Crystal structure and hydrogen bonding in the water-stabilized proton-transfer salt brucinium 4-aminophenylarsonate tetrahydrate

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In the structure of the brucinium salt of 4-aminophenylarsonic acid (p-arsanilic acid), systematically 2,3-dimethoxy-10-oxostyrchnidinium 4-aminophenylarsonate tetrahydrate, \(\text{C}_{23}\text{H}_{27}\text{N}_2\text{O}_4\)\[\text{As(C}_6\text{H}_7\text{N})\text{O}_2\text{(OH)}\]•4\(\text{H}_2\text{O}\), the brucinium cations form the characteristic undulating and overlapping head-to-tail layered brucine substructures packed along [010]. The arsanilate anions and the water molecules of solvation are accommodated between the layers and are linked to them through a primary cation N—H⋯O(anion) hydrogen bond, as well as through water O—H⋯O hydrogen bonds to brucinium and arsanilate ions as well as bridging water O-atom acceptors, giving an overall three-dimensional network structure.

1. Chemical context

The \textit{Strychnos} alkaloid base brucine, (2,3-dimethoxystrychnidin-10-one; BRU) has been extensively employed as a resolving agent for chiral organic compounds (Wilen, 1972). With chiral acids, the separation is achieved through proton-transfer to N19 of the styrchnidine cage (\(pK_{a2} = 11.7\); O’Neil, 2001), followed by separation of the resultant crystalline salt products by fractional crystallization. Similar effects are achieved with the essentially identical \textit{Strychnos} alkaloid strychnine but separation efficiency favours brucine. This is probably because of the formation in the crystal of characteristic brucinium host substructures comprising head-to-tail undulating layers of brucine molecules or cations which accommodate selectively the hydrogen-bonded guest molecules in the crystal structure. A characteristic of the substructure is the repeat interval in the layer of ca 12.3 Å along a 21 screw axis in the crystal, which is reflected in the unit-cell dimension, with brucine being predominantly in the monoclinic space group \(P2_1\) or the orthorhombic space group \(P2_12_12_1\) (Smith, Wermuth & White, 2006; Smith, Wermuth, Young & White, 2006).
This example of molecular recognition was described in the early structure determinations of brucinium benzoyl-dalaninate (Gould & Walkinshaw, 1984) and in the structures of the pseudopolymorphic brucinium solvates, brucine–MeOH (1:1) and brucine–EtOH–water (1/1/2) (Glover et al., 1985). The guest molecules are accommodated interstitially within the layers and are commonly accompanied by compatible polar solvent molecules, usually generating high-dimensional hydrogen-bonded crystal structures.

Currently, a large number of structures of brucinium compounds with chiral organic molecules, including both acids and non-acids are known, but in addition those with achiral compounds also feature. Of interest to us have been the structures of brucinium proton-transfer salts with largely simple organic acids, prepared under aqueous alcoholic conditions, the crystalline products being stabilized by solvent molecules. Water-stabilized achiral carboxylate examples include BRU⁺ hydrogen fumarate⁻·1,5H₂O (Dijksma, Gould, Parsons & Walkinshaw, 1998), BRU⁺ dihydrogen citrate⁻·3H₂O (Smith, Wermuth & White, 2005) and BRU⁺ benzoate⁻·3H₂O (Białońska & Ciunik, 2006b).

Other organic acids besides carboxylates may be included among the set but fewer structural examples are known, e.g. sulfonates (BRU⁺ toluene-4-sulfonate⁻·3H₂O; Smith, Wermuth, Healy et al., 2005). However, no brucinium arsonate structures are known, so that the reaction of brucine with 4-aminophenylarsonic acid (p-arsanilic acid) in 2-propanol/water was carried out, resulting in the formation of the crystalline hydrated title salt, C₂₀H₂₁N₂AsO₄⁻·C₆H₄AsNO₃⁻·4H₂O, and the structure is reported herein. The acid has biological significance as an anti-helminth in veterinary applications (Thomas, 1905; Steverding, 2010) and as a monohydrated sodium salt (atroxyl) which had early usage as an anti-syphilitic (Ehrlich & Bertheim, 1907; Bosch & Rosich, 2008). Simple p-arsanilate salt structures are not common in the Cambridge Structural Database (Groom et al., 2016), with only the NH₄⁺ and K⁺ salts (Smith & Wermuth, 2014) and the guanidinium salts (Smith & Wermuth, 2010; Latham et al., 2011) being known.

2. Structural commentary
The asymmetric unit of the title salt comprises a brucinium cation, a p-arsanilate anion A and four water molecules of solvation, (O1W–O4W), all inter-associated through hydrogen bonds (Fig. 1). Protonation has occurred as expected at N₁⁹ of the brucine cage, the invoked Peerdeman (1956) absolute overall Cahn–Ingold stereochemistry of the cation as C₇(−Ⅹ)₁₅C₂₀—H₂₀₁

Figure 1
Molecular configuration and atom-numbering scheme for the brucinium cation, p-arsanilate anion A and the four water molecules of solvation in the asymmetric unit of the title salt. Inter-species hydrogen bonds are shown as dashed lines. Non-H atoms are shown as 40% probability displacement ellipsoids.

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

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|-------|---------|
| N₁⁹—H₁¹⁹—O₁₂A | 0.91 (4) | 1.72 (4) | 2.610 (3) | 168 (4) |
| N₄₄—H₄₄A—O₄₄W | 0.89 (3) | 2.46 (4) | 3.291 (5) | 155 (4) |
| N₄₄—H₄₄A—O₃₃W | 0.90 (3) | 2.25 (3) | 3.137 (6) | 169 (4) |
| O₁₃A—H₁₃₆A—O₁₁₄W | 0.90 (4) | 1.67 (4) | 2.546 (3) | 165 (4) |
| O₁₆W—H₁₆₁W—O₂₅ | 0.90 (4) | 1.95 (4) | 2.843 (4) | 175 (3) |
| O₁₆W—H₁₆₂W—O₂₅W | 0.90 (3) | 1.87 (4) | 2.760 (5) | 168 (4) |
| O₂₆W—H₂₆₁W—O₁₂A | 0.90 (3) | 2.11 (3) | 2.945 (4) | 153 (4) |
| O₂₆W—H₂₆₂W—O₁₁₄W | 0.89 (3) | 2.07 (4) | 2.915 (4) | 158 (5) |
| O₃₆W—H₃₆₁W—O₂₅ | 0.91 (4) | 2.06 (4) | 2.922 (4) | 159 (3) |
| O₃₆W—H₃₆₂W—O₄₄W | 0.91 (3) | 1.91 (3) | 2.791 (4) | 164 (3) |
| O₄₄W—H₄₄₁W—O₁₁₄W | 0.90 (4) | 1.88 (4) | 2.770 (5) | 172 (5) |
| O₄₄W—H₄₄₂W—O₁₂A | 0.89 (4) | 1.91 (4) | 2.802 (4) | 174 (5) |
| C₁₄—H₁₄—O₃₃W | 1.00 | 2.52 | 3.363 (4) | 142 |
| C₁₅—H₁₅₁W—O₁₁₄W | 0.99 | 2.60 | 3.561 (4) | 165 |
| C₁₈—H₁₈₁W—O₂₅ | 0.99 | 2.58 | 3.422 (5) | 143 |
| C₂₀—H₂₀₁—O₁₁₄W | 0.99 | 2.41 | 3.388 (4) | 170 |
| C₂₀—H₂₀₂—O₁₃₄W | 0.99 | 2.43 | 3.229 (4) | 137 |

Symmetry codes: (i) x−½, y+½, z−½; (ii) x+½, y+½, z+½; (iii) −x, y−½, z−½; (iv) x+½, y+½, z+½; (v) −x+½, y−½, z+½; (vi) x+½, −y+½, −z+½; (vii) −x+1, y, −z+½; (viii) −x+1, y, −z−½.
3. Supramolecular features

The brucinium cations form into the previously described undulating sheet–host substructures which are considered to be the reason for the molecular recognition peculiar to brucine (Gould & Walkinshaw, 1984; Gould et al., 1985; Dijksma, Gould, Parsons & Walkinshaw, 1998; Dijksma, Gould, Parsons, Taylor & Walkinshaw, 1998; Oshikawa et al., 2002; Białońska & Ciunik, 2004). In the title salt, these substructures extend along the $b$-axis direction, with the previously described $2_1$ propagation of the brucinium cations along the ca 12.3 Å axis (Fig. 2). The $p$-arsanilate anions and the water molecules occupy the interstitial spaces in the structure. The protonated N19 atom of the cation gives a single hydrogen-bonding interaction with a $p$-arsanilate oxygen acceptor (O12A) while two of the solvent water molecules (O1W and O3W) form hydrogen bonds with the...

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**Figure 2**
The undulating brucinium sheet substructures in the unit cell of the title salt, less the inter-sheet anion and water molecules, viewed down $a$. All H atoms except that of the protonated N19 atom have also been removed.

**Figure 3**
A perspective view of the packing in the unit cell, viewed along the approximate $a$-axial direction, showing the associated anions and the water molecules in the interstitial regions of the brucinium layered substructures, with hydrogen-bonding interactions shown as dashed lines.
carbonyl O25 atom of the brucinium cation (Table 1). Within the inter-sheet channels, the p-arsanilate anions are linked head-to-head through an O13A—H···O11A bond while both H atoms of the amine group form hydrogen bonds with water molecules O3W and O4W. The water molecules O2W and O4A are further linked to the p-arsanilate O-atom O12A with O2W also linked to O11A. Water molecules O3W and O4W give inter-water hydrogen bonds and together with a number of inter-molecular C—H···O interactions (Table 1) result in an overall three-dimensional network structure (Fig. 3).

4. Database survey

Interstitial water molecules are present in the structures of the brucine pseudo-polymorphic structures, e.g. the common tetrahydrate form and the 5.2 hydrate (Smith et al., 2006a) and the dihydrate (Smith et al., 2007), as well as the mixed solvates BRU–EtOH–H2O (1/1/2) (Glover et al., 1985) and BRU–i-PrOH–H2O (1/1/2) (Białońska & Ciunik, 2004). A large number of water-stabilized brucinium salts of acids are known: with the inorganic sulfate (BRU)2SO4(7H2O) (Białońska & Ciunik, 2005) and most commonly with aromatic carboxylates, e.g. the benzoate (a trihydrate; Białońska & Ciunik, 2006b); the 4-nitrobenzoate (a dihydrate; Białońska & Ciunik, 2007); the 3,5-dinitrobenzoate (a dihydrate; Białońska & Ciunik, 2006a); the 3,5-dinitrosalicylate (a monohydrate; Smith et al., 2006a); the phthalate (a monohydrate; Krishnan, Gayathri, Sivakumar, Gunasekaran & Anbalagen, 2013); the hydrogen isophthalate (a trihydrate; Smith, Wermuth, Young & White, 2006); the hydrogen 3-nitrophthalate (a dihydrate; Smith, Wermuth, Young & Healy, 2005) and the picraminobenzoate (a monohydrate; Smith & Wermuth, 2011).

Aliphatic carboxylate examples are: with hydrogen oxalate (a dihydrate; Krishnan, Gayathri, Sivakumar, Chakkaravathi & Anbalagen, 2013); with hydrogen fumarate (a sesquihydrate; Dijksma, Gould, Parsons & Walkinshaw, 1998); with hydrogen (S)-malate (a pentahydrate; Smith, Wermuth & White, 2006); with dihydrogen citrate (a trihydrate; Smith, Wermuth & White, 2005); with L-glycerate (a 4.75 hydrate; Białońska et al., 2005) and with hydrogen cis-cyclohexane-1,2-dicarboxylate (a dihydrate; Smith et al., 2012). Some sulfonate salts are also known, e.g. with toluene-4-sulfonate (a trihydrate; Smith, Wermuth, Healy et al., 2005); with 3-carboxy-4-hydroxybenzenesulfonate (a pentahydrate; Smith et al., 2006b) and with biphenyl-4,4’-disulfonate (a hexahydrate; Smith et al., 2010).

5. Synthesis and crystallization

The title compound was synthesized by heating together under reflux for 10 min, 1 mmol quantities of brucine tetrahydrate and 4-aminophenylarsonic acid in 50 mL of 80% 2-propanol/water. After concentration to ca 30 mL, partial room-temperature evaporation of the hot-filtered solution gave thin colourless crystal plates of the title compound from which a specimen was cleaved for the X-ray analysis.

Table 2

| Property                          | Value                  |
|----------------------------------|------------------------|
| Crystal data                     | (C9H25N2O4)[As(C6H7NO3(OH)]4H2O |
| Chemical formula                 | (C9H25N2O4)[As(C6H7NO3(OH)]4H2O |
| M<sub>w</sub>                     | 683.58                 |
| Crystal system, space group      | Orthorhombic, P2₁2₁2₁  |
| Temperature (K)                  | 298.15                 |
| a, b, c (Å)                      | 7.6553 (3), 12.3238 (5), 31.960 (2) |
| V (Å³)                           | 3015.2 (3)             |
| Radiation type                   | Mo Kα                 |
| μ (mm⁻¹)                         | 1.19                  |
| Crystal size (mm)                | 0.36 × 0.34 × 0.10     |

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms potentially involved in hydrogen-bonding interactions were located by difference methods but their positional parameters were constrained in the refinement with N—H and O—H = 0.90 Å, and with Uiso(H) = 1.2Ueq(N) or 1.5Ueq(O). Other H atoms were included in the refinement at calculated positions [C—H( aromatic) = 0.95 Å and C—H (aliphatic) = 0.97–1.00 Å] and treated as riding with H atoms treated by a mixture of independent and constrained refinement

Acknowledgements

The authors acknowledge support from the Science and Engineering Faculty, Queensland University of Technology.

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Crystal structure and hydrogen bonding in the water-stabilized proton-transfer salt brucinium 4-aminophenylarsonate tetrahydrate

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Computing details
Data collection: CrysAlis PRO (Rigaku OD, 2015); cell refinement: CrysAlis PRO (Rigaku OD, 2015); data reduction: CrysAlis PRO (Rigaku OD, 2015); program(s) used to solve structure: SIR92 (Altomare et al., 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 2012); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON (Spek, 2009).

2,3-Dimethoxy-10-oxostrychnidinium 4-aminophenylarsonate tetrahydrate

Crystal data

\[
(C_{23}H_{27}N_2O_4)[\text{As}(C_6H_7N)O_2(OH)] \cdot 4\text{H}_2\text{O}
\]

\[
M_r = 683.58
\]

Orthorhombic, \(P\overline{2}_12_12_1\)

Hall symbol: \(P\ 2\ ac\ 2ab\)

\(a = 7.6553\ (3)\ \text{Å}\)

\(b = 12.3238\ (5)\ \text{Å}\)

\(c = 31.960\ (2)\ \text{Å}\)

\(V = 3015.2\ (3)\ \text{Å}^3\)

\(Z = 4\)

\(F(000) = 1432\)

\(D_\text{c} = 1.506\ \text{Mg m}^{-3}\)

Mo \(K\alpha\) radiation, \(\lambda = 0.71073\ \text{Å}\)

Cell parameters from 2822 reflections

\(\theta = 3.4-27.9\degree\)

\(\mu = 1.19\ \text{mm}^{-1}\)

\(T = 200\ \text{K}\)

Plate, colourless

\(0.36 \times 0.34 \times 0.10\ \text{mm}\)

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer

Radiation source: Enhance (Mo) X-ray source

Graphite monochromator

Detector resolution: 16.077 pixels mm\(^{-1}\)

\(\omega\) scans

Absorption correction: multi-scan

\(\text{(CrysAlis PRO; Rigaku OD, 2015)}\)

\(T\min = 0.811, \ T\max = 0.980\)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.048\)

\(wR(F^2) = 0.096\)

\(S = 1.05\)

6980 reflections

433 parameters

14 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement
$w = 1/\sigma^2(F_o^2) + (0.0414P^2 + 0.2011P)$
where $P = (F_o^2 + 2F_c^2)/3$

$\Delta/\sigma_{max} = 0.001$  
$\Delta\rho_{max} = 0.55 \text{ e} \AA^{-3}$

$\Delta\rho_{min} = -0.46 \text{ e} \AA^{-3}$

Absolute structure: Flack (1983), 3672 Friedel pairs

Absolute structure parameter: $-0.005 (9)$

**Special details**

**Geometry.** Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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

| Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\AA^2$) |
|---|---|---|---|---|
| O2 | 0.2664 (3) | 0.56076 (19) | 0.24172 (7) | 0.0288 (8) |
| O3 | 0.2324 (3) | 0.44912 (19) | 0.17363 (7) | 0.0251 (7) |
| O24 | 0.2010 (3) | $-0.13571 (17)$ | 0.32202 (7) | 0.0224 (7) |
| O25 | 0.2193 (4) | 0.0496 (2) | 0.19539 (7) | 0.0336 (9) |
| N9 | 0.1591 (3) | 0.1193 (2) | 0.25929 (8) | 0.0192 (7) |
| N19 | 0.1326 (4) | 0.2044 (2) | 0.39973 (8) | 0.0220 (8) |
| C1 | 0.2107 (4) | 0.4025 (3) | 0.28549 (10) | 0.0220 (10) |
| C2 | 0.2316 (4) | 0.4525 (3) | 0.24709 (10) | 0.0206 (10) |
| C3 | 0.2176 (4) | 0.3914 (3) | 0.21006 (10) | 0.0200 (9) |
| C4 | 0.1912 (4) | 0.2806 (2) | 0.21125 (9) | 0.0192 (9) |
| C5 | 0.1762 (5) | 0.2319 (2) | 0.25023 (10) | 0.0186 (9) |
| C6 | 0.1822 (5) | 0.2909 (3) | 0.28690 (9) | 0.0200 (9) |
| C7 | 0.1402 (4) | 0.2196 (3) | 0.32382 (10) | 0.0194 (9) |
| C8 | 0.1639 (4) | 0.1035 (3) | 0.30544 (9) | 0.0178 (9) |
| C10 | 0.2084 (5) | 0.0380 (3) | 0.23349 (10) | 0.0224 (10) |
| C11 | 0.2482 (5) | $-0.0701 (3)$ | 0.25362 (11) | 0.0244 (11) |
| C12 | 0.3195 (5) | $-0.0703 (3)$ | 0.29876 (10) | 0.0216 (10) |
| C13 | 0.3369 (4) | 0.0468 (3) | 0.31477 (9) | 0.0173 (9) |
| C14 | 0.3946 (4) | 0.0634 (3) | 0.36027 (10) | 0.0208 (10) |
| C15 | 0.4243 (4) | 0.1858 (3) | 0.36540 (11) | 0.0217 (10) |
| C16 | 0.2486 (5) | 0.2415 (3) | 0.36351 (10) | 0.0215 (10) |
| C17 | $-0.0479 (4)$ | 0.2361 (3) | 0.33974 (11) | 0.0236 (11) |
| C18 | $-0.0461 (4)$ | 0.1812 (3) | 0.38190 (10) | 0.0236 (10) |
| C20 | 0.2066 (5) | 0.1088 (3) | 0.42293 (9) | 0.0234 (10) |
| C21 | 0.2646 (4) | 0.0242 (3) | 0.39246 (10) | 0.0221 (10) |
| C22 | 0.2076 (5) | $-0.0761 (3)$ | 0.39424 (10) | 0.0235 (10) |
| C23 | 0.2581 (5) | $-0.1618 (3)$ | 0.36323 (11) | 0.0269 (11) |
| C25 | 0.2845 (6) | 0.6248 (3) | 0.27850 (12) | 0.0400 (14) |
| C26 | 0.2222 (4) | 0.3880 (3) | 0.13581 (10) | 0.0263 (10) |
| As1A | 0.18853 (4) | 0.38087 (2) | 0.50015 (1) | 0.0194 (1) |
| O11A | 0.0706 (3) | 0.2967 (2) | 0.52906 (7) | 0.0288 (8) |
| O12A | 0.1351 (3) | 0.37219 (19) | 0.44956 (7) | 0.0256 (7) |
| Atom | x    | y    | z    | Ueq |
|------|------|------|------|-----|
| O13A | 0.4046 (3) | 0.3544 (2) | 0.50798 (9) | 0.0361 (9) |
| N4A  | 0.1284 (6) | 0.8469 (3) | 0.55939 (14) | 0.0526 (15) |
| C1A  | 0.1723 (5) | 0.5265 (2) | 0.51885 (9) | 0.0213 (9) |
| C2A  | 0.0081 (5) | 0.5733 (3) | 0.52485 (11) | 0.0277 (11) |
| C3A  | -0.0043 (6) | 0.6792 (3) | 0.53827 (11) | 0.0320 (12) |
| C4A  | 0.1423 (6) | 0.7411 (3) | 0.54628 (12) | 0.0314 (13) |
| C5A  | 0.3047 (6) | 0.6939 (3) | 0.53962 (11) | 0.0324 (11) |
| C6A  | 0.3193 (5) | 0.5885 (3) | 0.52554 (10) | 0.0271 (10) |
| O1W  | 0.4311 (4) | -0.0600 (3) | 0.13578 (10) | 0.0461 (11) |
| O2W  | -0.2441 (4) | 0.3881 (3) | 0.43528 (11) | 0.0521 (11) |
| O3W  | 0.4514 (4) | 0.8770 (3) | 0.61869 (11) | 0.0587 (12) |
| O4W  | 0.2795 (4) | 0.5374 (3) | 0.40023 (10) | 0.0511 (11) |
| H1   | 0.21570 | 0.44380 | 0.31060 | 0.0260* |
| H4   | 0.18370 | 0.23920 | 0.18630 | 0.0230* |
| H8   | 0.06440 | 0.05630 | 0.31430 | 0.0210* |
| H12  | 0.43720 | -0.10550 | 0.29900 | 0.0260* |
| H13  | 0.42710 | 0.08270 | 0.29690 | 0.0210* |
| H14  | 0.50800 | 0.02480 | 0.36480 | 0.0250* |
| H16  | 0.26740 | 0.32150 | 0.36610 | 0.0260* |
| H19  | 0.122 (6) | 0.258 (3) | 0.4190 (11) | 0.0620* |
| H22  | 0.13050 | -0.09540 | 0.41630 | 0.0280* |
| H111 | 0.33410 | -0.10810 | 0.23570 | 0.0290* |
| H112 | 0.13960 | -0.11370 | 0.25330 | 0.0290* |
| H151 | 0.48150 | 0.20080 | 0.39260 | 0.0260* |
| H152 | 0.50090 | 0.21300 | 0.34270 | 0.0260* |
| H171 | -0.07610 | 0.31420 | 0.34240 | 0.0280* |
| H172 | -0.13360 | 0.20150 | 0.32080 | 0.0280* |
| H181 | -0.06470 | 0.10210 | 0.37890 | 0.0280* |
| H182 | -0.13850 | 0.21120 | 0.40020 | 0.0280* |
| H201 | 0.30700 | 0.13230 | 0.44020 | 0.0280* |
| H202 | 0.11670 | 0.07810 | 0.44180 | 0.0280* |
| H231 | 0.38680 | -0.17010 | 0.36320 | 0.0320* |
| H232 | 0.20630 | -0.23190 | 0.37180 | 0.0320* |
| H251 | 0.30870 | 0.70020 | 0.27070 | 0.0600* |
| H252 | 0.38120 | 0.59660 | 0.29540 | 0.0600* |
| H253 | 0.17600 | 0.62160 | 0.29470 | 0.0600* |
| H261 | 0.23380 | 0.43690 | 0.11180 | 0.0390* |
| H262 | 0.10930 | 0.35070 | 0.13440 | 0.0390* |
| H263 | 0.31660 | 0.33430 | 0.13520 | 0.0390* |
| H2A  | -0.09470 | 0.53230 | 0.51970 | 0.0330* |
| H3A  | -0.11650 | 0.71040 | 0.54210 | 0.0390* |
| H5A  | 0.40740 | 0.73490 | 0.54490 | 0.0390* |
| H6A  | 0.43160 | 0.55850 | 0.52040 | 0.0330* |
| H13A | 0.445 (6) | 0.298 (3) | 0.4931 (13) | 0.0770* |
| H41A | 0.022 (3) | 0.876 (4) | 0.5617 (15) | 0.0620* |
| H42A | 0.227 (3) | 0.861 (4) | 0.5735 (13) | 0.0620* |
| H11W | 0.360 (5) | -0.029 (4) | 0.1548 (10) | 0.0770* |
| H12W | 0.358 (5) | -0.071 (4) | 0.1141 (10) | 0.0770* |
### Atomic displacement parameters (Å²)

|     | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|-----|-----------|-----------|-----------|-----------|-----------|-----------|
| O2  | 0.0412 (17)| 0.0165 (12)| 0.0288 (13)| −0.0036 (11)| 0.0025 (12)| −0.0002 (10)|
| O3  | 0.0292 (13)| 0.0255 (13)| 0.0207 (12)| −0.0021 (11)| 0.0027 (10)| 0.0008 (10)|
| O24 | 0.0244 (12)| 0.0185 (11)| 0.0243 (11)| −0.0011 (11)| −0.0001 (11)| 0.0000 (9)|
| O25 | 0.0533 (19)| 0.0281 (14)| 0.0195 (12)| 0.0026 (14)| 0.0064 (13)| 0.0055 (10)|
| N9  | 0.0226 (14)| 0.0182 (12)| 0.0168 (12)| 0.0001 (13)| −0.0003 (11)| −0.0035 (11)|
| N19 | 0.0261 (15)| 0.0230 (15)| 0.0169 (14)| −0.0009 (12)| 0.0011 (12)| −0.0052 (12)|
| C1  | 0.0251 (19)| 0.0207 (17)| 0.0203 (16)| 0.0005 (14)| −0.0011 (15)| −0.0087 (13)|
| C2  | 0.0181 (18)| 0.0173 (16)| 0.0263 (18)| 0.0022 (13)| 0.0011 (15)| 0.0004 (14)|
| C3  | 0.0169 (17)| 0.0247 (17)| 0.0183 (15)| −0.0004 (15)| 0.0021 (13)| 0.0018 (14)|
| C4  | 0.0200 (16)| 0.0229 (16)| 0.0148 (14)| −0.0001 (15)| 0.0007 (15)| −0.0046 (12)|
| C5  | 0.0196 (17)| 0.0181 (15)| 0.0181 (15)| 0.0014 (14)| −0.0008 (15)| −0.0012 (12)|
| C6  | 0.0196 (16)| 0.0213 (15)| 0.0192 (15)| 0.0022 (15)| 0.0023 (15)| 0.0000 (13)|
| C7  | 0.0228 (17)| 0.0176 (16)| 0.0177 (16)| 0.0017 (13)| 0.0007 (14)| −0.0031 (13)|
| C8  | 0.0193 (16)| 0.0188 (16)| 0.0153 (14)| −0.0002 (14)| 0.0004 (13)| −0.0039 (12)|
| C10 | 0.0214 (18)| 0.0235 (17)| 0.0224 (17)| −0.0025 (16)| 0.0005 (16)| −0.0059 (14)|
| C11 | 0.030 (2)  | 0.0184 (17)| 0.0248 (18)| 0.0021 (14)| −0.0014 (16)| −0.0066 (14)|
| C12 | 0.0204 (17)| 0.0190 (16)| 0.0255 (17)| 0.0035 (16)| 0.0024 (16)| −0.0047 (13)|
| C13 | 0.0137 (16)| 0.0175 (15)| 0.0208 (16)| −0.0005 (13)| 0.0031 (13)| −0.0038 (12)|
| C14 | 0.0164 (17)| 0.0248 (18)| 0.0211 (17)| 0.0020 (14)| −0.0029 (14)| −0.0028 (14)|
| C15 | 0.0210 (18)| 0.0250 (18)| 0.0192 (17)| −0.0039 (15)| −0.0021 (15)| −0.0058 (15)|
| C16 | 0.0291 (18)| 0.0173 (16)| 0.0182 (16)| −0.0043 (13)| 0.0024 (15)| −0.0043 (13)|
| C17 | 0.0249 (19)| 0.0242 (19)| 0.0216 (17)| 0.0053 (15)| 0.0030 (15)| −0.0054 (14)|
| C18 | 0.0186 (17)| 0.0283 (19)| 0.0239 (18)| 0.0018 (15)| 0.0061 (15)| −0.0032 (15)|
| C20 | 0.0289 (18)| 0.0239 (17)| 0.0175 (15)| −0.0004 (17)| −0.0029 (15)| 0.0001 (14)|
| C21 | 0.0204 (17)| 0.0257 (18)| 0.0201 (16)| 0.0025 (14)| −0.0065 (14)| 0.0003 (14)|
| C22 | 0.0229 (18)| 0.0272 (17)| 0.0205 (16)| 0.0019 (15)| −0.0026 (16)| 0.0032 (13)|
| C23 | 0.0284 (19)| 0.0210 (17)| 0.0314 (19)| −0.0009 (14)| −0.0039 (16)| 0.0031 (15)|
| C25 | 0.062 (3)  | 0.0220 (19)| 0.036 (2)  | −0.004 (2)  | −0.002 (2)  | −0.0032 (17)|
| C26 | 0.0268 (19)| 0.0317 (19)| 0.0204 (15)| 0.0005 (17)| −0.0019 (14)| 0.0030 (16)|
| As1A| 0.0219 (2) | 0.0175 (1) | 0.0187 (1) | −0.0005 (1) | 0.0006 (2)  | −0.0038 (2) |
| O11A| 0.0363 (15)| 0.0273 (13)| 0.0229 (12)| −0.0063 (12)| 0.0047 (11)| −0.0041 (11)|
| O12A| 0.0368 (14)| 0.0204 (12)| 0.0197 (11)| 0.0008 (11)| 0.0016 (10)| −0.0054 (10)|
| O13A| 0.0239 (12)| 0.0339 (14)| 0.0505 (19)| 0.0038 (11)| −0.0055 (13)| −0.0178 (13)|
| N4A | 0.060 (3)  | 0.0279 (19)| 0.070 (3)  | 0.0095 (18)| −0.011 (2)  | −0.0174 (18)|
| C1A | 0.0328 (19)| 0.0171 (15)| 0.0139 (15)| −0.0015 (15)| 0.0001 (16)| −0.0007 (12)|
| C2A | 0.0270 (19)| 0.0250 (19)| 0.031 (2)  | 0.0009 (15)| 0.0045 (17)| −0.0009 (16)|
| C3A | 0.042 (2)  | 0.026 (2)  | 0.028 (2)  | 0.0090 (17)| 0.0073 (18)| 0.0008 (16)|
| C4A | 0.048 (3)  | 0.0208 (18)| 0.0254 (18)| 0.0027 (17)| −0.0018 (18)| 0.0023 (15)|
### Geometric parameters (Å, °)

| Bond                        | Length (Å) | Angle (°) |
|-----------------------------|------------|-----------|
| As1A—O12A                   | 1.671 (2)  |           |
| As1A—O13A                   | 1.704 (2)  |           |
| As1A—C1A                    | 1.896 (3)  |           |
| As1A—O11A                   | 1.657 (2)  |           |
| O2—C2                      | 1.371 (4)  |           |
| O2—C25                     | 1.423 (4)  |           |
| O3—C26                     | 1.426 (4)  |           |
| O3—C3                      | 1.369 (4)  |           |
| O24—C23                    | 1.425 (4)  |           |
| O24—C12                    | 1.423 (4)  |           |
| O25—C10                    | 1.229 (4)  |           |
| O13A—H13A                  | 0.90 (4)   |           |
| O1W—H12W                   | 0.90 (3)   |           |
| O1W—H11W                   | 0.90 (4)   |           |
| O2W—H22W                   | 0.89 (3)   |           |
| O2W—H21W                   | 0.90 (3)   |           |
| O3W—H32W                   | 0.91 (3)   |           |
| N9—C5                      | 1.424 (4)  |           |
| N9—C10                     | 1.351 (4)  |           |
| N9—C8                      | 1.488 (4)  |           |
| N19—C16                    | 1.529 (4)  |           |
| N19—C18                    | 1.509 (4)  |           |
| N19—C20                    | 1.503 (4)  |           |
| O4W—H42W                   | 0.89 (4)   |           |
| O4W—H41W                   | 0.90 (4)   |           |
| N19—H19                    | 0.91 (4)   |           |
| N4A—C4A                    | 1.374 (5)  |           |
| N4A—H41A                   | 0.89 (3)   |           |
| N4A—H42A                   | 0.90 (3)   |           |
| C1—C2                      | 1.383 (5)  |           |
| C1—C6                      | 1.393 (5)  |           |
| C2—C3                      | 1.407 (5)  |           |
| C3—C4                      | 1.381 (4)  |           |
| C4—C5                      | 1.388 (4)  |           |
| C5—C6                      | 1.380 (4)  |           |
| C6—C7                      | 1.506 (5)  |           |
| C7—C17                     | 1.541 (4)  |           |
| C7—C16                     | 1.540 (5)  |           |

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C7—C8  1.557 (5)  C5A—C6A  1.379 (5)
C8—C13  1.527 (5)  C2A—H2A  0.9500
C10—C11  1.511 (5)  C3A—H3A  0.9500
C11—C12  1.543 (5)  C5A—H5A  0.9500
C12—C13  1.537 (5)  C6A—H6A  0.9500

O12A—As1A—C1A  110.46 (12)  N9—C8—H8  110.00
O13A—As1A—C1A  101.45 (14)  C13—C8—H8  110.00
O12A—As1A—O13A  111.55 (13)  C12—C11—H11  108.00
O11A—As1A—C1A  112.41 (13)  C10—C11—H11  108.00
O11A—As1A—O12A  111.48 (11)  C10—C11—H112  108.00
O11A—As1A—O13A  109.09 (12)  C11—C12—H12  109.00
C2—O2—C25  117.1 (3)  H111—C11—H112  107.00
C3—O3—C26  116.2 (3)  C12—C11—H112  108.00
C12—O24—C23  114.5 (3)  O24—C12—H12  109.00
As1A—O13A—H13A  114 (3)  C13—C12—H12  109.00
H11W—O1W—H12W  102 (3)  C11—C12—H12  109.00
H21W—O2W—H22W  108 (5)  C8—C13—H13  107.00
H31W—O3W—H32W  100 (4)  C12—C13—H13  107.00
C8—N9—C10  120.1 (3)  C14—C13—H13  106.00
C5—N9—C10  125.0 (3)  C15—C14—H14  109.00
C5—N9—C8  109.1 (2)  C21—C14—H14  109.00
C16—N19—C18  107.3 (2)  C13—C14—H14  109.00
C16—N19—C20  112.9 (3)  H151—C15—H152  109.00
C18—N19—C20  112.3 (3)  C16—C15—H151  110.00
H41W—O4W—H42W  104 (4)  C16—C15—H152  110.00
C20—N19—H19  106 (2)  C14—C15—H151  110.00
C18—N19—H19  108 (3)  C14—C15—H152  110.00
C16—N19—H19  110 (3)  C15—N16—H16  108.00
H41A—N4A—H42A  130 (4)  C15—C16—H16  108.00
C4A—N4A—H42A  106 (3)  C7—C16—H16  109.00
C4A—N4A—H41A  119 (3)  C7—C17—H171  111.00
C2—C1—C6  119.1 (3)  C7—C17—H172  111.00
O2—C2—C1  124.6 (3)  C18—C17—H171  111.00
O2—C2—C3  115.5 (3)  C18—C17—H172  111.00
C1—C2—C3  120.0 (3)  H171—C17—H172  109.00
O3—C3—C2  115.5 (3)  H181—C18—H182  109.00
O3—C3—C4  123.3 (3)  C17—C18—H182  111.00
C2—C3—C4  121.2 (3)  N19—C18—H182  111.00
C3—C4—C5  117.7 (3)  C17—C18—H181  111.00
C4—C5—C6  122.1 (3)  N19—C18—H181  111.00
N9—C5—C6  127.7 (3)  C21—C20—H201  110.00
N9—C5—C4  110.1 (3)  N19—C20—H202  110.00
C5—C6—C7  110.5 (3)  H201—C20—H202  108.00
C1—C6—C7  129.4 (3)  N19—C20—H201  110.00
C1—C6—C5  119.9 (3)  C21—C20—H202  110.00
C6—C7—C8  102.5 (3)  C23—C22—H22  118.00
C16—C7—C17  102.0 (3)  C21—C22—H22  118.00
| Bond/Angle | Value 1 | Value 2 | Value 3 |
|------------|---------|---------|---------|
| C6—C7—C17 | 112.4 (3) | O24—C23—H232 | 109.00  |
| C8—C7—C17 | 110.8 (3) | O24—C23—H231 | 109.00  |
| C6—C7—C16 | 115.4 (3) | H231—C23—H232 | 108.00  |
| C8—C7—C16 | 114.1 (3) | C22—C23—H232 | 109.00  |
| C7—C8—C13 | 116.6 (3) | C22—C23—H231 | 109.00  |
| N9—C8—C7 | 104.5 (3) | H251—C25—H253 | 109.00  |
| N9—C8—C13 | 106.0 (2) | H252—C25—H253 | 110.00  |
| O25—C10—C11 | 120.7 (3) | H251—C25—H252 | 109.00  |
| O25—C10—N9 | 122.5 (3) | O2—C25—H253 | 109.00  |
| N9—C10—C11 | 116.8 (3) | O2—C25—H251 | 110.00  |
| C10—C11—C12 | 118.1 (3) | O2—C25—H252 | 109.00  |
| O24—C12—C11 | 105.3 (3) | O3—C26—H261 | 110.00  |
| O24—C12—C13 | 114.4 (3) | O3—C26—H262 | 109.00  |
| C8—C13—C12 | 106.8 (3) | O3—C26—H263 | 109.00  |
| C8—C13—C14 | 112.0 (3) | O3—C26—H263 | 109.00  |
| C12—C13—C14 | 117.8 (3) | O3—C26—H263 | 109.00  |
| C11—C12—C13 | 109.9 (3) | O3—C26—H263 | 109.00  |
| C8—C13—C12 | 106.8 (3) | O3—C26—H263 | 109.00  |
| C8—C13—C14 | 112.0 (3) | O3—C26—H263 | 109.00  |
| C12—C13—C14 | 117.8 (3) | O3—C26—H263 | 109.00  |
| C13—C14—C15 | 106.0 (3) | O3—C26—H263 | 109.00  |
| C15—C14—C21 | 109.8 (3) | As1A—C1A—C2A | 119.6 (3) |
| C13—C14—C21 | 114.4 (3) | As1A—C1A—C6A | 121.4 (3) |
| C14—C15—C16 | 108.1 (3) | As1A—C1A—C2A | 119.8 (4) |
| C7—C16—C15 | 115.7 (3) | As1A—C1A—C6A | 121.7 (4) |
| N19—C16—C7 | 105.0 (3) | N4A—C4A—C5A | 120.9 (4) |
| N19—C16—C15 | 110.5 (3) | N4A—C4A—C3A | 121.2 (4) |
| C7—C17—C18 | 103.1 (3) | C3A—C4A—C5A | 117.9 (4) |
| N19—C18—C17 | 105.1 (3) | C4A—C5A—C6A | 121.1 (4) |
| N19—C20—C21 | 109.7 (2) | C1A—C6A—C5A | 120.5 (4) |
| C14—C21—C20 | 114.6 (3) | C1A—C6A—C5A | 120.00  |
| C14—C21—C22 | 123.4 (3) | C1A—C6A—H2A | 120.00  |
| C20—C21—C22 | 122.0 (3) | C1A—C6A—H2A | 120.00  |
| C21—C22—C23 | 123.3 (3) | C1A—C6A—H2A | 120.00  |
| O24—C23—C22 | 111.9 (3) | C6A—C5A—H5A | 119.00  |
| C6—C1—H1 | 120.00 | C4A—C5A—H5A | 119.00  |
| C2—C1—H1 | 120.00 | C1A—C6A—H6A | 120.00  |
| C5—C4—H4 | 121.00 | C5A—C6A—H6A | 120.00  |
| C3—C4—H4 | 121.00 | C5A—C6A—H6A | 120.00  |
| Bond/Distance | Value (°/Å) |
|---------------|------------|
| C12—O24—C23—C22 | 87.0 (4) |
| C23—O24—C12—C11 | 170.0 (3) |
| C8—N9—C5—C6 | 3.2 (4) |
| C8—N9—C5—C4 | 174.7 (3) |
| C5—N9—C10—O25 | -24.5 (5) |
| C10—N9—C5—C4 | 22.1 (6) |
| C10—N9—C5—C6 | -155.9 (3) |
| C5—N9—C8—C7 | 13.4 (3) |
| C5—N9—C8—C13 | -110.4 (3) |
| C10—N9—C8—C7 | 167.6 (3) |
| C10—N9—C8—C13 | 43.9 (4) |
| C8—N9—C10—O25 | -174.4 (3) |
| C8—N9—C10—C11 | 6.3 (5) |
| C5—N9—C10—C11 | 156.3 (3) |
| C20—N19—C16—C15 | -10.7 (4) |
| C16—N19—C18—C17 | -16.7 (3) |
| C18—N19—C16—C7 | -9.6 (3) |
| C18—N19—C16—C15 | -134.9 (3) |
| C20—N19—C16—C7 | 114.7 (3) |
| C18—N19—C20—C21 | 74.2 (3) |
| C20—N19—C18—C17 | -141.3 (3) |
| C16—N19—C20—C21 | -47.3 (4) |
| C2—C1—C6—C7 | -174.0 (3) |
| C6—C1—C2—O2 | -177.6 (3) |
| C6—C1—C2—C3 | 2.3 (5) |
| C2—C1—C6—C5 | 0.4 (5) |
| C1—C2—C3—C4 | -3.1 (5) |
| O2—C2—C3—O3 | -3.0 (4) |
| O2—C2—C3—C4 | 176.9 (3) |
| C1—C2—C3—C4 | 177.1 (3) |
| O3—C3—C4—C5 | -179.2 (3) |
| C2—C3—C4—C5 | 1.0 (5) |
| C3—C4—C5—N9 | -176.0 (3) |
| C3—C4—C5—C6 | 1.8 (5) |
| N9—C5—C6—C7 | -9.0 (4) |
| N9—C5—C6—C1 | 175.6 (3) |
| C4—C5—C6—C1 | -2.5 (6) |
| C4—C5—C6—C7 | 172.9 (3) |
| C5—C6—C7—C16 | 141.2 (3) |
| C1—C6—C7—C8 | -168.6 (4) |
| C1—C6—C7—C16 | -44.0 (5) |
| C1—C6—C7—C17 | 72.5 (5) |
| C5—C6—C7—C17 | 16.6 (4) |
| C5—C6—C7—C17 | -102.4 (4) |

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Hydrogen-bond geometry (Å, º)

| D—H···A       | D—H  | H···A  | D···A  | D—H···A |
|---------------|------|--------|--------|---------|
| N19—H19···O12A | 0.91 (4) | 1.72 (4) | 2.610 (3) | 168 (4) |
| N4A—H41A···O4W | 0.89 (3) | 2.46 (4) | 3.291 (5) | 155 (4) |
| N4A—H42A···O3W | 0.90 (3) | 2.25 (3) | 3.137 (6) | 169 (4) |
| O13A—H13A···O11A | 0.90 (4) | 1.67 (4) | 2.546 (3) | 165 (4) |
| O1W—H11W···O25 | 0.90 (4) | 1.95 (4) | 2.843 (4) | 175 (3) |
| O1W—H12W···O2W | 0.90 (3) | 1.87 (4) | 2.760 (5) | 168 (4) |
| O2W—H21W···O12A | 0.90 (3) | 2.11 (3) | 2.945 (4) | 153 (4) |
| O2W—H22W···O11A | 0.89 (3) | 2.07 (4) | 2.915 (4) | 158 (5) |
| O3W—H31W···O25 | 0.91 (4) | 2.06 (4) | 2.922 (4) | 159 (3) |
| O3W—H32W···O4W | 0.91 (3) | 1.91 (3) | 2.791 (4) | 164 (3) |
| O4W—H41W···O1W | 0.90 (4) | 1.88 (4) | 2.770 (5) | 172 (5) |
| O4W—H42W···O12A | 0.89 (4) | 1.91 (4) | 2.802 (4) | 174 (5) |
| C4—H4···O25 | 0.95 | 2.37 | 2.900 (4) | 115 |
| C6A—H6A···O13A | 0.95 | 2.55 | 3.011 (4) | 110 |
| C8—H8···O24 | 1.00 | 2.60 | 3.009 (4) | 104 |
| C14—H14···O3vii | 1.00 | 2.52 | 3.363 (4) | 142 |
| C15—H151···O11A | 0.99 | 2.60 | 3.561 (4) | 165 |
| C18—H182···O2W | 0.99 | 2.58 | 3.422 (5) | 143 |
| C20—H201···O11A | 0.99 | 2.41 | 3.388 (4) | 170 |
| C20—H202···O13A | 0.99 | 2.43 | 3.229 (4) | 137 |

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