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1-(Pyridin-2-yl)-2,4-bis[(pyridin-2-yl)carbonyl]-3,5-bis(3,4,5-trimethoxyphenyl)cyclohexanol 2.25-hydrate

Era Dravida Thendral,[a] S. Gomathi,[b] U. Mohamooda Sumaya,[c] K. Biruntha[c] and G. Usha[a]*

[a]PG and Research Department of Physics, Queen Mary's College, Chennai-4, Tamilnadu, India, [b]PG Department of Physics, Bhaktavatsalam Memorial College for Women, Chennai-80, Tamilnadu, India, and [c]Department of Physics, Bharathi Women's College, Chennai-108, Tamilnadu, India. *Correspondence e-mail: guqmc@yahoo.com

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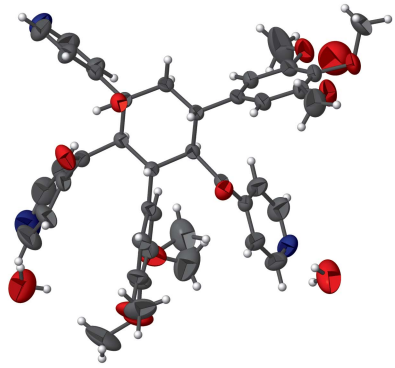
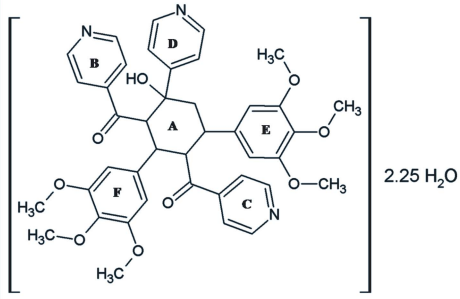
In the title compound, $C_{41}H_{41}N_3O_9\cdot 2.25H_2O$, the cyclohexanol ring adopts a chair conformation. The cyclohexanol ring makes dihedral angles of 87.89 (7) and 75.53 (8)° with the mean planes of the trimethoxyphenyl rings and dihedral angles of 84.18 (8), 85.07 (7) and 82.03 (8)° with the pyridine rings. In the crystal, the packing is stabilized by C—H···O, O—H···O and O—H···N hydrogen bonds involving water molecules and methoxy O atoms, resulting in a supramolecular network. One of the methoxy groups is disordered over two sets of sites with occupancy factors of 0.63 (3) and 0.37 (3).

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) has a central cyclohexanol ring attached to two trimethoxyphenyl, one pyridine and two pyridinecarbonyl rings. The dihedral angle between the trimethoxyphenyl rings *E* and *F* is 69.65 (7)°. The cyclohexanol ring *A* (C39–C44) makes dihedral angles of 75.53 (8) and 87.89 (7)°, respectively, with the mean planes of the rings *E* (C10–C15) and *F* (C1–C6). The pyridine rings *B* (N2/C20–C24), *C* (N1/C25–C29) and *D* (N1/C25–C29) are inclined to the central cyclohexanol ring by dihedral angles of 85.07 (7), 82.03 (8) and 84.18 (8)°, respectively. The cyclohexanol ring *A* adopts a chair conformation with puckering parameters q_2 = 0.034 (2), q_3 = 0.595 (2) and Q_T = 0.597 (2) Å, φ_2 = 288 (4) and θ_2 = 3.81 (19)°, and is similar to that observed in reported structures (Çelik *et al.*, 2016; Fun *et al.*, 2012).

In the crystal, the molecules are linked by C—H···O, O—H···O and O—H···N hydrogen bonds (Fig. 2, Table 1). The trifurcated atom O10 (acting as donor to two

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O3	0.96	2.40	2.918 (5)	114
C9—H9B···O7^{i}	0.96	2.63	3.575 (4)	168
C11—H11···O8^{ii}	0.93	2.55	3.450 (3)	163
C16—H16B···O12	0.96	2.16	2.951 (15)	139
C24—H24···O12^{iii}	0.93	2.57	3.192 (15)	125
C27—H27···O4^{iv}	0.93	2.62	3.408 (4)	143
C44—H44···O11^{v}	0.98	2.56	3.464 (4)	153
O9—H9···O7^{ii}	0.82	2.27	2.941 (2)	139
O9—H9···O8	0.82	2.17	2.760 (2)	129
O11—H11B···N2	0.85 (2)	2.02 (2)	2.861 (4)	169 (3)
O11—H11A···O10^{vi}	0.87 (2)	1.91 (2)	2.771 (4)	171 (3)
O10—H10A···N3	0.86 (2)	2.00 (2)	2.857 (4)	175 (4)
O10—H10B···O1^{vi}	0.86 (2)	2.16 (3)	2.939 (3)	151 (5)

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

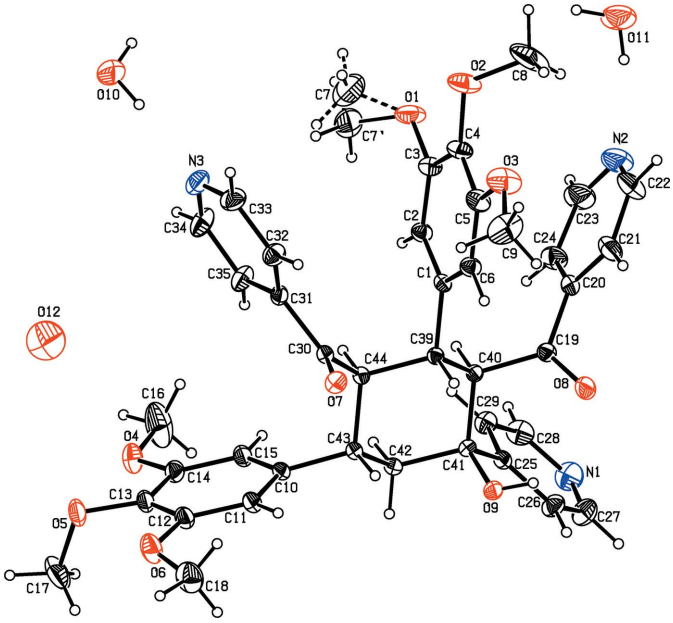


Figure 1
An *ORTEP* plot of the title compound with displacement ellipsoids drawn at the 20% probability level.

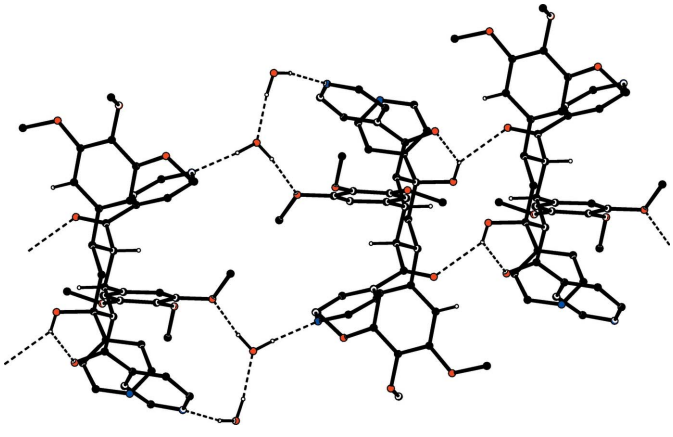


Figure 2
Linking of the molecules and water molecules by hydrogen bonds (dashed lines).

Table 2
Experimental details.

Crystal data	
Chemical formula	$4C_{41}H_{41}N_3O_9{\cdot}9H_2O$
M_r	3039.20
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (Å)	15.2367 (10), 14.8923 (7), 18.8149 (9)
β (°)	111.474 (2)
V (Å^3)	3972.9 (4)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.972, 0.982
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	38138, 6999, 4142
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.130, 1.08
No. of reflections	6995
No. of parameters	533
No. of restraints	13
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.20

Computer programs: *APEX2* (Bruker, 2004), *APEX2* and *SAINT* (Bruker, 2004), *SAINT* and *XPREP* (Bruker, 2004), *SIR92* (Altomare *et al.*, 1993), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

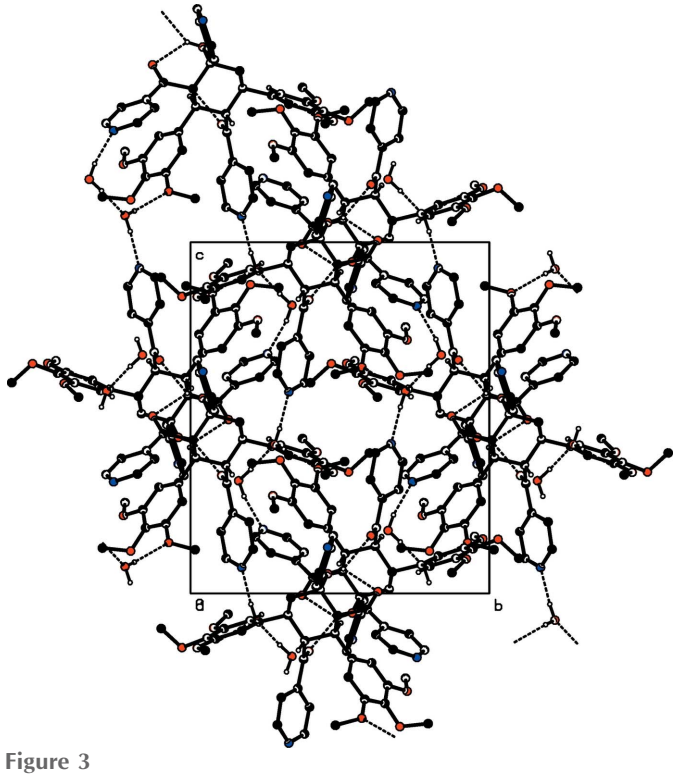


Figure 3
The crystal packing of the title compound, viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

hydrogen bonds and acceptor to one) and bifurcated atom O11 (acting as acceptor to two hydrogen bonds) of the water molecules are actively involved in the formation of hydrogen bonds through carbonyl O and pyridine N atoms. The O9 atom from the hydroxyl group generates intra- and intermolecular interactions with ketone atoms through O9—H9···O8 and O9—H9···O7 bonds. The packing of the molecules viewed along the *a* axis is shown in Fig. 3.

Synthesis and crystallization

The title compound was synthesized by the following Claisen–Schmidt reaction. In this procedure, 4-acetylpyridine(1.5 ml, 0.01 mol) and 3,4,5-trimethoxybenzaldehyde (2.6 g, 0.01 mol) were dissolved in 50 ml of ethanol in a round-bottomed flask. After 15 minutes, NaOH (10%) was added and the reaction mixture was maintained at 283 K and stirred for 5 h. Then the mixture was poured onto ice. The white-coloured precipitate was collected and washed with distilled water. A colourless, diffraction-quality crystal was obtained from repeated recrystallization from ethanol solution (yield: 87%, m.p. 398 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Atom C7 and its attached H atoms are disordered over two sets of sites with occupancy factors of 0.63 (3) and 0.37 (3). Atom O12 was refined with a fixed occupancy factor of 0.25 and no H atoms attached.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x171624 [https://doi.org/10.1107/S2414314617016248]

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Crystal data

$4C_{41}H_{41}N_3O_9·9H_2O$
M_r = 3039.20
Monoclinic, $P2_1/n$
a = 15.2367 (10) Å
b = 14.8923 (7) Å
c = 18.8149 (9) Å
β = 111.474 (2)°
V = 3972.9 (4) Å^3
Z = 1
$F(000)$ = 1608
D_x = 1.270 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7551 reflections
θ = 2.5–21.9°
μ = 0.09 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.972, T_{max} = 0.982
38138 measured reflections
6999 independent reflections
4142 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 25.0°, θ_{min} = 2.2°
h = −15→18
k = −17→17
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.130
S = 1.08
6995 reflections
533 parameters
13 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 2.0333P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Refinement. H atoms were positioned geometrically and treated as riding on their parent atoms and refined with, C—H = 0.93–0.96 Å and O—H = 0.82 Å, with U_{iso}(H) = 1.5 U_{eq}(c-methyl) and U_{iso}(H) = 1.2Ueq(O) for other H atoms. The water H atoms were identified through Fourier maps and fixed with their parent O atoms using *DFIX* 0.82 (2).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.47583 (16)	0.04020 (14)	0.68518 (13)	0.0309 (5)	
C2	0.43319 (18)	0.02820 (16)	0.73806 (13)	0.0393 (6)	
H2	0.3882	−0.0166	0.7308	0.047*	
C3	0.4578 (2)	0.08296 (18)	0.80163 (14)	0.0463 (7)	
C4	0.5238 (2)	0.15036 (18)	0.81334 (15)	0.0519 (7)	
C5	0.5690 (2)	0.15985 (17)	0.76169 (16)	0.0503 (7)	
C6	0.54417 (18)	0.10571 (16)	0.69746 (14)	0.0402 (6)	
H6	0.5736	0.1135	0.6624	0.048*	
C7	0.456 (2)	0.019 (2)	0.9148 (13)	0.113 (7)	0.37 (3)
H7A	0.4212	0.0208	0.9481	0.170*	0.37 (3)
H7B	0.5199	0.0377	0.9421	0.170*	0.37 (3)
H7C	0.4563	−0.0417	0.8969	0.170*	0.37 (3)
C7′	0.4051 (13)	−0.0152 (6)	0.8755 (10)	0.091 (4)	0.63 (3)
H7′1	0.4635	−0.0470	0.8872	0.136*	0.63 (3)
H7′2	0.3568	−0.0442	0.8337	0.136*	0.63 (3)
H7′3	0.3873	−0.0152	0.9195	0.136*	0.63 (3)
C8	0.5355 (4)	0.2949 (3)	0.8708 (3)	0.1271 (18)	
H8A	0.4757	0.3078	0.8314	0.191*	
H8B	0.5851	0.3203	0.8572	0.191*	
H8C	0.5372	0.3206	0.9181	0.191*	
C9	0.7029 (3)	0.2193 (3)	0.7414 (2)	0.0899 (12)	
H9A	0.7302	0.1603	0.7483	0.135*	
H9B	0.7517	0.2631	0.7629	0.135*	
H9C	0.6714	0.2309	0.6878	0.135*	
C10	0.48096 (18)	−0.27134 (15)	0.58138 (13)	0.0354 (6)	
C11	0.56584 (19)	−0.30086 (16)	0.57766 (14)	0.0424 (6)	
H11	0.6017	−0.2624	0.5602	0.051*	
C12	0.5971 (2)	−0.38757 (17)	0.59992 (15)	0.0466 (7)	
C13	0.5450 (2)	−0.44482 (16)	0.62708 (14)	0.0462 (7)	
C14	0.4599 (2)	−0.41605 (17)	0.62933 (14)	0.0453 (7)	
C15	0.42774 (19)	−0.32902 (16)	0.60684 (14)	0.0424 (6)	
H15	0.3707	−0.3099	0.6090	0.051*	
C16	0.3273 (3)	−0.4507 (3)	0.6625 (3)	0.1220 (18)	
H16A	0.3383	−0.3976	0.6935	0.183*	
H16B	0.3047	−0.4978	0.6861	0.183*	
H16C	0.2811	−0.4381	0.6129	0.183*	
C17	0.5445 (3)	−0.5983 (2)	0.6001 (2)	0.0919 (13)	
H17A	0.4768	−0.5969	0.5807	0.138*	
H17B	0.5661	−0.6552	0.6240	0.138*	
H17C	0.5664	−0.5899	0.5587	0.138*	
C18	0.7276 (2)	−0.3747 (2)	0.5590 (2)	0.0798 (10)	
H18A	0.6883	−0.3698	0.5060	0.120*	
H18B	0.7846	−0.4060	0.5638	0.120*	
H18C	0.7429	−0.3158	0.5807	0.120*	
C19	0.32405 (17)	0.09088 (16)	0.54529 (13)	0.0354 (6)	

C20	0.28709 (19)	0.14740 (16)	0.59363 (14)	0.0426 (7)
C21	0.3261 (3)	0.23114 (19)	0.61551 (18)	0.0694 (9)
H21	0.3738	0.2524	0.6002	0.083*
C22	0.2919 (4)	0.2829 (2)	0.6613 (2)	0.0973 (14)
H22	0.3200	0.3384	0.6781	0.117*
N2	0.2214 (3)	0.2575 (3)	0.68226 (19)	0.0988 (12)
C23	0.1847 (3)	0.1778 (3)	0.6599 (2)	0.0857 (11)
H23	0.1349	0.1593	0.6737	0.103*
C24	0.2162 (2)	0.1203 (2)	0.61726 (16)	0.0599 (8)
H24	0.1896	0.0636	0.6045	0.072*
C25	0.21191 (18)	−0.05937 (15)	0.44586 (13)	0.0360 (6)
C26	0.1745 (2)	−0.03033 (18)	0.37130 (14)	0.0513 (7)
H26	0.2139	−0.0160	0.3451	0.062*
C27	0.0774 (3)	−0.0226 (2)	0.33555 (18)	0.0712 (10)
H27	0.0537	−0.0032	0.2851	0.085*
N1	0.01609 (19)	−0.04114 (19)	0.36869 (16)	0.0731 (8)
C28	0.0532 (2)	−0.0694 (2)	0.44029 (18)	0.0602 (8)
H28	0.0121	−0.0832	0.4651	0.072*
C29	0.14823 (18)	−0.07962 (17)	0.48042 (15)	0.0457 (7)
H29	0.1697	−0.1001	0.5306	0.055*
C30	0.57500 (18)	−0.12177 (15)	0.68717 (13)	0.0332 (6)
C31	0.59889 (17)	−0.14134 (15)	0.77039 (13)	0.0354 (6)
C32	0.67705 (19)	−0.10218 (18)	0.82379 (14)	0.0481 (7)
H32	0.7160	−0.0650	0.8086	0.058*
C33	0.6968 (2)	−0.1188 (2)	0.90010 (16)	0.0590 (8)
H33	0.7498	−0.0917	0.9355	0.071*
N3	0.64484 (19)	−0.17117 (17)	0.92603 (13)	0.0616 (7)
C34	0.5709 (2)	−0.2095 (2)	0.87408 (17)	0.0668 (9)
H34	0.5343	−0.2477	0.8909	0.080*
C35	0.5443 (2)	−0.19640 (18)	0.79624 (15)	0.0541 (8)
H35	0.4908	−0.2242	0.7621	0.065*
C39	0.44920 (16)	−0.01741 (14)	0.61395 (12)	0.0284 (5)
H39	0.4867	0.0027	0.5843	0.034*
C40	0.34386 (16)	−0.00810 (14)	0.56285 (12)	0.0294 (5)
H40	0.3061	−0.0285	0.5922	0.035*
C41	0.31782 (16)	−0.06659 (14)	0.48983 (12)	0.0310 (5)
C42	0.34647 (17)	−0.16422 (15)	0.51199 (13)	0.0346 (6)
H42A	0.3326	−0.1996	0.4659	0.042*
H42B	0.3090	−0.1879	0.5397	0.042*
C43	0.45134 (16)	−0.17447 (14)	0.56144 (12)	0.0322 (6)
H43	0.4883	−0.1501	0.5328	0.039*
C44	0.47249 (16)	−0.11756 (14)	0.63428 (12)	0.0302 (5)
H44	0.4332	−0.1393	0.6619	0.036*
O1	0.41605 (17)	0.07301 (15)	0.85542 (12)	0.0709 (6)
O2	0.54795 (17)	0.20044 (15)	0.87925 (12)	0.0811 (7)
O3	0.63731 (17)	0.22451 (15)	0.77829 (13)	0.0807 (7)
O4	0.41173 (16)	−0.47771 (12)	0.65519 (12)	0.0662 (6)
O5	0.58008 (15)	−0.52884 (11)	0.65415 (11)	0.0642 (6)

O6	0.67918 (16)	−0.42277 (13)	0.59817 (13)	0.0700 (6)	
O7	0.63819 (12)	−0.10414 (11)	0.66429 (9)	0.0445 (4)	
O8	0.34263 (16)	0.12696 (11)	0.49476 (11)	0.0609 (6)	
O9	0.36986 (11)	−0.04089 (10)	0.44406 (8)	0.0382 (4)	
H9	0.3575	0.0113	0.4302	0.057*	
O10	0.6558 (3)	−0.2192 (2)	1.07603 (16)	0.1056 (10)	
O11	0.2246 (2)	0.3390 (2)	0.82088 (18)	0.1155 (11)	
O12	0.3307 (11)	−0.5495 (10)	0.7994 (8)	0.149 (5)*	0.25
H11B	0.219 (3)	0.321 (2)	0.7766 (11)	0.125 (16)*	
H11A	0.257 (2)	0.2982 (18)	0.8533 (14)	0.082 (13)*	
H10A	0.651 (3)	−0.202 (3)	1.0309 (15)	0.16 (2)*	
H10B	0.617 (3)	−0.188 (3)	1.089 (2)	0.16 (2)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0301 (14)	0.0274 (12)	0.0343 (13)	0.0018 (11)	0.0106 (11)	−0.0024 (10)
C2	0.0418 (16)	0.0362 (14)	0.0405 (14)	−0.0055 (12)	0.0159 (12)	−0.0059 (11)
C3	0.0505 (18)	0.0501 (16)	0.0425 (15)	0.0010 (14)	0.0221 (13)	−0.0081 (13)
C4	0.0568 (19)	0.0496 (17)	0.0471 (16)	−0.0063 (15)	0.0166 (15)	−0.0206 (14)
C5	0.0495 (18)	0.0410 (15)	0.0570 (17)	−0.0149 (14)	0.0155 (15)	−0.0133 (14)
C6	0.0425 (16)	0.0384 (14)	0.0414 (14)	−0.0050 (12)	0.0174 (12)	−0.0046 (12)
C7	0.117 (14)	0.153 (17)	0.076 (10)	0.041 (12)	0.041 (10)	0.031 (12)
C7′	0.120 (10)	0.091 (6)	0.093 (8)	0.019 (5)	0.078 (7)	0.024 (5)
C8	0.176 (5)	0.070 (3)	0.153 (4)	−0.023 (3)	0.081 (4)	−0.065 (3)
C9	0.080 (3)	0.108 (3)	0.078 (2)	−0.055 (2)	0.024 (2)	−0.008 (2)
C10	0.0428 (16)	0.0270 (13)	0.0323 (13)	0.0017 (12)	0.0090 (11)	−0.0036 (10)
C11	0.0492 (18)	0.0316 (14)	0.0450 (15)	0.0028 (12)	0.0155 (13)	−0.0015 (11)
C12	0.0476 (18)	0.0393 (15)	0.0483 (16)	0.0112 (14)	0.0119 (14)	−0.0007 (13)
C13	0.0540 (19)	0.0300 (14)	0.0437 (15)	0.0082 (13)	0.0052 (14)	0.0013 (12)
C14	0.0536 (19)	0.0338 (14)	0.0427 (15)	−0.0034 (14)	0.0108 (13)	0.0016 (12)
C15	0.0445 (17)	0.0330 (14)	0.0446 (15)	0.0048 (12)	0.0104 (13)	0.0003 (12)
C16	0.114 (4)	0.070 (3)	0.218 (5)	0.007 (3)	0.103 (4)	0.046 (3)
C17	0.098 (3)	0.0373 (18)	0.118 (3)	0.0069 (18)	0.012 (2)	−0.0150 (19)
C18	0.064 (2)	0.081 (2)	0.102 (3)	0.0197 (19)	0.040 (2)	0.008 (2)
C19	0.0358 (15)	0.0360 (14)	0.0352 (14)	0.0016 (11)	0.0137 (12)	−0.0010 (11)
C20	0.0503 (18)	0.0368 (15)	0.0420 (15)	0.0114 (13)	0.0184 (13)	0.0033 (12)
C21	0.097 (3)	0.0397 (17)	0.080 (2)	0.0060 (17)	0.042 (2)	−0.0069 (16)
C22	0.137 (4)	0.051 (2)	0.102 (3)	0.016 (2)	0.041 (3)	−0.023 (2)
N2	0.120 (3)	0.104 (3)	0.083 (2)	0.040 (2)	0.049 (2)	−0.016 (2)
C23	0.081 (3)	0.105 (3)	0.083 (3)	0.031 (2)	0.044 (2)	−0.009 (2)
C24	0.053 (2)	0.068 (2)	0.0656 (19)	0.0098 (16)	0.0309 (17)	−0.0069 (16)
C25	0.0397 (16)	0.0300 (13)	0.0355 (13)	0.0021 (11)	0.0103 (12)	−0.0025 (11)
C26	0.0461 (19)	0.0629 (18)	0.0377 (15)	0.0048 (14)	0.0068 (14)	0.0035 (13)
C27	0.065 (3)	0.081 (2)	0.0473 (18)	0.0097 (19)	−0.0024 (18)	0.0069 (16)
N1	0.0465 (17)	0.088 (2)	0.0683 (19)	0.0058 (15)	0.0021 (15)	−0.0030 (15)
C28	0.0429 (19)	0.069 (2)	0.065 (2)	−0.0062 (15)	0.0156 (16)	−0.0116 (16)
C29	0.0373 (17)	0.0508 (16)	0.0438 (15)	−0.0030 (13)	0.0085 (13)	−0.0009 (13)

C30	0.0390 (16)	0.0264 (12)	0.0327 (13)	0.0021 (11)	0.0113 (12)	−0.0001 (10)
C31	0.0369 (15)	0.0321 (13)	0.0336 (13)	0.0025 (11)	0.0086 (12)	0.0034 (11)
C32	0.0464 (18)	0.0527 (17)	0.0415 (15)	−0.0063 (14)	0.0114 (13)	0.0014 (13)
C33	0.061 (2)	0.066 (2)	0.0405 (16)	−0.0066 (16)	0.0071 (15)	−0.0051 (15)
N3	0.0672 (19)	0.0712 (17)	0.0400 (14)	0.0056 (15)	0.0121 (13)	0.0119 (13)
C34	0.071 (2)	0.074 (2)	0.0531 (19)	−0.0096 (18)	0.0193 (18)	0.0245 (17)
C35	0.0544 (19)	0.0562 (17)	0.0420 (16)	−0.0145 (15)	0.0063 (14)	0.0118 (13)
C39	0.0298 (14)	0.0268 (12)	0.0295 (12)	−0.0002 (10)	0.0121 (10)	0.0004 (10)
C40	0.0338 (14)	0.0273 (12)	0.0301 (12)	0.0010 (10)	0.0155 (11)	0.0016 (10)
C41	0.0353 (15)	0.0317 (13)	0.0267 (12)	0.0010 (11)	0.0121 (11)	0.0018 (10)
C42	0.0413 (16)	0.0303 (13)	0.0306 (12)	0.0005 (11)	0.0112 (11)	−0.0032 (10)
C43	0.0387 (15)	0.0262 (12)	0.0312 (12)	0.0016 (11)	0.0122 (11)	−0.0005 (10)
C44	0.0337 (14)	0.0266 (12)	0.0308 (12)	0.0006 (10)	0.0125 (11)	0.0008 (10)
O1	0.0972 (18)	0.0766 (15)	0.0572 (13)	−0.0113 (13)	0.0500 (13)	−0.0206 (12)
O2	0.1008 (19)	0.0757 (16)	0.0674 (14)	−0.0199 (13)	0.0314 (13)	−0.0427 (12)
O3	0.0823 (17)	0.0742 (15)	0.0899 (16)	−0.0475 (13)	0.0366 (14)	−0.0343 (13)
O4	0.0750 (16)	0.0400 (11)	0.0843 (15)	0.0002 (11)	0.0298 (13)	0.0160 (10)
O5	0.0787 (15)	0.0327 (10)	0.0642 (13)	0.0145 (10)	0.0059 (11)	0.0074 (9)
O6	0.0645 (15)	0.0540 (12)	0.0939 (16)	0.0241 (11)	0.0319 (13)	0.0107 (11)
O7	0.0390 (11)	0.0520 (11)	0.0433 (10)	0.0027 (9)	0.0160 (9)	0.0043 (8)
O8	0.1010 (17)	0.0377 (10)	0.0628 (12)	0.0081 (10)	0.0523 (12)	0.0098 (9)
O9	0.0454 (11)	0.0391 (9)	0.0359 (9)	0.0048 (8)	0.0217 (8)	0.0044 (7)
O10	0.161 (3)	0.105 (2)	0.0690 (18)	0.051 (2)	0.0637 (19)	0.0157 (15)
O11	0.127 (3)	0.152 (3)	0.0771 (19)	0.063 (2)	0.0488 (19)	−0.011 (2)

Geometric parameters (Å, º)

C1—C6	1.383 (3)	C19—C40	1.517 (3)
C1—C2	1.385 (3)	C20—C24	1.371 (4)
C1—C39	1.516 (3)	C20—C21	1.378 (4)
C2—C3	1.381 (3)	C21—C22	1.392 (5)
C2—H2	0.9300	C21—H21	0.9300
C3—C4	1.380 (4)	C22—N2	1.328 (5)
C3—O1	1.386 (3)	C22—H22	0.9300
C4—O2	1.377 (3)	N2—C23	1.314 (5)
C4—C5	1.389 (4)	C23—C24	1.374 (4)
C5—O3	1.368 (3)	C23—H23	0.9300
C5—C6	1.385 (3)	C24—H24	0.9300
C6—H6	0.9300	C25—C26	1.376 (3)
C7—O1	1.333 (11)	C25—C29	1.385 (3)
C7—H7A	0.9600	C25—C41	1.524 (3)
C7—H7B	0.9600	C26—C27	1.387 (4)
C7—H7C	0.9600	C26—H26	0.9300
C7′—O1	1.394 (7)	C27—N1	1.329 (4)
C7′—H7′1	0.9600	C27—H27	0.9300
C7′—H7′2	0.9600	N1—C28	1.324 (4)
C7′—H7′3	0.9600	C28—C29	1.375 (4)
C8—O2	1.421 (4)	C28—H28	0.9300

C8—H8A	0.9600	C29—H29	0.9300
C8—H8B	0.9600	C30—O7	1.218 (3)
C8—H8C	0.9600	C30—C31	1.501 (3)
C9—O3	1.413 (4)	C30—C44	1.515 (3)
C9—H9A	0.9600	C31—C32	1.375 (3)
C9—H9B	0.9600	C31—C35	1.377 (3)
C9—H9C	0.9600	C32—C33	1.377 (4)
C10—C15	1.382 (3)	C32—H32	0.9300
C10—C11	1.392 (3)	C33—N3	1.325 (4)
C10—C43	1.517 (3)	C33—H33	0.9300
C11—C12	1.387 (3)	N3—C34	1.321 (4)
C11—H11	0.9300	C34—C35	1.383 (4)
C12—O6	1.368 (3)	C34—H34	0.9300
C12—C13	1.383 (4)	C35—H35	0.9300
C13—C14	1.382 (4)	C39—C40	1.546 (3)
C13—O5	1.383 (3)	C39—C44	1.548 (3)
C14—O4	1.371 (3)	C39—H39	0.9800
C14—C15	1.395 (3)	C40—C41	1.551 (3)
C15—H15	0.9300	C40—H40	0.9800
C16—O4	1.402 (4)	C41—O9	1.420 (3)
C16—H16A	0.9600	C41—C42	1.531 (3)
C16—H16B	0.9600	C42—C43	1.534 (3)
C16—H16C	0.9600	C42—H42A	0.9700
C17—O5	1.412 (3)	C42—H42B	0.9700
C17—H17A	0.9600	C43—C44	1.542 (3)
C17—H17B	0.9600	C43—H43	0.9800
C17—H17C	0.9600	C44—H44	0.9800
C18—O6	1.413 (4)	O9—H9	0.8200
C18—H18A	0.9600	O10—H10A	0.864 (18)
C18—H18B	0.9600	O10—H10B	0.855 (19)
C18—H18C	0.9600	O11—H11B	0.851 (18)
C19—O8	1.212 (3)	O11—H11A	0.873 (17)
C19—C20	1.493 (3)		

C6—C1—C2	119.6 (2)	C21—C22—H22	118.2
C6—C1—C39	119.2 (2)	C23—N2—C22	117.3 (3)
C2—C1—C39	121.2 (2)	N2—C23—C24	123.4 (4)
C3—C2—C1	119.7 (2)	N2—C23—H23	118.3
C3—C2—H2	120.1	C24—C23—H23	118.3
C1—C2—H2	120.1	C20—C24—C23	119.4 (3)
C4—C3—C2	121.2 (2)	C20—C24—H24	120.3
C4—C3—O1	117.6 (2)	C23—C24—H24	120.3
C2—C3—O1	121.2 (2)	C26—C25—C29	116.6 (2)
O2—C4—C3	118.5 (3)	C26—C25—C41	122.5 (2)
O2—C4—C5	122.5 (3)	C29—C25—C41	121.0 (2)
C3—C4—C5	118.8 (2)	C25—C26—C27	119.4 (3)
O3—C5—C6	123.4 (3)	C25—C26—H26	120.3
O3—C5—C4	116.3 (2)	C27—C26—H26	120.3

C6—C5—C4	120.3 (2)	N1—C27—C26	124.3 (3)
C1—C6—C5	120.3 (2)	N1—C27—H27	117.9
C1—C6—H6	119.9	C26—C27—H27	117.9
C5—C6—H6	119.9	C28—N1—C27	115.6 (3)
O1—C7—H7A	109.5	N1—C28—C29	124.4 (3)
O1—C7—H7B	109.5	N1—C28—H28	117.8
H7A—C7—H7B	109.5	C29—C28—H28	117.8
O1—C7—H7C	109.5	C28—C29—C25	119.8 (3)
H7A—C7—H7C	109.5	C28—C29—H29	120.1
H7B—C7—H7C	109.5	C25—C29—H29	120.1
O1—C7′—H7′1	109.5	O7—C30—C31	119.3 (2)
O1—C7′—H7′2	109.5	O7—C30—C44	121.1 (2)
H7′1—C7′—H7′2	109.5	C31—C30—C44	119.4 (2)
O1—C7′—H7′3	109.5	C32—C31—C35	117.9 (2)
H7′1—C7′—H7′3	109.5	C32—C31—C30	119.6 (2)
H7′2—C7′—H7′3	109.5	C35—C31—C30	122.5 (2)
O2—C8—H8A	109.5	C31—C32—C33	119.0 (3)
O2—C8—H8B	109.5	C31—C32—H32	120.5
H8A—C8—H8B	109.5	C33—C32—H32	120.5
O2—C8—H8C	109.5	N3—C33—C32	123.9 (3)
H8A—C8—H8C	109.5	N3—C33—H33	118.0
H8B—C8—H8C	109.5	C32—C33—H33	118.0
O3—C9—H9A	109.5	C34—N3—C33	116.4 (2)
O3—C9—H9B	109.5	N3—C34—C35	124.2 (3)
H9A—C9—H9B	109.5	N3—C34—H34	117.9
O3—C9—H9C	109.5	C35—C34—H34	117.9
H9A—C9—H9C	109.5	C31—C35—C34	118.5 (3)
H9B—C9—H9C	109.5	C31—C35—H35	120.7
C15—C10—C11	119.7 (2)	C34—C35—H35	120.7
C15—C10—C43	121.2 (2)	C1—C39—C40	112.33 (18)
C11—C10—C43	119.1 (2)	C1—C39—C44	111.33 (17)
C12—C11—C10	120.1 (2)	C40—C39—C44	109.38 (18)
C12—C11—H11	120.0	C1—C39—H39	107.9
C10—C11—H11	120.0	C40—C39—H39	107.9
O6—C12—C13	115.2 (2)	C44—C39—H39	107.9
O6—C12—C11	124.4 (3)	C19—C40—C39	107.52 (18)
C13—C12—C11	120.4 (3)	C19—C40—C41	112.77 (18)
C14—C13—O5	120.7 (3)	C39—C40—C41	111.79 (18)
C14—C13—C12	119.5 (2)	C19—C40—H40	108.2
O5—C13—C12	119.8 (3)	C39—C40—H40	108.2
O4—C14—C13	115.7 (2)	C41—C40—H40	108.2
O4—C14—C15	123.8 (3)	O9—C41—C25	111.54 (18)
C13—C14—C15	120.5 (3)	O9—C41—C42	104.72 (18)
C10—C15—C14	119.8 (3)	C25—C41—C42	110.76 (19)
C10—C15—H15	120.1	O9—C41—C40	111.55 (18)
C14—C15—H15	120.1	C25—C41—C40	108.53 (18)
O4—C16—H16A	109.5	C42—C41—C40	109.70 (17)
O4—C16—H16B	109.5	C41—C42—C43	112.92 (19)

H16A—C16—H16B	109.5	C41—C42—H42A	109.0
O4—C16—H16C	109.5	C43—C42—H42A	109.0
H16A—C16—H16C	109.5	C41—C42—H42B	109.0
H16B—C16—H16C	109.5	C43—C42—H42B	109.0
O5—C17—H17A	109.5	H42A—C42—H42B	107.8
O5—C17—H17B	109.5	C10—C43—C42	113.21 (19)
H17A—C17—H17B	109.5	C10—C43—C44	110.89 (18)
O5—C17—H17C	109.5	C42—C43—C44	108.46 (18)
H17A—C17—H17C	109.5	C10—C43—H43	108.0
H17B—C17—H17C	109.5	C42—C43—H43	108.0
O6—C18—H18A	109.5	C44—C43—H43	108.0
O6—C18—H18B	109.5	C30—C44—C43	113.00 (19)
H18A—C18—H18B	109.5	C30—C44—C39	106.86 (18)
O6—C18—H18C	109.5	C43—C44—C39	110.83 (17)
H18A—C18—H18C	109.5	C30—C44—H44	108.7
H18B—C18—H18C	109.5	C43—C44—H44	108.7
O8—C19—C20	118.5 (2)	C39—C44—H44	108.7
O8—C19—C40	121.1 (2)	C7—O1—C3	119.0 (8)
C20—C19—C40	120.3 (2)	C3—O1—C7′	115.6 (4)
C24—C20—C21	118.3 (3)	C4—O2—C8	117.1 (3)
C24—C20—C19	123.4 (2)	C5—O3—C9	117.7 (2)
C21—C20—C19	118.3 (3)	C14—O4—C16	118.2 (2)
C20—C21—C22	117.9 (4)	C13—O5—C17	113.7 (2)
C20—C21—H21	121.0	C12—O6—C18	118.4 (2)
C22—C21—H21	121.0	C41—O9—H9	109.5
N2—C22—C21	123.6 (4)	H10A—O10—H10B	107 (3)
N2—C22—H22	118.2	H11B—O11—H11A	107 (2)

C6—C1—C2—C3	1.4 (4)	C30—C31—C35—C34	−179.0 (3)
C39—C1—C2—C3	−178.8 (2)	N3—C34—C35—C31	1.3 (5)
C1—C2—C3—C4	0.6 (4)	C6—C1—C39—C40	−119.8 (2)
C1—C2—C3—O1	179.6 (2)	C2—C1—C39—C40	60.3 (3)
C2—C3—C4—O2	−177.6 (3)	C6—C1—C39—C44	117.1 (2)
O1—C3—C4—O2	3.4 (4)	C2—C1—C39—C44	−62.8 (3)
C2—C3—C4—C5	−3.0 (4)	O8—C19—C40—C39	81.9 (3)
O1—C3—C4—C5	177.9 (3)	C20—C19—C40—C39	−94.0 (3)
O2—C4—C5—O3	−2.3 (4)	O8—C19—C40—C41	−41.8 (3)
C3—C4—C5—O3	−176.6 (3)	C20—C19—C40—C41	142.3 (2)
O2—C4—C5—C6	177.8 (3)	C1—C39—C40—C19	55.1 (2)
C3—C4—C5—C6	3.5 (4)	C44—C39—C40—C19	179.28 (18)
C2—C1—C6—C5	−0.9 (4)	C1—C39—C40—C41	179.42 (18)
C39—C1—C6—C5	179.3 (2)	C44—C39—C40—C41	−56.4 (2)
O3—C5—C6—C1	178.5 (3)	C26—C25—C41—O9	−0.6 (3)
C4—C5—C6—C1	−1.6 (4)	C29—C25—C41—O9	−178.7 (2)
C15—C10—C11—C12	−0.4 (4)	C26—C25—C41—C42	−116.8 (2)
C43—C10—C11—C12	176.2 (2)	C29—C25—C41—C42	65.1 (3)
C10—C11—C12—O6	179.8 (2)	C26—C25—C41—C40	122.7 (2)
C10—C11—C12—C13	−1.0 (4)	C29—C25—C41—C40	−55.4 (3)

O6—C12—C13—C14	−178.5 (2)	C19—C40—C41—O9	60.3 (2)
C11—C12—C13—C14	2.3 (4)	C39—C40—C41—O9	−61.0 (2)
O6—C12—C13—O5	3.8 (4)	C19—C40—C41—C25	−63.0 (2)
C11—C12—C13—O5	−175.4 (2)	C39—C40—C41—C25	175.68 (18)
O5—C13—C14—O4	−3.9 (4)	C19—C40—C41—C42	175.8 (2)
C12—C13—C14—O4	178.4 (2)	C39—C40—C41—C42	54.5 (2)
O5—C13—C14—C15	175.6 (2)	O9—C41—C42—C43	63.8 (2)
C12—C13—C14—C15	−2.1 (4)	C25—C41—C42—C43	−175.82 (19)
C11—C10—C15—C14	0.6 (4)	C40—C41—C42—C43	−56.0 (3)
C43—C10—C15—C14	−175.9 (2)	C15—C10—C43—C42	−46.1 (3)
O4—C14—C15—C10	−179.9 (2)	C11—C10—C43—C42	137.3 (2)
C13—C14—C15—C10	0.6 (4)	C15—C10—C43—C44	76.0 (3)
O8—C19—C20—C24	139.8 (3)	C11—C10—C43—C44	−100.6 (2)
C40—C19—C20—C24	−44.3 (4)	C41—C42—C43—C10	−178.05 (19)
O8—C19—C20—C21	−40.1 (4)	C41—C42—C43—C44	58.4 (2)
C40—C19—C20—C21	135.9 (3)	O7—C30—C44—C43	54.1 (3)
C24—C20—C21—C22	0.8 (4)	C31—C30—C44—C43	−130.5 (2)
C19—C20—C21—C22	−179.3 (3)	O7—C30—C44—C39	−68.1 (3)
C20—C21—C22—N2	−2.8 (6)	C31—C30—C44—C39	107.3 (2)
C21—C22—N2—C23	2.1 (6)	C10—C43—C44—C30	55.8 (3)
C22—N2—C23—C24	0.6 (6)	C42—C43—C44—C30	−179.26 (18)
C21—C20—C24—C23	1.7 (4)	C10—C43—C44—C39	175.7 (2)
C19—C20—C24—C23	−178.2 (3)	C42—C43—C44—C39	−59.4 (2)
N2—C23—C24—C20	−2.5 (5)	C1—C39—C44—C30	−52.7 (2)
C29—C25—C26—C27	0.3 (4)	C40—C39—C44—C30	−177.41 (18)
C41—C25—C26—C27	−177.9 (2)	C1—C39—C44—C43	−176.18 (19)
C25—C26—C27—N1	0.4 (5)	C40—C39—C44—C43	59.1 (2)
C26—C27—N1—C28	−0.7 (5)	C4—C3—O1—C7	−90 (2)
C27—N1—C28—C29	0.3 (5)	C2—C3—O1—C7	91 (2)
N1—C28—C29—C25	0.4 (4)	C4—C3—O1—C7′	−138.1 (10)
C26—C25—C29—C28	−0.6 (4)	C2—C3—O1—C7′	42.8 (10)
C41—C25—C29—C28	177.6 (2)	C3—C4—O2—C8	−122.9 (3)
O7—C30—C31—C32	29.5 (3)	C5—C4—O2—C8	62.8 (4)
C44—C30—C31—C32	−146.1 (2)	C6—C5—O3—C9	−18.4 (4)
O7—C30—C31—C35	−151.7 (3)	C4—C5—O3—C9	161.7 (3)
C44—C30—C31—C35	32.8 (3)	C13—C14—O4—C16	176.7 (3)
C35—C31—C32—C33	−0.6 (4)	C15—C14—O4—C16	−2.8 (4)
C30—C31—C32—C33	178.3 (2)	C14—C13—O5—C17	84.7 (3)
C31—C32—C33—N3	0.2 (4)	C12—C13—O5—C17	−97.6 (3)
C32—C33—N3—C34	0.9 (5)	C13—C12—O6—C18	169.1 (3)
C33—N3—C34—C35	−1.6 (5)	C11—C12—O6—C18	−11.8 (4)
C32—C31—C35—C34	−0.1 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*B*···O3	0.96	2.40	2.918 (5)	114
C9—H9*B*···O7^{i}	0.96	2.63	3.575 (4)	168

C11—H11···O8^{ii}	0.93	2.55	3.450 (3)	163
C16—H16*B*···O12	0.96	2.16	2.951 (15)	139
C24—H24···O12^{iii}	0.93	2.57	3.192 (15)	125
C27—H27···O4^{iv}	0.93	2.62	3.408 (4)	143
C44—H44···O11^{v}	0.98	2.56	3.464 (4)	153
O9—H9···O7^{ii}	0.82	2.27	2.941 (2)	139
O9—H9···O8	0.82	2.17	2.760 (2)	129
O11—H11*B*···N2	0.85 (2)	2.02 (2)	2.861 (4)	169 (3)
O11—H11*A*···O10^{vi}	0.87 (2)	1.91 (2)	2.771 (4)	171 (3)
O10—H10*A*···N3	0.86 (2)	2.00 (2)	2.857 (4)	175 (4)
O10—H10*B*···O1^{vi}	0.86 (2)	2.16 (3)	2.939 (3)	151 (5)

Symmetry codes: (i) $-x+3/2, y+1/2, -z+3/2$; (ii) $-x+1, -y, -z+1$; (iii) $-x+1/2, y+1/2, -z+3/2$; (iv) $x-1/2, -y-1/2, z-1/2$; (v) $-x+1/2, y-1/2, -z+3/2$; (vi) $-x+1, -y, -z+2$.