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1,1′-(Hexane-1,6-diyl)bis(indoline-2,3-dione)

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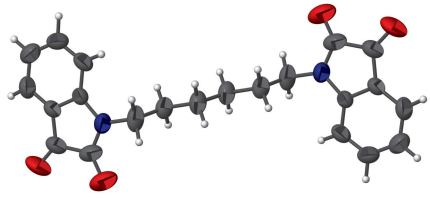
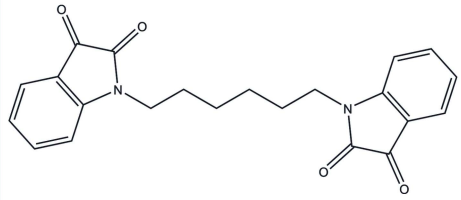
Structural data: full structural data are available from iucrdata.iucr.org

The molecule of the title compound, $C_{22}H_{20}N_2O_4$, is situated on a crystallographic centre of symmetry. The two indoline-2,3-dione fragments are linked by a hexylene group at each N atom. In the crystal, supramolecular layers propagating in the *ac* plane are formed *via* C—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

Isatin (1*H*-indole-2,3-dione) its derivatives have aroused great attention in recent years due to their wide variety of biological activities, relevant to application as insecticides and fungicides and in a broad range of drug therapies, including anticancer drugs, antibiotics and antidepressants (Bhrigu *et al.*, 2010; Malhotra *et al.*, 2011; Da Silva *et al.*, 2001; Qachchachi *et al.*, 2014; Ramachandran, 2011; Smitha *et al.*, 2008).

As a continuation of our research work devoted to the development of substituted isatin derivatives, we report in this paper the synthesis of a new isatin derivative obtained by the reaction of 1,6-dibromohexane with indoline-2,3-dione in the presence of a catalytic quantity of tetra-*n*-butylammonium bromide under mild conditions to furnish the title compound.

The crystallographically centrosymmetric molecule of the title compound contains two indoline-2,3-dione fragments being linked by the hexylene group at the N atoms (Fig. 1). The sum of valence angles around N1 is 359.9, indicating an sp^2 hybridization of this atom.

In the crystal, the molecules are linked by C—H···O hydrogen bonds (Table 1), forming supramolecular layers propagating in the *ac* plane (Figs. 2 and 3).

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1^{i}	0.88 (2)	2.52 (2)	3.316 (3)	150.6 (19)
C4—H4···O2^{ii}	0.91 (3)	2.51 (3)	3.349 (3)	154 (2)

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

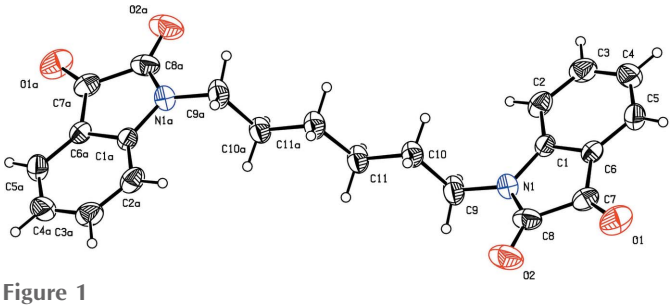


Figure 1
The title molecule with the atomic numbering scheme. Displacement ellipsoids are shown at the 30% probability level. [Symmetry code: (*a*) $-x+1, -y+1, -z+1$.]

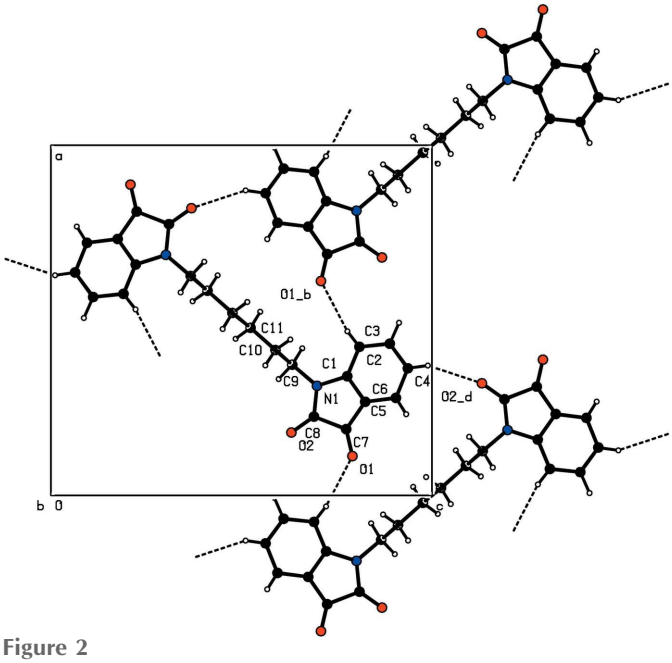


Figure 2
View down the *b* axis of the packing structure of the title compound. The dashed lines indicate intermolecular C—H···O interactions. [Symmetry codes: (ii) $x+\frac{1}{2}, y, -z+\frac{3}{2}$; (iii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$.]

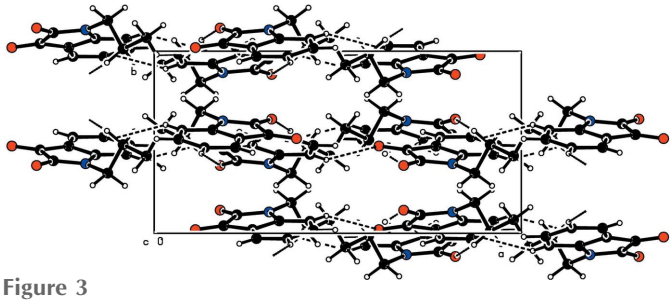


Figure 3
View of the title compound structure along the *c* axis, showing the layers parallel to the *ac* plane.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{20}N_2O_4$
M_r	376.40
Crystal system, space group	Orthorhombic, *Pbca*
Temperature (K)	296
a, *b*, *c* (Å)	15.1498 (3), 7.4949 (2), 16.4795 (3)
V (Å^3)	1871.19 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.48 × 0.37 × 0.15
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.925, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	52362, 2303, 1596
R_{int}	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.666
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.053, 0.171, 1.03
No. of reflections	2303
No. of parameters	167
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.23

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Synthesis and crystallization

To a solution of isatin (0.2 g, 1.2 mmol), potassium carbonate (0.41 g, 3 mmol) and tetra-*n*-butylammonium bromide (0.05 g, 0.15 mmol) in DMF (10 ml) was added dropwise 1,6-dibromohexane (0.3 ml, 3 mmol) at room temperature. The mixture was stirred for 48 h. The solid obtained was removed by filtration and the filtrate concentrated under reduced pressure, leading to a residue which was separated by chromatography on a column of silica gel with ethyl acetate/hexane (1/4) as eluent. The solid that was obtained was recrystallized from ethanol solution to afford the title compound as orange crystals in 68% yield.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Two reflections, *i.e.* 0 0 2 and 2 0 0, were omitted from the final refinement owing to poor agreement.

References

Bhrigu, B., Pathak, D., Siddiqui, N., Alam, M. S. & Ahsan, W. (2010). *Int. J. Pharm. Sci. Drug Res.* **2**, 229–235.
Bruker (2009). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Malhotra, S., Balwani, S., Dhawan, A., Singh, B. K., Kumar, S., Thimmulappa, R., Biswal, S., Olsen, C. E., Van der Eycken, E., Prasad, A. K., Ghosh, B. & Parmar, V. S. (2011). *Med. Chem. Commun.* **2**, 743–751.

Qachchachi, F.-Z., Kandri Rodi, Y., Essassi, E. M., Bodensteiner, M. & El Ammari, L. (2014). *Acta Cryst.* E**70**, o588.
Ramachandran, S. (2011). *Int. J. Res. Pharm. Chem.* **1**, 289–294.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Silva, J. F. M. da, Garden, S. J. & Pinto, A. C. (2001). *J. Braz. Chem. Soc.* **12**, 273–324.
Smitha, S., Pandeya, S. N., Stables, J. P. & Ganapathy, S. (2008). *Sci. Pharm.* **76**, 621–636.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

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1,1′-(Hexane-1,6-diyl)bis(indoline-2,3-dione)

Crystal data

$C_{22}H_{20}N_2O_4$
M_r = 376.40
Orthorhombic, *Pbca*
a = 15.1498 (3) Å
b = 7.4949 (2) Å
c = 16.4795 (3) Å
V = 1871.19 (7) Å^3
Z = 4
F(000) = 792
D_x = 1.336 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9889 reflections
θ = 2.5–23.9°
μ = 0.09 mm^{-1}
T = 296 K
Prism, orange
0.48 × 0.37 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.925, T_{max} = 1.000
52362 measured reflections
2303 independent reflections
1596 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 28.3°, θ_{min} = 2.8°
h = −19→20
k = −9→9
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.171
S = 1.03
2303 reflections
167 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0802P)^2 + 0.5456P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.34040 (11)	0.4314 (2)	0.77758 (10)	0.0504 (4)
C2	0.42431 (12)	0.4338 (3)	0.80961 (14)	0.0664 (6)
C3	0.43333 (15)	0.4836 (3)	0.88930 (14)	0.0755 (6)
C4	0.36311 (18)	0.5300 (3)	0.93673 (14)	0.0799 (7)
C5	0.27861 (16)	0.5286 (3)	0.90539 (13)	0.0704 (6)
C6	0.26756 (10)	0.4786 (3)	0.82489 (11)	0.0536 (4)
C7	0.18931 (12)	0.4580 (3)	0.77498 (14)	0.0647 (5)
C8	0.22517 (14)	0.3985 (3)	0.69028 (13)	0.0686 (6)
C9	0.3733 (2)	0.3309 (3)	0.63322 (14)	0.0758 (6)
C10	0.41541 (15)	0.4874 (3)	0.58917 (12)	0.0624 (5)
C11	0.47878 (17)	0.4251 (3)	0.52375 (13)	0.0665 (5)
H2	0.4687 (16)	0.404 (3)	0.7780 (15)	0.082 (7)*
H3	0.4932 (17)	0.481 (3)	0.9136 (14)	0.082 (7)*
H4	0.3711 (18)	0.562 (3)	0.9892 (18)	0.102 (9)*
H5	0.2320 (18)	0.557 (4)	0.9323 (17)	0.092 (8)*
H9A	0.3325 (14)	0.264 (3)	0.5976 (15)	0.082 (7)*
H10A	0.3714 (15)	0.563 (3)	0.5626 (14)	0.075 (6)*
H11A	0.4474 (16)	0.346 (3)	0.4847 (16)	0.093 (8)*
H9B	0.4203 (15)	0.261 (3)	0.6596 (15)	0.084 (7)*
H10B	0.4454 (15)	0.560 (3)	0.6282 (15)	0.079 (7)*
H11B	0.5246 (15)	0.357 (3)	0.5493 (13)	0.074 (6)*
N1	0.31307 (11)	0.3827 (2)	0.69831 (9)	0.0627 (5)
O1	0.11235 (9)	0.4797 (3)	0.79127 (13)	0.1002 (6)
O2	0.17970 (13)	0.3727 (3)	0.63114 (11)	0.1027 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0448 (8)	0.0572 (9)	0.0492 (8)	−0.0027 (7)	0.0037 (7)	0.0050 (7)
C2	0.0424 (9)	0.0803 (13)	0.0766 (13)	−0.0013 (9)	0.0044 (9)	0.0121 (11)
C3	0.0634 (12)	0.0906 (16)	0.0725 (13)	−0.0168 (11)	−0.0204 (11)	0.0176 (11)
C4	0.0950 (17)	0.0897 (16)	0.0552 (12)	−0.0218 (13)	−0.0126 (11)	0.0024 (11)
C5	0.0754 (13)	0.0762 (13)	0.0597 (11)	−0.0055 (11)	0.0187 (10)	−0.0037 (10)
C6	0.0416 (9)	0.0593 (10)	0.0600 (10)	−0.0032 (7)	0.0049 (7)	0.0015 (8)
C7	0.0445 (9)	0.0663 (12)	0.0833 (13)	−0.0033 (8)	−0.0028 (9)	0.0049 (10)
C8	0.0733 (13)	0.0610 (11)	0.0715 (12)	−0.0109 (9)	−0.0263 (10)	0.0044 (9)
C9	0.1044 (17)	0.0660 (13)	0.0571 (11)	0.0016 (13)	0.0252 (11)	−0.0030 (10)
C10	0.0753 (12)	0.0591 (11)	0.0529 (10)	0.0048 (10)	0.0100 (9)	−0.0021 (9)
C11	0.0846 (14)	0.0597 (11)	0.0551 (10)	0.0080 (10)	0.0166 (10)	−0.0002 (9)
N1	0.0684 (10)	0.0705 (10)	0.0491 (8)	−0.0028 (8)	0.0072 (7)	−0.0026 (7)
O1	0.0392 (7)	0.1247 (14)	0.1366 (17)	0.0028 (8)	0.0011 (8)	0.0002 (12)
O2	0.1175 (14)	0.1024 (13)	0.0881 (12)	−0.0207 (11)	−0.0450 (11)	−0.0030 (10)

Geometric parameters (Å, º)

C1—C2	1.377 (3)	C7—C8	1.563 (3)
C1—C6	1.397 (2)	C8—O2	1.209 (2)
C1—N1	1.418 (2)	C8—N1	1.343 (3)
C2—C3	1.372 (3)	C9—N1	1.461 (3)
C2—H2	0.88 (2)	C9—C10	1.520 (3)
C3—C4	1.365 (4)	C9—H9A	0.99 (2)
C3—H3	0.99 (3)	C9—H9B	0.98 (2)
C4—C5	1.380 (3)	C10—C11	1.517 (3)
C4—H4	0.91 (3)	C10—H10A	0.98 (2)
C5—C6	1.389 (3)	C10—H10B	0.96 (3)
C5—H5	0.86 (3)	C11—C11^{i}	1.512 (4)
C6—C7	1.451 (3)	C11—H11A	1.00 (3)
C7—O1	1.207 (2)	C11—H11B	0.96 (2)
C2—C1—C6	120.82 (18)	N1—C8—C7	106.35 (15)
C2—C1—N1	128.77 (17)	N1—C9—C10	114.06 (18)
C6—C1—N1	110.40 (15)	N1—C9—H9A	100.3 (14)
C3—C2—C1	117.55 (19)	C10—C9—H9A	111.8 (14)
C3—C2—H2	124.1 (16)	N1—C9—H9B	105.6 (14)
C1—C2—H2	118.4 (16)	C10—C9—H9B	108.4 (14)
C4—C3—C2	122.7 (2)	H9A—C9—H9B	116 (2)
C4—C3—H3	119.1 (14)	C11—C10—C9	111.55 (17)
C2—C3—H3	118.2 (14)	C11—C10—H10A	107.0 (13)
C3—C4—C5	120.4 (2)	C9—C10—H10A	112.1 (13)
C3—C4—H4	120.7 (18)	C11—C10—H10B	110.7 (14)
C5—C4—H4	118.9 (18)	C9—C10—H10B	108.6 (14)
C4—C5—C6	118.1 (2)	H10A—C10—H10B	106.9 (19)
C4—C5—H5	124.6 (18)	C11^{i}—C11—C10	114.1 (2)
C6—C5—H5	117.3 (18)	C11^{i}—C11—H11A	108.0 (15)
C5—C6—C1	120.41 (17)	C10—C11—H11A	109.9 (14)
C5—C6—C7	131.97 (18)	C11^{i}—C11—H11B	108.3 (13)
C1—C6—C7	107.59 (17)	C10—C11—H11B	108.1 (13)
O1—C7—C6	130.4 (2)	H11A—C11—H11B	108.3 (19)
O1—C7—C8	124.9 (2)	C8—N1—C1	110.96 (16)
C6—C7—C8	104.63 (15)	C8—N1—C9	124.76 (19)
O2—C8—N1	129.1 (2)	C1—N1—C9	124.22 (18)
O2—C8—C7	124.6 (2)		
C6—C1—C2—C3	0.2 (3)	C6—C7—C8—O2	−177.7 (2)
N1—C1—C2—C3	−178.8 (2)	O1—C7—C8—N1	−177.4 (2)
C1—C2—C3—C4	−0.1 (3)	C6—C7—C8—N1	2.6 (2)
C2—C3—C4—C5	−0.1 (4)	N1—C9—C10—C11	178.6 (2)
C3—C4—C5—C6	0.2 (4)	C9—C10—C11—C11^{i}	178.6 (3)
C4—C5—C6—C1	0.0 (3)	O2—C8—N1—C1	178.1 (2)
C4—C5—C6—C7	177.8 (2)	C7—C8—N1—C1	−2.2 (2)
C2—C1—C6—C5	−0.1 (3)	O2—C8—N1—C9	0.9 (4)

N1—C1—C6—C5	179.06 (18)	C7—C8—N1—C9	−179.46 (18)
C2—C1—C6—C7	−178.48 (18)	C2—C1—N1—C8	−179.8 (2)
N1—C1—C6—C7	0.7 (2)	C6—C1—N1—C8	1.1 (2)
C5—C6—C7—O1	0.0 (4)	C2—C1—N1—C9	−2.6 (3)
C1—C6—C7—O1	178.1 (2)	C6—C1—N1—C9	178.33 (17)
C5—C6—C7—C8	180.0 (2)	C10—C9—N1—C8	95.2 (3)
C1—C6—C7—C8	−1.9 (2)	C10—C9—N1—C1	−81.6 (3)
O1—C7—C8—O2	2.3 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1^{ii}	0.88 (2)	2.52 (2)	3.316 (3)	150.6 (19)
C4—H4···O2^{iii}	0.91 (3)	2.51 (3)	3.349 (3)	154 (2)
C9—H9*A*···O2	0.99 (2)	2.52 (2)	2.950 (4)	106.3 (16)

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