Acta Crystallographica Section C
Structural Chemistry
ISSN 2053-2296

Three closely-related cyclohexanols ($C_{35}H_{27}X_2N_3O_3$; X = F, Cl or Br): similar molecular structures but different crystal structures

Seranthimata Samshuddin,[a] Jerry P. Jasinski,[b] Ray J. Butcher,[c] Elizabeth A. Neuhardt,[b] Badiadka Narayana,[a] Hemmige S. Yathirajan[d] and Christopher Glidewell[e]*

[a]Department of Studies in Chemistry, Mangalore University, Mangalagangotri 574 199, India, [b]Department of Chemistry, Keene State College, 229 Main Street, Keene, NH 03435-2001, USA, [c]Department of Chemistry, Howard University, 525 College Street, Washington NW, DC 20059, USA, [d]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and [e]School of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland
Correspondence e-mail: cg@st-andrews.ac.uk

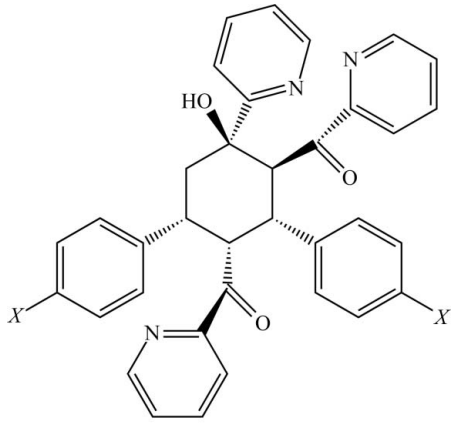
Received 2 September 2014
Accepted 3 September 2014

Three highly-substituted cyclohexanol derivatives have been prepared from 2-acetylpyridine and 4-halogenobenzaldehydes under mild conditions. (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)-3,5-Bis(4-fluorophenyl)-2,4-bis(pyridine-2-carbonyl)-1-(pyridin-2-yl)cyclohexanol, $C_{35}H_{27}F_2N_3O_3$, (I), (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)-3,5-bis(4-chlorophenyl)-2,4-bis(pyridine-2-carbonyl)-1-(pyridin-2-yl)cyclohexanol acetone 0.951-solvate, $C_{35}H_{27}Cl_2N_3O_3\cdot 0.951C_3H_6O$, (II), and (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)-3,5-bis(4-bromophenyl)-2,4-bis(pyridine-2-carbonyl)-1-(pyridin-2-yl)cyclohexanol, $C_{35}H_{27}Br_2N_3O_3$, (III), all crystallize in different space groups, *viz*. *Pbca*, *Fdd*2 and $P\overline{1}$, respectively. In compound (II), the acetone molecule is disordered over two sets of atomic sites having occupancies of 0.690 (13) and 0.261 (13). Each of the cyclohexanol molecules contains an intramolecular O—H···N hydrogen bond and their overall molecular conformations are fairly similar. The molecules of (I) are linked by two independent C—H···O hydrogen bonds to form a $C(5)C(10)[R_2^2(15)]$ chain of rings, and those of (III) are linked by a combination of C—H···O and C—H···N hydrogen bonds, forming a chain of alternating $R_2^2(16)$ and $R_2^2(18)$ rings. The cyclohexanol molecules in (II) are linked by a single C—H···N hydrogen bond to form simple $C(4)$ chains and these chains are linked by a π–π stacking interaction to form sheets, to which the disordered acetone molecules are weakly linked *via* a number of C—H···O contacts.

Keywords: crystal structure; highly substituted cyclohexanols; supramolecular assembly; hydrogen bonding; Michael addition; condensation reactions.

1. Introduction

Chalcones, *i.e.* 1,3-disubstituted prop-2-en-1-one derivatives of the form $R^1COCH{=}CHR^2$, are versatile intermediates for organic synthesis which are readily prepared *via* Claisen reactions between acetyl compounds (R^1COCH_3) and aldehydes (R^2CHO). Their versatility stems from their dual modes of reactivity, involving either Michael-type addition across the C=C double bond or condensation reactions at the carbonyl group. When these two modes are active in tandem, new cyclic structures result which can be either carbocyclic (Tabba *et al.*, 1995; Ravindran *et al.*, 2008; Fischer *et al.*, 2008; Fun *et al.*, 2010*b*) or heterocyclic (Fun *et al.*, 2010*a*; Jasinski, Guild *et al.*, 2010; Jasinski, Pek *et al.*, 2010; Samshuddin *et al.*, 2010).



(I) X = F
(II*a*) X = Cl
(III) X = Br
(IV*a*) X = Ph

(II) = (II*a*)·0.951MeCOMe
(IV) = (IV*a*)· EtCOMe

Scheme 1

We now report the synthesis and structures of three closely related cyclohexanol derivatives, namely (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)-3,5-bis(4-fluorophenyl)-2,4-bis(pyridine-2-carbonyl)-1-(pyridin-2-yl)cyclohexanol, (I), (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)-3,5-bis(4-chlorophenyl)-2,4-bis(pyridine-2-carbonyl)-1-(pyridin-2-yl)cyclohexanol acetone 0.951-solvate, (II), and (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)-3,5-bis(4-bromophenyl)-2,4-bis(pyridine-2-carbonyl)-1-(pyridin-2-yl)cyclohexanol, (III) (see Scheme 1). These cyclohexanols were prepared, in a one-pot procedure exploiting the dual reactivity of chalcones and giving satisfactory yields, by the reaction of 3:2 molar ratios of 2-acetylpyridine with a 4-halogenobenzaldehyde in alkaline aqueous ethanol, without the need for any form of heating. The purposes of this study were the definition of the relative stereochemistry of the five stereogenic centres, the comparison of the molecular conformations, and the exploration of the supramolecular assembly. In addition, we compare the structures of compounds (I)–(III) with that of the recently reported analogue (IV) (Fun *et al.*, 2012) (see Scheme 1).

Table 1
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{35}H_{27}F_2N_3O_3$	$C_{35}H_{27}Cl_2N_3O_3{\cdot}0.951C_3H_6O$	$C_{35}H_{27}Br_2N_3O_3$
M_r	575.60	663.73	697.41
Crystal system, space group	Orthorhombic, *Pbca*	Orthorhombic, *Fdd*2	Triclinic, $P\overline{1}$
Temperature (K)	200	200	200
a, *b*, *c* (Å)	21.8864 (9), 11.2268 (4), 24.2670 (9)	16.5446 (6), 53.4204 (17), 15.5857 (4)	9.5741 (4), 10.7061 (3), 15.9952 (6)
α, β, γ (°)	90, 90, 90	90, 90, 90	92.863 (3), 97.443 (3), 103.618 (3)
V (Å^3)	5962.7 (4)	13774.9 (8)	1574.49 (10)
Z	8	16	2
Radiation type	Cu *K*α	Cu *K*α	Cu *K*α
μ (mm^{-1})	0.75	2.05	3.58
Crystal size (mm)	0.40 × 0.09 × 0.08	0.30 × 0.25 × 0.20	0.25 × 0.20 × 0.15
Data collection			
Diffractometer	Agilent Eos Gemini diffractometer	Agilent Xcalibur Ruby Gemini diffractometer	Agilent Xcalibur Ruby Gemini diffractometer
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2012)	Multi-scan (*CrysAlis PRO*; Agilent, 2012)	Multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min}, T_{max}	0.693, 0.942	0.388, 0.664	0.109, 0.584
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37077, 5754, 4489	9544, 4224, 3490	11667, 5922, 4948
R_{int}	0.036	0.027	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.614	0.610	0.610
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.046, 0.131, 1.03	0.039, 0.109, 0.97	0.046, 0.141, 1.05
No. of reflections	5754	4224	5922
No. of parameters	388	436	388
No. of restraints	0	7	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.20	0.16, −0.16	0.67, −0.64
Absolute structure	–	Flack x determined using 613 quotients [(*I*+)−(*I*−)]/[(*I*+)+(*I*−)] (Parsons *et al.*, 2013).	–
Absolute structure parameter	–	0.089 (18)	–

Computer programs: *CrysAlis PRO* (Agilent, 2012), *CrysAlis RED* (Agilent, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2014) and *PLATON* (Spek, 2009).

2. Experimental

2.1. Synthesis and crystallization

For the synthesis of the solvent-free forms (I), (II*a*) and (III) (see Scheme 1), 2-acetylpyridine (0.03 mol) and the appropriate 4-halogenobenzaldehyde (0.02 mol) were dissolved in ethanol (30 ml). Aqueous potassium hydroxide solution (10 ml of a 10% *w*/*v* solution) was added and the mixtures were stirred at 293 K for 4 h and then permitted to stand overnight at ambient temperature. The resulting colourless products were collected by filtration and dried in air. Data for compound (I): yield 64%, m.p. 493–495 K; NMR (dimethyl sulfoxide-d_6): δ(H) 1.69 (*m*, 2H, CH_2), 3.62 (*dd*, 1H, H-3), 4.04 (*m*, 1H, H-5), 4.40 (*dd*, 1H, H-4), 5.52 (*s*, 1H, OH), 6.04 (*d*, 1H, H-2), 6.61–8.44 (*m*, 20H, aryl and pyridyl); IR (KBr, cm^{-1}): 3430 (OH), 1682 (C=O); analysis found: C 72.9, H 4.7, N 6.5%; $C_{35}H_{27}F_2N_3O_3$ requires: C 73.0, H 4.7, N 6.6%. Data for compound (II*a*): yield 66%, m.p. 416–419 K; NMR (dimethyl sulfoxide-d_6): δ(H) 1.75 (*m*, 2H, CH_2), 3.70 (*dd*, 1H, H-3), 4.05 (*m*, 1H, H-5), 4.45 (*dd*, 1H, H-4), 5.50 (*s*, 1H, OH), 6.05 (*d*, 1H, H-2), 6.84–8.44 (*m*, 20H, aryl and pyridyl); IR (KBr, cm^{-1}): 3434 (OH), 1689 (C=O); analysis found:C 68.9, H 4.5, N 6.9%; $C_{35}H_{27}Cl_2N_3O_3$ requires: C 69.3, H 4.5, N 6.9%. Data for compound (III): yield 72%, m.p. 508–510 K; NMR (chloroform-d_3): δ(H) 1.95 (*m*, 2H, CH_2), 3.50 (*dd*, 1H, H-3), 4.15 (*m*, 1H, H-5), 4.40 (*dd*, 1H, H-4), 5.50 (*s*, 1H, OH), 6.22 (*m*, 1H, H-2), 6.86–7.49 (*m*, 20H, aryl and pyridyl); δ(C) 37.9, 40.6, 47.7, 199.9, 120.9, 121.0, 121.6, 121.8, 126.0, 126.1, 126.5, 130.2, 130.7, 130.8, 136.0, 136.1, 139.7, 141.0, 146.9, 147.9, 148.0, 153.7, 153.9, 162.2, 203.0, 205.0; IR (KBr, cm^{-1}): 3429 (OH), 1682 (C=O); analysis found: C 60.2, H 3.9, N 6.0%; $C_{35}H_{27}Br_2N_3O_3$ requires: C 60.3, H 3.9, N 6.0%. Crystals of (I)–(III) suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in acetone.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms in the cycohexanol molecules were located in difference maps and then treated as riding atoms. H atoms bonded to C atoms were treated as riding atoms in geometrically idealized positions, with C—H = 0.95 (aromatic and heteroaromatic), 0.99 (CH_2) or 1.00 Å (aliphatic), and with U_{iso}(H) = 1.2U_{eq}(C). H atoms bonded to O atoms were permitted to ride at the positions located in difference maps, with U_{iso}(H) = 1.5U_{eq}(O), giving the O—H distances shown in Table 2. Chloro compound (II) crystallizes as an acetone solvate where the solvent molecules are disordered over two sets of atomic sites. For the minor component, the bonded distances and the one-angle nonbonded distances were restrained to the corresponding

Table 2
Hydrogen bonds and short intermolecular contacts (Å, °) for compounds (I)–(III).

*Cg*1 represents the centroid of the C31–C36 ring.

Compound	*D*—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)	O11—H11···N11	0.82	2.13	2.624 (2)	119
	C13—H13···O11^{i}	0.95	2.54	3.385 (3)	148
	C55—H55···O27^{i}	0.95	2.53	3.244 (2)	132
(I)	O11—H11···N11	0.88	1.92	2.587 (4)	130
	C14—H14···O71	0.95	2.62	3.517 (12)	157
	C14—H14···O81	0.95	2.72	3.499 (12)	140
	C43—H43···N41^{ii}	0.95	2.61	3.433 (4)	145
	C45—H45···O71^{iii}	0.95	2.57	3.251 (11)	129
	C16—H16···*Cg*1^{iv}	0.95	3.00	3.869 (5)	153
(I)	O11—H11···N11	0.81	2.06	2.595 (4)	123
	C16—H16···O27^{v}	0.95	2.46	3.385 (6)	165
	C53—H53···N41^{vi}	0.95	2.57	3.473 (5)	158

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, z$; (ii) $x+\frac{1}{4}, -y+\frac{3}{4}, z-\frac{1}{4}$; (iii) $x-\frac{3}{4}, -y+\frac{3}{4}, z-\frac{1}{4}$; (iv) $x+\frac{1}{2}, y, z+\frac{1}{2}$; (v) $-x, -y, -z+1$; (vi) $-x, -y+1, -z$.

values in the major component, subject to variations of 0.005 and 0.01 Å, respectively. In addition, the anisotropic displacement parameters for atoms C71 and C81 were constrained to be identical, as were those of atoms O71 and O81, and those of methyl atoms C72, C73, C82 and C83. The H atoms of both components of the disordered acetone solvent molecule were included in calculated positions as riding atoms, with C—H = 0.95 Å and U_{iso}(H) = 1.5U_{eq}(C). Subject to these conditions, the occupancies of the major and minor components refined to 0.690 (13) and 0.261 (13), respectively. The correct orientation of the structure of (II) with respect to the polar-axis direction was determined using the Flack *x* parameter (Flack, 1983), calculated using 613 quotients of the type [(*I*+)−(*I*−)]/[(*I*+)+(*I*−)] (Parsons *et al.*, 2013), although this has no chemical significance. Examination of the refined structures using *PLATON* (Spek, 2009) showed that none of them contained any solvent-accessible voids.

3. Results and discussion

Although all of the cyclohexanols reported here, (I)–(III), were recrystallized from acetone, fluoro and bromo derivatives (I) and (III) crystallized in the solvent-free form but chloro derivative (II) crystallized as a nonstoichiometric acetone solvate, in which the acetone solvent molecule is disordered over two adjacent sets of atomic sites having occupancies of 0.690 (13) and 0.261 (13) in the crystal selected for data collection. However, the s.u. values associated with these refined occupancies cannot definitively rule out the possibility that (II) might just, in fact, be a stoichiometric 1:1 solvate. In addition to the contrast between solvate (II) and solvent-free forms (I) and (III), all of these compounds crystallize in different space groups, *viz.* *Pbca*, *Fdd*2 and $P\overline{1}$, respectively, for compounds (I), (II) and (III), despite the very small differences in their overall molecular constitutions. This behaviour contrasts with the isomorphous behaviour sometimes seen in compounds which differ only in the identity of their halogen substituents (*e.g.* Glidewell *et al.*, 2005; Nayak *et al.*, 2014). [2,6-Bis(biphenyl-4-yl)-4-hydroxy-4-(pyridin-2-yl)-cyclohexane-1,3-diyl]bis[(pyridin-2-yl)methanone]–butan-2-one (1/1), (IV), crystallizes in the space group $P2_1/c$ as a stoichiometric 1:1 solvate with butanone, although the reported synthetic procedure for (IV) specifies that the compound was crystallized from acetone (Fun *et al.*, 2012).

The cyclohexanol molecules in compounds (I)–(III) contain five stereogenic centres and, for each compound, the reference molecule was selected as one having the *R* configuration at atom C1 (Figs. 1–3). On this basis, the reference molecules all have the configuration (1*R*,2*S*,3*S*,4*R*,5*R*) and the space groups confirm that all three compounds crystallize as racemic mixtures. Compound (IV) has exactly the same stereochemistry, although this aspect of its structure was not mentioned in the original structure report (Fun *et al.*, 2012). The isolated yields of the (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*) forms for compounds (I)–(IV), in the range 64–74%, suggest a high degree of stereoselectivity in the formation of the substituted cyclohexane ring.

The formation of these polysubstituted cyclohexanols from very simple precursors can be envisaged as starting with a condensation reaction between the substituted benzaldehyde (ArCHO) and 2-acetylpyridine (MeCOPy) to form a chalcone intermediate (*A*) (see Scheme 2). This step is followed by two Michael additions, the first of which is addition of MeCOPy to chalcone (*A*) to form intermediate (*B*), and the second addition is that of (*B*) to a further molecule of (*A*) giving (*C*). Finally, an intramolecular aldol reaction converts intermediate (*C*) to the cyclohexanol product. The formation of intermediate (*B*) introduces a stereogenic centre, at the site which will eventually become atom C5 of the cyclohexane ring (*cf.* Figs. 1–3). Intermediate (*B*) is necessarily racemic, as no

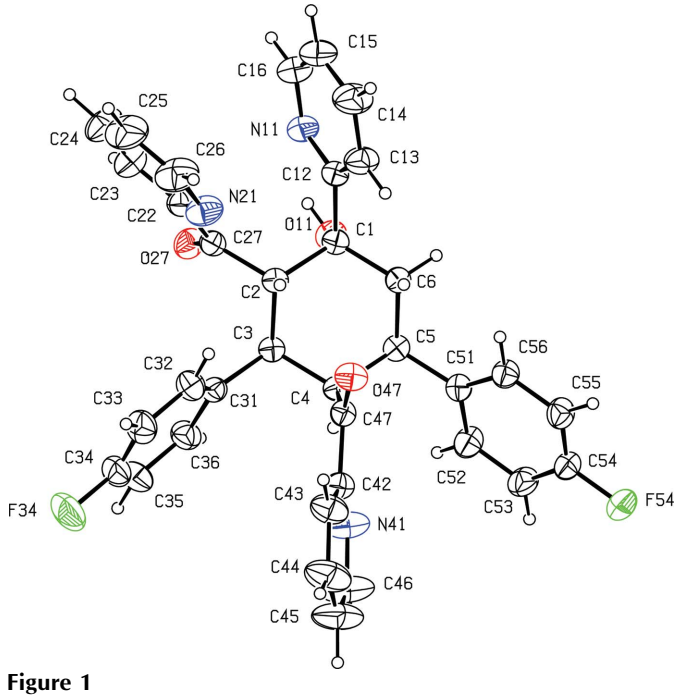


Figure 1
The molecular structure of the (1*R*,2*S*,3*S*,4*R*,5*R*) enantiomer of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

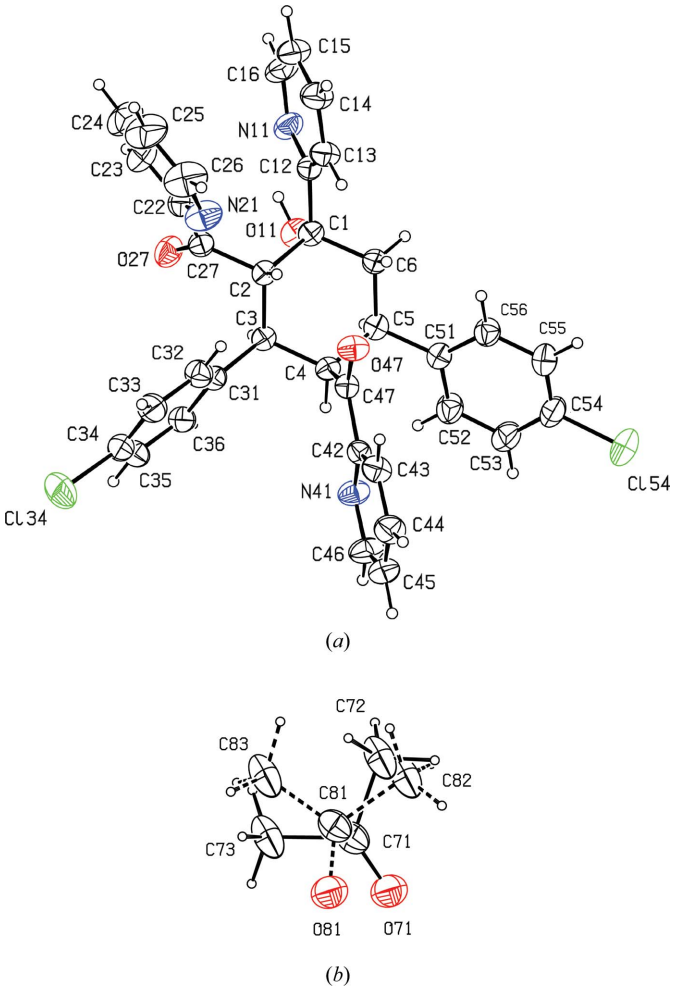


Figure 2
The independent molecular components of compound (II), showing the atom-labelling scheme, for (*a*) the (1*R*,2*S*,3*S*,4*R*,5*R*) enantiomer of the cyclohexanol molecule and (*b*) the disordered acetone solvent molecule. The occupancies of the two components of the disordered acetone molecule are 0.690 (13) and 0.261 (13). Displacement ellipsoids are drawn at the 30% probability level for part (*a*) and at the 10% probability level for part (*b*).

component in the initial reaction mixtures is capable of inducing enantioselectivity. However, the presence of a stereogenic centre in (*B*) appears to control the relative stereochemistry at the remaining stereogenic centres subsequently formed at the final positions C1, C2, C3 and C4. This reaction scheme accounts for the reaction stoichiometry, the formation of five adjacent stereogenic centres and the

Table 3
Selected dihedral angles (°) for compounds (I)–(III).

Compound	θ_1	θ_2	θ_3	θ_4	θ_5
(I)	30.80 (12)	85.33 (11)	44.19 (13)	67.09 (13)	28.94 (10)
(II)	24.3 (2)	88.0 (2)	62.66 (19)	57.3 (2)	53.3 (2)
(III)	31.5 (2)	79.4 (2)	63.9 (2)	42.5 (2)	49.05 (18)

Notes: θ_1 represents the dihedral angle between the N11/C12–C16 and N21/C22–C26 planes; θ_2 represents the dihedral angle between the N21/C22–C26 and C31–C36 planes; θ_3 represents the dihedral angle between the C31–C36 and N41/C42–C46 planes; θ_4 represents the dihedral angle between the N41/C42–C46 and C51–C56 planes; θ_5 represents the dihedral angle between the C51–C56 and N11/C12–C16 planes.

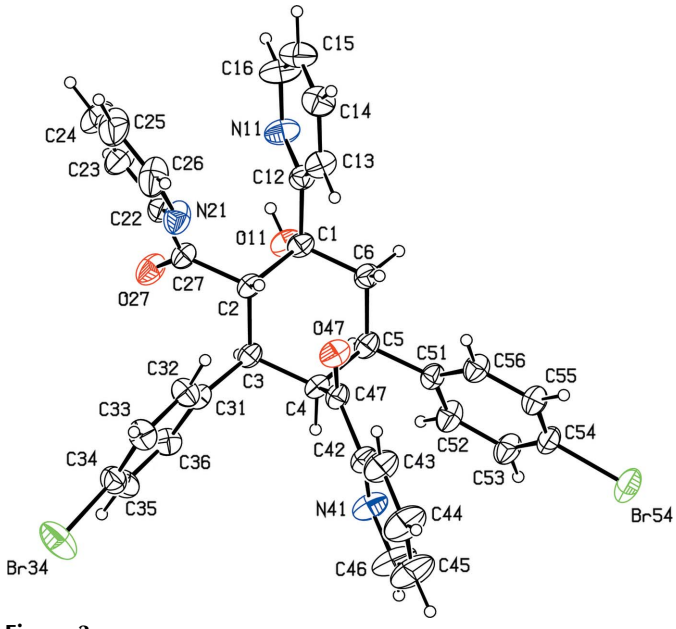
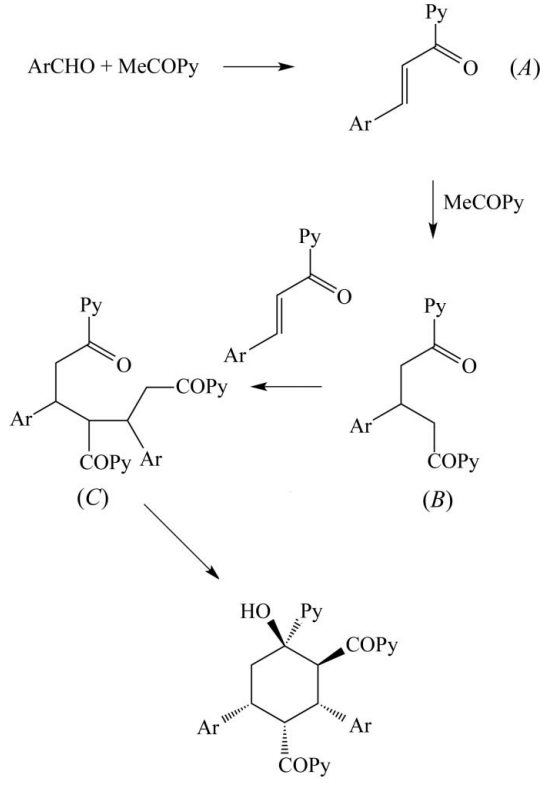


Figure 3
The molecular structure of the (1*R*,2*S*,3*S*,4*R*,5*R*) enantiomer of compound (III), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

formation of the final products as racemic mixtures, but it must be emphasized that this scheme is largely conjectural, albeit firmly based on the straightforward application of simple organic processes.



Scheme 2

Within the cyclohexanol molecules, the cyclohexane rings all adopt chair conformations and the ring-puckering angles θ,

calculated (Cremer & Pople, 1975) for the atom sequence C1–C2–C3–C4–C5–C6, are 5.36 (17), 5.5 (3) and 0.0 (3)° for compounds (I), (II) and (III), respectively; the corresponding value for compound (IV) is 4.69 (15)°. The ideal value for a perfect chair conformation is θ = 0.0° (Boeyens, 1978). The organic substituents at atoms C1, C2, C3 and C5 all occupy equatorial sites, and both the hydroxy group at atom C1 and the acyl substituent at atom C4 occupy axial sites.

In each of compounds (I)–(III) there is an intramolecular O—H···N hydrogen bond (Table 2), which may serve effectively to lock the orientation of the pyridine ring bonded to atom C1. The overall molecular conformation can be conveniently summarized in terms of the dihedral angles between the successive rings bonded to the central cyclohexane ring (Table 3); the corresponding values for (I)–(III) are in general similar, although with some small differences in detail. The overall similarity between compounds (I)–(III) in terms of their molecular constitutions, stereochemistry and molecular conformations makes their marked differences in crystallization behaviour difficult to understand.

The supramolecular assembly in compounds (I)–(III) is fairly simple for molecules of this complexity. Thus, for example, despite the presence in each cyclohexanol molecule of five independent aryl or pyridyl rings, there are no π–π interactions in the structure of (I) and only one such interaction in each of (II) and (III). Similarly, there are no C—H···π interactions in the structures of (I) and (III), while the single interaction of this type in (II) has long H···*Cg* and C···*Cg* distances (Table 2), and it is not considered to be structurally significant. The principal intermolecular interactions are thus C—H···N and C—H···O hydrogen bonds, augmented by the π–π interactions in (II) and (III). In the structure of compound (I), a combination of two C—H···O hydrogen bonds (Table 2) links molecules related by a *b*-glide plane to form a $C(5)C(10)[R_2^2(15)]$ chain of rings (Bernstein *et al.*, 1995) running parallel to the [010] direction (Fig. 4).

There are several C—H···O contacts between the molecular components in compound (II) (Table 2). Within the selected asymmetric unit, the cyclohexanol molecule forms

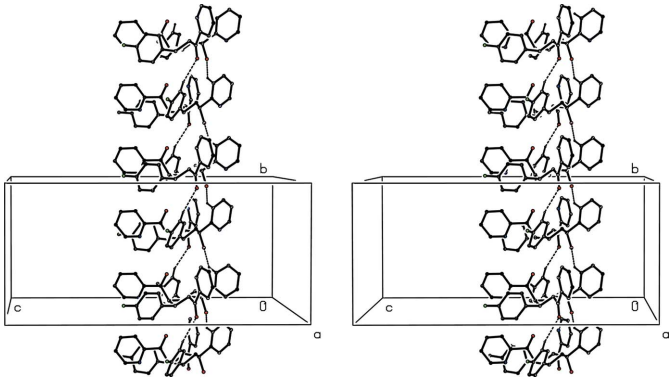


Figure 4
A stereoview of part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded $C(5)C(10)[R_2^2(15)]$ chain of rings parallel to [010]. Hydrogen bonds are shown as dashed lines and H atoms not involved in the motifs shown have been omitted.

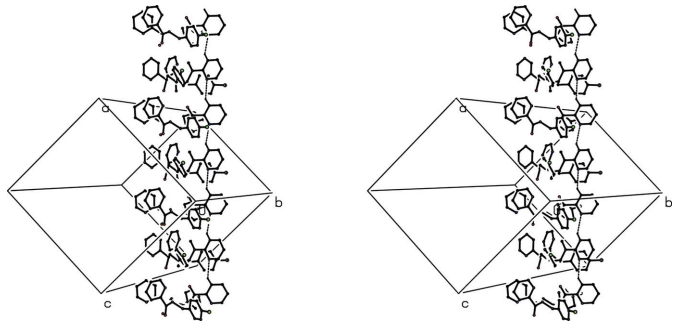


Figure 5
A stereoview of part of the crystal structure of compound (II), showing the formation of a hydrogen-bonded $C(4)$ chain parallel to [10$\bar{1}$]. Hydrogen bonds are shown as dashed lines and the disordered acetone molecules and H atoms not involved in the motif shown have been omitted.

rather long C—H···O contacts with the two partial-occupancy components of the disordered acetone molecule, one of them with an H···O distance beyond the sum (2.65 Å) of the van der Waals radii for H and O (Rowland & Taylor, 1996). A third such contact has a C—H···O angle of only 129°, so that this contact is probably not structurally significant (Wood *et al.*, 2009). The weak bonding of the acetone molecule to the cyclohexanol molecule may account for the nonstoichiometric nature of the solvation, for the positional disorder of the solvent molecule and for the relatively large displacement parameters of the solvent molecule. Subject to these provisos, and that mentioned above concerning the C—H···π contact, the sole significant intermolecular hydrogen bond, of C—H···N type, links cyclohexanol molecules related by the *d*-glide plane at y = 0.375 to form a simple $C(4)$ chain running parallel to the [10$\bar{1}$] direction (Fig. 5). These simple chains are linked into sheets by a single π–π stacking interaction. The planes of the pyridine rings containing atoms N11 and N41 in the molecules at (x, y, z) and ($x+\frac{1}{2}$, y, $z+\frac{1}{2}$), respectively, make a dihedral angle of 4.7 (2)°. The ring-centroid separation is 3.846 (2) Å and the shortest perpendicular distance from the centroid of one ring to the plane of the other is 3.3909 (17) Å, corresponding to a nearly ideal ring-centroid offset of *ca* 1.52 Å (Fig. 6). This interaction links cyclohexanol molecules

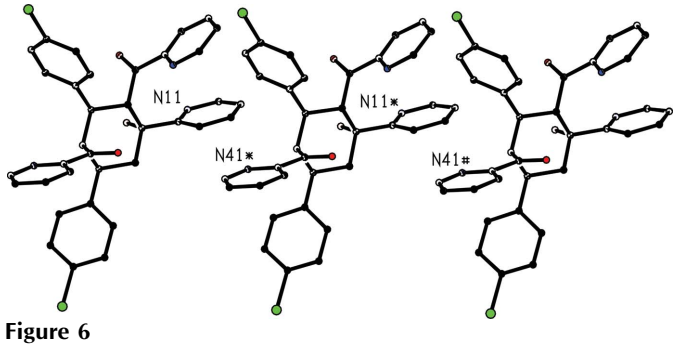


Figure 6
Part of the crystal structure of compound (II), showing the π–π stacking interaction which generates chains along [101]. For the sake of clarity, the H atoms, the solvent molecules and the unit-cell outline have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($x+\frac{1}{2}$, y, $z+\frac{1}{2}$) and ($x+1$, y, $z+1$), respectively.

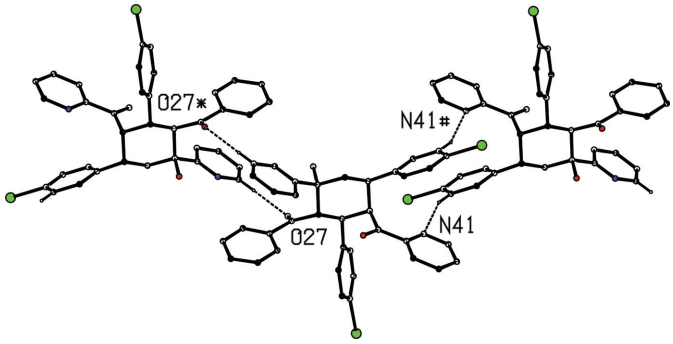


Figure 7
Part of the crystal structure of compound (III), showing the formation of the hydrogen-bonded $R_2^2(16)$ and $R_2^2(18)$ rings which generate a chain parallel to [01$\bar{1}$]. For the sake of clarity, H atoms not involved in the motifs shown and the unit-cell outline have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions ($-x$, $-y$, $-z+1$) and ($-x$, $-y+1$, $-z$), respectively.

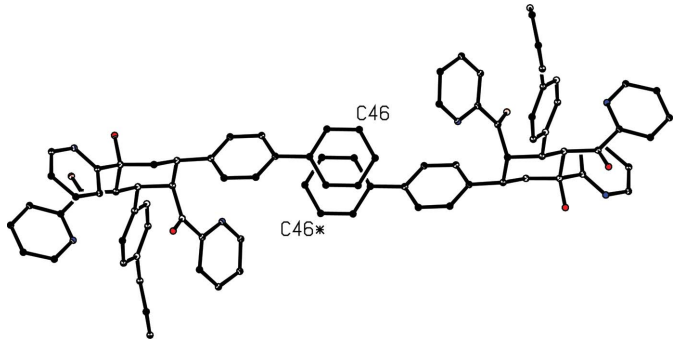


Figure 8
Part of the crystal structure of compound (IV), showing the π–π stacking interaction which links hydrogen-bonded chains along [001] into a sheet parallel to (100). The original atomic coordinates (Fun *et al.*, 2012) have been used. For the sake of clarity, the H atoms, the solvent molecules and the unit-cell outline have been omitted. The atom marked with an asterisk (*) is at the symmetry position ($-x$, $-y$, $-z$).

related by translation to form a π-stacked chain running parallel to the [101] direction. The combination of the hydrogen-bonded chains along [10$\bar{1}$] and the π-stacked chains along [101] links the cyclohexanol molecules into a sheet parallel to (010) to which the acetone molecules are linked only weakly, if at all.

A combination of one C—H···N hydrogen bond and one C—H···O hydrogen bond links the molecules of compound (III) into a chain of centrosymmetric rings running parallel to the [01$\bar{1}$] direction (Fig. 7). Inversion-related pairs of C—H···N hydrogen bonds link pairs of molecules into $R_2^2(18)$ rings centred at (0, $\frac{1}{2}-n$, n), where n represents an integer, and these rings alternate with $R_2^2(16)$ rings containing inversion-related pairs of C—H···O hydrogen bonds which are centred at (0, n, $\frac{1}{2}-n$), where n again represents an integer. There is a single π–π interaction in this structure; the planes of the pyridine rings containing atom N21 in the molecules at (x, y, z) and ($-x+1$, $-y$, $-z+1$) are parallel. The interplanar spacing is 3.555 (2) Å, the ring-centroid separation is 3.680 (3) Å and the ring-centroid offset is 0.951 (3) Å; the effect of this interaction is to link the hydrogen-bonded chains into a sheet parallel to (011).

In the structure of compound (III), there is a short intermolecular Br···Br contact between inversion-related molecules, with Br54···Br54^{i} = 3.3802 (6) Å and C54—Br54···Br54^{i} = 159.23 (9)° [symmetry code: (i) $-x$, $-y+1$, $-z$]. A database study of the angular distribution of such contacts (Ramasubbu *et al.*, 1986) has shown that the C—X···X angles (where X = Cl, Br or I) are clustered either around 100° or around 165°, and the angle observed in compound (III) is consistent with this finding. Although the observed Br···Br distance in (III) is shorter that the conventional sum of the van der Waals radii (3.70 Å; Rowland & Taylor, 1996), a database study of the nonbonded distances in such contacts (Nyburg & Faerman, 1985) found that atoms such as halogens bonded to C atoms do not behave in this context as though they were spherical but instead they behave as oblate ellipsoids, with the major axis normal to the direction of the C—X bond and the minor axis parallel to the C—X bond. For Br, these characteristic radii were found to be 2.01 and 1.64 Å, respectively, and, on this basis, the observed Br···Br distance in compound (III) does not seem to be exceptional.

In compound (IV) (Fun *et al.*, 2012), the supramolecular assembly consists of $C(5)$ chains containing molecules related by *c*-glide planes which are linked by a single C—H···O hydrogen bond, and the butanone solvent molecules are linked to these chains, also *via* C—H···O hydrogen bonds. Although the presence of a π–π stacking interaction was mentioned in the original structure report, no discussion of its action was given. In fact, this interaction involves a pair of inversion-related molecules (Fig. 8) and, in combination with the hydrogen-bonded chains, it generates a sheet lying parallel to (100).

BN and SS thank Mangalore University and the UGC–SAP for financial assistance for the purchase of chemicals. HSY thanks the University of Mysore for research facilities. JPJ and RJB acknowledge the NSF–MRI program (grant Nos. CHE-1039027 and CHE-0619278) for funds to purchase an X-ray diffractometer.

References

Agilent (2012). *CrysAlis PRO* and *CrysAlis RED*. Agilent Technologies, Yarnton, Oxfordshire, England.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Boeyens, J. C. A. (1978). *J. Cryst. Mol. Struct.* **8**, 317–320.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Fischer, A., Swamy, M. T., Narayana, B. & Yathirajan, H. S. (2008). *Acta Cryst.* E**64**, o2152.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Fun, H.-K., Hemamalini, M., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2010*a*). *Acta Cryst.* E**66**, o582–o583.
Fun, H.-K., Hemamalini, M., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2010*b*). *Acta Cryst.* E**66**, o864–o865.
Fun, H.-K., Ooi, C. W., Samshuddin, S., Narayana, B. & Sarojini, B. K. (2012). *Acta Cryst.* E**68**, o1633–o1634.
Glidewell, C., Low, J. N., Skakle, J. M. S. & Wardell, J. L. (2005). *Acta Cryst.* C**61**, o276–o280.
Jasinski, J. P., Guild, C. J., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2010). *Acta Cryst.* E**66**, o1948–o1949.

Jasinski, J. P., Pek, A. E., Samshuddin, S., Narayana, B. & Yathirajan, H. S. (2010). *Acta Cryst.* E**66**, o1950–o1951.
Nayak, P. S., Jasinski, J. P., Golen, J. A., Narayana, B., Kaur, M., Yathirajan, H. S. & Glidewell, C. (2014). *Acta Cryst.* C**70**, 889–894.
Nyburg, S. C. & Faerman, C. H. (1985). *Acta Cryst.* B**41**, 274–279.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Ramasubbu, N., Parthasarathy, R. & Murray-Rust, P. (1986). *J. Am. Chem. Soc.* **108**, 4308–4314.
Ravindran, G., Muthusubramanian, S. & Perumal, S. (2008). *Arkivoc*, **xiii**, 57–64.
Rowland, R. S. & Taylor, R. (1996). *J. Phys. Chem.* **100**, 7384–7391.
Samshuddin, S., Narayana, B., Yathirajan, H. S., Safwan, A. P. & Tiekink, E. R. T. (2010). *Acta Cryst.* E**66**, o1279–o1280.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2014). *SHELXL2014*. University of Göttingen, Germany.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tabba, H. D., Yousef, N. M. & Al-Arab, M. M. (1995). *Collect. Czech. Chem. Commun.* **60**, 594–604.
Wood, P. A., Allen, F. H. & Pidcock, E. (2009). *CrystEngComm*, **11**, 1563–1571.

supporting information

Acta Cryst. (2014). C**70**, 953-959 [doi:10.1107/S2053229614019937]

Three closely-related cyclohexanols ($C_{35}H_{27}X_2N_3O_3$; X = F, Cl or Br): similar molecular structures but different crystal structures

Seranthimata Samshuddin, Jerry P. Jasinski, Ray J. Butcher, Elizabeth A. Neuhardt, Badiadka Narayana, Hemmige S. Yathirajan and Christopher Glidewell

Computing details

For all compounds, data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis RED* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2014); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2014) and *PLATON* (Spek, 2009).

(I) (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)-3,5-Bis(4-fluorophenyl)-2,4-bis(pyridine-2-carbonyl)1-(pyridin-2-yl)cyclohexanol

Crystal data

$C_{35}H_{27}F_2N_3O_3$
M_r = 575.60
Orthorhombic, *Pbca*
a = 21.8864 (9) Å
b = 11.2268 (4) Å
c = 24.2670 (9) Å
V = 5962.7 (4) Å^3
Z = 8
F(000) = 2400
D_x = 1.282 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 5754 reflections
θ = 3.6–71.2°
μ = 0.75 mm^{-1}
T = 200 K
Needle, colourless
0.40 × 0.09 × 0.08 mm

Data collection

Agilent Eos Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.693, T_{max} = 0.942
37077 measured reflections
5754 independent reflections
4489 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 71.2°, θ_{min} = 3.6°
h = −18→26
k = −13→13
l = −27→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.131
S = 1.03
5754 reflections
388 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

$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 1.1285P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.19695 (8)	0.56058 (14)	0.29755 (6)	0.0469 (4)
O11	0.20061 (6)	0.43914 (10)	0.28039 (5)	0.0587 (3)
H11	0.1716	0.4286	0.2598	0.088*
C2	0.15217 (7)	0.57345 (13)	0.34750 (6)	0.0436 (3)
H2	0.1509	0.6589	0.3590	0.052*
C3	0.17385 (7)	0.49773 (13)	0.39661 (6)	0.0434 (3)
H3	0.1731	0.4128	0.3842	0.052*
C4	0.24067 (7)	0.52586 (13)	0.41351 (6)	0.0433 (3)
H4	0.2539	0.4617	0.4395	0.052*
C5	0.28430 (7)	0.52110 (13)	0.36324 (7)	0.0463 (3)
H5	0.2842	0.4368	0.3500	0.056*
C6	0.26119 (7)	0.59731 (15)	0.31527 (7)	0.0495 (4)
H6A	0.2609	0.6821	0.3265	0.059*
H6B	0.2894	0.5890	0.2836	0.059*
N11	0.13521 (8)	0.57704 (16)	0.21601 (6)	0.0662 (4)
C12	0.17203 (8)	0.63612 (16)	0.25019 (7)	0.0513 (4)
C13	0.18284 (11)	0.75631 (19)	0.24506 (9)	0.0716 (5)
H13	0.2101	0.7960	0.2694	0.086*
C14	0.15306 (14)	0.8180 (2)	0.20361 (11)	0.0942 (8)
H14	0.1597	0.9010	0.1990	0.113*
C15	0.11414 (13)	0.7586 (3)	0.16938 (10)	0.0927 (8)
H15	0.0927	0.7995	0.1411	0.111*
C16	0.10686 (12)	0.6393 (3)	0.17684 (9)	0.0853 (7)
H16	0.0800	0.5980	0.1527	0.102*
C27	0.08834 (8)	0.53511 (15)	0.33041 (7)	0.0493 (4)
O27	0.07514 (6)	0.43068 (11)	0.32607 (6)	0.0646 (3)
N21	0.05596 (9)	0.74111 (16)	0.32838 (8)	0.0758 (5)
C22	0.04146 (8)	0.62888 (18)	0.31706 (7)	0.0573 (4)
C23	−0.01306 (10)	0.5955 (3)	0.29329 (10)	0.0846 (7)
H23	−0.0222	0.5142	0.2861	0.102*
C24	−0.05428 (13)	0.6857 (4)	0.28028 (13)	0.1123 (10)
H24	−0.0920	0.6669	0.2630	0.135*
C25	−0.04035 (15)	0.8010 (3)	0.29238 (13)	0.1112 (10)
H25	−0.0684	0.8633	0.2847	0.133*
C26	0.01477 (14)	0.8246 (3)	0.31575 (11)	0.1002 (9)
H26	0.0246	0.9052	0.3236	0.120*

C31	0.13233 (7)	0.50623 (14)	0.44664 (7)	0.0473 (4)
C32	0.10194 (9)	0.60966 (17)	0.46100 (8)	0.0604 (4)
H32	0.1041	0.6769	0.4373	0.072*
C33	0.06841 (10)	0.6169 (2)	0.50932 (9)	0.0737 (6)
H33	0.0479	0.6885	0.5190	0.088*
C34	0.06538 (10)	0.5205 (2)	0.54226 (9)	0.0771 (6)
F34	0.03235 (8)	0.52803 (18)	0.58975 (6)	0.1177 (6)
C35	0.09327 (12)	0.4160 (2)	0.52967 (9)	0.0830 (7)
H35	0.0899	0.3491	0.5535	0.100*
C36	0.12679 (10)	0.40895 (18)	0.48118 (8)	0.0649 (5)
H36	0.1462	0.3362	0.4716	0.078*
C47	0.24466 (7)	0.64265 (13)	0.44453 (7)	0.0456 (3)
O47	0.23444 (6)	0.73801 (9)	0.42281 (5)	0.0575 (3)
N41	0.28713 (11)	0.54117 (16)	0.52388 (8)	0.0865 (6)
C42	0.26141 (9)	0.63990 (15)	0.50466 (7)	0.0561 (4)
C43	0.25094 (12)	0.73865 (19)	0.53713 (8)	0.0764 (6)
H43	0.2322	0.8079	0.5223	0.092*
C44	0.26832 (17)	0.7342 (3)	0.59181 (10)	0.1086 (10)
H44	0.2610	0.8002	0.6154	0.130*
C45	0.29594 (18)	0.6346 (3)	0.61140 (11)	0.1234 (12)
H45	0.3091	0.6302	0.6486	0.148*
C46	0.30429 (18)	0.5414 (3)	0.57659 (12)	0.1228 (12)
H46	0.3236	0.4719	0.5907	0.147*
C51	0.34937 (7)	0.54824 (14)	0.38108 (7)	0.0481 (4)
C52	0.37957 (9)	0.46579 (17)	0.41399 (9)	0.0653 (5)
H52	0.3597	0.3934	0.4234	0.078*
C53	0.43805 (9)	0.48691 (19)	0.43340 (9)	0.0705 (5)
H53	0.4584	0.4302	0.4560	0.085*
C54	0.46566 (8)	0.59140 (18)	0.41921 (8)	0.0598 (4)
F54	0.52375 (5)	0.61192 (12)	0.43809 (5)	0.0820 (4)
C55	0.43852 (8)	0.67500 (17)	0.38715 (8)	0.0621 (5)
H55	0.4592	0.7468	0.3780	0.074*
C56	0.37968 (8)	0.65291 (15)	0.36802 (8)	0.0550 (4)
H56	0.3599	0.7106	0.3456	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0533 (9)	0.0418 (8)	0.0455 (8)	0.0003 (6)	−0.0003 (7)	−0.0037 (7)
O11	0.0640 (7)	0.0490 (6)	0.0631 (7)	0.0062 (5)	−0.0044 (6)	−0.0149 (6)
C2	0.0496 (8)	0.0366 (7)	0.0447 (8)	0.0021 (6)	−0.0022 (6)	−0.0004 (6)
C3	0.0501 (8)	0.0328 (7)	0.0474 (8)	0.0012 (6)	−0.0015 (6)	0.0008 (6)
C4	0.0496 (8)	0.0340 (7)	0.0462 (8)	0.0013 (6)	−0.0034 (6)	0.0028 (6)
C5	0.0495 (9)	0.0381 (7)	0.0514 (8)	0.0022 (6)	−0.0018 (7)	−0.0023 (6)
C6	0.0483 (9)	0.0523 (9)	0.0478 (8)	0.0008 (7)	0.0011 (7)	0.0016 (7)
N11	0.0690 (10)	0.0781 (11)	0.0516 (8)	−0.0047 (8)	−0.0112 (7)	−0.0038 (8)
C12	0.0541 (9)	0.0577 (10)	0.0421 (8)	−0.0008 (7)	0.0008 (7)	−0.0011 (7)
C13	0.0880 (14)	0.0628 (12)	0.0641 (12)	−0.0082 (10)	−0.0169 (10)	0.0112 (9)

C14	0.119 (2)	0.0785 (15)	0.0852 (16)	−0.0030 (14)	−0.0186 (15)	0.0296 (13)
C15	0.0962 (18)	0.116 (2)	0.0661 (14)	0.0048 (15)	−0.0201 (12)	0.0294 (14)
C16	0.0844 (15)	0.113 (2)	0.0583 (12)	−0.0039 (14)	−0.0201 (11)	0.0039 (12)
C27	0.0525 (9)	0.0505 (9)	0.0450 (8)	−0.0014 (7)	0.0009 (7)	0.0017 (7)
O27	0.0643 (8)	0.0548 (7)	0.0748 (8)	−0.0110 (6)	−0.0088 (6)	0.0017 (6)
N21	0.0841 (12)	0.0654 (11)	0.0780 (11)	0.0245 (9)	−0.0153 (9)	−0.0006 (9)
C22	0.0535 (9)	0.0708 (12)	0.0476 (9)	0.0096 (8)	0.0003 (7)	0.0047 (8)
C23	0.0627 (12)	0.1074 (18)	0.0838 (15)	0.0044 (12)	−0.0151 (11)	0.0066 (13)
C24	0.0685 (15)	0.160 (3)	0.109 (2)	0.0285 (18)	−0.0282 (14)	0.014 (2)
C25	0.104 (2)	0.126 (3)	0.104 (2)	0.058 (2)	−0.0170 (17)	0.0143 (19)
C26	0.112 (2)	0.0883 (17)	0.1003 (18)	0.0465 (16)	−0.0226 (16)	0.0019 (14)
C31	0.0469 (8)	0.0490 (8)	0.0461 (8)	−0.0031 (7)	−0.0036 (7)	0.0001 (7)
C32	0.0650 (11)	0.0566 (10)	0.0595 (10)	0.0019 (8)	0.0082 (8)	−0.0039 (8)
C33	0.0722 (13)	0.0803 (14)	0.0686 (12)	−0.0002 (11)	0.0124 (10)	−0.0182 (11)
C34	0.0707 (13)	0.1070 (18)	0.0534 (11)	−0.0127 (12)	0.0120 (9)	−0.0094 (11)
F34	0.1163 (12)	0.1697 (16)	0.0670 (8)	−0.0229 (11)	0.0383 (8)	−0.0117 (9)
C35	0.0904 (16)	0.0956 (17)	0.0630 (12)	−0.0111 (13)	0.0090 (11)	0.0248 (12)
C36	0.0704 (12)	0.0625 (11)	0.0619 (11)	0.0012 (9)	0.0033 (9)	0.0128 (9)
C47	0.0474 (8)	0.0381 (8)	0.0513 (8)	−0.0013 (6)	0.0000 (7)	0.0017 (7)
O47	0.0793 (8)	0.0351 (6)	0.0580 (7)	0.0012 (5)	−0.0048 (6)	0.0018 (5)
N41	0.1270 (17)	0.0641 (11)	0.0682 (11)	0.0086 (10)	−0.0376 (11)	0.0004 (9)
C42	0.0673 (11)	0.0477 (9)	0.0531 (9)	−0.0099 (8)	−0.0066 (8)	0.0002 (7)
C43	0.1111 (18)	0.0619 (12)	0.0562 (11)	−0.0032 (11)	0.0002 (11)	−0.0056 (9)
C44	0.173 (3)	0.0959 (19)	0.0572 (13)	−0.0161 (19)	−0.0056 (16)	−0.0175 (13)
C45	0.196 (4)	0.112 (2)	0.0613 (15)	−0.022 (2)	−0.0468 (19)	0.0030 (15)
C46	0.196 (4)	0.0907 (19)	0.0821 (17)	0.010 (2)	−0.066 (2)	0.0076 (15)
C51	0.0480 (9)	0.0433 (8)	0.0529 (9)	0.0031 (6)	0.0018 (7)	−0.0014 (7)
C52	0.0575 (10)	0.0523 (10)	0.0862 (13)	−0.0029 (8)	−0.0101 (9)	0.0141 (9)
C53	0.0605 (11)	0.0714 (13)	0.0795 (13)	0.0020 (9)	−0.0142 (10)	0.0209 (10)
C54	0.0480 (9)	0.0718 (12)	0.0597 (10)	−0.0051 (8)	−0.0060 (8)	−0.0029 (9)
F54	0.0588 (6)	0.1041 (9)	0.0831 (8)	−0.0149 (6)	−0.0181 (6)	0.0077 (7)
C55	0.0576 (10)	0.0542 (10)	0.0744 (12)	−0.0093 (8)	−0.0008 (9)	0.0043 (9)
C56	0.0523 (9)	0.0492 (9)	0.0634 (10)	0.0012 (7)	−0.0025 (8)	0.0062 (8)

Geometric parameters (Å, º)

C1—O11	1.4279 (19)	C25—C26	1.359 (4)
C1—C6	1.527 (2)	C25—H25	0.9500
C1—C12	1.529 (2)	C26—H26	0.9500
C1—C2	1.565 (2)	C31—C36	1.382 (2)
O11—H11	0.8158	C31—C32	1.383 (2)
C2—C27	1.519 (2)	C32—C33	1.386 (3)
C2—C3	1.539 (2)	C32—H32	0.9500
C2—H2	1.0000	C33—C34	1.347 (3)
C3—C31	1.520 (2)	C33—H33	0.9500
C3—C4	1.551 (2)	C34—C35	1.357 (4)
C3—H3	1.0000	C34—F34	1.363 (2)
C4—C47	1.514 (2)	C35—C36	1.389 (3)

C4—C5	1.550 (2)	C35—H35	0.9500
C4—H4	1.0000	C36—H36	0.9500
C5—C51	1.519 (2)	C47—O47	1.2140 (19)
C5—C6	1.531 (2)	C47—C42	1.505 (2)
C5—H5	1.0000	N41—C42	1.328 (3)
C6—H6A	0.9900	N41—C46	1.333 (3)
C6—H6B	0.9900	C42—C43	1.379 (3)
N11—C12	1.333 (2)	C43—C44	1.381 (3)
N11—C16	1.333 (3)	C43—H43	0.9500
C12—C13	1.376 (3)	C44—C45	1.357 (4)
C13—C14	1.384 (3)	C44—H44	0.9500
C13—H13	0.9500	C45—C46	1.357 (4)
C14—C15	1.364 (4)	C45—H45	0.9500
C14—H14	0.9500	C46—H46	0.9500
C15—C16	1.361 (4)	C51—C56	1.386 (2)
C15—H15	0.9500	C51—C52	1.390 (2)
C16—H16	0.9500	C52—C53	1.384 (3)
C27—O27	1.212 (2)	C52—H52	0.9500
C27—C22	1.505 (2)	C53—C54	1.364 (3)
N21—C22	1.328 (3)	C53—H53	0.9500
N21—C26	1.336 (3)	C54—C55	1.356 (3)
C22—C23	1.378 (3)	C54—F54	1.371 (2)
C23—C24	1.393 (4)	C55—C56	1.391 (2)
C23—H23	0.9500	C55—H55	0.9500
C24—C25	1.362 (4)	C56—H56	0.9500
C24—H24	0.9500		

O11—C1—C6	106.76 (13)	C25—C24—H24	120.1
O11—C1—C12	109.29 (13)	C23—C24—H24	120.1
C6—C1—C12	112.97 (13)	C26—C25—C24	118.3 (2)
O11—C1—C2	110.43 (13)	C26—C25—H25	120.9
C6—C1—C2	109.49 (13)	C24—C25—H25	120.9
C12—C1—C2	107.91 (13)	N21—C26—C25	123.9 (3)
C1—O11—H11	105.9	N21—C26—H26	118.0
C27—C2—C3	109.78 (12)	C25—C26—H26	118.0
C27—C2—C1	109.77 (13)	C36—C31—C32	117.94 (17)
C3—C2—C1	110.82 (12)	C36—C31—C3	119.17 (15)
C27—C2—H2	108.8	C32—C31—C3	122.80 (15)
C3—C2—H2	108.8	C31—C32—C33	121.13 (19)
C1—C2—H2	108.8	C31—C32—H32	119.4
C31—C3—C2	113.55 (13)	C33—C32—H32	119.4
C31—C3—C4	109.86 (12)	C34—C33—C32	118.8 (2)
C2—C3—C4	112.52 (12)	C34—C33—H33	120.6
C31—C3—H3	106.8	C32—C33—H33	120.6
C2—C3—H3	106.8	C33—C34—C35	122.59 (19)
C4—C3—H3	106.8	C33—C34—F34	118.6 (2)
C47—C4—C5	112.67 (12)	C35—C34—F34	118.9 (2)
C47—C4—C3	111.22 (12)	C34—C35—C36	118.5 (2)

C5—C4—C3	111.44 (12)	C34—C35—H35	120.7
C47—C4—H4	107.1	C36—C35—H35	120.7
C5—C4—H4	107.1	C31—C36—C35	121.0 (2)
C3—C4—H4	107.1	C31—C36—H36	119.5
C51—C5—C6	114.48 (13)	C35—C36—H36	119.5
C51—C5—C4	110.25 (13)	O47—C47—C42	118.94 (15)
C6—C5—C4	112.07 (13)	O47—C47—C4	122.49 (14)
C51—C5—H5	106.5	C42—C47—C4	118.56 (13)
C6—C5—H5	106.5	C42—N41—C46	117.1 (2)
C4—C5—H5	106.5	N41—C42—C43	122.75 (18)
C1—C6—C5	111.55 (13)	N41—C42—C47	117.45 (16)
C1—C6—H6A	109.3	C43—C42—C47	119.79 (17)
C5—C6—H6A	109.3	C42—C43—C44	118.3 (2)
C1—C6—H6B	109.3	C42—C43—H43	120.9
C5—C6—H6B	109.3	C44—C43—H43	120.9
H6A—C6—H6B	108.0	C45—C44—C43	119.3 (3)
C12—N11—C16	117.64 (19)	C45—C44—H44	120.4
N11—C12—C13	122.39 (17)	C43—C44—H44	120.4
N11—C12—C1	114.04 (15)	C44—C45—C46	118.5 (2)
C13—C12—C1	123.42 (16)	C44—C45—H45	120.7
C12—C13—C14	118.4 (2)	C46—C45—H45	120.7
C12—C13—H13	120.8	N41—C46—C45	124.1 (3)
C14—C13—H13	120.8	N41—C46—H46	118.0
C15—C14—C13	119.5 (2)	C45—C46—H46	118.0
C15—C14—H14	120.3	C56—C51—C52	117.92 (16)
C13—C14—H14	120.3	C56—C51—C5	123.61 (15)
C16—C15—C14	118.2 (2)	C52—C51—C5	118.43 (15)
C16—C15—H15	120.9	C53—C52—C51	121.41 (18)
C14—C15—H15	120.9	C53—C52—H52	119.3
N11—C16—C15	123.8 (2)	C51—C52—H52	119.3
N11—C16—H16	118.1	C54—C53—C52	118.12 (18)
C15—C16—H16	118.1	C54—C53—H53	120.9
O27—C27—C22	119.69 (16)	C52—C53—H53	120.9
O27—C27—C2	121.13 (15)	C55—C54—C53	123.09 (17)
C22—C27—C2	119.16 (15)	C55—C54—F54	118.79 (17)
C22—N21—C26	117.2 (2)	C53—C54—F54	118.11 (17)
N21—C22—C23	123.49 (19)	C54—C55—C56	118.26 (17)
N21—C22—C27	117.14 (16)	C54—C55—H55	120.9
C23—C22—C27	119.36 (19)	C56—C55—H55	120.9
C22—C23—C24	117.3 (3)	C51—C56—C55	121.20 (16)
C22—C23—H23	121.4	C51—C56—H56	119.4
C24—C23—H23	121.4	C55—C56—H56	119.4
C25—C24—C23	119.8 (3)		

O11—C1—C2—C27	−61.80 (16)	C22—C23—C24—C25	−1.5 (4)
C6—C1—C2—C27	−179.08 (13)	C23—C24—C25—C26	1.7 (5)
C12—C1—C2—C27	57.59 (16)	C22—N21—C26—C25	0.1 (4)
O11—C1—C2—C3	59.62 (16)	C24—C25—C26—N21	−1.0 (5)

C6—C1—C2—C3	−57.65 (16)	C2—C3—C31—C36	148.77 (15)
C12—C1—C2—C3	179.02 (13)	C4—C3—C31—C36	−84.24 (18)
C27—C2—C3—C31	−58.61 (16)	C2—C3—C31—C32	−34.7 (2)
C1—C2—C3—C31	179.97 (12)	C4—C3—C31—C32	92.30 (18)
C27—C2—C3—C4	175.80 (12)	C36—C31—C32—C33	1.9 (3)
C1—C2—C3—C4	54.37 (16)	C3—C31—C32—C33	−174.72 (17)
C31—C3—C4—C47	−51.99 (16)	C31—C32—C33—C34	−0.5 (3)
C2—C3—C4—C47	75.59 (16)	C32—C33—C34—C35	−0.8 (4)
C31—C3—C4—C5	−178.62 (12)	C32—C33—C34—F34	180.0 (2)
C2—C3—C4—C5	−51.05 (16)	C33—C34—C35—C36	0.7 (4)
C47—C4—C5—C51	54.51 (16)	F34—C34—C35—C36	179.9 (2)
C3—C4—C5—C51	−179.66 (12)	C32—C31—C36—C35	−1.9 (3)
C47—C4—C5—C6	−74.26 (16)	C3—C31—C36—C35	174.78 (19)
C3—C4—C5—C6	51.57 (16)	C34—C35—C36—C31	0.7 (3)
O11—C1—C6—C5	−60.66 (17)	C5—C4—C47—O47	58.7 (2)
C12—C1—C6—C5	179.18 (13)	C3—C4—C47—O47	−67.30 (19)
C2—C1—C6—C5	58.90 (17)	C5—C4—C47—C42	−121.88 (15)
C51—C5—C6—C1	176.87 (13)	C3—C4—C47—C42	112.16 (16)
C4—C5—C6—C1	−56.61 (17)	C46—N41—C42—C43	−1.6 (4)
C16—N11—C12—C13	−2.1 (3)	C46—N41—C42—C47	177.1 (3)
C16—N11—C12—C1	173.54 (18)	O47—C47—C42—N41	−163.26 (19)
O11—C1—C12—N11	28.5 (2)	C4—C47—C42—N41	17.3 (3)
C6—C1—C12—N11	147.25 (15)	O47—C47—C42—C43	15.5 (3)
C2—C1—C12—N11	−91.57 (17)	C4—C47—C42—C43	−164.01 (18)
O11—C1—C12—C13	−155.82 (18)	N41—C42—C43—C44	0.3 (4)
C6—C1—C12—C13	−37.1 (2)	C47—C42—C43—C44	−178.3 (2)
C2—C1—C12—C13	84.1 (2)	C42—C43—C44—C45	1.2 (5)
N11—C12—C13—C14	1.6 (3)	C43—C44—C45—C46	−1.4 (6)
C1—C12—C13—C14	−173.7 (2)	C42—N41—C46—C45	1.4 (5)
C12—C13—C14—C15	0.1 (4)	C44—C45—C46—N41	0.1 (6)
C13—C14—C15—C16	−1.1 (4)	C6—C5—C51—C56	18.5 (2)
C12—N11—C16—C15	1.0 (4)	C4—C5—C51—C56	−108.96 (18)
C14—C15—C16—N11	0.6 (4)	C6—C5—C51—C52	−163.96 (16)
C3—C2—C27—O27	−45.1 (2)	C4—C5—C51—C52	68.6 (2)
C1—C2—C27—O27	76.93 (19)	C56—C51—C52—C53	0.1 (3)
C3—C2—C27—C22	136.74 (14)	C5—C51—C52—C53	−177.57 (19)
C1—C2—C27—C22	−101.21 (16)	C51—C52—C53—C54	−0.2 (3)
C26—N21—C22—C23	0.1 (3)	C52—C53—C54—C55	0.0 (3)
C26—N21—C22—C27	179.0 (2)	C52—C53—C54—F54	−179.53 (19)
O27—C27—C22—N21	172.63 (17)	C53—C54—C55—C56	0.2 (3)
C2—C27—C22—N21	−9.2 (2)	F54—C54—C55—C56	179.70 (17)
O27—C27—C22—C23	−8.4 (3)	C52—C51—C56—C55	0.1 (3)
C2—C27—C22—C23	169.76 (18)	C5—C51—C56—C55	177.62 (16)
N21—C22—C23—C24	0.6 (4)	C54—C55—C56—C51	−0.2 (3)
C27—C22—C23—C24	−178.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H11···N11	0.82	2.13	2.624 (2)	119
C13—H13···O11^{i}	0.95	2.54	3.385 (3)	148
C55—H55···O27^{i}	0.95	2.53	3.244 (2)	132

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

(II) (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)-3,5-Bis(4-chlorophenyl)-2,4-bis(pyridine-2-carbonyl)-1-(pyridin-2-yl)cyclohexanol acetone 0.951-solvate

Crystal data

$C_{35}H_{27}Cl_2N_3O_3 \cdot 0.951C_3H_6O$
M_r = 663.73
Orthorhombic, *Fdd*2
a = 16.5446 (6) Å
b = 53.4204 (17) Å
c = 15.5857 (4) Å
V = 13774.9 (8) Å^3
Z = 16
F(000) = 5543
D_x = 1.280 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54178 Å
Cell parameters from 4224 reflections
θ = 4.6–70.1°
μ = 2.05 mm^{-1}
T = 200 K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.388, T_{max} = 0.664
9544 measured reflections
4224 independent reflections
3490 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 70.1°, θ_{min} = 4.6°
h = −19→20
k = −56→64
l = −18→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 0.97
4224 reflections
436 parameters
7 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
$w = 1/[\sigma^2(F_o^2) + (0.0744P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack x determined using 613 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013).
Absolute structure parameter: 0.089 (18)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.25172 (19)	0.35490 (6)	0.4632 (2)	0.0505 (6)	
O11	0.20607 (15)	0.35606 (5)	0.54074 (15)	0.0647 (6)	
H11	0.2379	0.3467	0.5731	0.097*	
C2	0.21280 (17)	0.33568 (5)	0.40064 (19)	0.0481 (6)	
H2	0.2440	0.3357	0.3457	0.058*	
C3	0.12475 (17)	0.34306 (5)	0.38147 (19)	0.0460 (6)	
H3	0.0962	0.3435	0.4380	0.055*	
C4	0.11812 (16)	0.36986 (5)	0.34352 (19)	0.0454 (6)	
H4	0.0596	0.3745	0.3438	0.054*	
C5	0.16302 (18)	0.38897 (5)	0.4017 (2)	0.0489 (6)	
H5	0.1336	0.3891	0.4577	0.059*	
C6	0.24983 (19)	0.38087 (5)	0.4218 (2)	0.0526 (6)	
H6A	0.2818	0.3806	0.3681	0.063*	
H6B	0.2750	0.3932	0.4611	0.063*	
N11	0.3517 (2)	0.33975 (6)	0.5638 (2)	0.0679 (7)	
C12	0.33845 (19)	0.34663 (6)	0.4829 (2)	0.0546 (7)	
C13	0.3972 (2)	0.34512 (7)	0.4207 (3)	0.0649 (8)	
H13	0.3865	0.3509	0.3642	0.078*	
C14	0.4712 (2)	0.33528 (9)	0.4409 (3)	0.0809 (11)	
H14	0.5121	0.3338	0.3984	0.097*	
C15	0.4854 (3)	0.32758 (9)	0.5246 (4)	0.0876 (13)	
H15	0.5356	0.3204	0.5407	0.105*	
C16	0.4251 (3)	0.33070 (9)	0.5827 (3)	0.0862 (13)	
H16	0.4357	0.3262	0.6405	0.103*	
C27	0.21686 (19)	0.30956 (6)	0.4398 (2)	0.0541 (7)	
O27	0.16848 (16)	0.30235 (5)	0.4917 (2)	0.0735 (7)	
N21	0.3165 (2)	0.29660 (7)	0.3350 (3)	0.0837 (10)	
C22	0.2864 (2)	0.29305 (6)	0.4138 (3)	0.0620 (8)	
C23	0.3160 (3)	0.27562 (7)	0.4698 (4)	0.0841 (12)	
H23	0.2924	0.2736	0.5249	0.101*	
C24	0.3807 (4)	0.26106 (10)	0.4446 (5)	0.1104 (18)	
H24	0.4038	0.2492	0.4829	0.132*	
C25	0.4108 (4)	0.26401 (11)	0.3641 (5)	0.116 (2)	
H25	0.4540	0.2537	0.3445	0.139*	
C26	0.3785 (3)	0.28192 (12)	0.3115 (4)	0.1066 (17)	
H26	0.4009	0.2841	0.2559	0.128*	
C31	0.08089 (18)	0.32368 (6)	0.3270 (2)	0.0513 (6)	
C32	0.1182 (2)	0.31044 (7)	0.2615 (3)	0.0637 (8)	
H32	0.1729	0.3141	0.2477	0.076*	
C33	0.0783 (2)	0.29211 (8)	0.2158 (3)	0.0746 (10)	
H33	0.1049	0.2832	0.1712	0.090*	
C34	−0.0008 (3)	0.28699 (7)	0.2360 (3)	0.0757 (10)	
Cl34	−0.05239 (11)	0.26346 (3)	0.18053 (12)	0.1225 (6)	
C35	−0.0399 (2)	0.29974 (8)	0.2996 (3)	0.0764 (11)	
H35	−0.0946	0.2960	0.3126	0.092*	

C36	0.0003 (2)	0.31802 (7)	0.3446 (3)	0.0631 (8)	
H36	−0.0272	0.3269	0.3884	0.076*	
C47	0.14717 (16)	0.37140 (5)	0.25098 (19)	0.0451 (6)	
O47	0.21661 (12)	0.36662 (5)	0.23092 (16)	0.0607 (6)	
N41	0.01329 (16)	0.38537 (6)	0.2066 (2)	0.0646 (7)	
C42	0.08830 (17)	0.37945 (5)	0.1827 (2)	0.0488 (6)	
C43	0.1135 (2)	0.38102 (7)	0.0983 (2)	0.0618 (8)	
H43	0.1672	0.3765	0.0831	0.074*	
C44	0.0603 (2)	0.38919 (8)	0.0363 (3)	0.0751 (10)	
H44	0.0768	0.3907	−0.0218	0.090*	
C45	−0.0167 (3)	0.39508 (8)	0.0603 (3)	0.0782 (11)	
H45	−0.0553	0.4005	0.0192	0.094*	
C46	−0.0367 (2)	0.39294 (9)	0.1454 (3)	0.0794 (11)	
H46	−0.0903	0.3972	0.1616	0.095*	
C51	0.15672 (19)	0.41541 (6)	0.3660 (2)	0.0508 (6)	
C52	0.0822 (2)	0.42707 (7)	0.3642 (3)	0.0706 (10)	
H52	0.0362	0.4185	0.3856	0.085*	
C53	0.0733 (3)	0.45099 (8)	0.3318 (4)	0.0804 (11)	
H53	0.0215	0.4587	0.3302	0.096*	
C54	0.1397 (3)	0.46354 (7)	0.3021 (3)	0.0725 (10)	
Cl54	0.12896 (10)	0.49384 (2)	0.26117 (12)	0.1142 (5)	
C55	0.2146 (3)	0.45280 (7)	0.3033 (3)	0.0726 (10)	
H55	0.2603	0.4617	0.2825	0.087*	
C56	0.2225 (2)	0.42862 (6)	0.3355 (3)	0.0623 (8)	
H56	0.2744	0.4210	0.3366	0.075*	
C71	0.6495 (16)	0.2977 (7)	0.2767 (10)	0.125 (3)	0.690 (13)
O71	0.5974 (5)	0.3120 (2)	0.2778 (8)	0.159 (4)	0.690 (13)
C72	0.7036 (14)	0.2906 (3)	0.2035 (9)	0.174 (5)	0.690 (13)
H72A	0.7137	0.2726	0.2047	0.261*	0.690 (13)
H72B	0.7550	0.2996	0.2087	0.261*	0.690 (13)
H72C	0.6776	0.2951	0.1492	0.261*	0.690 (13)
C73	0.6809 (14)	0.2869 (3)	0.3563 (8)	0.174 (5)	0.690 (13)
H73A	0.7396	0.2893	0.3587	0.261*	0.690 (13)
H73B	0.6686	0.2690	0.3581	0.261*	0.690 (13)
H73C	0.6557	0.2953	0.4055	0.261*	0.690 (13)
C81	0.664 (4)	0.293 (2)	0.285 (2)	0.125 (3)	0.261 (11)
O81	0.6093 (14)	0.2987 (6)	0.326 (2)	0.159 (4)	0.261 (11)
C82	0.680 (3)	0.2969 (10)	0.1916 (17)	0.174 (5)	0.261 (11)
H82A	0.7179	0.2841	0.1711	0.261*	0.261 (11)
H82B	0.7035	0.3135	0.1825	0.261*	0.261 (11)
H82C	0.6291	0.2956	0.1599	0.261*	0.261 (11)
C83	0.731 (2)	0.2787 (7)	0.323 (2)	0.174 (5)	0.261 (11)
H83A	0.7726	0.2758	0.2799	0.261*	0.261 (11)
H83B	0.7101	0.2626	0.3440	0.261*	0.261 (11)
H83C	0.7539	0.2881	0.3714	0.261*	0.261 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0563 (14)	0.0556 (15)	0.0397 (13)	0.0015 (13)	0.0020 (13)	0.0001 (12)
O11	0.0749 (13)	0.0745 (14)	0.0445 (12)	0.0068 (12)	0.0082 (11)	−0.0015 (11)
C2	0.0501 (13)	0.0502 (13)	0.0440 (14)	0.0029 (12)	0.0040 (12)	0.0005 (12)
C3	0.0469 (13)	0.0504 (13)	0.0409 (13)	0.0026 (11)	0.0056 (11)	0.0026 (11)
C4	0.0413 (12)	0.0505 (14)	0.0443 (14)	0.0055 (11)	0.0058 (11)	0.0000 (12)
C5	0.0552 (14)	0.0497 (13)	0.0419 (13)	0.0031 (12)	0.0075 (13)	−0.0010 (11)
C6	0.0576 (15)	0.0507 (14)	0.0495 (15)	−0.0007 (13)	−0.0049 (13)	−0.0007 (13)
N11	0.0849 (19)	0.0655 (15)	0.0533 (15)	0.0078 (15)	−0.0128 (16)	0.0091 (13)
C12	0.0625 (16)	0.0502 (14)	0.0512 (16)	−0.0030 (13)	−0.0109 (15)	0.0016 (13)
C13	0.0551 (16)	0.080 (2)	0.0597 (19)	−0.0013 (16)	−0.0068 (16)	0.0029 (17)
C14	0.0621 (19)	0.092 (3)	0.088 (3)	0.0039 (19)	−0.008 (2)	0.002 (2)
C15	0.074 (2)	0.086 (3)	0.104 (3)	0.015 (2)	−0.029 (3)	0.006 (3)
C16	0.105 (3)	0.084 (3)	0.070 (2)	0.019 (2)	−0.030 (3)	0.015 (2)
C27	0.0583 (15)	0.0555 (15)	0.0485 (15)	−0.0016 (13)	−0.0029 (14)	0.0026 (13)
O27	0.0774 (14)	0.0687 (14)	0.0743 (17)	0.0041 (12)	0.0120 (14)	0.0199 (13)
N21	0.088 (2)	0.093 (2)	0.070 (2)	0.0334 (19)	0.0021 (18)	−0.0136 (18)
C22	0.0672 (17)	0.0499 (15)	0.069 (2)	0.0054 (14)	−0.0081 (17)	−0.0079 (15)
C23	0.097 (3)	0.0609 (19)	0.094 (3)	0.019 (2)	−0.003 (3)	0.011 (2)
C24	0.123 (4)	0.077 (3)	0.131 (5)	0.042 (3)	−0.014 (4)	0.004 (3)
C25	0.122 (4)	0.102 (4)	0.123 (5)	0.061 (3)	−0.013 (4)	−0.039 (4)
C26	0.110 (3)	0.128 (4)	0.081 (3)	0.047 (3)	0.011 (3)	−0.030 (3)
C31	0.0516 (14)	0.0498 (14)	0.0526 (16)	0.0002 (12)	−0.0016 (13)	0.0070 (13)
C32	0.0637 (16)	0.0635 (17)	0.064 (2)	0.0005 (15)	−0.0012 (17)	−0.0067 (16)
C33	0.086 (2)	0.067 (2)	0.071 (2)	0.0000 (18)	−0.016 (2)	−0.0121 (18)
C34	0.090 (2)	0.0581 (17)	0.079 (3)	−0.0081 (18)	−0.031 (2)	0.0038 (18)
Cl34	0.1446 (11)	0.0949 (8)	0.1281 (12)	−0.0398 (8)	−0.0502 (10)	−0.0146 (8)
C35	0.0620 (18)	0.081 (2)	0.086 (3)	−0.0154 (19)	−0.0148 (19)	0.014 (2)
C36	0.0564 (16)	0.0667 (19)	0.066 (2)	−0.0026 (15)	−0.0054 (16)	0.0083 (17)
C47	0.0442 (13)	0.0472 (12)	0.0440 (15)	0.0009 (11)	0.0069 (12)	−0.0014 (11)
O47	0.0481 (10)	0.0849 (15)	0.0491 (11)	0.0098 (10)	0.0084 (10)	0.0015 (11)
N41	0.0513 (13)	0.0835 (18)	0.0588 (16)	0.0139 (13)	0.0057 (13)	0.0086 (15)
C42	0.0477 (13)	0.0507 (13)	0.0479 (15)	−0.0038 (11)	0.0003 (13)	0.0001 (12)
C43	0.0542 (16)	0.082 (2)	0.0494 (17)	−0.0068 (15)	0.0071 (15)	0.0027 (16)
C44	0.079 (2)	0.093 (3)	0.0528 (19)	−0.010 (2)	−0.0045 (18)	0.0080 (19)
C45	0.080 (2)	0.083 (2)	0.072 (2)	0.003 (2)	−0.022 (2)	0.016 (2)
C46	0.0548 (17)	0.101 (3)	0.083 (3)	0.0193 (19)	−0.0018 (19)	0.014 (2)
C51	0.0590 (15)	0.0487 (14)	0.0448 (14)	0.0031 (13)	0.0035 (13)	−0.0028 (12)
C52	0.0645 (18)	0.0581 (17)	0.089 (3)	0.0055 (16)	0.0117 (19)	0.0037 (18)
C53	0.076 (2)	0.062 (2)	0.103 (3)	0.0158 (18)	−0.003 (2)	0.008 (2)
C54	0.098 (3)	0.0495 (16)	0.070 (2)	0.0066 (18)	−0.007 (2)	0.0090 (16)
Cl54	0.1386 (10)	0.0630 (5)	0.1409 (12)	0.0055 (6)	−0.0158 (10)	0.0338 (6)
C55	0.084 (2)	0.0572 (17)	0.077 (2)	−0.0077 (18)	0.006 (2)	0.0091 (17)
C56	0.0596 (16)	0.0565 (16)	0.071 (2)	0.0020 (14)	0.0073 (17)	0.0020 (16)
C71	0.138 (11)	0.106 (16)	0.132 (7)	−0.032 (6)	0.022 (7)	0.008 (6)
O71	0.130 (5)	0.161 (8)	0.186 (11)	0.012 (5)	0.014 (6)	0.015 (7)

C72	0.286 (14)	0.131 (8)	0.104 (5)	−0.013 (8)	0.028 (7)	0.016 (4)
C73	0.286 (14)	0.131 (8)	0.104 (5)	−0.013 (8)	0.028 (7)	0.016 (4)
C81	0.138 (11)	0.106 (16)	0.132 (7)	−0.032 (6)	0.022 (7)	0.008 (6)
O81	0.130 (5)	0.161 (8)	0.186 (11)	0.012 (5)	0.014 (6)	0.015 (7)
C82	0.286 (14)	0.131 (8)	0.104 (5)	−0.013 (8)	0.028 (7)	0.016 (4)
C83	0.286 (14)	0.131 (8)	0.104 (5)	−0.013 (8)	0.028 (7)	0.016 (4)

Geometric parameters (Å, º)

C1—O11	1.426 (4)	C34—C35	1.365 (7)
C1—C6	1.531 (4)	C34—Cl34	1.748 (4)
C1—C12	1.533 (4)	C35—C36	1.374 (6)
C1—C2	1.556 (4)	C35—H35	0.9500
O11—H11	0.8847	C36—H36	0.9500
C2—C27	1.524 (4)	C47—O47	1.218 (3)
C2—C3	1.539 (4)	C47—C42	1.506 (4)
C2—H2	1.0000	N41—C46	1.326 (5)
C3—C31	1.523 (4)	N41—C42	1.334 (4)
C3—C4	1.552 (4)	C42—C43	1.382 (5)
C3—H3	1.0000	C43—C44	1.378 (5)
C4—C47	1.523 (4)	C43—H43	0.9500
C4—C5	1.555 (4)	C44—C45	1.364 (6)
C4—H4	1.0000	C44—H44	0.9500
C5—C51	1.521 (4)	C45—C46	1.372 (7)
C5—C6	1.532 (4)	C45—H45	0.9500
C5—H5	1.0000	C46—H46	0.9500
C6—H6A	0.9900	C51—C52	1.381 (5)
C6—H6B	0.9900	C51—C56	1.382 (5)
N11—C12	1.331 (5)	C52—C53	1.382 (5)
N11—C16	1.340 (5)	C52—H52	0.9500
C12—C13	1.375 (5)	C53—C54	1.368 (6)
C13—C14	1.370 (6)	C53—H53	0.9500
C13—H13	0.9500	C54—C55	1.365 (6)
C14—C15	1.387 (8)	C54—Cl54	1.749 (4)
C14—H14	0.9500	C55—C56	1.392 (5)
C15—C16	1.357 (7)	C55—H55	0.9500
C15—H15	0.9500	C56—H56	0.9500
C16—H16	0.9500	C71—O71	1.151 (13)
C27—O27	1.201 (4)	C71—C73	1.464 (17)
C27—C22	1.506 (5)	C71—C72	1.498 (14)
N21—C22	1.338 (6)	C72—H72A	0.9800
N21—C26	1.342 (5)	C72—H72B	0.9800
C22—C23	1.366 (6)	C72—H72C	0.9800
C23—C24	1.380 (7)	C73—H73A	0.9800
C23—H23	0.9500	C73—H73B	0.9800
C24—C25	1.360 (10)	C73—H73C	0.9800
C24—H24	0.9500	C81—O81	1.150 (13)
C25—C26	1.369 (9)	C81—C83	1.463 (18)

C25—H25	0.9500	C81—C82	1.497 (15)
C26—H26	0.9500	C82—H82A	0.9800
C31—C32	1.386 (5)	C82—H82B	0.9800
C31—C36	1.394 (5)	C82—H82C	0.9800
C32—C33	1.380 (5)	C83—H83A	0.9800
C32—H32	0.9500	C83—H83B	0.9800
C33—C34	1.373 (6)	C83—H83C	0.9800
C33—H33	0.9500		

O11—C1—C6	107.9 (2)	C34—C33—H33	120.7
O11—C1—C12	109.8 (3)	C32—C33—H33	120.7
C6—C1—C12	111.4 (3)	C35—C34—C33	121.2 (4)
O11—C1—C2	109.9 (2)	C35—C34—Cl34	119.1 (3)
C6—C1—C2	109.0 (2)	C33—C34—Cl34	119.6 (4)
C12—C1—C2	108.8 (2)	C34—C35—C36	119.7 (3)
C1—O11—H11	98.2	C34—C35—H35	120.1
C27—C2—C3	110.7 (2)	C36—C35—H35	120.1
C27—C2—C1	109.6 (2)	C35—C36—C31	121.1 (4)
C3—C2—C1	110.1 (2)	C35—C36—H36	119.5
C27—C2—H2	108.8	C31—C36—H36	119.5
C3—C2—H2	108.8	O47—C47—C42	119.2 (3)
C1—C2—H2	108.8	O47—C47—C4	122.0 (3)
C31—C3—C2	112.6 (2)	C42—C47—C4	118.8 (2)
C31—C3—C4	112.4 (2)	C46—N41—C42	116.8 (3)
C2—C3—C4	112.2 (2)	N41—C42—C43	122.2 (3)
C31—C3—H3	106.4	N41—C42—C47	118.1 (3)
C2—C3—H3	106.4	C43—C42—C47	119.6 (3)
C4—C3—H3	106.4	C44—C43—C42	119.5 (3)
C47—C4—C3	112.9 (2)	C44—C43—H43	120.2
C47—C4—C5	111.5 (2)	C42—C43—H43	120.2
C3—C4—C5	110.5 (2)	C45—C44—C43	118.6 (4)
C47—C4—H4	107.2	C45—C44—H44	120.7
C3—C4—H4	107.2	C43—C44—H44	120.7
C5—C4—H4	107.2	C44—C45—C46	118.1 (4)
C51—C5—C6	113.7 (2)	C44—C45—H45	121.0
C51—C5—C4	111.3 (2)	C46—C45—H45	121.0
C6—C5—C4	112.4 (2)	N41—C46—C45	124.8 (4)
C51—C5—H5	106.3	N41—C46—H46	117.6
C6—C5—H5	106.3	C45—C46—H46	117.6
C4—C5—H5	106.3	C52—C51—C56	117.7 (3)
C1—C6—C5	111.2 (2)	C52—C51—C5	119.2 (3)
C1—C6—H6A	109.4	C56—C51—C5	123.1 (3)
C5—C6—H6A	109.4	C51—C52—C53	121.4 (4)
C1—C6—H6B	109.4	C51—C52—H52	119.3
C5—C6—H6B	109.4	C53—C52—H52	119.3
H6A—C6—H6B	108.0	C54—C53—C52	119.4 (4)
C12—N11—C16	117.2 (4)	C54—C53—H53	120.3
N11—C12—C13	122.3 (3)	C52—C53—H53	120.3

N11—C12—C1	115.1 (3)	C55—C54—C53	121.2 (3)
C13—C12—C1	122.5 (3)	C55—C54—Cl54	119.1 (3)
C14—C13—C12	119.5 (4)	C53—C54—Cl54	119.7 (3)
C14—C13—H13	120.3	C54—C55—C56	118.7 (4)
C12—C13—H13	120.3	C54—C55—H55	120.6
C13—C14—C15	118.7 (5)	C56—C55—H55	120.6
C13—C14—H14	120.6	C51—C56—C55	121.6 (3)
C15—C14—H14	120.6	C51—C56—H56	119.2
C16—C15—C14	117.8 (4)	C55—C56—H56	119.2
C16—C15—H15	121.1	O71—C71—C73	120.9 (14)
C14—C15—H15	121.1	O71—C71—C72	128.7 (13)
N11—C16—C15	124.3 (4)	C73—C71—C72	109.5 (11)
N11—C16—H16	117.8	C71—C72—H72A	109.5
C15—C16—H16	117.8	C71—C72—H72B	109.5
O27—C27—C22	120.2 (3)	H72A—C72—H72B	109.5
O27—C27—C2	122.2 (3)	C71—C72—H72C	109.5
C22—C27—C2	117.5 (3)	H72A—C72—H72C	109.5
C22—N21—C26	116.8 (4)	H72B—C72—H72C	109.5
N21—C22—C23	123.3 (4)	C71—C73—H73A	109.5
N21—C22—C27	116.6 (3)	C71—C73—H73B	109.5
C23—C22—C27	120.1 (4)	H73A—C73—H73B	109.5
C22—C23—C24	118.7 (5)	C71—C73—H73C	109.5
C22—C23—H23	120.6	H73A—C73—H73C	109.5
C24—C23—H23	120.6	H73B—C73—H73C	109.5
C25—C24—C23	118.8 (5)	O81—C81—C83	120.7 (17)
C25—C24—H24	120.6	O81—C81—C82	129.5 (16)
C23—C24—H24	120.6	C83—C81—C82	109.7 (13)
C24—C25—C26	119.3 (5)	C81—C82—H82A	109.5
C24—C25—H25	120.3	C81—C82—H82B	109.5
C26—C25—H25	120.3	H82A—C82—H82B	109.5
N21—C26—C25	123.0 (6)	C81—C82—H82C	109.5
N21—C26—H26	118.5	H82A—C82—H82C	109.5
C25—C26—H26	118.5	H82B—C82—H82C	109.5
C32—C31—C36	117.4 (3)	C81—C83—H83A	109.5
C32—C31—C3	123.0 (3)	C81—C83—H83B	109.5
C36—C31—C3	119.5 (3)	H83A—C83—H83B	109.5
C33—C32—C31	121.9 (3)	C81—C83—H83C	109.5
C33—C32—H32	119.0	H83A—C83—H83C	109.5
C31—C32—H32	119.0	H83B—C83—H83C	109.5
C34—C33—C32	118.6 (4)		

O11—C1—C2—C27	−63.9 (3)	C22—C23—C24—C25	−2.2 (8)
C6—C1—C2—C27	178.1 (2)	C23—C24—C25—C26	2.5 (10)
C12—C1—C2—C27	56.4 (3)	C22—N21—C26—C25	0.0 (8)
O11—C1—C2—C3	58.2 (3)	C24—C25—C26—N21	−1.4 (10)
C6—C1—C2—C3	−59.9 (3)	C2—C3—C31—C32	−36.8 (4)
C12—C1—C2—C3	178.5 (2)	C4—C3—C31—C32	91.0 (4)
C27—C2—C3—C31	−53.4 (3)	C2—C3—C31—C36	140.7 (3)

C1—C2—C3—C31	−174.8 (2)	C4—C3—C31—C36	−91.4 (3)
C27—C2—C3—C4	178.6 (2)	C36—C31—C32—C33	−0.8 (5)
C1—C2—C3—C4	57.3 (3)	C3—C31—C32—C33	176.8 (3)
C31—C3—C4—C47	−55.0 (3)	C31—C32—C33—C34	0.2 (6)
C2—C3—C4—C47	73.1 (3)	C32—C33—C34—C35	0.3 (6)
C31—C3—C4—C5	179.5 (2)	C32—C33—C34—Cl34	−178.8 (3)
C2—C3—C4—C5	−52.4 (3)	C33—C34—C35—C36	−0.1 (6)
C47—C4—C5—C51	54.1 (3)	Cl34—C34—C35—C36	179.0 (3)
C3—C4—C5—C51	−179.6 (2)	C34—C35—C36—C31	−0.6 (6)
C47—C4—C5—C6	−74.7 (3)	C32—C31—C36—C35	1.0 (5)
C3—C4—C5—C6	51.6 (3)	C3—C31—C36—C35	−176.7 (3)
O11—C1—C6—C5	−59.7 (3)	C3—C4—C47—O47	−61.9 (4)
C12—C1—C6—C5	179.7 (3)	C5—C4—C47—O47	63.1 (4)
C2—C1—C6—C5	59.6 (3)	C3—C4—C47—C42	119.0 (3)
C51—C5—C6—C1	176.0 (3)	C5—C4—C47—C42	−116.0 (3)
C4—C5—C6—C1	−56.4 (3)	C46—N41—C42—C43	−0.1 (5)
C16—N11—C12—C13	−1.2 (5)	C46—N41—C42—C47	178.5 (3)
C16—N11—C12—C1	175.4 (3)	O47—C47—C42—N41	−177.8 (3)
O11—C1—C12—N11	6.8 (4)	C4—C47—C42—N41	1.3 (4)
C6—C1—C12—N11	126.3 (3)	O47—C47—C42—C43	0.8 (4)
C2—C1—C12—N11	−113.6 (3)	C4—C47—C42—C43	179.9 (3)
O11—C1—C12—C13	−176.6 (3)	N41—C42—C43—C44	0.7 (5)
C6—C1—C12—C13	−57.1 (4)	C47—C42—C43—C44	−177.9 (3)
C2—C1—C12—C13	63.1 (4)	C42—C43—C44—C45	−1.1 (6)
N11—C12—C13—C14	2.7 (6)	C43—C44—C45—C46	1.0 (7)
C1—C12—C13—C14	−173.7 (4)	C42—N41—C46—C45	0.0 (7)
C12—C13—C14—C15	−1.4 (7)	C44—C45—C46—N41	−0.5 (7)
C13—C14—C15—C16	−1.3 (7)	C6—C5—C51—C52	−165.0 (3)
C12—N11—C16—C15	−1.7 (7)	C4—C5—C51—C52	66.9 (4)
C14—C15—C16—N11	2.9 (8)	C6—C5—C51—C56	14.5 (4)
C3—C2—C27—O27	−39.9 (4)	C4—C5—C51—C56	−113.6 (3)
C1—C2—C27—O27	81.8 (4)	C56—C51—C52—C53	1.0 (6)
C3—C2—C27—C22	142.5 (3)	C5—C51—C52—C53	−179.5 (4)
C1—C2—C27—C22	−95.8 (3)	C51—C52—C53—C54	−0.9 (7)
C26—N21—C22—C23	0.3 (7)	C52—C53—C54—C55	0.4 (7)
C26—N21—C22—C27	−179.9 (4)	C52—C53—C54—Cl54	−179.9 (4)
O27—C27—C22—N21	152.5 (4)	C53—C54—C55—C56	0.0 (7)
C2—C27—C22—N21	−29.9 (5)	Cl54—C54—C55—C56	−179.7 (3)
O27—C27—C22—C23	−27.8 (5)	C52—C51—C56—C55	−0.5 (6)
C2—C27—C22—C23	149.9 (4)	C5—C51—C56—C55	180.0 (4)
N21—C22—C23—C24	0.8 (7)	C54—C55—C56—C51	0.0 (6)
C27—C22—C23—C24	−178.9 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H11···N11	0.88	1.92	2.587 (4)	130
C14—H14···O71	0.95	2.62	3.517 (12)	157

C14—H14···O81	0.95	2.72	3.499 (12)	140
C43—H43···N41^{i}	0.95	2.61	3.433 (4)	145
C45—H45···O71^{ii}	0.95	2.57	3.251 (11)	129
C16—H16···*Cg*1^{iii}	0.95	3.00	3.869 (5)	153

Symmetry codes: (i) *x*+1/4, −*y*+3/4, *z*−1/4; (ii) *x*−3/4, −*y*+3/4, *z*−1/4; (iii) *x*+1/2, *y*, *z*+1/2.

(III) (1*RS*,2*SR*,3*SR*,4*RS*,5*RS*)- 3,5-Bis(4-bromophenyl)-2,4-bis(pyridine-2-carbonyl)-1-(pyridin-2-yl)cyclohexanol

Crystal data

$C_{35}H_{27}Br_2N_3O_3$
M_r = 697.41
Triclinic, $P\overline{1}$
a = 9.5741 (4) Å
b = 10.7061 (3) Å
c = 15.9952 (6) Å
α = 92.863 (3)°
β = 97.443 (3)°
γ = 103.618 (3)°
V = 1574.49 (10) Å^3
Z = 2
$F(000)$ = 704
D_x = 1.471 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 5922 reflections
θ = 4.8–70.0°
μ = 3.58 mm^{-1}
T = 200 K
Block, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.109, T_{max} = 0.584
11667 measured reflections
5922 independent reflections
4948 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 70.0°, θ_{min} = 4.8°
h = −10→11
k = −13→13
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.141
S = 1.05
5922 reflections
388 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
$w = 1/[\sigma^2(F_o^2) + (0.0808P)^2 + 0.8269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1094 (3)	0.2890 (3)	0.37522 (18)	0.0469 (6)
O11	−0.0179 (2)	0.1914 (2)	0.34356 (14)	0.0597 (5)

H11	−0.0388	0.1511	0.3839	0.075*
C2	0.2459 (3)	0.2500 (3)	0.34883 (17)	0.0431 (6)
H2	0.3345	0.3181	0.3736	0.052*
C3	0.2393 (3)	0.2382 (2)	0.25268 (17)	0.0423 (5)
H3	0.1509	0.1686	0.2300	0.051*
C4	0.2191 (3)	0.3631 (3)	0.21229 (17)	0.0428 (6)
H4	0.2020	0.3448	0.1494	0.051*
C5	0.0830 (3)	0.3990 (3)	0.23934 (18)	0.0474 (6)
H5	−0.0014	0.3245	0.2185	0.057*
C6	0.0928 (3)	0.4136 (3)	0.33570 (19)	0.0505 (6)
H6A	0.0040	0.4358	0.3508	0.061*
H6B	0.1771	0.4851	0.3592	0.061*
N11	0.0326 (3)	0.2241 (3)	0.50752 (18)	0.0679 (8)
C12	0.1245 (3)	0.3091 (3)	0.47110 (19)	0.0496 (6)
C13	0.2307 (4)	0.4069 (4)	0.5184 (2)	0.0618 (8)
H13	0.2964	0.4669	0.4914	0.074*
C14	0.2392 (4)	0.4156 (4)	0.6059 (2)	0.0722 (9)
H14	0.3110	0.4819	0.6396	0.087*
C15	0.1442 (4)	0.3286 (5)	0.6429 (2)	0.0778 (11)
H15	0.1477	0.3333	0.7026	0.093*
C16	0.0443 (5)	0.2349 (5)	0.5928 (2)	0.0857 (13)
H16	−0.0211	0.1735	0.6190	0.103*
C27	0.2561 (3)	0.1226 (3)	0.38517 (19)	0.0489 (6)
O27	0.1950 (3)	0.0192 (2)	0.34738 (15)	0.0666 (6)
N21	0.4626 (4)	0.2257 (3)	0.4895 (2)	0.0679 (8)
C22	0.3450 (4)	0.1270 (3)	0.4699 (2)	0.0541 (7)
C23	0.3069 (5)	0.0303 (4)	0.5227 (3)	0.0768 (11)
H23	0.2229	−0.0384	0.5068	0.092*
C24	0.3957 (7)	0.0364 (6)	0.6000 (3)	0.1017 (17)
H24	0.3722	−0.0276	0.6384	0.122*
C25	0.5172 (7)	0.1359 (5)	0.6197 (3)	0.0977 (17)
H25	0.5802	0.1416	0.6715	0.117*
C26	0.5455 (6)	0.2268 (4)	0.5633 (3)	0.0884 (14)
H26	0.6300	0.2954	0.5776	0.106*
C31	0.3678 (3)	0.1972 (2)	0.22351 (17)	0.0439 (6)
C32	0.5090 (3)	0.2443 (3)	0.2641 (2)	0.0550 (7)
H32	0.5266	0.2992	0.3148	0.066*
C33	0.6256 (3)	0.2124 (3)	0.2318 (2)	0.0597 (8)
H33	0.7222	0.2468	0.2593	0.072*
C34	0.5991 (4)	0.1310 (3)	0.1601 (2)	0.0575 (7)
Br34	0.75694 (5)	0.09296 (5)	0.11223 (4)	0.0979 (2)
C35	0.4591 (4)	0.0767 (4)	0.1205 (2)	0.0655 (9)
H35	0.4417	0.0171	0.0721	0.079*
C36	0.3452 (4)	0.1110 (3)	0.1528 (2)	0.0561 (7)
H36	0.2487	0.0745	0.1257	0.067*
C47	0.3521 (3)	0.4760 (3)	0.23542 (18)	0.0441 (6)
O47	0.4088 (2)	0.5068 (2)	0.30802 (13)	0.0568 (5)
N41	0.3478 (4)	0.5267 (3)	0.0881 (2)	0.0725 (8)

C42	0.4133 (3)	0.5553 (3)	0.1677 (2)	0.0512 (6)
C43	0.5344 (4)	0.6559 (4)	0.1917 (3)	0.0752 (10)
H43	0.5767	0.6742	0.2495	0.090*
C44	0.5924 (6)	0.7290 (5)	0.1310 (4)	0.1031 (17)
H44	0.6775	0.7971	0.1456	0.124*
C45	0.5261 (6)	0.7024 (6)	0.0496 (4)	0.113 (2)
H45	0.5621	0.7530	0.0064	0.135*
C46	0.4060 (6)	0.6008 (6)	0.0310 (3)	0.1027 (17)
H46	0.3615	0.5822	−0.0263	0.123*
C51	0.0525 (3)	0.5151 (3)	0.19609 (18)	0.0466 (6)
C52	−0.0392 (4)	0.4954 (3)	0.1203 (2)	0.0611 (8)
H52	−0.0860	0.4096	0.0980	0.073*
C53	−0.0653 (4)	0.5979 (3)	0.0755 (2)	0.0643 (8)
H53	−0.1278	0.5828	0.0229	0.077*
C54	0.0009 (3)	0.7206 (3)	0.1089 (2)	0.0544 (7)
Br54	−0.03457 (5)	0.86020 (4)	0.04693 (3)	0.07606 (16)
C55	0.0917 (4)	0.7454 (3)	0.1847 (2)	0.0577 (7)
H55	0.1369	0.8315	0.2068	0.069*
C56	0.1159 (4)	0.6418 (3)	0.2283 (2)	0.0553 (7)
H56	0.1772	0.6578	0.2813	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0392 (13)	0.0497 (15)	0.0488 (15)	0.0037 (11)	0.0059 (11)	0.0116 (11)
O11	0.0459 (11)	0.0660 (13)	0.0585 (12)	−0.0037 (10)	0.0042 (9)	0.0149 (10)
C2	0.0421 (13)	0.0399 (13)	0.0450 (14)	0.0050 (10)	0.0043 (10)	0.0108 (10)
C3	0.0393 (13)	0.0383 (12)	0.0457 (14)	0.0030 (10)	0.0023 (10)	0.0102 (10)
C4	0.0433 (13)	0.0421 (13)	0.0408 (13)	0.0061 (11)	0.0027 (10)	0.0114 (10)
C5	0.0409 (14)	0.0458 (14)	0.0525 (15)	0.0062 (11)	0.0005 (11)	0.0126 (11)
C6	0.0473 (15)	0.0552 (16)	0.0536 (16)	0.0161 (12)	0.0129 (12)	0.0159 (13)
N11	0.0625 (17)	0.0764 (19)	0.0571 (16)	−0.0042 (14)	0.0157 (13)	0.0159 (13)
C12	0.0454 (15)	0.0543 (16)	0.0517 (16)	0.0126 (12)	0.0119 (12)	0.0139 (12)
C13	0.0581 (18)	0.0651 (19)	0.0566 (18)	0.0054 (15)	0.0049 (14)	0.0089 (14)
C14	0.063 (2)	0.085 (3)	0.063 (2)	0.0140 (18)	0.0011 (16)	−0.0032 (18)
C15	0.068 (2)	0.114 (3)	0.0501 (19)	0.018 (2)	0.0123 (16)	0.0069 (19)
C16	0.079 (3)	0.113 (3)	0.057 (2)	−0.004 (2)	0.0232 (19)	0.022 (2)
C27	0.0526 (16)	0.0413 (14)	0.0510 (15)	0.0072 (12)	0.0066 (12)	0.0116 (11)
O27	0.0818 (16)	0.0433 (11)	0.0637 (14)	−0.0001 (11)	−0.0038 (12)	0.0116 (10)
N21	0.077 (2)	0.0548 (15)	0.0665 (18)	0.0189 (14)	−0.0131 (15)	0.0022 (13)
C22	0.0646 (19)	0.0483 (15)	0.0513 (16)	0.0208 (14)	0.0012 (13)	0.0073 (12)
C23	0.096 (3)	0.068 (2)	0.067 (2)	0.020 (2)	0.0048 (19)	0.0260 (18)
C24	0.148 (5)	0.104 (4)	0.064 (2)	0.053 (4)	0.004 (3)	0.037 (2)
C25	0.133 (4)	0.095 (3)	0.066 (2)	0.056 (3)	−0.031 (3)	−0.006 (2)
C26	0.102 (3)	0.069 (2)	0.083 (3)	0.028 (2)	−0.033 (2)	−0.008 (2)
C31	0.0465 (14)	0.0387 (13)	0.0453 (14)	0.0083 (11)	0.0038 (11)	0.0105 (10)
C32	0.0480 (16)	0.0565 (17)	0.0559 (17)	0.0096 (13)	0.0012 (12)	−0.0086 (13)
C33	0.0432 (15)	0.0594 (18)	0.073 (2)	0.0076 (13)	0.0068 (14)	−0.0022 (15)

C34	0.0583 (18)	0.0496 (16)	0.0683 (19)	0.0139 (13)	0.0215 (15)	0.0060 (14)
Br34	0.0719 (3)	0.0935 (3)	0.1280 (4)	0.0138 (2)	0.0402 (3)	−0.0285 (3)
C35	0.070 (2)	0.0626 (19)	0.0597 (19)	0.0105 (16)	0.0115 (16)	−0.0109 (15)
C36	0.0533 (17)	0.0572 (17)	0.0509 (16)	0.0043 (13)	0.0014 (13)	−0.0014 (13)
C47	0.0407 (13)	0.0398 (13)	0.0507 (16)	0.0072 (10)	0.0049 (11)	0.0102 (11)
O47	0.0561 (12)	0.0521 (11)	0.0518 (12)	−0.0030 (9)	−0.0009 (9)	0.0071 (9)
N41	0.0667 (18)	0.081 (2)	0.0631 (18)	0.0009 (15)	0.0096 (14)	0.0287 (15)
C42	0.0459 (15)	0.0511 (15)	0.0564 (17)	0.0085 (12)	0.0079 (12)	0.0181 (13)
C43	0.064 (2)	0.068 (2)	0.084 (3)	−0.0065 (17)	0.0105 (18)	0.0255 (19)
C44	0.085 (3)	0.093 (3)	0.115 (4)	−0.020 (3)	0.021 (3)	0.045 (3)
C45	0.098 (4)	0.121 (4)	0.113 (4)	−0.007 (3)	0.033 (3)	0.068 (3)
C46	0.099 (3)	0.128 (4)	0.071 (3)	−0.002 (3)	0.013 (2)	0.049 (3)
C51	0.0423 (14)	0.0468 (14)	0.0518 (15)	0.0126 (11)	0.0049 (11)	0.0117 (11)
C52	0.065 (2)	0.0478 (16)	0.0630 (19)	0.0093 (14)	−0.0101 (15)	0.0076 (14)
C53	0.071 (2)	0.0616 (19)	0.0572 (18)	0.0187 (16)	−0.0089 (15)	0.0128 (15)
C54	0.0542 (17)	0.0510 (16)	0.0645 (18)	0.0192 (13)	0.0126 (14)	0.0249 (14)
Br54	0.0745 (3)	0.0655 (2)	0.0962 (3)	0.02530 (19)	0.0135 (2)	0.0409 (2)
C55	0.0626 (19)	0.0443 (15)	0.0655 (19)	0.0107 (13)	0.0079 (15)	0.0117 (13)
C56	0.0569 (17)	0.0517 (16)	0.0535 (17)	0.0111 (13)	−0.0026 (13)	0.0080 (13)

Geometric parameters (Å, º)

C1—O11	1.422 (3)	C25—C26	1.364 (7)
C1—C12	1.521 (4)	C25—H25	0.9500
C1—C6	1.537 (4)	C26—H26	0.9500
C1—C2	1.563 (4)	C31—C36	1.384 (4)
O11—H11	0.8144	C31—C32	1.387 (4)
C2—C27	1.528 (4)	C32—C33	1.394 (4)
C2—C3	1.529 (4)	C32—H32	0.9500
C2—H2	1.0000	C33—C34	1.363 (5)
C3—C31	1.519 (4)	C33—H33	0.9500
C3—C4	1.555 (3)	C34—C35	1.383 (5)
C3—H3	1.0000	C34—Br34	1.896 (3)
C4—C47	1.526 (4)	C35—C36	1.381 (5)
C4—C5	1.548 (4)	C35—H35	0.9500
C4—H4	1.0000	C36—H36	0.9500
C5—C51	1.521 (4)	C47—O47	1.211 (4)
C5—C6	1.530 (4)	C47—C42	1.503 (4)
C5—H5	1.0000	N41—C46	1.330 (5)
C6—H6A	0.9900	N41—C42	1.331 (4)
C6—H6B	0.9900	C42—C43	1.382 (5)
N11—C12	1.322 (4)	C43—C44	1.369 (6)
N11—C16	1.351 (5)	C43—H43	0.9500
C12—C13	1.387 (5)	C44—C45	1.358 (8)
C13—C14	1.388 (5)	C44—H44	0.9500
C13—H13	0.9500	C45—C46	1.373 (7)
C14—C15	1.357 (6)	C45—H45	0.9500
C14—H14	0.9500	C46—H46	0.9500

C15—C16	1.353 (6)	C51—C52	1.377 (4)
C15—H15	0.9500	C51—C56	1.391 (4)
C16—H16	0.9500	C52—C53	1.394 (4)
C27—O27	1.213 (4)	C52—H52	0.9500
C27—C22	1.496 (4)	C53—C54	1.364 (5)
N21—C26	1.333 (5)	C53—H53	0.9500
N21—C22	1.341 (5)	C54—C55	1.372 (5)
C22—C23	1.381 (5)	C54—Br54	1.905 (3)
C23—C24	1.397 (6)	C55—C56	1.388 (4)
C23—H23	0.9500	C55—H55	0.9500
C24—C25	1.369 (8)	C56—H56	0.9500
C24—H24	0.9500		

O11—C1—C12	110.1 (2)	C25—C24—H24	120.5
O11—C1—C6	107.2 (2)	C23—C24—H24	120.5
C12—C1—C6	109.9 (2)	C26—C25—C24	118.5 (4)
O11—C1—C2	109.9 (2)	C26—C25—H25	120.7
C12—C1—C2	110.0 (2)	C24—C25—H25	120.7
C6—C1—C2	109.7 (2)	N21—C26—C25	124.4 (4)
C1—O11—H11	105.3	N21—C26—H26	117.8
C27—C2—C3	110.6 (2)	C25—C26—H26	117.8
C27—C2—C1	108.8 (2)	C36—C31—C32	117.9 (3)
C3—C2—C1	111.4 (2)	C36—C31—C3	119.4 (3)
C27—C2—H2	108.6	C32—C31—C3	122.7 (3)
C3—C2—H2	108.6	C31—C32—C33	121.1 (3)
C1—C2—H2	108.6	C31—C32—H32	119.4
C31—C3—C2	113.6 (2)	C33—C32—H32	119.4
C31—C3—C4	110.4 (2)	C34—C33—C32	119.0 (3)
C2—C3—C4	112.4 (2)	C34—C33—H33	120.5
C31—C3—H3	106.7	C32—C33—H33	120.5
C2—C3—H3	106.7	C33—C34—C35	121.4 (3)
C4—C3—H3	106.7	C33—C34—Br34	119.6 (3)
C47—C4—C5	110.4 (2)	C35—C34—Br34	119.0 (3)
C47—C4—C3	112.7 (2)	C36—C35—C34	118.6 (3)
C5—C4—C3	109.4 (2)	C36—C35—H35	120.7
C47—C4—H4	108.1	C34—C35—H35	120.7
C5—C4—H4	108.1	C35—C36—C31	121.8 (3)
C3—C4—H4	108.1	C35—C36—H36	119.1
C51—C5—C6	113.8 (3)	C31—C36—H36	119.1
C51—C5—C4	111.2 (2)	O47—C47—C42	118.1 (2)
C6—C5—C4	111.4 (2)	O47—C47—C4	121.7 (2)
C51—C5—H5	106.6	C42—C47—C4	120.1 (2)
C6—C5—H5	106.6	C46—N41—C42	116.2 (4)
C4—C5—H5	106.6	N41—C42—C43	123.2 (3)
C5—C6—C1	111.5 (3)	N41—C42—C47	118.9 (3)
C5—C6—H6A	109.3	C43—C42—C47	117.9 (3)
C1—C6—H6A	109.3	C44—C43—C42	118.9 (4)
C5—C6—H6B	109.3	C44—C43—H43	120.6

C1—C6—H6B	109.3	C42—C43—H43	120.6
H6A—C6—H6B	108.0	C45—C44—C43	118.7 (4)
C12—N11—C16	118.2 (3)	C45—C44—H44	120.6
N11—C12—C13	121.5 (3)	C43—C44—H44	120.6
N11—C12—C1	115.7 (3)	C44—C45—C46	118.7 (4)
C13—C12—C1	122.7 (3)	C44—C45—H45	120.7
C12—C13—C14	118.8 (3)	C46—C45—H45	120.7
C12—C13—H13	120.6	N41—C46—C45	124.2 (5)
C14—C13—H13	120.6	N41—C46—H46	117.9
C15—C14—C13	119.4 (4)	C45—C46—H46	117.9
C15—C14—H14	120.3	C52—C51—C56	117.8 (3)
C13—C14—H14	120.3	C52—C51—C5	119.1 (3)
C16—C15—C14	118.5 (4)	C56—C51—C5	123.2 (3)
C16—C15—H15	120.7	C51—C52—C53	121.7 (3)
C14—C15—H15	120.7	C51—C52—H52	119.2
N11—C16—C15	123.5 (4)	C53—C52—H52	119.2
N11—C16—H16	118.2	C54—C53—C52	118.6 (3)
C15—C16—H16	118.2	C54—C53—H53	120.7
O27—C27—C22	119.5 (3)	C52—C53—H53	120.7
O27—C27—C2	122.0 (3)	C53—C54—C55	122.0 (3)
C22—C27—C2	118.4 (2)	C53—C54—Br54	118.3 (2)
C26—N21—C22	116.9 (4)	C55—C54—Br54	119.8 (2)
N21—C22—C23	123.1 (3)	C54—C55—C56	118.5 (3)
N21—C22—C27	116.8 (3)	C54—C55—H55	120.7
C23—C22—C27	120.1 (3)	C56—C55—H55	120.7
C22—C23—C24	118.1 (4)	C55—C56—C51	121.5 (3)
C22—C23—H23	121.0	C55—C56—H56	119.3
C24—C23—H23	121.0	C51—C56—H56	119.3
C25—C24—C23	119.0 (4)		

O11—C1—C2—C27	−59.6 (3)	C22—C23—C24—C25	−1.0 (8)
C12—C1—C2—C27	61.8 (3)	C23—C24—C25—C26	1.0 (8)
C6—C1—C2—C27	−177.2 (2)	C22—N21—C26—C25	−0.9 (7)
O11—C1—C2—C3	62.6 (3)	C24—C25—C26—N21	0.0 (8)
C12—C1—C2—C3	−176.0 (2)	C2—C3—C31—C36	140.5 (3)
C6—C1—C2—C3	−55.0 (3)	C4—C3—C31—C36	−92.2 (3)
C27—C2—C3—C31	−57.4 (3)	C2—C3—C31—C32	−40.6 (4)
C1—C2—C3—C31	−178.5 (2)	C4—C3—C31—C32	86.7 (3)
C27—C2—C3—C4	176.4 (2)	C36—C31—C32—C33	4.0 (5)
C1—C2—C3—C4	55.2 (3)	C3—C31—C32—C33	−174.9 (3)
C31—C3—C4—C47	−59.7 (3)	C31—C32—C33—C34	−1.6 (5)
C2—C3—C4—C47	68.3 (3)	C32—C33—C34—C35	−1.9 (5)
C31—C3—C4—C5	177.1 (2)	C32—C33—C34—Br34	177.0 (3)
C2—C3—C4—C5	−54.9 (3)	C33—C34—C35—C36	2.9 (6)
C47—C4—C5—C51	59.7 (3)	Br34—C34—C35—C36	−176.1 (3)
C3—C4—C5—C51	−175.8 (2)	C34—C35—C36—C31	−0.3 (5)
C47—C4—C5—C6	−68.5 (3)	C32—C31—C36—C35	−3.0 (5)
C3—C4—C5—C6	56.0 (3)	C3—C31—C36—C35	175.9 (3)

C51—C5—C6—C1	174.7 (2)	C5—C4—C47—O47	72.3 (3)
C4—C5—C6—C1	−58.6 (3)	C3—C4—C47—O47	−50.3 (4)
O11—C1—C6—C5	−62.5 (3)	C5—C4—C47—C42	−105.5 (3)
C12—C1—C6—C5	177.9 (2)	C3—C4—C47—C42	131.9 (3)
C2—C1—C6—C5	56.8 (3)	C46—N41—C42—C43	0.4 (6)
C16—N11—C12—C13	0.1 (6)	C46—N41—C42—C47	179.4 (4)
C16—N11—C12—C1	177.9 (4)	O47—C47—C42—N41	−176.7 (3)
O11—C1—C12—N11	8.8 (4)	C4—C47—C42—N41	1.3 (4)
C6—C1—C12—N11	126.6 (3)	O47—C47—C42—C43	2.5 (5)
C2—C1—C12—N11	−112.5 (3)	C4—C47—C42—C43	−179.6 (3)
O11—C1—C12—C13	−173.5 (3)	N41—C42—C43—C44	−1.3 (7)
C6—C1—C12—C13	−55.7 (4)	C47—C42—C43—C44	179.7 (4)
C2—C1—C12—C13	65.2 (4)	C42—C43—C44—C45	2.0 (9)
N11—C12—C13—C14	−0.4 (5)	C43—C44—C45—C46	−1.9 (10)
C1—C12—C13—C14	−178.0 (3)	C42—N41—C46—C45	−0.3 (9)
C12—C13—C14—C15	0.1 (6)	C44—C45—C46—N41	1.1 (11)
C13—C14—C15—C16	0.5 (7)	C6—C5—C51—C52	−141.5 (3)
C12—N11—C16—C15	0.6 (7)	C4—C5—C51—C52	91.7 (3)
C14—C15—C16—N11	−0.9 (8)	C6—C5—C51—C56	40.2 (4)
C3—C2—C27—O27	−34.8 (4)	C4—C5—C51—C56	−86.7 (4)
C1—C2—C27—O27	88.0 (4)	C56—C51—C52—C53	1.9 (5)
C3—C2—C27—C22	145.0 (3)	C5—C51—C52—C53	−176.6 (3)
C1—C2—C27—C22	−92.3 (3)	C51—C52—C53—C54	−0.9 (6)
C26—N21—C22—C23	0.9 (6)	C52—C53—C54—C55	0.0 (6)
C26—N21—C22—C27	−177.2 (4)	C52—C53—C54—Br54	179.7 (3)
O27—C27—C22—N21	146.8 (3)	C53—C54—C55—C56	−0.1 (5)
C2—C27—C22—N21	−32.9 (4)	Br54—C54—C55—C56	−179.8 (3)
O27—C27—C22—C23	−31.3 (5)	C54—C55—C56—C51	1.1 (5)
C2—C27—C22—C23	148.9 (3)	C52—C51—C56—C55	−2.0 (5)
N21—C22—C23—C24	0.1 (7)	C5—C51—C56—C55	176.4 (3)
C27—C22—C23—C24	178.1 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O11—H11···N11	0.81	2.06	2.595 (4)	123
C16—H16···O27^{i}	0.95	2.46	3.385 (6)	165
C53—H53···N41^{ii}	0.95	2.57	3.473 (5)	158

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