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N'-Cyclohexylidene-2-hydroxybenzohydrazide

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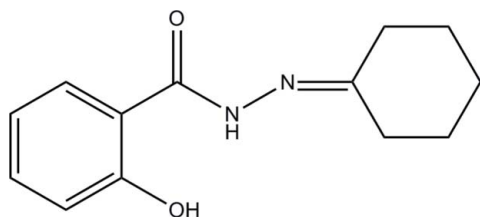
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.047; wR factor = 0.138; data-to-parameter ratio = 7.1.

In the title molecule, $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_2$, the cyclohexylidene ring adopts a chair conformation. The intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond influences the molecular conformation: the benzene ring and the mean plane of the central $\text{C}(\text{O})\text{NHN}$ fragment form a dihedral angle of $4.9(1)$ Å. In the crystal, intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into chains propagated along $[001]$.

Related literature

For properties of Schiff-base derivatives, see Sreeja *et al.* (2003). For a related structure, see Luo *et al.* (2007).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_2$
 $M_r = 232.28$
 Monoclinic, Cc
 $a = 18.376(2)$ Å
 $b = 5.3386(10)$ Å
 $c = 12.9435(15)$ Å

 $\beta = 102.241(2)^\circ$
 $V = 1240.9(3)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.39 \times 0.29 \times 0.27$ mm

Data collection

 Bruker SMART APEX CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.968$, $T_{\max} = 0.977$

 2969 measured reflections
 1090 independent reflections
 898 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.138$
 $S = 1.08$
 1090 reflections
 154 parameters

 2 restraints
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.28$ e Å⁻³
 $\Delta\rho_{\min} = -0.15$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{N1}-\text{H1}\cdots\text{O2}$ | 0.86 | 1.97 | 2.655 (4) | 136 |
| $\text{O2}-\text{H2}\cdots\text{O1}^1$ | 0.82 | 2.15 | 2.704 (4) | 125 |

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

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; 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: CV2521).

References

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 Sreeja, P. B., Sreekanth, A., Nayar, C. R., Prathapachandra Kurup, M. R., Usman, A., Razak, I. A., Chantrapromma, S. & Fun, H. K. (2003). *J. Mol. Struct.* **645**, 221–226.

supplementary materials

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N'-Cyclohexylidene-2-hydroxybenzohydrazide

D. Liu

Comment

Chemistry of Schiff bases has been intensively investigated in recent years, owing to their coordination properties and diverse applications. Schiff base derivatives and their complexes have been studied for their antifungal and antibacterial activity, and as antiviral drugs (Sreeja *et al.*, 2003). In this paper, we present the crystal structure of the title compound, (I), which was synthesized by the reaction of cyclohexanone and salicyloyl hydrazide.

In (I) (Fig. 1), the bond lengths and angles are normal and comparable to those observed in the compound reported by Luo *et al.* (2007). The cyclohexylidene ring adopts a chair conformation. Intramolecular N—H \cdots O hydrogen bond (Table 1) influences the molecular conformation. The dihedral angle between the benzene ring and the plane C1/N1/N2 is 4.9 (1) Å. The plane C1/N1/N2 and ring C8-C13 form a dihedral angle of 37.7 (3) Å. Intermolecular O—H \cdots O hydrogen bonds (Table 1) link the molecules into chains propagated in direction [001].

Experimental

Salicyloyl hydrazide (5 mmol) and cyclohexanone (5 mmol), 20 ml ethanol were mixed in 50 ml flask. After stirring 30 min at 353 K, the mixture then cooling slowly to room temperature and affording the title compound, then recrystallized from ethanol, affording the title compound as a red crystalline solid. Elemental analysis: calculated for C₁₃H₁₆N₂O₂: C 67.22, H 6.94, N 12.06%; found: C 67.29, H 6.85, N 12.24%.

Refinement

All H atoms were placed in geometrically idealized positions (N—H 0.86 Å, O—H 0.82 Å and C—H=0.93–0.97 Å) and treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}$ of the parent atom. In the absence of any significant anomalous scatterers in the molecule, 330 Friedel pairs were merged before the final refinement.

Figures

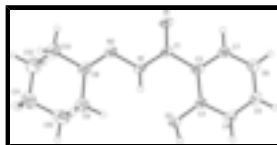


Fig. 1. The molecular structure of (I) with the atomic numbering scheme and 30% probability displacement ellipsoids.

(I)

Crystal data

C₁₃H₁₆N₂O₂

$F_{000} = 496$

supplementary materials

$M_r = 232.28$

Monoclinic, Cc

$a = 18.376$ (2) Å

$b = 5.3386$ (10) Å

$c = 12.9435$ (15) Å

$\beta = 102.241$ (2)°

$V = 1240.9$ (3) Å³

$Z = 4$

$D_x = 1.243$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1322 reflections

$\theta = 3.2$ – 27.5 °

$\mu = 0.09$ mm⁻¹

$T = 293$ K

Block, red

$0.39 \times 0.29 \times 0.27$ mm

Data collection

Bruker SMART Apex CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ K

phi and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.968$, $T_{\max} = 0.977$

2969 measured reflections

1090 independent reflections

898 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.3$ °

$h = -21 \rightarrow 11$

$k = -6 \rightarrow 6$

$l = -15 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.138$

$S = 1.08$

1090 reflections

154 parameters

2 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

$$w = 1/[\sigma^2(F_o^2) + (0.0866P)^2 + 0.3353P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.28$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.15$ e Å⁻³

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|-------------|-------------|----------------------------------|
| N1 | 0.18964 (17) | 0.6052 (6) | 0.1521 (2) | 0.0432 (8) |
| H1 | 0.1776 | 0.6179 | 0.2126 | 0.052* |
| N2 | 0.15740 (18) | 0.7600 (7) | 0.0684 (2) | 0.0475 (8) |
| O1 | 0.2584 (2) | 0.4124 (5) | 0.0501 (2) | 0.0577 (8) |
| O2 | 0.20359 (16) | 0.4399 (5) | 0.3488 (2) | 0.0511 (8) |
| H2 | 0.1965 | 0.4152 | 0.4084 | 0.077* |
| C1 | 0.24015 (19) | 0.4354 (7) | 0.1362 (2) | 0.0371 (8) |
| C2 | 0.27283 (19) | 0.2675 (7) | 0.2264 (3) | 0.0369 (8) |
| C3 | 0.25343 (18) | 0.2669 (6) | 0.3267 (3) | 0.0370 (8) |
| C4 | 0.2836 (2) | 0.0878 (7) | 0.4024 (3) | 0.0465 (10) |
| H4 | 0.2699 | 0.0859 | 0.4675 | 0.056* |
| C5 | 0.3333 (2) | -0.0845 (8) | 0.3810 (3) | 0.0524 (11) |
| H5 | 0.3533 | -0.2032 | 0.4317 | 0.063* |
| C6 | 0.3545 (2) | -0.0838 (7) | 0.2838 (3) | 0.0505 (11) |
| H6 | 0.3890 | -0.1997 | 0.2699 | 0.061* |
| C7 | 0.3240 (2) | 0.0891 (7) | 0.2088 (3) | 0.0422 (9) |
| H7 | 0.3380 | 0.0871 | 0.1439 | 0.051* |
| C8 | 0.1097 (3) | 0.9202 (8) | 0.0854 (3) | 0.0531 (11) |
| C9 | 0.0802 (3) | 0.9560 (10) | 0.1848 (4) | 0.0681 (13) |
| H9A | 0.0993 | 0.8241 | 0.2348 | 0.082* |
| H9B | 0.0976 | 1.1151 | 0.2170 | 0.082* |
| C10 | -0.0029 (3) | 0.9511 (13) | 0.1616 (5) | 0.0863 (18) |
| H10A | -0.0200 | 0.9941 | 0.2253 | 0.104* |
| H10B | -0.0198 | 0.7824 | 0.1415 | 0.104* |
| C11 | -0.0380 (3) | 1.1323 (12) | 0.0730 (5) | 0.0874 (17) |
| H11A | -0.0917 | 1.1123 | 0.0572 | 0.105* |
| H11B | -0.0266 | 1.3034 | 0.0961 | 0.105* |
| C12 | -0.0081 (3) | 1.0816 (13) | -0.0264 (5) | 0.0849 (17) |
| H12A | -0.0253 | 0.9184 | -0.0543 | 0.102* |
| H12B | -0.0280 | 1.2059 | -0.0795 | 0.102* |
| C13 | 0.0746 (3) | 1.0884 (9) | -0.0060 (4) | 0.0690 (14) |
| H13A | 0.0914 | 1.2591 | 0.0096 | 0.083* |
| H13B | 0.0908 | 1.0354 | -0.0691 | 0.083* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| N1 | 0.0527 (19) | 0.059 (2) | 0.0189 (15) | 0.0072 (17) | 0.0087 (13) | 0.0043 (13) |
| N2 | 0.0493 (18) | 0.067 (2) | 0.0256 (16) | 0.0066 (17) | 0.0075 (13) | 0.0104 (14) |
| O1 | 0.082 (2) | 0.0684 (18) | 0.0267 (14) | 0.0165 (16) | 0.0210 (13) | 0.0059 (12) |
| O2 | 0.0656 (18) | 0.0681 (18) | 0.0219 (13) | 0.0188 (15) | 0.0142 (12) | 0.0037 (12) |
| C1 | 0.044 (2) | 0.046 (2) | 0.0212 (19) | -0.0032 (16) | 0.0065 (15) | -0.0014 (14) |

supplementary materials

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C2 | 0.0394 (19) | 0.046 (2) | 0.0239 (18) | -0.0062 (16) | 0.0031 (14) | -0.0038 (14) |
| C3 | 0.0407 (19) | 0.0477 (19) | 0.0219 (17) | -0.0004 (18) | 0.0048 (14) | -0.0020 (15) |
| C4 | 0.052 (2) | 0.059 (2) | 0.029 (2) | 0.004 (2) | 0.0115 (17) | 0.0057 (17) |
| C5 | 0.056 (3) | 0.056 (2) | 0.043 (3) | 0.008 (2) | 0.0052 (19) | 0.0111 (18) |
| C6 | 0.052 (2) | 0.055 (2) | 0.045 (3) | 0.0075 (19) | 0.014 (2) | -0.0001 (18) |
| C7 | 0.048 (2) | 0.051 (2) | 0.0291 (19) | 0.0004 (18) | 0.0118 (16) | -0.0045 (16) |
| C8 | 0.053 (2) | 0.073 (3) | 0.033 (2) | 0.002 (2) | 0.0079 (18) | 0.0075 (19) |
| C9 | 0.075 (3) | 0.086 (3) | 0.043 (3) | 0.027 (3) | 0.012 (2) | 0.005 (2) |
| C10 | 0.082 (4) | 0.110 (5) | 0.076 (4) | 0.015 (3) | 0.037 (3) | -0.001 (3) |
| C11 | 0.072 (3) | 0.116 (4) | 0.072 (4) | 0.034 (3) | 0.008 (3) | -0.001 (3) |
| C12 | 0.075 (4) | 0.115 (5) | 0.057 (3) | 0.023 (3) | -0.002 (3) | 0.003 (3) |
| C13 | 0.075 (3) | 0.076 (3) | 0.053 (3) | 0.007 (3) | 0.007 (2) | 0.016 (2) |

Geometric parameters (Å, °)

| | | | |
|----------|-----------|---------------|-----------|
| N1—C1 | 1.344 (5) | C7—H7 | 0.9300 |
| N1—N2 | 1.391 (4) | C8—C9 | 1.510 (6) |
| N1—H1 | 0.8600 | C8—C13 | 1.516 (6) |
| N2—C8 | 1.277 (5) | C9—C10 | 1.493 (8) |
| O1—C1 | 1.236 (5) | C9—H9A | 0.9700 |
| O2—C3 | 1.373 (4) | C9—H9B | 0.9700 |
| O2—H2 | 0.8200 | C10—C11 | 1.534 (9) |
| C1—C2 | 1.493 (5) | C10—H10A | 0.9700 |
| C2—C7 | 1.391 (5) | C10—H10B | 0.9700 |
| C2—C3 | 1.417 (4) | C11—C12 | 1.526 (9) |
| C3—C4 | 1.398 (5) | C11—H11A | 0.9700 |
| C4—C5 | 1.365 (6) | C11—H11B | 0.9700 |
| C4—H4 | 0.9300 | C12—C13 | 1.486 (8) |
| C5—C6 | 1.394 (6) | C12—H12A | 0.9700 |
| C5—H5 | 0.9300 | C12—H12B | 0.9700 |
| C6—C7 | 1.370 (6) | C13—H13A | 0.9700 |
| C6—H6 | 0.9300 | C13—H13B | 0.9700 |
| C1—N1—N2 | 118.4 (3) | C10—C9—H9A | 109.4 |
| C1—N1—H1 | 120.8 | C8—C9—H9A | 109.4 |
| N2—N1—H1 | 120.8 | C10—C9—H9B | 109.4 |
| C8—N2—N1 | 117.3 (3) | C8—C9—H9B | 109.4 |
| C3—O2—H2 | 109.5 | H9A—C9—H9B | 108.0 |
| O1—C1—N1 | 122.2 (3) | C9—C10—C11 | 112.9 (5) |
| O1—C1—C2 | 120.3 (3) | C9—C10—H10A | 109.0 |
| N1—C1—C2 | 117.5 (3) | C11—C10—H10A | 109.0 |
| C7—C2—C3 | 117.3 (3) | C9—C10—H10B | 109.0 |
| C7—C2—C1 | 117.2 (3) | C11—C10—H10B | 109.0 |
| C3—C2—C1 | 125.4 (3) | H10A—C10—H10B | 107.8 |
| O2—C3—C4 | 119.8 (3) | C12—C11—C10 | 110.4 (5) |
| O2—C3—C2 | 119.9 (3) | C12—C11—H11A | 109.6 |
| C4—C3—C2 | 120.3 (3) | C10—C11—H11A | 109.6 |
| C5—C4—C3 | 120.1 (4) | C12—C11—H11B | 109.6 |
| C5—C4—H4 | 119.9 | C10—C11—H11B | 109.6 |
| C3—C4—H4 | 119.9 | H11A—C11—H11B | 108.1 |

| | | | |
|-----------|-----------|---------------|-----------|
| C4—C5—C6 | 120.5 (4) | C13—C12—C11 | 112.5 (5) |
| C4—C5—H5 | 119.7 | C13—C12—H12A | 109.1 |
| C6—C5—H5 | 119.7 | C11—C12—H12A | 109.1 |
| C7—C6—C5 | 119.4 (4) | C13—C12—H12B | 109.1 |
| C7—C6—H6 | 120.3 | C11—C12—H12B | 109.1 |
| C5—C6—H6 | 120.3 | H12A—C12—H12B | 107.8 |
| C6—C7—C2 | 122.3 (4) | C12—C13—C8 | 112.0 (5) |
| C6—C7—H7 | 118.8 | C12—C13—H13A | 109.2 |
| C2—C7—H7 | 118.8 | C8—C13—H13A | 109.2 |
| N2—C8—C9 | 128.0 (4) | C12—C13—H13B | 109.2 |
| N2—C8—C13 | 117.1 (4) | C8—C13—H13B | 109.2 |
| C9—C8—C13 | 114.8 (4) | H13A—C13—H13B | 107.9 |
| C10—C9—C8 | 111.3 (4) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|--------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N1—H1 \cdots O2 | 0.86 | 1.97 | 2.655 (4) | 136 |
| O2—H2 \cdots O1 ⁱ | 0.82 | 2.15 | 2.704 (4) | 125 |

Symmetry codes: (i) $x, -y+1, z+1/2$.

Fig. 1

