Acta Cryst E STRUCTURE REPORTS

ISSN 1600-5368

Conformation and crystal structures of 1-amino-cyclohexaneacetic acid ($\beta^{3,3}$Ac$_6$c) in N-protected derivatives

Naiem Ahmad Wani,[a] Vivek K. Gupta,[b] Rajni Kant,[b] Subrayashastry Aravinda[a]* and Rajkishor Rai[a]*

[a]Medicinal Chemistry Division, Indian Institute of Integrative Medicine, Canal Road, Jammu Tawi 180 001, India, and [b]X-ray Crystallography Laboratory, Post-Graduate Department of Physics & Electronics, University of Jammu, Jammu Tawi 180 006, India. *Correspondence e-mail: saravinda@iiim.ac.in, raj@iiim.ac.in

Received 25 August 2014
Accepted 16 September 2014

Edited by G. Smith, Queensland University of Technology, Australia

IIIM Communication number: IIIM/1553/2013.

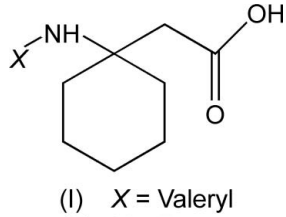
Keywords: crystal structure; disubstituted-β-amino acids; π–π interaction; hydrogen bonds; conformation

CCDC references: 1024489; 1024490
Supporting information: this article has supporting information at journals.iucr.org/e

N-Protected derivatives of 1-aminocyclohexaneacetic acid ($\beta^{3,3}$-Ac$_6$c), namely Valeroyl-$\beta^{3,3}$-Ac$_6$c-OH [2-(1-pentanamidocyclohexyl)acetic acid, $C_{13}H_{23}NO_3$], (I), Fmoc-$\beta^{3,3}$-Ac$_6$c-OH [2-(1-{[(9*H*-fluoren-9-yloxy)carbonyl]amino}cyclohexyl)acetic acid, $C_{23}H_{25}NO_4$], (II), and Pyr-$\beta^{3,3}$-Ac$_6$c-OH {2-[1-(pyrazine-2-amido)cyclohexyl]acetic acid, $C_{13}H_{17}N_3O_3$}, (III), were synthesized and their conformational properties were determined by X-ray diffraction analysis. The backbone torsion angles (φ, θ) for $\beta^{3,3}$-Ac$_6$c-OH are restricted to *gauche* conformations in all the derivatives, with a chair conformation of the cyclohexane ring. In the crystal structure of (I), the packing of molecules shows both carboxylic acid $R_2^2(8)$ O—H···O and centrosymmetric $R_2^2(14)$ N—H···O hydrogen-bonding interactions, giving rise to chains along the *c*-axis direction. In (II), centrosymmetric carboxylic acid $R_2^2(8)$ O—H···O dimers are extended through N—H···O hydrogen bonds and together with inter-ring π–π interactions between Fmoc groups [ring centroid distance = 3.786 (2) Å], generate a layered structure lying parallel to (010). In the case of compound (III), carboxylic acid O—H···N$_{pyrazine}$ hydrogen bonds give rise to zigzag ribbon structures extending along the *c*-axis direction.

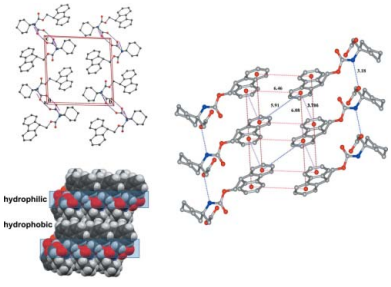
1. Chemical context

β-Amino acids are homologues of α-amino acids, which are constituents of several bioactive natural and synthetic products. β-Amino acids have been used as building blocks in peptidomimetic drug design (Cheng *et al.* 2001). The introduction of β-amino acids into pharmacologically active peptide sequences has shown improved biological activity and metabolic stability (Yamazaki *et al.*, 1991; Huang *et al.*, 1993). The backbone conformation of a β-amino acid is defined by the torsional angles φ, θ and ψ (Banerjee & Balaram, 1997), as shown in Fig. 1. The monosubstitution at the α- and β-carbon atoms plays an important role in the folding of oligomers of β-amino acids (Seebach *et al.*, 2009).



(I) *X* = Valeryl
(II) *X* = Fmoc
(III) *X* = Pyr

In order to investigate the effect of protecting groups and disubstitution on the conformation of β-amino acids, N-



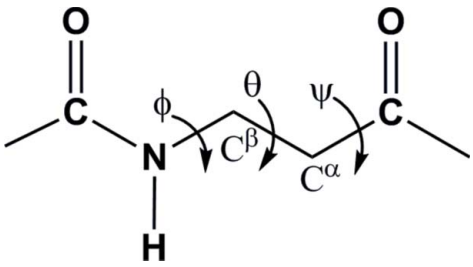
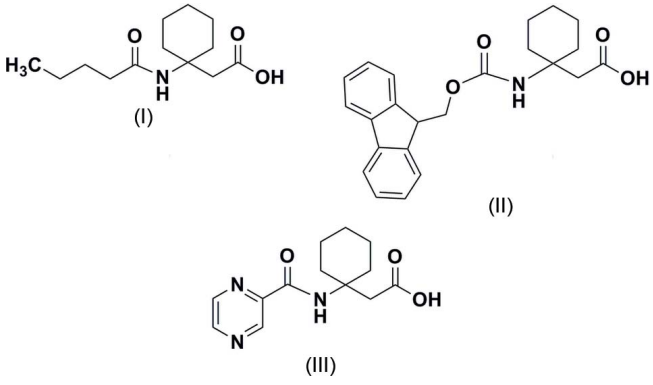


Figure 1
Definition of backbone torsion angles for β-amino acids.

protected derivatives of 1-aminocyclohexaneacetic acid ($\beta^{3,3}$Ac$_6$c), *i.e.* Valeroyl-$\beta^{3,3}$-Ac$_6$c-OH (I), Fmoc-$\beta^{3,3}$-Ac$_6$c-OH (II) and Pyr-$\beta^{3,3}$-Ac$_6$c-OH (III) were synthesized. The crystal structures of the three compounds were determined and are reported herein, together with their comparative conformational features.



2. Structural commentary

The molecular conformations of Valeroyl-$\beta^{3,3}$-Ac$_6$c-OH (I), Fmoc-$\beta^{3,3}$-Ac$_6$c-OH (II) and Pyr-$\beta^{3,3}$-Ac$_6$c-OH (III) are shown in Fig. 2. The backbone torsion angles (φ, θ) (C0′—N1—C1*B* —C1*A* and N1—C1*B*—C1*A*—C1′) adopt a *gauche* conformation in all three compounds [φ = 61.9 (3)°, θ = 57.2 (3)° for (I); φ = 56.7 (3)°, θ = 66.1 (3)° for (II) and φ = 65.5 (2)°, θ = 55.0 (2)° for (III). The torsional angle ψ restricts the extended (*trans*) conformation for (I) [166.9 (2)°] and (III) [157.9 (2)°]. In the case of (II), it is restricted to a *gauche* conformation [*i.e.* ψ = −63.6 (3)°]. In a 3,3-disubstituted β-amino acid residue, $\beta^{3,3}$-Ac$_6$c-OH, the cyclohexane ring imposes a restriction on the torsion angles φ and θ. The protecting groups at the *N*-terminus of (I) adopts a *trans* geometry [ω_0 (C4—C0′—N1—C1*B*) = 177.4 (2) for (I), ω_0 (O—C0′—N1—C1*B*) = −175.64 (19) for (II) and ω_0 (C6—C0—N1— C1*A*) = −170.04 (17)° for (III)]. In the case of the N-protected *tert*-butyloxycarbonyl (Boc) group, the protecting group adopts a *cis* geometry with ω_0 = 14.50° (Vasudev *et al.*, 2008). The cyclohexane ring adopts a chair conformation with axial amino and equatorial CH$_2$CO groups in all the derivatives. In Pyr-$\beta^{3,3}$-Ac$_6$c-OH (III), an intramolecular N—H···N interaction is observed between NH of the $\beta^{3,3}$-Ac$_6$c-OH residue and N3 of the pyrazine ring as shown in Fig. 3*c*. There

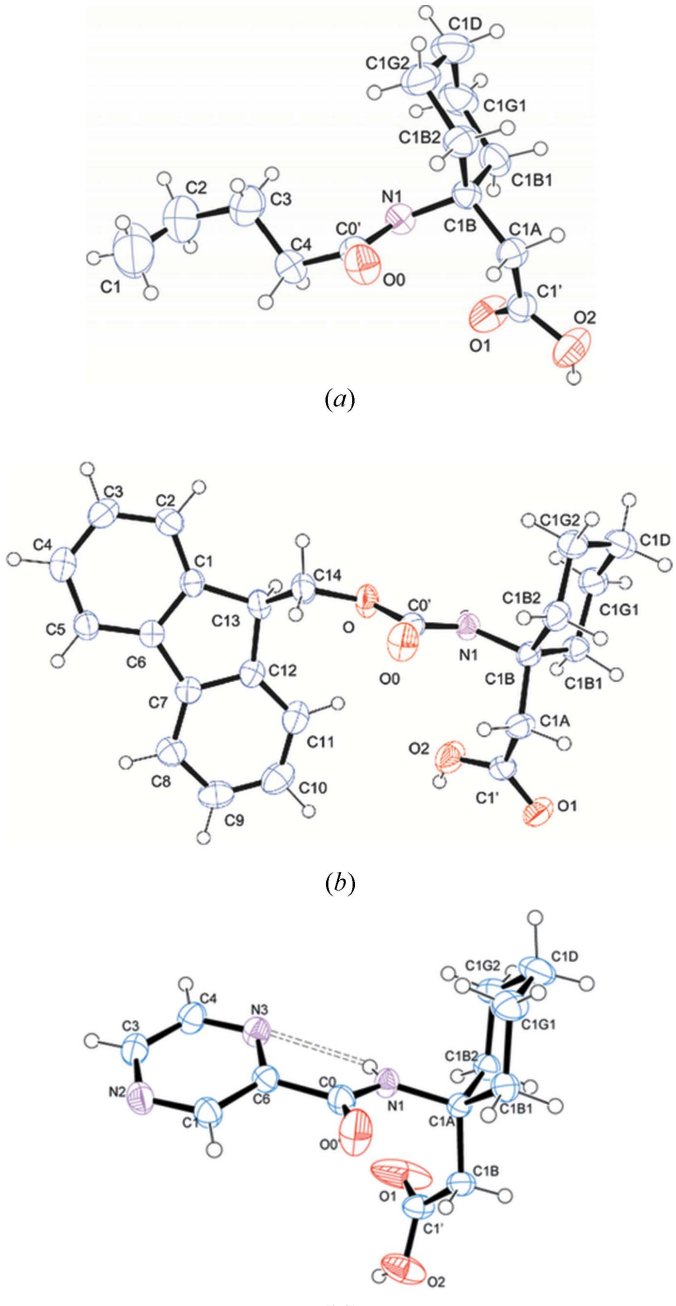


Figure 2
ORTEP view of the molecular conformation with the atom-labelling scheme. for Valeroyl-$\beta^{3,3}$-Ac$_6$c-OH (I), (*b*) Fmoc-$\beta^{3,3}$-Ac$_6$c-OH (II) and (*c*) Pyr-$\beta^{3,3}$-Ac$_6$c-OH (III). The displacement ellipsoids are drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii.

are no intramolecular hydrogen bonding interactions observed in the crystal structures of derivatives (I) and (II).

3. Supramolecular features

In the crystals of compounds (I) and (II), intermolecular hydrogen-bonding interactions generate primary centrosymmetric dimeric but different substructures (Figs. 4 and 5). In (I), N1—H···O1^{ii} bond pairs (Table 1) give a cyclic $R_2^2(14)$

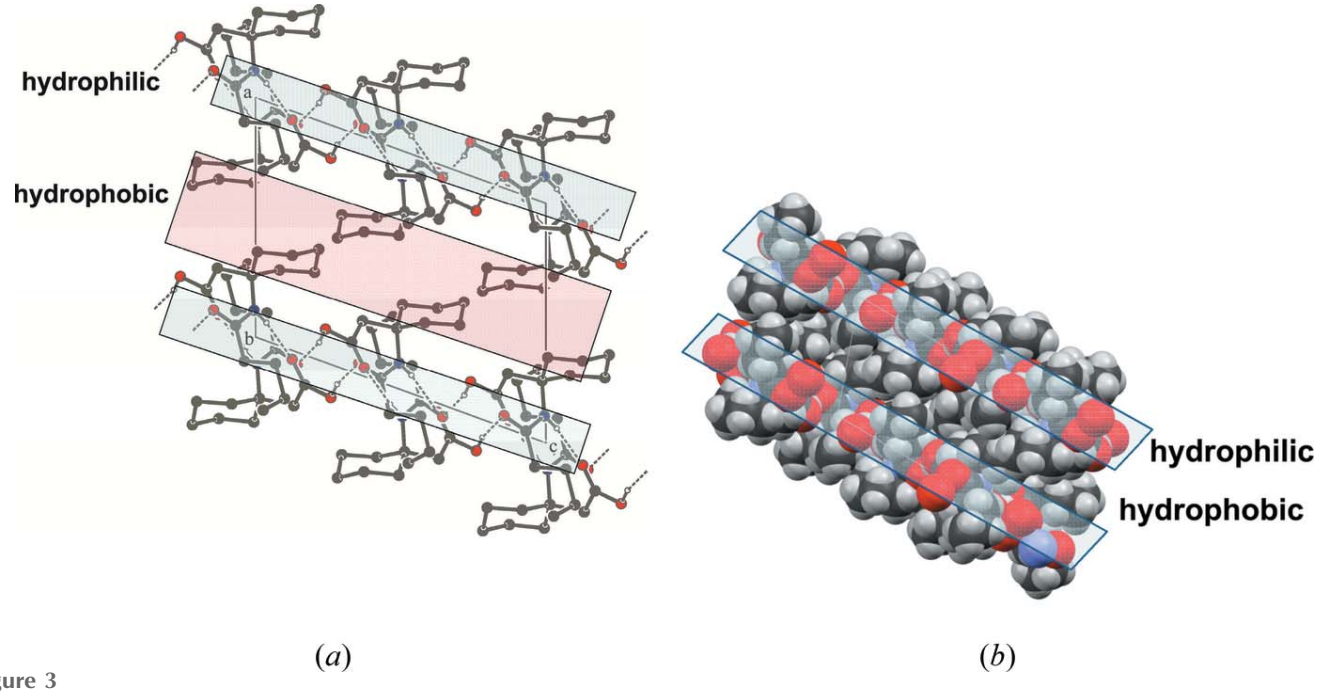


Figure 3
(*a*) Packing of Valeroyl-$\beta^{3,3}$-Ac$_6$c-OH (I) down the *b*-axis showing the alternative hydrophilic and hydrophobic layers (*b*) space-filling model.

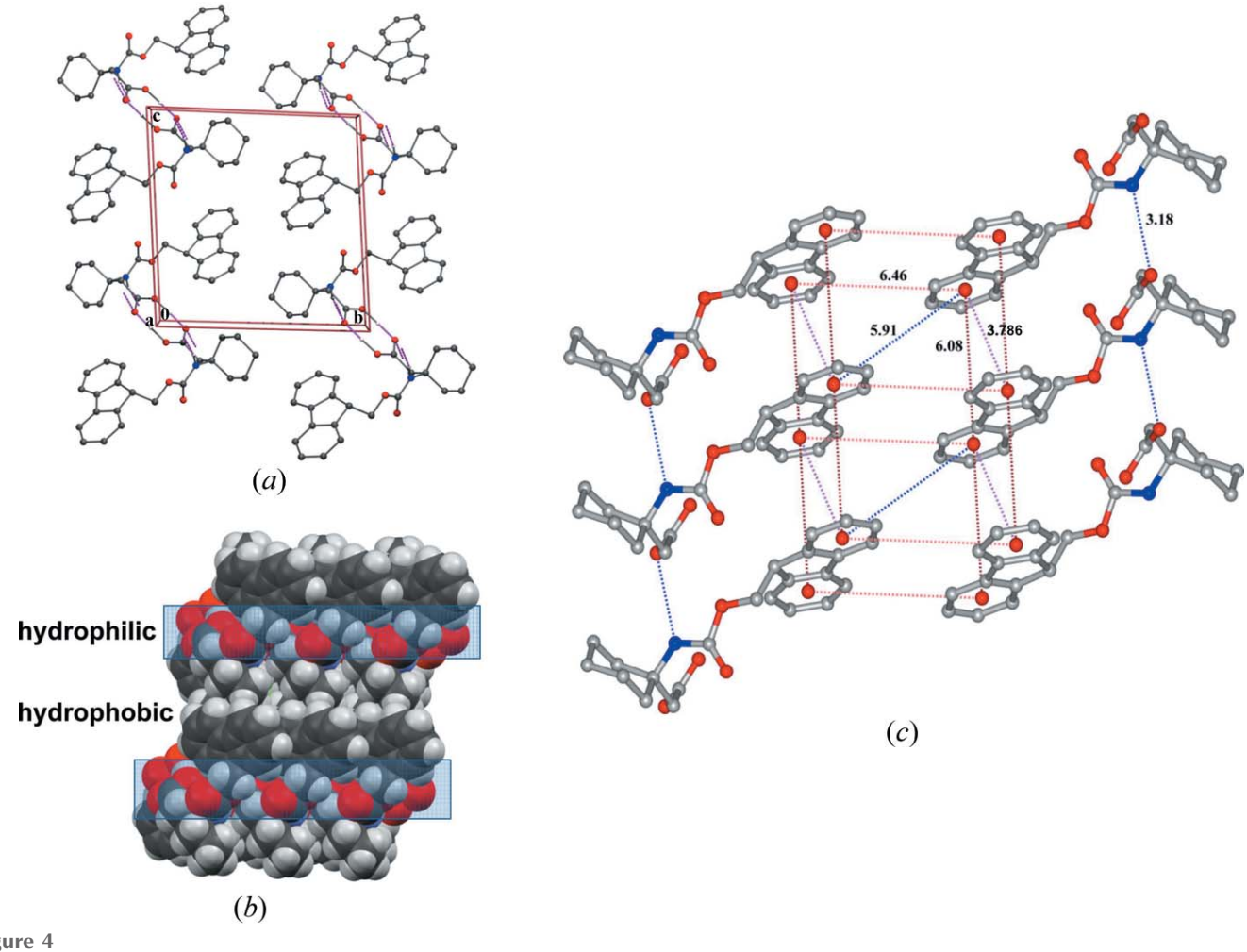


Figure 4
(*a*) Packing of Fmoc-$\beta^{3,3}$-Ac$_6$c-OH (II) down the *a*-axis. (*b*) Space-filling model showing the alternative hydrophilic and hydrophobic layers (packing down the *c*-axis). (*c*) The environment of the Fmoc group showing the aromatic interaction. The centroid–centroid distances are shown.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O0^{i}	0.87 (4)	1.74 (4)	2.599 (3)	166 (4)
N1—H1N···O1^{ii}	0.82 (3)	2.16 (3)	2.981 (3)	172 (2)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.86 (2)	2.35 (2)	3.182 (3)	161 (2)
O2—H2O···O1^{ii}	0.84 (3)	1.83 (3)	2.673 (3)	177 (1)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H21···N2^{i}	0.93 (4)	1.86 (4)	2.791 (3)	177 (4)
N1—H1N···N3	0.79 (2)	2.34 (2)	2.729 (2)	111.3 (19)

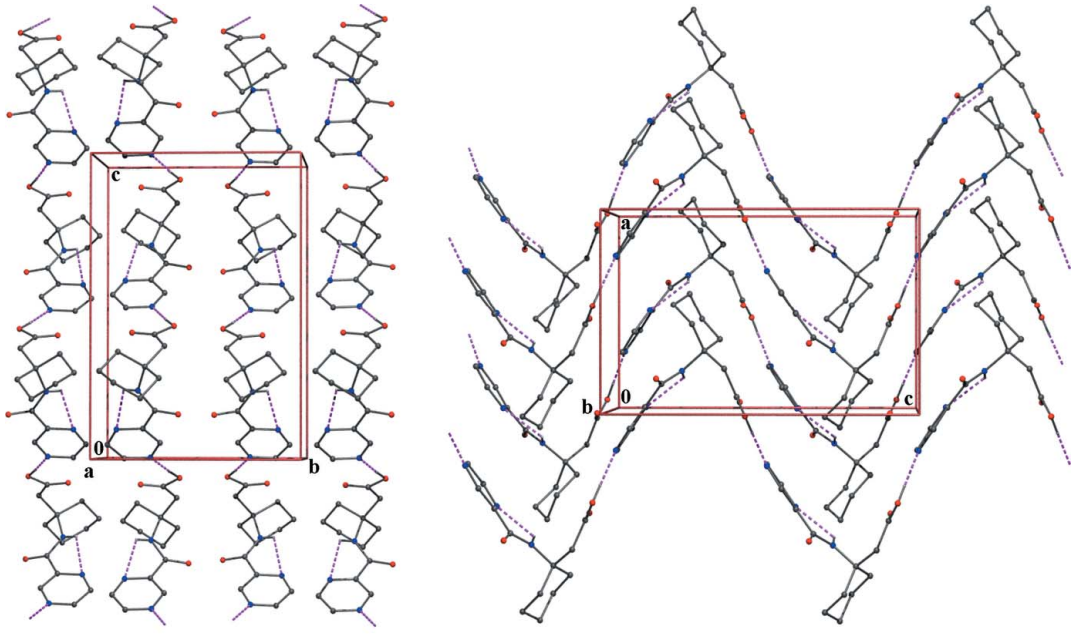
Symmetry code: (i) $-x+\frac{3}{2}, y, z+\frac{1}{2}$.

motif which is extended into a ribbon structure along the *c*-axis direction through a second but non-centrosymmetric cyclic carboxylic acid $R_2^2(8)$ O2—H···O^{i} hydrogen-bond motif (Fig. 4*a*). In (II), the intermolecular dimeric association is through the centrosymmetric $R_2^2(8)$ carboxylic acid hydrogen-bonding motif. Structure extension is through N1—H···O1′ (carboxyl) hydrogen bonds (Table 2), generating a two-dimensional layered structure lying parallel to (010) (Fig. 4*c*). Also present in the structure are π–π interactions between the Fmoc groups with an intercentroid distance of 3.786 (2) Å. Fig. 4*c* shows the aromatic rings of Fmoc groups stacked in a face-to-face and edge-to-face manner, together with interplane distances that are within the range for stabilizing π–π interactions (Burley & Petsko, 1985; Sengupta *et al.*, 2005) and have been reported to induce self-assembly in peptides (Wang & Chau, 2011). In the case of (I) and (II), the molecular packing in the crystals leads to the formation of alternating hydrophobic and hydrophilic layers. In the crystals of (III), in which no dimer substructure formation is present, the molecules are linked by an intermolecular carboxylic acid O2—H···N2^{i} hydrogen bond (Table 3) with a pyrazine N-atom acceptor, leading to the formation of a zigzag ribbon structure extending along the *c*-axis direction.

4. Synthesis and crystallization

Preparation of Valeroyl-$\beta^{3,3}$Ac$_6$c-OH (I): $\beta^{3,3}$Ac$_6$c-OH (5 mmol, 785 mg) was dissolved in 5 ml of a 2*M* NaOH solution and a solution of 5 mmol of valeric anhydride (931 mg) dissolved in 1,4-dioxane was added, after which the mixture was stirred for 4 h at room temperature. On completion of the reaction, the 1,4-dioxane was evaporated and the product was extracted with diethyl ether (3 × 5 ml). The aqueous layer was acidified with 2*M* HCl and extracted with ethyl acetate (3 × 10ml) and the combined organic layer was washed with brine solution. The organic layer was passed over anhydrous Na_2SO_4 and evaporated to give Valeroyl-β^{3}Ac$_6$c-OH (yield: 1.1 g, 85.2%). Single crystals were grown by slow evaporation from a solution in methanol/water.

Preparation of Fmoc-$\beta^{3,3}$Ac$_6$c-OH (II): $\beta^{3,3}$Ac$_6$c-OH (10 mmol, 1.57 g) was dissolved in 1*M* Na_2CO_3 solution and Fmoc-OSu (10 mmol, 3.37 g) dissolved in CH_3CN was added.



(*a*) (*b*)

Figure 5
(*a*) Packing of Pyr-$\beta^{3,3}$-Ac$_6$c-OH (III) down the *a*-axis showing the ribbon structure. (*b*) Zigzag arrangement of the ribbons along the *c*-axis.

Table 4
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{13}H_{23}NO_3$	$C_{23}H_{25}NO_4$	$C_{13}H_{17}N_3O_3$
M_r	241.32	379.44	263.30
Crystal system, space group	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$	Orthorhombic, $Pca2_1$
Temperature (K)	291	291	291
a, b, c (Å)	9.5894 (5), 12.5007 (7), 12.3709 (8)	6.0834 (4), 12.7642 (9), 12.8399 (9)	8.7135 (1), 10.5321 (1), 14.3907 (2)
α, β, γ (°)	90, 109.984 (7), 90	94.018 (6), 92.295 (6), 100.489 (6)	90, 90, 90
V (Å^3)	1393.66 (14)	976.53 (12)	1320.66 (3)
Z	4	2	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.08	0.09	0.10
Crystal size (mm)	0.30 × 0.08 × 0.08	0.30 × 0.05 × 0.03	0.25 × 0.25 × 0.25
Data collection			
Diffractometer	Oxford Diffraction Xcalibur, Sapphire3 CCD	Oxford Diffraction Xcalibur, Sapphire3 CCD	Oxford Difraction Xcalibur, Sapphire3 CCD
Absorption correction	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min}, T_{max}	0.797, 1.000	0.947, 1.000	0.931, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14087, 2737, 1717	7781, 4166, 2037	68869, 2878, 2670
R_{int}	0.047	0.047	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617	0.639	0.639
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.068, 0.213, 1.03	0.054, 0.086, 0.97	0.042, 0.106, 1.04
No. of reflections	2737	4166	2878
No. of parameters	162	353	240
No. of restraints	0	1	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	All H-atom parameters refined	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.30	0.15, −0.20	0.27, −0.26
Absolute structure	–	–	(Flack, 1983): 1585 Friedel pairs
Absolute structure parameter	–	–	0.2 (14)

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2010), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009).

The reaction mixture was stirred at room temperature for 6 h. After completion of the reaction, the CH_3CN was evaporated and the residue was extracted with diethyl ether (3 × 10 ml). The aqueous layer was acidified with 2*M* HCl and extracted with ethyl acetate (3 × 15 ml). The combined organic layer was washed with brine solution. The ethyl acetate layer was passed over anhydrous Na_2SO_4 and evaporated. The residue was purified by crystallization in ethyl acetate/*n*-hexane, affording Fmoc-$\beta^{3,3}$Ac$_6$c-OH (yield: 3.0 g, 79%). Single crystals were obtained by slow evaporation from an ethyl acetate/*n*-hexane solution.

Preparation of Pyr-$\beta^{3,3}$Ac$_6$c-OH (III): Pyrazine carboxylic acid (3 mmol, 372 mg) was dissolved in dry CH_2Cl_2 and then 200 µl of *N*-methylmorpholine was added, followed by $\beta^{3,3}$Ac$_6$c-OMe. HCl (3 mmol, 622.5 mg) and EDCI. HCl (3 mmol,576 mg) at 273 K. The reaction mixture was stirred at room temperature for 12 h. After completion of the reaction, water was added and the reaction mixture was extracted with CH_2Cl_2 (3 × 5ml). The combined organic layer was washed with 2*M* HCl (2 × 5ml), Na_2CO_3 (2 × 5ml) and brine solution (2 × 5ml). The organic layer was passed over anhydrous Na_2SO_4 and evaporated to give Pyr-$\beta^{3,3}$Ac$_6$c-OMe (Yield: 600 mg, 72.2%). Pyr-$\beta^{3,3}$Ac$_6$c-OMe (2 mmol, 554 mg) was dissolved in 2 ml of methanol and 1 ml of 2*M* NaOH, and the reaction mixture was stirred at room temperature for 4 h. Methanol was evaporated and the residue was extracted with diethyl ether (2 × 5ml). The aqueous layer was acidified with 2*M* HCl and extracted with ethyl acetate (3 × 5ml). The combined organic layer was washed with brine solution (1 × 5ml). The ethyl acetate layer was passed over anhydrous Na_2SO_4 and evaporated to give Pyr-$\beta^{3,3}$Ac$_6$c-OH (yield: 370 mg, 70.3%). Single crystals were grown from an ethanol/water solution.

5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 4. For derivative (I), H atoms for N1 and O2 were located in a difference Fourier map and both their coordinates and U_{iso} values were refined. The remaining H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.96–0.98 Å and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). For derivatives (II) and (III), all hydrogen atoms were located from a difference Fourier map and both their coordinates and U_{iso} values were refined. In (II), the carboxyl O—H distance was constrained to 0.84 Å. Although not of consequence with the achiral molecule of (III), which crystallized in the non-

centrosymmetric space group $Pca2_1$, the structure was inverted in the final cycles of refinement as the Flack parameter was 0.8 (14). The inverted structure gave a value of 0.2 (14) for 1585 Friedel pairs.

Acknowledgements

RR acknowledges the Council of Scientific and Industrial Research (CSIR), India, for financial assistance under MLP5009and BSC-120. RK wishes to acknowledge the Department of Science and Technology, India, for sanctioning the single-crystal X-ray diffractometer as a National Facility under project No: SR/S2 /CMP/47.

References

Banerjee, A. & Balaram, P. (1997). *Curr. Sci.* **73**, 1067–1077.
Burley, S. K. & Petsko, G. A. (1985). *Science*, **229**, 23–28.
Cheng, R. P., Gellman, S. H. & DeGrado, W. F. (2001). *Chem. Rev.* **101**, 3219–3232.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Huang, Z., Pröbstl, A., Spencer, J. R., Yamazaki, T. & Goodman, M. (1993). *Int. J. Pept. Protein Res.* **42**, 352–365.
Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Seebach, D., Beck, A. K., Capone, S., Deniau, G., Grošelj, U. & Zass, E. (2009). *Synthesis*, **1**, 1–32.
Sengupta, A., Mahalakshmi, R., Shamala, N. & Balaram, P. (2005). *J. Pept. Res.* **65**, 113–129.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Vasudev, P. G., Rai, R., Shamala, N. & Balaram, P. (2008). *Biopolymers*, **90**, 138–150.
Wang, W. & Chau, Y. (2011). *Chem. Commun.* **47**, 10224.
Yamazaki, T., Pröbsti, A., Schiller, P. W. & Goodman, M. (1991). *Int. J. Pept. Protein Res.* **37**, 364–381.

supporting information

Acta Cryst. (2014). E**70**, 272-277 [doi:10.1107/S1600536814020777]

Conformation and crystal structures of 1-aminocyclohexaneacetic acid ($\beta^{3,3}$Ac$_6$c) in N-protected derivatives

Naiem Ahmad Wani, Vivek K. Gupta, Rajni Kant, Subrayashastry Aravinda and Rajkishor Rai

Computing details

For all compounds, data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2010); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009).

(I) 2-(1-Pentanamidocyclohexyl)acetic acid

Crystal data

$C_{13}H_{23}NO_3$
M_r = 241.32
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.5894 (5) Å
b = 12.5007 (7) Å
c = 12.3709 (8) Å
β = 109.984 (7)°
V = 1393.66 (14) Å^3
Z = 4
$F(000)$ = 528
D_x = 1.150 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4978 reflections
θ = 3.5–29.1°
μ = 0.08 mm^{-1}
T = 291 K
Needle, colourless
0.30 × 0.08 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur, Sapphire3 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1049 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.797, T_{max} = 1.000
14087 measured reflections
2737 independent reflections
1717 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 26.0°, θ_{min} = 3.5°
h = −11→11
k = −15→15
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.213
S = 1.03
2737 reflections
162 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0999P)^2 + 0.474P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

Special details

Experimental. *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27–08-2010 CrysAlis171. NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2941 (6)	−0.4516 (4)	−0.0262 (5)	0.141 (2)
H1A	0.3775	−0.4719	−0.0479	0.211*
H1B	0.2304	−0.5123	−0.0328	0.211*
H1C	0.3286	−0.4266	0.0519	0.211*
C2	0.2142 (5)	−0.3689 (4)	−0.0997 (4)	0.1151 (15)
H2A	0.1810	−0.3938	−0.1787	0.138*
H2B	0.2790	−0.3080	−0.0937	0.138*
C3	0.0798 (4)	−0.3343 (3)	−0.0684 (4)	0.0977 (13)
H3A	0.0124	−0.2948	−0.1326	0.117*
H3B	0.0282	−0.3977	−0.0571	0.117*
C4	0.1176 (3)	−0.2653 (2)	0.0389 (2)	0.0560 (7)
H4A	0.1902	−0.3019	0.1026	0.067*
H4B	0.1610	−0.1985	0.0261	0.067*
C0′	−0.0183 (3)	−0.2422 (2)	0.0688 (2)	0.0471 (6)
O0	−0.0486 (2)	−0.29372 (16)	0.14429 (16)	0.0630 (6)
N1	−0.1087 (2)	−0.16761 (17)	0.00548 (18)	0.0442 (5)
C1B	−0.2512 (3)	−0.1329 (2)	0.0135 (2)	0.0435 (6)
C1A	−0.2298 (3)	−0.0852 (2)	0.1317 (2)	0.0476 (6)
H1A1	−0.3261	−0.0627	0.1328	0.057*
H1A2	−0.1946	−0.1415	0.1884	0.057*
C1′	−0.1254 (3)	0.0078 (2)	0.1691 (2)	0.0467 (6)
O1	−0.0372 (2)	0.03565 (17)	0.12608 (18)	0.0726 (7)
O2	−0.1390 (3)	0.0553 (2)	0.2593 (2)	0.0803 (8)
C1B1	−0.3163 (3)	−0.0502 (2)	−0.0824 (2)	0.0586 (7)
H1B3	−0.4034	−0.0182	−0.0727	0.070*
H1B4	−0.2439	0.0061	−0.0745	0.070*
C1B2	−0.3582 (3)	−0.2283 (2)	−0.0062 (2)	0.0558 (7)
H1B5	−0.3131	−0.2834	0.0501	0.067*
H1B6	−0.4487	−0.2053	0.0053	0.067*

C1D	−0.4625 (4)	−0.1922 (3)	−0.2179 (3)	0.0884 (12)
H1D1	−0.5568	−0.1683	−0.2138	0.106*
H1D2	−0.4809	−0.2238	−0.2932	0.106*
C1G1	−0.3594 (4)	−0.0972 (3)	−0.2031 (3)	0.0772 (10)
H1G1	−0.4080	−0.0425	−0.2589	0.093*
H1G2	−0.2706	−0.1194	−0.2176	0.093*
C1G2	−0.3966 (4)	−0.2751 (3)	−0.1265 (3)	0.0744 (10)
H1G3	−0.3075	−0.3042	−0.1358	0.089*
H1G4	−0.4667	−0.3333	−0.1358	0.089*
H2O	−0.074 (5)	0.107 (3)	0.281 (3)	0.106 (14)*
H1N	−0.076 (3)	−0.1325 (19)	−0.037 (2)	0.035 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.140 (4)	0.134 (4)	0.153 (5)	0.044 (4)	0.056 (4)	−0.004 (4)
C2	0.101 (3)	0.127 (4)	0.123 (4)	0.025 (3)	0.046 (3)	−0.021 (3)
C3	0.081 (2)	0.120 (3)	0.100 (3)	0.006 (2)	0.040 (2)	−0.039 (2)
C4	0.0516 (15)	0.0561 (16)	0.0601 (17)	0.0054 (12)	0.0187 (13)	0.0066 (13)
C0′	0.0490 (14)	0.0473 (14)	0.0439 (14)	−0.0011 (11)	0.0146 (11)	−0.0019 (12)
O0	0.0706 (13)	0.0647 (12)	0.0586 (12)	0.0133 (10)	0.0284 (10)	0.0201 (10)
N1	0.0458 (12)	0.0478 (12)	0.0420 (12)	−0.0006 (9)	0.0190 (10)	0.0062 (10)
C1B	0.0407 (12)	0.0485 (14)	0.0420 (13)	−0.0018 (10)	0.0150 (10)	0.0008 (11)
C1A	0.0489 (14)	0.0520 (15)	0.0461 (14)	−0.0040 (11)	0.0215 (11)	−0.0007 (12)
C1′	0.0503 (14)	0.0480 (14)	0.0426 (14)	0.0015 (11)	0.0170 (12)	0.0011 (12)
O1	0.0871 (15)	0.0757 (14)	0.0694 (13)	−0.0327 (12)	0.0452 (12)	−0.0172 (11)
O2	0.0884 (16)	0.0858 (17)	0.0840 (16)	−0.0313 (13)	0.0519 (13)	−0.0394 (13)
C1B1	0.0521 (15)	0.0675 (18)	0.0536 (16)	0.0124 (13)	0.0148 (12)	0.0101 (14)
C1B2	0.0504 (15)	0.0630 (17)	0.0563 (17)	−0.0102 (12)	0.0209 (13)	−0.0064 (13)
C1D	0.064 (2)	0.138 (3)	0.0529 (19)	−0.009 (2)	0.0072 (16)	−0.020 (2)
C1G1	0.0656 (19)	0.109 (3)	0.0491 (18)	0.0071 (18)	0.0090 (14)	0.0109 (17)
C1G2	0.0663 (19)	0.084 (2)	0.073 (2)	−0.0235 (17)	0.0246 (16)	−0.0300 (18)

Geometric parameters (Å, º)

C1—C2	1.416 (7)	C1A—C1′	1.500 (3)
C1—H1A	0.9600	C1A—H1A1	0.9700
C1—H1B	0.9600	C1A—H1A2	0.9700
C1—H1C	0.9600	C1′—O1	1.194 (3)
C2—C3	1.529 (5)	C1′—O2	1.309 (3)
C2—H2A	0.9700	O2—H2O	0.88 (4)
C2—H2B	0.9700	C1B1—C1G1	1.524 (4)
C3—C4	1.519 (4)	C1B1—H1B3	0.9700
C3—H3A	0.9700	C1B1—H1B4	0.9700
C3—H3B	0.9700	C1B2—C1G2	1.523 (4)
C4—C0′	1.499 (3)	C1B2—H1B5	0.9700
C4—H4A	0.9700	C1B2—H1B6	0.9700
C4—H4B	0.9700	C1D—C1G2	1.505 (5)

C0′—O0	1.248 (3)	C1D—C1G1	1.516 (5)
C0′—N1	1.331 (3)	C1D—H1D1	0.9700
N1—C1B	1.469 (3)	C1D—H1D2	0.9700
N1—H1N	0.82 (2)	C1G1—H1G1	0.9700
C1B—C1A	1.526 (3)	C1G1—H1G2	0.9700
C1B—C1B1	1.535 (4)	C1G2—H1G3	0.9700
C1B—C1B2	1.538 (3)	C1G2—H1G4	0.9700

C2—C1—H1A	109.5	C1B—C1A—H1A1	108.0
C2—C1—H1B	109.5	C1′—C1A—H1A2	108.0
H1A—C1—H1B	109.5	C1B—C1A—H1A2	108.0
C2—C1—H1C	109.5	H1A1—C1A—H1A2	107.3
H1A—C1—H1C	109.5	O1—C1′—O2	122.7 (2)
H1B—C1—H1C	109.5	O1—C1′—C1A	126.0 (2)
C1—C2—C3	111.2 (4)	O2—C1′—C1A	111.3 (2)
C1—C2—H2A	109.4	C1′—O2—H2O	109 (3)
C3—C2—H2A	109.4	C1G1—C1B1—C1B	113.6 (2)
C1—C2—H2B	109.4	C1G1—C1B1—H1B3	108.8
C3—C2—H2B	109.4	C1B—C1B1—H1B3	108.8
H2A—C2—H2B	108.0	C1G1—C1B1—H1B4	108.8
C4—C3—C2	114.3 (3)	C1B—C1B1—H1B4	108.8
C4—C3—H3A	108.7	H1B3—C1B1—H1B4	107.7
C2—C3—H3A	108.7	C1G2—C1B2—C1B	112.3 (2)
C4—C3—H3B	108.7	C1G2—C1B2—H1B5	109.2
C2—C3—H3B	108.7	C1B—C1B2—H1B5	109.2
H3A—C3—H3B	107.6	C1G2—C1B2—H1B6	109.2
C0′—C4—C3	110.9 (2)	C1B—C1B2—H1B6	109.2
C0′—C4—H4A	109.5	H1B5—C1B2—H1B6	107.9
C3—C4—H4A	109.5	C1G2—C1D—C1G1	111.1 (3)
C0′—C4—H4B	109.5	C1G2—C1D—H1D1	109.4
C3—C4—H4B	109.5	C1G1—C1D—H1D1	109.4
H4A—C4—H4B	108.0	C1G2—C1D—H1D2	109.4
O0—C0′—N1	122.0 (2)	C1G1—C1D—H1D2	109.4
O0—C0′—C4	122.0 (2)	H1D1—C1D—H1D2	108.0
N1—C0′—C4	115.9 (2)	C1D—C1G1—C1B1	111.6 (3)
C0′—N1—C1B	127.0 (2)	C1D—C1G1—H1G1	109.3
C0′—N1—H1N	115.9 (16)	C1B1—C1G1—H1G1	109.3
C1B—N1—H1N	116.7 (16)	C1D—C1G1—H1G2	109.3
N1—C1B—C1A	110.86 (19)	C1B1—C1G1—H1G2	109.3
N1—C1B—C1B1	106.79 (19)	H1G1—C1G1—H1G2	108.0
C1A—C1B—C1B1	111.3 (2)	C1D—C1G2—C1B2	111.6 (3)
N1—C1B—C1B2	110.2 (2)	C1D—C1G2—H1G3	109.3
C1A—C1B—C1B2	108.6 (2)	C1B2—C1G2—H1G3	109.3
C1B1—C1B—C1B2	109.1 (2)	C1D—C1G2—H1G4	109.3
C1′—C1A—C1B	117.1 (2)	C1B2—C1G2—H1G4	109.3
C1′—C1A—H1A1	108.0	H1G3—C1G2—H1G4	108.0

C1—C2—C3—C4	75.6 (5)	C1B—C1A—C1′—O1	−14.9 (4)

C2—C3—C4—C0′	−175.5 (3)	C1B—C1A—C1′—O2	166.9 (2)
C3—C4—C0′—O0	99.1 (3)	N1—C1B—C1B1—C1G1	66.6 (3)
C3—C4—C0′—N1	−77.3 (3)	C1A—C1B—C1B1—C1G1	−172.3 (2)
O0—C0′—N1—C1B	1.0 (4)	C1B2—C1B—C1B1—C1G1	−52.5 (3)
C4—C0′—N1—C1B	177.4 (2)	N1—C1B—C1B2—C1G2	−63.3 (3)
C0′—N1—C1B—C1A	61.9 (3)	C1A—C1B—C1B2—C1G2	175.1 (2)
C0′—N1—C1B—C1B1	−176.7 (2)	C1B1—C1B—C1B2—C1G2	53.6 (3)
C0′—N1—C1B—C1B2	−58.4 (3)	C1G2—C1D—C1G1—C1B1	−54.3 (4)
N1—C1B—C1A—C1′	57.2 (3)	C1B—C1B1—C1G1—C1D	53.7 (3)
C1B1—C1B—C1A—C1′	−61.5 (3)	C1G1—C1D—C1G2—C1B2	56.2 (4)
C1B2—C1B—C1A—C1′	178.5 (2)	C1B—C1B2—C1G2—C1D	−57.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···O0^{i}	0.87 (4)	1.74 (4)	2.599 (3)	166 (4)
N1—H1*N*···O1^{ii}	0.82 (3)	2.16 (3)	2.981 (3)	172 (2)

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

(II) 2-(1-{[(9H-fluoren-9-yloxy)carbonyl]amino}cyclohexyl)acetic acid

Crystal data

$C_{23}H_{25}NO_4$
M_r = 379.44
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.0834 (4) Å
b = 12.7642 (9) Å
c = 12.8399 (9) Å
α = 94.018 (6)°
β = 92.295 (6)°
γ = 100.489 (6)°
V = 976.53 (12) Å^3
Z = 2
$F(000)$ = 404
D_x = 1.290 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2812 reflections
θ = 3.5–27.0°
μ = 0.09 mm^{-1}
T = 291 K
Needle, colorless
0.30 × 0.05 × 0.03 mm

Data collection

Oxford Diffraction Xcalibur, Sapphire3 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.947, T_{max} = 1.000
7781 measured reflections
4166 independent reflections
2037 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.0°, θ_{min} = 3.5°
h = −7→6
k = −15→16
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.086
S = 0.97
4166 reflections
353 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0085P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Experimental. *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27–08-2010 CrysAlis171. NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8803 (4)	−0.19879 (17)	−0.39027 (18)	0.0398 (6)
C2	1.0680 (4)	−0.1766 (2)	−0.4476 (2)	0.0490 (7)
C3	1.1160 (5)	−0.2575 (2)	−0.5173 (2)	0.0548 (8)
C4	0.9775 (5)	−0.3561 (2)	−0.5289 (2)	0.0544 (8)
C5	0.7899 (4)	−0.3786 (2)	−0.47135 (19)	0.0471 (7)
C6	0.7413 (4)	−0.29817 (17)	−0.40208 (18)	0.0381 (6)
C7	0.5582 (4)	−0.29905 (18)	−0.33106 (18)	0.0399 (6)
C8	0.3733 (4)	−0.3763 (2)	−0.3161 (2)	0.0503 (7)
C9	0.2213 (5)	−0.3534 (3)	−0.2458 (2)	0.0598 (8)
C10	0.2509 (5)	−0.2550 (3)	−0.1896 (2)	0.0631 (9)
C11	0.4356 (5)	−0.1777 (2)	−0.2032 (2)	0.0555 (8)
C12	0.5888 (4)	−0.19843 (17)	−0.27442 (18)	0.0411 (6)
C13	0.7945 (4)	−0.12711 (19)	−0.3089 (2)	0.0437 (7)
C14	0.7453 (5)	−0.0259 (2)	−0.3520 (2)	0.0478 (7)
O	0.7045 (3)	0.04337 (11)	−0.26348 (13)	0.0488 (5)
C0′	0.5479 (4)	0.10582 (18)	−0.2765 (2)	0.0395 (6)
O0	0.4294 (3)	0.10122 (13)	−0.35483 (14)	0.0588 (5)
N1	0.5526 (3)	0.17073 (15)	−0.18907 (17)	0.0372 (5)
C1B	0.3973 (4)	0.24494 (17)	−0.17036 (18)	0.0361 (6)
C1B1	0.4539 (4)	0.2995 (2)	−0.0596 (2)	0.0430 (7)
C1G1	0.6806 (5)	0.3738 (2)	−0.0476 (2)	0.0521 (8)
C1D	0.7028 (6)	0.4553 (2)	−0.1294 (3)	0.0630 (9)
C1G2	0.6576 (5)	0.4007 (2)	−0.2385 (2)	0.0529 (8)
C1B2	0.4262 (4)	0.3298 (2)	−0.2497 (2)	0.0455 (7)
C1A	0.1535 (4)	0.1842 (2)	−0.1806 (2)	0.0442 (7)
C1′	0.0895 (4)	0.10713 (19)	−0.0994 (2)	0.0435 (7)
O1	−0.0602 (3)	0.11924 (12)	−0.03783 (13)	0.0536 (5)
O2	0.1903 (3)	0.02715 (16)	−0.09706 (17)	0.0658 (6)

H1	1.169 (3)	−0.1087 (16)	−0.4336 (16)	0.057 (8)*
H2	1.250 (4)	−0.2404 (17)	−0.5581 (18)	0.071 (8)*
H3	1.010 (3)	−0.4159 (15)	−0.5793 (17)	0.057 (7)*
H4	0.697 (3)	−0.4510 (15)	−0.4749 (15)	0.048 (7)*
H5	0.355 (3)	−0.4462 (16)	−0.3585 (17)	0.057 (7)*
H6	0.086 (4)	−0.4073 (18)	−0.2391 (18)	0.076 (9)*
H7	0.130 (4)	−0.2404 (17)	−0.1414 (19)	0.079 (9)*
H8	0.465 (3)	−0.1068 (16)	−0.1668 (17)	0.059 (8)*
H9	0.901 (3)	−0.1079 (14)	−0.2496 (15)	0.034 (6)*
H10	0.877 (3)	0.0150 (15)	−0.3877 (16)	0.047 (7)*
H11	0.613 (3)	−0.0384 (15)	−0.4024 (17)	0.051 (7)*
H1N	0.638 (4)	0.1617 (17)	−0.1365 (18)	0.056 (9)*
H1B1	0.444 (3)	0.2449 (15)	−0.0120 (16)	0.037 (7)*
H1B2	0.325 (3)	0.3423 (15)	−0.0449 (16)	0.052 (7)*
H1G1	0.812 (4)	0.3321 (16)	−0.0523 (18)	0.068 (8)*
H1G2	0.702 (3)	0.4120 (15)	0.0257 (18)	0.060 (8)*
H1D1	0.587 (4)	0.5056 (18)	−0.116 (2)	0.084 (9)*
H1D2	0.857 (4)	0.4989 (18)	−0.1182 (19)	0.079 (9)*
H1G3	0.670 (3)	0.4543 (16)	−0.2899 (17)	0.049 (7)*
H1G4	0.778 (4)	0.3541 (16)	−0.2585 (17)	0.066 (8)*
H1B3	0.393 (3)	0.2939 (14)	−0.3222 (16)	0.046 (7)*
H1B4	0.309 (3)	0.3775 (16)	−0.2388 (17)	0.064 (8)*
H1A1	0.127 (3)	0.1439 (15)	−0.2528 (17)	0.055 (7)*
H1A2	0.052 (3)	0.2388 (15)	−0.1722 (16)	0.047 (7)*
H2O	0.145 (7)	−0.020 (2)	−0.056 (3)	0.24 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0446 (16)	0.0360 (14)	0.0398 (16)	0.0111 (12)	−0.0001 (13)	0.0028 (11)
C2	0.0463 (17)	0.0416 (17)	0.058 (2)	0.0050 (14)	0.0053 (15)	0.0040 (14)
C3	0.0535 (19)	0.061 (2)	0.054 (2)	0.0183 (16)	0.0160 (16)	0.0093 (15)
C4	0.067 (2)	0.0512 (18)	0.0471 (19)	0.0202 (15)	0.0060 (16)	−0.0031 (14)
C5	0.0555 (18)	0.0407 (16)	0.0435 (17)	0.0061 (14)	0.0051 (14)	−0.0028 (13)
C6	0.0416 (15)	0.0383 (14)	0.0353 (15)	0.0102 (11)	−0.0006 (12)	0.0037 (11)
C7	0.0448 (15)	0.0407 (15)	0.0353 (15)	0.0093 (12)	0.0023 (12)	0.0060 (11)
C8	0.0568 (18)	0.0487 (18)	0.0450 (18)	0.0062 (14)	0.0049 (15)	0.0087 (14)
C9	0.054 (2)	0.069 (2)	0.056 (2)	0.0042 (17)	0.0124 (16)	0.0213 (17)
C10	0.066 (2)	0.077 (2)	0.054 (2)	0.0248 (18)	0.0204 (17)	0.0204 (17)
C11	0.071 (2)	0.0522 (19)	0.0488 (19)	0.0232 (16)	0.0135 (16)	0.0056 (15)
C12	0.0479 (16)	0.0407 (15)	0.0379 (16)	0.0143 (12)	0.0042 (13)	0.0072 (12)
C13	0.0486 (17)	0.0367 (15)	0.0454 (18)	0.0107 (12)	−0.0022 (14)	−0.0042 (13)
C14	0.0536 (19)	0.0356 (16)	0.055 (2)	0.0122 (14)	0.0030 (16)	−0.0020 (14)
O	0.0498 (11)	0.0413 (10)	0.0570 (13)	0.0208 (8)	−0.0078 (9)	−0.0100 (9)
C0′	0.0348 (15)	0.0326 (14)	0.0521 (19)	0.0067 (11)	0.0058 (14)	0.0065 (13)
O0	0.0638 (13)	0.0649 (13)	0.0499 (13)	0.0251 (10)	−0.0115 (10)	−0.0056 (10)
N1	0.0358 (13)	0.0366 (12)	0.0408 (14)	0.0120 (9)	−0.0009 (11)	0.0014 (10)
C1B	0.0363 (14)	0.0370 (14)	0.0382 (15)	0.0120 (11)	0.0072 (12)	0.0098 (11)

C1B1	0.0435 (17)	0.0433 (17)	0.0435 (18)	0.0086 (13)	0.0103 (14)	0.0071 (14)
C1G1	0.0495 (18)	0.0536 (19)	0.050 (2)	0.0031 (14)	0.0040 (15)	−0.0040 (15)
C1D	0.065 (2)	0.051 (2)	0.068 (2)	−0.0042 (17)	0.0182 (19)	−0.0004 (17)
C1G2	0.059 (2)	0.0454 (18)	0.058 (2)	0.0103 (15)	0.0223 (17)	0.0186 (15)
C1B2	0.0498 (18)	0.0445 (17)	0.0470 (19)	0.0172 (13)	0.0094 (15)	0.0112 (14)
C1A	0.0352 (16)	0.0480 (17)	0.0523 (19)	0.0111 (13)	0.0065 (14)	0.0121 (14)
C1′	0.0312 (15)	0.0429 (16)	0.0561 (19)	0.0070 (12)	0.0001 (13)	0.0035 (13)
O1	0.0495 (11)	0.0609 (12)	0.0569 (13)	0.0208 (9)	0.0189 (10)	0.0115 (9)
O2	0.0678 (14)	0.0521 (13)	0.0884 (17)	0.0271 (10)	0.0316 (12)	0.0240 (11)

Geometric parameters (Å, º)

C1—C2	1.381 (3)	O—C0′	1.362 (3)
C1—C6	1.385 (3)	C0′—O0	1.205 (3)
C1—C13	1.511 (3)	C0′—N1	1.344 (3)
C2—C3	1.398 (3)	N1—C1B	1.469 (3)
C2—H1	0.967 (19)	N1—H1N	0.86 (2)
C3—C4	1.375 (3)	C1B—C1B2	1.530 (3)
C3—H2	0.98 (2)	C1B—C1B1	1.536 (3)
C4—C5	1.382 (3)	C1B—C1A	1.540 (3)
C4—H3	1.017 (18)	C1B1—C1G1	1.521 (3)
C5—C6	1.391 (3)	C1B1—H1B1	0.95 (2)
C5—H4	0.989 (18)	C1B1—H1B2	1.048 (19)
C6—C7	1.467 (3)	C1G1—C1D	1.522 (4)
C7—C8	1.384 (3)	C1G1—H1G1	1.04 (2)
C7—C12	1.409 (3)	C1G1—H1G2	1.02 (2)
C8—C9	1.372 (3)	C1D—C1G2	1.512 (4)
C8—H5	0.997 (18)	C1D—H1D1	1.05 (2)
C9—C10	1.384 (3)	C1D—H1D2	1.00 (2)
C9—H6	0.98 (2)	C1G2—C1B2	1.524 (3)
C10—C11	1.379 (3)	C1G2—H1G3	0.98 (2)
C10—H7	1.01 (2)	C1G2—H1G4	1.05 (2)
C11—C12	1.378 (3)	C1B2—H1B3	1.005 (19)
C11—H8	0.971 (19)	C1B2—H1B4	1.03 (2)
C12—C13	1.509 (3)	C1A—C1′	1.497 (3)
C13—C14	1.514 (3)	C1A—H1A1	1.020 (19)
C13—H9	0.968 (19)	C1A—H1A2	1.015 (18)
C14—O	1.449 (3)	C1′—O1	1.253 (2)
C14—H10	1.017 (19)	C1′—O2	1.284 (3)
C14—H11	0.99 (2)	O2—H2O	0.84 (1)
C2—C1—C6	121.1 (2)	N1—C0′—O	108.3 (2)
C2—C1—C13	128.7 (2)	C0′—N1—C1B	124.5 (2)
C6—C1—C13	110.2 (2)	C0′—N1—H1N	118.0 (15)
C1—C2—C3	118.2 (2)	C1B—N1—H1N	116.8 (15)
C1—C2—H1	119.2 (12)	N1—C1B—C1B2	110.33 (19)
C3—C2—H1	122.3 (12)	N1—C1B—C1B1	107.6 (2)
C4—C3—C2	120.5 (3)	C1B2—C1B—C1B1	109.3 (2)

C4—C3—H2	122.1 (13)	N1—C1B—C1A	110.39 (19)
C2—C3—H2	117.4 (13)	C1B2—C1B—C1A	108.7 (2)
C3—C4—C5	121.3 (2)	C1B1—C1B—C1A	110.59 (19)
C3—C4—H3	121.6 (12)	C1G1—C1B1—C1B	113.6 (2)
C5—C4—H3	117.0 (12)	C1G1—C1B1—H1B1	111.6 (13)
C4—C5—C6	118.3 (2)	C1B—C1B1—H1B1	107.8 (12)
C4—C5—H4	121.6 (11)	C1G1—C1B1—H1B2	110.5 (10)
C6—C5—H4	119.9 (11)	C1B—C1B1—H1B2	105.2 (12)
C5—C6—C1	120.5 (2)	H1B1—C1B1—H1B2	107.7 (15)
C5—C6—C7	130.4 (2)	C1D—C1G1—C1B1	111.4 (3)
C1—C6—C7	109.07 (18)	C1D—C1G1—H1G1	109.3 (13)
C8—C7—C12	120.0 (2)	C1B1—C1G1—H1G1	112.1 (12)
C8—C7—C6	131.7 (2)	C1D—C1G1—H1G2	110.1 (12)
C12—C7—C6	108.2 (2)	C1B1—C1G1—H1G2	109.5 (12)
C9—C8—C7	119.2 (2)	H1G1—C1G1—H1G2	104.4 (18)
C9—C8—H5	122.7 (12)	C1G2—C1D—C1G1	111.0 (2)
C7—C8—H5	118.1 (12)	C1G2—C1D—H1D1	108.9 (15)
C8—C9—C10	121.1 (3)	C1G1—C1D—H1D1	109.0 (15)
C8—C9—H6	118.4 (13)	C1G2—C1D—H1D2	112.7 (14)
C10—C9—H6	120.4 (13)	C1G1—C1D—H1D2	106.5 (16)
C11—C10—C9	120.2 (3)	H1D1—C1D—H1D2	108.6 (18)
C11—C10—H7	121.5 (13)	C1D—C1G2—C1B2	110.8 (2)
C9—C10—H7	118.2 (13)	C1D—C1G2—H1G3	109.8 (12)
C10—C11—C12	119.6 (3)	C1B2—C1G2—H1G3	110.1 (13)
C10—C11—H8	124.3 (13)	C1D—C1G2—H1G4	112.2 (13)
C12—C11—H8	116.0 (13)	C1B2—C1G2—H1G4	109.0 (11)
C11—C12—C7	119.8 (2)	H1G3—C1G2—H1G4	104.7 (16)
C11—C12—C13	130.4 (2)	C1G2—C1B2—C1B	112.3 (2)
C7—C12—C13	109.7 (2)	C1G2—C1B2—H1B3	111.5 (11)
C1—C13—C12	102.78 (18)	C1B—C1B2—H1B3	109.3 (11)
C1—C13—C14	112.5 (2)	C1G2—C1B2—H1B4	108.2 (12)
C12—C13—C14	113.2 (2)	C1B—C1B2—H1B4	109.2 (13)
C1—C13—H9	110.8 (11)	H1B3—C1B2—H1B4	106.1 (17)
C12—C13—H9	108.6 (12)	C1′—C1A—C1B	115.4 (2)
C14—C13—H9	108.9 (11)	C1′—C1A—H1A1	109.0 (12)
O—C14—C13	106.8 (2)	C1B—C1A—H1A1	108.1 (12)
O—C14—H10	106.4 (11)	C1′—C1A—H1A2	105.6 (12)
C13—C14—H10	113.1 (12)	C1B—C1A—H1A2	107.8 (11)
O—C14—H11	109.1 (11)	H1A1—C1A—H1A2	110.9 (17)
C13—C14—H11	113.5 (12)	O1—C1′—O2	121.5 (2)
H10—C14—H11	107.6 (18)	O1—C1′—C1A	121.1 (2)
C0′—O—C14	118.1 (2)	O2—C1′—C1A	117.4 (2)
O0—C0′—N1	127.8 (2)	C1′—O2—H2O	118 (3)
O0—C0′—O	123.9 (2)		

C6—C1—C2—C3	−0.7 (4)	C11—C12—C13—C1	−178.4 (3)
C13—C1—C2—C3	178.6 (3)	C7—C12—C13—C1	−0.9 (3)
C1—C2—C3—C4	0.8 (4)	C11—C12—C13—C14	−56.8 (4)

C2—C3—C4—C5	−1.0 (5)	C7—C12—C13—C14	120.6 (2)
C3—C4—C5—C6	1.1 (4)	C1—C13—C14—O	−169.9 (2)
C4—C5—C6—C1	−1.0 (4)	C12—C13—C14—O	74.2 (3)
C4—C5—C6—C7	−179.3 (3)	C13—C14—O—C0′	−144.5 (2)
C2—C1—C6—C5	0.9 (4)	C14—O—C0′—O0	5.7 (4)
C13—C1—C6—C5	−178.5 (2)	C14—O—C0′—N1	−174.0 (2)
C2—C1—C6—C7	179.4 (2)	O0—C0′—N1—C1B	4.6 (4)
C13—C1—C6—C7	0.0 (3)	O—C0′—N1—C1B	−175.64 (19)
C5—C6—C7—C8	−4.6 (5)	C0′—N1—C1B—C1B2	−63.4 (3)
C1—C6—C7—C8	177.0 (3)	C0′—N1—C1B—C1B1	177.4 (2)
C5—C6—C7—C12	177.7 (3)	C0′—N1—C1B—C1A	56.7 (3)
C1—C6—C7—C12	−0.6 (3)	N1—C1B—C1B1—C1G1	67.5 (3)
C12—C7—C8—C9	0.1 (4)	C1B2—C1B—C1B1—C1G1	−52.3 (3)
C6—C7—C8—C9	−177.3 (3)	C1A—C1B—C1B1—C1G1	−171.9 (2)
C7—C8—C9—C10	−0.4 (5)	C1B—C1B1—C1G1—C1D	53.3 (3)
C8—C9—C10—C11	−0.2 (5)	C1B1—C1G1—C1D—C1G2	−54.8 (3)
C9—C10—C11—C12	1.1 (5)	C1G1—C1D—C1G2—C1B2	57.1 (3)
C10—C11—C12—C7	−1.4 (4)	C1D—C1G2—C1B2—C1B	−57.8 (3)
C10—C11—C12—C13	175.9 (3)	N1—C1B—C1B2—C1G2	−63.9 (3)
C8—C7—C12—C11	0.8 (4)	C1B1—C1B—C1B2—C1G2	54.1 (3)
C6—C7—C12—C11	178.8 (2)	C1A—C1B—C1B2—C1G2	174.9 (2)
C8—C7—C12—C13	−177.0 (2)	N1—C1B—C1A—C1′	66.1 (3)
C6—C7—C12—C13	1.0 (3)	C1B2—C1B—C1A—C1′	−172.8 (2)
C2—C1—C13—C12	−178.8 (3)	C1B1—C1B—C1A—C1′	−52.8 (3)
C6—C1—C13—C12	0.5 (3)	C1B—C1A—C1′—O1	117.9 (2)
C2—C1—C13—C14	59.1 (4)	C1B—C1A—C1′—O2	−63.6 (3)
C6—C1—C13—C14	−121.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.86 (2)	2.35 (2)	3.182 (3)	161 (2)
O2—H2*O*···O1^{ii}	0.84 (3)	1.83 (3)	2.673 (3)	177 (1)

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

(III) 2-[1-(Pyrazine-2-amido)cyclohexyl]acetic acid

Crystal data

$C_{13}H_{17}N_3O_3$
M_r = 263.30
Orthorhombic, *Pca*2$_1$
Hall symbol: P 2c -2ac
a = 8.7135 (1) Å
b = 10.5321 (1) Å
c = 14.3907 (2) Å
V = 1320.66 (3) Å^3
Z = 4

F(000) = 560
D_x = 1.324 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 28796 reflections
θ = 3.7–27.0°
μ = 0.10 mm^{-1}
T = 291 K
Cube, colorless
0.25 × 0.25 × 0.25 mm

Data collection

Oxford Difraction Xcalibur, Sapphire3 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1049 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.931, T_{max} = 1.000

68869 measured reflections
2878 independent reflections
2670 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.0°, θ_{min} = 3.9°
h = −11→11
k = −13→13
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.106
S = 1.04
2878 reflections
240 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.4913P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.032
$\Delta\rho_{max}$ = 0.27 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.26 e $Å^{-3}$
Absolute structure: (Flack, 1983): 1585 Friedel pairs
Absolute structure parameter: 0.2 (14)

Special details

Experimental. *CrysAlis PRO*, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27–08-2010 CrysAlis171. NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
H1	0.902 (2)	0.412 (2)	1.0592 (15)	0.030 (5)*
H1B2	1.400 (3)	0.435 (2)	1.2329 (16)	0.041 (6)*
H1B5	1.272 (3)	0.384 (2)	1.4161 (18)	0.049 (7)*
H1B6	1.183 (3)	0.442 (2)	1.3342 (17)	0.048 (6)*
H1B1	1.499 (2)	0.380 (2)	1.3191 (16)	0.033 (5)*
H1D2	1.651 (3)	0.100 (3)	1.200 (2)	0.061 (7)*
H1N	1.161 (2)	0.172 (2)	1.2259 (16)	0.036 (6)*
H1G1	1.481 (3)	0.256 (2)	1.1391 (19)	0.045 (6)*
H2	0.709 (3)	0.107 (2)	0.971 (2)	0.051 (7)*
H3	0.894 (3)	−0.021 (2)	1.0429 (17)	0.047 (6)*
H1B4	1.269 (3)	0.107 (2)	1.3669 (17)	0.045 (6)*

H1G4	1.401 (3)	0.050 (3)	1.232 (2)	0.061 (7)*
H1G2	1.616 (3)	0.323 (3)	1.173 (2)	0.066 (8)*
H1B3	1.415 (3)	0.192 (2)	1.3989 (19)	0.046 (6)*
H1G3	1.502 (3)	0.005 (3)	1.3184 (19)	0.065 (8)*
H1D1	1.667 (4)	0.176 (3)	1.298 (2)	0.071 (9)*
H21	0.885 (5)	0.352 (4)	1.458 (3)	0.106 (13)*
C1	0.9028 (2)	0.3259 (2)	1.05370 (16)	0.0401 (4)
N2	0.7893 (2)	0.27249 (19)	1.00502 (14)	0.0442 (4)
C3	0.7875 (3)	0.1459 (2)	1.00191 (15)	0.0433 (5)
C4	0.8954 (3)	0.0736 (2)	1.04730 (16)	0.0450 (5)
N3	1.0074 (2)	0.12584 (16)	1.09722 (13)	0.0398 (4)
C6	1.0105 (2)	0.25193 (18)	1.10026 (14)	0.0343 (4)
C0	1.1318 (2)	0.31669 (18)	1.15876 (15)	0.0382 (4)
O0′	1.1604 (2)	0.42890 (15)	1.14626 (15)	0.0658 (6)
N1	1.19610 (18)	0.24153 (15)	1.22264 (12)	0.0348 (4)
C1A	1.2995 (2)	0.28157 (17)	1.29864 (13)	0.0311 (4)
C1B	1.2100 (2)	0.36660 (19)	1.36671 (16)	0.0381 (4)
C1′	1.0648 (2)	0.31013 (19)	1.40619 (15)	0.0423 (5)
O1	1.0357 (3)	0.2011 (2)	1.4129 (3)	0.1197 (13)
O2	0.9700 (2)	0.39599 (18)	1.43574 (16)	0.0677 (6)
C1B1	1.4369 (2)	0.3562 (2)	1.26078 (16)	0.0394 (4)
C1G1	1.5373 (3)	0.2777 (2)	1.19617 (18)	0.0504 (5)
C1D	1.5948 (3)	0.1582 (3)	1.2448 (2)	0.0575 (6)
C1B2	1.3586 (2)	0.15966 (19)	1.34564 (14)	0.0366 (4)
C1G2	1.4634 (3)	0.0817 (2)	1.28311 (18)	0.0483 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0386 (10)	0.0391 (10)	0.0428 (10)	0.0036 (9)	−0.0049 (9)	0.0025 (10)
N2	0.0374 (8)	0.0538 (11)	0.0414 (9)	0.0036 (8)	−0.0067 (7)	0.0039 (8)
C3	0.0402 (11)	0.0513 (13)	0.0386 (11)	−0.0063 (9)	−0.0045 (9)	−0.0018 (10)
C4	0.0510 (12)	0.0399 (11)	0.0440 (10)	−0.0049 (10)	−0.0066 (9)	−0.0016 (9)
N3	0.0445 (9)	0.0361 (8)	0.0389 (9)	−0.0002 (7)	−0.0048 (8)	0.0035 (7)
C6	0.0340 (9)	0.0368 (10)	0.0321 (9)	0.0012 (7)	0.0002 (7)	0.0023 (8)
C0	0.0388 (10)	0.0335 (9)	0.0422 (11)	0.0024 (8)	−0.0048 (8)	0.0032 (8)
O0′	0.0769 (12)	0.0360 (8)	0.0843 (13)	−0.0095 (8)	−0.0359 (11)	0.0160 (8)
N1	0.0365 (8)	0.0291 (8)	0.0388 (9)	−0.0040 (6)	−0.0074 (7)	0.0016 (6)
C1A	0.0299 (8)	0.0305 (9)	0.0329 (9)	−0.0015 (7)	0.0010 (7)	−0.0030 (7)
C1B	0.0352 (10)	0.0327 (10)	0.0464 (11)	−0.0002 (8)	0.0058 (9)	−0.0069 (8)
C1′	0.0418 (11)	0.0358 (10)	0.0494 (12)	−0.0009 (9)	0.0134 (9)	−0.0037 (9)
O1	0.0886 (16)	0.0440 (10)	0.227 (3)	−0.0088 (10)	0.099 (2)	−0.0111 (15)
O2	0.0533 (10)	0.0462 (9)	0.1035 (15)	0.0071 (8)	0.0373 (10)	0.0115 (9)
C1B1	0.0322 (9)	0.0395 (11)	0.0464 (11)	−0.0066 (8)	0.0044 (9)	−0.0020 (9)
C1G1	0.0453 (12)	0.0557 (13)	0.0500 (13)	−0.0058 (11)	0.0180 (11)	−0.0036 (11)
C1D	0.0454 (12)	0.0660 (16)	0.0611 (16)	0.0172 (11)	0.0121 (12)	−0.0050 (12)
C1B2	0.0371 (10)	0.0398 (9)	0.0329 (10)	0.0042 (8)	−0.0012 (8)	0.0026 (8)
C1G2	0.0551 (13)	0.0404 (11)	0.0493 (12)	0.0171 (10)	0.0039 (11)	−0.0012 (9)

Geometric parameters (Å, º)

C1—N2	1.337 (3)	C1B—H1B6	0.95 (3)
C1—C6	1.391 (3)	C1′—O1	1.180 (3)
C1—H1	0.91 (2)	C1′—O2	1.297 (3)
N2—C3	1.334 (3)	O2—H21	0.93 (5)
C3—C4	1.375 (3)	C1B1—C1G1	1.521 (3)
C3—H2	0.91 (3)	C1B1—H1B2	0.97 (2)
C4—N3	1.331 (3)	C1B1—H1B1	1.03 (2)
C4—H3	1.00 (3)	C1G1—C1D	1.525 (4)
N3—C6	1.329 (3)	C1G1—H1G1	0.98 (3)
C6—C0	1.514 (3)	C1G1—H1G2	0.90 (3)
C0—O0′	1.221 (2)	C1D—C1G2	1.505 (4)
C0—N1	1.336 (3)	C1D—H1D2	1.01 (3)
N1—C1A	1.478 (2)	C1D—H1D1	1.00 (3)
N1—H1N	0.79 (2)	C1B2—C1G2	1.523 (3)
C1A—C1B1	1.532 (3)	C1B2—H1B4	1.00 (2)
C1A—C1B	1.539 (2)	C1B2—H1B3	0.97 (3)
C1A—C1B2	1.540 (3)	C1G2—H1G4	0.97 (3)
C1B—C1′	1.509 (3)	C1G2—H1G3	1.01 (3)
C1B—H1B5	0.91 (3)		

N2—C1—C6	121.03 (19)	O1—C1′—C1B	126.5 (2)
N2—C1—H1	117.5 (14)	O2—C1′—C1B	112.51 (18)
C6—C1—H1	121.3 (14)	C1′—O2—H21	106 (3)
C3—N2—C1	116.57 (18)	C1G1—C1B1—C1A	112.85 (17)
N2—C3—C4	122.0 (2)	C1G1—C1B1—H1B2	113.6 (14)
N2—C3—H2	118.2 (16)	C1A—C1B1—H1B2	109.0 (14)
C4—C3—H2	119.8 (16)	C1G1—C1B1—H1B1	108.9 (12)
N3—C4—C3	121.9 (2)	C1A—C1B1—H1B1	104.3 (12)
N3—C4—H3	117.3 (15)	H1B2—C1B1—H1B1	107.7 (18)
C3—C4—H3	120.8 (15)	C1B1—C1G1—C1D	110.9 (2)
C6—N3—C4	116.44 (19)	C1B1—C1G1—H1G1	110.5 (15)
N3—C6—C1	122.0 (2)	C1D—C1G1—H1G1	111.0 (15)
N3—C6—C0	118.85 (18)	C1B1—C1G1—H1G2	112.3 (19)
C1—C6—C0	119.08 (17)	C1D—C1G1—H1G2	111.1 (19)
O0′—C0—N1	126.08 (19)	H1G1—C1G1—H1G2	101 (2)
O0′—C0—C6	119.79 (18)	C1G2—C1D—C1G1	111.1 (2)
N1—C0—C6	114.12 (17)	C1G2—C1D—H1D2	106.1 (16)
C0—N1—C1A	126.55 (16)	C1G1—C1D—H1D2	111.2 (16)
C0—N1—H1N	115.3 (17)	C1G2—C1D—H1D1	107.4 (17)
C1A—N1—H1N	116.8 (17)	C1G1—C1D—H1D1	113.3 (18)
N1—C1A—C1B1	111.06 (16)	H1D2—C1D—H1D1	107 (2)
N1—C1A—C1B	109.15 (15)	C1G2—C1B2—C1A	113.00 (17)
C1B1—C1A—C1B	108.90 (15)	C1G2—C1B2—H1B4	110.6 (13)
N1—C1A—C1B2	106.91 (15)	C1A—C1B2—H1B4	109.3 (13)
C1B1—C1A—C1B2	108.81 (16)	C1G2—C1B2—H1B3	110.4 (15)
C1B—C1A—C1B2	112.02 (16)	C1A—C1B2—H1B3	102.9 (15)

C1′—C1B—C1A	115.79 (16)	H1B4—C1B2—H1B3	110 (2)
C1′—C1B—H1B5	106.5 (16)	C1D—C1G2—C1B2	112.6 (2)
C1A—C1B—H1B5	108.4 (17)	C1D—C1G2—H1G4	109.7 (17)
C1′—C1B—H1B6	108.0 (15)	C1B2—C1G2—H1G4	107.0 (16)
C1A—C1B—H1B6	107.2 (15)	C1D—C1G2—H1G3	111.0 (17)
H1B5—C1B—H1B6	111 (2)	C1B2—C1G2—H1G3	109.4 (16)
O1—C1′—O2	121.0 (2)	H1G4—C1G2—H1G3	107 (2)
C6—C1—N2—C3	−1.5 (3)	C0—N1—C1A—C1B2	−173.2 (2)
C1—N2—C3—C4	0.8 (3)	N1—C1A—C1B—C1′	55.0 (2)
N2—C3—C4—N3	0.3 (4)	C1B1—C1A—C1B—C1′	176.42 (19)
C3—C4—N3—C6	−0.6 (3)	C1B2—C1A—C1B—C1′	−63.2 (2)
C4—N3—C6—C1	−0.2 (3)	C1A—C1B—C1′—O1	23.6 (4)
C4—N3—C6—C0	177.82 (19)	C1A—C1B—C1′—O2	−157.9 (2)
N2—C1—C6—N3	1.3 (3)	N1—C1A—C1B1—C1G1	−62.6 (2)
N2—C1—C6—C0	−176.68 (19)	C1B—C1A—C1B1—C1G1	177.20 (19)
N3—C6—C0—O0′	162.9 (2)	C1B2—C1A—C1B1—C1G1	54.8 (2)
C1—C6—C0—O0′	−19.1 (3)	C1A—C1B1—C1G1—C1D	−57.0 (3)
N3—C6—C0—N1	−18.5 (3)	C1B1—C1G1—C1D—C1G2	55.1 (3)
C1—C6—C0—N1	159.53 (19)	N1—C1A—C1B2—C1G2	67.3 (2)
O0′—C0—N1—C1A	8.4 (4)	C1B1—C1A—C1B2—C1G2	−52.7 (2)
C6—C0—N1—C1A	−170.04 (17)	C1B—C1A—C1B2—C1G2	−173.13 (18)
C0—N1—C1A—C1B1	−54.6 (2)	C1G1—C1D—C1G2—C1B2	−53.8 (3)
C0—N1—C1A—C1B	65.5 (2)	C1A—C1B2—C1G2—C1D	53.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H21···N2^{i}	0.93 (4)	1.86 (4)	2.791 (3)	177 (4)
N1—H1*N*···N3	0.79 (2)	2.34 (2)	2.729 (2)	111.3 (19)

Symmetry code: (i) −*x*+3/2, *y*, *z*+1/2.