# organic compounds

# 6-(4-Methoxyphenyl)-6a-nitro-6,6a,6b,7,8,9,10,12a-octahydrospiro-[chromeno[3,4-a]indolizine-12,3'-indolin]-2'-one
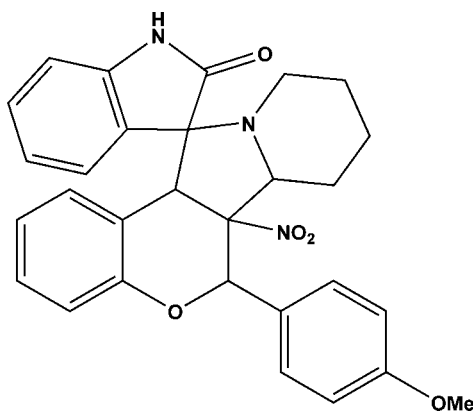
Seenivasan Karthiga Devi,[a] Thothadri Srinivasan,[a] Jonnalagadda Naga Siva Rao,[b] Raghavachary Raghunathan[b] and Devadasan Velmurugan[a]*

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In the title compound, $C_{29}H_{27}N_3O_5$, the hydropyran ring adopts an envelope conformation with the methine C atom bearing the *para*-methoxybenzene ring as the flap. The central pyrrolidine ring has a twist conformation on the N—C bond involving the spiro C atom. The piperidine ring adopts a chair conformation. An intramolecular C—H⋯O contact closes an $S(7)$ ring. In the crystal, inversion dimers linked by C—H⋯O interactions generate $R_2^2(18)$ loops and N—H⋯O hydrogen bonds connect the dimers into [100] chains.

## Related literature

For a related structure and background to 4*H*-chromene derivatives, see: Devi *et al.* (2013).

## Experimental

### Crystal data

| | |
|---|---|
| $C_{29}H_{27}N_3O_5$ | $\gamma$ = 66.757 (1)° |
| $M_r$ = 497.54 | $V$ = 1222.49 (7) Å³ |
| Triclinic, $P\bar{1}$ | $Z$ = 2 |
| $a$ = 9.3438 (3) Å | Mo $K\alpha$ radiation |
| $b$ = 11.3626 (4) Å | $\mu$ = 0.09 mm⁻¹ |
| $c$ = 13.5713 (4) Å | $T$ = 293 K |
| $\alpha$ = 68.687 (1)° | 0.30 × 0.25 × 0.20 mm |
| $\beta$ = 88.284 (1)° | |

### Data collection

| | |
|---|---|
| Bruker SMART APEXII diffractometer | 17765 measured reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2008) | 4964 independent reflections |
| $T_{min}$ = 0.973, $T_{max}$ = 0.982 | 4116 reflections with $I > 2\sigma(I)$ |
| | $R_{int}$ = 0.027 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.042 | 335 parameters |
| $wR(F^2)$ = 0.119 | H-atom parameters constrained |
| $S$ = 1.03 | $\Delta\rho_{max}$ = 0.22 e Å⁻³ |
| 4964 reflections | $\Delta\rho_{min}$ = −0.23 e Å⁻³ |

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N3—H3$A$⋯O3[i] | 0.86 | 2.24 | 3.085 (2) | 167 |
| C14—H14⋯O5[ii] | 0.93 | 2.54 | 3.358 (2) | 147 |
| C8—H8⋯O5 | 0.98 | 2.40 | 3.234 (2) | 143 |

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

## References

Bruker (2008). *APEX2, SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Devi, S. K., Srinivasan, T., Rao, J. N. S., Raghunathan, R. & Velmurugan, D. (2013). *Acta Cryst.* E**69**, o993.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

## 6-(4-Methoxyphenyl)-6a-nitro-6,6a,6b,7,8,9,10,12a-octahydrospiro-[chromeno[3,4-*a*]indolizine-12,3′-indolin]-2′-one

**Seenivasan Karthiga Devi, Thothadri Srinivasan, Jonnalagadda Naga Siva Rao, Raghavachary Raghunathan and Devadasan Velmurugan**

### S1. Comment

As part of our ongoing studies of 4H-chromene derivatives (Devi *et al.*, 2013), we have synthesized the title compound (Fig. 1) and report herein its crystal structure.

The hydropyrrolidine ring adopts an *envelope* conformation, the piperidine ring adopts a *chair* conformation and the pyran ring adopts an *envelope* conformation. The pyrrolidine ring (N2/C9/C10/C17/C22) makes a dihedral angle of 86.78 (8)° with the other pyrrolidine ring (N3/C22/C23/C28/C29) which shows that they are almost orthogonal to each other. The pyrrolidine ring makes a dihedral angle of 29.65 (8)° with the pyran ring (O2/C8-C11/C16), it makes a dihedral angle of 8.88 (9)° with the piperidine ring (N2/C17-C21).

The other pyrrolidine ring (N3/C22/C23/C28/C29) makes a dihedral angle of 71.63 (8)° with the pyran ring, it makes a dihedral angle of 86.84 (8)° with the piperidine ring which shows that they are almost at right angles to each other. The dihedral angle between the pyran ring and the piperidine ring is 30.34 (8)° . The oxygen atom O5 attached with pyrrolidine ring deviates by -0.0200 (1)Å. The nitrogroup attached with the pyrrolidine ring makes a diherdal angle of 88.83 (1)° which shows it is in orthogonal orientation . The crystal packing features N—H···O, C—H···O hydrogen bonds and intramolecular C—H···O hydrogen bonds.

### S2. Experimental

To a solution of isatin (1equiv) and piperidine-2-carboxylic acid (1.4 equiv) in dry toluene, was added 2-(4-methoxyphenyl)-3-nitro-2H-chromene (1equiv) under nitrogen atmosphere. The reaction mixture was refluxed for 24h in Dean-Stark apparatus to give the cycloadducts. After completion of the reaction as indicated by TLC, the solvent was evaporated under reduced pressure. The crude product was extracted with dichloromethane. The organic layer was dried with anhydrous sodium sulphate and concentrated in vacuo. Then the crude product was purified by column chromatography using hexane/EtOAc (7:3) as eluent. Colourless blocks were obtained by slow evaporation of a solution of the title compound in ethyl acetate at room temperature.

### S3. Refinement

The hydrogen atoms were placed in calculated positions and treated as riding atoms: C—H = 0.93 Å to 0.97 Å, and N—H =0.86 Å with Uiso(H) = 1.5Ueq(C) for methyl H atoms and = 1.2Ueq(C) for other H atoms.
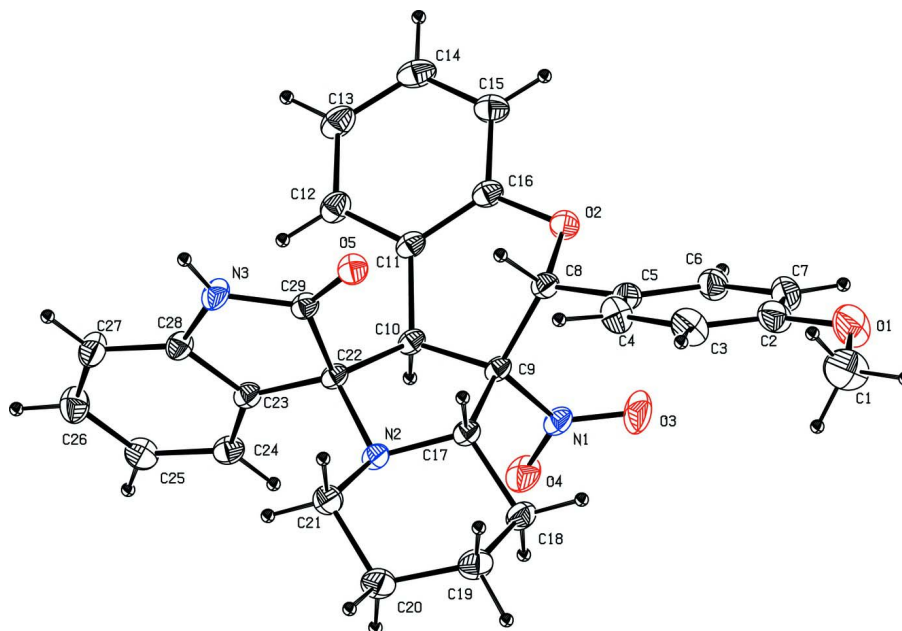
**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.
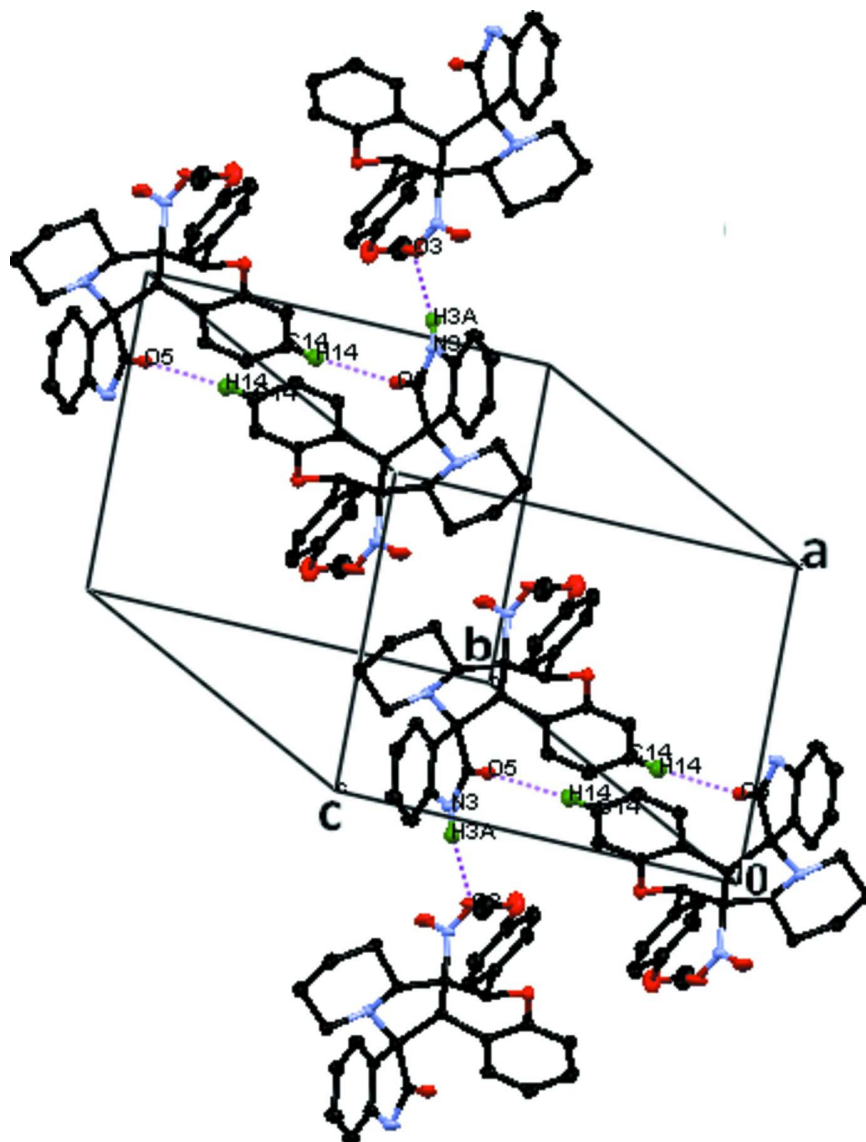
**Figure 2**

The crystal packing of the title compound viewed down *b* axis. H-atoms not involved in H-bonds have been excluded for clarity.

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*Crystal data*

| | |
|---|---|
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| $M_r = 497.54$ | $V = 1222.49\ (7)$ Å$^3$ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| Hall symbol: -P 1 | $F(000) = 524$ |
| $a = 9.3438\ (3)$ Å | $D_x = 1.352$ Mg m$^{-3}$ |
| $b = 11.3626\ (4)$ Å | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $c = 13.5713\ (4)$ Å | Cell parameters from 4964 reflections |
| $\alpha = 68.687\ (1)°$ | $\theta = 1.6–26.4°$ |
| $\beta = 88.284\ (1)°$ | $\mu = 0.09$ mm$^{-1}$ |

$T = 293$ K

Block, colourless

0.30 × 0.25 × 0.20 mm

*Data collection*

| | |
|---|---|
| Bruker SMART APEXII diffractometer | 17765 measured reflections |
| Radiation source: fine-focus sealed tube | 4964 independent reflections |
| Graphite monochromator | 4116 reflections with $I > 2\sigma(I)$ |
| $\omega$ and $\varphi$ scans | $R_{int} = 0.027$ |
| Absorption correction: multi-scan | $\theta_{max} = 26.4°$, $\theta_{min} = 1.6°$ |
| (*SADABS*; Bruker, 2008) | $h = -11 \rightarrow 11$ |
| $T_{min} = 0.973$, $T_{max} = 0.982$ | $k = -14 \rightarrow 14$ |
| | $l = -16 \rightarrow 16$ |

*Refinement*

Refinement on $F^2$

Least-squares matrix: full

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

$wR(F^2) = 0.119$

$S = 1.03$

4964 reflections

335 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

$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 0.279P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.22$ 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.

**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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.2341 (3) | 1.0337 (2) | 0.38130 (16) | 0.0993 (8) |
| H1A | 0.3106 | 1.0725 | 0.3690 | 0.149* |
| H1B | 0.1336 | 1.1041 | 0.3434 | 0.149* |
| H1C | 0.2654 | 0.9592 | 0.3564 | 0.149* |
| C2 | 0.3493 (2) | 0.87030 (17) | 0.55807 (14) | 0.0637 (5) |
| C3 | 0.4983 (2) | 0.82230 (18) | 0.53115 (14) | 0.0682 (5) |
| H3 | 0.5192 | 0.8662 | 0.4634 | 0.082* |
| C4 | 0.6175 (2) | 0.70767 (16) | 0.60584 (13) | 0.0592 (4) |
| H4 | 0.7191 | 0.6778 | 0.5883 | 0.071* |
| C5 | 0.58892 (17) | 0.63663 (15) | 0.70583 (11) | 0.0455 (3) |
| C6 | 0.43845 (19) | 0.68872 (17) | 0.73146 (13) | 0.0545 (4) |
| H6 | 0.4165 | 0.6446 | 0.7988 | 0.065* |
| C7 | 0.3206 (2) | 0.80484 (19) | 0.65897 (14) | 0.0650 (5) |
| H7 | 0.2207 | 0.8393 | 0.6783 | 0.078* |

| | | | |
|---|---|---|---|
| C8 | 0.72252 (16) | 0.51114 (14) | 0.78326 (10) | 0.0408 (3) |
| H8 | 0.8151 | 0.5325 | 0.7724 | 0.049* |
| C9 | 0.77152 (15) | 0.36977 (13) | 0.77426 (10) | 0.0367 (3) |
| C10 | 0.91910 (15) | 0.26057 (13) | 0.85468 (10) | 0.0366 (3) |
| H10 | 0.9014 | 0.1758 | 0.8865 | 0.044* |
| C11 | 0.94489 (16) | 0.29989 (14) | 0.94587 (10) | 0.0397 (3) |
| C12 | 1.07826 (18) | 0.21855 (17) | 1.02158 (11) | 0.0507 (4) |
| H12 | 1.1586 | 0.1446 | 1.0117 | 0.061* |
| C13 | 1.0931 (2) | 0.2457 (2) | 1.11079 (12) | 0.0600 (4) |
| H13 | 1.1843 | 0.1920 | 1.1595 | 0.072* |
| C14 | 0.9731 (2) | 0.35255 (19) | 1.12802 (12) | 0.0594 (4) |
| H14 | 0.9817 | 0.3685 | 1.1896 | 0.071* |
| C15 | 0.8405 (2) | 0.43555 (17) | 1.05410 (12) | 0.0536 (4) |
| H15 | 0.7598 | 0.5081 | 1.0652 | 0.064* |
| C16 | 0.82846 (17) | 0.40991 (15) | 0.96263 (11) | 0.0434 (3) |
| C17 | 0.81967 (15) | 0.37337 (14) | 0.66400 (10) | 0.0383 (3) |
| H17 | 0.8471 | 0.4528 | 0.6321 | 0.046* |
| C18 | 0.70480 (18) | 0.38308 (17) | 0.58150 (11) | 0.0499 (4) |
| H18A | 0.6086 | 0.4661 | 0.5662 | 0.060* |
| H18B | 0.6790 | 0.3032 | 0.6082 | 0.060* |
| C19 | 0.7827 (2) | 0.38777 (19) | 0.48083 (12) | 0.0598 (4) |
| H19A | 0.7147 | 0.3871 | 0.4286 | 0.072* |
| H19B | 0.7975 | 0.4731 | 0.4505 | 0.072* |
| C20 | 0.9400 (2) | 0.26524 (19) | 0.50478 (13) | 0.0596 (4) |
| H20A | 0.9914 | 0.2758 | 0.4409 | 0.071* |
| H20B | 0.9232 | 0.1811 | 0.5244 | 0.071* |
| C21 | 1.04685 (18) | 0.25141 (17) | 0.59464 (11) | 0.0491 (3) |
| H21A | 1.1419 | 0.1668 | 0.6125 | 0.059* |
| H21B | 1.0764 | 0.3292 | 0.5718 | 0.059* |
| C22 | 1.05240 (15) | 0.22993 (13) | 0.78451 (10) | 0.0366 (3) |
| C23 | 1.18780 (16) | 0.08993 (14) | 0.83232 (10) | 0.0392 (3) |
| C24 | 1.19077 (18) | −0.04125 (15) | 0.87141 (11) | 0.0470 (3) |
| H24 | 1.0985 | −0.0538 | 0.8694 | 0.056* |
| C25 | 1.3337 (2) | −0.15412 (16) | 0.91379 (13) | 0.0548 (4) |
| H25 | 1.3372 | −0.2432 | 0.9416 | 0.066* |
| C26 | 1.4710 (2) | −0.13609 (17) | 0.91531 (14) | 0.0615 (4) |
| H26 | 1.5660 | −0.2133 | 0.9431 | 0.074* |
| C27 | 1.46969 (18) | −0.00471 (17) | 0.87613 (14) | 0.0586 (4) |
| H27 | 1.5621 | 0.0077 | 0.8773 | 0.070* |
| C28 | 1.32679 (16) | 0.10650 (15) | 0.83550 (11) | 0.0438 (3) |
| C29 | 1.13735 (15) | 0.32878 (14) | 0.76355 (10) | 0.0389 (3) |
| N1 | 0.64091 (14) | 0.31789 (13) | 0.79590 (9) | 0.0446 (3) |
| N2 | 0.96439 (13) | 0.24825 (11) | 0.68832 (8) | 0.0386 (3) |
| N3 | 1.29296 (14) | 0.24880 (12) | 0.79328 (10) | 0.0497 (3) |
| H3A | 1.3632 | 0.2813 | 0.7871 | 0.060* |
| O1 | 0.22311 (19) | 0.98242 (14) | 0.49209 (11) | 0.0914 (5) |
| O2 | 0.69437 (12) | 0.49773 (11) | 0.89032 (8) | 0.0493 (3) |
| O3 | 0.50728 (14) | 0.39968 (14) | 0.78486 (14) | 0.0836 (4) |

| | | | | |
|---|---|---|---|---|
| O4 | 0.67564 (14) | 0.19579 (13) | 0.81793 (11) | 0.0701 (4) |
| O5 | 1.07592 (12) | 0.45448 (10) | 0.72573 (8) | 0.0485 (3) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.128 (2) | 0.0676 (13) | 0.0613 (12) | −0.0123 (13) | −0.0229 (13) | −0.0090 (10) |
| C2 | 0.0710 (11) | 0.0486 (9) | 0.0556 (10) | −0.0075 (8) | −0.0074 (8) | −0.0206 (7) |
| C3 | 0.0848 (13) | 0.0505 (9) | 0.0519 (9) | −0.0195 (9) | 0.0094 (9) | −0.0099 (7) |
| C4 | 0.0602 (10) | 0.0478 (8) | 0.0600 (10) | −0.0199 (7) | 0.0149 (8) | −0.0135 (7) |
| C5 | 0.0483 (8) | 0.0446 (7) | 0.0462 (8) | −0.0193 (6) | 0.0045 (6) | −0.0199 (6) |
| C6 | 0.0531 (9) | 0.0602 (9) | 0.0457 (8) | −0.0154 (7) | 0.0068 (7) | −0.0241 (7) |
| C7 | 0.0541 (10) | 0.0673 (11) | 0.0596 (10) | −0.0054 (8) | 0.0015 (8) | −0.0304 (8) |
| C8 | 0.0400 (7) | 0.0477 (7) | 0.0402 (7) | −0.0220 (6) | 0.0063 (6) | −0.0187 (6) |
| C9 | 0.0339 (6) | 0.0453 (7) | 0.0378 (7) | −0.0239 (6) | 0.0051 (5) | −0.0151 (5) |
| C10 | 0.0362 (7) | 0.0427 (7) | 0.0359 (6) | −0.0236 (6) | 0.0053 (5) | −0.0125 (5) |
| C11 | 0.0420 (7) | 0.0513 (8) | 0.0331 (6) | −0.0291 (6) | 0.0071 (5) | −0.0134 (6) |
| C12 | 0.0474 (8) | 0.0644 (9) | 0.0392 (7) | −0.0252 (7) | 0.0042 (6) | −0.0161 (7) |
| C13 | 0.0591 (10) | 0.0822 (12) | 0.0394 (8) | −0.0333 (9) | −0.0015 (7) | −0.0187 (8) |
| C14 | 0.0792 (12) | 0.0772 (11) | 0.0373 (8) | −0.0457 (10) | 0.0069 (8) | −0.0236 (7) |
| C15 | 0.0669 (10) | 0.0598 (9) | 0.0446 (8) | −0.0330 (8) | 0.0120 (7) | −0.0241 (7) |
| C16 | 0.0484 (8) | 0.0513 (8) | 0.0378 (7) | −0.0288 (7) | 0.0068 (6) | −0.0158 (6) |
| C17 | 0.0380 (7) | 0.0444 (7) | 0.0362 (6) | −0.0213 (6) | 0.0051 (5) | −0.0146 (5) |
| C18 | 0.0468 (8) | 0.0604 (9) | 0.0435 (8) | −0.0216 (7) | −0.0022 (6) | −0.0209 (7) |
| C19 | 0.0664 (11) | 0.0727 (11) | 0.0398 (8) | −0.0267 (9) | 0.0001 (7) | −0.0228 (7) |
| C20 | 0.0666 (11) | 0.0744 (11) | 0.0442 (8) | −0.0288 (9) | 0.0105 (7) | −0.0301 (8) |
| C21 | 0.0486 (8) | 0.0600 (9) | 0.0450 (8) | −0.0253 (7) | 0.0138 (6) | −0.0243 (7) |
| C22 | 0.0360 (7) | 0.0426 (7) | 0.0376 (6) | −0.0234 (6) | 0.0056 (5) | −0.0144 (5) |
| C23 | 0.0392 (7) | 0.0450 (7) | 0.0379 (7) | −0.0217 (6) | 0.0059 (5) | −0.0160 (5) |
| C24 | 0.0510 (8) | 0.0494 (8) | 0.0487 (8) | −0.0293 (7) | 0.0091 (6) | −0.0182 (6) |
| C25 | 0.0640 (10) | 0.0435 (8) | 0.0549 (9) | −0.0243 (7) | 0.0090 (7) | −0.0145 (7) |
| C26 | 0.0498 (9) | 0.0505 (9) | 0.0681 (11) | −0.0124 (7) | 0.0006 (8) | −0.0138 (8) |
| C27 | 0.0394 (8) | 0.0575 (9) | 0.0737 (11) | −0.0205 (7) | 0.0006 (7) | −0.0184 (8) |
| C28 | 0.0390 (7) | 0.0470 (8) | 0.0484 (8) | −0.0220 (6) | 0.0055 (6) | −0.0169 (6) |
| C29 | 0.0387 (7) | 0.0455 (7) | 0.0396 (7) | −0.0244 (6) | 0.0089 (5) | −0.0164 (6) |
| N1 | 0.0391 (6) | 0.0572 (7) | 0.0465 (6) | −0.0290 (6) | 0.0070 (5) | −0.0194 (5) |
| N2 | 0.0374 (6) | 0.0472 (6) | 0.0363 (6) | −0.0217 (5) | 0.0050 (5) | −0.0167 (5) |
| N3 | 0.0365 (6) | 0.0502 (7) | 0.0676 (8) | −0.0271 (5) | 0.0056 (6) | −0.0181 (6) |
| O1 | 0.0928 (11) | 0.0680 (8) | 0.0653 (8) | 0.0075 (7) | −0.0170 (7) | −0.0163 (7) |
| O2 | 0.0470 (6) | 0.0582 (6) | 0.0428 (5) | −0.0181 (5) | 0.0061 (4) | −0.0235 (5) |
| O3 | 0.0361 (6) | 0.0715 (8) | 0.1459 (13) | −0.0282 (6) | 0.0131 (7) | −0.0380 (8) |
| O4 | 0.0591 (7) | 0.0610 (7) | 0.1013 (10) | −0.0415 (6) | 0.0089 (7) | −0.0243 (7) |
| O5 | 0.0477 (6) | 0.0437 (6) | 0.0584 (6) | −0.0260 (5) | 0.0113 (5) | −0.0166 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O1 | 1.418 (3) | C16—O2 | 1.3797 (17) |
| C1—H1A | 0.9600 | C17—N2 | 1.4584 (17) |

| C1—H1B | 0.9600 | C17—C18 | 1.5187 (18) |
| C1—H1C | 0.9600 | C17—H17 | 0.9800 |
| C2—O1 | 1.365 (2) | C18—C19 | 1.522 (2) |
| C2—C3 | 1.373 (3) | C18—H18A | 0.9700 |
| C2—C7 | 1.377 (3) | C18—H18B | 0.9700 |
| C3—C4 | 1.388 (2) | C19—C20 | 1.513 (2) |
| C3—H3 | 0.9300 | C19—H19A | 0.9700 |
| C4—C5 | 1.384 (2) | C19—H19B | 0.9700 |
| C4—H4 | 0.9300 | C20—C21 | 1.523 (2) |
| C5—C6 | 1.383 (2) | C20—H20A | 0.9700 |
| C5—C8 | 1.5101 (19) | C20—H20B | 0.9700 |
| C6—C7 | 1.377 (2) | C21—N2 | 1.4634 (18) |
| C6—H6 | 0.9300 | C21—H21A | 0.9700 |
| C7—H7 | 0.9300 | C21—H21B | 0.9700 |
| C8—O2 | 1.4321 (16) | C22—N2 | 1.4681 (16) |
| C8—C9 | 1.5374 (19) | C22—C23 | 1.5041 (19) |
| C8—H8 | 0.9800 | C22—C29 | 1.5590 (17) |
| C9—N1 | 1.5309 (16) | C23—C24 | 1.3774 (19) |
| C9—C17 | 1.5404 (18) | C23—C28 | 1.3879 (19) |
| C9—C10 | 1.5452 (17) | C24—C25 | 1.385 (2) |
| C10—C11 | 1.5146 (18) | C24—H24 | 0.9300 |
| C10—C22 | 1.5516 (18) | C25—C26 | 1.378 (2) |
| C10—H10 | 0.9800 | C25—H25 | 0.9300 |
| C11—C16 | 1.384 (2) | C26—C27 | 1.386 (2) |
| C11—C12 | 1.3929 (19) | C26—H26 | 0.9300 |
| C12—C13 | 1.377 (2) | C27—C28 | 1.375 (2) |
| C12—H12 | 0.9300 | C27—H27 | 0.9300 |
| C13—C14 | 1.379 (3) | C28—N3 | 1.4060 (18) |
| C13—H13 | 0.9300 | C29—O5 | 1.2126 (16) |
| C14—C15 | 1.376 (2) | C29—N3 | 1.3522 (18) |
| C14—H14 | 0.9300 | N1—O3 | 1.1997 (17) |
| C15—C16 | 1.391 (2) | N1—O4 | 1.2118 (16) |
| C15—H15 | 0.9300 | N3—H3A | 0.8600 |
| | | | |
| O1—C1—H1A | 109.5 | C18—C17—H17 | 107.3 |
| O1—C1—H1B | 109.5 | C9—C17—H17 | 107.3 |
| H1A—C1—H1B | 109.5 | C17—C18—C19 | 107.80 (12) |
| O1—C1—H1C | 109.5 | C17—C18—H18A | 110.1 |
| H1A—C1—H1C | 109.5 | C19—C18—H18A | 110.1 |
| H1B—C1—H1C | 109.5 | C17—C18—H18B | 110.1 |
| O1—C2—C3 | 124.88 (18) | C19—C18—H18B | 110.1 |
| O1—C2—C7 | 115.56 (17) | H18A—C18—H18B | 108.5 |
| C3—C2—C7 | 119.55 (16) | C20—C19—C18 | 111.05 (13) |
| C2—C3—C4 | 119.46 (17) | C20—C19—H19A | 109.4 |
| C2—C3—H3 | 120.3 | C18—C19—H19A | 109.4 |
| C4—C3—H3 | 120.3 | C20—C19—H19B | 109.4 |
| C5—C4—C3 | 121.68 (16) | C18—C19—H19B | 109.4 |
| C5—C4—H4 | 119.2 | H19A—C19—H19B | 108.0 |

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| C3—C4—H4 | 119.2 | C19—C20—C21 | 112.21 (13) |
| C6—C5—C4 | 117.54 (14) | C19—C20—H20A | 109.2 |
| C6—C5—C8 | 122.97 (13) | C21—C20—H20A | 109.2 |
| C4—C5—C8 | 119.43 (14) | C19—C20—H20B | 109.2 |
| C7—C6—C5 | 121.09 (16) | C21—C20—H20B | 109.2 |
| C7—C6—H6 | 119.5 | H20A—C20—H20B | 107.9 |
| C5—C6—H6 | 119.5 | N2—C21—C20 | 109.45 (12) |
| C2—C7—C6 | 120.55 (17) | N2—C21—H21A | 109.8 |
| C2—C7—H7 | 119.7 | C20—C21—H21A | 109.8 |
| C6—C7—H7 | 119.7 | N2—C21—H21B | 109.8 |
| O2—C8—C5 | 109.55 (11) | C20—C21—H21B | 109.8 |
| O2—C8—C9 | 109.49 (11) | H21A—C21—H21B | 108.2 |
| C5—C8—C9 | 119.53 (11) | N2—C22—C23 | 112.92 (11) |
| O2—C8—H8 | 105.8 | N2—C22—C10 | 100.87 (10) |
| C5—C8—H8 | 105.8 | C23—C22—C10 | 115.71 (10) |
| C9—C8—H8 | 105.8 | N2—C22—C29 | 113.85 (10) |
| N1—C9—C8 | 112.02 (11) | C23—C22—C29 | 101.58 (10) |
| N1—C9—C17 | 108.63 (10) | C10—C22—C29 | 112.49 (10) |
| C8—C9—C17 | 112.21 (11) | C24—C23—C28 | 119.79 (13) |
| N1—C9—C10 | 108.43 (10) | C24—C23—C22 | 130.71 (12) |
| C8—C9—C10 | 110.21 (10) | C28—C23—C22 | 109.49 (11) |
| C17—C9—C10 | 105.04 (10) | C23—C24—C25 | 118.73 (14) |
| C11—C10—C9 | 113.46 (11) | C23—C24—H24 | 120.6 |
| C11—C10—C22 | 118.45 (10) | C25—C24—H24 | 120.6 |
| C9—C10—C22 | 104.58 (10) | C26—C25—C24 | 120.80 (14) |
| C11—C10—H10 | 106.5 | C26—C25—H25 | 119.6 |
| C9—C10—H10 | 106.5 | C24—C25—H25 | 119.6 |
| C22—C10—H10 | 106.5 | C25—C26—C27 | 121.06 (15) |
| C16—C11—C12 | 117.84 (13) | C25—C26—H26 | 119.5 |
| C16—C11—C10 | 120.54 (12) | C27—C26—H26 | 119.5 |
| C12—C11—C10 | 121.28 (13) | C28—C27—C26 | 117.54 (15) |
| C13—C12—C11 | 121.10 (15) | C28—C27—H27 | 121.2 |
| C13—C12—H12 | 119.5 | C26—C27—H27 | 121.2 |
| C11—C12—H12 | 119.5 | C27—C28—C23 | 122.06 (13) |
| C12—C13—C14 | 120.11 (15) | C27—C28—N3 | 128.86 (13) |
| C12—C13—H13 | 119.9 | C23—C28—N3 | 109.08 (12) |
| C14—C13—H13 | 119.9 | O5—C29—N3 | 126.03 (12) |
| C15—C14—C13 | 120.08 (14) | O5—C29—C22 | 126.45 (12) |
| C15—C14—H14 | 120.0 | N3—C29—C22 | 107.51 (11) |
| C13—C14—H14 | 120.0 | O3—N1—O4 | 122.10 (12) |
| C14—C15—C16 | 119.42 (15) | O3—N1—C9 | 119.37 (12) |
| C14—C15—H15 | 120.3 | O4—N1—C9 | 118.39 (12) |
| C16—C15—H15 | 120.3 | C17—N2—C21 | 111.30 (11) |
| O2—C16—C11 | 121.60 (12) | C17—N2—C22 | 107.05 (10) |
| O2—C16—C15 | 117.02 (13) | C21—N2—C22 | 116.93 (10) |
| C11—C16—C15 | 121.37 (14) | C29—N3—C28 | 112.30 (11) |
| N2—C17—C18 | 110.64 (11) | C29—N3—H3A | 123.8 |
| N2—C17—C9 | 104.11 (10) | C28—N3—H3A | 123.8 |

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| C18—C17—C9 | 119.60 (11) | C2—O1—C1 | 118.13 (17) |
| N2—C17—H17 | 107.3 | C16—O2—C8 | 112.06 (10) |
| | | | |
| O1—C2—C3—C4 | 179.39 (17) | C9—C10—C22—C23 | −154.69 (10) |
| C7—C2—C3—C4 | 0.6 (3) | C11—C10—C22—C29 | −38.33 (15) |
| C2—C3—C4—C5 | 2.6 (3) | C9—C10—C22—C29 | 89.17 (12) |
| C3—C4—C5—C6 | −3.7 (3) | N2—C22—C23—C24 | −59.95 (18) |
| C3—C4—C5—C8 | 178.97 (15) | C10—C22—C23—C24 | 55.57 (19) |
| C4—C5—C6—C7 | 1.7 (2) | C29—C22—C23—C24 | 177.72 (14) |
| C8—C5—C6—C7 | 178.92 (15) | N2—C22—C23—C28 | 121.06 (12) |
| O1—C2—C7—C6 | 178.54 (17) | C10—C22—C23—C28 | −123.42 (12) |
| C3—C2—C7—C6 | −2.5 (3) | C29—C22—C23—C28 | −1.27 (14) |
| C5—C6—C7—C2 | 1.4 (3) | C28—C23—C24—C25 | 0.4 (2) |
| C6—C5—C8—O2 | −26.75 (18) | C22—C23—C24—C25 | −178.55 (13) |
| C4—C5—C8—O2 | 150.40 (14) | C23—C24—C25—C26 | −1.1 (2) |
| C6—C5—C8—C9 | 100.70 (17) | C24—C25—C26—C27 | 1.0 (3) |
| C4—C5—C8—C9 | −82.15 (18) | C25—C26—C27—C28 | −0.2 (3) |
| O2—C8—C9—N1 | 63.21 (13) | C26—C27—C28—C23 | −0.5 (2) |
| C5—C8—C9—N1 | −64.26 (16) | C26—C27—C28—N3 | 178.64 (15) |
| O2—C8—C9—C17 | −174.28 (10) | C24—C23—C28—C27 | 0.5 (2) |
| C5—C8—C9—C17 | 58.25 (16) | C22—C23—C28—C27 | 179.58 (14) |
| O2—C8—C9—C10 | −57.60 (13) | C24—C23—C28—N3 | −178.85 (12) |
| C5—C8—C9—C10 | 174.92 (11) | C22—C23—C28—N3 | 0.27 (16) |
| N1—C9—C10—C11 | −102.41 (12) | N2—C22—C29—O5 | 58.98 (18) |
| C8—C9—C10—C11 | 20.53 (14) | C23—C22—C29—O5 | −179.33 (13) |
| C17—C9—C10—C11 | 141.59 (11) | C10—C22—C29—O5 | −54.99 (17) |
| N1—C9—C10—C22 | 127.09 (10) | N2—C22—C29—N3 | −119.81 (12) |
| C8—C9—C10—C22 | −109.97 (11) | C23—C22—C29—N3 | 1.87 (13) |
| C17—C9—C10—C22 | 11.09 (12) | C10—C22—C29—N3 | 126.22 (12) |
| C9—C10—C11—C16 | 11.56 (17) | C8—C9—N1—O3 | 22.46 (18) |
| C22—C10—C11—C16 | 134.74 (13) | C17—C9—N1—O3 | −102.05 (16) |
| C9—C10—C11—C12 | −175.29 (12) | C10—C9—N1—O3 | 144.30 (14) |
| C22—C10—C11—C12 | −52.11 (17) | C8—C9—N1—O4 | −161.64 (12) |
| C16—C11—C12—C13 | 0.7 (2) | C17—C9—N1—O4 | 73.85 (15) |
| C10—C11—C12—C13 | −172.64 (14) | C10—C9—N1—O4 | −39.80 (16) |
| C11—C12—C13—C14 | 1.8 (3) | C18—C17—N2—C21 | 63.88 (14) |
| C12—C13—C14—C15 | −2.4 (3) | C9—C17—N2—C21 | −166.40 (10) |
| C13—C14—C15—C16 | 0.5 (2) | C18—C17—N2—C22 | −167.21 (11) |
| C12—C11—C16—O2 | 178.36 (12) | C9—C17—N2—C22 | −37.49 (12) |
| C10—C11—C16—O2 | −8.27 (19) | C20—C21—N2—C17 | −58.79 (15) |
| C12—C11—C16—C15 | −2.6 (2) | C20—C21—N2—C22 | 177.76 (12) |
| C10—C11—C16—C15 | 170.76 (12) | C23—C22—N2—C17 | 168.07 (10) |
| C14—C15—C16—O2 | −178.89 (13) | C10—C22—N2—C17 | 43.95 (12) |
| C14—C15—C16—C11 | 2.0 (2) | C29—C22—N2—C17 | −76.77 (13) |
| N1—C9—C17—N2 | −101.06 (11) | C23—C22—N2—C21 | −66.33 (15) |
| C8—C9—C17—N2 | 134.53 (11) | C10—C22—N2—C21 | 169.55 (11) |
| C10—C9—C17—N2 | 14.79 (12) | C29—C22—N2—C21 | 48.83 (16) |
| N1—C9—C17—C18 | 23.05 (16) | O5—C29—N3—C28 | 179.32 (13) |

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| C8—C9—C17—C18 | −101.35 (14) | C22—C29—N3—C28 | −1.88 (16) |
| C10—C9—C17—C18 | 138.91 (12) | C27—C28—N3—C29 | −178.18 (15) |
| N2—C17—C18—C19 | −60.27 (15) | C23—C28—N3—C29 | 1.07 (17) |
| C9—C17—C18—C19 | 178.82 (12) | C3—C2—O1—C1 | 16.0 (3) |
| C17—C18—C19—C20 | 54.91 (18) | C7—C2—O1—C1 | −165.1 (2) |
| C18—C19—C20—C21 | −53.21 (19) | C11—C16—O2—C8 | −30.37 (17) |
| C19—C20—C21—N2 | 53.69 (18) | C15—C16—O2—C8 | 150.56 (13) |
| C11—C10—C22—N2 | −160.01 (11) | C5—C8—O2—C16 | −163.66 (11) |
| C9—C10—C22—N2 | −32.51 (12) | C9—C8—O2—C16 | 63.46 (14) |
| C11—C10—C22—C23 | 77.81 (14) | | |

*Hydrogen-bond geometry (Å, °)*

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N3—H3$A$⋯O3[i] | 0.86 | 2.24 | 3.085 (2) | 167 |
| C14—H14⋯O5[ii] | 0.93 | 2.54 | 3.358 (2) | 147 |
| C8—H8⋯O5 | 0.98 | 2.40 | 3.234 (2) | 143 |

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