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Hexaaquacobalt(II) tetraaquabis(2-aminopyrazine-κN^4)cobalt(II) disulfate dihydrate

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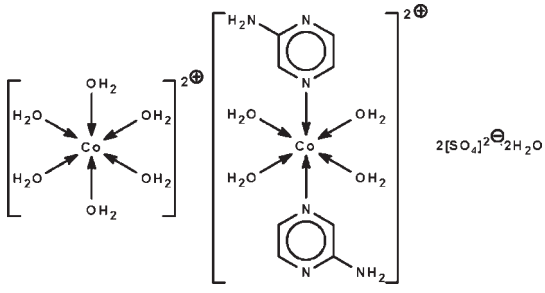
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 13.3.

The reaction of cobalt(II) sulfate and 2-aminopyrazine affords the title salt, $[Co(H_2O)_6][Co(C_4H_5N_3)_2(H_2O)_4](SO_4)_2{\cdot}2H_2O$. The metal atoms in the tetraaqua-coordinated and hexaaqua-coordinated complex cations lie on centers of inversion in slightly distorted octahedral geometries. The cations, anions and solvent water molecules are linked by O—H···O, O—H···N and N—H···O hydrogen bonds into a three-dimensional network.

Related literature

The reaction of cobalt(II) chloride and 3-aminopyrazine yields tetrakis(3-aminopyrazine)dichloridocobalt(II); see: Csöregh *et al.* (2000); Kang *et al.* (2009).



Experimental

Crystal data

$[Co(H_2O)_6][Co(C_4H_5N_3)_2(H_2O)_4](SO_4)_2{\cdot}2H_2O$
M_r = 716.40
Triclinic, $P\overline{1}$
a = 6.5722 (3) Å
b = 8.3264 (4) Å
c = 13.2337 (7) Å
α = 75.732 (2)°
β = 78.571 (1)°
γ = 78.795 (1)°
V = 679.81 (6) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.47 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.668, T_{max} = 0.758
6692 measured reflections
3071 independent reflections
2762 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.05
3071 reflections
231 parameters
14 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1w—H1w1···O1	0.84 (1)	1.93 (1)	2.755 (2)	168 (3)
O1w—H1w2···N2^{i}	0.85 (1)	1.95 (1)	2.795 (2)	175 (3)
O2w—H2w1···O3	0.84 (1)	1.94 (1)	2.769 (2)	170 (2)
O2w—H2w2···O1^{ii}	0.84 (1)	1.93 (1)	2.765 (2)	170 (3)
O3w—H3w1···O2	0.85 (1)	1.91 (1)	2.743 (2)	169 (3)
O3w—H3w2···O6w	0.85 (1)	1.89 (1)	2.730 (2)	170 (3)
O4w—H4w1···O6w^{iii}	0.85 (1)	1.95 (1)	2.781 (2)	168 (3)
O4w—H4w2···O2^{iii}	0.85 (1)	1.91 (1)	2.745 (2)	167 (2)
O5w—H5w1···O3^{iv}	0.85 (1)	1.98 (1)	2.816 (2)	170 (3)
O5w—H5w2···O4^{v}	0.84 (1)	1.90 (1)	2.737 (2)	174 (3)
O6w—H6w1···O3^{i}	0.85 (1)	1.94 (1)	2.782 (2)	171 (4)
O6w—H6w2···O4^{iv}	0.85 (1)	1.89 (1)	2.711 (2)	164 (3)
N3—H3n2···O1^{vi}	0.85 (1)	2.20 (1)	3.036 (2)	168 (3)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2657).

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supporting information

Acta Cryst. (2009). E**65**, m1503 [doi:10.1107/S1600536809045310]

Hexaaquacobalt(II) tetraaquabis(2-aminopyrazine-κN^4)cobalt(II) disulfate dihydrate

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S1. Experimental

To an aqueous solution of 3-aminopyrazine (0.19 g, 2 mmol) was added cobalt(II) sulfate heptahydrate (0.56 g, 2 mmol). Red crystals of the salt separated from the solution after a few days. CH&N elemental analysis. Calc. for $C_8H_{34}N_6O_{20}S_2Co_2$: C 13.41, H 4.78, N 11.73%; found: C 13.39, H 4.72, N 11.76%.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U(C). The amino and water H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H = O–H = 0.85±0.01 Å; their temperature factors were refined.

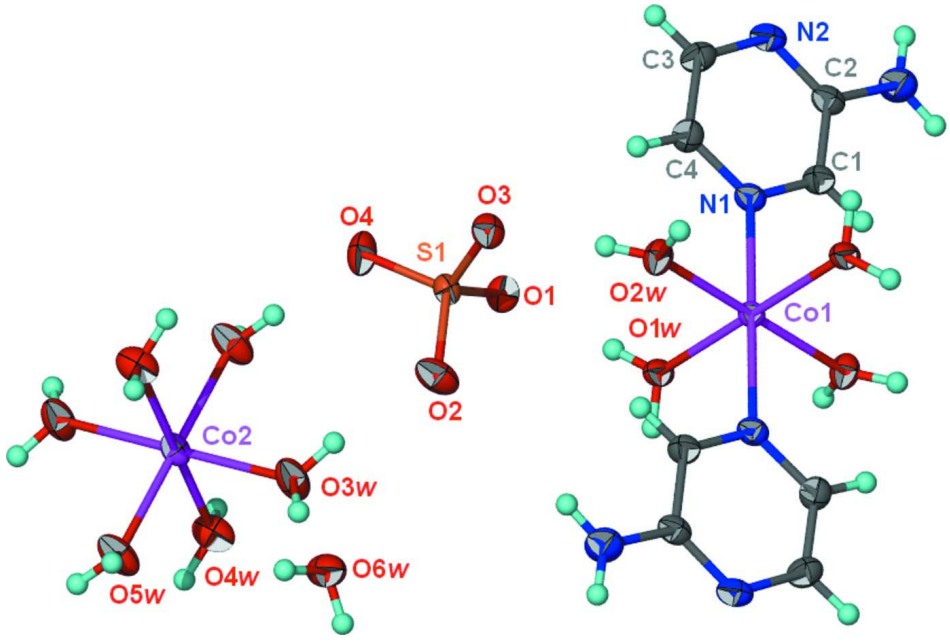

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[Co(H_2O)_6]$ $[Co(H_2O)_4(C_4H_5N_3)_2]$ $2[SO_4]\cdot 2H_2O$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

Hexaaquacobalt(II) tetraaquabis(2-aminopyrazine-κN^4)cobalt(II) disulfate dihydrate

Crystal data

$[Co(H_2O)_6][Co(C_4H_5N_3)_2(H_2O)_4](SO_4)_2\cdot 2H_2O$
$M_r = 716.40$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.5722$ (3) Å
$b = 8.3264$ (4) Å
$c = 13.2337$ (7) Å
$\alpha = 75.732$ (2)°
$\beta = 78.571$ (1)°
$\gamma = 78.795$ (1)°
$V = 679.81$ (6) Å^3
$Z = 1$
$F(000) = 370$
$D_x = 1.750$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6326 reflections
$\theta = 3.2$–$27.5°$
$\mu = 1.47$ mm^{-1}
$T = 293$ K
Prism, red
0.30 × 0.20 × 0.20 mm

Data collection

Rigaku RAXIS-RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.668$, $T_{max} = 0.758$
6692 measured reflections
3071 independent reflections
2762 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.2°$
$h = -7\rightarrow 8$
$k = -10\rightarrow 10$
$l = -17\rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.085$
$S = 1.05$
3071 reflections
231 parameters
14 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 0.1887P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Co1	1.0000	1.0000	0.5000	0.02058 (10)
Co2	0.0000	0.5000	1.0000	0.02713 (11)
S1	0.48109 (7)	0.88842 (5)	0.80066 (3)	0.02386 (12)
O1	0.4133 (2)	0.98162 (18)	0.70001 (10)	0.0309 (3)
O2	0.5369 (3)	0.70948 (18)	0.79992 (14)	0.0439 (4)
O3	0.6685 (2)	0.95119 (18)	0.81446 (11)	0.0317 (3)

O4	0.3094 (2)	0.9161 (2)	0.88747 (12)	0.0410 (4)
O1W	0.7178 (2)	0.91396 (17)	0.53377 (11)	0.0295 (3)
O2W	1.0213 (2)	0.96509 (18)	0.66021 (11)	0.0305 (3)
O3W	0.2836 (2)	0.4730 (2)	0.90039 (14)	0.0435 (4)
O4W	−0.1433 (3)	0.4747 (2)	0.87641 (13)	0.0399 (4)
O5W	0.0399 (2)	0.23895 (19)	1.05544 (14)	0.0421 (4)
O6W	0.6169 (3)	0.2195 (2)	0.91230 (13)	0.0397 (3)
N1	0.8413 (2)	1.25974 (19)	0.49825 (13)	0.0257 (3)
N2	0.7121 (3)	1.6016 (2)	0.49242 (14)	0.0302 (3)
N3	0.7876 (3)	1.6554 (2)	0.30956 (16)	0.0423 (4)
C1	0.8491 (3)	1.3722 (2)	0.40789 (15)	0.0289 (4)
H1	0.9012	1.3361	0.3452	0.035*
C2	0.7812 (3)	1.5450 (2)	0.40335 (16)	0.0283 (4)
C3	0.7021 (3)	1.4852 (3)	0.58309 (16)	0.0317 (4)
H3	0.6516	1.5207	0.6461	0.038*
C4	0.7624 (3)	1.3170 (2)	0.58746 (15)	0.0306 (4)
H4	0.7489	1.2415	0.6523	0.037*
H1W1	0.615 (3)	0.941 (3)	0.5778 (16)	0.039 (7)*
H1W2	0.708 (4)	0.822 (2)	0.521 (2)	0.043 (7)*
H2W1	0.924 (3)	0.961 (3)	0.7118 (14)	0.038 (7)*
H2W2	1.136 (3)	0.983 (4)	0.671 (2)	0.058 (9)*
H3W1	0.348 (5)	0.556 (3)	0.872 (3)	0.072 (10)*
H3W2	0.376 (3)	0.387 (2)	0.906 (2)	0.050 (8)*
H4W1	−0.199 (5)	0.387 (3)	0.886 (3)	0.068 (10)*
H4W2	−0.234 (3)	0.558 (2)	0.856 (2)	0.043 (7)*
H5W1	0.135 (3)	0.176 (3)	1.0881 (19)	0.046 (8)*
H5W2	−0.062 (4)	0.185 (4)	1.076 (2)	0.066 (9)*
H6W1	0.637 (6)	0.145 (4)	0.876 (3)	0.094 (13)*
H6W2	0.625 (5)	0.164 (4)	0.9747 (12)	0.065 (10)*
H3N1	0.830 (5)	1.618 (4)	0.2538 (15)	0.062 (9)*
H3N2	0.751 (5)	1.7602 (15)	0.307 (3)	0.064 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.02009 (17)	0.01865 (17)	0.02239 (18)	−0.00235 (12)	−0.00307 (13)	−0.00400 (12)
Co2	0.02194 (19)	0.02479 (19)	0.0323 (2)	−0.00440 (14)	−0.00566 (14)	0.00002 (14)
S1	0.0220 (2)	0.0245 (2)	0.0240 (2)	−0.00519 (17)	−0.00492 (16)	−0.00072 (16)
O1	0.0285 (7)	0.0353 (7)	0.0270 (7)	−0.0006 (6)	−0.0095 (5)	−0.0022 (5)
O2	0.0429 (9)	0.0237 (7)	0.0623 (10)	−0.0039 (6)	−0.0138 (8)	−0.0005 (7)
O3	0.0241 (6)	0.0394 (8)	0.0336 (7)	−0.0094 (6)	−0.0057 (5)	−0.0074 (6)
O4	0.0292 (7)	0.0610 (10)	0.0311 (7)	−0.0127 (7)	0.0028 (6)	−0.0080 (7)
O1W	0.0249 (7)	0.0264 (7)	0.0383 (8)	−0.0067 (5)	0.0030 (6)	−0.0134 (6)
O2W	0.0266 (7)	0.0413 (8)	0.0242 (7)	−0.0062 (6)	−0.0044 (5)	−0.0069 (6)
O3W	0.0280 (8)	0.0322 (8)	0.0594 (10)	−0.0041 (6)	0.0049 (7)	0.0007 (7)
O4W	0.0383 (8)	0.0344 (8)	0.0493 (9)	−0.0060 (7)	−0.0199 (7)	−0.0029 (7)
O5W	0.0314 (8)	0.0290 (8)	0.0618 (10)	−0.0075 (6)	−0.0170 (7)	0.0077 (7)
O6W	0.0501 (9)	0.0321 (8)	0.0387 (9)	−0.0050 (7)	−0.0107 (7)	−0.0089 (7)

N1	0.0231 (7)	0.0206 (7)	0.0326 (8)	−0.0019 (6)	−0.0050 (6)	−0.0053 (6)
N2	0.0271 (8)	0.0234 (8)	0.0425 (9)	−0.0026 (6)	−0.0079 (7)	−0.0104 (7)
N3	0.0564 (12)	0.0247 (9)	0.0399 (11)	0.0015 (8)	−0.0053 (9)	−0.0040 (8)
C1	0.0305 (9)	0.0241 (9)	0.0318 (10)	−0.0029 (7)	−0.0039 (8)	−0.0072 (7)
C2	0.0244 (9)	0.0224 (9)	0.0378 (10)	−0.0034 (7)	−0.0059 (7)	−0.0052 (7)
C3	0.0290 (9)	0.0318 (10)	0.0361 (10)	0.0005 (8)	−0.0061 (8)	−0.0138 (8)
C4	0.0305 (10)	0.0296 (10)	0.0294 (9)	−0.0018 (8)	−0.0040 (8)	−0.0052 (7)

Geometric parameters (Å, º)

Co1—O1W	2.0451 (13)	O3W—H3W1	0.846 (10)
Co1—O1W^{i}	2.0451 (13)	O3W—H3W2	0.846 (10)
Co1—O2W^{i}	2.0970 (13)	O4W—H4W1	0.847 (10)
Co1—O2W	2.0970 (13)	O4W—H4W2	0.850 (10)
Co1—N1^{i}	2.2076 (15)	O5W—H5W1	0.849 (10)
Co1—N1	2.2076 (15)	O5W—H5W2	0.840 (10)
Co2—O3W	2.0670 (16)	O6W—H6W1	0.849 (10)
Co2—O3W^{ii}	2.0670 (16)	O6W—H6W2	0.847 (10)
Co2—O5W	2.0977 (15)	N1—C1	1.324 (2)
Co2—O5W^{ii}	2.0977 (15)	N1—C4	1.352 (3)
Co2—O4W	2.1128 (16)	N2—C2	1.340 (3)
Co2—O4W^{ii}	2.1128 (16)	N2—C3	1.343 (3)
S1—O2	1.4656 (16)	N3—C2	1.349 (3)
S1—O4	1.4703 (15)	N3—H3N1	0.849 (10)
S1—O1	1.4717 (13)	N3—H3N2	0.852 (10)
S1—O3	1.4866 (14)	C1—C2	1.411 (3)
O1W—H1W1	0.836 (10)	C1—H1	0.9300
O1W—H1W2	0.846 (10)	C3—C4	1.370 (3)
O2W—H2W1	0.838 (10)	C3—H3	0.9300
O2W—H2W2	0.843 (10)	C4—H4	0.9300
O1W—Co1—O1W^{i}	180.0	Co1—O1W—H1W1	126.4 (17)
O1W—Co1—O2W^{i}	87.43 (6)	Co1—O1W—H1W2	120.7 (18)
O1W^{i}—Co1—O2W^{i}	92.57 (6)	H1W1—O1W—H1W2	110 (2)
O1W—Co1—O2W	92.57 (6)	Co1—O2W—H2W1	128.2 (18)
O1W^{i}—Co1—O2W	87.43 (6)	Co1—O2W—H2W2	114 (2)
O2W^{i}—Co1—O2W	180.000 (1)	H2W1—O2W—H2W2	116 (3)
O1W—Co1—N1^{i}	88.71 (6)	Co2—O3W—H3W1	121 (2)
O1W^{i}—Co1—N1^{i}	91.29 (6)	Co2—O3W—H3W2	125.2 (19)
O2W^{i}—Co1—N1^{i}	90.16 (6)	H3W1—O3W—H3W2	107 (3)
O2W—Co1—N1^{i}	89.84 (6)	Co2—O4W—H4W1	117 (2)
O1W—Co1—N1	91.29 (6)	Co2—O4W—H4W2	114.2 (18)
O1W^{i}—Co1—N1	88.71 (6)	H4W1—O4W—H4W2	107 (3)
O2W^{i}—Co1—N1	89.84 (6)	Co2—O5W—H5W1	128.5 (19)
O2W—Co1—N1	90.16 (6)	Co2—O5W—H5W2	122 (2)
N1^{i}—Co1—N1	180.000 (1)	H5W1—O5W—H5W2	103 (3)
O3W—Co2—O3W^{ii}	180.0	H6W1—O6W—H6W2	104 (3)
O3W—Co2—O5W	88.95 (7)	C1—N1—C4	117.08 (16)

O3W^{ii}—Co2—O5W	91.05 (7)	C1—N1—Co1	119.42 (13)
O3W—Co2—O5W^{ii}	91.05 (7)	C4—N1—Co1	122.75 (13)
O3W^{ii}—Co2—O5W^{ii}	88.95 (7)	C2—N2—C3	116.49 (17)
O5W—Co2—O5W^{ii}	180.0	C2—N3—H3N1	118 (2)
O3W—Co2—O4W	87.20 (7)	C2—N3—H3N2	121 (2)
O3W^{ii}—Co2—O4W	92.80 (7)	H3N1—N3—H3N2	121 (3)
O5W—Co2—O4W	89.82 (7)	N1—C1—C2	122.25 (18)
O5W^{ii}—Co2—O4W	90.18 (7)	N1—C1—H1	118.9
O3W—Co2—O4W^{ii}	92.80 (7)	C2—C1—H1	118.9
O3W^{ii}—Co2—O4W^{ii}	87.20 (7)	N2—C2—N3	119.21 (18)
O5W—Co2—O4W^{ii}	90.18 (7)	N2—C2—C1	120.28 (18)
O5W^{ii}—Co2—O4W^{ii}	89.82 (7)	N3—C2—C1	120.50 (19)
O4W—Co2—O4W^{ii}	180.0	N2—C3—C4	123.26 (19)
O2—S1—O4	110.72 (10)	N2—C3—H3	118.4
O2—S1—O1	109.81 (9)	C4—C3—H3	118.4
O4—S1—O1	108.83 (9)	N1—C4—C3	120.53 (18)
O2—S1—O3	108.89 (9)	N1—C4—H4	119.7
O4—S1—O3	109.26 (9)	C3—C4—H4	119.7
O1—S1—O3	109.31 (8)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1w—H1w1···O1	0.84 (1)	1.93 (1)	2.755 (2)	168 (3)
O1w—H1w2···N2^{iii}	0.85 (1)	1.95 (1)	2.795 (2)	175 (3)
O2w—H2w1···O3	0.84 (1)	1.94 (1)	2.769 (2)	170 (2)
O2w—H2w2···O1^{iv}	0.84 (1)	1.93 (1)	2.765 (2)	170 (3)
O3w—H3w1···O2	0.85 (1)	1.91 (1)	2.743 (2)	169 (3)
O3w—H3w2···O6w	0.85 (1)	1.89 (1)	2.730 (2)	170 (3)
O4w—H4w1···O6w^{v}	0.85 (1)	1.95 (1)	2.781 (2)	168 (3)
O4w—H4w2···O2^{v}	0.85 (1)	1.91 (1)	2.745 (2)	167 (2)
O5w—H5w1···O3^{vi}	0.85 (1)	1.98 (1)	2.816 (2)	170 (3)
O5w—H5w2···O4^{ii}	0.84 (1)	1.90 (1)	2.737 (2)	174 (3)
O6w—H6w1···O3^{iii}	0.85 (1)	1.94 (1)	2.782 (2)	171 (4)
O6w—H6w2···O4^{vi}	0.85 (1)	1.89 (1)	2.711 (2)	164 (3)
N3—H3n2···O1^{vii}	0.85 (1)	2.20 (1)	3.036 (2)	168 (3)

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