

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N,N'-DisalicyloylhydrazineYu-Ting Chen,^{a,b} Da-Cheng Li,^{a*} Da-Qi Wang^a and Yue-Hua Zhu^c

^aSchool of Chemistry and Chemical Engineering, Liaocheng University, Liaocheng 252059, People's Republic of China, ^bDepartment of Chemistry, Dezhou University, Dezhou 253023, People's Republic of China, and ^cSchool of Materials Science and Engineering, Liaocheng University, Liaocheng 252059, People's Republic of China
Correspondence e-mail: dougroup@163.com

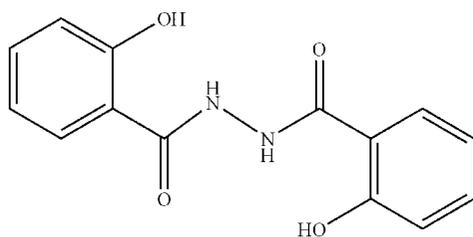
Received 21 November 2007; accepted 25 November 2007

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.047; wR factor = 0.163; data-to-parameter ratio = 12.0.

The approximately planar molecule of the title compound, $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_4$, is centrosymmetric and has an *E* configuration with respect to the N—N bond. This compound adopts the ketoamine form with C=O and C—N distances of 1.233 (3) and 1.331 (4) Å, respectively. Adjacent molecules are assembled into a two-dimensional supramolecular structure parallel to the (101) plane *via* intermolecular O—H...O hydrogen bonds.

Related literature

For metallacrowns with unsymmetrical aroylhydrazone ligands, see: John *et al.* (2006); Dou *et al.* (2006). For the crystal structure of an iron compound with *N,N'*-bis-picolinoyl hydrazine, see: Bernhardt *et al.* (2005). For the preparation of 2-acetyl-2-hydroxynaphthohydrazide, see: Liu *et al.* (2006).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_4$
 $M_r = 272.26$

Monoclinic, $P2_1/n$
 $a = 8.3816$ (18) Å

$b = 6.2909$ (15) Å
 $c = 12.376$ (2) Å
 $\beta = 105.463$ (2)°
 $V = 628.9$ (2) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 298$ (2) K
 $0.18 \times 0.15 \times 0.14$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.981$, $T_{\max} = 0.985$

3082 measured reflections
1102 independent reflections
618 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.163$
 $S = 1.03$
1102 reflections

92 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2}\cdots\text{O1}^{\text{ii}}$	0.82	1.81	2.617 (3)	166
$\text{N1}-\text{H1}\cdots\text{O2}$	0.86	1.89	2.580 (3)	136

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

Data collection: SMART (Siemens, 1996); cell refinement: SMART; data reduction: SAINT (Siemens, 1996); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

The authors acknowledge the support of the National Natural Science Foundation of China (20671048).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2060).

References

- Bernhardt, P. V., Chin, P., Sharpe, P. C., Wang, J. C. & Richardson, D. R. (2005). *Biol. Inorg. Chem.* **10**, 761–777.
 Dou, J. M., Liu, M. L., Li, D. C. & Wang, D. Q. (2006). *Eur. J. Inorg. Chem.* **23**, 4866–4871.
 John, R. P., Park, J., Moon, D., Lee, K. & Lah, M. S. (2006). *Chem. Commun.* pp. 3699–3701.
 Liu, M.-L., Dou, J.-M., Li, D.-C. & Wang, D.-Q. (2006). *Acta Cryst.* **E62**, o1009–o1010.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997a). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supporting information

Acta Cryst. (2008). E64, o120 [https://doi.org/10.1107/S160053680706312X]

N,N'-Disalicyloylhydrazine

Yu-Ting Chen, Da-Cheng Li, Da-Qi Wang and Yue-Hua Zhu

S1. Comment

Aroylhydrazine ligands have recently gained the increasing concern due to their quite interesting chemical activities (John *et al.*, 2006; Dou *et al.*, 2006). However, most of the studies are focused on unsymmetrical aroylhydrazine, while symmetrical diaroylhydrazines receive much less attention (Bernhardt *et al.*, 2005). In order to explore the impact of the structural character of symmetrical ligands on the properties of the complexes, the title compound, was synthesized by the self-combination of salicyloylhydrazine on the acidic environment.

The title molecule has crystallographic inversion symmetry (Fig. 1) and goes near to co-planar with the mean deviation of 0.0584 Å from the least-squares plane of all non-hydrogen atoms. An E configuration with respect to the N—N bond is observed. The distance of C1—O1 is 1.233 (3) Å, typical of a double bond, whereas the distances of C1—N1 and N1—N1ⁱ at 1.331 (4) Å and 1.373 (4) Å, respectively are typical for a single bond (Table. 1), which is in agreement with that of the analogous compound (Liu *et al.*, 2006), suggesting this diaroylhydrazine exists in the ketoamino form. All oxygen atoms in the title compound participate in intermolecular H-bond interactions with their neighbors, leading to one molecule bound with four molecules through O—H···O interactions. The dihedral angle of two adjacent molecules linked by O—H···O hydrogen bond is 65.7°. In such a recognition pattern, the two-dimensional network structure is assembled parallel to the (1 0 1) plane, as shown in Fig. 2.

S2. Experimental

The salicyloylhydrazine (6.08 g, 40 mmol) was added to the solution of ice acetic acid (3 ml) in methanol (20 ml). After refluxed for three hours, the mixture was filtrated. Then colorless needle crystals suitable for X-ray diffraction were obtained by vaporizing the filtrate at room temperature. Yield: 4.23 g, 77.76%. m.p.: 565–567 K. Anal. for C₁₄H₁₂N₂O₄: Calc. C, 61.76; H, 4.44; N, 10.29; Found: C, 61.52; H, 4.51; N, 10.28%. The No. of CCDC: 614757.

S3. Refinement

The H atoms on the ligands were allowed to ride on their parent atoms with C(*sp*₂ hybrid)-H distances of 0.93 Å and $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$.

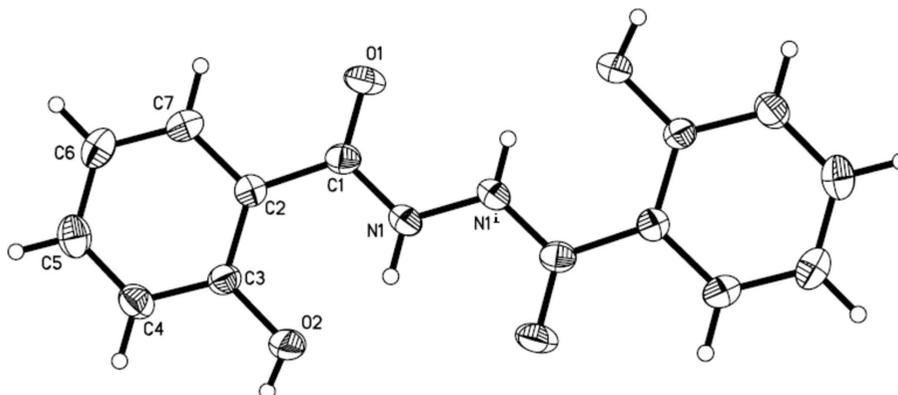


Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level. Symmetry code (i): $-x + 2, -y + 1, -z + 1$

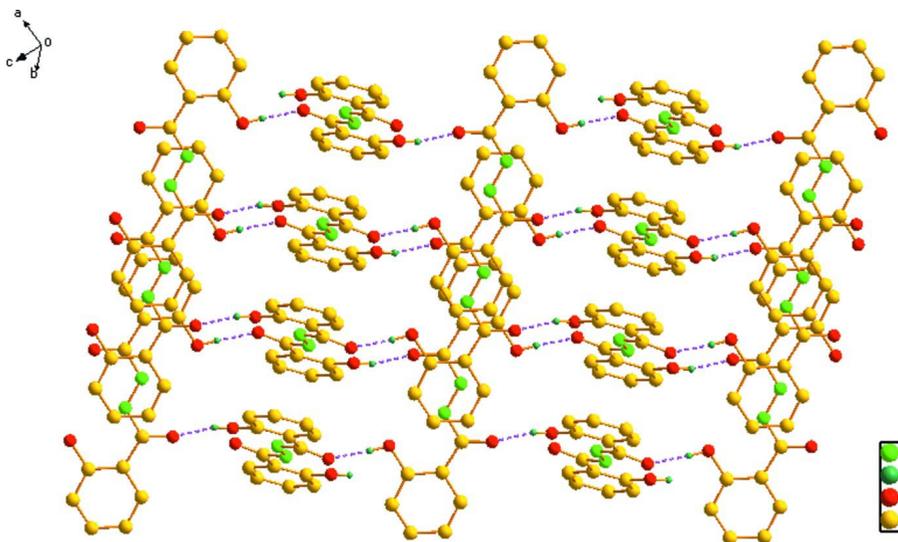


Figure 2

Two-dimensional network of the compound. Symmetry code (ii): $x + 1/2, -y + 3/2, z + 1/2$.

N,N'-Disalicyloylhydrazine

Crystal data

$C_{14}H_{12}N_2O_4$

$M_r = 272.26$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1n$

$a = 8.3816 (18) \text{ \AA}$

$b = 6.2909 (15) \text{ \AA}$

$c = 12.376 (2) \text{ \AA}$

$\beta = 105.463 (2)^\circ$

$V = 628.9 (2) \text{ \AA}^3$

$Z = 2$

$F(000) = 284$

$D_x = 1.438 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 678 reflections

$\theta = 2.6\text{--}25.5^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, colorless

$0.18 \times 0.15 \times 0.14 \text{ mm}$

Data collection

Bruker SMART 1000 CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.981$, $T_{\max} = 0.985$

3082 measured reflections
1102 independent reflections
618 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.6^\circ$
 $h = -9 \rightarrow 9$
 $k = -7 \rightarrow 7$
 $l = -6 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.163$
 $S = 1.03$
1102 reflections
92 parameters
0 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
 $w = 1/[\sigma^2(F_o^2) + (0.0827P)^2 + 0.0895P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.30 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.9962 (3)	0.5894 (4)	0.53095 (19)	0.0462 (7)
H1	1.0781	0.6229	0.5867	0.055*
O1	0.7444 (2)	0.6612 (3)	0.42420 (17)	0.0576 (7)
O2	1.1131 (3)	0.8180 (4)	0.70745 (18)	0.0696 (8)
H2	1.1607	0.8443	0.7730	0.104*
C1	0.8612 (4)	0.7109 (5)	0.5042 (2)	0.0419 (8)
C2	0.8605 (3)	0.9011 (4)	0.5739 (2)	0.0372 (7)
C3	0.9807 (3)	0.9503 (5)	0.6721 (2)	0.0419 (7)
C4	0.9670 (4)	1.1325 (5)	0.7318 (3)	0.0502 (9)
H4	1.0485	1.1646	0.7971	0.060*
C5	0.8344 (4)	1.2655 (6)	0.6953 (3)	0.0550 (9)
H5	0.8257	1.3872	0.7360	0.066*
C6	0.7137 (4)	1.2198 (5)	0.5984 (3)	0.0550 (9)
H6	0.6238	1.3105	0.5737	0.066*
C7	0.7265 (3)	1.0410 (5)	0.5388 (3)	0.0475 (8)
H7	0.6446	1.0114	0.4734	0.057*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0449 (14)	0.0496 (16)	0.0396 (15)	-0.0013 (12)	0.0033 (12)	-0.0122 (11)
O1	0.0524 (13)	0.0698 (16)	0.0397 (12)	-0.0048 (11)	-0.0066 (10)	-0.0061 (11)
O2	0.0648 (16)	0.0800 (17)	0.0481 (14)	0.0250 (13)	-0.0125 (11)	-0.0230 (13)
C1	0.0430 (18)	0.0471 (18)	0.0336 (15)	-0.0016 (14)	0.0067 (14)	0.0045 (14)
C2	0.0375 (16)	0.0409 (17)	0.0357 (16)	-0.0006 (13)	0.0141 (13)	0.0018 (13)
C3	0.0370 (15)	0.0487 (17)	0.0384 (16)	0.0066 (14)	0.0073 (13)	-0.0016 (15)
C4	0.0476 (19)	0.056 (2)	0.0465 (18)	-0.0038 (16)	0.0127 (15)	-0.0124 (16)
C5	0.063 (2)	0.0485 (19)	0.061 (2)	-0.0016 (17)	0.0303 (19)	-0.0077 (17)
C6	0.054 (2)	0.050 (2)	0.065 (2)	0.0137 (16)	0.0226 (18)	0.0076 (18)
C7	0.0389 (16)	0.055 (2)	0.0465 (18)	0.0052 (15)	0.0079 (14)	0.0074 (16)

Geometric parameters (\AA , $^\circ$)

N1—C1	1.331 (3)	C3—C4	1.384 (4)
N1—N1 ⁱ	1.372 (4)	C4—C5	1.368 (4)
N1—H1	0.8600	C4—H4	0.9300
O1—C1	1.233 (3)	C5—C6	1.377 (5)
O2—C3	1.363 (3)	C5—H5	0.9300
O2—H2	0.8200	C6—C7	1.365 (4)
C1—C2	1.476 (4)	C6—H6	0.9300
C2—C3	1.391 (4)	C7—H7	0.9300
C2—C7	1.402 (4)		
C1—N1—N1 ⁱ	119.7 (3)	C5—C4—C3	120.4 (3)
C1—N1—H1	120.2	C5—C4—H4	119.8
N1 ⁱ —N1—H1	120.2	C3—C4—H4	119.8
C3—O2—H2	109.5	C4—C5—C6	120.2 (3)
O1—C1—N1	119.6 (3)	C4—C5—H5	119.9
O1—C1—C2	123.3 (3)	C6—C5—H5	119.9
N1—C1—C2	117.1 (2)	C7—C6—C5	119.8 (3)
C3—C2—C7	117.7 (3)	C7—C6—H6	120.1
C3—C2—C1	125.2 (2)	C5—C6—H6	120.1
C7—C2—C1	117.1 (2)	C6—C7—C2	121.4 (3)
O2—C3—C4	120.7 (3)	C6—C7—H7	119.3
O2—C3—C2	118.8 (3)	C2—C7—H7	119.3
C4—C3—C2	120.5 (3)		
N1 ⁱ —N1—C1—O1	0.1 (5)	C1—C2—C3—C4	-179.4 (3)
N1 ⁱ —N1—C1—C2	-180.0 (3)	O2—C3—C4—C5	179.4 (3)
O1—C1—C2—C3	172.2 (3)	C2—C3—C4—C5	0.5 (4)
N1—C1—C2—C3	-7.7 (4)	C3—C4—C5—C6	-0.4 (5)
O1—C1—C2—C7	-6.7 (4)	C4—C5—C6—C7	0.1 (5)
N1—C1—C2—C7	173.3 (3)	C5—C6—C7—C2	0.1 (5)
C7—C2—C3—O2	-179.3 (3)	C3—C2—C7—C6	0.1 (4)

C1—C2—C3—O2	1.7 (4)	C1—C2—C7—C6	179.1 (3)
C7—C2—C3—C4	-0.4 (4)		

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

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
O2—H2...O1 ⁱⁱ	0.82	1.81	2.617 (3)	166
N1—H1...O2	0.86	1.89	2.580 (3)	136

Symmetry code: (ii) $x+1/2, -y+3/2, z+1/2$.