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3-*tert*-Butyl-1-(4-chlorophenyl)-7,7-dimethyl-5,6,7,8-tetrahydropyrazolo[3,4-*b*]quinolin-5-one: centrosymmetric dimers generated by C—H···π(arene) hydrogen bonds

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.053
wR factor = 0.122
Data-to-parameter ratio = 17.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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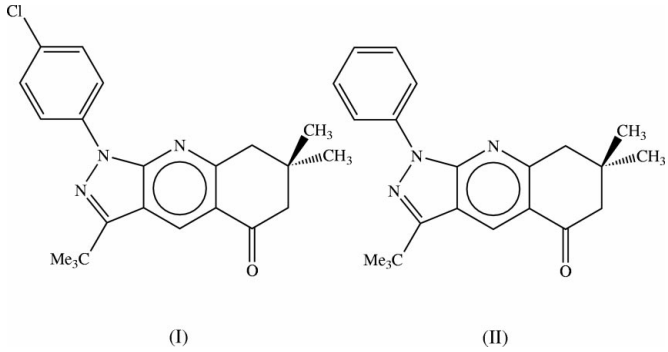
Molecules of the title compound, $C_{22}H_{24}ClN_3O$, are linked by two pairs of C—H···π(arene) hydrogen bonds into centrosymmetric dimers.

Comment

We report here the structure of the title compound, (I) (Fig. 1), whose supramolecular aggregation shows some interesting differences from that in the unsubstituted analogue (II) (Low *et al.*, 2004).



The bond lengths in (I) are very similar to those in (II) and require no further discussion here. The ring-puckering parameters (Cremer & Pople, 1975) for the carbocyclic rings in (I) and (II) are quite similar [for the atom sequence C4*A*—C5—

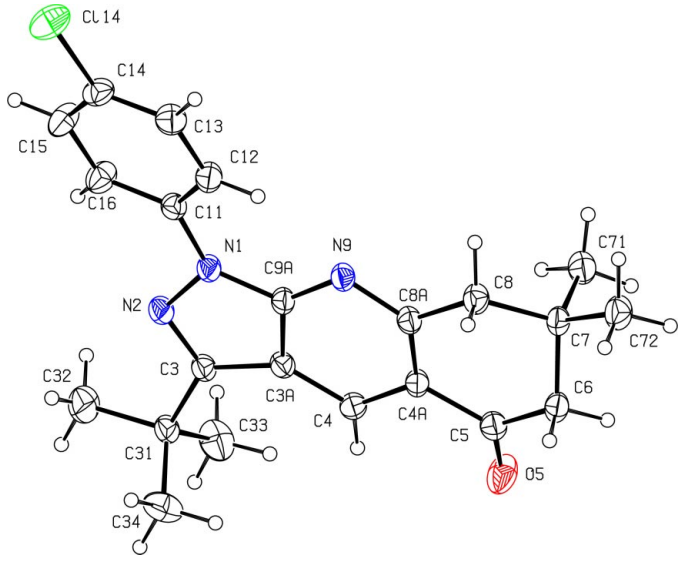


Figure 1
The molecule of compound (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

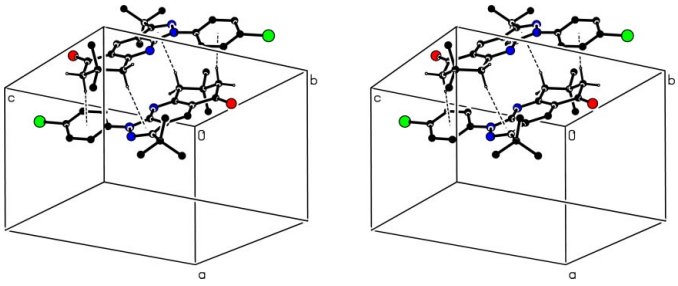


Figure 2
Stereoview of part of the crystal structure of compound (I), showing the formation of a centrosymmetric hydrogen-bonded dimer. For clarity, H atoms bonded to C atoms not involved in the motifs shown have been omitted. C—H···π hydrogen bonds are shown as dashed lines.

C6—C7—C8—C8*A*, $\theta = 132.1\ (4)^\circ$ and $\varphi = 351.0\ (5)$ in (I), and $\theta = 127.4\ (3)^\circ$ and $\varphi = 353.8\ (3)$ in (II)] and indicate an envelope conformation in each compound (Evans & Boeyens, 1989).

The principal difference between (I) and (II) arises from the intermolecular aggregation. In (I), the molecules are linked into centrosymmetric dimers by two pairs of C—H···π(arene) interactions (Table 1). Atoms C6 and C8 in the molecule at (x, y, z) act as donors, *via* the axial H atoms H6*A* and H8*A*, to the aryl and pyrazole rings, respectively, in the molecule at ($-x$, $1-y$, $1-z$) (Fig. 2). There are no other types of intermolecular hydrogen bond in the structure of (I) and there are no direction-specific interactions between the dimers. By contrast, in (II), the molecules are linked into chains by means of a C—H···N hydrogen bond, and C—H···π(arene) hydrogen bonds are absent from the structure of (II). It is striking that the presence of a single remote Cl substituent in (I) is associated with such a change in the hydrogen bonding.

Experimental

A mixture of 5-amino-3-*tert*-butyl-1-(4-chlorophenyl)pyrazole (1 mmol), 5,5-dimethyl-1,3-cyclohexanedione (dimedone) (1 mmol) and formaldehyde (3 mmol) was placed in an open Pyrex-glass vessel and irradiated in a domestic microwave oven for 3 min (at 600 watts). After reaction, the mixture was extracted with ethanol; the extract was filtered and the product, (I), was purified by column chromatography on silica gel, with dichloromethane/hexane (7:3, *v*/*v*) as eluant. Yield 39%, m.p. 428 K. MS (EI 70 eV) *m*/*z* (%): 383/381 (15/49), 382 (12), 368/366 (37/100), 149 (16), 57 (11). Crystals suitable for single-crystal X-ray diffraction were grown from ethanol.

Crystal data

$C_{22}H_{24}ClN_3O$
$M_r = 381.89$
Triclinic, $P\overline{1}$
$a = 8.6851\ (11)$ Å
$b = 10.6167\ (9)$ Å
$c = 12.4330\ (12)$ Å
$\alpha = 106.724\ (8)^\circ$
$\beta = 101.049\ (10)^\circ$
$\gamma = 107.406\ (8)^\circ$
$V = 998.1\ (2)$ Å^3
$Z = 2$
$D_x = 1.271$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4289 reflections
$\theta = 5.0$–27.5°
$\mu = 0.21$ mm^{-1}
$T = 293\ (2)$ K
Block, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*EVALCCD*; Duisenberg *et al.*, 2003)
$T_{min} = 0.925$, $T_{max} = 0.980$
13 573 measured reflections
4289 independent reflections
1940 reflections with $I > 2\sigma(I)$
$R_{int} = 0.091$
$\theta_{max} = 27.5^\circ$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.122$
$S = 0.93$
4289 reflections
250 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.3921P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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

	D—H	H···A	D···A	D—H···A
C6—H6*A*···*Cg*1^{i}	0.97	2.77	3.649 (3)	151
C8—H8*A*···*Cg*2^{i}	0.97	2.82	3.768 (3)	165

Symmetry code: (i) $-x, -y+1, -z+1$. Notes: *Cg*1 and *Cg*2 are the centroids of rings C11–C16 and N1/N2/C3/C3*A*/C9*A*, respectively.

All H atoms were located in difference maps and then treated as riding atoms, with C—H distances 0.93 Å (aromatic), 0.96 Å (CH_3) or 0.97 Å (CH_2), and with $U_{iso}(H) = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for the methyl groups. This structure was determined at room temperature and both the data completeness and the ratio of observed-to-unique reflections are rather low. Since this structure is, in all respects, similar to its non-chlorinated analogue (II), a second data-collection, at low temperature, was not justified.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DIRAX*/*LSQ* (Duisenberg *et al.*, 2000); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *OSCAIL* (McArdle, 2003) and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the 'Servicios Técnicos de Investigación', University of Jaén. JC thanks the Consejería de Educación y Ciencia (Junta de Andalucía, Spain) and the Universidad de Jaén for financial support. JQ and JM thank COLCIENCIAS and UNIVALLE (Universidad del Valle) for financial support. JNL thanks NCR Self-Service, Dundee, for grants which have provided computing facilities for this work.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Duisenberg, A. J. M., Hooft, R. W. W., Schreurs, A. M. M. & Kroon, J. (2000). *J. Appl. Cryst.* **33**, 893–898.
Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
Evans, D. G. & Boeyens, J. C. A. (1989). *Acta Cryst.* B**45**, 581–590.

Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Hooft, R. W. W. (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
Low, J. N., Cobo, J., Mera, J., Quiroga, J. & Glidewell, C. (2004). *Acta Cryst.* C**60**, o479–o482.
McArdle, P. (2003). *OSCAIL for Windows*. Version 10. Crystallography Centre, Chemistry Department, NUI Galway, Ireland.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

Acta Cryst. (2005). E**61**, o49–o51 [https://doi.org/10.1107/S1600536804031927]

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3-*tert*-Butyl-1-(4-chlorophenyl)-7,7-dimethyl-5,6,7,8- tetrahydroimidazo[3,4-*b*]quinolin-5-one

Crystal data

$C_{22}H_{24}ClN_3O$
$M_r = 381.89$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.6851\ (11)$ Å
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$c = 12.4330\ (12)$ Å
$\alpha = 106.724\ (8)°$
$\beta = 101.049\ (10)°$
$\gamma = 107.406\ (8)°$
$V = 998.1\ (2)$ Å^3
$Z = 2$
$F(000) = 404$
$D_x = 1.271$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4289 reflections
$\theta = 5.0–27.5°$
$\mu = 0.21$ mm^{-1}
$T = 293$ K
Block, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: fine-focus sealed X-ray tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan EvalCCD, (Duisenberg et al., 2003)
$T_{min} = 0.925$, $T_{max} = 0.980$
13573 measured reflections
4289 independent reflections
1940 reflections with $I > 2\sigma(I)$
$R_{int} = 0.091$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.0°$
$h = -11\rightarrow11$
$k = -13\rightarrow13$
$l = -13\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.122$
$S = 0.93$
4289 reflections
250 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
$w = 1/[\sigma^2(F_o^2) + (0.0419P)^2 + 0.3921P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl14	0.25042 (9)	0.12483 (9)	0.88047 (7)	0.0675 (3)
O5	0.1443 (2)	0.6703 (2)	0.18979 (16)	0.0659 (6)
N1	0.3639 (2)	0.5147 (2)	0.62204 (16)	0.0367 (5)
N2	0.4930 (2)	0.6473 (2)	0.68344 (16)	0.0388 (5)
N9	0.1388 (2)	0.4006 (2)	0.43698 (16)	0.0363 (5)
C3	0.4865 (3)	0.7244 (2)	0.61863 (19)	0.0350 (6)
C3A	0.3525 (3)	0.6432 (2)	0.50982 (19)	0.0348 (6)
C4	0.2845 (3)	0.6615 (3)	0.40730 (19)	0.0377 (6)
C4A	0.1440 (3)	0.5504 (3)	0.32209 (19)	0.0351 (6)
C5	0.0714 (3)	0.5675 (3)	0.2111 (2)	0.0411 (6)
C6	−0.0937 (3)	0.4546 (3)	0.13021 (19)	0.0403 (6)
C7	−0.1159 (3)	0.3051 (3)	0.12727 (19)	0.0373 (6)
C8	−0.0857 (3)	0.3079 (3)	0.25340 (19)	0.0410 (6)
C8A	0.0735 (3)	0.4244 (2)	0.34112 (19)	0.0353 (6)
C9A	0.2751 (3)	0.5102 (3)	0.51641 (19)	0.0341 (5)
C11	0.3375 (3)	0.4160 (2)	0.67918 (19)	0.0351 (6)
C12	0.1866 (3)	0.3007 (3)	0.6409 (2)	0.0436 (6)
C13	0.1620 (3)	0.2101 (3)	0.7022 (2)	0.0468 (7)
C14	0.2860 (3)	0.2358 (3)	0.8012 (2)	0.0441 (6)
C15	0.4363 (3)	0.3484 (3)	0.8392 (2)	0.0528 (7)
C16	0.4628 (3)	0.4393 (3)	0.7780 (2)	0.0484 (7)
C31	0.6009 (3)	0.8794 (2)	0.66531 (19)	0.0388 (6)
C32	0.7361 (4)	0.9172 (3)	0.7800 (2)	0.0710 (9)
C33	0.6879 (3)	0.9129 (3)	0.5760 (2)	0.0653 (8)
C34	0.4916 (3)	0.9676 (3)	0.6879 (3)	0.0637 (8)
C71	0.0095 (3)	0.2563 (3)	0.0723 (2)	0.0521 (7)
C72	−0.2966 (3)	0.2042 (3)	0.0534 (2)	0.0531 (7)
H4	0.3319	0.7459	0.3961	0.045*
H6A	−0.1847	0.4782	0.1540	0.048*
H6E	−0.1044	0.4544	0.0511	0.048*
H8A	−0.1814	0.3177	0.2793	0.049*
H8E	−0.0818	0.2174	0.2531	0.049*
H12	0.1020	0.2840	0.5741	0.052*
H13	0.0611	0.1317	0.6761	0.056*
H15	0.5206	0.3641	0.9058	0.063*
H16	0.5651	0.5161	0.8036	0.058*
H32A	0.6828	0.8979	0.8375	0.107*
H32B	0.8066	1.0162	0.8087	0.107*
H32C	0.8043	0.8614	0.7660	0.107*
H33A	0.7608	0.8610	0.5649	0.098*
H33B	0.7538	1.0128	0.6046	0.098*
H33C	0.6040	0.8859	0.5023	0.098*
H34A	0.4068	0.9443	0.6156	0.096*
H34B	0.5617	1.0668	0.7175	0.096*
H34C	0.4378	0.9472	0.7449	0.096*

H71A	−0.0036	0.2643	−0.0036	0.078*
H71B	−0.0121	0.1592	0.0635	0.078*
H71C	0.1230	0.3148	0.1225	0.078*
H72A	−0.3151	0.2014	−0.0260	0.080*
H72B	−0.3752	0.2370	0.0863	0.080*
H72C	−0.3130	0.1107	0.0536	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl14	0.0747 (5)	0.0743 (6)	0.0797 (5)	0.0312 (4)	0.0329 (4)	0.0558 (5)
O5	0.0719 (13)	0.0574 (14)	0.0505 (12)	−0.0006 (11)	0.0009 (10)	0.0324 (11)
N1	0.0409 (11)	0.0282 (12)	0.0303 (11)	0.0050 (9)	0.0027 (9)	0.0096 (9)
N2	0.0402 (11)	0.0304 (12)	0.0341 (11)	0.0060 (10)	0.0034 (9)	0.0080 (10)
N9	0.0400 (11)	0.0315 (12)	0.0290 (10)	0.0064 (10)	0.0053 (9)	0.0099 (9)
C3	0.0365 (13)	0.0309 (14)	0.0307 (13)	0.0087 (11)	0.0067 (10)	0.0079 (11)
C3A	0.0404 (13)	0.0293 (15)	0.0306 (13)	0.0092 (11)	0.0102 (11)	0.0095 (11)
C4	0.0455 (14)	0.0316 (15)	0.0350 (14)	0.0103 (12)	0.0119 (12)	0.0154 (12)
C4A	0.0389 (13)	0.0335 (15)	0.0272 (13)	0.0093 (12)	0.0059 (11)	0.0104 (11)
C5	0.0472 (15)	0.0380 (17)	0.0352 (14)	0.0138 (13)	0.0114 (12)	0.0125 (13)
C6	0.0426 (14)	0.0478 (17)	0.0286 (13)	0.0175 (13)	0.0074 (11)	0.0134 (12)
C7	0.0376 (13)	0.0388 (15)	0.0277 (13)	0.0114 (11)	0.0027 (10)	0.0092 (11)
C8	0.0416 (13)	0.0383 (16)	0.0339 (13)	0.0063 (12)	0.0062 (11)	0.0128 (12)
C8A	0.0392 (13)	0.0347 (15)	0.0288 (13)	0.0115 (12)	0.0103 (11)	0.0096 (11)
C9A	0.0400 (13)	0.0328 (15)	0.0260 (12)	0.0105 (12)	0.0079 (11)	0.0104 (11)
C11	0.0395 (13)	0.0331 (15)	0.0319 (13)	0.0124 (12)	0.0117 (11)	0.0120 (11)
C12	0.0478 (15)	0.0410 (17)	0.0356 (14)	0.0108 (14)	0.0072 (12)	0.0146 (12)
C13	0.0483 (15)	0.0391 (17)	0.0484 (16)	0.0087 (13)	0.0142 (13)	0.0180 (13)
C14	0.0527 (16)	0.0453 (18)	0.0472 (15)	0.0222 (14)	0.0213 (13)	0.0273 (13)
C15	0.0472 (16)	0.061 (2)	0.0522 (17)	0.0167 (16)	0.0046 (13)	0.0338 (15)
C16	0.0385 (13)	0.0490 (18)	0.0522 (16)	0.0095 (13)	0.0043 (12)	0.0246 (14)
C31	0.0421 (13)	0.0302 (15)	0.0324 (13)	0.0061 (12)	0.0043 (11)	0.0075 (11)
C32	0.0683 (19)	0.0462 (19)	0.0589 (19)	0.0005 (15)	−0.0160 (15)	0.0086 (15)
C33	0.0610 (18)	0.055 (2)	0.0609 (19)	−0.0015 (15)	0.0218 (15)	0.0169 (16)
C34	0.0635 (18)	0.0392 (18)	0.081 (2)	0.0181 (15)	0.0216 (16)	0.0134 (16)
C71	0.0578 (16)	0.0582 (19)	0.0378 (14)	0.0273 (15)	0.0102 (12)	0.0112 (13)
C72	0.0506 (15)	0.0494 (18)	0.0417 (15)	0.0086 (14)	−0.0018 (12)	0.0129 (13)

Geometric parameters (Å, º)

N1—C9A	1.370 (3)	C34—H34B	0.96
N1—N2	1.390 (2)	C34—H34C	0.96
N1—C11	1.415 (3)	C3A—C4	1.390 (3)
C11—C16	1.381 (3)	C3A—C9A	1.404 (3)
C11—C12	1.381 (3)	C4—C4A	1.388 (3)
C12—C13	1.383 (3)	C4—H4	0.93
C12—H12	0.93	C4A—C8A	1.403 (3)
C13—C14	1.368 (3)	C4A—C5	1.488 (3)

C13—H13	0.93	C5—O5	1.217 (3)
C14—C15	1.365 (3)	C5—C6	1.493 (3)
C14—Cl14	1.738 (2)	C6—C7	1.529 (3)
C15—C16	1.384 (3)	C6—H6E	0.97
C15—H15	0.93	C6—H6A	0.97
C16—H16	0.93	C7—C8	1.530 (3)
N2—C3	1.309 (3)	C7—C71	1.531 (3)
C3—C3A	1.433 (3)	C7—C72	1.526 (3)
C3—C31	1.511 (3)	C71—H71A	0.96
C31—C33	1.522 (3)	C71—H71B	0.96
C31—C32	1.523 (3)	C71—H71C	0.96
C31—C34	1.529 (3)	C72—H72A	0.96
C32—H32A	0.96	C72—H72B	0.96
C32—H32B	0.96	C72—H72C	0.96
C32—H32C	0.96	C8—C8A	1.501 (3)
C33—H33A	0.96	C8—H8A	0.97
C33—H33B	0.96	C8—H8E	0.97
C33—H33C	0.96	C8A—N9	1.344 (3)
C34—H34A	0.96	N9—C9A	1.341 (3)

C9A—N1—N2	110.15 (18)	C4—C3A—C9A	116.4 (2)
C9A—N1—C11	131.75 (19)	C4—C3A—C3	138.0 (2)
N2—N1—C11	117.86 (17)	C9A—C3A—C3	105.5 (2)
C16—C11—C12	119.6 (2)	C4A—C4—C3A	118.3 (2)
C16—C11—N1	119.0 (2)	C4A—C4—H4	120.9
C12—C11—N1	121.3 (2)	C3A—C4—H4	120.9
C11—C12—C13	119.8 (2)	C4—C4A—C8A	120.0 (2)
C11—C12—H12	120.1	C4—C4A—C5	118.9 (2)
C13—C12—H12	120.1	C8A—C4A—C5	121.1 (2)
C14—C13—C12	120.0 (2)	O5—C5—C4A	120.5 (2)
C14—C13—H13	120.0	O5—C5—C6	122.7 (2)
C12—C13—H13	120.0	C4A—C5—C6	116.8 (2)
C15—C14—C13	120.8 (2)	C5—C6—C7	114.50 (19)
C15—C14—Cl14	119.66 (19)	C5—C6—H6E	108.6
C13—C14—Cl14	119.6 (2)	C7—C6—H6E	108.6
C14—C15—C16	119.7 (2)	C5—C6—H6A	108.6
C14—C15—H15	120.2	C7—C6—H6A	108.6
C16—C15—H15	120.2	H6E—C6—H6A	107.6
C11—C16—C15	120.1 (2)	C72—C7—C6	108.89 (18)
C11—C16—H16	119.9	C72—C7—C8	109.43 (19)
C15—C16—H16	119.9	C6—C7—C8	108.65 (19)
C3—N2—N1	107.66 (17)	C72—C7—C71	109.6 (2)
N2—C3—C3A	110.1 (2)	C6—C7—C71	109.7 (2)
N2—C3—C31	120.81 (19)	C8—C7—C71	110.59 (18)
C3A—C3—C31	128.9 (2)	C7—C71—H71A	109.5
C3—C31—C33	110.5 (2)	C7—C71—H71B	109.5
C3—C31—C32	110.7 (2)	H71A—C71—H71B	109.5
C33—C31—C32	108.7 (2)	C7—C71—H71C	109.5

C3—C31—C34	107.98 (19)	H71A—C71—H71C	109.5
C33—C31—C34	109.6 (2)	H71B—C71—H71C	109.5
C32—C31—C34	109.4 (2)	C7—C72—H72A	109.5
C31—C32—H32A	109.5	C7—C72—H72B	109.5
C31—C32—H32B	109.5	H72A—C72—H72B	109.5
H32A—C32—H32B	109.5	C7—C72—H72C	109.5
C31—C32—H32C	109.5	H72A—C72—H72C	109.5
H32A—C32—H32C	109.5	H72B—C72—H72C	109.5
H32B—C32—H32C	109.5	C8A—C8—C7	114.8 (2)
C31—C33—H33A	109.5	C8A—C8—H8A	108.6
C31—C33—H33B	109.5	C7—C8—H8A	108.6
H33A—C33—H33B	109.5	C8A—C8—H8E	108.6
C31—C33—H33C	109.5	C7—C8—H8E	108.6
H33A—C33—H33C	109.5	H8A—C8—H8E	107.5
H33B—C33—H33C	109.5	N9—C8A—C4A	123.6 (2)
C31—C34—H34A	109.5	N9—C8A—C8	116.1 (2)
C31—C34—H34B	109.5	C4A—C8A—C8	120.3 (2)
H34A—C34—H34B	109.5	C9A—N9—C8A	114.3 (2)
C31—C34—H34C	109.5	N9—C9A—N1	126.1 (2)
H34A—C34—H34C	109.5	N9—C9A—C3A	127.4 (2)
H34B—C34—H34C	109.5	N1—C9A—C3A	106.52 (19)

C9A—N1—C11—C16	170.1 (2)	C3A—C4—C4A—C5	−179.1 (2)
N2—N1—C11—C16	−16.2 (3)	C4—C4A—C5—O5	7.9 (3)
C9A—N1—C11—C12	−12.6 (4)	C8A—C4A—C5—O5	−171.7 (2)
N2—N1—C11—C12	161.1 (2)	C4—C4A—C5—C6	−171.2 (2)
C16—C11—C12—C13	0.4 (3)	C8A—C4A—C5—C6	9.1 (3)
N1—C11—C12—C13	−176.9 (2)	O5—C5—C6—C7	145.9 (2)
C11—C12—C13—C14	0.8 (4)	C4A—C5—C6—C7	−35.0 (3)
C12—C13—C14—C15	−1.6 (4)	C5—C6—C7—C72	173.2 (2)
C12—C13—C14—Cl14	177.65 (19)	C5—C6—C7—C8	54.1 (2)
C13—C14—C15—C16	1.1 (4)	C5—C6—C7—C71	−66.9 (2)
Cl14—C14—C15—C16	−178.1 (2)	C72—C7—C8—C8A	−168.0 (2)
C12—C11—C16—C15	−0.8 (4)	C6—C7—C8—C8A	−49.2 (2)
N1—C11—C16—C15	176.5 (2)	C71—C7—C8—C8A	71.2 (3)
C14—C15—C16—C11	0.1 (4)	C4—C4A—C8A—N9	−3.0 (3)
C9A—N1—N2—C3	−0.2 (2)	C5—C4A—C8A—N9	176.6 (2)
C11—N1—N2—C3	−175.16 (18)	C4—C4A—C8A—C8	175.6 (2)
N1—N2—C3—C3A	−0.9 (2)	C5—C4A—C8A—C8	−4.8 (3)
N1—N2—C3—C31	174.51 (18)	C7—C8—C8A—N9	−155.06 (19)
N2—C3—C31—C33	131.2 (2)	C7—C8—C8A—C4A	26.2 (3)
C3A—C3—C31—C33	−54.4 (3)	C4A—C8A—N9—C9A	2.4 (3)
N2—C3—C31—C32	10.7 (3)	C8—C8A—N9—C9A	−176.26 (19)
C3A—C3—C31—C32	−174.8 (2)	C8A—N9—C9A—N1	179.0 (2)
N2—C3—C31—C34	−109.0 (2)	C8A—N9—C9A—C3A	0.6 (3)
C3A—C3—C31—C34	65.4 (3)	N2—N1—C9A—N9	−177.52 (19)
N2—C3—C3A—C4	−178.6 (2)	C11—N1—C9A—N9	−3.5 (4)
C31—C3—C3A—C4	6.5 (4)	N2—N1—C9A—C3A	1.2 (2)

N2—C3—C3A—C9A	1.6 (2)	C11—N1—C9A—C3A	175.2 (2)
C31—C3—C3A—C9A	−173.3 (2)	C4—C3A—C9A—N9	−2.8 (3)
C9A—C3A—C4—C4A	2.0 (3)	C3—C3A—C9A—N9	177.0 (2)
C3—C3A—C4—C4A	−177.7 (2)	C4—C3A—C9A—N1	178.53 (18)
C3A—C4—C4A—C8A	0.6 (3)	C3—C3A—C9A—N1	−1.6 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*A*···*Cg*1^{i}	0.97	2.77	3.649 (3)	151
C8—H8*A*···*Cg*2^{i}	0.97	2.82	3.768 (3)	165

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.