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3,3′-Diazenediyldiphthalic acid dihydrate

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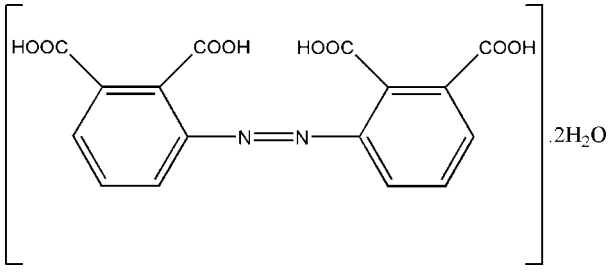
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.084; wR factor = 0.260; data-to-parameter ratio = 11.3.

In the crystal structure of the title compound, $C_{16}H_{10}N_2O_8\cdot 2H_2O$, the organic molecule is located on a centre of symmetry. The two benzene rings are parallel, but not coplanar, as indicated by N=N—C—C torsion angles involving the azo group of 12.1 (5) and −168.2 (3)°. The organic molecule and the water molecule are linked by O—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For related literature, 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 = 6.6914 (14) Å
b = 7.8566 (17) Å
c = 8.7665 (19) Å
α = 95.658 (3)°
β = 100.628 (3)°
γ = 105.601 (3)°
V = 430.90 (16) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 298 (2) K
0.27 × 0.19 × 0.15 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.966, T_{max} = 0.981
2297 measured reflections
1530 independent reflections
1248 reflections with $I > 2\sigma(I)$
R_{int} = 0.009

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.084
$wR(F^2)$ = 0.260
S = 1.11
1530 reflections
135 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.04 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
O1W—H1WA···O1	0.85 (3)	2.08 (2)	2.875 (4)	155 (4)
O4—H4···O1W^{i}	0.82	1.81	2.631 (4)	177
O1W—H1WB···O3^{ii}	0.85 (4)	2.62 (4)	3.104 (5)	117 (4)
O2—H2···O3^{iii}	0.82	2.00	2.657 (4)	137

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to SiChuan University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2247).

References

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supplementary materials

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3,3'-Diazenediyldiphthalic acid dihydrate

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Comment

In an attempt to prepare a Cd-containing coordination polmyer (Carlucci *et al.*, 2000), the title compound was obtained as an unexpected product.

The complete organic molecule (Fig. 1) is generated by inversion at the midpoint of the central N—N bond and a water molecule of crystallization completes the crystal structure. The components interact through O—H···O hydrogen bonds (Table 1) to generate a three-dimensional architecture.

Experimental

$CdSO_4$ (0.033 g, 0.012 mmol), 2,2',3,3'-diazenediyldiphthalic acid (0.026 g, 0.014 mmol) and NaOH (0.048 mmol, 0.12 mmol), were added in a mixed solvent of acetonitrile and the mixture was heated for ten hours under reflux. During the process stirring and influx were required. The resultant was then filtered to give a pure solution which was infiltrated by diethyl ether freely in a closed vessel. Single crystals suitable for X-ray diffraction were obtained for a week.

Refinement

C-bound H atoms were placed at calculated positions (C—H = 0.93 Å) and were treated as riding, with U_{iso}(H) = 1.2U_{eq}(C). Carboxy H atoms were constrained with O—H = 0.82 Å and U_{iso}(H) = 1.2U_{eq}(O), but each O—H group was allowed to rotate freely about its C—O bond. Water H atoms were tentatively located in a difference Fourier map and were refined, with distance restraints of O–H = 0.85 (1) Å and H···H = 1.39 (1) Å, and with U_{iso}(H) = 1.2U_{eq}(O). The maximum residual peak is located 1.34 Å from Br1.

Figures

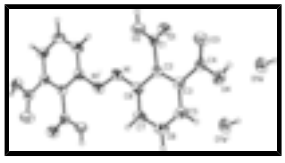

Fig. 1. The molecular structure of (I), showing the atomic numbering scheme. Non-H atoms are shown as 30% probability displacement ellipsoids. [symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*]

3,3'-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 = 1.519 Mg m^{-3}

Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.6914 (14) Å	Cell parameters from 1248 reflections
b = 7.8566 (17) Å	θ = 2.4–25.2º
c = 8.7665 (19) Å	μ = 0.13 mm^{-1}
α = 95.658 (3)º	T = 298 (2) K
β = 100.628 (3)º	Block, colourless
γ = 105.601 (3)º	0.27 × 0.19 × 0.15 mm
V = 430.90 (16) Å^3	

Data collection

Bruker APEXII area-detector diffractometer	1530 independent reflections
Radiation source: fine-focus sealed tube	1248 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.009
T = 298(2) K	θ_{max} = 25.2º
φ and ω scan	θ_{min} = 2.4º
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)	$h = -7\rightarrow8$
T_{min} = 0.966, T_{max} = 0.981	$k = -6\rightarrow9$
2297 measured reflections	$l = -10\rightarrow10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.084	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.260	$w = 1/[\sigma^2(F_o^2) + (0.16P)^2 + 0.1722P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.11	$(\Delta/\sigma)_{max} < 0.001$
1530 reflections	$\Delta\rho_{max}$ = 1.04 e Å^{-3}
135 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
3 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}
C1	0.1413 (5)	0.2366 (4)	0.1954 (4)	0.0537 (8)
C2	0.0818 (5)	0.2502 (4)	0.3535 (3)	0.0505 (8)
C3	−0.1122 (5)	0.1482 (4)	0.3774 (4)	0.0541 (8)
C4	−0.2663 (5)	0.0169 (4)	0.2462 (4)	0.0558 (8)
C5	−0.1571 (6)	0.1709 (5)	0.5259 (4)	0.0688 (10)
H5	−0.2867	0.1042	0.5425	0.083*
C6	−0.0133 (7)	0.2900 (6)	0.6479 (4)	0.0775 (11)
H6	−0.0472	0.3046	0.7457	0.093*
C7	0.1814 (7)	0.3884 (5)	0.6264 (4)	0.0741 (11)
H7	0.2792	0.4681	0.7095	0.089*
C8	0.2295 (5)	0.3672 (4)	0.4798 (4)	0.0585 (9)
N1	0.4269 (5)	0.4597 (4)	0.4434 (3)	0.0664 (9)
O1	0.1017 (4)	0.3301 (3)	0.1000 (3)	0.0728 (8)
O2	0.2414 (5)	0.1183 (4)	0.1782 (3)	0.0797 (9)
H2	0.2913	0.1285	0.0997	0.120*
O3	−0.2418 (5)	0.0128 (4)	0.1130 (3)	0.0933 (11)
O4	−0.4243 (4)	−0.0911 (4)	0.2846 (3)	0.0759 (8)
H4	−0.4939	−0.1658	0.2080	0.114*
O1W	0.3401 (5)	0.6712 (4)	0.0411 (4)	0.0934 (10)
H1WA	0.306 (7)	0.563 (3)	0.057 (8)	0.140*
H1WB	0.228 (5)	0.705 (6)	0.025 (8)	0.140*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0446 (15)	0.0528 (17)	0.0533 (17)	0.0005 (13)	0.0063 (12)	0.0074 (14)
C2	0.0540 (16)	0.0461 (15)	0.0499 (16)	0.0139 (13)	0.0078 (13)	0.0087 (12)
C3	0.0584 (18)	0.0510 (17)	0.0584 (18)	0.0205 (14)	0.0168 (14)	0.0137 (13)
C4	0.0509 (17)	0.0527 (17)	0.0627 (19)	0.0066 (14)	0.0185 (14)	0.0165 (14)
C5	0.078 (2)	0.075 (2)	0.070 (2)	0.0326 (19)	0.0336 (18)	0.0233 (18)
C6	0.101 (3)	0.085 (3)	0.056 (2)	0.038 (2)	0.028 (2)	0.0067 (18)
C7	0.096 (3)	0.070 (2)	0.0527 (19)	0.031 (2)	0.0049 (17)	−0.0023 (16)
C8	0.0645 (19)	0.0506 (17)	0.0566 (18)	0.0164 (15)	0.0064 (14)	0.0045 (13)
N1	0.0659 (18)	0.0644 (17)	0.0545 (16)	0.0066 (14)	0.0004 (12)	0.0012 (12)
O1	0.0810 (17)	0.0723 (16)	0.0667 (15)	0.0140 (13)	0.0225 (12)	0.0301 (13)
O2	0.100 (2)	0.100 (2)	0.0593 (15)	0.0501 (17)	0.0301 (13)	0.0201 (13)
O3	0.0813 (18)	0.093 (2)	0.0686 (18)	−0.0311 (15)	0.0182 (13)	−0.0046 (14)
O4	0.0589 (15)	0.0733 (17)	0.0910 (19)	0.0041 (12)	0.0260 (13)	0.0159 (13)
O1W	0.0775 (19)	0.0712 (18)	0.116 (2)	0.0007 (14)	0.0076 (17)	0.0286 (17)

Geometric parameters (Å, °)

C1—O1	1.205 (4)	C6—C7	1.381 (5)
C1—O2	1.296 (4)	C6—H6	0.9300
C1—C2	1.513 (4)	C7—C8	1.387 (5)
C2—C3	1.395 (4)	C7—H7	0.9300
C2—C8	1.398 (4)	C8—N1	1.435 (5)
C3—C5	1.394 (5)	N1—N1^{i}	1.236 (5)
C3—C4	1.486 (5)	O2—H2	0.8200
C4—O3	1.207 (4)	O4—H4	0.8200
C4—O4	1.287 (4)	O1W—H1WA	0.85 (3)
C5—C6	1.373 (6)	O1W—H1WB	0.85 (4)
C5—H5	0.9300		
O1—C1—O2	125.3 (3)	C3—C5—H5	119.4
O1—C1—C2	122.3 (3)	C5—C6—C7	120.4 (3)
O2—C1—C2	112.4 (3)	C5—C6—H6	119.8
C3—C2—C8	119.5 (3)	C7—C6—H6	119.8
C3—C2—C1	122.3 (3)	C6—C7—C8	119.4 (3)
C8—C2—C1	118.2 (3)	C6—C7—H7	120.3
C5—C3—C2	118.9 (3)	C8—C7—H7	120.3
C5—C3—C4	121.1 (3)	C7—C8—C2	120.7 (3)
C2—C3—C4	120.0 (3)	C7—C8—N1	124.6 (3)
O3—C4—O4	123.0 (3)	C2—C8—N1	114.7 (3)
O3—C4—C3	121.4 (3)	N1^{i}—N1—C8	116.2 (4)
O4—C4—C3	115.6 (3)	C1—O2—H2	109.5
C6—C5—C3	121.1 (3)	C4—O4—H4	109.5
C6—C5—H5	119.4	H1WA—O1W—H1WB	109 (5)

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—H1WA···O1	0.85 (3)	2.08 (2)	2.875 (4)	155 (4)
O4—H4···O1W^{ii}	0.82	1.81	2.631 (4)	177
O1W—H1WB···O3^{iii}	0.85 (4)	2.62 (4)	3.104 (5)	117 (4)
O2—H2···O3^{iv}	0.82	2.00	2.657 (4)	137

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

Fig. 1