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Solid-state conformations of linear depsipeptide amides with an alternating sequence of α,α-disubstituted α-amino acid and α-hydroxy acid

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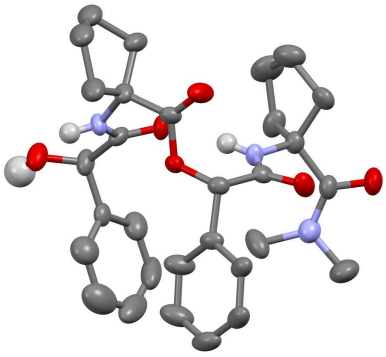
Depsipeptides and cyclodepsipeptides are analogues of the corresponding peptides in which one or more amide groups are replaced by ester functions. Reports of crystal structures of linear depsipeptides are rare. The crystal structures and conformational analyses of four depsipeptides with an alternating sequence of an α,α-disubstituted α-amino acid and an α-hydroxy acid are reported. The molecules in the linear hexadepsipeptide amide in (*S*)-Pms-Acp-(*S*)-Pms-Acp-(*S*)-Pms-Acp-NMe$_2$ acetonitrile solvate, $C_{47}H_{58}N_4O_9\cdot C_2H_3N$, (**3b**), as well as in the related linear tetradepsipeptide amide (*S*)-Pms-Aib-(*S*)-Pms-Aib-NMe$_2$, $C_{28}H_{37}N_3O_6$, (**5a**), the diastereoisomeric mixture (*S*,*R*)-Pms-Acp-(*R*,*S*)-Pms-Acp-NMe$_2$/(*R*,*S*)-Pms-Acp-(*R*,*S*)-Pms-Acp-NMe$_2$ (1:1), $C_{32}H_{41}N_3O_6$, (**5b**), and (*R*,*S*)-Mns-Acp-(*S*,*R*)-Mns-Acp-NMe$_2$, $C_{30}H_{37}N_3O_6$, (**5c**) (Pms is phenyllactic acid, Acp is 1-aminocyclopentanecarboxylic acid and Mns is mandelic acid), generally adopt a β-turn conformation in the solid state, which is stabilized by intramolecular N—H···O hydrogen bonds. Whereas β-turns of type I (or I′) are formed in the cases of (**3b**), (**5a**) and (**5b**), which contain phenyllactic acid, the torsion angles for (**5c**), which incorporates mandelic acid, indicate a β-turn in between type I and type III. Intermolecular N—H···O and O—H···O hydrogen bonds link the molecules of (**3a**) and (**5b**) into extended chains, and those of (**5a**) and (**5c**) into two-dimensional networks.

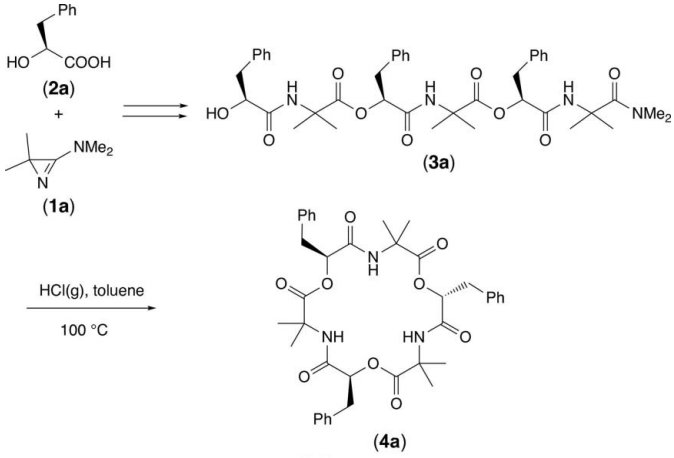
1. Introduction

Depsipeptides and cyclodepsipeptides are analogues of the corresponding peptides in which one or more amide groups are replaced by ester functions, *i.e.* amino acids are replaced by hydroxy acids. In particular, cyclic depsipeptides are still of broad chemical, biological and medicinal interest, which is reflected in a large number of recent review articles [*e.g.* structure and isolation (Wang *et al.*, 2018; Ding *et al.*, 2016; Tarsis *et al.*, 2015; Pelay-Gimeno *et al.*, 2013), synthesis (Köcher *et al.*, 2017; Qi *et al.*, 2016; Boecker *et al.*, 2016; Weiss *et al.*, 2013; Xu *et al.*, 2013) and biological activity (Wang *et al.*, 2018; Weiss *et al.*, 2017; Kitagaki *et al.*, 2015; Sivanathan & Scherkenbeck, 2014; Smelcerovic *et al.*, 2014)]. Their general structures can be assigned to different classes, containing, besides α-amino acids, a simple hydroxy acid, like in solonamide A (Kitir *et al.* 2014), or a complex hydroxy acid, as in calcaripeptide A (Silber *et al.*, 2013), being characterized by a head-to-side-chain lactonization, as in kahalalide A (Bourel-Bonnet *et al.*, 2005), or with an alternating sequence of α-amino and α-hydroxy acids. Two well-known examples of the last type are the 18-membered enniatin A (Shemyakin *et al.*, 1965; Quitt *et al.*, 1963) and the 36-membered valinomycin (Neupert-Laves & Dobler, 1975; Shemyakin *et al.*, 1963), which are of considerable interest as natural ionophores,



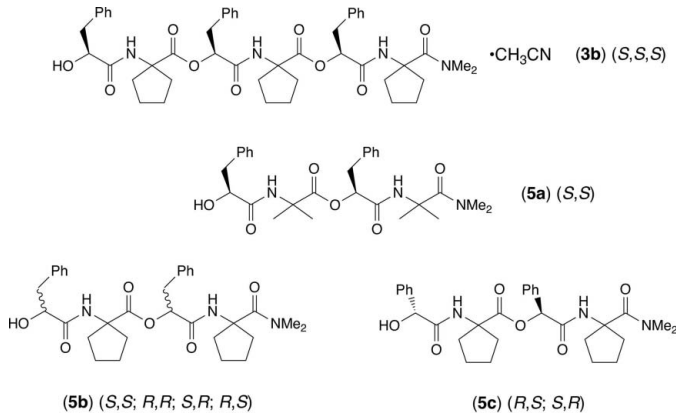
enabling the transport of monovalent cations across membranes (Ovchinnikov *et al.*, 1974; Dobler *et al.*, 1969).

Our studies on the use of 2,2-disubstituted 3-amino-2*H*-azirines in the synthesis of heterocycles and peptides (Heimgartner, 1981, 1986, 1991) have shown them to be suitable building blocks for α,α-disubstituted α-amino acids in peptide synthesis ('azirine/oxazolone method'; Arnhold *et al.*, 2014; Pradeille *et al.*, 2012; Altherr *et al.*, 2007; Stamm & Heimgartner, 2004; Wipf & Heimgartner, 1990; Obrecht & Heimgartner, 1987*a*). Furthermore, peptide and depsipeptide amides prepared by this method have been cyclized *via* 'direct amide cyclization' to give cyclic peptides (Arnhold *et al.*, 2015; Dannecker-Dörig *et al.*, 2009; Jeremic *et al.*, 2005) and cyclodepsipeptides (Koch *et al.*, 2000, 2001; Obrecht & Heimgartner, 1984, 1987*b*, 1990), respectively, which contain α,α-disubstituted α-amino acids. The studies toward the synthesis of such cyclic depsipeptides with enniatin-like structures *via* 'direct amide cyclization' of the linear depsipeptides of type (**3a**), prepared from azirine (**1a**) and α-hydroxy acid (**2a**), gave the corresponding cyclic depsipeptide (Scheme 1). The structure of the latter has been established by X-ray crystallography as the 18-membered (*R*,*S*,*S*)-isomer (**4a**) (Köttgen *et al.*, 2006). Similar results were obtained with analogous hexadepsipeptides (Köttgen *et al.*, 2006), as well as with a homologue of (**3a**) to give the corresponding 24-membered cyclodepsipeptide (Köttgen *et al.*, 2009). Unexpectedly, both (**4a**) and the 24-membered homologue were formed stereoselectively as epimers of the expected products. This fact was explained by a likely reaction mechanism (Köttgen *et al.*, 2006).

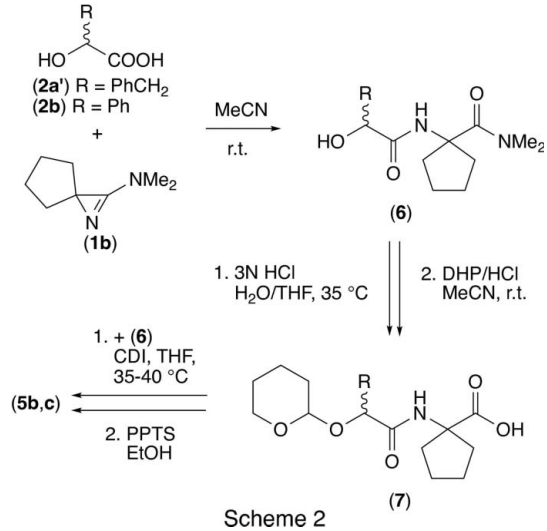


Scheme 1

These surprising results prompted us to study the detailed structure and conformation of the linear hexadepsipeptide amide (*S*)-Pms-Acp-(*S*)-Pms-Acp-(*S*)-Pms-Acp-NMe_2, (**3b**), by X-ray crystallography. In addition, the structures of three related linear tetradepsipeptide amides, namely, (*S*)-Pms-Aib-(*S*)-Pms-Aib-NMe_2, (**5a**), the diastereoisomeric mixture (*S*,*R*)-Pms-Acp-(*R*,*S*)-Pms-Acp-NMe_2/(*R*,*S*)-Pms-Acp-(*R*,*S*)-Pms-Acp-NMe_2 (1:1), (**5b**), and (*R*,*S*)-Mns-Acp-(*S*,*R*)-Mns-Acp-NMe_2, (**5c**), were examined (Pms is phenyllactic acid, Acp is 1-aminocyclopentanecarboxylic acid and Mns is mandelic acid). These compounds all contain an alternating sequence of an α,α-disubstituted α-amino acid and an α-hydroxy acid.



The syntheses of (**5b**) and (**5c**) were carried out according to Scheme 2 in analogy to previously reported methods (Obrecht & Heimgartner, 1984, 1990).



Scheme 2

2. Experimental

2.1. Synthesis and crystallization

The enantiomerically pure hexadepsipeptide amide (**3b**), with an alternating sequence of 1-aminocyclopentanecarboxylic acid (Acp) and (*S*)-phenyllactic acid [Pms, (**2a**)], was prepared *via* the 'azirine/oxazolone method' starting with 2-dimethylamino-1-azaspiro[2.4]hept-1-ene, *i.e.* (**1b**), and (**2a**) (Magirius, 1995; Köttgen *et al.*, 2006). In an analogous manner, the tetradepsipeptide amide (**5a**) was synthesized using 3-dimethylamino-2,2-dimethyl-2*H*-azirine, *i.e.* (**1a**), as the synthon for 2-aminoisobutyric acid (Aib) (Obrecht & Heimgartner, 1990; Köttgen *et al.*, 2009). Similarly, reaction of racemic mandelic acid [Mns, (**2b**), 3.16 g] with an equimolar amount of (**1b**) (2.87 g) in 120 ml of acetonitrile after 1.5 h at room temperature gave the diamide (**6**) (R = Ph, 5.55 g) in 92% yield (Magirius, 1995). Selective hydrolysis of the terminal amide function by treatment of 4.90 g of (**6**) (R = Ph) in 170 ml of 3 *N* HCl [tetrahydrofuran (THF)/H_2O, 1:1 *v*/*v*] at 308 K for 15 h led to the corresponding acid (yield 3.59 g, 82%). Subsequent protection of the hydroxy group by reaction of

Table 1
Experimental details.

	(**3b**)	(**5a**)	(**5b**)	(**5c**)
Crystal data				
Chemical formula	$C_{47}H_{58}N_4O_9{\cdot}C_2H_3N$	$C_{28}H_{37}N_3O_6$	$C_{32}H_{41}N_3O_6$	$C_{30}H_{37}N_3O_6$
M_r	864.02	511.60	563.68	535.62
Crystal system, space group	Monoclinic, $P2_1$	Monoclinic, $P2_1$	Monoclinic, $P2_1/n$	Orthorhombic, $Pna2_1$
Temperature (K)	173	173	173	173
a, b, c (Å)	10.645 (3), 19.161 (9), 11.503 (3)	10.388 (4), 12.756 (6), 21.624 (5)	9.937 (5), 18.319 (7), 16.436 (3)	18.922 (5), 18.300 (3), 8.072 (6)
α, β, γ (°)	90, 93.75 (2), 90	90, 93.55 (3), 90	90, 90.19 (3), 90	90, 90, 90
V (Å^3)	2341.5 (15)	2859.7 (18)	2992 (2)	2795 (2)
Z	2	4	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.09	0.08	0.09	0.09
Crystal size (mm)	0.48 × 0.33 × 0.23	0.42 × 0.35 × 0.35	0.40 × 0.40 × 0.25	0.30 × 0.23 × 0.20
Data collection				
Diffractometer	Rigaku AFC-5R	Rigaku AFC-5R	Rigaku AFC-5R	Rigaku AFC-5R
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5821, 5527, 3617	7056, 6868, 4297	7311, 6855, 4077	4363, 3889, 2106
R_{int}	0.066	0.033	0.027	0.043
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.650	0.650	0.650	0.649
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.075, 0.218, 1.04	0.052, 0.149, 1.04	0.070, 0.205, 1.04	0.055, 0.161, 1.01
No. of reflections	5527	6868	6855	3889
No. of parameters	632	703	509	394
No. of restraints	229	1	540	98
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement	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})	0.36, −0.28	0.33, −0.23	0.66, −0.66	0.32, −0.25

Computer programs: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991), *TEXSAN* (Molecular Structure Corporation, 1989), *SHELXS86* (Sheldrick, 1990), *ORTEPII* (Johnson, 1976), *Mercury* (Macrae *et al.*, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

526 mg of the acid with an excess of 3,4-dihydro-2*H*-pyran and HCl in acetonitrile (10 ml) and 3 drops of HCl-saturated acetonitrile for 60 min at room temperature yielded crude (**7**) (R = Ph) as a colourless oil. Coupling of the latter with (**6**) (R = Ph, 580 mg) by using 1,1′-carbonyldiimidazole (324 mg) and 0.5 ml of a sodium imidazolide suspension in THF gave, after 20 min at 308 K and chromatographic separation (SiO_2, Et_2O/AcOEt, 10:1 *v*/*v*), 810 mg (65%) of the tetrahydropyranyl-protected tetradepsipeptide amide. Finally, deprotection of 795 mg of the latter by treatment with pyridinium *p*-toluenesulfonate in ethanol and chromatographic purification (SiO_2, Et_2O/AcOEt, 30:1 *v*/*v*) gave (**5c**) (yield 589 mg) as a racemic mixture of diastereoisomers in 86% yield, although the studied crystal only contained a single diastereoisomer as its racemate. The analogous reaction starting with racemic phenyllactic acid (**2a′**) led to (**5b**), also as a racemic mixture of diastereoisomers (Magirius, 1995), which persisted in the chosen crystal. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of the solvents, *i.e.* acetonitrile in the cases of (**3b**) and (**5c**), diethyl ether in the case of (**5a**) and dichloromethane/diethyl ether/hexane in the case of (**5b**).

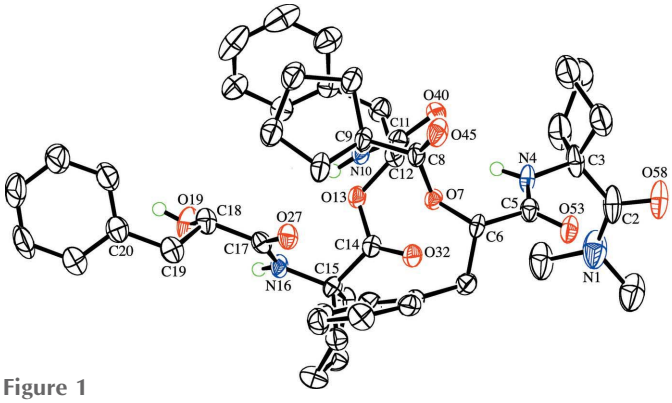


Figure 1
View of the depsipeptide molecule of (**3b**), showing the atom-labelling scheme for the peptide backbone. Displacement ellipsoids are drawn at the 50% probability level. Most H atoms and the minor-disorder conformations of the five-membered rings have been omitted for clarity.

2.2. Analytical and spectroscopic data

The data for (**3b**) and (**5a**) have been published previously (Köttgen *et al.*, 2006, 2009, respectively). Compound (**5b**) was obtained as a crude mixture of diastereoisomers from racemic (**2a′**) and (**1b**) in a preliminary experiment (Magirius, 1995). Compound (**5c**) (mixture of diastereoisomers): m.p. 371–373 K; IR ($CHCl_3$): 3400 (*m*), 3300 (*m*), 2995 (*m*), 2960 (*m*), 2870 (*m*), 1740 (*s*) (C═O ester), 1665 (*s*), 1630 (*s*) (C═O amide), 1525 (*m*), 1520 (*m*), 1495 (*m*), 1450 (*m*), 1395 (*m*), 1165 (*m*) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 7.96, 7.76, 7.75, 7.73 (4*s*, 2 NH), 7.4–7.3, 7.3–7.15 (2*m*, 9 arom. H), 7.05 (*d*, J = 7, 1

arom. H), 5.86, 5.73 (2*s*, PhCHO), 5.12, 5.10 (2*s*, PhCHOH), 2.71, 2.62, 2.59 (3 broad *s*, Me_2N), 2.5–2.35, 2.3–2.25, 2.2–1.85, 1.8–1.65, 1.6–1.35 (5*m*, 8 CH_2); ^{13}C NMR ($CDCl_3$): δ 172.7, 172.0, 171.7, 171.6, 171.5 (5*s*, 3 C═O amide); 166.5 (*s*, C═O ester); 138.7, 138.5, 134.0, 133.6 (4*s*, 2 arom. C); 128.5, 128.4, 128.3, 128.2, 128.1, 127.7, 127.2, 126.9, 126.7 (9*d*, 10 arom. CH); 76.7, 76.3 (2*d*, Ph*C*HO); 74.1, 74.0 (2*d*, Ph*C*HOH); 65.3, 65.0, 64.5, 64.1 (4*s*, 2 cyclopentyl-C1); 39.0, 38.1, 37.3, 37.0, 36.9, 36.5, 35.9, 35.7, 24.4, 24.3, 24.2, 24.1, 24.0 (13*t*, 8 CH_2); 37.6, 37.3 (2*q*, Me_2N); ESI–MS: 574 (18, $[M + K]^+$), 568 (100, $[M + Na]^+$), 536 (4, $M + 1]^+$); analysis calculated (%) for $C_{30}H_{37}N_3O_6$: C 67.27, H 6.96, N 7.84; found: C 66.99, H 6.95, N 7.67. The single crystal selected after crystallization from acetonitrile contained only the single racemic diastereoisomer (**5c**).

2.3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. Equivalent reflections, other than Friedel pairs in the noncentrosymmetric structures of (**3b**), (**5a**) and (**5c**), were merged. A correction for secondary extinction was applied only in the case of (**3b**).

In (**5a**), there are two symmetry-independent molecules in the asymmetric unit. The atomic coordinates of the two molecules were tested carefully for a relationship from a higher symmetry space group using the program *PLATON* (Spek, 2009), but none could be found. In (**3b**), the asymmetric unit contains one molecule of the peptide and one molecule of acetonitrile. Two five-membered rings and the terminal –NMe_2 group are disordered. Two sets of positions were defined for two of the –CH_2– groups in two of the five-membered rings and for the –NMe_2 methyl groups. The site-occupation factors of the major conformations of these groups refined to 0.597 (18), 0.59 (2) and 0.880 (18), respectively. For this and the other disordered structures described below, similarity restraints were applied to the chemically equivalent bond lengths involving all disordered C atoms, while neighbouring atoms within and between each conformation of the disordered components were restrained to have similar atomic displacement parameters and all non-H atoms were refined anisotropically. In (**5b**), both benzyl substituents exhibit disorder. Two orientations were defined for just the phenyl ring of the central benzyl group and for the entire terminal benzyl group, including the stereogenic C atom, C12, and the site-occupation factors of the major sites of these groups refined to 0.505 (7) and 0.520 (3), respectively. As the disorder inverts the chirality at C12, the crystal contains a mixture of diastereoisomers in the ratio 0.520 (3):0.480 (3) for (*S*,*R*)-Pms-Acp-(*R*,*S*)-Pms-Acp-NMe_2 and (*R*,*S*)-Pms-Acp-(*R*,*S*)-Pms-Acp-NMe_2, respectively. In (**5c**), one of the five-membered rings exhibits significant conformational disorder and two positions were refined for three of the ring atoms. The site-occupation factor of the major conformation of the ring refined to 0.658 (12). Models for disorder are rarely perfect and there remained a few reflections with significant discrepancies between their observed and calculated intensities. These reflections were omitted from the final refinements, *i.e.* 1 for (**3b**) and (**5b**), 7 for (**5a**) and 4 for (**5c**).

In general, except for (**3b**) and the two positions of the disordered hydroxy H atom of (**5b**), the amide and hydroxy H atoms were placed in the positions indicated by a difference electron-density map and their positions were allowed to refine together with individual isotropic displacement parameters. For (**5c**), the N—H and O—H distances were restrained to 0.86 (2) and 0.84 (2) Å, respectively. The methyl H atoms in all structures and the hydroxy H atoms in (**3b**) and (**5b**) were constrained to an ideal geometry (C—H = 0.98 Å and O—H = 0.84 Å), with U_{iso}(H) = 1.5U_{eq}(C,O), while each group was allowed to rotate freely about its parent C—C or C—O bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95 (aromatic), 0.99 (methylene) and 1.00 Å (methine), and with U_{iso}(H) = 1.2U_{eq}(C).

Table 2
Hydrogen-bond geometry (Å, °) for (**3b**), (**5a**), (**5b**) and (**5c**).

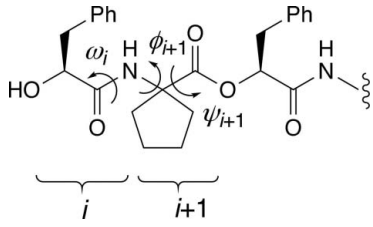
	D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(**3b**)	O19—H419···O45^{i}	0.84	2.11	2.814 (6)	142
	N4—H4···O40	0.88	2.19	3.019 (7)	156
	N10—H10···O27	0.88	2.07	3.882 (7)	154
	N16—H16···O53^{i}	0.88	2.23	3.052 (6)	156
(**5a**)†	O13—H13···O35^{ii}	0.90 (6)	1.79 (6)	2.670 (5)	169 (5)
	N4—H4···O21	0.90 (6)	1.99 (6)	2.881 (5)	171 (5)
	N10—H10···O53^{iii}	0.81 (5)	2.36 (5)	3.124 (5)	157 (4)
	O53—H53···O75^{iv}	0.86 (6)	1.83 (6)	2.684 (5)	171 (6)
	N44—H44···O61	0.85 (5)	2.01 (5)	2.824 (5)	159 (5)
	N50—H50···O13^{v}	0.84 (5)	2.40 (5)	3.185 (5)	156 (4)
(**5b**)‡	O13—H131···O34^{vi}	0.84	2.00	2.753 (3)	149
	O13—H132···O34^{vi}	0.84	1.92	2.753 (3)	172
	N4—H4···O21	0.87 (3)	2.06 (3)	2.920 (3)	170 (3)
	N10—H10···O39^{vi}	0.84 (3)	2.34 (3)	3.161 (3)	164 (3)
(**5c**)	O13—H13···O32^{vii}	0.86 (2)	1.87 (3)	2.713 (6)	166 (8)
	N4—H4···O20	0.84 (2)	2.09 (3)	2.907 (5)	164 (6)
	N10—H10···O37^{viii}	0.85 (2)	2.17 (3)	2.970 (6)	155 (5)

Symmetry codes: (i) $x, y, z-1$; (ii) $-x+1, y-\frac{1}{2}, -z$; (iii) $-x+1, y-\frac{1}{2}, -z+1$; (iv) $-x, y+\frac{1}{2}, -z+1$; (v) $-x+1, y+\frac{1}{2}, -z+1$; (vi) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (vii) $-x+\frac{1}{2}, y-\frac{1}{2}, z+\frac{1}{2}$; (viii) $-x+\frac{1}{2}, y-\frac{1}{2}, z-\frac{1}{2}$. † Two symmetry-independent molecules; the atom numbers of molecule *B* correspond with those of molecule *A* + 40. ‡ The hydroxy group is disordered.

3. Results and discussion

The asymmetric unit of (**3b**) contains one molecule of the hexadepsipeptide (Fig. 1) and one molecule of acetonitrile. Two of the five-membered rings and the terminal –NMe_2 group are disordered. The space group permits the compound in the crystal to be enantiomerically pure, but the absolute configuration of the molecule has not been determined. The enantiomer used in the refinement was based on the known *S*-configuration of the stereogenic centres of the hydroxy acids. The terminal hydroxy group and all amide N—H groups act as donors for hydrogen bonds. N4—H is involved in an intramolecular interaction with the central amide O atom, O40 (Table 2), to give a loop with a graph-set motif (Bernstein *et al.*, 1995) of *S*(10), *i.e.* a β-turn. N10—H forms a similar intramolecular interaction with amide atom O27 at the hydroxy end of the molecule to give another graph-set motif of

Table 3
Torsion angles ω, ϕ and ψ (°) of the backbone of the depsipeptide molecules in the structures of (**3b**), (**5a**), (**5b**) and (**5c**).



	Amino/ hydroxy acid†		Atoms	Torsion angles
(**3b**)	Pms(1)	ψ_1	O19—C18—C17—N16	−9.6 (9)
		ω_1	C18—C17—N16—C15	179.0 (6)
	Acp(2)	ϕ_2	C17—N16—C15—C14	−49.1 (8)
		ψ_2	N16—C15—C14—O13	−34.2 (7)
		ω_2	C15—C14—O13—C12	−171.3 (5)
	Pms(3)	ϕ_3	C14—O13—C12—C11	−77.1 (6)
		ψ_3	O13—C12—C11—N10	−17.0 (8)
		ω_3	C12—C11—N10—C9	179.0 (5)
	Acp(4)	ϕ_4	C11—N10—C9—C8	−49.7 (7)
		ψ_4	N10—C9—C8—O7	−35.2 (7)
		ω_4	C9—C8—O7—C6	−167.0 (5)
	Pms(5)	ϕ_5	C8—O7—C6—C5	−93.5 (6)
		ψ_5	O7—C6—C5—N4	−5.5 (7)
		ω_5	C6—C5—N4—C3	178.9 (6)
	Acp(6)	ϕ_6	C5—N4—C3—C2	51.6 (8)
		ψ_6	N4—C3—C2—N1	43.6 (10)
(**5a**)‡	Pms(1)	ψ_1	O13—C12—C11—N10	−18.9 (6); −17.2 (5)
		ω_1	C12—C11—N10—C9	−173.3 (4); −171.1 (4)
	Aib(2)	ϕ_2	C11—N10—C9—C8	−51.5 (6); −50.9 (6)
		ψ_2	N10—C9—C8—O7	−33.1 (6); −33.8 (6)
		ω_2	C9—C8—O7—C6	−169.9 (3); −173.1 (4)
	Pms(3)	ϕ_3	C8—O7—C6—C5	−98.6 (4); −96.0 (5)
		ψ_3	O7—C6—C5—N4	−2.1 (6); −6.9 (6)
		ω_3	C6—C5—N4—C3	178.5 (4); −179.4 (4)
	Aib(4)	ϕ_4	C5—N4—C3—C2	−48.8 (6); −49.1 (5)
		ψ_4	N4—C3—C2—N1	−43.2 (6); −44.3 (5)
(**5b**)	Pms(1)	ψ_1	O13—C12*a*—C11—N10§	22.1 (8)
			O13—C12*b*—C11—N10¶	−13.0 (8)
		ω_1	C12*a*—C11—N10—C9§	166.4 (4)
			C12*b*—C11—N10—C9¶	−174.9 (4)
	Acp(2)	ϕ_2	C11—N10—C9—C8	51.3 (4)
		ψ_2	N10—C9—C8—O7	37.3 (3)
		ω_2	C9—C8—O7—C6	169.0 (2)
	Pms(3)	ϕ_3	C8—O7—C6—C5	84.6 (3)
		ψ_3	O7—C6—C5—N4	2.5 (4)
		ω_3	C6—C5—N4—C3	175.7 (2)
	Acp(4)	ϕ_4	C5—N4—C3—C2	46.8 (3)
		ψ_4	N4—C3—C2—N1	54.9 (3)
(**5c**)	Mns(1)	ψ_1	O13—C12—C11—N10	15.3 (7)
		ω_1	C12—C11—N10—C9	−170.0 (5)
	Acp(2)	ϕ_2	C11—N10—C9—C8	−64.2 (7)
		ψ_2	N10—C9—C8—O7	−15.9 (7)
		ω_2	C9—C8—O7—C6	179.8 (4)
	Mns(3)	ϕ_3	C8—O7—C6—C5	−76.1 (5)
		ψ_3	O7—C6—C5—N4	−13.2 (6)
		ω_3	C6—C5—N4—C3	179.7 (5)
	Acp(4)	ϕ_4	C5—N4—C3—C2	54.5 (6)
		ψ_4	N4—C3—C2—N1	48.3 (7)

† Acp is 1-aminocyclopentanecarboxylic acid, Pms is phenyllactic acid, Aib is aminoisobutyric acid and Mns is mandelic acid. ‡ Two symmetry-independent molecules. The atom numbers for molecule *A* are given and those of molecule *B* are obtained by adding 40; the torsion angles for molecule *B* are listed second. § *S*,*R*-isomer. ¶ *R*,*R*-isomer.

S(10). These two intramolecular hydrogen bonds stabilize the hexadepsipeptide backbone in a distorted 3_{10}-helical conformation comparable with that of the hexa-Aib peptide Z-(Aib)$_6$-N(Me)Ph (Dannecker-Dörig *et al.*, 2011) and also the decapeptide Z-(Aib)$_{10}$-OH (Gessmann *et al.*, 2016). The torsion angles of Acp(2) and Acp(4), *i.e.* ϕ_2/ψ_2 and ϕ_4/ψ_4, respectively, are close to the characteristic values of −60/−30° (β-turn of type III) of a right-handed 3_{10}-helix (Table 3). On the other hand, the corresponding torsion angles of the phenyllactic acids Pms(3) and Pms(5) deviate significantly from the expected values in a 3_{10}-helical structure. The combination of the torsion angles ϕ_2/ψ_2; ϕ_3/ψ_3 and ϕ_4/ψ_4; ϕ_5/ψ_5, respectively, are in good agreement for two consecutive β-turns of type I, whereas in poly(Aib)-peptides, β-turns of type III dominate. The amide group at the hydroxy end of the molecule, N16—H, forms an intermolecular hydrogen bond with amide atom O53^{i} near the –NMe$_2$ end of an adjacent molecule (in this discussion, the symmetry codes are as defined in the relevant table). This interaction links the molecules into extended chains, which run parallel to the [001] direction and can be described by a graph-set motif of *C*(14). Hydroxy group O19—H forms an intermolecular hydrogen bond with ester carbonyl atom O45^{i} near the opposite end of the same neighbouring molecule. This interaction reinforces the chain described above to give a double-bridged chain and can also be described by a graph-set motif of *C*(14) (Fig. 2). Double-bridges produce rings within the chain links that have a graph-set motif of $R_2^2(12)$.

The crystals of the tetradepsipeptide (**5a**) are enantiomerically pure, but the absolute configuration of the molecule has not been determined. The enantiomer used in the refinement was based on the known *S*-configuration at C6 and

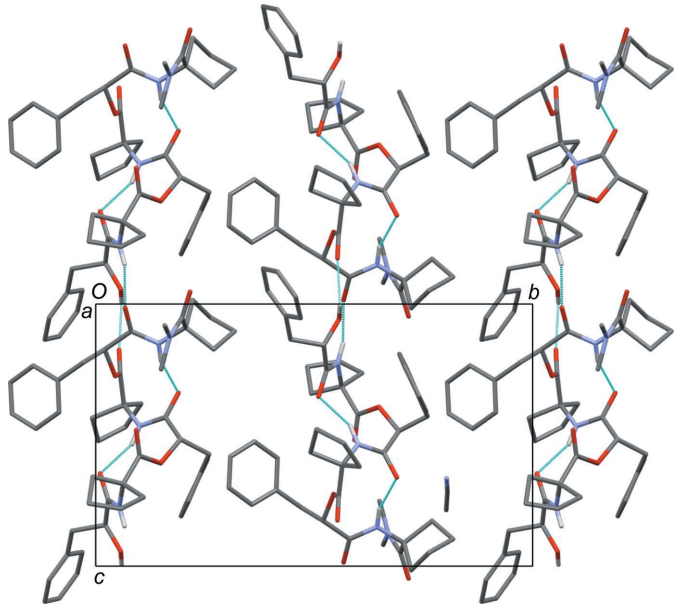


Figure 2
The crystal packing of (**3b**) projected down the *a* axis and showing the hydrogen-bonded chains progressing parallel to the [001] direction. H atoms not involved in hydrogen-bonding interactions and the minor-disorder conformations have been omitted for clarity.

C12. There are two independent molecules, denoted *A* and *B*, in the asymmetric unit (Figs. 3*a* and 3*b*). These molecules have very similar geometries and are of the same enantiomer, the main differences being in the orientations of the phenyl rings. The conformation of molecule *B* is attained when the phenyl rings of the benzyl groups at C6 and C12 of molecule *A* are twisted by approximately 40 and 21°, respectively, about their

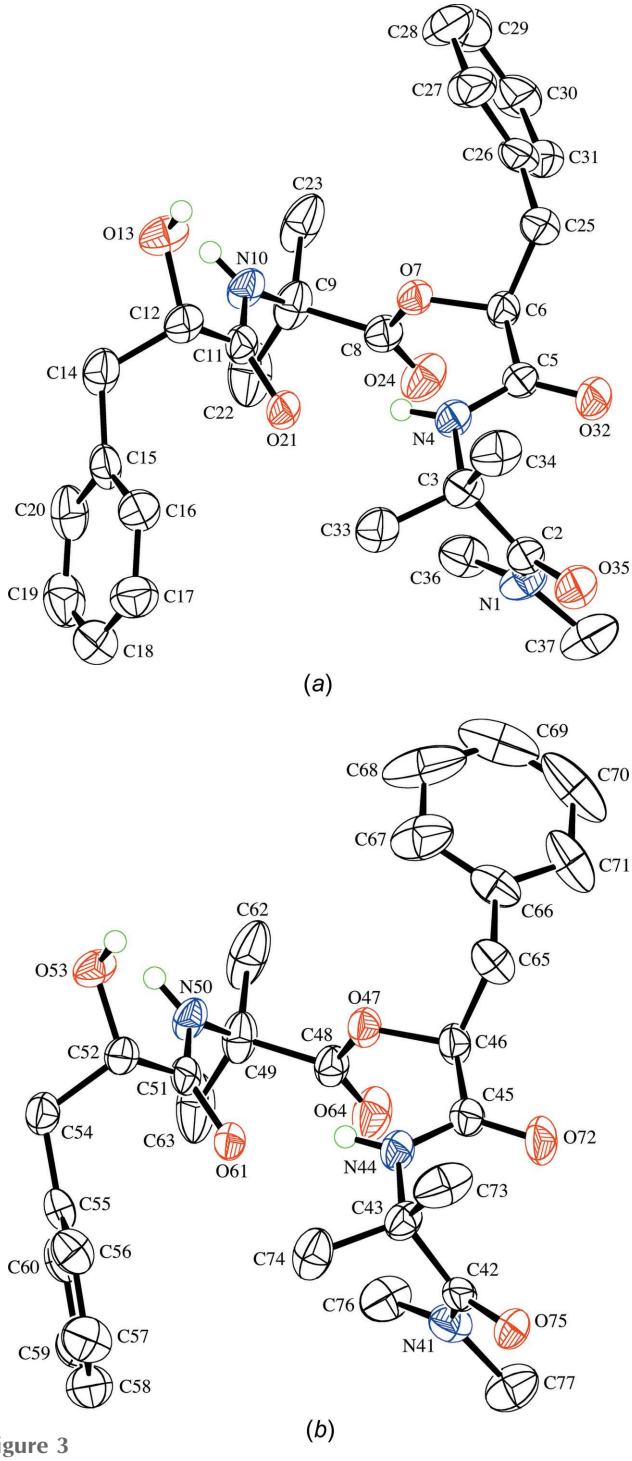


Figure 3
View of the symmetry-independent molecules of (**5a**), showing the atom-labelling scheme of (*a*) molecule *A* and (*b*) molecule *B*. Displacement ellipsoids are drawn at the 50% probability level. Most H atoms have been omitted for clarity.

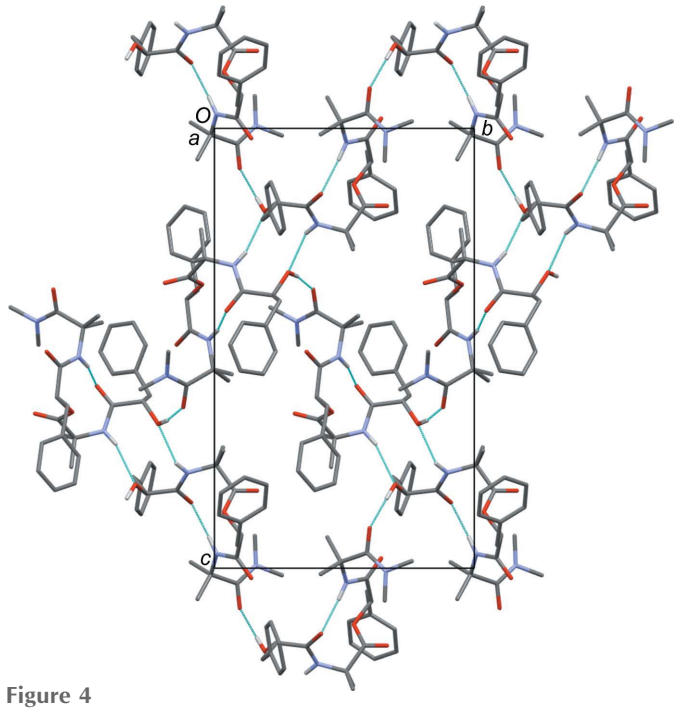


Figure 4
The crystal packing of (**5a**) projected down the *a* axis and showing one of the 2D hydrogen-bonded layers. H atoms not involved in hydrogen-bonding interactions have been omitted for clarity.

axes. The hydroxy and all amide groups act as donors for hydrogen bonds. Molecules *A* and *B* each form one intramolecular hydrogen bond between the amide group near the –NMe_2 end of the molecule (N4—H or N44—H) and the amide O atom (O21 or O61) at the hydroxy end of the molecule (Table 2). These interactions give loops with a graph-set motif (Bernstein *et al.*, 1995) of $S(10)$; the molecules form β-turns analogous to those in the hexadepsipeptide (**3b**). Again, the torsion angles of Aib(2), *i.e.* ϕ_2/ψ_2, are close to the characteristic values of −60/−30° of a right-handed 3_{10}-helix (Table 3), but those of Pms(3) involved in the β-turn deviate clearly from these ideal values, while being in good agreement with those of β-turns of type I. The hydroxy group of molecule *A*, O13—H, forms an intermolecular hydrogen bond with atom O35^{ii} of the terminal amide group at the opposite end of a neighbouring molecule *A*, thus forming extended chains, which run parallel to the [010] direction and have a graph-set motif of $C(14)$. Identical chains, also running parallel to the [010] direction, are formed among the type *B* molecules (O53—H···O75^{iv}). The remaining amide group of molecule *A*, N10—H, forms an intermolecular hydrogen bond with hydroxy atom O53^{iii} of a neighbouring molecule *B*, and the corresponding amide group of molecule *B*, N50—H, hydrogen bonds back to hydroxy atom O13^{v} of the same molecule *A*, thus forming a closed dimeric system with a graph-set motif of $R_2^2(10)$. The combination of all intermolecular hydrogen bonds links the molecules into two-dimensional (2D) networks, which lie parallel to the $(10\bar{1})$ plane (Fig. 4).

Since the space group of (**5b**) is centrosymmetric, the compound in the crystal is racemic. Both benzyl substituents exhibit disorder. Two approximately equally occupied orien-

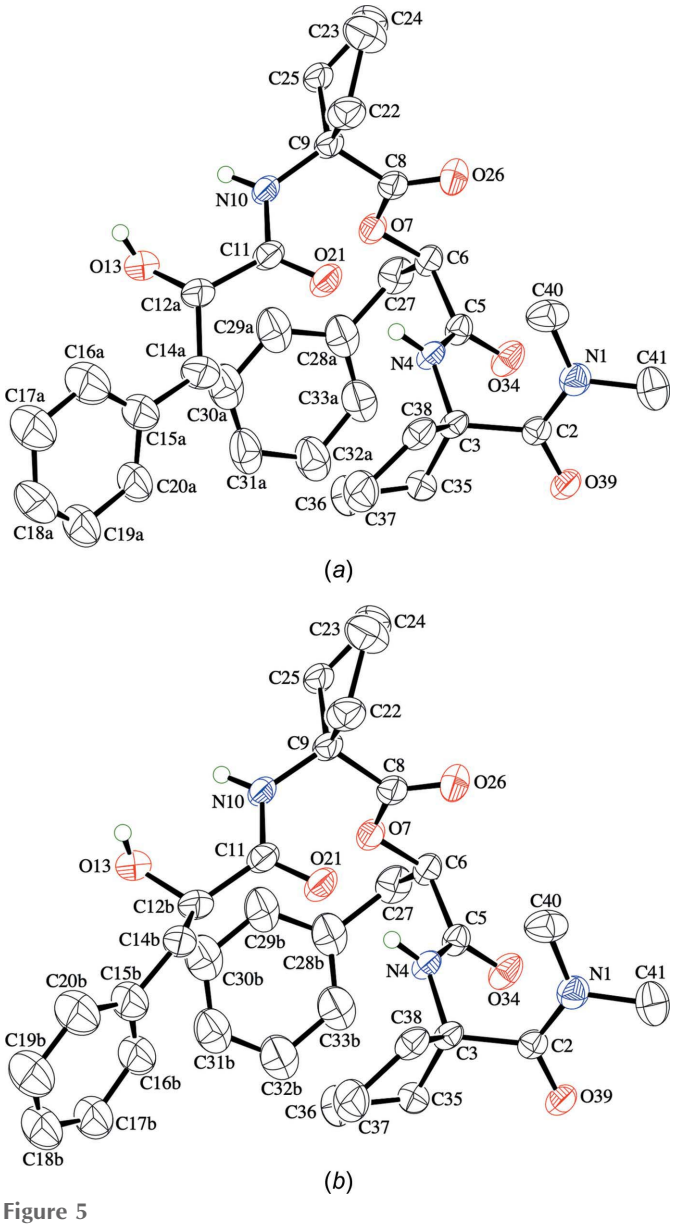


Figure 5
Views of the two diastereoisomers of (**5b**) disordered over the same crystallographic site with almost equal occupation. The atom-labelling scheme is shown for (*a*) the 6*R*,12*S* diastereoisomer and (*b*) the 6*R*,12*R* diastereoisomer (the enantiomers of these molecules are also present in this centrosymmetric structure). Displacement ellipsoids are drawn at the 50% probability level. Most H atoms and the minor-disorder conformations of the benzyl groups have been omitted for clarity.

tations were modelled for just the phenyl ring of the central benzyl group and for the entire terminal benzyl group, including the stereogenic C atom, C12 (Figs. 5*a* and 5*b*). The disorder at C12 is a consequence of inversion at that atom and indicates that the compound actually contains almost equal quantities of two racemic diastereoisomers, which crystallize disordered over the same crystallographic site. Each amide and hydroxy group of the molecule acts as a donor for hydrogen bonds, thereby forming one intramolecular and two intermolecular interactions. The two N—H groups in the molecule form hydrogen bonds to amide O atoms. N4—H is involved in an intramolecular interaction with amide atom O21 at the hydroxy end of the molecule (Table 2) to give a β-turn with a graph-set motif of $S(10)$. Similar to (**3b**) and (**5a**), the torsion angles ϕ_2/ψ_2 of Acp(2) with values of 51.3 (4) and 37.3 (3)°, and ϕ_3/ψ_3 of Pms(3) with values of 84.6 (3) and 2.5 (4)°, respectively, correspond well with those of a β-turn of type I′ (Table 3). As (**5b**) is racemic, an equal number of molecules with the corresponding type I turn are also present. N10—H forms an intermolecular hydrogen bond with the secondary amide O atom, O39^{vi}, at the opposite end of a neighbouring molecule. This interaction links the molecules into chains, which run parallel to the [010] direction and can be described by a graph-set motif of $C(11)$. Hydroxy group O13—H forms an intermolecular hydrogen bond with the primary amide O atom, O34^{vi}, near the –NMe$_2$ end of the same adjacent molecule. This interaction reinforces the chain described above to give a double-bridged chain and can also be described by a graph-set motif of $C(11)$. Double-bridges produce rings within the chain links that have a graph-set motif of $R_2^2(12)$. The intermolecular interactions link the molecules into extended chains, which run parallel to the [010] direction (Fig. 6).

Finally, the space group of (**5c**) is noncentrosymmetric, but the presence of glide planes indicates that the compound in the crystal is racemic. The absolute structure could not be confirmed by refinement of the absolute structure parameter and was chosen arbitrarily. One of the five-membered rings of the molecule exhibits conformational disorder, which appears

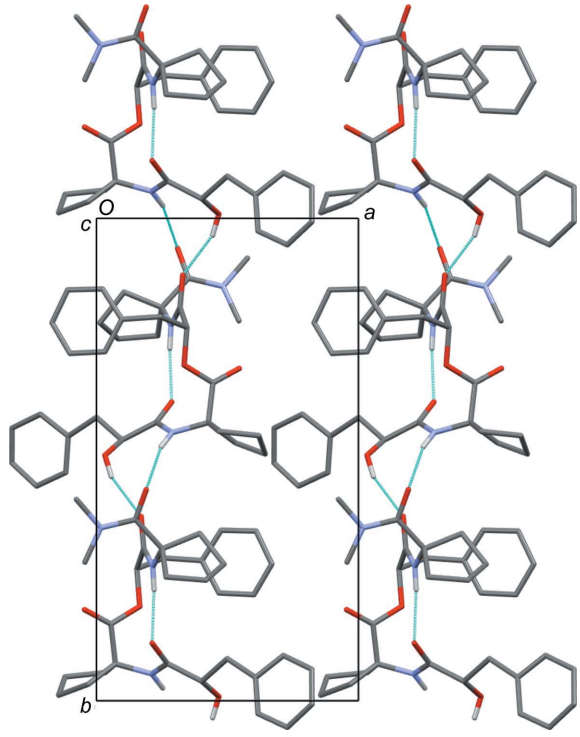


Figure 6
A partial packing diagram for (**5b**) projected down the *c* axis and showing the hydrogen-bonded chains progressing parallel to the [010] direction. The inversion-related chain that runs between the two chains shown has been excluded, while H atoms not involved in hydrogen-bonding interactions and the minor-disorder components have been omitted for clarity.

to correspond with a different atom forming the flap of the envelope conformation of the five-membered ring (Fig. 7). The two amide groups in the molecule act as hydrogen-bond donors. N4—H is involved in an intramolecular interaction with amide atom O20 at the hydroxy end of the molecule (Table 2) to give a loop with a graph-set motif of $S(10)$. The values of the torsion angles ϕ_2/ψ_2 of Acp(2) with values of −64.2 (7) and −15.9 (7)°, and ϕ_3/ψ_3 of Mns(3) with values of −76.1 (5) and −13.2 (6)°, respectively, are in between the typical values for β-turns of type I and type III (Table 3). N10—H forms an intermolecular hydrogen bond with amide atom O37^{viii} at the opposite end of a neighbouring molecule. This interaction links the molecules into extended chains, which run parallel to the [011] direction and can be described by a graph-set motif of $C(11)$. Hydroxy group O13—H forms an intermolecular hydrogen bond with amide atom O32^{vii} near the –NMe$_2$ end of a different adjacent molecule. This interaction links the molecules into chains, which run parallel to the [0$\bar{1}$1] direction and can also be described by a graph-set motif of $C(11)$. The combination of all intermolecular hydrogen bonds links the molecules into 2D networks, which lie parallel to the (100) plane (Fig. 8).

In summary, all of the studied depsipeptides with an alternating sequence of an α,α-disubstituted α-amino acid and an α-hydroxy acid exist in the crystal in a β-turn conformation. Whereas β-turns of type I (or I′) are formed in the cases of (**3b**), (**5a**) and (**5b**), which contain phenyllactic acid [Pms, (**2a**)], the torsion angles for (**5c**), which incorporates mandelic acid [Mns, (**2b**)], indicate a β-turn in between type I and type III. Crystal structures of linear depsipeptides are rare. The only detailed studies have been carried out by Katakai and coworkers on Boc-(Leu-Leu-Ala)$_2$-(Leu-Leu-Lac)$_3$-OEt (Ohyama *et al.*, 2000), Boc-(Leu-Leu-Lac)$_3$-Leu-Leu-OEt (Ohyama *et al.*, 2001) and Boc-Leu-Leu-Ala-(Leu-Leu-Lac)$_3$-Leu-Leu-OEt (Oku *et al.*, 2004) (Lac is lactic acid). In the first case, the crystal structure can be described as an α-helix with two 3_{10}-helical segments located at the N-terminus and in the middle of the chain, between the peptide and the depsipeptide units, whereas in the second case, an almost ideal α-helical conformation was found. The conformation of the third depsipeptide, which has been prepared by condensation of the tripeptide segment Boc-Leu-Leu-Ala-OH with the α-helical depsipeptide sequence H-(Leu-Leu-Lac)$_3$-Leu-Leu-OEt, was described as an '$\alpha/3_{10}$-conjugated helix with a kink at the junction of the peptide and depsipeptide segments'.

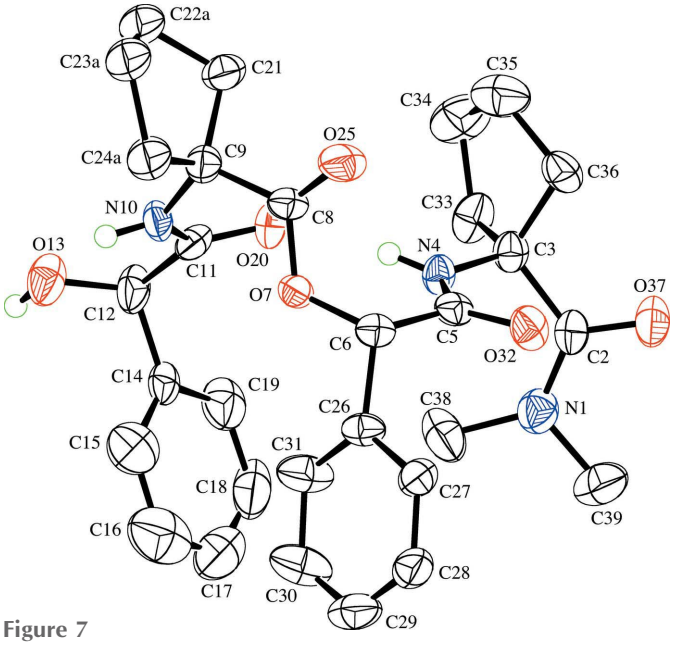


Figure 7
View of the molecule of (**5c**), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Most H atoms and one conformation of the disordered five-membered ring have been omitted for clarity.

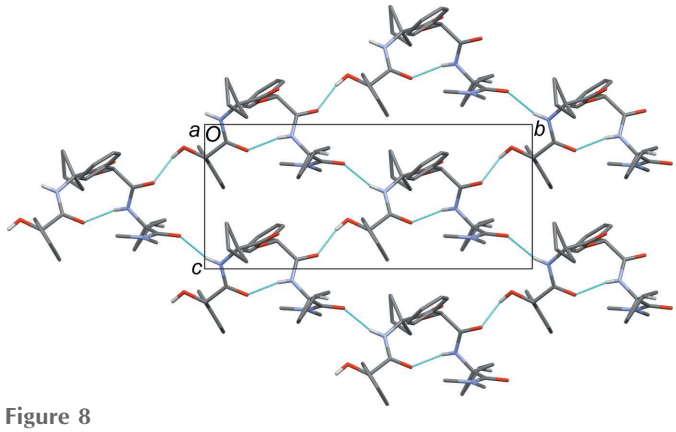


Figure 8
A partial packing diagram for (**5c**) projected down the *a* axis and showing one of the 2D hydrogen-bonded layers. H atoms not involved in hydrogen-bonding interactions have been omitted for clarity.

Theoretical conformational analysis of polydepsipeptides poly(Ala-Lac) with an alternating sequence of L-alanine and L-lactic acid showed that a helical structure similar to a right-handed α-helix of a polypeptide is energetically preferred (Ingwall & Goodman, 1974; Goodman, 1985). The 3_{10}-helix was identified as the second low-energy conformation. Experimentally, on the basis of circular dichroism (CD), IR and NMR measurements, the 3_{10}-helical conformation, *i.e.* the formation of β-turns of type I/II, was found as the dominant structure (Ingwall *et al.*, 1976). These and other studies in solution confirm that the conformational stability of depsipeptides is significantly lower than that of related peptides as a result of the smaller number of intramolecular hydrogen bonds (Wouters *et al.*, 1982; Becktel *et al.*, 1985; Arad & Goodman, 1990; Katakai *et al.*, 1996).

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supporting information

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Solid-state conformations of linear depsipeptide amides with an alternating sequence of α,α-disubstituted α-amino acid and α-hydroxy acid

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Computing details

For all structures, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991); cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991); data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEPII* (Johnson, 1976) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(*S*)-Pms-Acp-(*S*)-Pms-Acp-(*S*)-Pms-Acp-NMe$_2$ (3b)

Crystal data

$C_{47}H_{58}N_4O_9 \cdot C_2H_3N$
M_r = 864.02
Monoclinic, $P2_1$
a = 10.645 (3) Å
b = 19.161 (9) Å
c = 11.503 (3) Å
β = 93.75 (2)°
V = 2341.5 (15) Å^3
Z = 2
$F(000)$ = 924
D_x = 1.226 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 20 reflections
θ = 10.0–12.5°
μ = 0.09 mm^{-1}
T = 173 K
Prism, colourless
0.48 × 0.33 × 0.23 mm

Data collection

Rigaku AFC-5R diffractometer
Radiation source: Rigaku RU200 rotating anode generator
Graphite monochromator
ω–2θ scans
5821 measured reflections
5527 independent reflections
3617 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 27.5°, θ_{min} = 2.5°
h = 0→13
k = 0→24
l = −14→14
3 standard reflections every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.218
S = 1.04
5527 reflections
632 parameters
229 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0906P)^2 + 3.1019P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.009 (2)
Absolute structure: No quotients, so Flack parameter determined by classical intensity fit
Absolute structure parameter: 0 (2)

Special details

Experimental. Solvent used: MeCN

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

Refinement. The asymmetric unit contains one molecule of the peptide and one molecule of MeCN. Two five-membered rings and the terminal -NMe2 group are disordered. Two sets of positions were defined for two of the -CH2- groups in two of the five-membered rings and for the -NMe2 methyl groups. The site occupation factors of the major conformations of these groups refined to 0.597 (18), 0.59 (2) and 0.880 (18), respectively. Similarity restraints were applied to the chemically equivalent bond lengths and angles involving all disordered C-atoms, while neighbouring atoms within and between each conformation of the disordered groups were restrained to have similar atomic displacement parameters.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O7	0.8591 (4)	0.5444 (3)	0.7255 (3)	0.0263 (9)	
O13	0.9280 (4)	0.6485 (3)	0.3926 (3)	0.0288 (9)	
O19	0.7833 (5)	0.5572 (4)	0.0570 (4)	0.0494 (14)	
H19	0.7232	0.5592	0.0060	0.074*	
O27	0.8268 (4)	0.5100 (3)	0.3529 (3)	0.0351 (11)	
O32	1.0631 (4)	0.5925 (3)	0.5188 (3)	0.0358 (11)	
O40	0.7911 (4)	0.6893 (3)	0.6611 (3)	0.0332 (10)	
O45	0.6831 (4)	0.5534 (3)	0.8246 (4)	0.0451 (13)	
O53	1.0616 (4)	0.5672 (3)	0.9855 (3)	0.0384 (11)	
O58	1.2172 (5)	0.7160 (4)	1.0466 (4)	0.0549 (15)	
N1	1.2599 (6)	0.6696 (5)	0.8749 (5)	0.068 (2)	
N4	0.9841 (5)	0.6441 (3)	0.8501 (4)	0.0329 (12)	
H4	0.9416	0.6493	0.7825	0.040*	
N10	0.7488 (4)	0.6005 (3)	0.5350 (4)	0.0290 (11)	
H10	0.7591	0.5829	0.4656	0.035*	
N16	0.9577 (5)	0.5538 (3)	0.2256 (4)	0.0297 (11)	
H16	0.9681	0.5658	0.1530	0.036*	
C2	1.1778 (8)	0.6977 (5)	0.9482 (6)	0.050 (2)	
C3	1.0395 (7)	0.7056 (4)	0.9112 (6)	0.0419 (18)	
C5	0.9978 (6)	0.5811 (4)	0.8964 (5)	0.0303 (14)	
C6	0.9344 (5)	0.5204 (4)	0.8268 (5)	0.0262 (13)	
H6	0.8805	0.4931	0.8784	0.031*	
C8	0.7356 (6)	0.5559 (4)	0.7345 (5)	0.0316 (14)	
C9	0.6667 (5)	0.5644 (4)	0.6146 (5)	0.0300 (13)	
C11	0.8073 (6)	0.6595 (4)	0.5681 (5)	0.0313 (14)	

C12	0.8925 (6)	0.6941 (4)	0.4841 (5)	0.0322 (14)
H12	0.9711	0.7092	0.5295	0.039*
C14	1.0192 (5)	0.6003 (4)	0.4211 (5)	0.0279 (13)
C15	1.0631 (5)	0.5627 (4)	0.3139 (5)	0.0260 (12)
C17	0.8484 (6)	0.5284 (4)	0.2525 (5)	0.0290 (13)
C18	0.7437 (6)	0.5220 (4)	0.1568 (5)	0.0336 (15)
H18	0.6676	0.5464	0.1831	0.040*
C20	0.7098 (7)	0.4457 (4)	0.1344 (7)	0.0454 (18)
H201	0.7849	0.4210	0.1089	0.055*
H202	0.6872	0.4243	0.2085	0.055*
C21	0.6026 (7)	0.4351 (4)	0.0442 (6)	0.0411 (16)
C22	0.6134 (9)	0.3935 (6)	−0.0518 (8)	0.062 (2)
H22	0.6914	0.3709	−0.0619	0.075*
C23	0.5132 (9)	0.3833 (6)	−0.1353 (8)	0.071 (3)
H23	0.5231	0.3534	−0.2000	0.085*
C24	0.4015 (9)	0.4164 (6)	−0.1231 (8)	0.066 (3)
H24	0.3333	0.4109	−0.1799	0.079*
C25	0.3895 (8)	0.4575 (6)	−0.0282 (8)	0.067 (3)
H25	0.3107	0.4791	−0.0178	0.080*
C26	0.4875 (7)	0.4690 (6)	0.0530 (7)	0.061 (3)
H26	0.4769	0.5002	0.1158	0.073*
C28	1.1662 (6)	0.6094 (4)	0.2632 (5)	0.0301 (14)
H281	1.1296	0.6385	0.1983	0.036*
H282	1.2053	0.6404	0.3241	0.036*
C29	1.2641 (6)	0.5580 (4)	0.2192 (6)	0.0383 (15)
H291	1.2753	0.5658	0.1355	0.046*
H292	1.3464	0.5642	0.2632	0.046*
C30	1.2121 (7)	0.4844 (4)	0.2391 (6)	0.0425 (17)
H301	1.1620	0.4676	0.1691	0.051*
H302	1.2812	0.4510	0.2585	0.051*
C31	1.1294 (6)	0.4935 (4)	0.3414 (6)	0.0334 (14)
H311	1.0681	0.4549	0.3450	0.040*
H312	1.1808	0.4960	0.4161	0.040*
C33	0.8301 (6)	0.7593 (4)	0.4276 (6)	0.0366 (15)
H331	0.8140	0.7937	0.4890	0.044*
H332	0.8882	0.7808	0.3742	0.044*
C34	0.7080 (6)	0.7414 (4)	0.3609 (5)	0.0318 (14)
C35	0.5952 (7)	0.7561 (5)	0.4082 (7)	0.050 (2)
H35	0.5959	0.7782	0.4822	0.060*
C36	0.4816 (8)	0.7395 (6)	0.3506 (8)	0.064 (3)
H36	0.4047	0.7495	0.3847	0.077*
C37	0.4813 (8)	0.7072 (5)	0.2389 (9)	0.066 (3)
H37	0.4041	0.6943	0.1987	0.079*
C38	0.5913 (8)	0.6952 (5)	0.1909 (8)	0.063 (3)
H38	0.5910	0.6745	0.1158	0.076*
C39	0.7061 (7)	0.7128 (5)	0.2501 (6)	0.0464 (18)
H39	0.7828	0.7051	0.2143	0.056*
C41	0.5396 (6)	0.6037 (5)	0.6198 (6)	0.0446 (15)

H411	0.5093	0.6010	0.6992	0.054*	0.597 (18)
H412	0.5499	0.6534	0.5993	0.054*	0.597 (18)
H413	0.5505	0.6549	0.6163	0.054*	0.403 (18)
H414	0.4956	0.5912	0.6901	0.054*	0.403 (18)
C42A	0.4448 (10)	0.5678 (8)	0.5311 (12)	0.0503 (19)	0.597 (18)
H421	0.4157	0.6011	0.4693	0.060*	0.597 (18)
H422	0.3707	0.5509	0.5705	0.060*	0.597 (18)
C43A	0.5150 (10)	0.5057 (7)	0.4779 (11)	0.0478 (18)	0.597 (18)
H431	0.4600	0.4641	0.4703	0.057*	0.597 (18)
H432	0.5431	0.5180	0.4001	0.057*	0.597 (18)
C42B	0.4712 (19)	0.5743 (8)	0.5069 (11)	0.0474 (19)	0.403 (18)
H423	0.5131	0.5889	0.4364	0.057*	0.403 (18)
H424	0.3816	0.5884	0.4993	0.057*	0.403 (18)
C43B	0.4864 (8)	0.4949 (9)	0.531 (2)	0.0487 (19)	0.403 (18)
H433	0.4357	0.4793	0.5949	0.058*	0.403 (18)
H434	0.4640	0.4668	0.4600	0.058*	0.403 (18)
C44	0.6296 (6)	0.4919 (5)	0.5652 (6)	0.0428 (15)	
H441	0.6063	0.4602	0.6282	0.051*	0.597 (18)
H442	0.7000	0.4709	0.5252	0.051*	0.597 (18)
H443	0.6478	0.4554	0.6248	0.051*	0.403 (18)
H444	0.6773	0.4813	0.4964	0.051*	0.403 (18)
C46	1.0330 (6)	0.4724 (4)	0.7782 (6)	0.0347 (15)	
H461	1.0863	0.4995	0.7272	0.042*	
H462	1.0879	0.4531	0.8431	0.042*	
C47	0.9702 (5)	0.4133 (4)	0.7094 (5)	0.0278 (13)	
C48	0.9559 (6)	0.4160 (4)	0.5875 (5)	0.0352 (15)	
H48	0.9893	0.4544	0.5474	0.042*	
C49	0.8936 (7)	0.3632 (4)	0.5247 (6)	0.0394 (16)	
H49	0.8865	0.3649	0.4420	0.047*	
C50	0.8422 (7)	0.3083 (4)	0.5820 (6)	0.0422 (17)	
H50	0.7986	0.2725	0.5387	0.051*	
C51	0.8535 (7)	0.3048 (4)	0.7009 (6)	0.0426 (17)	
H51	0.8184	0.2665	0.7399	0.051*	
C52	0.9163 (6)	0.3572 (4)	0.7650 (5)	0.0340 (14)	
H52	0.9225	0.3548	0.8476	0.041*	
C54	1.0241 (9)	0.7699 (5)	0.8276 (7)	0.067 (2)	
H541	1.1061	0.7924	0.8160	0.080*	0.59 (2)
H542	0.9833	0.7567	0.7510	0.080*	0.59 (2)
H543	1.0968	0.8019	0.8401	0.080*	0.41 (2)
H544	1.0187	0.7545	0.7453	0.080*	0.41 (2)
C55A	0.9380 (16)	0.8178 (7)	0.8965 (16)	0.070 (2)	0.59 (2)
H551	0.8896	0.8507	0.8445	0.085*	0.59 (2)
H552	0.9867	0.8443	0.9580	0.085*	0.59 (2)
C56A	0.8513 (12)	0.7625 (8)	0.9489 (16)	0.068 (2)	0.59 (2)
H561	0.7917	0.7832	1.0018	0.081*	0.59 (2)
H562	0.8051	0.7339	0.8885	0.081*	0.59 (2)
C55B	0.9008 (17)	0.8070 (14)	0.8570 (19)	0.069 (2)	0.41 (2)
H553	0.8325	0.7960	0.7973	0.083*	0.41 (2)

H554	0.9133	0.8582	0.8587	0.083*	0.41 (2)
C56B	0.866 (2)	0.7807 (12)	0.9782 (17)	0.068 (2)	0.41 (2)
H563	0.8719	0.8193	1.0356	0.082*	0.41 (2)
H564	0.7784	0.7626	0.9737	0.082*	0.41 (2)
C57	0.9598 (8)	0.7219 (5)	1.0151 (7)	0.0610 (19)	
H571	1.0061	0.7512	1.0745	0.073*	0.59 (2)
H572	0.9295	0.6789	1.0520	0.073*	0.59 (2)
H573	1.0150	0.7370	1.0830	0.073*	0.41 (2)
H574	0.9135	0.6796	1.0374	0.073*	0.41 (2)
C59A	1.2331 (11)	0.6496 (9)	0.7517 (6)	0.067 (3)	0.880 (18)
H591	1.2298	0.5987	0.7454	0.100*	0.880 (18)
H592	1.2997	0.6676	0.7050	0.100*	0.880 (18)
H593	1.1520	0.6694	0.7231	0.100*	0.880 (18)
C60A	1.3921 (8)	0.6580 (8)	0.9182 (8)	0.072 (3)	0.880 (18)
H601	1.4372	0.7026	0.9209	0.107*	0.880 (18)
H602	1.4325	0.6258	0.8658	0.107*	0.880 (18)
H603	1.3942	0.6378	0.9966	0.107*	0.880 (18)
C59B	1.195 (7)	0.673 (7)	0.758 (3)	0.068 (3)	0.120 (18)
H594	1.1354	0.6344	0.7474	0.102*	0.120 (18)
H595	1.2567	0.6706	0.6985	0.102*	0.120 (18)
H596	1.1487	0.7176	0.7493	0.102*	0.120 (18)
C60B	1.363 (6)	0.717 (4)	0.915 (6)	0.070 (3)	0.120 (18)
H604	1.3276	0.7625	0.9356	0.105*	0.120 (18)
H605	1.4201	0.7238	0.8523	0.105*	0.120 (18)
H606	1.4096	0.6972	0.9832	0.105*	0.120 (18)
N63	0.3687 (16)	0.8031 (9)	0.6648 (12)	0.150 (6)	
C61	0.5884 (13)	0.8022 (9)	0.7855 (15)	0.127 (5)	
H611	0.5770	0.8187	0.8647	0.190*	
H612	0.6497	0.8320	0.7493	0.190*	
H613	0.6192	0.7540	0.7884	0.190*	
C62	0.4663 (13)	0.8050 (7)	0.7159 (12)	0.095 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.025 (2)	0.034 (3)	0.0193 (18)	0.0008 (18)	0.0000 (15)	0.0000 (17)
O13	0.030 (2)	0.036 (3)	0.0197 (19)	0.001 (2)	−0.0024 (15)	−0.0010 (18)
O19	0.051 (3)	0.069 (4)	0.028 (2)	−0.023 (3)	−0.002 (2)	0.010 (3)
O27	0.042 (3)	0.043 (3)	0.020 (2)	−0.001 (2)	0.0004 (18)	0.0017 (19)
O32	0.037 (2)	0.047 (3)	0.022 (2)	0.008 (2)	−0.0031 (17)	−0.004 (2)
O40	0.038 (2)	0.035 (3)	0.026 (2)	0.007 (2)	−0.0006 (18)	−0.0058 (19)
O45	0.036 (2)	0.079 (4)	0.021 (2)	−0.002 (3)	0.0027 (17)	−0.001 (2)
O53	0.035 (2)	0.055 (3)	0.024 (2)	−0.007 (2)	−0.0065 (18)	−0.002 (2)
O58	0.057 (3)	0.075 (4)	0.031 (2)	−0.034 (3)	−0.011 (2)	−0.002 (3)
N1	0.051 (3)	0.119 (6)	0.035 (3)	−0.027 (4)	0.008 (2)	−0.009 (3)
N4	0.033 (3)	0.040 (3)	0.024 (2)	−0.016 (2)	−0.007 (2)	−0.001 (2)
N10	0.028 (3)	0.041 (3)	0.017 (2)	0.002 (2)	−0.0002 (18)	−0.002 (2)
N16	0.034 (3)	0.040 (3)	0.015 (2)	−0.001 (2)	−0.0002 (18)	0.000 (2)

C2	0.060 (5)	0.058 (5)	0.031 (3)	−0.039 (4)	−0.006 (3)	0.001 (3)
C3	0.066 (5)	0.030 (4)	0.029 (3)	−0.019 (3)	−0.010 (3)	0.001 (3)
C5	0.029 (3)	0.039 (4)	0.023 (3)	−0.006 (3)	0.003 (2)	−0.002 (3)
C6	0.021 (3)	0.039 (4)	0.019 (3)	−0.008 (3)	−0.003 (2)	0.001 (2)
C8	0.031 (3)	0.044 (4)	0.020 (3)	−0.003 (3)	0.000 (2)	−0.004 (3)
C9	0.029 (3)	0.040 (4)	0.020 (3)	−0.002 (3)	−0.003 (2)	0.001 (3)
C11	0.032 (3)	0.036 (4)	0.026 (3)	0.006 (3)	0.000 (2)	−0.001 (3)
C12	0.032 (3)	0.037 (4)	0.027 (3)	0.001 (3)	0.000 (2)	−0.007 (3)
C14	0.026 (3)	0.033 (3)	0.024 (3)	−0.003 (3)	0.000 (2)	0.001 (3)
C15	0.026 (3)	0.030 (3)	0.022 (3)	−0.002 (3)	0.000 (2)	0.005 (2)
C17	0.035 (3)	0.024 (3)	0.028 (3)	−0.003 (3)	0.003 (2)	−0.002 (2)
C18	0.036 (3)	0.045 (4)	0.021 (3)	−0.009 (3)	0.009 (2)	0.000 (3)
C20	0.049 (4)	0.040 (4)	0.046 (4)	−0.003 (3)	−0.001 (3)	−0.009 (3)
C21	0.043 (4)	0.032 (4)	0.047 (4)	−0.009 (3)	0.000 (3)	−0.006 (3)
C22	0.062 (5)	0.067 (6)	0.056 (5)	0.006 (5)	−0.003 (4)	−0.026 (5)
C23	0.073 (6)	0.075 (7)	0.064 (6)	−0.012 (5)	−0.003 (5)	−0.045 (5)
C24	0.057 (5)	0.073 (7)	0.064 (6)	−0.019 (5)	−0.016 (4)	−0.022 (5)
C25	0.040 (4)	0.089 (8)	0.071 (6)	−0.005 (5)	−0.001 (4)	−0.034 (5)
C26	0.041 (4)	0.084 (7)	0.059 (5)	−0.005 (4)	0.005 (4)	−0.038 (5)
C28	0.034 (3)	0.032 (3)	0.025 (3)	−0.011 (3)	0.001 (2)	0.001 (3)
C29	0.032 (3)	0.045 (4)	0.038 (3)	−0.002 (3)	0.008 (3)	0.002 (3)
C30	0.047 (4)	0.033 (4)	0.049 (4)	0.004 (3)	0.013 (3)	0.000 (3)
C31	0.042 (4)	0.024 (3)	0.034 (3)	0.001 (3)	−0.001 (3)	0.002 (3)
C33	0.036 (4)	0.034 (4)	0.039 (3)	−0.005 (3)	0.000 (3)	0.001 (3)
C34	0.039 (3)	0.030 (3)	0.027 (3)	0.003 (3)	0.001 (2)	0.012 (3)
C35	0.044 (4)	0.065 (6)	0.041 (4)	0.007 (4)	0.005 (3)	0.012 (4)
C36	0.042 (4)	0.081 (7)	0.071 (6)	0.012 (4)	0.008 (4)	0.033 (5)
C37	0.045 (5)	0.064 (6)	0.084 (7)	−0.013 (4)	−0.026 (4)	0.032 (5)
C38	0.059 (5)	0.065 (6)	0.063 (5)	0.020 (5)	−0.021 (4)	−0.008 (5)
C39	0.044 (4)	0.056 (5)	0.038 (4)	0.008 (4)	−0.005 (3)	−0.004 (3)
C41	0.031 (3)	0.068 (4)	0.033 (3)	0.000 (3)	−0.003 (2)	−0.004 (3)
C42A	0.035 (3)	0.071 (4)	0.044 (3)	−0.003 (3)	−0.003 (3)	−0.003 (3)
C43A	0.035 (3)	0.070 (4)	0.038 (3)	−0.006 (3)	−0.001 (3)	−0.004 (3)
C42B	0.032 (3)	0.070 (4)	0.040 (4)	−0.005 (3)	−0.001 (3)	−0.001 (3)
C43B	0.036 (3)	0.069 (4)	0.040 (4)	−0.007 (3)	−0.002 (3)	−0.004 (3)
C44	0.035 (3)	0.063 (4)	0.030 (3)	−0.013 (3)	−0.003 (2)	−0.004 (3)
C46	0.028 (3)	0.042 (4)	0.033 (3)	0.002 (3)	−0.001 (3)	−0.002 (3)
C47	0.026 (3)	0.030 (3)	0.027 (3)	0.004 (2)	0.000 (2)	−0.003 (3)
C48	0.045 (4)	0.029 (4)	0.031 (3)	0.004 (3)	0.003 (3)	0.005 (3)
C49	0.044 (4)	0.038 (4)	0.035 (3)	0.007 (3)	−0.004 (3)	0.000 (3)
C50	0.048 (4)	0.038 (4)	0.041 (4)	−0.005 (3)	−0.004 (3)	−0.006 (3)
C51	0.054 (4)	0.035 (4)	0.040 (4)	−0.004 (3)	0.008 (3)	0.006 (3)
C52	0.040 (3)	0.037 (4)	0.025 (3)	0.010 (3)	0.002 (3)	0.006 (3)
C54	0.094 (5)	0.051 (4)	0.052 (4)	−0.012 (4)	−0.023 (4)	−0.002 (3)
C55A	0.096 (5)	0.053 (4)	0.059 (4)	−0.002 (4)	−0.022 (4)	−0.004 (4)
C56A	0.094 (5)	0.050 (4)	0.056 (4)	0.001 (4)	−0.018 (4)	−0.011 (3)
C55B	0.096 (5)	0.051 (4)	0.055 (4)	−0.004 (4)	−0.024 (4)	−0.006 (4)
C56B	0.093 (5)	0.052 (4)	0.056 (4)	−0.001 (4)	−0.020 (4)	−0.009 (4)

C57	0.086 (5)	0.046 (4)	0.048 (4)	−0.001 (4)	−0.016 (3)	−0.012 (3)
C59A	0.051 (4)	0.117 (7)	0.034 (3)	−0.026 (4)	0.012 (3)	−0.014 (4)
C60A	0.049 (4)	0.123 (7)	0.043 (4)	−0.022 (4)	0.005 (3)	−0.007 (4)
C59B	0.052 (5)	0.118 (7)	0.035 (4)	−0.027 (5)	0.011 (4)	−0.012 (5)
C60B	0.051 (5)	0.121 (7)	0.040 (4)	−0.025 (5)	0.007 (4)	−0.008 (5)
N63	0.169 (13)	0.131 (12)	0.140 (11)	0.028 (11)	−0.071 (10)	−0.018 (10)
C61	0.086 (9)	0.100 (11)	0.191 (16)	−0.005 (8)	−0.022 (10)	−0.027 (11)
C62	0.098 (9)	0.074 (8)	0.111 (10)	0.009 (7)	−0.012 (8)	−0.020 (7)

Geometric parameters (Å, º)

O7—C8	1.344 (7)	C37—H37	0.9500
O7—C6	1.445 (7)	C38—C39	1.401 (11)
O13—C14	1.364 (7)	C38—H38	0.9500
O13—C12	1.438 (7)	C39—H39	0.9500
O19—C18	1.418 (7)	C41—C42A	1.548 (6)
O19—H19	0.8400	C41—C42B	1.552 (6)
O27—C17	1.243 (7)	C41—H411	0.9900
O32—C14	1.198 (7)	C41—H412	0.9900
O40—C11	1.235 (7)	C41—H413	0.9900
O45—C8	1.210 (7)	C41—H414	0.9900
O53—C5	1.221 (7)	C42A—C43A	1.552 (6)
O58—C2	1.231 (8)	C42A—H421	0.9900
N1—C2	1.365 (11)	C42A—H422	0.9900
N1—C59A	1.477 (7)	C43A—C44	1.551 (6)
N1—C60B	1.477 (8)	C43A—H431	0.9900
N1—C59B	1.478 (8)	C43A—H432	0.9900
N1—C60A	1.478 (8)	C42B—C43B	1.553 (6)
N4—C5	1.322 (9)	C42B—H423	0.9900
N4—C3	1.475 (8)	C42B—H424	0.9900
N4—H4	0.8800	C43B—C44	1.551 (6)
N10—C11	1.335 (9)	C43B—H433	0.9900
N10—C9	1.478 (8)	C43B—H434	0.9900
N10—H10	0.8800	C44—H441	0.9900
N16—C17	1.317 (8)	C44—H442	0.9900
N16—C15	1.473 (7)	C44—H443	0.9900
N16—H16	0.8800	C44—H444	0.9900
C2—C3	1.514 (11)	C46—C47	1.511 (9)
C3—C57	1.542 (12)	C46—H461	0.9900
C3—C54	1.565 (11)	C46—H462	0.9900
C5—C6	1.542 (9)	C47—C52	1.393 (9)
C6—C46	1.530 (9)	C47—C48	1.402 (8)
C6—H6	1.0000	C48—C49	1.387 (10)
C8—C9	1.528 (8)	C48—H48	0.9500
C9—C44	1.542 (10)	C49—C50	1.374 (10)
C9—C41	1.552 (9)	C49—H49	0.9500
C11—C12	1.520 (9)	C50—C51	1.367 (10)
C12—C33	1.539 (10)	C50—H50	0.9500

C12—H12	1.0000	C51—C52	1.391 (11)
C14—C15	1.527 (8)	C51—H51	0.9500
C15—C31	1.525 (9)	C52—H52	0.9500
C15—C28	1.559 (8)	C54—C55B	1.549 (6)
C17—C18	1.520 (9)	C54—C55A	1.551 (6)
C18—C20	1.524 (10)	C54—H541	0.9900
C18—H18	1.0000	C54—H542	0.9900
C20—C21	1.506 (10)	C54—H543	0.9900
C20—H201	0.9900	C54—H544	0.9900
C20—H202	0.9900	C55A—C56A	1.553 (6)
C21—C22	1.372 (11)	C55A—H551	0.9900
C21—C26	1.396 (11)	C55A—H552	0.9900
C22—C23	1.402 (12)	C56A—C57	1.551 (6)
C22—H22	0.9500	C56A—H561	0.9900
C23—C24	1.363 (14)	C56A—H562	0.9900
C23—H23	0.9500	C55B—C56B	1.551 (6)
C24—C25	1.359 (12)	C55B—H553	0.9900
C24—H24	0.9500	C55B—H554	0.9900
C25—C26	1.371 (12)	C56B—C57	1.550 (6)
C25—H25	0.9500	C56B—H563	0.9900
C26—H26	0.9500	C56B—H564	0.9900
C28—C29	1.543 (9)	C57—H571	0.9900
C28—H281	0.9900	C57—H572	0.9900
C28—H282	0.9900	C57—H573	0.9900
C29—C30	1.537 (11)	C57—H574	0.9900
C29—H291	0.9900	C59A—H591	0.9800
C29—H292	0.9900	C59A—H592	0.9800
C30—C31	1.525 (9)	C59A—H593	0.9800
C30—H301	0.9900	C60A—H601	0.9800
C30—H302	0.9900	C60A—H602	0.9800
C31—H311	0.9900	C60A—H603	0.9800
C31—H312	0.9900	C59B—H594	0.9800
C33—C34	1.506 (9)	C59B—H595	0.9800
C33—H331	0.9900	C59B—H596	0.9800
C33—H332	0.9900	C60B—H604	0.9800
C34—C35	1.380 (10)	C60B—H605	0.9800
C34—C39	1.386 (10)	C60B—H606	0.9800
C35—C36	1.377 (12)	N63—C62	1.160 (16)
C35—H35	0.9500	C61—C62	1.482 (18)
C36—C37	1.426 (15)	C61—H611	0.9800
C36—H36	0.9500	C61—H612	0.9800
C37—C38	1.347 (13)	C61—H613	0.9800

C8—O7—C6	118.9 (4)	C42A—C41—H412	110.3
C14—O13—C12	116.9 (4)	C9—C41—H412	110.3
C18—O19—H19	109.5	H411—C41—H412	108.6
C2—N1—C59A	127.1 (7)	C9—C41—H413	112.0
C2—N1—C60B	93 (3)	C42B—C41—H413	112.0

C2—N1—C59B	105 (4)	C9—C41—H414	112.0
C60B—N1—C59B	123 (5)	C42B—C41—H414	112.0
C2—N1—C60A	119.0 (7)	H413—C41—H414	109.7
C59A—N1—C60A	113.9 (8)	C41—C42A—C43A	106.9 (8)
C5—N4—C3	120.4 (5)	C41—C42A—H421	110.3
C5—N4—H4	119.8	C43A—C42A—H421	110.3
C3—N4—H4	119.8	C41—C42A—H422	110.3
C11—N10—C9	120.2 (5)	C43A—C42A—H422	110.3
C11—N10—H10	119.9	H421—C42A—H422	108.6
C9—N10—H10	119.9	C44—C43A—C42A	104.7 (8)
C17—N16—C15	121.6 (5)	C44—C43A—H431	110.8
C17—N16—H16	119.2	C42A—C43A—H431	110.8
C15—N16—H16	119.2	C44—C43A—H432	110.8
O58—C2—N1	119.1 (7)	C42A—C43A—H432	110.8
O58—C2—C3	119.8 (8)	H431—C43A—H432	108.9
N1—C2—C3	121.1 (6)	C41—C42B—C43B	99.8 (11)
N4—C3—C2	113.9 (6)	C41—C42B—H423	111.8
N4—C3—C57	107.9 (6)	C43B—C42B—H423	111.8
C2—C3—C57	112.1 (6)	C41—C42B—H424	111.8
N4—C3—C54	108.3 (5)	C43B—C42B—H424	111.8
C2—C3—C54	108.2 (6)	H423—C42B—H424	109.5
C57—C3—C54	106.0 (7)	C44—C43B—C42B	99.8 (12)
O53—C5—N4	125.3 (6)	C44—C43B—H433	111.8
O53—C5—C6	118.1 (6)	C42B—C43B—H433	111.8
N4—C5—C6	116.5 (5)	C44—C43B—H434	111.8
O7—C6—C46	104.7 (4)	C42B—C43B—H434	111.8
O7—C6—C5	112.3 (5)	H433—C43B—H434	109.5
C46—C6—C5	110.9 (5)	C9—C44—C43B	106.6 (9)
O7—C6—H6	109.6	C9—C44—C43A	104.9 (7)
C46—C6—H6	109.6	C9—C44—H441	110.8
C5—C6—H6	109.6	C43A—C44—H441	110.8
O45—C8—O7	124.6 (5)	C9—C44—H442	110.8
O45—C8—C9	123.8 (5)	C43A—C44—H442	110.8
O7—C8—C9	111.3 (5)	H441—C44—H442	108.8
N10—C9—C8	109.9 (5)	C9—C44—H443	110.4
N10—C9—C44	109.9 (5)	C43B—C44—H443	110.4
C8—C9—C44	109.4 (6)	C9—C44—H444	110.4
N10—C9—C41	110.5 (6)	C43B—C44—H444	110.4
C8—C9—C41	112.7 (5)	H443—C44—H444	108.6
C44—C9—C41	104.4 (5)	C47—C46—C6	110.6 (5)
O40—C11—N10	123.4 (6)	C47—C46—H461	109.5
O40—C11—C12	118.1 (6)	C6—C46—H461	109.5
N10—C11—C12	118.4 (5)	C47—C46—H462	109.5
O13—C12—C11	113.4 (5)	C6—C46—H462	109.5
O13—C12—C33	108.1 (5)	H461—C46—H462	108.1
C11—C12—C33	111.3 (5)	C52—C47—C48	117.7 (6)
O13—C12—H12	107.9	C52—C47—C46	121.3 (5)
C11—C12—H12	107.9	C48—C47—C46	120.8 (6)

C33—C12—H12	107.9	C49—C48—C47	120.8 (6)
O32—C14—O13	122.5 (6)	C49—C48—H48	119.6
O32—C14—C15	125.3 (6)	C47—C48—H48	119.6
O13—C14—C15	112.1 (5)	C50—C49—C48	120.0 (6)
N16—C15—C31	111.4 (5)	C50—C49—H49	120.0
N16—C15—C14	110.6 (5)	C48—C49—H49	120.0
C31—C15—C14	113.9 (5)	C51—C50—C49	120.4 (7)
N16—C15—C28	109.3 (5)	C51—C50—H50	119.8
C31—C15—C28	104.4 (5)	C49—C50—H50	119.8
C14—C15—C28	106.9 (5)	C50—C51—C52	120.2 (7)
O27—C17—N16	122.9 (6)	C50—C51—H51	119.9
O27—C17—C18	118.6 (5)	C52—C51—H51	119.9
N16—C17—C18	118.5 (5)	C51—C52—C47	120.8 (6)
O19—C18—C17	107.8 (5)	C51—C52—H52	119.6
O19—C18—C20	113.5 (6)	C47—C52—H52	119.6
C17—C18—C20	110.8 (6)	C55B—C54—C3	106.5 (10)
O19—C18—H18	108.2	C55A—C54—C3	101.2 (9)
C17—C18—H18	108.2	C55A—C54—H541	111.5
C20—C18—H18	108.2	C3—C54—H541	111.5
C21—C20—C18	114.0 (6)	C55A—C54—H542	111.5
C21—C20—H201	108.8	C3—C54—H542	111.5
C18—C20—H201	108.8	H541—C54—H542	109.4
C21—C20—H202	108.8	C55B—C54—H543	110.4
C18—C20—H202	108.8	C3—C54—H543	110.4
H201—C20—H202	107.7	C55B—C54—H544	110.4
C22—C21—C26	116.7 (7)	C3—C54—H544	110.4
C22—C21—C20	122.2 (7)	H543—C54—H544	108.6
C26—C21—C20	121.1 (7)	C54—C55A—C56A	100.4 (11)
C21—C22—C23	122.1 (8)	C54—C55A—H551	111.7
C21—C22—H22	118.9	C56A—C55A—H551	111.7
C23—C22—H22	118.9	C54—C55A—H552	111.7
C24—C23—C22	119.6 (8)	C56A—C55A—H552	111.7
C24—C23—H23	120.2	H551—C55A—H552	109.5
C22—C23—H23	120.2	C57—C56A—C55A	95.2 (11)
C25—C24—C23	118.7 (8)	C57—C56A—H561	112.7
C25—C24—H24	120.6	C55A—C56A—H561	112.7
C23—C24—H24	120.6	C57—C56A—H562	112.7
C24—C25—C26	122.2 (9)	C55A—C56A—H562	112.7
C24—C25—H25	118.9	H561—C56A—H562	110.2
C26—C25—H25	118.9	C54—C55B—C56B	107.8 (13)
C25—C26—C21	120.5 (8)	C54—C55B—H553	110.2
C25—C26—H26	119.7	C56B—C55B—H553	110.2
C21—C26—H26	119.7	C54—C55B—H554	110.2
C29—C28—C15	105.3 (5)	C56B—C55B—H554	110.2
C29—C28—H281	110.7	H553—C55B—H554	108.5
C15—C28—H281	110.7	C57—C56B—C55B	107.0 (13)
C29—C28—H282	110.7	C57—C56B—H563	110.3
C15—C28—H282	110.7	C55B—C56B—H563	110.3

H281—C28—H282	108.8	C57—C56B—H564	110.3
C30—C29—C28	106.2 (5)	C55B—C56B—H564	110.3
C30—C29—H291	110.5	H563—C56B—H564	108.6
C28—C29—H291	110.5	C3—C57—C56B	108.3 (9)
C30—C29—H292	110.5	C3—C57—C56A	98.6 (9)
C28—C29—H292	110.5	C3—C57—H571	112.0
H291—C29—H292	108.7	C56A—C57—H571	112.0
C31—C30—C29	104.0 (6)	C3—C57—H572	112.0
C31—C30—H301	111.0	C56A—C57—H572	112.0
C29—C30—H301	111.0	H571—C57—H572	109.7
C31—C30—H302	111.0	C3—C57—H573	110.0
C29—C30—H302	111.0	C56B—C57—H573	110.0
H301—C30—H302	109.0	C3—C57—H574	110.0
C15—C31—C30	102.9 (5)	C56B—C57—H574	110.0
C15—C31—H311	111.2	H573—C57—H574	108.4
C30—C31—H311	111.2	N1—C59A—H591	109.5
C15—C31—H312	111.2	N1—C59A—H592	109.5
C30—C31—H312	111.2	H591—C59A—H592	109.5
H311—C31—H312	109.1	N1—C59A—H593	109.5
C34—C33—C12	111.4 (6)	H591—C59A—H593	109.5
C34—C33—H331	109.3	H592—C59A—H593	109.5
C12—C33—H331	109.3	N1—C60A—H601	109.5
C34—C33—H332	109.3	N1—C60A—H602	109.5
C12—C33—H332	109.3	H601—C60A—H602	109.5
H331—C33—H332	108.0	N1—C60A—H603	109.5
C35—C34—C39	118.9 (7)	H601—C60A—H603	109.5
C35—C34—C33	119.8 (6)	H602—C60A—H603	109.5
C39—C34—C33	121.3 (6)	N1—C59B—H594	109.5
C36—C35—C34	121.5 (8)	N1—C59B—H595	109.5
C36—C35—H35	119.3	H594—C59B—H595	109.5
C34—C35—H35	119.3	N1—C59B—H596	109.5
C35—C36—C37	118.9 (8)	H594—C59B—H596	109.5
C35—C36—H36	120.5	H595—C59B—H596	109.5
C37—C36—H36	120.5	N1—C60B—H604	109.5
C38—C37—C36	119.6 (8)	N1—C60B—H605	109.5
C38—C37—H37	120.2	H604—C60B—H605	109.5
C36—C37—H37	120.2	N1—C60B—H606	109.5
C37—C38—C39	120.9 (9)	H604—C60B—H606	109.5
C37—C38—H38	119.6	H605—C60B—H606	109.5
C39—C38—H38	119.6	C62—C61—H611	109.5
C34—C39—C38	120.1 (7)	C62—C61—H612	109.5
C34—C39—H39	119.9	H611—C61—H612	109.5
C38—C39—H39	119.9	C62—C61—H613	109.5
C42A—C41—C9	107.0 (6)	H611—C61—H613	109.5
C9—C41—C42B	98.9 (9)	H612—C61—H613	109.5
C42A—C41—H411	110.3	N63—C62—C61	175.4 (18)
C9—C41—H411	110.3		

C59A—N1—C2—O58	−175.0 (11)	C22—C21—C26—C25	3.2 (14)
C60B—N1—C2—O58	−38 (3)	C20—C21—C26—C25	−178.4 (9)
C59B—N1—C2—O58	−163 (5)	N16—C15—C28—C29	−97.1 (6)
C60A—N1—C2—O58	3.6 (14)	C31—C15—C28—C29	22.1 (6)
C59A—N1—C2—C3	6.0 (15)	C14—C15—C28—C29	143.1 (5)
C60B—N1—C2—C3	143 (3)	C15—C28—C29—C30	3.1 (7)
C59B—N1—C2—C3	18 (5)	C28—C29—C30—C31	−27.1 (7)
C60A—N1—C2—C3	−175.3 (9)	N16—C15—C31—C30	78.9 (6)
C5—N4—C3—C2	51.6 (8)	C14—C15—C31—C30	−155.2 (5)
C5—N4—C3—C57	−73.6 (8)	C28—C15—C31—C30	−38.9 (6)
C5—N4—C3—C54	172.0 (6)	C29—C30—C31—C15	40.9 (7)
O58—C2—C3—N4	−135.4 (7)	O13—C12—C33—C34	65.6 (7)
N1—C2—C3—N4	43.6 (10)	C11—C12—C33—C34	−59.6 (7)
O58—C2—C3—C57	−12.4 (10)	C12—C33—C34—C35	102.9 (8)
N1—C2—C3—C57	166.5 (8)	C12—C33—C34—C39	−79.7 (8)
O58—C2—C3—C54	104.1 (8)	C39—C34—C35—C36	3.6 (12)
N1—C2—C3—C54	−76.9 (9)	C33—C34—C35—C36	−179.0 (7)
C3—N4—C5—O53	−5.2 (10)	C34—C35—C36—C37	−0.8 (13)
C3—N4—C5—C6	178.9 (6)	C35—C36—C37—C38	−1.7 (14)
C8—O7—C6—C46	146.1 (5)	C36—C37—C38—C39	1.2 (14)
C8—O7—C6—C5	−93.5 (6)	C35—C34—C39—C38	−4.0 (12)
O53—C5—C6—O7	178.3 (5)	C33—C34—C39—C38	178.6 (7)
N4—C5—C6—O7	−5.5 (7)	C37—C38—C39—C34	1.6 (14)
O53—C5—C6—C46	−64.9 (7)	N10—C9—C41—C42A	95.3 (9)
N4—C5—C6—C46	111.3 (6)	C8—C9—C41—C42A	−141.3 (9)
C6—O7—C8—O45	6.5 (10)	C44—C9—C41—C42A	−22.7 (10)
C6—O7—C8—C9	−167.0 (5)	N10—C9—C41—C42B	81.3 (9)
C11—N10—C9—C8	−49.7 (7)	C8—C9—C41—C42B	−155.3 (9)
C11—N10—C9—C44	−170.1 (5)	C44—C9—C41—C42B	−36.7 (9)
C11—N10—C9—C41	75.3 (6)	C9—C41—C42A—C43A	2.3 (15)
O45—C8—C9—N10	151.2 (7)	C41—C42A—C43A—C44	18.8 (17)
O7—C8—C9—N10	−35.2 (8)	C9—C41—C42B—C43B	53.8 (15)
O45—C8—C9—C44	−88.1 (8)	C41—C42B—C43B—C44	−49.5 (19)
O7—C8—C9—C44	85.5 (7)	N10—C9—C44—C43B	−112.0 (11)
O45—C8—C9—C41	27.5 (10)	C8—C9—C44—C43B	127.2 (10)
O7—C8—C9—C41	−158.9 (6)	C41—C9—C44—C43B	6.4 (11)
C9—N10—C11—O40	−4.6 (9)	N10—C9—C44—C43A	−83.9 (8)
C9—N10—C11—C12	179.0 (5)	C8—C9—C44—C43A	155.3 (7)
C14—O13—C12—C11	−77.1 (6)	C41—C9—C44—C43A	34.5 (8)
C14—O13—C12—C33	159.0 (5)	C42B—C43B—C44—C9	26.5 (16)
O40—C11—C12—O13	166.4 (5)	C42A—C43A—C44—C9	−33.1 (14)
N10—C11—C12—O13	−17.0 (8)	O7—C6—C46—C47	−58.2 (6)
O40—C11—C12—C33	−71.4 (7)	C5—C6—C46—C47	−179.6 (5)
N10—C11—C12—C33	105.2 (6)	C6—C46—C47—C52	−75.6 (7)
C12—O13—C14—O32	4.6 (8)	C6—C46—C47—C48	99.4 (7)
C12—O13—C14—C15	−171.3 (5)	C52—C47—C48—C49	−2.1 (9)
C17—N16—C15—C31	78.6 (7)	C46—C47—C48—C49	−177.3 (6)
C17—N16—C15—C14	−49.1 (8)	C47—C48—C49—C50	1.7 (10)

C17—N16—C15—C28	−166.6 (6)	C48—C49—C50—C51	−0.9 (11)
O32—C14—C15—N16	150.1 (6)	C49—C50—C51—C52	0.6 (12)
O13—C14—C15—N16	−34.2 (7)	C50—C51—C52—C47	−1.0 (11)
O32—C14—C15—C31	23.8 (9)	C48—C47—C52—C51	1.8 (9)
O13—C14—C15—C31	−160.5 (5)	C46—C47—C52—C51	176.9 (6)
O32—C14—C15—C28	−91.0 (7)	N4—C3—C54—C55B	94.0 (13)
O13—C14—C15—C28	84.8 (6)	C2—C3—C54—C55B	−142.0 (13)
C15—N16—C17—O27	−0.4 (10)	C57—C3—C54—C55B	−21.6 (14)
C15—N16—C17—C18	179.0 (6)	N4—C3—C54—C55A	117.1 (10)
O27—C17—C18—O19	169.8 (6)	C2—C3—C54—C55A	−118.9 (10)
N16—C17—C18—O19	−9.6 (9)	C57—C3—C54—C55A	1.5 (11)
O27—C17—C18—C20	−65.4 (8)	C3—C54—C55A—C56A	−37.1 (15)
N16—C17—C18—C20	115.2 (7)	C54—C55A—C56A—C57	58.7 (18)
O19—C18—C20—C21	−61.2 (8)	C3—C54—C55B—C56B	17 (3)
C17—C18—C20—C21	177.4 (6)	C54—C55B—C56B—C57	−6 (3)
C18—C20—C21—C22	125.1 (8)	N4—C3—C57—C56B	−97.9 (14)
C18—C20—C21—C26	−53.2 (10)	C2—C3—C57—C56B	135.9 (14)
C26—C21—C22—C23	−2.1 (14)	C54—C3—C57—C56B	18.0 (15)
C20—C21—C22—C23	179.5 (9)	N4—C3—C57—C56A	−81.4 (9)
C21—C22—C23—C24	1.3 (17)	C2—C3—C57—C56A	152.3 (8)
C22—C23—C24—C25	−1.5 (16)	C54—C3—C57—C56A	34.5 (9)
C23—C24—C25—C26	2.7 (17)	C55B—C56B—C57—C3	−8 (3)
C24—C25—C26—C21	−3.7 (17)	C55A—C56A—C57—C3	−56.2 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O19—H19···O45^{i}	0.84	2.11	2.814 (6)	142
N4—H4···O40	0.88	2.19	3.019 (7)	156
N10—H10···O27	0.88	2.07	2.882 (7)	154
N16—H16···O53^{i}	0.88	2.23	3.052 (6)	156

Symmetry code: (i) *x*, *y*, *z*−1.

(*S*)-Pms-Aib-(*S*)-Pms-Aib-NMe$_2$ (5a)

Crystal data

$C_{28}H_{37}N_3O_6$
M_r = 511.60
Monoclinic, $P2_1$
a = 10.388 (4) Å
b = 12.756 (6) Å
c = 21.624 (5) Å
β = 93.55 (3)°
V = 2859.7 (18) Å^3
Z = 4

$F(000)$ = 1096
D_x = 1.188 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 17.5–19.5°
μ = 0.08 mm^{-1}
T = 173 K
Irregular prism, colourless
0.42 × 0.35 × 0.35 mm

Data collection

Rigaku AFC-5R diffractometer
Radiation source: Rigaku RU200 rotating anode generator
Graphite monochromator
ω–2θ scans
7056 measured reflections
6868 independent reflections
4297 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.5°$
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 0$
$l = -28 \rightarrow 0$
3 standard reflections every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.149$
$S = 1.03$
6868 reflections
703 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
$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 1.181P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: No quotients, so Flack parameter determined by classical intensity fit
Absolute structure parameter: 0.0 (14)

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O7	0.6610 (3)	0.5360 (2)	0.12783 (13)	0.0346 (7)
O13	0.6470 (3)	0.1953 (3)	0.19026 (16)	0.0484 (9)
H13	0.684 (5)	0.163 (5)	0.159 (3)	0.063 (18)*
O21	0.4396 (3)	0.4102 (3)	0.14925 (13)	0.0402 (8)
O24	0.5569 (4)	0.6639 (3)	0.17617 (16)	0.0551 (9)
O32	0.5683 (3)	0.6415 (3)	−0.02233 (15)	0.0511 (9)
O35	0.2719 (3)	0.5974 (3)	−0.09069 (15)	0.0562 (10)
N1	0.2739 (4)	0.6597 (4)	0.0063 (2)	0.0492 (11)
N4	0.4842 (4)	0.5082 (3)	0.03249 (18)	0.0375 (9)
H4	0.480 (5)	0.479 (5)	0.070 (3)	0.066 (18)*
N10	0.6122 (4)	0.3962 (4)	0.21722 (18)	0.0402 (9)
H10	0.653 (4)	0.353 (4)	0.238 (2)	0.029 (12)*
C2	0.3049 (4)	0.5873 (4)	−0.0349 (2)	0.0409 (11)
C3	0.3780 (4)	0.4862 (4)	−0.0143 (2)	0.0405 (11)
C5	0.5698 (4)	0.5855 (4)	0.0236 (2)	0.0360 (10)
C6	0.6743 (4)	0.6041 (4)	0.07540 (19)	0.0347 (10)
H6	0.6675	0.6783	0.0898	0.042*

C8	0.6024 (4)	0.5776 (4)	0.1761 (2)	0.0391 (11)
C9	0.6128 (5)	0.5074 (4)	0.2346 (2)	0.0464 (13)
C11	0.5214 (4)	0.3549 (4)	0.1777 (2)	0.0373 (11)
C12	0.5236 (4)	0.2380 (4)	0.1696 (2)	0.0386 (11)
H12	0.5056	0.2208	0.1248	0.046*
C14	0.4218 (5)	0.1856 (4)	0.2073 (2)	0.0512 (14)
H141	0.4289	0.1089	0.2016	0.061*
H142	0.4436	0.2006	0.2516	0.061*
C15	0.2826 (5)	0.2163 (4)	0.1929 (2)	0.0434 (12)
C16	0.2232 (5)	0.2002 (4)	0.1338 (2)	0.0461 (12)
H16	0.2732	0.1776	0.1010	0.055*
C17	0.0921 (5)	0.2170 (5)	0.1228 (3)	0.0562 (14)
H17	0.0533	0.2063	0.0823	0.067*
C18	0.0168 (6)	0.2489 (5)	0.1698 (3)	0.0636 (16)
H18	−0.0737	0.2575	0.1622	0.076*
C19	0.0748 (6)	0.2684 (5)	0.2280 (3)	0.0631 (16)
H19	0.0243	0.2924	0.2603	0.076*
C20	0.2067 (6)	0.2528 (5)	0.2393 (2)	0.0559 (14)
H20	0.2456	0.2672	0.2793	0.067*
C22	0.5005 (7)	0.5328 (5)	0.2745 (2)	0.0713 (18)
H221	0.4189	0.5153	0.2517	0.107*
H222	0.5017	0.6077	0.2847	0.107*
H223	0.5089	0.4917	0.3129	0.107*
C23	0.7432 (6)	0.5333 (5)	0.2670 (3)	0.0704 (19)
H231	0.7529	0.4954	0.3064	0.106*
H232	0.7486	0.6089	0.2748	0.106*
H233	0.8121	0.5123	0.2406	0.106*
C25	0.8093 (4)	0.5879 (4)	0.0527 (2)	0.0433 (11)
H251	0.8151	0.5168	0.0346	0.052*
H252	0.8244	0.6395	0.0197	0.052*
C26	0.9124 (4)	0.6002 (4)	0.1049 (2)	0.0442 (12)
C27	0.9936 (5)	0.5178 (5)	0.1217 (3)	0.0603 (15)
H27	0.9876	0.4546	0.0985	0.072*
C28	1.0826 (6)	0.5253 (5)	0.1710 (3)	0.075 (2)
H28	1.1385	0.4680	0.1811	0.089*
C29	1.0914 (5)	0.6162 (5)	0.2063 (3)	0.0654 (17)
H29	1.1504	0.6204	0.2415	0.078*
C30	1.0133 (5)	0.7005 (5)	0.1895 (3)	0.0576 (15)
H30	1.0203	0.7637	0.2128	0.069*
C31	0.9246 (4)	0.6935 (4)	0.1388 (2)	0.0457 (12)
H31	0.8722	0.7521	0.1271	0.055*
C33	0.2828 (5)	0.4091 (4)	0.0134 (2)	0.0505 (13)
H331	0.3271	0.3429	0.0235	0.076*
H332	0.2104	0.3962	−0.0168	0.076*
H333	0.2506	0.4393	0.0511	0.076*
C34	0.4343 (5)	0.4347 (5)	−0.0701 (2)	0.0573 (15)
H341	0.4911	0.4845	−0.0896	0.086*
H342	0.3641	0.4140	−0.1000	0.086*

H343	0.4838	0.3725	−0.0566	0.086*
C36	0.2952 (5)	0.6555 (5)	0.0734 (2)	0.0537 (14)
H361	0.3070	0.5824	0.0866	0.081*
H362	0.2204	0.6852	0.0927	0.081*
H363	0.3725	0.6959	0.0862	0.081*
C37	0.2188 (6)	0.7596 (5)	−0.0161 (3)	0.0753 (19)
H371	0.2791	0.8166	−0.0049	0.113*
H372	0.1370	0.7720	0.0030	0.113*
H373	0.2036	0.7569	−0.0612	0.113*
O47	0.0451 (3)	0.4527 (2)	0.64513 (13)	0.0356 (7)
O53	0.2356 (3)	0.7840 (3)	0.67259 (14)	0.0415 (8)
H53	0.165 (6)	0.818 (5)	0.664 (3)	0.061 (18)*
O61	0.2545 (3)	0.5468 (3)	0.58551 (13)	0.0369 (7)
O64	0.1722 (3)	0.3097 (3)	0.64854 (19)	0.0578 (10)
O72	−0.1299 (3)	0.3545 (3)	0.50820 (15)	0.0516 (9)
O75	−0.0157 (3)	0.3802 (3)	0.36654 (14)	0.0412 (8)
N41	0.1294 (4)	0.3169 (3)	0.43905 (18)	0.0425 (10)
N44	0.0280 (3)	0.4770 (3)	0.51904 (17)	0.0361 (9)
H44	0.084 (5)	0.512 (4)	0.541 (2)	0.041 (14)*
N50	0.2471 (4)	0.5744 (3)	0.68817 (18)	0.0374 (9)
H50	0.256 (4)	0.619 (4)	0.717 (2)	0.025 (12)*
C42	0.0477 (4)	0.3928 (4)	0.41641 (19)	0.0344 (10)
C43	0.0361 (4)	0.4959 (4)	0.4517 (2)	0.0379 (11)
C45	−0.0539 (4)	0.4068 (4)	0.5411 (2)	0.0398 (11)
C46	−0.0562 (4)	0.3936 (4)	0.6116 (2)	0.0365 (10)
H46	−0.0475	0.3176	0.6225	0.044*
C48	0.1549 (4)	0.3994 (4)	0.6620 (2)	0.0368 (10)
C49	0.2512 (5)	0.4624 (4)	0.7039 (2)	0.0457 (12)
C51	0.2558 (4)	0.6074 (4)	0.62956 (19)	0.0317 (10)
C52	0.2706 (4)	0.7252 (4)	0.6204 (2)	0.0328 (10)
H52	0.2135	0.7469	0.5837	0.039*
C54	0.4098 (4)	0.7512 (4)	0.6074 (2)	0.0405 (11)
H541	0.4165	0.8279	0.6014	0.049*
H542	0.4657	0.7328	0.6446	0.049*
C55	0.4624 (4)	0.6970 (4)	0.5519 (2)	0.0346 (10)
C56	0.4328 (4)	0.7354 (4)	0.4924 (2)	0.0451 (12)
H56	0.3784	0.7949	0.4866	0.054*
C57	0.4824 (5)	0.6872 (5)	0.4417 (2)	0.0556 (15)
H57	0.4626	0.7144	0.4013	0.067*
C58	0.5604 (5)	0.5998 (5)	0.4493 (3)	0.0586 (16)
H58	0.5939	0.5669	0.4143	0.070*
C59	0.5894 (5)	0.5606 (4)	0.5080 (2)	0.0481 (13)
H59	0.6427	0.5005	0.5137	0.058*
C60	0.5404 (4)	0.6094 (4)	0.5582 (2)	0.0434 (12)
H60	0.5609	0.5820	0.5985	0.052*
C62	0.2094 (7)	0.4486 (6)	0.7695 (3)	0.078 (2)
H621	0.2683	0.4873	0.7984	0.118*
H622	0.2115	0.3739	0.7804	0.118*

H623	0.1216	0.4755	0.7721	0.118*
C63	0.3868 (5)	0.4185 (5)	0.6979 (3)	0.0711 (19)
H631	0.4108	0.4261	0.6550	0.107*
H632	0.3882	0.3441	0.7093	0.107*
H633	0.4483	0.4570	0.7256	0.107*
C65	−0.1860 (4)	0.4350 (4)	0.6317 (2)	0.0458 (12)
H651	−0.1899	0.5115	0.6241	0.055*
H652	−0.2562	0.4020	0.6054	0.055*
C66	−0.2097 (4)	0.4148 (4)	0.6988 (2)	0.0455 (12)
C67	−0.1530 (7)	0.4779 (5)	0.7455 (3)	0.0712 (18)
H67	−0.0966	0.5328	0.7349	0.085*
C68	−0.1773 (10)	0.4620 (6)	0.8070 (3)	0.099 (3)
H68	−0.1363	0.5048	0.8383	0.119*
C69	−0.2588 (8)	0.3863 (8)	0.8223 (4)	0.090 (3)
H69	−0.2758	0.3770	0.8646	0.108*
C70	−0.3182 (6)	0.3219 (7)	0.7786 (4)	0.089 (3)
H70	−0.3768	0.2691	0.7899	0.107*
C71	−0.2899 (5)	0.3363 (5)	0.7160 (3)	0.0668 (18)
H71	−0.3273	0.2904	0.6853	0.080*
C73	−0.0861 (5)	0.5530 (5)	0.4267 (2)	0.0612 (16)
H731	−0.1605	0.5061	0.4286	0.092*
H732	−0.0765	0.5738	0.3836	0.092*
H733	−0.0995	0.6155	0.4518	0.092*
C74	0.1521 (5)	0.5662 (4)	0.4413 (2)	0.0526 (14)
H741	0.1400	0.6345	0.4608	0.079*
H742	0.1600	0.5759	0.3967	0.079*
H743	0.2308	0.5333	0.4596	0.079*
C76	0.2228 (5)	0.3228 (5)	0.4917 (2)	0.0546 (14)
H761	0.2431	0.3965	0.5008	0.082*
H762	0.3016	0.2858	0.4820	0.082*
H763	0.1867	0.2903	0.5278	0.082*
C77	0.1273 (6)	0.2154 (4)	0.4068 (3)	0.0592 (15)
H771	0.0542	0.2135	0.3759	0.089*
H772	0.1184	0.1588	0.4369	0.089*
H773	0.2079	0.2062	0.3862	0.089*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.0334 (15)	0.0385 (17)	0.0310 (16)	−0.0017 (14)	−0.0054 (12)	0.0038 (14)
O13	0.0488 (19)	0.061 (2)	0.0342 (18)	0.0094 (18)	−0.0121 (15)	−0.0085 (18)
O21	0.0389 (16)	0.052 (2)	0.0279 (15)	−0.0077 (15)	−0.0080 (13)	0.0125 (15)
O24	0.072 (2)	0.052 (2)	0.042 (2)	0.020 (2)	0.0058 (17)	0.0010 (18)
O32	0.0487 (19)	0.065 (2)	0.0384 (19)	−0.0080 (18)	−0.0073 (15)	0.0198 (18)
O35	0.050 (2)	0.078 (3)	0.0381 (19)	−0.0114 (19)	−0.0166 (15)	0.0151 (19)
N1	0.044 (2)	0.053 (3)	0.048 (3)	0.006 (2)	−0.0153 (19)	0.000 (2)
N4	0.037 (2)	0.046 (2)	0.028 (2)	−0.0034 (18)	−0.0057 (16)	0.0045 (18)
N10	0.045 (2)	0.041 (2)	0.031 (2)	−0.007 (2)	−0.0166 (17)	0.0045 (19)

C2	0.035 (2)	0.055 (3)	0.032 (2)	−0.007 (2)	−0.0063 (19)	0.007 (2)
C3	0.042 (2)	0.055 (3)	0.024 (2)	−0.003 (2)	−0.0050 (18)	−0.002 (2)
C5	0.033 (2)	0.042 (3)	0.032 (2)	−0.002 (2)	−0.0010 (18)	0.003 (2)
C6	0.036 (2)	0.037 (3)	0.031 (2)	−0.004 (2)	−0.0021 (18)	0.006 (2)
C8	0.037 (2)	0.044 (3)	0.035 (2)	−0.004 (2)	−0.0068 (19)	0.003 (2)
C9	0.066 (3)	0.042 (3)	0.029 (2)	−0.009 (2)	−0.013 (2)	0.001 (2)
C11	0.039 (2)	0.047 (3)	0.025 (2)	−0.008 (2)	−0.0026 (19)	0.008 (2)
C12	0.042 (2)	0.047 (3)	0.025 (2)	−0.003 (2)	−0.0095 (18)	0.000 (2)
C14	0.057 (3)	0.051 (3)	0.044 (3)	−0.019 (3)	−0.017 (2)	0.013 (3)
C15	0.049 (3)	0.042 (3)	0.039 (3)	−0.016 (2)	0.001 (2)	0.009 (2)
C16	0.045 (3)	0.053 (3)	0.039 (3)	−0.005 (2)	−0.004 (2)	0.000 (2)
C17	0.048 (3)	0.059 (4)	0.060 (3)	0.001 (3)	−0.005 (3)	0.001 (3)
C18	0.052 (3)	0.064 (4)	0.076 (4)	0.003 (3)	0.011 (3)	0.015 (3)
C19	0.077 (4)	0.055 (4)	0.061 (4)	0.006 (3)	0.031 (3)	0.006 (3)
C20	0.077 (4)	0.055 (4)	0.036 (3)	−0.015 (3)	0.007 (3)	0.004 (3)
C22	0.119 (5)	0.063 (4)	0.034 (3)	−0.001 (4)	0.020 (3)	−0.004 (3)
C23	0.095 (5)	0.046 (3)	0.063 (4)	−0.024 (3)	−0.044 (3)	0.016 (3)
C25	0.036 (2)	0.043 (3)	0.050 (3)	−0.003 (2)	0.000 (2)	0.004 (2)
C26	0.027 (2)	0.044 (3)	0.061 (3)	−0.006 (2)	0.002 (2)	0.004 (3)
C27	0.047 (3)	0.041 (3)	0.091 (4)	0.000 (3)	−0.011 (3)	0.006 (3)
C28	0.054 (3)	0.049 (4)	0.115 (6)	−0.002 (3)	−0.033 (4)	0.024 (4)
C29	0.047 (3)	0.066 (4)	0.080 (4)	−0.021 (3)	−0.020 (3)	0.016 (3)
C30	0.043 (3)	0.053 (3)	0.075 (4)	−0.019 (3)	−0.010 (3)	−0.006 (3)
C31	0.038 (2)	0.045 (3)	0.054 (3)	0.000 (2)	−0.002 (2)	0.003 (3)
C33	0.053 (3)	0.055 (3)	0.041 (3)	−0.010 (3)	−0.014 (2)	0.001 (3)
C34	0.060 (3)	0.072 (4)	0.037 (3)	0.004 (3)	−0.009 (2)	−0.009 (3)
C36	0.047 (3)	0.068 (4)	0.046 (3)	−0.001 (3)	0.001 (2)	−0.009 (3)
C37	0.072 (4)	0.069 (4)	0.082 (5)	0.019 (3)	−0.024 (3)	0.004 (4)
O47	0.0326 (15)	0.0390 (17)	0.0347 (16)	−0.0058 (14)	−0.0030 (13)	−0.0016 (14)
O53	0.0399 (19)	0.051 (2)	0.0321 (18)	0.0102 (17)	−0.0078 (14)	−0.0102 (16)
O61	0.0332 (15)	0.0441 (19)	0.0334 (16)	−0.0119 (14)	0.0016 (12)	−0.0074 (15)
O64	0.044 (2)	0.051 (2)	0.076 (3)	0.0011 (17)	−0.0185 (18)	−0.010 (2)
O72	0.0383 (18)	0.069 (3)	0.046 (2)	−0.0211 (18)	−0.0079 (15)	−0.0071 (19)
O75	0.0403 (17)	0.049 (2)	0.0330 (17)	−0.0070 (15)	−0.0049 (13)	−0.0084 (15)
N41	0.037 (2)	0.050 (3)	0.041 (2)	0.0055 (19)	0.0047 (17)	0.001 (2)
N44	0.0325 (19)	0.042 (2)	0.033 (2)	−0.0037 (18)	−0.0063 (16)	−0.0030 (18)
N50	0.044 (2)	0.041 (2)	0.026 (2)	−0.0081 (19)	−0.0088 (16)	−0.0020 (18)
C42	0.025 (2)	0.049 (3)	0.029 (2)	−0.004 (2)	0.0015 (16)	−0.005 (2)
C43	0.038 (2)	0.040 (3)	0.034 (2)	0.001 (2)	−0.0052 (19)	−0.006 (2)
C45	0.031 (2)	0.053 (3)	0.034 (2)	−0.007 (2)	0.0004 (19)	−0.007 (2)
C46	0.031 (2)	0.043 (3)	0.036 (2)	−0.009 (2)	−0.0027 (17)	0.000 (2)
C48	0.039 (2)	0.041 (3)	0.030 (2)	−0.008 (2)	−0.0042 (18)	0.002 (2)
C49	0.048 (3)	0.048 (3)	0.039 (3)	−0.013 (2)	−0.017 (2)	0.007 (2)
C51	0.0204 (19)	0.044 (3)	0.029 (2)	−0.0033 (18)	−0.0091 (16)	−0.002 (2)
C52	0.029 (2)	0.041 (3)	0.028 (2)	0.0003 (19)	−0.0066 (16)	−0.0020 (19)
C54	0.038 (2)	0.044 (3)	0.039 (3)	−0.009 (2)	−0.004 (2)	−0.003 (2)
C55	0.0224 (19)	0.038 (3)	0.043 (3)	−0.0047 (19)	−0.0016 (18)	0.006 (2)
C56	0.034 (2)	0.052 (3)	0.049 (3)	0.005 (2)	−0.003 (2)	0.008 (2)

C57	0.043 (3)	0.084 (4)	0.039 (3)	0.008 (3)	−0.007 (2)	0.005 (3)
C58	0.040 (3)	0.082 (4)	0.053 (3)	0.005 (3)	−0.003 (2)	−0.018 (3)
C59	0.039 (3)	0.047 (3)	0.058 (3)	0.010 (2)	−0.003 (2)	−0.002 (3)
C60	0.032 (2)	0.046 (3)	0.051 (3)	−0.002 (2)	−0.007 (2)	0.010 (2)
C62	0.119 (5)	0.068 (4)	0.044 (3)	−0.043 (4)	−0.027 (3)	0.009 (3)
C63	0.050 (3)	0.050 (4)	0.108 (5)	−0.004 (3)	−0.040 (3)	0.010 (3)
C65	0.034 (2)	0.059 (3)	0.044 (3)	−0.001 (2)	0.003 (2)	0.014 (3)
C66	0.037 (2)	0.046 (3)	0.054 (3)	0.016 (2)	0.012 (2)	0.019 (3)
C67	0.115 (5)	0.035 (3)	0.067 (4)	0.012 (3)	0.025 (4)	0.000 (3)
C68	0.179 (9)	0.063 (5)	0.060 (4)	0.046 (5)	0.039 (5)	−0.002 (4)
C69	0.085 (5)	0.113 (7)	0.077 (5)	0.051 (5)	0.042 (4)	0.044 (5)
C70	0.034 (3)	0.123 (7)	0.112 (6)	0.013 (4)	0.015 (3)	0.080 (6)
C71	0.031 (2)	0.085 (5)	0.084 (4)	−0.004 (3)	0.000 (3)	0.042 (4)
C73	0.073 (4)	0.067 (4)	0.042 (3)	0.029 (3)	−0.013 (3)	−0.007 (3)
C74	0.070 (3)	0.046 (3)	0.040 (3)	−0.018 (3)	−0.006 (2)	0.006 (2)
C76	0.053 (3)	0.058 (4)	0.051 (3)	0.020 (3)	−0.010 (2)	−0.005 (3)
C77	0.084 (4)	0.036 (3)	0.058 (3)	0.008 (3)	0.005 (3)	−0.001 (3)

Geometric parameters (Å, º)

O7—C8	1.350 (6)	O47—C48	1.358 (5)
O7—C6	1.442 (5)	O47—C46	1.452 (5)
O13—C12	1.438 (5)	O53—C52	1.421 (5)
O13—H13	0.90 (6)	O53—H53	0.86 (6)
O21—C11	1.238 (5)	O61—C51	1.227 (5)
O24—C8	1.198 (6)	O64—C48	1.198 (6)
O32—C5	1.222 (5)	O72—C45	1.226 (5)
O35—C2	1.239 (5)	O75—C42	1.239 (5)
N1—C2	1.336 (7)	N41—C42	1.359 (6)
N1—C36	1.457 (6)	N41—C76	1.451 (6)
N1—C37	1.466 (7)	N41—C77	1.470 (7)
N4—C5	1.350 (6)	N44—C45	1.343 (6)
N4—C3	1.476 (5)	N44—C43	1.483 (6)
N4—H4	0.90 (6)	N44—H44	0.85 (5)
N10—C11	1.341 (6)	N50—C51	1.344 (6)
N10—C9	1.467 (7)	N50—C49	1.468 (7)
N10—H10	0.81 (5)	N50—H50	0.84 (5)
C2—C3	1.549 (7)	C42—C43	1.529 (7)
C3—C34	1.522 (7)	C43—C74	1.531 (7)
C3—C33	1.541 (7)	C43—C73	1.533 (7)
C5—C6	1.530 (6)	C45—C46	1.535 (6)
C6—C25	1.528 (6)	C46—C65	1.536 (6)
C6—H6	1.0000	C46—H46	1.0000
C8—C9	1.547 (7)	C48—C49	1.534 (6)
C9—C23	1.522 (7)	C49—C62	1.521 (8)
C9—C22	1.529 (8)	C49—C63	1.529 (8)
C11—C12	1.502 (7)	C51—C52	1.524 (7)
C12—C14	1.528 (7)	C52—C54	1.527 (6)

C12—H12	1.0000	C52—H52	1.0000
C14—C15	1.512 (7)	C54—C55	1.516 (7)
C14—H141	0.9900	C54—H541	0.9900
C14—H142	0.9900	C54—H542	0.9900
C15—C20	1.394 (8)	C55—C60	1.383 (7)
C15—C16	1.399 (6)	C55—C56	1.393 (7)
C16—C17	1.384 (7)	C56—C57	1.383 (8)
C16—H16	0.9500	C56—H56	0.9500
C17—C18	1.381 (8)	C57—C58	1.381 (8)
C17—H17	0.9500	C57—H57	0.9500
C18—C19	1.384 (9)	C58—C59	1.381 (8)
C18—H18	0.9500	C58—H58	0.9500
C19—C20	1.391 (8)	C59—C60	1.375 (7)
C19—H19	0.9500	C59—H59	0.9500
C20—H20	0.9500	C60—H60	0.9500
C22—H221	0.9800	C62—H621	0.9800
C22—H222	0.9800	C62—H622	0.9800
C22—H223	0.9800	C62—H623	0.9800
C23—H231	0.9800	C63—H631	0.9800
C23—H232	0.9800	C63—H632	0.9800
C23—H233	0.9800	C63—H633	0.9800
C25—C26	1.515 (6)	C65—C66	1.509 (7)
C25—H251	0.9900	C65—H651	0.9900
C25—H252	0.9900	C65—H652	0.9900
C26—C27	1.382 (7)	C66—C71	1.369 (7)
C26—C31	1.399 (7)	C66—C67	1.393 (8)
C27—C28	1.371 (8)	C67—C68	1.384 (9)
C27—H27	0.9500	C67—H67	0.9500
C28—C29	1.388 (9)	C68—C69	1.338 (12)
C28—H28	0.9500	C68—H68	0.9500
C29—C30	1.382 (8)	C69—C70	1.371 (11)
C29—H29	0.9500	C69—H69	0.9500
C30—C31	1.391 (7)	C70—C71	1.413 (9)
C30—H30	0.9500	C70—H70	0.9500
C31—H31	0.9500	C71—H71	0.9500
C33—H331	0.9800	C73—H731	0.9800
C33—H332	0.9800	C73—H732	0.9800
C33—H333	0.9800	C73—H733	0.9800
C34—H341	0.9800	C74—H741	0.9800
C34—H342	0.9800	C74—H742	0.9800
C34—H343	0.9800	C74—H743	0.9800
C36—H361	0.9800	C76—H761	0.9800
C36—H362	0.9800	C76—H762	0.9800
C36—H363	0.9800	C76—H763	0.9800
C37—H371	0.9800	C77—H771	0.9800
C37—H372	0.9800	C77—H772	0.9800
C37—H373	0.9800	C77—H773	0.9800

C8—O7—C6	116.2 (4)	C48—O47—C46	116.4 (4)
C12—O13—H13	111 (4)	C52—O53—H53	110 (4)
C2—N1—C36	127.7 (4)	C42—N41—C76	128.1 (4)
C2—N1—C37	119.1 (4)	C42—N41—C77	117.8 (4)
C36—N1—C37	113.1 (5)	C76—N41—C77	114.1 (4)
C5—N4—C3	120.8 (4)	C45—N44—C43	122.2 (4)
C5—N4—H4	120 (4)	C45—N44—H44	125 (3)
C3—N4—H4	118 (4)	C43—N44—H44	113 (3)
C11—N10—C9	122.3 (4)	C51—N50—C49	121.4 (4)
C11—N10—H10	114 (3)	C51—N50—H50	118 (3)
C9—N10—H10	121 (3)	C49—N50—H50	119 (3)
O35—C2—N1	120.9 (5)	O75—C42—N41	120.5 (4)
O35—C2—C3	117.9 (5)	O75—C42—C43	119.5 (4)
N1—C2—C3	121.1 (4)	N41—C42—C43	120.0 (4)
N4—C3—C34	108.5 (4)	N44—C43—C42	111.2 (4)
N4—C3—C33	109.0 (4)	N44—C43—C74	109.5 (4)
C34—C3—C33	108.7 (4)	C42—C43—C74	110.1 (4)
N4—C3—C2	111.8 (4)	N44—C43—C73	109.2 (4)
C34—C3—C2	109.6 (4)	C42—C43—C73	108.8 (4)
C33—C3—C2	109.2 (4)	C74—C43—C73	108.2 (5)
O32—C5—N4	124.6 (4)	O72—C45—N44	123.8 (4)
O32—C5—C6	118.5 (4)	O72—C45—C46	117.8 (4)
N4—C5—C6	116.9 (4)	N44—C45—C46	118.3 (4)
O7—C6—C25	107.8 (3)	O47—C46—C45	112.6 (4)
O7—C6—C5	112.6 (3)	O47—C46—C65	107.5 (4)
C25—C6—C5	111.6 (4)	C45—C46—C65	108.1 (4)
O7—C6—H6	108.3	O47—C46—H46	109.5
C25—C6—H6	108.3	C45—C46—H46	109.5
C5—C6—H6	108.3	C65—C46—H46	109.5
O24—C8—O7	124.0 (4)	O64—C48—O47	123.3 (4)
O24—C8—C9	122.6 (4)	O64—C48—C49	122.8 (4)
O7—C8—C9	113.1 (4)	O47—C48—C49	113.7 (4)
N10—C9—C23	108.5 (5)	N50—C49—C62	108.8 (5)
N10—C9—C22	111.0 (5)	N50—C49—C63	110.6 (4)
C23—C9—C22	112.5 (5)	C62—C49—C63	110.7 (5)
N10—C9—C8	110.6 (4)	N50—C49—C48	111.4 (4)
C23—C9—C8	105.4 (4)	C62—C49—C48	106.1 (4)
C22—C9—C8	108.8 (4)	C63—C49—C48	109.2 (5)
O21—C11—N10	121.8 (5)	O61—C51—N50	122.4 (4)
O21—C11—C12	121.5 (4)	O61—C51—C52	121.1 (4)
N10—C11—C12	116.7 (4)	N50—C51—C52	116.4 (4)
O13—C12—C11	111.1 (4)	O53—C52—C51	112.6 (4)
O13—C12—C14	107.6 (4)	O53—C52—C54	108.8 (3)
C11—C12—C14	110.9 (4)	C51—C52—C54	110.0 (4)
O13—C12—H12	109.1	O53—C52—H52	108.4
C11—C12—H12	109.1	C51—C52—H52	108.4
C14—C12—H12	109.1	C54—C52—H52	108.4
C15—C14—C12	117.5 (4)	C55—C54—C52	116.0 (4)

C15—C14—H141	107.9	C55—C54—H541	108.3
C12—C14—H141	107.9	C52—C54—H541	108.3
C15—C14—H142	107.9	C55—C54—H542	108.3
C12—C14—H142	107.9	C52—C54—H542	108.3
H141—C14—H142	107.2	H541—C54—H542	107.4
C20—C15—C16	118.0 (5)	C60—C55—C56	118.0 (5)
C20—C15—C14	121.0 (5)	C60—C55—C54	121.8 (4)
C16—C15—C14	120.8 (5)	C56—C55—C54	120.2 (4)
C17—C16—C15	120.5 (5)	C57—C56—C55	120.3 (5)
C17—C16—H16	119.7	C57—C56—H56	119.9
C15—C16—H16	119.7	C55—C56—H56	119.9
C18—C17—C16	121.0 (5)	C58—C57—C56	120.6 (5)
C18—C17—H17	119.5	C58—C57—H57	119.7
C16—C17—H17	119.5	C56—C57—H57	119.7
C17—C18—C19	119.2 (5)	C59—C58—C57	119.6 (5)
C17—C18—H18	120.4	C59—C58—H58	120.2
C19—C18—H18	120.4	C57—C58—H58	120.2
C18—C19—C20	120.2 (5)	C60—C59—C58	119.4 (5)
C18—C19—H19	119.9	C60—C59—H59	120.3
C20—C19—H19	119.9	C58—C59—H59	120.3
C19—C20—C15	121.0 (5)	C59—C60—C55	122.1 (5)
C19—C20—H20	119.5	C59—C60—H60	119.0
C15—C20—H20	119.5	C55—C60—H60	119.0
C9—C22—H221	109.5	C49—C62—H621	109.5
C9—C22—H222	109.5	C49—C62—H622	109.5
H221—C22—H222	109.5	H621—C62—H622	109.5
C9—C22—H223	109.5	C49—C62—H623	109.5
H221—C22—H223	109.5	H621—C62—H623	109.5
H222—C22—H223	109.5	H622—C62—H623	109.5
C9—C23—H231	109.5	C49—C63—H631	109.5
C9—C23—H232	109.5	C49—C63—H632	109.5
H231—C23—H232	109.5	H631—C63—H632	109.5
C9—C23—H233	109.5	C49—C63—H633	109.5
H231—C23—H233	109.5	H631—C63—H633	109.5
H232—C23—H233	109.5	H632—C63—H633	109.5
C26—C25—C6	111.5 (4)	C66—C65—C46	114.3 (4)
C26—C25—H251	109.3	C66—C65—H651	108.7
C6—C25—H251	109.3	C46—C65—H651	108.7
C26—C25—H252	109.3	C66—C65—H652	108.7
C6—C25—H252	109.3	C46—C65—H652	108.7
H251—C25—H252	108.0	H651—C65—H652	107.6
C27—C26—C31	118.4 (5)	C71—C66—C67	117.6 (5)
C27—C26—C25	120.6 (5)	C71—C66—C65	121.7 (5)
C31—C26—C25	121.0 (4)	C67—C66—C65	120.7 (5)
C28—C27—C26	121.5 (6)	C68—C67—C66	121.1 (7)
C28—C27—H27	119.2	C68—C67—H67	119.4
C26—C27—H27	119.2	C66—C67—H67	119.4
C27—C28—C29	120.2 (6)	C69—C68—C67	119.9 (8)

C27—C28—H28	119.9	C69—C68—H68	120.0
C29—C28—H28	119.9	C67—C68—H68	120.0
C30—C29—C28	119.3 (5)	C68—C69—C70	121.8 (7)
C30—C29—H29	120.4	C68—C69—H69	119.1
C28—C29—H29	120.4	C70—C69—H69	119.1
C29—C30—C31	120.4 (6)	C69—C70—C71	118.0 (7)
C29—C30—H30	119.8	C69—C70—H70	121.0
C31—C30—H30	119.8	C71—C70—H70	121.0
C30—C31—C26	120.1 (5)	C66—C71—C70	121.5 (7)
C30—C31—H31	119.9	C66—C71—H71	119.3
C26—C31—H31	119.9	C70—C71—H71	119.3
C3—C33—H331	109.5	C43—C73—H731	109.5
C3—C33—H332	109.5	C43—C73—H732	109.5
H331—C33—H332	109.5	H731—C73—H732	109.5
C3—C33—H333	109.5	C43—C73—H733	109.5
H331—C33—H333	109.5	H731—C73—H733	109.5
H332—C33—H333	109.5	H732—C73—H733	109.5
C3—C34—H341	109.5	C43—C74—H741	109.5
C3—C34—H342	109.5	C43—C74—H742	109.5
H341—C34—H342	109.5	H741—C74—H742	109.5
C3—C34—H343	109.5	C43—C74—H743	109.5
H341—C34—H343	109.5	H741—C74—H743	109.5
H342—C34—H343	109.5	H742—C74—H743	109.5
N1—C36—H361	109.5	N41—C76—H761	109.5
N1—C36—H362	109.5	N41—C76—H762	109.5
H361—C36—H362	109.5	H761—C76—H762	109.5
N1—C36—H363	109.5	N41—C76—H763	109.5
H361—C36—H363	109.5	H761—C76—H763	109.5
H362—C36—H363	109.5	H762—C76—H763	109.5
N1—C37—H371	109.5	N41—C77—H771	109.5
N1—C37—H372	109.5	N41—C77—H772	109.5
H371—C37—H372	109.5	H771—C77—H772	109.5
N1—C37—H373	109.5	N41—C77—H773	109.5
H371—C37—H373	109.5	H771—C77—H773	109.5
H372—C37—H373	109.5	H772—C77—H773	109.5
C36—N1—C2—O35	174.2 (5)	C76—N41—C42—O75	171.2 (5)
C37—N1—C2—O35	−9.9 (7)	C77—N41—C42—O75	−7.7 (6)
C36—N1—C2—C3	−3.2 (7)	C76—N41—C42—C43	−7.2 (7)
C37—N1—C2—C3	172.7 (5)	C77—N41—C42—C43	173.9 (4)
C5—N4—C3—C34	72.2 (6)	C45—N44—C43—C42	−49.1 (5)
C5—N4—C3—C33	−169.6 (4)	C45—N44—C43—C74	−170.9 (4)
C5—N4—C3—C2	−48.8 (6)	C45—N44—C43—C73	70.9 (6)
O35—C2—C3—N4	139.3 (4)	O75—C42—C43—N44	137.3 (4)
N1—C2—C3—N4	−43.2 (6)	N41—C42—C43—N44	−44.3 (5)
O35—C2—C3—C34	19.0 (6)	O75—C42—C43—C74	−101.3 (5)
N1—C2—C3—C34	−163.6 (4)	N41—C42—C43—C74	77.2 (5)
O35—C2—C3—C33	−100.0 (5)	O75—C42—C43—C73	17.1 (6)

N1—C2—C3—C33	77.5 (5)	N41—C42—C43—C73	−164.5 (4)
C3—N4—C5—O32	−0.8 (7)	C43—N44—C45—O72	−1.5 (7)
C3—N4—C5—C6	178.5 (4)	C43—N44—C45—C46	−179.4 (4)
C8—O7—C6—C25	137.9 (4)	C48—O47—C46—C45	−96.0 (5)
C8—O7—C6—C5	−98.6 (4)	C48—O47—C46—C65	145.0 (4)
O32—C5—C6—O7	177.3 (4)	O72—C45—C46—O47	175.1 (4)
N4—C5—C6—O7	−2.1 (6)	N44—C45—C46—O47	−6.9 (6)
O32—C5—C6—C25	−61.4 (6)	O72—C45—C46—C65	−66.2 (6)
N4—C5—C6—C25	119.3 (4)	N44—C45—C46—C65	111.8 (5)
C6—O7—C8—O24	3.8 (6)	C46—O47—C48—O64	2.9 (6)
C6—O7—C8—C9	−169.9 (3)	C46—O47—C48—C49	−173.1 (4)
C11—N10—C9—C23	−166.7 (5)	C51—N50—C49—C62	−167.4 (4)
C11—N10—C9—C22	69.3 (6)	C51—N50—C49—C63	70.7 (5)
C11—N10—C9—C8	−51.5 (6)	C51—N50—C49—C48	−50.9 (6)
O24—C8—C9—N10	153.1 (5)	O64—C48—C49—N50	150.2 (5)
O7—C8—C9—N10	−33.1 (6)	O47—C48—C49—N50	−33.8 (6)
O24—C8—C9—C23	−89.8 (6)	O64—C48—C49—C62	−91.5 (6)
O7—C8—C9—C23	84.0 (5)	O47—C48—C49—C62	84.4 (5)
O24—C8—C9—C22	30.9 (6)	O64—C48—C49—C63	27.8 (7)
O7—C8—C9—C22	−155.2 (4)	O47—C48—C49—C63	−156.2 (4)
C9—N10—C11—O21	6.7 (7)	C49—N50—C51—O61	7.5 (6)
C9—N10—C11—C12	−173.3 (4)	C49—N50—C51—C52	−171.1 (4)
O21—C11—C12—O13	161.2 (4)	O61—C51—C52—O53	164.1 (3)
N10—C11—C12—O13	−18.9 (6)	N50—C51—C52—O53	−17.2 (5)
O21—C11—C12—C14	−79.3 (5)	O61—C51—C52—C54	−74.3 (5)
N10—C11—C12—C14	100.7 (5)	N50—C51—C52—C54	104.4 (4)
O13—C12—C14—C15	−178.7 (4)	O53—C52—C54—C55	−178.6 (4)
C11—C12—C14—C15	59.6 (6)	C51—C52—C54—C55	57.7 (5)
C12—C14—C15—C20	−125.4 (5)	C52—C54—C55—C60	−99.9 (5)
C12—C14—C15—C16	60.2 (7)	C52—C54—C55—C56	80.3 (6)
C20—C15—C16—C17	−2.1 (8)	C60—C55—C56—C57	−0.9 (7)
C14—C15—C16—C17	172.5 (5)	C54—C55—C56—C57	178.9 (5)
C15—C16—C17—C18	−0.6 (9)	C55—C56—C57—C58	0.8 (8)
C16—C17—C18—C19	2.6 (9)	C56—C57—C58—C59	−0.2 (8)
C17—C18—C19—C20	−1.9 (9)	C57—C58—C59—C60	−0.2 (8)
C18—C19—C20—C15	−0.8 (9)	C58—C59—C60—C55	0.1 (7)
C16—C15—C20—C19	2.7 (8)	C56—C55—C60—C59	0.5 (7)
C14—C15—C20—C19	−171.8 (5)	C54—C55—C60—C59	−179.3 (4)
O7—C6—C25—C26	−52.6 (5)	O47—C46—C65—C66	−65.5 (5)
C5—C6—C25—C26	−176.7 (4)	C45—C46—C65—C66	172.6 (4)
C6—C25—C26—C27	119.1 (5)	C46—C65—C66—C71	−102.5 (5)
C6—C25—C26—C31	−58.5 (6)	C46—C65—C66—C67	79.2 (6)
C31—C26—C27—C28	1.4 (9)	C71—C66—C67—C68	−0.3 (9)
C25—C26—C27—C28	−176.3 (6)	C65—C66—C67—C68	178.0 (6)
C26—C27—C28—C29	1.3 (10)	C66—C67—C68—C69	−1.4 (11)
C27—C28—C29—C30	−2.8 (10)	C67—C68—C69—C70	1.1 (11)
C28—C29—C30—C31	1.6 (9)	C68—C69—C70—C71	0.9 (11)
C29—C30—C31—C26	1.0 (8)	C67—C66—C71—C70	2.4 (8)

C27—C26—C31—C30	−2.5 (8)	C65—C66—C71—C70	−175.9 (5)
C25—C26—C31—C30	175.1 (5)	C69—C70—C71—C66	−2.7 (9)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13—H13···O35^{i}	0.90 (6)	1.79 (6)	2.670 (5)	169 (5)
N4—H4···O21	0.90 (6)	1.99 (6)	2.881 (5)	171 (5)
N10—H10···O53^{ii}	0.81 (5)	2.36 (5)	3.124 (5)	157 (4)
O53—H53···O75^{iii}	0.86 (6)	1.83 (6)	2.684 (5)	171 (6)
N44—H44···O61	0.85 (5)	2.01 (5)	2.824 (5)	159 (5)
N50—H50···O13^{iv}	0.84 (5)	2.40 (5)	3.185 (5)	156 (4)

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

(*S*,*R*)-Pms-Acp-(*R*,*S*)-Pms-Acp-NMe$_2$/(*R*,*S*)-Pms-Acp-(*R*,*S*)-Pms-Acp-NMe$_2$ (1:1) (5b)

Crystal data

$C_{32}H_{41}N_3O_6$
M_r = 563.68
Monoclinic, $P2_1/n$
a = 9.937 (5) Å
b = 18.319 (7) Å
c = 16.436 (3) Å
β = 90.19 (3)°
V = 2992 (2) Å^3
Z = 4
$F(000)$ = 1208
D_x = 1.251 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 15–20°
μ = 0.09 mm^{-1}
T = 173 K
Prism, colourless
0.40 × 0.40 × 0.25 mm

Data collection

Rigaku AFC-5R diffractometer
Radiation source: Rigaku RU200 rotating anode generator
Graphite monochromator
ω–2θ scans
7311 measured reflections
6855 independent reflections
4077 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°, θ_{min} = 2.6°
h = −12→12
k = −23→0
l = 0→21
3 standard reflections every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.205
S = 1.04
6855 reflections
509 parameters
540 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2 + 2.67P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

Special details

Experimental. Solvent used: MeCN

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

Refinement. The molecule contains disorder of both benzyl substituents. Two orientations of just the phenyl ring of the central benzyl group and the entire terminal benzyl group, including the stereogenic C-atom, were defined and the site occupation factors of the major sites of these groups refined to 0.507 (7) and 0.532 (4), respectively. Similarity restraints were applied to the chemically equivalent bond lengths and angles involving all disordered C-atoms, while neighbouring atoms within and between each disordered arrangement were restrained to have similar atomic displacement parameters.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O7	0.16539 (19)	0.80427 (10)	0.32978 (11)	0.0355 (5)	
O13	0.4674 (2)	0.99111 (12)	0.27404 (13)	0.0451 (5)	
H131	0.443233	1.034843	0.278838	0.068*	0.532 (4)
H132	0.426640	1.030960	0.268171	0.068*	0.468 (4)
O21	0.2127 (2)	0.88066 (11)	0.17927 (12)	0.0467 (6)	
O26	−0.0453 (2)	0.81105 (12)	0.28006 (14)	0.0473 (5)	
O34	0.1704 (2)	0.61769 (10)	0.26113 (11)	0.0409 (5)	
O39	0.1900 (2)	0.56239 (10)	0.07656 (11)	0.0356 (4)	
N1	0.0170 (2)	0.64103 (13)	0.08740 (14)	0.0384 (6)	
N4	0.2153 (2)	0.72171 (12)	0.19238 (12)	0.0294 (5)	
H4	0.220 (3)	0.7692 (17)	0.1946 (17)	0.032 (8)*	
N10	0.2112 (3)	0.94402 (13)	0.29672 (16)	0.0400 (6)	
H10	0.253 (3)	0.9732 (18)	0.3274 (19)	0.039 (9)*	
C2	0.1490 (3)	0.62417 (14)	0.09297 (15)	0.0294 (6)	
C3	0.2512 (3)	0.68470 (13)	0.11537 (15)	0.0296 (6)	
C5	0.1808 (3)	0.68462 (14)	0.25869 (15)	0.0309 (6)	
C6	0.1545 (3)	0.72606 (15)	0.33766 (16)	0.0343 (6)	
H6	0.062268	0.713672	0.357496	0.041*	
C8	0.0553 (3)	0.84091 (16)	0.30291 (16)	0.0354 (6)	
C9	0.0725 (3)	0.92330 (15)	0.31334 (17)	0.0367 (7)	
C11	0.2708 (3)	0.92252 (15)	0.22782 (18)	0.0410 (7)	
C12A	0.4022 (5)	0.9594 (4)	0.2049 (3)	0.0509 (15)	0.532 (4)
H121	0.378466	1.000607	0.167610	0.061*	0.532 (4)
C14A	0.4994 (7)	0.9125 (4)	0.1604 (4)	0.0604 (12)	0.532 (4)
H141	0.448336	0.881743	0.121602	0.072*	0.532 (4)
H142	0.542936	0.879333	0.200182	0.072*	0.532 (4)
C15A	0.6073 (6)	0.9514 (4)	0.1146 (6)	0.0634 (11)	0.532 (4)
C16A	0.6167 (7)	1.0265 (3)	0.1034 (5)	0.0813 (14)	0.532 (4)
H161	0.548935	1.058190	0.123708	0.098*	0.532 (4)
C17A	0.7284 (7)	1.0548 (4)	0.0613 (5)	0.0858 (15)	0.532 (4)
H171	0.740153	1.106089	0.056578	0.103*	0.532 (4)
C18A	0.8223 (8)	1.0070 (4)	0.0265 (6)	0.0781 (14)	0.532 (4)
H181	0.884415	1.023615	−0.013272	0.094*	0.532 (4)

C19A	0.8217 (7)	0.9346 (4)	0.0520 (6)	0.0725 (13)	0.532 (4)
H191	0.896498	0.904223	0.039793	0.087*	0.532 (4)
C20A	0.7137 (7)	0.9059 (4)	0.0948 (5)	0.0693 (12)	0.532 (4)
H201	0.712865	0.855927	0.110393	0.083*	0.532 (4)
C12B	0.4176 (5)	0.9397 (4)	0.2165 (3)	0.0487 (16)	0.468 (4)
H122	0.469313	0.893283	0.223864	0.058*	0.468 (4)
C14B	0.4420 (5)	0.9665 (4)	0.1310 (3)	0.0460 (12)	0.468 (4)
H143	0.388830	0.935194	0.093744	0.055*	0.468 (4)
H144	0.404411	1.016379	0.126985	0.055*	0.468 (4)
C15B	0.5767 (6)	0.9689 (4)	0.1009 (6)	0.0660 (12)	0.468 (4)
C16B	0.6590 (7)	0.9088 (4)	0.1153 (5)	0.0694 (13)	0.468 (4)
H162	0.623520	0.869335	0.146063	0.083*	0.468 (4)
C17B	0.7906 (7)	0.9033 (5)	0.0870 (6)	0.0759 (15)	0.468 (4)
H172	0.844703	0.862101	0.099806	0.091*	0.468 (4)
C18B	0.8405 (8)	0.9600 (5)	0.0394 (7)	0.0778 (14)	0.468 (4)
H182	0.927583	0.956351	0.016000	0.093*	0.468 (4)
C19B	0.7623 (8)	1.0222 (5)	0.0261 (7)	0.0858 (14)	0.468 (4)
H192	0.798956	1.062754	−0.002102	0.103*	0.468 (4)
C20B	0.6302 (8)	1.0246 (5)	0.0545 (6)	0.0814 (13)	0.468 (4)
H202	0.575631	1.065562	0.041500	0.098*	0.468 (4)
C22	−0.0308 (3)	0.96537 (18)	0.26110 (19)	0.0475 (8)	
H221	0.011713	1.008829	0.236250	0.057*	
H222	−0.066007	0.933767	0.217062	0.057*	
C23	−0.1440 (4)	0.9883 (2)	0.3178 (2)	0.0618 (10)	
H231	−0.143737	1.041876	0.325222	0.074*	
H232	−0.232170	0.973566	0.294845	0.074*	
C24	−0.1194 (3)	0.9502 (2)	0.39842 (19)	0.0543 (9)	
H241	−0.164651	0.902077	0.399811	0.065*	
H242	−0.152437	0.980203	0.444293	0.065*	
C25	0.0324 (3)	0.94154 (16)	0.40240 (17)	0.0380 (7)	
H251	0.058063	0.901449	0.439745	0.046*	
H252	0.075949	0.987242	0.420916	0.046*	
C27	0.2572 (3)	0.70386 (18)	0.40233 (17)	0.0448 (7)	
H271	0.244514	0.735184	0.450780	0.054*	0.507 (7)
H272	0.238786	0.652871	0.418871	0.054*	0.507 (7)
H273	0.235521	0.728621	0.454194	0.054*	0.493 (7)
H274	0.251508	0.650535	0.411495	0.054*	0.493 (7)
C28A	0.4026 (8)	0.7093 (5)	0.3756 (11)	0.0515 (13)	0.507 (7)
C29A	0.4673 (7)	0.7764 (5)	0.3641 (5)	0.0566 (13)	0.507 (7)
H291	0.419217	0.820412	0.373624	0.068*	0.507 (7)
C30A	0.6009 (8)	0.7799 (5)	0.3389 (5)	0.0606 (14)	0.507 (7)
H301	0.644592	0.825742	0.333925	0.073*	0.507 (7)
C31A	0.6693 (7)	0.7165 (6)	0.3214 (5)	0.0608 (15)	0.507 (7)
H311	0.758127	0.719081	0.300425	0.073*	0.507 (7)
C32A	0.6093 (7)	0.6481 (5)	0.3340 (5)	0.0618 (14)	0.507 (7)
H321	0.657915	0.604270	0.324774	0.074*	0.507 (7)
C33A	0.4765 (7)	0.6464 (5)	0.3605 (5)	0.0558 (13)	0.507 (7)
H331	0.434639	0.600379	0.368528	0.067*	0.507 (7)

C28B	0.3974 (8)	0.7234 (5)	0.3775 (12)	0.0516 (13)	0.493 (7)
C29B	0.4428 (7)	0.7955 (5)	0.3809 (5)	0.0531 (13)	0.493 (7)
H292	0.386201	0.832373	0.402721	0.064*	0.493 (7)
C30B	0.5705 (8)	0.8139 (5)	0.3525 (5)	0.0593 (15)	0.493 (7)
H302	0.600799	0.863020	0.354963	0.071*	0.493 (7)
C31B	0.6524 (7)	0.7604 (6)	0.3208 (5)	0.0594 (14)	0.493 (7)
H312	0.738523	0.773295	0.300313	0.071*	0.493 (7)
C32B	0.6110 (7)	0.6873 (6)	0.3181 (5)	0.0621 (13)	0.493 (7)
H322	0.668896	0.650182	0.298137	0.075*	0.493 (7)
C33B	0.4823 (7)	0.6709 (5)	0.3457 (5)	0.0574 (13)	0.493 (7)
H332	0.451674	0.621858	0.342673	0.069*	0.493 (7)
C35	0.3942 (3)	0.65298 (16)	0.12363 (18)	0.0371 (6)	
H351	0.410967	0.635509	0.179758	0.045*	
H352	0.407585	0.611914	0.085349	0.045*	
C36	0.4868 (4)	0.71660 (19)	0.1029 (2)	0.0542 (9)	
H361	0.492480	0.751966	0.148284	0.065*	
H362	0.578488	0.699407	0.089223	0.065*	
C37	0.4171 (4)	0.7498 (2)	0.0291 (2)	0.0567 (9)	
H371	0.441030	0.722834	−0.021011	0.068*	
H372	0.443183	0.801608	0.022384	0.068*	
C38	0.2658 (3)	0.74319 (15)	0.04646 (16)	0.0380 (7)	
H381	0.216728	0.727500	−0.003071	0.046*	
H382	0.228915	0.790689	0.064499	0.046*	
C40	−0.0437 (3)	0.71224 (19)	0.1009 (2)	0.0508 (8)	
H401	0.026906	0.749567	0.102728	0.076*	
H402	−0.092456	0.711921	0.152604	0.076*	
H403	−0.106403	0.723216	0.056394	0.076*	
C41	−0.0762 (3)	0.5825 (2)	0.0640 (2)	0.0566 (9)	
H411	−0.081552	0.579562	0.004510	0.085*	
H412	−0.165696	0.592898	0.086018	0.085*	
H413	−0.043677	0.535888	0.085745	0.085*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.0440 (11)	0.0297 (10)	0.0329 (10)	−0.0095 (9)	0.0073 (8)	−0.0065 (8)
O13	0.0428 (12)	0.0430 (12)	0.0495 (13)	0.0021 (10)	0.0088 (10)	−0.0100 (10)
O21	0.0672 (14)	0.0349 (11)	0.0380 (11)	−0.0159 (10)	0.0186 (10)	−0.0114 (9)
O26	0.0483 (13)	0.0410 (12)	0.0525 (13)	−0.0128 (10)	−0.0001 (10)	−0.0115 (10)
O34	0.0644 (14)	0.0269 (10)	0.0314 (10)	−0.0057 (9)	0.0023 (9)	0.0015 (8)
O39	0.0468 (11)	0.0261 (10)	0.0340 (10)	−0.0026 (8)	0.0044 (8)	−0.0048 (8)
N1	0.0393 (13)	0.0397 (14)	0.0363 (13)	−0.0009 (11)	−0.0034 (10)	−0.0045 (10)
N4	0.0428 (13)	0.0218 (11)	0.0236 (10)	−0.0029 (9)	0.0038 (9)	−0.0026 (8)
N10	0.0493 (15)	0.0314 (13)	0.0394 (13)	−0.0142 (11)	0.0187 (11)	−0.0136 (11)
C2	0.0369 (14)	0.0306 (14)	0.0207 (11)	−0.0019 (11)	0.0018 (10)	−0.0005 (10)
C3	0.0410 (15)	0.0240 (13)	0.0237 (12)	−0.0010 (11)	0.0031 (10)	−0.0018 (10)
C5	0.0375 (14)	0.0297 (14)	0.0253 (12)	−0.0051 (11)	−0.0020 (11)	−0.0013 (10)
C6	0.0461 (16)	0.0305 (14)	0.0263 (13)	−0.0104 (12)	0.0073 (11)	−0.0026 (11)

C8	0.0445 (16)	0.0364 (15)	0.0254 (13)	−0.0081 (13)	0.0120 (11)	−0.0058 (11)
C9	0.0467 (17)	0.0307 (14)	0.0330 (14)	−0.0084 (12)	0.0143 (12)	−0.0078 (11)
C11	0.0535 (18)	0.0282 (14)	0.0415 (16)	−0.0067 (13)	0.0162 (14)	−0.0071 (12)
C12A	0.053 (3)	0.050 (3)	0.049 (2)	−0.010 (2)	0.021 (2)	−0.019 (2)
C14A	0.062 (2)	0.061 (2)	0.059 (2)	−0.009 (2)	0.0259 (19)	−0.013 (2)
C15A	0.058 (2)	0.074 (2)	0.059 (2)	−0.017 (2)	0.022 (2)	−0.015 (2)
C16A	0.072 (3)	0.097 (3)	0.075 (3)	−0.013 (2)	0.026 (2)	−0.006 (3)
C17A	0.076 (3)	0.099 (3)	0.083 (3)	−0.015 (3)	0.025 (3)	0.001 (3)
C18A	0.060 (3)	0.090 (3)	0.084 (3)	−0.013 (3)	0.028 (2)	0.005 (3)
C19A	0.061 (2)	0.084 (3)	0.073 (2)	−0.024 (2)	0.023 (2)	−0.007 (2)
C20A	0.058 (3)	0.083 (2)	0.067 (2)	−0.027 (2)	0.024 (2)	−0.018 (2)
C12B	0.052 (3)	0.050 (3)	0.045 (3)	−0.006 (2)	0.018 (2)	−0.017 (2)
C14B	0.047 (2)	0.049 (2)	0.042 (2)	−0.010 (2)	0.016 (2)	−0.012 (2)
C15B	0.060 (2)	0.078 (2)	0.060 (2)	−0.021 (2)	0.021 (2)	−0.017 (2)
C16B	0.059 (3)	0.082 (3)	0.067 (2)	−0.021 (2)	0.022 (2)	−0.021 (2)
C17B	0.066 (3)	0.089 (3)	0.073 (3)	−0.024 (3)	0.021 (3)	−0.020 (3)
C18B	0.063 (3)	0.092 (3)	0.079 (3)	−0.021 (3)	0.025 (2)	−0.009 (3)
C19B	0.076 (3)	0.097 (3)	0.085 (3)	−0.019 (3)	0.030 (3)	0.000 (2)
C20B	0.072 (2)	0.094 (3)	0.078 (3)	−0.014 (2)	0.024 (2)	−0.003 (2)
C22	0.061 (2)	0.0433 (18)	0.0383 (16)	−0.0010 (15)	0.0120 (15)	−0.0005 (14)
C23	0.047 (2)	0.083 (3)	0.056 (2)	0.0033 (19)	0.0089 (16)	−0.0020 (19)
C24	0.0493 (19)	0.075 (2)	0.0383 (17)	−0.0112 (17)	0.0120 (14)	−0.0142 (16)
C25	0.0463 (17)	0.0365 (15)	0.0314 (14)	−0.0094 (13)	0.0114 (12)	−0.0096 (12)
C27	0.062 (2)	0.0490 (18)	0.0238 (13)	−0.0088 (15)	0.0020 (13)	−0.0005 (13)
C28A	0.052 (2)	0.078 (3)	0.025 (2)	−0.020 (2)	−0.0040 (19)	−0.001 (3)
C29A	0.054 (2)	0.083 (3)	0.033 (2)	−0.020 (2)	−0.0090 (18)	0.001 (2)
C30A	0.057 (2)	0.085 (3)	0.039 (2)	−0.022 (2)	−0.007 (2)	−0.003 (2)
C31A	0.054 (3)	0.085 (4)	0.043 (2)	−0.029 (3)	−0.003 (2)	−0.005 (3)
C32A	0.056 (2)	0.086 (4)	0.043 (2)	−0.024 (3)	0.002 (2)	−0.003 (3)
C33A	0.053 (2)	0.082 (3)	0.033 (2)	−0.021 (2)	0.0032 (19)	−0.003 (2)
C28B	0.051 (2)	0.079 (3)	0.025 (2)	−0.018 (2)	−0.0050 (19)	−0.002 (3)
C29B	0.051 (2)	0.079 (3)	0.029 (2)	−0.019 (2)	−0.009 (2)	−0.001 (2)
C30B	0.055 (3)	0.085 (4)	0.038 (2)	−0.016 (3)	−0.008 (2)	−0.001 (3)
C31B	0.052 (3)	0.086 (4)	0.040 (2)	−0.017 (3)	−0.004 (2)	−0.003 (3)
C32B	0.057 (2)	0.087 (3)	0.043 (2)	−0.018 (3)	0.001 (2)	−0.004 (2)
C33B	0.054 (2)	0.084 (3)	0.035 (2)	−0.016 (2)	−0.0020 (19)	−0.002 (2)
C35	0.0354 (15)	0.0388 (16)	0.0372 (15)	−0.0025 (12)	0.0040 (12)	−0.0031 (12)
C36	0.0478 (19)	0.051 (2)	0.064 (2)	−0.0145 (16)	0.0042 (16)	−0.0016 (17)
C37	0.068 (2)	0.050 (2)	0.052 (2)	−0.0196 (17)	0.0148 (17)	0.0051 (16)
C38	0.0597 (19)	0.0288 (14)	0.0257 (13)	−0.0033 (13)	0.0058 (12)	−0.0003 (11)
C40	0.0469 (18)	0.061 (2)	0.0446 (17)	0.0185 (16)	−0.0067 (14)	−0.0088 (16)
C41	0.0433 (18)	0.065 (2)	0.061 (2)	−0.0105 (17)	−0.0129 (16)	−0.0024 (18)

Geometric parameters (Å, º)

O7—C8	1.356 (4)	C19B—C20B	1.395 (4)
O7—C6	1.443 (3)	C19B—H192	0.9500
O13—C12B	1.423 (5)	C20B—H202	0.9500

O13—C12A	1.429 (5)	C22—C23	1.522 (5)
O13—H131	0.8400	C22—H221	0.9900
O13—H132	0.8400	C22—H222	0.9900
O21—C11	1.247 (4)	C23—C24	1.517 (5)
O26—C8	1.198 (3)	C23—H231	0.9900
O34—C5	1.231 (3)	C23—H232	0.9900
O39—C2	1.233 (3)	C24—C25	1.518 (4)
N1—C2	1.351 (4)	C24—H241	0.9900
N1—C40	1.455 (4)	C24—H242	0.9900
N1—C41	1.468 (4)	C25—H251	0.9900
N4—C5	1.330 (3)	C25—H252	0.9900
N4—C3	1.481 (3)	C27—C28B	1.497 (5)
N4—H4	0.87 (3)	C27—C28A	1.516 (5)
N10—C11	1.339 (4)	C27—H271	0.9900
N10—C9	1.457 (4)	C27—H272	0.9900
N10—H10	0.84 (3)	C27—H273	0.9900
C2—C3	1.547 (4)	C27—H274	0.9900
C3—C35	1.541 (4)	C28A—C33A	1.389 (7)
C3—C38	1.566 (4)	C28A—C29A	1.400 (7)
C5—C6	1.527 (4)	C29A—C30A	1.393 (7)
C6—C27	1.526 (4)	C29A—H291	0.9500
C6—H6	1.0000	C30A—C31A	1.376 (10)
C8—C9	1.529 (4)	C30A—H301	0.9500
C9—C22	1.542 (5)	C31A—C32A	1.403 (10)
C9—C25	1.555 (4)	C31A—H311	0.9500
C11—C12B	1.505 (5)	C32A—C33A	1.391 (7)
C11—C12A	1.519 (5)	C32A—H321	0.9500
C12A—C14A	1.488 (7)	C33A—H331	0.9500
C12A—H121	1.0000	C28B—C33B	1.382 (7)
C14A—C15A	1.494 (5)	C28B—C29B	1.398 (7)
C14A—H141	0.9900	C29B—C30B	1.395 (7)
C14A—H142	0.9900	C29B—H292	0.9500
C15A—C20A	1.386 (4)	C30B—C31B	1.377 (10)
C15A—C16A	1.393 (4)	C30B—H302	0.9500
C16A—C17A	1.408 (4)	C31B—C32B	1.402 (10)
C16A—H161	0.9500	C31B—H312	0.9500
C17A—C18A	1.403 (4)	C32B—C33B	1.391 (7)
C17A—H171	0.9500	C32B—H322	0.9500
C18A—C19A	1.391 (4)	C33B—H332	0.9500
C18A—H181	0.9500	C35—C36	1.525 (4)
C19A—C20A	1.390 (4)	C35—H351	0.9900
C19A—H191	0.9500	C35—H352	0.9900
C20A—H201	0.9500	C36—C37	1.521 (5)
C12B—C14B	1.508 (6)	C36—H361	0.9900
C12B—H122	1.0000	C36—H362	0.9900
C14B—C15B	1.430 (4)	C37—C38	1.536 (5)
C14B—H143	0.9900	C37—H371	0.9900
C14B—H144	0.9900	C37—H372	0.9900

C15B—C20B	1.382 (4)	C38—H381	0.9900
C15B—C16B	1.391 (4)	C38—H382	0.9900
C16B—C17B	1.393 (4)	C40—H401	0.9800
C16B—H162	0.9500	C40—H402	0.9800
C17B—C18B	1.393 (4)	C40—H403	0.9800
C17B—H172	0.9500	C41—H411	0.9800
C18B—C19B	1.395 (4)	C41—H412	0.9800
C18B—H182	0.9500	C41—H413	0.9800

C8—O7—C6	117.4 (2)	C9—C22—H221	110.4
C12A—O13—H131	109.5	C23—C22—H222	110.4
C12B—O13—H132	109.5	C9—C22—H222	110.4
C2—N1—C40	126.7 (2)	H221—C22—H222	108.6
C2—N1—C41	117.6 (2)	C24—C23—C22	106.9 (3)
C40—N1—C41	115.7 (3)	C24—C23—H231	110.3
C5—N4—C3	122.0 (2)	C22—C23—H231	110.3
C5—N4—H4	119.2 (19)	C24—C23—H232	110.3
C3—N4—H4	118.7 (19)	C22—C23—H232	110.3
C11—N10—C9	120.2 (3)	H231—C23—H232	108.6
C11—N10—H10	118 (2)	C23—C24—C25	104.1 (3)
C9—N10—H10	121 (2)	C23—C24—H241	110.9
O39—C2—N1	121.1 (2)	C25—C24—H241	110.9
O39—C2—C3	119.5 (2)	C23—C24—H242	110.9
N1—C2—C3	119.3 (2)	C25—C24—H242	110.9
N4—C3—C35	108.8 (2)	H241—C24—H242	109.0
N4—C3—C2	111.8 (2)	C24—C25—C9	103.9 (2)
C35—C3—C2	110.8 (2)	C24—C25—H251	111.0
N4—C3—C38	109.2 (2)	C9—C25—H251	111.0
C35—C3—C38	103.6 (2)	C24—C25—H252	111.0
C2—C3—C38	112.4 (2)	C9—C25—H252	111.0
O34—C5—N4	123.9 (2)	H251—C25—H252	109.0
O34—C5—C6	116.9 (2)	C28B—C27—C6	111.5 (8)
N4—C5—C6	119.2 (2)	C28A—C27—C6	114.6 (8)
O7—C6—C27	106.1 (2)	C28A—C27—H271	108.6
O7—C6—C5	113.9 (2)	C6—C27—H271	108.6
C27—C6—C5	110.1 (2)	C28A—C27—H272	108.6
O7—C6—H6	108.9	C6—C27—H272	108.6
C27—C6—H6	108.9	H271—C27—H272	107.6
C5—C6—H6	108.9	C28B—C27—H273	109.3
O26—C8—O7	123.2 (3)	C6—C27—H273	109.3
O26—C8—C9	125.4 (3)	C28B—C27—H274	109.3
O7—C8—C9	111.3 (2)	C6—C27—H274	109.3
N10—C9—C8	110.0 (2)	H273—C27—H274	108.0
N10—C9—C22	113.2 (2)	C33A—C28A—C29A	117.5 (5)
C8—C9—C22	110.9 (3)	C33A—C28A—C27	120.1 (6)
N10—C9—C25	111.5 (2)	C29A—C28A—C27	122.4 (6)
C8—C9—C25	106.8 (2)	C30A—C29A—C28A	121.2 (6)
C22—C9—C25	104.2 (2)	C30A—C29A—H291	119.4

O21—C11—N10	121.2 (3)	C28A—C29A—H291	119.4
O21—C11—C12B	119.7 (3)	C31A—C30A—C29A	119.7 (7)
N10—C11—C12B	118.3 (3)	C31A—C30A—H301	120.1
O21—C11—C12A	120.8 (3)	C29A—C30A—H301	120.1
N10—C11—C12A	117.5 (3)	C30A—C31A—C32A	120.8 (5)
O13—C12A—C14A	109.4 (5)	C30A—C31A—H311	119.6
O13—C12A—C11	111.8 (3)	C32A—C31A—H311	119.6
C14A—C12A—C11	115.1 (5)	C33A—C32A—C31A	118.1 (7)
O13—C12A—H121	106.7	C33A—C32A—H321	121.0
C14A—C12A—H121	106.7	C31A—C32A—H321	121.0
C11—C12A—H121	106.7	C28A—C33A—C32A	122.7 (6)
C12A—C14A—C15A	116.2 (5)	C28A—C33A—H331	118.7
C12A—C14A—H141	108.2	C32A—C33A—H331	118.7
C15A—C14A—H141	108.2	C33B—C28B—C29B	118.4 (5)
C12A—C14A—H142	108.2	C33B—C28B—C27	120.5 (6)
C15A—C14A—H142	108.2	C29B—C28B—C27	121.1 (6)
H141—C14A—H142	107.4	C30B—C29B—C28B	120.6 (6)
C20A—C15A—C16A	120.8 (5)	C30B—C29B—H292	119.7
C20A—C15A—C14A	112.4 (5)	C28B—C29B—H292	119.7
C16A—C15A—C14A	125.9 (5)	C31B—C30B—C29B	119.7 (7)
C15A—C16A—C17A	118.8 (4)	C31B—C30B—H302	120.2
C15A—C16A—H161	120.6	C29B—C30B—H302	120.2
C17A—C16A—H161	120.6	C30B—C31B—C32B	121.2 (6)
C18A—C17A—C16A	119.8 (5)	C30B—C31B—H312	119.4
C18A—C17A—H171	120.1	C32B—C31B—H312	119.4
C16A—C17A—H171	120.1	C33B—C32B—C31B	117.8 (7)
C19A—C18A—C17A	117.9 (6)	C33B—C32B—H322	121.1
C19A—C18A—H181	121.0	C31B—C32B—H322	121.1
C17A—C18A—H181	121.0	C28B—C33B—C32B	122.4 (6)
C20A—C19A—C18A	121.2 (6)	C28B—C33B—H332	118.8
C20A—C19A—H191	119.4	C32B—C33B—H332	118.8
C18A—C19A—H191	119.4	C36—C35—C3	104.4 (3)
C15A—C20A—C19A	118.8 (5)	C36—C35—H351	110.9
C15A—C20A—H201	120.6	C3—C35—H351	110.9
C19A—C20A—H201	120.6	C36—C35—H352	110.9
O13—C12B—C11	113.0 (4)	C3—C35—H352	110.9
O13—C12B—C14B	110.3 (5)	H351—C35—H352	108.9
C11—C12B—C14B	110.0 (5)	C37—C36—C35	102.1 (3)
O13—C12B—H122	107.8	C37—C36—H361	111.3
C11—C12B—H122	107.8	C35—C36—H361	111.3
C14B—C12B—H122	107.8	C37—C36—H362	111.3
C15B—C14B—C12B	119.1 (5)	C35—C36—H362	111.3
C15B—C14B—H143	107.5	H361—C36—H362	109.2
C12B—C14B—H143	107.5	C36—C37—C38	105.3 (3)
C15B—C14B—H144	107.5	C36—C37—H371	110.7
C12B—C14B—H144	107.5	C38—C37—H371	110.7
H143—C14B—H144	107.0	C36—C37—H372	110.7
C20B—C15B—C16B	116.9 (5)	C38—C37—H372	110.7

C20B—C15B—C14B	125.2 (6)	H371—C37—H372	108.8
C16B—C15B—C14B	117.8 (4)	C37—C38—C3	106.3 (2)
C15B—C16B—C17B	123.5 (6)	C37—C38—H381	110.5
C15B—C16B—H162	118.3	C3—C38—H381	110.5
C17B—C16B—H162	118.3	C37—C38—H382	110.5
C18B—C17B—C16B	118.1 (6)	C3—C38—H382	110.5
C18B—C17B—H172	121.0	H381—C38—H382	108.7
C16B—C17B—H172	121.0	N1—C40—H401	109.5
C17B—C18B—C19B	119.8 (6)	N1—C40—H402	109.5
C17B—C18B—H182	120.1	H401—C40—H402	109.5
C19B—C18B—H182	120.1	N1—C40—H403	109.5
C20B—C19B—C18B	119.9 (6)	H401—C40—H403	109.5
C20B—C19B—H192	120.1	H402—C40—H403	109.5
C18B—C19B—H192	120.1	N1—C41—H411	109.5
C15B—C20B—C19B	121.7 (6)	N1—C41—H412	109.5
C15B—C20B—H202	119.2	H411—C41—H412	109.5
C19B—C20B—H202	119.2	N1—C41—H413	109.5
C23—C22—C9	106.8 (3)	H411—C41—H413	109.5
C23—C22—H221	110.4	H412—C41—H413	109.5

C40—N1—C2—O39	−176.7 (3)	O13—C12B—C14B—C15B	69.8 (8)
C41—N1—C2—O39	2.1 (4)	C11—C12B—C14B—C15B	−164.8 (6)
C40—N1—C2—C3	−0.3 (4)	C12B—C14B—C15B—C20B	−138.8 (10)
C41—N1—C2—C3	178.5 (2)	C12B—C14B—C15B—C16B	44.9 (12)
C5—N4—C3—C35	−75.9 (3)	C20B—C15B—C16B—C17B	1.4 (15)
C5—N4—C3—C2	46.8 (3)	C14B—C15B—C16B—C17B	177.9 (9)
C5—N4—C3—C38	171.8 (2)	C15B—C16B—C17B—C18B	−2.3 (15)
O39—C2—C3—N4	−128.7 (2)	C16B—C17B—C18B—C19B	4.4 (16)
N1—C2—C3—N4	54.9 (3)	C17B—C18B—C19B—C20B	−5.7 (18)
O39—C2—C3—C35	−7.1 (3)	C16B—C15B—C20B—C19B	−2.6 (16)
N1—C2—C3—C35	176.5 (2)	C14B—C15B—C20B—C19B	−178.8 (10)
O39—C2—C3—C38	108.2 (3)	C18B—C19B—C20B—C15B	4.8 (18)
N1—C2—C3—C38	−68.2 (3)	N10—C9—C22—C23	−135.8 (3)
C3—N4—C5—O34	−3.1 (4)	C8—C9—C22—C23	100.0 (3)
C3—N4—C5—C6	175.7 (2)	C25—C9—C22—C23	−14.6 (3)
C8—O7—C6—C27	−154.2 (2)	C9—C22—C23—C24	−8.8 (4)
C8—O7—C6—C5	84.6 (3)	C22—C23—C24—C25	29.3 (4)
O34—C5—C6—O7	−178.6 (2)	C23—C24—C25—C9	−38.1 (3)
N4—C5—C6—O7	2.5 (4)	N10—C9—C25—C24	154.9 (3)
O34—C5—C6—C27	62.4 (3)	C8—C9—C25—C24	−85.0 (3)
N4—C5—C6—C27	−116.5 (3)	C22—C9—C25—C24	32.5 (3)
C6—O7—C8—O26	−5.7 (4)	O7—C6—C27—C28B	−61.0 (5)
C6—O7—C8—C9	169.0 (2)	C5—C6—C27—C28B	62.6 (5)
C11—N10—C9—C8	51.3 (4)	O7—C6—C27—C28A	−71.4 (5)
C11—N10—C9—C22	−73.4 (3)	C5—C6—C27—C28A	52.2 (5)
C11—N10—C9—C25	169.6 (3)	C6—C27—C28A—C33A	−107.8 (14)
O26—C8—C9—N10	−148.1 (3)	C6—C27—C28A—C29A	71.4 (15)
O7—C8—C9—N10	37.3 (3)	C33A—C28A—C29A—C30A	−0.4 (19)

O26—C8—C9—C22	−22.2 (4)	C27—C28A—C29A—C30A	−179.6 (11)
O7—C8—C9—C22	163.3 (2)	C28A—C29A—C30A—C31A	3.0 (14)
O26—C8—C9—C25	90.7 (3)	C29A—C30A—C31A—C32A	−4.6 (12)
O7—C8—C9—C25	−83.8 (3)	C30A—C31A—C32A—C33A	3.5 (11)
C9—N10—C11—O21	−4.8 (5)	C29A—C28A—C33A—C32A	−1 (2)
C9—N10—C11—C12B	−174.8 (4)	C27—C28A—C33A—C32A	178.5 (10)
C9—N10—C11—C12A	166.4 (4)	C31A—C32A—C33A—C28A	−0.8 (14)
O21—C11—C12A—O13	−166.6 (4)	C6—C27—C28B—C33B	−100.3 (14)
N10—C11—C12A—O13	22.1 (8)	C6—C27—C28B—C29B	75.6 (15)
O21—C11—C12A—C14A	−40.9 (8)	C33B—C28B—C29B—C30B	0 (2)
N10—C11—C12A—C14A	147.8 (5)	C27—C28B—C29B—C30B	−175.7 (11)
O13—C12A—C14A—C15A	−71.9 (8)	C28B—C29B—C30B—C31B	0.0 (14)
C11—C12A—C14A—C15A	161.2 (7)	C29B—C30B—C31B—C32B	−1.4 (12)
C12A—C14A—C15A—C20A	162.7 (7)	C30B—C31B—C32B—C33B	2.4 (12)
C12A—C14A—C15A—C16A	−6.7 (13)	C29B—C28B—C33B—C32B	1 (2)
C20A—C15A—C16A—C17A	8.7 (15)	C27—C28B—C33B—C32B	176.7 (11)
C14A—C15A—C16A—C17A	177.3 (9)	C31B—C32B—C33B—C28B	−2.1 (15)
C15A—C16A—C17A—C18A	4.7 (14)	N4—C3—C35—C36	−84.3 (3)
C16A—C17A—C18A—C19A	−16.3 (14)	C2—C3—C35—C36	152.4 (2)
C17A—C18A—C19A—C20A	15.1 (15)	C38—C3—C35—C36	31.7 (3)
C16A—C15A—C20A—C19A	−10.0 (14)	C3—C35—C36—C37	−43.1 (3)
C14A—C15A—C20A—C19A	180.0 (8)	C35—C36—C37—C38	37.4 (3)
C18A—C19A—C20A—C15A	−2.1 (14)	C36—C37—C38—C3	−17.8 (3)
O21—C11—C12B—O13	176.8 (4)	N4—C3—C38—C37	107.3 (3)
N10—C11—C12B—O13	−13.0 (8)	C35—C3—C38—C37	−8.5 (3)
O21—C11—C12B—C14B	53.1 (7)	C2—C3—C38—C37	−128.1 (3)
N10—C11—C12B—C14B	−136.8 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13—H131···O34^{i}	0.84	2.00	2.753 (3)	149
O13—H132···O34^{i}	0.84	1.92	2.753 (3)	172
N4—H4···O21	0.87 (3)	2.06 (3)	2.920 (3)	170 (3)
N10—H10···O39^{i}	0.84 (3)	2.34 (3)	3.161 (3)	164 (3)

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

(*R*,*S*)-Mns-Acp-(*S*,*R*)-Mns-Acp-NMe$_2$ (5c)

Crystal data

$C_{30}H_{37}N_3O_6$
M_r = 535.62
Orthorhombic, $Pna2_1$
a = 18.922 (5) Å
b = 18.300 (3) Å
c = 8.072 (6) Å
V = 2795 (2) Å^3
Z = 4
F(000) = 1144

D_x = 1.273 Mg m^{-3}
Melting point: 372 K
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 23 reflections
θ = 18–20°
μ = 0.09 mm^{-1}
T = 173 K
Prism, colourless
0.30 × 0.23 × 0.20 mm

Data collection

Rigaku AFC-5R diffractometer
Radiation source: Rigaku RU200 rotating anode generator
Graphite monochromator
ω–2θ scans
4363 measured reflections
3889 independent reflections
2106 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.8°$
$h = -1 \rightarrow 24$
$k = 0 \rightarrow 23$
$l = -10 \rightarrow 1$
3 standard reflections every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.161$
$S = 1.01$
3889 reflections
394 parameters
98 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0586P)^2 + 1.4867P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Absolute structure: Flack *x* determined using 231 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −3.2 (10)

Special details

Experimental. Solvent used: dichloromethane/diethyl ether/hexane

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

Refinement. One of the five-membered rings exhibits significant conformational disorder and two positions were refined for three of the ring-atoms. The site occupation factor of the major conformation of the ring refined to 0.658 (12). Similarity restraints were applied to the chemically equivalent bond lengths involving all disordered C atoms, while neighbouring atoms within and between each conformation of the disordered components were restrained to have similar atomic displacement parameters.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O7	0.27446 (16)	0.65935 (17)	0.3454 (5)	0.0299 (8)	
O13	0.3236 (2)	0.4369 (2)	0.6595 (7)	0.0529 (12)	
H13	0.308 (4)	0.403 (3)	0.725 (8)	0.08 (3)*	
O20	0.33321 (19)	0.62869 (18)	0.6795 (6)	0.0381 (9)	
O25	0.36813 (19)	0.7228 (2)	0.2567 (7)	0.0532 (13)	
O32	0.21090 (18)	0.84238 (19)	0.4054 (6)	0.0419 (10)	
O37	0.2006 (2)	0.9229 (2)	0.7711 (6)	0.0499 (11)	
N1	0.1471 (2)	0.8140 (2)	0.7832 (7)	0.0463 (13)	
N4	0.2682 (2)	0.7657 (2)	0.5800 (6)	0.0311 (10)	
H4	0.292 (2)	0.7272 (18)	0.592 (8)	0.035 (16)*	
N10	0.3518 (2)	0.5493 (2)	0.4666 (7)	0.0347 (11)	
H10	0.347 (3)	0.5065 (16)	0.426 (7)	0.036 (15)*	
C2	0.2041 (3)	0.8557 (3)	0.7550 (8)	0.0385 (14)	
C3	0.2765 (3)	0.8207 (3)	0.7111 (7)	0.0345 (13)	

C5	0.2375 (3)	0.7814 (3)	0.4359 (7)	0.0313 (12)	
C6	0.2312 (2)	0.7213 (3)	0.3053 (7)	0.0313 (12)	
H6	0.2462	0.7413	0.1956	0.038*	
C8	0.3444 (2)	0.6681 (3)	0.3171 (7)	0.0330 (13)	
C9	0.3889 (3)	0.6022 (3)	0.3607 (7)	0.0364 (13)	
C11	0.3308 (3)	0.5661 (3)	0.6214 (8)	0.0326 (13)	
C12	0.3037 (3)	0.5028 (3)	0.7310 (9)	0.0453 (17)	
H12	0.3279	0.5066	0.8408	0.054*	
C14	0.2250 (3)	0.5116 (3)	0.7606 (8)	0.0335 (13)	
C15	0.1775 (4)	0.4822 (4)	0.6556 (11)	0.064 (2)	
H15	0.1937	0.4564	0.5607	0.077*	
C16	0.1048 (5)	0.4890 (5)	0.6834 (14)	0.087 (3)	
H16	0.0714	0.4680	0.6096	0.104*	
C17	0.0832 (4)	0.5272 (4)	0.8217 (16)	0.090 (4)	
H17	0.0340	0.5334	0.8398	0.108*	
C18	0.1298 (5)	0.5571 (4)	0.9357 (15)	0.082 (3)	
H18	0.1137	0.5818	1.0322	0.099*	
C19	0.2010 (4)	0.5490 (3)	0.9010 (10)	0.0585 (19)	
H19	0.2347	0.5695	0.9749	0.070*	
C21	0.4590 (2)	0.6277 (3)	0.4484 (9)	0.0472 (17)	
H211	0.4714	0.6789	0.4214	0.057*	0.342 (12)
H212	0.4572	0.6211	0.5701	0.057*	0.342 (12)
H213	0.4619	0.6817	0.4441	0.057*	0.658 (12)
H214	0.4575	0.6130	0.5664	0.057*	0.658 (12)
C22A	0.5083 (10)	0.5727 (10)	0.364 (2)	0.059 (2)	0.342 (12)
H221	0.5577	0.5903	0.3662	0.071*	0.342 (12)
H222	0.5060	0.5247	0.4207	0.071*	0.342 (12)
C23A	0.4812 (8)	0.5667 (12)	0.186 (3)	0.060 (2)	0.342 (12)
H231	0.4978	0.5205	0.1359	0.072*	0.342 (12)
H232	0.4995	0.6079	0.1194	0.072*	0.342 (12)
C24A	0.4003 (9)	0.5685 (17)	0.1894 (19)	0.058 (2)	0.342 (12)
H241	0.3808	0.5997	0.1002	0.070*	0.342 (12)
H242	0.3797	0.5189	0.1814	0.070*	0.342 (12)
C22B	0.5250 (4)	0.5954 (6)	0.3684 (17)	0.060 (2)	0.658 (12)
H223	0.5587	0.5772	0.4527	0.072*	0.658 (12)
H224	0.5491	0.6314	0.2964	0.072*	0.658 (12)
C23B	0.4934 (4)	0.5328 (5)	0.2677 (17)	0.0573 (19)	0.658 (12)
H233	0.4875	0.4885	0.3367	0.069*	0.658 (12)
H234	0.5236	0.5208	0.1713	0.069*	0.658 (12)
C24B	0.4218 (5)	0.5629 (8)	0.2120 (12)	0.057 (2)	0.658 (12)
H243	0.3908	0.5225	0.1749	0.068*	0.658 (12)
H244	0.4281	0.5974	0.1187	0.068*	0.658 (12)
C26	0.1547 (2)	0.6970 (3)	0.2940 (7)	0.0334 (12)	
C27	0.1078 (3)	0.7397 (3)	0.2043 (9)	0.0446 (16)	
H27	0.1246	0.7817	0.1473	0.054*	
C28	0.0370 (3)	0.7219 (3)	0.1972 (10)	0.0518 (18)	
H28	0.0055	0.7513	0.1344	0.062*	
C29	0.0121 (3)	0.6626 (4)	0.2794 (10)	0.0565 (19)	

H29	−0.0367	0.6505	0.2739	0.068*
C30	0.0578 (3)	0.6197 (4)	0.3712 (10)	0.066 (2)
H30	0.0403	0.5786	0.4302	0.079*
C31	0.1294 (3)	0.6370 (4)	0.3772 (10)	0.0547 (18)
H31	0.1608	0.6071	0.4390	0.066*
C33	0.3102 (3)	0.7855 (3)	0.8668 (8)	0.0452 (15)
H331	0.2950	0.8116	0.9681	0.054*
H332	0.2960	0.7336	0.8763	0.054*
C34	0.3901 (3)	0.7917 (4)	0.8443 (11)	0.070 (2)
H341	0.4116	0.7428	0.8305	0.085*
H342	0.4119	0.8158	0.9415	0.085*
C35	0.4008 (3)	0.8379 (4)	0.6881 (11)	0.066 (2)
H351	0.4402	0.8728	0.7035	0.079*
H352	0.4115	0.8063	0.5918	0.079*
C36	0.3313 (3)	0.8784 (3)	0.6619 (9)	0.0426 (14)
H361	0.3255	0.8934	0.5448	0.051*
H362	0.3282	0.9221	0.7339	0.051*
C38	0.1422 (4)	0.7336 (3)	0.7716 (10)	0.061 (2)
H381	0.1272	0.7198	0.6597	0.092*
H382	0.1077	0.7157	0.8524	0.092*
H383	0.1885	0.7120	0.7950	0.092*
C39	0.0805 (3)	0.8526 (4)	0.8168 (12)	0.072 (2)
H391	0.0829	0.8757	0.9262	0.107*
H392	0.0412	0.8176	0.8146	0.107*
H393	0.0729	0.8901	0.7321	0.107*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.0282 (17)	0.0290 (17)	0.033 (2)	−0.0035 (15)	−0.0015 (17)	0.0000 (17)
O13	0.063 (3)	0.036 (2)	0.059 (3)	0.010 (2)	0.027 (3)	0.014 (2)
O20	0.050 (2)	0.0253 (18)	0.039 (2)	0.0087 (16)	−0.001 (2)	0.0017 (19)
O25	0.035 (2)	0.046 (2)	0.078 (4)	−0.0047 (17)	−0.001 (2)	0.032 (3)
O32	0.050 (2)	0.032 (2)	0.044 (3)	0.0000 (17)	−0.016 (2)	0.010 (2)
O37	0.061 (2)	0.030 (2)	0.058 (3)	0.0079 (17)	0.005 (2)	−0.002 (2)
N1	0.048 (3)	0.041 (3)	0.050 (3)	0.003 (2)	0.007 (3)	0.005 (3)
N4	0.038 (2)	0.026 (2)	0.029 (3)	0.004 (2)	−0.006 (2)	0.002 (2)
N10	0.041 (3)	0.020 (2)	0.043 (3)	−0.002 (2)	0.008 (2)	0.001 (2)
C2	0.047 (3)	0.028 (3)	0.040 (4)	0.005 (2)	0.000 (3)	0.003 (3)
C3	0.041 (3)	0.031 (3)	0.032 (3)	0.003 (2)	−0.003 (3)	0.001 (2)
C5	0.029 (3)	0.032 (3)	0.032 (3)	−0.005 (2)	0.001 (3)	0.011 (3)
C6	0.028 (2)	0.038 (3)	0.028 (3)	−0.003 (2)	−0.001 (2)	0.005 (3)
C8	0.026 (3)	0.039 (3)	0.034 (3)	−0.004 (2)	−0.004 (3)	0.004 (3)
C9	0.034 (3)	0.031 (3)	0.044 (4)	0.001 (2)	0.014 (3)	0.003 (3)
C11	0.027 (3)	0.035 (3)	0.036 (3)	0.008 (2)	0.008 (3)	0.011 (3)
C12	0.057 (3)	0.027 (3)	0.053 (5)	0.007 (3)	0.019 (3)	0.009 (3)
C14	0.045 (3)	0.022 (2)	0.034 (3)	0.003 (2)	0.005 (3)	0.008 (3)
C15	0.069 (5)	0.060 (4)	0.062 (5)	−0.007 (4)	0.009 (4)	0.015 (4)

C16	0.086 (6)	0.083 (6)	0.092 (8)	−0.018 (5)	−0.021 (6)	0.036 (6)
C17	0.065 (5)	0.058 (5)	0.146 (11)	0.014 (4)	0.047 (7)	0.049 (6)
C18	0.086 (6)	0.036 (4)	0.125 (9)	0.007 (4)	0.061 (6)	0.005 (5)
C19	0.070 (5)	0.043 (4)	0.063 (5)	−0.005 (3)	0.023 (4)	0.007 (4)
C21	0.030 (3)	0.043 (3)	0.068 (5)	0.003 (2)	0.006 (3)	0.016 (4)
C22A	0.039 (4)	0.052 (5)	0.086 (5)	0.002 (4)	0.022 (4)	−0.001 (4)
C23A	0.044 (4)	0.051 (4)	0.084 (5)	0.001 (4)	0.024 (4)	−0.003 (4)
C24A	0.046 (5)	0.049 (4)	0.080 (5)	0.000 (4)	0.025 (4)	−0.004 (4)
C22B	0.040 (4)	0.051 (4)	0.087 (5)	0.000 (3)	0.023 (4)	0.000 (4)
C23B	0.041 (3)	0.049 (4)	0.082 (5)	0.003 (3)	0.023 (4)	−0.005 (4)
C24B	0.044 (4)	0.049 (4)	0.077 (5)	0.000 (4)	0.022 (4)	−0.006 (4)
C26	0.027 (3)	0.048 (3)	0.025 (3)	−0.004 (2)	−0.001 (2)	−0.005 (3)
C27	0.033 (3)	0.044 (3)	0.057 (4)	0.005 (3)	−0.006 (3)	−0.016 (3)
C28	0.037 (3)	0.049 (4)	0.069 (5)	0.008 (3)	−0.014 (4)	−0.023 (4)
C29	0.030 (3)	0.080 (5)	0.059 (5)	−0.002 (3)	−0.002 (3)	−0.028 (4)
C30	0.051 (4)	0.083 (5)	0.064 (5)	−0.027 (4)	0.000 (4)	0.012 (5)
C31	0.038 (3)	0.071 (4)	0.055 (5)	−0.014 (3)	−0.004 (3)	0.020 (4)
C33	0.057 (4)	0.048 (3)	0.031 (3)	0.016 (3)	−0.012 (3)	−0.001 (3)
C34	0.052 (4)	0.097 (6)	0.062 (5)	0.013 (4)	−0.022 (4)	−0.005 (5)
C35	0.046 (4)	0.085 (5)	0.066 (5)	−0.012 (4)	−0.006 (4)	−0.006 (5)
C36	0.045 (3)	0.039 (3)	0.043 (4)	−0.009 (3)	−0.007 (3)	−0.008 (3)
C38	0.078 (5)	0.043 (4)	0.062 (5)	−0.018 (3)	0.025 (4)	−0.010 (4)
C39	0.041 (3)	0.074 (5)	0.099 (7)	0.008 (3)	0.012 (4)	0.005 (5)

Geometric parameters (Å, º)

O7—C8	1.352 (6)	C21—H214	0.9900
O7—C6	1.436 (6)	C22A—C23A	1.529 (6)
O13—C12	1.389 (7)	C22A—H221	0.9900
O13—H13	0.86 (2)	C22A—H222	0.9900
O20—C11	1.238 (6)	C23A—C24A	1.533 (6)
O25—C8	1.200 (6)	C23A—H231	0.9900
O32—C5	1.248 (6)	C23A—H232	0.9900
O37—C2	1.238 (6)	C24A—H241	0.9900
N1—C2	1.341 (7)	C24A—H242	0.9900
N1—C39	1.470 (7)	C22B—C23B	1.526 (6)
N1—C38	1.477 (7)	C22B—H223	0.9900
N4—C5	1.331 (7)	C22B—H224	0.9900
N4—C3	1.468 (7)	C23B—C24B	1.529 (6)
N4—H4	0.84 (2)	C23B—H233	0.9900
N10—C11	1.347 (8)	C23B—H234	0.9900
N10—C9	1.469 (7)	C24B—H243	0.9900
N10—H10	0.85 (2)	C24B—H244	0.9900
C2—C3	1.554 (8)	C26—C31	1.374 (8)
C3—C36	1.533 (7)	C26—C27	1.387 (8)
C3—C33	1.549 (8)	C27—C28	1.381 (8)
C5—C6	1.528 (7)	C27—H27	0.9500
C6—C26	1.516 (7)	C28—C29	1.356 (9)

C6—H6	1.0000	C28—H28	0.9500
C8—C9	1.512 (7)	C29—C30	1.381 (10)
C9—C24A	1.529 (6)	C29—H29	0.9500
C9—C24B	1.532 (6)	C30—C31	1.392 (8)
C9—C21	1.575 (8)	C30—H30	0.9500
C11—C12	1.545 (7)	C31—H31	0.9500
C12—C14	1.516 (7)	C33—C34	1.528 (9)
C12—H12	1.0000	C33—H331	0.9900
C14—C15	1.347 (9)	C33—H332	0.9900
C14—C19	1.401 (9)	C34—C35	1.531 (11)
C15—C16	1.400 (11)	C34—H341	0.9900
C15—H15	0.9500	C34—H342	0.9900
C16—C17	1.379 (13)	C35—C36	1.524 (8)
C16—H16	0.9500	C35—H351	0.9900
C17—C18	1.387 (15)	C35—H352	0.9900
C17—H17	0.9500	C36—H361	0.9900
C18—C19	1.384 (9)	C36—H362	0.9900
C18—H18	0.9500	C38—H381	0.9800
C19—H19	0.9500	C38—H382	0.9800
C21—C22B	1.525 (6)	C38—H383	0.9800
C21—C22A	1.530 (6)	C39—H391	0.9800
C21—H211	0.9900	C39—H392	0.9800
C21—H212	0.9900	C39—H393	0.9800
C21—H213	0.9900		

C8—O7—C6	115.3 (4)	H221—C22A—H222	108.8
C12—O13—H13	106 (5)	C22A—C23A—C24A	108.5 (15)
C2—N1—C39	116.6 (5)	C22A—C23A—H231	110.0
C2—N1—C38	127.4 (5)	C24A—C23A—H231	110.0
C39—N1—C38	115.8 (5)	C22A—C23A—H232	110.0
C5—N4—C3	121.9 (4)	C24A—C23A—H232	110.0
C5—N4—H4	121 (4)	H231—C23A—H232	108.4
C3—N4—H4	116 (4)	C9—C24A—C23A	99.4 (11)
C11—N10—C9	122.0 (4)	C9—C24A—H241	111.9
C11—N10—H10	122 (4)	C23A—C24A—H241	111.9
C9—N10—H10	116 (4)	C9—C24A—H242	111.9
O37—C2—N1	120.3 (5)	C23A—C24A—H242	111.9
O37—C2—C3	118.8 (5)	H241—C24A—H242	109.6
N1—C2—C3	120.8 (4)	C21—C22B—C23B	101.3 (6)
N4—C3—C36	111.0 (5)	C21—C22B—H223	111.5
N4—C3—C33	110.2 (4)	C23B—C22B—H223	111.5
C36—C3—C33	102.6 (4)	C21—C22B—H224	111.5
N4—C3—C2	110.6 (4)	C23B—C22B—H224	111.5
C36—C3—C2	111.8 (4)	H223—C22B—H224	109.3
C33—C3—C2	110.5 (5)	C22B—C23B—C24B	103.6 (8)
O32—C5—N4	122.8 (5)	C22B—C23B—H233	111.1
O32—C5—C6	118.4 (5)	C24B—C23B—H233	111.1
N4—C5—C6	118.8 (4)	C22B—C23B—H234	111.1

O7—C6—C26	109.0 (4)	C24B—C23B—H234	111.1
O7—C6—C5	111.6 (4)	H233—C23B—H234	109.0
C26—C6—C5	109.1 (4)	C23B—C24B—C9	107.4 (7)
O7—C6—H6	109.0	C23B—C24B—H243	110.2
C26—C6—H6	109.0	C9—C24B—H243	110.2
C5—C6—H6	109.0	C23B—C24B—H244	110.2
O25—C8—O7	122.2 (5)	C9—C24B—H244	110.2
O25—C8—C9	123.5 (4)	H243—C24B—H244	108.5
O7—C8—C9	114.3 (4)	C31—C26—C27	118.8 (5)
N10—C9—C8	113.2 (4)	C31—C26—C6	122.6 (5)
N10—C9—C24A	109.1 (12)	C27—C26—C6	118.4 (5)
C8—C9—C24A	100.9 (12)	C28—C27—C26	120.6 (6)
N10—C9—C24B	110.0 (7)	C28—C27—H27	119.7
C8—C9—C24B	114.7 (7)	C26—C27—H27	119.7
N10—C9—C21	109.7 (5)	C29—C28—C27	120.4 (6)
C8—C9—C21	109.7 (4)	C29—C28—H28	119.8
C24A—C9—C21	114.0 (8)	C27—C28—H28	119.8
C24B—C9—C21	98.5 (5)	C28—C29—C30	119.9 (6)
O20—C11—N10	123.5 (5)	C28—C29—H29	120.0
O20—C11—C12	119.3 (5)	C30—C29—H29	120.0
N10—C11—C12	117.3 (5)	C29—C30—C31	119.9 (7)
O13—C12—C14	115.1 (5)	C29—C30—H30	120.0
O13—C12—C11	108.8 (5)	C31—C30—H30	120.0
C14—C12—C11	109.7 (4)	C26—C31—C30	120.3 (6)
O13—C12—H12	107.7	C26—C31—H31	119.9
C14—C12—H12	107.7	C30—C31—H31	119.9
C11—C12—H12	107.7	C34—C33—C3	106.3 (6)
C15—C14—C19	119.2 (6)	C34—C33—H331	110.5
C15—C14—C12	120.9 (6)	C3—C33—H331	110.5
C19—C14—C12	119.9 (6)	C34—C33—H332	110.5
C14—C15—C16	121.3 (9)	C3—C33—H332	110.5
C14—C15—H15	119.3	H331—C33—H332	108.7
C16—C15—H15	119.3	C33—C34—C35	105.6 (5)
C17—C16—C15	117.7 (9)	C33—C34—H341	110.6
C17—C16—H16	121.1	C35—C34—H341	110.6
C15—C16—H16	121.1	C33—C34—H342	110.6
C16—C17—C18	123.3 (8)	C35—C34—H342	110.6
C16—C17—H17	118.3	H341—C34—H342	108.7
C18—C17—H17	118.3	C36—C35—C34	105.6 (6)
C19—C18—C17	116.2 (9)	C36—C35—H351	110.6
C19—C18—H18	121.9	C34—C35—H351	110.6
C17—C18—H18	121.9	C36—C35—H352	110.6
C18—C19—C14	122.2 (8)	C34—C35—H352	110.6
C18—C19—H19	118.9	H351—C35—H352	108.8
C14—C19—H19	118.9	C35—C36—C3	102.3 (5)
C22B—C21—C9	112.6 (6)	C35—C36—H361	111.3
C22A—C21—C9	96.8 (10)	C3—C36—H361	111.3
C22A—C21—H211	112.4	C35—C36—H362	111.3

C9—C21—H211	112.4	C3—C36—H362	111.3
C22A—C21—H212	112.4	H361—C36—H362	109.2
C9—C21—H212	112.4	N1—C38—H381	109.5
H211—C21—H212	110.0	N1—C38—H382	109.5
C22B—C21—H213	109.1	H381—C38—H382	109.5
C9—C21—H213	109.1	N1—C38—H383	109.5
C22B—C21—H214	109.1	H381—C38—H383	109.5
C9—C21—H214	109.1	H382—C38—H383	109.5
H213—C21—H214	107.8	N1—C39—H391	109.5
C23A—C22A—C21	105.1 (13)	N1—C39—H392	109.5
C23A—C22A—H221	110.7	H391—C39—H392	109.5
C21—C22A—H221	110.7	N1—C39—H393	109.5
C23A—C22A—H222	110.7	H391—C39—H393	109.5
C21—C22A—H222	110.7	H392—C39—H393	109.5

C39—N1—C2—O37	5.9 (10)	C12—C14—C15—C16	178.9 (6)
C38—N1—C2—O37	−179.3 (7)	C14—C15—C16—C17	0.4 (11)
C39—N1—C2—C3	−177.5 (6)	C15—C16—C17—C18	−1.9 (13)
C38—N1—C2—C3	−2.7 (10)	C16—C17—C18—C19	2.2 (12)
C5—N4—C3—C36	−70.1 (6)	C17—C18—C19—C14	−1.2 (10)
C5—N4—C3—C33	177.0 (5)	C15—C14—C19—C18	−0.2 (9)
C5—N4—C3—C2	54.5 (6)	C12—C14—C19—C18	−178.5 (6)
O37—C2—C3—N4	−135.0 (6)	N10—C9—C21—C22B	106.9 (7)
N1—C2—C3—N4	48.3 (7)	C8—C9—C21—C22B	−128.2 (7)
O37—C2—C3—C36	−10.8 (8)	C24B—C9—C21—C22B	−8.0 (10)
N1—C2—C3—C36	172.5 (6)	N10—C9—C21—C22A	94.7 (10)
O37—C2—C3—C33	102.7 (6)	C8—C9—C21—C22A	−140.4 (10)
N1—C2—C3—C33	−73.9 (7)	C24A—C9—C21—C22A	−28.1 (17)
C3—N4—C5—O32	−3.5 (7)	C9—C21—C22A—C23A	37.9 (17)
C3—N4—C5—C6	179.7 (5)	C21—C22A—C23A—C24A	−39 (3)
C8—O7—C6—C26	163.3 (5)	N10—C9—C24A—C23A	−116.8 (17)
C8—O7—C6—C5	−76.1 (5)	C8—C9—C24A—C23A	123.8 (17)
O32—C5—C6—O7	169.9 (4)	C21—C9—C24A—C23A	6 (2)
N4—C5—C6—O7	−13.2 (6)	C22A—C23A—C24A—C9	19 (3)
O32—C5—C6—C26	−69.6 (6)	C9—C21—C22B—C23B	−16.6 (12)
N4—C5—C6—C26	107.4 (5)	C21—C22B—C23B—C24B	34.5 (13)
C6—O7—C8—O25	−2.8 (8)	C22B—C23B—C24B—C9	−42.9 (14)
C6—O7—C8—C9	179.8 (4)	N10—C9—C24B—C23B	−84.5 (10)
C11—N10—C9—C8	−64.2 (7)	C8—C9—C24B—C23B	146.5 (8)
C11—N10—C9—C24A	−175.7 (9)	C21—C9—C24B—C23B	30.1 (10)
C11—N10—C9—C24B	166.0 (6)	O7—C6—C26—C31	25.7 (8)
C11—N10—C9—C21	58.7 (6)	C5—C6—C26—C31	−96.4 (6)
O25—C8—C9—N10	166.8 (6)	O7—C6—C26—C27	−158.4 (5)
O7—C8—C9—N10	−15.9 (7)	C5—C6—C26—C27	79.4 (6)
O25—C8—C9—C24A	−76.7 (11)	C31—C26—C27—C28	−0.7 (9)
O7—C8—C9—C24A	100.6 (10)	C6—C26—C27—C28	−176.7 (6)
O25—C8—C9—C24B	−65.8 (9)	C26—C27—C28—C29	0.7 (10)
O7—C8—C9—C24B	111.5 (7)	C27—C28—C29—C30	0.1 (10)

O25—C8—C9—C21	43.9 (8)	C28—C29—C30—C31	−0.9 (11)
O7—C8—C9—C21	−138.7 (5)	C27—C26—C31—C30	−0.1 (10)
C9—N10—C11—O20	8.9 (8)	C6—C26—C31—C30	175.8 (6)
C9—N10—C11—C12	−170.0 (5)	C29—C30—C31—C26	0.9 (12)
O20—C11—C12—O13	−163.6 (5)	N4—C3—C33—C34	88.4 (6)
N10—C11—C12—O13	15.3 (7)	C36—C3—C33—C34	−29.7 (6)
O20—C11—C12—C14	69.7 (7)	C2—C3—C33—C34	−149.0 (5)
N10—C11—C12—C14	−111.4 (6)	C3—C33—C34—C35	6.5 (7)
O13—C12—C14—C15	−34.0 (8)	C33—C34—C35—C36	19.6 (7)
C11—C12—C14—C15	89.1 (6)	C34—C35—C36—C3	−38.2 (7)
O13—C12—C14—C19	144.3 (5)	N4—C3—C36—C35	−76.3 (6)
C11—C12—C14—C19	−92.6 (7)	C33—C3—C36—C35	41.3 (6)
C19—C14—C15—C16	0.6 (10)	C2—C3—C36—C35	159.7 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13—H13···O32^{i}	0.86 (2)	1.87 (3)	2.713 (6)	166 (8)
N4—H4···O20	0.84 (2)	2.09 (3)	2.907 (5)	164 (6)
N10—H10···O37^{ii}	0.85 (2)	2.17 (3)	2.970 (6)	155 (5)

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