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4,4′-Diazenediyldiphthalic acid dihydrate

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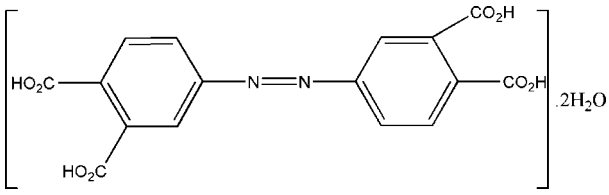
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.061; wR factor = 0.194; data-to-parameter ratio = 11.4.

In the crystal structure of the title compound, $C_{16}H_{10}N_2O_8\cdot 2H_2O$, the centrosymmetric organic molecules and water molecules interact by way of O—H···O hydrogen bonds.

Related literature

For background, see: Carlucci *et al.* (2000).



Experimental

Crystal data

$C_{16}H_{10}N_2O_8\cdot 2H_2O$
M_r = 394.29
Triclinic, $P\overline{1}$
a = 5.484 (2) Å
b = 6.491 (3) Å
c = 12.820 (5) Å
α = 86.779 (6)°
β = 82.921 (5)°
γ = 71.956 (6)°
V = 430.5 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 298 (2) K
0.28 × 0.24 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.965, T_{max} = 0.976
2278 measured reflections
1533 independent reflections
1378 reflections with $I > 2\sigma(I)$
R_{int} = 0.012

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.194
S = 1.10
1533 reflections
135 parameters
5 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.07 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WB···O3^{i}	0.84 (3)	1.95 (3)	2.782 (3)	173 (4)
O1W—H1WA···O2^{ii}	0.84 (3)	2.43 (2)	3.070 (3)	134 (3)
O1W—H1WA···O3^{iii}	0.84 (3)	2.20 (2)	2.895 (3)	139 (3)
O1—H1···O1W^{iv}	0.82	1.76	2.577 (2)	173
O4—H4A···O2^{v}	0.82	1.88	2.692 (2)	171

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2004); 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: HB2593).

References

Bruker (2004). *APEX2* (Version 6.12), *SAINT* (Version 6.12) and *SHELXTL* (Version 1.22). Bruker AXS Inc., Madison, Wisconsin, USA.
Carlucci, L., Ciani, G., Proserpion, D. M. & Rizzato, S. (2000). *Angew. Chem. Int. Ed.* **39**, 1506–1510.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.

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supplementary materials

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4,4'-Diazenediyldiphthalic acid dihydrate

J. Wang, L. Lu, J.-W. Bai and B.-Z. Zhao

Comment

In an attempt to prepare a Zn-containing coordination polymer (Carlucci *et al.*, 2000), the title compound, (I), arose as an unexpected product.

The complete organic molecule (Fig. 1) is generated by inversion at the mid-point of the central N=N bond and a water molecule of crystallization completes the structure. The components interact by way of O—H···O hydrogen bonds to generate a two-dimesnional supramolecular architecture (Table 2).

Experimental

4,4'-Azo-diphthalic acid (0.035 g, 0.16 mmol), NaOH (0.048 g, 0.12 mmol) and $ZnSO_4$ (0.0028 g, 0.014 mmol) were added to a mixed solvent of ethanol and acetonitrile. The mixture was heated for five hours under reflux with stirring. The resulting liquid was then filtered to give a solution which was infiltrated by diethyl ether in a closed vessel. After a week, colourless blocks of (I) were recovered.

Refinement

The C-bound and carboxylic acid H atoms were placed in calculated positions [C—H = 0.93 Å, O—H = 0.89 Å] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(carrier). The water H atoms were found in a dffierent map and refined with O—H = 0.84 (2)Å and U_{iso}(H) = 1.5U_{eq}(O). The maximum difference peak is located on the N=N bond, 1.07Å from N1.

Figures

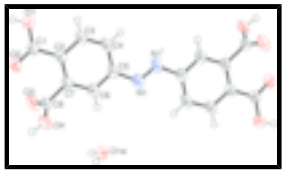

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids (arbitaray spheres for the H atoms). Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*.

4,4'-Diazenediyldiphthalic acid dihydrate

Crystal data

$C_{16}H_{10}N_2O_8·2H_2O$	Z = 1
M_r = 394.29	F_{000} = 204
Triclinic, $P\overline{1}$	D_x = 1.521 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å

a = 5.484 (2) Å	Cell parameters from 1533 reflections
b = 6.491 (3) Å	θ = 1.6–25.2º
c = 12.820 (5) Å	μ = 0.13 mm^{-1}
α = 86.779 (6)º	T = 298 (2) K
β = 82.921 (5)º	Block, colourless
γ = 71.956 (6)º	0.28 × 0.24 × 0.19 mm
V = 430.5 (3) $Å^3$	

Data collection

Bruker APEXII CCD diffractometer	1533 independent reflections
Radiation source: fine-focus sealed tube	1378 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.012
Detector resolution: 0 pixels mm^{-1}	θ_{max} = 25.2º
T = 298(2) K	θ_{min} = 1.6º
ω scans	$h = -6 \rightarrow 6$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$k = -4 \rightarrow 7$
T_{min} = 0.965, T_{max} = 0.976	$l = -15 \rightarrow 15$
2278 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.061	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.194	$w = 1/[\sigma^2(F_o^2) + (0.1258P)^2 + 0.1677P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.10	$(\Delta/\sigma)_{max} < 0.001$
1533 reflections	$\Delta\rho_{max}$ = 1.07 e $Å^{-3}$
135 parameters	$\Delta\rho_{min}$ = −0.23 e $Å^{-3}$
5 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2$sigma(F^2) is used only for calculat-

ing *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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.2911 (4)	−0.1176 (3)	0.13083 (13)	0.0536 (6)
O1W	0.8546 (4)	0.6214 (3)	0.15345 (14)	0.0490 (5)
O4	0.7036 (3)	0.1458 (4)	0.07814 (13)	0.0557 (6)
H4A	0.7194	0.1419	0.0138	0.084*
O1	−0.0028 (4)	−0.1294 (3)	0.26625 (14)	0.0546 (6)
H1	−0.0448	−0.2031	0.2261	0.082*
O3	0.2965 (4)	0.3246 (4)	0.05734 (13)	0.0579 (6)
C5	0.4209 (5)	0.3623 (4)	0.39868 (19)	0.0476 (7)
C2	0.2568 (5)	0.0939 (4)	0.27905 (18)	0.0426 (6)
N1	0.5149 (5)	0.5055 (4)	0.45109 (17)	0.0566 (7)
C8	0.4608 (5)	0.2336 (4)	0.11278 (17)	0.0391 (6)
C7	0.4044 (4)	0.2211 (4)	0.23069 (17)	0.0390 (6)
C6	0.4860 (5)	0.3540 (4)	0.29044 (19)	0.0450 (6)
H4	0.5847	0.4378	0.2578	0.054*
C1	0.1826 (5)	−0.0600 (4)	0.21811 (18)	0.0417 (6)
C4	0.2685 (7)	0.2389 (5)	0.4474 (2)	0.0604 (8)
H3	0.2205	0.2464	0.5196	0.072*
C3	0.1906 (6)	0.1070 (5)	0.3879 (2)	0.0577 (8)
H2	0.0911	0.0241	0.4208	0.069*
H1WA	0.748 (5)	0.683 (5)	0.111 (2)	0.087*
H1WB	0.981 (5)	0.525 (5)	0.124 (2)	0.087*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0578 (11)	0.0728 (13)	0.0361 (10)	−0.0289 (10)	0.0053 (8)	−0.0230 (9)
O1W	0.0522 (11)	0.0558 (11)	0.0423 (10)	−0.0195 (8)	−0.0075 (8)	−0.0064 (8)
O4	0.0476 (11)	0.0861 (14)	0.0287 (9)	−0.0145 (9)	0.0008 (8)	−0.0083 (9)
O1	0.0639 (12)	0.0696 (13)	0.0405 (10)	−0.0368 (10)	0.0040 (8)	−0.0129 (9)
O3	0.0552 (11)	0.0787 (14)	0.0293 (9)	−0.0048 (10)	−0.0052 (8)	−0.0039 (9)
C5	0.0587 (15)	0.0555 (15)	0.0333 (13)	−0.0220 (12)	−0.0058 (11)	−0.0109 (11)
C2	0.0483 (13)	0.0531 (14)	0.0279 (12)	−0.0178 (11)	−0.0012 (10)	−0.0073 (10)
N1	0.0789 (16)	0.0662 (15)	0.0329 (10)	−0.0319 (13)	−0.0078 (10)	−0.0109 (10)
C8	0.0463 (13)	0.0451 (12)	0.0276 (11)	−0.0167 (10)	−0.0006 (9)	−0.0075 (9)
C7	0.0422 (12)	0.0474 (13)	0.0266 (11)	−0.0117 (10)	−0.0031 (9)	−0.0063 (9)
C6	0.0544 (14)	0.0510 (14)	0.0340 (13)	−0.0222 (11)	−0.0040 (10)	−0.0060 (10)
C1	0.0438 (13)	0.0501 (13)	0.0322 (12)	−0.0154 (10)	−0.0028 (9)	−0.0068 (10)
C4	0.087 (2)	0.0779 (19)	0.0247 (12)	−0.0397 (17)	0.0024 (12)	−0.0093 (12)
C3	0.0796 (19)	0.0784 (19)	0.0286 (13)	−0.0466 (16)	0.0051 (12)	−0.0093 (12)

Geometric parameters (Å, °)

O2—C1	1.221 (3)	C2—C7	1.398 (3)
O1W—H1WA	0.84 (3)	C2—C3	1.399 (3)
O1W—H1WB	0.84 (3)	C2—C1	1.481 (3)
O4—C8	1.307 (3)	N1—N1^{i}	1.245 (4)
O4—H4A	0.8200	C8—C7	1.508 (3)
O1—C1	1.310 (3)	C7—C6	1.389 (3)
O1—H1	0.8200	C6—H4	0.9300
O3—C8	1.203 (3)	C4—C3	1.370 (4)
C5—C6	1.389 (4)	C4—H3	0.9300
C5—C4	1.400 (4)	C3—H2	0.9300
C5—N1	1.426 (4)		
H1WA—O1W—H1WB	111.0 (17)	C6—C7—C8	117.9 (2)
C8—O4—H4A	109.5	C2—C7—C8	121.7 (2)
C1—O1—H1	109.5	C7—C6—C5	120.3 (2)
C6—C5—C4	119.8 (2)	C7—C6—H4	119.8
C6—C5—N1	115.0 (2)	C5—C6—H4	119.8
C4—C5—N1	125.2 (2)	O2—C1—O1	123.9 (2)
C7—C2—C3	118.5 (2)	O2—C1—C2	121.5 (2)
C7—C2—C1	121.2 (2)	O1—C1—C2	114.5 (2)
C3—C2—C1	120.2 (2)	C3—C4—C5	119.5 (2)
N1^{i}—N1—C5	115.4 (3)	C3—C4—H3	120.3
O3—C8—O4	123.9 (2)	C5—C4—H3	120.3
O3—C8—C7	122.2 (2)	C4—C3—C2	121.7 (3)
O4—C8—C7	113.8 (2)	C4—C3—H2	119.1
C6—C7—C2	120.2 (2)	C2—C3—H2	119.1

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1WB···O3^{ii}	0.84 (3)	1.95 (3)	2.782 (3)	173 (4)
O1W—H1WA···O2^{iii}	0.84 (3)	2.43 (2)	3.070 (3)	134 (3)
O1W—H1WA···O3^{iv}	0.84 (3)	2.20 (2)	2.895 (3)	139 (3)
O1—H1···O1W^{v}	0.82	1.76	2.577 (2)	173
O4—H4A···O2^{vi}	0.82	1.88	2.692 (2)	171

Symmetry codes: (ii) *x*+1, *y*, *z*; (iii) *x*, *y*+1, *z*; (iv) −*x*+1, −*y*+1, −*z*; (v) *x*−1, *y*−1, *z*; (vi) −*x*+1, −*y*, −*z*.

Fig. 1