Crystal structures and Hirshfeld surface analyses of hypoxanthine salts involving 5-sulfosalicylate and perchlorate anions

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Two salts of 1,9-dihydropurin-6-one (hypoxanthine), namely, 6-oxo-1,9-dihydropurin-7-ium 5-sulfosalicylate dihydrate, C5H5N4O+·C12H7O6S−·2H2O, (I), and 6-oxo-1,9-dihydropurin-7-ium perchlorate monohydrate, C5H5N4O+·ClO4−·H2O, (II), have been synthesized and characterized using single-crystal X-ray diffraction and Hirshfeld analysis. In both salts, the hypoxanthine molecule is protonated at the N7 position of the purine ring. In salt (I), the cation and anion are connected through N—H–O interactions. The protonated hypoxanthine cations of salt (I) form base pairs with another symmetry-related hypoxanthine cation through N—H–O hydrogen bonds with an R22(8) ring motif, while in salt (II), the hypoxanthine cations are paired through a water molecule via N—H–O and O–H–N hydrogen bonds with an R32(11) ring motif. The packings within the crystal structures are stabilized by π–π stacking interactions in salt (I) and C–O–π interactions in salt (II). The combination of several interactions leads to the formation of supramolecular sheets extending parallel to (010) in salts (I) and (II). Hirshfeld surface analysis and fingerprint plots reveal that O–H···O contacts play the major role in the crystal packing of each of the salts, with a 54.1% contribution in salt (I) and 62.3% in salt (II).

1. Chemical context

1,9-Dihydropurin-6-one (hypoxanthine, C₅H₄N₄O), a notable purine-based nucleotide (Emel’yanenko et al., 2017), is present in the anticodon as nucleoside inosine in t-RNA (Costas & Acevedo-Chávez, 1997; Holley et al., 1965; Stryer, 1988; Plekan et al., 2012; Hughes, 1981; Schmalle et al., 1988). Hypoxanthine and xanthine are significant as drugs in the treatment of infections like gout and xanthinuria. Hypoxanthine is additionally utilized against hypoxia and is known to repress the impact of few medications (Dubler et al., 1987a,b; Biradha et al., 2010).

Hypoxanthine (HX), a potential oxygen-free radical generator, is a strong agent against cancer cells (Susithra et al., 2018; Latosinska et al., 2014; Rutledge et al., 2007). The presence of the imine group in its structure is responsible for its pharmacological activity. Hypoxanthine can exist in two stable tautomers, viz. as the oxo-N7(H) form and as the oxo-N9(H) form. When hypoxanthine interacts with strong acids, it becomes protonated at position N7 or N9. A limited number of hypoxanthine salts like hypoxanthine nitrate (Cabaj & Dominiak, 2021; Cabaj et al., 2019) and hypoxanthine hydrochloride monohydrate (Sletten & Jensen, 1969) have been reported so far in the literature.
The current article reports the crystal structures of hypoxanthinium 5-sulfosalicylate dihydrate, (I), and hypoxanthinium perchlorate monohydrate, (II), salts and the noncovalent interactions that govern their crystal packings.

2. Structural commentary

Salt (I) crystallizes with two hypoxanthinium cations (A⁺ and B⁺), two 5-sulfosalicylate anions (5SCA⁻; A and B) and four solvent water molecules (O1W, O2W, O3W and O4W) in the asymmetric unit, as shown in Fig. 1. In salt (I), the B cation is equally disordered over two sets of sites for atoms C5/C6B/C6C and O6B/O6C. Atoms H1B/H1C and H7B/H7C attached to N1B and N7B, respectively, are also disordered. The solvent water molecule O3W is also disordered over two positions. Atoms N7A and N7B are protonated, which is confirmed by widening of the C5A—N7A—C8A angle to 107.1 (4)° compared to the value of 103.8° in the two polymorphic forms of the neutral HX molecule (Schmalle et al., 1988; Yang & Xie, 2007); the situation for C5B—N7B—C8B is less clear due to the observed disorder. The torsion angles of N3A—C4A—C5A—N7A = −179.2 (4)° and N3B—C4B—C5C—N7B = −178.3 (6)° are similar to those of the two forms of the neutral HX molecule (−179.55 and −178.99°; Schmalle et al., 1988; Yang & Xie, 2007). The carboxylic acid group in each of the two 5SCA⁻ anions is coplanar with the benzene ring [O7A—C9A—C10A—C11A = −178.2 (4)° and O7B—C9B—C10B—C11B = 175.9 (4)°], a situation that is likewise observed for previously reported crystal structures involving 5SCA⁻ anions.

Salt (II) crystallizes with one hypoxanthinium cation, one perchlorate anion (PCA⁻) and one solvent water molecule in the asymmetric unit. The molecular structure of salt (II) is shown in Fig. 2. Again, the N7 atom of the purine ring is protonated, as confirmed by the widening of the C5—N7—C8 angle to 108.00 (12)°. The N3—C4—C5—N7 torsion angle of 179.34 (14)° is similar to the values determined for salt (I). The PCA⁻ anion has the characteristic tetrahedral shape, with Cl—O bond lengths between 1.4116 (15) and 1.4421 (15) Å, and O—Cl—O angles between 108.29 (9) and 111.24 (12)°.

3. Supramolecular features

In the crystal structure of salt (I), (010) sheets of cations and sheets of anions are stacked alternately along [010]. The crystal packing is governed by N—H···O, O—H···N and C—H···O hydrogen bonds (Table 1). Symmetry-related A⁺ cations interact through a pair of N1A—H1A···O6A hydrogen bonds with a robust R2(8) motif (Bernstein et al., 1995; Motherwell et al., 2000). Solvent water molecule OW1 connects the A⁺ cation via N7A—H7A···O1W and O1W—H1WA···O6A hydrogen bonds with an R4(4) motif. The A⁺ cations are further connected via C2A—H2A···O1W, C8A—H8A···O2W, N9A—H9A···O2W and O2W—H2WA···N3A, N1A—H1A···O6A hydrogen bonds with R2(7), R4(14), R2(10) and R2(10) motifs (Fig. 3).

Figure 1
The asymmetric unit of salt (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonding and the disorder of cation B⁺ is shown.

Figure 2
The asymmetric unit of salt (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonding.
The B\(^+\) cations interact with the O atom of the solvent water molecules O3W and O4W through N1B—H1C···O4W and N9B—H9B···O3WA, and with N9B—H9B···O6B with an \(R_2^2(7)\) motif. Short O3WA···O4W contacts with an \(R_3^2(20)\) motif are also observed (Fig. 4). Furthermore, the two 5SCA\(^-\) anions (A and B) self assemble into sheets by interaction of symmetry-related counterparts through O7A—H7D···O10A and O7B—H7E···O10B, respectively (Fig. 5). A and B sheets are interconnected through O9B—H9E···O12A and through O9A—H9D···O12B and C15B—H15B···O9A interactions, resulting in \(R_2^2(7)\), \(R_3^2(23)\) and \(R_4^2(26)\) ring motifs. Moreover, cation B\(^+\) interacts with 5SCA\(^-\) (A) via N1B—H1C···O11A and C2B—H2B···O11A with an \(R_1^2(5)\) motif. Another interconnection between cationic and anionic sheets involves the solvent water molecules through O1W—H1WA···O10B, O1W—H1WB···O12B, O2W—H2WA···N3A and O2W—H2WB···O12A (Fig. 6).
The crystal structure of (I) is consolidated by π-π interactions between the phenyl rings of the two 5SCA anions (C10A–C15A and C10B–C15B), and the imidazole ring (C4A–C4E). N9A) and the pyrimidine ring (N1A–C6A) of cation A⁺, with centroid-to-centroid distances of 3.547 (3), 3.562 (3), 3.554 (3) and 3.533 (3) Å, and slippages of 0.815, 1.300, 1.182 and 1.105 Å (Fig. 7).

In the crystal structure of salt (II), (010) sheets of cations and sheets of anions are stacked alternately along [010]. The crystal packing of salt (II) is dominated by N–H⋯O and O–H⋯O hydrogen bonds, and to a minor extent by C–H⋯O hydrogen bonds (Table 2). The protonated N atom of the cation forms an N7–H7⋯O1W ii hydrogen bond with the O atom of the water molecule. The water molecule disrupts the formation of base pairs but connects symmetry-related cations through O1W–H2W ii⋯O3 iii interactions with an R2₁(11) ring motif generate a cationic strand along [01]. Parallel cationic strands are connected through the solvent water molecule and the PCA⁺ anion through O1W–H1W⋯O3 and bifurcated N1–H1⋯O4 and N1–H1⋯O5 interactions, respectively, forming R2₁(9), R2₁(14) and R2₁(20) motifs. The crystal packing of salt (II) is shown in Fig. 8. The crystal structure is further stabilized by carbon–ylide–π (π refers to the ring system of the cation) interactions.

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

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N7B–H7B⋯O3WA i 0.86 2.26 3.08 158 |
| O7A–H7D⋯O10A ii 0.82 1.86 2.677 170 |
| O7B–H7E⋯O10B ii 0.82 1.84 2.655 175 |
| O9A–H9D⋯O12B ii 0.82 2.34 2.924 128 |
| O9B–H9E⋯O12A iii 0.82 2.54 3.143 131 |
| O1W–H1WA⋯O6A iii 0.85 2.31 2.801 117 |
| O1W–H1WA⋯O10B iv 0.85 2.28 2.917 132 |
| N9B–H9B⋯O6B ii 0.86 2.42 3.044 130 |
| N9B–H9B⋯O3WA iii 0.86 2.47 3.07 128 |
| N1A–H1A⋯O6A iii 0.86 2.05 2.898 170 |
| N1B–H1C⋯O4W 0.86 2.22 2.800 135 |
| N1B–H1C⋯O11A 0.86 2.45 2.998 122 |
| O1W–H1WB⋯O12B 0.85 2.01 2.844 169 |
| O2W–H2WA⋯N3A 0.83 2.07 2.849 157 |
| O2W–H2WB⋯O12A 0.82 2.03 2.815 160 |
| N7A–H7A⋯O1W 0.86 1.77 2.615 168 |
| N9A–H9A⋯O2W 0.86 1.89 2.697 157 |
| C2A–H2A⋯O1W ii 0.93 2.43 3.149 134 |
| C2B–H2B⋯O11A 0.93 2.46 2.974 114 |
| CS4–H8A⋯O2W iii 0.93 2.40 3.310 167 |
| C15B–H15B⋯O9A 0.93 2.59 3.510 172 |

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

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

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N1–H1⋯O4 0.82 2.60 3.249 138 |
| N1–H1⋯O5 0.82 2.09 2.879 162 |
| N7–H7⋯O2 i 0.91 2.60 3.031 110.2 |
| N7–H7⋯O1W ii 0.91 1.76 2.6489 165 |
| N9–H9⋯O6 iii 0.84 1.93 2.7602 166 |
| O1W–H1W⋯O3 iii 0.85 2.17 3.018 172 |
| O1W–H2W⋯N3 0.85 2.11 2.951 172 |
| C8–H8⋯O2 ii 0.93 2.47 2.970 114 |
| C8–H8⋯O3 iii 0.93 2.47 3.268 144 |
| C8–H8⋯O4 iii 0.93 2.55 3.072 116 |

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

Figure 7
π–π stacking interactions in (I) between the imidazole and pyrimidine rings of the cations and the phenyl rings of the anions.

Figure 8
A view of the supramolecular arrangement involving hydrogen-bonded rings in salt (II).
| Compound                                      | Space group       | Primary interaction between | Graph-set motif | Motif type | Secondary interaction between | Graph-set motif | Motif type |
|-----------------------------------------------|-------------------|----------------------------|----------------|-----------|-------------------------------|----------------|-----------|
| Guanidinium hydrochloride                     | Monoclinic P2_1/c | N−H⋯N, N−H⋯O              | R_2^*(8), R_2^*(10) | IV and V   | N−H⋯Cl, C−H⋯Cl, O−H⋯N, O−H⋯Cl | R_2^*(11)     | XII and XIII |
| Guanidinium hydrobromide                     | Monoclinic P2_1/c | N−H⋯N, N−H⋯O              | R_2^*(8), R_2^*(10) | IV and V   | N−H⋯Br, N−H⋯N, O−H⋯Br, C−H⋯Br | R_2^*(11)     | XII and XIII |
| Guanidinium dinitrate dihydrate               | Monoclinic P2_1/c | N−H⋯O                    | R_2^*(8)         | V          | N−H⋯O, O−H⋯O                 | R_2^*(12)     | XII        |
| Guanidinium phosphate monohydrate            | Monoclinic P2_1/c | N−H⋯N                    | R_2^*(8)         | IV         | N−H⋯N, N−H⋯O                 | R_2^*(6), R_2^*(10) | XII and XVIII |
| Guanidinium phosphate dihydrate form (I)      | Monoclinic P2_1/c | N−H⋯N                    | R_2^*(8)         | IV         | N−H⋯N, N−H⋯O                 | R_2^*(8), R_2^*(6) | XIII and XVIII |
| Guanidinium phosphate dihydrate form (II)     | Monoclinic P2_1/c | N−H⋯N                    | R_2^*(8)         | IV         | N−H⋯N, N−H⋯O                 | R_2^*(8), R_2^*(6) | XIII and XVIII |
| Guanidinium phosphate hydrate form (I)        | Triclinic, P\(\overline{1}\) | N−H⋯N                    | R_2^*(8)         | IV         | N−H⋯O, O−H⋯O                 | R_2^*(9), R_2^*(10) | XVI and XVII |
| Guanidinium phosphate monohydrate form (II)   | Monoclinic P2_1/n | N−H⋯N                    | R_2^*(8)         | IV         | N−H⋯O, O−H⋯O                 | R_2^*(8), R_2^*(9) | VI, XIII and XVI |
| Guanidinium sulfate monohydrate               | Monoclinic P2_1/c | N−H⋯O                    | R_2^*(8)         | VI         | N−H⋯O, O−H⋯O                 | R_2^*(12)     | XV         |
| Xanthinium nitrate monohydrate                | Triclinic, P\(\overline{1}\) | N−H⋯O                    | R_2^*(8)         | I          | O−H⋯N, O−H⋯O                 | R_2^*(4), R_2^*(8), R_2^*(14) | VIII, XI and XIII |
| Xanthinium sulfate monohydrate                | Monoclinic P2_1   | N−H⋯O                    | R_2^*(8)         | I          | O−H⋯N                        | R_2^*(8)       | XIII       |
| Xanthinium perchlorate dihydrate              | Triclinic, P\(\overline{1}\) | N−H⋯O                    | R_2^*(8)         | I          | O−H⋯N, O−H⋯O                 | R_2^*(8)       | XIII       |
| Hypoxanthinium hydrochloride monohydrate      | Monoclinic P2_1/c | N−H⋯Cl                    | R_2^*(9)         | III        | N−H⋯Cl, C−H⋯Cl, O−H⋯N       | R_2^*(11), R_2^*(16), R_2^*(14) | IX, X and XI |

Table 3: Comparison of salt forms of purine derivatives containing halides/nitrate/phosphite/phosphate/sulfate and perchlorates as anions.
with distances of 3.6097 (13), 3.2983 (13), 3.4580 (13) and 3.7236 (14) Å (Fig. 9).

4. Hirshfeld surface analysis

Hirshfeld surface (HS) analyses of salts (I) and (II) were performed using CrystalExplorer17 (Turner et al., 2017). The results of the HS analysis mapped over \( d_{\text{norm}} \) are shown in Figs. 10(a) and 10(b) for (I) and (II), respectively. Corresponding two-dimensional fingerprint plots (Spackman & Jayatilaka, 2009) for (I) and (II) are shown in Figs. 11 and 12, respectively. The contributions of the noncovalent interactions to the HS in the two salts are: O···H/H···O 54.1% (I), 62.3% (II); N···H/H···N 3.1% (I), 6.8% (II); C···H/H···C 5.9% (I), 5.4% (II); H···H/H···H 16.0% (I), 5.3% (II); C···C/C···C 0.9% (I), 0.1% (II).

5. Comparison with the structures of related compounds

Crystal data, supramolecular interactions and hydrogen bonding motifs of structurally similar halide/nitrate/phosphate/perchlorate or sulfate salts like guanidinium bromide (Wei, 1977), guanidinium chloride (Maixner & Zachoval, 1991), bis(guanidinium) hydrogen phosphate 2.5-hydrate (Low et al., 1986), guanidinium phosphate (Bendeif et al., 2007), guanidinium sulfate (Cherouana et al., 2003), guanidinium dinitrate dihydrate (Bouchouit et al., 2002), xanthinium nitrate, xanthinium sulfate (Sridhar, 2011), xanthinium perchlorate dihydrate (Biradha et al., 2010), hypoxanthinium chloride (Sletten & Jensen, 1969) and hypoxanthinium nitrate monohydrate (Cabaj et al., 2019) are listed and compared in Table 3.
Figure 11
Fingerprint plots of salt (I) showing all intermolecular interactions and delineated into O···H:H···C, H···N/N···H, C···O, C···N, C···H/H···C and H···H contacts.

Figure 12
Fingerprint plots of salt (II) showing all intermolecular interactions and delineated into O···H:H···O, H···N/N···H, C···O, C···H/H···C and H···H contacts.
a strong acid, the chloride/nitrate/sulfate/phosphite/phosphate or perchlorate salts of guanine/xanthine and hypoxanthine have different molecular recognition patterns. The most important primary and secondary motifs formed by hypox-

| Table 4 | Experimental details. |
|---------|-----------------------|
|         | (I) | (II) |
| Crystal data | C₅H₅N₄O⁺·C₅H₇O₆S⁻·2H₂O | C₅H₅N₄O⁺·ClO₄⁻·H₂O |
| Mᵣ      | 388.32 | 254.60 |
| Crystal system, space group | Monoclinic, P₂₁/c | Monoclinic, P₂₁/c |
| Temperature (K) | 293 | 296 |
| a, b, c (Å) | 8.7055 (3), 25.9927 (13), 13.6479 (5) | 5.0307 (6), 20.386 (2), 9.0181 (10) |
| β (°) | 91.864 (3) | 94.233 (2) |
| V (Å³) | 3086.6 (2) | 922.33 (18) |
| Z      | 8 | 4 |
| Radiation type | Mo Kα | Mo Kα |
| μ (mm⁻¹) | 0.27 | 0.44 |
| Crystal size (mm) | 0.55 × 0.20 × 0.10 | 0.45 × 0.02 × 0.003 |

Data collection

| Diffractometer | Bruker APEXII CCD | Bruker APEXII CCD |
|----------------|-------------------|-------------------|
| Absorption correction | – | Multi-scan (SADABS; Bruker, 2016) |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 21075, 7079, 5905 | 16360, 2752, 2370 |
| R_min, R_max | 0.032, 0.649 | 0.025, 0.711 |

Refinement

| R(F²) | 0.087, 0.190, 1.22 | 0.038, 0.111, 1.05 |
| No. of parameters | 7079 | 2752 |
| No. of restraints | 521 | 165 |
| 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 |
| Δρ_max (e Å⁻³) | 0.74, –0.43 | 0.37, –0.29 |

Computer programs: APEX2 and SAINT (Bruker, 2016), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), Mercury (Macrae et al., 2020), PovRay (Cason, 2004), PLATON (Spek, 2020) and publCIF (Westrip, 2010).

Figure 13

Primary motifs observed in xanthine/ hypoxanthine/ guanine

Primary ring motifs observed in purine derivatives.
anthine and similar compounds are summarized in Figs. 13 and 14. Crystallographic studies of salts involving perchlorate and sulfate anions reveal that most of these salts have similar crystal packing arrangements (Bishop et al., 2014). In general, salts of structurally similar systems will have similar molecular recognition patterns and supramolecular motifs. However, for salts (I) and (II) and related systems compiled in Table 3, great similarities are not observed. The differences in molecular recognition and supramolecular self-assembly might be due to the involvement of other functional groups or substituents in the structures, the intrusion of water molecules in the crystal structure, or the ratio of anions and cations present in the asymmetric unit.

6. Synthesis and crystallization
Salt (I) was synthesized by mixing an equimolar ratio of hypoxanthine (0.0340 g) and 5-sulfosalicylic acid (0.0545 g) in hot water. The solution was heated to 333 K for 1 h and then allowed to cool slowly to room temperature. Colourless needle-shaped crystals were harvested from the mother liquid after one week.

Salt (II) was synthesized by mixing an equimolar ratio of hypoxanthine (0.0340 g) and iron perchlorate monohydrate (0.0681 g) in hot water. The solution was heated to 333 K with constant stirring for 1 h and then allowed to cool slowly to room temperature. Colourless plate-like crystals were harvested from the mother liquid after one week.

Figure 14
Secondary ring motifs observed in purine derivatives.
7. Refinement

Crystal data, data collection and structure refinement details of salts (I) and (II) are summarized in Table 4. In salt (I), carbon (C5 and C6) and oxygen (O6) atoms of cation B are equally disordered over two sets of sites, with a refined occupancy ratio of 0.503 (18):0.497 (18). The solvent water molecule O3W is disordered over two positions, with a refined site-occupancy ratio of 0.58 (6):0.42 (6). The H atoms of water molecules O1W and O2W were located from a difference Fourier map, and the O—H distance restrained to 0.82 Å. Attempts to localize the H atoms of O3

H distance of 1.36 Å. All C-bound H atoms were treated as for salt (I).

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Crystal structures and Hirshfeld surface analyses of hypoxanthine salts involving 5-sulfosalicylate and perchlorate anions

Udhayasuriyan Sathya, Jeyaraman Selvaraj Nirmalram, Sundaramoorthy Gomathi, Franc Perdih, Samson Jegan Jennifer and Ibrahim Abdul Razak

Computing details
For both structures, data collection: APEX2 (Bruker, 2016); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: PLATON (Spek, 2020), Mercury (Macrae et al., 2020) and POVRay (Cason, 2004); software used to prepare material for publication: PLATON (Spek, 2020) and publCIF (Westrip, 2010).

6-Oxo-1,9-dihydropurin-7-ium 5-sulfosalicylate dihydrate (I)

Crystal data

\[ \text{C}_{5}\text{H}_{5}\text{N}_{4}\text{O}^+\cdot\text{C}_{7}\text{H}_{5}\text{O}_{6}\text{S}^-\cdot2\text{H}_{2}\text{O} \]

\[ M_r = 388.32 \]

Monoclinic, \( P2_1/c \)

\[ a = 8.7055 (3) \, \text{Å} \]

\[ b = 25.9927 (13) \, \text{Å} \]

\[ c = 13.6479 (5) \, \text{Å} \]

\[ \beta = 91.864 (3)^\circ \]

\[ V = 3086.6 (2) \, \text{Å}^3 \]

\[ Z = 8 \]

Data collection

Bruker APEXII CCD diffractometer

\( \phi \) and \( \omega \) scans

21075 measured reflections

7079 independent reflections

5905 reflections with \( I > 2\sigma(I) \)

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\[ R(F^2) = 0.032 \]

\[ wR(F^2) = 0.190 \]

\[ S = 1.22 \]

7079 reflections

521 parameters

2 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

\[ w = 1/\left[ \sigma^2(F_c^2) + (0.0167P)^2 + 13.1153P \right] \]

where \( P = (F_c^2 + 2F_s^2)/3 \)

\((\Delta\sigma)_{\text{max}} < 0.001\)

\[ \Delta\rho_{\text{max}} = 0.74 \, \text{e} \, \text{Å}^{-3} \]

\[ \Delta\rho_{\text{min}} = -0.43 \, \text{e} \, \text{Å}^{-3} \]
Extinction correction: SHELXL (Sheldrick, 2015b), \( F_c^{o} = kF_c[1+0.001xF_c^2]^{1/4} \)

**Extinction coefficient:** 0.0013 (3)

**Special details**

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

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

|    | x  | y  | z  | \( U_{eq} \) or \( U_{eq}^* \) | Occ. (<1) |
|----|----|----|----|-----------------------------|-----------|
| O6A| 0.1947 (4) | 0.49617 (15) | 0.5394 (2) | 0.0415 (8) |
| N1A| −0.0096 (4) | 0.49891 (15) | 0.6407 (3) | 0.0312 (8) |
| H1A| −0.072122 | 0.497638 | 0.590694 | 0.037* |
| N3A| 0.0062 (4) | 0.50335 (15) | 0.8125 (3) | 0.0311 (8) |
| N7A| 0.3886 (4) | 0.49922 (15) | 0.7338 (3) | 0.0321 (8) |
| H7A| 0.459823 | 0.497846 | 0.691626 | 0.038* |
| N9A| 0.2725 (4) | 0.50320 (16) | 0.8730 (3) | 0.0331 (9) |
| H9A| 0.257740 | 0.504829 | 0.934876 | 0.040* |
| C2A| −0.0710 (5) | 0.50158 (18) | 0.7299 (3) | 0.0325 (10) |
| H2A| −0.177576 | 0.502197 | 0.732324 | 0.039* |
| C4A| 0.1607 (5) | 0.50245 (17) | 0.8000 (3) | 0.0263 (9) |
| C5A| 0.2318 (4) | 0.50003 (17) | 0.7134 (3) | 0.0261 (9) |
| C6A| 0.1473 (5) | 0.49805 (18) | 0.6231 (3) | 0.0287 (9) |
| C8A| 0.4084 (5) | 0.50097 (19) | 0.8307 (3) | 0.0355 (10) |
| H8A| 0.503178 | 0.500683 | 0.864222 | 0.043* |
| N1B| 0.7702 (5) | 0.25698 (19) | 0.9705 (5) | 0.0620 (15) |
| H1C| 0.689866 | 0.258941 | 1.005445 | 0.074* |
| H1B| 0.716586 | 0.258513 | 1.022312 | 0.074* |
| C2B| 0.7345 (7) | 0.2589 (2) | 0.8774 (6) | 0.0581 (16) |
| H2B| 0.631879 | 0.262454 | 0.857329 | 0.070* |
| N3B| 0.8390 (5) | 0.25613 (17) | 0.8117 (3) | 0.0445 (10) |
| C4B| 0.9779 (5) | 0.25070 (18) | 0.8592 (3) | 0.0331 (10) |
| O6B| 0.9419 (18) | 0.2493 (6) | 1.1190 (8) | 0.054 (3) |
| C5C| 1.0267 (15) | 0.2496 (4) | 0.9577 (8) | 0.035 (3) |
| C6B| 0.9094 (14) | 0.2524 (4) | 1.0314 (7) | 0.035 (3) |
| O6C| 1.0195 (19) | 0.2484 (6) | 1.1206 (10) | 0.062 (3) |
| C5B| 0.9336 (17) | 0.2513 (4) | 0.9552 (8) | 0.040 (3) |
| C6C| 1.0442 (17) | 0.2479 (4) | 1.0326 (7) | 0.048 (4) |
| N7B| 1.1875 (6) | 0.2444 (2) | 0.9780 (5) | 0.0680 (16) |
| H7B| 1.241971 | 0.242864 | 1.031666 | 0.082* |
| H7C| 1.267726 | 0.243141 | 1.016423 | 0.082* |
| C8B| 1.2225 (7) | 0.2428 (2) | 0.8848 (6) | 0.0565 (16) |
| H8B| 1.324295 | 0.239139 | 0.867383 | 0.068* |
| N9B| 1.1141 (5) | 0.24629 (17) | 0.8172 (4) | 0.0479 (11) |
| H9B| 1.127857 | 0.245844 | 0.755081 | 0.058* |
| O3WA| 0.367 (4) | 0.2714 (4) | 1.1689 (17) | 0.063 (5) |

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| Atom  | U1   | U2   | U3   | C1   | C2   |
|-------|------|------|------|------|------|
| O3WB  | 0.296 (4) | 0.2700 (6) | 1.1468 (11) | 0.043 (6) | 0.42 (6) |
| S1A   | 0.40654 (12) | 0.36757 (5) | 0.96698 (8) | 0.0300 (3) |
| O7A   | −0.1915 (4) | 0.38972 (17) | 0.9153 (3) | 0.0473 (10) |
| H7D   | −0.284261 | 0.394380 | 0.919381 | 0.071* |
| O8A   | −0.2547 (4) | 0.38674 (17) | 0.7567 (3) | 0.0489 (10) |
| O9A   | −0.0354 (4) | 0.37374 (16) | 0.6296 (2) | 0.0453 (9) |
| H9D   | −0.123261 | 0.380142 | 0.645875 | 0.068* |
| O10A  | 0.5153 (4) | 0.40963 (14) | 0.9514 (3) | 0.0386 (8) |
| O11A  | 0.4769 (4) | 0.31756 (14) | 0.9629 (3) | 0.0467 (9) |
| O12A  | 0.3208 (4) | 0.37511 (15) | 1.0554 (2) | 0.0429 (9) |
| C9A   | −0.1574 (5) | 0.38438 (18) | 0.8225 (3) | 0.0309 (9) |
| C10A  | 0.0083 (5) | 0.37720 (17) | 0.8061 (3) | 0.0295 (9) |
| C11A  | 0.0590 (5) | 0.37254 (18) | 0.7101 (3) | 0.0322 (10) |
| C12A  | 0.2158 (5) | 0.3673 (2) | 0.6944 (4) | 0.0381 (11) |
| H12A  | 0.250192 | 0.364424 | 0.630852 | 0.046* |
| C13A  | 0.3201 (5) | 0.36641 (19) | 0.7726 (3) | 0.0352 (10) |
| H13A  | 0.424567 | 0.363245 | 0.761467 | 0.042* |
| C14A  | 0.2696 (5) | 0.37022 (17) | 0.8681 (3) | 0.0299 (9) |
| C15A  | 0.1145 (5) | 0.37553 (17) | 0.8842 (3) | 0.0287 (9) |
| H15A  | 0.080905 | 0.378003 | 0.948039 | 0.034* |
| S1B   | 0.56102 (12) | 0.37011 (5) | 0.45829 (9) | 0.0341 (3) |
| O7B   | 1.1577 (4) | 0.38612 (17) | 0.4147 (2) | 0.0443 (9) |
| H7E   | 1.248391 | 0.394576 | 0.418788 | 0.066* |
| O8B   | 1.2092 (4) | 0.37883 (17) | 0.2566 (3) | 0.0497 (10) |
| O9B   | 0.9832 (4) | 0.36982 (17) | 0.1295 (2) | 0.0492 (9) |
| H9E   | 1.073533 | 0.369136 | 0.148463 | 0.074* |
| O10B  | 0.4520 (4) | 0.41174 (15) | 0.4380 (3) | 0.0465 (9) |
| O11B  | 0.4889 (4) | 0.31994 (16) | 0.4549 (3) | 0.0542 (10) |
| O12B  | 0.6496 (4) | 0.37900 (17) | 0.5491 (3) | 0.0509 (10) |
| C9B   | 1.1165 (5) | 0.37948 (18) | 0.3224 (3) | 0.0317 (10) |
| C10B  | 0.9504 (5) | 0.37406 (17) | 0.3041 (3) | 0.0280 (9) |
| C11B  | 0.8925 (5) | 0.37007 (18) | 0.2071 (3) | 0.0333 (10) |
| C12B  | 0.7338 (6) | 0.3667 (2) | 0.1889 (4) | 0.0405 (11) |
| H12B  | 0.695190 | 0.363737 | 0.124781 | 0.049* |
| C13B  | 0.6350 (5) | 0.36764 (19) | 0.2646 (4) | 0.0365 (11) |
| H13B  | 0.529554 | 0.365968 | 0.251642 | 0.044* |
| C14B  | 0.6914 (5) | 0.37113 (17) | 0.3618 (3) | 0.0287 (9) |
| C15B  | 0.8480 (5) | 0.37456 (16) | 0.3811 (3) | 0.0269 (9) |
| H15B  | 0.885372 | 0.377214 | 0.445531 | 0.032* |
| O1W   | 0.6140 (4) | 0.48175 (16) | 0.6162 (2) | 0.0449 (9) |
| H1WA  | 0.605842 | 0.503025 | 0.569001 | 0.067* |
| H1WB  | 0.613001 | 0.452409 | 0.588798 | 0.067* |
| O2W   | 0.2476 (4) | 0.48039 (15) | 1.0649 (3) | 0.0412 (8) |
| O4W   | 0.6239 (8) | 0.2277 (2) | 1.1488 (4) | 0.100 (2) |
| H2WA  | 0.161 (5) | 0.487 (4) | 1.082 (7) | 0.150* |
| H2WB  | 0.270 (12) | 0.4502 (13) | 1.075 (8) | 0.150* |
Atomic displacement parameters (Å²)

|   | $U_{11}$  | $U_{22}$  | $U_{33}$  | $U_{12}$  | $U_{13}$  | $U_{23}$  |
|---|-----------|-----------|-----------|-----------|-----------|-----------|
| O6A | 0.0336 (17) | 0.066 (2) | 0.0244 (16) | −0.0003 (17) | 0.0008 (13) | −0.0022 (16) |
| N1A | 0.0239 (18) | 0.041 (2) | 0.0279 (19) | 0.0016 (16) | −0.0058 (14) | −0.0027 (17) |
| N3A | 0.0270 (18) | 0.040 (2) | 0.0268 (19) | 0.0015 (16) | 0.0048 (15) | 0.0003 (16) |
| N7A | 0.0220 (17) | 0.045 (2) | 0.0296 (19) | 0.0007 (16) | 0.0021 (14) | 0.0066 (17) |
| N9A | 0.0300 (19) | 0.049 (2) | 0.0205 (17) | 0.0018 (17) | −0.0007 (14) | 0.0037 (17) |
| C2A | 0.026 (2) | 0.036 (2) | 0.036 (2) | 0.0014 (18) | 0.0062 (18) | −0.002 (2) |
| C4A | 0.028 (2) | 0.031 (2) | 0.0198 (19) | 0.0007 (17) | 0.0007 (16) | 0.0010 (17) |
| C5A | 0.0191 (18) | 0.036 (2) | 0.023 (2) | 0.0006 (17) | 0.0023 (15) | 0.0005 (18) |
| C6A | 0.030 (2) | 0.034 (2) | 0.023 (2) | −0.0007 (18) | 0.0008 (16) | 0.0000 (18) |
| C8A | 0.025 (2) | 0.047 (3) | 0.034 (2) | −0.003 (2) | 0.0002 (18) | 0.007 (2) |
| N1B | 0.0220 (17) | 0.036 (2) | 0.023 (2) | −0.0007 (18) | 0.0008 (16) | 0.0000 (18) |
| O6B | 0.058 (7) | 0.077 (7) | 0.026 (4) | −0.009 (7) | −0.003 (5) | −0.003 (4) |
| C5C | 0.023 (6) | 0.030 (5) | 0.051 (7) | 0.004 (4) | −0.005 (5) | −0.003 (4) |
| C6B | 0.052 (8) | 0.034 (5) | 0.020 (5) | −0.003 (5) | −0.004 (4) | −0.001 (4) |
| N3B | 0.037 (2) | 0.043 (2) | 0.052 (3) | 0.0017 (19) | −0.008 (2) | 0.003 (2) |
| C4B | 0.038 (2) | 0.034 (2) | 0.032 (2) | 0.0005 (19) | 0.0027 (19) | 0.0034 (19) |
| O6C | 0.058 (7) | 0.077 (7) | 0.026 (4) | −0.009 (7) | −0.003 (5) | −0.003 (4) |
| C5B | 0.023 (6) | 0.030 (5) | 0.051 (7) | 0.004 (4) | −0.005 (5) | −0.003 (4) |
| C6C | 0.094 (12) | 0.032 (5) | 0.018 (5) | −0.013 (6) | 0.020 (5) | 0.004 (4) |
| N7B | 0.041 (3) | 0.047 (3) | 0.115 (5) | −0.003 (2) | −0.009 (3) | 0.002 (3) |
| O7A | 0.0204 (15) | 0.082 (3) | 0.0395 (19) | 0.0058 (18) | 0.0025 (14) | 0.0025 (19) |
| O8A | 0.0254 (16) | 0.081 (3) | 0.040 (2) | 0.0042 (17) | −0.0062 (14) | 0.0015 (19) |
| O9A | 0.0374 (19) | 0.064 (2) | 0.0339 (18) | 0.0009 (19) | −0.0081 (15) | 0.0020 (18) |
| O10A | 0.0233 (15) | 0.048 (2) | 0.045 (2) | −0.0038 (14) | −0.0008 (14) | 0.0051 (16) |
| O11A | 0.0363 (19) | 0.045 (2) | 0.058 (2) | 0.0119 (16) | −0.0007 (17) | 0.0030 (18) |
| S1A | 0.0190 (5) | 0.0406 (6) | 0.0304 (5) | 0.0041 (4) | 0.0014 (4) | 0.0024 (5) |
| O7B | 0.0216 (15) | 0.080 (3) | 0.0314 (17) | −0.0009 (17) | −0.0012 (13) | 0.0034 (18) |
| O8B | 0.0331 (18) | 0.080 (3) | 0.0366 (19) | −0.0020 (18) | 0.0085 (15) | −0.0028 (19) |
| O9B | 0.048 (2) | 0.069 (3) | 0.0303 (18) | 0.000 (2) | 0.0063 (15) | −0.0004 (18) |
| O10B | 0.0261 (17) | 0.053 (2) | 0.060 (2) | 0.0086 (16) | −0.0035 (16) | −0.0015 (19) |
| O11B | 0.041 (2) | 0.052 (2) | 0.070 (3) | −0.0094 (18) | 0.0085 (19) | 0.003 (2) |
### Geometric parameters (Å, °)

|                  |                  |                  |
|------------------|------------------|------------------|
| O6A—C6A         | 1.228 (5)        | S1A—O12A        |
| N1A—C2A         | 1.346 (6)        | S1A—O10A        |
| N1A—C6A         | 1.395 (5)        | S1A—C14A        |
| N1A—H1A         | 0.8600           | O7A—C9A         |
| N3A—C2A         | 1.293 (6)        | O7A—H7D         | 0.8200 |
| N3A—C4A         | 1.362 (5)        | O8A—C9A         | 1.216 (5) |
| N7A—C8A         | 1.330 (6)        | O9A—C11A        | 1.350 (5) |
| N7A—C5A         | 1.384 (5)        | O9A—H9D         | 0.8200 |
| N7A—H7A         | 0.8600           | C9A—C10A        | 1.479 (6) |
| N9A—C8A         | 1.334 (6)        | C10A—C15A       | 1.389 (6) |
| N9A—C4A         | 1.370 (5)        | C10A—C11A       | 1.402 (6) |
| N9A—H9A         | 0.8600           | C11A—C12A       | 1.395 (6) |
| C2A—H2A         | 0.9300           | C12A—C13A       | 1.378 (7) |
| C4A—C5A         | 1.354 (6)        | C12A—H12A       | 0.9300 |
| C5A—C6A         | 1.415 (6)        | C13A—C14A       | 1.394 (6) |
| C8A—H8A         | 0.9300           | C13A—H13A       | 0.9300 |
| N1B—C2B         | 1.300 (9)        | C14A—C15A       | 1.382 (6) |
| N1B—C6B         | 1.452 (12)       | C15A—H15A       | 0.9300 |
| N1B—C5B         | 1.452 (15)       | S1B—O11B        | 1.447 (4) |
| N1B—H1C         | 0.8600           | S1B—O12B        | 1.456 (4) |
| N1B—H1B         | 0.8600           | S1B—O10B        | 1.460 (4) |
| C2B—N3B         | 1.301 (7)        | S1B—C14B        | 1.767 (4) |
| C2B—C5B         | 2.013 (16)       | O7B—C9B         | 1.310 (5) |
| C2B—H2B         | 0.9300           | O7B—H7E         | 0.8200 |
| N3B—C4B         | 1.361 (6)        | O8B—C9B         | 1.227 (5) |
| C4B—N9B         | 1.339 (6)        | O9B—C11B        | 1.342 (6) |
| C4B—C5B         | 1.378 (12)       | O9B—H9E         | 0.8200 |
| C4B—C5C         | 1.396 (12)       | C9B—C10B        | 1.466 (6) |
| O6B—C6B         | 1.222 (15)       | C10B—C15B       | 1.401 (6) |
| C5C—N7B         | 1.425 (13)       | C10B—C11B       | 1.405 (6) |
| C5C—C6B         | 1.458 (18)       | C11B—C12B       | 1.399 (7) |
| C5C—C8B         | 2.009 (15)       | C12B—C13B       | 1.366 (7) |
| O6C—C6C         | 1.227 (16)       | C12B—H12B       | 0.9300 |
| C5B—C6C         | 1.41 (2)         | C13B—C14B       | 1.401 (6) |
C6C—N7B 1.476 (14) C13B—H13B 0.9300
N7B—C8B 1.319 (9) C14B—C15B 1.383 (6)
N7B—H7B 0.8600 C15B—H15B 0.9300
N7B—H7C 0.8600 O1W—H1WA 0.8501
C8B—N9B 1.301 (7) O1W—H1WB 0.8493
C8B—H8B 0.9300 O2W—H2WA 0.821 (10)
N9B—H9B 0.8600 O2W—H2WB 0.820 (10)
S1A—O11A 1.439 (4)

C2A—N1A—C6A 125.2 (4) N9B—C8B—C5C 74.8 (5)
C2A—N1A—H1A 117.4 N7B—C8B—C5C 45.0 (4)
C6A—N1A—H1A 117.4 N9B—C8B—H8B 120.1
C2A—N3A—C4A 112.2 (4) N7B—C8B—H8B 120.1
C8A—N7A—C5A 107.1 (4) C5C—C8B—H8B 165.1
C8A—N7A—H7A 126.4 C8B—N9B—C4B 109.5 (5)
C5A—N7A—H7A 126.4 C8B—N9B—H9B 125.2
C8A—N9A—C4A 107.7 (4) C4B—N9B—H9B 125.2
C8A—N9A—H9A 126.1 O11A—S1A—O12A 112.6 (2)
C4A—N9A—H9A 126.1 O11A—S1A—O10A 113.0 (2)
N3A—C2A—N1A 125.4 (4) O12A—S1A—O10A 111.8 (2)
N3A—C2A—C5B 93.9 (7) O11A—S1A—C14A 106.4 (2)
N3A—C2A—H1C 119.3 C4B—N9A—C14A 106.0 (2)
N1A—C2A—H2A 117.3 C5A—C4A—O11A 126.3 (4)
C5A—C4A—N3A 126.3 (4) C9A—O7A—H7D 109.5
C5A—C4A—N9A 107.5 (4) C11A—O9A—H9D 109.5
N3A—C4A—N9A 126.2 (4) C11A—C10A—C12A 114.2 (4)
C4A—C5A—N7A 107.5 (4) C11A—C10A—O9A 122.1 (4)
C4A—C5A—C6A 121.5 (4) C11A—C10A—O8A 123.6 (4)
N7A—C5A—C6A 131.0 (4) C7A—C9A—C10A 114.2 (4)
O6A—C6A—N1A 121.4 (4) C15A—C10A—C11A 119.5 (4)
O6A—C6A—C5A 129.1 (4) C15A—C10A—C9A 121.1 (4)
N1A—C6A—C5A 109.5 (4) C11A—O11A—O10A 119.4 (4)
N7A—C8A—N9A 110.1 (4) O9A—C11A—C12A 116.8 (4)
N7A—C8A—H8A 125.0 O9A—C11A—C10A 123.8 (4)
N9A—C8A—H8A 125.0 C12A—C11A—C10A 119.4 (4)
C2B—N1B—C6B 137.0 (7) C13A—C12A—C11A 120.5 (4)
C2B—N1B—C5B 93.9 (7) C13A—C12A—H12A 119.8
C2B—N1B—H1C 111.5 C11A—C12A—H12A 119.8
C6B—N1B—H1C 111.5 C12A—C13A—C14A 120.2 (4)
C2B—N1B—H1B 133.1 C12A—C13A—H13A 119.9
C5B—N1B—H1B 133.1 C14A—C13A—H13A 119.9
N1B—C2B—N3B 121.5 (6) C15A—C14A—C13A 119.7 (4)
N1B—C2B—C5B 46.0 (5) C15A—C14A—S1A 121.3 (3)
N3B—C2B—C5B 75.5 (5) C13A—C14A—S1A 119.0 (3)
N1B—C2B—H2B 119.3 C14A—C15A—C10A 120.7 (4)
N3B—C2B—H2B 119.3 C14A—C15A—H15A 119.6
C5B—C2B—H2B 165.3 C10A—C15A—H15A 119.6
C2B—N3B—C4B 107.9 (5) O11B—S1B—O12B 112.8 (3)
N9B—C4B—N3B 126.2 (5) O11B—S1B—O10B 112.5 (2)
| Bond  | Angle (°) | Bond  | Angle (°) | Bond  | Angle (°) |
|-------|-----------|-------|-----------|-------|-----------|
| N9B—C4B—C5B | 133.4 (8) | O12B—S1B—O10B | 111.5 (2) |
| N3B—C4B—C5B | 100.4 (7) | O11B—S1B—C14B | 106.2 (2) |
| N9B—C4B—C5C | 99.5 (7) | O12B—S1B—C14B | 107.3 (2) |
| N3B—C4B—C5C | 134.3 (7) | O10B—S1B—C14B | 106.1 (2) |
| C4B—C5C—N7B | 117.1 (10) | C9B—O7B—H7E | 109.5 |
| C4B—C5C—C6B | 117.7 (10) | C11B—O9B—H9E | 109.5 |
| N7B—C5C—C6B | 125.2 (10) | O8B—C9B—O7B | 122.7 (4) |
| C4B—C5C—C8B | 76.2 (6) | O8B—C9B—C10B | 122.9 (4) |
| N7B—C5C—C8B | 40.9 (5) | O7B—C9B—C10B | 114.4 (4) |
| C6B—C5C—C8B | 166.0 (9) | C15B—C10B—C11B | 119.4 (4) |
| O6B—C6B—N1B | 136.7 (11) | C15B—C10B—C9B | 121.3 (4) |
| O6B—C6B—C5C | 121.8 (11) | C11B—C10B—C9B | 119.3 (4) |
| N1B—C6B—C5C | 101.5 (8) | O9B—C11B—C12B | 117.6 (4) |
| C4B—C5B—C6C | 120.4 (12) | O9B—C11B—C10B | 122.8 (4) |
| C4B—C5B—N1B | 116.4 (10) | C12B—C11B—C10B | 119.6 (4) |
| C6B—C5B—N1B | 123.2 (10) | C13B—C12B—C11B | 120.5 (4) |
| C4B—C5B—C2B | 76.3 (7) | C13B—C12B—H12B | 119.8 |
| C6B—C5B—C2B | 163.3 (10) | C11B—C12B—H12B | 119.8 |
| N1B—C5B—C2B | 40.1 (5) | C12B—C13B—C14B | 120.5 (4) |
| O6C—C6C—C5B | 126.6 (13) | C12B—C13B—H13B | 119.8 |
| O6C—C6C—N7B | 132.2 (13) | C14B—C13B—H13B | 119.8 |
| C5B—C6C—N7B | 101.2 (8) | C15B—C14B—C13B | 119.8 (4) |
| C8B—N7B—C5C | 94.1 (7) | C15B—C14B—S1B | 120.8 (3) |
| C8B—N7B—C6C | 135.6 (7) | C13B—C14B—S1B | 119.4 (3) |
| C8B—N7B—H7B | 133.0 | C14B—C13B—H13B | 120.2 (4) |
| C5C—N7B—H7B | 133.0 | C14B—C13B—H13B | 119.9 |
| C8B—N7B—H7C | 112.2 | C10B—C15B—H15B | 119.9 |
| C6C—N7B—H7C | 112.2 | H1WA—O1W—H1WB | 104.5 |
| N9B—C8B—N7B | 119.8 (6) | H2WA—O2W—H2WB | 112 (10) |
| C4A—N3A—C2A—N1A | 0.3 (7) | C6B—C5C—N7B—C8B | 178.1 (9) |
| C6A—N1A—C2A—N3A | −0.5 (8) | O6C—C6C—N7B—C8B | 178.6 (13) |
| C2A—N3A—C4A—C5A | 0.0 (7) | C5B—C6C—N7B—C8B | −2.0 (12) |
| C2A—N3A—C4A—N9A | −179.1 (4) | C5C—N7B—C8B—N9B | 0.3 (8) |
| C8A—N9A—C4A—C5A | −0.4 (5) | C6C—N7B—C8B—N9B | 2.6 (12) |
| C8A—N9A—C4A—N3A | 179.0 (4) | N7B—C8B—N9B—C4B | −0.6 (8) |
| N3A—C4A—C5A—N7A | −179.2 (4) | C5C—C8B—N9B—C4B | −0.4 (5) |
| N9A—C4A—C5A—N7A | 0.1 (5) | N3B—C4B—N9B—C8B | 178.7 (5) |
| N3A—C4A—C5A—C6A | −0.1 (7) | C5B—C4B—N9B—C8B | −1.3 (10) |
| N9A—C4A—C5A—C6A | 179.2 (4) | C5C—C4B—N9B—C8B | 0.5 (7) |
| C8A—N7A—C5A—C4A | 0.2 (5) | O8A—C9A—C10A—C15A | 179.1 (5) |
| C8A—N7A—C5A—C6A | −178.8 (5) | O7A—C9A—C10A—C15A | 1.1 (6) |
| C2A—N1A—C6A—O6A | −179.1 (5) | O8A—C9A—C10A—C11A | −0.3 (7) |
| C2A—N1A—C6A—C5A | 0.4 (6) | O7A—C9A—C10A—C11A | −178.2 (4) |
| C4A—C5A—C6A—O6A | 179.4 (5) | C15A—C10A—C11A—O9A | −180.0 (4) |
| N7A—C5A—C6A—O6A | −1.7 (9) | C9A—C10A—C11A—O9A | −0.6 (7) |
| C4A—C5A—C6A—N1A | −0.1 (6) | C15A—C10A—C11A—C12A | −1.3 (7) |
| N7A—C5A—C6A—N1A | 178.7 (4) | C9A—C10A—C11A—C12A | 178.1 (4) |
The table below lists the hydrogen-bond geometry (Å, °) for the molecules in the crystal structure:

| D     | H   | A   | D     | H   | A   | D     | H   | A   |
|-------|-----|-----|-------|-----|-----|-------|-----|-----|
| N7B-H7B···O3W i | 0.86 | 2.26 | 3.08  | 158 |
| O7A-H7D···O10A i | 0.82 | 1.86 | 2.677 | 170 |
| O7B-H7E···O10B i | 0.82 | 1.84 | 2.655 | 175 |
| O9A-H9D···O12A i | 0.82 | 2.34 | 2.924 | 128 |
| O9B-H9E···O12A i | 0.82 | 2.54 | 3.143 | 131 |
| O1W-H1W···O6A iv | 0.85 | 2.31 | 2.801 | 117 |

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O1W—H1WA···O10Biv 0.85 2.28 2.917 132
N9B—H9B···O6Biv 0.86 2.42 3.044 130
N9B—H9B···O3Wvi 0.86 2.47 3.07 128
N1A—H1A···O6Wvi 0.86 2.05 2.898 170
N1B—H1C···O4W 0.86 2.22 2.890 135
N1B—H1C···O11A 0.86 2.45 2.998 122
O1W—H1WB···O12B 0.85 2.01 2.844 169
O2W—H2WB···O12A 0.82 2.03 2.815 160
N7A—H7A···O1W 0.86 1.77 2.615 168
N9A—H9A···O2W 0.86 1.89 2.697 157
C2A—H2A···O1W 0.93 2.43 3.149 134
C2B—H2B···O11A 0.93 2.46 2.974 114
C8A—H8A···O2Wvii 0.93 2.40 3.310 167
C15B—H15B···O9A 0.93 2.59 3.510 172

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

6-Oxo-1,9-dihydropurin-7-ium perchlorate monohydrate (II)

Crystal data

C₅H₅N₄O⁺·ClO₄⁻·H₂O  \hspace{10pt} F(000) = 520
Mᵣ = 254.60  \hspace{10pt} Dₐ = 1.833 Mg m⁻³
Monoclinic, P₂₁/c  \hspace{10pt} Mo Kα radiation, λ = 0.71073 Å
a = 5.0307 (6) Å  \hspace{10pt} Cell parameters from 2752 reflections
b = 20.386 (2) Å  \hspace{10pt} θ = 2.0–30.3°
c = 9.0181 (10) Å  \hspace{10pt} µ = 0.44 mm⁻¹
β = 94.233 (2)°  \hspace{10pt} T = 296 K
V = 922.33 (18) Å³  \hspace{10pt} Plate, colourless
Z = 4  \hspace{10pt} 0.45 × 0.02 × 0.003 mm

Data collection

Bruker APEXII CCD  \hspace{10pt} 2752 independent reflections
φ and ω scans  \hspace{10pt} 2370 reflections with I > 2σ(I)
Absorption correction: multi-scan  \hspace{10pt} R_m = 0.025
(SADABS; Bruker, 2016)  \hspace{10pt} θ_max = 30.3°, θ_min = 2.0°
T_min = 0.957, T_max = 1.000  \hspace{10pt} h = −7→7
16360 measured reflections  \hspace{10pt} k = −28→28
l = −12→12

Refinement

Refinement on F²  \hspace{10pt} Hydrogen site location: mixed
Least-squares matrix: full  \hspace{10pt} H atoms treated by a mixture of independent
R[F² > 2σ(F²)] = 0.038 and constrained refinement
wR(F²) = 0.111  \hspace{10pt} w = 1/[σ²(Fo²) + (0.0576P)² + 0.3728P]
S = 1.05  \hspace{10pt} \text{where } P = (Fo² + 2Fc²)/3
2752 reflections  \hspace{10pt} (Δ/σ)_max < 0.001
165 parameters  \hspace{10pt} Δρ_max = 0.37 e Å⁻³
3 restraints  \hspace{10pt} Δρ_min = −0.29 e Å⁻³
**Special details**

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

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

|    | x            | y            | z            | U\(_{eq}^*/U_{eq}\) |
|----|--------------|--------------|--------------|----------------------|
| Cl1| 1.32826 (7)  | 0.47612 (2)  | 0.23036 (5)  | 0.03437 (13)         |
| O6 | 1.1975 (2)   | 0.28099 (6)  | 0.28926 (13) | 0.0395 (3)           |
| O2 | 1.2250 (4)   | 0.53538 (8)  | 0.1690 (2)   | 0.0699 (5)           |
| O3 | 1.6141 (3)   | 0.47706 (8)  | 0.2342 (2)   | 0.0627 (4)           |
| O4 | 1.2288 (3)   | 0.42071 (7)  | 0.14577 (18) | 0.0571 (4)           |
| O5 | 1.2456 (3)   | 0.46991 (8)  | 0.37946 (17) | 0.0598 (4)           |
| N1 | 0.9409 (3)   | 0.35230 (6)  | 0.41720 (15) | 0.0334 (3)           |
| H1 | 1.018 (4)    | 0.3843 (12)  | 0.387 (3)    | 0.049 (6)*           |
| N7 | 0.8829 (2)   | 0.17434 (6)  | 0.43852 (14) | 0.0281 (3)           |
| H7 | 0.992 (5)    | 0.1492 (12)  | 0.387 (3)    | 0.060 (7)*           |
| N9 | 0.5789 (2)   | 0.20049 (6)  | 0.58838 (14) | 0.0284 (3)           |
| H9 | 0.462 (5)    | 0.1993 (11)  | 0.650 (3)    | 0.046 (6)*           |
| C2 | 0.7509 (3)   | 0.36488 (7)  | 0.51528 (18) | 0.0342 (3)           |
| H2 | 0.716229     | 0.408591     | 0.533422     | 0.041*               |
| N3 | 0.6127 (3)   | 0.32029 (6)  | 0.57756 (14) | 0.0312 (3)           |
| C4 | 0.6819 (3)   | 0.25866 (7)  | 0.53983 (15) | 0.0246 (3)           |
| C5 | 0.8730 (3)   | 0.24168 (7)  | 0.44524 (15) | 0.0243 (3)           |
| C6 | 1.0223 (3)   | 0.29051 (7)  | 0.37512 (16) | 0.0272 (3)           |
| C8 | 0.7053 (3)   | 0.15077 (7)  | 0.52526 (17) | 0.0311 (3)           |
| H8 | 0.672604     | 0.106462     | 0.540420     | 0.037*               |
| O1W| 0.2214 (3)   | 0.38054 (6)  | 0.76425 (18) | 0.0511 (4)           |
| H1W| 0.252 (5)    | 0.4216 (5)   | 0.765 (3)    | 0.066 (7)*           |
| H2W| 0.344 (4)    | 0.3627 (10)  | 0.718 (3)    | 0.081 (9)*           |

**Atomic displacement parameters (Å²)**

|    | U\(_{11}\)   | U\(_{22}\)   | U\(_{33}\)   | U\(_{12}\)   | U\(_{13}\)   | U\(_{23}\)   |
|----|--------------|--------------|--------------|--------------|--------------|--------------|
| Cl1| 0.0347 (2)   | 0.02074 (17) | 0.0495 (2)   | −0.00172 (12)| 0.01561 (16)| −0.00075 (13)|
| O6 | 0.0398 (6)   | 0.0391 (6)   | 0.0428 (6)   | −0.0012 (5)  | 0.0248 (5)   | 0.0054 (5)   |
| O2 | 0.0807 (12)  | 0.0369 (7)   | 0.0952 (13)  | 0.0172 (7)   | 0.0277 (10)  | 0.0222 (8)   |
| O3 | 0.0351 (7)   | 0.0545 (9)   | 0.1004 (13)  | −0.0050 (6)  | 0.0182 (7)   | −0.0109 (8)  |
| O4 | 0.0551 (8)   | 0.0419 (8)   | 0.0757 (10)  | −0.0102 (6)  | 0.0147 (7)   | −0.0226 (7)  |
| O5 | 0.0734 (10)  | 0.0587 (9)   | 0.0503 (8)   | −0.0211 (8)  | 0.0244 (7)   | −0.0040 (6)  |
| N1 | 0.0377 (7)   | 0.0255 (6)   | 0.0388 (7)   | −0.0043 (5)  | 0.0146 (5)   | 0.0030 (5)   |
| N7 | 0.0305 (6)   | 0.0236 (5)   | 0.0317 (6)   | 0.0031 (4)   | 0.0130 (5)   | 0.0006 (4)   |
| N9 | 0.0282 (6)   | 0.0285 (6)   | 0.0304 (6)   | −0.0020 (4)  | 0.0141 (5)   | 0.0016 (4)   |
| C2 | 0.0410 (8)   | 0.0242 (6)   | 0.0385 (8)   | 0.0007 (6)   | 0.0120 (6)   | −0.0031 (6)  |
| N3 | 0.0331 (6)   | 0.0269 (6)   | 0.0351 (6)   | 0.0024 (5)   | 0.0132 (5)   | −0.0026 (5)  |
| C4 | 0.0233 (6)   | 0.0261 (6)   | 0.0254 (6)   | −0.0003 (5)  | 0.0075 (5)   | 0.0005 (5)   |

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### Geometric parameters (Å, °)

|        | Cl1—O2     | Cl1—O4     | Cl1—O3     | Cl1—O5     | O6—C6     | N1—C2     | N1—C6     | N1—H1     | O6—C6     | N1—C2     | N1—H1     | O6—C6     | N1—C2     | N1—H1     | O6—C6     | N1—C2     | N1—H1     | O6—C6     |
|--------|------------|------------|------------|------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
|        | 1.4116 (15)| 1.4324 (13)| 1.4359 (15)| 1.4421 (15)| 1.2307 (17)| 1.357 (2)  | 1.3860 (19)| 0.82 (2)  | 1.3204 (18)| 1.3752 (18)| 0.90 (3)  | 111.24 (12)| 109.68 (11)| 109.46 (9) | 108.50 (11)| 108.29 (9) | 109.63 (11)| 125.56 (13)| 115.8 (16) |
|        | N9—C8     | N9—C4     | N9—H9     | C2—N3     | C2—H2     | N3—C4     | C4—C5     | C5—C6     | C8—N7     | C5—N7     | C8—H8     | O1W—H1W   | O1W—H2W   | C2—N1     | C2—N1     | N1—C6     | C6—N1     | C6—N1     |
|        | 1.3452 (19)| 1.3784 (17)| 0.84 (2)  | 1.309 (2) | 0.9300    | 1.3539 (18)| 1.3758 (17)| 1.4229 (18)| 0.9300    | 0.852 (9) | 0.850 (9) | 117.4     | 117.4     | 112.14 (12)| 126.43 (12)| 127.50 (12)| 106.07 (12)| 107.91 (11)| 131.06 (12)|
|        | 118.5 (16) | 121.02 (13)| 123.73 (13)| 126.53 (14)| 109.75 (12)| 109.76 (12)| 125.1     | 125.1     | 106.9 (14) |
|        | N3—C2—H2  | N1—C2—H2  | O6—C6—N1 | O6—C6—C5 | N1—C6—C5 | N7—C5—C4 | N7—C5—C6 | N7—C8—H8 | H1W—O1W   |
|        | −1.1 (3)   | 0.5 (2)    | C2—N1—C6 | C2—N1—C6 | N1—C6—C5 | N7—C5—C6 | N7—C8—C9 | N9—C8—C9 | O1W—H1W   |
|        | N1—C2—N3  | N9—C4—C5 | C2—N1—C6 | N1—C6—C5 | N7—C5—C6 | N7—C8—N9 | N9—C8—N9 | C4—N9—C8 | O1W—H2W   |
|        | 0.0 (2)    | 0.0 (2)    | 179.42 (15)| 179.25 (14)| 0.24 (16) | 0.01 (17) | 179.31 (15)| 179.34 (14)| 0.15 (18) | 0.24 (18) |
|        | 179.54 (13)| −199.27 (16)| 1.0 (2)    | 0.6 (3)   | 179.83 (15)| 179.65 (14)| 0.4 (2)   | 0.15 (18) | −0.24 (18) |
Hydrogen-bond geometry (Å, °)

| D—H···A             | D—H | H···A | D···A  | D—H···A |
|---------------------|-----|-------|--------|---------|
| N1—H1···O4          | 0.82| 2.60  | 3.249  | 138     |
| N1—H1···O5          | 0.82| 2.09  | 2.879  | 162     |
| N7—H7···O2^i        | 0.91| 2.60  | 3.031  | 110.2   |
| N7—H7···O1^ii       | 0.91| 1.76  | 2.6489 | 165     |
| N9—H9···O6^iii      | 0.84| 1.93  | 2.7602 | 166     |
| O1W—H1W···O3^iv     | 0.85| 2.17  | 3.018  | 172     |
| O1W—H2W···N3        | 0.85| 2.11  | 2.951  | 172     |
| C8—H8···O2^i        | 0.93| 2.47  | 2.970  | 114     |
| C8—H8···O3^iii      | 0.93| 2.47  | 3.268  | 144     |
| C8—H8···O4^iii      | 0.93| 2.55  | 3.072  | 116     |

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