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2′-(1,3-Benzodioxol-5-ylmethylene)-2-methoxybenzohydrazide

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Key indicators

Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.102
Data-to-parameter ratio = 16.1

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

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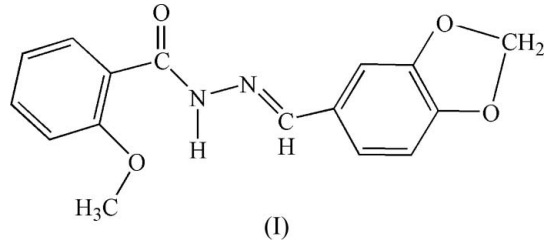
The non-H atoms of the title compound, $C_{16}H_{14}N_2O_4$, are essentially coplanar. An intramolecular N—H···O hydrogen bond stabilizes the molecular structure. Molecules are linked *via* weak intermolecular C—H···O hydrogen bonds, forming a three-dimensional network.

Comment

In order to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Belloni *et al.*, 2005; Tynan *et al.*, 2005; Parashar *et al.*, 1988), the synthesis of new and designed crystal structures has become a major strand of modern chemistry. Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of the active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Schiff bases functioning as ligands, we report the synthesis and crystal structure of the title compound, (I).



(I)

In the molecular structure of the compound, (I) (Fig. 1), the geometric parameters are normal. The benzo[1,3]dioxole ring system (C10–C16/O3/O4) is planar, with an r.m.s. deviation for fitted atoms of 0.021 Å. The 2-methoxybenzoic acid methylene hydrazide group (O1/O2/N1/N2/C1–C9) is also planar, with an r.m.s. deviation of 0.074 Å. The dihedral angle between these planes is 3.20 (3)°.

Intramolecular N—H···O hydrogen bonding stabilizes the molecular conformation, while intermolecular C—H···O hydrogen bonding stabilizes the crystal structure; geometric details are given in Table 1. The molecules associate to form a supramolecular structure, as illustrated in Fig. 2.

Experimental

An anhydrous ethanol solution (50 ml) of benzo[1,3]dioxole-5-carbaldehyde (1.50 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 2-methoxybenzoic acid hydrazide (1.66 g, 10 mmol), and the mixture was stirred at 350 K for 6 h under N_2, whereupon a red precipitate appeared. The product was isolated,

recrystallized from anhydrous ethanol and then dried *in vacuo* to give pure compound (I) in 81% yield. Red single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Crystal data

$C_{16}H_{14}N_2O_4$
M_r = 298.29
Monoclinic, $P2_1/n$
a = 7.9913 (5) Å
b = 12.5941 (7) Å
c = 14.1263 (10) Å
β = 102.231 (3)°
V = 1389.45 (15) Å^3
Z = 4
D_x = 1.426 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 113 (2) K
Prism, red
0.22 × 0.20 × 0.16 mm

Data collection

Rigaku SATURN diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.977, T_{max} = 0.984
12843 measured reflections
3307 independent reflections
2725 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.9°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.102
S = 1.09
3307 reflections
205 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 0.0088P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.020 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.893 (12)	1.928 (13)	2.6502 (13)	136.8 (12)
C4—H4···O1^{i}	0.95	2.45	3.2350 (15)	140
C6—H6···O3^{ii}	0.95	2.53	3.4306 (17)	160
C9—H9···O4^{iii}	0.95	2.50	3.4127 (14)	161
C16—H16B···O1^{iv}	0.99	2.28	3.2587 (17)	169

Symmetry codes: (i) $x-\frac{1}{2}, -y-\frac{1}{2}, z-\frac{1}{2}$; (ii) $x-1, y-1, z$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

The N-bound H atom was located in a difference Fourier map and its positional parameters were refined, with U_{iso}(H) = 1.2U_{eq}(N). C-bound H atoms were included in calculated positions, with C—H = 0.93 (aromatic) or 0.96 Å (methyl), and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for aromatic H or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

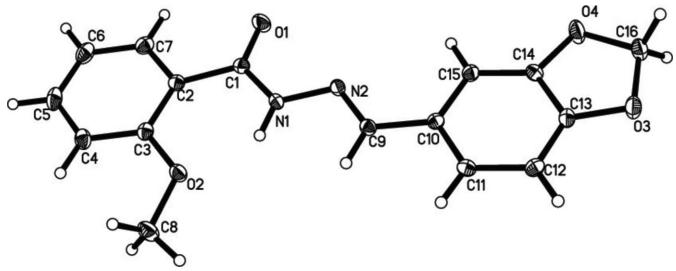


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

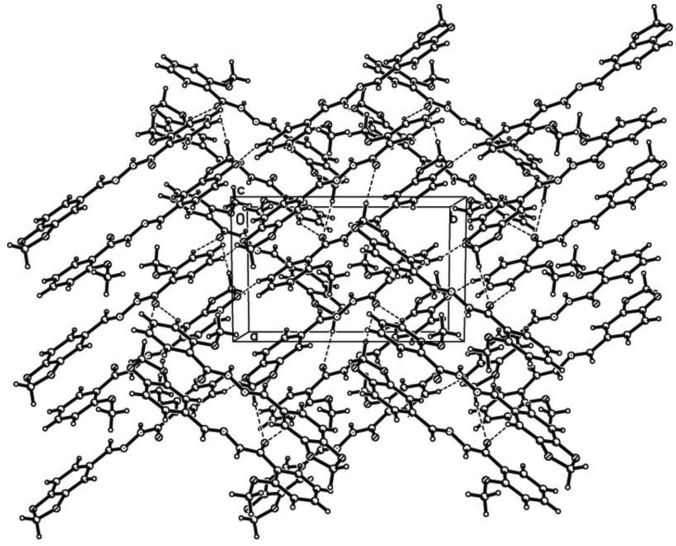


Figure 2
The crystal packing of (I), viewed down the *c* axis. Hydrogen bonds are indicated by dashed lines.

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