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Crystal structure of L-leucyl-L-isoleucine 2,2,2-trifluoroethanol monosolvate

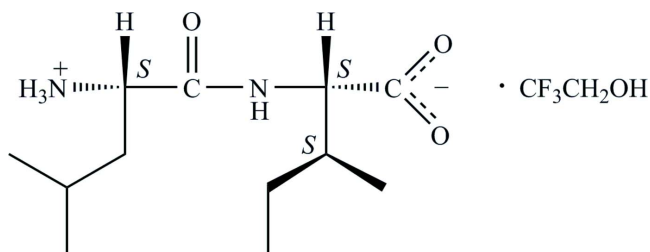
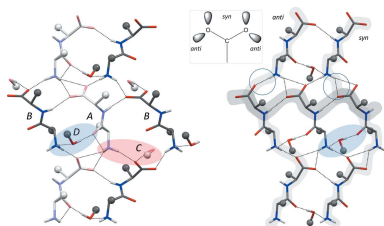
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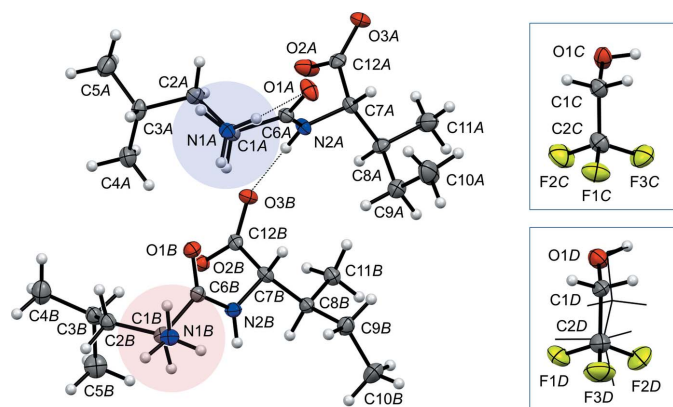
Hydrophobic dipeptides with either L-Leu or L-Phe constitute a rather heterogeneous group of crystal structures. Some form materials with large water-filled channels, but there is also a pronounced tendency to incorporate organic solvent molecules, which then act as acceptors for one of the three H atoms of the charged N-terminal amino group. L-Leu-L-Ile has uniquely been obtained as two distinct hydrates, but has so far failed to co-crystallize with a simple alcohol. The present structure of $C_{12}H_{24}N_2O_3 \cdot CF_3CH_2OH$, which crystallizes with two dipeptide and two solvent molecules in the asymmetric unit, demonstrates that when 2,2,2-trifluoroethanol is used as a solvent, its high capacity as a hydrogen-bond donor leads to formation of an alcohol solvate.

1. Chemical context

Dipeptides with at least one hydrophobic residue (*i.e.* lacking a functional group) such as Val, Leu, Ile and Phe have a high propensity to form crystal structures that are divided into hydrophobic and hydrophilic layers (Görbitz, 2010). The latter include two $C(8)$ head-to-tail chains with two of the three N-terminal amino H atoms acting as donors and the C-terminal carboxylate group as acceptor, and also a $C(4)$ or $C(5)$ chain using the peptide $>N-H$ group as donor and, respectively, the peptide carbonyl group or the carboxylate group as acceptor. The third amino H atom finds an acceptor in a polar side chain or, when both residues are hydrophobic, in a co-crystallized solvent molecule. L-Leu-L-Val has thus been obtained as a series of alcohol solvates (Görbitz & Torgersen, 1999), but also as a non-layered hydrate (Görbitz & Gundersen, 1996). The same is true for L-Leu-L-Leu (Görbitz, 1998, 2001). L-Leu-L-Ile (LI) has, on the other hand, been obtained as two distinct hydrates; a 0.75 hydrate (Görbitz, 2004; CSD refcode ETIWIN) that is isostructural to the Leu-Val analogue (Görbitz & Gundersen, 1996), and a 2.5 hydrate with extensive water channels (Görbitz & Rise, 2008; CSD refcode HIZCOJ). Crystallization using methanol, ethanol or 2-propanol as precipitating agents did not result in formations of alcohol solvates.



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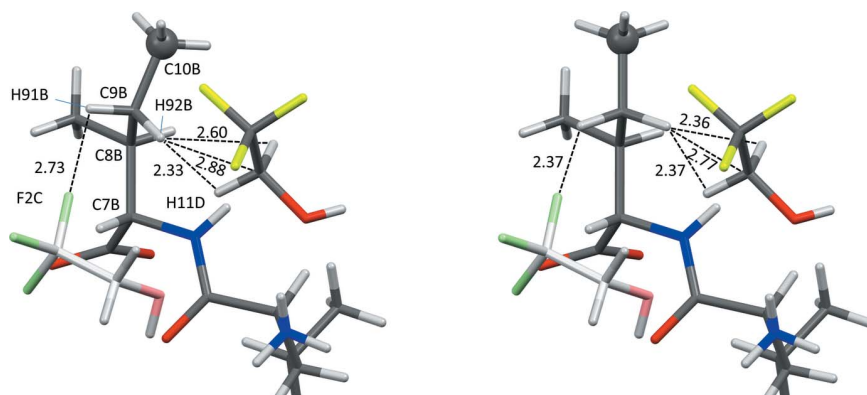

Figure 1

The asymmetric unit of (I), solvent molecules being shown in different positions relative to the peptide molecules than they have in the unit cell to avoid extensive overlap. The minor disorder orientation for TFE molecule *D* is shown in wireframe representation. The amino group of molecule *A* has an unusual eclipsed conformation (blue shade) resulting from formation of an intramolecular hydrogen bond to O1A, while a normal staggered conformation (red shade) is observed for molecule *B*. Thermal displacement ellipsoids are shown at the 50% probability level.

Recently we have become interested in the use of fluorinated alcohols like 2,2,2-trifluoroethanol (TFE) and 1,1,1,3,3,3-hexafluoro-2-propanol during crystallization, not only due to their superior abilities to dissolve a large range of organic molecules (abandoning the use of water if that is desirable), but also as crystal engineering tools to manipulate hydrogen-bonding patterns in solid-state structures by being incorporated into the crystal lattice by virtue of their strong hydrogen-bond-donating capacity. The crystal structure of the LI TFE solvate (I) presented here provides an example of how this can take place.

2. Structural commentary

The four molecules (two dipeptides and two solvent species) in the asymmetric unit are shown in Fig. 1. The structure is well behaved with normal bond lengths and bond angles. Disorder for TFE molecule *D* was easily resolved (see *Refinement*


Figure 2

In the experimental crystal structure of (I) (left) the ethyl group of the Ile residue of molecule *B* is rotated to relieve a short distance between H91B and F2C. If the C7B–C8B–C9B–C10B torsion angle had been exactly 180°, this distance would have been too short (right). The terminal methyl group, with C10B as a sphere, is not involved in any short contacts.

Table 1
Selected torsion angles (°).

Torsion angle	Name	Molecule <i>A</i> /Molecule <i>B</i>	Conformation <i>A</i> / <i>B</i>
N1–C1–C6–N2	ψ_1	162.6 (3)/117.8 (3)	–/–
C1–C6–N2–C7	ω_1	168.6 (3)/173.1 (3)	–/–
C6–N2–C7–C12	φ_2	–99.6 (4)/–65.5 (4)	–/–
N2–C7–C12–O2	ψ_T	–52.8 (4)/–41.1 (4)	–/–
N1–C1–C2–C3	χ_1^1	–69.5 (4)/177.8 (3)	<i>gauche</i> –/ <i>trans</i>
C1–C2–C3–C4	$\chi_1^{2,1}$	–68.2 (4)/–168.3 (3)	<i>gauche</i> –/ <i>trans</i>
C1–C2–C3–C5	$\chi_1^{2,2}$	170.1 (3)/69.1 (4)	<i>trans</i> / <i>gauche</i> +
N2–C7–C8–C9	$\chi_2^{1,1}$	–60.9 (4)/–72.7 (3)	<i>gauche</i> –/ <i>gauche</i> –
N2–C7–C8–C11	$\chi_2^{1,2}$	173.8 (3)/161.1 (3)	<i>trans</i> / <i>trans</i>
C7–C8–C9–C10	χ_2^2	–59.5 (4)/155.5 (3)	<i>gauche</i> –/ <i>trans</i>

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N1A–H1A...O3A ⁱ	0.91	2.13	2.928 (4)	146
N1A–H2A...O1A	0.91	2.07	2.607 (4)	116
N1A–H3A...O2B ⁱⁱ	0.91	1.87	2.767 (4)	168
N2A–H4A...O3B	0.88	2.00	2.883 (4)	177
N1B–H1B...O2A ⁱⁱ	0.91	1.79	2.695 (4)	179
N1B–H2B...O3B ⁱⁱ	0.91	1.89	2.721 (4)	151
N1B–H3B...O1D	0.91	1.98	2.838 (5)	156
O1D–H1D...O1A ⁱⁱⁱ	0.86 (3)	1.87 (4)	2.695 (4)	159 (5)
O1C–H1C...O2B ⁱⁱ	0.86 (3)	1.85 (3)	2.693 (4)	167 (4)

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

details). The molecular conformations of the two peptide molecules are quite different in terms of the side-chain conformations, Table 1. The overall molecular conformation of molecule *B* is very close to that of molecule *B* in the 2.5 hydrate (Görbitz & Rise, 2008). A substantial 24.5° deviation from the idealized *trans* orientation at 180° for χ_2^2 of molecule *B* is needed to relieve a short contact between H91B and F2C, Fig. 2.

3. Supramolecular features

The unit cell and crystal-packing arrangement is illustrated in Fig. 3a), hydrogen-bond parameters are listed in Table 2.

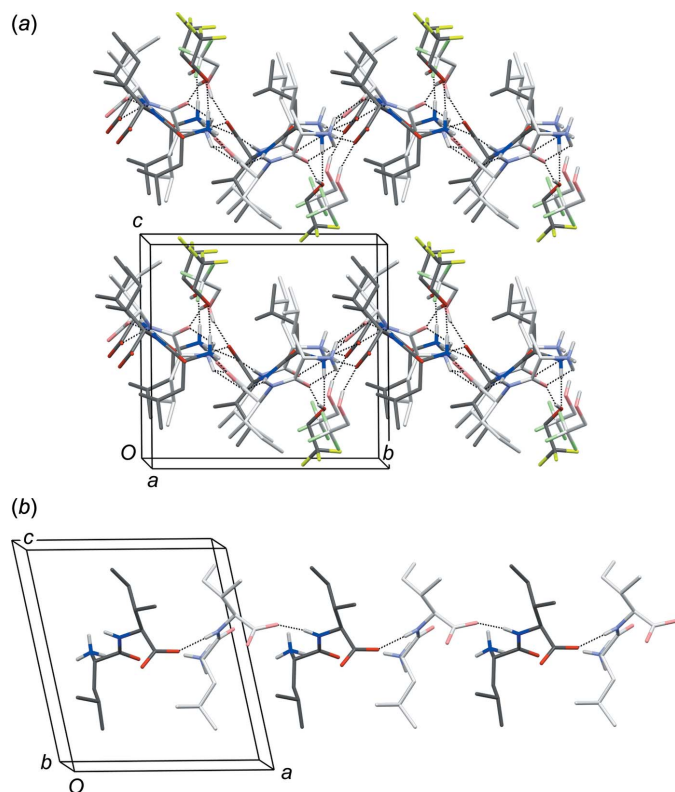


Figure 3
 Crystal packing of (I) viewed (a) approximately along the *x* axis. (b) View approximately along the *y* axis showing a single hydrogen-bonded *C*(5) chain parallel to the *x* axis. Only H atoms involved in strong hydrogen bonds are included; peptide molecule *A* and TFE molecule *C* are shown with atoms in lighter colors.

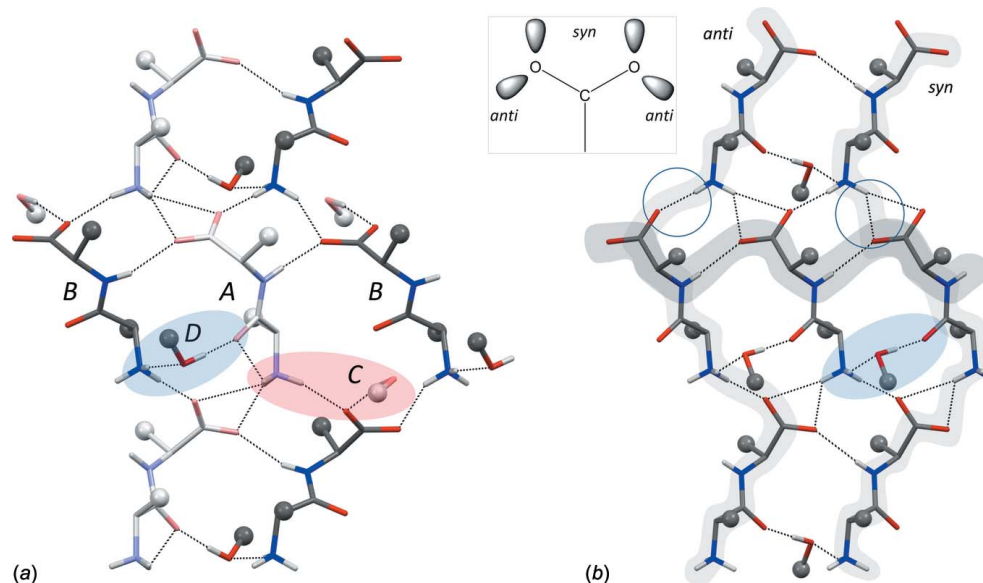


Figure 4
 Hydrogen bonds in (a) the crystal structure of (I) and (b) the crystal structure of *L*-Leu-*L*-Val 2-propanol solvate (Görbitz & Torgersen, 1999; CSD refcode JUCSEF01). Peptide *C* β atoms and solvent *C* atoms carrying hydroxyl groups are shown as small spheres, other side-chain and solvent atoms have been omitted for clarity. The archetype **S5** pattern in (b) is characterized by the presence of one *syn* and one *anti* head-to-tail *C*(8) chain with alternating molecules being related by Screw symmetry (light grey shades), as well as a *C*(5) chain involving an amide >*N*-H donor and a carboxylate acceptor. An **S4** pattern has the same symmetry, but a *C*(4) chain to O=C< carbonyl acceptor, while consecutive molecules in **T5** and **T4** sheets are related by Translation rather than by a screw operation (Görbitz, 2010). See text for details on the red and blue shades.

While the two molecules in the asymmetric unit of structures like *L*-Met-*L*-Ala 2-propanol solvate (Görbitz, 2000; CSD refcode CAQTOD) and *L*-Leu-*L*-Phe 2-propanol solvate (Görbitz, 1999; CSD refcode COCGOQ) are quite similar and related by pseudotranslational symmetry along a 10 Å long axis, the differences between the conformations (as discussed above) and relative positions of LI molecules *A* and *B* are readily observed in Fig. 3*b*). The *C*(5) hydrogen-bonded chain is part of an **S5** hydrogen-bonded sheet, one out of four distinct types of sheets observed in layered dipeptide crystal structures (Görbitz, 2010).

This sheet is compared in Fig. 4 to the corresponding sheet of *L*-Leu-*L*-Val 2-propanol solvate (Görbitz & Torgersen, 1999), where the third amino hydrogen atom is accepted by the co-crystallized alcohol molecule (shaded blue in Fig. 4*b*). At the same time, the hydroxyl group serves as a hydrogen-bond donor to the peptide carbonyl group, which is not involved in any other strong hydrogen bonds (in distinction to the related **S4** pattern). Precisely the same function is taken by TFE molecule *D* in Fig. 4*a*), but solvent molecule *C* is different; it seeks out and forms a hydrogen bond to the carboxylate group of peptide molecule *B*, uniquely abandoning its role as a hydrogen-bond acceptor (red shade in Fig. 4*b*). The third amino H atom of molecule *A* is then left to participate in only a bent intramolecular interaction that leads to the inherently less favorable eclipsed amino conformation shown in Fig. 1.

In summary, TFE has been shown to be co-crystallized with *L*-Leu-*L*-Ile, thus radically changing the hydrogen bonding pattern. It is the first dipeptide alcohol solvate where an

Table 3
Experimental details.

Crystal data	
Chemical formula	C ₁₂ H ₂₄ N ₂ O ₃ ·C ₂ H ₃ F ₃ O
<i>M</i> _r	344.37
Crystal system, space group	Monoclinic, <i>P</i> ₂ ₁
Temperature (K)	120
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.947 (3), 12.999 (4), 12.440 (4)
β (°)	101.833 (4)
<i>V</i> (Å ³)	1732.6 (9)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.12
Crystal size (mm)	0.77 × 0.43 × 0.07
Data collection	
Diffractometer	Bruker D8 Advance single crystal CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2014)
<i>T</i> _{min} , <i>T</i> _{max}	0.643, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	10435, 5594, 4796
<i>R</i> _{int}	0.039
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.598
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.041, 0.095, 1.03
No. of reflections	5594
No. of parameters	454
No. of restraints	39
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.27, -0.18

Computer programs: *APEX2* and *SAINTE-Plus* (Bruker, 2014), *SHELXT* (Sheldrick, 2015a), *Mercury* (Macrae *et al.*, 2008) and *SHELXL2014* (Sheldrick, 2015b).

alcohol molecule does not act as a hydrogen bond acceptor, but rather forms a strong hydrogen bond donor to a peptide carboxylate acceptor.

4. Synthesis and crystallization

L-Leu-L-Val was purchased from Sigma–Aldrich and used as received. Colorless plates of the title compound were grown by vapor diffusion of acetonitrile into 30 μ l of a saturated trifluoroethanol solution of the dipeptide.

5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. Solvent molecule *D* is disordered over a major and a minor position with occupancies 0.825 (5) and 0.175 (5), respectively. The O1 and C1 atoms of the minor component were constrained to have the same set of anisotropic displacement parameters as the corresponding atoms of the major component, while C2 and the three F atoms were refined isotropically.

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supporting information

Acta Cryst. (2016). E72, 635-638 [doi:10.1107/S2056989016005302]

Crystal structure of L-leucyl-L-isoleucine 2,2,2-trifluoroethanol monosolvate

Carl Henrik Görbitz

Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINTE-Plus* (Bruker, 2014); data reduction: *SAINTE-Plus* (Bruker, 2014); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXT* (Sheldrick, 2015a); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015b).

L-Leucyl-L-isoleucine 2,2,2-trifluoroethanol monosolvate

Crystal data

$C_{12}H_{24}N_2O_3 \cdot C_2H_3F_3O$

$M_r = 344.37$

Monoclinic, $P2_1$

$a = 10.947$ (3) Å

$b = 12.999$ (4) Å

$c = 12.440$ (4) Å

$\beta = 101.833$ (4)°

$V = 1732.6$ (9) Å³

$Z = 4$

$F(000) = 736$

$D_x = 1.320$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4820 reflections

$\theta = 2.3$ – 25.0 °

$\mu = 0.12$ mm⁻¹

$T = 120$ K

Plate, colorless

$0.77 \times 0.43 \times 0.07$ mm

Data collection

Bruker D8 Advance single crystal CCD diffractometer

Radiation source: fine-focus sealed tube
Graphite monochromator

Detector resolution: 8.3 pixels mm⁻¹

Sets of exposures each taken over 0.5° ω rotation scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2014)

$T_{\min} = 0.643$, $T_{\max} = 1.000$

10435 measured reflections

5594 independent reflections

4796 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 25.1$ °, $\theta_{\min} = 1.7$ °

$h = -13$ → 13

$k = -15$ → 12

$l = -14$ → 14

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.095$

$S = 1.03$

5594 reflections

454 parameters

39 restraints

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.0691P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.27$ e Å⁻³

$\Delta\rho_{\min} = -0.18$ e Å⁻³

Special details

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

Refinement. One of the solvent molecules is disordered over two positions.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1A	0.0423 (2)	0.6609 (2)	0.3719 (2)	0.0280 (6)	
O2A	0.0083 (2)	0.3213 (2)	0.4273 (2)	0.0325 (7)	
O3A	-0.1629 (2)	0.3989 (2)	0.3414 (2)	0.0305 (7)	
N1A	0.2285 (3)	0.7482 (2)	0.5048 (2)	0.0199 (7)	
H1A	0.2165	0.7717	0.5708	0.030*	
H2A	0.1656	0.7711	0.4505	0.030*	
H3A	0.3028	0.7718	0.4929	0.030*	
N2A	0.1323 (2)	0.5031 (2)	0.3755 (2)	0.0190 (7)	
H4A	0.2002	0.4671	0.4009	0.023*	
C1A	0.2293 (3)	0.6338 (3)	0.5053 (3)	0.0183 (8)	
H11A	0.3123	0.6076	0.4960	0.022*	
C2A	0.1980 (3)	0.5909 (3)	0.6115 (3)	0.0222 (8)	
H21A	0.1192	0.6229	0.6220	0.027*	
H22A	0.1826	0.5161	0.6018	0.027*	
C3A	0.2968 (3)	0.6072 (3)	0.7160 (3)	0.0225 (8)	
H31A	0.3203	0.6816	0.7215	0.027*	
C4A	0.4133 (3)	0.5437 (3)	0.7136 (3)	0.0307 (10)	
H41A	0.4465	0.5625	0.6489	0.046*	
H42A	0.3919	0.4704	0.7103	0.046*	
H43A	0.4764	0.5573	0.7802	0.046*	
C5A	0.2433 (4)	0.5779 (4)	0.8157 (3)	0.0376 (11)	
H51A	0.1661	0.6164	0.8144	0.056*	
H52A	0.3040	0.5945	0.8830	0.056*	
H53A	0.2255	0.5040	0.8138	0.056*	
C6A	0.1269 (3)	0.5994 (3)	0.4086 (3)	0.0192 (8)	
C7A	0.0299 (3)	0.4538 (3)	0.2983 (3)	0.0180 (8)	
H71A	-0.0258	0.5090	0.2592	0.022*	
C8A	0.0799 (3)	0.3913 (3)	0.2128 (3)	0.0207 (8)	
H81A	0.1408	0.3403	0.2533	0.025*	
C9A	0.1501 (3)	0.4587 (3)	0.1444 (3)	0.0270 (9)	
H91A	0.2233	0.4895	0.1941	0.032*	
H92A	0.1820	0.4141	0.0918	0.032*	
C10A	0.0738 (4)	0.5449 (4)	0.0804 (3)	0.0442 (12)	
H12A	0.1226	0.5782	0.0324	0.066*	
H13A	0.0525	0.5957	0.1319	0.066*	
H14A	-0.0030	0.5165	0.0358	0.066*	
C11A	-0.0252 (3)	0.3306 (4)	0.1401 (3)	0.0350 (10)	
H15A	-0.0647	0.2854	0.1861	0.052*	

H16A	0.0093	0.2890	0.0877	0.052*
H17A	-0.0873	0.3785	0.1000	0.052*
C12A	-0.0472 (3)	0.3866 (3)	0.3615 (3)	0.0215 (8)
O1B	0.5502 (2)	0.6220 (2)	0.50128 (19)	0.0238 (6)
O2B	0.5573 (2)	0.3479 (2)	0.52421 (19)	0.0215 (6)
O3B	0.3600 (2)	0.3917 (2)	0.45792 (19)	0.0216 (6)
N1B	0.7727 (3)	0.7368 (2)	0.4733 (2)	0.0226 (7)
H1B	0.8461	0.7665	0.5066	0.034*
H2B	0.7080	0.7715	0.4921	0.034*
H3B	0.7655	0.7392	0.3991	0.034*
N2B	0.6296 (2)	0.5066 (2)	0.3983 (2)	0.0192 (7)
H4B	0.6958	0.4877	0.3732	0.023*
C1B	0.7706 (3)	0.6274 (3)	0.5093 (3)	0.0193 (8)
H11B	0.8320	0.5861	0.4779	0.023*
C2B	0.8047 (3)	0.6239 (3)	0.6345 (3)	0.0227 (8)
H21B	0.8894	0.6530	0.6585	0.027*
H22B	0.7462	0.6691	0.6636	0.027*
C3B	0.8024 (3)	0.5183 (3)	0.6864 (3)	0.0248 (9)
H31B	0.7208	0.4848	0.6546	0.030*
C4B	0.8116 (4)	0.5314 (4)	0.8093 (3)	0.0371 (11)
H41B	0.8115	0.4636	0.8438	0.056*
H42B	0.7401	0.5715	0.8221	0.056*
H43B	0.8891	0.5676	0.8411	0.056*
C5B	0.9073 (4)	0.4492 (3)	0.6641 (3)	0.0343 (10)
H51B	0.9013	0.3815	0.6974	0.052*
H52B	0.9881	0.4804	0.6960	0.052*
H53B	0.8997	0.4414	0.5847	0.052*
C6B	0.6391 (3)	0.5850 (3)	0.4685 (3)	0.0189 (8)
C7B	0.5130 (3)	0.4513 (3)	0.3620 (3)	0.0184 (8)
H71B	0.4463	0.5021	0.3316	0.022*
C8B	0.5294 (3)	0.3774 (3)	0.2694 (3)	0.0197 (8)
H81B	0.6128	0.3439	0.2930	0.024*
C9B	0.5323 (3)	0.4366 (3)	0.1635 (3)	0.0261 (9)
H91B	0.4456	0.4501	0.1243	0.031*
H92B	0.5735	0.5038	0.1826	0.031*
C10B	0.6011 (4)	0.3791 (4)	0.0867 (3)	0.0393 (11)
H12B	0.5968	0.4190	0.0192	0.059*
H13B	0.5620	0.3118	0.0685	0.059*
H14B	0.6886	0.3696	0.1231	0.059*
C11B	0.4328 (3)	0.2917 (3)	0.2499 (3)	0.0249 (9)
H15B	0.4419	0.2489	0.3159	0.037*
H16B	0.4453	0.2493	0.1879	0.037*
H17B	0.3489	0.3217	0.2332	0.037*
C12B	0.4741 (3)	0.3938 (3)	0.4562 (3)	0.0175 (8)
O1C	0.5265 (2)	0.7674 (2)	0.3049 (2)	0.0299 (7)
H1C	0.501 (4)	0.784 (3)	0.364 (3)	0.045*
F1C	0.2472 (2)	0.77562 (19)	0.09991 (19)	0.0419 (6)
F2C	0.3722 (2)	0.64892 (18)	0.1427 (2)	0.0478 (7)

F3C	0.2751 (2)	0.7092 (2)	0.26117 (19)	0.0454 (7)	
C1C	0.4374 (3)	0.8097 (3)	0.2199 (3)	0.0257 (9)	
H11C	0.4774	0.8281	0.1580	0.031*	
H12C	0.4032	0.8734	0.2459	0.031*	
C2C	0.3341 (3)	0.7359 (3)	0.1812 (3)	0.0285 (9)	
O1D	0.8241 (3)	0.7359 (3)	0.2588 (3)	0.0329 (10)	0.825 (5)
H1D	0.897 (4)	0.710 (4)	0.279 (4)	0.049*	0.825 (5)
F1D	0.6835 (3)	0.8066 (3)	0.0612 (3)	0.0441 (9)	0.825 (5)
F2D	0.8335 (3)	0.7120 (3)	0.0332 (3)	0.0576 (12)	0.825 (5)
F3D	0.6481 (3)	0.6516 (3)	0.0016 (2)	0.0572 (12)	0.825 (5)
C1D	0.7461 (4)	0.6710 (4)	0.1859 (3)	0.0280 (13)	0.825 (5)
H11D	0.6640	0.6662	0.2072	0.034*	0.825 (5)
H12D	0.7829	0.6012	0.1901	0.034*	0.825 (5)
C2D	0.7286 (4)	0.7099 (4)	0.0713 (3)	0.0353 (14)	0.825 (5)
O11D	0.8098 (19)	0.6853 (16)	0.2667 (12)	0.0329 (10)	0.175 (5)
H11E	0.8867	0.6965	0.2746	0.049*	0.175 (5)
F11D	0.7114 (17)	0.8328 (14)	0.1014 (19)	0.090 (11)*	0.175 (5)
F12D	0.8965 (12)	0.7813 (13)	0.1012 (14)	0.077 (6)*	0.175 (5)
F13D	0.7446 (19)	0.7253 (17)	-0.0213 (10)	0.116 (10)*	0.175 (5)
C11D	0.760 (2)	0.6624 (12)	0.1569 (13)	0.0280 (13)	0.175 (5)
H13D	0.6694	0.6480	0.1477	0.034*	0.175 (5)
H14D	0.8006	0.6000	0.1351	0.034*	0.175 (5)
C12D	0.7788 (13)	0.7499 (11)	0.0850 (10)	0.038 (8)*	0.175 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1A	0.0237 (14)	0.0214 (16)	0.0342 (15)	0.0061 (12)	-0.0051 (11)	-0.0037 (12)
O2A	0.0238 (13)	0.0317 (17)	0.0442 (17)	0.0035 (13)	0.0125 (12)	0.0137 (15)
O3A	0.0144 (13)	0.0460 (19)	0.0315 (15)	0.0020 (12)	0.0056 (11)	-0.0025 (14)
N1A	0.0179 (15)	0.0213 (19)	0.0198 (15)	-0.0020 (13)	0.0025 (12)	-0.0007 (13)
N2A	0.0143 (14)	0.0186 (19)	0.0224 (16)	0.0012 (12)	-0.0003 (12)	-0.0011 (14)
C1A	0.0182 (17)	0.014 (2)	0.0214 (19)	0.0003 (14)	0.0022 (14)	-0.0015 (15)
C2A	0.0227 (18)	0.021 (2)	0.023 (2)	-0.0005 (16)	0.0049 (15)	0.0016 (17)
C3A	0.030 (2)	0.017 (2)	0.0181 (18)	-0.0010 (16)	0.0003 (15)	-0.0002 (16)
C4A	0.026 (2)	0.040 (3)	0.024 (2)	0.0019 (19)	0.0003 (16)	0.0031 (19)
C5A	0.042 (2)	0.046 (3)	0.025 (2)	0.008 (2)	0.0083 (19)	0.003 (2)
C6A	0.0159 (17)	0.020 (2)	0.0212 (19)	-0.0007 (15)	0.0033 (14)	-0.0014 (16)
C7A	0.0162 (16)	0.018 (2)	0.0188 (18)	-0.0002 (15)	0.0007 (13)	0.0008 (15)
C8A	0.0222 (17)	0.021 (2)	0.0206 (19)	0.0013 (16)	0.0073 (14)	-0.0026 (17)
C9A	0.0266 (19)	0.032 (3)	0.023 (2)	-0.0011 (17)	0.0055 (15)	-0.0006 (18)
C10A	0.037 (2)	0.058 (3)	0.034 (3)	-0.003 (2)	0.000 (2)	0.018 (2)
C11A	0.031 (2)	0.038 (3)	0.038 (2)	-0.0091 (19)	0.0136 (19)	-0.014 (2)
C12A	0.0215 (19)	0.022 (2)	0.0221 (19)	0.0014 (17)	0.0079 (14)	-0.0087 (18)
O1B	0.0221 (13)	0.0259 (16)	0.0240 (14)	0.0017 (11)	0.0064 (11)	-0.0043 (12)
O2B	0.0196 (12)	0.0230 (16)	0.0219 (13)	0.0017 (10)	0.0044 (11)	0.0019 (11)
O3B	0.0152 (12)	0.0232 (15)	0.0265 (13)	0.0016 (11)	0.0048 (10)	0.0023 (12)
N1B	0.0214 (15)	0.0222 (19)	0.0251 (17)	-0.0002 (13)	0.0070 (13)	-0.0002 (14)

N2B	0.0169 (14)	0.0201 (18)	0.0213 (16)	0.0002 (12)	0.0060 (12)	-0.0024 (14)
C1B	0.0221 (18)	0.014 (2)	0.0229 (19)	-0.0009 (15)	0.0075 (15)	-0.0016 (16)
C2B	0.0206 (18)	0.023 (2)	0.0233 (19)	-0.0019 (15)	0.0026 (15)	-0.0022 (17)
C3B	0.0213 (18)	0.029 (2)	0.022 (2)	-0.0048 (16)	0.0010 (15)	0.0006 (17)
C4B	0.042 (2)	0.040 (3)	0.027 (2)	-0.006 (2)	0.0026 (19)	0.001 (2)
C5B	0.040 (2)	0.025 (3)	0.037 (2)	0.000 (2)	0.0062 (18)	0.003 (2)
C6B	0.0189 (17)	0.020 (2)	0.0174 (18)	0.0003 (15)	0.0037 (14)	0.0050 (16)
C7B	0.0146 (16)	0.019 (2)	0.0211 (19)	0.0007 (15)	0.0010 (14)	0.0012 (16)
C8B	0.0165 (17)	0.022 (2)	0.0202 (19)	-0.0003 (15)	0.0024 (14)	-0.0034 (16)
C9B	0.029 (2)	0.029 (2)	0.0203 (19)	-0.0030 (18)	0.0060 (16)	0.0003 (17)
C10B	0.038 (2)	0.059 (3)	0.024 (2)	-0.004 (2)	0.0127 (17)	-0.005 (2)
C11B	0.0212 (18)	0.028 (3)	0.024 (2)	-0.0013 (17)	0.0014 (15)	-0.0021 (17)
C12B	0.0185 (18)	0.013 (2)	0.0196 (18)	0.0022 (15)	0.0011 (14)	-0.0045 (16)
O1C	0.0289 (14)	0.0399 (19)	0.0208 (14)	0.0083 (13)	0.0045 (11)	0.0017 (13)
F1C	0.0378 (13)	0.0410 (17)	0.0400 (14)	-0.0028 (12)	-0.0079 (11)	0.0016 (12)
F2C	0.0526 (15)	0.0330 (17)	0.0551 (16)	0.0018 (12)	0.0048 (12)	-0.0159 (13)
F3C	0.0418 (14)	0.0557 (19)	0.0410 (15)	-0.0157 (12)	0.0140 (12)	0.0048 (13)
C1C	0.028 (2)	0.026 (2)	0.022 (2)	0.0010 (17)	0.0035 (16)	0.0003 (17)
C2C	0.035 (2)	0.026 (3)	0.024 (2)	0.0008 (18)	0.0042 (18)	0.0010 (18)
O1D	0.0277 (17)	0.039 (3)	0.0311 (17)	0.003 (2)	0.0032 (14)	0.004 (2)
F1D	0.054 (2)	0.041 (2)	0.035 (2)	0.0164 (17)	0.0036 (17)	0.0132 (18)
F2D	0.055 (2)	0.084 (3)	0.044 (2)	0.0129 (19)	0.0334 (17)	0.0123 (19)
F3D	0.073 (2)	0.062 (3)	0.0291 (17)	-0.0009 (19)	-0.0069 (16)	-0.0075 (16)
C1D	0.026 (2)	0.039 (3)	0.020 (3)	0.007 (2)	0.007 (2)	0.002 (2)
C2D	0.034 (3)	0.045 (4)	0.028 (3)	0.007 (3)	0.007 (2)	0.003 (3)
O11D	0.0277 (17)	0.039 (3)	0.0311 (17)	0.003 (2)	0.0032 (14)	0.004 (2)
C11D	0.026 (2)	0.039 (3)	0.020 (3)	0.007 (2)	0.007 (2)	0.002 (2)

Geometric parameters (Å, °)

O1A—C6A	1.238 (4)	C1B—H11B	1.0000
O2A—C12A	1.247 (5)	C2B—C3B	1.519 (5)
O3A—C12A	1.251 (4)	C2B—H21B	0.9900
N1A—C1A	1.488 (5)	C2B—H22B	0.9900
N1A—H1A	0.9100	C3B—C4B	1.520 (5)
N1A—H2A	0.9100	C3B—C5B	1.526 (5)
N1A—H3A	0.9100	C3B—H31B	1.0000
N2A—C6A	1.323 (5)	C4B—H41B	0.9800
N2A—C7A	1.466 (4)	C4B—H42B	0.9800
N2A—H4A	0.8800	C4B—H43B	0.9800
C1A—C6A	1.533 (5)	C5B—H51B	0.9800
C1A—C2A	1.536 (5)	C5B—H52B	0.9800
C1A—H11A	1.0000	C5B—H53B	0.9800
C2A—C3A	1.525 (5)	C7B—C12B	1.523 (5)
C2A—H21A	0.9900	C7B—C8B	1.538 (5)
C2A—H22A	0.9900	C7B—H71B	1.0000
C3A—C4A	1.525 (5)	C8B—C11B	1.521 (5)
C3A—C5A	1.525 (5)	C8B—C9B	1.532 (5)

C3A—H31A	1.0000	C8B—H81B	1.0000
C4A—H41A	0.9800	C9B—C10B	1.527 (5)
C4A—H42A	0.9800	C9B—H91B	0.9900
C4A—H43A	0.9800	C9B—H92B	0.9900
C5A—H51A	0.9800	C10B—H12B	0.9800
C5A—H52A	0.9800	C10B—H13B	0.9800
C5A—H53A	0.9800	C10B—H14B	0.9800
C7A—C8A	1.526 (5)	C11B—H15B	0.9800
C7A—C12A	1.538 (5)	C11B—H16B	0.9800
C7A—H71A	1.0000	C11B—H17B	0.9800
C8A—C11A	1.528 (5)	O1C—C1C	1.396 (4)
C8A—C9A	1.534 (5)	O1C—H1C	0.86 (3)
C8A—H81A	1.0000	F1C—C2C	1.342 (4)
C9A—C10A	1.522 (6)	F2C—C2C	1.328 (4)
C9A—H91A	0.9900	F3C—C2C	1.338 (4)
C9A—H92A	0.9900	C1C—C2C	1.486 (5)
C10A—H12A	0.9800	C1C—H11C	0.9900
C10A—H13A	0.9800	C1C—H12C	0.9900
C10A—H14A	0.9800	O1D—C1D	1.394 (4)
C11A—H15A	0.9800	O1D—H1D	0.86 (3)
C11A—H16A	0.9800	F1D—C2D	1.347 (4)
C11A—H17A	0.9800	F2D—C2D	1.329 (4)
O1B—C6B	1.228 (4)	F3D—C2D	1.338 (5)
O2B—C12B	1.259 (4)	C1D—C2D	1.487 (5)
O3B—C12B	1.254 (4)	C1D—H11D	0.9900
N1B—C1B	1.493 (5)	C1D—H12D	0.9900
N1B—H1B	0.9100	O11D—C11D	1.396 (6)
N1B—H2B	0.9100	O11D—H11E	0.8400
N1B—H3B	0.9100	F11D—C12D	1.345 (6)
N2B—C6B	1.333 (5)	F12D—C12D	1.327 (6)
N2B—C7B	1.453 (4)	F13D—C12D	1.337 (6)
N2B—H4B	0.8800	C11D—C12D	1.488 (6)
C1B—C2B	1.526 (5)	C11D—H13D	0.9900
C1B—C6B	1.528 (5)	C11D—H14D	0.9900
C1A—N1A—H1A	109.5	H21B—C2B—H22B	107.4
C1A—N1A—H2A	109.5	C2B—C3B—C4B	108.8 (3)
H1A—N1A—H2A	109.5	C2B—C3B—C5B	112.1 (3)
C1A—N1A—H3A	109.5	C4B—C3B—C5B	110.6 (3)
H1A—N1A—H3A	109.5	C2B—C3B—H31B	108.4
H2A—N1A—H3A	109.5	C4B—C3B—H31B	108.4
C6A—N2A—C7A	122.7 (3)	C5B—C3B—H31B	108.4
C6A—N2A—H4A	118.7	C3B—C4B—H41B	109.5
C7A—N2A—H4A	118.7	C3B—C4B—H42B	109.5
N1A—C1A—C6A	106.6 (3)	H41B—C4B—H42B	109.5
N1A—C1A—C2A	111.4 (3)	C3B—C4B—H43B	109.5
C6A—C1A—C2A	108.2 (3)	H41B—C4B—H43B	109.5
N1A—C1A—H11A	110.2	H42B—C4B—H43B	109.5

C6A—C1A—H11A	110.2	C3B—C5B—H51B	109.5
C2A—C1A—H11A	110.2	C3B—C5B—H52B	109.5
C3A—C2A—C1A	116.0 (3)	H51B—C5B—H52B	109.5
C3A—C2A—H21A	108.3	C3B—C5B—H53B	109.5
C1A—C2A—H21A	108.3	H51B—C5B—H53B	109.5
C3A—C2A—H22A	108.3	H52B—C5B—H53B	109.5
C1A—C2A—H22A	108.3	O1B—C6B—N2B	123.9 (3)
H21A—C2A—H22A	107.4	O1B—C6B—C1B	120.3 (3)
C4A—C3A—C5A	110.0 (3)	N2B—C6B—C1B	115.8 (3)
C4A—C3A—C2A	111.0 (3)	N2B—C7B—C12B	111.7 (3)
C5A—C3A—C2A	109.6 (3)	N2B—C7B—C8B	108.2 (3)
C4A—C3A—H31A	108.7	C12B—C7B—C8B	111.3 (3)
C5A—C3A—H31A	108.7	N2B—C7B—H71B	108.5
C2A—C3A—H31A	108.7	C12B—C7B—H71B	108.5
C3A—C4A—H41A	109.5	C8B—C7B—H71B	108.5
C3A—C4A—H42A	109.5	C11B—C8B—C9B	111.5 (3)
H41A—C4A—H42A	109.5	C11B—C8B—C7B	113.2 (3)
C3A—C4A—H43A	109.5	C9B—C8B—C7B	110.9 (3)
H41A—C4A—H43A	109.5	C11B—C8B—H81B	107.0
H42A—C4A—H43A	109.5	C9B—C8B—H81B	107.0
C3A—C5A—H51A	109.5	C7B—C8B—H81B	107.0
C3A—C5A—H52A	109.5	C10B—C9B—C8B	113.1 (3)
H51A—C5A—H52A	109.5	C10B—C9B—H91B	109.0
C3A—C5A—H53A	109.5	C8B—C9B—H91B	109.0
H51A—C5A—H53A	109.5	C10B—C9B—H92B	109.0
H52A—C5A—H53A	109.5	C8B—C9B—H92B	109.0
O1A—C6A—N2A	125.0 (3)	H91B—C9B—H92B	107.8
O1A—C6A—C1A	118.2 (3)	C9B—C10B—H12B	109.5
N2A—C6A—C1A	116.6 (3)	C9B—C10B—H13B	109.5
N2A—C7A—C8A	110.7 (3)	H12B—C10B—H13B	109.5
N2A—C7A—C12A	109.8 (3)	C9B—C10B—H14B	109.5
C8A—C7A—C12A	111.5 (3)	H12B—C10B—H14B	109.5
N2A—C7A—H71A	108.3	H13B—C10B—H14B	109.5
C8A—C7A—H71A	108.3	C8B—C11B—H15B	109.5
C12A—C7A—H71A	108.3	C8B—C11B—H16B	109.5
C7A—C8A—C11A	110.7 (3)	H15B—C11B—H16B	109.5
C7A—C8A—C9A	112.0 (3)	C8B—C11B—H17B	109.5
C11A—C8A—C9A	111.6 (3)	H15B—C11B—H17B	109.5
C7A—C8A—H81A	107.4	H16B—C11B—H17B	109.5
C11A—C8A—H81A	107.4	O3B—C12B—O2B	124.3 (3)
C9A—C8A—H81A	107.4	O3B—C12B—C7B	117.4 (3)
C10A—C9A—C8A	115.3 (3)	O2B—C12B—C7B	118.2 (3)
C10A—C9A—H91A	108.5	C1C—O1C—H1C	104 (3)
C8A—C9A—H91A	108.5	O1C—C1C—C2C	111.0 (3)
C10A—C9A—H92A	108.5	O1C—C1C—H11C	109.4
C8A—C9A—H92A	108.5	C2C—C1C—H11C	109.4
H91A—C9A—H92A	107.5	O1C—C1C—H12C	109.4
C9A—C10A—H12A	109.5	C2C—C1C—H12C	109.4

C9A—C10A—H13A	109.5	H11C—C1C—H12C	108.0
H12A—C10A—H13A	109.5	F2C—C2C—F3C	106.4 (3)
C9A—C10A—H14A	109.5	F2C—C2C—F1C	106.4 (3)
H12A—C10A—H14A	109.5	F3C—C2C—F1C	106.5 (3)
H13A—C10A—H14A	109.5	F2C—C2C—C1C	113.1 (3)
C8A—C11A—H15A	109.5	F3C—C2C—C1C	112.2 (3)
C8A—C11A—H16A	109.5	F1C—C2C—C1C	111.7 (3)
H15A—C11A—H16A	109.5	C1D—O1D—H1D	111 (4)
C8A—C11A—H17A	109.5	O1D—C1D—C2D	111.0 (4)
H15A—C11A—H17A	109.5	O1D—C1D—H11D	109.4
H16A—C11A—H17A	109.5	C2D—C1D—H11D	109.4
O2A—C12A—O3A	123.8 (3)	O1D—C1D—H12D	109.4
O2A—C12A—C7A	118.6 (3)	C2D—C1D—H12D	109.4
O3A—C12A—C7A	117.6 (3)	H11D—C1D—H12D	108.0
C1B—N1B—H1B	109.5	F2D—C2D—F3D	106.4 (3)
C1B—N1B—H2B	109.5	F2D—C2D—F1D	106.0 (4)
H1B—N1B—H2B	109.5	F3D—C2D—F1D	106.7 (4)
C1B—N1B—H3B	109.5	F2D—C2D—C1D	113.4 (4)
H1B—N1B—H3B	109.5	F3D—C2D—C1D	111.5 (4)
H2B—N1B—H3B	109.5	F1D—C2D—C1D	112.3 (3)
C6B—N2B—C7B	121.8 (3)	C11D—O11D—H11E	109.5
C6B—N2B—H4B	119.1	O11D—C11D—C12D	110.5 (7)
C7B—N2B—H4B	119.1	O11D—C11D—H13D	109.6
N1B—C1B—C2B	108.6 (3)	C12D—C11D—H13D	109.6
N1B—C1B—C6B	108.4 (3)	O11D—C11D—H14D	109.6
C2B—C1B—C6B	110.2 (3)	C12D—C11D—H14D	109.6
N1B—C1B—H11B	109.9	H13D—C11D—H14D	108.1
C2B—C1B—H11B	109.9	F12D—C12D—F13D	106.9 (7)
C6B—C1B—H11B	109.9	F12D—C12D—F11D	106.0 (7)
C3B—C2B—C1B	116.0 (3)	F13D—C12D—F11D	106.8 (7)
C3B—C2B—H21B	108.3	F12D—C12D—C11D	113.3 (6)
C1B—C2B—H21B	108.3	F13D—C12D—C11D	111.5 (7)
C3B—C2B—H22B	108.3	F11D—C12D—C11D	111.9 (6)
C1B—C2B—H22B	108.3		
N1A—C1A—C2A—C3A	-69.5 (4)	C7B—N2B—C6B—C1B	173.1 (3)
C6A—C1A—C2A—C3A	173.6 (3)	N1B—C1B—C6B—O1B	-63.1 (4)
C1A—C2A—C3A—C4A	-68.2 (4)	C2B—C1B—C6B—O1B	55.6 (4)
C1A—C2A—C3A—C5A	170.1 (3)	N1B—C1B—C6B—N2B	117.8 (3)
C7A—N2A—C6A—O1A	-6.9 (5)	C2B—C1B—C6B—N2B	-123.5 (3)
C7A—N2A—C6A—C1A	168.6 (3)	C6B—N2B—C7B—C12B	-65.5 (4)
N1A—C1A—C6A—O1A	-21.6 (4)	C6B—N2B—C7B—C8B	171.7 (3)
C2A—C1A—C6A—O1A	98.4 (4)	N2B—C7B—C8B—C11B	161.1 (3)
N1A—C1A—C6A—N2A	162.6 (3)	C12B—C7B—C8B—C11B	38.0 (4)
C2A—C1A—C6A—N2A	-77.5 (4)	N2B—C7B—C8B—C9B	-72.7 (3)
C6A—N2A—C7A—C8A	136.9 (3)	C12B—C7B—C8B—C9B	164.2 (3)
C6A—N2A—C7A—C12A	-99.6 (4)	C11B—C8B—C9B—C10B	-77.4 (4)
N2A—C7A—C8A—C11A	173.8 (3)	C7B—C8B—C9B—C10B	155.5 (3)

C12A—C7A—C8A—C11A	51.3 (4)	N2B—C7B—C12B—O3B	142.0 (3)
N2A—C7A—C8A—C9A	-60.9 (4)	C8B—C7B—C12B—O3B	-97.0 (4)
C12A—C7A—C8A—C9A	176.5 (3)	N2B—C7B—C12B—O2B	-41.1 (4)
C7A—C8A—C9A—C10A	-59.5 (4)	C8B—C7B—C12B—O2B	80.0 (4)
C11A—C8A—C9A—C10A	65.2 (4)	O1C—C1C—C2C—F2C	-60.4 (4)
N2A—C7A—C12A—O2A	-52.8 (4)	O1C—C1C—C2C—F3C	60.0 (4)
C8A—C7A—C12A—O2A	70.2 (4)	O1C—C1C—C2C—F1C	179.6 (3)
N2A—C7A—C12A—O3A	129.1 (3)	O1D—C1D—C2D—F2D	63.6 (5)
C8A—C7A—C12A—O3A	-107.9 (4)	O1D—C1D—C2D—F3D	-176.3 (4)
N1B—C1B—C2B—C3B	177.8 (3)	O1D—C1D—C2D—F1D	-56.6 (5)
C6B—C1B—C2B—C3B	59.2 (4)	O11D—C11D—C12D—F12D	52.3 (19)
C1B—C2B—C3B—C4B	-168.3 (3)	O11D—C11D—C12D—F13D	172.9 (18)
C1B—C2B—C3B—C5B	69.1 (4)	O11D—C11D—C12D—F11D	-67.5 (19)
C7B—N2B—C6B—O1B	-5.9 (5)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1A—H1A \cdots O3A ⁱ	0.91	2.13	2.928 (4)	146
N1A—H2A \cdots O1A	0.91	2.07	2.607 (4)	116
N1A—H3A \cdots O2B ⁱⁱ	0.91	1.87	2.767 (4)	168
N2A—H4A \cdots O3B	0.88	2.00	2.883 (4)	177
N1B—H1B \cdots O2A ⁱⁱ	0.91	1.79	2.695 (4)	179
N1B—H2B \cdots O3B ⁱⁱ	0.91	1.89	2.721 (4)	151
N1B—H3B \cdots O1D	0.91	1.98	2.838 (5)	156
O1D—H1D \cdots O1A ⁱⁱⁱ	0.86 (3)	1.87 (4)	2.695 (4)	159 (5)
O1C—H1C \cdots O2B ⁱⁱ	0.86 (3)	1.85 (3)	2.693 (4)	167 (4)

Symmetry codes: (i) $-x, y+1/2, -z+1$; (ii) $-x+1, y+1/2, -z+1$; (iii) $x+1, y, z$.