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 Tetraaquabis(biuret- κ^2O,O')-yttrium(III) trichloride

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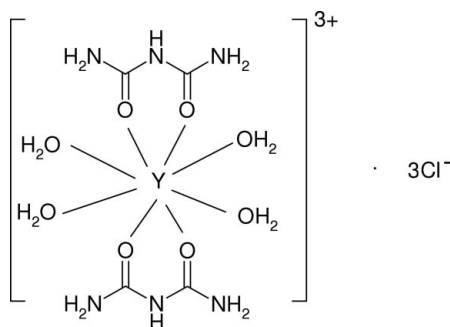
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{N}-\text{C}) = 0.002$ Å; R factor = 0.025; wR factor = 0.060; data-to-parameter ratio = 30.7.

In the title compound, $[\text{Y}(\text{C}_2\text{H}_5\text{N}_3\text{O}_2)_2(\text{H}_2\text{O})_4]\text{Cl}_3$, the Y^{3+} ion (site symmetry 2) is bonded to eight O atoms (arising from two O,O' -bidentate biuret molecules and four water molecules) in a distorted square-antiprismatic arrangement. A network of $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{Cl}$ and $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds help to establish the packing, leading to a three-dimensional network. One of the chloride ions has site symmetry 2.

Related literature

For related structures, see: Carugo *et al.* (1992); Haddad (1987, 1988). For related literature, see: Bernstein *et al.* (1995). For valence-sum calculations, see: Brese & O'Keeffe (1991).



Experimental

Crystal data

 $[\text{Y}(\text{C}_2\text{H}_5\text{N}_3\text{O}_2)_2(\text{H}_2\text{O})_4]\text{Cl}_3$
 $M_r = 473.50$
 Monoclinic, $C2/c$
 $a = 7.6510$ (4) Å
 $b = 13.2534$ (7) Å
 $c = 17.2547$ (9) Å
 $\beta = 100.817$ (1)°

 $V = 1718.57$ (16) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.90$ mm⁻¹
 $T = 293$ (2) K
 $0.36 \times 0.24 \times 0.14$ mm

Data collection

 Bruker SMART1000 CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
 $T_{\min} = 0.340$, $T_{\max} = 0.611$
 (expected range = 0.322–0.579)

 8075 measured reflections
 3105 independent reflections
 2464 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.059$
 $S = 0.98$
 3105 reflections

 101 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.37$ e Å⁻³
 $\Delta\rho_{\min} = -0.34$ e Å⁻³

Table 1

Selected bond lengths (Å).

| | | | |
|-------|-------------|-------|-------------|
| Y1–O1 | 2.3157 (11) | Y1–O4 | 2.3536 (12) |
| Y1–O2 | 2.3349 (11) | Y1–O3 | 2.3660 (10) |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|-------|-------------|-------------|---------------|
| N1–H1 \cdots O1 ⁱ | 0.86 | 2.10 | 2.9111 (18) | 157 |
| N1–H2 \cdots Cl1 ⁱⁱ | 0.86 | 2.39 | 3.1897 (17) | 155 |
| N2–H3 \cdots Cl1 ⁱⁱ | 0.86 | 2.53 | 3.3184 (14) | 153 |
| N3–H4 \cdots Cl1 ⁱⁱⁱ | 0.86 | 2.54 | 3.3633 (15) | 161 |
| N3–H5 \cdots Cl1 ^{iv} | 0.86 | 2.54 | 3.3232 (15) | 151 |
| O3–H6 \cdots Cl1 ^v | 0.80 | 2.39 | 3.1607 (12) | 161 |
| O3–H7 \cdots Cl2 ^{vi} | 0.79 | 2.27 | 3.0504 (12) | 168 |
| O4–H8 \cdots Cl1 | 0.75 | 2.45 | 3.2028 (13) | 177 |
| O4–H9 \cdots Cl2 | 0.80 | 2.40 | 3.1238 (12) | 150 |

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2260).

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supporting information

Acta Cryst. (2008). E64, m619 [doi:10.1107/S1600536808008659]

Tetraaquabis(biuret- κ^2O,O')yttrium(III) trichloride

William T. A. Harrison

S1. Comment

No complexes of yttrium(III) with biuret (biur), $H_2N-CO-NH-CO-NH_2$ (or $C_2H_5N_3O_2$) have been structurally characterized. The structures of two samarium-biuret complexes, $Sm(biur)_4(NO_3)_3$ (Haddad, 1987) and $Sm(biur)_4(ClO_4)_3$ (Haddad, 1988) have been described. In both cases, an SmO_8 square antiprismatic coordination arises for the metal ion. Based on X-ray photographs, it was suggested that all the $Ln(biur)_4(NO_3)_3$ and $Ln(biur)_4(ClO_4)_3$ compounds are isostructural with their samarium prototypes. In this paper, we describe the synthesis and structure of the title compound, (I).

Compound (I) is an ionic salt containing a new $[Y(biur)_2(H_2O)_4]^{3+}$ complex ion. The complete complex ion is generated by crystallographic 2-fold symmetry, with the Y atom lying on the rotation axis. Two uncoordinated chloride ions, one of which has crystallographic site symmetry 2, complete the structure of (I), Fig. 1.

The resulting YO_8 polyhedral geometry in (I) (Table 1) is a distorted square antiprism (Fig. 2). The nominal square face formed by atoms O1, O2, O1ⁱ and O2ⁱ ($i = -x, y, 3/2 - z$) is reasonably regular, but the second face formed by the four water molecules (O3, O4, O3ⁱ and O4ⁱ) is much more distorted, and the diagonal O3 \cdots O3ⁱ and O4 \cdots O4ⁱ distances of 4.223 (2) Å and 3.5197 (19) Å, respectively, are very different. The Y1 atom deviates from the mean planes of O1/O2/O1ⁱ/O2ⁱ and O3/O4/O3ⁱ/O4ⁱ by 1.1616 (8) Å and 1.3151 (9) Å, respectively. The two O atom mean planes are constrained to be parallel by symmetry. The bond valence sum (Brese & O'Keeffe, 1991) for Y1 of 3.34 in (I) indicates that its valence requirement is easily satisfied by this geometry.

The O,O-bidentate coordination of the biuret molecule to the yttrium ion in (I) results in a six-membered chelate ring that is non-planar. As noted previously (Carugo *et al.*, 1992), the biuret molecule can be regarded as two planar amide fragments linked by the NH bridge. Here, the dihedral angle between the N1/C1/O1/N2 and N2/C2/O2/N3 units is 5.06 (10)°. The yttrium cation deviates from the N1/C1/O1/N2 and N2/C2/O2/N3 mean planes by 0.894 (4) Å and 0.606 (4) Å, respectively.

The component species in (I) are linked by a dense array of N—H \cdots O, N—H \cdots Cl and O—H \cdots Cl hydrogen bonds (Table 2) resulting in a three-dimensional network. Of note is the N—H \cdots O hydrogen bond, which results in [100] chains (Fig. 3) of cations, in which $R^2_2(8)$ loops (Bernstein *et al.*, 1995) linking the molecules are apparent.

S2. Experimental

0.1 M Aqueous solutions of YCl_3 (10 ml) and biuret (10 ml) were mixed and a small quantity of dilute hydrochloric acid was added, to result in a colourless solution. Colourless blocks of (I) grew over several days as the water slowly evaporated.

S3. Refinement

The N-bound hydrogen atoms were geometrically placed (N—H = 0.88 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. The water H atoms were located in difference maps and refined as riding in their as-found relative positions with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$; see Table 2 for O—H distances.

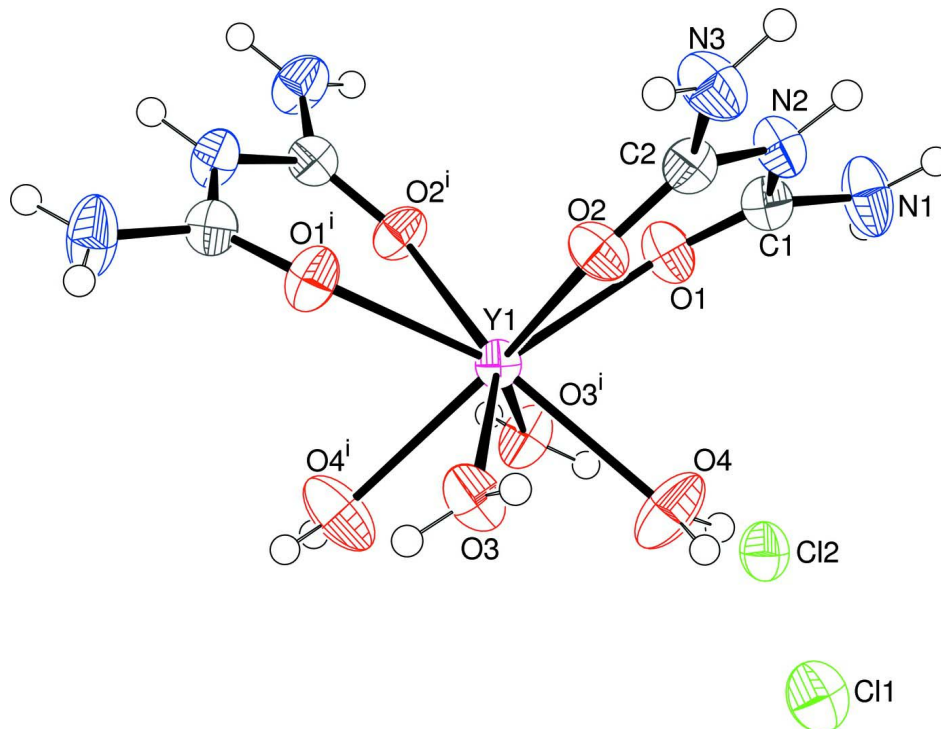
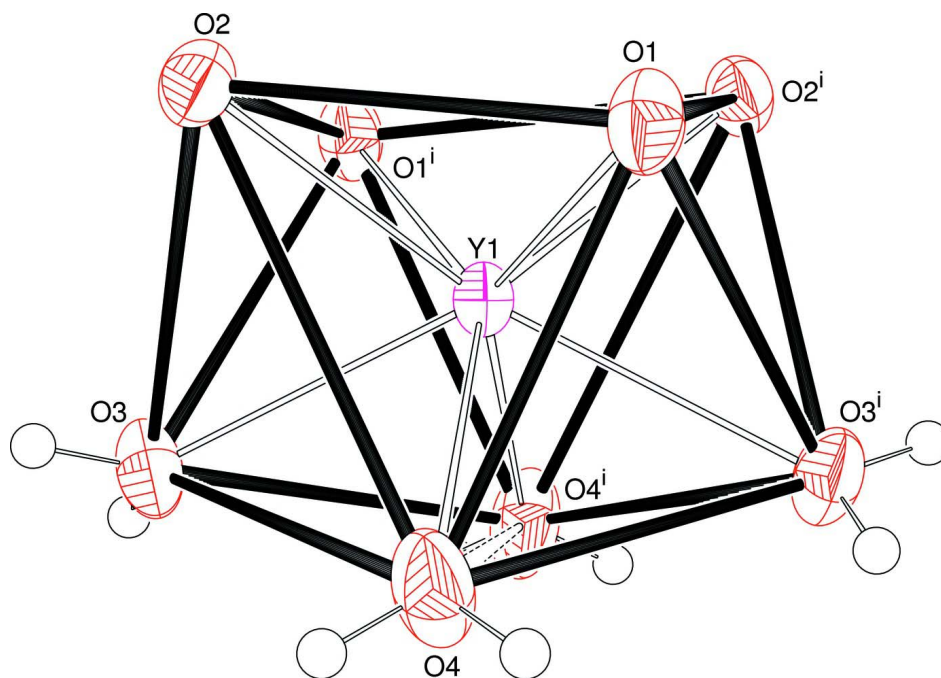
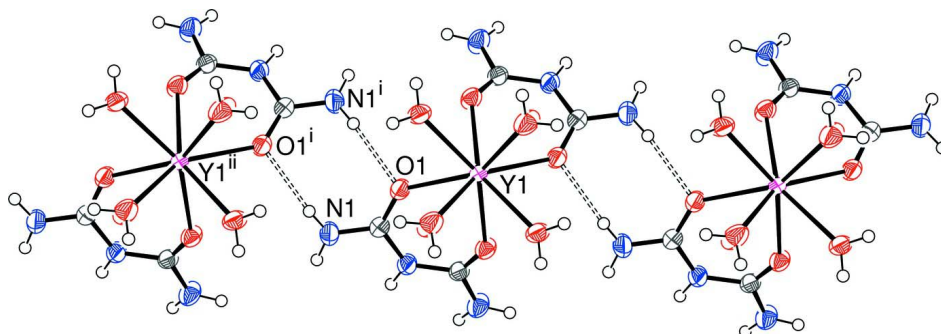


Figure 1

View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). Symmetry code: (i) $-x, y, 3/2 - z$.


Figure 2

Detail of (I) showing the distorted square antiprismatic coordination of the Y atom. Symmetry code: (i) $-x, y, 3/2 - z$.


Figure 3

Fragment of a [100] chain of cations in (I) with the hydrogen bonds indicated by double-dashed lines. Symmetry codes:

(i) $1 - x, y, 3/2 - z$; (ii) $x + 1, y, z$.

Tetraaquabis(biuret- κ^2O, O')yttrium(III) trichloride

Crystal data

$[\text{Y}(\text{C}_2\text{H}_5\text{N}_3\text{O}_2)_2(\text{H}_2\text{O})_4]\text{Cl}_3$

$M_r = 473.50$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 7.6510 (4) \text{ \AA}$

$b = 13.2534 (7) \text{ \AA}$

$c = 17.2547 (9) \text{ \AA}$

$\beta = 100.817 (1)^\circ$

$V = 1718.57 (16) \text{ \AA}^3$

$Z = 4$

$F(000) = 952$

$D_x = 1.830 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3637 reflections

$\theta = 3.1\text{--}32.4^\circ$

$\mu = 3.90 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colourless

$0.36 \times 0.24 \times 0.14 \text{ mm}$

Data collection

| | |
|--|--|
| Bruker SMART1000 CCD diffractometer | 8075 measured reflections |
| Radiation source: fine-focus sealed tube | 3105 independent reflections |
| Graphite monochromator | 2464 reflections with $I > 2\sigma(I)$ |
| ω scans | $R_{\text{int}} = 0.019$ |
| Absorption correction: multi-scan (SADABS; Bruker, 1999) | $\theta_{\text{max}} = 32.6^\circ$, $\theta_{\text{min}} = 2.4^\circ$ |
| $T_{\text{min}} = 0.340$, $T_{\text{max}} = 0.611$ | $h = -11 \rightarrow 11$ |
| | $k = -20 \rightarrow 19$ |
| | $l = -16 \rightarrow 26$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: difmap (O-H) and geom (N-H) |
| $R[F^2 > 2\sigma(F^2)] = 0.025$ | H-atom parameters constrained |
| $wR(F^2) = 0.059$ | $w = 1/[\sigma^2(F_o^2) + (0.0303P)^2]$ |
| $S = 0.98$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3105 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 101 parameters | $\Delta\rho_{\text{max}} = 0.37 \text{ e } \text{\AA}^{-3}$ |
| 0 restraints | $\Delta\rho_{\text{min}} = -0.34 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | |

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.

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|--------------|----------------------------------|
| Y1 | 0.0000 | 0.107273 (15) | 0.7500 | 0.02386 (6) |
| Cl1 | -0.04205 (5) | 0.33346 (3) | 0.50811 (2) | 0.03905 (10) |
| Cl2 | 0.5000 | 0.29705 (5) | 0.7500 | 0.03992 (14) |
| O1 | 0.25038 (14) | 0.02884 (9) | 0.72093 (7) | 0.0348 (3) |
| O2 | -0.08317 (13) | 0.01042 (8) | 0.63600 (7) | 0.0329 (2) |
| N1 | 0.45654 (18) | -0.01014 (13) | 0.64850 (10) | 0.0476 (4) |
| H1 | 0.5399 | 0.0182 | 0.6817 | 0.057* |
| H2 | 0.4804 | -0.0382 | 0.6068 | 0.057* |
| N2 | 0.17105 (17) | -0.05973 (11) | 0.60525 (8) | 0.0332 (3) |
| H3 | 0.2113 | -0.0980 | 0.5724 | 0.040* |
| N3 | -0.10477 (19) | -0.11253 (11) | 0.54451 (9) | 0.0410 (3) |
| H4 | -0.2191 | -0.1099 | 0.5351 | 0.049* |
| H5 | -0.0506 | -0.1546 | 0.5193 | 0.049* |
| C1 | 0.2922 (2) | -0.01064 (12) | 0.66153 (10) | 0.0308 (3) |
| C2 | -0.0118 (2) | -0.05156 (11) | 0.59806 (9) | 0.0295 (3) |
| O3 | -0.23875 (15) | 0.18780 (9) | 0.66560 (7) | 0.0377 (3) |

| | | | | |
|----|--------------|--------------|-------------|------------|
| H6 | -0.2731 | 0.1747 | 0.6201 | 0.045* |
| H7 | -0.3170 | 0.2159 | 0.6812 | 0.045* |
| O4 | 0.11867 (16) | 0.22520 (10) | 0.67195 (8) | 0.0519 (4) |
| H8 | 0.0828 | 0.2492 | 0.6326 | 0.062* |
| H9 | 0.2176 | 0.2465 | 0.6742 | 0.062* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|--------------|-------------|--------------|--------------|---------------|
| Y1 | 0.02161 (9) | 0.02849 (10) | 0.02041 (9) | 0.000 | 0.00117 (7) | 0.000 |
| Cl1 | 0.0364 (2) | 0.0491 (2) | 0.0297 (2) | 0.00110 (17) | 0.00118 (17) | -0.00610 (18) |
| Cl2 | 0.0316 (3) | 0.0479 (3) | 0.0418 (3) | 0.000 | 0.0107 (2) | 0.000 |
| O1 | 0.0255 (5) | 0.0467 (6) | 0.0309 (6) | 0.0049 (5) | 0.0014 (5) | -0.0080 (5) |
| O2 | 0.0282 (5) | 0.0358 (6) | 0.0327 (6) | 0.0034 (5) | 0.0004 (5) | -0.0092 (5) |
| N1 | 0.0266 (7) | 0.0778 (11) | 0.0394 (8) | -0.0078 (7) | 0.0089 (6) | -0.0204 (8) |
| N2 | 0.0266 (6) | 0.0415 (7) | 0.0315 (7) | 0.0016 (6) | 0.0053 (5) | -0.0088 (6) |
| N3 | 0.0321 (7) | 0.0459 (8) | 0.0426 (8) | -0.0014 (6) | 0.0012 (6) | -0.0179 (7) |
| C1 | 0.0271 (7) | 0.0350 (8) | 0.0293 (8) | 0.0013 (6) | 0.0025 (6) | 0.0004 (7) |
| C2 | 0.0290 (7) | 0.0320 (7) | 0.0261 (7) | -0.0009 (6) | 0.0021 (6) | 0.0012 (6) |
| O3 | 0.0353 (6) | 0.0498 (7) | 0.0253 (6) | 0.0145 (5) | -0.0011 (5) | -0.0026 (5) |
| O4 | 0.0390 (7) | 0.0677 (9) | 0.0436 (7) | -0.0157 (6) | -0.0061 (6) | 0.0281 (7) |

Geometric parameters (Å, °)

| | | | |
|-------------------------------------|-------------|------------------------|-------------|
| Y1—O1 ⁱ | 2.3157 (11) | N1—H2 | 0.8600 |
| Y1—O1 | 2.3157 (11) | N2—C1 | 1.374 (2) |
| Y1—O2 | 2.3349 (11) | N2—C2 | 1.385 (2) |
| Y1—O2 ⁱ | 2.3349 (11) | N2—H3 | 0.8600 |
| Y1—O4 ⁱ | 2.3536 (12) | N3—C2 | 1.329 (2) |
| Y1—O4 | 2.3536 (12) | N3—H4 | 0.8600 |
| Y1—O3 ⁱ | 2.3660 (10) | N3—H5 | 0.8600 |
| Y1—O3 | 2.3660 (10) | O3—H6 | 0.7990 |
| O1—C1 | 1.2450 (19) | O3—H7 | 0.7934 |
| O2—C2 | 1.2392 (18) | O4—H8 | 0.7541 |
| N1—C1 | 1.318 (2) | O4—H9 | 0.8023 |
| N1—H1 | 0.8600 | | |
| O1 ⁱ —Y1—O1 | 126.66 (6) | O4—Y1—O3 | 71.61 (4) |
| O1 ⁱ —Y1—O2 | 80.21 (4) | O3 ⁱ —Y1—O3 | 126.37 (6) |
| O1—Y1—O2 | 71.12 (4) | C1—O1—Y1 | 135.89 (10) |
| O1 ⁱ —Y1—O2 ⁱ | 71.12 (4) | C2—O2—Y1 | 137.05 (10) |
| O1—Y1—O2 ⁱ | 80.21 (4) | C1—N1—H1 | 120.0 |
| O2—Y1—O2 ⁱ | 113.30 (6) | C1—N1—H2 | 120.0 |
| O1 ⁱ —Y1—O4 ⁱ | 75.55 (5) | H1—N1—H2 | 120.0 |
| O1—Y1—O4 ⁱ | 147.74 (4) | C1—N2—C2 | 124.22 (14) |
| O2—Y1—O4 ⁱ | 140.68 (4) | C1—N2—H3 | 117.9 |
| O2 ⁱ —Y1—O4 ⁱ | 87.51 (4) | C2—N2—H3 | 117.9 |
| O1 ⁱ —Y1—O4 | 147.74 (4) | C2—N3—H4 | 120.0 |

| | | | |
|-------------------------------------|------------|----------|-------------|
| O1—Y1—O4 | 75.55 (5) | C2—N3—H5 | 120.0 |
| O2—Y1—O4 | 87.51 (4) | H4—N3—H5 | 120.0 |
| O2 ⁱ —Y1—O4 | 140.68 (4) | O1—C1—N1 | 122.63 (15) |
| O4 ⁱ —Y1—O4 | 96.78 (8) | O1—C1—N2 | 122.49 (14) |
| O1 ⁱ —Y1—O3 ⁱ | 130.07 (4) | N1—C1—N2 | 114.87 (15) |
| O1—Y1—O3 ⁱ | 76.19 (4) | O2—C2—N3 | 122.60 (14) |
| O2—Y1—O3 ⁱ | 145.42 (4) | O2—C2—N2 | 122.84 (14) |
| O2 ⁱ —Y1—O3 ⁱ | 70.89 (4) | N3—C2—N2 | 114.49 (15) |
| O4 ⁱ —Y1—O3 ⁱ | 71.61 (4) | Y1—O3—H6 | 125.5 |
| O4—Y1—O3 ⁱ | 73.53 (4) | Y1—O3—H7 | 123.2 |
| O1 ⁱ —Y1—O3 | 76.19 (4) | H6—O3—H7 | 107.8 |
| O1—Y1—O3 | 130.07 (4) | Y1—O4—H8 | 133.0 |
| O2—Y1—O3 | 70.89 (4) | Y1—O4—H9 | 132.0 |
| O2 ⁱ —Y1—O3 | 145.42 (4) | H8—O4—H9 | 94.3 |
| O4 ⁱ —Y1—O3 | 73.53 (4) | | |

Symmetry code: (i) $-x, y, -z+3/2$.

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N1—H1 \cdots O1 ⁱⁱ | 0.86 | 2.10 | 2.9111 (18) | 157 |
| N1—H2 \cdots C11 ⁱⁱⁱ | 0.86 | 2.39 | 3.1897 (17) | 155 |
| N2—H3 \cdots C11 ⁱⁱⁱ | 0.86 | 2.53 | 3.3184 (14) | 153 |
| N3—H4 \cdots C11 ^{iv} | 0.86 | 2.54 | 3.3633 (15) | 161 |
| N3—H5 \cdots C11 ^v | 0.86 | 2.54 | 3.3232 (15) | 151 |
| O3—H6 \cdots C11 ^{vi} | 0.80 | 2.39 | 3.1607 (12) | 161 |
| O3—H7 \cdots C12 ^{vii} | 0.79 | 2.27 | 3.0504 (12) | 168 |
| O4—H8 \cdots C11 | 0.75 | 2.45 | 3.2028 (13) | 177 |
| O4—H9 \cdots C12 | 0.80 | 2.40 | 3.1238 (12) | 150 |

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