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# Methyl 3′-benzyl-4′-(2,4-dichlorophenyl)-1′-methyl-2-oxo-1-propylspiro[indoline-3,2′-pyrrolidine]-3′-carboxylate

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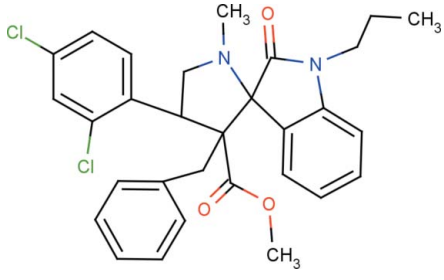
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.051; $wR$ factor = 0.167; data-to-parameter ratio = 23.1.

In the title compound, $C_{30}H_{30}Cl_2N_2O_3$, the indole ring system is roughly planar, with a maximum deviation of 0.1039 (18) Å for the carbonyl C atom, and makes a dihedral angle of 86.61 (9)° with the mean plane of the pyrrolidine ring. This spiro pyrrolidine ring adopts an envelope conformation with the N atom at the flap position. The pyrrole ring of the indole ring system adopts a twisted conformation on the C—C(=O) bond. The molecular structure is stabilized by an intramolecular C—H···O hydrogen bond, which generates an $S$(6) ring motif, and a $\pi$–$\pi$ interaction [centroid–centroid distance = 3.6577 (12) Å] involving the 2,4-dichlorophenyl ring and the benzyl ring. In the crystal, molecules are linked *via* C—H···O hydrogen bonds, forming $C$(9) chains running parallel to [10$\bar{1}$].

## Related literature

For the biological activity of spiro-pyrrolidine and oxindole derivatives, see: Peddi *et al.* (2004); Rajeswaran *et al.* (1999). For a related crystal structure, see: Jagadeesan *et al.* (2013). For graph-set motif notation, see: Bernstein *et al.* (1995). For ring puckering analysis, see: Cremer & Pople (1975). For bond-length distortions in small rings, see: Allen (1981).




## Experimental

### Crystal data

$C_{30}H_{30}Cl_2N_2O_3$
$M_r$ = 537.46
Monoclinic, $P2_1/n$
$a$ = 7.5563 (4) Å
$b$ = 28.8497 (16) Å
$c$ = 12.4274 (8) Å
$\beta$ = 90.433 (3)°
$V$ = 2709.1 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.27 mm$^{-1}$
$T$ = 293 K
0.35 × 0.30 × 0.25 mm

### Data collection

Bruker Kappa APEXII CCD diffractometer
31802 measured reflections
7794 independent reflections
4985 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.167
$S$ = 1.00
7794 reflections
337 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.58 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C18—H18$B$···O1 | 0.97 | 2.39 | 3.073 (2) | 127 |
| C13—H13···O2$^{i}$ | 0.93 | 2.46 | 3.132 (2) | 129 |

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

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) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

SK and KS thank Dr Babu Varghese, SAIF, IIT, Chennai, India, for the data collection.

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2694).

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# supporting information

*Acta Cryst.* (2014). E**70**, o272 [doi:10.1107/S1600536814002621]

## Methyl 3′-benzyl-4′-(2,4-dichlorophenyl)-1′-methyl-2-oxo-1-propylspiro-[indoline-3,2′-pyrrolidine]-3′-carboxylate

**S. Karthikeyan, K. Sethusankar, Anthonisamy Devaraj and Manickam Bakthadoss**

### S1. Comment

Spiro-pyrrolidine derivatives are unique tetracyclic 5-HT(2 A) receptor antagonists (Peddi *et al.*, 2004). Oxindole derivatives help to treat and prevent diabetic complications arising from elevated levels of sorbitol and act as aldose reductase inhibitor (Rajeswaran *et al.*, 1999).

The molecular structure of the title compound is illustrated in Fig 1. In the molecule, there is C-H···O hydrogen bond, forming an *S*(6) ring motif (Bernstein *et al.*, 1995), and a $\pi$–$\pi$ interaction present [*Cg*(1)···*Cg*(2) = 3.6577 (12) Å, where Cg1 and Cg2 are the centroids of rings C1-C6 and C19-C24, respectively]. The indole ring system is essentially planar with a maximum deviation of 0.1039 (18) Å for atom C10. The mean plane of the indole ring system forms a dihedral angle of 86.61 (9) ° with the mean plane of the pyrrolidine five membered ring. The latter forms a dihedral angle of 80.80 (11) ° with the benzyl ring which shows that they are almost orthogonal. Atom O1 deviates from the mean plane of the indole ring system by 0.2776 (15) Å. The molecular dimensions in the title compound are in excellent agreement with the corresponding dimensions reported in another closely related compound (Jagadeesan *et al.*, 2013).

The spiro-pyrrolidine ring adopts an envelope conformation with atom N1 at the flap position. The distance to the flap position from the mean plane of the spiro carbon is 0.2524 (17) Å; the puckering parameters (Cremer & Pople, 1975) of the ring are $Q_2$ = 0.4033 (18) Å and $\varphi_2$ = 8.5 (3)°. The pyrrole ring system adopts a twisted conformation on bond C9-C10 with the deviation of -0.0559 (18) Å. The central spiro-pyrrolidine ring is perpendicular to the dichloro phenyl ring with a dihedral angle of 87.33 (10)°. The carbonyl group and the benzyl ring have a (+)anti-clinal conformation with torsion angle C18—C17—C25—O2 = 148.57 (17)°.

In the benzene ring (C11—C16) of the indole ring system, the expansion of the ipso angles at C11, C13 and C14 [121.37 (16), 121.56 (18) and 120.37 (18)°, respectively] and contraction of the apical angles at C12, C15 and C16 [117.81 (18), 118.82 (18) and 119.97 (18)°, respectively] are caused by the fusion of the smaller pyrrole ring to the six-membered benzene ring and the strain is taken up by the angular distortion rather than by bond-length distortions (Allen, 1981). The carboxyl group and oxindole ring system are (-)*syn*-clinal to each other with the torsion angle C9—C17—C25—O2 = -89.82 (19)°.

The crystal packing (Fig. 2 and Table 1) is stabilized by a C-H···O hydrogen bond resulting in the formation of *C*(9) chains running parallel to [1 0 -1].

### S2. Experimental

A mixture of (*E*)-methyl 2-benzyl-3-(2,4-dichlorophenyl)acrylate (2 mmol), *N*-propyl isatin (2 mmol) and sarcosine (2 mmol) in acetonitrile (8 ml) was refluxed for 12 h. After the completion of the reaction as indicated by TLC, the reaction mixture was concentrated. The resulting crude mass was diluted with water (10 ml) and extracted with ethyl acetate (3 × 10 ml). The combined organic layers were washed with brine (2 × 10 ml) and dried over anhydrous $Na_2SO_4$. The organic

layer was concentrated and the residue purified by column chromatography on silica gel (Acme 100–200 mesh), using ethyl acetate:hexanes (2:8) to afford the title compound as a colourless solid in (62%) yield. Block-like colourless crystals were obtained by slow evaporation of a solution in $CHCl_3$.

## S3. Refinement

The H atoms could all be localed in difference electron-density maps. In the final cycles of refinement they were treated as riding atoms and their distances were geometrically constrained: C-H = 0.93 and 0.96 Å for CH and $CH_3$ H atoms, respectively, with $U_{iso}$(H) = 1.5 $U_{eq}$(C-methyl) and = 1.2$U_{eq}$(C) for other H atoms.

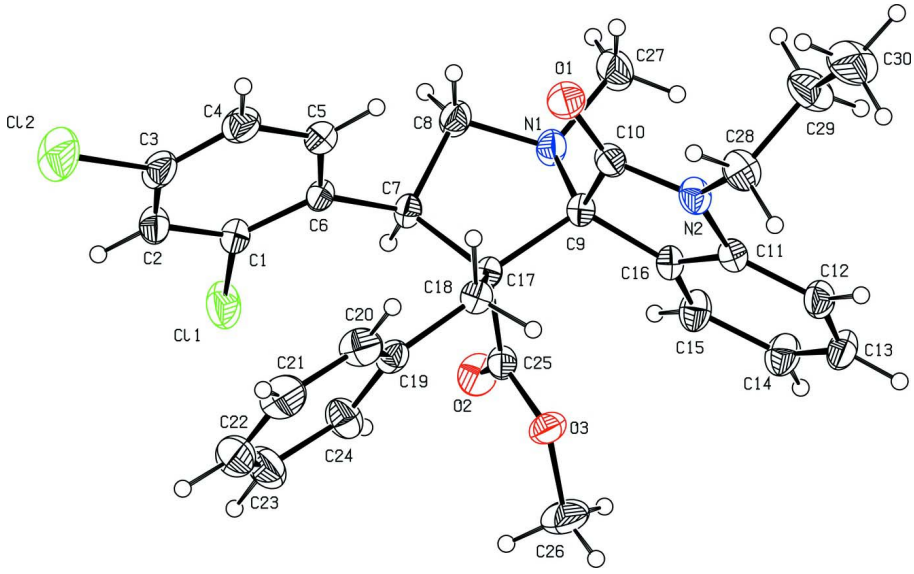


**Figure 1**

The molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at 30% probability level.

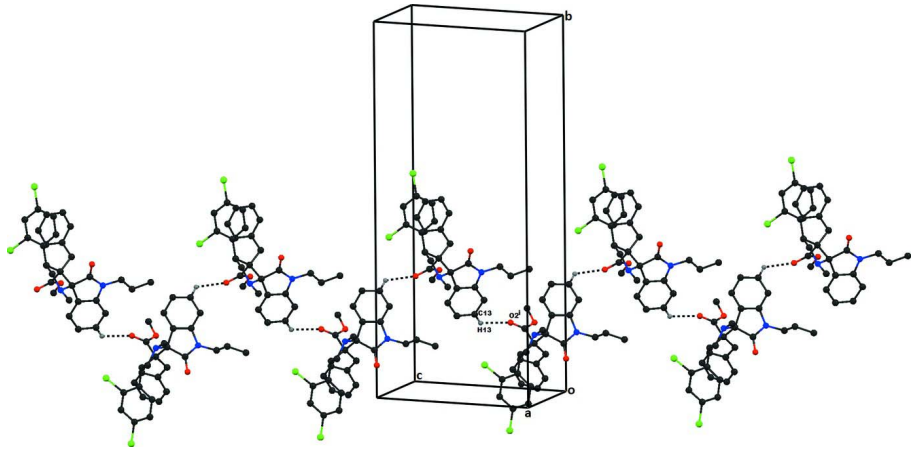


**Figure 2**

The crystal packing of the title compound viewed along the a axis, showing the formation of infinite *C*(9) chains. Dashed lines indicate C—H···O hydrogen bonds - see Table 1 for details.

## Methyl 3′-benzyl-4′-(2,4-dichlorophenyl)-1′-methyl-2-oxo-1-propylspiro[indoline-3,2′-pyrrolidine]-3′-carboxylate

### *Crystal data*

$C_{30}H_{30}Cl_2N_2O_3$
$M_r$ = 537.46
Monoclinic, $P2_1/n$
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$a$ = 7.5563 (4) Å
$b$ = 28.8497 (16) Å
$c$ = 12.4274 (8) Å
$\beta$ = 90.433 (3)°
$V$ = 2709.1 (3) Å$^3$
$Z$ = 4
$F(000)$ = 1128
$D_x$ = 1.318 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 7794 reflections
$\theta$ = 2.2–29.9°
$\mu$ = 0.27 mm$^{-1}$
$T$ = 293 K
Block, colourless
0.35 × 0.30 × 0.25 mm

### *Data collection*

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
31802 measured reflections
7794 independent reflections
4985 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029
$\theta_{max}$ = 29.9°, $\theta_{min}$ = 2.2°
$h$ = −10→7
$k$ = −37→40
$l$ = −17→16

### *Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.167
$S$ = 1.00
7794 reflections
337 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
$w = 1/[\sigma^2(F_o^2) + (0.0918P)^2 + 0.3965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.58 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.3859 (2) | 0.04008 (6) | 0.32495 (15) | 0.0424 (4) |
| C2 | 0.3770 (3) | −0.00713 (7) | 0.34519 (18) | 0.0512 (5) |
| H2 | 0.3748 | −0.0183 | 0.4153 | 0.061* |
| C3 | 0.3714 (2) | −0.03682 (7) | 0.25925 (19) | 0.0520 (5) |

| | | | | |
|---|---|---|---|---|
| C4 | 0.3773 (3) | −0.02096 (7) | 0.15521 (18) | 0.0509 (5) |
| H4 | 0.3762 | −0.0416 | 0.0977 | 0.061* |
| C5 | 0.3851 (2) | 0.02637 (6) | 0.13764 (16) | 0.0441 (4) |
| H5 | 0.3897 | 0.0373 | 0.0673 | 0.053* |
| C6 | 0.3861 (2) | 0.05816 (6) | 0.22188 (14) | 0.0361 (4) |
| C7 | 0.3906 (2) | 0.10974 (6) | 0.20185 (14) | 0.0349 (3) |
| H7 | 0.4141 | 0.1247 | 0.2713 | 0.042* |
| C8 | 0.5393 (2) | 0.12406 (6) | 0.12614 (17) | 0.0456 (4) |
| H8A | 0.6510 | 0.1268 | 0.1645 | 0.055* |
| H8B | 0.5524 | 0.1019 | 0.0680 | 0.055* |
| C9 | 0.2936 (2) | 0.16388 (5) | 0.05789 (13) | 0.0330 (3) |
| C10 | 0.2552 (2) | 0.13941 (6) | −0.05080 (14) | 0.0385 (4) |
| C11 | 0.1097 (2) | 0.20864 (6) | −0.05612 (13) | 0.0358 (4) |
| C12 | 0.0117 (2) | 0.24610 (7) | −0.09082 (16) | 0.0455 (4) |
| H12 | −0.0537 | 0.2448 | −0.1545 | 0.055* |
| C13 | 0.0137 (3) | 0.28541 (7) | −0.02830 (18) | 0.0519 (5) |
| H13 | −0.0537 | 0.3109 | −0.0494 | 0.062* |
| C14 | 0.1123 (3) | 0.28797 (7) | 0.06395 (18) | 0.0530 (5) |
| H14 | 0.1141 | 0.3153 | 0.1036 | 0.064* |
| C15 | 0.2100 (3) | 0.24996 (6) | 0.09897 (16) | 0.0454 (4) |
| H15 | 0.2778 | 0.2516 | 0.1616 | 0.054* |
| C16 | 0.2047 (2) | 0.20999 (5) | 0.03956 (13) | 0.0342 (3) |
| C17 | 0.21755 (19) | 0.13252 (5) | 0.15373 (12) | 0.0298 (3) |
| C18 | 0.0724 (2) | 0.09876 (6) | 0.11544 (14) | 0.0358 (4) |
| H18A | −0.0209 | 0.1169 | 0.0820 | 0.043* |
| H18B | 0.1227 | 0.0793 | 0.0598 | 0.043* |
| C19 | −0.0128 (2) | 0.06708 (6) | 0.19792 (15) | 0.0387 (4) |
| C20 | −0.0855 (3) | 0.02613 (7) | 0.1602 (2) | 0.0550 (5) |
| H20 | −0.0829 | 0.0197 | 0.0869 | 0.066* |
| C21 | −0.1623 (3) | −0.00549 (8) | 0.2298 (3) | 0.0697 (7) |
| H21 | −0.2107 | −0.0328 | 0.2028 | 0.084* |
| C22 | −0.1672 (3) | 0.00321 (9) | 0.3375 (2) | 0.0694 (7) |
| H22 | −0.2169 | −0.0183 | 0.3842 | 0.083* |
| C23 | −0.0990 (3) | 0.04354 (9) | 0.3761 (2) | 0.0653 (6) |
| H23 | −0.1033 | 0.0498 | 0.4494 | 0.078* |
| C24 | −0.0228 (3) | 0.07536 (7) | 0.30671 (17) | 0.0517 (5) |
| H24 | 0.0225 | 0.1029 | 0.3344 | 0.062* |
| C25 | 0.1402 (2) | 0.16665 (6) | 0.23502 (13) | 0.0357 (4) |
| C26 | −0.1178 (4) | 0.20778 (9) | 0.2834 (2) | 0.0752 (7) |
| H26A | −0.1124 | 0.1962 | 0.3558 | 0.113* |
| H26B | −0.2392 | 0.2110 | 0.2614 | 0.113* |
| H26C | −0.0605 | 0.2374 | 0.2802 | 0.113* |
| C27 | 0.5916 (3) | 0.18816 (9) | 0.0023 (2) | 0.0626 (6) |
| H27A | 0.7088 | 0.1930 | 0.0305 | 0.094* |
| H27B | 0.5430 | 0.2172 | −0.0214 | 0.094* |
| H27C | 0.5966 | 0.1671 | −0.0574 | 0.094* |
| C28 | 0.0617 (3) | 0.15200 (8) | −0.21054 (15) | 0.0505 (5) |
| H28A | −0.0588 | 0.1634 | −0.2150 | 0.061* |

| | | | | |
|---|---|---|---|---|
| H28B | 0.0571 | 0.1184 | −0.2136 | 0.061* |
| C29 | 0.1629 (3) | 0.16950 (10) | −0.30642 (18) | 0.0690 (7) |
| H29A | 0.1616 | 0.2031 | −0.3065 | 0.083* |
| H29B | 0.2852 | 0.1595 | −0.3007 | 0.083* |
| C30 | 0.0850 (4) | 0.15207 (12) | −0.41017 (19) | 0.0840 (8) |
| H30A | 0.0914 | 0.1188 | −0.4118 | 0.126* |
| H30B | 0.1501 | 0.1646 | −0.4695 | 0.126* |
| H30C | −0.0365 | 0.1616 | −0.4156 | 0.126* |
| N1 | 0.48008 (18) | 0.16871 (5) | 0.08585 (13) | 0.0412 (3) |
| N2 | 0.1382 (2) | 0.16591 (5) | −0.10730 (11) | 0.0412 (3) |
| O1 | 0.3160 (2) | 0.10260 (5) | −0.08116 (11) | 0.0526 (3) |
| O2 | 0.21833 (19) | 0.18395 (5) | 0.30852 (11) | 0.0523 (3) |
| O3 | −0.02944 (16) | 0.17580 (4) | 0.21260 (10) | 0.0455 (3) |
| Cl1 | 0.39756 (9) | 0.07585 (2) | 0.43710 (4) | 0.06494 (18) |
| Cl2 | 0.36248 (10) | −0.09601 (2) | 0.28361 (7) | 0.0864 (2) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0429 (9) | 0.0406 (9) | 0.0438 (10) | 0.0073 (7) | 0.0002 (7) | 0.0091 (8) |
| C2 | 0.0486 (11) | 0.0475 (11) | 0.0574 (12) | 0.0055 (8) | 0.0043 (9) | 0.0206 (9) |
| C3 | 0.0426 (10) | 0.0336 (9) | 0.0799 (15) | 0.0030 (7) | 0.0064 (9) | 0.0123 (9) |
| C4 | 0.0512 (11) | 0.0383 (10) | 0.0631 (13) | 0.0060 (8) | 0.0058 (9) | −0.0036 (9) |
| C5 | 0.0458 (10) | 0.0399 (10) | 0.0467 (10) | 0.0081 (8) | 0.0065 (8) | 0.0042 (8) |
| C6 | 0.0334 (8) | 0.0331 (8) | 0.0419 (9) | 0.0061 (6) | 0.0006 (7) | 0.0072 (7) |
| C7 | 0.0353 (8) | 0.0329 (8) | 0.0363 (9) | 0.0024 (6) | −0.0057 (6) | 0.0058 (6) |
| C8 | 0.0335 (9) | 0.0448 (10) | 0.0586 (12) | 0.0027 (7) | −0.0015 (8) | 0.0143 (8) |
| C9 | 0.0354 (8) | 0.0310 (8) | 0.0324 (8) | 0.0005 (6) | −0.0035 (6) | 0.0026 (6) |
| C10 | 0.0463 (9) | 0.0366 (9) | 0.0328 (9) | −0.0014 (7) | 0.0035 (7) | 0.0020 (7) |
| C11 | 0.0383 (8) | 0.0363 (9) | 0.0328 (8) | −0.0024 (7) | −0.0006 (6) | 0.0044 (6) |
| C12 | 0.0428 (9) | 0.0507 (11) | 0.0429 (10) | 0.0034 (8) | −0.0064 (8) | 0.0115 (8) |
| C13 | 0.0530 (11) | 0.0409 (10) | 0.0617 (13) | 0.0120 (8) | −0.0024 (9) | 0.0129 (9) |
| C14 | 0.0661 (13) | 0.0320 (9) | 0.0607 (13) | 0.0036 (9) | −0.0033 (10) | −0.0016 (8) |
| C15 | 0.0566 (11) | 0.0331 (9) | 0.0462 (10) | −0.0008 (8) | −0.0112 (8) | 0.0003 (7) |
| C16 | 0.0383 (8) | 0.0300 (8) | 0.0344 (8) | −0.0008 (6) | −0.0033 (6) | 0.0054 (6) |
| C17 | 0.0314 (7) | 0.0287 (7) | 0.0292 (8) | 0.0016 (6) | −0.0038 (6) | 0.0016 (6) |
| C18 | 0.0356 (8) | 0.0346 (8) | 0.0373 (9) | −0.0021 (6) | −0.0042 (7) | −0.0016 (7) |
| C19 | 0.0296 (8) | 0.0363 (9) | 0.0500 (10) | 0.0000 (6) | −0.0019 (7) | 0.0034 (7) |
| C20 | 0.0485 (11) | 0.0447 (11) | 0.0719 (14) | −0.0079 (8) | 0.0065 (10) | −0.0063 (10) |
| C21 | 0.0538 (12) | 0.0422 (12) | 0.113 (2) | −0.0126 (9) | 0.0122 (13) | 0.0043 (13) |
| C22 | 0.0523 (12) | 0.0620 (14) | 0.094 (2) | −0.0061 (11) | 0.0183 (12) | 0.0274 (13) |
| C23 | 0.0617 (13) | 0.0741 (16) | 0.0603 (14) | −0.0106 (11) | 0.0110 (11) | 0.0193 (12) |
| C24 | 0.0517 (11) | 0.0534 (12) | 0.0499 (12) | −0.0129 (9) | 0.0014 (9) | 0.0072 (9) |
| C25 | 0.0415 (9) | 0.0326 (8) | 0.0329 (8) | 0.0021 (7) | −0.0021 (7) | 0.0020 (6) |
| C26 | 0.0779 (16) | 0.0790 (17) | 0.0689 (16) | 0.0340 (13) | 0.0173 (12) | −0.0145 (13) |
| C27 | 0.0469 (11) | 0.0669 (14) | 0.0742 (15) | −0.0074 (10) | 0.0080 (10) | 0.0274 (12) |
| C28 | 0.0590 (11) | 0.0574 (12) | 0.0349 (10) | −0.0063 (9) | −0.0075 (8) | −0.0045 (8) |
| C29 | 0.0680 (14) | 0.101 (2) | 0.0381 (11) | −0.0087 (13) | 0.0017 (10) | 0.0019 (11) |

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|---|---|---|---|---|---|---|
| C30 | 0.0926 (19) | 0.124 (3) | 0.0351 (12) | 0.0123 (17) | 0.0002 (12) | −0.0111 (13) |
| N1 | 0.0341 (7) | 0.0395 (8) | 0.0498 (9) | −0.0018 (6) | −0.0025 (6) | 0.0130 (6) |
| N2 | 0.0517 (8) | 0.0428 (8) | 0.0289 (7) | 0.0006 (7) | −0.0058 (6) | −0.0009 (6) |
| O1 | 0.0706 (9) | 0.0416 (7) | 0.0459 (8) | 0.0095 (6) | 0.0077 (6) | −0.0063 (6) |
| O2 | 0.0649 (8) | 0.0495 (8) | 0.0424 (8) | 0.0023 (6) | −0.0125 (6) | −0.0131 (6) |
| O3 | 0.0418 (7) | 0.0473 (7) | 0.0473 (7) | 0.0131 (5) | 0.0022 (5) | −0.0050 (6) |
| Cl1 | 0.0957 (4) | 0.0589 (3) | 0.0401 (3) | 0.0111 (3) | −0.0062 (3) | 0.0068 (2) |
| Cl2 | 0.0968 (5) | 0.0375 (3) | 0.1251 (6) | −0.0044 (3) | 0.0081 (4) | 0.0214 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.383 (2) | C17—C18 | 1.539 (2) |
| C1—C2 | 1.387 (3) | C18—C19 | 1.520 (2) |
| C1—Cl1 | 1.736 (2) | C18—H18A | 0.9700 |
| C2—C3 | 1.369 (3) | C18—H18B | 0.9700 |
| C2—H2 | 0.9300 | C19—C24 | 1.376 (3) |
| C3—C4 | 1.373 (3) | C19—C20 | 1.384 (3) |
| C3—Cl2 | 1.7356 (19) | C20—C21 | 1.388 (3) |
| C4—C5 | 1.384 (3) | C20—H20 | 0.9300 |
| C4—H4 | 0.9300 | C21—C22 | 1.362 (4) |
| C5—C6 | 1.392 (3) | C21—H21 | 0.9300 |
| C5—H5 | 0.9300 | C22—C23 | 1.359 (4) |
| C6—C7 | 1.509 (2) | C22—H22 | 0.9300 |
| C7—C8 | 1.527 (2) | C23—C24 | 1.388 (3) |
| C7—C17 | 1.577 (2) | C23—H23 | 0.9300 |
| C7—H7 | 0.9800 | C24—H24 | 0.9300 |
| C8—N1 | 1.451 (2) | C25—O2 | 1.193 (2) |
| C8—H8A | 0.9700 | C25—O3 | 1.336 (2) |
| C8—H8B | 0.9700 | C26—O3 | 1.442 (2) |
| C9—N1 | 1.456 (2) | C26—H26A | 0.9600 |
| C9—C16 | 1.507 (2) | C26—H26B | 0.9600 |
| C9—C10 | 1.550 (2) | C26—H26C | 0.9600 |
| C9—C17 | 1.605 (2) | C27—N1 | 1.455 (2) |
| C10—O1 | 1.218 (2) | C27—H27A | 0.9600 |
| C10—N2 | 1.360 (2) | C27—H27B | 0.9600 |
| C11—C12 | 1.378 (2) | C27—H27C | 0.9600 |
| C11—C16 | 1.385 (2) | C28—N2 | 1.460 (2) |
| C11—N2 | 1.404 (2) | C28—C29 | 1.508 (3) |
| C12—C13 | 1.375 (3) | C28—H28A | 0.9700 |
| C12—H12 | 0.9300 | C28—H28B | 0.9700 |
| C13—C14 | 1.364 (3) | C29—C30 | 1.500 (3) |
| C13—H13 | 0.9300 | C29—H29A | 0.9700 |
| C14—C15 | 1.390 (3) | C29—H29B | 0.9700 |
| C14—H14 | 0.9300 | C30—H30A | 0.9600 |
| C15—C16 | 1.370 (2) | C30—H30B | 0.9600 |
| C15—H15 | 0.9300 | C30—H30C | 0.9600 |
| C17—C25 | 1.530 (2) | | |

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|---|---|---|---|
| C6—C1—C2 | 122.61 (19) | C19—C18—H18A | 107.7 |
| C6—C1—Cl1 | 121.26 (14) | C17—C18—H18A | 107.7 |
| C2—C1—Cl1 | 116.14 (15) | C19—C18—H18B | 107.7 |
| C3—C2—C1 | 118.30 (18) | C17—C18—H18B | 107.7 |
| C3—C2—H2 | 120.9 | H18A—C18—H18B | 107.1 |
| C1—C2—H2 | 120.9 | C24—C19—C20 | 117.20 (18) |
| C2—C3—C4 | 121.67 (18) | C24—C19—C18 | 125.81 (16) |
| C2—C3—Cl2 | 118.71 (17) | C20—C19—C18 | 116.99 (17) |
| C4—C3—Cl2 | 119.60 (18) | C19—C20—C21 | 121.1 (2) |
| C3—C4—C5 | 118.64 (19) | C19—C20—H20 | 119.4 |
| C3—C4—H4 | 120.7 | C21—C20—H20 | 119.4 |
| C5—C4—H4 | 120.7 | C22—C21—C20 | 120.4 (2) |
| C4—C5—C6 | 122.10 (18) | C22—C21—H21 | 119.8 |
| C4—C5—H5 | 118.9 | C20—C21—H21 | 119.8 |
| C6—C5—H5 | 118.9 | C23—C22—C21 | 119.5 (2) |
| C1—C6—C5 | 116.61 (16) | C23—C22—H22 | 120.3 |
| C1—C6—C7 | 121.66 (16) | C21—C22—H22 | 120.3 |
| C5—C6—C7 | 121.72 (16) | C22—C23—C24 | 120.3 (2) |
| C6—C7—C8 | 112.70 (14) | C22—C23—H23 | 119.8 |
| C6—C7—C17 | 116.98 (13) | C24—C23—H23 | 119.8 |
| C8—C7—C17 | 105.37 (13) | C19—C24—C23 | 121.5 (2) |
| C6—C7—H7 | 107.1 | C19—C24—H24 | 119.3 |
| C8—C7—H7 | 107.1 | C23—C24—H24 | 119.3 |
| C17—C7—H7 | 107.1 | O2—C25—O3 | 123.05 (16) |
| N1—C8—C7 | 103.09 (14) | O2—C25—C17 | 125.83 (15) |
| N1—C8—H8A | 111.1 | O3—C25—C17 | 111.11 (14) |
| C7—C8—H8A | 111.1 | O3—C26—H26A | 109.5 |
| N1—C8—H8B | 111.1 | O3—C26—H26B | 109.5 |
| C7—C8—H8B | 111.1 | H26A—C26—H26B | 109.5 |
| H8A—C8—H8B | 109.1 | O3—C26—H26C | 109.5 |
| N1—C9—C16 | 112.40 (13) | H26A—C26—H26C | 109.5 |
| N1—C9—C10 | 115.24 (14) | H26B—C26—H26C | 109.5 |
| C16—C9—C10 | 100.96 (13) | N1—C27—H27A | 109.5 |
| N1—C9—C17 | 103.19 (12) | N1—C27—H27B | 109.5 |
| C16—C9—C17 | 116.61 (13) | H27A—C27—H27B | 109.5 |
| C10—C9—C17 | 108.92 (12) | N1—C27—H27C | 109.5 |
| O1—C10—N2 | 125.15 (17) | H27A—C27—H27C | 109.5 |
| O1—C10—C9 | 126.75 (16) | H27B—C27—H27C | 109.5 |
| N2—C10—C9 | 108.09 (14) | N2—C28—C29 | 113.74 (17) |
| C12—C11—C16 | 121.37 (16) | N2—C28—H28A | 108.8 |
| C12—C11—N2 | 129.12 (16) | C29—C28—H28A | 108.8 |
| C16—C11—N2 | 109.46 (14) | N2—C28—H28B | 108.8 |
| C13—C12—C11 | 117.81 (18) | C29—C28—H28B | 108.8 |
| C13—C12—H12 | 121.1 | H28A—C28—H28B | 107.7 |
| C11—C12—H12 | 121.1 | C30—C29—C28 | 111.6 (2) |
| C14—C13—C12 | 121.56 (18) | C30—C29—H29A | 109.3 |
| C14—C13—H13 | 119.2 | C28—C29—H29A | 109.3 |
| C12—C13—H13 | 119.2 | C30—C29—H29B | 109.3 |

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|---|---|---|---|
| C13—C14—C15 | 120.37 (18) | C28—C29—H29B | 109.3 |
| C13—C14—H14 | 119.8 | H29A—C29—H29B | 108.0 |
| C15—C14—H14 | 119.8 | C29—C30—H30A | 109.5 |
| C16—C15—C14 | 118.82 (18) | C29—C30—H30B | 109.5 |
| C16—C15—H15 | 120.6 | H30A—C30—H30B | 109.5 |
| C14—C15—H15 | 120.6 | C29—C30—H30C | 109.5 |
| C15—C16—C11 | 119.97 (15) | H30A—C30—H30C | 109.5 |
| C15—C16—C9 | 130.58 (16) | H30B—C30—H30C | 109.5 |
| C11—C16—C9 | 109.42 (14) | C8—N1—C27 | 114.20 (15) |
| C25—C17—C18 | 109.67 (13) | C8—N1—C9 | 107.01 (13) |
| C25—C17—C7 | 109.73 (13) | C27—N1—C9 | 115.56 (15) |
| C18—C17—C7 | 116.11 (13) | C10—N2—C11 | 111.17 (14) |
| C25—C17—C9 | 105.54 (12) | C10—N2—C28 | 123.42 (16) |
| C18—C17—C9 | 112.66 (13) | C11—N2—C28 | 125.32 (15) |
| C7—C17—C9 | 102.46 (11) | C25—O3—C26 | 116.54 (17) |
| C19—C18—C17 | 118.45 (14) | | |

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|---|---|---|---|
| C6—C1—C2—C3 | 1.2 (3) | C16—C9—C17—C25 | −28.00 (17) |
| Cl1—C1—C2—C3 | −178.64 (15) | C10—C9—C17—C25 | −141.37 (13) |
| C1—C2—C3—C4 | 1.1 (3) | N1—C9—C17—C18 | −144.62 (13) |
| C1—C2—C3—Cl2 | 179.34 (14) | C16—C9—C17—C18 | 91.65 (16) |
| C2—C3—C4—C5 | −1.5 (3) | C10—C9—C17—C18 | −21.72 (17) |
| Cl2—C3—C4—C5 | −179.75 (15) | N1—C9—C17—C7 | −19.13 (15) |
| C3—C4—C5—C6 | −0.3 (3) | C16—C9—C17—C7 | −142.86 (14) |
| C2—C1—C6—C5 | −2.9 (3) | C10—C9—C17—C7 | 103.78 (14) |
| Cl1—C1—C6—C5 | 176.96 (13) | C25—C17—C18—C19 | −62.32 (18) |
| C2—C1—C6—C7 | 178.20 (16) | C7—C17—C18—C19 | 62.74 (19) |
| Cl1—C1—C6—C7 | −2.0 (2) | C9—C17—C18—C19 | −179.56 (13) |
| C4—C5—C6—C1 | 2.4 (3) | C17—C18—C19—C24 | 24.6 (3) |
| C4—C5—C6—C7 | −178.66 (16) | C17—C18—C19—C20 | −154.86 (16) |
| C1—C6—C7—C8 | 127.31 (18) | C24—C19—C20—C21 | −1.1 (3) |
| C5—C6—C7—C8 | −51.6 (2) | C18—C19—C20—C21 | 178.42 (18) |
| C1—C6—C7—C17 | −110.32 (18) | C19—C20—C21—C22 | −0.1 (4) |
| C5—C6—C7—C17 | 70.8 (2) | C20—C21—C22—C23 | 1.1 (4) |
| C6—C7—C8—N1 | 158.73 (15) | C21—C22—C23—C24 | −0.8 (4) |
| C17—C7—C8—N1 | 30.04 (18) | C20—C19—C24—C23 | 1.4 (3) |
| N1—C9—C10—O1 | 50.1 (2) | C18—C19—C24—C23 | −178.06 (18) |
| C16—C9—C10—O1 | 171.47 (17) | C22—C23—C24—C19 | −0.5 (4) |
| C17—C9—C10—O1 | −65.2 (2) | C18—C17—C25—O2 | 148.57 (17) |
| N1—C9—C10—N2 | −130.73 (15) | C7—C17—C25—O2 | 19.9 (2) |
| C16—C9—C10—N2 | −9.37 (17) | C9—C17—C25—O2 | −89.82 (19) |
| C17—C9—C10—N2 | 113.91 (14) | C18—C17—C25—O3 | −32.52 (18) |
| C16—C11—C12—C13 | 1.2 (3) | C7—C17—C25—O3 | −161.18 (13) |
| N2—C11—C12—C13 | −175.90 (17) | C9—C17—C25—O3 | 89.09 (15) |
| C11—C12—C13—C14 | 1.5 (3) | N2—C28—C29—C30 | 176.8 (2) |
| C12—C13—C14—C15 | −1.9 (3) | C7—C8—N1—C27 | −174.07 (17) |
| C13—C14—C15—C16 | −0.3 (3) | C7—C8—N1—C9 | −44.87 (18) |
| C14—C15—C16—C11 | 2.9 (3) | C16—C9—N1—C8 | 166.61 (15) |

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|---|---|---|---|
| C14—C15—C16—C9 | −179.25 (17) | C10—C9—N1—C8 | −78.45 (18) |
| C12—C11—C16—C15 | −3.5 (3) | C17—C9—N1—C8 | 40.15 (17) |
| N2—C11—C16—C15 | 174.18 (16) | C16—C9—N1—C27 | −65.0 (2) |
| C12—C11—C16—C9 | 178.32 (15) | C10—C9—N1—C27 | 50.0 (2) |
| N2—C11—C16—C9 | −4.06 (18) | C17—C9—N1—C27 | 168.57 (16) |
| N1—C9—C16—C15 | −46.7 (2) | O1—C10—N2—C11 | −173.11 (16) |
| C10—C9—C16—C15 | −170.01 (18) | C9—C10—N2—C11 | 7.72 (19) |
| C17—C9—C16—C15 | 72.2 (2) | O1—C10—N2—C28 | 3.8 (3) |
| N1—C9—C16—C11 | 131.31 (15) | C9—C10—N2—C28 | −175.39 (15) |
| C10—C9—C16—C11 | 7.97 (16) | C12—C11—N2—C10 | 174.91 (17) |
| C17—C9—C16—C11 | −109.83 (16) | C16—C11—N2—C10 | −2.48 (19) |
| C6—C7—C17—C25 | 115.66 (16) | C12—C11—N2—C28 | −1.9 (3) |
| C8—C7—C17—C25 | −118.25 (15) | C16—C11—N2—C28 | −179.30 (16) |
| C6—C7—C17—C18 | −9.4 (2) | C29—C28—N2—C10 | −93.2 (2) |
| C8—C7—C17—C18 | 116.72 (16) | C29—C28—N2—C11 | 83.2 (2) |
| C6—C7—C17—C9 | −132.57 (14) | O2—C25—O3—C26 | −0.9 (3) |
| C8—C7—C17—C9 | −6.48 (17) | C17—C25—O3—C26 | −179.80 (17) |
| N1—C9—C17—C25 | 95.73 (14) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C18—H18*B*···O1 | 0.97 | 2.39 | 3.073 (2) | 127 |
| C13—H13···O2$^{i}$ | 0.93 | 2.46 | 3.132 (2) | 129 |

Symmetry code: (i) $x-1/2, -y+1/2, z-1/2$.