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Different intra- and intermolecular hydrogen-bonding patterns in (3*S*,4a*S*,8a*S*)-2-[(2*R*,3*S*)-3-(2,5-X_2-benzamido)-2-(2,5-X_2-benzoyloxy)-4-phenylbutyl]-*N*-*tert*-butyldecahydroisoquinoline-3-carboxamides (*X* = H or Cl): compounds with moderate aspartyl protease inhibition activity

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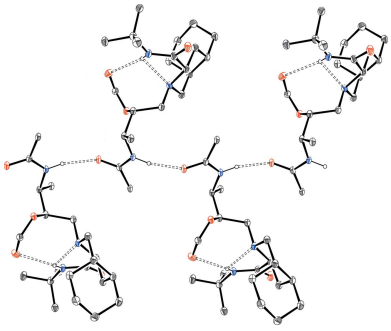
The crystal structures of (3*S*,4a*S*,8a*S*)-2-[(2*R*,3*S*)-3-benzamido-2-benzoyloxy-4-phenylbutyl]-*N*-*tert*-butyldecahydroisoquinoline-3-carboxamide, $C_{38}H_{47}N_3O_4$, (I), and (3*S*,4a*S*,8a*S*)-2-[(2*R*,3*S*)-3-(2,5-dichlorobenzamido)-2-(2,5-dichlorobenzoyloxy)-4-phenylbutyl]-*N*-*tert*-butyldecahydroisoquinoline-3-carboxamide, $C_{38}H_{43}Cl_4N_3O_4$, (II), are described. Despite their chemical similarity, they adopt different conformations in the solid state: (I) features a bifurcated intramolecular N—H···(N,O) hydrogen bond from the *tert*-butylamide NH group to the piperidine N atom and the benzoate O atom, whereas (II) has an intramolecular N—H···O link from the benzamide NH group to the *tert*-butylamide O atom. In the crystal of (I), molecules are linked by *C*(4) amide N—H···O hydrogen bonds into chains propagating in the [010] direction, with both donor and acceptor parts of the benzamide group. In the extended structure of (II), *C*(11) N—H···O chains propagating in the [010] direction arise, with the donor being the *tert*-butylamide NH group and the acceptor being the O atom of the benzamide group.

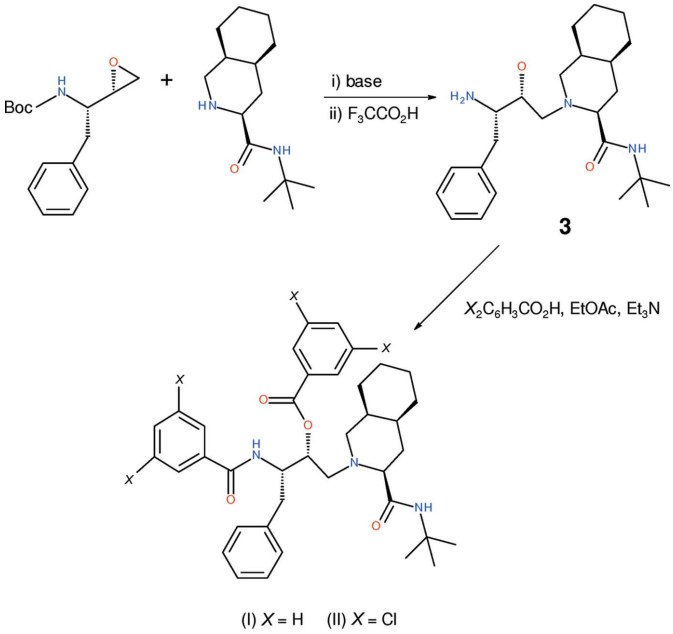
1. Chemical context

Malaria remains one of the most devastating infectious diseases with over 200 million cases and more than 600 000 deaths each year – primarily children under the age of five in sub-Saharan Africa. There is an urgent need for effective drugs with new mechanisms of action, due to the high rate of mutation of the parasite, which leads to the development of resistance of current drugs.

One of the critical stages of the life cycle of the parasite during human infection is the degradation of haemoglobin, which provides nutrients for its growth and maturation (Coombs *et al.*, 2001). Plasmepsins are a family of aspartic proteases involved in the degradation of human haemoglobin by Plasmodium falciparum (Huizing *et al.*, 2015). As the parasite needs the resulting amino acid building blocks for its growth and development, plasmepsins are an important anti-malarial drug target. Secondary alcohols (Muthas *et al.*, 2005; Ersmark *et al.*, 2006) and tertiary alcohols (Motwani *et al.*, 2015) have been successfully used to develop potent inhibitors of these enzymes.



Cunico *et al.* (2008) reported the moderate *in vitro* anti-malarial activities of the products of reactions of the 2-aminoethyl compound, **3** (see Scheme 1) with various sulfonyl chlorides and acyl chlorides. In the present article, we report the crystal structures of two compounds (see Scheme 2), $C_{38}H_{47}N_3O_4$, (I), and $C_{38}H_{43}Cl_4N_3O_4$, (II), obtained in that study from reactions with acyl chlorides.



Scheme 1

2. Structural commentary

Compound (I) crystallizes in the space group $P2_1$ with a single molecule in the asymmetric unit (Fig. 1). The absolute structure was not definitively established based on refinement of the Flack parameter (Parsons *et al.*, 2013) and the configurations of the stereogenic centres (C2 *R*, C3 *S*, C7 *S*, C9 *S*, C14 *S*) were set to match those in (II): they are those expected based on the known starting materials. Each atom in the C1—C2—C3—C4 'backbone' of (I) bears a different substituent: C1 is attached to a piperidine+cyclohexane fused-ring system, which in turn bears a *tert*-butylamide group. C2 is attached to a benzoate group and C3 bears a benzamide group. Finally, C4 is attached to a simple phenyl ring, *i.e.* a benzyl group. Some key torsion angles are presented in Table 1. These show that with respect to the C2—C3 bond, the C1 + C4, C1 + N3 and N3 + O4 pairings are *gauche*, whereas the C4 + O4 atoms are mutually *anti*. In terms of the H atoms, H2 is *anti* to N3 (171°) and H3 is *anti* to C1 (176°); the *gauche* torsion angle between the H atoms is 54°. The N1—C1—C2—C3 torsion angle of 170.4 (3)° indicates an *anti* conformation and the N1/C7/C8/C9/C14/C5 and C9–C14 rings have a *cis*-fused junction (H9—C9—C14—H14 = −52°). The amide torsion angles C3—N3—C5—C27 and C17—N2—C16—C7 are −178.3 (3) and −164.7 (4)°, respectively, which reflect the expected near-planar conformations for these groups. The dihedral angles between the aromatic rings C21–C26 (*A*), C27–C32 (*B*) and C33–C38 (*C*) are *A*/*B* = 85.7 (2), *A*/*C* = 79.2 (2) and *B*/*C* = 17.3 (2)°. The conformation of (I) is supported by a bifurcated intramolecular N—H···(N,O) hydrogen bond (Table 2) arising from the *tert*-butylamide group: the acceptor atoms are the N atom of the piperidine ring and the O atom of the C=O group of the benzoate group. The bifurcated bond is very asymmetric in terms of angles and the H···O link is long, but given that the assemblage is close to planar (bond-angle sum for the H atom = 353°), we regard it as being just significant.

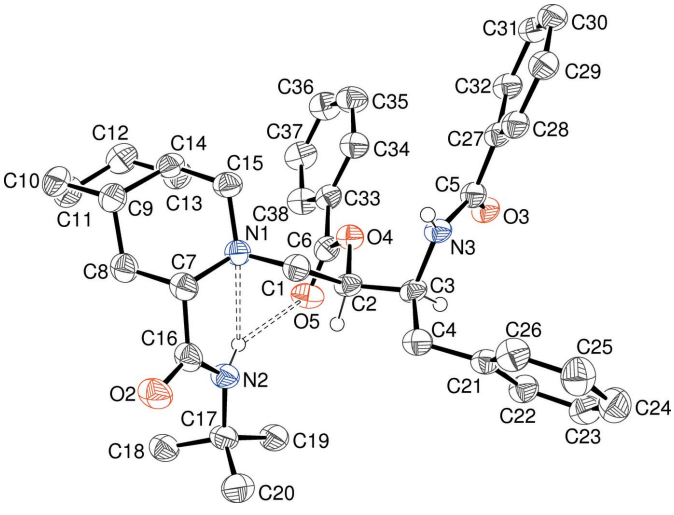
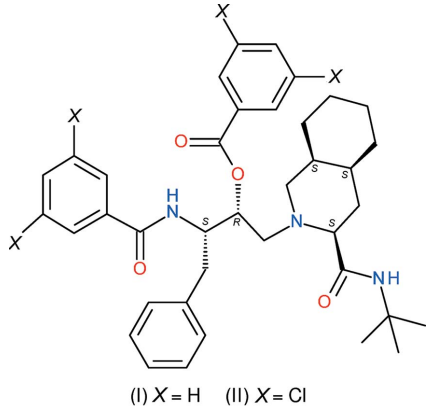


Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids, with most H atoms omitted for clarity. The bifurcated intramolecular hydrogen bond is shown as a double-dashed line.

Table 1
Selected torsion angles (°) for (I).

N1—C1—C2—C3	170.4 (3)	C1—C2—C3—C4	59.4 (4)
C1—C2—C3—N3	−66.3 (4)	C4—C3—N3—C5	138.6 (4)
O4—C2—C3—C4	178.4 (3)	C3—C2—O4—C6	131.5 (3)



Scheme 2

Compound (II) crystallizes in the space group $P2_12_12_1$ with one molecule in the asymmetric unit (Fig. 2). Here, the absolute structure is very well established (C2 *R*, C3 *S*, C7 *S*, C9 *S*, C14 *S*) and is consistent with the starting materials (Cunico *et al.*, 2008). The C1—C2—C3—C4 backbone bears the equivalent substituents to (I), with the difference that the benzyl and amide rings both bear a pair of Cl atoms at the

Table 2
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1*N*···O5	0.90 (5)	2.55 (5)	3.384 (5)	154 (4)
N2—H1*N*···N1	0.90 (5)	2.32 (5)	2.773 (4)	111 (4)
N3—H3*N*···O3^{i}	0.93 (5)	2.04 (5)	2.929 (4)	161 (4)
C18—H18*B*···O2^{ii}	0.98	2.39	3.310 (5)	157
C20—H20*A*···O2	0.98	2.35	2.963 (6)	120
C29—H29···O5^{i}	0.95	2.58	3.467 (5)	157

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

meta positions. Selected torsion angles for (II) (Table 3) show similarities but also one major difference with respect to (I). In terms of the central C2—C3 bond in (II), the C1 + C4, C1 + N3 and N3 + O4 pairings are *gauche*, whereas the C4 + O4 atoms are mutually *anti*. With respect to the H atoms, H2 is *anti* to N3 (−175°) and H3 is *anti* to C1 (−166°); the torsion angle between the H atoms is 69°. Thus, the overall conformation of the atoms about the C2—C3 bond in (II) is essentially the same as in (I), although some of the torsion angles differ by as much as 20°. The N1—C1—C2—C3 *gauche* torsion angle of −69.1 (3)° in (II) is quite different to the value for (I) above, whereas the amide torsion angles C3—N3—C5—C27 [180.0 (3)°] and C17—N2—C16—C7 [–177.5 (3)°] in (II) are similar. The dihedral angles between the aromatic rings C21–C26 (*A*), C27–C32 (*B*) and C33–C38 (*C*) are *A*/*B* = 74.84 (17), *A*/*C* = 67.99 (17) and *B*/*C* = 68.91 (15)°: it may be seen that the first two of these values are similar to the equivalent data for (I), but the third value is very different, possibly reflecting a reorientation in (II) to minimize unfavourable steric interactions between the bichlorinated rings. Compound (II) features a completely different intramolecular N—H···O hydrogen bond (Table 4) to (I): in (II), a much shorter (and presumably stronger) bond arises from the benzamide NH group to the *tert*-butylamide O atom, which no doubt correlates with the very different N1—C1—C2—C3 torsion angles for (I) and (II) already mentioned.

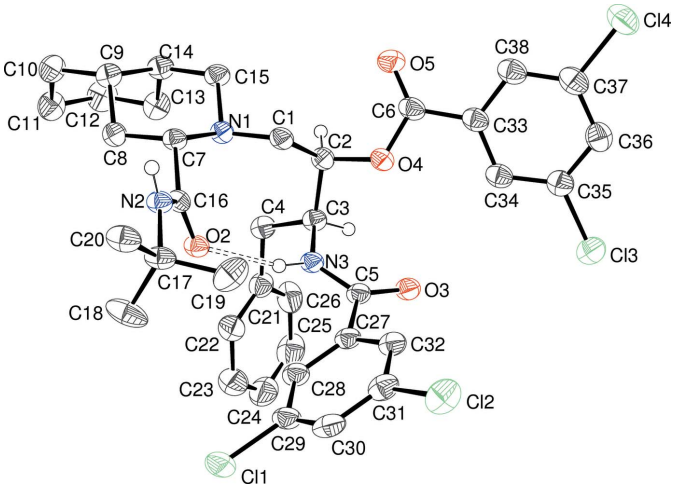


Figure 2
The asymmetric unit of (II), showing 50% probability displacement ellipsoids, with most H atoms omitted for clarity. The intramolecular hydrogen bond is shown as a double-dashed line.

Table 3
Selected torsion angles (°) for (II).

N1—C1—C2—C3	−69.1 (3)	C1—C2—C3—C4	74.4 (3)
C1—C2—C3—N3	−49.5 (3)	C4—C3—N3—C5	136.6 (3)
O4—C2—C3—C4	−167.3 (2)	C3—C2—O4—C6	158.0 (2)

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1*N*···O3^{i}	0.84 (4)	2.13 (4)	2.931 (3)	160 (3)
N3—H2*N*···O2	0.88 (4)	1.99 (4)	2.834 (3)	159 (3)
C4—H4*A*···N1	0.99	2.55	3.149 (4)	119
C18—H18*A*···O2	0.98	2.36	2.975 (4)	120
C34—H34···O3	0.95	2.40	3.324 (4)	163

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

3. Supramolecular features

In the crystal of (I), molecules are linked by classical $C(4)$ amide N—H···O hydrogen bonds into chains propagating in the [010] direction, with adjacent molecules related by the 2_1 screw axis. Both donor and acceptor are part of the benzamide group (Fig. 3). Two weak C—H···O interactions are also observed.

In the extended structure of (II), $C(11)$ [010] N—H···O chains arise, with the donor being the *tert*-butylamide NH group and the acceptor being the O atom of the benzamide ring (Fig. 4). Adjacent molecules are again related by a 2_1 screw axis.

In short, for (I), the *tert*-butylamide NH moiety forms an intramolecular hydrogen bond and the benzamide NH group forms an intermolecular link, whereas for (II), the situation is reversed: the benzamide NH group forms the intramolecular

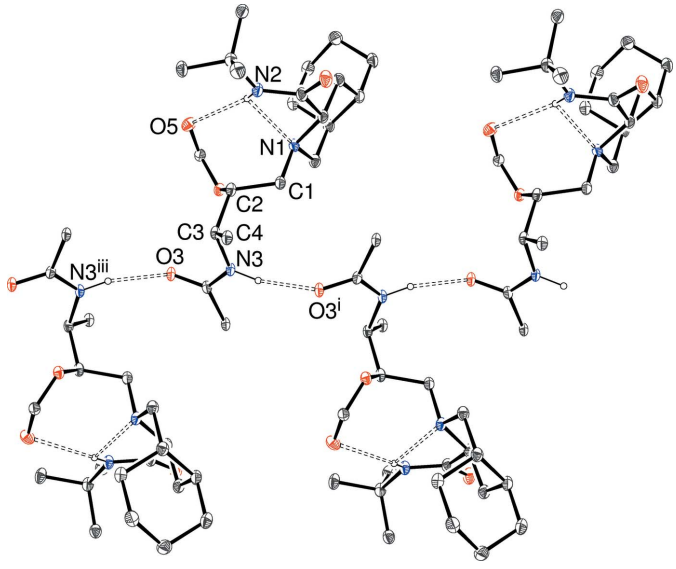


Figure 3
A fragment of a [010] hydrogen-bonded chain in (I), showing 20% probability displacement ellipsoids; the pendant rings and C-bound H atoms have been omitted for clarity. [Symmetry code as in Table 2; additionally (iii) $-x, y-\frac{1}{2}, -z$.]

Table 5
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{38}H_{47}N_3O_4$	$C_{38}H_{43}Cl_4N_3O_4$
M_r	609.78	747.55
Crystal system, space group	Monoclinic, $P2_1$	Orthorhombic, $P2_12_12_1$
Temperature (K)	100	100
a, b, c (Å)	11.4866 (3), 9.4448 (2), 16.8257 (5)	10.4539 (1), 15.1917 (1), 24.3677 (2)
α, β, γ (°)	90, 109.227 (3), 90	90, 90, 90
V (Å^3)	1723.58 (8)	3869.90 (6)
Z	2	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	0.60	3.12
Crystal size (mm)	0.52 × 0.15 × 0.05	0.25 × 0.20 × 0.04
Data collection		
Diffractometer	Rigaku Mercury CCD	Rigaku Mercury CCD
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2004)	Multi-scan (*SADABS*; Sheldrick, 2004)
T_{min}, T_{max}	0.654, 0.971	0.611, 0.886
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	24074, 5349, 4547	44109, 7278, 7140
R_{int}	0.068	0.046
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.610	0.610
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.151, 1.07	0.038, 0.100, 1.05
No. of reflections	5349	7278
No. of parameters	415	451
No. of restraints	1	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.26	0.28, −0.32
Absolute structure	Flack x determined using 1316 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)	Flack x determined using 3021 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.4 (2)	−0.006 (7)

Computer programs: *CrysAlis PRO* (Rigaku, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

bond and the *tert*-butyl NH group forms the intermolecular link.

4. Database survey

A survey of of the Cambridge Structural Database (Groom *et al.*, 2016: updated to April 2017) for the grouping of atoms about the C1—C2—C3—C4 fragment in (I) and (II) yielded 24 matches. The most similar are the isostructural halide salts YURSUB and YURTAI of the anti-HIV drug saquinavir mesylate (Fandaruff *et al.*, 2015), which also act as protease inhibitors. The other hits have little similarity to the title compounds.

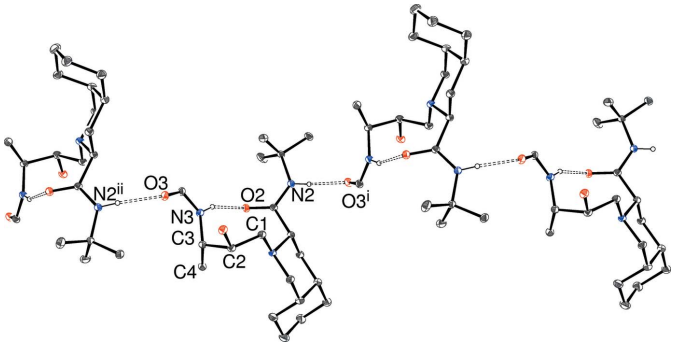


Figure 4
A fragment of a [010] hydrogen-bonded chain in (II), showing 20% probability displacement ellipsoids; the pendant rings and C-bound H atoms have been omitted for clarity. [Symmetry code as in Table 4; additionally (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.]

5. Synthesis and crystallisation

As summarized in Scheme 1, compounds (I) and (II) were prepared as described previously (Cunico *et al.*, 2008) and recrystallized from methanol solution. (I): colourless needles, m.p. 475–476 K, ESI–HRMS (M + H): calculated for $C_{38}H_{48}N_3O_4$: 610.3645, found: 610.3638. (II): colourless slabs, m.p. 459–460 K, ESI–HRMS (M + H): calculated for $C_{38}H_{44}{}^{35}Cl_4N_3O_4$: 746.2086, found: 746.2078.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. The N-bound H atoms were located in difference maps and their positions were freely refined. The C-bound H atoms were placed geometrically (C—H = 0.95–1.00 Å) and refined as riding atoms. The

constraint $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C) was applied in all cases. The methyl groups were allowed to rotate, but not to tip, to best fit the electron density.

Acknowledgements

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supporting information

Acta Cryst. (2017). E**73**, 913-917 [https://doi.org/10.1107/S2056989017007800]

Different intra- and intermolecular hydrogen-bonding patterns in (3*S*,4a*S*,8a*S*)-2-[(2*R*,3*S*)-3-(2,5-X_2-benzamido)-2-(2,5-X_2-benzoyloxy)-4-phenylbutyl]-*N*-*tert*-butyldecahydroisoquinoline-3-carboxamides (*X* = H or Cl): compounds with moderate aspartyl protease inhibition activity

Wilson Cunico, Maria de Lourdes G. Ferreira, James L. Wardell and William T. A. Harrison

Computing details

For both compounds, data collection: *CrysAlis PRO* (Rigaku, 2014); cell refinement: *CrysAlis PRO* (Rigaku, 2014); data reduction: *CrysAlis PRO* (Rigaku, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3* (Farrugia, 2012); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(I) (3*S*,4a*S*,8a*S*)-2-[(2*R*,3*S*)-3-Benzamido-2-benzoyloxy-4-phenylbutyl]-*N*-*tert*-butyldecahydroisoquinoline-3-carboxamide

Crystal data

$C_{38}H_{47}N_3O_4$
M_r = 609.78
Monoclinic, $P2_1$
a = 11.4866 (3) Å
b = 9.4448 (2) Å
c = 16.8257 (5) Å
β = 109.227 (3)°
V = 1723.58 (8) Å^3
Z = 2
$F(000)$ = 656
D_x = 1.175 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 8813 reflections
θ = 5.4–69.6°
μ = 0.60 mm^{-1}
T = 100 K
Needle, colourless
0.52 × 0.15 × 0.05 mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
T_{min} = 0.654, T_{max} = 0.971
24074 measured reflections
5349 independent reflections
4547 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 70.1°, θ_{min} = 2.8°
h = −14→13
k = −11→9
l = −20→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.151
S = 1.07
5349 reflections
415 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0894P)^2 + 0.2672P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Absolute structure: Flack *x* determined using 1316 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.4 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1094 (4)	0.3195 (4)	0.1956 (2)	0.0347 (8)
H1A	0.1950	0.3530	0.2069	0.042*
H1B	0.0566	0.3672	0.1438	0.042*
C2	0.1046 (3)	0.1585 (4)	0.1806 (2)	0.0322 (8)
H2	0.1668	0.1113	0.2295	0.039*
C3	0.1262 (3)	0.1138 (4)	0.0997 (2)	0.0310 (8)
H3	0.1265	0.0079	0.0988	0.037*
C4	0.2513 (3)	0.1625 (4)	0.0978 (2)	0.0353 (8)
H4A	0.2491	0.2665	0.0900	0.042*
H4B	0.3140	0.1414	0.1530	0.042*
C5	−0.0678 (3)	0.0764 (4)	−0.0162 (2)	0.0322 (8)
C6	−0.0309 (4)	0.0410 (4)	0.2415 (2)	0.0344 (9)
C7	0.1555 (3)	0.4553 (4)	0.3252 (2)	0.0362 (8)
H7	0.1668	0.5406	0.2933	0.043*
C8	0.1086 (4)	0.5037 (5)	0.3955 (3)	0.0408 (9)
H8A	0.1674	0.5733	0.4311	0.049*
H8B	0.1060	0.4213	0.4312	0.049*
C9	−0.0194 (4)	0.5709 (5)	0.3631 (3)	0.0408 (9)
H9	−0.0135	0.6587	0.3314	0.049*
C10	−0.0665 (4)	0.6127 (5)	0.4343 (3)	0.0469 (10)
H10A	−0.1408	0.6726	0.4114	0.056*
H10B	−0.0027	0.6698	0.4759	0.056*
C11	−0.0984 (4)	0.4852 (5)	0.4784 (3)	0.0510 (12)
H11A	−0.0224	0.4307	0.5069	0.061*
H11B	−0.1330	0.5179	0.5218	0.061*
C12	−0.1913 (4)	0.3899 (5)	0.4161 (3)	0.0497 (11)
H12A	−0.2702	0.4415	0.3917	0.060*
H12B	−0.2072	0.3054	0.4457	0.060*
C13	−0.1435 (4)	0.3435 (5)	0.3454 (3)	0.0430 (10)
H13A	−0.0704	0.2821	0.3691	0.052*
H13B	−0.2079	0.2871	0.3038	0.052*
C14	−0.1088 (4)	0.4686 (5)	0.3014 (2)	0.0381 (9)
H14	−0.1863	0.5225	0.2732	0.046*

C15	−0.0557 (3)	0.4256 (4)	0.2330 (3)	0.0379 (9)
H15A	−0.1128	0.3580	0.1944	0.046*
H15B	−0.0501	0.5105	0.1999	0.046*
C16	0.2805 (4)	0.3896 (5)	0.3683 (3)	0.0389 (9)
C17	0.3889 (4)	0.1660 (5)	0.4317 (3)	0.0395 (9)
C18	0.4024 (4)	0.1975 (5)	0.5232 (3)	0.0430 (10)
H18A	0.4144	0.2995	0.5336	0.064*
H18B	0.4737	0.1462	0.5604	0.064*
H18C	0.3277	0.1673	0.5345	0.064*
C19	0.3574 (4)	0.0085 (5)	0.4131 (3)	0.0484 (11)
H19A	0.2764	−0.0113	0.4182	0.073*
H19B	0.4200	−0.0499	0.4533	0.073*
H19C	0.3556	−0.0135	0.3557	0.073*
C20	0.5072 (4)	0.1970 (6)	0.4129 (3)	0.0516 (12)
H20A	0.5284	0.2973	0.4235	0.077*
H20B	0.4952	0.1751	0.3538	0.077*
H20C	0.5742	0.1386	0.4492	0.077*
C21	0.2909 (3)	0.0951 (4)	0.0296 (2)	0.0345 (8)
C22	0.3112 (4)	−0.0514 (5)	0.0303 (3)	0.0381 (9)
H22	0.2960	−0.1089	0.0721	0.046*
C23	0.3533 (4)	−0.1124 (5)	−0.0299 (3)	0.0449 (10)
H23	0.3680	−0.2115	−0.0282	0.054*
C24	0.3743 (4)	−0.0325 (6)	−0.0919 (3)	0.0508 (11)
H24	0.4034	−0.0756	−0.1328	0.061*
C25	0.3523 (4)	0.1131 (6)	−0.0941 (3)	0.0523 (11)
H25	0.3657	0.1698	−0.1369	0.063*
C26	0.3109 (4)	0.1745 (5)	−0.0337 (3)	0.0444 (10)
H26	0.2959	0.2736	−0.0358	0.053*
C27	−0.1602 (3)	0.1360 (4)	−0.0933 (2)	0.0315 (8)
C28	−0.1291 (4)	0.2311 (4)	−0.1467 (2)	0.0335 (8)
H28	−0.0470	0.2653	−0.1323	0.040*
C29	−0.2175 (4)	0.2756 (4)	−0.2206 (3)	0.0370 (9)
H29	−0.1955	0.3395	−0.2568	0.044*
C30	−0.3380 (4)	0.2273 (4)	−0.2417 (3)	0.0386 (9)
H30	−0.3984	0.2582	−0.2923	0.046*
C31	−0.3700 (4)	0.1339 (4)	−0.1889 (3)	0.0382 (9)
H31	−0.4525	0.1011	−0.2033	0.046*
C32	−0.2825 (3)	0.0883 (4)	−0.1156 (3)	0.0353 (8)
H32	−0.3052	0.0240	−0.0798	0.042*
C33	−0.1626 (3)	0.0098 (4)	0.2289 (2)	0.0346 (8)
C34	−0.2557 (4)	0.0418 (5)	0.1540 (3)	0.0400 (9)
H34	−0.2354	0.0806	0.1081	0.048*
C35	−0.3772 (4)	0.0176 (5)	0.1463 (3)	0.0481 (11)
H35	−0.4406	0.0405	0.0952	0.058*
C36	−0.4071 (4)	−0.0400 (5)	0.2126 (3)	0.0493 (11)
H36	−0.4910	−0.0566	0.2068	0.059*
C37	−0.3155 (4)	−0.0735 (5)	0.2872 (3)	0.0506 (11)
H37	−0.3363	−0.1127	0.3328	0.061*

C38	−0.1931 (4)	−0.0496 (5)	0.2950 (3)	0.0449 (10)
H38	−0.1298	−0.0738	0.3458	0.054*
N1	0.0677 (3)	0.3598 (3)	0.26662 (19)	0.0332 (7)
N2	0.2845 (3)	0.2467 (4)	0.3755 (2)	0.0369 (8)
H1N	0.208 (4)	0.209 (6)	0.358 (3)	0.044*
N3	0.0279 (3)	0.1602 (3)	0.02500 (19)	0.0301 (7)
H3N	0.027 (4)	0.252 (6)	0.005 (3)	0.036*
O2	0.3711 (3)	0.4674 (3)	0.3992 (2)	0.0506 (8)
O3	−0.0799 (2)	−0.0457 (3)	0.00716 (17)	0.0344 (6)
O4	−0.0177 (2)	0.1111 (3)	0.17533 (16)	0.0329 (6)
O5	0.0540 (2)	0.0080 (4)	0.30361 (18)	0.0449 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.036 (2)	0.0253 (19)	0.040 (2)	−0.0010 (16)	0.0092 (16)	0.0009 (15)
C2	0.0261 (17)	0.0251 (19)	0.043 (2)	0.0008 (15)	0.0086 (15)	0.0016 (16)
C3	0.0292 (17)	0.0212 (18)	0.0410 (19)	0.0052 (15)	0.0094 (14)	0.0021 (15)
C4	0.0318 (18)	0.027 (2)	0.045 (2)	−0.0015 (16)	0.0095 (15)	0.0010 (17)
C5	0.0328 (19)	0.023 (2)	0.039 (2)	−0.0003 (15)	0.0090 (15)	−0.0032 (15)
C6	0.037 (2)	0.030 (2)	0.038 (2)	0.0008 (16)	0.0138 (17)	0.0014 (16)
C7	0.037 (2)	0.0251 (19)	0.042 (2)	−0.0047 (16)	0.0062 (16)	0.0014 (17)
C8	0.042 (2)	0.031 (2)	0.045 (2)	−0.0043 (18)	0.0092 (17)	−0.0029 (17)
C9	0.045 (2)	0.027 (2)	0.047 (2)	0.0004 (18)	0.0100 (18)	0.0000 (17)
C10	0.050 (2)	0.032 (2)	0.057 (3)	0.006 (2)	0.015 (2)	−0.005 (2)
C11	0.057 (3)	0.049 (3)	0.048 (2)	0.008 (2)	0.020 (2)	−0.002 (2)
C12	0.052 (3)	0.043 (3)	0.059 (3)	0.002 (2)	0.024 (2)	0.005 (2)
C13	0.043 (2)	0.033 (2)	0.053 (2)	−0.0001 (18)	0.0147 (18)	−0.0012 (19)
C14	0.035 (2)	0.033 (2)	0.044 (2)	0.0068 (17)	0.0097 (16)	0.0013 (17)
C15	0.033 (2)	0.032 (2)	0.045 (2)	0.0014 (16)	0.0073 (16)	0.0013 (16)
C16	0.039 (2)	0.030 (2)	0.043 (2)	−0.0064 (17)	0.0076 (17)	−0.0028 (16)
C17	0.0319 (19)	0.033 (2)	0.046 (2)	0.0001 (17)	0.0033 (16)	−0.0008 (18)
C18	0.041 (2)	0.035 (2)	0.047 (2)	0.0005 (18)	0.0055 (17)	0.0029 (18)
C19	0.044 (2)	0.030 (2)	0.059 (3)	0.0076 (19)	0.001 (2)	−0.004 (2)
C20	0.041 (2)	0.051 (3)	0.060 (3)	0.003 (2)	0.013 (2)	−0.004 (2)
C21	0.0264 (17)	0.032 (2)	0.043 (2)	−0.0004 (16)	0.0082 (14)	0.0002 (17)
C22	0.036 (2)	0.031 (2)	0.047 (2)	−0.0002 (17)	0.0132 (16)	−0.0019 (18)
C23	0.036 (2)	0.036 (2)	0.060 (3)	−0.0019 (18)	0.0131 (19)	−0.007 (2)
C24	0.048 (2)	0.050 (3)	0.057 (3)	−0.008 (2)	0.020 (2)	−0.011 (2)
C25	0.059 (3)	0.055 (3)	0.050 (2)	−0.006 (2)	0.026 (2)	0.001 (2)
C26	0.047 (2)	0.035 (2)	0.052 (2)	−0.0017 (19)	0.0174 (18)	0.003 (2)
C27	0.0311 (18)	0.0192 (18)	0.042 (2)	0.0015 (15)	0.0095 (15)	−0.0018 (15)
C28	0.0353 (19)	0.0226 (18)	0.041 (2)	0.0007 (16)	0.0099 (16)	−0.0031 (16)
C29	0.043 (2)	0.023 (2)	0.042 (2)	0.0030 (16)	0.0095 (17)	0.0019 (16)
C30	0.038 (2)	0.028 (2)	0.043 (2)	0.0039 (17)	0.0031 (17)	−0.0025 (17)
C31	0.0310 (18)	0.031 (2)	0.048 (2)	0.0012 (16)	0.0063 (16)	−0.0010 (17)
C32	0.0333 (19)	0.0219 (19)	0.048 (2)	−0.0015 (16)	0.0099 (16)	−0.0005 (16)
C33	0.0347 (19)	0.0245 (18)	0.046 (2)	−0.0012 (16)	0.0156 (16)	0.0015 (16)

C34	0.038 (2)	0.034 (2)	0.047 (2)	−0.0014 (17)	0.0115 (17)	0.0035 (18)
C35	0.036 (2)	0.045 (3)	0.059 (3)	−0.004 (2)	0.0104 (19)	0.003 (2)
C36	0.040 (2)	0.043 (3)	0.065 (3)	−0.008 (2)	0.018 (2)	0.004 (2)
C37	0.047 (2)	0.047 (3)	0.062 (3)	−0.002 (2)	0.024 (2)	0.010 (2)
C38	0.042 (2)	0.042 (3)	0.051 (2)	−0.001 (2)	0.0156 (18)	0.008 (2)
N1	0.0307 (16)	0.0262 (17)	0.0396 (17)	−0.0022 (13)	0.0075 (13)	−0.0028 (13)
N2	0.0325 (16)	0.0274 (18)	0.0445 (18)	0.0014 (14)	0.0040 (14)	0.0008 (14)
N3	0.0289 (15)	0.0188 (16)	0.0398 (16)	0.0015 (12)	0.0075 (12)	0.0012 (13)
O2	0.0407 (16)	0.0357 (17)	0.0652 (19)	−0.0084 (14)	0.0038 (14)	−0.0029 (15)
O3	0.0356 (13)	0.0185 (13)	0.0470 (15)	0.0024 (11)	0.0111 (11)	0.0009 (11)
O4	0.0303 (12)	0.0283 (14)	0.0391 (13)	−0.0033 (11)	0.0100 (10)	0.0022 (11)
O5	0.0361 (15)	0.0494 (18)	0.0457 (16)	0.0009 (14)	0.0087 (12)	0.0121 (14)

Geometric parameters (Å, º)

C1—N1	1.476 (5)	C17—N2	1.472 (5)
C1—C2	1.539 (5)	C17—C20	1.522 (6)
C1—H1A	0.9900	C17—C18	1.526 (6)
C1—H1B	0.9900	C17—C19	1.538 (6)
C2—O4	1.449 (4)	C18—H18A	0.9800
C2—C3	1.521 (5)	C18—H18B	0.9800
C2—H2	1.0000	C18—H18C	0.9800
C3—N3	1.453 (4)	C19—H19A	0.9800
C3—C4	1.519 (5)	C19—H19B	0.9800
C3—H3	1.0000	C19—H19C	0.9800
C4—C21	1.507 (6)	C20—H20A	0.9800
C4—H4A	0.9900	C20—H20B	0.9800
C4—H4B	0.9900	C20—H20C	0.9800
C5—O3	1.241 (5)	C21—C26	1.383 (6)
C5—N3	1.346 (5)	C21—C22	1.403 (6)
C5—C27	1.490 (5)	C22—C23	1.383 (6)
C6—O5	1.213 (5)	C22—H22	0.9500
C6—O4	1.346 (5)	C23—C24	1.372 (7)
C6—C33	1.486 (5)	C23—H23	0.9500
C7—N1	1.465 (5)	C24—C25	1.397 (8)
C7—C16	1.512 (6)	C24—H24	0.9500
C7—C8	1.522 (6)	C25—C26	1.383 (7)
C7—H7	1.0000	C25—H25	0.9500
C8—C9	1.527 (6)	C26—H26	0.9500
C8—H8A	0.9900	C27—C28	1.397 (5)
C8—H8B	0.9900	C27—C32	1.403 (5)
C9—C10	1.520 (6)	C28—C29	1.386 (5)
C9—C14	1.537 (6)	C28—H28	0.9500
C9—H9	1.0000	C29—C30	1.388 (6)
C10—C11	1.522 (7)	C29—H29	0.9500
C10—H10A	0.9900	C30—C31	1.384 (6)
C10—H10B	0.9900	C30—H30	0.9500
C11—C12	1.520 (7)	C31—C32	1.380 (6)

C11—H11A	0.9900	C31—H31	0.9500
C11—H11B	0.9900	C32—H32	0.9500
C12—C13	1.529 (6)	C33—C38	1.390 (6)
C12—H12A	0.9900	C33—C34	1.391 (5)
C12—H12B	0.9900	C34—C35	1.378 (6)
C13—C14	1.516 (6)	C34—H34	0.9500
C13—H13A	0.9900	C35—C36	1.382 (6)
C13—H13B	0.9900	C35—H35	0.9500
C14—C15	1.524 (6)	C36—C37	1.384 (7)
C14—H14	1.0000	C36—H36	0.9500
C15—N1	1.479 (5)	C37—C38	1.387 (6)
C15—H15A	0.9900	C37—H37	0.9500
C15—H15B	0.9900	C38—H38	0.9500
C16—O2	1.240 (5)	N2—H1N	0.90 (5)
C16—N2	1.354 (5)	N3—H3N	0.93 (5)

N1—C1—C2	112.5 (3)	N2—C17—C18	109.8 (3)
N1—C1—H1A	109.1	C20—C17—C18	111.8 (3)
C2—C1—H1A	109.1	N2—C17—C19	106.4 (3)
N1—C1—H1B	109.1	C20—C17—C19	108.1 (4)
C2—C1—H1B	109.1	C18—C17—C19	109.5 (4)
H1A—C1—H1B	107.8	C17—C18—H18A	109.5
O4—C2—C3	107.4 (3)	C17—C18—H18B	109.5
O4—C2—C1	107.3 (3)	H18A—C18—H18B	109.5
C3—C2—C1	114.4 (3)	C17—C18—H18C	109.5
O4—C2—H2	109.2	H18A—C18—H18C	109.5
C3—C2—H2	109.2	H18B—C18—H18C	109.5
C1—C2—H2	109.2	C17—C19—H19A	109.5
N3—C3—C4	111.1 (3)	C17—C19—H19B	109.5
N3—C3—C2	112.4 (3)	H19A—C19—H19B	109.5
C4—C3—C2	111.7 (3)	C17—C19—H19C	109.5
N3—C3—H3	107.1	H19A—C19—H19C	109.5
C4—C3—H3	107.1	H19B—C19—H19C	109.5
C2—C3—H3	107.1	C17—C20—H20A	109.5
C21—C4—C3	114.4 (3)	C17—C20—H20B	109.5
C21—C4—H4A	108.7	H20A—C20—H20B	109.5
C3—C4—H4A	108.7	C17—C20—H20C	109.5
C21—C4—H4B	108.7	H20A—C20—H20C	109.5
C3—C4—H4B	108.7	H20B—C20—H20C	109.5
H4A—C4—H4B	107.6	C26—C21—C22	118.1 (4)
O3—C5—N3	122.8 (3)	C26—C21—C4	121.7 (4)
O3—C5—C27	120.3 (3)	C22—C21—C4	120.1 (4)
N3—C5—C27	116.9 (3)	C23—C22—C21	120.2 (4)
O5—C6—O4	124.2 (4)	C23—C22—H22	119.9
O5—C6—C33	124.3 (4)	C21—C22—H22	119.9
O4—C6—C33	111.5 (3)	C24—C23—C22	121.3 (4)
N1—C7—C16	113.8 (3)	C24—C23—H23	119.4
N1—C7—C8	111.5 (3)	C22—C23—H23	119.4

C16—C7—C8	105.9 (3)	C23—C24—C25	119.0 (4)
N1—C7—H7	108.5	C23—C24—H24	120.5
C16—C7—H7	108.5	C25—C24—H24	120.5
C8—C7—H7	108.5	C26—C25—C24	119.8 (5)
C7—C8—C9	113.1 (3)	C26—C25—H25	120.1
C7—C8—H8A	109.0	C24—C25—H25	120.1
C9—C8—H8A	109.0	C25—C26—C21	121.5 (4)
C7—C8—H8B	109.0	C25—C26—H26	119.2
C9—C8—H8B	109.0	C21—C26—H26	119.2
H8A—C8—H8B	107.8	C28—C27—C32	118.7 (3)
C10—C9—C8	112.2 (3)	C28—C27—C5	122.9 (3)
C10—C9—C14	111.3 (4)	C32—C27—C5	118.3 (3)
C8—C9—C14	109.3 (3)	C29—C28—C27	120.3 (4)
C10—C9—H9	108.0	C29—C28—H28	119.9
C8—C9—H9	108.0	C27—C28—H28	119.9
C14—C9—H9	108.0	C28—C29—C30	120.3 (4)
C9—C10—C11	112.6 (4)	C28—C29—H29	119.9
C9—C10—H10A	109.1	C30—C29—H29	119.9
C11—C10—H10A	109.1	C31—C30—C29	119.8 (4)
C9—C10—H10B	109.1	C31—C30—H30	120.1
C11—C10—H10B	109.1	C29—C30—H30	120.1
H10A—C10—H10B	107.8	C32—C31—C30	120.3 (4)
C12—C11—C10	111.0 (4)	C32—C31—H31	119.9
C12—C11—H11A	109.4	C30—C31—H31	119.9
C10—C11—H11A	109.4	C31—C32—C27	120.6 (4)
C12—C11—H11B	109.4	C31—C32—H32	119.7
C10—C11—H11B	109.4	C27—C32—H32	119.7
H11A—C11—H11B	108.0	C38—C33—C34	119.5 (4)
C11—C12—C13	110.8 (4)	C38—C33—C6	118.6 (3)
C11—C12—H12A	109.5	C34—C33—C6	121.9 (4)
C13—C12—H12A	109.5	C35—C34—C33	120.1 (4)
C11—C12—H12B	109.5	C35—C34—H34	119.9
C13—C12—H12B	109.5	C33—C34—H34	119.9
H12A—C12—H12B	108.1	C34—C35—C36	120.2 (4)
C14—C13—C12	112.1 (4)	C34—C35—H35	119.9
C14—C13—H13A	109.2	C36—C35—H35	119.9
C12—C13—H13A	109.2	C35—C36—C37	120.4 (4)
C14—C13—H13B	109.2	C35—C36—H36	119.8
C12—C13—H13B	109.2	C37—C36—H36	119.8
H13A—C13—H13B	107.9	C36—C37—C38	119.5 (4)
C13—C14—C15	113.3 (4)	C36—C37—H37	120.2
C13—C14—C9	112.6 (3)	C38—C37—H37	120.2
C15—C14—C9	109.4 (3)	C37—C38—C33	120.3 (4)
C13—C14—H14	107.0	C37—C38—H38	119.8
C15—C14—H14	107.0	C33—C38—H38	119.8
C9—C14—H14	107.0	C7—N1—C1	111.6 (3)
N1—C15—C14	113.2 (3)	C7—N1—C15	111.0 (3)
N1—C15—H15A	108.9	C1—N1—C15	108.9 (3)

C14—C15—H15A	108.9	C16—N2—C17	124.8 (3)
N1—C15—H15B	108.9	C16—N2—H1N	111 (3)
C14—C15—H15B	108.9	C17—N2—H1N	120 (3)
H15A—C15—H15B	107.7	C5—N3—C3	122.5 (3)
O2—C16—N2	123.6 (4)	C5—N3—H3N	117 (3)
O2—C16—C7	119.5 (4)	C3—N3—H3N	121 (3)
N2—C16—C7	116.8 (3)	C6—O4—C2	118.2 (3)
N2—C17—C20	111.1 (4)		

N1—C1—C2—O4	51.4 (4)	N3—C5—C27—C32	−151.0 (4)
N1—C1—C2—C3	170.4 (3)	C32—C27—C28—C29	−0.7 (6)
O4—C2—C3—N3	52.7 (4)	C5—C27—C28—C29	175.6 (4)
C1—C2—C3—N3	−66.3 (4)	C27—C28—C29—C30	0.7 (6)
O4—C2—C3—C4	178.4 (3)	C28—C29—C30—C31	−0.2 (6)
C1—C2—C3—C4	59.4 (4)	C29—C30—C31—C32	−0.2 (6)
N3—C3—C4—C21	−66.6 (4)	C30—C31—C32—C27	0.1 (6)
C2—C3—C4—C21	167.0 (3)	C28—C27—C32—C31	0.3 (6)
N1—C7—C8—C9	−54.5 (5)	C5—C27—C32—C31	−176.2 (3)
C16—C7—C8—C9	−178.9 (3)	O5—C6—C33—C38	6.1 (6)
C7—C8—C9—C10	177.6 (4)	O4—C6—C33—C38	−173.6 (4)
C7—C8—C9—C14	53.6 (5)	O5—C6—C33—C34	−175.7 (4)
C8—C9—C10—C11	−70.2 (5)	O4—C6—C33—C34	4.6 (6)
C14—C9—C10—C11	52.6 (5)	C38—C33—C34—C35	1.3 (7)
C9—C10—C11—C12	−55.8 (5)	C6—C33—C34—C35	−176.8 (4)
C10—C11—C12—C13	56.2 (5)	C33—C34—C35—C36	−0.6 (7)
C11—C12—C13—C14	−55.1 (5)	C34—C35—C36—C37	0.1 (8)
C12—C13—C14—C15	177.6 (3)	C35—C36—C37—C38	−0.3 (8)
C12—C13—C14—C9	52.7 (5)	C36—C37—C38—C33	1.0 (7)
C10—C9—C14—C13	−51.0 (5)	C34—C33—C38—C37	−1.5 (7)
C8—C9—C14—C13	73.5 (4)	C6—C33—C38—C37	176.7 (4)
C10—C9—C14—C15	−178.0 (3)	C16—C7—N1—C1	−63.7 (4)
C8—C9—C14—C15	−53.6 (4)	C8—C7—N1—C1	176.5 (3)
C13—C14—C15—N1	−69.4 (4)	C16—C7—N1—C15	174.5 (3)
C9—C14—C15—N1	57.3 (4)	C8—C7—N1—C15	54.8 (4)
N1—C7—C16—O2	159.3 (4)	C2—C1—N1—C7	130.4 (3)
C8—C7—C16—O2	−77.9 (5)	C2—C1—N1—C15	−106.6 (3)
N1—C7—C16—N2	−25.0 (5)	C14—C15—N1—C7	−57.9 (4)
C8—C7—C16—N2	97.9 (4)	C14—C15—N1—C1	178.8 (3)
C3—C4—C21—C26	117.9 (4)	O2—C16—N2—C17	10.8 (7)
C3—C4—C21—C22	−63.6 (5)	C7—C16—N2—C17	−164.7 (4)
C26—C21—C22—C23	1.7 (6)	C20—C17—N2—C16	−57.7 (6)
C4—C21—C22—C23	−176.8 (3)	C18—C17—N2—C16	66.5 (5)
C21—C22—C23—C24	−1.0 (6)	C19—C17—N2—C16	−175.1 (4)
C22—C23—C24—C25	−0.1 (7)	O3—C5—N3—C3	1.0 (6)
C23—C24—C25—C26	0.5 (7)	C27—C5—N3—C3	−178.3 (3)
C24—C25—C26—C21	0.2 (7)	C4—C3—N3—C5	138.6 (4)
C22—C21—C26—C25	−1.3 (6)	C2—C3—N3—C5	−95.4 (4)
C4—C21—C26—C25	177.2 (4)	O5—C6—O4—C2	−4.2 (6)

O3—C5—C27—C28	−146.7 (4)	C33—C6—O4—C2	175.5 (3)
N3—C5—C27—C28	32.6 (5)	C3—C2—O4—C6	131.5 (3)
O3—C5—C27—C32	29.7 (5)	C1—C2—O4—C6	−105.0 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*···O5	0.90 (5)	2.55 (5)	3.384 (5)	154 (4)
N2—H1*N*···N1	0.90 (5)	2.32 (5)	2.773 (4)	111 (4)
N3—H3*N*···O3^{i}	0.93 (5)	2.04 (5)	2.929 (4)	161 (4)
C18—H18*B*···O2^{ii}	0.98	2.39	3.310 (5)	157
C20—H20*A*···O2	0.98	2.35	2.963 (6)	120
C29—H29···O5^{i}	0.95	2.58	3.467 (5)	157

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

(II) (3*S*,4a*S*,8a*S*)-2-[(2*R*,3*S*)-3-(2,5-Dichlorobenzamido)-2-(2,5-dichlorobenzoyloxy)-4-phenylbutyl]-*N*-*tert*-butyldecahydroisoquinoline-3-carboxamide

Crystal data

$C_{38}H_{43}Cl_4N_3O_4$
M_r = 747.55
Orthorhombic, $P2_12_12_1$
a = 10.4539 (1) Å
b = 15.1917 (1) Å
c = 24.3677 (2) Å
V = 3869.90 (6) Å^3
Z = 4
F(000) = 1568
D_x = 1.283 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 40379 reflections
θ = 3.4–70.0°
μ = 3.12 mm^{-1}
T = 100 K
Slab, colourless
0.25 × 0.20 × 0.04 mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
T_{min} = 0.611, T_{max} = 0.886
44109 measured reflections
7278 independent reflections
7140 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 70.1°, θ_{min} = 3.4°
h = −12→12
k = −15→18
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.100
S = 1.05
7278 reflections
451 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 1.8039P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Absolute structure: Flack *x* determined using 3021 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.006 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3339 (3)	0.3116 (2)	0.28983 (12)	0.0291 (6)
H1A	0.4228	0.3055	0.2764	0.035*
H1B	0.2948	0.3629	0.2712	0.035*
C2	0.2587 (3)	0.2289 (2)	0.27568 (12)	0.0300 (6)
H2	0.1702	0.2335	0.2910	0.036*
C3	0.3188 (3)	0.1407 (2)	0.29311 (12)	0.0291 (6)
H3	0.2717	0.0929	0.2734	0.035*
C4	0.3109 (3)	0.1206 (2)	0.35483 (13)	0.0330 (7)
H4A	0.3595	0.1657	0.3755	0.040*
H4B	0.2205	0.1235	0.3669	0.040*
C5	0.4901 (3)	0.09795 (19)	0.22901 (12)	0.0283 (6)
C6	0.1687 (3)	0.2720 (2)	0.18833 (14)	0.0339 (7)
C7	0.4476 (3)	0.37726 (19)	0.36694 (12)	0.0269 (6)
H7	0.4450	0.4368	0.3495	0.032*
C8	0.4452 (3)	0.3883 (2)	0.42941 (12)	0.0304 (6)
H8A	0.4500	0.3297	0.4471	0.036*
H8B	0.5209	0.4228	0.4411	0.036*
C9	0.3234 (3)	0.4354 (2)	0.44809 (13)	0.0327 (7)
H9	0.3238	0.4952	0.4309	0.039*
C10	0.3151 (4)	0.4484 (2)	0.51013 (14)	0.0399 (7)
H10A	0.3968	0.4735	0.5235	0.048*
H10B	0.2464	0.4913	0.5183	0.048*
C11	0.2880 (4)	0.3630 (3)	0.54077 (14)	0.0455 (8)
H11A	0.2758	0.3760	0.5802	0.055*
H11B	0.3625	0.3232	0.5372	0.055*
C12	0.1691 (4)	0.3171 (3)	0.51853 (16)	0.0503 (9)
H12A	0.0930	0.3542	0.5257	0.060*
H12B	0.1571	0.2603	0.5378	0.060*
C13	0.1815 (3)	0.3005 (2)	0.45668 (14)	0.0407 (8)
H13A	0.2534	0.2596	0.4498	0.049*
H13B	0.1021	0.2727	0.4428	0.049*
C14	0.2048 (3)	0.3865 (2)	0.42625 (13)	0.0347 (7)
H14	0.1289	0.4252	0.4327	0.042*
C15	0.2175 (3)	0.3736 (2)	0.36460 (13)	0.0318 (6)
H15A	0.1428	0.3399	0.3511	0.038*
H15B	0.2164	0.4319	0.3464	0.038*
C16	0.5715 (3)	0.33129 (19)	0.35033 (12)	0.0255 (6)
C17	0.7959 (3)	0.3617 (2)	0.31935 (14)	0.0311 (6)
C18	0.8574 (3)	0.2958 (3)	0.3576 (2)	0.0518 (10)

H18A	0.8061	0.2418	0.3584	0.078*
H18B	0.8622	0.3208	0.3947	0.078*
H18C	0.9439	0.2821	0.3446	0.078*
C19	0.7847 (4)	0.3250 (3)	0.26108 (16)	0.0478 (9)
H19A	0.7327	0.2714	0.2616	0.072*
H19B	0.8702	0.3112	0.2470	0.072*
H19C	0.7441	0.3689	0.2373	0.072*
C20	0.8752 (3)	0.4461 (2)	0.31835 (15)	0.0369 (7)
H20A	0.8331	0.4897	0.2949	0.055*
H20B	0.9606	0.4333	0.3038	0.055*
H20C	0.8829	0.4694	0.3557	0.055*
C21	0.3644 (4)	0.0305 (2)	0.36758 (13)	0.0378 (7)
C22	0.4947 (4)	0.0212 (3)	0.38043 (15)	0.0451 (8)
H22	0.5464	0.0723	0.3843	0.054*
C23	0.5494 (5)	−0.0614 (3)	0.38766 (17)	0.0558 (10)
H23	0.6376	−0.0664	0.3965	0.067*
C24	0.4763 (6)	−0.1352 (3)	0.38198 (17)	0.0620 (12)
H24	0.5144	−0.1916	0.3858	0.074*
C25	0.3478 (5)	−0.1284 (3)	0.37081 (18)	0.0601 (12)
H25	0.2969	−0.1800	0.3679	0.072*
C26	0.2912 (4)	−0.0445 (2)	0.36356 (16)	0.0503 (9)
H26	0.2024	−0.0400	0.3559	0.060*
C27	0.6317 (3)	0.10128 (19)	0.21805 (13)	0.0296 (6)
C28	0.7207 (3)	0.0856 (2)	0.25959 (14)	0.0335 (7)
H28	0.6934	0.0722	0.2958	0.040*
C29	0.8505 (3)	0.0899 (2)	0.24673 (15)	0.0363 (7)
C30	0.8922 (3)	0.1106 (2)	0.19463 (16)	0.0385 (7)
H30	0.9810	0.1148	0.1867	0.046*
C31	0.8013 (3)	0.1254 (2)	0.15382 (15)	0.0365 (7)
C32	0.6711 (3)	0.12013 (19)	0.16489 (13)	0.0318 (6)
H32	0.6100	0.1293	0.1366	0.038*
C33	0.1663 (3)	0.2464 (2)	0.12927 (13)	0.0331 (7)
C34	0.2152 (3)	0.1651 (2)	0.11265 (14)	0.0341 (7)
H34	0.2583	0.1278	0.1379	0.041*
C35	0.1993 (3)	0.1402 (2)	0.05848 (14)	0.0361 (7)
C36	0.1413 (3)	0.1945 (2)	0.02003 (14)	0.0396 (7)
H36	0.1330	0.1769	−0.0172	0.047*
C37	0.0960 (3)	0.2754 (2)	0.03776 (15)	0.0396 (7)
C38	0.1059 (3)	0.3019 (2)	0.09184 (15)	0.0377 (7)
H38	0.0721	0.3570	0.1033	0.045*
N1	0.3352 (2)	0.32700 (16)	0.34911 (10)	0.0269 (5)
N2	0.6672 (2)	0.38628 (17)	0.33856 (10)	0.0271 (5)
H1N	0.651 (4)	0.440 (3)	0.3363 (14)	0.033*
N3	0.4523 (2)	0.13695 (16)	0.27536 (10)	0.0278 (5)
H2N	0.510 (4)	0.166 (2)	0.2945 (15)	0.033*
O2	0.58152 (19)	0.24951 (13)	0.34972 (9)	0.0289 (4)
O3	0.4177 (2)	0.06170 (14)	0.19576 (9)	0.0328 (5)
O4	0.2526 (2)	0.22167 (14)	0.21627 (9)	0.0322 (4)

O5	0.1021 (2)	0.32785 (16)	0.20878 (10)	0.0423 (6)
Cl1	0.96231 (8)	0.07105 (6)	0.29804 (4)	0.0481 (2)
Cl2	0.85139 (8)	0.14928 (6)	0.08777 (4)	0.0480 (2)
Cl3	0.24876 (9)	0.03602 (6)	0.03774 (4)	0.0465 (2)
Cl4	0.01738 (10)	0.34193 (6)	−0.00931 (4)	0.0512 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0196 (12)	0.0323 (15)	0.0355 (15)	0.0010 (11)	−0.0018 (12)	−0.0014 (12)
C2	0.0208 (13)	0.0339 (15)	0.0352 (15)	−0.0008 (12)	−0.0024 (12)	−0.0035 (12)
C3	0.0211 (13)	0.0293 (14)	0.0370 (15)	−0.0019 (11)	0.0016 (12)	−0.0011 (12)
C4	0.0298 (15)	0.0296 (15)	0.0395 (16)	−0.0019 (12)	0.0019 (13)	−0.0014 (13)
C5	0.0230 (14)	0.0251 (13)	0.0369 (15)	0.0010 (11)	−0.0014 (12)	−0.0036 (12)
C6	0.0234 (14)	0.0331 (16)	0.0452 (17)	0.0005 (13)	−0.0064 (13)	0.0006 (13)
C7	0.0178 (13)	0.0267 (14)	0.0362 (15)	−0.0012 (11)	0.0003 (11)	−0.0007 (11)
C8	0.0228 (13)	0.0319 (15)	0.0364 (15)	−0.0038 (12)	−0.0005 (12)	−0.0019 (12)
C9	0.0299 (15)	0.0318 (15)	0.0365 (16)	−0.0012 (13)	0.0037 (13)	0.0006 (12)
C10	0.0424 (18)	0.0403 (18)	0.0370 (16)	−0.0024 (14)	0.0042 (14)	−0.0017 (14)
C11	0.054 (2)	0.047 (2)	0.0358 (17)	−0.0020 (16)	0.0048 (15)	0.0032 (15)
C12	0.052 (2)	0.052 (2)	0.048 (2)	−0.0100 (18)	0.0109 (17)	0.0066 (17)
C13	0.0362 (17)	0.0417 (18)	0.0442 (18)	−0.0095 (14)	0.0065 (15)	0.0009 (15)
C14	0.0253 (14)	0.0387 (17)	0.0401 (17)	0.0016 (13)	0.0059 (12)	−0.0006 (13)
C15	0.0199 (14)	0.0337 (16)	0.0417 (17)	0.0023 (12)	0.0016 (12)	−0.0036 (13)
C16	0.0180 (12)	0.0292 (15)	0.0292 (13)	−0.0016 (11)	−0.0014 (10)	0.0004 (11)
C17	0.0180 (13)	0.0272 (14)	0.0480 (17)	−0.0006 (11)	0.0045 (12)	0.0042 (13)
C18	0.0219 (15)	0.046 (2)	0.088 (3)	−0.0029 (14)	−0.0063 (17)	0.026 (2)
C19	0.049 (2)	0.044 (2)	0.051 (2)	−0.0072 (16)	0.0195 (17)	−0.0077 (16)
C20	0.0225 (14)	0.0325 (16)	0.0557 (19)	−0.0026 (12)	0.0025 (14)	0.0076 (14)
C21	0.0472 (19)	0.0304 (16)	0.0358 (16)	−0.0009 (15)	0.0006 (14)	0.0002 (13)
C22	0.049 (2)	0.0421 (19)	0.0438 (19)	0.0047 (16)	−0.0052 (16)	0.0011 (15)
C23	0.062 (3)	0.053 (2)	0.052 (2)	0.012 (2)	−0.006 (2)	0.0051 (18)
C24	0.096 (4)	0.042 (2)	0.048 (2)	0.016 (2)	−0.006 (2)	0.0015 (17)
C25	0.091 (4)	0.0340 (19)	0.056 (2)	−0.012 (2)	−0.006 (2)	−0.0001 (17)
C26	0.066 (3)	0.0379 (19)	0.0471 (19)	−0.0139 (18)	−0.0119 (18)	0.0062 (16)
C27	0.0217 (14)	0.0256 (14)	0.0416 (16)	0.0008 (11)	0.0001 (12)	−0.0066 (12)
C28	0.0255 (15)	0.0314 (15)	0.0437 (17)	0.0010 (12)	0.0003 (12)	−0.0056 (13)
C29	0.0214 (14)	0.0329 (16)	0.0546 (19)	0.0036 (12)	−0.0048 (14)	−0.0096 (14)
C30	0.0255 (15)	0.0283 (15)	0.062 (2)	0.0003 (12)	0.0058 (14)	−0.0119 (14)
C31	0.0311 (16)	0.0308 (16)	0.0476 (18)	−0.0032 (12)	0.0060 (14)	−0.0076 (14)
C32	0.0256 (14)	0.0263 (14)	0.0436 (17)	−0.0019 (12)	0.0003 (13)	−0.0057 (12)
C33	0.0241 (14)	0.0340 (16)	0.0411 (16)	−0.0003 (13)	−0.0041 (13)	0.0017 (13)
C34	0.0244 (14)	0.0362 (16)	0.0418 (16)	0.0002 (12)	−0.0024 (12)	0.0021 (14)
C35	0.0332 (16)	0.0354 (16)	0.0397 (16)	−0.0014 (13)	−0.0010 (13)	0.0010 (13)
C36	0.0371 (18)	0.0451 (18)	0.0365 (17)	−0.0038 (15)	−0.0013 (14)	0.0021 (14)
C37	0.0325 (16)	0.0402 (18)	0.0460 (18)	−0.0040 (14)	−0.0052 (14)	0.0090 (15)
C38	0.0287 (15)	0.0354 (17)	0.0491 (19)	−0.0012 (13)	−0.0061 (14)	0.0028 (14)
N1	0.0177 (11)	0.0294 (12)	0.0336 (12)	−0.0025 (10)	0.0009 (10)	−0.0021 (10)

N2	0.0201 (11)	0.0249 (12)	0.0364 (13)	−0.0005 (10)	0.0006 (10)	0.0002 (10)
N3	0.0198 (11)	0.0277 (12)	0.0358 (13)	0.0006 (10)	−0.0018 (10)	−0.0039 (10)
O2	0.0217 (9)	0.0253 (10)	0.0395 (11)	−0.0011 (8)	−0.0028 (8)	0.0001 (8)
O3	0.0254 (10)	0.0300 (11)	0.0432 (12)	0.0010 (8)	−0.0046 (9)	−0.0076 (9)
O4	0.0250 (10)	0.0356 (11)	0.0361 (11)	0.0038 (9)	−0.0054 (9)	−0.0030 (9)
O5	0.0332 (11)	0.0429 (13)	0.0507 (14)	0.0121 (10)	−0.0066 (10)	−0.0081 (11)
Cl1	0.0266 (4)	0.0544 (5)	0.0632 (5)	0.0083 (4)	−0.0099 (4)	−0.0119 (4)
Cl2	0.0419 (4)	0.0490 (5)	0.0529 (5)	−0.0093 (4)	0.0139 (4)	−0.0051 (4)
Cl3	0.0514 (5)	0.0428 (4)	0.0452 (4)	0.0061 (4)	0.0023 (4)	−0.0044 (3)
Cl4	0.0557 (5)	0.0457 (5)	0.0523 (5)	0.0007 (4)	−0.0155 (4)	0.0113 (4)

Geometric parameters (Å, º)

C1—N1	1.463 (4)	C17—N2	1.473 (4)
C1—C2	1.522 (4)	C17—C18	1.512 (5)
C1—H1A	0.9900	C17—C20	1.527 (4)
C1—H1B	0.9900	C17—C19	1.530 (5)
C2—O4	1.453 (4)	C18—H18A	0.9800
C2—C3	1.540 (4)	C18—H18B	0.9800
C2—H2	1.0000	C18—H18C	0.9800
C3—N3	1.462 (4)	C19—H19A	0.9800
C3—C4	1.537 (4)	C19—H19B	0.9800
C3—H3	1.0000	C19—H19C	0.9800
C4—C21	1.511 (4)	C20—H20A	0.9800
C4—H4A	0.9900	C20—H20B	0.9800
C4—H4B	0.9900	C20—H20C	0.9800
C5—O3	1.238 (4)	C21—C26	1.376 (5)
C5—N3	1.335 (4)	C21—C22	1.405 (5)
C5—C27	1.505 (4)	C22—C23	1.391 (5)
C6—O5	1.206 (4)	C22—H22	0.9500
C6—O4	1.348 (4)	C23—C24	1.364 (7)
C6—C33	1.491 (5)	C23—H23	0.9500
C7—N1	1.467 (3)	C24—C25	1.374 (8)
C7—C16	1.526 (4)	C24—H24	0.9500
C7—C8	1.532 (4)	C25—C26	1.416 (6)
C7—H7	1.0000	C25—H25	0.9500
C8—C9	1.530 (4)	C26—H26	0.9500
C8—H8A	0.9900	C27—C32	1.389 (5)
C8—H8B	0.9900	C27—C28	1.395 (4)
C9—C10	1.527 (4)	C28—C29	1.395 (4)
C9—C14	1.540 (4)	C28—H28	0.9500
C9—H9	1.0000	C29—C30	1.379 (5)
C10—C11	1.523 (5)	C29—Cl1	1.735 (3)
C10—H10A	0.9900	C30—C31	1.393 (5)
C10—H10B	0.9900	C30—H30	0.9500
C11—C12	1.525 (6)	C31—C32	1.391 (4)
C11—H11A	0.9900	C31—Cl2	1.731 (4)
C11—H11B	0.9900	C32—H32	0.9500

C12—C13	1.534 (5)	C33—C38	1.394 (5)
C12—H12A	0.9900	C33—C34	1.397 (5)
C12—H12B	0.9900	C34—C35	1.383 (5)
C13—C14	1.522 (5)	C34—H34	0.9500
C13—H13A	0.9900	C35—C36	1.388 (5)
C13—H13B	0.9900	C35—Cl3	1.740 (3)
C14—C15	1.521 (4)	C36—C37	1.385 (5)
C14—H14	1.0000	C36—H36	0.9500
C15—N1	1.470 (4)	C37—C38	1.382 (5)
C15—H15A	0.9900	C37—Cl4	1.736 (3)
C15—H15B	0.9900	C38—H38	0.9500
C16—O2	1.247 (4)	N2—H1N	0.84 (4)
C16—N2	1.335 (4)	N3—H2N	0.88 (4)
N1—C1—C2	111.1 (2)	N2—C17—C20	106.8 (2)
N1—C1—H1A	109.4	C18—C17—C20	109.6 (3)
C2—C1—H1A	109.4	N2—C17—C19	108.5 (3)
N1—C1—H1B	109.4	C18—C17—C19	111.4 (3)
C2—C1—H1B	109.4	C20—C17—C19	109.4 (3)
H1A—C1—H1B	108.0	C17—C18—H18A	109.5
O4—C2—C1	108.1 (2)	C17—C18—H18B	109.5
O4—C2—C3	103.1 (2)	H18A—C18—H18B	109.5
C1—C2—C3	116.5 (2)	C17—C18—H18C	109.5
O4—C2—H2	109.6	H18A—C18—H18C	109.5
C1—C2—H2	109.6	H18B—C18—H18C	109.5
C3—C2—H2	109.6	C17—C19—H19A	109.5
N3—C3—C4	109.5 (2)	C17—C19—H19B	109.5
N3—C3—C2	110.0 (2)	H19A—C19—H19B	109.5
C4—C3—C2	114.9 (2)	C17—C19—H19C	109.5
N3—C3—H3	107.4	H19A—C19—H19C	109.5
C4—C3—H3	107.4	H19B—C19—H19C	109.5
C2—C3—H3	107.4	C17—C20—H20A	109.5
C21—C4—C3	111.2 (3)	C17—C20—H20B	109.5
C21—C4—H4A	109.4	H20A—C20—H20B	109.5
C3—C4—H4A	109.4	C17—C20—H20C	109.5
C21—C4—H4B	109.4	H20A—C20—H20C	109.5
C3—C4—H4B	109.4	H20B—C20—H20C	109.5
H4A—C4—H4B	108.0	C26—C21—C22	118.2 (3)
O3—C5—N3	124.7 (3)	C26—C21—C4	122.0 (3)
O3—C5—C27	120.0 (3)	C22—C21—C4	119.7 (3)
N3—C5—C27	115.3 (3)	C23—C22—C21	121.2 (4)
O5—C6—O4	124.5 (3)	C23—C22—H22	119.4
O5—C6—C33	124.9 (3)	C21—C22—H22	119.4
O4—C6—C33	110.5 (3)	C24—C23—C22	119.9 (4)
N1—C7—C16	111.3 (2)	C24—C23—H23	120.0
N1—C7—C8	109.8 (2)	C22—C23—H23	120.0
C16—C7—C8	109.1 (2)	C23—C24—C25	120.4 (4)
N1—C7—H7	108.9	C23—C24—H24	119.8

C16—C7—H7	108.9	C25—C24—H24	119.8
C8—C7—H7	108.9	C24—C25—C26	120.1 (4)
C9—C8—C7	111.1 (2)	C24—C25—H25	120.0
C9—C8—H8A	109.4	C26—C25—H25	120.0
C7—C8—H8A	109.4	C21—C26—C25	120.3 (4)
C9—C8—H8B	109.4	C21—C26—H26	119.9
C7—C8—H8B	109.4	C25—C26—H26	119.9
H8A—C8—H8B	108.0	C32—C27—C28	121.0 (3)
C10—C9—C8	113.7 (3)	C32—C27—C5	117.6 (3)
C10—C9—C14	111.0 (3)	C28—C27—C5	121.4 (3)
C8—C9—C14	110.0 (2)	C29—C28—C27	118.5 (3)
C10—C9—H9	107.3	C29—C28—H28	120.7
C8—C9—H9	107.3	C27—C28—H28	120.7
C14—C9—H9	107.3	C30—C29—C28	121.7 (3)
C11—C10—C9	112.7 (3)	C30—C29—Cl1	119.2 (2)
C11—C10—H10A	109.1	C28—C29—Cl1	119.1 (3)
C9—C10—H10A	109.1	C29—C30—C31	118.6 (3)
C11—C10—H10B	109.1	C29—C30—H30	120.7
C9—C10—H10B	109.1	C31—C30—H30	120.7
H10A—C10—H10B	107.8	C32—C31—C30	121.3 (3)
C10—C11—C12	111.5 (3)	C32—C31—Cl2	119.2 (3)
C10—C11—H11A	109.3	C30—C31—Cl2	119.4 (3)
C12—C11—H11A	109.3	C27—C32—C31	118.9 (3)
C10—C11—H11B	109.3	C27—C32—H32	120.6
C12—C11—H11B	109.3	C31—C32—H32	120.6
H11A—C11—H11B	108.0	C38—C33—C34	120.8 (3)
C11—C12—C13	110.8 (3)	C38—C33—C6	118.8 (3)
C11—C12—H12A	109.5	C34—C33—C6	120.3 (3)
C13—C12—H12A	109.5	C35—C34—C33	118.3 (3)
C11—C12—H12B	109.5	C35—C34—H34	120.8
C13—C12—H12B	109.5	C33—C34—H34	120.8
H12A—C12—H12B	108.1	C34—C35—C36	122.3 (3)
C14—C13—C12	110.6 (3)	C34—C35—Cl3	119.3 (3)
C14—C13—H13A	109.5	C36—C35—Cl3	118.3 (3)
C12—C13—H13A	109.5	C37—C36—C35	117.8 (3)
C14—C13—H13B	109.5	C37—C36—H36	121.1
C12—C13—H13B	109.5	C35—C36—H36	121.1
H13A—C13—H13B	108.1	C38—C37—C36	122.1 (3)
C15—C14—C13	112.6 (3)	C38—C37—Cl4	119.7 (3)
C15—C14—C9	109.5 (2)	C36—C37—Cl4	118.2 (3)
C13—C14—C9	112.0 (3)	C37—C38—C33	118.7 (3)
C15—C14—H14	107.5	C37—C38—H38	120.6
C13—C14—H14	107.5	C33—C38—H38	120.6
C9—C14—H14	107.5	C1—N1—C7	112.5 (2)
N1—C15—C14	112.9 (3)	C1—N1—C15	108.8 (2)
N1—C15—H15A	109.0	C7—N1—C15	110.1 (2)
C14—C15—H15A	109.0	C16—N2—C17	126.5 (3)
N1—C15—H15B	109.0	C16—N2—H1N	118 (3)

C14—C15—H15B	109.0	C17—N2—H1N	114 (3)
H15A—C15—H15B	107.8	C5—N3—C3	123.4 (3)
O2—C16—N2	123.9 (3)	C5—N3—H2N	118 (2)
O2—C16—C7	122.1 (2)	C3—N3—H2N	119 (2)
N2—C16—C7	114.0 (2)	C6—O4—C2	119.3 (2)
N2—C17—C18	111.1 (3)		

N1—C1—C2—O4	175.5 (2)	C27—C28—C29—Cl1	179.7 (2)
N1—C1—C2—C3	−69.1 (3)	C28—C29—C30—C31	−1.6 (5)
O4—C2—C3—N3	68.7 (3)	Cl1—C29—C30—C31	179.8 (2)
C1—C2—C3—N3	−49.5 (3)	C29—C30—C31—C32	0.5 (5)
O4—C2—C3—C4	−167.3 (2)	C29—C30—C31—Cl2	−178.7 (2)
C1—C2—C3—C4	74.4 (3)	C28—C27—C32—C31	−1.6 (5)
N3—C3—C4—C21	−59.4 (3)	C5—C27—C32—C31	178.7 (3)
C2—C3—C4—C21	176.4 (3)	C30—C31—C32—C27	1.1 (5)
N1—C7—C8—C9	−58.6 (3)	Cl2—C31—C32—C27	−179.7 (2)
C16—C7—C8—C9	179.2 (2)	O5—C6—C33—C38	15.3 (5)
C7—C8—C9—C10	179.9 (3)	O4—C6—C33—C38	−167.0 (3)
C7—C8—C9—C14	54.7 (3)	O5—C6—C33—C34	−159.9 (3)
C8—C9—C10—C11	−72.6 (4)	O4—C6—C33—C34	17.8 (4)
C14—C9—C10—C11	52.1 (4)	C38—C33—C34—C35	−1.6 (5)
C9—C10—C11—C12	−53.9 (4)	C6—C33—C34—C35	173.5 (3)
C10—C11—C12—C13	55.9 (4)	C33—C34—C35—C36	2.6 (5)
C11—C12—C13—C14	−57.1 (4)	C33—C34—C35—Cl3	−175.4 (2)
C12—C13—C14—C15	−179.9 (3)	C34—C35—C36—C37	−1.5 (5)
C12—C13—C14—C9	56.2 (4)	Cl3—C35—C36—C37	176.5 (3)
C10—C9—C14—C15	−179.2 (3)	C35—C36—C37—C38	−0.7 (5)
C8—C9—C14—C15	−52.4 (3)	C35—C36—C37—Cl4	−177.4 (3)
C10—C9—C14—C13	−53.5 (4)	C36—C37—C38—C33	1.7 (5)
C8—C9—C14—C13	73.3 (3)	Cl4—C37—C38—C33	178.3 (2)
C13—C14—C15—N1	−69.1 (3)	C34—C33—C38—C37	−0.5 (5)
C9—C14—C15—N1	56.2 (3)	C6—C33—C38—C37	−175.6 (3)
N1—C7—C16—O2	−35.0 (4)	C2—C1—N1—C7	153.7 (2)
C8—C7—C16—O2	86.4 (3)	C2—C1—N1—C15	−84.0 (3)
N1—C7—C16—N2	147.1 (2)	C16—C7—N1—C1	−57.2 (3)
C8—C7—C16—N2	−91.6 (3)	C8—C7—N1—C1	−178.2 (2)
C3—C4—C21—C26	−85.2 (4)	C16—C7—N1—C15	−178.8 (2)
C3—C4—C21—C22	90.5 (4)	C8—C7—N1—C15	60.3 (3)
C26—C21—C22—C23	1.5 (5)	C14—C15—N1—C1	175.6 (2)
C4—C21—C22—C23	−174.3 (3)	C14—C15—N1—C7	−60.7 (3)
C21—C22—C23—C24	0.4 (6)	O2—C16—N2—C17	4.7 (5)
C22—C23—C24—C25	−2.0 (6)	C7—C16—N2—C17	−177.5 (3)
C23—C24—C25—C26	1.8 (7)	C18—C17—N2—C16	−53.9 (4)
C22—C21—C26—C25	−1.7 (5)	C20—C17—N2—C16	−173.3 (3)
C4—C21—C26—C25	174.0 (4)	C19—C17—N2—C16	68.9 (4)
C24—C25—C26—C21	0.1 (6)	O3—C5—N3—C3	0.8 (5)
O3—C5—C27—C32	42.1 (4)	C27—C5—N3—C3	180.0 (3)
N3—C5—C27—C32	−137.1 (3)	C4—C3—N3—C5	136.6 (3)

O3—C5—C27—C28	−137.6 (3)	C2—C3—N3—C5	−96.3 (3)
N3—C5—C27—C28	43.2 (4)	O5—C6—O4—C2	4.8 (5)
C32—C27—C28—C29	0.5 (5)	C33—C6—O4—C2	−172.9 (2)
C5—C27—C28—C29	−179.8 (3)	C1—C2—O4—C6	−78.2 (3)
C27—C28—C29—C30	1.2 (5)	C3—C2—O4—C6	158.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*···O3^{i}	0.84 (4)	2.13 (4)	2.931 (3)	160 (3)
N3—H2*N*···O2	0.88 (4)	1.99 (4)	2.834 (3)	159 (3)
C4—H4*A*···N1	0.99	2.55	3.149 (4)	119
C18—H18*A*···O2	0.98	2.36	2.975 (4)	120
C34—H34···O3	0.95	2.40	3.324 (4)	163

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.