organic compounds

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10-Hydroxy-2-azapentacyclo[10.8.0.-0^{2,10}.0^{4,9}.0^{15,20}]icosa-1(12),4(9),5,7,13,-15(20),16,18-octaene-3,11-dione

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Key indicators: single-crystal X-ray study; T = 298 K; mean σ (C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.136; data-to-parameter ratio = 17.1.

In the title compound, $C_{19}H_{11}NO_3$, the isoindolinone ring system is approximately planar with a maximum atomic deviation of 0.071 (1) Å and the five-membered ring of the dihydrobenzo[g]indol-3-one unit assumes an envelope conformation. The naphthalene ring system makes a dihedral angle of 39.47 $(4)^{\circ}$ with the mean plane of the isoindolinone system. Intermolecular $O-H \cdots O$ and $C-H \cdots O$ hydrogen bonding helps to stabilize the crystal structure.

Related literature

For applications of naphthylamines, see Valenti et al. (2006); Black et al. (1994).



Experimental

Crystal data

C ₁₉ H ₁₁ NO ₃	$\gamma = 75.862 \ (12)^{\circ}$
$M_r = 301.29$	V = 683.94 (19) Å
Triclinic, $P\overline{1}$	Z = 2
a = 7.3250 (11) Å	Mo $K\alpha$ radiation
b = 9.7916 (16) Å	$\mu = 0.10 \text{ mm}^{-1}$
c = 10.4532 (17) Å	T = 298 K
$\alpha = 70.401 \ (13)^{\circ}$	$0.40 \times 0.30 \times 0.29$
$\beta = 82.503 \ (13)^{\circ}$	

Data collection

Stoe IPDS II diffractometer 7737 measured reflections 3627 independent reflections

Refinement

D-

02

C6

$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of
$wR(F^2) = 0.136$	independent and constrained
S = 1.13	refinement
3627 reflections	$\Delta \rho_{\rm max} = 0.29 \ {\rm e} \ {\rm \AA}^{-3}$
212 parameters	$\Delta \rho_{\rm min} = -0.22 \text{ e } \text{\AA}^{-3}$

mm

3161 reflections with $I > 2\sigma(I)$

 $R_{\rm int}=0.040$

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

-H···A	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdot \cdot \cdot A$
$-H2\cdots O1^{i}$	0.96 (2)	1.85 (2)	2.7978 (15)	172.2 (17)
$-H6\cdots O3^{ii}$	0.93	2.52	3.1609 (18)	126

Symmetry codes: (i) -x + 1, -y + 1, -z + 1; (ii) -x + 1, -y + 2, -z.

Data collection: X-RED32 (Stoe & Cie, 2005); cell refinement: X-AREA (Stoe & Cie, 2005); data reduction: X-AREA; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

We are grateful to the Islamic Azad University, Yazd Branch, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5159).

References

- Black, D. S. C., Bowyer, M. C., Condie, G. C., Craig, D. C. & Kumar, N. (1994). Tetrahedron, 50, 10983-10994.
- Farrugia, L. J. (1997). J. Appl. Cryst. 30, 565.

Farrugia, L. J. (1999). J. Appl. Cryst. 32, 837-838.

Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

- Stoe & Cie (2005). X-AREA and X-RED32. Stoe & Cie, Darmstadt, Germany.
- Valenti, L. E., De Pauli, C. P. & Giacomelli, C. E. (2006). J. Inorg. Biochem. 100, 192-200.

supplementary materials

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10-Hydroxy-2-azapentacyclo[10.8.0.0^{2,10}.0^{4,9}.0^{15,20}]icosa-1(12),4(9),5,7,13,15(20),16,18-octaene-3,11-dione

S. Hashemian and B. Notash

Comment

Naphthylamine is an aromatic amine which can be obtained from nitronaphthalene. Naphthylamines are used in the manufacture of dyes, condensation colors, and rubber and in the synthesis of a large number of chemical (Black *et al.* 1994; Valenti *et al.* 2006). Ninhydrin (2,2-dihydroxyindane-1,3-dione) is a chemical used to detect ammonia or primary and secondary amines. The carbon atom of a carbonyl bears a partial positive charge enhanced by neighboring electron withdrawing groups like carbonyl itself. So the central carbon of a 1,2,3-tricarbonyl compound is much more electrophilic than one in a simple ketone. Thus indane-1,2,3-trione reacts readily with nucleophiles, including water. Whereas for most carbonyl compounds, a carbonyl form is more stable than a product of water addition (hydrate), ninhydrin forms a stable hydrate of the central carbon because of the destabilizing effect of the adjacent carbonyl groups.

We report here the crystal structure of 7-hydroxy-7-aH-benzo[g] isoindolino[1,2- α]indole-7,10-dione. The title compound was prepared by the reaction of α -naphthylamine and ninhydrine in molar ratio of 1: 1 in acetone. The structure of title complex is shown in Fig. 1. There are intermolecular hydrogen bondings between O—H···O and C—H···O which play important role in the stabilization of crystalline network (Table 1 & Fig. 2).

Experimental

The title compound was prepared by the reaction of α - naphthylamine and ninhydrine in molar ratio of 1: 1 in acetone. The mixture was stirred for about 2 h at room temperature. The resulting solution was kept in air. After slow evaporation, single crystals of the title compound suitable for an X-ray diffraction study formed at the bottom of the vessel after 2 weeks (m.p. 138 °C).

Refinement

Hydroxyl H atom was found in a difference Fourier map and refined isotropically. Other H atoms were positioned geometrically and refined as riding atoms with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures



Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at 30% probability level.



Fig. 2. The packing diagram of the title compound. The intermolecular O—H…O and C—H…O hydrogen bonds are shown as blue dashed lines.

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Crvstal	data
0. 90000	

C ₁₉ H ₁₁ NO ₃	Z = 2
$M_r = 301.29$	F(000) = 312
Triclinic, $P\overline{1}$	$D_{\rm x} = 1.463 {\rm Mg m}^{-3}$
Hall symbol: -P 1	Mo <i>K</i> α radiation, $\lambda = 0.71073$ Å
a = 7.3250 (11) Å	Cell parameters from 1011 reflections
b = 9.7916 (16) Å	$\theta = 2.3 - 25.0^{\circ}$
c = 10.4532 (17) Å	$\mu = 0.10 \text{ mm}^{-1}$
$\alpha = 70.401 \ (13)^{\circ}$	T = 298 K
$\beta = 82.503 \ (13)^{\circ}$	Block, colorless
$\gamma = 75.862 \ (12)^{\circ}$	$0.40 \times 0.30 \times 0.29 \text{ mm}$
$V = 683.94 (19) \text{ Å}^3$	

Data collection

3161 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.040$
$\theta_{\text{max}} = 29.3^\circ, \ \theta_{\text{min}} = 2.3^\circ$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.136$	H atoms treated by a mixture of independent and constrained refinement
<i>S</i> = 1.13	$w = 1/[\sigma^{2}(F_{o}^{2}) + (0.072P)^{2} + 0.1037P]$ where $P = (F_{o}^{2} + 2F_{c}^{2})/3$
3627 reflections	$(\Delta/\sigma)_{\rm max} < 0.001$
212 parameters	$\Delta \rho_{max} = 0.29 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta \rho_{min} = -0.22 \text{ e } \text{\AA}^{-3}$

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.

 $U_{iso}*/U_{eq}$ \boldsymbol{Z} х y C1 0.0298(2)0.60706 (15) 0.62937 (12) 0.49240(11) C2 0.69780 (16) 0.61664 (13) 0.36032 (12) 0.0324(2)C3 0.0410 (3) 0.87442 (18) 0.53663 (16) 0.33404 (14) H3 0.049* 0.9488 0.4711 0.4042 C4 0.9358 (2) 0.55825 (19) 0.19890 (16) 0.0494(3)H4 1.0540 0.059* 0.5065 0.1779 C5 0.8237(2)0.65604 (19) 0.09393 (15) 0.0502(3)Н5 0.8694 0.0042 0.060* 0.6697 C6 0.6452(2)0.73333 (16) 0.12066 (13) 0.0425(3)H6 0.5690 0.7968 0.0507 0.051* C7 0.25611 (12) 0.58481 (16) 0.71222 (13) 0.0332 (2) C8 0.40104 (16) 0.31604 (11) 0.77784 (12) 0.0318(2)C9 0.33814 (17) 0.94765 (13) 0.28511 (13) 0.0374 (3) C10 0.29102 (17) 0.97079 (13) 0.41761 (13) 0.0358 (3) C11 0.19816 (19) 1.10289 (14) 0.44465 (16) 0.0441(3)H11 0.1640 1.1909 0.3742 0.053* C12 0.15988 (19) 1.09807 (16) 0.57670 (17) 0.0469 (3) H12 0.056* 0.0981 1.1843 0.5963 C13 0.21185 (17) 0.96457 (16) 0.68549 (15) 0.0413 (3) C14 0.1701 (2) 0.9614 (2) 0.82289 (17) 0.0541 (4) H14 0.1102 1.0485 0.8415 0.065* C15 0.2156 (2) 0.8347 (2) 0.92698 (17) 0.0596 (4) H15 0.1890 0.8359 1.0161 0.071* C16 0.7010(2) 0.3028(2)0.90168 (15) 0.0545(4)H16 0.3306 0.6140 0.9742 0.065* C17 0.34726 (19) 0.69747 (16) 0.0422 (3) 0.77121 (13) H17 0.4051 0.6082 0.7558 0.051* C18 0.30558 (16) 0.65996 (12) 0.0344(2)0.82910 (14) C19 0.34334 (15) 0.83897 (12) 0.52100 (12) 0.0310(2) 01 0.67384 (13) 0.57343 (10) 0.60386 (9) 0.0374 (2) O2 0.24447 (12) 0.73626 (10) 0.28658 (9) 0.0376(2) H2 0.268 (3) 0.630(2)0.317(2)0.066 (6)* O3 0.32112 (19) 1.03639 (12) 0.17284 (11) 0.0592 (3)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (A^2)

supplementary materials

N1	0.43075 (13)	0.72309	(10) 0.	.46469 (9)	0.0303 (2)	
Atomic dis	placement parameters	$(Å^2)$				
	U^{11}	U ²²	U ³³	U^{12}	U^{13}	U ²³
C1	0.0310 (5)	0.0265 (5)	0.0314 (5)	-0.0044 (4	-0.0037 (4)	-0.0091 (4)
C2	0.0318 (5)	0.0332 (5)	0.0334 (5)	-0.0057 (4	-0.0021 (4)	-0.0128 (4)
C3	0.0329 (6)	0.0468 (7)	0.0462 (7)	-0.0022 (5	-0.0040(5)	-0.0221 (6)
C4	0.0366 (6)	0.0628 (9)	0.0545 (8)	-0.0073 (6	b) 0.0071 (6)	-0.0317 (7)
C5	0.0490 (8)	0.0656 (9)	0.0402 (7)	-0.0176 (7	<i>(</i>) 0.0120 (6)	-0.0237 (7)
C6	0.0460 (7)	0.0487 (7)	0.0309 (6)	-0.0114 (5) 0.0016 (5)	-0.0106 (5)
C7	0.0339 (5)	0.0342 (5)	0.0308 (5)	-0.0075 (4	-0.0004 (4)	-0.0096 (4)
C8	0.0328 (5)	0.0321 (5)	0.0268 (5)	-0.0041 (4	-0.0038 (4)	-0.0057 (4)
C9	0.0367 (6)	0.0319 (5)	0.0370 (6)	-0.0045 (4	-0.0026 (4)	-0.0040 (4)
C10	0.0323 (5)	0.0302 (5)	0.0430 (6)	-0.0053 (4	-0.0014 (4)	-0.0104 (5)
C11	0.0384 (6)	0.0293 (6)	0.0628 (8)	-0.0041 (5	·) -0.0020 (6)	-0.0146 (5)
C12	0.0378 (6)	0.0398 (7)	0.0697 (9)	-0.0068 (5	6) 0.0044 (6)	-0.0297 (7)
C13	0.0310 (5)	0.0490 (7)	0.0540 (7)	-0.0105 (5	b) 0.0038 (5)	-0.0301 (6)
C14	0.0438 (7)	0.0725 (10)	0.0611 (9)	-0.0126 (7	() 0.0084 (6)	-0.0447 (9)
C15	0.0510 (8)	0.0929 (13)	0.0467 (8)	-0.0148 (8	6) 0.0065 (6)	-0.0413 (9)
C16	0.0540 (8)	0.0735 (10)	0.0354 (7)	-0.0126 (7	<i>(</i>) 0.0017 (6)	-0.0188 (7)
C17	0.0428 (7)	0.0495 (7)	0.0346 (6)	-0.0087 (5	6) 0.0009 (5)	-0.0158 (5)
C18	0.0290 (5)	0.0408 (6)	0.0379 (6)	-0.0088 (4) 0.0016 (4)	-0.0184 (5)
C19	0.0278 (5)	0.0304 (5)	0.0353 (5)	-0.0060 (4) 0.0001 (4)	-0.0119 (4)
01	0.0403 (5)	0.0356 (4)	0.0330 (4)	-0.0006 (3	-0.0097(3)	-0.0091 (3)
02	0.0338 (4)	0.0389 (5)	0.0380 (4)	-0.0049 (3	-0.0085 (3)	-0.0089 (4)
03	0.0753 (8)	0.0408 (6)	0.0421 (5)	-0.0017 (5	·) -0.0040 (5)	0.0047 (4)
N1	0.0325 (5)	0.0292 (4)	0.0265 (4)	-0.0019 (3	-0.0028(3)	-0.0080 (3)

Geometric parameters (Å, °)

C101	1.2192 (14)	C10—C19	1.3826 (16)
C1—N1	1.3908 (14)	C10—C11	1.4075 (17)
C1—C2	1.4829 (16)	C11—C12	1.359 (2)
C2—C3	1.3855 (16)	C11—H11	0.9300
С2—С7	1.3858 (16)	C12—C13	1.421 (2)
C3—C4	1.387 (2)	C12—H12	0.9300
С3—Н3	0.9300	C13—C14	1.420 (2)
C4—C5	1.393 (2)	C13—C18	1.4307 (17)
C4—H4	0.9300	C14—C15	1.349 (3)
С5—С6	1.387 (2)	C14—H14	0.9300
С5—Н5	0.9300	C15—C16	1.404 (3)
С6—С7	1.3885 (17)	C15—H15	0.9300
С6—Н6	0.9300	C16—C17	1.3704 (18)
С7—С8	1.4983 (16)	C16—H16	0.9300
C8—O2	1.4071 (14)	C17—C18	1.4148 (19)
C8—N1	1.4905 (14)	С17—Н17	0.9300
С8—С9	1.5445 (16)	C18—C19	1.4177 (16)
С9—ОЗ	1.2037 (16)	C19—N1	1.4278 (14)

C9—C10	1.4616 (18)	O2—H2	0.96 (2)
01—C1—N1	125.83 (10)	C11—C10—C9	127.75 (12)
O1—C1—C2	127.52 (10)	C12—C11—C10	118.08 (13)
N1—C1—C2	106.62 (9)	С12—С11—Н11	121.0
C3—C2—C7	121.55 (11)	C10-C11-H11	121.0
C3—C2—C1	128.81 (11)	C11—C12—C13	121.64 (12)
C7—C2—C1	109.36 (10)	С11—С12—Н12	119.2
C2—C3—C4	117.33 (12)	С13—С12—Н12	119.2
С2—С3—Н3	121.3	C14—C13—C12	120.91 (13)
С4—С3—Н3	121.3	C14—C13—C18	118.03 (14)
C3—C4—C5	121.23 (13)	C12—C13—C18	121.06 (12)
C3—C4—H4	119.4	C15—C14—C13	121.45 (14)
C5—C4—H4	119.4	C15-C14-H14	119.3
C6—C5—C4	121 25 (12)	C13—C14—H14	119.3
С6—С5—Н5	119.4	C14-C15-C16	120 45 (14)
C4—C5—H5	119.4	C14-C15-H15	119.8
$C_{5} - C_{6} - C_{7}$	117 34 (13)	C16-C15-H15	119.8
C5—C6—H6	121.3	C_{17} $-C_{16}$ $-C_{15}$	120.69 (16)
C7_C6_H6	121.5	C17_C16_H16	119.7
C^{2} C^{7} C^{6}	121.5 121.28(11)	C15-C16-H16	119.7
$C_{2}^{} C_{1}^{} C_{0}^{} C_{0}^{$	121.20(11) 100 15 (10)	$C_{15} = C_{10} = 110$	119.7
$C_2 - C_7 - C_8$	109.13(10) 120.57(11)	$C_{10} = C_{17} = C_{18}$	120.21 (14)
$C_{0} = C_{0} = C_{0}$	129.37 (11)	$C_{10} - C_{17} - H_{17}$	119.9
02 - 03 - 01	111.02 (9)	$C_{10} - C_{17} - C_{10}$	119.9
02 - 03 - 07	113.85 (9)	C17 - C18 - C19	123.32 (11)
NI = C8 = C7	103.29 (9)	C1/-C18-C13	119.13 (12)
02-08-09	104.10 (9)	C19 - C18 - C13	115.52 (12)
NI-C8-C9	103.13 (9)	C10-C19-C18	121.92 (11)
C7—C8—C9	120.25 (10)	C10—C19—N1	109.84 (10)
O3—C9—C10	129.58 (12)	C18—C19—N1	128.21 (10)
03—C9—C8	124.77 (12)	С8—О2—Н2	107.6 (12)
C10—C9—C8	105.45 (9)	C1—N1—C19	126.89 (9)
C19—C10—C11	121.77 (12)	C1—N1—C8	111.06 (9)
C19—C10—C9	110.43 (10)	C19—N1—C8	108.74 (9)
O1—C1—C2—C3	1.8 (2)	C11—C12—C13—C18	-0.5 (2)
N1—C1—C2—C3	179.64 (12)	C12—C13—C14—C15	178.73 (14)
O1—C1—C2—C7	-172.16 (12)	C18—C13—C14—C15	-0.5 (2)
N1—C1—C2—C7	5.70 (13)	C13-C14-C15-C16	-1.3 (2)
C7—C2—C3—C4	1.3 (2)	C14—C15—C16—C17	1.6 (3)
C1—C2—C3—C4	-171.95 (12)	C15-C16-C17-C18	0.0 (2)
C2—C3—C4—C5	-0.3 (2)	C16-C17-C18-C19	-179.45 (13)
C3—C4—C5—C6	-1.2 (2)	C16-C17-C18-C13	-1.7 (2)
C4—C5—C6—C7	1.6 (2)	C14—C13—C18—C17	2.00 (18)
C3—C2—C7—C6	-0.90 (19)	C12-C13-C18-C17	-177.24 (12)
C1—C2—C7—C6	173.56 (11)	C14—C13—C18—C19	179.92 (11)
C3—C2—C7—C8	177.95 (11)	C12-C13-C18-C19	0.68 (17)
C1—C2—C7—C8	-7.60 (13)	C11—C10—C19—C18	0.37 (18)
C5—C6—C7—C2	-0.6 (2)	C9—C10—C19—C18	-177.05 (10)
C5—C6—C7—C8	-179.17 (13)	C11-C10-C19-N1	178.75 (11)

supplementary materials

C2—C7—C8—O2	-115.06 (11)	C9-C10-C19-N1	1.33 (14)
C6—C7—C8—O2	63.66 (17)	C17—C18—C19—C10	177.17 (12)
C2—C7—C8—N1	6.39 (12)	C13—C18—C19—C10	-0.61 (17)
C6—C7—C8—N1	-174.89 (13)	C17-C18-C19-N1	-0.9 (2)
C2—C7—C8—C9	120.48 (11)	C13-C18-C19-N1	-178.66 (10)
C6—C7—C8—C9	-60.81 (18)	O1—C1—N1—C19	40.41 (18)
O2—C8—C9—O3	-72.49 (16)	C2-C1-N1-C19	-137.50 (11)
N1—C8—C9—O3	170.63 (13)	O1—C1—N1—C8	176.42 (11)
С7—С8—С9—О3	56.47 (18)	C2-C1-N1-C8	-1.48 (12)
O2—C8—C9—C10	102.75 (10)	C10-C19-N1-C1	125.80 (12)
N1-C8-C9-C10	-14.13 (12)	C18—C19—N1—C1	-55.95 (17)
C7—C8—C9—C10	-128.29 (11)	C10-C19-N1-C8	-11.01 (13)
O3—C9—C10—C19	-176.69 (14)	C18—C19—N1—C8	167.23 (11)
C8—C9—C10—C19	8.38 (13)	O2—C8—N1—C1	119.96 (10)
O3—C9—C10—C11	6.1 (2)	C7—C8—N1—C1	-2.84 (12)
C8—C9—C10—C11	-168.84 (12)	C9—C8—N1—C1	-128.76 (10)
C19—C10—C11—C12	-0.18 (19)	O2-C8-N1-C19	-95.95 (11)
C9—C10—C11—C12	176.77 (12)	C7—C8—N1—C19	141.25 (9)
C10-C11-C12-C13	0.3 (2)	C9—C8—N1—C19	15.33 (11)
C11—C12—C13—C14	-179.76 (13)		

Hydrogen-bond geometry (Å, °)

D—H···A	<i>D</i> —Н	$H \cdots A$	$D \cdots A$	D—H··· A	
O2—H2···O1 ⁱ	0.96 (2)	1.85 (2)	2.7978 (15)	172.2 (17)	
C6—H6···O3 ⁱⁱ	0.93	2.52	3.1609 (18)	126	
Symmetry codes: (i) $-x+1$, $-y+1$, $-z+1$; (ii) $-x+1$, $-y+2$, $-z$.					



Fig. 1



