Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,4′-Azinodibenzoic acid

Qun-Di Yu[a] and Yun-Yu Liu[b]*

[a]Food Science and Pharmacy College, Zhejiang Ocean University, Zhoushan 316000, People's Republic of China, and [b]Department of Chemistry, Northeast Normal University, Changchun 130024, People's Republic of China
Correspondence e-mail: yunyuliu888@yahoo.com.cn

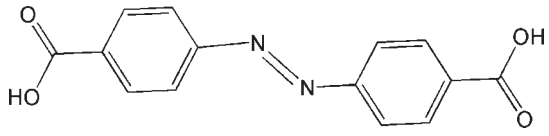
Received 13 August 2009; accepted 20 August 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.100; data-to-parameter ratio = 14.8.

The title compound, $C_{14}H_{10}N_2O_4$, 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 O—H···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

$C_{14}H_{10}N_2O_4$
M_r = 270.16
Triclinic, $P\overline{1}$
a = 3.772 (2) Å
b = 6.322 (5) Å
c = 12.692 (3) Å
α = 79.323 (5)°
β = 88.199 (4)°
γ = 88.435 (5)°
V = 297.2 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.16 × 0.14 × 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_{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
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···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: *SAINT* (Bruker, 1998); data reduction: *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*.

We thank Zhejiang Ocean University and the Science Foundation for Young Teachers of Northeast Normal University (No. 20080305) for support.

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

References

Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, Z.-F., Zhang, Z.-L., Tan, Y.-H., Tang, Y.-Z., Fun, H.-K., Zhou, Z.-Y., Abrahams, B. F. & Liang, H. (2008). *CrystEngComm*, **10**, 217–231.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2326 [doi:10.1107/S1600536809033224]

4,4′-Azinodibenzoic acid

Qun-Di Yu and Yun-Yu Liu

S1. 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).

S2. Experimental

A mixture of $ZnCl_2·2H_2O$ (0.5 mmol), 4,4′-azodibenzoatic acid (0.5 mmol), and H_2O (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).

S3. Refinement

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

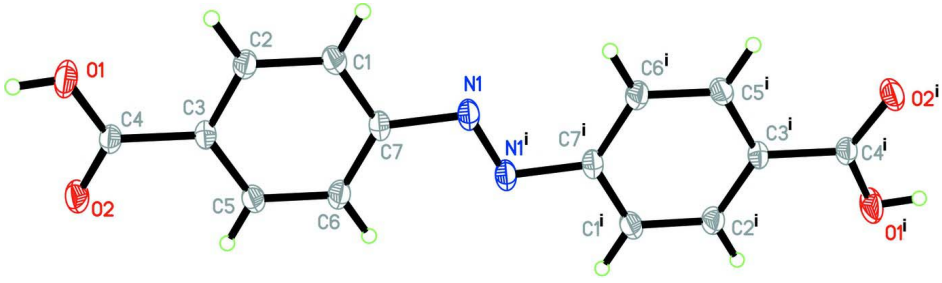


Figure 1

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

$C_{14}H_{10}N_2O_4$
M_r = 270.16
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 3.772 (2) Å
b = 6.322 (5) Å
c = 12.692 (3) Å
α = 79.323 (5)°
β = 88.199 (4)°
γ = 88.435 (5)°
V = 297.2 (3) Å^3
Z = 1
F(000) = 140
D_x = 1.509 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1351 reflections
θ = 3.0–29.0°
μ = 0.11 mm^{-1}

T = 293 K
Block, pale yellow

0.16 × 0.14 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
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_{int} = 0.017
θ_{max} = 29.0°, θ_{min} = 3.3°
h = −5→4
k = −8→5
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.100
S = 0.86
1351 reflections
91 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
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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 (Å²)

	x	y	z	U_{iso}*/U_{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 (Å²)

	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 (Å, º)

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 code: (i) −*x*, −*y*, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{ii}	0.82	1.81	2.6181 (17)	170

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