The effect of partial methylation of the glycine amino group on crystal structure in *N,N*-dimethylglycine and its hemihydrate

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N,N-Dimethylglycine, $C_4H_9NO_2$, and its hemihydrate, $C_4H_9NO_2 \cdot 0.5H_2O$, are discussed in order to follow the effect of the methylation of the glycine amino group (and thus its ability to form several hydrogen bonds) on crystal structure, in particular on the possibility of the formation of hydrogen-bonded 'head-to-tail' chains, which are typical for the crystal structures of amino acids and essential for considering amino acid crystals as mimics of peptide chains. Both compounds crystallize in centrosymmetric space groups (*Pbca* and *C*2/*c*, respectively) and have two *N,N*-dimethylglycine zwitterions in the asymmetric unit. In the anhydrous compound, there are no head-to-tail chains but the zwitterions form $R_4^4(20)$ ring motifs, which are not bonded to each other by any hydrogen bonds. In contrast, in the crystal structure of *N,N*-dimethylglycinium hemihydrate, the zwitterions are linked to each other by N—H⋯O hydrogen bonds into infinite $C_2^2(10)$ head-to-tail chains, while the water molecules outside the chains provide additional hydrogen bonds to the carboxylate groups.

Comment

Glycine is the smallest amino acid and has no chiral C atom. Despite this simplicity it attracts great interest, having been studied extensively both as an individual molecule in the gas phase or in inert matrixes, and in the bulk in solution and in the solid state (Boldyreva, 2008, and references therein). Under ambient conditions, as shown in the pioneering work by Bernal (1931), glycine crystallizes in three different polymorphs, namely the α-, β- and γ-forms, depending on subtle changes in the crystallization conditions. The zwitterions of glycine in these polymorphs form 'head-to-tail' chains, linked *via* N—H⋯O hydrogen bonds between the terminal amino and carboxylate groups (Marsh, 1958; Iitaka, 1960, 1961), which can mimic peptide chains. These head-to-tail chains are very robust, and are preserved after solid-state polymorphic

transitions induced by variations in temperature (Boldyreva *et al.*, 2003) or pressure (Boldyreva *et al.*, 2004, 2005; Dawson *et al.*, 2005; Goryainov *et al.*, 2005, 2006).

Such head-to-tail chains are present in the crystal structures of all other α-amino acids (Suresh & Vijayan, 1983; Boldyreva, 2008; Görbitz, 2010) and they are essential for considering amino acid crystals as mimics of peptide chains. The formation of these head-to-tail chains is favoured by dipole–dipole interactions and agrees with optimizing the close packing. For example, in *N,N,N*-trimethylglycine (Viertorinne *et al.*, 1999), where no hydrogen bonds are possible, head-to-tail chains of zwitterions are formed because of Coulombic forces between the dipoles. In addition to these factors, the very possibility of the formation of these chains, as well as their robustness and dynamics on variations in pressure and temperature, have been supposed to be closely related to the ability of the terminal amino groups to form additional hydrogen bonds to neighbouring molecules outside the chain (Kolesov & Boldyreva, 2010, 2011). Excluding these additional hydrogen bonds, for example, by partial methylation of the amino group, may therefore have a significant effect on the crystal packing.

Hitherto, only the crystal structure of *N*-methylglycine (Trzebiatowska-Gusowska & Gagor, 2007; Dittrich & Spackman, 2007) has been published, and the head-to-tail chains are present therein. In *N*-methylglycine, an amino group forms a second hydrogen bond, in addition to that linking the zwitterions within a chain, and this agrees with the hypothesis that such additional hydrogen bonds are essential for head-to-tail chain formation. We have therefore decided to study the crystal structures of *N,N*-dimethylglycine, (I), and its hemihydrate, (II). In (I), only one type of N—H⋯O hydrogen bond is possible and one might expect that no head-to-tail chains can be formed. In (II), the presence of water molecules could give a chance of forming additional hydrogen bonds, and thus head-to-tail chains might become possible. Our present study has confirmed our original hypothesis and provides a successful example of crystal engineering.
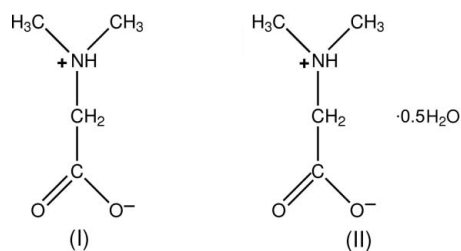
Compound (I) crystallizes in the orthorhombic system (space group *Pbca*), as is the case for mono- and trimethylglycine, whereas (II) crystallizes in the monoclinic space group *C*2/*c*. In both crystal structures, there are two *N,N*-dimethylglycine zwitterions, *A* and *B*, in the asymmetric unit with different molecular conformations (see Fig. 1 and selected torsion angles in Table 1). For previously known structures, an increase in methylation of an amino group results in a decrease in the twisting of the molecule, which can be char-
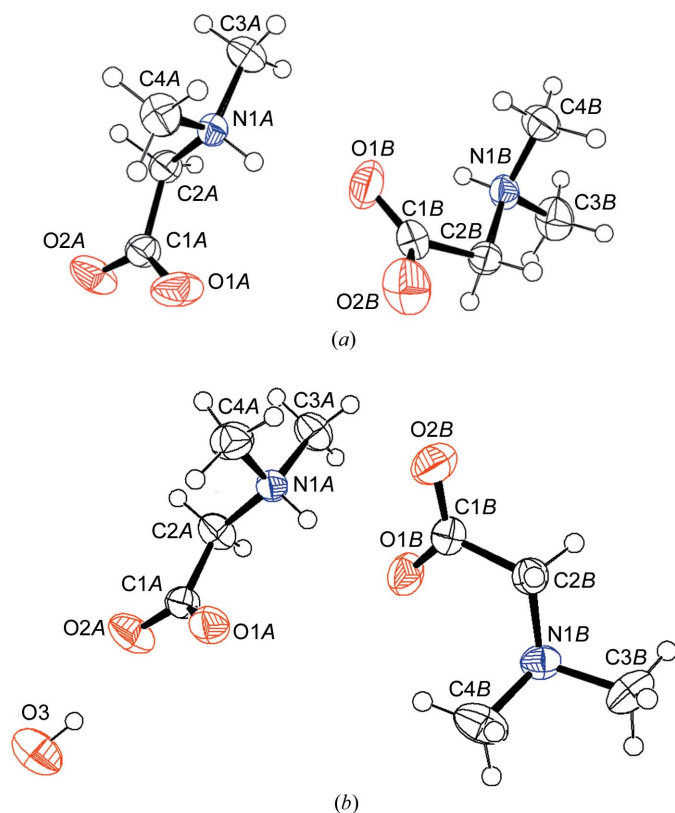
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Figure 1
The asymmetric units of (*a*) (I) and (*b*) (II), showing the atom-labelling schemes. Displacement ellipsoids are drawn at the 50% probability level.

acterized by the value of the N—C—C—O torsion angle, *i.e.* the main backbone of the molecule becomes more planar. Thus, the largest value of this torsion angle was observed for the three polymorphs of glycine: 25.03 (12)° in β-glycine (Drebushchak, Boldyreva & Shutova, 2002), 19.0 (1)° in α-glycine (Drebushchak, Boldyreva, Seretkin & Shutova, 2002) and 15.4 (1)° in γ-glycine (Kvick *et al.*, 1980), whereas its value in *N*-methylglycine is 5.6 (2)° (Trzebiatowska-Gusowska & Gagor, 2007) and just 0.0 (2)° in *N*,*N*,*N*-trimethylglycine (Viertorinne *et al.*, 1999). A nearly planar backbone conformation was found earlier in two salts of *N*,*N*-dimethylglycine where the molecule is in the cationic form, namely in *N*,*N*-dimethylglycine hydrochloride [2.2 (2)°; Santarsiero & Marsh, 1983] and *N*,*N*-dimethylglycinium trifluoroacetate [3.2 (3)°; Rodrigues *et al.*, 2001]. The molecular conformation in *N*,*N*-dimethylglycine hydrate, (II) [N—C—C—O torsion angles for the two different molecules in the asymmetric unit are 5.21 (17) and 8.71 (18)°], also follows the general trend. However, the molecular conformation of *N*,*N*-dimethylglycine in (I) does not conform to this correlation and is close to that found in the glycine polymorphs.

The difference in the conformations of the *N*,*N*-dimethylglycine zwitterions in (I) and (II) is mainly related to the different orientations of the carboxylate group, whereas the difference in the orientations of the methyl groups in the two structures is almost negligible (Table 1). Interestingly, in contrast with (I) and (II), the difference in the N—C—C—O

torsion angle in the crystal structures of *N*,*N*,*N*-trimethylglycine and its hydrate is not large [0.0 (2) and 2.3 (3)°, respectively; Mak, 1990].

Several conformations of an isolated neutral molecule of *N*,*N*-dimethylglycine (Headley & Starnes, 1996, 1998; Gómez-Zavaglia *et al.*, 2003; Lesarri *et al.*, 2005) and of its isolated zwitterion (Headley & Starnes, 1998) have been predicted by *ab initio* calculations. For a neutral molecule, the predicted average value of the N—C—C—O torsion angle in the lowest energy conformer is 20 (4)°. The existence of this conformer, together with two others (with a *syn* arrangement for N—C—C—O), has been proven experimentally by means of low-temperature matrix isolation FT–IR spectroscopy (Gómez-Zavaglia *et al.*, 2003) and combined laser ablation with molecular-beam Fourier-transform microwave spectroscopy (Lesarri *et al.*, 2005). For isolated zwitterions, the optimized molecular geometry for the two most stable conformers has a planar N—C—C—O torsion angle (Headley & Starnes, 1998). Interestingly, in (I), the average value of the N—C—C—O torsion angle in the *N*,*N*-dimethylglycine zwitterion is similar to that predicted for the ground state of a neutral molecule, whereas in (II) the zwitterions are planar and their geometry is in good agreement with theoretical calculations. Although the nonplanar conformation of a ground-state conformer of a neutral molecule should facilitate the formation of an intramolecular O—H···N hydrogen bond, in turn resulting in additional stabilization of the molecular conformation, this intramolecular hydrogen bond is not formed in the crystal structures of either (I) or (II), and was not observed in *ab initio* calculations for isolated zwitterions.

The orientation of the methyl groups in the crystal structures of (I) and (II) is quite similar (Table 1). It is interesting that such an orientation of the methyl groups is inherent in the most stable conformer of neutral *N*,*N*-dimethylglycine [CH₃—N—C—C torsion angles of about 152 and −82° (Headley & Starnes, 1996, 1998; Gómez-Zavaglia *et al.*, 2003)], in contrast with that predicted for *N*,*N*-dimethylglycine zwitterions [±63.4° for the lowest energy zwitterion and ±114.2° for the next highest (Headley & Starnes, 1998)].

Geometric data for the hydrogen bonds in the crystal structures of (I) and (II) are summarized in Tables 2 and 3, respectively. Since the amino group is doubly methylated and has only one H atom, there is only one N—H···O hydrogen bond per molecule in the asymmetric units of the two structures. Substitution of the H atoms of ammonia for alkyl groups is known to increase the basicity of amines. Hence, the hydrogen bonds formed in the crystal structure of *N*,*N*-dimethylglycine can be expected to be stronger than those in glycine. Indeed, even the hydrogen bonds in the most stable γ-polymorph of glycine (Drebushchak *et al.*, 2003), with N···O distances of 2.809 (2), 2.809 (2) and 2.987 (2) Å (Kvick *et al.*, 1980), are considerably longer than those in (I) and (II). The hydrogen bonds in *N*-methylglycine, with N···O distances of 2.7886 (16) and 2.7599 (17) Å (Trzebiatowska-Gusowska & Gagor, 2007), are also longer than those in (I) and (II). Not only are the N—H···O hydrogen bonds in (I) shorter than those in (II), but also the nature of these hydrogen bonds in
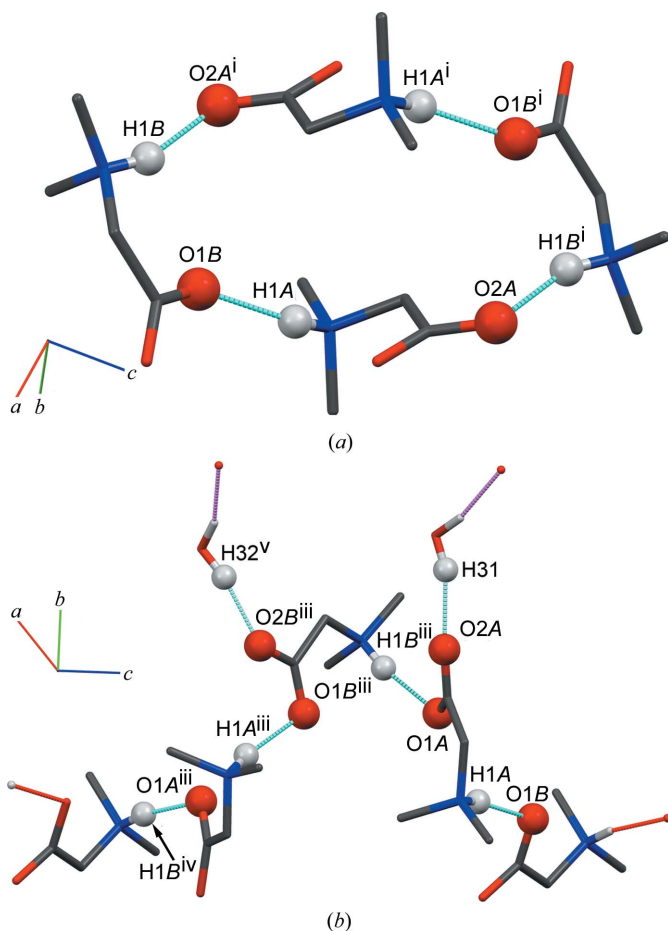
Figure 2
Fragments of the crystal structures of (I) and (II), representing the hydrogen-bonding and structural motifs. Hydrogen bonds are shown as dashed lines, and H atoms of the CH$_2$ and CH$_3$ groups have been omitted. (a) An $R_4^4(20)$ ring motif in (I) [symmetry code: (i) $-x, -y + 1, -z + 1$]. (b) A $C_2^2(10)$ infinite chain in (II), extending along the crystallographic c axis, and the O—H···O hydrogen bonds formed by water molecules, which function as bridges between these chains [symmetry codes: (iii) x, $-y + 1, z - \frac{1}{2}$; (iv) $x, y, z - 1$; (v) $x, -y + 2, z - \frac{1}{2}$].

the two structures is different. In (II), the N—H···O hydrogen bonds include atoms O1 as acceptors, while in (I) a zwitterion A forms a hydrogen bond with atom O1 (*cis* with respect to the N atom) of a zwitterion B, which is in turn bonded to atom O2 (*trans* to the N atom) of a zwitterion A (Fig. 2). A water molecule in (II) forms two O—H···O hydrogen bonds with two carboxylate groups of *N,N*-dimethylglycine, using atoms O2 as acceptors.

In the anhydrous compound, (I), four *N,N*-dimethylglycine zwitterions, two each of A and B, are linked to each other *via* N1A—H1A···O1B and N1B—H1B···O2$A(-x, -y + 1, -z + 1)$ hydrogen bonds to form a ring motif of $R_4^4(20)$ type (Etter *et al.*, 1990) centred at the centre of inversion $\bar{1}$. The formation of such insular motifs, which are not hydrogen bonded to each other, is very unusual for crystalline amino acids. In contrast, in the crystal structure of the hemihydrate, (II), the A and B zwitterions are linked to each other *via* N1A—H1A···O1B and N1B—H1B···O1$A(x, -y + 1, z + \frac{1}{2})$ hydrogen bonds into infinite $C_2^2(10)$ head-to-tail chains
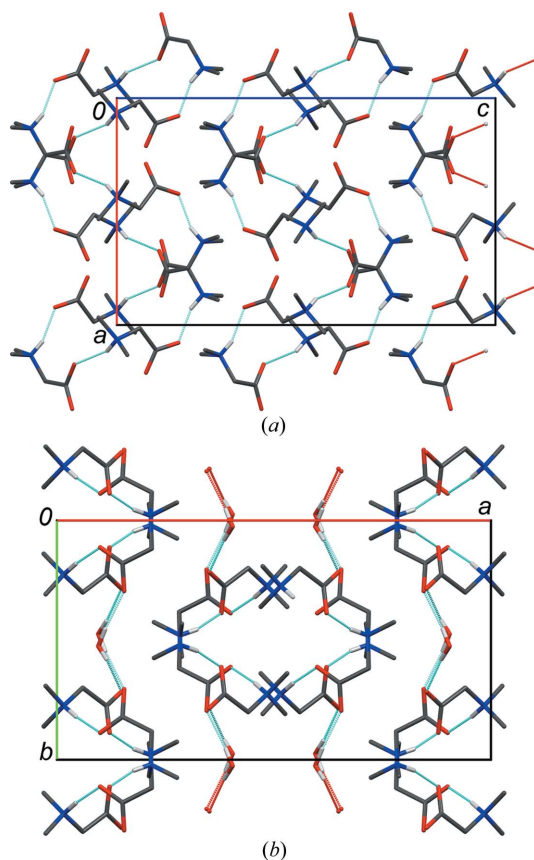
Figure 3
Fragments of the crystal structures of (a) (I) and (b) (II). Hydrogen bonds are shown as dashed lines, and non-hydrogen-bonded H atoms of the CH$_2$ and CH$_3$ groups have been omitted.

extending along the crystallographic c axis, whereas the water molecules outside the chains provide additional O—H···O hydrogen bonds to the carboxylate groups (Figs. 2 and 3). Such infinite head-to-tail chains [if the chain were formed by zwitterions of the same crystallographic symmetry, it would be termed a $C(5)$ chain] are common structure-forming motifs, not only in the glycine family but for all α-amino acids known at the present time (Suresh & Vijayan, 1983; Allen, 2002; Boldyreva, 2008; Görbitz, 2010). In this way, two-dimensional layers parallel to the crystallographic (100) plane are formed (Figs. 2 and 3). These layers are not linked to each other by hydrogen bonds, but are packed so that the distances between the layers are not the same. Therefore, two alternating domains are formed, *viz.* dense double layers, with an interlayer distance of 3.713 (2) Å, and loose layers, with a distance of 6.019 (2) Å.

In contrast, the crystal structure of (I) does not have such alternating domains, and the packing density is distributed homogeneously throughout the structure. Although the ring motifs in (I) are not linked to each other by hydrogen bonds, one can find 'pseudolayers' parallel to the (100) crystallographic plane and formed by assembling these motifs (Fig. 3). The layers in (II) and the pseudolayers in (I) are antiparallel to each other, and are stacked along the [100] crystallographic direction.

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Experimental

Prismatic crystals of (I) were obtained by sublimation. A purchased sample of N,N-dimethylglycine (Aldrich) was found to be a hydrate. It was ground and then placed in a vacuum chamber. The chamber was kept at 353 K and 1 mbar (1 bar = 100 000 Pa) for 27 h, after which colourless well-shaped crystals of (I) were found condensed on the cold part of the vacuum chamber. Prismatic colourless crystals of (II) were crystallized from solution in an ethanol–water mixture (1:1 v/v) by slow evaporation under ambient conditions. All mounted crystals were covered with a thin layer of CryoOil to protect them from the ambient atmosphere. Compound (I) is very sensitive to ambient humidity, and if the value exceeds 15% the crystals absorb water within a few minutes.

Compound (I)

Crystal data

$C_4H_9NO_2$
$M_r = 103.12$
Orthorhombic, $Pbca$
$a = 11.2228\ (3)$ Å
$b = 10.0097\ (3)$ Å
$c = 18.7285\ (4)$ Å

$V = 2103.90\ (10)$ Å^3
$Z = 16$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 295$ K
$0.4 \times 0.35 \times 0.2$ mm

Data collection

Oxford Gemini Ultra R diffractometer
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)
$T_{min} = 0.960$, $T_{max} = 0.980$

56342 measured reflections
2712 independent reflections
2475 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.115$
$S = 1.06$
2712 reflections

181 parameters
Only H-atom coordinates refined
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Compound (II)

Crystal data

$C_4H_9NO_2 \cdot 0.5H_2O$
$M_r = 112.13$
Monoclinic, $C2/c$
$a = 20.0403\ (8)$ Å
$b = 10.7329\ (4)$ Å
$c = 11.1120\ (5)$ Å
$\beta = 103.780\ (4)°$

$V = 2321.29\ (17)$ Å^3
$Z = 16$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 295$ K
$0.4 \times 0.25 \times 0.2$ mm

Data collection

Oxford Gemini Ultra R diffractometer
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)
$T_{min} = 0.959$, $T_{max} = 0.979$

23107 measured reflections
2891 independent reflections
2311 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.119$
$S = 1.03$
2891 reflections

196 parameters
Only H-atom coordinates refined
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Table 1
Selected torsion angles (°) in (I) and (II).

In producing this table, the coordinates of molecule B of (II) have been inverted, as allowed by the space-group symmetry, in order to facilitate the comparison of the torsion angles.

	(I)	(II)
C1A—C2A—N1A—C3A	168.16 (9)	167.30 (12)
C1B—C2B—N1B—C3B	−167.84 (10)	−167.47 (11)
C1A—C2A—N1A—C4A	−69.04 (11)	−68.95 (15)
C1B—C2B—N1B—C4B	68.80 (12)	69.06 (14)
N1A—C2A—C1A—O1A	−15.45 (15)	−8.71 (18)
N1B—C2B—C1B—O1B	22.50 (14)	5.21 (17)
N1A—C2A—C1A—O2A	165.70 (10)	171.84 (12)
N1B—C2B—C1B—O2B	−158.74 (10)	−175.43 (11)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1A—H1A⋯O1B	0.890 (14)	1.907 (14)	2.7334 (12)	153.7 (12)
N1B—H1B⋯O2A^i	0.876 (15)	1.828 (15)	2.6645 (12)	158.9 (13)

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

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1A—H1A⋯O1B	0.898 (15)	1.901 (15)	2.7614 (14)	160.0 (13)
N1A—H1A⋯O2B	0.898 (15)	2.651 (14)	3.1959 (14)	120.0 (11)
O3—H31⋯O2A	0.86 (2)	1.92 (2)	2.7538 (16)	162 (2)
N1B—H1B⋯O1A^i	0.902 (15)	1.874 (15)	2.7657 (14)	169.3 (13)
O3—H32⋯O2B^{ii}	0.88 (2)	1.86 (2)	2.7215 (17)	169 (2)

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

All H atoms were found in a difference Fourier map and their positions were refined freely, with $U_{iso}(H) = 1.5U_{eq}(C)$ for terminal methyl H atoms or $1.2U_{eq}(parent)$ otherwise.

For both compounds, data collection: CrysAlis CCD (Oxford Diffraction, 2008); cell refinement: CrysAlis CCD; data reduction: CrysAlis RED (Oxford Diffraction, 2008); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: Mercury (Macrae et al., 2008) and ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: publCIF (Westrip, 2010).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KU3073). Services for accessing these data are described at the back of the journal.

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supplementary materials

The effect of partial methylation of the glycine amino group on crystal structure in *N,N*-dimethylglycine and its hemihydrate

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(I) 2-(Dimethylamino)acetic acid

Crystal data

$C_4H_9NO_2$	$D_x = 1.302$ Mg m^{-3}
$M_r = 103.12$	Melting point: 451 K
Orthorhombic, *Pbca*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 19159 reflections
$a = 11.2228$ (3) Å	$\theta = 4.2–45.4°$
$b = 10.0097$ (3) Å	$\mu = 0.10$ mm^{-1}
$c = 18.7285$ (4) Å	$T = 295$ K
$V = 2103.90$ (10) Å^3	Prism, colourless
$Z = 16$	$0.4 \times 0.35 \times 0.2$ mm
$F(000) = 896$	

Data collection

Oxford Gemini Ultra R diffractometer	56342 measured reflections
Radiation source: fine-focus sealed tube	2712 independent reflections
Graphite monochromator	2475 reflections with $I > 2\sigma(I)$
Detector resolution: 10.3457 pixels mm^{-1}	$R_{int} = 0.031$
ω scans	$\theta_{max} = 28.7°$, $\theta_{min} = 4.2°$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)	$h = -15 \rightarrow 15$
	$k = -13 \rightarrow 13$
$T_{min} = 0.960$, $T_{max} = 0.980$	$l = -25 \rightarrow 25$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.041$	Secondary atom site location: difference Fourier map
$wR(F^2) = 0.115$	
$S = 1.06$	Hydrogen site location: difference Fourier map
2712 reflections	Only H-atom coordinates refined
181 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.3986P]$ where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	
0 constraints	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.28$ e Å^{-3}
	$\Delta\rho_{min} = -0.19$ e Å^{-3}

Special details

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}
N1A	0.06371 (7)	0.73440 (9)	0.50066 (4)	0.0316 (2)
H1A	0.1135 (12)	0.6847 (13)	0.4750 (7)	0.038*
N1B	0.13313 (8)	0.45069 (9)	0.27281 (4)	0.0335 (2)
H1B	0.0744 (13)	0.4604 (13)	0.3032 (7)	0.040*
O2A	0.07817 (8)	0.53742 (12)	0.66264 (5)	0.0585 (3)
O2B	0.36999 (9)	0.58032 (11)	0.38201 (6)	0.0597 (3)
C1B	0.26546 (10)	0.55743 (11)	0.36546 (5)	0.0360 (2)
C2B	0.24866 (9)	0.44448 (11)	0.31153 (5)	0.0343 (2)
H22B	0.2514 (12)	0.3612 (14)	0.3374 (8)	0.041*
H21B	0.3083 (14)	0.4467 (13)	0.2776 (7)	0.041*
O1B	0.17369 (10)	0.61252 (11)	0.38790 (5)	0.0603 (3)
C2A	0.01419 (9)	0.65464 (11)	0.56079 (5)	0.0339 (2)
H21A	−0.0328 (13)	0.7181 (14)	0.5914 (7)	0.041*
H22A	−0.0413 (13)	0.5899 (14)	0.5419 (7)	0.041*
O1A	0.21041 (9)	0.57217 (11)	0.57718 (6)	0.0626 (3)
C1A	0.11077 (9)	0.58250 (11)	0.60357 (6)	0.0363 (2)
C4A	0.13201 (12)	0.85164 (12)	0.52758 (7)	0.0434 (3)
H41A	0.0761 (16)	0.9082 (17)	0.5563 (9)	0.065*
H43A	0.1953 (16)	0.8174 (17)	0.5594 (9)	0.065*
H42A	0.1646 (16)	0.8963 (18)	0.4872 (10)	0.065*
C4B	0.12858 (12)	0.56642 (14)	0.22350 (7)	0.0472 (3)
H41B	0.1925 (18)	0.5572 (18)	0.1859 (10)	0.071*
H43B	0.0524 (19)	0.5645 (18)	0.1984 (10)	0.071*
H42B	0.1367 (16)	0.6474 (18)	0.2513 (10)	0.071*
C3B	0.11100 (13)	0.32456 (15)	0.23337 (7)	0.0519 (3)
H31B	0.1709 (19)	0.3162 (19)	0.1963 (10)	0.078*
H33B	0.1150 (18)	0.2498 (19)	0.2676 (10)	0.078*
H32B	0.0327 (19)	0.3294 (19)	0.2115 (10)	0.078*
C3A	−0.03296 (12)	0.77908 (14)	0.45193 (7)	0.0477 (3)
H31A	−0.0913 (18)	0.8256 (19)	0.4808 (10)	0.072*
H32A	−0.0656 (17)	0.7007 (19)	0.4288 (9)	0.072*
H33A	−0.0003 (17)	0.8388 (18)	0.4152 (10)	0.072*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.0311 (4)	0.0360 (4)	0.0277 (4)	0.0046 (3)	0.0010 (3)	−0.0021 (3)

N1B	0.0268 (4)	0.0472 (5)	0.0267 (4)	0.0002 (3)	0.0003 (3)	−0.0016 (3)
O2A	0.0337 (4)	0.1009 (8)	0.0410 (5)	0.0053 (4)	0.0030 (3)	0.0255 (5)
O2B	0.0469 (5)	0.0760 (7)	0.0563 (6)	−0.0187 (5)	−0.0095 (4)	−0.0127 (5)
C1B	0.0400 (5)	0.0412 (5)	0.0268 (4)	−0.0038 (4)	−0.0010 (4)	−0.0005 (4)
C2B	0.0284 (4)	0.0420 (5)	0.0326 (5)	0.0032 (4)	−0.0033 (4)	−0.0040 (4)
O1B	0.0580 (6)	0.0770 (7)	0.0460 (5)	0.0133 (5)	0.0023 (4)	−0.0253 (5)
C2A	0.0283 (4)	0.0396 (5)	0.0340 (5)	0.0023 (4)	0.0035 (4)	0.0010 (4)
O1A	0.0423 (5)	0.0827 (7)	0.0629 (6)	0.0283 (5)	0.0198 (4)	0.0279 (5)
C1A	0.0305 (5)	0.0433 (5)	0.0352 (5)	0.0033 (4)	0.0027 (4)	0.0032 (4)
C4A	0.0461 (6)	0.0407 (6)	0.0435 (6)	−0.0069 (5)	−0.0013 (5)	0.0002 (5)
C4B	0.0433 (6)	0.0605 (8)	0.0378 (6)	0.0093 (5)	−0.0004 (5)	0.0110 (5)
C3B	0.0491 (7)	0.0603 (8)	0.0463 (6)	−0.0105 (6)	−0.0086 (5)	−0.0127 (6)
C3A	0.0477 (6)	0.0547 (7)	0.0406 (6)	0.0071 (5)	−0.0127 (5)	0.0036 (5)

Geometric parameters (Å, º)

N1A—C3A	1.4866 (14)	C2A—H21A	1.005 (14)
N1A—C2A	1.4881 (13)	C2A—H22A	0.966 (15)
N1A—C4A	1.4897 (14)	O1A—C1A	1.2269 (14)
N1A—H1A	0.890 (14)	C4A—H41A	1.002 (18)
N1B—C4B	1.4823 (15)	C4A—H43A	0.989 (18)
N1B—C3B	1.4837 (16)	C4A—H42A	0.952 (19)
N1B—C2B	1.4868 (13)	C4B—H41B	1.01 (2)
N1B—H1B	0.876 (15)	C4B—H43B	0.98 (2)
O2A—C1A	1.2496 (13)	C4B—H42B	0.968 (18)
O2B—C1B	1.2349 (14)	C3B—H31B	0.97 (2)
C1B—O1B	1.2415 (15)	C3B—H33B	0.987 (19)
C1B—C2B	1.5278 (14)	C3B—H32B	0.97 (2)
C2B—H22B	0.965 (14)	C3A—H31A	0.97 (2)
C2B—H21B	0.923 (15)	C3A—H32A	0.97 (2)
C2A—C1A	1.5292 (14)	C3A—H33A	0.982 (19)
C3A—N1A—C2A	110.70 (9)	O1A—C1A—O2A	126.38 (11)
C3A—N1A—C4A	110.26 (9)	O1A—C1A—C2A	118.35 (10)
C2A—N1A—C4A	111.02 (8)	O2A—C1A—C2A	115.26 (9)
C3A—N1A—H1A	107.2 (8)	N1A—C4A—H41A	107.7 (10)
C2A—N1A—H1A	110.1 (8)	N1A—C4A—H43A	107.5 (10)
C4A—N1A—H1A	107.5 (8)	H41A—C4A—H43A	108.8 (14)
C4B—N1B—C3B	110.44 (10)	N1A—C4A—H42A	107.4 (11)
C4B—N1B—C2B	111.51 (9)	H41A—C4A—H42A	113.7 (14)
C3B—N1B—C2B	110.68 (9)	H43A—C4A—H42A	111.5 (15)
C4B—N1B—H1B	107.0 (9)	N1B—C4B—H41B	109.8 (10)
C3B—N1B—H1B	107.0 (9)	N1B—C4B—H43B	108.4 (11)
C2B—N1B—H1B	110.0 (9)	H41B—C4B—H43B	106.5 (14)
O2B—C1B—O1B	128.37 (11)	N1B—C4B—H42B	108.4 (11)
O2B—C1B—C2B	114.87 (10)	H41B—C4B—H42B	112.7 (15)
O1B—C1B—C2B	116.75 (10)	H43B—C4B—H42B	111.0 (15)
N1B—C2B—C1B	113.52 (9)	N1B—C3B—H31B	108.3 (12)
N1B—C2B—H22B	108.0 (8)	N1B—C3B—H33B	108.3 (11)
C1B—C2B—H22B	107.6 (8)	H31B—C3B—H33B	111.7 (16)

N1B—C2B—H21B	107.2 (9)	N1B—C3B—H32B	108.6 (12)
C1B—C2B—H21B	110.4 (9)	H31B—C3B—H32B	109.2 (15)
H22B—C2B—H21B	110.1 (12)	H33B—C3B—H32B	110.7 (17)
N1A—C2A—C1A	112.65 (8)	N1A—C3A—H31A	107.2 (11)
N1A—C2A—H21A	106.8 (8)	N1A—C3A—H32A	107.9 (11)
C1A—C2A—H21A	111.8 (8)	H31A—C3A—H32A	112.5 (16)
N1A—C2A—H22A	108.9 (8)	N1A—C3A—H33A	109.9 (11)
C1A—C2A—H22A	109.4 (8)	H31A—C3A—H33A	110.5 (15)
H21A—C2A—H22A	107.1 (12)	H32A—C3A—H33A	108.7 (14)
C4A—N1A—C2A—C1A	−69.04 (11)	C4B—N1B—C2B—C1B	68.80 (12)
C3A—N1A—C2A—C1A	168.16 (9)	C3B—N1B—C2B—C1B	−167.84 (10)
O2A—C1A—C2A—N1A	165.70 (10)	O2B—C1B—C2B—N1B	−158.74 (10)
O1A—C1A—C2A—N1A	−15.45 (15)	O1B—C1B—C2B—N1B	22.50 (14)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O1B	0.890 (14)	1.907 (14)	2.7334 (12)	153.7 (12)
N1B—H1B···O2A^i	0.876 (15)	1.828 (15)	2.6645 (12)	158.9 (13)

Symmetry code: (i) −x, −y+1, −z+1.

(II) 2-(Dimethylamino)acetic acid hemihydrate

Crystal data

$C_4H_9NO_2$·0.5H_2O	$F(000) = 976$
$M_r = 112.13$	$D_x = 1.283$ Mg m⁻³
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 5711 reflections
$a = 20.0403$ (8) Å	$\theta = 3.0$–37.5°
$b = 10.7329$ (4) Å	$\mu = 0.11$ mm⁻¹
$c = 11.1120$ (5) Å	$T = 295$ K
$\beta = 103.780$ (4)°	Prism, colourless
$V = 2321.29$ (17) Å³	$0.4 \times 0.25 \times 0.2$ mm
$Z = 16$	

Data collection

Oxford Gemini Ultra R diffractometer	23107 measured reflections
Radiation source: fine-focus sealed tube	2891 independent reflections
Graphite monochromator	2311 reflections with $I > 2\sigma(I)$
Detector resolution: 10.3457 pixels mm⁻¹	$R_{int} = 0.041$
ω scans	$\theta_{max} = 28.3°$, $\theta_{min} = 3.0°$
Absorption correction: multi-scan	$h = -26 \rightarrow 26$
(*CrysAlis RED*; Oxford Diffraction, 2008)	$k = -14 \rightarrow 14$
$T_{min} = 0.959$, $T_{max} = 0.979$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	2891 reflections
Least-squares matrix: full	196 parameters
$R[F^2 > 2\sigma(F^2)] = 0.042$	0 restraints
$wR(F^2) = 0.119$	0 constraints
$S = 1.03$	

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
Only H-atom coordinates refined

$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.6228P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Special details

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}
N1A	0.28381 (5)	0.47991 (9)	0.13230 (9)	0.0320 (2)
H1A	0.3219 (8)	0.4557 (13)	0.1879 (13)	0.038*
N1B	0.48450 (5)	0.27436 (10)	0.50065 (10)	0.0357 (2)
H1B	0.4565 (8)	0.3192 (13)	0.5367 (14)	0.043*
C2B	0.44060 (6)	0.19935 (12)	0.39996 (12)	0.0354 (3)
H21B	0.4695 (8)	0.1604 (14)	0.3550 (14)	0.043*
H22B	0.4162 (8)	0.1402 (14)	0.4387 (14)	0.043*
O1A	0.40011 (4)	0.61262 (9)	0.13228 (9)	0.0445 (3)
O1B	0.37882 (5)	0.38664 (9)	0.33421 (9)	0.0490 (3)
C1A	0.34878 (6)	0.67936 (12)	0.12587 (11)	0.0361 (3)
C1B	0.38577 (6)	0.27469 (12)	0.31117 (11)	0.0362 (3)
C2A	0.28173 (7)	0.61760 (12)	0.13521 (14)	0.0406 (3)
H21A	0.2449 (8)	0.6435 (15)	0.0688 (15)	0.049*
H22A	0.2705 (8)	0.6446 (14)	0.2125 (15)	0.049*
O2B	0.35118 (5)	0.21550 (10)	0.22104 (9)	0.0534 (3)
O2A	0.34510 (6)	0.79447 (9)	0.11332 (12)	0.0588 (3)
O3	0.40461 (7)	1.02552 (12)	0.11151 (13)	0.0681 (4)
H32	0.3860 (11)	1.079 (2)	0.154 (2)	0.082*
H31	0.3891 (11)	0.955 (2)	0.1293 (19)	0.082*
C3B	0.52585 (8)	0.19174 (18)	0.59824 (15)	0.0534 (4)
H32B	0.4946 (11)	0.1348 (19)	0.6293 (19)	0.080*
H33B	0.5517 (11)	0.2454 (19)	0.666 (2)	0.080*
H31B	0.5575 (11)	0.143 (2)	0.5602 (19)	0.080*
C4A	0.28773 (8)	0.43160 (16)	0.00943 (13)	0.0463 (3)
H43A	0.3233 (10)	0.4784 (18)	−0.0174 (16)	0.070*
H41A	0.2451 (11)	0.4461 (17)	−0.0407 (18)	0.070*
H42A	0.2958 (9)	0.3460 (19)	0.0182 (17)	0.070*
C3A	0.22366 (8)	0.42524 (16)	0.16962 (16)	0.0486 (4)
H32A	0.2220 (10)	0.4516 (17)	0.2448 (19)	0.073*
H33A	0.2291 (10)	0.338 (2)	0.1690 (18)	0.073*
H31A	0.1838 (11)	0.4486 (19)	0.1070 (18)	0.073*

C4B	0.53029 (9)	0.36074 (17)	0.45253 (19)	0.0576 (4)
H42B	0.5558 (11)	0.408 (2)	0.524 (2)	0.086*
H43B	0.5009 (11)	0.415 (2)	0.388 (2)	0.086*
H41B	0.5623 (11)	0.309 (2)	0.417 (2)	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.0285 (5)	0.0326 (5)	0.0341 (5)	−0.0011 (4)	0.0057 (4)	0.0034 (4)
N1B	0.0270 (5)	0.0394 (6)	0.0411 (6)	0.0015 (4)	0.0089 (4)	−0.0049 (4)
C2B	0.0364 (6)	0.0326 (6)	0.0362 (6)	0.0017 (5)	0.0064 (5)	−0.0003 (5)
O1A	0.0311 (4)	0.0484 (6)	0.0543 (6)	−0.0025 (4)	0.0104 (4)	0.0064 (4)
O1B	0.0583 (6)	0.0371 (5)	0.0457 (5)	0.0114 (4)	0.0003 (5)	0.0018 (4)
C1A	0.0361 (6)	0.0363 (6)	0.0354 (6)	−0.0061 (5)	0.0078 (5)	−0.0017 (5)
C1B	0.0374 (6)	0.0377 (6)	0.0339 (6)	0.0026 (5)	0.0090 (5)	0.0028 (5)
C2A	0.0346 (6)	0.0335 (6)	0.0571 (8)	0.0014 (5)	0.0176 (6)	0.0016 (6)
O2B	0.0541 (6)	0.0501 (6)	0.0473 (6)	0.0074 (5)	−0.0051 (5)	−0.0086 (4)
O2A	0.0588 (7)	0.0335 (5)	0.0858 (8)	−0.0088 (5)	0.0208 (6)	−0.0021 (5)
O3	0.0762 (9)	0.0491 (7)	0.0910 (9)	−0.0116 (6)	0.0437 (7)	−0.0114 (6)
C3B	0.0377 (7)	0.0710 (11)	0.0458 (8)	0.0114 (7)	−0.0015 (6)	−0.0007 (7)
C4A	0.0457 (8)	0.0538 (8)	0.0400 (7)	−0.0072 (7)	0.0110 (6)	−0.0069 (6)
C3A	0.0444 (8)	0.0519 (8)	0.0531 (9)	−0.0122 (7)	0.0188 (7)	0.0054 (7)
C4B	0.0455 (8)	0.0551 (9)	0.0784 (12)	−0.0147 (7)	0.0270 (8)	−0.0091 (8)

Geometric parameters (Å, º)

N1A—C2A	1.4790 (16)	C2A—H21A	0.952 (17)
N1A—C4A	1.4799 (17)	C2A—H22A	0.982 (16)
N1A—C3A	1.4857 (16)	O3—H32	0.88 (2)
N1A—H1A	0.898 (15)	O3—H31	0.86 (2)
N1B—C2B	1.4849 (16)	C3B—H32B	0.99 (2)
N1B—C3B	1.4902 (19)	C3B—H33B	0.99 (2)
N1B—C4B	1.4905 (18)	C3B—H31B	0.99 (2)
N1B—H1B	0.902 (15)	C4A—H43A	0.98 (2)
C2B—C1B	1.5226 (17)	C4A—H41A	0.92 (2)
C2B—H21B	0.947 (15)	C4A—H42A	0.93 (2)
C2B—H22B	0.963 (15)	C3A—H32A	0.89 (2)
O1A—C1A	1.2416 (16)	C3A—H33A	0.94 (2)
O1B—C1B	1.2432 (15)	C3A—H31A	0.96 (2)
C1A—O2A	1.2436 (16)	C4B—H42B	0.98 (2)
C1A—C2A	1.5238 (17)	C4B—H43B	1.00 (2)
C1B—O2B	1.2476 (16)	C4B—H41B	1.00 (2)
C2A—N1A—C4A	112.22 (11)	C1A—C2A—H22A	108.7 (9)
C2A—N1A—C3A	111.07 (11)	H21A—C2A—H22A	107.2 (13)
C4A—N1A—C3A	110.12 (11)	H32—O3—H31	103.4 (19)
C2A—N1A—H1A	107.3 (9)	N1B—C3B—H32B	109.3 (12)
C4A—N1A—H1A	108.1 (9)	N1B—C3B—H33B	107.9 (12)
C3A—N1A—H1A	107.9 (9)	H32B—C3B—H33B	110.6 (17)
C2B—N1B—C3B	110.65 (11)	N1B—C3B—H31B	107.9 (12)

C2B—N1B—C4B	111.83 (11)	H32B—C3B—H31B	109.9 (17)
C3B—N1B—C4B	110.36 (12)	H33B—C3B—H31B	111.2 (16)
C2B—N1B—H1B	107.8 (10)	N1A—C4A—H43A	107.8 (11)
C3B—N1B—H1B	106.9 (9)	N1A—C4A—H41A	104.7 (12)
C4B—N1B—H1B	109.2 (9)	H43A—C4A—H41A	111.8 (16)
N1B—C2B—C1B	114.01 (10)	N1A—C4A—H42A	107.1 (12)
N1B—C2B—H21B	108.1 (9)	H43A—C4A—H42A	114.7 (16)
C1B—C2B—H21B	109.2 (9)	H41A—C4A—H42A	110.1 (16)
N1B—C2B—H22B	107.0 (9)	N1A—C3A—H32A	110.6 (13)
C1B—C2B—H22B	106.0 (9)	N1A—C3A—H33A	106.6 (12)
H21B—C2B—H22B	112.5 (13)	H32A—C3A—H33A	110.6 (17)
O1A—C1A—O2A	127.29 (12)	N1A—C3A—H31A	106.6 (11)
O1A—C1A—C2A	118.50 (11)	H32A—C3A—H31A	112.7 (17)
O2A—C1A—C2A	114.21 (12)	H33A—C3A—H31A	109.4 (17)
O1B—C1B—O2B	126.11 (12)	N1B—C4B—H42B	106.1 (12)
O1B—C1B—C2B	118.61 (11)	N1B—C4B—H43B	108.0 (12)
O2B—C1B—C2B	115.27 (11)	H42B—C4B—H43B	112.8 (18)
N1A—C2A—C1A	113.78 (10)	N1B—C4B—H41B	107.6 (12)
N1A—C2A—H21A	107.2 (10)	H42B—C4B—H41B	110.6 (17)
C1A—C2A—H21A	110.6 (10)	H43B—C4B—H41B	111.5 (17)
N1A—C2A—H22A	109.1 (9)		
C4A—N1A—C2A—C1A	−68.95 (15)	C4B—N1B—C2B—C1B	−69.06 (14)
C3A—N1A—C2A—C1A	167.30 (12)	C3B—N1B—C2B—C1B	167.47 (11)
O2A—C1A—C2A—N1A	171.84 (12)	O2B—C1B—C2B—N1B	175.43 (11)
O1A—C1A—C2A—N1A	−8.71 (18)	O1B—C1B—C2B—N1B	−5.21 (17)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A···O1B	0.898 (15)	1.901 (15)	2.7614 (14)	160.0 (13)
N1A—H1A···O2B	0.898 (15)	2.651 (14)	3.1959 (14)	120.0 (11)
O3—H31···O2A	0.86 (2)	1.92 (2)	2.7538 (16)	162 (2)
N1B—H1B···O1A[i]	0.902 (15)	1.874 (15)	2.7657 (14)	169.3 (13)
O3—H32···O2B[ii]	0.88 (2)	1.86 (2)	2.7215 (17)	169 (2)

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

Selected torsion angles (°) in (I) and (II)

Torsion angle	(I)	(II)
C1A—C2A—N1A—C3A	168.16 (9)	167.30 (12)
C1B—C2B—N1B—C3B	-167.84 (10)	-167.47 (11)
C1A—C2A—N1A—C4A	-69.04 (11)	-68.95 (15)
C1B—C2B—N1B—C4B	68.80 (12)	69.06 (14)
N1A—C2A—C1A—O1A	-15.45 (15)	-8.71 (18)
N1B—C2B—C1B—O1B	22.50 (14)	5.21 (17)
N1A—C2A—C1A—O2A	165.70 (10)	171.84 (12)
N1B—C2B—C1B—O2B	-158.74 (10)	-175.43 (11)