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(*E*)-5,5′-(Diazene-1,2-diyl)diisophthalic acid *N*,*N*-dimethylformamide disolvate

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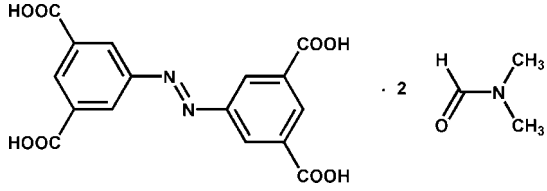
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.154; data-to-parameter ratio = 14.1.

The title compound, $C_{16}H_{10}N_2O_8\cdot 2C_3H_7NO$, was synthesized by the reductive condensation reaction of 5-nitroisophthalic acid in the presence of NaOH. The tetra-acid molecule, which has a crystallographically imposed centre of symmetry, adopts an *E* configuration with respect to the azo group. In the crystal packing, molecules are linked through intermolecular O—H···O and C—H···O hydrogen-bonding interactions, forming chains propagating in [2$\bar{1}$0].

Related literature

For general background information on the applications of azo compounds, see: Chung & Stevens (1993); Carliell *et al.* (1995).



Experimental

Crystal data

$C_{16}H_{10}N_2O_8\cdot 2C_3H_7NO$
M_r = 504.45
Triclinic, $P\bar{1}$
a = 6.2926 (13) Å
b = 7.2114 (13) Å
c = 13.653 (4) Å
α = 80.94 (4)°
β = 85.30 (4)°
γ = 81.72 (3)°
V = 604.3 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.971, T_{max} = 0.979
5593 measured reflections
2363 independent reflections
1607 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.154
S = 1.04
2363 reflections
167 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 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—H1···O5^{i}	0.82	1.72	2.541 (2)	174
O3—H3···O2^{ii}	0.82	1.94	2.697 (2)	154
C4—H4···O3^{ii}	0.93	2.42	3.305 (2)	159
C11—H11···O2^{iii}	0.93	2.58	3.240 (3)	128

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2248).

References

Carliell, C. M., Barclay, S. J., Naidoo, N., Buckley, C. A., Mulholland, D. A. & Senior, E. (1995). *Water SA*, **21**, 61–69.
Chung, K.-T. & Stevens, S. E. Jr (1993). *Environ. Toxicol. Chem.* **2**, 2121–2132.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, o2202 [doi:10.1107/S1600536808032819]

(*E*)-5,5′-(Diazene-1,2-diyl)diisophthalic acid *N*,*N*-dimethylformamide disolvate

Li Zhang and Zhi-Rong Qu

S1. Comment

Azo compounds are used as dyes in textile, paper manufacturing, pharmaceutial and food industries (Chung & Stevens, 1993; Carliell *et al.*, 1995). Herein, we report the crystal structure of the title compound, which was obtained by reductive condensation reaction of 5-nitroisophthalic acid in the presence of NaOH.

The acid molecule of the title compound (Fig. 1) has a crystallographically imposed centre of symmetry and adopts an E-configuration with respect to the azo group. The molecular conformation is stabilized by intramolecular C—H···O hydrogen bonds (Table 1). In the crystal packing (Fig. 2), molecules are linked into layers parallel to the $(2\bar{1}0)$ plane by intermolecular O—H···O and C—H···O hydrogen bonds (Table 1).

S2. Experimental

A solution of sodium hydroxide (35.9 g, 0.9 mol) in H_2O (125 ml) was added dropwise to a suspension of 5-nitro-isophthalic acid (10 g, 50.3 mmol) in H_2O (125 ml). The mixture was heated at 50°C for 18 h. After filtration, the yellow solid obtained was dissolved in H_2O and acidified with HCl. Crystals suitable for X-ray analysis were obtained after 10 days by slow evaporation of a DMF solution.

S3. Refinement

All H atoms were positioned geometrically and were allowed to ride on their parent atoms, with C—H = 0.93-0.96 Å, O—H = 0.82 Å, and with U_{iso}(H) = 1.5U_{eq}(C, O) or 1.2U_{eq}(C) for aromatic and aldehyde H atoms.

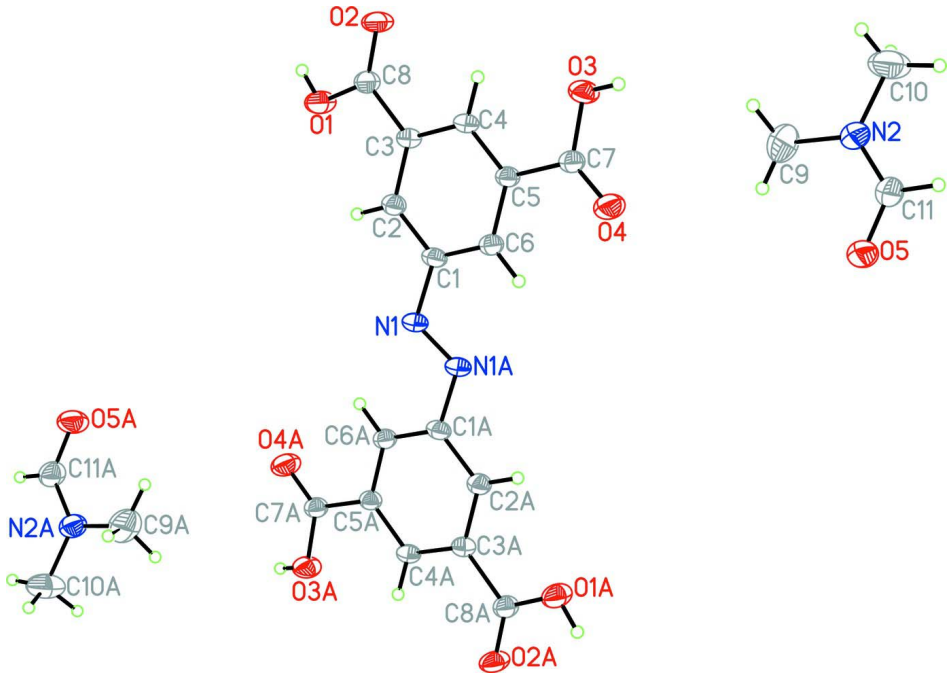

Figure 1

The molecular structure of the title compound with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level. [Symmetry code: (A) -x+3, -y, -z+1].

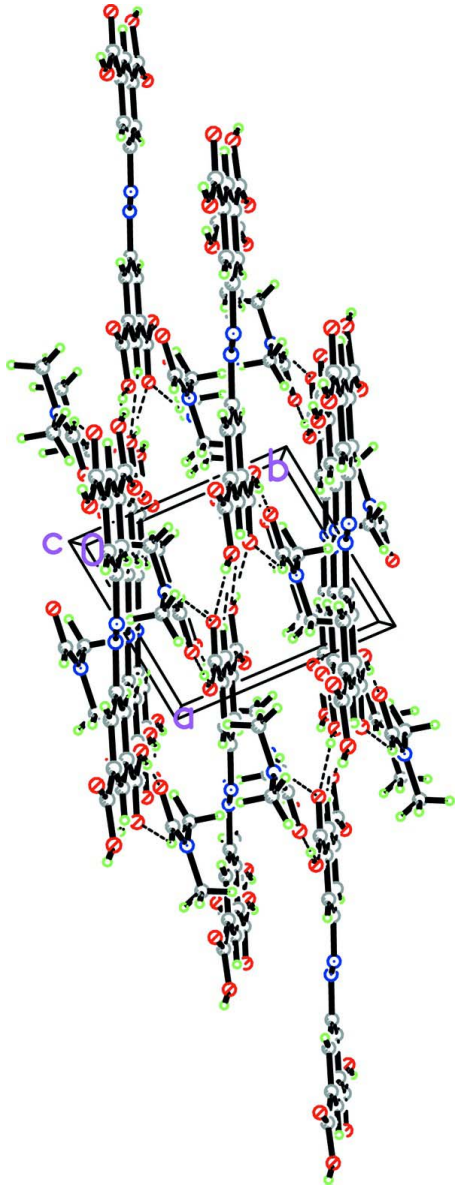

Figure 2

Packing diagram of the title compound, showing the structure along the *c* axis. Intermolecular hydrogen bonds are shown as dashed lines.

(E)-5,5′-(Diazene-1,2-diyl)diisophthalic acid N,N-dimethylformamide disolvate

Crystal data

$C_{16}H_{10}N_2O_8\cdot 2C_3H_7NO$
$M_r = 504.45$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.2926\ (13)$ Å
$b = 7.2114\ (13)$ Å
$c = 13.653\ (4)$ Å
$\alpha = 80.94\ (4)°$
$\beta = 85.30\ (4)°$
$\gamma = 81.72\ (3)°$
$V = 604.3\ (3)$ Å^3
$Z = 1$
$F(000) = 264$
$D_x = 1.386$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1381 reflections
$\theta = 2.9–27.4°$
$\mu = 0.11$ mm^{-1}

T = 293 K
Cuboid, colourless

0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.971, T_{max} = 0.979

5593 measured reflections
2363 independent reflections
1607 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 26.0°, θ_{min} = 2.9°
h = −7→7
k = −8→8
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.154
S = 1.04
2363 reflections
167 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
$w = 1/[\sigma^2(F_o^2) + (0.0869P)^2 + 0.0094P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	1.2504 (3)	0.1247 (3)	0.53879 (14)	0.0368 (5)
C2	1.1485 (3)	0.1486 (3)	0.63147 (14)	0.0388 (5)
H2	1.2213	0.1037	0.6889	0.047*
C3	0.9387 (3)	0.2393 (3)	0.63829 (14)	0.0362 (4)
C4	0.8321 (3)	0.3072 (3)	0.55177 (14)	0.0361 (5)
H4	0.6909	0.3664	0.5558	0.043*
C5	0.9357 (3)	0.2870 (2)	0.45901 (13)	0.0340 (4)
C6	1.1450 (3)	0.1962 (2)	0.45217 (14)	0.0360 (4)
H6	1.2144	0.1830	0.3903	0.043*
C7	0.8248 (3)	0.3644 (3)	0.36581 (14)	0.0393 (5)
C8	0.8253 (3)	0.2634 (3)	0.73691 (14)	0.0429 (5)
C9	0.5021 (5)	0.1873 (5)	0.1616 (2)	0.0951 (10)
H9A	0.6554	0.1852	0.1536	0.143*
H9B	0.4689	0.0633	0.1894	0.143*

H9C	0.4427	0.2763	0.2052	0.143*
C10	0.1791 (4)	0.2888 (5)	0.0659 (2)	0.0897 (10)
H10A	0.1367	0.3194	−0.0013	0.135*
H10B	0.1337	0.3953	0.1003	0.135*
H10C	0.1133	0.1817	0.0988	0.135*
C11	0.5328 (4)	0.2465 (4)	−0.01670 (17)	0.0583 (6)
H11	0.4662	0.2823	−0.0764	0.070*
N1	1.4637 (2)	0.0230 (2)	0.54104 (12)	0.0396 (4)
N2	0.4107 (3)	0.2433 (3)	0.06578 (13)	0.0560 (5)
O1	0.9368 (2)	0.1852 (3)	0.81263 (10)	0.0609 (5)
H1	0.8628	0.1957	0.8643	0.091*
O2	0.6425 (2)	0.3456 (2)	0.74637 (11)	0.0587 (5)
O3	0.6225 (2)	0.4372 (2)	0.38429 (11)	0.0559 (5)
H3	0.5702	0.4880	0.3321	0.084*
O4	0.9084 (2)	0.3631 (3)	0.28388 (11)	0.0658 (5)
O5	0.7304 (3)	0.2054 (3)	−0.02061 (11)	0.0725 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0248 (10)	0.0368 (10)	0.0480 (12)	−0.0002 (7)	0.0004 (8)	−0.0085 (9)
C2	0.0308 (10)	0.0441 (11)	0.0393 (10)	0.0020 (8)	−0.0022 (8)	−0.0062 (9)
C3	0.0287 (10)	0.0376 (10)	0.0404 (11)	0.0008 (7)	0.0016 (8)	−0.0067 (8)
C4	0.0256 (10)	0.0376 (10)	0.0428 (11)	0.0035 (7)	0.0003 (8)	−0.0074 (8)
C5	0.0280 (10)	0.0340 (9)	0.0390 (10)	0.0004 (7)	0.0000 (7)	−0.0077 (8)
C6	0.0313 (10)	0.0375 (10)	0.0383 (10)	−0.0012 (8)	0.0023 (8)	−0.0083 (8)
C7	0.0334 (11)	0.0427 (11)	0.0401 (11)	0.0018 (8)	0.0006 (8)	−0.0084 (8)
C8	0.0368 (11)	0.0525 (12)	0.0356 (11)	0.0035 (9)	−0.0004 (8)	−0.0042 (9)
C9	0.097 (2)	0.142 (3)	0.0424 (15)	−0.011 (2)	−0.0023 (14)	−0.0094 (17)
C10	0.0544 (18)	0.110 (2)	0.093 (2)	0.0092 (15)	0.0136 (15)	−0.0082 (19)
C11	0.0547 (15)	0.0755 (16)	0.0417 (12)	0.0016 (12)	−0.0043 (10)	−0.0081 (11)
N1	0.0257 (9)	0.0437 (9)	0.0473 (9)	0.0046 (7)	0.0009 (7)	−0.0097 (8)
N2	0.0496 (12)	0.0716 (13)	0.0446 (10)	−0.0024 (9)	0.0042 (8)	−0.0110 (9)
O1	0.0468 (9)	0.0897 (12)	0.0349 (8)	0.0200 (8)	−0.0010 (7)	−0.0018 (8)
O2	0.0395 (9)	0.0859 (11)	0.0401 (8)	0.0211 (8)	0.0030 (6)	−0.0069 (8)
O3	0.0372 (9)	0.0786 (11)	0.0427 (8)	0.0188 (7)	−0.0053 (6)	−0.0032 (8)
O4	0.0494 (10)	0.1019 (13)	0.0383 (9)	0.0144 (8)	0.0017 (7)	−0.0113 (9)
O5	0.0458 (10)	0.1218 (16)	0.0445 (10)	0.0002 (9)	0.0043 (7)	−0.0103 (10)

Geometric parameters (Å, º)

C1—C6	1.394 (3)	C8—O1	1.304 (2)
C1—C2	1.395 (3)	C9—N2	1.447 (3)
C1—N1	1.434 (2)	C9—H9A	0.9600
C2—C3	1.389 (2)	C9—H9B	0.9600
C2—H2	0.9300	C9—H9C	0.9600
C3—C4	1.392 (3)	C10—N2	1.447 (3)
C3—C8	1.494 (3)	C10—H10A	0.9600

C4—C5	1.395 (3)	C10—H10B	0.9600
C4—H4	0.9300	C10—H10C	0.9600
C5—C6	1.387 (2)	C11—O5	1.235 (3)
C5—C7	1.492 (3)	C11—N2	1.309 (3)
C6—H6	0.9300	C11—H11	0.9300
C7—O4	1.197 (2)	N1—N1^{i}	1.251 (3)
C7—O3	1.325 (2)	O1—H1	0.8200
C8—O2	1.222 (2)	O3—H3	0.8200
C6—C1—C2	120.34 (17)	O1—C8—C3	114.04 (17)
C6—C1—N1	124.35 (17)	N2—C9—H9A	109.5
C2—C1—N1	115.31 (17)	N2—C9—H9B	109.5
C3—C2—C1	120.22 (18)	H9A—C9—H9B	109.5
C3—C2—H2	119.9	N2—C9—H9C	109.5
C1—C2—H2	119.9	H9A—C9—H9C	109.5
C2—C3—C4	119.34 (17)	H9B—C9—H9C	109.5
C2—C3—C8	121.08 (18)	N2—C10—H10A	109.5
C4—C3—C8	119.57 (17)	N2—C10—H10B	109.5
C3—C4—C5	120.42 (17)	H10A—C10—H10B	109.5
C3—C4—H4	119.8	N2—C10—H10C	109.5
C5—C4—H4	119.8	H10A—C10—H10C	109.5
C6—C5—C4	120.25 (17)	H10B—C10—H10C	109.5
C6—C5—C7	118.99 (17)	O5—C11—N2	124.4 (2)
C4—C5—C7	120.75 (16)	O5—C11—H11	117.8
C5—C6—C1	119.40 (18)	N2—C11—H11	117.8
C5—C6—H6	120.3	N1^{i}—N1—C1	113.5 (2)
C1—C6—H6	120.3	C11—N2—C10	122.1 (2)
O4—C7—O3	123.70 (19)	C11—N2—C9	121.0 (2)
O4—C7—C5	124.31 (18)	C10—N2—C9	116.8 (2)
O3—C7—C5	111.99 (17)	C8—O1—H1	109.5
O2—C8—O1	122.62 (18)	C7—O3—H3	109.5
O2—C8—C3	123.32 (18)		

Symmetry code: (i) $-x+3, -y, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O3	0.93	2.38	2.704 (3)	100
C9—H9*A*···O5	0.96	2.37	2.763 (3)	104
O1—H1···O5^{ii}	0.82	1.72	2.541 (2)	174
O3—H3···O2^{iii}	0.82	1.94	2.697 (2)	154
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