.7661	0.063*
C25	0.5981 (16)	0.2594 (6)	0.7660 (5)	0.0470 (17)
H25	0.5369	0.3086	0.8205	0.056*
C26	0.7973 (14)	0.3177 (6)	0.7173 (5)	0.0446 (17)
H26	0.8772	0.4089	0.7381	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.045 (2)	0.027 (2)	0.054 (3)	0.0010 (19)	−0.005 (2)	0.001 (2)
O2	0.095 (4)	0.041 (3)	0.047 (3)	0.020 (3)	−0.005 (3)	−0.001 (2)
O3	0.036 (2)	0.035 (2)	0.043 (3)	−0.0023 (18)	0.005 (2)	0.004 (2)
O4	0.051 (3)	0.032 (3)	0.066 (4)	−0.004 (2)	0.006 (2)	0.003 (2)
C1	0.043 (3)	0.033 (4)	0.048 (5)	0.002 (3)	0.003 (3)	0.008 (3)
C2	0.044 (3)	0.033 (3)	0.053 (4)	0.006 (3)	0.006 (3)	0.014 (3)
C3	0.037 (3)	0.031 (3)	0.056 (4)	−0.008 (3)	−0.001 (3)	0.009 (3)
C4	0.041 (3)	0.040 (4)	0.054 (5)	0.002 (3)	0.015 (3)	0.020 (3)
C5	0.034 (3)	0.027 (3)	0.062 (5)	0.002 (3)	−0.001 (3)	0.002 (3)
C11	0.046 (3)	0.025 (3)	0.046 (4)	0.004 (3)	0.005 (3)	−0.002 (3)
C12	0.053 (4)	0.027 (3)	0.048 (5)	0.007 (3)	0.003 (3)	0.001 (3)
N13	0.072 (4)	0.039 (3)	0.051 (4)	0.013 (3)	−0.006 (3)	0.003 (3)
C14	0.057 (4)	0.041 (4)	0.057 (5)	0.012 (3)	−0.009 (4)	0.008 (4)
C15	0.059 (4)	0.043 (4)	0.049 (5)	0.008 (3)	0.007 (4)	−0.010 (3)
C16	0.066 (4)	0.030 (3)	0.051 (5)	0.006 (3)	0.009 (4)	0.001 (3)
C21	0.031 (3)	0.029 (3)	0.044 (4)	0.001 (2)	−0.007 (3)	−0.002 (3)
C22	0.045 (3)	0.024 (3)	0.063 (5)	0.001 (3)	0.003 (3)	0.003 (3)
N23	0.054 (3)	0.041 (3)	0.048 (4)	−0.002 (3)	0.004 (3)	0.008 (3)
C24	0.058 (4)	0.040 (4)	0.055 (5)	0.004 (3)	0.011 (4)	0.010 (4)
C25	0.050 (4)	0.034 (3)	0.047 (4)	0.006 (3)	−0.002 (3)	−0.002 (3)
C26	0.042 (3)	0.028 (3)	0.051 (5)	0.004 (3)	0.000 (3)	−0.008 (3)

Geometric parameters (Å, º)

O1—C1	1.353 (8)	C12—N13	1.329 (10)
O1—C2	1.472 (8)	C12—H12	0.9500
O2—C1	1.194 (8)	N13—C14	1.353 (10)
O3—C5	1.340 (8)	C14—C15	1.402 (10)
O3—C4	1.452 (7)	C14—H14	0.9500
O4—C5	1.193 (8)	C15—C16	1.349 (12)
C1—C11	1.475 (10)	C15—H15	0.9500
C2—C3	1.537 (9)	C16—H16	0.9500
C2—H2A	0.9900	C21—C22	1.379 (8)
C2—H2B	0.9900	C21—C26	1.384 (9)
C3—C4	1.528 (10)	C22—N23	1.328 (9)
C3—H3A	0.9900	C22—H22	0.9500
C3—H3B	0.9900	N23—C24	1.364 (10)
C4—H4A	0.9900	C24—C25	1.393 (9)
C4—H4B	0.9900	C24—H24	0.9500
C5—C21	1.504 (10)	C25—C26	1.368 (10)
C11—C16	1.389 (10)	C25—H25	0.9500
C11—C12	1.400 (8)	C26—H26	0.9500
C1—O1—C2	114.8 (5)	N13—C12—C11	123.2 (7)
C5—O3—C4	116.2 (5)	N13—C12—H12	118.4

O2—C1—O1	122.4 (6)	C11—C12—H12	118.4
O2—C1—C11	125.2 (6)	C12—N13—C14	118.4 (7)
O1—C1—C11	112.4 (6)	N13—C14—C15	121.1 (7)
O1—C2—C3	105.9 (5)	N13—C14—H14	119.5
O1—C2—H2A	110.6	C15—C14—H14	119.5
C3—C2—H2A	110.6	C16—C15—C14	119.9 (7)
O1—C2—H2B	110.6	C16—C15—H15	120.0
C3—C2—H2B	110.6	C14—C15—H15	120.0
H2A—C2—H2B	108.7	C15—C16—C11	119.8 (7)
C4—C3—C2	109.8 (6)	C15—C16—H16	120.1
C4—C3—H3A	109.7	C11—C16—H16	120.1
C2—C3—H3A	109.7	C22—C21—C26	117.6 (7)
C4—C3—H3B	109.7	C22—C21—C5	123.1 (6)
C2—C3—H3B	109.7	C26—C21—C5	119.3 (6)
H3A—C3—H3B	108.2	N23—C22—C21	125.0 (7)
O3—C4—C3	111.0 (5)	N23—C22—H22	117.5
O3—C4—H4A	109.4	C21—C22—H22	117.5
C3—C4—H4A	109.4	C22—N23—C24	115.8 (6)
O3—C4—H4B	109.4	N23—C24—C25	123.6 (7)
C3—C4—H4B	109.4	N23—C24—H24	118.2
H4A—C4—H4B	108.0	C25—C24—H24	118.2
O4—C5—O3	124.4 (6)	C26—C25—C24	117.7 (7)
O4—C5—C21	123.4 (7)	C26—C25—H25	121.2
O3—C5—C21	112.2 (5)	C24—C25—H25	121.2
C16—C11—C12	117.6 (7)	C25—C26—C21	120.3 (6)
C16—C11—C1	118.2 (6)	C25—C26—H26	119.8
C12—C11—C1	124.1 (6)	C21—C26—H26	119.8

C2—O1—C1—O2	−3.1 (9)	N13—C14—C15—C16	1.9 (12)
C2—O1—C1—C11	177.9 (5)	C14—C15—C16—C11	−2.9 (11)
C1—O1—C2—C3	171.4 (5)	C12—C11—C16—C15	3.2 (10)
O1—C2—C3—C4	173.9 (5)	C1—C11—C16—C15	−178.9 (7)
C5—O3—C4—C3	−84.3 (7)	O4—C5—C21—C22	−179.8 (7)
C2—C3—C4—O3	−70.1 (7)	O3—C5—C21—C22	−1.8 (9)
C4—O3—C5—O4	−1.7 (10)	O4—C5—C21—C26	0.8 (10)
C4—O3—C5—C21	−179.6 (6)	O3—C5—C21—C26	178.7 (6)
O2—C1—C11—C16	0.4 (11)	C26—C21—C22—N23	−0.4 (11)
O1—C1—C11—C16	179.4 (6)	C5—C21—C22—N23	−179.9 (7)
O2—C1—C11—C12	178.2 (7)	C21—C22—N23—C24	0.9 (11)
O1—C1—C11—C12	−2.8 (9)	C22—N23—C24—C25	−0.9 (11)
C16—C11—C12—N13	−2.5 (10)	N23—C24—C25—C26	0.3 (12)
C1—C11—C12—N13	179.7 (7)	C24—C25—C26—C21	0.2 (10)
C11—C12—N13—C14	1.4 (11)	C22—C21—C26—C25	−0.2 (10)
C12—N13—C14—C15	−1.1 (11)	C5—C21—C26—C25	179.3 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*A*···O4^{i}	0.99	2.49	3.341 (8)	144
C16—H16···O2^{ii}	0.95	2.45	3.198 (10)	136
C24—H24···O2^{iii}	0.95	2.45	3.218 (10)	138

Symmetry codes: (i) −*x*+3, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*, −*z*; (iii) −*x*+1, −*y*, −*z*+1.