Tetraammonium $\mu$-ethylenediaminetetraacetato-$1\kappa^3O,N,O':2\kappa^3O'',N'',O'''-bis[trioxidotungstate(VI)]
tetrahydrate

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The title compound, $(\text{NH}_4)_4[W_2(C_{10}H_{12}N_2O_8)O_6] \cdot \text{C}_1 \text{H}_2 \text{O}$, was obtained from a mixture of tungstic acid, ammonia and ethylenediaminetetraacetic acid ($\text{H}_4\text{edta}$) in a 2:4:1 ratio. The anion of the complex contains two $\text{WO}_3$ units and one bridging $\text{edta}^{4-}$ ligand. Each central metal atom is tridentately coordinated by nitrogen and two carboxylate groups of the $\text{edta}^{4-}$ ligand, together with the three oxido ligands, producing a distorted octahedral coordination environment around each tungsten atom. The center of the carbon–carbon bond of the ethylene bridge represents a crystallographic inversion center. The crystal structure consists of a three-dimensional supramolecular framework built up by the dinuclear bridge, the ammonium counter-ions and the solvent water molecules via hydrogen bonds of the $\text{N} - \text{H} \cdots \text{O}$ and $\text{O} - \text{H} \cdots \text{O}$ type.

Structure description

Research on inorganic–organic framework materials is one of the fastest growing areas in materials chemistry because of their unique hybrid nature, which enables the combination of properties from both inorganic and organic materials (Cheetham & Rao, 2007). As organic ligands, polycarboxylates are multidentate chelating agents that are widespread in nature and industry because of their ability to coordinate with various transition metals in different ratios (Nicolau & Guy, 1995; Langer, 2000).

As a part of this field, molybdenum polycarboxylate complexes have thus been thoroughly investigated over the past three decades (Lee & Holm, 2004). Some well-characterized mono-, bi- and polynuclear molybdenum and tungsten complexes have been reported, for example $\text{Mo}_2(\text{O}_2\text{CCH}_2\text{OH})_4$, $\text{M}_2[\text{MoO}_3(\text{C}_2\text{O}_4)]$ ($\text{M} = \text{Na}, \text{K}, \text{Rb}, \text{Cs}$),

Received 25 August 2021
Accepted 21 September 2021

Edited by W. Imhof, University Koblenz-Landau, Germany

Keywords: crystal structure; ethylenediaminetetraacetate; tungstic acid; binuclear complex.

Structural data: full structural data are available from iucrdata.iucr.org
Table 1
Hydrogen-bond geometry (Å, °).

| D—H—A   | D—H  | D···A  | D—D···A | D—H···A |
|----------|-------|--------|----------|----------|
| O2—H2A·O3 | 0.87  | 1.88   | 2.743 (3) | 171      |
| O2—H2B·O6  | 0.87  | 1.90   | 2.763 (3) | 170      |
| O1—H1A·O7  | 0.87  | 2.72   | 3.468 (3) | 145      |
| O1—H1A·O4  | 0.87  | 2.06   | 2.900 (3) | 161      |
| O1—H1B·O2  | 0.87  | 2.49   | 3.177 (3) | 137      |
| N10—H10A·O6 | 0.95  | 1.78 (4)| 2.727 (3) | 175 (3)  |
| N10—H10B·O4  | 0.81 (4)| 2.20 (4) | 2.966 (3) | 168 (3)  |
| N10—H10C·O7  | 0.90 (4)| 2.01 (4) | 2.876 (3) | 160 (3)  |
| N10—H10D·O8  | 0.87 (4)| 1.84 (4) | 2.736 (3) | 175 (4)  |
| N11—H11A·O8 | 0.87 (5)| 2.13 (5) | 2.947 (3) | 156 (4)  |
| N11—H11B·O3  | 0.84 (5)| 2.31 (5) | 3.109 (3) | 160 (4)  |
| N11—H11C·O1  | 0.83 (4)| 2.25 (4) | 2.979 (4) | 147 (4)  |
| N11—H11D·O2  | 0.92 (4)| 1.93 (4) | 2.846 (4) | 173 (4)  |

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

Na₃[MO₂(C₈H₁₂N₂O₈)₃]·3H₂O (M = Mo, W) (Cotton et al., 2002; Cindric et al., 2000; Zhou et al., 1999). Structural analyses of W⁶⁺–edta complexes are rare in the literature. Together with the structure of Na₃K₆[Mo₆O₁₈(edta)₃]·10H₂O, the structure of Na₃[W₂O₇(edta)]·8H₂O has been published (Lin et al., 2006).

Nevertheless, tungsten has been reported to incorporate into several enzymes (Johnson et al., 1996). In fact, tungsten could be a useful probe for the active site of molybdenum enzymes. As a consequence, more effort has been put into tungsten chemistry by inorganic and bioinorganic chemists (Bagno & Bonchio, 2000; Enemark et al., 2004; Sung & Holm, 2001; Zhou et al., 2004).

In this study, the reaction of H₄edta with tungstic acid has been investigated and a new binuclear 2:1 W–edta complex, (NH₄)₄[W₂(C₁₀H₁₂N₂O₈)O₆]·4H₂O, was isolated and structurally characterized.

As shown in Fig. 1, the dinuclear anion of the title compound shows one edta⁴⁻ ligand bonded to two tungstate WO₄ units. Each W atom is six-coordinate in a distorted octahedral environment built up by the tridentate facial coordination of one N and two O atoms of the edta⁴⁻ ligand as well as by three oxido ligands. The edta⁴⁻ ligand itself therefore acts as a bridge between the two WO₄ units, with the central carbon–carbon bond also representing a crystallographic center of inversion. The anion is accompanied by four ammonium cations and four solvent water molecules.

![Figure 1](image1.png)

Figure 1
Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Synthesis and crystallization
Tungstic acid (4 mmol, 0.999 g) and ammonia solution (8 mmol, 1.001 g) were mixed in 30 ml of water to solubilize the W⁶⁺ source. To this mixture was slowly added ethylenediammine-tetraacetic acid (H₄edta) (2 mmol, 0.584 g) under vigorous stirring. The solution was then stirred for two h at room temperature. The colorless solution thus obtained was left at room temperature for slow evaporation of water. After two weeks, colorless crystals (yield 11.6% based on W) were obtained from the solution.

The FT–infrared spectra of the title compound shows well-resolved absorption bands for the carboxylate of the coordinating edta⁴⁻ at 1651 cm⁻¹ and 1402 cm⁻¹, which are attributed to the antisymmetric and symmetric stretching vibrations ν(COO⁻). The bands at 926, 857 and 666 cm⁻¹ can be attributed to symmetric and asymmetric W=O stretching vibrations (Lin et al., 2006; Li et al., 2007). The range of 3500–2800 cm⁻¹ shows many bands ascribed to O–H stretching of water molecules, as well as N–H stretching vibrations of ammonium cations (Yaffa et al., 2020).

Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2.
Table 2
Experimental details.

| Crystal data |  |
|--------------|---|
| Chemical formula | (NH₄)₄[W₂(C₁₀H₁₂N₂O₈)O₆]·4H₂O |
| M_r | 896.15 |
| Crystal system, space group | Monoclinic, P2₁/c |
| Temperature (K) | 150 |
| a, b, c (Å) | 6.8017 (5), 7.7194 (5), 23.9807 (19) |
| V (Å³) | 1253.63 (16) |
| Z | 2 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 9.26 |
| Crystal size (mm) | 0.18 × 0.18 × 0.14 |

Data collection

| Diffractometer | Bruker APEXII CCD |
|----------------|-----------------|
| Absorption correction | Multi-scan (SADABS; Bruker, 2016) |
| T_min, T_max | 0.444, 0.746 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 55419, 2890, 2703 |
| R(int) | 0.053 |
| (sin θ/λ)max (Å⁻¹) | 0.652 |

Refinement

| R[F² > 2σ(F²)], wR(F²), S | 0.014, 0.034, 1.07 |
| No. of reflections | 2890 |
| No. of parameters | 201 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³) | 0.71, -0.85 |

References

Bagni, A. & Bonchio, M. (2000). *Chem. Phys. Lett.* **317**, 123–128.

Bruker (2016). Bruker (2016). *APEX2, SAINT* and *SADABS.*

Cheetham, A. K. & Rao, C. N. R. (2007). *Science* **318**, 58–59.

Cindric, M., Strukan, N., Vrdoljak, V., Devecić, M., Veksli, Z. & Kamenar, B. (2000). *Inorg. Chim. Acta* **304**, 260–267.

Cotton, F. A., Barnard, T. S., Daniels, L. M. & Murillo, C. A. (2002). *Inorg. Chem. Commun.* **5**, 527–532.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Enemark, J. H., Cooney, J. J. A., Wang, J. J. & Holm, R. H. (2004). *Chem. Rev.* **104**, 1175–1200.

Johnson, M. K., Rees, D. C. & Adams, M. W. W. (1996). *Chem. Rev.* **96**, 2817–2840.

Langer, R. (2000). *Acc. Chem. Res.* **33**, 94–101.

Lee, S. C. & Holm, R. H. (2004). *Chem. Rev.* **104**, 1135–1158.

Lin, H. B., Chen, C. Y., Liao, X. L., Lin, T. R. & Zhou, Z. H. (2006). *Synth. React. Inorg. Met.-Org. Nano-Met. Chem.* **36**, 411–414.

Nicolaou, K. C. & Guy, R. K. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 2079–2090.

Yaffa, L., Kama, A. B., Sall, M. L., Diop, C. A. K., Sidibé, M., Giorgi, M., Diop, M. & Gautier, R. (2020). *J. Inorg. Biochem.* **191**, 1–6.

Zhou, Z.-H., Hou, S. Y., Cao, Z.-X., Wan, H. L. & Ng, S. W. (2004). *J. Inorg. Biochem.* **98**, 1037–1044.

Zhou, Z. H., Wan, H. L. & Tsai, K. R. (1999). *J. Chem. Soc. Dalton Trans.* pp. 4289–4290.
full crystallographic data

*IUCrData* (2021). *6*, x210982  [https://doi.org/10.1107/S2414314621009822]

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**Crystal data**

(NH₄)₄[W₂(C₁₀H₁₂N₂O₈)O₆]·4H₂O  

$F(000) = 860$  

$D_x = 2.374$ Mg m$^{-3}$  

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å  

Cell parameters from 9817 reflections  

$\theta = 2.8–27.6^\circ$  

$\mu = 9.26$ mm$^{-1}$  

$T = 150$ K  

Block, colourless  

$0.18 \times 0.18 \times 0.14$ mm

**Data collection**

Bruker APEXII CCD diffractometer  

$\varphi$ and $\omega$ scans  

Absorption correction: multi-scan  

(SADABS; Bruker, 2016)  

$T_{\text{min}} = 0.444$, $T_{\text{max}} = 0.746$  

55419 measured reflections

2890 independent reflections  

2703 reflections with $I > 2\sigma(I)$  

$R_{\text{int}} = 0.053$  

$\theta_{\text{max}} = 27.6^\circ$, $\theta_{\text{min}} = 2.8^\circ$  

$h = -8$ to $8$  

$k = -10$ to $10$  

$l = -31$ to $31$

**Refinement**

Refinement on $F^2$  

Least-squares matrix: full  

$R[F^2 > 2\sigma(F^2)] = 0.014$  

$wR(F^2) = 0.034$  

$S = 1.07$  

2890 reflections  

201 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  

$w = 1/[\sigma(F_c^2) + (0.0138P)^2 + 1.4846P]$  

where $P = (F_c^2 + 2F_s^2)/3$  

$(\Delta/\sigma)_{\text{max}} = 0.002$  

$\Delta \rho_{\text{max}} = 0.71$ e Å$^{-3}$  

$\Delta \rho_{\text{min}} = -0.85$ 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.

**Refinement.** All non-hydrogen atoms were refined anisotropically. Hydrogen atoms bonded to carbon and oxygen were placed in idealized positions and refined using a riding model with isotropic displacement parameters calculated as $U_{	ext{iso}}(H) = 1.2U_{	ext{eq}}(C)$ for ethylene and methylene hydrogen atoms and $U_{	ext{iso}}(H) = 1.5U_{	ext{eq}}(O)$ for solvent water molecules. Hydrogen atoms of the ammonium cations were located in the difference-Fourier map and refined isotropically.

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

|   | $x$          | $y$          | $z$          | $U_{	ext{iso}}/U_{	ext{eq}}$ |
|---|--------------|--------------|--------------|---------------------------------|
| W1| 0.40177 (2)  | 0.69956 (2)  | 0.10889 (2)  | 0.01546 (4)                     |
| O6| 0.4717 (3)   | 0.7513 (3)   | 0.04235 (8)  | 0.0290 (4)                      |
| O3| 0.5793 (3)   | 0.7989 (2)   | 0.15614 (9)  | 0.0270 (4)                      |
| O9| 0.2095 (3)   | 0.6721 (2)   | 0.17381 (8)  | 0.0237 (4)                      |
| N4| 0.0791 (3)   | 0.6278 (2)   | 0.06743 (8)  | 0.0143 (4)                      |
| O8| 0.4519 (3)   | 0.4764 (2)   | 0.11461 (8)  | 0.0276 (4)                      |
| O5| −0.0230 (3)  | 0.5116 (3)   | 0.20717 (8)  | 0.0351 (5)                      |
| C7| −0.0056 (4)  | 0.5133 (3)   | 0.10897 (10) | 0.0192 (5)                      |
| H7A| 0.0366       | 0.3924       | 0.1033       | 0.023*                          |
| H7B| −0.1515      | 0.5173       | 0.1031       | 0.023*                          |
| C11| 0.0609 (4)   | 0.5690 (3)   | 0.16809 (11) | 0.0200 (5)                      |
| C12| 0.0980 (4)   | 0.5353 (3)   | 0.01373 (11) | 0.0190 (5)                      |
| H12A| 0.1915       | 0.4378       | 0.0208       | 0.023*                          |
| H12B| 0.1546       | 0.6157       | −0.0127      | 0.023*                          |
| O7| −0.0275 (3)  | 1.0904 (2)   | 0.07169 (9)  | 0.0280 (4)                      |
| O4| 0.2366 (3)   | 0.9388 (2)   | 0.10315 (9)  | 0.0253 (4)                      |
| C1| 0.0597 (4)   | 0.9519 (3)   | 0.07921 (11) | 0.0183 (5)                      |
| C8| −0.0402 (4)  | 0.7886 (3)   | 0.05795 (12) | 0.0219 (5)                      |
| H8A| −0.1639      | 0.7748       | 0.0762       | 0.026*                          |
| H8B| −0.0769      | 0.8012       | 0.0172       | 0.026*                          |
| N10| 0.4415 (4)   | 0.8304 (3)   | −0.06894 (11)| 0.0225 (5)                      |
| O2| 0.7912 (3)   | 1.0837 (3)   | 0.19478 (9)  | 0.0357 (5)                      |
| H2A| 0.7148       | 0.9966       | 0.1847       | 0.054*                          |
| H2B| 0.8584       | 1.0492       | 0.2254       | 0.054*                          |
| O1| 0.2481 (3)   | 1.1729 (3)   | 0.19796 (10)| 0.0367 (5)                      |
| H1A| 0.2243       | 1.1196       | 0.1661       | 0.055*                          |
| H1B| 0.1322       | 1.2036       | 0.2070       | 0.055*                          |
| N11| 0.5898 (4)   | 0.3808 (4)   | 0.23069 (12) | 0.0262 (5)                      |
| H10A| 0.456 (5)    | 0.808 (4)    | −0.0300 (16) | 0.032 (9)*                      |
| H10B| 0.521 (5)    | 0.904 (5)    | −0.0757 (14) | 0.034 (9)*                      |
| H10C| 0.316 (6)    | 0.864 (5)    | −0.0785 (16) | 0.045 (10)*                     |
| H10D| 0.471 (6)    | 0.735 (5)    | −0.0853 (17) | 0.046 (11)*                     |
| H11A| 0.515 (6)    | 0.409 (6)    | 0.2004 (19)  | 0.060 (13)*                     |
| H11B| 0.517 (7)    | 0.355 (6)    | 0.256 (2)    | 0.069 (14)*                     |
| H11C| 0.662 (6)    | 0.466 (6)    | 0.2389 (17)  | 0.054 (12)*                     |
### Atomic displacement parameters (Å²)

|   | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$   | $U^{13}$   | $U^{23}$   |
|---|------------|------------|------------|------------|------------|------------|
| W1| 0.01185 (5)| 0.01695 (6)| 0.01687 (6)| −0.00047 (3)| −0.00242 (4)| 0.00027 (4)|
| O6| 0.0201 (10)| 0.0457 (11)| 0.0211 (10)| −0.0102 (9) | 0.0011 (8)  | 0.0012 (9)  |
| O3| 0.0216 (10)| 0.0330 (11)| 0.0253 (10)| −0.0030 (8) | −0.0044 (8) | −0.0031 (8) |
| O9| 0.0244 (10)| 0.0285 (10)| 0.0182 (9) | −0.0082 (8) | 0.0019 (8)  | −0.0016 (7) |
| N4| 0.0136 (9) | 0.0125 (9) | 0.0161 (10)| −0.0008 (7) | −0.0026 (8) | 0.0005 (8)  |
| O8| 0.0255 (10)| 0.0208 (9) | 0.0344 (11)| 0.0069 (8)  | −0.0087 (8) | −0.0021 (8) |
| O5| 0.0297 (11)| 0.0522 (13)| 0.0233 (10)| −0.0139 (10)| 0.0015 (8)  | 0.0115 (10) |
| C7| 0.0173 (12)| 0.0174 (12)| 0.0225 (13)| −0.0051 (9) | −0.0006 (10)| 0.0026 (10) |
| C11| 0.0174 (12)| 0.0193 (12)| 0.0227 (13)| 0.0011 (9)  | −0.0011 (10)| 0.0044 (10) |
| C12| 0.0165 (12)| 0.0215 (12)| 0.0186 (12)| −0.0022 (9) | −0.0010 (10)| −0.0051 (10)|
| O7| 0.0256 (10)| 0.0166 (9) | 0.0411 (12)| 0.0055 (8)  | −0.0012 (8) | 0.0011 (8)  |
| O4| 0.0190 (9) | 0.0153 (9) | 0.0396 (12)| −0.0016 (7) | −0.0083 (8) | −0.0005 (8) |
| C1| 0.0169 (12)| 0.0176 (12)| 0.0202 (13)| 0.0004 (9)  | 0.0013 (9)  | 0.0015 (9)  |
| C8| 0.0177 (12)| 0.0142 (12)| 0.0320 (15)| 0.0019 (9)  | −0.0070 (11)| 0.0018 (10) |
| N10| 0.0215 (12)| 0.0178 (11)| 0.0279 (14)| −0.0025 (9) | 0.0010 (10) | 0.0034 (10) |
| O2| 0.0462 (13)| 0.0282 (11)| 0.0316 (12)| −0.0025 (10)| −0.0022 (10)| −0.0008 (9) |
| O1| 0.0333 (12)| 0.0386 (12)| 0.0377 (13)| −0.0046 (10)| 0.0013 (10) | −0.0024 (10)|
| N11| 0.0272 (13)| 0.0256 (13)| 0.0254 (13)| −0.0032 (11)| 0.0000 (11)| 0.0033 (11)|

### Geometric parameters (Å, °)

|   | $d_{ij}$   | $d_{ij}$   | $d_{ij}$   | $d_{ij}$   | $d_{ij}$   | $d_{ij}$   |
|---|------------|------------|------------|------------|------------|------------|
| W1—O6| 1.7529 (19) | O7—C1     | 1.228 (3)  |
| W1—O3| 1.7534 (19) | O4—C1     | 1.288 (3)  |
| W1—O9| 2.1350 (19) | C1—C8     | 1.499 (3)  |
| W1—N4| 2.3884 (19) | C8—H8A    | 0.9900     |
| W1—O8| 1.7590 (19) | C8—H8B    | 0.9900     |
| W1—O4| 2.1590 (17) | N10—H10A  | 0.95 (4)   |
| O9—C11| 1.284 (3)  | N10—H10B  | 0.81 (4)   |
| N4—C7| 1.487 (3)   | N10—H10C  | 0.90 (4)   |
| N4—C12| 1.488 (3)  | N10—H10D  | 0.87 (4)   |
| N4—C8| 1.489 (3)   | O2—H2A    | 0.8701     |
| O5—C11| 1.225 (3)  | O2—H2B    | 0.8698     |
| C7—H7A| 0.9900     | O1—H1A    | 0.8701     |
| C7—H7B| 0.9900     | O1—H1B    | 0.8698     |
| C7—C11| 1.510 (4)  | N11—H11A  | 0.87 (5)   |
| C12—C12| 1.531 (5)  | N11—H11B  | 0.84 (5)   |
| C12—H12A| 0.9900    | N11—H11C  | 0.83 (4)   |
| C12—H12B| 0.9900    | N11—H11D  | 0.92 (4)   |
| O6—W1—O3| 105.05 (9) | N4—C12—C12i| 113.7 (3) |
| O6—W1—O9| 157.36 (9) | N4—C12—H12A| 108.8 |
| O6—W1—N4| 89.46 (8)  | N4—C12—H12B| 108.8 |
| O6—W1—O8| 103.12 (10)| C12—C12—H12A| 108.8 |
| Bond | Symmetry Code | Distance (Å) | Angle (°) |
|------|---------------|--------------|-----------|
| O6—W1—O4  | 86.03 (9)      | C12—C12—H12B | 108.8     |
| O3—W1—O9  | 90.22 (8)      | H12A—C12—H12B | 107.7     |
| O3—W1—N4  | 157.08 (8)     | C1—O4—W1      | 123.67 (15) |
| O3—W1—O8  | 105.14 (9)     | O7—C1—O4      | 123.5 (2)  |
| O3—W1—O4  | 89.42 (8)      | O7—C1—C8      | 119.0 (2)  |
| O9—W1—N4  | 71.28 (7)      | O4—C1—C8      | 117.4 (2)  |
| O9—W1—O4  | 77.36 (8)      | N4—C8—C1      | 115.2 (2)  |
| O8—W1—O9  | 88.46 (8)      | N4—C8—H8A     | 108.5     |
| O8—W1—N4  | 88.23 (8)      | N4—C8—H8B     | 108.5     |
| O8—W1—O4  | 159.79 (8)     | C1—C8—H8A     | 108.5     |
| O4—W1—N4  | 73.71 (7)      | C1—C8—H8B     | 108.5     |
| C11—O9—W1 | 120.89 (17)    | H8A—C8—H8B    | 107.5     |
| C7—N4—W1  | 104.91 (14)    | H10A—N10—H10B | 108 (3)   |
| C7—N4—C12 | 111.36 (19)    | H10A—N10—H10C | 108 (3)   |
| C7—N4—C8  | 110.97 (19)    | H10A—N10—H10D | 106 (3)   |
| C12—N4—W1 | 108.76 (14)    | H10B—N10—H10C | 112 (3)   |
| C12—N4—C8 | 110.94 (19)    | H10B—N10—H10D | 108 (4)   |
| C8—N4—W1  | 109.70 (14)    | H10C—N10—H10D | 113 (4)   |
| N4—C7—H7A | 109.4          | H2A—O2—H2B    | 104.5     |
| N4—C7—H7B | 109.4          | H1A—O1—H1B    | 104.5     |
| N4—C7—C11 | 111.03 (19)    | H11A—N11—H11B | 109 (4)   |
| H7A—C7—H7B| 108.0          | H11A—N11—H11C | 106 (4)   |
| C11—C7—H7A| 109.4          | H11A—N11—H11D | 106 (4)   |
| C11—C7—H7B| 109.4          | H11B—N11—H11C | 113 (4)   |
| O9—C11—C7 | 116.1 (2)      | H11B—N11—H11D | 112 (4)   |
| O5—C11—O9 | 124.1 (3)      | H11C—N11—H11D | 111 (4)   |
| O5—C11—C7 | 119.7 (2)      |                |           |

Symmetry code: (i) −x, −y+1, −z.

**Hydrogen-bond geometry (Å, °)**

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|------|--------|
| C7—H7A···O7ii | 0.99 | 2.48 | 3.384 (3) | 152 |
| C12—H12B···O7iii | 0.99 | 2.77 | 3.548 (3) | 136 |
| C8—H8A···O6iv | 0.99 | 2.54 | 3.319 (3) | 135 |
| C8—H8B···O7iii | 0.99 | 2.46 | 3.319 (4) | 145 |
| O2—H2A···O3 | 0.87 | 1.88 | 2.743 (3) | 171 |

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|          | d (Å) | d (Å) | R (Å)   | θ (°) |
|----------|-------|-------|---------|-------|
| O2—H2B···O5v | 0.87  | 2.90  | 2.763 (3) | 170   |
| O1—H1A···O7  | 0.87  | 2.06  | 2.900 (3) | 161   |
| O1—H1A···O4  | 0.87  | 2.49  | 3.177 (3) | 137   |
| O1—H1B···O2iv | 0.87  | 2.72  | 3.468 (3) | 145   |
| N10—H10A···O6 | 0.95 (4) | 1.78 (4) | 2.727 (3) | 175 (3) |
| N10—H10B···O4vi | 0.81 (4) | 2.20 (4) | 2.996 (3) | 168 (3) |
| N10—H10C···O7iii | 0.90 (4) | 2.01 (4) | 2.876 (3) | 160 (3) |
| N10—H10D···O8vii | 0.87 (4) | 1.87 (4) | 2.736 (3) | 175 (4) |
| N11—H11A···O8  | 0.87 (5) | 2.13 (5) | 2.947 (3) | 156 (4) |
| N11—H11B···O3viii | 0.84 (5) | 2.31 (5) | 3.109 (3) | 160 (4) |
| N11—H11C···O1viii | 0.83 (4) | 2.25 (4) | 2.979 (4) | 147 (4) |
| N11—H11D···O2ii | 0.92 (4) | 1.93 (4) | 2.846 (4) | 173 (4) |

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