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1,2-Bis(4-nitrobenzoyl)hydrazine

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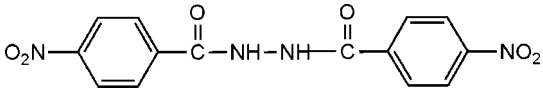
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.070; wR factor = 0.220; data-to-parameter ratio = 12.5.

The title molecule, $C_{14}H_{10}N_4O_6$, crystallizes with one half-molecule in the asymmetric unit; the mid-point of the N—N bond lies on an inversion centre. The nitro and amide groups are twisted with respect to the benzene ring, making dihedral angles of 14.6 (5) and 31.1 (5)°, respectively. In the crystal structure, molecules are linked through N—H···O hydrogen bonding between the imino and carbonyl groups.

Related literature

For the biological activity of hydrazides, see: Cui *et al.* (2007); Li & Ban (2009). For related structures, see: Shang *et al.* (2005*a*,*b*); Zhang *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{10}N_4O_6$
M_r = 330.26
Monoclinic, $P2_1/n$
a = 4.7947 (6) Å
b = 9.8750 (11) Å
c = 14.9094 (17) Å
β = 99.05 (3)°
V = 697.13 (14) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.975, T_{max} = 0.988
1364 measured reflections
1364 independent reflections
673 reflections with $I > 2\sigma(I)$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.220
S = 1.10
1364 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.12	2.881 (5)	147

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: XU2587).

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supplementary materials

Acta Cryst. (2009). E**65**, o2189 [**doi:10.1107/S160053680903181X**]

1,2-Bis(4-nitrobenzoyl)hydrazine

X.-Y. Jiang, X.-J. Feng, S. Yang, H.-J. Xu and L.-Y. Hao

Comment

Hydrazides have been demonstrated to possess excellent biological activities (Cui *et al.*, 2007; Li & Ban, 2009). Recently a great deal of hydrazides have been synthesized and characterized (Shang *et al.*, 2005*a*,b; Zhang *et al.*, 2009; Li & Ban, 2009). We also are interested in this field of research, we report here the crystal structure of the title compound.

The molecular structure of the title compound has crystallographically imposed inversion symmetry located in the middle of the N—N bond (Fig. 1). One intermolecular hydrogen bond N—H···O is observed in the crystal structure (Table 1).

Experimental

4-Nitrobenzohydrazide (0.371 g, 2.0 mmol) and 20 ml chloroform were introduced into a round-bottomed flask at 281 K and stirred. 4-Nitrobenzoyl chloride (0.362 g, 2.0 mmol) was added to the mixture, which was stirred for 2 h at room temperature. A colourless solid product was filtered, and washed three times with ethyl ether. Crystals of the title compound suitable for X-ray structural determination was obtained by slow evaporation a methanol solution in air.

Refinement

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 and N—H = 0.86 Å and with U_{iso}(H) = 1.2 U_{eq}(C,N).

Figures

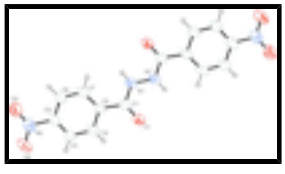

Fig. 1. The molecular structure of the title copound, showing 30% probability displacement ellipsoids [symmetry code: (i) 2-x, -y, 1-z].

1,2-Bis(4-nitrobenzoyl)hydrazine

Crystal data

$C_{14}H_{10}N_4O_6$	F_{000} = 340
M_r = 330.26	D_x = 1.573 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 25 reflections
a = 4.7947 (6) Å	θ = 8–12°
b = 9.8750 (11) Å	μ = 0.13 mm^{-1}

c = 14.9094 (17) Å	T = 293 K
β = 99.05 (3)º	Block, colorless
V = 697.13 (14) Å^3	0.20 × 0.10 × 0.10 mm
Z = 2	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.0000
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 2.5º
T = 293 K	h = −5→5
ω/2θ scans	k = 0→12
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→18
T_{min} = 0.975, T_{max} = 0.988	3 standard reflections
1364 measured reflections	every 200 reflections
1364 independent reflections	intensity decay: none
673 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.070	H-atom parameters constrained
$wR(F^2)$ = 0.220	$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.1296P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.10	$(\Delta/\sigma)_{max} < 0.001$
1364 reflections	$\Delta\rho_{max}$ = 0.13 e Å^{-3}
109 parameters	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
O1	0.5717 (7)	−0.0146 (4)	0.4016 (2)	0.0940 (11)
O2	0.8216 (9)	0.1386 (5)	−0.0278 (3)	0.1227 (16)
O3	1.2216 (11)	0.2315 (5)	0.0270 (3)	0.1219 (15)
N1	1.0280 (8)	0.0187 (4)	0.4580 (2)	0.0842 (12)
H1A	1.1951	0.0419	0.4498	0.101*
N2	1.0017 (12)	0.1715 (5)	0.0358 (3)	0.0949 (13)
C1	0.8102 (10)	0.0176 (5)	0.3890 (3)	0.0794 (12)
C2	0.8783 (10)	0.0610 (5)	0.2994 (3)	0.0775 (12)
C3	0.7163 (12)	0.0022 (6)	0.2224 (4)	0.1007 (16)
H3A	0.5823	−0.0633	0.2297	0.121*
C4	0.7531 (11)	0.0399 (6)	0.1371 (3)	0.0920 (15)
H4A	0.6379	0.0047	0.0865	0.110*
C5	0.9622 (12)	0.1304 (6)	0.1272 (3)	0.0897 (14)
C6	1.1218 (12)	0.1918 (5)	0.2030 (4)	0.0926 (15)
H6A	1.2522	0.2587	0.1952	0.111*
C7	1.0864 (12)	0.1540 (5)	0.2868 (3)	0.0939 (15)
H7A	1.2021	0.1903	0.3370	0.113*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.087 (2)	0.124 (3)	0.0660 (19)	−0.004 (2)	−0.0060 (16)	0.0027 (19)
O2	0.140 (3)	0.145 (4)	0.070 (2)	0.015 (3)	−0.024 (2)	0.009 (3)
O3	0.156 (4)	0.117 (3)	0.090 (3)	−0.013 (3)	0.010 (3)	0.015 (2)
N1	0.077 (2)	0.102 (3)	0.065 (2)	−0.004 (2)	−0.0141 (18)	0.012 (2)
N2	0.117 (3)	0.085 (3)	0.082 (3)	0.014 (3)	0.009 (3)	0.014 (2)
C1	0.086 (3)	0.076 (3)	0.070 (3)	0.000 (2)	−0.007 (2)	0.003 (2)
C2	0.081 (3)	0.079 (3)	0.064 (2)	0.006 (2)	−0.017 (2)	0.005 (2)
C3	0.109 (4)	0.098 (4)	0.080 (3)	−0.017 (3)	−0.029 (3)	0.002 (3)
C4	0.099 (3)	0.102 (4)	0.066 (3)	−0.008 (3)	−0.012 (3)	0.001 (3)
C5	0.113 (4)	0.079 (3)	0.070 (3)	0.017 (3)	−0.009 (3)	0.011 (3)
C6	0.114 (4)	0.075 (3)	0.079 (3)	−0.008 (3)	−0.017 (3)	0.005 (3)
C7	0.112 (4)	0.079 (3)	0.075 (3)	−0.005 (3)	−0.035 (3)	0.003 (3)

Geometric parameters (Å, °)

O1—C1	1.229 (5)	C2—C3	1.407 (7)
O2—N2	1.221 (6)	C3—C4	1.362 (7)
O3—N2	1.234 (5)	C3—H3A	0.9300
N1—C1	1.346 (6)	C4—C5	1.368 (7)
N1—N1^{i}	1.372 (7)	C4—H4A	0.9300
N1—H1A	0.8600	C5—C6	1.400 (7)
N2—C5	1.462 (6)	C6—C7	1.340 (7)
C1—C2	1.488 (6)	C6—H6A	0.9300

C2—C7	1.390 (7)	C7—H7A	0.9300
C1—N1—N1^{i}	117.1 (5)	C2—C3—H3A	119.6
C1—N1—H1A	121.5	C3—C4—C5	119.0 (5)
N1^{i}—N1—H1A	121.5	C3—C4—H4A	120.5
O2—N2—O3	123.8 (5)	C5—C4—H4A	120.5
O2—N2—C5	118.0 (5)	C4—C5—C6	120.8 (5)
O3—N2—C5	118.1 (5)	C4—C5—N2	119.1 (5)
O1—C1—N1	121.0 (4)	C6—C5—N2	119.8 (5)
O1—C1—C2	123.5 (4)	C7—C6—C5	119.9 (5)
N1—C1—C2	115.5 (4)	C7—C6—H6A	120.0
C7—C2—C3	118.6 (5)	C5—C6—H6A	120.0
C7—C2—C1	125.1 (5)	C6—C7—C2	120.6 (5)
C3—C2—C1	116.3 (5)	C6—C7—H7A	119.7
C4—C3—C2	120.9 (5)	C2—C7—H7A	119.7
C4—C3—H3A	119.6		
N1^{i}—N1—C1—O1	0.7 (8)	C3—C4—C5—N2	−179.8 (5)
N1^{i}—N1—C1—C2	179.0 (5)	O2—N2—C5—C4	10.3 (7)
O1—C1—C2—C7	148.0 (5)	O3—N2—C5—C4	−166.2 (5)
N1—C1—C2—C7	−30.3 (7)	O2—N2—C5—C6	−164.5 (5)
O1—C1—C2—C3	−31.9 (7)	O3—N2—C5—C6	19.0 (7)
N1—C1—C2—C3	149.8 (5)	C4—C5—C6—C7	5.5 (8)
C7—C2—C3—C4	−3.0 (8)	N2—C5—C6—C7	−179.8 (5)
C1—C2—C3—C4	176.9 (5)	C5—C6—C7—C2	−4.6 (8)
C2—C3—C4—C5	3.8 (9)	C3—C2—C7—C6	3.4 (8)
C3—C4—C5—C6	−5.0 (8)	C1—C2—C7—C6	−176.5 (5)

Symmetry codes: (i) −*x*+2, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1^{ii}	0.86	2.12	2.881 (5)	147

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

Fig. 1