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2′,2′-Dibenzylideneisophthalohydrazide methanol solvate

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Key indicators

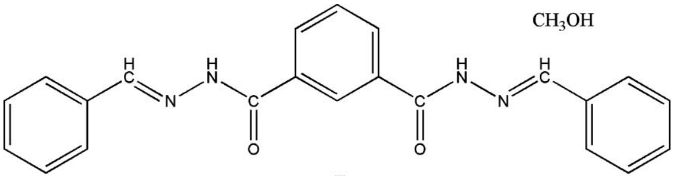
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.155
Data-to-parameter ratio = 18.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{22}H_{18}N_4O_2 \cdot CH_3OH$, was synthesized by the reaction of benzaldehyde and isophthaloyl hydrazine in methanol. The molecule is non-planar, the dihedral angles between the pairs of aromatic rings being 13.2 (1), 27.0 (1) and 18.4 (1)°. The hydrazide molecules are linked *via* hydrogen bonds into a chain along the *c* axis.

Comment

The chemistry of aroylhydrazone compounds has received increasing attention because the hydrazone group is strongly coordinated to many metal atoms and aroylhydrazone compounds possess widespread applications in the treatment of tuberculosis. They also exhibit fungicidal activity (Edwards *et al.*, 1975; Zhi *et al.*, 2003; Yang & Pan, 2004). We report here the synthesis and crystal structure of the title compound, (I), obtained by the condensation of benzaldehyde with isophthaloyl hydrazine.



(I)

The molecular structure of (I) is shown in Fig. 1. The title molecule is non-planar. The dihedral angle between rings C1–C6 and C9–C14 is 13.2 (1)°, between rings C1–C6 and C17–C22 is 27.0 (1)° and between rings C9–C14 and C17–C22 is 18.4 (1)°. Similar C═O distances (Table 1) have been

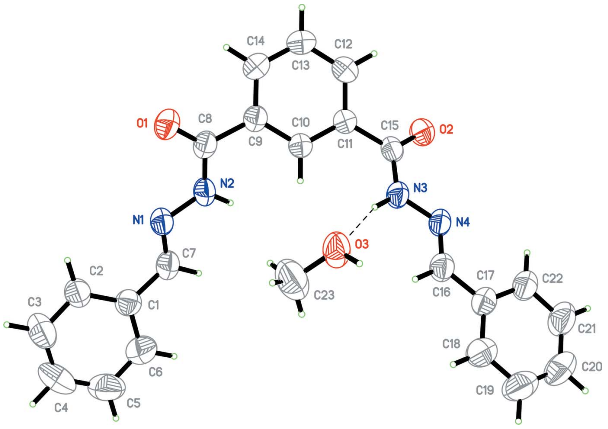


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids. The dashed line represents a hydrogen bond.

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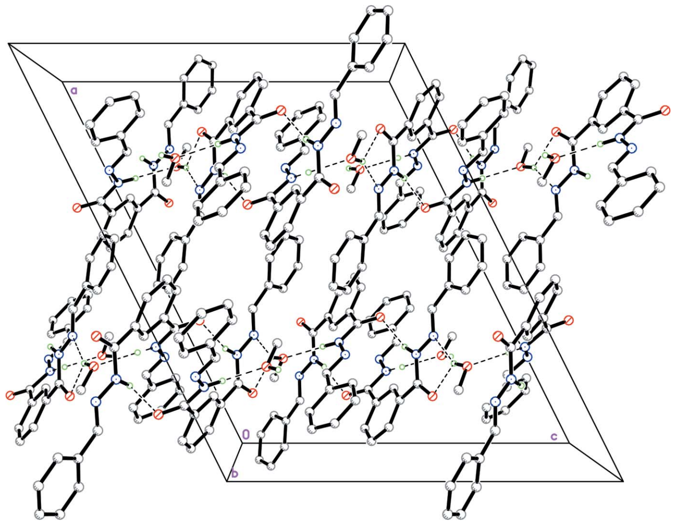

Figure 2
The packing of (I), showing the intermolecular hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

observed in many hydrazone compounds. The N1—C7 and N4—C16 bond lengths are close to the value of 1.280 (5) Å found for the imine bond length in *p*-dimethylenedioxybenzaldehyde 2,4-dinitrobenzoylhydrazone (Wang *et al.*, 2004) and shorter than the value of 1.337 (2) Å found for the C—N single bond in the 1:1 complex of 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone and nicotinoylhydrazine (Liu *et al.*, 2001). The N1—N2 and N3—N4 bond lengths are close to the values 1.3794 (19) and 1.388 (2) Å in *p*-dimethylenedioxybenzaldehyde benzoylhydrazone (Fun *et al.*, 1997), indicating that a partially conjugated system operates in this hydrazone.

There are four intermolecular hydrogen bonds in the crystal structure (Table 2), which link adjacent molecules to form a chain. The H atom on O3 engages in binding to atoms N1^{ii} and O1^{ii} simultaneously.

Experimental

Isophthaloyl hydrazine (17.8 mmol, 3.45 g) was dissolved in anhydrous methanol (50 ml), and benzaldehyde (35.7 mmol, 3.65 ml) was added. The mixture was refluxed for 3 h and the resulting precipitate was collected by filtration and washed with methanol and diethyl ether. The product (0.37 g) was dissolved in methanol (15 ml) and CH_2Cl_2 (15 ml), and kept at room temperature for 20 d to obtain colourless single crystals.

Crystal data

$C_{23}H_{22}N_4O_3$
$M_r = 402.45$
Monoclinic, $C2/c$
$a = 20.168$ (4) Å
$b = 14.737$ (3) Å
$c = 16.357$ (3) Å
$\beta = 116.54$ (3)°
$V = 4349.0$ (15) Å^3
$Z = 8$
$D_x = 1.229$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4999 reflections
$\theta = 1.8$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.55 × 0.42 × 0.36 mm

Data collection

Rigaku Weissenberg IP diffractometer
ω scans
Absorption correction: multi-scan (*TEXRAY*; Molecular Structure Corporation, 1999)
$T_{min} = 0.778$, $T_{max} = 0.970$
20697 measured reflections
4999 independent reflections
3243 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 26$
$k = -19 \rightarrow 19$
$l = -21 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.155$
$S = 1.04$
4999 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0901P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C7	1.454 (2)	C15—O2	1.2305 (18)
C7—N1	1.270 (2)	C15—N3	1.348 (2)
C8—O1	1.2219 (17)	C16—N4	1.272 (2)
C8—N2	1.351 (2)	C16—C17	1.463 (2)
C8—C9	1.499 (2)	N1—N2	1.3838 (18)
C11—C15	1.497 (2)	N3—N4	1.3837 (16)
O1—C8—N2	122.96 (15)	C7—N1—N2	114.50 (13)
O2—C15—N3	122.84 (14)	C16—N4—N3	115.12 (13)
O1—C8—C9—C10	153.04 (15)	O1—C8—N2—N1	−4.5 (2)
C10—C11—C15—O2	151.34 (15)	O2—C15—N3—N4	0.7 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2^{i}	0.86	2.17	3.0048 (17)	164
O3—H3*C*···N1^{ii}	0.89	2.28	3.0954 (18)	153
O3—H3*C*···O1^{ii}	0.89	2.30	2.9550 (19)	131
N3—H3*A*···O3	0.86	2.05	2.8848 (19)	163

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Atoms H3*A* and H3*C* were located in difference Fourier maps, but were then allowed to ride on N3 and O3, with N—H = 0.86 Å and O—H = 0.89 Å. The other H atoms were placed in idealized positions (aromatic C—H = 0.93 Å, methanol C—H = 0.96 Å and N—H = 0.86 Å) and were refined using a riding model, with $U_{iso}(H) = 1.5U_{eq}(C)$.

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97-2* (Sheldrick, 1997).

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