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# 5,7-Dimethyl-1*H*-indole-2,3-dione

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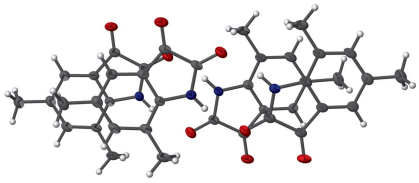
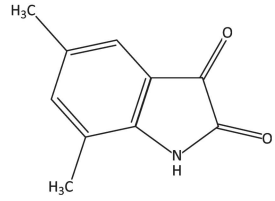
**Structural data**: full structural data are available from iucrdata.iucr.org

The title compound, $C_{10}H_9NO_2$, crystallizes with four molecules in the asymmetric unit. The molecules are all near planar, with the non-H atoms possessing mean deviations from planarity of 0.017, 0.026, 0.020 and 0.007 Å. In the crystal, the molecules form two dimers, each linked by a pair of N—H···O hydrogen bonds.

**3D view**



**Chemical scheme**




## Structure description

Herein we report the crystal structure of 5,7-dimethyl-1*H*-indole-2,3-dione: there are four of these 5,7-dimethylisatin molecules in the asymmetric unit (Fig. 1). The molecules are near planar, with the non-H atoms demonstrating r.m.s. deviations of 0.017, 0.026, 0.020 and 0.007 Å. The bond lengths and angles of the title compound are consistent with those observed in the other reported 5,7-substituted isatin structure (Golen & Manke, 2016).

In the crystal, the four molecules combine into pairs, each linked through two N—H···O hydrogen bonds (Table 1). This dimerization is also observed in 7-methylisatin (Lyncee *et al.*, 2017). The packing of the title compound, including hydrogen bonding, is shown in Fig. 2.

## Synthesis and crystallization

A commercial sample (Acros) of 5,7-dimethyl-1*H*-indole-2,3-dione was used for the crystallization. A sample suitable for single-crystal X-ray analysis was grown from the slow evaporation of its methyl acetate solution.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

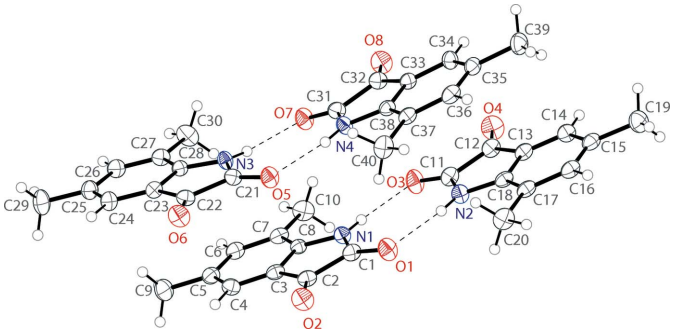



**Figure 1**
The molecular structure of the title compound, showing the atom-labeling scheme of the four molecules present in the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

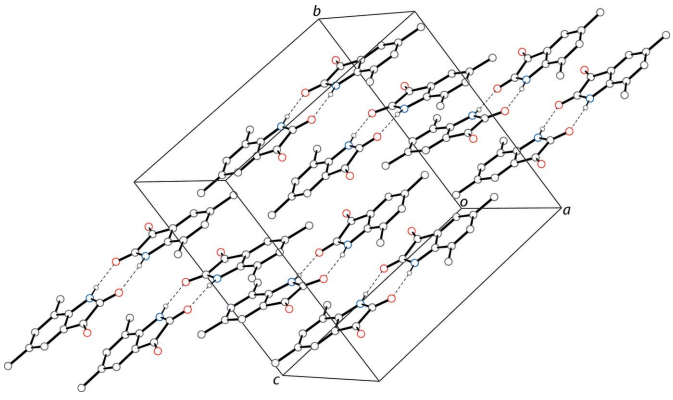



**Figure 2**
The molecular packing of the title compound with hydrogen bonds drawn as dashed lines.

## Funding information

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## References

Bruker (2014). *APEX2*, *SAINT*, and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O3 | 0.86 (1) | 2.07 (1) | 2.903 (3) | 164 (3) |
| N2—H2···O1 | 0.86 (1) | 2.04 (1) | 2.890 (3) | 169 (3) |
| N3—H3···O7 | 0.86 (1) | 2.09 (1) | 2.922 (3) | 163 (3) |
| N4—H4*A*···O5 | 0.86 (1) | 2.02 (1) | 2.867 (3) | 168 (3) |

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{10}H_9NO_2$ |
| $M_r$ | 175.18 |
| Crystal system, space group | Triclinic, $P\overline{1}$ |
| Temperature (K) | 200 |
| $a$, $b$, $c$ (Å) | 8.7148 (9), 13.5652 (17), 14.7832 (19) |
| $\alpha$, $\beta$, $\gamma$ (°) | 91.562 (5), 96.341 (4), 103.776 (4) |
| $V$ (Å$^3$) | 1684.4 (4) |
| $Z$ | 8 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.10 |
| Crystal size (mm) | 0.4 × 0.4 × 0.04 |
| | |
| Data collection | |
| Diffractometer | Bruker D8 Venture |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2014) |
| $T_{min}$, $T_{max}$ | 0.692, 0.745 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 56302, 6187, 4523 |
| $R_{int}$ | 0.060 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.603 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.063, 0.189, 1.10 |
| No. of reflections | 6187 |
| No. of parameters | 489 |
| No. of restraints | 4 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.39, −0.27 |

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

Golen, J. A. & Manke, D. R. (2016). *IUCrData*, **1**, x161510.
Lyncee, M. A., Desikan, V., Golen, J. A. & Manke, D. R. (2017). *IUCrData*, **2**, x170378.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

# full crystallographic data

*IUCrData* (2017). **2**, x170444 [https://doi.org/10.1107/S2414314617004448]

## 5,7-Dimethyl-1*H*-indole-2,3-dione

**Matthew F. Perez, Vasumathi Desikan, James A. Golen and David R. Manke**

### 5,7-Dimethyl-1*H*-indole-2,3-dione

*Crystal data*

$C_{10}H_9NO_2$
$M_r$ = 175.18
Triclinic, $P\overline{1}$
$a$ = 8.7148 (9) Å
$b$ = 13.5652 (17) Å
$c$ = 14.7832 (19) Å
$\alpha$ = 91.562 (5)°
$\beta$ = 96.341 (4)°
$\gamma$ = 103.776 (4)°
$V$ = 1684.4 (4) Å$^3$
$Z$ = 8
$F(000)$ = 736
$D_x$ = 1.382 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9884 reflections
$\theta$ = 3.1–25.3°
$\mu$ = 0.10 mm$^{-1}$
$T$ = 200 K
PLATE, orange
0.4 × 0.4 × 0.04 mm

*Data collection*

Bruker D8 Venture diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
$T_{min}$ = 0.692, $T_{max}$ = 0.745
56302 measured reflections
6187 independent reflections
4523 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060
$\theta_{max}$ = 25.4°, $\theta_{min}$ = 2.8°
$h$ = −10→10
$k$ = −16→16
$l$ = −17→17

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.189
$S$ = 1.10
6187 reflections
489 parameters
4 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0802P)^2 + 1.7614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.27 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.4016 (2) | 0.11027 (16) | 0.69510 (15) | 0.0238 (5) |
| H1 | 0.353 (3) | 0.110 (2) | 0.6412 (8) | 0.029* |
| O1 | 0.6533 (2) | 0.10964 (16) | 0.66010 (14) | 0.0363 (5) |
| O2 | 0.7175 (2) | 0.10836 (17) | 0.86028 (14) | 0.0384 (5) |
| C1 | 0.5564 (3) | 0.10994 (19) | 0.71358 (19) | 0.0251 (6) |
| C2 | 0.5897 (3) | 0.10911 (19) | 0.81955 (19) | 0.0252 (6) |
| C3 | 0.4366 (3) | 0.10900 (18) | 0.85238 (18) | 0.0232 (6) |
| C4 | 0.3923 (3) | 0.10889 (19) | 0.93965 (18) | 0.0260 (6) |
| H4 | 0.4675 | 0.1094 | 0.9914 | 0.031* |
| C5 | 0.2352 (3) | 0.10795 (19) | 0.94964 (19) | 0.0270 (6) |
| C6 | 0.1284 (3) | 0.10665 (19) | 0.87147 (19) | 0.0271 (6) |
| H6 | 0.0214 | 0.1059 | 0.8792 | 0.033* |
| C7 | 0.1693 (3) | 0.10639 (18) | 0.78225 (18) | 0.0237 (6) |
| C8 | 0.3269 (3) | 0.10886 (18) | 0.77573 (17) | 0.0204 (5) |
| C9 | 0.1800 (4) | 0.1088 (2) | 1.0429 (2) | 0.0368 (7) |
| H9A | 0.2189 | 0.0584 | 1.0792 | 0.055* |
| H9B | 0.0635 | 0.0921 | 1.0367 | 0.055* |
| H9C | 0.2220 | 0.1765 | 1.0734 | 0.055* |
| C10 | 0.0509 (3) | 0.1030 (2) | 0.7002 (2) | 0.0333 (7) |
| H10A | 0.0857 | 0.1629 | 0.6652 | 0.050* |
| H10B | −0.0532 | 0.1026 | 0.7196 | 0.050* |
| H10C | 0.0425 | 0.0413 | 0.6620 | 0.050* |
| N2 | 0.5531 (3) | 0.12309 (17) | 0.46883 (15) | 0.0263 (5) |
| H2 | 0.595 (3) | 0.119 (2) | 0.5236 (8) | 0.032* |
| O3 | 0.3010 (2) | 0.12231 (16) | 0.50270 (13) | 0.0366 (5) |
| O4 | 0.2490 (2) | 0.14359 (17) | 0.30327 (15) | 0.0412 (5) |
| C11 | 0.4008 (3) | 0.1263 (2) | 0.44925 (19) | 0.0271 (6) |
| C12 | 0.3738 (3) | 0.1373 (2) | 0.34425 (19) | 0.0274 (6) |
| C13 | 0.5289 (3) | 0.14013 (19) | 0.31343 (18) | 0.0242 (6) |
| C14 | 0.5803 (3) | 0.1514 (2) | 0.22828 (19) | 0.0274 (6) |
| H14 | 0.5083 | 0.1564 | 0.1764 | 0.033* |
| C15 | 0.7394 (3) | 0.15539 (19) | 0.21970 (19) | 0.0281 (6) |
| C16 | 0.8409 (3) | 0.14914 (19) | 0.2986 (2) | 0.0274 (6) |
| H16 | 0.9495 | 0.1528 | 0.2926 | 0.033* |
| C17 | 0.7926 (3) | 0.13796 (19) | 0.38523 (19) | 0.0255 (6) |
| C18 | 0.6332 (3) | 0.13328 (18) | 0.39022 (18) | 0.0238 (6) |
| C19 | 0.8017 (4) | 0.1678 (2) | 0.1285 (2) | 0.0389 (7) |
| H19A | 0.7569 | 0.1056 | 0.0897 | 0.058* |
| H19B | 0.9180 | 0.1805 | 0.1372 | 0.058* |
| H19C | 0.7707 | 0.2253 | 0.0994 | 0.058* |
| C20 | 0.9053 (3) | 0.1327 (2) | 0.4685 (2) | 0.0354 (7) |
| H20A | 0.9159 | 0.1924 | 0.5097 | 0.053* |
| H20B | 1.0097 | 0.1314 | 0.4504 | 0.053* |
| H20C | 0.8638 | 0.0708 | 0.4996 | 0.053* |
| N3 | 0.3688 (2) | 0.35893 (17) | 0.85114 (15) | 0.0238 (5) |

| | | | | |
|---|---|---|---|---|
| H3 | 0.318 (3) | 0.344 (2) | 0.7975 (9) | 0.029* |
| O5 | 0.6154 (2) | 0.34703 (15) | 0.81535 (13) | 0.0318 (5) |
| O6 | 0.6912 (2) | 0.37267 (16) | 1.01601 (14) | 0.0358 (5) |
| C21 | 0.5233 (3) | 0.35717 (19) | 0.86965 (18) | 0.0243 (6) |
| C22 | 0.5623 (3) | 0.36974 (19) | 0.97550 (18) | 0.0248 (6) |
| C23 | 0.4129 (3) | 0.37657 (18) | 1.00930 (17) | 0.0225 (5) |
| C24 | 0.3723 (3) | 0.3857 (2) | 1.09591 (18) | 0.0270 (6) |
| H24 | 0.4497 | 0.3905 | 1.1475 | 0.032* |
| C25 | 0.2169 (3) | 0.3880 (2) | 1.10712 (19) | 0.0286 (6) |
| C26 | 0.1068 (3) | 0.3803 (2) | 1.02911 (19) | 0.0278 (6) |
| H26 | 0.0009 | 0.3825 | 1.0371 | 0.033* |
| C27 | 0.1434 (3) | 0.36965 (19) | 0.94057 (18) | 0.0245 (6) |
| C28 | 0.3001 (3) | 0.36819 (18) | 0.93224 (17) | 0.0216 (5) |
| C29 | 0.1678 (4) | 0.3968 (3) | 1.2013 (2) | 0.0401 (7) |
| H29A | 0.1848 | 0.3388 | 1.2356 | 0.060* |
| H29B | 0.0549 | 0.3974 | 1.1962 | 0.060* |
| H29C | 0.2318 | 0.4600 | 1.2331 | 0.060* |
| C30 | 0.0199 (3) | 0.3593 (2) | 0.8587 (2) | 0.0361 (7) |
| H30A | 0.0486 | 0.4179 | 0.8213 | 0.054* |
| H30B | −0.0843 | 0.3565 | 0.8791 | 0.054* |
| H30C | 0.0151 | 0.2967 | 0.8226 | 0.054* |
| N4 | 0.5103 (2) | 0.35401 (17) | 0.62544 (15) | 0.0252 (5) |
| H4A | 0.554 (3) | 0.350 (2) | 0.6800 (8) | 0.030* |
| O7 | 0.2527 (2) | 0.34008 (15) | 0.65678 (13) | 0.0339 (5) |
| O8 | 0.2033 (2) | 0.37014 (16) | 0.45880 (14) | 0.0369 (5) |
| C31 | 0.3544 (3) | 0.35165 (19) | 0.60458 (18) | 0.0256 (6) |
| C32 | 0.3291 (3) | 0.36678 (19) | 0.50041 (18) | 0.0255 (6) |
| C33 | 0.4871 (3) | 0.37722 (18) | 0.47077 (17) | 0.0221 (5) |
| C34 | 0.5404 (3) | 0.39321 (19) | 0.38603 (18) | 0.0255 (6) |
| H34 | 0.4687 | 0.3990 | 0.3342 | 0.031* |
| C35 | 0.7010 (3) | 0.40072 (19) | 0.37810 (18) | 0.0260 (6) |
| C36 | 0.8016 (3) | 0.39118 (19) | 0.45634 (19) | 0.0280 (6) |
| H36 | 0.9104 | 0.3955 | 0.4506 | 0.034* |
| C37 | 0.7510 (3) | 0.37566 (19) | 0.54253 (18) | 0.0240 (6) |
| C38 | 0.5905 (3) | 0.36892 (18) | 0.54667 (17) | 0.0219 (5) |
| C39 | 0.7648 (4) | 0.4188 (2) | 0.2880 (2) | 0.0372 (7) |
| H39A | 0.7093 | 0.3631 | 0.2438 | 0.056* |
| H39B | 0.8789 | 0.4217 | 0.2956 | 0.056* |
| H39C | 0.7476 | 0.4832 | 0.2659 | 0.056* |
| C40 | 0.8614 (3) | 0.3659 (2) | 0.6257 (2) | 0.0349 (7) |
| H40A | 0.8644 | 0.4202 | 0.6714 | 0.052* |
| H40B | 0.9686 | 0.3713 | 0.6088 | 0.052* |
| H40C | 0.8232 | 0.2997 | 0.6511 | 0.052* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0199 (11) | 0.0291 (12) | 0.0230 (12) | 0.0071 (9) | 0.0016 (9) | 0.0024 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O1 | 0.0239 (10) | 0.0527 (13) | 0.0358 (12) | 0.0127 (9) | 0.0098 (9) | 0.0065 (9) |
| O2 | 0.0222 (11) | 0.0554 (13) | 0.0399 (12) | 0.0146 (9) | 0.0006 (9) | 0.0065 (10) |
| C1 | 0.0161 (13) | 0.0262 (13) | 0.0350 (15) | 0.0072 (10) | 0.0069 (11) | 0.0036 (11) |
| C2 | 0.0190 (13) | 0.0238 (13) | 0.0333 (15) | 0.0062 (10) | 0.0025 (11) | 0.0033 (11) |
| C3 | 0.0191 (13) | 0.0195 (12) | 0.0311 (15) | 0.0053 (9) | 0.0019 (10) | 0.0020 (10) |
| C4 | 0.0229 (14) | 0.0271 (14) | 0.0283 (15) | 0.0065 (10) | 0.0026 (11) | 0.0047 (11) |
| C5 | 0.0260 (14) | 0.0231 (13) | 0.0329 (15) | 0.0052 (10) | 0.0092 (11) | 0.0031 (11) |
| C6 | 0.0197 (13) | 0.0251 (13) | 0.0384 (16) | 0.0058 (10) | 0.0101 (11) | 0.0043 (11) |
| C7 | 0.0172 (12) | 0.0196 (12) | 0.0341 (15) | 0.0036 (9) | 0.0042 (10) | 0.0020 (10) |
| C8 | 0.0185 (12) | 0.0182 (12) | 0.0245 (13) | 0.0038 (9) | 0.0046 (10) | 0.0027 (10) |
| C9 | 0.0326 (16) | 0.0420 (17) | 0.0375 (17) | 0.0072 (13) | 0.0150 (13) | 0.0051 (13) |
| C10 | 0.0191 (14) | 0.0399 (16) | 0.0407 (17) | 0.0080 (11) | 0.0005 (12) | 0.0041 (13) |
| N2 | 0.0188 (11) | 0.0338 (12) | 0.0268 (12) | 0.0075 (9) | 0.0019 (9) | 0.0012 (10) |
| O3 | 0.0230 (10) | 0.0578 (14) | 0.0319 (11) | 0.0126 (9) | 0.0094 (9) | 0.0029 (10) |
| O4 | 0.0188 (10) | 0.0629 (15) | 0.0436 (13) | 0.0160 (9) | −0.0017 (9) | 0.0034 (11) |
| C11 | 0.0201 (13) | 0.0289 (14) | 0.0321 (15) | 0.0060 (10) | 0.0033 (11) | −0.0015 (11) |
| C12 | 0.0193 (14) | 0.0300 (14) | 0.0333 (15) | 0.0072 (10) | 0.0028 (11) | 0.0001 (11) |
| C13 | 0.0187 (13) | 0.0225 (13) | 0.0311 (15) | 0.0057 (10) | 0.0007 (11) | −0.0017 (11) |
| C14 | 0.0236 (14) | 0.0279 (14) | 0.0305 (15) | 0.0067 (10) | 0.0027 (11) | 0.0013 (11) |
| C15 | 0.0274 (14) | 0.0204 (13) | 0.0372 (16) | 0.0051 (10) | 0.0088 (12) | 0.0002 (11) |
| C16 | 0.0187 (13) | 0.0245 (13) | 0.0407 (16) | 0.0061 (10) | 0.0085 (11) | 0.0013 (11) |
| C17 | 0.0184 (13) | 0.0204 (13) | 0.0376 (16) | 0.0051 (10) | 0.0031 (11) | −0.0015 (11) |
| C18 | 0.0185 (13) | 0.0200 (12) | 0.0336 (15) | 0.0059 (9) | 0.0030 (11) | −0.0002 (10) |
| C19 | 0.0359 (17) | 0.0427 (17) | 0.0395 (18) | 0.0075 (13) | 0.0145 (14) | 0.0045 (14) |
| C20 | 0.0220 (14) | 0.0430 (17) | 0.0413 (18) | 0.0106 (12) | −0.0015 (12) | −0.0007 (13) |
| N3 | 0.0190 (11) | 0.0321 (12) | 0.0207 (11) | 0.0063 (9) | 0.0044 (9) | 0.0020 (9) |
| O5 | 0.0221 (10) | 0.0433 (12) | 0.0323 (11) | 0.0099 (8) | 0.0086 (8) | 0.0037 (9) |
| O6 | 0.0199 (10) | 0.0514 (13) | 0.0364 (12) | 0.0112 (9) | −0.0012 (8) | 0.0056 (9) |
| C21 | 0.0212 (13) | 0.0256 (13) | 0.0267 (14) | 0.0056 (10) | 0.0042 (11) | 0.0042 (10) |
| C22 | 0.0209 (14) | 0.0249 (13) | 0.0287 (14) | 0.0057 (10) | 0.0028 (11) | 0.0056 (11) |
| C23 | 0.0178 (12) | 0.0231 (13) | 0.0256 (14) | 0.0039 (10) | 0.0013 (10) | −0.0002 (10) |
| C24 | 0.0235 (14) | 0.0312 (14) | 0.0258 (14) | 0.0057 (11) | 0.0031 (11) | −0.0007 (11) |
| C25 | 0.0264 (14) | 0.0322 (15) | 0.0293 (15) | 0.0085 (11) | 0.0088 (11) | 0.0016 (11) |
| C26 | 0.0211 (13) | 0.0303 (14) | 0.0337 (15) | 0.0074 (10) | 0.0078 (11) | 0.0042 (11) |
| C27 | 0.0166 (13) | 0.0274 (14) | 0.0306 (15) | 0.0067 (10) | 0.0032 (10) | 0.0042 (11) |
| C28 | 0.0196 (12) | 0.0213 (12) | 0.0246 (13) | 0.0051 (9) | 0.0046 (10) | 0.0024 (10) |
| C29 | 0.0326 (16) | 0.058 (2) | 0.0316 (17) | 0.0118 (14) | 0.0108 (13) | −0.0025 (14) |
| C30 | 0.0191 (14) | 0.0547 (19) | 0.0364 (17) | 0.0132 (12) | 0.0008 (12) | 0.0108 (14) |
| N4 | 0.0198 (11) | 0.0353 (12) | 0.0233 (12) | 0.0101 (9) | 0.0056 (9) | 0.0059 (10) |
| O7 | 0.0240 (10) | 0.0474 (12) | 0.0331 (11) | 0.0106 (8) | 0.0112 (8) | 0.0061 (9) |
| O8 | 0.0200 (10) | 0.0542 (13) | 0.0386 (12) | 0.0135 (9) | 0.0015 (9) | 0.0056 (10) |
| C31 | 0.0205 (13) | 0.0285 (14) | 0.0300 (15) | 0.0090 (10) | 0.0055 (11) | 0.0050 (11) |
| C32 | 0.0191 (13) | 0.0273 (14) | 0.0306 (15) | 0.0063 (10) | 0.0035 (11) | 0.0035 (11) |
| C33 | 0.0178 (13) | 0.0239 (13) | 0.0257 (14) | 0.0068 (10) | 0.0033 (10) | 0.0016 (10) |
| C34 | 0.0244 (14) | 0.0293 (14) | 0.0221 (13) | 0.0061 (10) | 0.0009 (10) | −0.0013 (10) |
| C35 | 0.0238 (14) | 0.0250 (13) | 0.0299 (15) | 0.0047 (10) | 0.0088 (11) | 0.0007 (11) |
| C36 | 0.0195 (13) | 0.0260 (14) | 0.0387 (16) | 0.0050 (10) | 0.0062 (11) | −0.0021 (11) |
| C37 | 0.0171 (12) | 0.0234 (13) | 0.0319 (15) | 0.0065 (10) | 0.0015 (10) | −0.0010 (11) |

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|---|---|---|---|---|---|---|
| C38 | 0.0199 (13) | 0.0204 (12) | 0.0263 (14) | 0.0056 (9) | 0.0045 (10) | 0.0016 (10) |
| C39 | 0.0380 (17) | 0.0395 (17) | 0.0359 (17) | 0.0082 (13) | 0.0155 (13) | 0.0006 (13) |
| C40 | 0.0199 (14) | 0.0480 (18) | 0.0370 (17) | 0.0113 (12) | −0.0006 (12) | −0.0044 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—H1 | 0.860 (5) | N3—H3 | 0.860 (5) |
| N1—C1 | 1.348 (3) | N3—C21 | 1.350 (3) |
| N1—C8 | 1.418 (3) | N3—C28 | 1.413 (3) |
| O1—C1 | 1.219 (3) | O5—C21 | 1.223 (3) |
| O2—C2 | 1.209 (3) | O6—C22 | 1.204 (3) |
| C1—C2 | 1.562 (4) | C21—C22 | 1.560 (4) |
| C2—C3 | 1.469 (3) | C22—C23 | 1.467 (3) |
| C3—C4 | 1.387 (4) | C23—C24 | 1.375 (4) |
| C3—C8 | 1.399 (4) | C23—C28 | 1.403 (3) |
| C4—H4 | 0.9500 | C24—H24 | 0.9500 |
| C4—C5 | 1.390 (4) | C24—C25 | 1.390 (4) |
| C5—C6 | 1.398 (4) | C25—C26 | 1.401 (4) |
| C5—C9 | 1.510 (4) | C25—C29 | 1.511 (4) |
| C6—H6 | 0.9500 | C26—H26 | 0.9500 |
| C6—C7 | 1.404 (4) | C26—C27 | 1.393 (4) |
| C7—C8 | 1.380 (3) | C27—C28 | 1.389 (3) |
| C7—C10 | 1.496 (4) | C27—C30 | 1.508 (4) |
| C9—H9A | 0.9800 | C29—H29A | 0.9800 |
| C9—H9B | 0.9800 | C29—H29B | 0.9800 |
| C9—H9C | 0.9800 | C29—H29C | 0.9800 |
| C10—H10A | 0.9800 | C30—H30A | 0.9800 |
| C10—H10B | 0.9800 | C30—H30B | 0.9800 |
| C10—H10C | 0.9800 | C30—H30C | 0.9800 |
| N2—H2 | 0.860 (5) | N4—H4A | 0.862 (5) |
| N2—C11 | 1.338 (3) | N4—C31 | 1.352 (3) |
| N2—C18 | 1.414 (3) | N4—C38 | 1.418 (3) |
| O3—C11 | 1.231 (3) | O7—C31 | 1.223 (3) |
| O4—C12 | 1.208 (3) | O8—C32 | 1.207 (3) |
| C11—C12 | 1.561 (4) | C31—C32 | 1.558 (4) |
| C12—C13 | 1.465 (4) | C32—C33 | 1.467 (3) |
| C13—C14 | 1.382 (4) | C33—C34 | 1.388 (4) |
| C13—C18 | 1.394 (4) | C33—C38 | 1.384 (4) |
| C14—H14 | 0.9500 | C34—H34 | 0.9500 |
| C14—C15 | 1.395 (4) | C34—C35 | 1.398 (4) |
| C15—C16 | 1.401 (4) | C35—C36 | 1.401 (4) |
| C15—C19 | 1.507 (4) | C35—C39 | 1.503 (4) |
| C16—H16 | 0.9500 | C36—H36 | 0.9500 |
| C16—C17 | 1.393 (4) | C36—C37 | 1.399 (4) |
| C17—C18 | 1.386 (3) | C37—C38 | 1.388 (3) |
| C17—C20 | 1.502 (4) | C37—C40 | 1.505 (4) |
| C19—H19A | 0.9800 | C39—H39A | 0.9800 |
| C19—H19B | 0.9800 | C39—H39B | 0.9800 |

| | | | |
|---|---|---|---|
| C19—H19C | 0.9800 | C39—H39C | 0.9800 |
| C20—H20A | 0.9800 | C40—H40A | 0.9800 |
| C20—H20B | 0.9800 | C40—H40B | 0.9800 |
| C20—H20C | 0.9800 | C40—H40C | 0.9800 |
| | | | |
| C1—N1—H1 | 125 (2) | C21—N3—H3 | 122.3 (19) |
| C1—N1—C8 | 111.6 (2) | C21—N3—C28 | 111.0 (2) |
| C8—N1—H1 | 124 (2) | C28—N3—H3 | 126 (2) |
| N1—C1—C2 | 106.0 (2) | N3—C21—C22 | 106.2 (2) |
| O1—C1—N1 | 128.3 (3) | O5—C21—N3 | 127.6 (2) |
| O1—C1—C2 | 125.7 (2) | O5—C21—C22 | 126.2 (2) |
| O2—C2—C1 | 124.1 (2) | O6—C22—C21 | 124.2 (2) |
| O2—C2—C3 | 131.1 (3) | O6—C22—C23 | 130.6 (3) |
| C3—C2—C1 | 104.8 (2) | C23—C22—C21 | 105.2 (2) |
| C4—C3—C2 | 131.6 (2) | C24—C23—C22 | 132.2 (2) |
| C4—C3—C8 | 121.2 (2) | C24—C23—C28 | 121.3 (2) |
| C8—C3—C2 | 107.2 (2) | C28—C23—C22 | 106.4 (2) |
| C3—C4—H4 | 120.8 | C23—C24—H24 | 120.4 |
| C3—C4—C5 | 118.5 (2) | C23—C24—C25 | 119.2 (2) |
| C5—C4—H4 | 120.8 | C25—C24—H24 | 120.4 |
| C4—C5—C6 | 118.7 (2) | C24—C25—C26 | 118.4 (2) |
| C4—C5—C9 | 121.0 (3) | C24—C25—C29 | 120.6 (3) |
| C6—C5—C9 | 120.3 (2) | C26—C25—C29 | 121.0 (2) |
| C5—C6—H6 | 117.9 | C25—C26—H26 | 118.1 |
| C5—C6—C7 | 124.2 (2) | C27—C26—C25 | 123.8 (2) |
| C7—C6—H6 | 117.9 | C27—C26—H26 | 118.1 |
| C6—C7—C10 | 122.6 (2) | C26—C27—C30 | 122.0 (2) |
| C8—C7—C6 | 115.0 (2) | C28—C27—C26 | 116.0 (2) |
| C8—C7—C10 | 122.4 (2) | C28—C27—C30 | 122.0 (2) |
| C3—C8—N1 | 110.3 (2) | C23—C28—N3 | 111.1 (2) |
| C7—C8—N1 | 127.3 (2) | C27—C28—N3 | 127.7 (2) |
| C7—C8—C3 | 122.3 (2) | C27—C28—C23 | 121.2 (2) |
| C5—C9—H9A | 109.5 | C25—C29—H29A | 109.5 |
| C5—C9—H9B | 109.5 | C25—C29—H29B | 109.5 |
| C5—C9—H9C | 109.5 | C25—C29—H29C | 109.5 |
| H9A—C9—H9B | 109.5 | H29A—C29—H29B | 109.5 |
| H9A—C9—H9C | 109.5 | H29A—C29—H29C | 109.5 |
| H9B—C9—H9C | 109.5 | H29B—C29—H29C | 109.5 |
| C7—C10—H10A | 109.5 | C27—C30—H30A | 109.5 |
| C7—C10—H10B | 109.5 | C27—C30—H30B | 109.5 |
| C7—C10—H10C | 109.5 | C27—C30—H30C | 109.5 |
| H10A—C10—H10B | 109.5 | H30A—C30—H30B | 109.5 |
| H10A—C10—H10C | 109.5 | H30A—C30—H30C | 109.5 |
| H10B—C10—H10C | 109.5 | H30B—C30—H30C | 109.5 |
| C11—N2—H2 | 122 (2) | C31—N4—H4A | 123 (2) |
| C11—N2—C18 | 111.5 (2) | C31—N4—C38 | 110.7 (2) |
| C18—N2—H2 | 126 (2) | C38—N4—H4A | 126 (2) |
| N2—C11—C12 | 106.3 (2) | N4—C31—C32 | 106.4 (2) |

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|---|---|---|---|
| O3—C11—N2 | 127.5 (3) | O7—C31—N4 | 127.1 (3) |
| O3—C11—C12 | 126.2 (2) | O7—C31—C32 | 126.5 (2) |
| O4—C12—C11 | 124.1 (2) | O8—C32—C31 | 124.3 (2) |
| O4—C12—C13 | 131.4 (3) | O8—C32—C33 | 131.2 (3) |
| C13—C12—C11 | 104.5 (2) | C33—C32—C31 | 104.5 (2) |
| C14—C13—C12 | 131.6 (2) | C34—C33—C32 | 131.6 (2) |
| C14—C13—C18 | 121.1 (2) | C38—C33—C32 | 107.4 (2) |
| C18—C13—C12 | 107.2 (2) | C38—C33—C34 | 121.0 (2) |
| C13—C14—H14 | 120.5 | C33—C34—H34 | 120.5 |
| C13—C14—C15 | 119.0 (3) | C33—C34—C35 | 119.0 (2) |
| C15—C14—H14 | 120.5 | C35—C34—H34 | 120.5 |
| C14—C15—C16 | 118.2 (3) | C34—C35—C36 | 118.3 (2) |
| C14—C15—C19 | 121.1 (3) | C34—C35—C39 | 121.0 (3) |
| C16—C15—C19 | 120.7 (2) | C36—C35—C39 | 120.7 (2) |
| C15—C16—H16 | 117.9 | C35—C36—H36 | 118.1 |
| C17—C16—C15 | 124.1 (2) | C37—C36—C35 | 123.8 (2) |
| C17—C16—H16 | 117.9 | C37—C36—H36 | 118.1 |
| C16—C17—C20 | 122.7 (2) | C36—C37—C40 | 122.8 (2) |
| C18—C17—C16 | 115.6 (2) | C38—C37—C36 | 115.6 (2) |
| C18—C17—C20 | 121.7 (3) | C38—C37—C40 | 121.6 (2) |
| C13—C18—N2 | 110.5 (2) | C33—C38—N4 | 111.0 (2) |
| C17—C18—N2 | 127.6 (2) | C33—C38—C37 | 122.4 (2) |
| C17—C18—C13 | 121.9 (2) | C37—C38—N4 | 126.6 (2) |
| C15—C19—H19A | 109.5 | C35—C39—H39A | 109.5 |
| C15—C19—H19B | 109.5 | C35—C39—H39B | 109.5 |
| C15—C19—H19C | 109.5 | C35—C39—H39C | 109.5 |
| H19A—C19—H19B | 109.5 | H39A—C39—H39B | 109.5 |
| H19A—C19—H19C | 109.5 | H39A—C39—H39C | 109.5 |
| H19B—C19—H19C | 109.5 | H39B—C39—H39C | 109.5 |
| C17—C20—H20A | 109.5 | C37—C40—H40A | 109.5 |
| C17—C20—H20B | 109.5 | C37—C40—H40B | 109.5 |
| C17—C20—H20C | 109.5 | C37—C40—H40C | 109.5 |
| H20A—C20—H20B | 109.5 | H40A—C40—H40B | 109.5 |
| H20A—C20—H20C | 109.5 | H40A—C40—H40C | 109.5 |
| H20B—C20—H20C | 109.5 | H40B—C40—H40C | 109.5 |

| | | | |
|---|---|---|---|
| N1—C1—C2—O2 | 179.8 (2) | N3—C21—C22—O6 | −179.1 (2) |
| N1—C1—C2—C3 | 0.0 (3) | N3—C21—C22—C23 | 0.8 (3) |
| O1—C1—C2—O2 | 0.2 (4) | O5—C21—C22—O6 | 1.4 (4) |
| O1—C1—C2—C3 | −179.5 (3) | O5—C21—C22—C23 | −178.7 (2) |
| O2—C2—C3—C4 | 0.6 (5) | O6—C22—C23—C24 | −1.8 (5) |
| O2—C2—C3—C8 | −179.3 (3) | O6—C22—C23—C28 | −179.7 (3) |
| C1—N1—C8—C3 | 0.8 (3) | C21—N3—C28—C23 | 2.1 (3) |
| C1—N1—C8—C7 | −178.4 (2) | C21—N3—C28—C27 | −178.0 (2) |
| C1—C2—C3—C4 | −179.6 (3) | C21—C22—C23—C24 | 178.2 (3) |
| C1—C2—C3—C8 | 0.5 (3) | C21—C22—C23—C28 | 0.4 (3) |
| C2—C3—C4—C5 | −179.5 (2) | C22—C23—C24—C25 | −178.4 (3) |
| C2—C3—C8—N1 | −0.8 (3) | C22—C23—C28—N3 | −1.4 (3) |

| | | | |
|---|---|---|---|
| C2—C3—C8—C7 | 178.5 (2) | C22—C23—C28—C27 | 178.7 (2) |
| C3—C4—C5—C6 | 0.3 (4) | C23—C24—C25—C26 | 0.2 (4) |
| C3—C4—C5—C9 | −179.4 (2) | C23—C24—C25—C29 | 179.4 (3) |
| C4—C3—C8—N1 | 179.3 (2) | C24—C23—C28—N3 | −179.6 (2) |
| C4—C3—C8—C7 | −1.4 (4) | C24—C23—C28—C27 | 0.5 (4) |
| C4—C5—C6—C7 | −0.1 (4) | C24—C25—C26—C27 | 0.6 (4) |
| C5—C6—C7—C8 | −0.8 (4) | C25—C26—C27—C28 | −0.9 (4) |
| C5—C6—C7—C10 | 178.8 (2) | C25—C26—C27—C30 | 178.6 (3) |
| C6—C7—C8—N1 | −179.3 (2) | C26—C27—C28—N3 | −179.6 (2) |
| C6—C7—C8—C3 | 1.6 (3) | C26—C27—C28—C23 | 0.3 (4) |
| C8—N1—C1—O1 | 179.0 (3) | C28—N3—C21—O5 | 177.8 (3) |
| C8—N1—C1—C2 | −0.5 (3) | C28—N3—C21—C22 | −1.7 (3) |
| C8—C3—C4—C5 | 0.4 (4) | C28—C23—C24—C25 | −0.8 (4) |
| C9—C5—C6—C7 | 179.6 (2) | C29—C25—C26—C27 | −178.5 (3) |
| C10—C7—C8—N1 | 1.1 (4) | C30—C27—C28—N3 | 1.0 (4) |
| C10—C7—C8—C3 | −178.1 (2) | C30—C27—C28—C23 | −179.1 (2) |
| N2—C11—C12—O4 | −179.2 (3) | N4—C31—C32—O8 | −178.9 (3) |
| N2—C11—C12—C13 | −0.4 (3) | N4—C31—C32—C33 | −0.1 (3) |
| O3—C11—C12—O4 | 0.2 (4) | O7—C31—C32—O8 | 1.0 (4) |
| O3—C11—C12—C13 | 179.0 (3) | O7—C31—C32—C33 | 179.8 (3) |
| O4—C12—C13—C14 | 0.5 (5) | O8—C32—C33—C34 | −0.7 (5) |
| O4—C12—C13—C18 | 177.8 (3) | O8—C32—C33—C38 | 178.9 (3) |
| C11—N2—C18—C13 | −2.1 (3) | C31—N4—C38—C33 | 0.2 (3) |
| C11—N2—C18—C17 | 176.9 (2) | C31—N4—C38—C37 | 179.7 (2) |
| C11—C12—C13—C14 | −178.2 (3) | C31—C32—C33—C34 | −179.3 (3) |
| C11—C12—C13—C18 | −0.8 (3) | C31—C32—C33—C38 | 0.2 (3) |
| C12—C13—C14—C15 | 177.4 (3) | C32—C33—C34—C35 | 179.6 (2) |
| C12—C13—C18—N2 | 1.8 (3) | C32—C33—C38—N4 | −0.3 (3) |
| C12—C13—C18—C17 | −177.3 (2) | C32—C33—C38—C37 | −179.8 (2) |
| C13—C14—C15—C16 | −0.9 (4) | C33—C34—C35—C36 | 0.3 (4) |
| C13—C14—C15—C19 | −179.9 (2) | C33—C34—C35—C39 | −179.6 (2) |
| C14—C13—C18—N2 | 179.5 (2) | C34—C33—C38—N4 | 179.3 (2) |
| C14—C13—C18—C17 | 0.4 (4) | C34—C33—C38—C37 | −0.2 (4) |
| C14—C15—C16—C17 | 0.8 (4) | C34—C35—C36—C37 | −0.8 (4) |
| C15—C16—C17—C18 | −0.1 (4) | C35—C36—C37—C38 | 0.7 (4) |
| C15—C16—C17—C20 | −179.3 (2) | C35—C36—C37—C40 | −179.9 (2) |
| C16—C17—C18—N2 | −179.4 (2) | C36—C37—C38—N4 | −179.6 (2) |
| C16—C17—C18—C13 | −0.5 (4) | C36—C37—C38—C33 | −0.2 (4) |
| C18—N2—C11—O3 | −177.9 (3) | C38—N4—C31—O7 | −180.0 (3) |
| C18—N2—C11—C12 | 1.5 (3) | C38—N4—C31—C32 | 0.0 (3) |
| C18—C13—C14—C15 | 0.3 (4) | C38—C33—C34—C35 | 0.1 (4) |
| C19—C15—C16—C17 | 179.8 (2) | C39—C35—C36—C37 | 179.1 (2) |
| C20—C17—C18—N2 | −0.2 (4) | C40—C37—C38—N4 | 0.9 (4) |
| C20—C17—C18—C13 | 178.7 (2) | C40—C37—C38—C33 | −179.6 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O3 | 0.86 (1) | 2.07 (1) | 2.903 (3) | 164 (3) |
| N2—H2···O1 | 0.86 (1) | 2.04 (1) | 2.890 (3) | 169 (3) |
| N3—H3···O7 | 0.86 (1) | 2.09 (1) | 2.922 (3) | 163 (3) |
| N4—H4*A*···O5 | 0.86 (1) | 2.02 (1) | 2.867 (3) | 168 (3) |