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# 12-(4-Methoxybenzoyl)-2-methylbenzo[*f*]pyrido[1,2-*a*]indole-6,11-dione

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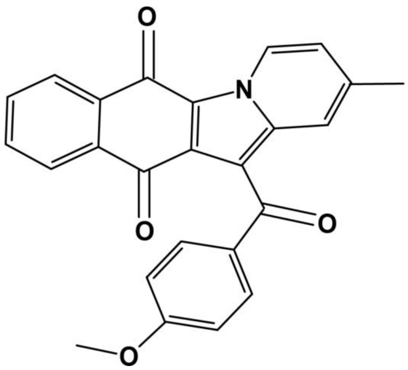
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.043; $wR$ factor = 0.120; data-to-parameter ratio = 14.2.

In the title compound, $C_{25}H_{17}NO_4$, the indolizine fused naphthaquinone unit is approximately planar [r.m.s deviation = 0.0678 Å] and makes a dihedral angle of 57.82 (5)° with the benzene ring of the methoxybenzene group. The naphthoquinone O atoms deviate, in the same sense, from the mean plane of the fused six-membered rings by 0.2001 (14) and 0.0516 (14) Å. In the crystal there is $\pi$–$\pi$ stacking of inversion-related pairs of molecules [interplanar spacing = 3.514 (2) Å].

## Related literature

For general background to the applications and biological activity of indolizine derivatives, see: Švorc *et al.* (2009). For the synthesis of indolizines, see: Babaev *et al.* (2005), and for their use as intermediates in the synthesis of indolizidines, see: Kloubert *et al.* (2012). For the crystal structures of similar compounds, see: Liu *et al.* (2011); Ramesh *et al.* (2009). For standard bond lengths, see: Allen *et al.* (1987).



## Experimental

### *Crystal data*

$C_{25}H_{17}NO_4$
$M_r$ = 395.40
Monoclinic, $P2_1/c$
$a$ = 8.1346 (3) Å
$b$ = 23.2926 (8) Å
$c$ = 10.1505 (3) Å
$\beta$ = 97.304 (2)°
$V$ = 1907.67 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
0.30 × 0.20 × 0.20 mm

### *Data collection*

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{\min}$ = 0.972, $T_{\max}$ = 0.982
18096 measured reflections
3852 independent reflections
2856 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.029

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.120
$S$ = 1.04
3852 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{\min}$ = −0.20 e Å$^{-3}$

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); 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: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2443).

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# supporting information

*Acta Cryst.* (2012). E**68**, o3040 [https://doi.org/10.1107/S1600536812040408]

## 12-(4-Methoxybenzoyl)-2-methylbenzo[*f*]pyrido[1,2-*a*]indole-6,11-dione

**J. Josephine Novina, G. Vasuki, Yun Liu and Jin-Wei Sun**

### S1. Comment

Indolizines, the nitrogen containing heterocyclic systems, are widely distributed in nature. In particular, indolizine derivatives are an important class of heterocyclic bioactive compounds with a wide range of applications, such as pharmaceutical drugs, potential central nervous system depressants, calcium entry blockers, cardiovascular agents, spectral sensitizers and novel dyes. Polycyclic indolizine derivatives have been found to have high-efficiency long-wavelength fluorescence quantum yield. Several polyhydroxylated indolizines are interesting as inhibitors of glycosides. They have also been tested as antimycobacterial agents against mycobacterial tuberculosis, for the treatment of angina pectoris, aromatase inhibitory, antiinflammatory, antiviral, analgesic and antitumor activities (Švorc *et al.*, 2009). Moreover, the application of indolizines themselves are as intermediates in the synthesis of indolizidines (Kloubert *et al.*, 2012) and many natural alkaloids contain in their structure a saturated (swainsonine) or aromatic (camptothecin) indolizine moiety (Babaev *et al.*, 2005). The benzo[*f*]pyrido[1,2-*a*]indole-6,11-diones are benzo-fused indolizines, and occur in several marine alkaloids (Liu *et al.*, 2011). The synthesis of these compounds has drawn much research interest. In view of their importance, the crystal structure determination of the title compound was carried out and results are presented herein.

In the title compound, $C_{25}H_{17}NO_4$, the fused naphthaquione–indolizine ring system (N/C1–C16/O1/O2) is approximately planar with a maximum deviation of 0.1193 (14) Å for atom C11 and -0.2001 (14) Å for atom O1, respectively. The fused ring systems make a dihedral angle of 57.82 (5)° with that of benzene ring of the methoxybenzene group. The torsion angles C11—C18—C19—C20 = -21.0 (2)° and C11—C18—C19—C24 = 161.52 (16)° also indicate that the aromatic ring is at different plane from the plane of the fused ring systems. The sum of bond angles around N [359.99 (43)°] indicates that atom N exhibits sp$^2$ hybridization. The geometric parameters of the title compound (Fig. 1) agree well with a reported similar structure 12-benzoyl-2-methylnaphtho[2,3-*b*]-indolizine-6,11-dione [Liu *et al.*, 2011]. The O2 atom is essentially coplanar with the ring, deviating by only -0.0516 (14) Å, while O1 deviates by -0.2001 (14) Å from the best-fit plane. The discrepancy in bond length is also observed for C9—C11 [1.400 (2) Å], which is slightly shorter than the average of 1.434 (1) Å calculated for indoles in the Cambridge Structure Database (Allen *et al.*, 1987).

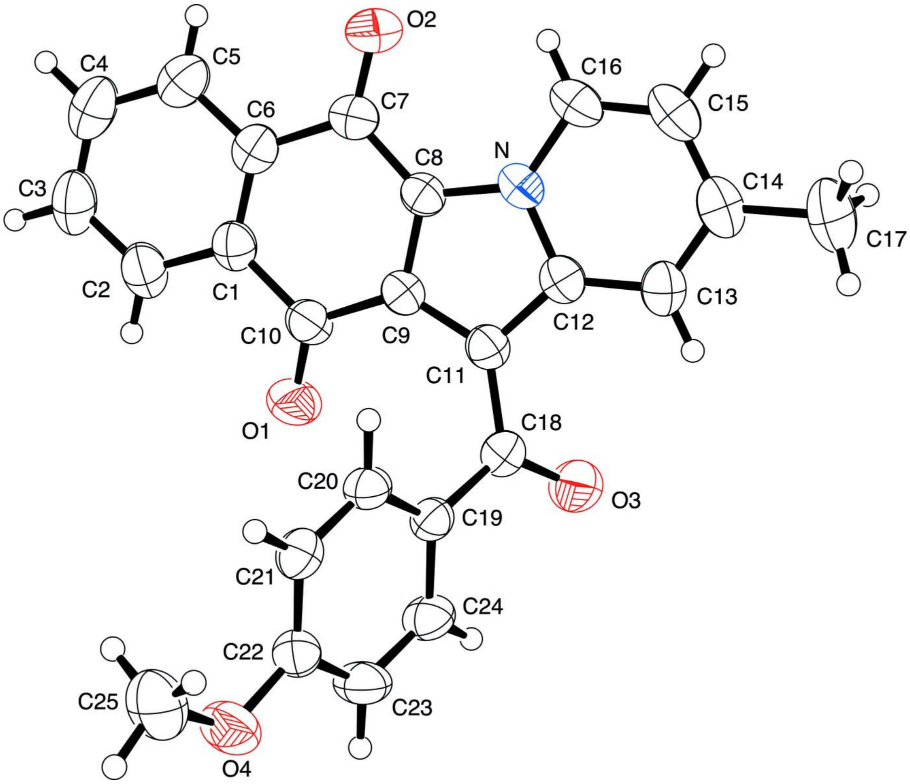
The endocyclic angle at C7 is contracted to 114.76 (15)° while those at C8 is expanded to 125.71 (15)°, respectively. This would appear to be a real effect caused by the fusion of the indolizine with naphthalene ring resulting an angular distortion as observed in the reported structure 3′-benzyloxy-3-hydroxy-3,3′-bi-1*H*-indole-2,2′(3*H*,3′H)-dione monohydrate [Ramesh *et al.*, 2009]. The widening of exocyclic angle O4—C22—C21 [125.05 (16)°] from the normal value of 120°, may be due to steric repulsion between atoms H21 and H25C (H21—H25C = 2.367 Å). In the crystal, there is $\pi$-$\pi$ stacking of inversion-related pairs of molecules [interplanar spacing = 3.514 (2) Å].

## S2. Experimental

4-Methyl pyridine (3.0 mmol), 2-bromo-1-(4-methoxyphenyl)ethanone (1.0 mmol), 1,4-naphthaquionone (1.0 mmol), and hydrated copper chloride (0.1 mmol) were mixed in 15 ml of $CH_3CN$ and heated to reflux for 12 h. After completion of the reaction, the reaction mixture was separated by silica gel column chromatography to afford the title compound (yield: 91%).

## S3. Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms: C—H =0.93 and 0.96 Å for CH and $CH_3$ H atoms, respectively, with $U_{iso}(H) = KU_{eq}$ (parent C-atom), where K=1.5 for $CH_3$ H atoms and K=1.2 for CH H-atoms.



### Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

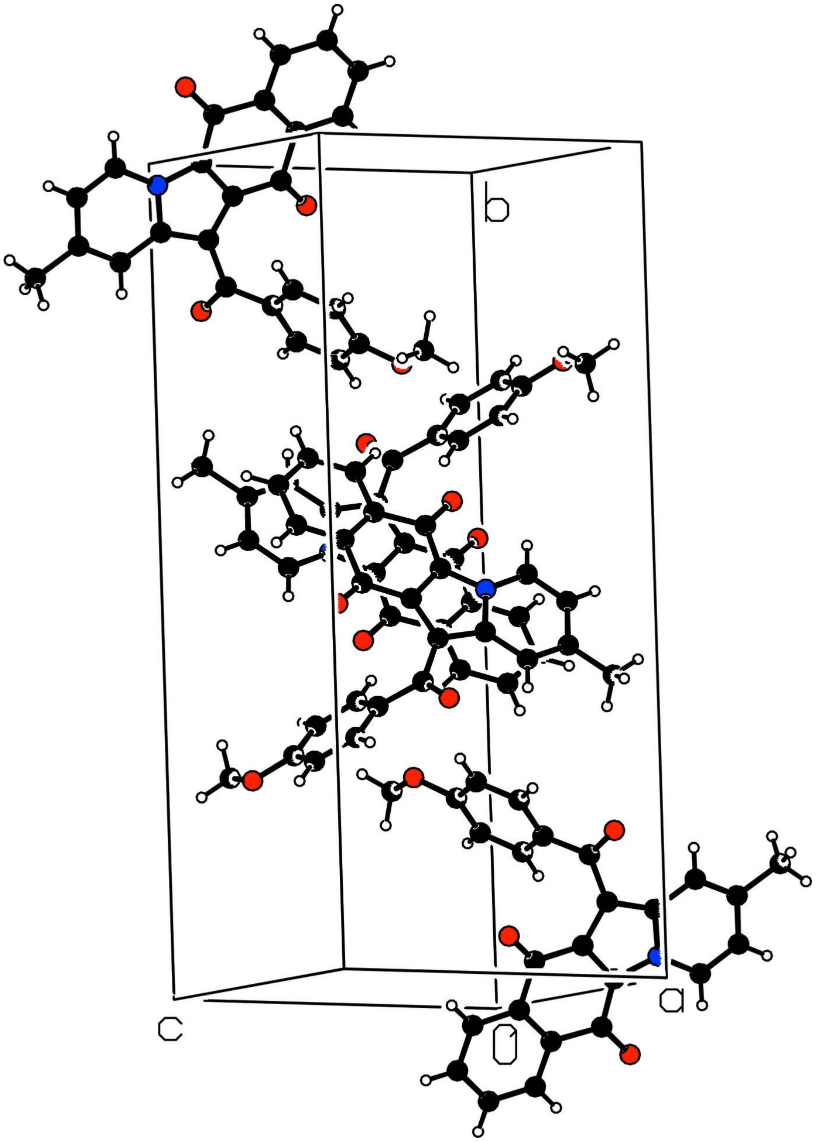


**Figure 2**

Crystal packing of the title compound viewed approximately down the bisector of the *a* and *c* axes.

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### *Crystal data*

$C_{25}H_{17}NO_4$
$M_r = 395.40$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.1346$ (3) Å
$b = 23.2926$ (8) Å
$c = 10.1505$ (3) Å
$\beta = 97.304$ (2)°
$V = 1907.67$ (11) Å$^3$
$Z = 4$

$F(000) = 824$
$D_x = 1.377$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5015 reflections
$\theta = 2.2$–26.3°
$\mu = 0.09$ mm$^{-1}$
$T = 293$ K
Block, brown
0.30 × 0.20 × 0.20 mm

*Data collection*

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{\min} = 0.972$, $T_{\max} = 0.982$
18096 measured reflections
3852 independent reflections
2856 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.029$
$\theta_{\max} = 26.3°$, $\theta_{\min} = 2.2°$
$h = -10 \rightarrow 10$
$k = -28 \rightarrow 29$
$l = -12 \rightarrow 10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.120$
$S = 1.04$
3852 reflections
271 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
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.4967P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.26$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.20$ 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{\text{iso}}$*/$U_{\text{eq}}$ |
|---|---|---|---|---|
| N | 0.17994 (16) | 0.53308 (6) | 0.57068 (13) | 0.0388 (3) |
| O2 | 0.34773 (17) | 0.42161 (6) | 0.56349 (14) | 0.0628 (4) |
| C8 | 0.24197 (19) | 0.50723 (7) | 0.46488 (15) | 0.0376 (4) |
| C9 | 0.21764 (19) | 0.54500 (7) | 0.35825 (16) | 0.0383 (4) |
| O1 | 0.2104 (2) | 0.55356 (6) | 0.12630 (12) | 0.0652 (4) |
| C6 | 0.3785 (2) | 0.43608 (7) | 0.33619 (18) | 0.0435 (4) |
| C18 | 0.0992 (2) | 0.64903 (7) | 0.32447 (17) | 0.0439 (4) |
| C19 | 0.2224 (2) | 0.67504 (7) | 0.24748 (16) | 0.0379 (4) |
| C1 | 0.3460 (2) | 0.47115 (7) | 0.22307 (18) | 0.0443 (4) |
| C21 | 0.5041 (2) | 0.68874 (7) | 0.20229 (17) | 0.0431 (4) |
| H21 | 0.6165 | 0.6806 | 0.2219 | 0.052* |
| O3 | −0.03169 (17) | 0.67344 (7) | 0.33481 (16) | 0.0714 (5) |
| C16 | 0.1778 (2) | 0.51365 (9) | 0.69868 (16) | 0.0477 (4) |
| H16 | 0.2195 | 0.4776 | 0.7238 | 0.057* |
| C10 | 0.2554 (2) | 0.52654 (7) | 0.22716 (17) | 0.0444 (4) |
| C12 | 0.11594 (19) | 0.58666 (7) | 0.53067 (16) | 0.0399 (4) |

| | | | | |
|---|---|---|---|---|
| C7 | 0.3231 (2) | 0.45262 (8) | 0.46517 (17) | 0.0426 (4) |
| C14 | 0.0491 (2) | 0.60280 (9) | 0.75177 (18) | 0.0509 (5) |
| C23 | 0.2821 (2) | 0.74026 (8) | 0.0772 (2) | 0.0539 (5) |
| H23 | 0.2453 | 0.7662 | 0.0101 | 0.065* |
| O4 | 0.54786 (16) | 0.75454 (6) | 0.02430 (14) | 0.0634 (4) |
| C20 | 0.3900 (2) | 0.66254 (7) | 0.27206 (16) | 0.0409 (4) |
| H20 | 0.4265 | 0.6358 | 0.3373 | 0.049* |
| C13 | 0.0496 (2) | 0.62101 (8) | 0.62437 (18) | 0.0464 (4) |
| H13 | 0.0052 | 0.6567 | 0.5991 | 0.056* |
| C22 | 0.4494 (2) | 0.72723 (7) | 0.10299 (17) | 0.0443 (4) |
| C2 | 0.3967 (2) | 0.45322 (9) | 0.1042 (2) | 0.0553 (5) |
| H2 | 0.3732 | 0.4758 | 0.0285 | 0.066* |
| C11 | 0.14110 (19) | 0.59490 (7) | 0.39734 (16) | 0.0403 (4) |
| C24 | 0.1712 (2) | 0.71529 (8) | 0.14967 (18) | 0.0483 (4) |
| H24 | 0.0598 | 0.7253 | 0.1334 | 0.058* |
| C15 | 0.1142 (2) | 0.54769 (9) | 0.78676 (18) | 0.0539 (5) |
| H15 | 0.1129 | 0.5346 | 0.8732 | 0.065* |
| C4 | 0.5135 (3) | 0.36798 (9) | 0.2080 (2) | 0.0644 (6) |
| H4 | 0.5702 | 0.3335 | 0.2030 | 0.077* |
| C5 | 0.4614 (2) | 0.38467 (8) | 0.3266 (2) | 0.0538 (5) |
| H5 | 0.4824 | 0.3611 | 0.4008 | 0.065* |
| C17 | −0.0160 (3) | 0.63983 (11) | 0.8546 (2) | 0.0731 (7) |
| H17A | −0.0058 | 0.6197 | 0.9379 | 0.110* |
| H17B | −0.1305 | 0.6486 | 0.8270 | 0.110* |
| H17C | 0.0467 | 0.6748 | 0.8649 | 0.110* |
| C3 | 0.4816 (3) | 0.40227 (9) | 0.0974 (2) | 0.0646 (6) |
| H3 | 0.5174 | 0.3911 | 0.0178 | 0.077* |
| C25 | 0.7210 (2) | 0.74482 (11) | 0.0475 (3) | 0.0754 (7) |
| H25A | 0.7748 | 0.7666 | −0.0149 | 0.113* |
| H25B | 0.7430 | 0.7047 | 0.0368 | 0.113* |
| H25C | 0.7624 | 0.7565 | 0.1362 | 0.113* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N | 0.0378 (7) | 0.0458 (8) | 0.0328 (7) | −0.0053 (6) | 0.0042 (6) | 0.0017 (6) |
| O2 | 0.0696 (9) | 0.0613 (9) | 0.0577 (8) | 0.0171 (7) | 0.0086 (7) | 0.0195 (7) |
| C8 | 0.0367 (8) | 0.0409 (9) | 0.0352 (9) | −0.0036 (7) | 0.0045 (7) | 0.0012 (7) |
| C9 | 0.0375 (8) | 0.0406 (9) | 0.0370 (9) | −0.0022 (7) | 0.0061 (7) | 0.0005 (7) |
| O1 | 0.1040 (11) | 0.0559 (8) | 0.0363 (7) | 0.0112 (8) | 0.0117 (7) | 0.0047 (6) |
| C6 | 0.0366 (9) | 0.0411 (10) | 0.0529 (11) | −0.0042 (7) | 0.0058 (8) | −0.0034 (8) |
| C18 | 0.0452 (10) | 0.0427 (10) | 0.0444 (10) | 0.0067 (8) | 0.0081 (8) | 0.0021 (8) |
| C19 | 0.0417 (9) | 0.0344 (9) | 0.0377 (9) | 0.0031 (7) | 0.0049 (7) | −0.0014 (7) |
| C1 | 0.0436 (9) | 0.0431 (10) | 0.0479 (10) | −0.0067 (7) | 0.0123 (8) | −0.0052 (8) |
| C21 | 0.0389 (9) | 0.0444 (10) | 0.0453 (10) | 0.0049 (7) | 0.0033 (7) | −0.0012 (8) |
| O3 | 0.0561 (8) | 0.0741 (10) | 0.0893 (11) | 0.0229 (7) | 0.0302 (8) | 0.0266 (8) |
| C16 | 0.0476 (10) | 0.0604 (12) | 0.0346 (9) | −0.0068 (9) | 0.0031 (8) | 0.0086 (9) |
| C10 | 0.0543 (10) | 0.0411 (10) | 0.0388 (10) | −0.0048 (8) | 0.0097 (8) | 0.0002 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C12 | 0.0353 (8) | 0.0445 (9) | 0.0402 (9) | −0.0055 (7) | 0.0056 (7) | −0.0012 (8) |
| C7 | 0.0374 (9) | 0.0446 (10) | 0.0447 (10) | −0.0034 (7) | 0.0014 (7) | 0.0053 (8) |
| C14 | 0.0436 (10) | 0.0670 (13) | 0.0433 (10) | −0.0125 (9) | 0.0095 (8) | −0.0108 (9) |
| C23 | 0.0517 (11) | 0.0515 (11) | 0.0579 (12) | 0.0064 (9) | 0.0044 (9) | 0.0211 (9) |
| O4 | 0.0549 (8) | 0.0670 (9) | 0.0710 (9) | −0.0051 (7) | 0.0179 (7) | 0.0200 (7) |
| C20 | 0.0460 (9) | 0.0394 (9) | 0.0365 (9) | 0.0073 (7) | 0.0017 (7) | 0.0036 (7) |
| C13 | 0.0422 (9) | 0.0507 (10) | 0.0473 (10) | −0.0040 (8) | 0.0102 (8) | −0.0081 (8) |
| C22 | 0.0485 (10) | 0.0394 (9) | 0.0462 (10) | −0.0028 (8) | 0.0108 (8) | −0.0006 (8) |
| C2 | 0.0608 (12) | 0.0542 (11) | 0.0543 (11) | −0.0064 (9) | 0.0202 (9) | −0.0091 (9) |
| C11 | 0.0399 (9) | 0.0423 (9) | 0.0395 (9) | −0.0012 (7) | 0.0079 (7) | 0.0011 (7) |
| C24 | 0.0383 (9) | 0.0473 (10) | 0.0582 (11) | 0.0071 (8) | 0.0024 (8) | 0.0112 (9) |
| C15 | 0.0534 (11) | 0.0751 (14) | 0.0335 (9) | −0.0127 (10) | 0.0073 (8) | −0.0006 (9) |
| C4 | 0.0555 (12) | 0.0530 (12) | 0.0865 (16) | 0.0040 (10) | 0.0153 (11) | −0.0176 (12) |
| C5 | 0.0478 (10) | 0.0449 (10) | 0.0682 (13) | 0.0010 (8) | 0.0054 (9) | −0.0037 (9) |
| C17 | 0.0779 (15) | 0.0912 (17) | 0.0537 (12) | −0.0094 (13) | 0.0219 (11) | −0.0256 (12) |
| C3 | 0.0655 (13) | 0.0615 (13) | 0.0712 (14) | −0.0054 (11) | 0.0266 (11) | −0.0222 (12) |
| C25 | 0.0506 (12) | 0.0877 (17) | 0.0921 (17) | −0.0101 (11) | 0.0251 (12) | 0.0067 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N—C16 | 1.378 (2) | C14—C13 | 1.361 (3) |
| N—C8 | 1.381 (2) | C14—C15 | 1.416 (3) |
| N—C12 | 1.393 (2) | C14—C17 | 1.502 (3) |
| O2—C7 | 1.228 (2) | C23—C24 | 1.365 (2) |
| C8—C9 | 1.389 (2) | C23—C22 | 1.386 (3) |
| C8—C7 | 1.433 (2) | C23—H23 | 0.9300 |
| C9—C11 | 1.400 (2) | O4—C22 | 1.359 (2) |
| C9—C10 | 1.467 (2) | O4—C25 | 1.416 (2) |
| O1—C10 | 1.218 (2) | C20—H20 | 0.9300 |
| C6—C5 | 1.384 (2) | C13—H13 | 0.9300 |
| C6—C1 | 1.407 (2) | C2—C3 | 1.379 (3) |
| C6—C7 | 1.489 (2) | C2—H2 | 0.9300 |
| C18—O3 | 1.223 (2) | C24—H24 | 0.9300 |
| C18—C19 | 1.477 (2) | C15—H15 | 0.9300 |
| C18—C11 | 1.479 (2) | C4—C3 | 1.375 (3) |
| C19—C20 | 1.385 (2) | C4—C5 | 1.382 (3) |
| C19—C24 | 1.390 (2) | C4—H4 | 0.9300 |
| C1—C2 | 1.388 (2) | C5—H5 | 0.9300 |
| C1—C10 | 1.489 (2) | C17—H17A | 0.9600 |
| C21—C20 | 1.379 (2) | C17—H17B | 0.9600 |
| C21—C22 | 1.380 (2) | C17—H17C | 0.9600 |
| C21—H21 | 0.9300 | C3—H3 | 0.9300 |
| C16—C15 | 1.347 (3) | C25—H25A | 0.9600 |
| C16—H16 | 0.9300 | C25—H25B | 0.9600 |
| C12—C13 | 1.402 (2) | C25—H25C | 0.9600 |
| C12—C11 | 1.407 (2) | | |

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| C16—N—C8 | 129.76 (15) | C21—C20—C19 | 121.72 (15) |

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| C16—N—C12 | 121.34 (15) | C21—C20—H20 | 119.1 |
| C8—N—C12 | 108.89 (13) | C19—C20—H20 | 119.1 |
| N—C8—C9 | 107.44 (14) | C14—C13—C12 | 120.95 (18) |
| N—C8—C7 | 126.80 (14) | C14—C13—H13 | 119.5 |
| C9—C8—C7 | 125.71 (15) | C12—C13—H13 | 119.5 |
| C8—C9—C11 | 109.24 (14) | O4—C22—C21 | 125.05 (16) |
| C8—C9—C10 | 119.70 (15) | O4—C22—C23 | 115.09 (16) |
| C11—C9—C10 | 130.71 (15) | C21—C22—C23 | 119.86 (16) |
| C5—C6—C1 | 119.22 (17) | C3—C2—C1 | 120.6 (2) |
| C5—C6—C7 | 119.38 (17) | C3—C2—H2 | 119.7 |
| C1—C6—C7 | 121.39 (15) | C1—C2—H2 | 119.7 |
| O3—C18—C19 | 120.77 (16) | C9—C11—C12 | 106.48 (14) |
| O3—C18—C11 | 120.06 (16) | C9—C11—C18 | 130.43 (15) |
| C19—C18—C11 | 119.03 (14) | C12—C11—C18 | 122.99 (15) |
| C20—C19—C24 | 117.97 (15) | C23—C24—C19 | 120.92 (16) |
| C20—C19—C18 | 122.49 (15) | C23—C24—H24 | 119.5 |
| C24—C19—C18 | 119.50 (15) | C19—C24—H24 | 119.5 |
| C2—C1—C6 | 119.25 (17) | C16—C15—C14 | 122.00 (17) |
| C2—C1—C10 | 119.19 (17) | C16—C15—H15 | 119.0 |
| C6—C1—C10 | 121.54 (15) | C14—C15—H15 | 119.0 |
| C20—C21—C22 | 119.14 (16) | C3—C4—C5 | 120.08 (19) |
| C20—C21—H21 | 120.4 | C3—C4—H4 | 120.0 |
| C22—C21—H21 | 120.4 | C5—C4—H4 | 120.0 |
| C15—C16—N | 119.00 (18) | C4—C5—C6 | 120.7 (2) |
| C15—C16—H16 | 120.5 | C4—C5—H5 | 119.6 |
| N—C16—H16 | 120.5 | C6—C5—H5 | 119.6 |
| O1—C10—C9 | 122.39 (16) | C14—C17—H17A | 109.5 |
| O1—C10—C1 | 121.42 (16) | C14—C17—H17B | 109.5 |
| C9—C10—C1 | 116.13 (15) | H17A—C17—H17B | 109.5 |
| N—C12—C13 | 118.38 (15) | C14—C17—H17C | 109.5 |
| N—C12—C11 | 107.93 (14) | H17A—C17—H17C | 109.5 |
| C13—C12—C11 | 133.63 (17) | H17B—C17—H17C | 109.5 |
| O2—C7—C8 | 123.46 (17) | C4—C3—C2 | 120.1 (2) |
| O2—C7—C6 | 121.77 (16) | C4—C3—H3 | 119.9 |
| C8—C7—C6 | 114.76 (15) | C2—C3—H3 | 119.9 |
| C13—C14—C15 | 118.33 (17) | O4—C25—H25A | 109.5 |
| C13—C14—C17 | 121.7 (2) | O4—C25—H25B | 109.5 |
| C15—C14—C17 | 119.98 (18) | H25A—C25—H25B | 109.5 |
| C24—C23—C22 | 120.34 (17) | O4—C25—H25C | 109.5 |
| C24—C23—H23 | 119.8 | H25A—C25—H25C | 109.5 |
| C22—C23—H23 | 119.8 | H25B—C25—H25C | 109.5 |
| C22—O4—C25 | 118.39 (16) | | |

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| C16—N—C8—C9 | −178.53 (15) | C22—C21—C20—C19 | −1.8 (3) |
| C12—N—C8—C9 | 0.38 (17) | C24—C19—C20—C21 | −0.2 (3) |
| C16—N—C8—C7 | −1.0 (3) | C18—C19—C20—C21 | −177.69 (16) |
| C12—N—C8—C7 | 177.96 (15) | C15—C14—C13—C12 | −1.4 (3) |
| N—C8—C9—C11 | 0.40 (18) | C17—C14—C13—C12 | 177.84 (17) |

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| C7—C8—C9—C11 | −177.21 (15) | N—C12—C13—C14 | 0.7 (2) |
| N—C8—C9—C10 | −173.46 (14) | C11—C12—C13—C14 | −176.11 (17) |
| C7—C8—C9—C10 | 8.9 (2) | C25—O4—C22—C21 | −2.3 (3) |
| O3—C18—C19—C20 | 154.62 (18) | C25—O4—C22—C23 | 178.04 (18) |
| C11—C18—C19—C20 | −21.0 (2) | C20—C21—C22—O4 | −177.84 (17) |
| O3—C18—C19—C24 | −22.9 (3) | C20—C21—C22—C23 | 1.8 (3) |
| C11—C18—C19—C24 | 161.52 (16) | C24—C23—C22—O4 | 179.80 (17) |
| C5—C6—C1—C2 | 0.4 (3) | C24—C23—C22—C21 | 0.1 (3) |
| C7—C6—C1—C2 | −178.47 (16) | C6—C1—C2—C3 | −1.5 (3) |
| C5—C6—C1—C10 | 179.22 (16) | C10—C1—C2—C3 | 179.66 (17) |
| C7—C6—C1—C10 | 0.3 (2) | C8—C9—C11—C12 | −1.00 (18) |
| C8—N—C16—C15 | 177.94 (16) | C10—C9—C11—C12 | 171.95 (17) |
| C12—N—C16—C15 | −0.9 (2) | C8—C9—C11—C18 | 175.43 (16) |
| C8—C9—C10—O1 | 167.00 (17) | C10—C9—C11—C18 | −11.6 (3) |
| C11—C9—C10—O1 | −5.3 (3) | N—C12—C11—C9 | 1.22 (18) |
| C8—C9—C10—C1 | −10.4 (2) | C13—C12—C11—C9 | 178.26 (17) |
| C11—C9—C10—C1 | 177.31 (16) | N—C12—C11—C18 | −175.55 (14) |
| C2—C1—C10—O1 | 7.4 (3) | C13—C12—C11—C18 | 1.5 (3) |
| C6—C1—C10—O1 | −171.35 (17) | O3—C18—C11—C9 | 140.6 (2) |
| C2—C1—C10—C9 | −175.19 (16) | C19—C18—C11—C9 | −43.8 (3) |
| C6—C1—C10—C9 | 6.0 (2) | O3—C18—C11—C12 | −43.5 (3) |
| C16—N—C12—C13 | 0.5 (2) | C19—C18—C11—C12 | 132.13 (17) |
| C8—N—C12—C13 | −178.57 (14) | C22—C23—C24—C19 | −2.1 (3) |
| C16—N—C12—C11 | 178.02 (14) | C20—C19—C24—C23 | 2.1 (3) |
| C8—N—C12—C11 | −1.00 (17) | C18—C19—C24—C23 | 179.74 (17) |
| N—C8—C7—O2 | −0.2 (3) | N—C16—C15—C14 | 0.2 (3) |
| C9—C8—C7—O2 | 176.99 (17) | C13—C14—C15—C16 | 1.0 (3) |
| N—C8—C7—C6 | −179.40 (14) | C17—C14—C15—C16 | −178.26 (18) |
| C9—C8—C7—C6 | −2.2 (2) | C3—C4—C5—C6 | −0.7 (3) |
| C5—C6—C7—O2 | −0.6 (3) | C1—C6—C5—C4 | 0.7 (3) |
| C1—C6—C7—O2 | 178.29 (16) | C7—C6—C5—C4 | 179.60 (16) |
| C5—C6—C7—C8 | 178.63 (15) | C5—C4—C3—C2 | −0.4 (3) |
| C1—C6—C7—C8 | −2.5 (2) | C1—C2—C3—C4 | 1.5 (3) |