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

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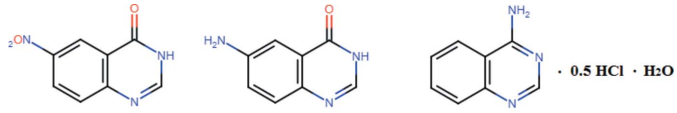
The title compounds, 6-nitroquinazolin-4(3*H*)-one ($C_8H_5N_3O_3$; **I**), 6-aminoquinazolin-4(3*H*)-one ($C_8H_7N_3O$; **II**) and 4-aminoquinazolin-1-ium chloride–4-aminoquinazoline–water (1/1/2), ($C_8H_8N_3^+\cdot Cl^-\cdot C_8H_7N_3\cdot 2H_2O$; **III**) were synthesized and their structures were 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), anti-tuberculosis (Nandy *et al.*, 2006), antihypertension (Hess *et al.*, 1968) and antidiabetic (Paneersalvam *et al.*, 2010) activities.



I II III

In line with this, we synthesized 6-nitroquinazolin-4(3*H*)-one (**I**), 6-aminoquinazolin-4(3*H*)-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

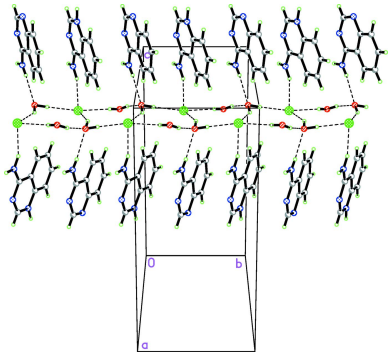


Table 1
Selected bond lengths (Å) for **I**.

N1—C2	1.287 (3)	N3—C4	1.366 (3)
C2—N3	1.354 (3)	C6—N9	1.464 (3)

Table 2
Selected bond lengths (Å) for **II**.

N1*A*—C2*A*	1.291 (5)	N1*B*—C2*B*	1.290 (5)
C2*A*—N3*A*	1.369 (4)	C2*B*—N3*B*	1.364 (4)
N3*A*—C4*A*	1.376 (4)	N3*B*—C4*B*	1.366 (4)
C6*A*—N9*A*	1.374 (4)	C6*B*—N9*B*	1.392 (5)

Table 3
Selected bond lengths (Å) for **III**.

N1*A*—C2*A*	1.315 (4)	N1*B*—C2*B*	1.309 (4)
C2*A*—N3*A*	1.328 (4)	C2*B*—N3*B*	1.340 (4)
N3*A*—C4*A*	1.363 (4)	N3*B*—C4*B*	1.347 (4)
C4*A*—N9*A*	1.293 (4)	C4*B*—N9*B*	1.323 (4)

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)°, 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 $Pca2_1$ 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° in molecules *A* and *B*, respectively. All bond lengths and angles

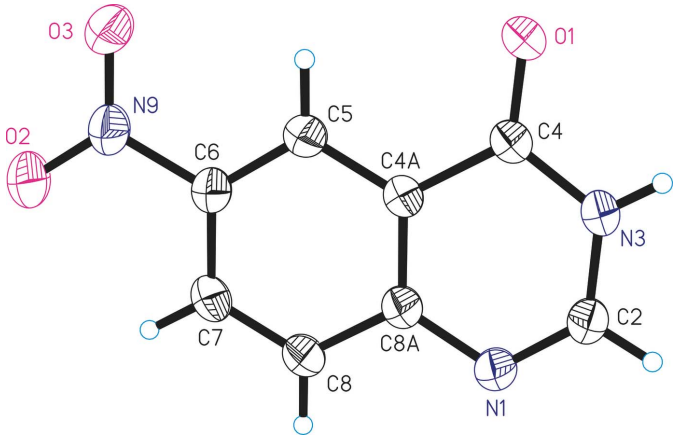


Figure 1
The molecular structure of 6-nitroquinazolin-4(3*H*)-one (**I**), with displacement ellipsoids drawn at the 50% probability level.

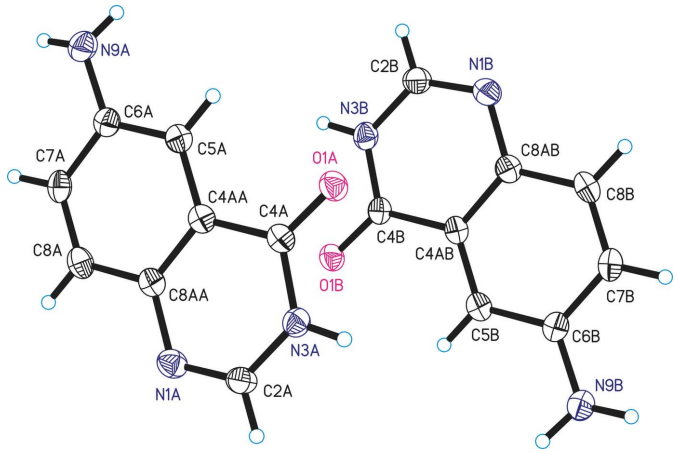


Figure 2
The molecular structure of 6-aminoquinazolin-4(3*H*)-one (**II**), showing the two independent molecules, with displacement ellipsoids drawn at the 50% probability level.

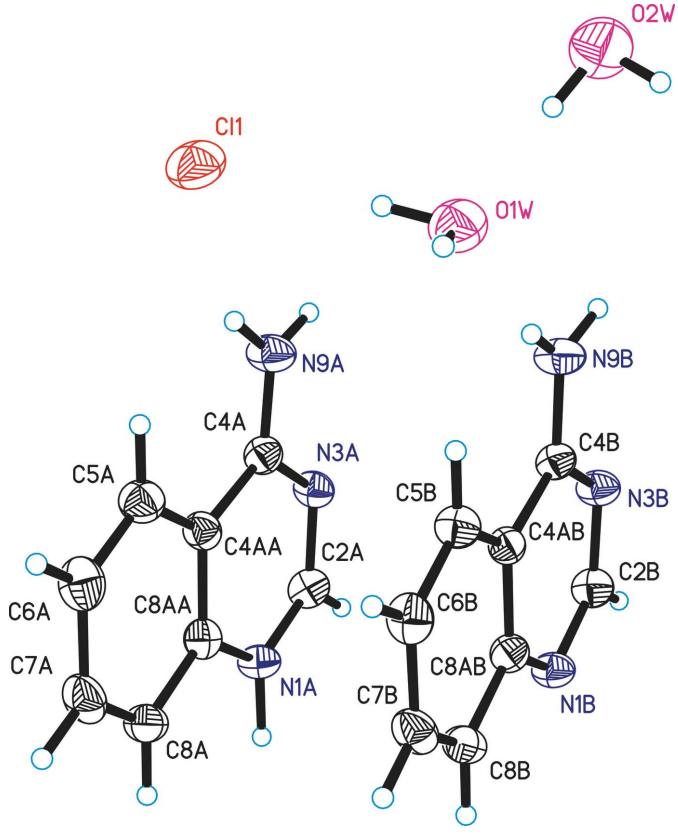


Figure 3
The asymmetric unit of compound **III** with displacement ellipsoids drawn at the 50% probability level.

Table 4
Hydrogen-bond geometry (Å, °) for **I**.

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O1^{i}	0.80 (3)	2.02 (3)	2.814 (2)	178 (4)
C8—H8···N1^{ii}	0.93	2.53	3.450 (3)	172
C2—H2···O2^{iii}	0.93	2.57	3.466 (4)	163
C7—H7···O2^{iv}	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) $-x+1, -y+2, -z+2$.

Table 5
Hydrogen-bond geometry (Å, °) for **II**.

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

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

are normal. Selected geometric parameters are listed in Table 2.

In the case of compound **III**, there are protonated (A) and unprotonated (B) 4-aminoquinazoline molecules (Fig. 3) in the asymmetric unit and they both have a planar structure. Molecule A is protonated at the N1 nitrogen atom and this leads to an elongation of the N1—C2 and N3—C4 bonds and a shortening of the C2—N3 and C4—N9 bonds with respect to the unprotonated molecule B. In both A and B, the nitrogen atom of the amino group is in an sp^2 hybridized state. The sum of the valence angles around the nitrogen atoms in molecules A and B are 360 and 359°, respectively, and the carbon-to-amino group nitrogen bond lengths C4—N9 are shorter than the bond lengths observed in compound **II** (Table 3).

3. Supramolecular features

In the crystal of **I**, intermolecular N—H···O hydrogen bonds link the molecules into centrosymmetric dimers, forming $R_2^2(8)$ motifs. Other head-to-head $R_2^2(10)$ and $R_2^2(8)$ motifs are formed by weak intermolecular C—H···O and C—H···N hydrogen bonds, producing layers parallel to the $(1\bar{1}2)$ plane (Table 4, Fig. 4). In addition, an $R_3^2(8)$ ring motif is formed by the interactions between three adjacent molecules. The layers are linked though π–π stacking interactions with centroid–centroid distances of 3.8264 (13) and 3.9600 (14) Å into a three-dimensional network.

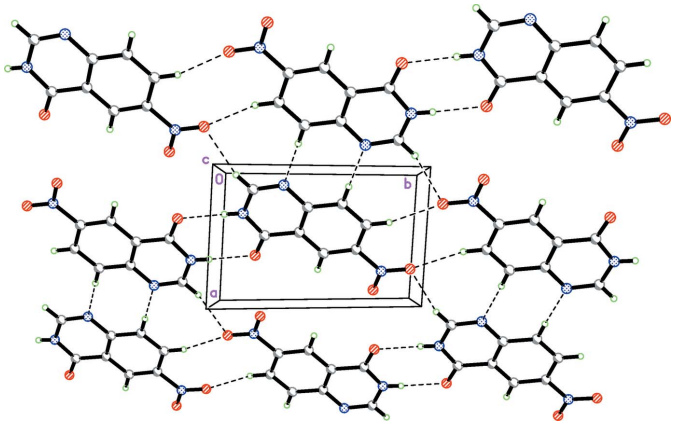


Figure 4
Hydrogen bonding in the crystal of 6-nitroquinazolin-4(3*H*)-one (**I**). Colour code: C grey, H green, N blue, O red.

The two independent molecules of compound **II** are related by a pseudo-center of symmetry and are linked by two N—H···O hydrogen bonds, forming an $R_2^2(8)$ motif. An N—H···N hydrogen bond generates a three-dimensional network (Table 5, Fig. 5).

The packing analysis of **III** shows that the protonated and unprotonated 4-aminoquinazoline molecules are linked by intermolecular N—H···N hydrogen bonds, forming pseudo-centrosymmetric dimers characterized by a donor–acceptor distance of 2.786 (3) Å. Other N—H···N hydrogen bonds form centrosymmetric $R_2^2(8)$ 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* N—H···Cl and N—H···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 *self-assembly* of molecules has also been observed in other quinazoline hydrochloride crystals

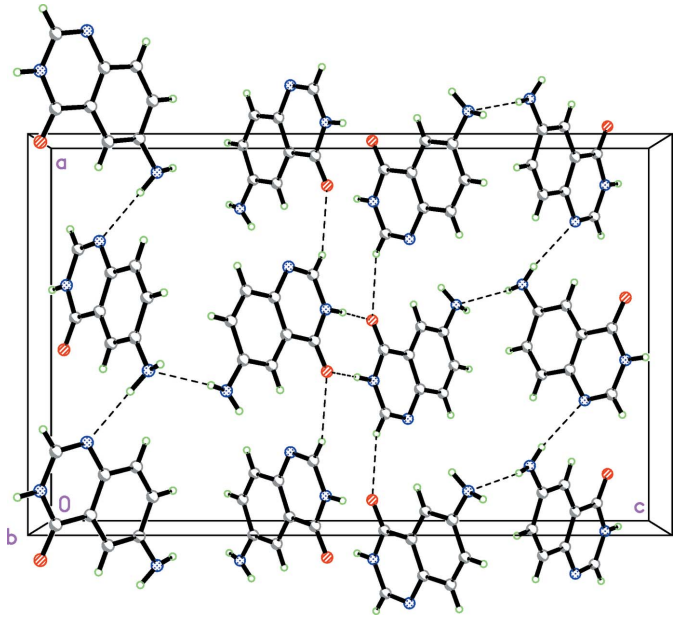


Figure 5
Hydrogen bonding in the crystal of 6-aminoquinazolin-4(3*H*)-one (**II**). Colour code: C grey, H green, N blue, O red.

Table 6
Hydrogen-bond geometry (Å, °) for **III**.

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—H9AA···N3B^{ii}	0.91 (4)	2.00 (4)	2.907 (4)	175 (3)
N9A—H9AB···Cl1	0.94 (5)	2.34 (5)	3.206 (2)	153 (5)
N9B—H9BA···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···Cl1	0.90 (3)	2.25 (3)	3.157 (4)	178 (6)
O1W—H2W1···Cl1^{iii}	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···Cl1^{iv}	0.89 (5)	2.40 (5)	3.215 (4)	153 (5)

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

(Tashkhodzhaev *et al.*, 1995; Turgunov *et al.*, 1998, 2003). The above mentioned N—H···N hydrogen bonds link the molecules into a three-dimensional network. The crystal structure of **III** is stabilized by π–π interactions [centroid–centroid distances in the range 3.4113 (16)–3.9080 (18) Å].

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(3*H*)-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(3*H*)-one and its first metal coordination compound have also been reported [BIHJIO (Liao *et al.*, 2018) and NALFEN (Turgunov & Englert, 2010)].

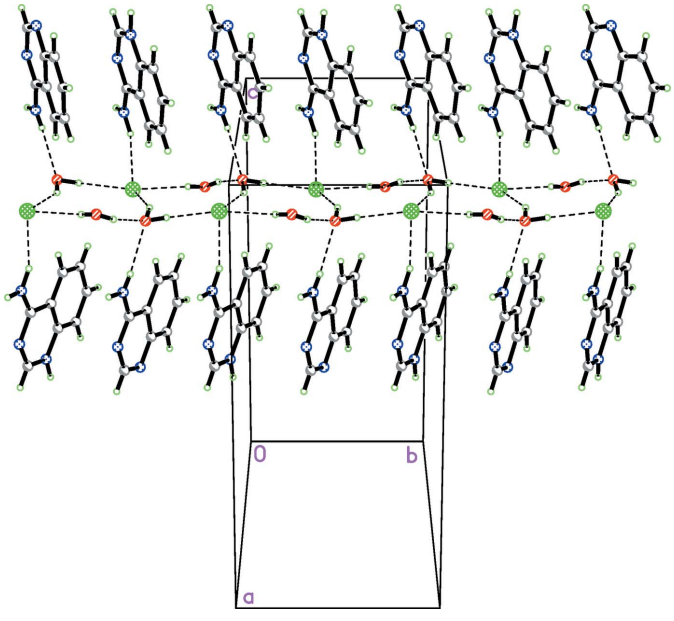


Figure 6
Part of the crystal structure of **III** showing the hydrogen-bonding scheme. Colour code: C grey, H light green, Cl bright green, N blue, O red.

5. Synthesis and crystallization

Compound I: In a three-necked flask equipped with a mechanical stirrer and reflux condenser, quinazolin-4(3*H*)-one (22.5 g, 0.15 mol) was dissolved in 78 ml of concentrated sulfuric acid at 303 K for 1 h. Then a nitrating 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 ($SnCl_2{\cdot}2H_2O$) 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 20 ml of ethanol and 7 ml of HCl (36%) were added portionwise with stirring of the mixture. The reaction was carried out for 10-15 minutes at ∼273 K, 30 min at room temperature and 2 h in a water bath (∼363 K). The reaction mixture was left overnight at room temperature, diluted with water, and brought to a strongly alkaline medium (pH = 10–11) with 10% of sodium hydroxide, in which the expected 6-amino-3*N*-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-chloro-quinazoline 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 U_{iso}(H) = 1.2U_{eq}(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

X-ray diffraction studies were performed at the Centre of Collective Usage of Equipment of the Institute of Bioorganic Chemistry of the Uzbekistan Academy of Sciences. Professor Bakhtiyar Ibragimov is acknowledged for support with the diffraction measurements.

Table 7
Experimental details.

	I	II	III
Crystal data			
Chemical formula	$C_8H_5N_3O_3$	$C_8H_7N_3O$	$C_8H_8N_3^+\cdot Cl^-\cdot C_8H_7N_3\cdot 2H_2O$
M_r	191.15	161.17	362.82
Crystal system, space group	Triclinic, $P\overline{1}$	Orthorhombic, $Pca2_1$	Monoclinic, $P2_1/n$
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 (Å^3)	401.45 (11)	1439.99 (10)	1763.5 (2)
Z	2	8	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Cu $K\alpha$
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*; Rigaku OD, 2018)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.742, 1.000	0.720, 1.000	0.934, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2652, 1598, 1124	22195, 2976, 2489	6703, 3563, 2207
R_{int}	0.024	0.070	0.052
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.630	0.630	0.629
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, 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
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (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)	–

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*), *XP* in *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

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supporting information

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Crystal structures of 6-nitroquinazolin-4(3*H*)-one, 6-aminoquinazolin-4(3*H*)-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(3*H*)-one (I)

Crystal data

$C_8H_5N_3O_3$
M_r = 191.15
Triclinic, $P\overline{1}$
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) Å^3
Z = 2
$F(000)$ = 196
D_x = 1.581 Mg m^{-3}
Melting point: 560(2) K
Cu $K\alpha$ 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)
T_{min} = 0.742, T_{max} = 1.000
2652 measured reflections
1598 independent reflections
1124 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 76.3°, θ_{min} = 5.3°
h = −6→7
k = −7→10
l = −10→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 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
$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: SHELXL2014/7 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
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 (Å^2)

	x	*y*	*z*	U_{iso}*/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)	0.7678 (3)	0.0635 (5)
H3	0.334 (5)	0.038 (3)	0.598 (3)	0.057 (6)*

Atomic displacement parameters (Å^2)

	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)

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, º)

O1—C4	1.233 (2)	C4A—C5	1.395 (3)
O2—N9	1.218 (2)	C4A—C8A	1.399 (3)
O3—N9	1.223 (2)	C5—C6	1.374 (3)
N1—C2	1.287 (3)	C5—H5	0.9300
N1—C8A	1.388 (3)	C6—C7	1.395 (3)
C2—N3	1.354 (3)	C6—N9	1.464 (3)
C2—H2	0.9300	C7—C8	1.363 (3)
N3—C4	1.366 (3)	C7—H7	0.9300
N3—H3	0.80 (3)	C8—C8A	1.412 (3)
C4—C4A	1.463 (3)	C8—H8	0.9300
C2—N1—C8A	115.80 (17)	C5—C6—C7	122.54 (19)
N1—C2—N3	125.57 (19)	C5—C6—N9	118.89 (18)
N1—C2—H2	117.2	C7—C6—N9	118.57 (18)
N3—C2—H2	117.2	C8—C7—C6	119.31 (18)
C2—N3—C4	123.57 (17)	C8—C7—H7	120.3
C2—N3—H3	122.0 (18)	C6—C7—H7	120.3
C4—N3—H3	114.3 (18)	C7—C8—C8A	120.21 (19)
O1—C4—N3	122.34 (17)	C7—C8—H8	119.9
O1—C4—C4A	124.23 (18)	C8A—C8—H8	119.9
N3—C4—C4A	113.43 (16)	N1—C8A—C4A	122.73 (18)
C5—C4A—C8A	120.89 (18)	N1—C8A—C8	118.18 (18)
C5—C4A—C4	120.21 (17)	C4A—C8A—C8	119.09 (19)
C8A—C4A—C4	118.90 (18)	O2—N9—O3	122.9 (2)
C6—C5—C4A	117.95 (18)	O2—N9—C6	118.0 (2)
C6—C5—H5	121.0	O3—N9—C6	119.03 (18)
C4A—C5—H5	121.0		
C8A—N1—C2—N3	−0.1 (4)	C6—C7—C8—C8A	−0.5 (4)
N1—C2—N3—C4	−0.7 (4)	C2—N1—C8A—C4A	0.5 (3)
C2—N3—C4—O1	−178.3 (2)	C2—N1—C8A—C8	−179.8 (2)
C2—N3—C4—C4A	1.0 (3)	C5—C4A—C8A—N1	−179.2 (2)
O1—C4—C4A—C5	−2.2 (3)	C4—C4A—C8A—N1	0.0 (3)
N3—C4—C4A—C5	178.5 (2)	C5—C4A—C8A—C8	1.1 (3)
O1—C4—C4A—C8A	178.6 (2)	C4—C4A—C8A—C8	−179.8 (2)
N3—C4—C4A—C8A	−0.7 (3)	C7—C8—C8A—N1	180.0 (2)
C8A—C4A—C5—C6	−1.0 (3)	C7—C8—C8A—C4A	−0.2 (3)
C4—C4A—C5—C6	179.8 (2)	C5—C6—N9—O2	−175.0 (2)
C4A—C5—C6—C7	0.3 (3)	C7—C6—N9—O2	4.9 (4)
C4A—C5—C6—N9	−179.9 (2)	C5—C6—N9—O3	6.0 (3)
C5—C6—C7—C8	0.5 (4)	C7—C6—N9—O3	−174.2 (2)
N9—C6—C7—C8	−179.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O1^{i}	0.80 (3)	2.02 (3)	2.814 (2)	178 (4)
C8—H8···N1^{ii}	0.93	2.53	3.450 (3)	172
C2—H2···O2^{iii}	0.93	2.57	3.466 (4)	163
C7—H7···O2^{iv}	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(3*H*)-one (II)

Crystal data

$C_8H_7N_3O$
M_r = 161.17
Orthorhombic, $Pca2_1$
a = 13.4535 (5) Å
b = 4.9510 (2) Å
c = 21.6188 (8) Å
V = 1439.99 (10) Å^3
Z = 8
$F(000)$ = 672
D_x = 1.487 Mg m^{-3}
Melting point: 589(2) K
Cu $K\alpha$ 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
0.60 × 0.45 × 0.35 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)
T_{min} = 0.720, T_{max} = 1.000
22195 measured reflections
2976 independent reflections
2489 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 76.1°, θ_{min} = 4.1°
h = −16→16
k = −6→6
l = −26→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 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
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.144P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: SHELXL-2014/7 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0034 (4)
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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
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)
H8A	0.6763	0.5982	0.3198	0.049*
C8AA	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)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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)

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, º)

O1A—C4A	1.235 (4)	O1B—C4B	1.239 (4)
N1A—C2A	1.291 (5)	N1B—C2B	1.290 (5)
N1A—C8AA	1.397 (4)	N1B—C8AB	1.391 (4)
C2A—N3A	1.369 (4)	C2B—N3B	1.364 (4)
C2A—H2A	0.9300	C2B—H2B	0.9300
N3A—C4A	1.376 (4)	N3B—C4B	1.366 (4)
N3A—H3A	0.94 (3)	N3B—H3B	0.92 (2)
C4A—C4AA	1.457 (4)	C4B—C4AB	1.461 (5)
C4AA—C5A	1.397 (4)	C4AB—C5B	1.393 (5)
C4AA—C8AA	1.405 (4)	C4AB—C8AB	1.408 (4)
C5A—C6A	1.395 (5)	C5B—C6B	1.387 (5)
C5A—H5A	0.9300	C5B—H5B	0.9300
C6A—N9A	1.374 (4)	C6B—N9B	1.392 (5)
C6A—C7A	1.417 (5)	C6B—C7B	1.413 (5)
C7A—C8A	1.365 (5)	C7B—C8B	1.370 (5)
C7A—H7A	0.9300	C7B—H7B	0.9300
C8A—C8AA	1.403 (5)	C8B—C8AB	1.400 (5)
C8A—H8A	0.9300	C8B—H8B	0.9300
N9A—H9AA	0.93 (4)	N9B—H9BB	0.90 (5)
N9A—H9AB	0.81 (4)	N9B—H9BA	0.93 (5)
C2A—N1A—C8AA	116.3 (3)	C2B—N1B—C8AB	116.6 (3)
N1A—C2A—N3A	124.8 (3)	N1B—C2B—N3B	124.9 (3)
N1A—C2A—H2A	117.6	N1B—C2B—H2B	117.6

N3A—C2A—H2A	117.6	N3B—C2B—H2B	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)	N9B—C6B—C7B	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)
C8A—C8AA—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)

C8AA—N1A—C2A—N3A	−0.4 (5)	C8AB—N1B—C2B—N3B	−0.9 (5)
N1A—C2A—N3A—C4A	0.2 (5)	N1B—C2B—N3B—C4B	1.6 (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)

C5A—C4AA—C8AA—N1A	179.8 (3)	C5B—C4AB—C8AB—N1B	−178.8 (3)
C4A—C4AA—C8AA—N1A	−0.5 (4)	C4B—C4AB—C8AB—N1B	1.2 (5)
C5A—C4AA—C8AA—C8A	−0.3 (4)	C5B—C4AB—C8AB—C8B	0.4 (4)
C4A—C4AA—C8AA—C8A	179.4 (3)	C4B—C4AB—C8AB—C8B	−179.6 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N9*A*—H9*AA*···N9*B*^{i}	0.93 (4)	2.55 (4)	3.435 (5)	160 (4)
N9*A*—H9*AB*···N1*A*^{ii}	0.81 (4)	2.34 (4)	3.144 (5)	170 (4)
N9*B*—H9*BB*···N1*B*^{iii}	0.91 (4)	2.19 (4)	3.092 (5)	174 (4)
N3*A*—H3*A*···O1*B*^{iv}	0.95 (3)	1.89 (3)	2.832 (4)	175 (3)
N3*B*—H3*B*···O1*A*^{v}	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

$C_8H_8N_3^+·Cl^-·C_8H_7N_3·2H_2O$
M_r = 362.82
Monoclinic, $P2_1/n$
a = 14.3512 (12) Å
b = 7.5867 (6) Å
c = 16.2282 (9) Å
β = 93.544 (7)°
V = 1763.5 (2) Å^3
Z = 4
$F(000)$ = 760
D_x = 1.367 Mg m^{-3}
Cu $K\alpha$ 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
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)
T_{min} = 0.934, T_{max} = 1.000
6703 measured reflections
3563 independent reflections
2207 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 75.8°, θ_{min} = 4.0°
h = −17→15
k = −9→9
l = −15→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 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
$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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_{iso}*/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	1.0664	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)*

H9BB	0.370 (3)	0.406 (5)	0.596 (2)	0.047 (9)*
H1W1	0.345 (4)	0.601 (4)	0.733 (3)	0.11 (2)*
H2W1	0.271 (2)	0.497 (11)	0.736 (4)	0.19 (4)*
H1W2	0.437 (5)	0.346 (8)	0.759 (5)	0.190*
H2W2	0.437 (4)	0.183 (7)	0.766 (4)	0.15 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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, º)

O1W—H1W1	0.91 (2)	N9A—H9AA	0.91 (4)
O1W—H2W1	0.89 (2)	N9A—H9AB	0.94 (5)
O2W—H1W2	0.91 (2)	N1B—C2B	1.309 (4)
O2W—H2W2	0.89 (2)	N1B—C8AB	1.383 (4)
N1A—C2A	1.315 (4)	C2B—N3B	1.340 (4)
N1A—C8AA	1.393 (4)	C2B—H2BA	0.9300
N1A—H1A	1.03 (4)	N3B—C4B	1.347 (4)
C2A—N3A	1.328 (4)	C4B—N9B	1.323 (4)
C2A—H2AA	0.9300	C4B—C4AB	1.443 (4)
N3A—C4A	1.363 (4)	C4AB—C5B	1.408 (4)
C4A—N9A	1.293 (4)	C4AB—C8AB	1.409 (4)

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 (3)	N9B—C4B—C4AB—C5B	−1.5 (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 (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 (3)	C4B—C4AB—C5B—C6B	−178.4 (3)
C4AA—C5A—C6A—C7A	−0.4 (6)	C4AB—C5B—C6B—C7B	0.9 (5)
C5A—C6A—C7A—C8A	0.1 (6)	C5B—C6B—C7B—C8B	−0.9 (5)
C6A—C7A—C8A—C8AA	0.0 (5)	C6B—C7B—C8B—C8AB	0.6 (5)
C5A—C4AA—C8AA—N1A	−179.5 (3)	C2B—N1B—C8AB—C8B	−178.1 (3)
C4A—C4AA—C8AA—N1A	−0.4 (4)	C2B—N1B—C8AB—C4AB	1.9 (4)
C5A—C4AA—C8AA—C8A	−0.2 (5)	C7B—C8B—C8AB—N1B	179.7 (3)
C4A—C4AA—C8AA—C8A	178.8 (3)	C7B—C8B—C8AB—C4AB	−0.3 (5)
C2A—N1A—C8AA—C4AA	0.3 (4)	C5B—C4AB—C8AB—N1B	−179.7 (3)
C2A—N1A—C8AA—C8A	−178.9 (3)	C4B—C4AB—C8AB—N1B	−1.8 (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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H1*A*···N1*B*^{i}	1.03 (3)	1.76 (3)	2.786 (3)	173 (3)
N9*A*—H9*AA*···N3*B*^{ii}	0.91 (4)	2.00 (4)	2.907 (4)	175 (3)
N9*A*—H9*AB*···Cl1	0.94 (5)	2.34 (5)	3.206 (2)	153 (5)
N9*B*—H9*BA*···N3*A*^{ii}	0.96 (4)	2.12 (4)	3.074 (4)	174 (3)
N9*B*—H9*BB*···O1*W*	0.79 (4)	2.22 (3)	2.999 (4)	167 (4)
O1*W*—H1*W*1···Cl1	0.90 (3)	2.25 (3)	3.157 (4)	178 (6)
O1*W*—H2*W*1···Cl1^{iii}	0.89 (4)	2.37 (4)	3.183 (3)	151 (7)
O2*W*—H1*W*2···O1*W*	0.91 (7)	1.96 (7)	2.857 (5)	169 (6)
O2*W*—H2*W*2···Cl1^{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*.