Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(3*Z*)-3-[(*Z*)-2-(2-Oxoindolin-3-ylidene)hydrazin-1-ylidene]indolin-2-one 0.17-hydrate

Yong-Hong Liu,[a]* Lei Zhao,[b] Ming-Xuan Liu,[b] Hai Lin[b] and Jing-Jing Li[b]

[a]College of Chemistry and Chemical Engineering, Yangzhou University, Jiangsu Provincial Key Laboratory of Environmental Material & Engineering, Yangzhou, 225002, People's Republic of China, and [b]College of Chemistry and Chemical Engineering, Yangzhou University, Yangzhou 225002, People's Republic of China
Correspondence e-mail: yhliu@yzu.edu.cn

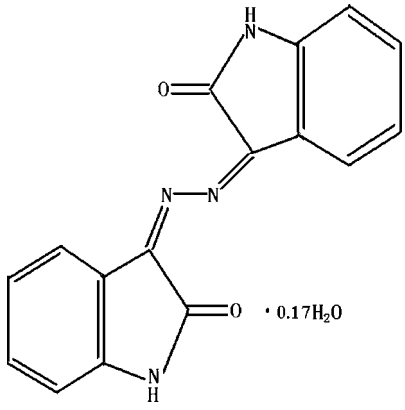
Received 4 May 2014; accepted 21 May 2014

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; H-atom completeness 97%; disorder in solvent or counterion; R factor = 0.039; wR factor = 0.108; data-to-parameter ratio = 15.0.

In the title compound, $C_{16}H_{10}N_4O_2\cdot0.17H_2O$, prepared by the one-step condensation reaction of isatin with hydrazine hydrate under microwave irradiation, the complete organic molecule is generated by crystallographic inversion symmetry and therefore exists in an *S-trans* conformation. In the crystal, molecules are linked by N—H···O hydrogen bonds, generating a three-dimensional framework with [001] channels, which are occupied by the disordered water molecules.

Related literature

For background to microwave synthesis, see: Hoz *et al.* (2004); Jagani *et al.* (2012). For our previous work in this area, see: Liu *et al.* (2008); Wang *et al.* (2010). For the coventional synthesis of the title compound, see: Ali & Alam (1994).



Experimental

Crystal data

$C_{16}H_{10}N_4O_2\cdot0.17H_2O$
M_r = 308.30
Trigonal, $R\overline{3}$
a = 24.8699 (18) Å
c = 5.6603 (8) Å
V = 3031.9 (5) Å^3
Z = 9
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.38 × 0.16 × 0.14 mm

Data collection

Bruker SMART1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.963, T_{max} = 0.986
8691 measured reflections
1547 independent reflections
1290 reflections with $I > 2\sigma$igma(I)
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.108
S = 1.01
1547 reflections
103 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.13	2.8951 (17)	148

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

The authors thank the Priority Academic Program Development of Jiangsu Higher Education Institutions and the National Disciplining and Cultivating Key of Physics and Chemistry in Yangzhou University (070304) for financial support of this work.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7227).

References

Ali, S. & Alam, M. (1994). *Arch. Pharm. Res.* **17**, 131–133.
Bruker (2002). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hoz, A. D. L., Ortiz, A. D. & Moreno, A. (2004). *Curr. Org. Chem.* **8**, 903–918.
Jagani, C. L., Vanparia, S. F., Patel, T. S., Dixit, R. B. & Dixit, B. C. (2012). *Arkivoc*, **vi**, 281–294.
Liu, Y.-H., Liu, X.-L., Dai, X.-Q., Xu, W. & Guo, R. (2008). *J. Chem. Crystallogr.* **38**, 109–113.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Wang, Y.-B., Shi, Y., Liu, X.-L. & Liu, Y.-H. (2010). *Acta Cryst.* E**66**, o955–o956.

supporting information

Acta Cryst. (2014). E**70**, o721 [doi:10.1107/S1600536814011805]

(3*Z*)-3-[(*Z*)-2-(2-Oxoindolin-3-ylidene)hydrazin-1-ylidene]indolin-2-one 0.17-hydrate

Yong-Hong Liu, Lei Zhao, Ming-Xuan Liu, Hai Lin and Jing-Jing Li

S1. Comment

Microwave irradiated and solvent-free synthesis have aroused great attention in recent years due to rapid, convenient, green, environment friendly, inexpensive and efficient (Hoz *et al.*, 2004; Jagani *et al.*, 2012). As a continuation of our research work on Schiff bases (Liu *et al.*, 2008; Wang *et al.*,2010), we report here one step synthesis of the title compound under microwave irradiated and free-solvent condition, which was prepared by two steps in the normal method (Ali & Alam, 1994), and its structure.

In the central symmetric molecule of the compound, the non-hydrogen atoms are conjugated by a couple of double bonds of C7=N2 and C7a=N2a, because whose bond length [1.2891 (17) Å] is shorter than the single bond one of C1—N1or C1a—N1a [1.4022 (17) Å] but longer than normal double one of C=N [1.271 (5) Å]. The molecule exists as the most stable configuration of (*E, E*)-isomer and conformation of *s-trans* (Fig. 1, Table 1).

In its pack structure there are two couples of N1–H1···O1 inter-molecular hydrogen bonds in the neighbor molecules which link many molecules into three dimensional net-work frames, and the disorder water molecules merge into the net-work (Fig. 2, Table 1). Thus the guest molecules of the water and the host molecules of the compound form into a super-molecular net-work structure.

S2. Experimental

In refluxing equipment, isatin (2.94 g, 20 mmol), 50% hydrazine hydrate (0.62 ml, 9.5 mmol) were heated under microwave irradiation for 10 min. After cooling, the red crystalline mixture was recrystallized from dimethylformamide to give 2.5 g (86.2%) of the title compound, m.p. 494.5–495.5 K (ref. 494.5~495.5 K, Ali *et al.*, 1994).

S3. Refinement

After their location in a difference map, all H atoms were fixed geometrically at ideal positions and allowed to ride on the parent C atoms, with C — H distances of 0.93 (aromaticl CH), O — H distances of 0.84 and N— H distances of 0.86, and with U_{iso}(H) values of 1.2U_{eq}(C).

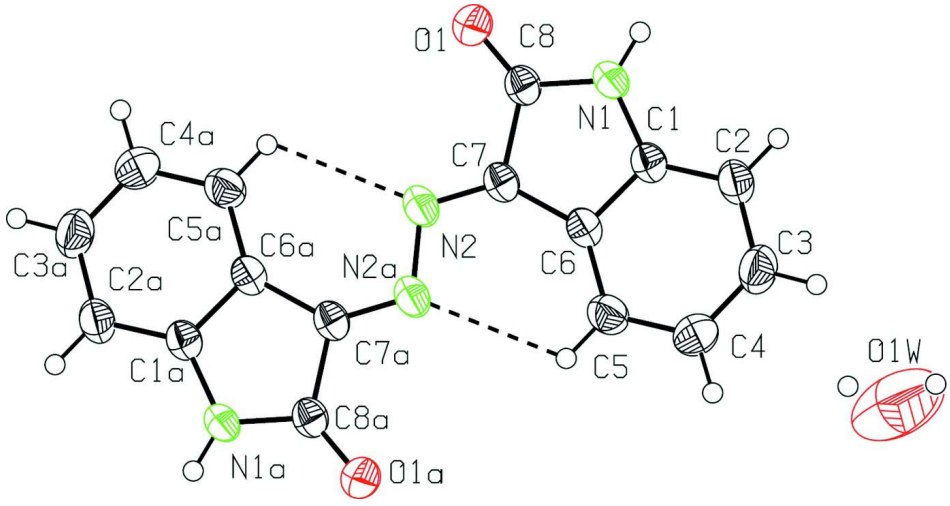


Figure 1

The molecular structure of the title compound, showing 30% probability ellipsoids.

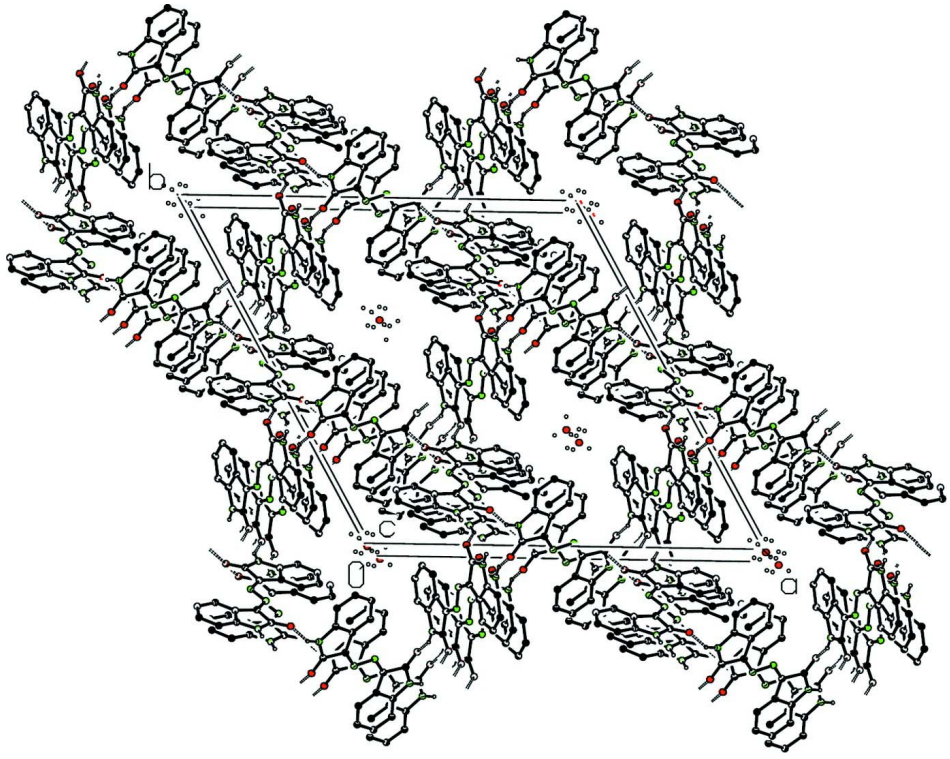


Figure 2

Part of the crystal structure of the title compound, showing two couples of N–H···O inter-molecular hydrogen bonds as dashed lines linking the molecules and disorder water molecules into a super-molecular net-work structure. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted.

(3*Z*)-3-[(*Z*)-2-(2-Oxoindolin-3-ylidene)hydrazin-1-ylidene]indolin-2-one 0.17-hydrate

Crystal data

$C_{16}H_{10}N_4O_2 \cdot 0.17H_2O$
$M_r = 308.30$
Trigonal, $R\overline{3}$
Hall symbol: -R 3
$a = 24.8699\ (18)$ Å
$c = 5.6603\ (8)$ Å
$V = 3031.9\ (5)$ Å^3
$Z = 9$
$F(000) = 1369$
$D_x = 1.450$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 2.8–27.2°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, brown
0.38 × 0.16 × 0.14 mm

Data collection

Bruker SMART1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
thin–slice ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.963$, $T_{max} = 0.986$
8691 measured reflections
1547 independent reflections
1290 reflections with $I > 2\sigma$igma(I)
$R_{int} = 0.025$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -32 \rightarrow 32$
$k = -29 \rightarrow 32$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.108$
$S = 1.01$
1547 reflections
103 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
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 2.5765P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Experimental. The title compound was synthesized under microwave irradiation.

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.25001 (6)	0.06795 (6)	0.2063 (2)	0.0345 (3)	
C2	0.20279 (7)	0.05119 (7)	0.3672 (3)	0.0429 (3)	
H2	0.1975	0.0250	0.4934	0.051*	
C3	0.16336 (8)	0.07495 (8)	0.3331 (3)	0.0515 (4)	
H3	0.1310	0.0644	0.4390	0.062*	

C4	0.17099 (8)	0.11403 (8)	0.1452 (3)	0.0521 (4)	
H4	0.1434	0.1286	0.1257	0.063*	
C5	0.21929 (7)	0.13147 (7)	−0.0134 (3)	0.0439 (3)	
H5	0.2247	0.1581	−0.1379	0.053*	
C6	0.25943 (6)	0.10844 (6)	0.0170 (2)	0.0341 (3)	
C7	0.31363 (6)	0.11575 (6)	−0.1095 (2)	0.0336 (3)	
C8	0.33439 (6)	0.07495 (6)	0.0163 (2)	0.0349 (3)	
N1	0.29469 (5)	0.04931 (5)	0.20131 (19)	0.0386 (3)	
H1	0.2967	0.0246	0.3026	0.046*	
N2	0.34522 (6)	0.14844 (5)	−0.2881 (2)	0.0406 (3)	
O1	0.37735 (5)	0.06693 (5)	−0.03535 (18)	0.0459 (3)	
O1W	0.0000	0.0000	0.248 (4)	0.177 (8)	0.25

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0402 (7)	0.0309 (6)	0.0306 (6)	0.0163 (5)	0.0026 (5)	0.0005 (5)
C2	0.0495 (8)	0.0409 (7)	0.0372 (7)	0.0217 (6)	0.0112 (6)	0.0069 (6)
C3	0.0532 (9)	0.0532 (9)	0.0516 (9)	0.0291 (8)	0.0181 (7)	0.0037 (7)
C4	0.0582 (9)	0.0549 (9)	0.0574 (10)	0.0388 (8)	0.0097 (7)	0.0033 (7)
C5	0.0547 (9)	0.0410 (7)	0.0422 (8)	0.0287 (7)	0.0055 (6)	0.0060 (6)
C6	0.0412 (7)	0.0297 (6)	0.0297 (6)	0.0165 (5)	0.0034 (5)	0.0017 (5)
C7	0.0385 (7)	0.0309 (6)	0.0281 (6)	0.0149 (5)	0.0011 (5)	0.0005 (5)
C8	0.0394 (7)	0.0345 (6)	0.0292 (6)	0.0173 (5)	0.0018 (5)	0.0003 (5)
N1	0.0461 (6)	0.0415 (6)	0.0327 (6)	0.0252 (5)	0.0067 (5)	0.0103 (5)
N2	0.0460 (7)	0.0416 (6)	0.0335 (6)	0.0214 (5)	0.0066 (5)	0.0095 (5)
O1	0.0479 (6)	0.0568 (6)	0.0415 (6)	0.0325 (5)	0.0074 (4)	0.0050 (5)
O1W	0.093 (6)	0.093 (6)	0.34 (3)	0.047 (3)	0.000	0.000

Geometric parameters (Å, º)

C1—C2	1.3756 (19)	C5—C6	1.388 (2)
C1—N1	1.4022 (17)	C5—H5	0.9300
C1—C6	1.4073 (17)	C6—C7	1.4554 (18)
C2—C3	1.389 (2)	C7—N2	1.2891 (17)
C2—H2	0.9300	C7—C8	1.5254 (18)
C3—C4	1.388 (2)	C8—O1	1.2165 (17)
C3—H3	0.9300	C8—N1	1.3594 (17)
C4—C5	1.384 (2)	N1—H1	0.8593
C4—H4	0.9300	N2—N2^{i}	1.404 (2)
C2—C1—N1	127.63 (12)	C6—C5—H5	120.6
C2—C1—C6	122.16 (13)	C5—C6—C1	119.53 (12)
N1—C1—C6	110.21 (11)	C5—C6—C7	134.43 (12)
C1—C2—C3	117.17 (13)	C1—C6—C7	106.04 (11)
C1—C2—H2	121.4	N2—C7—C6	134.34 (12)
C3—C2—H2	121.4	N2—C7—C8	118.92 (12)
C4—C3—C2	121.72 (14)	C6—C7—C8	106.71 (10)

C4—C3—H3	119.1	O1—C8—N1	126.85 (12)
C2—C3—H3	119.1	O1—C8—C7	127.86 (12)
C5—C4—C3	120.64 (14)	N1—C8—C7	105.29 (11)
C5—C4—H4	119.7	C8—N1—C1	111.72 (10)
C3—C4—H4	119.7	C8—N1—H1	124.1
C4—C5—C6	118.75 (13)	C1—N1—H1	124.2
C4—C5—H5	120.6	C7—N2—N2^{i}	111.89 (14)
N1—C1—C2—C3	−178.92 (14)	C5—C6—C7—C8	−177.99 (15)
C6—C1—C2—C3	1.3 (2)	C1—C6—C7—C8	1.50 (14)
C1—C2—C3—C4	0.0 (2)	N2—C7—C8—O1	−2.8 (2)
C2—C3—C4—C5	−1.1 (3)	C6—C7—C8—O1	178.67 (13)
C3—C4—C5—C6	0.9 (2)	N2—C7—C8—N1	176.85 (12)
C4—C5—C6—C1	0.3 (2)	C6—C7—C8—N1	−1.65 (14)
C4—C5—C6—C7	179.71 (15)	O1—C8—N1—C1	−179.13 (13)
C2—C1—C6—C5	−1.4 (2)	C7—C8—N1—C1	1.18 (14)
N1—C1—C6—C5	178.74 (12)	C2—C1—N1—C8	179.94 (13)
C2—C1—C6—C7	178.98 (12)	C6—C1—N1—C8	−0.26 (15)
N1—C1—C6—C7	−0.83 (14)	C6—C7—N2—N2^{i}	0.1 (2)
C5—C6—C7—N2	3.8 (3)	C8—C7—N2—N2^{i}	−177.92 (13)
C1—C6—C7—N2	−176.67 (15)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{ii}	0.86	2.13	2.8951 (17)	148

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