

1,4-Bis(piperidin-1-ylcarbonyl)benzene

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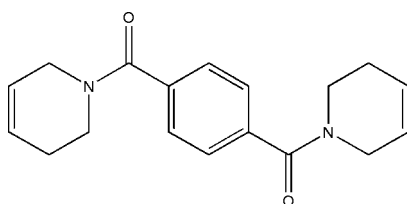
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.124; data-to-parameter ratio = 17.5.

The title compound, $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2$, has been synthesized by the reaction of terephthaloyl chloride and 1,2,3,6-tetrahydropyridine. This compound crystallizes as discrete molecular species disposed about a crystallographic centre of symmetry, such that half the molecule constitutes the asymmetric unit. The structure shows an envelope conformation for the dehydropiperidine ring with the amide carbonyl twisted out of the benzene ring plane by 57.3 (2)°.

Related literature

For background literature, see: Pang *et al.* (2006). For related structures, see: Jones *et al.* (2002).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_2$
 $M_r = 296.36$
 Monoclinic, $P2_1/n$
 $a = 9.1255$ (19) Å
 $b = 10.060$ (3) Å
 $c = 8.6941$ (16) Å
 $\beta = 106.991$ (14)°

$V = 763.3$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 295$ (2) K
 $0.35 \times 0.30 \times 0.15$ mm

Data collection

Rigaku AFC-7R diffractometer
 Absorption correction: none
 1985 measured reflections
 1753 independent reflections
 1122 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$
 3 standard reflections
 every 150 reflections
 intensity decay: 0.9%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.124$
 $S = 1.01$
 1753 reflections

100 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C9}-\text{H9}\cdots\text{O1}^i$	0.9500	2.5100	3.290 (2)	140.00

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

Data collection: *MSC/AFC7 Diffractometer Control* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC7 Diffractometer Control*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *TEXSAN*; program(s) used to refine structure: *TEXSAN*, *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN*, *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2448).

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supporting information

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S1. Comment

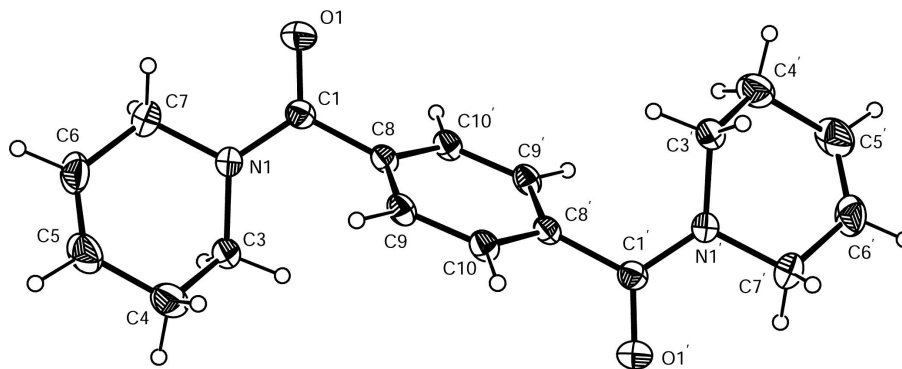
Derivatives of terephthalic acid are widely used in a range of polymer applications (Pang *et al.*, 2006). As part of our work on the synthesis of these compounds for use in new coating technologies, we have synthesized and determined the solid state structure of the title compound (I). This compound crystallizes as discrete molecular species (Fig. 1) disposed about a crystallographic centre of symmetry such that half the molecule constitutes the asymmetric unit of the crystal lattice. The bond lengths and bond angles in (I) are in accord with values for similar structures reported in the literature (Jones *et al.*, 2002). The tertiary nitrogen lies in the C1—C3—C7 plane with the sum of the C—N—C angles 359.3°. The amide plane is twisted out of the plane of the central phenyl ring as reflected in the O1—C1—C8—C10 torsion angle of -57.6 (2)°. C7 approaches coplanarity with amide plane with C7—N1—C1—O1 - 4.3 (2)°. C3 bends out of this plane with C3—N1—C1—C8 = -16.4 (2)° to give an envelope conformation to the dehydropiperidine ring. A weak intermolecular C—H...O interaction is observed between C9—H9 and the carbonyl oxygen (Table 1).

S2. Experimental

1,2,3,6-tetrahydropyridine (494.4 ml, 5.418 mmol) was added to a solution of terephthaloyl chloride (500 mg, 2.46 mmol) in toluene (50 ml) under an N₂ atmosphere with stirring. The reaction mixture was stirred and heated under reflux for 24 hr. On cooling to room temperature the mixture was washed with aqueous acid (2M HCl) and then aqueous base (2M NaOH) followed by two water washes with the organic layer collected and dried (MgSO₄). Removal of the solvent under vacuum resulted in isolation of a solid white product. Recrystallization from ethanol resulted in the formation of small white crystals (Yield 601 mg, 82.4%).

S3. Refinement

H atoms attached to carbon were constrained as riding atoms, with C—H set to 0.95 Å. $U_{\text{iso}}(\text{H})$ values were set to $1.2U_{\text{eq}}$ of the parent atom.

**Figure 1**

View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. Primed atoms were generated by symmetry $(-x, -y, 1 - z)$.

1,4-Bis(piperidin-1-ylcarbonyl)benzene

Crystal data

$C_{18}H_{20}N_2O_2$
 $M_r = 296.36$
 Monoclinic, $P2_1/n$
 Hall symbol: $-P\ 2_1n$
 $a = 9.1255\ (19)\ \text{\AA}$
 $b = 10.060\ (3)\ \text{\AA}$
 $c = 8.6941\ (16)\ \text{\AA}$
 $\beta = 106.991\ (14)^\circ$
 $V = 763.3\ (3)\ \text{\AA}^3$
 $Z = 2$

$F(000) = 316$
 $D_x = 1.289\ \text{Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.7107\ \text{\AA}$
 Cell parameters from 25 reflections
 $\theta = 12.6\text{--}16.9^\circ$
 $\mu = 0.09\ \text{mm}^{-1}$
 $T = 295\ \text{K}$
 Plate, colourless
 $0.35 \times 0.30 \times 0.15\ \text{mm}$

Data collection

Rigaku AFC7R
 diffractometer
 Radiation source: Rigaku rotating anode
 Graphite monochromator
 ω - 2θ scans
 1985 measured reflections
 1753 independent reflections
 1122 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 2.9^\circ$
 $h = -5 \rightarrow 11$
 $k = 0 \rightarrow 13$
 $l = -11 \rightarrow 10$
 3 standard reflections every 150 reflections
 intensity decay: 0.9%

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.124$
 $S = 1.01$
 1753 reflections
 100 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
 $w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.1363P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.16\ \text{e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.22\ \text{e \AA}^{-3}$

Special details

Experimental. The scan width was $(1.63 + 0.30 \tan \theta)^\circ$ with an ω scan speed of 16° per minute (up to 4 scans to achieve $I/\sigma(I) > 10$). Stationary background counts were recorded at each end of the scan, and the scan time:background time ratio was 2:1.

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > 2\sigma(F^2)$ 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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.34253 (15)	0.20263 (14)	0.59031 (15)	0.0576 (5)
N1	0.33853 (15)	0.09638 (14)	0.36082 (16)	0.0378 (4)
C1	0.28168 (18)	0.12259 (16)	0.48421 (18)	0.0360 (5)
C3	0.2956 (2)	−0.01623 (17)	0.2505 (2)	0.0417 (5)
C4	0.2517 (3)	0.0308 (2)	0.0776 (2)	0.0584 (7)
C5	0.3692 (3)	0.1256 (2)	0.0550 (2)	0.0660 (8)
C6	0.4691 (3)	0.1846 (2)	0.1752 (3)	0.0575 (7)
C7	0.4706 (2)	0.17209 (18)	0.3463 (2)	0.0448 (6)
C8	0.13664 (17)	0.05471 (15)	0.48833 (17)	0.0327 (4)
C9	0.00064 (18)	0.07421 (17)	0.36538 (17)	0.0369 (5)
C10	−0.13454 (17)	0.01931 (17)	0.37753 (17)	0.0364 (4)
H3A	0.38000	−0.07560	0.26880	0.0500*
H3B	0.21100	−0.06100	0.26960	0.0500*
H4A	0.24540	−0.04340	0.00850	0.0700*
H4B	0.15500	0.07400	0.05230	0.0700*
H5	0.37180	0.14410	−0.05130	0.0790*
H6	0.54520	0.23860	0.15170	0.0690*
H7A	0.46810	0.25840	0.38980	0.0540*
H7B	0.56190	0.12800	0.40490	0.0540*
H9	0.00080	0.12510	0.27340	0.0440*
H10	−0.22810	0.03330	0.29200	0.0420*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0557 (8)	0.0690 (9)	0.0491 (7)	−0.0176 (7)	0.0168 (6)	−0.0240 (7)
N1	0.0370 (7)	0.0413 (8)	0.0384 (7)	−0.0063 (6)	0.0161 (6)	−0.0035 (6)
C1	0.0360 (8)	0.0389 (9)	0.0324 (8)	0.0014 (7)	0.0089 (6)	−0.0019 (7)
C3	0.0473 (10)	0.0410 (9)	0.0425 (9)	−0.0050 (8)	0.0219 (8)	−0.0055 (8)
C4	0.0721 (14)	0.0630 (13)	0.0404 (10)	−0.0048 (11)	0.0168 (9)	−0.0049 (9)
C5	0.0999 (18)	0.0626 (13)	0.0434 (11)	−0.0070 (13)	0.0331 (11)	0.0084 (10)
C6	0.0686 (14)	0.0517 (11)	0.0640 (12)	−0.0070 (10)	0.0378 (11)	0.0114 (10)
C7	0.0379 (9)	0.0466 (10)	0.0517 (10)	−0.0053 (7)	0.0159 (8)	0.0057 (8)
C8	0.0322 (8)	0.0387 (8)	0.0284 (7)	0.0041 (6)	0.0109 (6)	−0.0027 (6)

C9	0.0394 (9)	0.0463 (9)	0.0258 (7)	0.0052 (7)	0.0109 (6)	0.0039 (7)
C10	0.0314 (7)	0.0481 (9)	0.0288 (7)	0.0059 (7)	0.0072 (6)	0.0007 (7)

Geometric parameters (Å, °)

O1—C1	1.228 (2)	C9—C10	1.384 (2)
N1—C1	1.347 (2)	C3—H3A	0.9500
N1—C3	1.462 (2)	C3—H3B	0.9500
N1—C7	1.462 (2)	C4—H4A	0.9500
C1—C8	1.499 (2)	C4—H4B	0.9500
C3—C4	1.514 (2)	C5—H5	0.9500
C4—C5	1.490 (4)	C6—H6	0.9500
C5—C6	1.311 (3)	C7—H7A	0.9500
C6—C7	1.489 (3)	C7—H7B	0.9500
C8—C9	1.395 (2)	C9—H9	0.9500
C8—C10 ⁱ	1.389 (2)	C10—H10	0.9600
O1…C9 ⁱⁱ	3.290 (2)	H3B…C8	2.4900
O1…H7A	2.4200	H3B…C9	2.6800
O1…H3A ⁱⁱⁱ	2.7800	H3B…H7A ^{ix}	2.5600
O1…H10 ⁱ	2.9000	H4A…O1 ^{ix}	2.7400
O1…H4A ^{iv}	2.7400	H4A…H10 ^x	2.5700
O1…H9 ⁱⁱ	2.5100	H4B…H7A ^v	2.5200
C3…C9	3.262 (3)	H6…C10 ^{iv}	2.9700
C9…C3	3.262 (3)	H6…C8 ^{xi}	2.7800
C9…O1 ^v	3.290 (2)	H6…C9 ^{xi}	3.0500
C1…H7B ⁱⁱⁱ	2.9200	H7A…O1	2.4200
C6…H3A	2.9300	H7A…H3B ^{iv}	2.5600
C6…H10 ^{vi}	3.0600	H7A…H4B ⁱⁱ	2.5200
C8…H3B	2.4900	H7B…C10 ^{vi}	3.0500
C8…H6 ^{vii}	2.7800	H7B…H10 ^{vi}	2.5800
C9…H3B	2.6800	H7B…C1 ⁱⁱⁱ	2.9200
C9…H6 ^{vii}	3.0500	H9…O1 ^v	2.5100
C10…H7B ^{viii}	3.0500	H10…C6 ^{viii}	3.0600
C10…H6 ^{ix}	2.9700	H10…H7B ^{viii}	2.5800
H3A…C6	2.9300	H10…O1 ⁱ	2.9000
H3A…O1 ⁱⁱⁱ	2.7800	H10…H4A ^x	2.5700
C1—N1—C3	125.62 (14)	H3A—C3—H3B	109.00
C1—N1—C7	119.07 (14)	C3—C4—H4A	109.00
C3—N1—C7	114.63 (14)	C3—C4—H4B	109.00
O1—C1—N1	122.12 (16)	C5—C4—H4A	109.00
O1—C1—C8	119.30 (15)	C5—C4—H4B	109.00
N1—C1—C8	118.56 (14)	H4A—C4—H4B	109.00
N1—C3—C4	110.59 (14)	C4—C5—H5	119.00
C3—C4—C5	109.82 (17)	C6—C5—H5	119.00
C4—C5—C6	122.97 (19)	C5—C6—H6	118.00
C5—C6—C7	124.0 (2)	C7—C6—H6	118.00

N1—C7—C6	111.29 (16)	N1—C7—H7A	109.00
C1—C8—C9	120.77 (14)	N1—C7—H7B	109.00
C1—C8—C10 ⁱ	119.56 (14)	C6—C7—H7A	109.00
C9—C8—C10 ⁱ	119.44 (15)	C6—C7—H7B	109.00
C8—C9—C10	119.92 (14)	H7A—C7—H7B	109.00
C8 ⁱ —C10—C9	120.64 (14)	C8—C9—H9	120.00
N1—C3—H3A	109.00	C10—C9—H9	120.00
N1—C3—H3B	109.00	C9—C10—H10	119.00
C4—C3—H3A	109.00	C8 ⁱ —C10—H10	120.00
C4—C3—H3B	109.00		
C3—N1—C1—O1	165.61 (16)	N1—C1—C8—C10 ⁱ	124.35 (17)
C3—N1—C1—C8	−16.4 (2)	N1—C3—C4—C5	47.6 (2)
C7—N1—C1—O1	−4.3 (2)	C3—C4—C5—C6	−16.9 (3)
C7—N1—C1—C8	173.66 (14)	C4—C5—C6—C7	−4.6 (4)
C1—N1—C3—C4	128.43 (19)	C5—C6—C7—N1	−5.4 (3)
C7—N1—C3—C4	−61.2 (2)	C1—C8—C9—C10	−174.84 (15)
C1—N1—C7—C6	−150.58 (16)	C10 ⁱ —C8—C9—C10	−0.4 (2)
C3—N1—C7—C6	38.4 (2)	C1—C8—C10 ⁱ —C9 ⁱ	174.91 (15)
O1—C1—C8—C9	116.88 (18)	C9—C8—C10 ⁱ —C9 ⁱ	0.4 (2)
O1—C1—C8—C10 ⁱ	−57.6 (2)	C8—C9—C10—C8 ⁱ	0.4 (2)
N1—C1—C8—C9	−61.2 (2)		

Symmetry codes: (i) $-x, -y, -z+1$; (ii) $x+1/2, -y+1/2, z+1/2$; (iii) $-x+1, -y, -z+1$; (iv) $-x+1/2, y+1/2, -z+1/2$; (v) $x-1/2, -y+1/2, z-1/2$; (vi) $x+1, y, z$; (vii) $x-1/2, -y+1/2, z+1/2$; (viii) $x-1, y, z$; (ix) $-x+1/2, y-1/2, -z+1/2$; (x) $-x, -y, -z$; (xi) $x+1/2, -y+1/2, z-1/2$.

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

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
C9—H9 $\cdots$ O1 ^v	0.9500	2.5100	3.290 (2)	140.00

Symmetry code: (v) $x-1/2, -y+1/2, z-1/2$.