research communications

Tetra-*n*-butylammonium orotate monohydrate: knowledge-based comparison of the results of accurate and lower-resolution analyses and a non-routine disorder refinement

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The title hydrated molecular salt (systematic name: tetra-*n*-butylammonium 2,6-dioxo-1,2,3,6-tetrahydropyrimidine-4-carboxylate monohydrate), $C_{16}H_{36}N^+ \cdot C_5H_3N_2O_4^- \cdot H_2O$, crystallizes with N—H⋯O and O—H⋯O hydrogen-bonded double-stranded antiparallel ribbons consisting of the hydrophilic orotate monoanions and water molecules, separated by the bulky hydrophobic cations. The hydrophobic and hydrophilic regions of the structure are joined by weaker non-classical C—H⋯O hydrogen bonds. An accurate structure analysis conducted at T = 100 K is compared to a lower-resolution less accurate determination using data measured at T = 295 K. The results of both analyses are evaluated using a knowledge-based approach, and it is found that the less accurate room-temperature structure analysis provides geometric data that are similar to those derived from the accurate low-temperature analysis, with both sets of results consistent with previously analyzed structures. A minor disorder of one methyl group in the cation at low temperature was found to be slightly more complex at room temperature; while still involving a minor fraction of the structure, the disorder at room temperature was found to require a non-routine treatment, which is described in detail.

1. Chemical context

We report here the structure analysis at two temperatures (**1** at 100 K and **2** at 295 K) of an organic salt formed by a bulky, hydrophobic cation, $^nBu_4N^+$, and the compact, hydrophilic anion $C_5H_3N_2O_4^-$, formed by single deprotonation of orotic acid. Crystals of this material are monohydrated, and the water molecule plays an integral role in the structure.
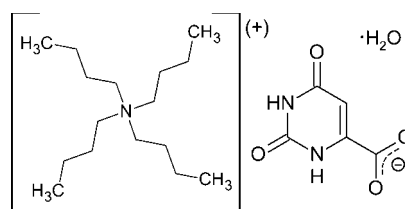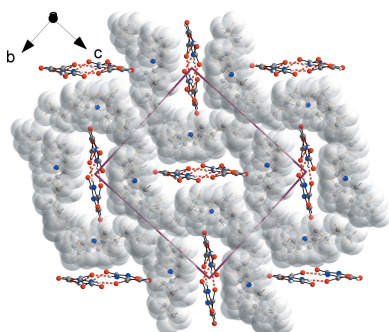
Orotic acid, 2,4-dioxo-1*H*-pyrimidine-6-carboxylic acid, $C_5H_4N_2O_4$, is important in a multitude of roles in biochemistry, among them as a precursor in the synthesis of uridine monophosphate (UMP) and thus of the pyrimidine nucleotides (Löffler *et al.*, 2015, 2016, 2018).

Our own interest in orotic acid, and in the conjugate bases formed by single and double deprotonation of orotic acid, arises from the functional groups that they present to their surroundings, which endow them with the ability to bind to a transition metal while at the same time forming energetically significant, directed and possibly structure-directing, interactions with their environment in a crystal. We have encountered, for example, a system in which stereoisomer selection for a six-coordinate Ni^{II} complex is achieved by enabling or vitiating hydrogen-bond formation in crystals of the product (Falvello *et al.*, 2007). In another study (Castro *et al.*, 2017), it was found that the $^nBu_4N^+$ salt of a Co^{III} orotate complex, namely $(^nBu_4N)[Co(orot)_2(bipy)]\cdot3H_2O$, undergoes an order–disorder phase transition, which upon recycling and repeating suffers arrest, which leaves the sample in a two-domain, two-structure form (monoclinic/triclinic).

It is in the context of phase transitions that we find the simple cation tetra-*n*-butylammonium, $^nBu_4N^+$ or $C_{16}H_{36}N^+$, to be of interest. It is known that the presence of even a single *n*-butyl group can be sufficient to incite an order–disorder transition when, for example, the temperature is varied (Willett *et al.*, 2005).

While our interest in orotic acid and orotates stems from their utility in coordination chemistry, it is also pertinent to explore molecular solids in which these fragments are present without metals. To date, six unique crystal structures have been analyzed of solids containing orotic acid in the absence of coordination compounds (ten analyses, including duplicates); and three analyses have been reported with orotic acid
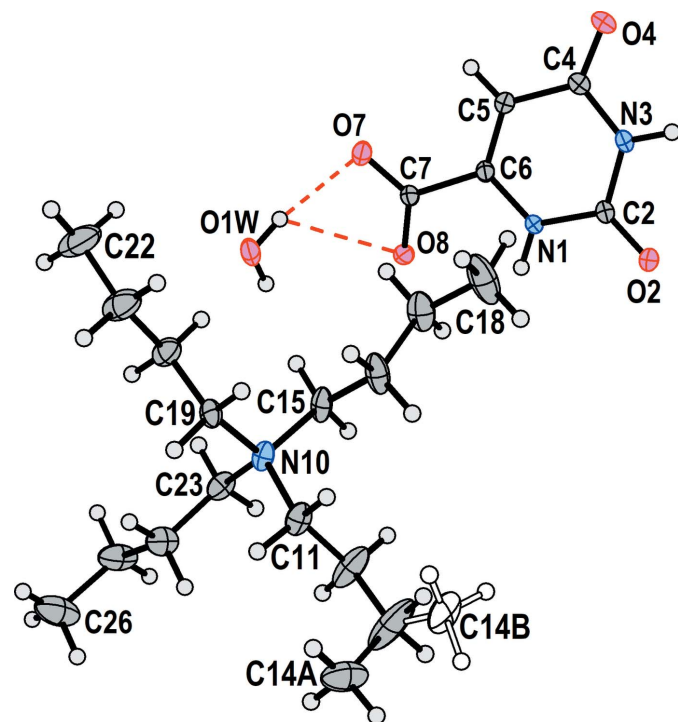
Figure 1
The asymmetric unit of **1** at 100 K. Non-hydrogen atoms are represented by their 50% probability displacement ellipsoids. Dashed red lines represent hydrogen bonds. C14*A* and C14*B* are the major and minor components of the disordered methyl group.
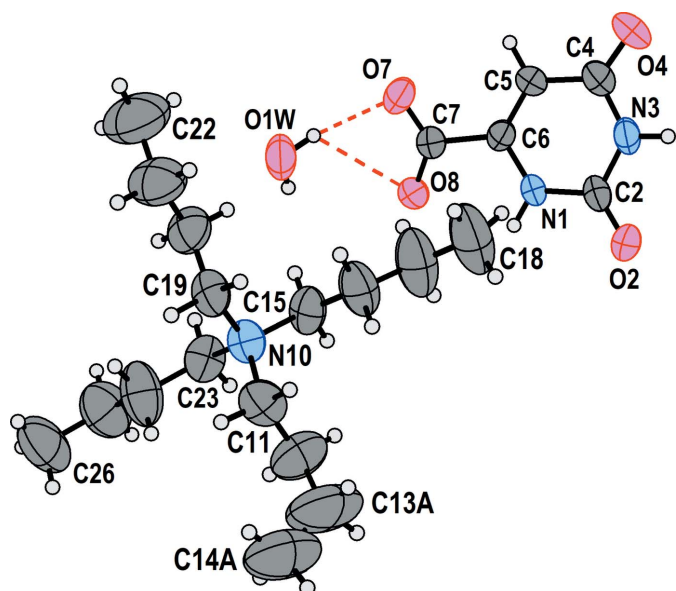
Figure 2
The asymmetric unit of **2** at 295 K. Non-hydrogen atoms are represented by their 50% probability displacement ellipsoids. Dashed red lines represent hydrogen bonds. C13*A* and C14*A* represent one component of a disordered ethyl fragment, whose other components are not shown.

co-crystallized with orotate complexes of Co, Pr and Nd. Singly deprotonated orotate – Horot⁻, deprotonated at the carboxylate function – figures in some 46 previously reported structure analyses, 16 of which also have *d*-block elements and six of which are lanthanoid compounds. There is also one structure of a uranium complex of Horot⁻. Some 15 Horot⁻-containing structures have no metal atom present.

With this as background, we undertook the structure analysis of the monohydrate of tetra-*n*-butylammonium 2,4-dioxo-1*H*-pyrimidine-6-carboxylate, $(^nBu_4N)(C_5H_3N_2O_4)$, at room temperature and at 100 K, to establish the structural organization adopted by this hydrophobic–hydrophilic ion pair and to explore the possibility of an order–disorder phase transition, as is seen with some regularity in *n*-butyl-containing molecular crystals.

2. Structural commentary

One of the motivations for this study was to observe the packing pattern adopted by a bulky hydrophobic cation and a compact hydrophilic anion when crystallized together. In the event, there are no solvent-accessible voids, as calculated by *PLATON* (Spek, 2009); however, this full packing arrangement is achieved with the incorporation of one water molecule per formula unit. Packing and scattering are more efficient at low temperature; we will discuss the structure first with reference to the analysis at $T = 100$ K; some comparisons between the two analyses will be presented at the end.

Displacement ellipsoid plots of the two structures are shown in Fig. 1 (100 K) and Fig. 2 (295 K). The two drawings have the same scale, and it is clear that, as expected, the lower-

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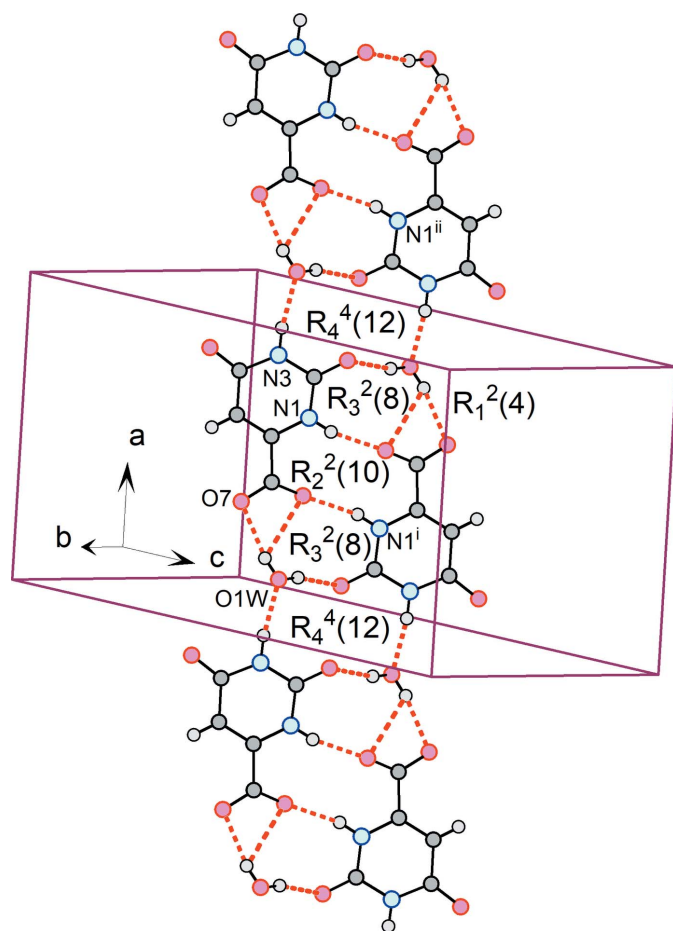Hydrogen-bond geometry (Å, °) for **1**.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1⋯O8^{i}	0.860 (16)	1.924 (16)	2.7668 (12)	166.4 (14)
N3—H3⋯O1W^{ii}	0.901 (17)	1.913 (17)	2.8081 (12)	171.8 (15)
C11—H11B⋯O7^{iii}	0.99	2.25	3.1462 (15)	151
C19—H19A⋯O4^{iv}	0.99	2.28	3.1878 (14)	151
C23—H23A⋯O2^{v}	0.99	2.37	3.3305 (14)	164
C24—H24A⋯O4^{iv}	0.99	2.34	3.3197 (19)	171
O1W—H1WA⋯O7	0.85 (2)	2.00 (2)	2.8396 (12)	169.2 (18)
O1W—H1WA⋯O8	0.85 (2)	2.64 (2)	3.1155 (12)	117.3 (15)
O1W—H1WB⋯O2^{i}	0.87 (2)	2.01 (2)	2.8618 (13)	168.3 (19)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1⋯O8^{i}	0.866 (19)	1.96 (2)	2.800 (3)	162.1 (18)
N3—H3⋯O1W^{ii}	0.86 (2)	1.96 (2)	2.807 (2)	170 (2)
C11—H11A⋯O7^{iii}	0.97	2.26	3.168 (3)	155
C19—H19A⋯O4^{iv}	0.97	2.28	3.226 (3)	164
C23—H23B⋯O2^{v}	0.97	2.44	3.386 (3)	166
C24—H24B⋯O4^{iv}	0.97	2.47	3.346 (4)	151
O1W—H1WA⋯O7	0.85 (2)	2.03 (2)	2.874 (2)	172 (2)
O1W—H1WA⋯O8	0.85 (2)	2.59 (3)	3.074 (2)	118 (2)
O1W—H1WB⋯O2^{i}	0.76 (3)	2.15 (3)	2.895 (2)	164 (3)

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

temperature structure has notably reduced displacement as compared to the structure at room temperature.

2.1. Supramolecular features

The structure is segregated into hydrophilic and hydrophobic zones. Firstly, a network of N—H⋯O and O—H⋯O hydrogen bonds link the Horot$^-$ anions and water molecules into a ladder-like chain propagating in the a-axis direction and lying in the (011) plane (Table 1 for $T = 100$ and Table 2 for $T = 295$ K; Fig. 3). Four different types of hydrogen-bonded rings form an uninterrupted fused-ring system along the length of this chain. Symmetry relatives of the $R_2^2(10)$ ring at the center of the segment shown in Fig. 3 occupy inversion centers at $(1/2 + n, 1/2, 1/2)$, where n is an integer. The chain is further propagated through an $R_4^4(12)$ ring whose congeners are on inversion centers at $(n, 1/2, 1/2)$, with n an integer. The components of this chain are related by the 2_1 screw axis and the c-glide to the constituent fragments of chains – also parallel to the a axis of the cell but lying in (01$\bar{1}$) planes – passing through centers of inversion at $(0, 0, 0)$, $(1/2, 0, 0)$ and lattice-related positions.

The hydrophobic cations surround the orotate–water chains, filling in the remaining space in the structure (Fig. 4). That the cell is efficiently filled can be seen in the Kitaigorodsky packing indices (KPI: percent filled space; Kitaigorodsky, 1973) of 66.8 for **1** and 63.2 for **2**. [In order to perform the calculation of the KPI using *PLATON* (Spek, 2009) it was necessary to create a structure model with only the principal

Figure 3
The ladder-like chain formed by the hydrophilic fragments, from the structure at $T = 100$ K. [Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + 2, -y + 1, -z + 1$.]
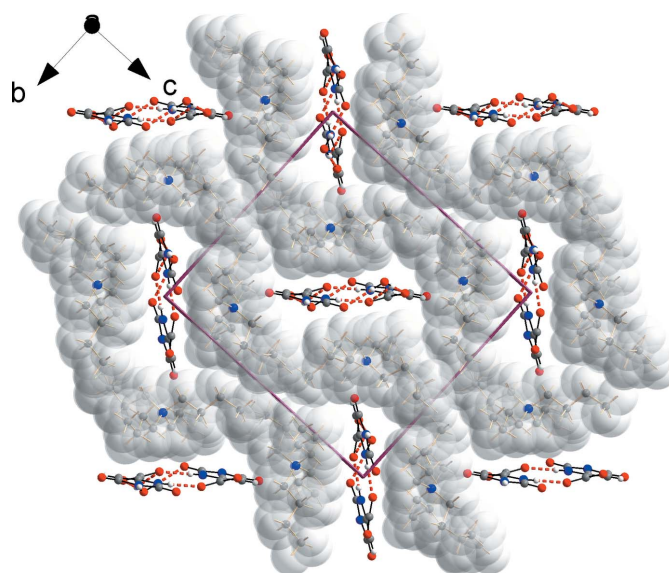

Figure 4
Packing of the ^{n}Bu$_4$N$^+$ cations and Horot$^{(-)}$–H$_2$O chains in **1**. Dashed red lines represent hydrogen bonds within the hydrophilic zones. The H atoms of the cations are represented by spheres with the van der Waals radius of 1.2 Å.

components of disorder present.] For comparison purposes, we note that a structure consisting of close-packed spheres fills 74.0% of its FCC unit cell.

The hydrophobic and hydrophilic sectors of the structure are not strictly separated, as there are favorable interactions between them (Table 1, Fig. 5). Each orotate anion accepts a total of four non-classical hydrogen bonds from the methylene groups of three surrounding $^nBu_4N^{(+)}$ cations. Fig. 5 shows a segment of the hydrophilic chain, with its hydrogen bonds in red, and three neighboring cations with the C—H···O interactions in blue. The flexibility of the butyl groups along with their capacity for forming directed interactions with the anions and dispersion-based interactions among themselves, is key to the ability of this material to form well-packed crystals.

The overall layout of the structure and the interactions that consolidate it are summarized graphically in the Hirshfeld surfaces (Spackman & Jayatilaka, 2009), which are presented here only for the low-temperature determination (Fig. 6). Fig. 6a shows the Hirshfeld surface for the anion from two viewpoints, within its crystalline surroundings. It is clear that

Figure 6
Hirshfeld surfaces based on d_{norm} for (a) the orotate anion, seen from two viewpoints in the antiparallel chains; (b) the water molecule, seen from two angles; (c) and (d) the cation, viewed from two opposite sides.

its principal interactions lie within the hydrophilic zone. Fig. 6b rounds out the picture, showing through the Hirshfeld surfaces that the major interactions of the water molecules also lie within the hydrophilic sector of the structure. Fig. 6c and 6d show the Hirshfeld surface of the cation from two opposite view directions. The scarce interactions consist of two non-classical hydrogen bonds on each side. Fingerprint calculations reveal that the close H(internal)···O(external) interactions account for only 14.1% of the points on the surface. These can be compared to H(i)···O(e) and O(i)···H(e) values of 27.5% and 32.3%, respectively, for the water molecule and 6.2% and 49.2% for the orotate anion.

3. Database survey: knowledge-based comparison of the analyses at two temperatures

The presence of bulky aliphatic groups clearly influences the diffraction from these crystals. The structure at room temperature suffers not only from a more complex disorder of one terminal ethyl group, but also produces weak diffraction, to the extent that from intensity statistics we estimate the effective resolution of the data to be about 1.0 Å. The data at $T = 100$ K are much stronger and give what in present times is regarded as an accurate result, which includes the observation of positive difference density at the centers of most of the bonds not involving H atoms.

It is thus instructive to compare the geometric parameters derived from the two analyses.

An overlay of all corresponding non-H atoms in the two structures, excluding the disordered Et fragment at C13 and C14, gives an r.m.s. deviation of 0.144 Å. As can be seen in Fig. 7, most of the deviation resides in the slightly different conformations of two of the terminal Et groups of the cation,

Figure 5
Partial view of the packing in **1**, showing hydrogen bonds within the hydrophilic chain (red dashed lines) and non-classical C—H···O hydrogen bonds (blue dashed lines) between methylene H atoms of the cations and oxygen atoms of the orotate anions. [Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (iii) $x + 1, y, z$; (iv): $x + 1, -y + \frac{3}{2}, z - \frac{1}{2}$; (v) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.] Butyl-group H atoms not involved in hydrogen bonds have been omitted. The minor congener of the terminal methyl group has been omitted.

Figure 7
Overlay of the asymmetric units for the analyses at T = 100 K (blue) and T = 295 K (red).

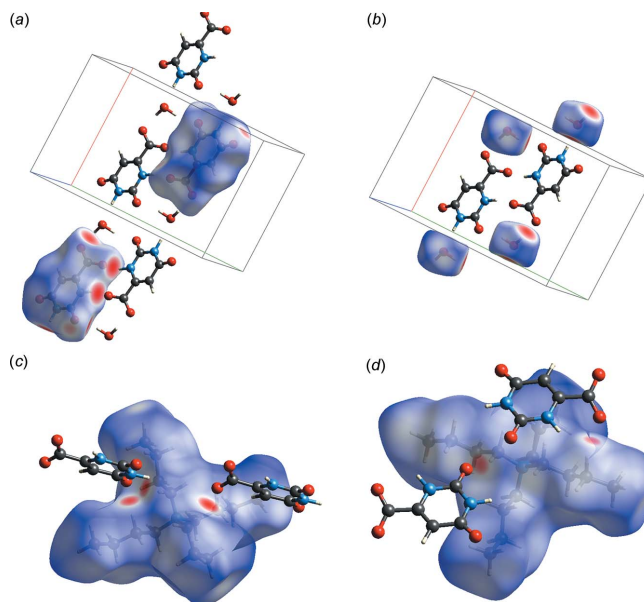namely C17/C18 (0.35, 0.21 Å deviation for C17 and C18, respectively) and C25/C26 (0.17, 0.18 Å for C25, C26, respectively).

3.1. *Mogul* geometry check

Extending the geometric comparisons to the possible differences between these two determinations, on one hand, and prior analyses involving similar chemical fragments, on the other, we performed a *Mogul* geometry check, in which bond lengths and bond angles found in these structures are compared to those of fragments of the same chemical nature found in the CSD (Cambridge Structural Database; Groom *et al.*, 2016). All results are compiled in the supporting information. Briefly, there are no gross outliers in these two analyses; however, the area of the carboxylate group and its linkage to the ring of the anion shows an interesting trend in both analyses. A relevant fact in this regard is that the dihedral angle between the orotate ring and the pendant carboxylate group is 23.14 (8)° at T = 100 K and 20.4 (2)° at room temperature. While the *Mogul* geometry check does not encounter any important outliers for either analysis, it does signal some slightly larger deviations from previous results, in the conjugated region where the ring and carboxylate group are joined, in keeping with the torsion angle that reduces π–π overlap between C6 and C7. Thus, considering the mean and standard deviation σ of the bond distances found in previous structure analyses with chemically similar groups, at low temperature the two nominally delocalized C$\cdots$O bonds C7—O7 and C7—O8 are 0.734 σ and 1.620 σ shorter than the mean; C5—C6 is 1.411 σ shorter; and C6—C7 is 1.985 σ longer. For T = 295 K the analogous deviations are 3.047 and 4.820 σ for C7—O7 and C7—O8, 3.065 σ for C5—C6 and 1.665 σ for C6—C7 – all in the expected direction from the mean. These variations are not extreme and might be taken as barely significant statistically. However, we consider it noteworthy that they stand out in comparison with the analogous values for the rest of the structure, and that similar results are

obtained at both temperatures (Tables S1 and S2 in the supporting information).

If we consider the reported structures of Horot$^-$ with alkali counter-ions, the Horot fragments in the anhydrous K$^+$ and Rb$^+$ salts (both: Bekiroglu & Kristiansson, 2002; K$^+$: Clegg & Nichol, 2018a; Rb$^+$: Martínez *et al.*, 2008) are co-planar. However, for the hydrated compounds they are not coplanar, although the angles are smaller than in the NBu$_4$$^+$ compound. For K(Horot)·H$_2$O the analogue of the O—C—C6—N1 torsion angle is −9.59° (CSD refcode MIJLUN, Yeşilel *et al.*, 2007); and for three analyses of Li(Horot)·H$_2$O, smaller values were found for the analogous torsion angle: SIMZOD 3.07° (Bach *et al.*, 1990); SIMZOD01 3.83°, (Lutz, 2001); SIMZOD02 3.82° (Clegg & Nichol, 2018b).

4. Synthesis and crystallization

2 ml of a 1.5 M aqueous solution of NBu$_4$OH (3 mmol) was added to a suspension of 0.7 g (4 mmol) of orotic acid monohydrate, H$_2$Orot·H$_2$O, in 2 ml of water. The suspension was stirred for 3 h at room temperature and then filtered through paper in order to remove the excess of H$_2$Orot·H$_2$O. Partial evaporation of the solution at 303 K produced colorless crystals of [NBu$_4$][HOrot]·H$_2$O, which were removed from the solution and dried with paper (0.643 g, 49.5% yield).

5. Refinement

Crystal data, data collection parameters and structure refinement residuals are given in Table 3. Single-crystal diffraction data were gathered from two crystals, one at T = 100 K, **1**, and the other at room temperature, **2**. The structure was solved *ab initio* from each of the two data sets using iterative methods (*SHELXT* 2014/5; Sheldrick, 2015a) and refined using full-matrix least-squares analysis (*SHELXL2018/1*; Sheldrick, 2015b). For **1**, one of the *n*-Bu groups of the cation, namely C11–C14, had its terminal CH$_3$ group disordered over two sets of sites, whose occupancy ratio was refined to 0.698 (4)/ 0.302 (4). For **2**, measured at room temperature, the same *n*-Bu group suffered a more complex disorder, with the γ-C atom, C13, disordered over two positions and the δ-C atom, C14, disordered over three positions. This disorder assembly was interpreted as being composed of four disorder groups; the structure model was composed and refined so as to produce chemically sound stoichiometry for the individual disorder groups and for the assembly as a whole. The interested reader is referred to the supporting information and the embedded, commented instruction file in the CIF for full details. The H atoms of methylene groups in both structures were placed at idealized positions and refined as riding atoms. The H atoms of all methyl groups in **1** and of the ordered methyl groups in **2** were placed at positions derived from local Fourier calculations and permitted to rotate but not tilt in the refinement. The H atoms of disordered CH$_3$ groups in **2** were placed at positions calculated to give staggered conformations about the local C—C bond and refined as riding atoms. For CH$_2$ groups, U_{iso}(H) were set to 1.2U_{eq} of their respective bonding partners. For CH$_3$, U_{iso}(H) were set to 1.5U_{eq}(C). The

Table 3
Experimental details.

	1	**2**
Crystal data		
Chemical formula	$C_{16}H_{36}N^+\cdot C_5H_3N_2O_4{}^-\cdot H_2O$	$C_{16}H_{36}N^+\cdot C_5H_3N_2O_4{}^-\cdot H_2O$
M_r	415.57	415.57
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/c$
Temperature (K)	100	295
a, b, c (Å)	10.0905 (5), 14.8664 (8), 16.1261 (9)	10.1335 (5), 14.6690 (7), 16.9205 (8)
β (°)	97.347 (5)	96.630 (4)
V (Å^3)	2399.2 (2)	2498.4 (2)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.08	0.08
Crystal size (mm)	$0.31 \times 0.18 \times 0.16$	$0.55 \times 0.23 \times 0.09$
Data collection		
Diffractometer	Bruker APEXII CCD	Rigaku Oxford Diffraction Xcalibur, Sapphire3
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.632, 1.000	0.980, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26211, 6560, 5274	27640, 4273, 1621
R_{int}	0.025	0.070
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.707	0.595
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.118, 1.03	0.047, 0.095, 1.04
No. of reflections	6560	4273
No. of parameters	297	307
No. of restraints	1	56
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, -0.36	0.26, -0.19

Computer programs: *APEX2* (Bruker, 2005), *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL2018/1* (Sheldrick, 2015b), *SIR92* (Altomare *et al.*, 1994), *DIAMOND* (Brandenburg, 2007), *Mercury* (Macrae *et al.*, 2006), *WinGX* (Farrugia, 2012) and *CrystalExplorer* (Spackman & Jayatilaka, 2009).

H atoms of the orotate anion and the water molecule were located in difference Fourier maps for both analyses; their positions were refined freely and their U_{iso} were refined freely for **1** and set to $1.2U_{eq}$ of their respective bonding partners for **2**.

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supporting information

Tetra-*n*-butylammonium orotate monohydrate: knowledge-based comparison of the results of accurate and lower-resolution analyses and a non-routine disorder refinement

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Computing details

Data collection: *APEX2* (Bruker, 2005) for 100K; *CrysAlis PRO* (Rigaku OD, 2018) for 295K. For both structures, cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018). Program(s) used to solve structure: *SHELXT2014/5* (Sheldrick, 2015a) for 100K; *SIR92* (Altomare *et al.*, 1994). for 295K. For both structures, program(s) used to refine structure: *SHELXL2018/1* (Sheldrick, 2015b). Molecular graphics: *DIAMOND* (Brandenburg, 2007) and *Mercury* (Macrae *et al.*, 2006) for 100K; *DIAMOND* (Brandenburg, 2007) for 295K. Software used to prepare material for publication: *SHELXL2018/1* (Sheldrick, 2015b), *WinGX* (Farrugia, 2012), *CrystalExplorer* (Spackman & Jayatilaka, 2009) for 100K; *SHELXL2018/1* (Sheldrick, 2015b) for 295K.

Tetra-*n*-butylammonium 2,6-dioxo-1,2,3,6-tetrahydropyrimidine-4-carboxylate monohydrate (100K)

Crystal data

$C_{16}H_{36}N^+ \cdot C_5H_3N_2O_4^- \cdot H_2O$	$F(000) = 912$
$M_r = 415.57$	$D_x = 1.150$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 10.0905$ (5) Å	Cell parameters from 17418 reflections
$b = 14.8664$ (8) Å	$\theta = 2.0$–$30.2°$
$c = 16.1261$ (9) Å	$\mu = 0.08$ mm^{-1}
$\beta = 97.347$ (5)°	$T = 100$ K
$V = 2399.2$ (2) Å^3	Irregular, colourless
$Z = 4$	$0.31 \times 0.18 \times 0.16$ mm

Data collection

Bruker APEXII CCD diffractometer	26211 measured reflections
Radiation source: fine-focus sealed X-ray tube	6560 independent reflections
Detector resolution: 7.9 pixels mm^{-1}	5274 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.025$
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)	$\theta_{max} = 30.1°$, $\theta_{min} = 1.9°$
	$h = -14 \rightarrow 13$
	$k = -19 \rightarrow 20$
$T_{min} = 0.632$, $T_{max} = 1.000$	$l = -19 \rightarrow 22$

Refinement

Refinement on F^2	$wR(F^2) = 0.118$
Least-squares matrix: full	$S = 1.03$
$R[F^2 > 2\sigma(F^2)] = 0.046$	6560 reflections

297 parameters
1 restraint
Primary atom site location: iterative
Secondary atom site location: difference Fourier
  map
Hydrogen site location: mixed

H atoms treated by a mixture of independent
  and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0533P)^2 + 0.9257P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.66470 (9)	0.57393 (6)	0.44839 (5)	0.01392 (17)	
H1	0.6305 (15)	0.5427 (10)	0.4853 (9)	0.027 (4)*	
C2	0.80132 (10)	0.58337 (7)	0.46003 (6)	0.0153 (2)	
O2	0.87264 (8)	0.55019 (6)	0.51914 (5)	0.02277 (18)	
N3	0.85324 (9)	0.63250 (7)	0.39951 (6)	0.01872 (19)	
H3	0.9431 (17)	0.6356 (11)	0.4049 (10)	0.033 (4)*	
C4	0.78097 (12)	0.67160 (9)	0.32996 (8)	0.0257 (3)	
O4	0.83964 (9)	0.70891 (9)	0.27739 (7)	0.0466 (3)	
C5	0.63733 (11)	0.66355 (9)	0.32642 (8)	0.0234 (2)	
H5	0.5824 (16)	0.6918 (11)	0.2842 (10)	0.032 (4)*	
C6	0.58455 (10)	0.61536 (7)	0.38472 (6)	0.01468 (19)	
C7	0.43431 (10)	0.60106 (7)	0.38434 (6)	0.0162 (2)	
O7	0.35897 (8)	0.65773 (6)	0.34528 (6)	0.02556 (19)	
O8	0.40227 (8)	0.53413 (6)	0.42361 (5)	0.02183 (18)	
N10	0.16527 (9)	0.64890 (6)	0.71583 (6)	0.01999 (19)	
C11	0.23130 (12)	0.65152 (8)	0.80568 (7)	0.0252 (2)	
H11A	0.162731	0.666953	0.842093	0.030*	
H11B	0.298703	0.700260	0.811208	0.030*	
C12	0.29905 (18)	0.56466 (11)	0.83718 (10)	0.0440 (4)	
H12A	0.361837	0.544834	0.798430	0.053*	
H12B	0.231249	0.516936	0.839666	0.053*	
C13	0.3754 (2)	0.58039 (15)	0.92478 (12)	0.0700 (7)	
H13A	0.420281	0.523517	0.943974	0.084*	0.698 (4)
H13B	0.446069	0.625580	0.919995	0.084*	0.698 (4)
H13C	0.312396	0.612618	0.956597	0.084*	0.302 (4)
H13D	0.388045	0.520048	0.950543	0.084*	0.302 (4)
C14A	0.2987 (3)	0.60960 (18)	0.98669 (13)	0.0476 (7)	0.698 (4)
H14A	0.224674	0.567621	0.989942	0.071*	0.698 (4)
H14B	0.263000	0.669712	0.972396	0.071*	0.698 (4)
H14C	0.355099	0.611802	1.040852	0.071*	0.698 (4)
C14B	0.4798 (5)	0.6179 (4)	0.9408 (3)	0.0421 (15)	0.302 (4)
H14D	0.471116	0.680281	0.921200	0.063*	0.302 (4)

H14E	0.547700	0.587096	0.912845	0.063*	0.302 (4)
H14F	0.506507	0.617144	1.001350	0.063*	0.302 (4)
C15	0.26768 (12)	0.62951 (9)	0.65638 (8)	0.0259 (3)	
H15A	0.297867	0.566407	0.664641	0.031*	
H15B	0.222976	0.634978	0.598272	0.031*	
C16	0.39030 (12)	0.69031 (10)	0.66631 (9)	0.0332 (3)	
H16A	0.361841	0.753793	0.669715	0.040*	
H16B	0.447696	0.675243	0.718934	0.040*	
C17	0.46970 (14)	0.67900 (10)	0.59284 (9)	0.0347 (3)	
H17A	0.492567	0.614683	0.587486	0.042*	
H17B	0.413228	0.697239	0.540801	0.042*	
C18	0.59751 (16)	0.73424 (14)	0.60233 (11)	0.0524 (5)	
H18A	0.656351	0.713928	0.651862	0.079*	
H18B	0.575709	0.797908	0.608666	0.079*	
H18C	0.642885	0.726506	0.552580	0.079*	
C19	0.10337 (11)	0.74153 (7)	0.69748 (7)	0.0201 (2)	
H19A	0.043909	0.754953	0.740200	0.024*	
H19B	0.175833	0.786861	0.703483	0.024*	
C20	0.02375 (13)	0.75215 (9)	0.61164 (8)	0.0276 (3)	
H20A	−0.056570	0.713362	0.607492	0.033*	
H20B	0.078800	0.732696	0.568292	0.033*	
C21	−0.01832 (18)	0.84942 (10)	0.59638 (9)	0.0409 (4)	
H21A	0.062117	0.888186	0.603271	0.049*	
H21B	−0.075909	0.867821	0.638714	0.049*	
C22	−0.0936 (2)	0.86374 (12)	0.50956 (11)	0.0526 (5)	
H22A	−0.175693	0.827766	0.503399	0.079*	
H22B	−0.037301	0.845143	0.467404	0.079*	
H22C	−0.116462	0.927527	0.501895	0.079*	
C23	0.05896 (12)	0.57558 (8)	0.70357 (7)	0.0238 (2)	
H23A	0.015447	0.577728	0.644991	0.029*	
H23B	0.103353	0.516338	0.712323	0.029*	
C24	−0.04814 (14)	0.58272 (9)	0.76131 (8)	0.0301 (3)	
H24A	−0.083940	0.644790	0.759398	0.036*	
H24B	−0.008146	0.570105	0.819443	0.036*	
C25	−0.16213 (16)	0.51665 (11)	0.73614 (8)	0.0388 (4)	
H25A	−0.200163	0.528091	0.677417	0.047*	
H25B	−0.126628	0.454496	0.739651	0.047*	
C26	−0.27213 (18)	0.52528 (14)	0.79220 (10)	0.0524 (5)	
H26A	−0.309482	0.586185	0.787442	0.079*	
H26B	−0.234829	0.513751	0.850363	0.079*	
H26C	−0.342804	0.481451	0.774853	0.079*	
O1W	0.13336 (8)	0.63464 (7)	0.43187 (6)	0.02528 (19)	
H1WA	0.193 (2)	0.6418 (13)	0.4002 (12)	0.048 (5)*	
H1WB	0.144 (2)	0.5788 (15)	0.4471 (13)	0.054 (6)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0109 (4)	0.0163 (4)	0.0148 (4)	−0.0007 (3)	0.0026 (3)	0.0026 (3)
C2	0.0119 (4)	0.0161 (5)	0.0181 (5)	−0.0002 (4)	0.0026 (4)	−0.0011 (4)
O2	0.0132 (4)	0.0317 (4)	0.0226 (4)	−0.0008 (3)	−0.0007 (3)	0.0078 (3)
N3	0.0109 (4)	0.0228 (5)	0.0230 (5)	−0.0005 (3)	0.0039 (3)	0.0052 (4)
C4	0.0167 (5)	0.0318 (6)	0.0292 (6)	−0.0005 (5)	0.0052 (4)	0.0130 (5)
O4	0.0195 (4)	0.0752 (8)	0.0465 (6)	−0.0017 (5)	0.0091 (4)	0.0391 (6)
C5	0.0143 (5)	0.0304 (6)	0.0252 (5)	0.0010 (4)	0.0014 (4)	0.0120 (5)
C6	0.0118 (4)	0.0152 (4)	0.0170 (5)	0.0001 (4)	0.0020 (4)	0.0000 (4)
C7	0.0121 (4)	0.0204 (5)	0.0158 (5)	−0.0010 (4)	0.0012 (4)	0.0018 (4)
O7	0.0139 (4)	0.0272 (4)	0.0350 (5)	0.0016 (3)	0.0009 (3)	0.0129 (4)
O8	0.0145 (4)	0.0270 (4)	0.0232 (4)	−0.0043 (3)	−0.0004 (3)	0.0102 (3)
N10	0.0175 (4)	0.0187 (4)	0.0225 (5)	0.0035 (4)	−0.0024 (3)	−0.0062 (4)
C11	0.0240 (6)	0.0257 (6)	0.0235 (6)	0.0031 (5)	−0.0058 (4)	−0.0094 (5)
C12	0.0537 (9)	0.0381 (8)	0.0336 (7)	0.0191 (7)	−0.0205 (7)	−0.0099 (6)
C13	0.0857 (15)	0.0685 (13)	0.0436 (10)	0.0356 (12)	−0.0387 (10)	−0.0153 (9)
C14A	0.0655 (16)	0.0519 (14)	0.0238 (10)	−0.0146 (12)	−0.0007 (10)	0.0023 (9)
C14B	0.030 (2)	0.042 (3)	0.047 (3)	−0.014 (2)	−0.022 (2)	0.000 (2)
C15	0.0186 (5)	0.0293 (6)	0.0287 (6)	0.0079 (5)	−0.0013 (4)	−0.0149 (5)
C16	0.0189 (5)	0.0451 (8)	0.0359 (7)	0.0006 (5)	0.0048 (5)	−0.0230 (6)
C17	0.0265 (6)	0.0421 (8)	0.0358 (7)	0.0029 (6)	0.0054 (5)	−0.0189 (6)
C18	0.0301 (7)	0.0788 (13)	0.0516 (9)	−0.0103 (8)	0.0173 (7)	−0.0362 (9)
C19	0.0175 (5)	0.0173 (5)	0.0264 (5)	0.0034 (4)	0.0055 (4)	−0.0032 (4)
C20	0.0311 (6)	0.0244 (6)	0.0270 (6)	0.0065 (5)	0.0017 (5)	0.0020 (5)
C21	0.0595 (10)	0.0311 (7)	0.0326 (7)	0.0181 (7)	0.0072 (7)	0.0056 (6)
C22	0.0720 (12)	0.0442 (9)	0.0404 (9)	0.0195 (9)	0.0023 (8)	0.0180 (7)
C23	0.0269 (6)	0.0190 (5)	0.0231 (5)	−0.0018 (4)	−0.0062 (4)	−0.0029 (4)
C24	0.0316 (7)	0.0304 (6)	0.0269 (6)	−0.0105 (5)	−0.0009 (5)	−0.0013 (5)
C25	0.0432 (8)	0.0486 (9)	0.0223 (6)	−0.0243 (7)	−0.0044 (5)	0.0026 (6)
C26	0.0499 (9)	0.0774 (13)	0.0296 (7)	−0.0363 (9)	0.0038 (7)	−0.0009 (8)
O1W	0.0137 (4)	0.0286 (5)	0.0344 (5)	0.0012 (3)	0.0063 (3)	0.0043 (4)

Geometric parameters (Å, º)

N1—C6	1.3693 (13)	C15—H15A	0.9900
N1—C2	1.3745 (13)	C15—H15B	0.9900
N1—H1	0.860 (16)	C16—C17	1.5220 (18)
C2—O2	1.2226 (13)	C16—H16A	0.9900
C2—N3	1.3751 (14)	C16—H16B	0.9900
N3—C4	1.3851 (15)	C17—C18	1.520 (2)
N3—H3	0.901 (17)	C17—H17A	0.9900
C4—O4	1.2268 (14)	C17—H17B	0.9900
C4—C5	1.4482 (16)	C18—H18A	0.9800
C5—C6	1.3449 (15)	C18—H18B	0.9800
C5—H5	0.922 (16)	C18—H18C	0.9800
C6—C7	1.5301 (14)	C19—C20	1.5173 (17)

C7—O8	1.2441 (13)	C19—H19A	0.9900
C7—O7	1.2498 (13)	C19—H19B	0.9900
N10—C11	1.5160 (15)	C20—C21	1.5182 (18)
N10—C15	1.5243 (15)	C20—H20A	0.9900
N10—C23	1.5243 (15)	C20—H20B	0.9900
N10—C19	1.5254 (14)	C21—C22	1.520 (2)
C11—C12	1.5177 (19)	C21—H21A	0.9900
C11—H11A	0.9900	C21—H21B	0.9900
C11—H11B	0.9900	C22—H22A	0.9800
C12—C13	1.538 (2)	C22—H22B	0.9800
C12—H12A	0.9900	C22—H22C	0.9800
C12—H12B	0.9900	C23—C24	1.5177 (19)
C13—C14B	1.191 (5)	C23—H23A	0.9900
C13—C14A	1.408 (3)	C23—H23B	0.9900
C13—H13A	0.9900	C24—C25	1.5274 (18)
C13—H13B	0.9900	C24—H24A	0.9900
C13—H13C	0.9900	C24—H24B	0.9900
C13—H13D	0.9900	C25—C26	1.524 (2)
C14A—H14A	0.9800	C25—H25A	0.9900
C14A—H14B	0.9800	C25—H25B	0.9900
C14A—H14C	0.9800	C26—H26A	0.9800
C14B—H14D	0.9800	C26—H26B	0.9800
C14B—H14E	0.9800	C26—H26C	0.9800
C14B—H14F	0.9800	O1W—H1WA	0.85 (2)
C15—C16	1.5242 (18)	O1W—H1WB	0.87 (2)
C6—N1—C2	122.84 (9)	H15A—C15—H15B	107.5
C6—N1—H1	120.7 (10)	C17—C16—C15	110.72 (11)
C2—N1—H1	116.4 (10)	C17—C16—H16A	109.5
O2—C2—N1	123.05 (9)	C15—C16—H16A	109.5
O2—C2—N3	121.87 (9)	C17—C16—H16B	109.5
N1—C2—N3	115.07 (9)	C15—C16—H16B	109.5
C2—N3—C4	126.07 (9)	H16A—C16—H16B	108.1
C2—N3—H3	115.3 (10)	C18—C17—C16	112.72 (11)
C4—N3—H3	118.5 (10)	C18—C17—H17A	109.0
O4—C4—N3	119.91 (11)	C16—C17—H17A	109.0
O4—C4—C5	125.39 (11)	C18—C17—H17B	109.0
N3—C4—C5	114.69 (10)	C16—C17—H17B	109.0
C6—C5—C4	120.02 (10)	H17A—C17—H17B	107.8
C6—C5—H5	120.3 (10)	C17—C18—H18A	109.5
C4—C5—H5	119.7 (10)	C17—C18—H18B	109.5
C5—C6—N1	121.00 (10)	H18A—C18—H18B	109.5
C5—C6—C7	123.50 (10)	C17—C18—H18C	109.5
N1—C6—C7	115.49 (9)	H18A—C18—H18C	109.5
O8—C7—O7	127.93 (10)	H18B—C18—H18C	109.5
O8—C7—C6	115.52 (9)	C20—C19—N10	115.27 (9)
O7—C7—C6	116.54 (9)	C20—C19—H19A	108.5
C11—N10—C15	110.83 (9)	N10—C19—H19A	108.5

C11—N10—C23	110.99 (9)	C20—C19—H19B	108.5
C15—N10—C23	107.80 (9)	N10—C19—H19B	108.5
C11—N10—C19	106.33 (8)	H19A—C19—H19B	107.5
C15—N10—C19	110.03 (9)	C19—C20—C21	110.58 (11)
C23—N10—C19	110.88 (9)	C19—C20—H20A	109.5
N10—C11—C12	114.97 (10)	C21—C20—H20A	109.5
N10—C11—H11A	108.5	C19—C20—H20B	109.5
C12—C11—H11A	108.5	C21—C20—H20B	109.5
N10—C11—H11B	108.5	H20A—C20—H20B	108.1
C12—C11—H11B	108.5	C20—C21—C22	112.28 (13)
H11A—C11—H11B	107.5	C20—C21—H21A	109.1
C11—C12—C13	109.23 (13)	C22—C21—H21A	109.1
C11—C12—H12A	109.8	C20—C21—H21B	109.1
C13—C12—H12A	109.8	C22—C21—H21B	109.1
C11—C12—H12B	109.8	H21A—C21—H21B	107.9
C13—C12—H12B	109.8	C21—C22—H22A	109.5
H12A—C12—H12B	108.3	C21—C22—H22B	109.5
C14B—C13—C12	126.5 (4)	H22A—C22—H22B	109.5
C14A—C13—C12	116.25 (19)	C21—C22—H22C	109.5
C14A—C13—H13A	108.2	H22A—C22—H22C	109.5
C12—C13—H13A	108.2	H22B—C22—H22C	109.5
C14A—C13—H13B	108.2	C24—C23—N10	114.52 (10)
C12—C13—H13B	108.2	C24—C23—H23A	108.6
H13A—C13—H13B	107.4	N10—C23—H23A	108.6
C14B—C13—H13C	105.7	C24—C23—H23B	108.6
C12—C13—H13C	105.7	N10—C23—H23B	108.6
C14B—C13—H13D	105.7	H23A—C23—H23B	107.6
C12—C13—H13D	105.7	C23—C24—C25	111.29 (11)
H13C—C13—H13D	106.1	C23—C24—H24A	109.4
C13—C14A—H14A	109.5	C25—C24—H24A	109.4
C13—C14A—H14B	109.5	C23—C24—H24B	109.4
H14A—C14A—H14B	109.5	C25—C24—H24B	109.4
C13—C14A—H14C	109.5	H24A—C24—H24B	108.0
H14A—C14A—H14C	109.5	C26—C25—C24	111.66 (13)
H14B—C14A—H14C	109.5	C26—C25—H25A	109.3
C13—C14B—H14D	109.5	C24—C25—H25A	109.3
C13—C14B—H14E	109.5	C26—C25—H25B	109.3
H14D—C14B—H14E	109.5	C24—C25—H25B	109.3
C13—C14B—H14F	109.5	H25A—C25—H25B	107.9
H14D—C14B—H14F	109.5	C25—C26—H26A	109.5
H14E—C14B—H14F	109.5	C25—C26—H26B	109.5
C16—C15—N10	115.36 (9)	H26A—C26—H26B	109.5
C16—C15—H15A	108.4	C25—C26—H26C	109.5
N10—C15—H15A	108.4	H26A—C26—H26C	109.5
C16—C15—H15B	108.4	H26B—C26—H26C	109.5
N10—C15—H15B	108.4	H1WA—O1W—H1WB	102.7 (18)
C6—N1—C2—O2	176.23 (10)	N10—C11—C12—C13	173.31 (15)

C6—N1—C2—N3	−4.41 (15)	C11—C12—C13—C14B	−76.8 (4)
O2—C2—N3—C4	179.14 (12)	C11—C12—C13—C14A	59.0 (3)
N1—C2—N3—C4	−0.22 (16)	C11—N10—C15—C16	−52.03 (14)
C2—N3—C4—O4	−175.20 (13)	C23—N10—C15—C16	−173.69 (11)
C2—N3—C4—C5	4.54 (18)	C19—N10—C15—C16	65.28 (13)
O4—C4—C5—C6	175.13 (14)	N10—C15—C16—C17	−167.64 (11)
N3—C4—C5—C6	−4.60 (19)	C15—C16—C17—C18	−176.63 (14)
C4—C5—C6—N1	0.54 (18)	C11—N10—C19—C20	−176.33 (10)
C4—C5—C6—C7	−178.16 (11)	C15—N10—C19—C20	63.60 (12)
C2—N1—C6—C5	4.30 (16)	C23—N10—C19—C20	−55.57 (13)
C2—N1—C6—C7	−176.90 (9)	N10—C19—C20—C21	−173.01 (11)
C5—C6—C7—O8	157.63 (11)	C19—C20—C21—C22	177.68 (13)
N1—C6—C7—O8	−21.14 (14)	C11—N10—C23—C24	55.88 (13)
C5—C6—C7—O7	−22.68 (16)	C15—N10—C23—C24	177.43 (10)
N1—C6—C7—O7	158.55 (10)	C19—N10—C23—C24	−62.08 (13)
C15—N10—C11—C12	−60.83 (15)	N10—C23—C24—C25	170.92 (11)
C23—N10—C11—C12	58.93 (15)	C23—C24—C25—C26	−178.31 (13)
C19—N10—C11—C12	179.62 (12)		

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O8[i]	0.860 (16)	1.924 (16)	2.7668 (12)	166.4 (14)
N3—H3⋯O1W[ii]	0.901 (17)	1.913 (17)	2.8081 (12)	171.8 (15)
C11—H11B⋯O7[iii]	0.99	2.25	3.1462 (15)	151
C19—H19A⋯O4[iv]	0.99	2.28	3.1878 (14)	151
C23—H23A⋯O2[v]	0.99	2.37	3.3305 (14)	164
C24—H24A⋯O4[iv]	0.99	2.34	3.3197 (19)	171
O1W—H1WA⋯O7	0.85 (2)	2.00 (2)	2.8396 (12)	169.2 (18)
O1W—H1WA⋯O8	0.85 (2)	2.64 (2)	3.1155 (12)	117.3 (15)
O1W—H1WB⋯O2[i]	0.87 (2)	2.01 (2)	2.8618 (13)	168.3 (19)

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

(295K)

Crystal data

$C_{16}H_{36}N^+ \cdot C_5H_3N_2O_4^- \cdot H_2O$	$F(000) = 912$
$M_r = 415.57$	$D_x = 1.105$ Mg m⁻³
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 10.1335$ (5) Å	Cell parameters from 3106 reflections
$b = 14.6690$ (7) Å	$\theta = 3.0–21.2°$
$c = 16.9205$ (8) Å	$\mu = 0.08$ mm⁻¹
$\beta = 96.630$ (4)°	$T = 295$ K
$V = 2498.4$ (2) Å³	Block, colourless
$Z = 4$	0.55 × 0.23 × 0.09 mm

Data collection

Rigaku Oxford Diffraction Xcalibur, Sapphire3 diffractometer	27640 measured reflections
Radiation source: fine-focus sealed X-ray tube	4273 independent reflections
Detector resolution: 16.3990 pixels mm^{-1}	1621 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.070$
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)	$\theta_{max} = 25.0°$, $\theta_{min} = 2.8°$
	$h = -12 \rightarrow 12$
	$k = -16 \rightarrow 16$
$T_{min} = 0.980$, $T_{max} = 1.000$	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.047$	Hydrogen site location: mixed
$wR(F^2) = 0.095$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.04$	
4273 reflections	$w = 1/[\sigma^2(F_o^2) + (0.030P)^2]$
307 parameters	where $P = (F_o^2 + 2F_c^2)/3$
56 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.26$ e Å^{-3}
	$\Delta\rho_{min} = -0.19$ e Å^{-3}

Special details

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

Refinement. Please see the Supporting Information for a full description of the refinement of the disorder assembly involving one of the n-butyl groups.

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

	x	y	z	$U_{iso}*/U_{eq}$	Occ. (<1)
N1	0.65997 (17)	0.57519 (13)	0.44599 (11)	0.0472 (5)	
H1	0.6254 (18)	0.5453 (13)	0.4826 (12)	0.057*	
C2	0.7952 (2)	0.58205 (16)	0.45510 (17)	0.0499 (6)	
O2	0.86644 (14)	0.55274 (11)	0.51216 (10)	0.0734 (6)	
N3	0.84553 (18)	0.62622 (13)	0.39389 (14)	0.0579 (6)	
H3	0.931 (2)	0.6305 (14)	0.3974 (12)	0.069*	
C4	0.7732 (3)	0.65988 (18)	0.32579 (19)	0.0774 (8)	
O4	0.83182 (17)	0.69171 (15)	0.27272 (13)	0.1322 (9)	
C5	0.6322 (2)	0.65389 (19)	0.32529 (17)	0.0718 (8)	
H5	0.578 (2)	0.6779 (14)	0.2804 (13)	0.086*	
C6	0.5795 (2)	0.61233 (15)	0.38384 (15)	0.0479 (6)	
C7	0.4310 (2)	0.60074 (19)	0.38728 (16)	0.0555 (7)	
O7	0.35729 (15)	0.65242 (12)	0.34479 (11)	0.0857 (6)	
O8	0.39840 (14)	0.54039 (12)	0.43105 (10)	0.0815 (6)	
N10	0.14286 (19)	0.65933 (14)	0.70428 (12)	0.0630 (6)	
C11	0.2037 (3)	0.66598 (19)	0.79054 (16)	0.0841 (9)	
H11A	0.266990	0.715812	0.794999	0.101*	
H11B	0.133751	0.681476	0.822808	0.101*	

C12	0.2727 (3)	0.5820 (2)	0.82472 (19)	0.1144 (11)	
H12A	0.207327	0.535090	0.831215	0.137*	0.55
H12B	0.331876	0.559208	0.788087	0.137*	0.55
H12C	0.355850	0.573372	0.802451	0.137*	0.45
H12D	0.217376	0.528770	0.812206	0.137*	0.45
C13A	0.3493 (13)	0.6010 (10)	0.9019 (6)	0.193 (4)	0.55
H13A	0.427192	0.635204	0.891178	0.232*	0.2
H13B	0.295887	0.641730	0.930300	0.232*	0.2
H13E	0.410871	0.550797	0.913505	0.232*	0.35
H13F	0.402359	0.654935	0.895443	0.232*	0.35
C14A	0.2872 (13)	0.6139 (10)	0.9661 (6)	0.180 (4)	0.35
H14A	0.351458	0.625437	1.011269	0.270*	0.35
H14B	0.228369	0.665130	0.957613	0.270*	0.35
H14C	0.236933	0.560366	0.975783	0.270*	0.35
C13B	0.2992 (16)	0.5948 (12)	0.9159 (7)	0.193 (4)	0.45
H13C	0.329722	0.656607	0.927175	0.232*	0.2
H13D	0.216411	0.586889	0.938731	0.232*	0.2
H13G	0.240945	0.640475	0.934596	0.232*	0.25
H13H	0.288846	0.537957	0.943909	0.232*	0.25
C14B	0.4340 (15)	0.6244 (12)	0.9250 (10)	0.182 (4)	0.25
H14D	0.462783	0.634885	0.980312	0.272*	0.25
H14E	0.488744	0.578362	0.905009	0.272*	0.25
H14F	0.441292	0.679919	0.895783	0.272*	0.25
C14C	0.3913 (10)	0.5353 (6)	0.9520 (6)	0.180 (4)	0.4
H14G	0.453563	0.497510	0.928456	0.270*	0.2
H14H	0.433849	0.561535	1.000342	0.270*	0.2
H14J	0.317066	0.499029	0.963613	0.270*	0.2
H14K	0.347631	0.480510	0.965991	0.270*	0.2
H14L	0.455048	0.520762	0.916123	0.270*	0.2
H14M	0.435875	0.562833	0.999198	0.270*	0.2
C15	0.2480 (2)	0.63645 (16)	0.65027 (15)	0.0750 (8)	
H15A	0.205410	0.632749	0.596007	0.090*	
H15B	0.283681	0.576591	0.664703	0.090*	
C16	0.3631 (3)	0.70328 (19)	0.65243 (17)	0.0967 (9)	
H16A	0.328748	0.762690	0.635352	0.116*	
H16B	0.404331	0.709057	0.706854	0.116*	
C17	0.4627 (3)	0.6762 (2)	0.6026 (2)	0.1374 (13)	
H17A	0.420044	0.669633	0.548602	0.165*	
H17B	0.495856	0.616595	0.620048	0.165*	
C18	0.5782 (3)	0.7383 (2)	0.60122 (19)	0.1481 (14)	
H18A	0.635629	0.732614	0.650213	0.178*	
H18B	0.547327	0.800037	0.595035	0.178*	
H18C	0.626182	0.722348	0.557510	0.178*	
C19	0.0819 (2)	0.75089 (16)	0.68169 (16)	0.0702 (8)	
H19A	0.018345	0.765289	0.718533	0.084*	
H19B	0.151486	0.796579	0.688643	0.084*	
C20	0.0128 (3)	0.75878 (18)	0.59841 (18)	0.0884 (9)	
H20A	0.073923	0.741475	0.560917	0.106*	

H20B	−0.061720	0.716915	0.591922	0.106*
C21	−0.0359 (3)	0.8531 (2)	0.58024 (19)	0.1206 (12)
H21A	0.038787	0.894827	0.587842	0.145*
H21B	−0.097424	0.869888	0.617650	0.145*
C22	−0.1041 (4)	0.8642 (2)	0.4970 (2)	0.1652 (16)
H22A	−0.184583	0.829057	0.490978	0.198*
H22B	−0.046435	0.843303	0.459575	0.198*
H22C	−0.124951	0.927360	0.487189	0.198*
C23	0.0391 (2)	0.58363 (15)	0.69378 (15)	0.0750 (8)
H23A	0.083208	0.525582	0.704889	0.090*
H23B	0.000089	0.582772	0.638666	0.090*
C24	−0.0726 (3)	0.5930 (2)	0.7470 (2)	0.1180 (12)
H24A	−0.033422	0.594611	0.802102	0.142*
H24B	−0.117241	0.650779	0.735494	0.142*
C25	−0.1686 (3)	0.5225 (2)	0.7383 (2)	0.1292 (12)
H25A	−0.123636	0.464660	0.749059	0.155*
H25B	−0.208397	0.521395	0.683314	0.155*
C26	−0.2779 (3)	0.5302 (2)	0.79096 (19)	0.1455 (14)
H26A	−0.317042	0.589774	0.785299	0.175*
H26B	−0.241944	0.520775	0.845354	0.175*
H26C	−0.344529	0.484942	0.775853	0.175*
O1W	0.12369 (17)	0.63080 (13)	0.42446 (14)	0.0860 (7)
H1WA	0.188 (2)	0.6385 (17)	0.3966 (15)	0.103*
H1WB	0.139 (3)	0.5857 (19)	0.4461 (17)	0.103*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0313 (11)	0.0604 (15)	0.0514 (15)	−0.0036 (10)	0.0107 (10)	0.0089 (11)
C2	0.0380 (17)	0.0521 (18)	0.062 (2)	−0.0062 (13)	0.0139 (14)	−0.0010 (15)
O2	0.0381 (9)	0.1015 (14)	0.0779 (13)	−0.0049 (9)	−0.0047 (9)	0.0245 (11)
N3	0.0333 (11)	0.0624 (14)	0.0797 (16)	−0.0024 (11)	0.0139 (13)	0.0126 (13)
C4	0.054 (2)	0.088 (2)	0.093 (2)	−0.0012 (16)	0.0212 (18)	0.0369 (19)
O4	0.0754 (14)	0.197 (2)	0.131 (2)	−0.0048 (14)	0.0396 (13)	0.0974 (18)
C5	0.0474 (18)	0.096 (2)	0.073 (2)	−0.0019 (14)	0.0113 (14)	0.0428 (18)
C6	0.0376 (14)	0.0489 (17)	0.0566 (17)	0.0033 (12)	0.0027 (13)	0.0102 (14)
C7	0.0401 (16)	0.067 (2)	0.0598 (19)	−0.0025 (14)	0.0059 (13)	0.0134 (15)
O7	0.0449 (10)	0.0949 (14)	0.1154 (16)	0.0094 (9)	0.0011 (10)	0.0499 (12)
O8	0.0428 (10)	0.1129 (15)	0.0883 (14)	−0.0103 (10)	0.0052 (9)	0.0527 (12)
N10	0.0656 (13)	0.0544 (15)	0.0706 (16)	0.0000 (12)	0.0148 (12)	−0.0165 (12)
C11	0.095 (2)	0.091 (2)	0.066 (2)	−0.0159 (18)	0.0094 (17)	−0.0229 (18)
C12	0.125 (3)	0.107 (3)	0.102 (3)	−0.001 (2)	−0.030 (2)	0.001 (2)
C13A	0.217 (10)	0.237 (6)	0.106 (5)	−0.034 (6)	−0.061 (6)	0.041 (5)
C14A	0.222 (9)	0.196 (9)	0.108 (5)	−0.028 (6)	−0.046 (6)	0.025 (6)
C13B	0.218 (10)	0.237 (6)	0.106 (5)	−0.035 (6)	−0.060 (6)	0.042 (5)
C14B	0.221 (9)	0.197 (9)	0.109 (6)	−0.029 (6)	−0.052 (6)	0.025 (6)
C14C	0.220 (9)	0.194 (9)	0.110 (5)	−0.026 (6)	−0.048 (6)	0.032 (6)
C15	0.0656 (18)	0.078 (2)	0.082 (2)	0.0122 (15)	0.0130 (15)	−0.0291 (16)

C16	0.078 (2)	0.116 (3)	0.102 (2)	−0.0080 (19)	0.0348 (18)	−0.030 (2)
C17	0.089 (2)	0.171 (4)	0.161 (3)	−0.008 (2)	0.049 (2)	−0.056 (3)
C18	0.093 (2)	0.212 (4)	0.149 (3)	−0.030 (3)	0.054 (2)	−0.055 (3)
C19	0.0719 (17)	0.0452 (19)	0.098 (2)	0.0068 (14)	0.0280 (16)	−0.0108 (17)
C20	0.087 (2)	0.070 (2)	0.109 (3)	0.0185 (17)	0.0141 (18)	0.0040 (19)
C21	0.155 (3)	0.092 (3)	0.117 (3)	0.033 (2)	0.021 (2)	0.016 (2)
C22	0.200 (4)	0.135 (3)	0.158 (4)	0.048 (3)	0.007 (3)	0.046 (3)
C23	0.0700 (18)	0.0534 (18)	0.099 (2)	−0.0105 (15)	0.0007 (16)	−0.0140 (16)
C24	0.086 (2)	0.098 (2)	0.177 (4)	−0.023 (2)	0.043 (2)	−0.036 (2)
C25	0.117 (3)	0.123 (3)	0.150 (3)	−0.039 (2)	0.028 (2)	−0.002 (2)
C26	0.115 (3)	0.202 (4)	0.125 (3)	−0.057 (3)	0.039 (2)	−0.007 (3)
O1W	0.0474 (11)	0.0889 (17)	0.125 (2)	0.0066 (11)	0.0233 (10)	0.0163 (14)

Geometric parameters (Å, º)

N1—C2	1.365 (2)	C14C—H14G	0.9600
N1—C6	1.367 (2)	C14C—H14H	0.9600
N1—H1	0.866 (19)	C14C—H14J	0.9600
C2—O2	1.215 (2)	C14C—H14K	0.9600
C2—N3	1.369 (3)	C14C—H14L	0.9600
N3—C4	1.384 (3)	C14C—H14M	0.9600
N3—H3	0.86 (2)	C15—C16	1.520 (3)
C4—O4	1.225 (3)	C15—H15A	0.9700
C4—C5	1.430 (3)	C15—H15B	0.9700
C5—C6	1.327 (3)	C16—C17	1.443 (3)
C5—H5	0.95 (2)	C16—H16A	0.9700
C6—C7	1.523 (3)	C16—H16B	0.9700
C7—O8	1.224 (2)	C17—C18	1.485 (3)
C7—O7	1.235 (2)	C17—H17A	0.9700
N10—C19	1.509 (3)	C17—H17B	0.9700
N10—C15	1.519 (3)	C18—H18A	0.9600
N10—C11	1.520 (3)	C18—H18B	0.9600
N10—C23	1.526 (2)	C18—H18C	0.9600
C11—C12	1.500 (3)	C19—C20	1.504 (3)
C11—H11A	0.9700	C19—H19A	0.9700
C11—H11B	0.9700	C19—H19B	0.9700
C12—C13A	1.468 (9)	C20—C21	1.490 (3)
C12—C13B	1.547 (11)	C20—H20A	0.9700
C12—H12A	0.9700	C20—H20B	0.9700
C12—H12B	0.9700	C21—C22	1.505 (4)
C12—H12C	0.9700	C21—H21A	0.9700
C12—H12D	0.9700	C21—H21B	0.9700
C13A—C14C	1.321 (12)	C22—H22A	0.9600
C13A—C14A	1.330 (13)	C22—H22B	0.9600
C13A—H13A	0.9700	C22—H22C	0.9600
C13A—H13B	0.9700	C23—C24	1.532 (3)
C13A—H13E	0.9700	C23—H23A	0.9700
C13A—H13F	0.9700	C23—H23B	0.9700

C14A—H14A	0.9600	C24—C25	1.416 (3)
C14A—H14B	0.9600	C24—H24A	0.9700
C14A—H14C	0.9600	C24—H24B	0.9700
C13B—C14C	1.369 (13)	C25—C26	1.504 (4)
C13B—C14B	1.425 (15)	C25—H25A	0.9700
C13B—H13C	0.9700	C25—H25B	0.9700
C13B—H13D	0.9700	C26—H26A	0.9600
C13B—H13G	0.9700	C26—H26B	0.9600
C13B—H13H	0.9700	C26—H26C	0.9600
C14B—H14D	0.9600	O1W—H1WA	0.85 (2)
C14B—H14E	0.9600	O1W—H1WB	0.76 (3)
C14B—H14F	0.9600		
C2—N1—C6	124.0 (2)	H14G—C14C—H14H	109.5
C2—N1—H1	116.2 (13)	C13A—C14C—H14J	109.5
C6—N1—H1	119.8 (13)	H14G—C14C—H14J	109.5
O2—C2—N1	124.1 (2)	H14H—C14C—H14J	109.5
O2—C2—N3	122.0 (2)	C13B—C14C—H14K	109.5
N1—C2—N3	114.0 (2)	C13B—C14C—H14L	109.5
C2—N3—C4	126.2 (2)	H14K—C14C—H14L	109.5
C2—N3—H3	116.1 (14)	C13B—C14C—H14M	109.5
C4—N3—H3	117.6 (15)	H14K—C14C—H14M	109.5
O4—C4—N3	119.4 (2)	H14L—C14C—H14M	109.5
O4—C4—C5	126.0 (3)	N10—C15—C16	115.61 (19)
N3—C4—C5	114.6 (3)	N10—C15—H15A	108.4
C6—C5—C4	120.8 (2)	C16—C15—H15A	108.4
C6—C5—H5	121.5 (13)	N10—C15—H15B	108.4
C4—C5—H5	117.7 (13)	C16—C15—H15B	108.4
C5—C6—N1	120.1 (2)	H15A—C15—H15B	107.4
C5—C6—C7	124.4 (2)	C17—C16—C15	113.2 (2)
N1—C6—C7	115.5 (2)	C17—C16—H16A	108.9
O8—C7—O7	127.5 (2)	C15—C16—H16A	108.9
O8—C7—C6	116.2 (2)	C17—C16—H16B	108.9
O7—C7—C6	116.3 (2)	C15—C16—H16B	108.9
C19—N10—C15	109.83 (19)	H16A—C16—H16B	107.7
C19—N10—C11	107.1 (2)	C16—C17—C18	116.5 (3)
C15—N10—C11	110.92 (18)	C16—C17—H17A	108.2
C19—N10—C23	111.21 (17)	C18—C17—H17A	108.2
C15—N10—C23	106.93 (18)	C16—C17—H17B	108.2
C11—N10—C23	110.85 (19)	C18—C17—H17B	108.2
C12—C11—N10	115.9 (2)	H17A—C17—H17B	107.3
C12—C11—H11A	108.3	C17—C18—H18A	109.5
N10—C11—H11A	108.3	C17—C18—H18B	109.5
C12—C11—H11B	108.3	H18A—C18—H18B	109.5
N10—C11—H11B	108.3	C17—C18—H18C	109.5
H11A—C11—H11B	107.4	H18A—C18—H18C	109.5
C13A—C12—C11	111.3 (6)	H18B—C18—H18C	109.5
C11—C12—C13B	107.7 (6)	C20—C19—N10	116.0 (2)

C13A—C12—H12A	109.4	C20—C19—H19A	108.3
C11—C12—H12A	109.4	N10—C19—H19A	108.3
C13A—C12—H12B	109.4	C20—C19—H19B	108.3
C11—C12—H12B	109.4	N10—C19—H19B	108.3
H12A—C12—H12B	108.0	H19A—C19—H19B	107.4
C11—C12—H12C	110.2	C21—C20—C19	111.9 (2)
C13B—C12—H12C	110.2	C21—C20—H20A	109.2
C11—C12—H12D	110.2	C19—C20—H20A	109.2
C13B—C12—H12D	110.2	C21—C20—H20B	109.2
H12C—C12—H12D	108.5	C19—C20—H20B	109.2
C14C—C13A—C12	122.0 (12)	H20A—C20—H20B	107.9
C14A—C13A—C12	120.2 (12)	C20—C21—C22	113.5 (3)
C14C—C13A—H13A	106.8	C20—C21—H21A	108.9
C12—C13A—H13A	106.8	C22—C21—H21A	108.9
C14C—C13A—H13B	106.8	C20—C21—H21B	108.9
C12—C13A—H13B	106.8	C22—C21—H21B	108.9
H13A—C13A—H13B	106.7	H21A—C21—H21B	107.7
C14A—C13A—H13E	107.3	C21—C22—H22A	109.5
C12—C13A—H13E	107.3	C21—C22—H22B	109.5
C14A—C13A—H13F	107.3	H22A—C22—H22B	109.5
C12—C13A—H13F	107.3	C21—C22—H22C	109.5
H13E—C13A—H13F	106.9	H22A—C22—H22C	109.5
C13A—C14A—H14A	109.5	H22B—C22—H22C	109.5
C13A—C14A—H14B	109.5	N10—C23—C24	114.4 (2)
H14A—C14A—H14B	109.5	N10—C23—H23A	108.7
C13A—C14A—H14C	109.5	C24—C23—H23A	108.7
H14A—C14A—H14C	109.5	N10—C23—H23B	108.7
H14B—C14A—H14C	109.5	C24—C23—H23B	108.7
C14C—C13B—C12	113.5 (12)	H23A—C23—H23B	107.6
C14B—C13B—C12	101.4 (12)	C25—C24—C23	114.7 (3)
C14C—C13B—H13C	108.9	C25—C24—H24A	108.6
C12—C13B—H13C	108.9	C23—C24—H24A	108.6
C14C—C13B—H13D	108.9	C25—C24—H24B	108.6
C12—C13B—H13D	108.9	C23—C24—H24B	108.6
H13C—C13B—H13D	107.7	H24A—C24—H24B	107.6
C14B—C13B—H13G	111.5	C24—C25—C26	115.3 (3)
C12—C13B—H13G	111.5	C24—C25—H25A	108.5
C14B—C13B—H13H	111.5	C26—C25—H25A	108.5
C12—C13B—H13H	111.5	C24—C25—H25B	108.5
H13G—C13B—H13H	109.3	C26—C25—H25B	108.5
C13B—C14B—H14D	109.5	H25A—C25—H25B	107.5
C13B—C14B—H14E	109.5	C25—C26—H26A	109.5
H14D—C14B—H14E	109.5	C25—C26—H26B	109.5
C13B—C14B—H14F	109.5	H26A—C26—H26B	109.5
H14D—C14B—H14F	109.5	C25—C26—H26C	109.5
H14E—C14B—H14F	109.5	H26A—C26—H26C	109.5
C13A—C14C—H14G	109.5	H26B—C26—H26C	109.5
C13A—C14C—H14H	109.5	H1WA—O1W—H1WB	105 (3)

C6—N1—C2—O2	176.4 (2)	N10—C11—C12—C13B	−167.6 (7)
C6—N1—C2—N3	−3.0 (3)	C11—C12—C13A—C14C	163.1 (11)
O2—C2—N3—C4	178.3 (2)	C11—C12—C13A—C14A	73.6 (15)
N1—C2—N3—C4	−2.2 (3)	C11—C12—C13B—C14C	−163.6 (11)
C2—N3—C4—O4	−174.3 (3)	C11—C12—C13B—C14B	−98.0 (12)
C2—N3—C4—C5	6.1 (4)	C19—N10—C15—C16	60.0 (3)
O4—C4—C5—C6	175.4 (3)	C11—N10—C15—C16	−58.2 (3)
N3—C4—C5—C6	−5.0 (4)	C23—N10—C15—C16	−179.2 (2)
C4—C5—C6—N1	0.5 (4)	N10—C15—C16—C17	177.5 (2)
C4—C5—C6—C7	−178.5 (2)	C15—C16—C17—C18	179.7 (3)
C2—N1—C6—C5	3.9 (3)	C15—N10—C19—C20	60.8 (3)
C2—N1—C6—C7	−177.1 (2)	C11—N10—C19—C20	−178.6 (2)
C5—C6—C7—O8	159.8 (3)	C23—N10—C19—C20	−57.4 (3)
N1—C6—C7—O8	−19.2 (3)	N10—C19—C20—C21	−176.2 (2)
C5—C6—C7—O7	−19.2 (4)	C19—C20—C21—C22	179.2 (3)
N1—C6—C7—O7	161.7 (2)	C19—N10—C23—C24	−62.8 (3)
C19—N10—C11—C12	−178.8 (2)	C15—N10—C23—C24	177.3 (2)
C15—N10—C11—C12	−58.9 (3)	C11—N10—C23—C24	56.3 (3)
C23—N10—C11—C12	59.7 (3)	N10—C23—C24—C25	−179.4 (3)
N10—C11—C12—C13A	168.9 (7)	C23—C24—C25—C26	179.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O8[i]	0.866 (19)	1.96 (2)	2.800 (3)	162.1 (18)
N3—H3···O1*W*[ii]	0.86 (2)	1.96 (2)	2.807 (2)	170 (2)
C11—H11*A*···O7[iii]	0.97	2.26	3.168 (3)	155
C19—H19*A*···O4[iv]	0.97	2.28	3.226 (3)	164
C23—H23*B*···O2[v]	0.97	2.44	3.386 (3)	166
C24—H24*B*···O4[iv]	0.97	2.47	3.346 (4)	151
O1*W*—H1*WA*···O7	0.85 (2)	2.03 (2)	2.874 (2)	172 (2)
O1*W*—H1*WA*···O8	0.85 (2)	2.59 (3)	3.074 (2)	118 (2)
O1*W*—H1*WB*···O2[i]	0.76 (3)	2.15 (3)	2.895 (2)	164 (3)

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