

4,4'-Azinodibenzoic acid

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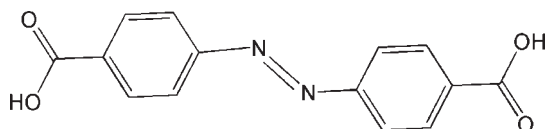
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.040; wR factor = 0.100; data-to-parameter ratio = 14.8.

The title compound, $\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_4$, shows crystallographic inversion symmetry and has one half-molecule in the asymmetric unit. In the crystal, molecules are linked into chains running along the cell diagonal by $\text{O}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions.

Related literature

For the use of azodibenzoate-based systems as bridging aromatic carboxylate ligands in coordination networks, see: Chen *et al.* (2008).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{10}\text{N}_2\text{O}_4$
 $M_r = 270.16$
Triclinic, $P\bar{1}$
 $a = 3.772$ (2) Å

$b = 6.322$ (5) Å
 $c = 12.692$ (3) Å
 $\alpha = 79.323$ (5)°
 $\beta = 88.199$ (4)°

$\gamma = 88.435$ (5)°
 $V = 297.2$ (3) Å³
 $Z = 1$
Mo $K\alpha$ radiation

$\mu = 0.11$ mm⁻¹
 $T = 293$ K
 $0.16 \times 0.14 \times 0.12$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick 1996)
 $T_{\min} = 0.962$, $T_{\max} = 0.971$

2173 measured reflections
1351 independent reflections
786 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.100$
 $S = 0.86$
1351 reflections

91 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1A}\cdots\text{O2}^i$	0.82	1.81	2.6181 (17)	170

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *S SAINT* (Bruker, 1998); data reduction: *S SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5033).

References

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supplementary materials

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Comment

Azodibenzoate-based systems represent one type of bridging aromatic carboxylate ligand employed in the generation of coordination networks (Chen *et al.*, 2008). There is half a molecule in the asymmetric unit of the title compound (Fig. 1). In the crystal, molecules are linked into chains by O—H...O hydrogen-bonding interactions (Table 2).

Experimental

A mixture of ZnCl₂·2H₂O (0.5 mmol), 4,4'-azodibenzoic acid (0.5 mmol), and H₂O (500 mmol) was heated at 140 °C for 3 days. After the mixture was slowly cooled to room temperature, pale yellow crystals of the title compound were yielded (22% yield).

Refinement

All H atoms on C atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding, with $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{carrier})$.

Figures

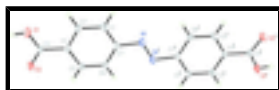


Fig. 1. The structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Symmetry code: (i) $-x, -y, -z$.

4,4'-Azinodibenzoic acid

Crystal data

C₁₄H₁₀N₂O₄

$M_r = 270.16$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 3.772\ (2)\ \text{\AA}$

$b = 6.322\ (5)\ \text{\AA}$

$c = 12.692\ (3)\ \text{\AA}$

$\alpha = 79.323\ (5)^\circ$

$\beta = 88.199\ (4)^\circ$

$\gamma = 88.435\ (5)^\circ$

$V = 297.2\ (3)\ \text{\AA}^3$

$Z = 1$

$F_{000} = 140$

$D_x = 1.509\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1351 reflections

$\theta = 3.0\text{--}29.0^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, pale yellow

$0.16 \times 0.14 \times 0.12\ \text{mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1351 independent reflections
Radiation source: fine-focus sealed tube	786 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.017$
$T = 293$ K	$\theta_{\text{max}} = 29.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 3.3^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick 1996)	$h = -5 \rightarrow 4$
$T_{\text{min}} = 0.962$, $T_{\text{max}} = 0.971$	$k = -8 \rightarrow 5$
2173 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.100$	$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2]$
$S = 0.86$	where $P = (F_o^2 + 2F_c^2)/3$
1351 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
91 parameters	$\Delta\rho_{\text{max}} = 0.19 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0410 (4)	-0.0391 (3)	0.23081 (12)	0.0356 (4)
H1	-0.0514	-0.1767	0.2450	0.043*
C2	0.1284 (4)	0.0608 (3)	0.31492 (12)	0.0343 (4)
H2	0.0924	-0.0090	0.3855	0.041*
C3	0.2692 (4)	0.2646 (2)	0.29319 (11)	0.0292 (4)

C4	0.3691 (4)	0.3697 (2)	0.38301 (12)	0.0315 (4)
C5	0.3203 (4)	0.3700 (2)	0.18790 (12)	0.0337 (4)
H5	0.4147	0.5070	0.1738	0.040*
C6	0.2312 (4)	0.2720 (3)	0.10404 (12)	0.0355 (4)
H6	0.2640	0.3426	0.0334	0.043*
C7	0.0913 (4)	0.0659 (2)	0.12647 (12)	0.0315 (4)
N1	-0.0103 (4)	-0.0518 (2)	0.04644 (9)	0.0372 (4)
O1	0.2870 (4)	0.2717 (2)	0.47782 (9)	0.0545 (4)
H1A	0.3524	0.3417	0.5217	0.082*
O2	0.5255 (3)	0.54435 (18)	0.36449 (9)	0.0453 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0452 (10)	0.0306 (9)	0.0329 (9)	-0.0099 (7)	-0.0026 (7)	-0.0095 (7)
C2	0.0438 (10)	0.0360 (9)	0.0243 (8)	-0.0081 (8)	-0.0014 (7)	-0.0072 (7)
C3	0.0321 (9)	0.0322 (8)	0.0261 (8)	-0.0037 (7)	-0.0030 (6)	-0.0115 (7)
C4	0.0377 (9)	0.0334 (9)	0.0253 (8)	-0.0060 (7)	-0.0040 (6)	-0.0092 (7)
C5	0.0436 (10)	0.0295 (8)	0.0299 (9)	-0.0082 (7)	-0.0009 (7)	-0.0092 (7)
C6	0.0465 (10)	0.0376 (9)	0.0240 (8)	-0.0067 (7)	-0.0030 (7)	-0.0084 (7)
C7	0.0333 (9)	0.0349 (9)	0.0299 (9)	-0.0023 (7)	-0.0048 (7)	-0.0144 (7)
N1	0.0471 (8)	0.0382 (8)	0.0300 (7)	-0.0081 (7)	-0.0058 (7)	-0.0143 (6)
O1	0.0875 (10)	0.0544 (8)	0.0254 (6)	-0.0314 (7)	-0.0023 (6)	-0.0128 (6)
O2	0.0674 (8)	0.0397 (7)	0.0321 (7)	-0.0215 (6)	-0.0027 (6)	-0.0119 (5)

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

C1—C7	1.377 (2)	C4—O1	1.2800 (18)
C1—C2	1.389 (2)	C5—C6	1.381 (2)
C1—H1	0.9300	C5—H5	0.9300
C2—C3	1.384 (2)	C6—C7	1.396 (2)
C2—H2	0.9300	C6—H6	0.9300
C3—C5	1.388 (2)	C7—N1	1.4327 (19)
C3—C4	1.485 (2)	N1—N1 ⁱ	1.239 (2)
C4—O2	1.246 (2)	O1—H1A	0.8200
C7—C1—C2	119.91 (16)	C6—C5—C3	120.22 (15)
C7—C1—H1	120.0	C6—C5—H5	119.9
C2—C1—H1	120.0	C3—C5—H5	119.9
C3—C2—C1	119.70 (15)	C5—C6—C7	119.22 (15)
C3—C2—H2	120.2	C5—C6—H6	120.4
C1—C2—H2	120.2	C7—C6—H6	120.4
C2—C3—C5	120.28 (14)	C1—C7—C6	120.67 (14)
C2—C3—C4	119.73 (14)	C1—C7—N1	115.05 (15)
C5—C3—C4	119.99 (15)	C6—C7—N1	124.28 (14)
O2—C4—O1	123.10 (14)	N1 ⁱ —N1—C7	114.04 (17)
O2—C4—C3	120.27 (14)	C4—O1—H1A	109.5
O1—C4—C3	116.63 (15)		

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

supplementary materials

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A \cdots O2 ⁱⁱ	0.82	1.81	2.6181 (17)	170

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

Fig. 1

