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# (*E*)-1,3-Benzodioxole-5-carbaldehyde 4-nitrophenylhydrazone

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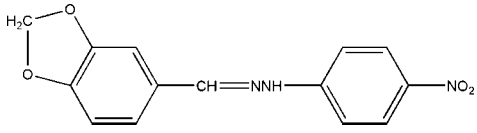
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.051; $wR$ factor = 0.088; data-to-parameter ratio = 8.7.

In the title compound, $C_{14}H_{11}N_3O_4$, the two benzene rings are nearly coplanar, making a dihedral angle of 5.2 (16)°. The crystal packing is consolidated by intermolecular N—H···O hydrogen bonding.

## Related literature

For general background, see: Okabe *et al.* (1993). For related structures, see: Shan *et al.* (2002, 2003).




## Experimental

### *Crystal data*

$C_{14}H_{11}N_3O_4$
$M_r$ = 285.26
Orthorhombic, $Pca2_1$
$a$ = 23.719 (3) Å
$b$ = 4.9052 (18) Å
$c$ = 12.353 (2) Å
$V$ = 1437.2 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 291 (2) K
0.30 × 0.26 × 0.24 mm

### *Data collection*

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
14432 measured reflections
1687 independent reflections
1316 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.062

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.088
$S$ = 1.07
1687 reflections
194 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$A$···O4$^{i}$ | 0.82 (4) | 2.30 (4) | 3.110 (4) | 171 (3) |

Symmetry code: (i) $-x+\frac{3}{2}, y-1, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); 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: XU2338).

## References

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o4406 [ **doi:10.1107/S1600536807051719** ]

# (*E*)-1,3-Benzodioxole-5-carbaldehyde 4-nitrophenylhydrazone

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## Comment

4-Nitrophenylhydrazine has applications in organic synthesis and some of its derivatives have been shown to be potentially DNA-damaging and mutagenic agents (Okabe *et al.*, 1993). As a continuation of this work, we report here the crystal structure of the title compound.

The structure of the title compound is shown in Fig. 1, Selected bond lengths and angles are listed in Table 1. The C6—C1 and C6—C5 bond, close to the imino group, are longer than other aromatic C—C bonds in the same benzene ring, which are consistent with the reported by Shan *et al.* (2002, 2003).

The title molecule crystallizes in the E conformation, with an N2—N1—C7—C1 torsion angle of −176.6 (3)°. The two benzene rings make a dihedral angle of 5.2 (16)°.

The intermolecular N—H···O hydrogen bonding (Table 2) helps to stabilize the crystal structure (Fig. 2).

## Experimental

4-Nitrophenylhydrazine (1 mmol, 0.153 g) was dissolved in anhydrous methanol, $H_2SO_4$ (98% 0.5 ml) was added to this, the mixture was stirred for several minutes at 351 K, 1,3-Benzodioxole-5-carbaldehyde (1 mmol, 0.150 g) in methanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized in dichloromethane, brown single crystals of the title compound were obtained after 2 d.

## Refinement

Imino H atom was located in a difference Fourier map and positional parameters were refined, $U_{iso}$(H) = 1.2$U_{eq}$(N). Other H atoms were placed in calculated positions with C—H = 0.93 Å (aromatic) and 0.97 Å (methylene), and refined in riding mode with $U_{iso}$(H)=1.2$U_{eq}$(C). Friedel pairs were merged.

## Figures

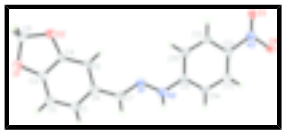

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radii.

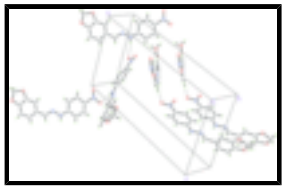

Fig. 2. Packing of (I), showing the intermolecular hydrogen bonds as dashed lines.

### (*E*)-1,3-Benzodioxole-5-carbaldehyde 4-nitrophenylhydrazone

*Crystal data*

| | |
|---|---|
| $C_{14}H_{11}N_3O_4$ | $F_{000}$ = 592 |
| $M_r$ = 285.26 | $D_x$ = 1.318 Mg m$^{-3}$ |
| Orthorhombic, *Pca*2$_1$ | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: P 2c -2ac | Cell parameters from 1143 reflections |
| *a* = 23.719 (3) Å | θ = 2.1–25.6º |
| *b* = 4.9052 (18) Å | μ = 0.10 mm$^{-1}$ |
| *c* = 12.353 (2) Å | *T* = 291 (2) K |
| *V* = 1437.2 (6) Å$^3$ | Block, brown |
| *Z* = 4 | 0.30 × 0.26 × 0.24 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 1316 reflections with $I > 2\sigma(I)$ |
| Radiation source: sealed tube | $R_{int}$ = 0.062 |
| Monochromator: graphite | $\theta_{max}$ = 27.4º |
| *T* = 298(2) K | $\theta_{min}$ = 2.4º |
| φ and ω scans | *h* = −30→29 |
| Absorption correction: none | *k* = −6→6 |
| 14432 measured reflections | *l* = −15→15 |
| 1687 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)]$ = 0.051 | $w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.088 | $(\Delta/\sigma)_{max} < 0.001$ |
| *S* = 1.07 | $\Delta\rho_{max}$ = 0.14 e Å$^{-3}$ |
| 1687 reflections | $\Delta\rho_{min}$ = −0.14 e Å$^{-3}$ |
| 194 parameters | Extinction correction: none |
| 1 restraint | |
| Primary atom site location: structure-invariant direct methods | |
| Secondary atom site location: difference Fourier map | |

*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 $F^2 > 2\text{sigma}(F^2)$ 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 | 0.97237 (14) | −0.2533 (5) | 0.2885 (3) | 0.0355 (7) |
| H1 | 0.9499 | −0.2448 | 0.3502 | 0.043* |
| C2 | 1.01832 (13) | −0.4287 (7) | 0.2850 (3) | 0.0405 (8) |
| H2 | 1.0268 | −0.5372 | 0.3446 | 0.049* |
| C3 | 1.05156 (14) | −0.4423 (7) | 0.1928 (3) | 0.0422 (8) |
| C4 | 1.03942 (13) | −0.2802 (7) | 0.1041 (3) | 0.0443 (8) |
| C5 | 0.99349 (14) | −0.1041 (7) | 0.1062 (3) | 0.0427 (8) |
| H5 | 0.9851 | 0.0034 | 0.0463 | 0.051* |
| C6 | 0.95999 (13) | −0.0902 (6) | 0.1992 (3) | 0.0350 (7) |
| C7 | 0.91205 (14) | 0.0874 (7) | 0.2049 (3) | 0.0452 (8) |
| H7 | 0.8909 | 0.0741 | 0.2680 | 0.054* |
| C8 | 1.11926 (16) | −0.5179 (8) | 0.0645 (3) | 0.0512 (9) |
| H8A | 1.1557 | −0.4302 | 0.0722 | 0.061* |
| H8B | 1.1232 | −0.6724 | 0.0161 | 0.061* |
| C9 | 0.82653 (14) | 0.6057 (7) | 0.0746 (3) | 0.0415 (8) |
| C10 | 0.85405 (17) | 0.6553 (7) | −0.0236 (3) | 0.0464 (9) |
| H10 | 0.8865 | 0.5586 | −0.0414 | 0.056* |
| C11 | 0.83259 (14) | 0.8510 (6) | −0.0950 (3) | 0.0410 (8) |
| H11 | 0.8513 | 0.8880 | −0.1596 | 0.049* |
| C12 | 0.78322 (13) | 0.9903 (7) | −0.0692 (3) | 0.0392 (8) |
| C13 | 0.75576 (14) | 0.9381 (6) | 0.0283 (3) | 0.0360 (7) |
| H13 | 0.7229 | 1.0317 | 0.0456 | 0.043* |
| C14 | 0.77741 (13) | 0.7470 (8) | 0.0995 (3) | 0.0473 (9) |
| H14 | 0.7590 | 0.7129 | 0.1646 | 0.057* |
| N1 | 0.89330 (11) | 0.2693 (6) | 0.1327 (2) | 0.0411 (7) |
| N2 | 0.84735 (12) | 0.4281 (5) | 0.1523 (2) | 0.0346 (6) |
| H2A | 0.8303 (14) | 0.416 (7) | 0.210 (3) | 0.042* |
| N3 | 0.76391 (11) | 1.2097 (5) | −0.1526 (2) | 0.0377 (6) |
| O1 | 1.09794 (10) | −0.6063 (5) | 0.1701 (2) | 0.0479 (6) |
| O2 | 1.07755 (9) | −0.3256 (4) | 0.0227 (2) | 0.0419 (5) |
| O3 | 0.78600 (10) | 1.2221 (4) | −0.23909 (18) | 0.0416 (6) |
| O4 | 0.71404 (9) | 1.3080 (5) | −0.1304 (2) | 0.0454 (6) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0421 (19) | 0.0279 (13) | 0.0365 (17) | −0.0115 (12) | −0.0002 (14) | −0.0008 (13) |
| C2 | 0.0315 (17) | 0.0506 (19) | 0.039 (2) | −0.0092 (15) | −0.0065 (14) | 0.0090 (15) |
| C3 | 0.0454 (19) | 0.0382 (18) | 0.043 (2) | 0.0026 (14) | −0.0013 (16) | 0.0078 (16) |
| C4 | 0.0420 (19) | 0.0357 (17) | 0.055 (2) | −0.0038 (14) | 0.0117 (17) | 0.0213 (16) |
| C5 | 0.0334 (19) | 0.053 (2) | 0.042 (2) | −0.0006 (14) | −0.0035 (15) | 0.0153 (17) |
| C6 | 0.0373 (17) | 0.0304 (14) | 0.0372 (18) | −0.0116 (12) | −0.0058 (15) | −0.0018 (14) |
| C7 | 0.0354 (18) | 0.056 (2) | 0.044 (2) | −0.0034 (15) | −0.0021 (16) | 0.0095 (17) |
| C8 | 0.054 (2) | 0.0439 (19) | 0.056 (2) | 0.0059 (17) | 0.0079 (17) | 0.0071 (17) |
| C9 | 0.0350 (17) | 0.0442 (18) | 0.045 (2) | −0.0027 (14) | 0.0084 (15) | 0.0062 (16) |
| C10 | 0.067 (2) | 0.0361 (16) | 0.037 (2) | 0.0120 (16) | 0.0168 (18) | −0.0051 (15) |
| C11 | 0.0433 (18) | 0.0350 (17) | 0.045 (2) | −0.0080 (14) | 0.0096 (15) | 0.0024 (15) |
| C12 | 0.0322 (16) | 0.0409 (17) | 0.0444 (19) | −0.0121 (14) | −0.0005 (16) | 0.0025 (14) |
| C13 | 0.0408 (18) | 0.0266 (13) | 0.0407 (19) | −0.0054 (13) | 0.0035 (15) | −0.0078 (15) |
| C14 | 0.0349 (18) | 0.048 (2) | 0.059 (2) | −0.0019 (14) | 0.0173 (16) | 0.0171 (17) |
| N1 | 0.0356 (14) | 0.0468 (15) | 0.0409 (16) | −0.0009 (13) | 0.0149 (12) | 0.0120 (13) |
| N2 | 0.0391 (16) | 0.0358 (13) | 0.0290 (14) | −0.0037 (12) | 0.0019 (11) | −0.0101 (12) |
| N3 | 0.0377 (16) | 0.0385 (14) | 0.0369 (17) | −0.0112 (11) | −0.0082 (14) | −0.0055 (12) |
| O1 | 0.0472 (15) | 0.0455 (13) | 0.0509 (16) | 0.0054 (10) | −0.0010 (12) | 0.0072 (11) |
| O2 | 0.0417 (13) | 0.0405 (12) | 0.0436 (13) | −0.0056 (10) | 0.0021 (11) | 0.0026 (11) |
| O3 | 0.0494 (13) | 0.0406 (12) | 0.0346 (14) | 0.0218 (10) | −0.0060 (12) | −0.0059 (10) |
| O4 | 0.0431 (13) | 0.0433 (12) | 0.0497 (15) | −0.0070 (10) | 0.0105 (12) | 0.0169 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.389 (5) | C8—H8B | 0.9700 |
| C1—C6 | 1.394 (5) | C9—N2 | 1.388 (4) |
| C1—H1 | 0.9300 | C9—C14 | 1.390 (5) |
| C2—C3 | 1.387 (5) | C9—C10 | 1.399 (5) |
| C2—H2 | 0.9300 | C10—C11 | 1.399 (5) |
| C3—C4 | 1.385 (5) | C10—H10 | 0.9300 |
| C3—O1 | 1.392 (4) | C11—C12 | 1.393 (5) |
| C4—O2 | 1.371 (4) | C11—H11 | 0.9300 |
| C4—C5 | 1.391 (5) | C12—C13 | 1.393 (4) |
| C5—C6 | 1.399 (5) | C12—N3 | 1.558 (4) |
| C5—H5 | 0.9300 | C13—C14 | 1.384 (5) |
| C6—C7 | 1.434 (4) | C13—H13 | 0.9300 |
| C7—N1 | 1.338 (4) | C14—H14 | 0.9300 |
| C7—H7 | 0.9300 | N1—N2 | 1.361 (4) |
| C8—O2 | 1.461 (4) | N2—H2A | 0.82 (4) |
| C8—O1 | 1.464 (5) | N3—O3 | 1.192 (4) |
| C8—H8A | 0.9700 | N3—O4 | 1.307 (3) |
| C2—C1—C6 | 119.8 (3) | N2—C9—C14 | 117.2 (3) |
| C2—C1—H1 | 120.1 | N2—C9—C10 | 122.9 (3) |
| C6—C1—H1 | 120.1 | C14—C9—C10 | 119.8 (3) |

| | | | |
|---|---|---|---|
| C3—C2—C1 | 120.1 (3) | C9—C10—C11 | 119.7 (3) |
| C3—C2—H2 | 120.0 | C9—C10—H10 | 120.1 |
| C1—C2—H2 | 120.0 | C11—C10—H10 | 120.1 |
| C4—C3—C2 | 120.3 (3) | C12—C11—C10 | 119.9 (3) |
| C4—C3—O1 | 109.7 (3) | C12—C11—H11 | 120.1 |
| C2—C3—O1 | 130.0 (3) | C10—C11—H11 | 120.1 |
| O2—C4—C3 | 110.5 (3) | C11—C12—C13 | 120.1 (3) |
| O2—C4—C5 | 129.1 (3) | C11—C12—N3 | 115.8 (3) |
| C3—C4—C5 | 120.3 (3) | C13—C12—N3 | 124.1 (3) |
| C4—C5—C6 | 119.4 (3) | C14—C13—C12 | 120.0 (3) |
| C4—C5—H5 | 120.3 | C14—C13—H13 | 120.0 |
| C6—C5—H5 | 120.3 | C12—C13—H13 | 120.0 |
| C1—C6—C5 | 120.2 (3) | C13—C14—C9 | 120.5 (3) |
| C1—C6—C7 | 118.5 (3) | C13—C14—H14 | 119.8 |
| C5—C6—C7 | 121.3 (3) | C9—C14—H14 | 119.8 |
| N1—C7—C6 | 129.5 (3) | C7—N1—N2 | 121.9 (3) |
| N1—C7—H7 | 115.2 | N1—N2—C9 | 121.4 (3) |
| C6—C7—H7 | 115.2 | N1—N2—H2A | 121 (2) |
| O2—C8—O1 | 105.8 (3) | C9—N2—H2A | 118 (2) |
| O2—C8—H8A | 110.6 | O3—N3—O4 | 124.6 (3) |
| O1—C8—H8A | 110.6 | O3—N3—C12 | 119.9 (3) |
| O2—C8—H8B | 110.6 | O4—N3—C12 | 112.5 (2) |
| O1—C8—H8B | 110.6 | C3—O1—C8 | 106.4 (2) |
| H8A—C8—H8B | 108.7 | C4—O2—C8 | 107.0 (3) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2A···O4$^{i}$ | 0.82 (4) | 2.30 (4) | 3.110 (4) | 171 (3) |

Symmetry codes: (i) −*x*+3/2, *y*−1, *z*+1/2.

**Fig. 1**

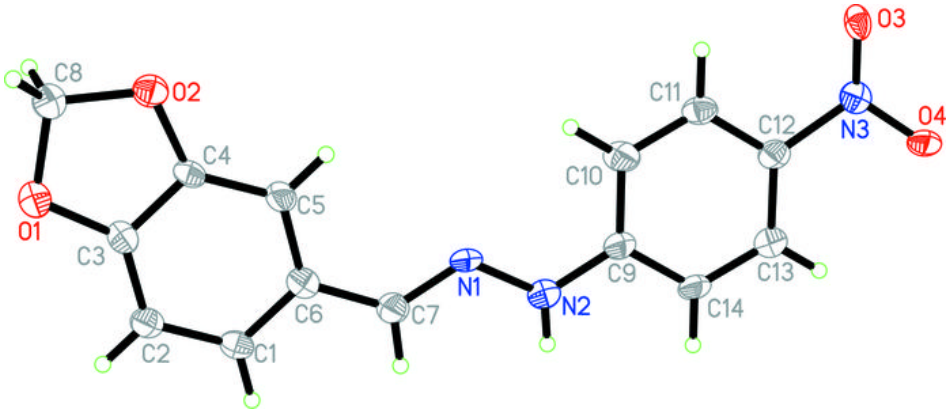

**Fig. 2**