Crystal structures of 6-nitroquinazolin-4(3H)-one, 6-aminoquinazolin-4(3H)-one and 4-aminoquinazoline hemihydrochloride dihydrate

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The title compounds, 6-nitroquinazolin-4(3H)-one (C₈H₅N₃O₃; I), 6-aminoquinazolin-4(3H)-one (C₈H₇N₃O; II) and 4-aminoquinazolin-1-ium chloride–4-aminoquinazoline–water (1/1/2), (C₈H₈N₃⁺/C₁Cl⁻/C₀/C₁C₈H₇N₃/2H₂O; III) were synthesized and their structures were determined by single-crystal X-ray analysis. In the crystals of I and II, the quinazoline molecules form hydrogen-bonded dimers via N—H···O interactions. The dimers are connected by weak intermolecular C—H···N and C—H···O hydrogen bonds, forming a layered structure in the case of I. In the crystal of II, N—H···N and C—H···O interactions link the dimers into a three-dimensional network structure. The asymmetric unit of III consists of two quinazoline molecules, one of which is protonated, a chloride ion, and two water molecules. The chloride anion and the water molecules form hydrogen-bonded chains consisting of fused five-membered rings. The protonated and unprotonated quinazolin molecules are linked to the chloride ions and water molecules of the chain by their amino groups.

1. Chemical context

Heterocyclic compounds play an important role in the lives of plant and living organisms because of their properties, including anti-inflammatory (Azab et al., 2016), antitumor (Ishikawa et al., 2009), antiviral (De Clercq & Field, 2006) and other activities (Ding et al., 1999). Quinazoline derivatives occupy a distinct position among nitrogen-containing heterocycles because of their wide spectrum of pharmaceutical and biopharmaceutical properties, amongst them anticancer (Chandregowda et al., 2009), antibacterial (Antipenko et al., 2009), anti-inflammatory (Alagarsamy et al., 2007), antituberculosis (Nandy et al., 2006), antihypertension (Hess et al., 1968) and antidiabetic (Paneersalvam et al., 2010) activities.

In line with this, we synthesized 6-nitroquinazolin-4(3H)-one (I), 6-aminoquinazolin-4(3H)-one (II) and 4-aminoquinazoline hemihydrochloride dihydrate (III), which are important intermediates in drug synthesis, and their crystal structures were determined. The hemi-protonated structures may be useful for the preparation of materials important to
various branches of science, ranging from biology to nano-
device fabrication and to pharmaceuticals (Perumalla et al., 2013).

2. Structural commentary

Compound I crystallizes in the triclinic space group $P\overline{1}$ with
one molecule in the asymmetric unit. As a whole, the molecule
is nearly planar. The nitro group is rotated slightly with respect
to the quinazoline ring system, the $C5-C6-N9-O3$ and
$C7-C6-N9-O2$ torsion angles being 6.0 (3) and 4.9 (4)$^\circ$,
respectively. All bond lengths and angles are normal and in
good agreement with those reported previously (Liao et al., 2018; Yong et al., 2008). Fig. 1 shows the molecular structure of
I in the solid state. Selected geometric parameters are listed in
Table 1.

Compound II crystallizes in the orthorhombic space group $Pca_2_1$
with two crystallographically independent molecules, A
and B, in the asymmetric unit (Fig. 2). All the atoms of the
molecule (except the amino-group hydrogens) lie in the same
plane. The nitrogen atom of the amino group is somewhere
between the $sp^2$ and $sp^3$ hybridized states, the sum of the
valence angles at the nitrogen atom being 349 and 342$^\circ$ in
molecules A and B, respectively. All bond lengths and angles

![Figure 1](image1.png)

**Figure 1**
The molecular structure of 6-nitroquinazolin-4(3H)-one (I), with
displacement ellipsoids drawn at the 50% probability level.

![Figure 2](image2.png)

**Figure 2**
The molecular structure of 6-aminoquinazolin-4(3H)-one (II), showing
the two independent molecules, with displacement ellipsoids drawn at the
50% probability level.

![Figure 3](image3.png)

**Figure 3**
The asymmetric unit of compound III with displacement ellipsoids drawn
at the 50% probability level.

| Table 1 | Selected bond lengths (Å) for I. |
|---------|--------------------------------|
| N1—C2  | 1.287 (3)                      |
| C2—N3  | 1.354 (3)                      |

| Table 2 | Selected bond lengths (Å) for II. |
|---------|---------------------------------|
| N1A—C2A| 1.291 (5)                       |
| C2A—N3A| 1.369 (4)                       |
| N3A—C4A| 1.376 (4)                       |
| C6A—N9A| 1.374 (4)                       |

| Table 3 | Selected bond lengths (Å) for III. |
|---------|----------------------------------|
| N1A—C2A| 1.315 (4)                        |
| C2A—N3A| 1.328 (4)                        |
| N3A—C4A| 1.363 (4)                        |
| C4A—N9A| 1.293 (4)                        |

Turgunov et al. • C$_6$H$_5$N$_3$O$_3$, C$_8$H$_7$N$_3$O and C$_8$H$_8$N$_3$$^+$Cl$^-$C$_6$H$_5$N$_3$2H$_2$O
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hydrogen bonds, producing layers parallel to the \( (1_2 \cdot 1/2) \) plane formed by weak intermolecular \( \text{C—H} \ldots \text{C} \) hydrogen bonds. The packing analysis of \( \text{III} \) shows that the protonated and unprotonated 4-aminoquinazoline molecules are linked by intermolecular \( \text{N—H} \ldots \text{N} \) hydrogen bonds, forming pseudocentrosymmetric dimers characterized by a donor–acceptor distance of 2.786 (3) \( \text{Å} \). Other \( \text{N—H} \ldots \text{N} \) hydrogen bonds form centrosymmetric \( R_2^1(8) \) ring motifs. The chloride anion and water molecules form hydrogen-bonded chains consisting of fused five-membered rings with the participation of two chloride anions and three water molecules. A chain of rings runs through the twofold screw axis parallel to the \([010]\) direction (Fig. 6). The protonated and unprotonated quinazoline molecules link to the chain via \( \text{N—H} \ldots \text{Cl} \) and \( \text{N—H} \ldots \text{Ow} \) hydrogen bonds from the lower and upper side (Table 6, Fig. 6). The chain direction corresponds to the smallest unit-cell edge and such \textit{self-assembly} of molecules has also been observed in other quinazoline hydrochloride crystals.

3. Supramolecular features

In the crystal of \( \text{I} \), intermolecular \( \text{N—H} \ldots \text{O} \) hydrogen bonds link the molecules into centrosymmetric dimers, forming \( R_2^1(8) \) motifs. Other head-to-head \( R_2^1(10) \) and \( R_2^1(8) \) motifs are formed by weak intermolecular \( \text{C—H} \ldots \text{O} \) and \( \text{C—H} \ldots \text{N} \) hydrogen bonds, producing layers parallel to the \((1\overline{1}2)\) plane (Table 4, Fig. 4). In addition, an \( R_2^1(8) \) ring motif is formed by the interactions between three adjacent molecules. The layers are linked though \( \pi \ldots \pi \) stacking interactions with centroid–centroid distances of 3.8264 (13) and 3.9600 (14) \( \text{Å} \) into a three-dimensional network.

The two independent molecules of compound \( \text{II} \) are related by a pseudo-center of symmetry and are linked by two \( \text{N—H} \ldots \text{O} \) hydrogen bonds, forming an \( R_2^1(8) \) motif. An \( \text{N—H} \ldots \text{N} \) hydrogen bond generates a three-dimensional network (Table 5, Fig. 5).

### Table 4

| \( \text{D—H} \cdot \cdot \cdot \text{A} \) | \( \text{D—H} \) | \( \text{H} \cdot \cdot \cdot \text{A} \) | \( \text{D—H} \cdot \cdot \cdot \text{A} \) | \( \text{D—H} \cdot \cdot \cdot \text{A} \) |
|------------------|-------|-------|------------------|------------------|
| \( \text{N3—H3} \cdot \cdot \cdot \text{O1}^1 \) | 0.80 (3) | 2.02 (3) | 2.818 (2) | 178 (4) |
| \( \text{C8—H8} \cdot \cdot \cdot \text{N1}^a \) | 0.93 | 2.53 | 3.450 (3) | 172 |
| \( \text{C2—H2} \cdot \cdot \cdot \text{O2}^a \) | 0.93 | 2.57 | 3.466 (4) | 163 |
| \( \text{C7—H7} \cdot \cdot \cdot \text{O2}^a \) | 0.93 | 2.56 | 3.437 (3) | 158 |

Symmetry codes: (i) \( x, y—z, y, z \); (ii) \( x, y, z \); (iii) \( x, y, z \); (iv) \( x, y, z \); (v) \( x, y, z \).
4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.41, including the update of January 2020; Groom et al., 2016) confirmed that three related compounds had been structurally characterized in which the benzene ring of the quinazolin-4(3H)-ones contains a nitro group [refcodes GAPPUK (Yu et al., 2012), GISXOW (Yong et al., 2008) and RUGKEK (Wu et al., 2009)].

The crystal structures of quinazolin-4(3H)-one and its first metal coordination compound have also been reported [BIHJJO (Liao et al., 2018) and NALFEN (Turgunov & Englert, 2010)].

5. Synthesis and crystallization

**Compound I:** In a three-necked flask equipped with a mechanical stirrer and reflux condenser, quinazolin-4(3H)-one (22.5 g, 0.15 mol) was dissolved in 78 ml of concentrated sulfuric acid at 303 K for 1 h. Then a nitration mixture (21 ml of nitric acid and 18 ml of concentrated sulfuric acid) was added to the flask under vigorous stirring of the mixture. The reaction mixture was stirred for another hour, maintaining a temperature not higher than 303 K, and then for another hour at room temperature. At room temperature, 45 ml of nitric acid were added dropwise to the reaction mixture over a period of 1 h. The reaction mixture was left at room temperature for 10 h. The contents of the flask were poured into a dish containing ice, the resulting precipitate was filtered off, washed with water and dried and recrystallized from ethanol to obtain 25.7 g of pure compound I as single crystals in 87.4% yield, m.p. 560–562 K.

**Compound II:** In a three-necked flask equipped with a mechanical stirrer and reflux condenser, 12.6 g (56 mmol) of tin (II) chloride dihydrate (SnCl2·2H2O) were cooled in an ice bath and 16.98 ml of concentrated (36%) HCl were added, then 3 g (16 mmol) of quinazolin-4-one as a suspension in water, and brought to a strongly alkaline medium (pH = 10–11) with 10% of sodium hydroxide, in which the expected 6-amino-3N-quinazoline-4-one was dissolved, so that the chloride was brought to a neutral medium in the presence of acid, and precipitated when converted to an alkaline medium with ammonia. The precipitate was filtered, washed with water until it reached a neutral medium, and dried at room temperature. The precipitate was recrystallized from ethanol and 6.67 g of pure compound II were obtained representing an 88.1% yield, m.p. 589–591 K.

**Compound III:** Crystals of compound III were obtained as a minor additional product in the reaction of 4-chloroquinazoline with ammonia.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 7. C-bound H atoms were placed in calculated positions and refined to ride on their parent atoms: C—H = 0.93 Å with Uiso(H) = 1.2Ueq(C). Hydrogen atoms of the water molecules and those bonded to nitrogen atoms were located in electron density difference maps and were freely refined.

Acknowledgements

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Table 7
Experimental details.

|                  | I                              | II                              | III                             |
|------------------|--------------------------------|---------------------------------|---------------------------------|
| Crystal data    |                                |                                 |                                 |
| Chemical formula | C₈H₇N₃C₂H₂O₂                 | C₈H₇N₃O                  | C₈H₇N₃·Cl⁻·C₈H₈N₃·2H₂O       |
| Mₐ               | 191.15                         | 161.17                        | 362.82                          |
| Crystal system, space group | Triclinic, P T | Orthorhombic, Pca2₁ | Monoclinic, P2₁/n              |
| Temperature (K)  | 293                            | 293                           | 298                            |
| a, b, c (Å)      | 5.5587 (9), 8.6673 (13), 8.7649 (12) | 13.4535 (5), 4.9510 (2), 21.6188 (8) | 14.3512 (12), 7.5867 (6), 16.2282 (9) |
| α, β, γ (°)      | 105.654 (12), 98.560 (13), 90.784 (13) | 90, 90, 90                      | 90, 93.544 (7), 90             |
| V (Å³)           | 401.45 (11)                    | 1439.99 (10)                 | 1763.5 (2)                      |
| Z                 | 2                              | 8                             | 4                              |
| Radiation type   | CuKa                          | CuKa                          | CuKa                           |
| μ (mm⁻¹)         | 1.07                           | 0.86                          | 2.12                           |
| Crystal size (mm) | 0.45 × 0.30 × 0.25             | 0.60 × 0.45 × 0.35            | 0.50 × 0.08 × 0.05             |
| Data collection  |                                |                                 |                                 |
| Diffractometer   | Rigaku Xcalibur, Ruby          | Rigaku Xcalibur, Ruby         | Rigaku Xcalibur, Ruby          |
| Absorption correction | Multi-scan (CrysAlis PRO) | Multi-scan (CrysAlis PRO) | Multi-scan (CrysAlis PRO) |
| T max, T max     | 0.742, 1.000                   | 0.720, 1.000                  | 0.934, 1.000                   |
| No. of reflections | 2652, 1598, 1124               | 2219S, 2976, 2489             | 6703, 3563, 2207               |
| R int (sin θ/λ) max (Å⁻¹) | 0.024                           | 0.070                          | 0.052                          |
| Refinement       |                                |                                 |                                 |
| R[F² > 2σ(F²)], wR(F²), S | 0.047, 0.145, 1.02             | 0.036, 0.098, 1.02             | 0.054, 0.151, 1.01             |
| No. of reflections | 1598                           | 2976                           | 3563                           |
| No. of parameters | 132                            | 242                           | 261                           |
| No. of restraints | 0                              | 2                             | 4                             |
| 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 | H atoms treated by a mixture of independent and constrained refinement |
| Δρ max, Δρ max (e Å⁻³) | 0.18, −0.17                   | 0.17, −0.15                   | 0.23, −0.22                   |
| Absolute structure | –                             | Flack x determined using 1053 quotients [(I')−(I')][(I')+(I')] (Parsons et al., 2013) | –                             |
| Absolute structure parameter | –                             | 0.2 (2)                       | –                             |

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Crystal structures of 6-nitroquinazolin-4(3H)-one, 6-aminoquinazolin-4(3H)-one and 4-aminoquinazoline hemihydrochloride dihydrate

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Computing details
For all structures, data collection: CrysAlis PRO (Rigaku OD, 2018); cell refinement: CrysAlis PRO (Rigaku OD, 2018); data reduction: CrysAlis PRO (Rigaku OD, 2018); program(s) used to solve structure: SHELXT2018/2 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2014/7 (Sheldrick, 2015b); molecular graphics: XP in SHELXTL (Sheldrick, 2008); software used to prepare material for publication: publCIF (Westrip, 2010).

6-Nitroquinazolin-4(3H)-one (I)

Crystal data

C₈H₅N₃O₃

Mr = 191.15

Triclinic, P T

a = 5.5587 (9) Å

b = 8.6673 (13) Å

c = 8.7649 (12) Å

α = 105.654 (12)°

β = 98.560 (13)°

γ = 90.784 (13)°

V = 401.45 (11) Å³

Z = 2

F(000) = 196

Dₐ = 1.581 Mg m⁻³

Melting point: 560(2) K

Cu Kα radiation, λ = 1.54184 Å

Cell parameters from 950 reflections

θ = 5.3–74.5°

μ = 1.07 mm⁻¹

T = 293 K

Prism, colourless

0.45 × 0.30 × 0.25 mm

Data collection

Rigaku Xcalibur, Ruby diffractometer

Radiation source: Enhance (Cu) X-ray Source

Graphite monochromator

Detector resolution: 10.2576 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2018)

Tmin = 0.742, Tmax = 1.000

2652 measured reflections

1598 independent reflections

1124 reflections with I > 2σ(I)

Rint = 0.024

θmax = 76.3°, θmin = 5.3°

h = −6→7

k = −7→10

l = −10→9

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.047

wR(F²) = 0.145

S = 1.02

1598 reflections

132 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

\[ w = \frac{1}{[\sigma^2(F_o^2) + (0.0615P)^2]} \]

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

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

\( \Delta \rho_{\text{max}} = 0.18 \text{ e Å}^{-3} \)

\( \Delta \rho_{\text{min}} = -0.17 \text{ e Å}^{-3} \)

**Extinction correction:** SHELXL2014/7 (Sheldrick, 2015b),

\[ F_c^* = kF_c[1+0.001xF_c^2\lambda^3/\sin(2\theta)]^{1/4} \]

**Extinction coefficient:** 0.012 (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} |
|---|-------|-------|-------|--------|
| O1 | 0.6353 (3) | 0.19086 (17) | 0.52777 (19) | 0.0644 (5) |
| O2 | 0.7328 (5) | 0.9420 (2) | 0.8525 (3) | 0.0988 (8) |
| O3 | 0.8935 (3) | 0.7763 (2) | 0.6717 (2) | 0.0739 (5) |
| N1 | 0.1305 (3) | 0.3183 (2) | 0.8279 (2) | 0.0587 (5) |
| C2 | 0.1601 (4) | 0.1736 (3) | 0.7482 (3) | 0.0585 (5) |
| H2 | 0.0576 | 0.0929 | 0.7592 | 0.070* |
| N3 | 0.3277 (4) | 0.1287 (2) | 0.6497 (2) | 0.0566 (5) |
| C4 | 0.4881 (4) | 0.2343 (2) | 0.6209 (2) | 0.0517 (5) |
| C4A | 0.4645 (4) | 0.4012 (2) | 0.7094 (2) | 0.0476 (5) |
| C5 | 0.6197 (4) | 0.5228 (2) | 0.6955 (3) | 0.0519 (5) |
| H5 | 0.7399 | 0.4998 | 0.6306 | 0.062* |
| C6 | 0.5897 (4) | 0.6775 (2) | 0.7807 (3) | 0.0529 (5) |
| C7 | 0.4120 (4) | 0.7162 (2) | 0.8796 (3) | 0.0569 (5) |
| H7 | 0.3956 | 0.8224 | 0.9349 | 0.068* |
| C8 | 0.2626 (4) | 0.5968 (2) | 0.8944 (3) | 0.0567 (5) |
| H8 | 0.1446 | 0.6214 | 0.9607 | 0.068* |
| C8A | 0.2866 (4) | 0.4359 (2) | 0.8092 (2) | 0.0507 (5) |
| N9 | 0.7504 (4) | 0.8071 (2) | 0.7683 (3) | 0.0635 (5) |
| H3 | 0.334 (5) | 0.038 (3) | 0.598 (3) | 0.057 (6)* |

**Atomic displacement parameters (Å²)**

|      | U^11   | U^22   | U^33   | U^12   | U^13   | U^23   |
|------|--------|--------|--------|--------|--------|--------|
| O1   | 0.0731 (10) | 0.0452 (8) | 0.0736 (11) | 0.0019 (7) | 0.0352 (9) | 0.0014 (7) |
| O2   | 0.1272 (18) | 0.0417 (9) | 0.1230 (17) | -0.0165 (10) | 0.0525 (15) | -0.0014 (9) |
| O3   | 0.0730 (11) | 0.0618 (10) | 0.0918 (13) | -0.0050 (8) | 0.0287 (10) | 0.0215 (9) |
| N1   | 0.0605 (10) | 0.0490 (9) | 0.0681 (11) | -0.0004 (7) | 0.0260 (9) | 0.0102 (8) |
| C2   | 0.0621 (12) | 0.0457 (11) | 0.0690 (14) | -0.0031 (9) | 0.0213 (11) | 0.0126 (9) |
| N3   | 0.0674 (11) | 0.0368 (8) | 0.0646 (11) | 0.0004 (7) | 0.0226 (9) | 0.0058 (8) |
| C4   | 0.0553 (11) | 0.0431 (10) | 0.0557 (11) | 0.0035 (8) | 0.0169 (9) | 0.0073 (8) |
| C4A  | 0.0534 (10) | 0.0398 (9) | 0.0486 (10) | 0.0030 (8) | 0.0129 (8) | 0.0082 (8) |
| C5   | 0.0549 (11) | 0.0478 (11) | 0.0542 (11) | 0.0042 (8) | 0.0154 (9) | 0.0122 (8) |
| C6   | 0.0571 (11) | 0.0430 (10) | 0.0579 (11) | -0.0013 (8) | 0.0111 (9) | 0.0118 (8) |
| C7   | 0.0674 (13) | 0.0396 (10) | 0.0600 (12) | 0.0067 (9) | 0.0143 (10) | 0.0052 (8) |
|     | x    | y    | z    | x    | y    | z    |
|-----|------|------|------|------|------|------|
| C8  | 0.0617 (12) | 0.0464 (11) | 0.0606 (12) | 0.0070 (9) | 0.0211 (10) | 0.0061 (9) |
| C8A | 0.0535 (11) | 0.0436 (10) | 0.0542 (11) | 0.0027 (8) | 0.0135 (9) | 0.0092 (8) |
| N9  | 0.0694 (12) | 0.0453 (10) | 0.0744 (12) | -0.0043 (8) | 0.0133 (10) | 0.0138 (8) |

**Geometric parameters (Å, °)**

| Bond | Distance (Å) | Angle (°) |
|------|--------------|-----------|
| O1—C4 | 1.233 (2) | C4A—C5 |
| O2—N9 | 1.218 (2) | C4A—C8A |
| O3—N9 | 1.223 (2) | C5—C6 |
| N1—C2 | 1.287 (3) | C5—H5 |
| N1—C8A | 1.388 (3) | C6—C7 |
| C2—N3 | 1.354 (3) | C6—N9 |
| C2—H2 | 0.9300 | C7—C8 |
| N3—C4 | 1.366 (3) | C7—H7 |
| N3—H3 | 0.80 (3) | C8—C8A |
| C4—C4A | 1.463 (3) | C8—H8 |
| C2—N1—C8A | 115.80 (17) | C5—C6—C7 |
| N1—C2—N3 | 125.57 (19) | C5—C6—N9 |
| N1—C2—H2 | 117.2 | C7—C6—N9 |
| N3—C2—H2 | 117.2 | C8—C7—C6 |
| C2—N3—C4 | 123.57 (17) | C8—C7—H7 |
| C2—N3—H3 | 122.0 (18) | C6—C7—H7 |
| C4—N3—H3 | 114.3 (18) | C7—C8—C8A |
| O1—C4—C4A | 122.34 (17) | C7—C8—H8 |
| O1—C4—C4A | 124.23 (18) | C8A—C8—H8 |
| N3—C4—C4A | 113.43 (16) | N1—C8A—C4A |
| C5—C4A—C8A | 120.89 (18) | N1—C8A—C8 |
| C5—C4A—C4 | 120.21 (17) | C4A—C8A—C8 |
| C8A—C4A—C4 | 118.90 (18) | O2—N9—O3 |
| C6—C5—C4A | 117.95 (18) | O2—N9—C6 |
| C6—C5—H5 | 121.0 | O3—N9—C6 |
| C4A—C5—H5 | 121.0 | C6—C7—C8—C8A |

**Supporting Information**

*Acta Cryst. (2021). E77, 989-993*
Hydrogen-bond geometry (Å, °)

| D—H···A   | D—H | H···A | D···A | D—H···A |
|-----------|------|-------|-------|---------|
| N3—H3···O1i | 0.80 (3) | 2.02 (3) | 2.814 (2) | 178 (4) |
| C8—H8···N1ii | 0.93 | 2.53 | 3.450 (3) | 172 |
| C2—H2···O2iii | 0.93 | 2.57 | 3.466 (4) | 163 |
| C7—H7···O2iv | 0.93 | 2.56 | 3.437 (3) | 158 |

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

6-Aminoquinazolin-4(3H)-one (II)

Crystal data

C₈H₇N₃O  
Mr = 161.17  
Orthorhombic, Pca₂₁  
a = 13.4535 (5) Å  
b = 4.9510 (2) Å  
c = 21.6188 (8) Å  
V = 1439.99 (10) Å³  
Z = 8  
F(000) = 672  
Dₐ = 1.487 Mg m⁻³  
Melting point: 589(2) K  
Cu Kα radiation, λ = 1.54184 Å

Cell parameters from 5076 reflections  
θ = 4.1–75.8°  
µ = 0.86 mm⁻¹  
T = 293 K  
Prism, colourless

Data collection

Rigaku Xcalibur, Ruby diffractometer  
Radiation source: Enhance (Cu) X-ray Source  
Graphite monochromator  
Detector resolution: 10.2576 pixels mm⁻¹  
Detector resolution: 10.2576 pixels mm⁻¹  
ω scans  
Detector resolution: 10.2576 pixels mm⁻¹  
Absorption correction: multi-scan  
CrysAlisPro; Rigaku OD, 2018  
Tmin = 0.720, Tmax = 1.000  
22195 measured reflections  
2976 independent reflections  
2489 reflections with I > 2σ(I)  
θ = 4.1–75.8°  
h = −16→16  
k = −6→6  
l = −26→27  
Refinement

Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.036  
wR(F²) = 0.098  
S = 1.02  
2976 reflections  
242 parameters  
2 restraints  
Primary atom site location: structure-invariant direct methods  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement

Absolute structure: Flack x determined using 1053 quotients [(I+)−(I)][(I+)−(I)] (Parsons et al., 2013)

Absolute structure parameter: 0.2 (2)

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.

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

|       | x          | y          | z          | Uiso*/Ueq |
|-------|------------|------------|------------|-----------|
| O1A   | 0.40087 (16) | 0.0190 (4) | 0.46170 (11) | 0.0394 (5) |
| N1A   | 0.6791 (2) | 0.2101 (6) | 0.39888 (13) | 0.0427 (7) |
| C2A   | 0.6607 (2) | 0.0205 (7) | 0.43824 (16) | 0.0408 (8) |
| H2A   | 0.7142 | -0.0786 | 0.4532 | 0.049* |
| N3A   | 0.5680 (2) | -0.0455 (6) | 0.45958 (13) | 0.0373 (6) |
| C4A   | 0.4828 (2) | 0.0850 (6) | 0.44094 (14) | 0.0337 (7) |
| C4AA  | 0.4994 (2) | 0.3010 (6) | 0.39636 (14) | 0.0338 (7) |
| C5A   | 0.4197 (2) | 0.4509 (6) | 0.37325 (14) | 0.0361 (7) |
| H5A   | 0.3555 | 0.4132 | 0.3867 | 0.043* |
| C6A   | 0.4353 (2) | 0.6566 (7) | 0.33025 (15) | 0.0368 (7) |
| C7A   | 0.5343 (3) | 0.7079 (7) | 0.31113 (15) | 0.0394 (8) |
| H7A   | 0.5464 | 0.8449 | 0.2827 | 0.047* |
| C8A   | 0.6122 (3) | 0.5608 (7) | 0.33352 (16) | 0.0412 (8) |
| C8AA  | 0.6763 | 0.5982 | 0.3198 | 0.049* |
| C8A   | 0.5971 (3) | 0.3541 (6) | 0.37691 (15) | 0.0364 (7) |
| N9A   | 0.3578 (3) | 0.8001 (6) | 0.30523 (15) | 0.0469 (8) |
| O1B   | 0.53939 (17) | 0.5050 (4) | 0.53859 (12) | 0.0403 (6) |
| N1B   | 0.2624 (2) | 0.3084 (6) | 0.60178 (14) | 0.0451 (7) |
| C2B   | 0.2799 (2) | 0.4952 (7) | 0.56163 (16) | 0.0430 (8) |
| H2B   | 0.2257 | 0.5891 | 0.5458 | 0.052* |
| N3B   | 0.3720 (2) | 0.5655 (5) | 0.54065 (13) | 0.0385 (6) |
| C4B   | 0.4570 (3) | 0.4384 (6) | 0.55929 (14) | 0.0332 (7) |
| C4AB  | 0.4421 (2) | 0.2222 (6) | 0.60434 (15) | 0.0332 (7) |
| C5B   | 0.5223 (3) | 0.0742 (6) | 0.62695 (14) | 0.0367 (7) |
| H5B   | 0.5863 | 0.1136 | 0.6134 | 0.044* |
| C6B   | 0.5075 (3) | -0.1315 (6) | 0.66954 (15) | 0.0371 (7) |
| C7B   | 0.4094 (3) | -0.1838 (7) | 0.68957 (15) | 0.0409 (8) |
| H7B   | 0.3983 | -0.3203 | 0.7183 | 0.049* |
| C8B   | 0.3303 (3) | -0.0383 (7) | 0.66766 (15) | 0.0415 (8) |
| H8B   | 0.2666 | -0.0768 | 0.6818 | 0.050* |
| C8AB  | 0.3444 (2) | 0.1679 (6) | 0.62419 (15) | 0.0373 (7) |
| N9B   | 0.5875 (3) | -0.2736 (7) | 0.69421 (15) | 0.0470 (8) |
| H3A   | 0.560 (2) | -0.190 (7) | 0.4877 (16) | 0.039 (10)* |
| H3B   | 0.383 (3) | 0.702 (7) | 0.5125 (17) | 0.059 (12)* |
| H9AA  | 0.373 (3) | 0.957 (9) | 0.2838 (19) | 0.054 (12)* |
| H9BB  | 0.638 (3) | -0.297 (10) | 0.667 (2) | 0.071 (14)* |
| H9BA  | 0.571 (3) | -0.431 (9) | 0.715 (2) | 0.062 (12)* |
| H9AB  | 0.307 (3) | 0.804 (8) | 0.3255 (19) | 0.050 (12)* |

Atomic displacement parameters (Å²)

|       | U11 | U22 | U33 | U12 | U13 | U23 |
|-------|-----|-----|-----|-----|-----|-----|
| O1A   | 0.0381 (13) | 0.0371 (12) | 0.0429 (11) | -0.0020 (9) | 0.0049 (10) | 0.0039 (10) |
| N1A   | 0.0368 (15) | 0.0409 (16) | 0.0505 (18) | -0.0034 (13) | 0.0035 (13) | 0.0010 (13) |
| C2A   | 0.0340 (18) | 0.0394 (19) | 0.0491 (19) | 0.0022 (14) | 0.0003 (15) | 0.0011 (16) |

*Estimated standard deviations in parentheses.
| Atom  | U1  (Å²) | U2  (Å²) | U3  (Å²) | U12 (Å²) | U13 (Å²) | U23 (Å²) |
|-------|---------|---------|---------|---------|---------|---------|
| N3A   | 0.0399 (15) | 0.0334 (14) | 0.0385 (15) | -0.0004 (11) | 0.0010 (13) | 0.0035 (13) |
| C4A   | 0.0358 (17) | 0.0317 (15) | 0.0337 (16) | -0.0019 (12) | 0.0017 (13) | -0.0064 (13) |
| C4AA  | 0.0376 (17) | 0.0307 (15) | 0.0332 (16) | -0.0035 (13) | 0.0013 (12) | -0.0058 (12) |
| C5A   | 0.0375 (17) | 0.0344 (16) | 0.0363 (17) | -0.0031 (13) | 0.0016 (14) | -0.0022 (14) |
| C6A   | 0.0446 (19) | 0.0337 (16) | 0.0322 (16) | 0.0004 (14) | 0.0007 (14) | -0.0045 (13) |
| C7A   | 0.053 (2) | 0.0329 (17) | 0.0320 (18) | -0.0053 (14) | 0.0051 (14) | 0.0019 (14) |
| C8A   | 0.043 (2) | 0.0395 (18) | 0.0409 (18) | -0.0083 (14) | 0.0084 (15) | -0.0016 (14) |
| C8AA  | 0.0383 (17) | 0.0338 (17) | 0.0371 (17) | -0.0015 (14) | 0.0043 (14) | -0.0029 (14) |
| N9A   | 0.049 (2) | 0.0437 (17) | 0.0482 (18) | 0.0016 (14) | -0.0013 (15) | 0.0106 (15) |
| O1B   | 0.0384 (13) | 0.0388 (12) | 0.0437 (12) | -0.0036 (10) | 0.0049 (11) | 0.0058 (10) |
| N1B   | 0.0365 (15) | 0.0463 (17) | 0.0525 (17) | -0.0017 (12) | 0.0019 (13) | 0.0057 (13) |
| C2B   | 0.0386 (19) | 0.0428 (18) | 0.0475 (19) | 0.0008 (14) | -0.0012 (15) | 0.0015 (16) |
| N3B   | 0.0422 (16) | 0.0333 (15) | 0.0399 (15) | -0.0017 (11) | 0.0032 (13) | 0.0033 (12) |
| C4B   | 0.0393 (19) | 0.0291 (16) | 0.0313 (16) | -0.0029 (12) | 0.0016 (13) | -0.0014 (13) |
| C4AB  | 0.0384 (18) | 0.0299 (15) | 0.0314 (16) | -0.0033 (13) | 0.0040 (13) | -0.0031 (13) |
| C5B   | 0.0389 (19) | 0.0350 (16) | 0.0361 (17) | -0.0041 (14) | 0.0047 (14) | -0.0041 (13) |
| C6B   | 0.0457 (19) | 0.0323 (16) | 0.0332 (17) | -0.0005 (14) | -0.0009 (14) | -0.0015 (14) |
| C7B   | 0.051 (2) | 0.0362 (18) | 0.0350 (17) | -0.0071 (15) | 0.0032 (15) | 0.0027 (14) |
| C8B   | 0.0396 (19) | 0.0450 (19) | 0.0399 (18) | -0.0070 (14) | 0.0101 (14) | -0.0022 (15) |
| C8AB  | 0.0391 (17) | 0.0335 (16) | 0.0392 (17) | -0.0032 (13) | 0.0018 (14) | -0.0020 (14) |
| N9B   | 0.0504 (19) | 0.0457 (18) | 0.0448 (18) | 0.0022 (14) | 0.0025 (15) | 0.0079 (14) |

**Geometric parameters (Å, °)**

| Bond       | Length (Å) | Angle (°) |
|------------|------------|-----------|
| O1A—C4A   | 1.235 (4)  |           |
| N1A—C2A   | 1.291 (5)  |           |
| N1A—C8AA  | 1.397 (4)  |           |
| C2A—N3A   | 1.369 (4)  |           |
| C2A—H2A   | 0.9300     |           |
| N3A—C4A   | 1.376 (4)  |           |
| N3A—H3A   | 0.94 (3)   |           |
| C4A—C4AA  | 1.457 (4)  |           |
| C4AA—C5A  | 1.397 (4)  |           |
| C4AA—C8AA | 1.405 (4)  |           |
| C5A—C6A   | 1.395 (5)  |           |
| C5A—H5A   | 0.9300     |           |
| C6A—N9A   | 1.374 (4)  |           |
| C6A—C7A   | 1.417 (5)  |           |
| C7A—C8A   | 1.365 (5)  |           |
| C7A—H7A   | 0.9300     |           |
| C8A—C8AA  | 1.403 (5)  |           |
| C8A—H8A   | 0.9300     |           |
| N9A—H9AA  | 0.93 (4)   |           |
| N9A—H9AB  | 0.81 (4)   |           |
| C2A—N1A   | 116.3 (3)  |           |
| N1A—C2A—N3A | 124.8 (3) |           |
| N1A—C2A—H2A | 117.6     |           |

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| Bond/Crossbond       | N3A—C2A—H2A | N3B—C2B—H2B |
|---------------------|--------------|--------------|
|                     | 117.6        | 117.6        |
| C2A—N3A—C4A        | 123.2 (3)    | C2B—N3B—C4B |
|                     | 123.0 (3)    |              |
| C2A—N3A—H3A        | 120 (2)      | C2B—N3B—H3B |
|                     | 123 (2)      |              |
| C4A—N3A—H3A        | 117 (2)      | C4B—N3B—H3B |
|                     | 114 (2)      |              |
| O1A—C4A—N3A        | 120.9 (3)    | O1B—C4B—N3B |
|                     | 121.3 (3)    |              |
| O1A—C4A—C4AA       | 124.9 (3)    | O1B—C4B—C4AB|
|                     | 123.9 (3)    |              |
| N3A—C4A—C4AA       | 114.3 (3)    | N3B—C4B—C4AB|
|                     | 114.7 (3)    |              |
| C5A—C4AA—C8AA      | 120.8 (3)    | C5B—C4AB—C8AB|
|                     | 121.0 (3)    |              |
| C5A—C4AA—C4A       | 120.6 (3)    | C5B—C4AB—C4B|
|                     | 120.9 (3)    |              |
| C8AA—C4AA—C4A      | 118.6 (3)    | C8AB—C4AB—C4B|
|                     | 118.1 (3)    |              |
| C6A—C5A—C4AA       | 120.7 (3)    | C6B—C5B—C4AB|
|                     | 120.6 (3)    |              |
| C6A—C5A—H5A        | 119.6        | C6B—C5B—H5B |
|                     | 119.7        |              |
| C4AA—C5A—H5A       | 119.6        | C4AB—C5B—H5B|
|                     | 119.7        |              |
| N9A—C6A—C5A        | 121.7 (3)    | C5B—C6B—N9B |
|                     | 121.0 (3)    |              |
| N9A—C6A—C7A        | 120.4 (3)    | C5B—C6B—C7B |
|                     | 118.1 (3)    |              |
| C5A—C6A—C7A        | 117.8 (3)    | C6B—C7B—C6B |
|                     | 120.8 (3)    |              |
| C8A—C7A—C6A        | 121.5 (3)    | C8B—C7B—C6B |
|                     | 121.6 (3)    |              |
| C8A—C7A—H7A        | 119.3        | C8B—C7B—H7B |
|                     | 119.2        |              |
| C6A—C7A—H7A        | 119.3        | C6B—C7B—H7B |
|                     | 119.2        |              |
| C7A—C8A—C8AA       | 121.0 (3)    | C7B—C8B—C8AB|
|                     | 120.7 (3)    |              |
| C7A—C8A—H8A        | 119.5        | C7B—C8B—H8B |
|                     | 119.7        |              |
| C8AA—C8A—H8A       | 119.5        | C8AB—C8B—H8B|
|                     | 119.7        |              |
| N1A—C8AA—C8A       | 119.0 (3)    | N1B—C8AB—C8B|
|                     | 119.4 (3)    |              |
| N1A—C8AA—C4AA      | 122.8 (3)    | N1B—C8AB—C4AB|
|                     | 122.6 (3)    |              |
| C8AA—C8A—C4AA      | 118.2 (3)    | C8B—C8AB—C4AB|
|                     | 118.0 (3)    |              |
| C6A—N9A—H9AA       | 117 (3)      | C6B—N9B—H9BB|
|                     | 113 (3)      |              |
| C6A—N9A—H9AB       | 116 (3)      | C6B—N9B—H9BA|
|                     | 116 (3)      |              |
| H9AA—N9A—H9AB      | 116 (4)      | H9BB—N9B—H9BA|
|                     | 113 (4)      |              |

| Bond/Crossbond       | C8AA—N1A—C2A—N3A | C8AB—N1B—C2B—N3B |
|---------------------|-------------------|-------------------|
|                     | −0.4 (5)          | −0.9 (5)          |
| N1A—C2A—N3A—C4A    | 0.2 (5)           | 1.6 (5)           |
|                     | 0.2 (5)           |                  |
| C2A—N3A—C4A—O1A    | −179.7 (3)        | C2B—N3B—C4B—O1B |
|                     | 178.9 (3)         |                  |
| C2A—N3A—C4A—C4AA   | −0.1 (4)          | C2B—N3B—C4B—C4AB|
|                     | −0.8 (4)          |                  |
| O1A—C4A—C4AA—C5A   | −0.5 (5)          | O1B—C4B—C4AB—C5B|
|                     | −0.2 (5)          |                  |
| N3A—C4A—C4AA—C5A   | 179.9 (3)         | N3B—C4B—C4AB—C5B|
|                     | 179.5 (3)         |                  |
| O1A—C4A—C4AA—C8AA  | 179.8 (3)         | O1B—C4B—C4AB—C8AB|
|                     | 179.8 (3)         |                  |
| N3A—C4A—C4AA—C8AA  | 0.2 (4)           | N3B—C4B—C4AB—C8AB|
|                     | −0.5 (4)          |                  |
| C8AA—C4AA—C5A—C6A  | 0.1 (5)           | C8AB—C4AB—C5B—C6B|
|                     | 0.2 (5)           |                  |
| C4A—C4AA—C5A—C6A   | −179.6 (3)        | C4B—C4AB—C5B—C6B|
|                     | −179.8 (3)        |                  |
| C4AA—C5A—C6A—N9A   | 177.6 (3)         | C4AB—C5B—C6B—N9B|
|                     | −177.5 (3)        |                  |
| C4AA—C5A—C6A—C7A   | −0.1 (5)          | C4AB—C5B—C6B—C7B|
|                     | −0.6 (5)          |                  |
| N9A—C6A—C7A—C8A    | −177.4 (3)        | C5B—C6B—C7B—C8B |
|                     | 0.4 (5)           |                  |
| C5A—C6A—C7A—C8A    | 0.4 (5)           | N9B—C6B—C7B—C8B |
|                     | 177.3 (3)         |                  |
| C6A—C7A—C8A—C8AA   | −0.6 (5)          | C6B—C7B—C8B—C8AB|
|                     | 0.2 (5)           |                  |
| C2A—N1A—C8AA—C8A   | −179.3 (3)        | C2B—N1B—C8AB—C8B|
|                     | −179.7 (3)        |                  |
| C2A—N1A—C8AA—C4AA  | 0.6 (5)           | C2B—N1B—C8AB—C4AB|
|                     | −0.5 (5)          |                  |
| C7A—C8A—C8AA—N1A   | −179.6 (3)        | C7B—C8B—C8AB—N1B|
|                     | 178.6 (3)         |                  |
| C7A—C8A—C8AA—C4AA  | 0.5 (5)           | C7B—C8B—C8AB—C4AB|
|                     | −0.6 (5)          |                  |
Hydrogen-bond geometry (Å, °)

| D—H···A | D—H     | H···A | D···A     | D—H···A |
|---------|---------|-------|-----------|---------|
| N9A—H9AA···N9B  | 0.93 (4) | 2.55 (4) | 3.435 (5) | 160 (4)   |
| N9A—H9AB···N1A  | 0.81 (4) | 2.34 (4) | 3.144 (5) | 170 (4)   |
| N9B—H9BB···N1B  | 0.91 (4) | 2.19 (4) | 3.092 (5) | 174 (4)   |
| N3A—H3A···O1B  | 0.95 (3) | 1.89 (3) | 2.832 (4) | 175 (3)   |
| N3B—H3B···O1A  | 0.92 (4) | 1.93 (4) | 2.847 (3) | 173 (4)   |

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

4-Aminoquinazolin-1-ium chloride–4-aminoquinazoline–water (1/1/2) (III)

Crystal data

C₈H₈N₃⁺·Cl⁻·C₈H₇N₃·2H₂O

Mr = 362.82

Monoclinic, P2₁/n

a = 14.3512 (12) Å

b = 7.5867 (6) Å

c = 16.2282 (9) Å

β = 93.544 (7)°

V = 1763.5 (2) Å³

Z = 4

Dₐ = 1.367 Mg m⁻³

Cu Kα radiation, λ = 1.54184 Å

Cell parameters from 1071 reflections

θ = 4.0–71.2°

µ = 2.12 mm⁻¹

T = 298 K

Needle, colourless

0.50 × 0.08 × 0.05 mm

Data collection

Rigaku Xcalibur, Ruby

diffractometer

6703 measured reflections

3563 independent reflections

2207 reflections with I > 2σ(I)

Rint = 0.052

θmax = 75.8°, θmin = 4.0°

h = −17→15

k = −9→9

l = −15→19

Tmin = 0.934, Tmax = 1.000

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.054

wR(F²) = 0.151

S = 1.00

3563 reflections

261 parameters

4 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ²(Fo²) + (0.0514P)²]

where P = (Fo² + 2Fc²)/3

(Δ/σ)max = 0.003

Δρmax = 0.23 e Å⁻³

Δρmin = −0.21 e Å⁻³

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sup-8
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) |
|----|------------|------------|------------|-------|
| Cl1| 0.38300 (8)| 0.88404 (15)| 0.76044 (5)| 0.0719 (3) |
| O1W| 0.3293 (2) | 0.4885 (5) | 0.72013 (16)| 0.0732 (8) |
| O2W| 0.4789 (2) | 0.2659 (6) | 0.7806 (2) | 0.0896 (10) |
| N1A| 0.28852 (17)| 0.9176 (4) | 0.34296 (12)| 0.0423 (6) |
| C2A| 0.3708 (2) | 0.8430 (4) | 0.35562 (16)| 0.0440 (7) |
| H2AA| 0.4007 | 0.8052 | 0.3095 | 0.053* |
| N3A| 0.41460 (17)| 0.8174 (4) | 0.42915 (13)| 0.0408 (5) |
| C4A| 0.3713 (2) | 0.8717 (4)| 0.49712 (16)| 0.0396 (6) |
| C4AA| 0.28014 (19)| 0.9559 (4)| 0.48907 (16)| 0.0389 (6) |
| C5A| 0.2313 (2) | 1.0135 (5) | 0.55595 (17)| 0.0484 (7) |
| H5AA| 0.2578 | 1.0010 | 0.6094 | 0.058* |
| C6A| 0.1456 (2) | 1.0874 (5)| 0.5433 (2) | 0.0551 (8) |
| H6AA| 0.1133 | 1.1248 | 0.5881 | 0.066* |
| C7A| 0.1056 (2) | 1.1074 (5)| 0.4628 (2) | 0.0527 (7) |
| H7AA| 0.0468 | 1.1584 | 0.4546 | 0.063* |
| C8A| 0.1520 (2) | 1.0528 (4)| 0.39587 (17)| 0.0469 (7) |
| H8AA| 0.1253 | 1.1066 | 0.3426 | 0.056* |
| C8AA| 0.23995 (19)| 0.9765 (4)| 0.40943 (16)| 0.0385 (6) |
| N9A| 0.41535 (19)| 0.8422 (4)| 0.56770 (14)| 0.0505 (7) |
| N1B| 0.28423 (18)| 0.4265 (4)| 0.32016 (12)| 0.0435 (6) |
| C2B| 0.3657 (2) | 0.3578 (4)| 0.34201 (16)| 0.0447 (7) |
| H2BA| 0.4010 | 0.3203 | 0.2991 | 0.054* |
| N3B| 0.40470 (17)| 0.3348 (4)| 0.41844 (14)| 0.0431 (6) |
| C4B| 0.35638 (19)| 0.3916 (4)| 0.48186 (15)| 0.0382 (6) |
| C4AB| 0.26521 (19)| 0.4695 (4)| 0.46662 (15)| 0.0359 (5) |
| C5B| 0.2086 (2) | 0.5269 (4)| 0.52924 (16)| 0.0426 (6) |
| H5BA| 0.2304 | 0.5181 | 0.5843 | 0.051* |
| C6B| 0.1221 (2) | 0.5954 (4)| 0.50991 (19)| 0.0486 (7) |
| H6BA| 0.0856 | 0.6345 | 0.5516 | 0.058* |
| C7B| 0.0883 (2) | 0.6069 (4)| 0.42737 (19)| 0.0481 (7) |
| H7BA| 0.0290 | 0.6522 | 0.4146 | 0.058* |
| C8B| 0.1419 (2) | 0.5522 (4)| 0.36512 (16)| 0.0439 (7) |
| H8BA| 0.1189 | 0.5615 | 0.3104 | 0.053* |
| C8AB| 0.23112 (19)| 0.4821 (4)| 0.38354 (15)| 0.0383 (6) |
| N9B| 0.39542 (19)| 0.3709 (4)| 0.55724 (14)| 0.0493 (7) |
| H1A| 0.259 (3) | 0.929 (5) | 0.284 (2) | 0.060 (10)* |
| H9AA| 0.472 (3) | 0.789 (5) | 0.569 (2) | 0.062 (11)* |
| H9AB| 0.390 (4) | 0.880 (7) | 0.617 (3) | 0.103 (17)* |
| H9BA| 0.453 (3) | 0.308 (5) | 0.565 (2) | 0.057 (10)* |
### Atomic displacement parameters (Å²)****

| Atom | U₁₁    | U₂₂    | U₃₃    | U₁₂    | U₁₃    | U₂₃    |
|------|--------|--------|--------|--------|--------|--------|
| Cl1  | 0.0881 (7) | 0.0841 (7) | 0.0431 (4) | −0.0031 (6) | 0.0015 (4) | −0.0039 (4) |
| O1W  | 0.082 (2) | 0.081 (2) | 0.0566 (13) | 0.0059 (17) | 0.0021 (13) | −0.0063 (14) |
| O2W  | 0.0612 (18) | 0.097 (3) | 0.109 (2) | 0.0033 (19) | −0.0051 (17) | −0.011 (2) |
| N1A  | 0.0404 (13) | 0.0530 (16) | 0.0329 (10) | −0.0007 (11) | −0.0021 (9) | 0.0007 (10) |
| C2A  | 0.0400 (15) | 0.0517 (18) | 0.0409 (12) | −0.0035 (13) | 0.0075 (11) | −0.0031 (12) |
| N3A  | 0.0333 (11) | 0.0498 (15) | 0.0396 (10) | 0.0055 (10) | 0.0051 (9) | 0.0006 (10) |
| C4A  | 0.0392 (14) | 0.0415 (15) | 0.0382 (12) | −0.0001 (12) | 0.0038 (10) | −0.0021 (11) |
| C4AA | 0.0346 (14) | 0.0380 (15) | 0.0439 (13) | 0.0001 (12) | 0.0013 (10) | 0.0021 (11) |
| C5A  | 0.0524 (18) | 0.0528 (19) | 0.0399 (13) | 0.0034 (15) | 0.0030 (12) | 0.0010 (12) |
| C6A  | 0.0546 (19) | 0.055 (2) | 0.0569 (16) | 0.0085 (16) | 0.0165 (14) | −0.0047 (15) |
| C7A  | 0.0345 (15) | 0.0489 (18) | 0.0751 (19) | 0.0105 (14) | 0.0058 (13) | 0.0000 (16) |
| C8A  | 0.0469 (17) | 0.0473 (18) | 0.0453 (13) | −0.0030 (14) | −0.0075 (12) | 0.0040 (12) |
| C8AA | 0.0377 (14) | 0.0373 (14) | 0.0409 (12) | −0.0032 (12) | 0.0068 (10) | 0.0018 (11) |
| N9A  | 0.0406 (14) | 0.072 (2) | 0.0390 (11) | 0.0133 (13) | 0.0009 (10) | −0.0045 (11) |
| N1B  | 0.0443 (13) | 0.0537 (16) | 0.0320 (9) | 0.0025 (12) | −0.0013 (9) | −0.0011 (10) |
| C2B  | 0.0423 (15) | 0.0527 (18) | 0.0397 (12) | 0.0029 (13) | 0.0060 (11) | −0.0032 (12) |
| N3B  | 0.0338 (12) | 0.0563 (16) | 0.0391 (10) | 0.0066 (11) | 0.0010 (9) | −0.0042 (10) |
| C4B  | 0.0340 (13) | 0.0436 (15) | 0.0368 (11) | −0.0008 (12) | 0.0008 (10) | −0.0041 (11) |
| C4AB | 0.0337 (13) | 0.0346 (14) | 0.0392 (12) | −0.0010 (11) | 0.0011 (10) | 0.0001 (10) |
| C5B  | 0.0418 (15) | 0.0499 (17) | 0.0367 (12) | 0.0016 (13) | 0.0065 (10) | 0.0000 (12) |
| C6B  | 0.0420 (16) | 0.0505 (18) | 0.0546 (15) | 0.0029 (14) | 0.0138 (12) | −0.0033 (14) |
| C7B  | 0.0314 (14) | 0.0487 (18) | 0.0643 (16) | 0.0060 (13) | 0.0031 (12) | 0.0073 (15) |
| C8B  | 0.0398 (15) | 0.0471 (17) | 0.0441 (13) | 0.0003 (13) | −0.0042 (11) | 0.0064 (12) |
| C8AB | 0.0360 (14) | 0.0402 (15) | 0.0388 (12) | −0.0019 (12) | 0.0028 (10) | −0.0012 (11) |
| N9B  | 0.0391 (14) | 0.072 (2) | 0.0366 (11) | 0.0112 (13) | −0.0015 (10) | −0.0058 (12) |

### Geometric parameters (Å, °)****

| Bond/Angle | Distance/Angle |
|------------|----------------|
| O1W—H1W1   | 0.91 (2)       |
| O1W—H2W1   | 0.89 (2)       |
| O2W—H1W2   | 0.91 (2)       |
| O2W—H2W2   | 0.89 (2)       |
| N1A—C2A    | 1.315 (4)      |
| N1A—C8AA   | 1.393 (4)      |
| N1A—H1A    | 1.03 (4)       |
| C2A—N3A    | 1.328 (4)      |
| C2A—H2AA   | 0.9300         |
| N3A—C4A    | 1.363 (4)      |
| C4A—N9A    | 1.293 (4)      |
| Bond/Angle/Distance | Length/Angle (deg) | Bond/Length/Angle (deg) | Length/Angle (deg) |
|---------------------|--------------------|--------------------------|--------------------|
| C4A—C4AA           | 1.455 (4)          | C5B—C6B                 | 1.364 (4)          |
| C4AA—C8AA          | 1.391 (4)          | C5B—H5BA                | 0.9300             |
| C4AA—C5A           | 1.397 (4)          | C6B—C7B                 | 1.399 (4)          |
| C5A—C6A            | 1.356 (5)          | C6B—H6BA                | 0.9300             |
| C5A—H5AA           | 0.9300             | C7B—C8B                 | 1.371 (4)          |
| C6A—C7A            | 1.403 (5)          | C7B—H7BA                | 0.9300             |
| C6A—H6AA           | 0.9300             | C8B—C8AB                | 1.402 (4)          |
| C7A—C8A            | 1.372 (5)          | C8B—H8BA                | 0.9300             |
| C7A—H7AA           | 0.9300             | N9B—H9BA                | 0.95 (4)           |
| C8A—C8AA           | 1.394 (4)          | N9B—H9BB                | 0.80 (4)           |
| C8A—H8AA           | 0.9300             |                          |                    |
| H1W1—O1W—H2W1      | 95 (6)             | C4A—N9A—H9AB            | 120 (3)            |
| H1W2—O2W—H2W2      | 87 (6)             | H9AA—N9A—H9AB           | 120 (4)            |
| C2A—N1A—C8AA       | 120.3 (2)          | C2B—N1B—C8AB            | 116.4 (2)          |
| C2A—N1A—H1A        | 119 (2)            | N1B—C2B—N3B             | 128.1 (3)          |
| C8AA—N1A—H1A       | 120 (2)            | N1B—C2B—H2BA            | 115.9              |
| N1A—C2A—N3A        | 125.0 (3)          | N3B—C2B—H2BA            | 115.9              |
| N1A—C2A—H2AA       | 117.5              | C2B—N3B—C4B             | 117.4 (2)          |
| N3A—C2A—H2AA       | 117.5              | N9B—C4B—N3B             | 117.4 (3)          |
| C2A—N3A—C4A        | 118.0 (2)          | N9B—C4B—C4AB            | 122.3 (3)          |
| N9A—C4A—N3A        | 116.2 (3)          | N3B—C4B—C4AB            | 120.3 (2)          |
| N9A—C4A—C4AA       | 122.9 (3)          | C5B—C4AB—C8AB           | 119.2 (3)          |
| N3A—C4A—C4AA       | 120.8 (2)          | C5B—C4AB—C4B            | 124.1 (2)          |
| C8AA—C4AA—C5A      | 119.2 (3)          | C8AB—C4AB—C4B           | 116.7 (2)          |
| C8AA—C4AA—C4A      | 116.8 (2)          | C6B—C5B—C4AB            | 120.6 (2)          |
| C5A—C4AA—C4A       | 124.0 (2)          | C6B—C5B—H5BA            | 119.7              |
| C6A—C5A—C4AA       | 120.4 (3)          | C4AB—C5B—H5BA           | 119.7              |
| C6A—C5A—H5AA       | 119.8              | C5B—C6B—C7B             | 120.1 (3)          |
| C4AA—C5A—H5AA      | 119.8              | C5B—C6B—H6BA            | 120.0              |
| C5A—C6A—C7A        | 120.0 (3)          | C7B—C6B—H6BA            | 120.0              |
| C5A—C6A—H6AA       | 120.0              | C8B—C7B—C6B             | 120.6 (3)          |
| C7A—C6A—H6AA       | 120.0              | C8B—C7B—H7BA            | 119.7              |
| C8A—C7A—C6A        | 120.9 (3)          | C6B—C7B—H7BA            | 119.7              |
| C8A—C7A—H7AA       | 119.5              | C7B—C8B—C8AB            | 120.3 (2)          |
| C6A—C7A—H7AA       | 119.5              | C7B—C8B—H8BA            | 119.9              |
| C7A—C8A—C8AA       | 118.6 (3)          | C8AB—C8B—H8BA           | 119.9              |
| C7A—C8A—H8AA       | 120.7              | N1B—C8AB—C8B            | 119.7 (2)          |
| C8AA—C8A—H8AA      | 120.7              | N1B—C8AB—C4AB           | 121.1 (3)          |
| C4AA—C8AA—N1A      | 119.0 (3)          | C8B—C8AB—C4AB           | 119.2 (3)          |
| C4AA—C8AA—C8A      | 120.8 (3)          | C4B—N9B—H9BA            | 119 (2)            |
| N1A—C8AA—C8A       | 120.2 (2)          | C4B—N9B—H9BB            | 120 (3)            |
| C4A—N9A—H9AA       | 120 (2)            | H9BA—N9B—H9BB           | 120 (3)            |
| C8AA—N1A—C2A—N3A   | -0.2 (5)           | C8AB—N1B—C2B—N3B       | -0.2 (5)           |
| N1A—C2A—N3A—C4A    | 0.2 (5)            | N1B—C2B—N3B—C4B        | -1.5 (5)           |
| C2A—N3A—C4A—N9A    | 179.0 (3)          | C2B—N3B—C4B—N9B        | -179.1 (3)         |
| C2A—N3A—C4A—C4AA   | -0.3 (4)           | C2B—N3B—C4B—C4AB       | 1.6 (5)            |
N9A—C4A—C4AA—C8AA $-178.8 \pm 3$ N9B—C4B—C4AB—C5B $-1.5 \pm 5$
N3A—C4A—C4AA—C8AA 0.4 (4) N3B—C4B—C4AB—C5B 177.8 (3)
N9A—C4A—C4AA—C5A 0.2 (5) N9B—C4B—C4AB—C8AB $-179.3 \pm 3$
N3A—C4A—C4AA—C5A 179.4 (3) N3B—C4B—C4AB—C8AB 0.0 (4)
C8AA—C4AA—C5A—C6A 0.4 (5) C8AB—C4AB—C5B—C6B 0.6 (5)
C4A—C4AA—C5A—C6A $-178.5 \pm 3$ C4B—C4AB—C5B—C6B $-178.4 \pm 3$
C4AA—C5A—C6A—C7A $-0.4 \pm 6$ C4AB—C5B—C6B—C7B 0.9 (5)
C5A—C6A—C7A—C8A 0.1 (6) C5B—C6B—C7B—C8B $-0.9 \pm 5$
C5A—C4AA—C8AA—N1A 179.5 (3) C5B—C4AB—C8AB—N1B $-179.7 \pm 3$
C4A—C4AA—C8AA—N1A $-179.5 \pm 3$ C2B—N1B—C8AB—C8B $-178.1 \pm 3$
C4A—C4AA—C8AA—N1A $-0.4 \pm 4$ C2B—N1B—C8AB—C4AB 1.9 (4)
C5A—C4AA—C8AA—C8A $-0.2 \pm 5$ C7B—C8B—C8AB—N1B 179.7 (3)
C4A—C4AA—C8AA—C8A 178.8 (3) C7B—C8B—C8AB—C4AB $-0.3 \pm 5$
C2A—N1A—C8AA—C4AA 0.3 (4) C5B—C4AB—C8AB—N1B $-179.7 \pm 3$
C2A—N1A—C8AA—C8A $-178.9 \pm 3$ C4B—C4AB—C8AB—N1B $-1.8 \pm 4$
C7A—C8A—C8AA—C4AA 0.0 (5) C5B—C4AB—C8AB—C8B 0.3 (4)
C7A—C8A—C8AA—N1A 179.2 (3) C4B—C4AB—C8AB—C8B 178.2 (3)

**Hydrogen-bond geometry (Å, °)**

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|------|---------|
| N1A—H1A···N1B$^i$ | 1.03 (3) | 1.76 (3) | 2.786 (3) | 173 (3) |
| N9A—H9A···N3B$^ii$ | 0.91 (4) | 2.00 (4) | 2.907 (4) | 175 (3) |
| N9A—H9AB···C11 | 0.94 (5) | 2.34 (5) | 3.206 (2) | 153 (5) |
| N9B—H9B···N3A$^ii$ | 0.96 (4) | 2.12 (4) | 3.074 (4) | 174 (3) |
| N9B—H9BB···O1W | 0.79 (4) | 2.22 (3) | 2.999 (4) | 167 (4) |
| O1W—H1W1···C11 | 0.90 (3) | 2.25 (3) | 3.157 (4) | 178 (6) |
| O1W—H2W1···C11$^ii$ | 0.89 (4) | 2.37 (4) | 3.183 (3) | 151 (7) |
| O2W—H1W2···O1W | 0.91 (7) | 1.96 (7) | 2.857 (5) | 169 (6) |
| O2W—H2W2···C11$^iv$ | 0.89 (5) | 2.40 (5) | 3.215 (4) | 153 (5) |

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

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*Acta Cryst.* (2021). E77, 989-993 sup-12