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Diethyl 4,4′-(diazenediyl)dibenzoate

Yongsheng Niu, Jianping Huang, Chunpu Zhao, Pan Gao and Youzhu Yu*

Department of Chemistry and Environmental Engineering, Anyang Institute of Technology, Henan 455000, People's Republic of China
Correspondence e-mail: 119yyz@163.com

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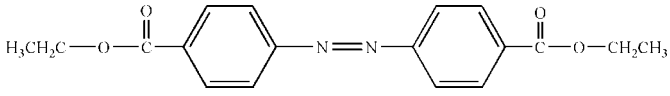
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.050; wR factor = 0.149; data-to-parameter ratio = 18.1.

The full molecule of the title compound, $C_{18}H_{18}N_2O_4$, is generated by the application of an inversion centre. There are strong π–π interactions between adjacent molecules with a centroid–centroid distance of 3.298 (2)Å.

Related literature

For the properties and structures of related compounds, see: Altomare *et al.* (2005;) Kubo *et al.* (2005); Harada *et al.* (1997).



Experimental

Crystal data

$C_{18}H_{18}N_2O_4$
M_r = 326.34
Monoclinic, $P2_1/c$
a = 14.844 (3) Å
b = 4.5731 (9) Å
c = 11.814 (2) Å
β = 95.88 (3)°
V = 797.7 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.40 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
13606 measured reflections
1976 independent reflections
1639 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.149
S = 1.07
1976 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AA2020).

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supporting information

Acta Cryst. (2011). E**67**, o2671 [https://doi.org/10.1107/S1600536811036877]

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S1. Comment

Synthesis, elucidation of crystal structures, and investigation of physical properties of new liquid crystals are important for studying the relationship between molecular stuctures and mesophases. (Kubo *et al.*, 2005). As a contribution to these fields, We report here the synthesis and structure of the title compound.

The title compound (Fig. 1), $C_{18}H_{18}N_2O_4$, shows crystallographic inversion symmetry. The intersection angle between two benzene rings is consistent with that of azobenzene (0 °). (Harada *et al.*, 1997) No classic hydrogen bonds are observed in the crystal. There are strong π-π interactions between planar adjacent molecules with the interplanar distance 3.298 Å.

S2. Experimental

1.0 g of azobenzene-4,4′-dicarbonylchloride and 20 ml of ethanol were stirred at 353 K for 4 h. After cooling to room temperature a red deposit was obtained. It was then recrystallized from CH_2Cl_2 to give red crystals suitable for X-ray diffraction analysis.

S3. Refinement

All H atoms were placed in geometrically idealized positions (C—H = 0.93, 0.96 and 0.97 Å) and treated as riding on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C).

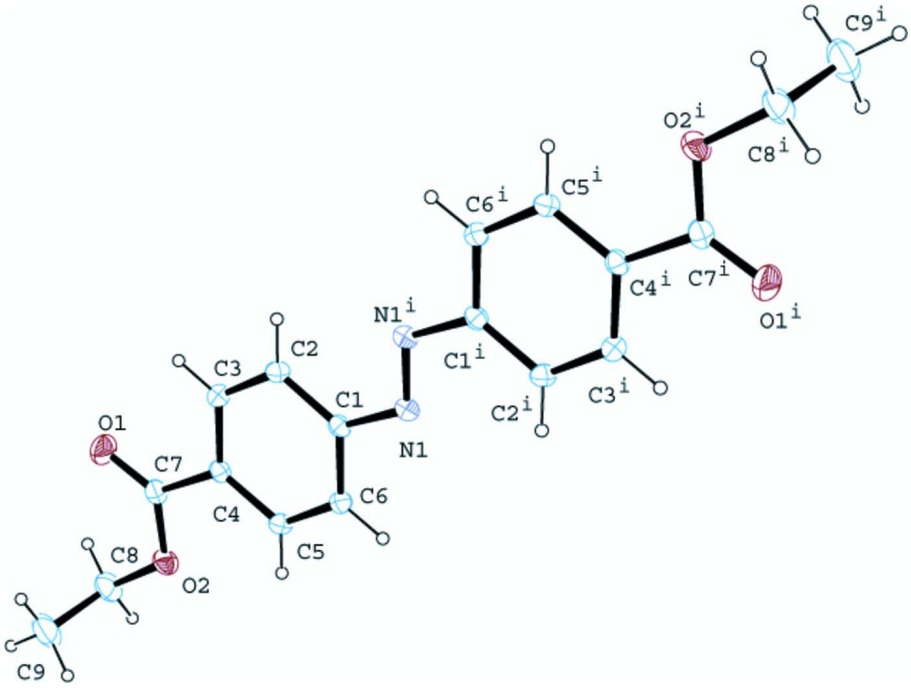

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. Symmetry code: (i) 2 - *x*, - *y*, 2 - *z*

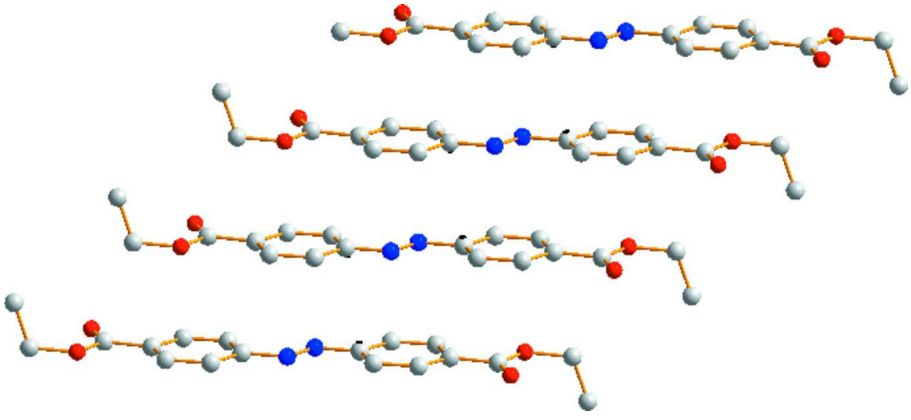

Figure 2

π-π interaction of the molecules.

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Crystal data

$C_{18}H_{18}N_2O_4$
M_r = 326.34
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 14.844 (3) Å
b = 4.5731 (9) Å
c = 11.814 (2) Å
β = 95.88 (3)°
V = 797.7 (3) Å^3
Z = 2
F(000) = 344
D_x = 1.359 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5335 reflections
θ = 3.5–28.4°
μ = 0.10 mm^{-1}

T = 293 K
Block, red

0.40 × 0.20 × 0.20 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
13606 measured reflections
1976 independent reflections

1639 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 28.4°, θ_{min} = 3.5°
h = −19→19
k = −6→6
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.149
S = 1.07
1976 reflections
109 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
$w = 1/[\sigma^2(F_o^2) + (0.0736P)^2 + 0.2526P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	0.92686 (8)	0.2685 (3)	0.93120 (11)	0.0327 (3)
C2	0.86559 (9)	0.3422 (3)	1.00838 (12)	0.0390 (3)
H2A	0.8699	0.2558	1.0800	0.047*
C3	0.92142 (9)	0.4028 (3)	0.82545 (11)	0.0353 (3)
H3A	0.9633	0.3565	0.7748	0.042*
C4	0.85380 (9)	0.6061 (3)	0.79487 (11)	0.0360 (3)
H4A	0.8502	0.6954	0.7238	0.043*
C5	0.79835 (9)	0.5450 (3)	0.97752 (12)	0.0398 (3)
H5A	0.7573	0.5946	1.0288	0.048*
C6	0.79149 (8)	0.6754 (3)	0.87074 (11)	0.0340 (3)
C7	0.71536 (9)	0.8857 (3)	0.84138 (13)	0.0407 (3)
C8	0.64597 (12)	1.2071 (4)	0.69866 (17)	0.0604 (5)
H8A	0.6696	1.3527	0.6498	0.072*
H8B	0.6251	1.3072	0.7635	0.072*
C9	0.56912 (14)	1.0525 (6)	0.6352 (2)	0.0848 (8)
H9A	0.5226	1.1908	0.6103	0.127*
H9B	0.5453	0.9099	0.6838	0.127*
H9C	0.5896	0.9565	0.5702	0.127*
N1	0.99820 (7)	0.0589 (2)	0.95208 (9)	0.0354 (3)
O1	0.65887 (8)	0.9411 (3)	0.90410 (11)	0.0645 (4)
O2	0.71756 (7)	1.0021 (3)	0.73787 (10)	0.0502 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0323 (6)	0.0305 (6)	0.0344 (7)	−0.0026 (5)	−0.0018 (5)	−0.0015 (5)
C2	0.0417 (7)	0.0438 (8)	0.0315 (7)	−0.0004 (6)	0.0032 (5)	0.0052 (5)
C3	0.0359 (6)	0.0373 (7)	0.0329 (7)	0.0013 (5)	0.0039 (5)	−0.0006 (5)
C4	0.0392 (6)	0.0380 (7)	0.0303 (6)	0.0012 (5)	0.0008 (5)	0.0017 (5)
C5	0.0363 (6)	0.0469 (8)	0.0368 (7)	0.0007 (6)	0.0071 (5)	−0.0010 (6)
C6	0.0311 (6)	0.0337 (6)	0.0361 (7)	−0.0019 (5)	−0.0016 (5)	−0.0033 (5)
C7	0.0344 (6)	0.0404 (7)	0.0462 (8)	0.0016 (5)	−0.0005 (5)	−0.0048 (6)
C8	0.0546 (9)	0.0584 (10)	0.0651 (11)	0.0187 (8)	−0.0086 (8)	0.0054 (9)
C9	0.0547 (10)	0.1064 (19)	0.0875 (16)	0.0183 (11)	−0.0209 (10)	−0.0167 (14)
N1	0.0370 (6)	0.0347 (6)	0.0334 (6)	−0.0007 (4)	−0.0009 (4)	0.0008 (4)
O1	0.0515 (7)	0.0768 (9)	0.0673 (8)	0.0215 (6)	0.0171 (6)	0.0054 (7)
O2	0.0468 (6)	0.0546 (7)	0.0475 (6)	0.0157 (5)	−0.0033 (5)	0.0054 (5)

Geometric parameters (Å, º)

C1—C3	1.3870 (18)	C6—C7	1.4972 (18)
C1—C2	1.3934 (19)	C7—O1	1.2015 (18)
C1—N1	1.4311 (17)	C7—O2	1.3371 (19)
C2—C5	1.384 (2)	C8—O2	1.4568 (18)
C2—H2A	0.9300	C8—C9	1.479 (3)
C3—C4	1.3883 (18)	C8—H8A	0.9700
C3—H3A	0.9300	C8—H8B	0.9700
C4—C6	1.3890 (19)	C9—H9A	0.9600
C4—H4A	0.9300	C9—H9B	0.9600
C5—C6	1.3896 (19)	C9—H9C	0.9600
C5—H5A	0.9300	N1—N1^{i}	1.250 (2)
C3—C1—C2	120.05 (12)	O1—C7—O2	124.33 (14)
C3—C1—N1	115.19 (12)	O1—C7—C6	123.40 (14)
C2—C1—N1	124.76 (12)	O2—C7—C6	112.27 (12)
C5—C2—C1	119.41 (13)	O2—C8—C9	110.67 (16)
C5—C2—H2A	120.3	O2—C8—H8A	109.5
C1—C2—H2A	120.3	C9—C8—H8A	109.5
C1—C3—C4	120.31 (12)	O2—C8—H8B	109.5
C1—C3—H3A	119.8	C9—C8—H8B	109.5
C4—C3—H3A	119.8	H8A—C8—H8B	108.1
C3—C4—C6	119.77 (12)	C8—C9—H9A	109.5
C3—C4—H4A	120.1	C8—C9—H9B	109.5
C6—C4—H4A	120.1	H9A—C9—H9B	109.5
C2—C5—C6	120.71 (13)	C8—C9—H9C	109.5
C2—C5—H5A	119.6	H9A—C9—H9C	109.5
C6—C5—H5A	119.6	H9B—C9—H9C	109.5
C4—C6—C5	119.73 (12)	N1^{i}—N1—C1	114.02 (14)
C4—C6—C7	122.27 (12)	C7—O2—C8	117.50 (13)
C5—C6—C7	117.99 (12)		

C3—C1—C2—C5	1.4 (2)	C4—C6—C7—O1	177.29 (15)
N1—C1—C2—C5	−178.86 (12)	C5—C6—C7—O1	−2.0 (2)
C2—C1—C3—C4	−1.5 (2)	C4—C6—C7—O2	−2.41 (19)
N1—C1—C3—C4	178.77 (11)	C5—C6—C7—O2	178.28 (12)
C1—C3—C4—C6	0.1 (2)	C3—C1—N1—N1^{i}	179.91 (13)
C1—C2—C5—C6	0.0 (2)	C2—C1—N1—N1^{i}	0.2 (2)
C3—C4—C6—C5	1.2 (2)	O1—C7—O2—C8	−0.7 (2)
C3—C4—C6—C7	−178.06 (12)	C6—C7—O2—C8	179.05 (12)
C2—C5—C6—C4	−1.3 (2)	C9—C8—O2—C7	−91.4 (2)
C2—C5—C6—C7	178.04 (12)		

Symmetry code: (i) −*x*+2, −*y*, −*z*+2.