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3,3'-Diazenediylidiphthalic acid dihydrate

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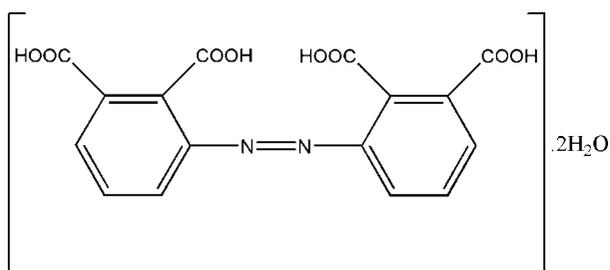
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.084; wR factor = 0.260; data-to-parameter ratio = 11.3.

In the crystal structure of the title compound, $\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$, the organic molecule is located on a centre of symmetry. The two benzene rings are parallel, but not coplanar, as indicated by $\text{N}=\text{N}-\text{C}-\text{C}$ torsion angles involving the azo group of 12.1 (5) and -168.2 (3)°. The organic molecule and the water molecule are linked by $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds, forming a three-dimensional network.

Related literature

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

Experimental

Crystal data

$\text{C}_{16}\text{H}_{10}\text{N}_2\text{O}_8 \cdot 2\text{H}_2\text{O}$
 $M_r = 394.29$
Triclinic, $P\bar{1}$
 $a = 6.6914$ (14) Å

$b = 7.8566$ (17) Å
 $c = 8.7665$ (19) Å
 $\alpha = 95.658$ (3)°
 $\beta = 100.628$ (3)°

$\gamma = 105.601$ (3)°
 $V = 430.90$ (16) Å³
 $Z = 1$
Mo $K\alpha$ radiation

$\mu = 0.13$ mm⁻¹
 $T = 298$ (2) K
 $0.27 \times 0.19 \times 0.15$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.009$

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
 $\Delta\rho_{\text{max}} = 1.04$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1W}-\text{H1WA} \cdots \text{O1}$	0.85 (3)	2.08 (2)	2.875 (4)	155 (4)
$\text{O4}-\text{H4} \cdots \text{O1W}^i$	0.82	1.81	2.631 (4)	177
$\text{O1W}-\text{H1WB} \cdots \text{O3}^{ii}$	0.85 (4)	2.62 (4)	3.104 (5)	117 (4)
$\text{O2}-\text{H2} \cdots \text{O3}^{iii}$	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.

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

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Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
Sheldrick, G. M. (2004). SADABS. University of Göttingen, Germany.

supporting information

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3,3'-Diazenediylidiphthalic acid dihydrate

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S1. Comment

In an attempt to prepare a Cd-containing coordination polymer (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 \cdots O hydrogen bonds (Table 1) to generate a three-dimensional architecture.

S2. Experimental

CdSO₄ (0.033 g, 0.012 mmol), 2,2',3,3'-diazenediylidiphthalic 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.

S3. Refinement

C-bound H atoms were placed at calculated positions (C—H = 0.93 Å) and were treated as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Carboxy H atoms were constrained with O—H = 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$, 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 \cdots H = 1.39 (1) Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. The maximum residual peak is located 1.34 Å from Br1.

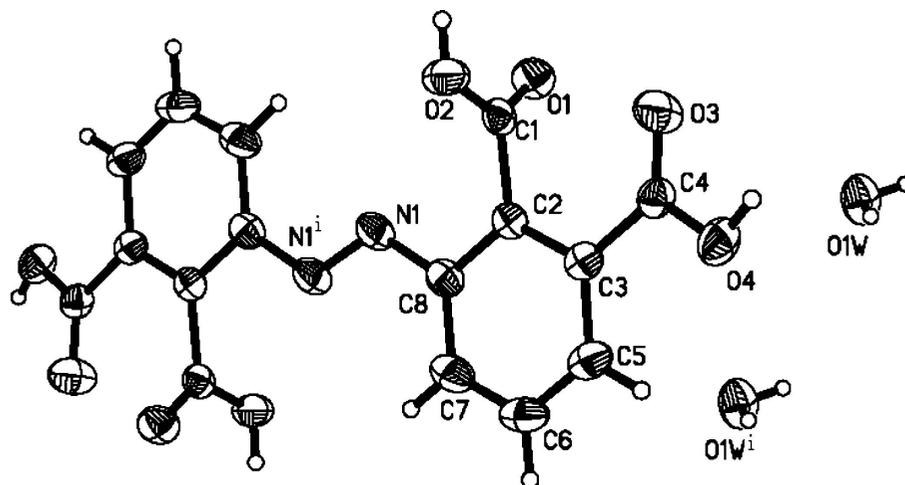


Figure 1

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

$C_{16}H_{10}N_2O_8 \cdot 2H_2O$

$M_r = 394.29$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.6914$ (14) Å

$b = 7.8566$ (17) Å

$c = 8.7665$ (19) Å

$\alpha = 95.658$ (3)°

$\beta = 100.628$ (3)°

$\gamma = 105.601$ (3)°

$V = 430.90$ (16) Å³

$Z = 1$

$F(000) = 204$

$D_x = 1.519$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1248 reflections

$\theta = 2.4$ – 25.2 °

$\mu = 0.13$ mm⁻¹

$T = 298$ K

Block, colourless

$0.27 \times 0.19 \times 0.15$ mm

Data collection

Bruker APEXII area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scan

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2004)

$T_{\min} = 0.966$, $T_{\max} = 0.981$

2297 measured reflections

1530 independent reflections

1248 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.009$

$\theta_{\max} = 25.2$ °, $\theta_{\min} = 2.4$ °

$h = -7 \rightarrow 8$

$k = -6 \rightarrow 9$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.084$

$wR(F^2) = 0.260$

$S = 1.11$

1530 reflections

135 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

$$w = 1/[\sigma^2(F_o^2) + (0.16P)^2 + 0.1722P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 1.04 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^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 (\AA , $^\circ$)

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 code: (i) $-x+1, -y+1, -z+1$.Hydrogen-bond geometry (\AA , $^\circ$)

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

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