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Propane-1,3-diyl bis(4-aminobenzoate)

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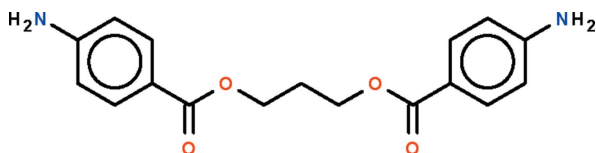
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.043; wR factor = 0.109; data-to-parameter ratio = 9.6.

Molecules of the title compound, $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_4$, lie on a twofold rotation axis that passes through the central methylene C atom. The molecules adopt a 'V' shape and the trimethylene unit assumes a *gauche-gauche* conformation. The amino N atom shows a nonplanar coordination. Adjacent molecules are connected by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds into chains running along [001]. Furthermore, $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds connect these chains into a three-dimensional network.

Related literature

For the crystal structure of 1,3-propandiyl-bis(benzoate), see: Pérez & Brisse (1977).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_4$
 $M_r = 314.33$
Monoclinic, $C2$

$a = 23.725$ (5) Å
 $b = 4.5109$ (9) Å
 $c = 8.2171$ (17) Å

$\beta = 107.173$ (3)°
 $V = 840.2$ (3) Å³
 $Z = 2$
Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹
 $T = 100$ K
 $0.35 \times 0.35 \times 0.02$ mm

Data collection

Bruker SMART APEX
diffractometer
3936 measured reflections

1082 independent reflections
788 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.090$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.109$
 $S = 0.96$
1082 reflections
113 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H11}\cdots\text{O2}^{\text{i}}$	0.86 (1)	2.15 (2)	2.958 (3)	157 (5)
$\text{N1}-\text{H12}\cdots\text{N1}^{\text{ii}}$	0.86 (1)	2.25 (1)	3.104 (3)	169 (2)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5289).

References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2009). *APEX2* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
Pérez, S. & Brisse, F. (1977). *Acta Cryst.* **B33**, 3259–3262.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

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Propane-1,3-diyl bis(4-aminobenzoate)

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S1. Comment

The chemical is a commercially available chemical that should be compatible of condensing with carbonyl compounds to yield Schiff bases; its special feature is its trimethylene portion, which assumes a *V* shape. The $C_{17}H_{18}N_2O_4$ molecule (Scheme I) lies on a twofold rotation axis that passes through the central methylene carbon atom; this symmetry element relates one 4-aminobenzoate unit to the other. The molecule assumes a *V* shape and the trimethylene portion assumes a *gauche-gauche* conformation. The amino nitrogen atom shows non-planar coordination (Fig. 1). Adjacent molecules are connected by $N-H\cdots O$ and $N-H\cdots N$ hydrogen bonds to form a three-dimensional network.

S2. Experimental

The compound was returned unchanged but in a crystalline form in an unsuccessful condensation with *o*-vanillin in ethanol medium.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [$C-H$ 0.95–0.99 Å, $U(H)$ 1.2 $U(C)$] and were included in the refinement in the riding model approximation. The amino H-atoms were located in a difference Fourier map, and were refined isotropically with a distance restraint of $N-H$ 0.86±0.01 Å. 822 Friedel pairs were merged.

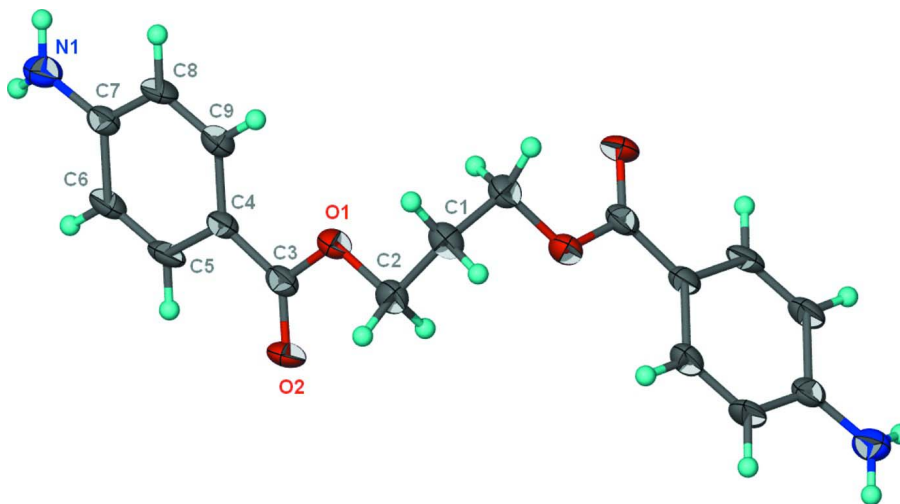


Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{17}H_{18}N_2O_4$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Propane-1,3-diyl bis(4-aminobenzoate)

Crystal data

C₁₇H₁₈N₂O₄ $M_r = 314.33$ Monoclinic, *C*2Hall symbol: *C* 2y $a = 23.725$ (5) Å $b = 4.5109$ (9) Å $c = 8.2171$ (17) Å $\beta = 107.173$ (3)° $V = 840.2$ (3) Å³ $Z = 2$ $F(000) = 332$ $D_x = 1.242$ Mg m⁻³Mo *K* α radiation, $\lambda = 0.71073$ Å

Cell parameters from 915 reflections

 $\theta = 2.6$ – 26.8 ° $\mu = 0.09$ mm⁻¹ $T = 100$ K

Plate, yellow

 $0.35 \times 0.35 \times 0.02$ mm

Data collection

Bruker SMART APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω scans

3936 measured reflections

1082 independent reflections

788 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.090$ $\theta_{\text{max}} = 27.5$ °, $\theta_{\text{min}} = 1.8$ ° $h = -30$ → 29 $k = -5$ → 5 $l = -10$ → 10

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.109$ $S = 0.96$

1082 reflections

113 parameters

3 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0523P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.42724 (7)	0.4987 (4)	0.0488 (2)	0.0262 (5)	
O2	0.35969 (8)	0.7580 (5)	-0.1476 (2)	0.0304 (5)	
N1	0.29701 (11)	1.0783 (6)	0.5367 (3)	0.0298 (6)	
C1	0.5000	0.1862 (10)	0.0000	0.0279 (10)	
H1A	0.5095	0.0569	-0.0857	0.033*	0.50
H1B	0.4905	0.0569	0.0857	0.033*	0.50
C2	0.44676 (11)	0.3705 (7)	-0.0862 (3)	0.0263 (7)	
H2A	0.4572	0.5279	-0.1562	0.032*	
H2B	0.4153	0.2454	-0.1609	0.032*	
C3	0.38135 (11)	0.6876 (7)	0.0010 (3)	0.0244 (7)	
C4	0.36179 (11)	0.7924 (7)	0.1432 (3)	0.0228 (6)	
C5	0.31600 (11)	0.9969 (7)	0.1134 (3)	0.0260 (7)	
H5	0.2991	1.0721	0.0016	0.031*	

C6	0.29486 (12)	1.0917 (7)	0.2423 (3)	0.0286 (7)
H6	0.2631	1.2289	0.2187	0.034*
C7	0.31969 (11)	0.9879 (7)	0.4094 (3)	0.0252 (7)
C8	0.36569 (11)	0.7851 (8)	0.4393 (3)	0.0295 (7)
H8	0.3830	0.7123	0.5514	0.035*
C9	0.38646 (11)	0.6885 (8)	0.3103 (3)	0.0282 (7)
H9	0.4179	0.5495	0.3338	0.034*
H11	0.3137 (17)	1.032 (12)	0.641 (2)	0.098 (16)*
H12	0.2748 (10)	1.233 (4)	0.519 (3)	0.025 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0204 (9)	0.0351 (12)	0.0206 (9)	0.0041 (10)	0.0021 (7)	0.0031 (9)
O2	0.0239 (10)	0.0453 (13)	0.0165 (9)	0.0042 (10)	-0.0024 (7)	0.0029 (10)
N1	0.0272 (14)	0.0364 (17)	0.0227 (13)	0.0056 (12)	0.0025 (11)	0.0016 (12)
C1	0.021 (2)	0.030 (3)	0.029 (2)	0.000	0.0034 (16)	0.000
C2	0.0205 (13)	0.0329 (17)	0.0243 (14)	-0.0040 (13)	0.0046 (11)	-0.0024 (13)
C3	0.0172 (13)	0.0302 (17)	0.0224 (13)	-0.0048 (13)	0.0005 (11)	0.0009 (13)
C4	0.0163 (12)	0.0304 (17)	0.0183 (12)	-0.0025 (12)	-0.0001 (10)	0.0023 (11)
C5	0.0190 (13)	0.0322 (17)	0.0193 (13)	-0.0007 (14)	-0.0058 (11)	0.0043 (13)
C6	0.0191 (14)	0.0368 (19)	0.0236 (14)	0.0029 (13)	-0.0031 (12)	0.0038 (13)
C7	0.0186 (13)	0.0335 (18)	0.0202 (13)	-0.0060 (14)	0.0006 (10)	0.0005 (13)
C8	0.0218 (14)	0.043 (2)	0.0179 (13)	0.0039 (14)	-0.0026 (11)	0.0090 (14)
C9	0.0181 (14)	0.0397 (19)	0.0237 (14)	0.0030 (14)	0.0012 (11)	0.0050 (14)

Geometric parameters (Å, °)

O1—C3	1.347 (3)	C3—C4	1.457 (4)
O1—C2	1.444 (3)	C4—C5	1.391 (4)
O2—C3	1.219 (3)	C4—C9	1.405 (3)
N1—C7	1.372 (3)	C5—C6	1.368 (4)
N1—H11	0.858 (10)	C5—H5	0.9500
N1—H12	0.861 (10)	C6—C7	1.405 (4)
C1—C2	1.503 (4)	C6—H6	0.9500
C1—C2 ⁱ	1.503 (4)	C7—C8	1.389 (4)
C1—H1A	0.9900	C8—C9	1.365 (4)
C1—H1B	0.9900	C8—H8	0.9500
C2—H2A	0.9900	C9—H9	0.9500
C2—H2B	0.9900		
C3—O1—C2	116.39 (19)	C5—C4—C9	118.1 (2)
C7—N1—H11	121 (3)	C5—C4—C3	119.4 (2)
C7—N1—H12	118.1 (19)	C9—C4—C3	122.4 (2)
H11—N1—H12	116 (4)	C6—C5—C4	121.1 (2)
C2—C1—C2 ⁱ	112.9 (4)	C6—C5—H5	119.4
C2—C1—H1A	109.0	C4—C5—H5	119.4
C2 ⁱ —C1—H1A	109.0	C5—C6—C7	120.6 (3)

C2—C1—H1B	109.0	C5—C6—H6	119.7
C2 ⁱ —C1—H1B	109.0	C7—C6—H6	119.7
H1A—C1—H1B	107.8	N1—C7—C8	121.7 (2)
O1—C2—C1	105.94 (18)	N1—C7—C6	120.0 (3)
O1—C2—H2A	110.5	C8—C7—C6	118.2 (2)
C1—C2—H2A	110.5	C9—C8—C7	121.2 (3)
O1—C2—H2B	110.5	C9—C8—H8	119.4
C1—C2—H2B	110.5	C7—C8—H8	119.4
H2A—C2—H2B	108.7	C8—C9—C4	120.7 (3)
O2—C3—O1	121.5 (2)	C8—C9—H9	119.7
O2—C3—C4	125.4 (3)	C4—C9—H9	119.7
O1—C3—C4	113.1 (2)		
C3—O1—C2—C1	176.5 (2)	C3—C4—C5—C6	177.4 (3)
C2 ⁱ —C1—C2—O1	-71.92 (18)	C4—C5—C6—C7	1.1 (4)
C2—O1—C3—O2	-3.8 (4)	C5—C6—C7—N1	-178.2 (3)
C2—O1—C3—C4	176.2 (2)	C5—C6—C7—C8	-0.6 (4)
O2—C3—C4—C5	-2.2 (4)	N1—C7—C8—C9	177.6 (3)
O1—C3—C4—C5	177.8 (3)	C6—C7—C8—C9	0.1 (5)
O2—C3—C4—C9	176.1 (3)	C7—C8—C9—C4	0.1 (5)
O1—C3—C4—C9	-4.0 (4)	C5—C4—C9—C8	0.3 (4)
C9—C4—C5—C6	-0.9 (4)	C3—C4—C9—C8	-177.9 (3)

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H11 \cdots O2 ⁱⁱ	0.86 (1)	2.15 (2)	2.958 (3)	157 (5)
N1—H12 \cdots N1 ⁱⁱⁱ	0.86 (1)	2.25 (1)	3.104 (3)	169 (2)

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