Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structures of *Z*–Gly–Aib–O^{-}·0.5Ca^{2+}·H$_2$O and *Z*–Gly–Aib–OH

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Received 8 June 2018
Accepted 24 July 2018

Edited by J. Ellena, Universidade de São Paulo, Brazil

Keywords: α-aminoisobutyric acid; dipeptide; peptide–calcium complex; hydrogen bonding; crystal structure.

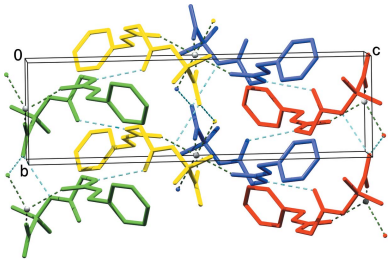
CCDC references: 1833520; 1833521

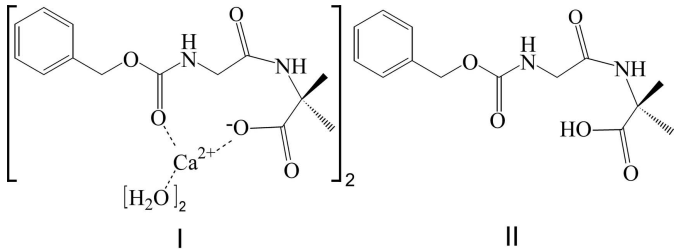
Supporting information: this article has supporting information at journals.iucr.org/e

Both deprotonated and neutral achiral title dipeptides assume similar structures of two conformations, which are related by a unit-cell inversion centre. Two molecules of both conformations of the metal-free neutral dipeptide are linked by two hydrogen bonds, while two molecules of both conformations of the ionized form coordinate a calcium ion in calcium(II) bis[2-(2-{[(benzyloxy)carbonyl]amino}acetamido)-2-methylpropanoate] monohydrate, 0.5Ca^{2+}·C$_{14}$H$_{17}$N$_2$O$_5^-$·0.5H$_2$O, which lies on an inversion centre and forms a distorted octahedral complex with the metal ion. These Ca^{II} complexes are connected in the crystal *via* hydrogen bonds in the *b*- and *c*-axis directions, whereas in the *a*-axis direction, they stack *via* apolar contacts. In the metal-free crystal, namely 2-(2-{[(benzyloxy)carbonyl]amino}acetamido)-2-methylpropanoic acid, C$_{14}$H$_{18}$N$_2$O$_5$, molecules are hydrogen bonded in the *a*- and *c*-axis directions, and stack in the *b*–axis direction *via* apolar contacts.

1. Chemical context

The presence of Gly and Aib (α-aminoisobutyric acid) combines a residue with the greatest conformational flexibility (Gly) with a severely restricted residue (Aib) because of the second methyl group attached to the Cα atom. The space available for Aib comprises the left-handed and right-handed helical region of the Ramachandran plot. Because of the absent side-chain atoms, Gly can adopt almost all conformations in contrast to all other residues. This makes Gly a conserved residue in peptides and proteins because a mutation of Gly could change the flexibility necessary for function or cause significant alteration of the secondary structure. Gly is incorporated in about half of all known peptaibol sequences (Stoppacher *et al.*, 2013) and frequently as a –Aib–Gly– dipeptide or as a –Aib–Gly–Aib– tripeptide unit. Peptides composed of Aib and Gly only show an enormous structural flexibility (Gessmann *et al.*, 1991; Gessmann, Brückner, Aivaliotis *et al.*, 2015; Gessmann, Brückner & Petratos, 2015) and therefore normally normally do not yield suitable sized crystals for structure analysis with X-rays.





I II

Table 1
Selected geometric parameters (Å, °) for **I**.

O—Ca_3	2.3200 (16)	Ca_3—O_4^{i}	2.3702 (18)
O2_2—Ca_3	2.2343 (16)		
O^{i}—Ca_3—O	180.0	O—Ca_3—O_4^{i}	93.21 (6)
O—Ca_3—O2_2	94.24 (6)	O2_2—Ca_3—O_4	86.27 (7)
O^{i}—Ca_3—O2_2	85.76 (6)	O2_2—Ca_3—O_4^{i}	93.73 (7)
O—Ca_3—O_4	86.79 (6)		

Symmetry code: (i) $-x, -y, -z+1$.

2. Structural commentary

In the crystal structure of **I** (*Z*–Gly–Aib-O^{-}·0.5Ca^{2+}·H$_2$O) all expected non H-atoms in both dipeptides were readily visible in the first electron-density map as the highest peaks. In addition, a heavy atom was detected, which at a later stage was identified as calcium by energy-dispersive X-ray spectroscopy (EDS), together with a water oxygen atom.

The backbone conformation of both peptides is very similar (Fig. 1). Gly is in the semi-extended conformation of both handednesses with torsion angles $\varphi = \mp 62.2\ (2)^{\circ}$, $\psi = \pm 153.37\ (18)^{\circ}$ in **I** and $\varphi = \mp 59.37\ (14)^{\circ}$, $\psi = \pm 153.66\ (10)^{\circ}$ in **II** (*Z*–Gly–Aib–OH). Aib adopts $\varphi = \pm 54.8\ (3)$ and $\pm 55.86\ (14)^{\circ}$ in **I** and **II**, respectively, while the values of ψ with both O atoms are $\mp 154.7\ (2)$ or $\pm 29.7\ (3)^{\circ}$ in **I** and $\mp 145.5\ (1)$ or $\pm 41.0\ (1)^{\circ}$ in **II** and therefore lies in the helical region of the Ramachandran plot. The *Z*-protection groups (benzyloxycarbonyl) adopt different conformations in **I** and **II** (Fig. 2). The r.m.s. deviation for the non-hydrogen atoms of Gly and Aib is 0.2 Å, whereby the most distant carbon atoms of the *Z* protection group of the two peptides are 4.75 Å apart in the superposition of the non-hydrogen atoms of the amino acid residues.

The similar backbone conformation is also visible in Fig. 2. Structure **I** crystallized with a water molecule and a half calcium ion (lying on a special position) per peptide molecule while **II**, which crystallized without any solvent molecules, forms two direct hydrogen bonds between two inversion-related molecules. In **I** the Ca^{2+} ion is coordinated by the carbonyl group of *Z* and the deprotonated carboxylate group of Aib2. It is worth noting that in both crystal structures the same oxygen atoms participate in the hydrogen bonding and coordination interactions (Fig. 2). One dipeptide molecule and its inverted mate provide four of the six ligand atoms for the calcium ion. The remaining two ligands are two water molecules, which are also related *via* the inversion centres. The metal coordination parameters are listed in Table 1. As the calcium ion sits on the inversion centre, the values of the fifteen angles between the ligands are reduced to seven values, each one occurring twice and the 180° angle between the metal ion and inverted atoms occurs three times.

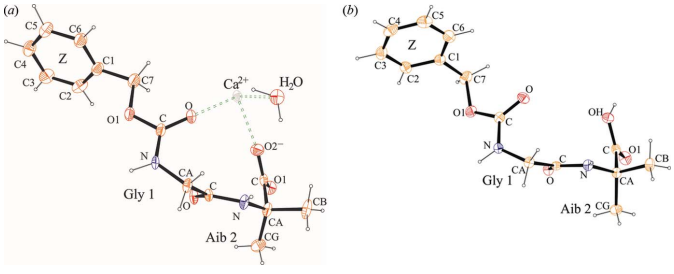


Figure 1
The molecular structures of *Z*–Gly–Aib–OH showing the 50% probability displacement ellipsoids and simplified atom numbering (Farrugia, 2012). (*a*) The asymmetric unit of the complex with Ca^{2+} and H$_2$O (I). One metal ion is coordinated by two symmetry-inverted peptides and water molecules. (*b*) The structure of the neutral dipeptide *Z*-Gly-Aib-OH (II).

3. Supramolecular features

The crystal packing is quite different in **I** and **II**. In the crystal structure of **I**, there are four hydrogen bonds between symmetry-related molecules (Table 2). The first hydrogen bond connects the NH group of Gly to the carbonyl group of a

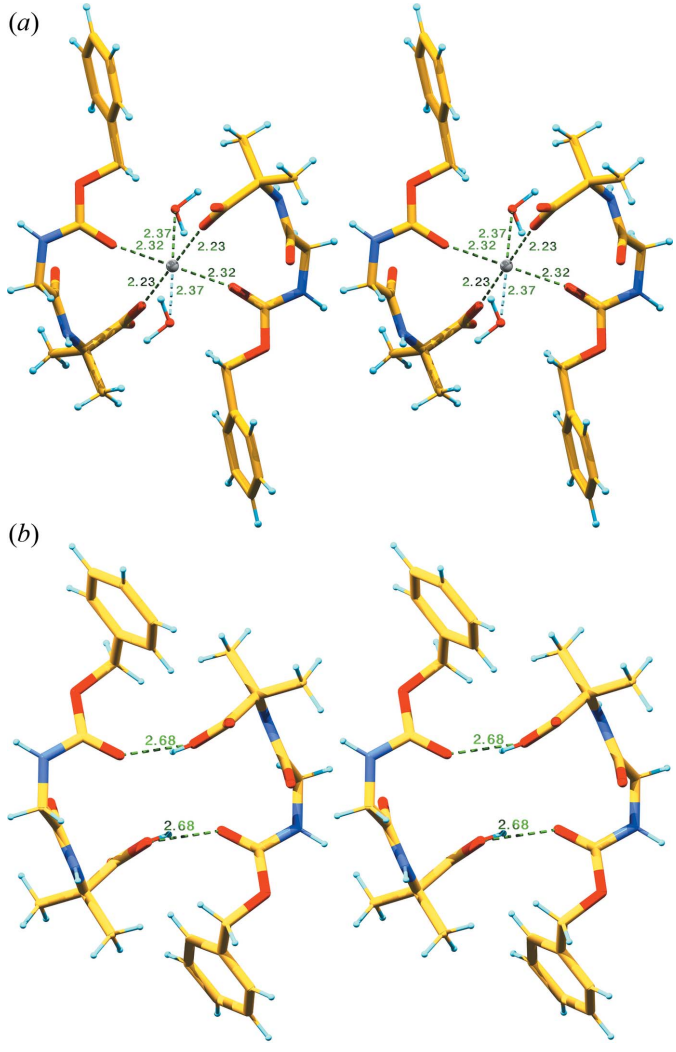


Figure 2
Wall-eyed stereo figure of two inversion-related molecules of the metal-bound structure **I** (*a*) with the Ca^{2+} ion in grey and the free, neutral dipeptide **II** (*b*). Distances for the Ca^{2+} co-ordination (*a*) and hydrogen bonds (*b*) are shown in Å.

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

D—H···A	D—H	H···A	D···A	D—H···A
N_1—H_1···O_1^{ii}	0.90 (3)	1.91 (3)	2.800 (2)	169 (3)
N_2—H_2···O1_2^{iii}	0.73 (4)	2.18 (4)	2.864 (2)	156 (4)
O_4—H1_4···O1_2^{iii}	1.00 (5)	1.75 (6)	2.741 (3)	172 (5)
O_4—H2_4···O^{iv}	1.07 (6)	1.99 (6)	3.053 (2)	176 (6)

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

Table 3
Hydrogen-bond geometry (Å, °) for **II**.

D—H···A	D—H	H···A	D···A	D—H···A
N_1—H_1···O_1^{i}	0.92 (2)	2.12 (2)	3.0298 (14)	168.7 (16)
N_2—H_2···O_1^{ii}	0.85 (2)	2.09 (2)	2.9304 (15)	167.3 (18)
O*H*_2—H*H*_2···O^{iii}	0.88 (2)	1.82 (2)	2.6789 (14)	162 (2)

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

Gly residue, which belongs to a symmetry-related molecule, *via* a screw axis. In Fig. 3*a* the green and yellow molecules are hydrogen bonded in a zigzag manner down the *b* axis and the blue and red symmetry-related ones in zigzag manner along the *b* axis. The second intermolecular hydrogen bond is formed between the NH group of Aib and the carbonyl group of Aib of a *y*-translated (+1 or −1) molecule, shown as pairs of the same color in Fig. 3*a*. The same carbonyl group accepts the hydrogen bond from the water molecule. As this water molecule also coordinates the calcium ion, which is bonded to the translated and inverted molecules, multiple bonded molecule layers are formed in the *bc* plane. These single molecule layers stack together *via* apolar contacts between the *Z* protection groups and the Aib side chains, along the *a*-axis direction (Fig. 4*a*). The shortest distance between two symmetry-related rings is 3.54 Å and 3.91 Å between the Aib side chain and a symmetry-related ring. The staggering angles between the *Z* rings of successive sheets are 119.9° while the distance between the centre of the rings is 4.79 Å. Finally, in one layer the rings of the *Z* protection groups are staggered parallel with a distance of 5.57 Å, which is equal to the length of the *b* axis.

In the crystal structure of **II**, one molecule (the left green molecule in Fig. 3*b*) is hydrogen bonded to four other molecules. The carbonyl group of *Z* and the C-terminal OH group of Aib are hydrogen bonded to the same molecule (Fig. 3*b*) and to the left green molecule. The NH group of Gly is hydrogen bonded to the carbonyl group of Gly1 of the right

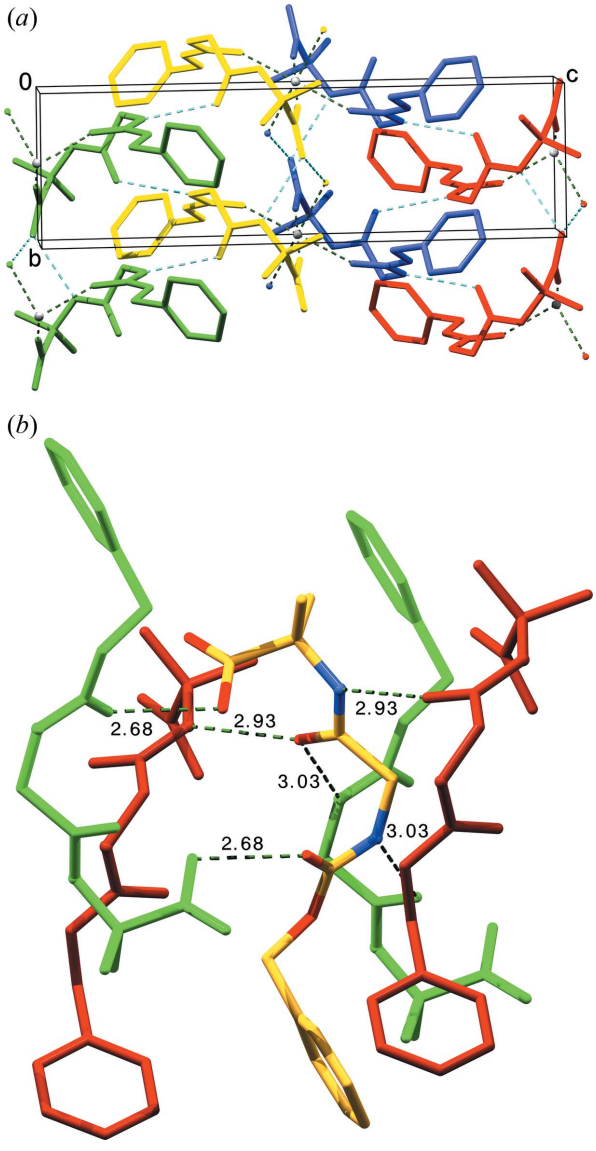


Figure 3
Molecular packing of **I** and **II** showing the bonding to neighbouring molecules. (*a*) In **I**, the Ca^{2+} ions are shown as a grey spheres at the inversion centres of the unit cell while the other spheres signify water molecules. The content of two *y*-translated unit cells is shown. Hydrogen bonds are shown in cyan and the metal coordination bonds are shown in dark green. (*b*) The bonding of the central molecule **II** (coloured atoms) to four neighbouring molecules *via* hydrogen bonds.

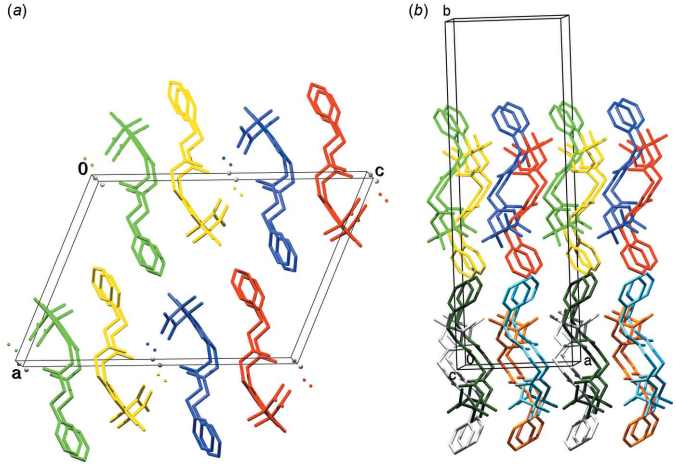


Figure 4
Molecular packing of **I** and **II** showing the assembly in the crystal. (*a*) The content of two *x*- and two *y*-translated unit cells is shown. (*b*) The content of two *x*- and two *z*-translated unit cells is shown. The view is along the *b* axis in (*a*) and down the *c* axis in (*b*).

Table 4
Experimental details.

	I	II
Crystal data		
Chemical formula	$0.5Ca^{2+}\cdot C_{14}H_{17}N_2O_5^-\cdot 0.5H_2O$	$C_{14}H_{18}N_2O_5$
M_r	331.35	294.30
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, *Pbca*
Temperature (K)	100	100
a, b, c (Å)	14.996 (3), 5.5740 (11), 20.607 (4)	9.5260 (19), 28.608 (6), 10.270 (2)
α, β, γ (°)	90, 112.55 (3), 90	90, 90, 90
V (Å^3)	1590.8 (6)	2798.8 (10)
Z	4	8
Radiation type	Synchrotron, λ = 0.59038 Å	Cu $K\alpha$
μ (mm^{-1})	0.14	0.90
Crystal size (mm)	0.18 × 0.06 × 0.03	0.2 × 0.1 × 0.05
Data collection		
Diffractometer	Pilatus3 6M detector on beamline I24 of Diamond Light Source	Bruker Venture D8
Absorption correction	–	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	–	0.90, 0.96
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28071, 4716, 3941	44443, 2843, 2522
R_{int}	0.128	0.067
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.750	0.634
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.078, 0.214, 1.06	0.036, 0.090, 1.09
No. of reflections	4716	2843
No. of parameters	279	262
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.27, −0.56	0.27, −0.21

Computer programs: *PROTEUM2* and *SAINT* (Bruker, 2008), *XDS* (Kabsch, 2010), *SHELXS86* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *COOT* (Emsley *et al.*, 2010), *SwissPDBViewer* (Guex & Peitsch, 1997), *CHEMDRAW* (Mills, 2006), *ORTEPIII* (Burnett & Johnson, (1996), *ORTEP-3 for Windows* (Farrugia, 2012), *POVRAY* (Persistence of Vision, 2004), *pyMOL* (DeLano, 2002) and *publCIF* (Westrip, 2010).

green molecule in Fig. 3*b* and Table 3. From the latter molecule the NH-group of Gly1 is a hydrogen-bond donor to the carbonyl group of Gly1 of the central molecule. The same carbonyl group of Gly1 is hydrogen bonded to the NH group of Aib2 of the left red molecule, while the NH group of the central molecule is hydrogen bonded to the carbonyl group of Aib2 of the right red molecule in Fig. 3*b*. From the same NH group of Aib2 there is a hydrogen-bonding distance of 3.31 Å to the carbonyl group of the same red molecule. The N—H···O distance is 2.95 Å and the N—H···O angle is 107°, which are too long and too acute for hydrogen bonding; thus this carbonyl oxygen, which is the only potential hydrogen-bond former, remains non-bonded. Fig. 4*b* shows all eight symmetry-related molecules of the space group in different colors, zooming out from the central yellow molecule in Fig. 3*b*. Layers of hydrogen-bonded molecules are formed in the *ac* plane, which stack together with the next layers along the *b* axis *via* apolar contacts. The rings of the *Z* groups interact between the layers through π–π stacking with an angle 120.1° and a distance between the centres of the rings of 5.73 Å. The shortest van der Waals distance between two layers is 3.86 Å, measured between two ring atoms of the *Z* protection groups. The staggering angles between the *Z* rings inside a sheet are 111.0° and the distance between the centres of the rings is 5.31 Å.

4. Database survey

The crystal structure of *t*-butyloxycarbonyl–Gly–Aib–OH has been determined [CSD (Groom *et al.*, 2016) refcode CALFEA; Smith *et al.*, 1981]. The dipeptide assumes a different structure to the ones reported here. The N-terminal protection group points in opposite directions compared to the benzyloxycarbonyl (*Z*) of the present work. In addition, the crystal structure of an Aib containing peptide complexed with metal ions has also been determined, namely H–Aib–Gly–OH complexed with copper(II) (CSD refcode MUYNID; Tiliakos *et al.*, 2003). In this structure, one peptide takes part in the coordination of three copper ions and a metal ion is bonded to three peptides. This coordination is quite different from the one we have observed in the present work, where two peptide anions coordinate one calcium ion.

5. Synthesis and crystallization

The dipeptide *Z*–Gly–Aib–O^{t}Bu (O^{t}Bu, *tert*-butoxy) was synthesized in DMF (dimethylformamide) from *Z*–Gly–OH (purchased from Bachem) and H–Aib–O^{t}Bu using HOB^t and DCC (*N*,*N*′-dicyclohexylcarbodiimide) as coupling reagents. *Z*–Gly–Aib–OH was obtained from *Z*–Gly–Aib–O^{t}Bu with removal of the *tert*-butyl-protecting group by disolving in

DCM (dichloromethane) and by adding TFA (trifluoro-acetic acid). The peptides were crystallized by slow evaporation from a methanol/water mixture (*v*:*v* = 50:50). Crystals of **I** and **II** were selected from different crystallization batches. In one crystallization batch, a small amount of calcium salt was present in the solvent, yielding the peptide–metal complex.

6. Measurement and refinement

Both crystals measured have a tiny third dimension. They were mounted on cryoloops without cryoprotectant and were kept in place with a minimal amount of vacuum grease and measured at 100 K. Photographs of the crystals are provided in Fig. S1 of the supporting information.

Diffraction data for the calcium-bound peptide (**I**) were collected on the microfocus beamline I24 (Evans *et al.*, 2011) of Diamond Light Source in Didcot, England, using a Pilatus3 6M detector (Dectris Ltd, Baden, Switzerland). A dataset of 1800 images covering 360° of rotation was collected in the resolution range 30.0–0.67 Å. 28517 reflections were recorded in total. Of these observed reflections, 4976 were unique. The data were integrated and scaled using the software package XDS (Kabsch, 2010). The initial space group $P2_1/n$ was changed to the conventional space group $P2_1/c$ with the *CCP4* programme suite (Winn *et al.*, 2011).

One single plate of the neutral peptide (**II**) was used for data collection at our in-house diffractometer and data were integrated and scaled with the Bruker software (Bruker, 2008). Crystal data, data collection and structure refinement details for both crystals are summarized in Table 4.

All non-hydrogen atoms and one water oxygen in **I** were detected in the direct methods solutions as highest peaks. The highest peak in **I** (583:220 to the second highest peak in relative units) in a special position was interpreted from this height as a metal ion. The electron density of the metal ion pointed to more than double the number of electrons as oxygen and was assumed to be calcium. Additional supporting evidence came from the octahedral arrangement of six oxygen atoms around the metal. The central metal ion was unequivocally identified as calcium *via* Energy-dispersive X-ray spectroscopy (EDS, Jeol Scanning Microscope 7000 F) by the occurrence of the characteristic peaks at 0.3 (*L*), 3.7 ($K\alpha$) and 4.0 ($K\beta$) keV. The spectrum is shown in the supporting information section (Fig. S2).

Acknowledgements

We are grateful to Dr Danny Axford for assistance at Diamond beamline I24 during the data collection and to Aleka Manousaki for measurements using the EDS facility of IESL–FORTH, Heraklion.

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supporting information

Acta Cryst. (2018). E**74**, 1173-1177 [https://doi.org/10.1107/S2056989018010745]

Crystal structures of *Z*–Gly–Aib–O^{-}·0.5Ca^{2+}·H$_2$O and *Z*–Gly–Aib–OH

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Computing details

Data collection: *PROTEUM2* (Bruker, 2008) for (II). Cell refinement: *XDS* (Kabsch, 2010) for (I); *SAINT* (Bruker, 2008) for (II). Data reduction: *XDS* (Kabsch, 2010) for (I); *SAINT* (Bruker, 2008) for (II). For both structures, program(s) used to solve structure: *SHELXS86* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *COOT* (Emsley *et al.*, 2010), *SwissPDBViewer* (Guex & Peitsch, 1997). Software used to prepare material for publication: *CHEMDRAW* (Mills, 2006), *ORTEPIII* (Burnett & Johnson, (1996), *ORTEP-3 for Windows* (Farrugia, 2012), *POVRAY* (Persistence of Vision, 2004), *pyMOL* (DeLano, 2002) for (I); *publCIF* (Westrip, 2010) for (II).

Calcium(II) bis[2-(2-{[(benzyloxy)carbonyl]amino}acetamido)-2-methylpropanoate] monohydrate (I)

Crystal data

0.5Ca^{2+}·C$_{14}$H$_{17}$N$_2$O$_5^{-}$·0.5H$_2$O
M_r = 331.35
Monoclinic, $P2_1/c$
a = 14.996 (3) Å
b = 5.5740 (11) Å
c = 20.607 (4) Å
β = 112.55 (3)°
V = 1590.8 (6) Å^3
Z = 4
$F(000)$ = 700
D_x = 1.384 Mg m^{-3}
Synchrotron radiation, λ = 0.59038 Å
Cell parameters from 4716 reflections
θ = 0.7–30°
μ = 0.14 mm^{-1}
T = 100 K
Brick, colourless
0.18 × 0.06 × 0.03 mm

Data collection

Pilatus3 6M detector on beamline I24 of Diamond Light Source diffractometer
φ–scans
28071 measured reflections
4716 independent reflections
3941 reflections with $I > 2\sigma(I)$
R_{int} = 0.128
θ_{max} = 26.3°, θ_{min} = 1.2°
h = −19→19
k = −7→7
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.214
S = 1.06
4716 reflections
279 parameters
0 restraints
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1201P)^2 + 1.1073P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.31673 (15)	−0.0754 (5)	0.26150 (12)	0.0259 (5)
C2	−0.33650 (17)	0.1220 (4)	0.21663 (15)	0.0312 (5)
H2	−0.285 (2)	0.245 (6)	0.2275 (17)	0.030 (8)*
C3	−0.42513 (18)	0.1428 (4)	0.16126 (15)	0.0310 (5)
H3	−0.435 (2)	0.268 (6)	0.1291 (17)	0.026 (7)*
C4	−0.49525 (17)	−0.0319 (5)	0.15046 (14)	0.0291 (5)
H4	−0.558 (2)	−0.007 (6)	0.1123 (19)	0.032 (8)*
C5	−0.47630 (16)	−0.2259 (5)	0.19555 (14)	0.0294 (5)
H5	−0.527 (2)	−0.367 (6)	0.1923 (18)	0.033 (8)*
C6	−0.38720 (16)	−0.2489 (5)	0.25022 (13)	0.0276 (5)
H6	−0.373 (3)	−0.377 (8)	0.283 (2)	0.049 (11)*
C7	−0.21987 (17)	−0.1049 (5)	0.31939 (13)	0.0315 (5)
H71	−0.212 (2)	−0.252 (6)	0.3567 (19)	0.036 (8)*
H72	−0.188 (3)	0.053 (7)	0.343 (2)	0.039 (9)*
O1	−0.15523 (11)	−0.1822 (3)	0.28613 (8)	0.0257 (4)
C	−0.06325 (14)	−0.2165 (4)	0.32957 (10)	0.0191 (4)
O	−0.03479 (11)	−0.1969 (3)	0.39390 (8)	0.0225 (3)
N_1	−0.00723 (12)	−0.2754 (3)	0.29610 (9)	0.0172 (3)
H_1	−0.033 (2)	−0.300 (6)	0.2494 (18)	0.027 (7)*
CA_1	0.09504 (14)	−0.3025 (4)	0.33509 (11)	0.0182 (4)
HA1_1	0.1043 (18)	−0.429 (5)	0.3668 (15)	0.014 (6)*
HA2_1	0.125 (2)	−0.342 (6)	0.3040 (17)	0.025 (7)*
C_1	0.14182 (13)	−0.0694 (3)	0.37032 (10)	0.0157 (4)
O_1	0.10901 (11)	0.1280 (3)	0.34679 (8)	0.0207 (3)
N_2	0.22149 (12)	−0.0984 (3)	0.42780 (9)	0.0179 (3)
H_2	0.229 (3)	−0.223 (8)	0.440 (2)	0.043 (10)*
CA_2	0.27454 (14)	0.1042 (4)	0.47072 (11)	0.0197 (4)
CB_2	0.34920 (18)	−0.0022 (4)	0.53830 (14)	0.0303 (6)
HB1_2	0.388 (2)	−0.101 (5)	0.5219 (16)	0.021 (7)*
HB2_2	0.316 (2)	−0.089 (5)	0.5596 (15)	0.018 (6)*
HB3_2	0.385 (3)	0.117 (8)	0.571 (2)	0.055 (11)*
CG_2	0.32397 (17)	0.2482 (4)	0.43140 (14)	0.0268 (5)
HG1_2	0.366 (2)	0.152 (6)	0.4210 (17)	0.027 (7)*
HG2_2	0.283 (2)	0.306 (6)	0.3910 (18)	0.030 (8)*
HG3_2	0.361 (3)	0.369 (7)	0.463 (2)	0.042 (9)*
C_2	0.20651 (14)	0.2604 (4)	0.49373 (10)	0.0184 (4)
O1_2	0.22838 (12)	0.4764 (3)	0.50765 (9)	0.0242 (3)
O2_2	0.13732 (12)	0.1601 (3)	0.50138 (9)	0.0259 (4)
Ca_3	0.0000	0.0000	0.5000	0.01693 (17)

O_4	0.09395 (13)	−0.3401 (3)	0.55420 (9)	0.0267 (4)
H1_4	0.148 (4)	−0.398 (10)	0.541 (3)	0.079 (15)*
H2_4	0.071 (4)	−0.503 (10)	0.570 (3)	0.095*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0171 (9)	0.0340 (12)	0.0251 (10)	0.0014 (8)	0.0064 (8)	−0.0075 (9)
C2	0.0234 (10)	0.0269 (11)	0.0442 (15)	−0.0030 (8)	0.0140 (10)	−0.0072 (10)
C3	0.0266 (11)	0.0263 (11)	0.0388 (13)	0.0048 (8)	0.0111 (10)	0.0031 (10)
C4	0.0221 (10)	0.0285 (11)	0.0311 (12)	0.0046 (8)	0.0041 (9)	−0.0030 (9)
C5	0.0203 (10)	0.0299 (11)	0.0340 (12)	−0.0023 (8)	0.0059 (9)	−0.0061 (9)
C6	0.0224 (10)	0.0313 (12)	0.0267 (11)	0.0013 (8)	0.0067 (9)	−0.0006 (9)
C7	0.0197 (10)	0.0482 (15)	0.0251 (11)	0.0010 (9)	0.0070 (9)	−0.0115 (10)
O1	0.0161 (7)	0.0405 (9)	0.0164 (7)	0.0025 (6)	0.0018 (6)	−0.0046 (6)
C	0.0173 (9)	0.0199 (9)	0.0154 (8)	−0.0030 (6)	0.0010 (7)	−0.0015 (7)
O	0.0214 (7)	0.0287 (8)	0.0137 (7)	−0.0026 (5)	0.0026 (5)	−0.0029 (6)
N_1	0.0151 (7)	0.0202 (8)	0.0099 (7)	−0.0005 (5)	−0.0021 (6)	−0.0024 (6)
CA_1	0.0155 (8)	0.0174 (9)	0.0160 (8)	0.0001 (6)	−0.0003 (7)	−0.0048 (7)
C_1	0.0171 (8)	0.0152 (8)	0.0112 (7)	−0.0010 (6)	0.0016 (6)	−0.0017 (6)
O_1	0.0245 (7)	0.0154 (7)	0.0137 (6)	0.0009 (5)	−0.0022 (5)	0.0020 (5)
N_2	0.0178 (7)	0.0106 (7)	0.0175 (8)	−0.0007 (5)	−0.0018 (6)	0.0010 (6)
CA_2	0.0169 (8)	0.0154 (8)	0.0188 (9)	−0.0015 (6)	−0.0019 (7)	0.0005 (7)
CB_2	0.0233 (11)	0.0206 (10)	0.0292 (12)	−0.0004 (7)	−0.0097 (9)	0.0016 (8)
CG_2	0.0231 (10)	0.0229 (10)	0.0349 (12)	−0.0037 (8)	0.0118 (9)	−0.0022 (9)
C_2	0.0198 (9)	0.0178 (8)	0.0116 (8)	−0.0025 (6)	−0.0006 (7)	0.0020 (6)
O1_2	0.0279 (8)	0.0148 (7)	0.0284 (8)	−0.0012 (5)	0.0090 (7)	−0.0002 (6)
O2_2	0.0265 (8)	0.0286 (8)	0.0240 (8)	−0.0100 (6)	0.0113 (6)	−0.0071 (6)
Ca_3	0.0189 (3)	0.0166 (3)	0.0123 (3)	−0.00324 (17)	0.0026 (2)	−0.00157 (17)
O_4	0.0304 (8)	0.0194 (7)	0.0290 (8)	0.0002 (6)	0.0101 (7)	0.0000 (6)

Geometric parameters (Å, º)

C1—C6	1.385 (3)	CA_1—HA2_1	0.94 (3)
C1—C2	1.394 (4)	C_1—O_1	1.227 (2)
C1—C7	1.494 (3)	C_1—N_2	1.331 (2)
C2—C3	1.385 (4)	N_2—CA_2	1.467 (3)
C2—H2	0.99 (3)	N_2—H_2	0.73 (4)
C3—C4	1.387 (4)	CA_2—CG_2	1.520 (3)
C3—H3	0.94 (3)	CA_2—CB_2	1.533 (3)
C4—C5	1.382 (4)	CA_2—C_2	1.547 (3)
C4—H4	0.98 (3)	CB_2—HB1_2	0.95 (3)
C5—C6	1.384 (3)	CB_2—HB2_2	0.92 (3)
C5—H5	1.08 (3)	CB_2—HB3_2	0.96 (5)
C6—H6	0.95 (4)	CG_2—HG1_2	0.91 (3)
C7—O1	1.451 (3)	CG_2—HG2_2	0.89 (3)
C7—H71	1.10 (4)	CG_2—HG3_2	0.96 (4)
C7—H72	1.03 (4)	C_2—O2_2	1.241 (3)

O1—C	1.339 (2)	C_2—O1_2	1.252 (2)
C—O	1.232 (2)	O2_2—Ca_3	2.2343 (16)
C—N_1	1.317 (3)	Ca_3—O2_2^{i}	2.2342 (16)
O—Ca_3	2.3200 (16)	Ca_3—O^{i}	2.3200 (16)
N_1—CA_1	1.441 (2)	Ca_3—O_4^{i}	2.3702 (18)
N_1—H_1	0.90 (3)	Ca_3—O_4	2.3702 (17)
CA_1—C_1	1.522 (3)	O_4—H1_4	1.00 (5)
CA_1—HA1_1	0.93 (3)	O_4—H2_4	1.07 (6)

C6—C1—C2	119.0 (2)	C_1—N_2—H_2	113 (3)
C6—C1—C7	120.2 (2)	CA_2—N_2—H_2	123 (3)
C2—C1—C7	120.7 (2)	N_2—CA_2—CG_2	110.34 (19)
C3—C2—C1	120.4 (2)	N_2—CA_2—CB_2	106.75 (16)
C3—C2—H2	122.9 (19)	CG_2—CA_2—CB_2	110.68 (19)
C1—C2—H2	116.7 (19)	N_2—CA_2—C_2	110.43 (16)
C2—C3—C4	120.1 (2)	CG_2—CA_2—C_2	112.33 (17)
C2—C3—H3	119.1 (19)	CB_2—CA_2—C_2	106.08 (19)
C4—C3—H3	121 (2)	CA_2—CB_2—HB1_2	103.5 (18)
C5—C4—C3	119.6 (2)	CA_2—CB_2—HB2_2	107.3 (18)
C5—C4—H4	122.3 (19)	HB1_2—CB_2—HB2_2	112 (3)
C3—C4—H4	118.0 (19)	CA_2—CB_2—HB3_2	113 (3)
C4—C5—C6	120.3 (2)	HB1_2—CB_2—HB3_2	113 (3)
C4—C5—H5	124.7 (18)	HB2_2—CB_2—HB3_2	107 (3)
C6—C5—H5	115.0 (18)	CA_2—CG_2—HG1_2	110 (2)
C1—C6—C5	120.5 (2)	CA_2—CG_2—HG2_2	113 (2)
C1—C6—H6	117 (2)	HG1_2—CG_2—HG2_2	107 (3)
C5—C6—H6	122 (2)	CA_2—CG_2—HG3_2	107 (2)
O1—C7—C1	106.06 (19)	HG1_2—CG_2—HG3_2	107 (3)
O1—C7—H71	101.6 (18)	HG2_2—CG_2—HG3_2	114 (3)
C1—C7—H71	116.7 (18)	O2_2—C_2—O1_2	124.2 (2)
O1—C7—H72	102 (2)	O2_2—C_2—CA_2	117.83 (18)
C1—C7—H72	115 (2)	O1_2—C_2—CA_2	117.76 (18)
H71—C7—H72	113 (3)	C_2—O2_2—Ca_3	171.82 (15)
C—O1—C7	115.48 (17)	O2_2^{i}—Ca_3—O2_2	180.0
O—C—N_1	123.95 (19)	O2_2^{i}—Ca_3—O^{i}	94.24 (6)
O—C—O1	123.3 (2)	O2_2—Ca_3—O^{i}	85.75 (6)
N_1—C—O1	112.70 (17)	O^{i}—Ca_3—O	180.0
C—O—Ca_3	156.34 (15)	O—Ca_3—O2_2	94.24 (6)
C—N_1—CA_1	119.38 (17)	O^{i}—Ca_3—O2_2	85.76 (6)
C—N_1—H_1	120 (2)	O—Ca_3—O_4	86.79 (6)
CA_1—N_1—H_1	121 (2)	O—Ca_3—O_4^{i}	93.21 (6)
N_1—CA_1—C_1	111.96 (16)	O2_2—Ca_3—O_4	86.27 (7)
N_1—CA_1—HA1_1	108.0 (16)	O2_2—Ca_3—O_4^{i}	93.73 (7)
C_1—CA_1—HA1_1	113.1 (17)	O2_2^{i}—Ca_3—O_4	93.73 (7)
N_1—CA_1—HA2_1	109.4 (18)	O2_2—Ca_3—O_4	86.27 (7)
C_1—CA_1—HA2_1	105.6 (19)	O^{i}—Ca_3—O_4	93.21 (6)
HA1_1—CA_1—HA2_1	109 (3)	O—Ca_3—O_4	86.79 (6)
O_1—C_1—N_2	123.21 (18)	O_4^{i}—Ca_3—O_4	180.0

O_1—C_1—CA_1	122.38 (17)	Ca_3—O_4—H1_4	122 (3)
N_2—C_1—CA_1	114.41 (17)	Ca_3—O_4—H2_4	128 (3)
C_1—N_2—CA_2	122.52 (17)	H1_4—O_4—H2_4	101 (4)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N_1—H_1···O_1^{ii}	0.90 (3)	1.91 (3)	2.800 (2)	169 (3)
N_2—H_2···O1_2^{iii}	0.73 (4)	2.18 (4)	2.864 (2)	156 (4)
O_4—H1_4···O1_2^{iii}	1.00 (5)	1.75 (6)	2.741 (3)	172 (5)
O_4—H2_4···O^{iv}	1.07 (6)	1.99 (6)	3.053 (2)	176 (6)

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

2-(2-{[(Benzyloxy)carbonyl]amino}acetamido)-2-methylpropanoic acid (II)

Crystal data

$C_{14}H_{18}N_2O_5$
M_r = 294.30
Orthorhombic, *Pbca*
a = 9.5260 (19) Å
b = 28.608 (6) Å
c = 10.270 (2) Å
V = 2798.8 (10) Å^3
Z = 8
F(000) = 1248
D_x = 1.397 Mg m^{-3}
Cu *Kα* radiation, λ = 1.54178 Å
Cell parameters from 312 reflections
θ = 3.1–44.6°
μ = 0.90 mm^{-1}
T = 100 K
Plate, colourless
0.2 × 0.1 × 0.05 mm

Data collection

Bruker Venture D8 diffractometer
profile data from φ or ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
T_{min} = 0.90, T_{max} = 0.96
44443 measured reflections
2843 independent reflections
2522 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 77.7°, θ_{min} = 3.1°
h = −11→12
k = −36→36
l = −12→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.090
S = 1.09
2843 reflections
262 parameters
0 restraints
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 1.1904P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.96843 (14)	0.33651 (5)	0.17611 (13)	0.0204 (3)
C2	0.91669 (14)	0.31322 (5)	0.06711 (13)	0.0220 (3)
H2	0.8387 (18)	0.3269 (6)	0.0193 (16)	0.027 (4)*
C3	0.97610 (15)	0.27138 (5)	0.02714 (14)	0.0257 (3)
H3	0.9404 (18)	0.2549 (6)	−0.0516 (17)	0.030 (4)*
C4	1.08684 (15)	0.25222 (5)	0.09657 (16)	0.0287 (3)
H4	1.130 (2)	0.2242 (7)	0.0670 (18)	0.042 (5)*
C5	1.13629 (16)	0.27456 (5)	0.20693 (15)	0.0302 (3)
H5	1.2082 (19)	0.2620 (6)	0.2553 (18)	0.031 (4)*
C6	1.07709 (15)	0.31656 (5)	0.24705 (14)	0.0259 (3)
H6	1.1110 (17)	0.3317 (6)	0.3233 (16)	0.024 (4)*
C7	0.90948 (16)	0.38351 (5)	0.21154 (15)	0.0262 (3)
H71	0.810 (2)	0.3851 (6)	0.1938 (16)	0.026 (4)*
H72	0.9308 (18)	0.3923 (6)	0.3014 (18)	0.030 (4)*
O1	0.96954 (10)	0.41880 (3)	0.12554 (9)	0.0222 (2)
C	1.06743 (13)	0.44778 (4)	0.17393 (12)	0.0163 (3)
O	1.10316 (10)	0.45014 (3)	0.28856 (9)	0.0215 (2)
N_1	1.12335 (11)	0.47415 (4)	0.07933 (10)	0.0158 (2)
H_1	1.0918 (19)	0.4703 (6)	−0.0046 (19)	0.033 (5)*
CA_1	1.20786 (12)	0.51416 (4)	0.11432 (12)	0.0161 (2)
H1_1	1.2928 (17)	0.5050 (5)	0.1588 (15)	0.017 (4)*
H2_1	1.2366 (16)	0.5302 (6)	0.0336 (15)	0.020 (4)*
C_1	1.12366 (12)	0.54897 (4)	0.19605 (11)	0.0138 (2)
O_1	0.99489 (9)	0.55194 (3)	0.18472 (8)	0.0163 (2)
N_2	1.19880 (11)	0.57585 (4)	0.27582 (10)	0.0147 (2)
H_2	1.287 (2)	0.5720 (6)	0.2796 (16)	0.026 (4)*
CA_2	1.13637 (12)	0.61511 (4)	0.34700 (12)	0.0153 (2)
CB_2	1.24310 (14)	0.63395 (5)	0.44538 (13)	0.0209 (3)
HB1_2	1.2728 (17)	0.6095 (6)	0.5053 (16)	0.026 (4)*
HB2_2	1.200 (2)	0.6593 (7)	0.4966 (17)	0.036 (5)*
HB3_2	1.3238 (19)	0.6467 (6)	0.4009 (16)	0.028 (4)*
CG_2	1.09477 (15)	0.65365 (5)	0.25267 (14)	0.0220 (3)
HG1_2	1.0301 (19)	0.6422 (6)	0.1863 (16)	0.029 (4)*
HG2_2	1.0499 (19)	0.6787 (7)	0.2964 (18)	0.032 (5)*
HG3_2	1.1808 (19)	0.6660 (6)	0.2125 (16)	0.028 (4)*
C_2	1.01118 (13)	0.59836 (4)	0.42891 (11)	0.0161 (3)
O_2	0.90772 (9)	0.62163 (3)	0.44834 (9)	0.0226 (2)
OH_2	1.03729 (10)	0.55748 (3)	0.48645 (9)	0.0193 (2)
HH_2	0.976 (2)	0.5538 (7)	0.550 (2)	0.045 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0193 (6)	0.0213 (6)	0.0207 (6)	−0.0080 (5)	0.0065 (5)	0.0012 (5)
C2	0.0197 (6)	0.0257 (7)	0.0206 (6)	−0.0036 (5)	0.0020 (5)	0.0004 (5)

C3	0.0259 (7)	0.0256 (7)	0.0257 (7)	−0.0064 (6)	0.0056 (6)	−0.0044 (6)
C4	0.0263 (7)	0.0212 (7)	0.0385 (8)	−0.0012 (6)	0.0107 (6)	0.0023 (6)
C5	0.0215 (7)	0.0322 (8)	0.0368 (8)	−0.0019 (6)	−0.0009 (6)	0.0102 (6)
C6	0.0227 (7)	0.0310 (7)	0.0241 (7)	−0.0107 (6)	−0.0011 (5)	0.0025 (6)
C7	0.0266 (8)	0.0227 (7)	0.0294 (7)	−0.0089 (6)	0.0121 (6)	−0.0028 (6)
O1	0.0218 (5)	0.0211 (5)	0.0237 (5)	−0.0070 (4)	0.0022 (4)	−0.0006 (4)
C	0.0127 (6)	0.0159 (6)	0.0204 (6)	0.0032 (4)	0.0028 (5)	−0.0015 (4)
O	0.0193 (5)	0.0266 (5)	0.0186 (5)	−0.0008 (4)	0.0012 (3)	0.0018 (4)
N_1	0.0144 (5)	0.0170 (5)	0.0160 (5)	−0.0009 (4)	0.0012 (4)	−0.0014 (4)
CA_1	0.0101 (6)	0.0188 (6)	0.0193 (6)	−0.0005 (5)	0.0024 (5)	−0.0010 (5)
C_1	0.0115 (6)	0.0149 (6)	0.0150 (5)	−0.0001 (4)	0.0016 (4)	0.0024 (4)
O_1	0.0089 (4)	0.0206 (4)	0.0194 (4)	0.0003 (3)	0.0008 (3)	−0.0027 (3)
N_2	0.0072 (5)	0.0183 (5)	0.0186 (5)	0.0014 (4)	0.0002 (4)	−0.0017 (4)
CA_2	0.0119 (5)	0.0164 (6)	0.0177 (6)	0.0014 (4)	−0.0006 (4)	−0.0020 (5)
CB_2	0.0150 (6)	0.0227 (6)	0.0249 (7)	−0.0031 (5)	−0.0022 (5)	−0.0048 (5)
CG_2	0.0223 (7)	0.0194 (6)	0.0242 (6)	0.0022 (5)	0.0002 (5)	0.0025 (5)
C_2	0.0137 (6)	0.0198 (6)	0.0148 (6)	−0.0004 (5)	−0.0025 (4)	−0.0031 (4)
O_2	0.0141 (4)	0.0288 (5)	0.0250 (5)	0.0056 (4)	0.0014 (3)	−0.0031 (4)
OH_2	0.0170 (4)	0.0220 (5)	0.0190 (4)	0.0000 (3)	0.0025 (4)	0.0025 (4)

Geometric parameters (Å, º)

C1—C6	1.388 (2)	CA_1—C_1	1.5296 (16)
C1—C2	1.3929 (19)	CA_1—H1_1	0.966 (16)
C1—C7	1.502 (2)	CA_1—H2_1	0.987 (16)
C2—C3	1.386 (2)	C_1—O_1	1.2351 (15)
C2—H2	0.972 (18)	C_1—N_2	1.3322 (16)
C3—C4	1.386 (2)	N_2—CA_2	1.4662 (15)
C3—H3	0.996 (18)	N_2—H_2	0.85 (2)
C4—C5	1.384 (2)	CA_2—CG_2	1.5203 (17)
C4—H4	0.95 (2)	CA_2—CB_2	1.5314 (17)
C5—C6	1.390 (2)	CA_2—C_2	1.5360 (17)
C5—H5	0.919 (18)	CB_2—HB1_2	0.973 (17)
C6—H6	0.951 (17)	CB_2—HB2_2	0.987 (19)
C7—O1	1.4583 (16)	CB_2—HB3_2	0.966 (18)
C7—H71	0.963 (18)	CG_2—HG1_2	0.975 (18)
C7—H72	0.978 (18)	CG_2—HG2_2	0.948 (19)
O1—C	1.3431 (16)	CG_2—HG3_2	0.984 (18)
C—O	1.2274 (16)	C_2—O_2	1.2061 (15)
C—N_1	1.3403 (16)	C_2—OH_2	1.3336 (16)
N_1—CA_1	1.4449 (16)	OH_2—HH_2	0.89 (2)
N_1—H_1	0.92 (2)		

C6—C1—C2	119.27 (13)	C_1—CA_1—H1_1	110.9 (9)
C6—C1—C7	121.31 (13)	N_1—CA_1—H2_1	108.3 (9)
C2—C1—C7	119.38 (13)	C_1—CA_1—H2_1	107.7 (9)
C3—C2—C1	120.44 (13)	H1_1—CA_1—H2_1	106.9 (13)
C3—C2—H2	120.6 (10)	O_1—C_1—N_2	123.49 (11)

C1—C2—H2	118.9 (10)	O_1—C_1—CA_1	120.93 (11)
C2—C3—C4	119.99 (14)	N_2—C_1—CA_1	115.56 (10)
C2—C3—H3	120.7 (10)	C_1—N_2—CA_2	122.06 (10)
C4—C3—H3	119.3 (10)	C_1—N_2—H_2	119.0 (11)
C5—C4—C3	119.85 (14)	CA_2—N_2—H_2	118.6 (11)
C5—C4—H4	120.3 (12)	N_2—CA_2—CG_2	110.09 (10)
C3—C4—H4	119.8 (12)	N_2—CA_2—CB_2	109.22 (10)
C4—C5—C6	120.26 (14)	CG_2—CA_2—CB_2	109.77 (11)
C4—C5—H5	121.0 (11)	N_2—CA_2—C_2	110.42 (10)
C6—C5—H5	118.7 (11)	CG_2—CA_2—C_2	111.90 (10)
C1—C6—C5	120.16 (14)	CB_2—CA_2—C_2	105.30 (10)
C1—C6—H6	119.9 (10)	CA_2—CB_2—HB1_2	111.0 (10)
C5—C6—H6	119.9 (10)	CA_2—CB_2—HB2_2	109.4 (11)
O1—C7—C1	109.05 (11)	HB1_2—CB_2—HB2_2	108.2 (14)
O1—C7—H71	103.7 (10)	CA_2—CB_2—HB3_2	110.5 (10)
C1—C7—H71	111.4 (10)	HB1_2—CB_2—HB3_2	109.8 (14)
O1—C7—H72	108.1 (10)	HB2_2—CB_2—HB3_2	108.0 (15)
C1—C7—H72	112.4 (11)	CA_2—CG_2—HG1_2	111.5 (10)
H71—C7—H72	111.7 (14)	CA_2—CG_2—HG2_2	111.3 (11)
C—O1—C7	118.40 (11)	HG1_2—CG_2—HG2_2	107.5 (15)
O—C—N_1	123.65 (12)	CA_2—CG_2—HG3_2	108.1 (10)
O—C—O1	125.54 (12)	HG1_2—CG_2—HG3_2	110.8 (14)
N_1—C—O1	110.80 (11)	HG2_2—CG_2—HG3_2	107.6 (15)
C—N_1—CA_1	119.15 (10)	O_2—C_2—OH_2	124.28 (12)
C—N_1—H_1	118.8 (11)	O_2—C_2—CA_2	123.55 (11)
CA_1—N_1—H_1	120.7 (11)	OH_2—C_2—CA_2	111.81 (10)
N_1—CA_1—C_1	111.11 (10)	C_2—OH_2—HH_2	107.8 (13)
N_1—CA_1—H1_1	111.7 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N_1—H_1···O_1^{i}	0.92 (2)	2.12 (2)	3.0298 (14)	168.7 (16)
N_2—H_2···O_1^{ii}	0.85 (2)	2.09 (2)	2.9304 (15)	167.3 (18)
O*H*_2—H*H*_2···O^{iii}	0.88 (2)	1.82 (2)	2.6789 (14)	162 (2)

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