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# 1-(Morpholinomethyl)indoline-2,3-dione

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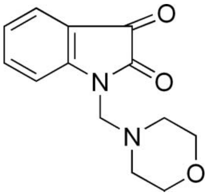
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.042; $wR$ factor = 0.144; data-to-parameter ratio = 20.8.

In the title compound, $C_{13}H_{14}N_2O_3$, the morpholine ring displays a chair conformation, with the (2,3-dioxoindolin-1-yl)methyl group in an equatorial position. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds.

## Related literature

For the synthesis of isatin-*N*-Mannich bases, see: Varma & Nobles ((1966). For the bioactivity of isatin derivatives, see: Glover *et al.* (1980, 1988); Maysinger *et al.* (1980).



## Experimental

### *Crystal data*

$C_{13}H_{14}N_2O_3$
$M_r$ = 246.26
Monoclinic, $P2_1/c$
$a$ = 11.608 (2) Å
$b$ = 8.2818 (17) Å
$c$ = 12.595 (3) Å
$\beta$ = 100.20 (3)°
$V$ = 1191.7 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 293 K
0.26 × 0.18 × 0.16 mm

### *Data collection*

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.979, $T_{max}$ = 0.984
7614 measured reflections
3556 independent reflections
2275 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.021

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.144
$S$ = 0.99
3556 reflections
171 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C6—H6$A$···O1$^{i}$ | 0.93 | 2.47 | 3.349 (2) | 158 |
| C5—H5$A$···O2$^{ii}$ | 0.93 | 2.52 | 3.216 (2) | 131 |

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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: RZ2460).

## References

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# supplementary materials

*Acta Cryst.* (2010). E**66**, o1748 [ **doi:10.1107/S1600536810023160** ]

# 1-(Morpholinomethyl)indoline-2,3-dione

**Y. Tang, J. Zhang, Y. Miao and G. Chen**

## Comment

Isatin has draw great attention from being discovered as a component of endogenous monoamine oxidase (MAO) inhibitory activity (tribulin) and subsequently identified as a selective inhibitor of MAO B at low concentrations (Glover *et al.*, 1980, 1988). In the following years, many isatin derivatives, such as isatin hydrazono, isatin Mannich bases and isatin based spiroazetidinones, have also been reported to possess anticonvulsant activity (Maysinger, *et al.*, 1980). Herein we report the synthesis and crystal structure of the title compound.

The X-ray structural analysis confirmed the assignment of the structure from spectroscopic data. The molecular structure of the title compound is depicted in Fig. 1. Geometric parameters of the title compound are in the usual ranges. The morpholin ring displays a typical chair conformation, with the (2,3-dioxoindolin-1-yl)methyl group in equatorial position. In the crystal structure, the molecules are linked into a three-dimensional network by C—H···O hydrogen bonds (Table 1).

## Experimental

The title compound was synthesized according the literature method (Varma & Nobles, 1966). Isatin (1 mmol), formaldehyde (1.2 mmol) and morpholin (1.2 mmol) were dissolved in methanol (20 ml). The mixture was refluxed until the disappearance of isatin, as evidenced by thin-layer chromatography. The solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 1:1 *v*/*v*), giving the title compound. $^1$H-NMR ($D_6$—DMSO, 400 MHz): 7.61 (2*H*, m), 7.15 (1*H*, t, J = 7.2 Hz), 7.09 (1*H*, d, J = 8.0 Hz), 4.45 (2*H*, s), 3.70 (4*H*, t, J = 4.8 Hz), 2.63 (4*H*, t, J = 4.8 Hz); MS (EI) m/*z*: 246 ($M^+$). 20 mg of the title compound was dissolved in 50 ml methanol and the solution was kept at room temperature for 4 d. Slow evaporation of the solvent gave orange single crystals of the title compound suitable for X-ray analysis.

## Refinement

The H atoms attached to atom C9 were located in a difference Fourier map and refined freely. All other H atoms were placed at calculated positions and refined as riding, with C—H = 0.93-0.98 Å, and with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C})$.

## Figures

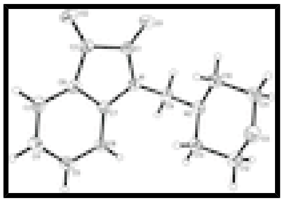

Fig. 1. An *ORTEP-3* drawing of the title compound, with the atom-numbering scheme and 30% probability displacement ellipsoids.

## 1-(Morpholinomethyl)indoline-2,3-dione

### *Crystal data*

$C_{13}H_{14}N_2O_3$

$M_r = 246.26$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.608$ (2) Å

$b = 8.2818$ (17) Å

$c = 12.595$ (3) Å

$\beta = 100.20$ (3)°

$V = 1191.7$ (4) Å$^3$

$Z = 4$

$F(000) = 520$

$D_x = 1.373$ Mg m$^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7356 reflections

$\theta = 1.5$–25.0°

$\mu = 0.10$ mm$^{-1}$

$T = 293$ K

Block, orange

0.26 × 0.18 × 0.16 mm

### *Data collection*

Bruker SMART CCD diffractometer

Radiation source: fine-focus sealed tube

graphite

phi and $\omega$ scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2001)

$T_{min} = 0.979$, $T_{max} = 0.984$

7614 measured reflections

3556 independent reflections

2275 reflections with $I > 2\sigma(I)$

$R_{int} = 0.021$

$\theta_{max} = 30.4°$, $\theta_{min} = 1.8°$

$h = -15 \rightarrow 15$

$k = -11 \rightarrow 10$

$l = -17 \rightarrow 15$

### *Refinement*

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.144$

$S = 0.99$

3556 reflections

171 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 atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.039$

$\Delta\rho_{max} = 0.19$ e Å$^{-3}$

$\Delta\rho_{min} = -0.22$ e Å$^{-3}$

### *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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{\mathrm{iso}}$*/$U_{\mathrm{eq}}$ |
|---|---|---|---|---|
| N2 | 0.91766 (9) | 0.23385 (12) | 0.11391 (8) | 0.0368 (3) |
| N1 | 0.73127 (8) | 0.33226 (11) | 0.00535 (8) | 0.0347 (3) |
| C7 | 0.68098 (9) | 0.48053 (14) | 0.03062 (9) | 0.0313 (3) |
| O2 | 0.59676 (8) | 0.46330 (13) | −0.24949 (7) | 0.0527 (3) |
| C10 | 0.98088 (11) | 0.23531 (16) | 0.02322 (11) | 0.0414 (3) |
| H10A | 0.9591 | 0.3302 | −0.0209 | 0.050* |
| H10B | 0.9604 | 0.1404 | −0.0212 | 0.050* |
| O3 | 1.14402 (7) | 0.37200 (13) | 0.13337 (9) | 0.0564 (3) |
| O1 | 0.73752 (10) | 0.19019 (12) | −0.15122 (8) | 0.0567 (3) |
| C6 | 0.68346 (10) | 0.54811 (15) | 0.13085 (10) | 0.0379 (3) |
| H6A | 0.7192 | 0.4957 | 0.1934 | 0.046* |
| C8 | 0.62332 (10) | 0.55651 (14) | −0.06320 (9) | 0.0345 (3) |
| C2 | 0.63447 (10) | 0.45189 (15) | −0.15409 (10) | 0.0377 (3) |
| C9 | 0.79252 (11) | 0.21364 (15) | 0.08256 (11) | 0.0373 (3) |
| C5 | 0.63034 (11) | 0.69807 (15) | 0.13481 (11) | 0.0456 (3) |
| H5A | 0.6323 | 0.7477 | 0.2014 | 0.055* |
| C1 | 0.70789 (11) | 0.30626 (15) | −0.10310 (10) | 0.0385 (3) |
| C3 | 0.56961 (11) | 0.70484 (16) | −0.05764 (11) | 0.0444 (3) |
| H3A | 0.5311 | 0.7557 | −0.1197 | 0.053* |
| C13 | 0.95237 (11) | 0.37262 (17) | 0.18371 (11) | 0.0435 (3) |
| H13A | 0.9126 | 0.3692 | 0.2452 | 0.052* |
| H13B | 0.9303 | 0.4718 | 0.1443 | 0.052* |
| C4 | 0.57469 (12) | 0.77531 (16) | 0.04263 (12) | 0.0495 (4) |
| H4A | 0.5403 | 0.8757 | 0.0481 | 0.059* |
| C12 | 1.08328 (12) | 0.3691 (2) | 0.22195 (12) | 0.0540 (4) |
| H12A | 1.1065 | 0.4616 | 0.2680 | 0.065* |
| H12B | 1.1043 | 0.2721 | 0.2642 | 0.065* |
| C11 | 1.11103 (11) | 0.23690 (17) | 0.06581 (12) | 0.0514 (4) |
| H11A | 1.1329 | 0.1385 | 0.1062 | 0.062* |
| H11B | 1.1529 | 0.2397 | 0.0057 | 0.062* |
| H9A | 0.7745 (10) | 0.1067 (17) | 0.0422 (11) | 0.038 (3)* |
| H9B | 0.7566 (11) | 0.2191 (14) | 0.1471 (11) | 0.036 (3)* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N2 | 0.0360 (5) | 0.0363 (5) | 0.0385 (6) | 0.0006 (4) | 0.0077 (4) | 0.0042 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N1 | 0.0366 (5) | 0.0373 (5) | 0.0306 (5) | 0.0046 (4) | 0.0074 (4) | −0.0023 (4) |
| C7 | 0.0285 (5) | 0.0345 (6) | 0.0319 (6) | −0.0018 (4) | 0.0082 (4) | −0.0003 (4) |
| O2 | 0.0613 (6) | 0.0656 (7) | 0.0290 (5) | −0.0100 (5) | 0.0018 (4) | 0.0040 (4) |
| C10 | 0.0420 (7) | 0.0404 (7) | 0.0440 (7) | 0.0003 (5) | 0.0135 (6) | −0.0044 (5) |
| O3 | 0.0414 (5) | 0.0612 (6) | 0.0685 (7) | −0.0114 (4) | 0.0150 (5) | −0.0142 (5) |
| O1 | 0.0671 (7) | 0.0587 (6) | 0.0452 (6) | 0.0075 (5) | 0.0125 (5) | −0.0178 (5) |
| C6 | 0.0385 (6) | 0.0456 (7) | 0.0302 (6) | 0.0014 (5) | 0.0073 (5) | −0.0019 (5) |
| C8 | 0.0316 (6) | 0.0402 (6) | 0.0322 (6) | −0.0026 (5) | 0.0072 (5) | 0.0032 (5) |
| C2 | 0.0352 (6) | 0.0475 (7) | 0.0304 (6) | −0.0072 (5) | 0.0058 (5) | 0.0025 (5) |
| C9 | 0.0361 (6) | 0.0362 (6) | 0.0403 (7) | −0.0010 (5) | 0.0086 (5) | 0.0055 (5) |
| C5 | 0.0474 (8) | 0.0460 (7) | 0.0458 (8) | 0.0010 (6) | 0.0146 (6) | −0.0114 (6) |
| C1 | 0.0384 (6) | 0.0449 (7) | 0.0331 (6) | −0.0028 (5) | 0.0089 (5) | −0.0053 (5) |
| C3 | 0.0423 (7) | 0.0432 (7) | 0.0474 (8) | 0.0050 (5) | 0.0074 (6) | 0.0115 (6) |
| C13 | 0.0416 (7) | 0.0487 (7) | 0.0409 (7) | 0.0013 (6) | 0.0092 (6) | −0.0057 (6) |
| C4 | 0.0488 (8) | 0.0391 (7) | 0.0623 (9) | 0.0076 (6) | 0.0145 (7) | −0.0014 (6) |
| C12 | 0.0440 (7) | 0.0652 (9) | 0.0509 (9) | −0.0030 (7) | 0.0033 (7) | −0.0131 (7) |
| C11 | 0.0405 (7) | 0.0549 (8) | 0.0611 (9) | −0.0008 (6) | 0.0157 (7) | −0.0116 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N2—C9 | 1.4460 (16) | C8—C3 | 1.3850 (17) |
| N2—C13 | 1.4596 (16) | C8—C2 | 1.4597 (17) |
| N2—C10 | 1.4631 (16) | C2—C1 | 1.5490 (18) |
| N1—C1 | 1.3618 (15) | C9—H9A | 1.024 (14) |
| N1—C7 | 1.4199 (14) | C9—H9B | 0.979 (14) |
| N1—C9 | 1.4736 (15) | C5—C4 | 1.382 (2) |
| C7—C6 | 1.3765 (16) | C5—H5A | 0.9300 |
| C7—C8 | 1.4000 (15) | C3—C4 | 1.383 (2) |
| O2—C2 | 1.2075 (15) | C3—H3A | 0.9300 |
| C10—C11 | 1.5110 (18) | C13—C12 | 1.5109 (18) |
| C10—H10A | 0.9700 | C13—H13A | 0.9700 |
| C10—H10B | 0.9700 | C13—H13B | 0.9700 |
| O3—C11 | 1.4168 (16) | C4—H4A | 0.9300 |
| O3—C12 | 1.4217 (17) | C12—H12A | 0.9700 |
| O1—C1 | 1.2180 (14) | C12—H12B | 0.9700 |
| C6—C5 | 1.3914 (17) | C11—H11A | 0.9700 |
| C6—H6A | 0.9300 | C11—H11B | 0.9700 |
| C9—N2—C13 | 114.36 (10) | C4—C5—C6 | 121.82 (12) |
| C9—N2—C10 | 113.97 (10) | C4—C5—H5A | 119.1 |
| C13—N2—C10 | 109.92 (10) | C6—C5—H5A | 119.1 |
| C1—N1—C7 | 110.17 (10) | O1—C1—N1 | 126.89 (12) |
| C1—N1—C9 | 122.94 (10) | O1—C1—C2 | 126.25 (12) |
| C7—N1—C9 | 126.73 (10) | N1—C1—C2 | 106.86 (10) |
| C6—C7—C8 | 121.37 (11) | C4—C3—C8 | 118.27 (12) |
| C6—C7—N1 | 127.92 (11) | C4—C3—H3A | 120.9 |
| C8—C7—N1 | 110.71 (10) | C8—C3—H3A | 120.9 |
| N2—C10—C11 | 109.34 (11) | N2—C13—C12 | 109.35 (11) |
| N2—C10—H10A | 109.8 | N2—C13—H13A | 109.8 |
| C11—C10—H10A | 109.8 | C12—C13—H13A | 109.8 |

| | | | |
|---|---|---|---|
| N2—C10—H10B | 109.8 | N2—C13—H13B | 109.8 |
| C11—C10—H10B | 109.8 | C12—C13—H13B | 109.8 |
| H10A—C10—H10B | 108.3 | H13A—C13—H13B | 108.3 |
| C11—O3—C12 | 109.81 (11) | C3—C4—C5 | 120.66 (13) |
| C7—C6—C5 | 117.28 (11) | C3—C4—H4A | 119.7 |
| C7—C6—H6A | 121.4 | C5—C4—H4A | 119.7 |
| C5—C6—H6A | 121.4 | O3—C12—C13 | 111.11 (12) |
| C3—C8—C7 | 120.55 (11) | O3—C12—H12A | 109.4 |
| C3—C8—C2 | 132.02 (11) | C13—C12—H12A | 109.4 |
| C7—C8—C2 | 107.44 (10) | O3—C12—H12B | 109.4 |
| O2—C2—C8 | 131.85 (13) | C13—C12—H12B | 109.4 |
| O2—C2—C1 | 123.32 (12) | H12A—C12—H12B | 108.0 |
| C8—C2—C1 | 104.82 (10) | O3—C11—C10 | 111.53 (11) |
| N2—C9—N1 | 116.55 (10) | O3—C11—H11A | 109.3 |
| N2—C9—H9A | 110.1 (7) | C10—C11—H11A | 109.3 |
| N1—C9—H9A | 102.5 (7) | O3—C11—H11B | 109.3 |
| N2—C9—H9B | 108.9 (7) | C10—C11—H11B | 109.3 |
| N1—C9—H9B | 106.9 (7) | H11A—C11—H11B | 108.0 |
| H9A—C9—H9B | 111.9 (10) | | |

### *Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C6—H6A···O1$^{i}$ | 0.93 | 2.47 | 3.349 (2) | 158 |
| C5—H5A···O2$^{ii}$ | 0.93 | 2.52 | 3.216 (2) | 131 |

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

**Fig. 1**