The title compound, (C₄H₁₂N₅)₄(C₂H₇N₄O)₂[V₁₀O₂₈]·4H₂O, is a by-product obtained by reacting ammonium metavanadate(V), metformin hydrochloride and acetic acid in the presence of sodium hypochlorite, at pH = 5. The crystal structure comprises a decavanadate(V) anion (V₁₀O₂₈)⁶⁻ lying on an inversion centre in space group P1, while cations and solvent water molecules are placed in general positions, surrounding the anion, and forming numerous N—H···O and O—H···O hydrogen bonds. Metforminium (C₄H₁₂N₅)⁺ and guanylurea (C₂H₇N₄O)⁺ cations display the expected shape. Interestingly, in physiology the latter cation is known to be the main metabolite of the former one. The reported structure thus supports the role of sodium hypochlorite as an oxidizing reagent being able to degrade metformin hydrochloride to form guanylurea.

**Structure description**

Metformin hydrochloride (Metf-HCl: 1,1-dimethylbiguanide hydrochloride) is one of the most commonly prescribed medications for the treatment of type 2 diabetes (Maruthur et al., 2016). On the other hand, coordination compounds of vanadium, including polyoxidovanadates resulting from the condensation of the vanadate anion, likewise exhibit an antidiabetic effect, among other biological activities of interest in medicinal applications (Thompson et al., 2009; Rehder, 2020). We are involved in studies about the chemical crystallography of compounds including both types of antidiabetic species. In
this context, we report here the crystal structure of a compound including a decavanadate(V) anion, metforminium cations, and a degradation product of the latter, guanylurea cation (1-carbamoylguanidinium).

The asymmetric unit of the title compound comprises one-half of the decavanadate(V) anion (V$_{10}$O$_{28}$)$^{6-}$, three cations and two water molecules of solvation. The chemical formula is thus (HMetf)$_4$(HGu)$_2$[V$_{10}$O$_{28}$]·4H$_2$O, where HMetf$^+$ is the metforminium cation (C$_4$H$_7$N$_4$O)$_+$ and HGu$^+$ is the guanylurea cation (C$_9$H$_7$N$_4$O)$_+$. All hydrogen-atom positions in the cations were refined, ensuring that the right tautomers are thus (HMetf)$_4$(HGu)$_2$[V$_{10}$O$_{28}$]·4H$_2$O, where HMetf$^+$ is the metforminium cation (C$_4$H$_7$N$_4$O)$_+$ and HGu$^+$ is the guanylurea cation (C$_9$H$_7$N$_4$O)$_+$. All hydrogen-atom positions in the cations were obtained from difference-Fourier maps, and their positions were refined, ensuring that the right tautomers are included in the structure model (Fig. 1). The decavanadate(V) anion is unprotonated, and displays its usual shape, with a point-group symmetry close to D$_{2h}$ (real C$_2$). The twisted shape of both metforminium cations is also similar to that observed in other compounds (e.g. Sánchez-Lombardo et al., 2014; Farzanfar et al., 2015). For the first cation, the dihedral angle between C1/N12/C25/C26 planes is 60.39 (9)°. Regarding the guanylurea cation, it is nearly planar [maximum distance of 0.009 (4) Å for N12], as in a closely related salt, namely (HMetf)$_3$(HGu)$_2$[V$_{10}$O$_{28}$]·2H$_2$O (Chatkon et al., 2014). In the metforminium cations, the positive charges are not clearly localized, since all C—N bond lengths span a short range, here between 1.321 (3) and 1.355 (3) Å (N—CH$_3$ bonds are omitted). These cations are thus stabilized by resonance, with delocalized π-bonds, a common feature of guanidinium derivatives. In the case of the present guanylurea cation, one π-bond is probably delocalized over C9—N11 and C9—N12.

The cation conformation, as well as their orientations with respect to the highly charged anion favour the formation of numerous hydrogen bonds, the NH$_2$ groups of HMetf$^+$ and HGu$^+$ being the main donors, and the O sites in the anion being the main acceptors (Table 1, Fig. 2). Empty channels oriented parallel to [100] are available in the crystal structure to accommodate water molecules (O16, O17). These mol-

![Figure 1](image1.png)

**Figure 1**
The structures of the molecular entities of the title compound, with displacement ellipsoids drawn at the 40% probability level. The centrosymmetric anion is shown, while the content for cations and water molecules is limited to the asymmetric unit.

![Figure 2](image2.png)

**Figure 2**
Main interactions between the decavanadate(V) anion (polyhedral representation) and the first shell including six cations and four water molecules (ball-and-stick representation). Hydrogen bonds are represented by blue dashed lines, and the label associated to each hydrogen bond refers to its entry in Table 1.
Acetic acid (commercial vinegar) and 2 ml of 5% HCl extracted from a commercial brand; metavanadate (NH4VO3, 1.50 g, 12.1 mmol) and metformin hydrochloride (Metf) were obtained during the reaction between ammonium metavanadate (NH4VO3, 1.50 g, 12.1 mmol) and metformin hydrochloride (Metf-HCl extracted from a commercial brand; 1.70 g, 10.2 mmol) in 50 ml of distilled water, 20 ml of 5% v/v acetic acid (commercial vinegar) and 2 ml of 5% v/v sodium hypochlorite (commercial bleach). In a typical procedure, NH4VO3 was dissolved by gently heating in a water bath followed by addition of Metf-HCl and stirring until dissolution. The water bath was removed, and once the mixture cooled down to room temperature, CH3COOH and NaOCl were evaporated at ambient conditions and the two major products, (H2Metf)3[V10O28]·8H2O (Sánchez-Lombardo et al., 2014) and (HMetr)4(HGu)2[V10O28]·4H2O (estimated yields of ca 30 and 10%, respectively), were separated by fractional crystallization over the course of 5 to 10 d.

Synthesis and crystallization

Orange good-quality single crystals of the title compound were obtained during the reaction between ammonium metavanadate (NH4VO3, 1.50 g, 12.1 mmol) and metformin hydrochloride (Metf-HCl extracted from a commercial brand; 1.70 g, 10.2 mmol) in 50 ml of distilled water, 20 ml of 5% v/v acetic acid (commercial vinegar) and 2 ml of 5% v/v sodium hypochlorite (commercial bleach). In a typical procedure, NH4VO3 was dissolved by gently heating in a water bath followed by addition of Metf-HCl and stirring until dissolution. The water bath was removed, and once the mixture cooled down to room temperature, CH3COOH and NaOCl were evaporated at ambient conditions and the two major products, (H2Metf)3[V10O28]·8H2O (Sánchez-Lombardo et al., 2014) and (HMetr)4(HGu)2[V10O28]·4H2O (estimated yields of ca 30 and 10%, respectively), were separated by fractional crystallization over the course of 5 to 10 d.

Refinement

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

Table 2: Experimental details.

| Crystal data | Description |
|--------------|-------------|
| Chemical formula | (C4H12N5)4(C2H7N4O)2[V10O28]·4H2O |
| Mw | 1756.44 |
| Crystal system, space group | Triclinic, P1 |
| Temperature (K) | 263 |
| a, b, c (Å) | 8.9701 (3), 13.2202 (5), 14.0861 (5) |
| α, β, γ (°) | 99.609 (3), 103.133 (5), 107.676 (3) |
| V (Å³) | 1499.00 (10) |
| Z | 1 |
| Radiation type | Ag | Kα, λ = 0.56083 Å |
| μ (mm⁻¹) | 0.82 |
| Crystal size (mm) | 0.35 × 0.09 × 0.08 |

Data collection

| Diffraactometer | Stoe Stadivari |
| Radiation type | Multi-scan (X-AREA; Stoe & Cie, 2019) |
| Tmin, Tmax | 0.471, 1.000 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 66181, 11970, 7120 |
| Rint | 0.064 |
| R1 | 0.040, 0.101, 0.83 |
| No. of parameters | 488 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³) | 0.50, −0.75 |

Computer programs: X-AREA (Stoe & Cie, 2019), SHELXTL2018/2 (Sheldrick, 2015a), SHELXL2018/3 (Sheldrick, 2015b), Mercury (Macrae et al., 2020) and publCIF (Westrip, 2010).

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full crystallographic data

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Bis[(amino(iminiumyl)methyl)urea] tetrakis{2-[(dimethylamino)(iminiumyl)methyl]guanidine} di-µ₆-oxo-tetra-µ₃-oxido-tetradeca-µ₂-oxido-octaoxidodecavanadium(V) tetrahydrate

Aarón Pérez-Benítez, Jorge Luis Ariza-Ramírez, Monserrat Fortis-Valera, Rosa Elena Arroyo-Carmona, María Isabel Martínez de la Luz, Diego Ramírez-Contreras and Sylvain Bernès

Bis[(amino(iminiumyl)methyl)urea] tetrakis{2-[(dimethylamino)(iminiumyl)methyl]guanidine} di-µ₆-oxo-tetra-µ₃-oxido-tetradeca-µ₂-oxido-octaoxidodecavanadium(V) tetrahydrate

Crystal data

(C₆H₁₂N₅)₄(C₂H₇N₄O₂)[V₁₀O₂₈]·₄H₂O

Mr = 1756.44
Triclinic, P

a = 8.9701 (3) Å
b = 13.2202 (5) Å
c = 14.0861 (5) Å

α = 99.609 (3)°
β = 103.133 (3)°
γ = 107.676 (3)°

V = 1499.00 (10) Å³

Z = 1

F(000) = 888

Dₐ = 1.946 Mg m⁻³

Ag Kα radiation, λ = 0.56083 Å

Cell parameters from 44097 reflections

θ = 2.2–30.9°

μ = 0.82 mm⁻¹

T = 263 K

0.35 × 0.09 × 0.08 mm

Data collection

Stoe Stadivari

radiation source: Sealed X-ray tube, Axo Astix-

f Microfocus source

Graded multilayer mirror monochromator

Detector resolution: 5.81 pixels mm⁻¹

ω scans

(X-AREA; Stoe & Cie, 2019)

T = 0.471, T_max = 1.000

66181 measured reflections

11970 independent reflections

7120 reflections with I > 2σ(I)

R-factor = 0.064

θ_max = 26.0°, θ_min = 2.4°

h = −12→14

k = −20→20

l = −22→22

Refinement

Refinement on F²

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.040

wR(F²) = 0.101

S = 0.83

11970 reflections

488 parameters

0 restraints

0 constraints

Primary atom site location: dual

Secondary atom site location: difference Fourier

map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ²(Fc²) + (0.0523P)²]

where P = (Fc² + 2Fos²)/3

(Δ/σ)max = 0.001

Δρ_max = 0.50 e Å⁻³

Δρ_min = −0.75 e Å⁻³
Special details

**Refinement.** High-resolution data were collected ($d_{	ext{min}} = 0.64$ Å), and all H atoms were discernible in difference-Fourier maps. Methyl H atoms were placed in calculated positions, with $U_{	ext{iso}}(H) = 1.5U_{	ext{eq}}$(carrier C). The positions for other H atoms were freely refined, and their isotropic displacements were calculated as $U_{	ext{iso}}(H) = 1.2U_{	ext{eq}}$(carrier N) and $U_{	ext{iso}}(H) = 1.5U_{	ext{eq}}$(carrier O).

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

|   | x       | y       | z       | $U_{	ext{iso}}*/U_{	ext{eq}}$ |
|---|---------|---------|---------|-------------------------------|
| V1| −0.18346 (3) | 0.50285 (3) | 0.48387 (2) | 0.02031 (7) |
| V2| 0.11454 (4)  | 0.67569 (3)  | 0.66199 (2)  | 0.02333 (7) |
| V3| 0.29658 (4)  | 0.73708 (3)  | 0.50272 (3)  | 0.02746 (8) |
| V4| 0.00747 (4)  | 0.56616 (3)  | 0.32784 (2)  | 0.02919 (7) |
| V5| −0.04938 (4) | 0.74348 (3)  | 0.47654 (3)  | 0.02779 (8) |
| O1| −0.35393 (15)| 0.39698 (12) | 0.47679 (11) | 0.0256 (3)  |
| O2| 0.14821 (19) | 0.71282 (13) | 0.78213 (11) | 0.0346 (3)  |
| O3| 0.47419 (18) | 0.82185 (14) | 0.51151 (13) | 0.0389 (4)  |
| O4| −0.02963 (18)| 0.52272 (14) | 0.20790 (11) | 0.0334 (3)  |
| O5| −0.1391 (2)  | 0.83039 (14) | 0.46158 (13) | 0.0401 (4)  |
| O6| −0.18582 (14)| 0.45457 (11) | 0.34529 (9)  | 0.0205 (3)  |
| O7| −0.00402 (17)| 0.75691 (12) | 0.61355 (11) | 0.0275 (3)  |
| O8| 0.16426 (17) | 0.82016 (12) | 0.48728 (11) | 0.0293 (3)  |
| O9| −0.08722 (16)| 0.66811 (12) | 0.34085 (11) | 0.0266 (3)  |
| O10| −0.24798 (15)| 0.60808 (12)| 0.46689 (11) | 0.0257 (3)  |
| O11| −0.09736 (14)| 0.54661 (11) | 0.62762 (10) | 0.0209 (3)  |
| O12| 0.30173 (16) | 0.75443 (12) | 0.64019 (11) | 0.0271 (3)  |
| O13| 0.21944 (16) | 0.66484 (12) | 0.36802 (11) | 0.0270 (3)  |
| O14| 0.05231 (14) | 0.60398 (11) | 0.49536 (10) | 0.0199 (2)  |
| C1| −0.2523 (3)  | 0.8595 (2)   | 0.76688 (18) | 0.0349 (5)  |
| C2| 0.0296 (3)   | 0.95180 (18) | 0.81639 (17) | 0.0315 (4)  |
| C3| 0.1549 (3)   | 0.8988 (2)   | 0.96161 (19) | 0.0466 (6)  |
| H3A| 0.191943 (3)| 0.952085 | 1.025219 | 0.070* |
| H3B| 0.222871 (3)| 0.854931 | 0.962427 | 0.070* |
| H3C| 0.043527 (3)| 0.852377 | 0.950131 | 0.070* |
| C4| 0.3258 (3)   | 1.0073 (4)  | 0.8715 (3)  | 0.0729 (11) |
| H4C| 0.344844 (3) | 0.959712 | 0.819942 | 0.109* |
| H4D| 0.407592 (3) | 1.021958 | 0.934421 | 0.109* |
| H4E| 0.331692 (3) | 1.075066 | 0.853596 | 0.109* |
| N1| −0.2665 (3)  | 0.79810 (18)| 0.67858 (16)| 0.0376 (5) |
| H1A| −0.178 (3)  | 0.794 (2)  | 0.662 (2)  | 0.045*   |
| H1B| −0.351 (3)  | 0.771 (2)  | 0.636 (2)  | 0.045*   |
| N2| −0.3897 (3)  | 0.8602 (2) | 0.7888 (2) | 0.0537 (7) |
| H2A| −0.379 (4)  | 0.902 (3)  | 0.848 (3)  | 0.064*   |
| H2B| −0.484 (4)  | 0.829 (3)  | 0.747 (3)  | 0.064*   |
| N3| −0.1127 (2)  | 0.91530 (19)| 0.83847 (15)| 0.0404 (5)|
| N4| 0.0405 (3)   | 0.99452 (18)| 0.73832 (17)| 0.0370 (4)|
| H4A| −0.045 (3)  | 1.001 (2)  | 0.700 (2)  | 0.044*   |
| H4B| 0.117 (3)   | 1.006 (2)  | 0.717 (2)  | 0.044*   |
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|     | V3      | V4      | V5      | O1      | O2      | O3      | O4      | O5      | O6      | O7      | O8      | O9      | O10     | O11     | O12     | O13     | O14     | C1      | C2      | C3      | C4      | N1      | N2      | N3      | N4      | N5      | C5      | C6      | C7      | C8      | N6      | N7      | N8      | N9      | N10     | C9      | C10     | N11     | N12     | N13     | N14     | O15     | O16     | O17     |
|-----|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
### Geometric parameters (Å, °)

| Bond/Angle | Distance/Angle | Distance/Angle | Distance/Angle |
|------------|----------------|---------------|---------------|
| V1—O10     | 1.6925 (14)    | C4—N5         | 1.454 (3)     |
| V1—O1      | 1.7005 (13)    | C4—H4C        | 0.9600        |
| V1—O11     | 1.9126 (13)    | C4—H4D        | 0.9600        |
| V1—O6      | 1.9430 (13)    | C4—H4E        | 0.9600        |
| V1—O14     | 2.0836 (12)    | N1—H1A        | 0.89 (3)      |
| V1—O14\*   | 2.1105 (13)    | N1—H1B        | 0.79 (3)      |
| V1—V5      | 3.0691 (5)     | N2—H2A        | 0.89 (4)      |
| V1—V3\*    | 3.0755 (5)     | N2—H2B        | 0.85 (3)      |
| V2—O2      | 1.6113 (15)    | N4—H4A        | 0.87 (3)      |
| V2—O12     | 1.8102 (14)    | N4—H4B        | 0.79 (3)      |
| V2—O7      | 1.8324 (14)    | C5—N6         | 1.329 (3)     |
| V2—O6\*    | 2.0062 (14)    | C5—N8         | 1.334 (3)     |
| V2—O11     | 2.0238 (13)    | C5—N7         | 1.334 (3)     |
| V2—O14     | 2.2495 (13)    | C6—N9         | 1.324 (3)     |
| V2—V4\*    | 3.0888 (5)     | C6—N10        | 1.331 (2)     |
| V2—V5      | 3.1024 (5)     | C6—N8         | 1.355 (3)     |
| V3—O3      | 1.6091 (15)    | C7—N10        | 1.457 (3)     |
| V3—O13     | 1.8407 (15)    | C7—H7C        | 0.9600        |
| V3—O8      | 1.8484 (15)    | C7—H7D        | 0.9600        |
| V3—O12     | 1.8998 (15)    | C7—H7E        | 0.9600        |
| V3—O1\*    | 2.0361 (15)    | C8—N10        | 1.455 (3)     |
| V3—O14     | 2.3243 (12)    | C8—H8A        | 0.9600        |
| V3—V5      | 3.0697 (5)     | C8—H8B        | 0.9600        |
| V3—V4      | 3.0951 (5)     | C8—H8C        | 0.9600        |
| V4—O4      | 1.6122 (15)    | N6—H6A        | 0.91 (3)      |
| V4—O9      | 1.8042 (15)    | N6—H6B        | 0.80 (3)      |
| V4—O13     | 1.8427 (13)    | N7—H7A        | 0.75 (3)      |
| V4—O6      | 2.0012 (13)    | N7—H7B        | 0.81 (3)      |
| V4—O1\*\* | 2.0196 (14)    | N9—H9A        | 0.84 (3)      |
| V4—O14     | 2.2434 (13)    | N9—H9B        | 0.81 (3)      |
| V4—V5      | 3.1169 (5)     | C9—N11        | 1.296 (3)     |
| V5—O5      | 1.6054 (16)    | C9—N12        | 1.309 (3)     |
| V5—O8      | 1.8331 (14)    | C9—N13        | 1.361 (3)     |
| V5—O7      | 1.8457 (15)    | C10—O15       | 1.223 (3)     |
| V5—O9      | 1.9040 (15)    | C10—N14       | 1.316 (3)     |
| V5—O10     | 2.0660 (14)    | C10—N13       | 1.405 (3)     |
| V5—O14     | 2.3191 (13)    | N11—H11A      | 0.84 (3)      |
| C1—N3      | 1.321 (3)      | N11—H11B      | 0.70 (3)      |
| C1—N1      | 1.323 (3)      | N12—H12A      | 0.83 (3)      |
| C1—N2      | 1.339 (3)      | N12—H12B      | 0.82 (3)      |
| C2—N4      | 1.327 (3)      | N13—H13       | 0.82 (3)      |
| C2—N5      | 1.328 (3)      | N14—H14A      | 0.88 (4)      |
| C2—N3      | 1.347 (3)      | N14—H14B      | 0.74 (3)      |
| C3—N5      | 1.453 (3)      | O16—H16A      | 0.80 (3)      |
| C3—H3A     | 0.9600        | O16—H16B      | 0.71 (3)      |
| C3—H3B     | 0.9600        | O17—H17A      | 0.75 (3)      |
C3—H3C  0.9600  O17—H17B  0.78 (3)

| Bond | Length (Å) | Bond | Length (Å) |
|------|------------|------|------------|
| O10—V1—O1 | 105.46 (7) | O10—V5—O14 | 74.12 (5) |
| O10—V1—O11 | 98.71 (6) | O5—V5—V1 | 131.07 (7) |
| O1—V1—O11 | 96.92 (6) | O8—V5—V1 | 125.08 (5) |
| O10—V1—O6 | 95.97 (6) | O7—V5—V1 | 78.36 (5) |
| O1—V1—O6 | 95.74 (6) | O9—V5—V1 | 78.38 (4) |
| O11—V1—O6 | 157.30 (5) | O10—V5—V1 | 31.41 (4) |
| O10—V1—O14 | 88.53 (6) | O14—V5—V1 | 42.73 (3) |
| O1—V1—O14 | 165.98 (6) | O5—V5—V3 | 137.49 (7) |
| O11—V1—O14 | 81.62 (5) | O8—V5—V3 | 33.66 (5) |
| O6—V1—O14 | 81.53 (5) | O7—V5—V3 | 85.71 (4) |
| O10—V1—O14i | 167.14 (6) | O9—V5—V3 | 83.45 (4) |
| O1—V1—O14i | 87.35 (6) | O10—V5—V3 | 122.77 (4) |
| O11—V1—O14i | 80.52 (5) | O14—V5—V3 | 48.69 (3) |
| O6—V1—O14i | 81.33 (5) | V1—V5—V3 | 91.420 (13) |
| O14—V1—O14i | 78.65 (5) | O5—V5—V2 | 134.32 (7) |
| O10—V1—V5 | 39.51 (5) | O8—V5—V2 | 82.26 (5) |
| O1—V1—V5 | 144.96 (5) | O7—V5—V2 | 32.36 (4) |
| O11—V1—V5 | 89.98 (4) | O9—V5—V2 | 123.48 (4) |
| O6—V1—V5 | 90.41 (4) | O10—V5—V2 | 82.77 (4) |
| O14—V1—V5 | 49.05 (4) | O14—V5—V2 | 46.29 (3) |
| O14i—V1—V5 | 127.69 (3) | V1—V5—V2 | 61.613 (12) |
| O10—V1—V3i | 143.79 (5) | V3—V5—V2 | 61.042 (11) |
| O1—V1—V3i | 38.33 (5) | O5—V5—V4 | 133.62 (7) |
| O11—V1—V3i | 88.31 (4) | O8—V5—V4 | 81.96 (5) |
| O6—V1—V3i | 90.02 (4) | O7—V5—V4 | 123.82 (5) |
| O14—V1—V3i | 127.68 (4) | O9—V5—V4 | 31.82 (4) |
| O14i—V1—V3i | 49.04 (3) | O10—V5—V4 | 80.43 (4) |
| V5—V1—V3i | 176.557 (14) | O10—V5—V4 | 45.90 (3) |
| O2—V2—O12 | 104.71 (7) | O14—V5—V4 | 61.206 (11) |
| O2—V2—O7 | 103.32 (7) | V1—V5—V4 | 60.033 (12) |
| O12—V2—O7 | 95.37 (7) | V2—V5—V4 | 91.958 (13) |
| O2—V2—O6i | 99.03 (7) | V1—O1—V3i | 110.47 (6) |
| O12—V2—O6i | 89.97 (6) | V1—O6—V4 | 105.96 (6) |
| O7—V2—O6i | 154.83 (6) | V1—O6—V2i | 106.54 (6) |
| O2—V2—O11 | 98.92 (7) | V4—O6—V2i | 100.85 (6) |
| O12—V2—O11 | 154.25 (6) | V2—O7—V5 | 115.02 (8) |
| O7—V2—O11 | 88.94 (6) | V5—O8—V3 | 112.99 (8) |
| O6i—V2—O11 | 76.20 (5) | V4—O9—V5 | 114.37 (7) |
| O2—V2—O14 | 173.33 (7) | V1—O10—V5 | 109.08 (6) |
| O12—V2—O14 | 80.50 (6) | V1—O11—V4i | 108.12 (6) |
| O7—V2—O14 | 80.05 (6) | V1—O11—V2 | 106.80 (6) |
| O6i—V2—O14 | 76.60 (5) | V4i—O11—V2 | 99.62 (5) |
| O11—V2—O14 | 75.26 (5) | V2—O12—V3 | 115.31 (7) |
| O2—V2—V4i | 88.84 (6) | V3—O13—V4 | 114.34 (7) |
| O12—V2—V4i | 129.46 (5) | V1—O14—V1i | 101.35 (5) |
| O7—V2—V4i | 129.07 (5) | V1—O14—V4 | 93.34 (5) |
| Bond                  | Distance (Å) | Angle (°)          |
|----------------------|--------------|--------------------|
| O6—V2—V4i           | 39.52 (4)    | V1i—O14—V4        | 93.95 (5) |
| O11—V2—V4i          | 40.14 (4)    | V1—O14—V2         | 93.60 (5) |
| O14—V2—V4i          | 84.60 (3)    | V1i—O14—V2        | 93.05 (5) |
| O2—V2—V5            | 135.78 (6)   | V4—O14—V2         | 168.99 (7) |
| O12—V2—V5           | 83.17 (5)    | V1—O14—V5         | 88.22 (4) |
| O7—V2—V5            | 32.62 (5)    | V1i—O14—V5        | 170.40 (6) |
| O6—V2—V5            | 124.76 (4)   | V4—O14—V5         | 86.16 (4) |
| O11—V2—V5           | 87.04 (4)    | V2—O14—V5         | 85.53 (5) |
| O14—V2—V5           | 48.18 (3)    | V1—O14—V3         | 170.95 (7) |
| V4—V2—V5            | 119.903 (14) | V1i—O14—V3        | 87.68 (4) |
| O3—V3—O13           | 102.26 (8)   | V4—O14—V3         | 85.29 (4) |
| O3—V3—O8            | 103.30 (8)   | V2—O14—V3         | 86.51 (4) |
| O13—V3—O8           | 92.71 (7)    | V5—O14—V3         | 82.77 (4) |
| O3—V3—O12           | 101.81 (7)   | N3—C1—N1          | 125.0 (2) |
| O13—V3—O12          | 154.56 (6)   | N3—C1—N2          | 116.8 (2) |
| O8—V3—O12           | 89.66 (7)    | N1—C1—N2          | 118.1 (2) |
| O3—V3—O1i           | 100.28 (7)   | N4—C2—N5          | 119.8 (2) |
| O13—V3—O1i          | 85.14 (6)    | N4—C2—N3          | 122.1 (2) |
| O8—V3—O1i           | 156.22 (6)   | N5—C2—N3          | 117.8 (2) |
| O12—V3—O1i          | 82.61 (6)    | N5—C3—H3A         | 109.5 |
| O3—V3—O14           | 174.67 (7)   | N5—C3—H3B         | 109.5 |
| O13—V3—O14          | 78.49 (5)    | H3A—C3—H3B        | 109.5 |
| O8—V3—O14           | 81.89 (5)    | N5—C3—H3C         | 109.5 |
| O12—V3—O14          | 76.79 (5)    | H3A—C3—H3C        | 109.5 |
| O1i—V3—O14          | 74.47 (5)    | H3B—C3—H3C        | 109.5 |
| O3—V3—V5            | 136.65 (7)   | N5—C4—H4C         | 109.5 |
| O13—V3—V5           | 85.37 (4)    | N5—C4—H4D         | 109.5 |
| O8—V3—V5            | 33.35 (4)    | H4C—C4—H4D        | 109.5 |
| O12—V3—V5           | 82.78 (4)    | N5—C4—H4E         | 109.5 |
| O1i—V3—V5           | 122.98 (4)   | H4C—C4—H4E        | 109.5 |
| O14—V3—V5           | 48.54 (3)    | H4D—C4—H4E        | 109.5 |
| O3—V3—V1i           | 131.48 (7)   | C1—N1—H1A         | 120.9 (18) |
| O13—V3—V1i          | 79.52 (5)    | C1—N1—H1B         | 121 (2) |
| O8—V3—V1i           | 125.17 (5)   | H1A—N1—H1B        | 117 (3) |
| O12—V3—V1i          | 78.46 (5)    | C1—N2—H2A         | 117 (2) |
| O1i—V3—V1i          | 31.20 (4)    | C1—N2—H2B         | 123 (2) |
| O14—V3—V1i          | 43.29 (3)    | H2A—N2—H2B        | 119 (3) |
| V5—V3—V1i           | 91.829 (13)  | C1—N3—C2          | 121.3 (2) |
| O3—V3—V4            | 134.99 (6)   | C2—N4—H4A         | 120.2 (18) |
| O13—V3—V4           | 32.85 (4)    | C2—N4—H4B         | 125 (2) |
| O8—V3—V4            | 82.37 (5)    | H4A—N4—H4B        | 114 (3) |
| O12—V3—V4           | 123.02 (4)   | C2—N5—C3          | 120.9 (2) |
| O1i—V3—V4           | 83.12 (4)    | C2—N5—C4          | 121.5 (2) |
| O14—V3—V4           | 46.25 (3)    | C3—N5—C4          | 117.5 (2) |
| V5—V3—V4            | 60.738 (12)  | N6—C5—N8          | 118.3 (2) |
| V1i—V3—V4           | 62.131 (11)  | N6—C5—N7          | 117.9 (2) |
| O4—V4—O9            | 104.49 (7)   | N8—C5—N7          | 123.7 (2) |
| O4—V4—O13           | 103.24 (7)   | N9—C6—N10         | 119.0 (2) |
| Bond         | Angle 1 | Angle 2 | Angle 3 |
|--------------|---------|---------|---------|
| O9—V4—O13   | 95.73 (7) | N9—C6—N8 | 122.24 (18) |
| O4—V4—O6    | 98.59 (7) | N10—C6—N8 | 118.44 (19) |
| O9—V4—O6    | 90.50 (6) | N10—C7—H7C | 109.5 |
| O13—V4—O6   | 154.96 (6) | N10—C7—H7D | 109.5 |
| O4—V4—O11i  | 98.58 (7) | H7C—C7—H7D | 109.5 |
| O9—V4—O11i  | 154.97 (6) | N10—C7—H7E | 109.5 |
| O13—V4—O11i | 88.24 (6) | H7C—C7—H7E | 109.5 |
| O6—V4—O11i  | 76.41 (5) | H7D—C7—H7E | 109.5 |
| O4—V4—O14   | 172.62 (7) | N10—C8—H8A | 109.5 |
| O9—V4—O14   | 81.17 (6) | N10—C8—H8B | 109.5 |
| O13—V4—O14  | 80.64 (5) | H8A—C8—H8B | 109.5 |
| O6—V4—O14   | 76.39 (5) | N10—C8—H8C | 109.5 |
| O11i—V4—O14 | 75.10 (5) | H8A—C8—H8C | 109.5 |
| O4—V4—V2i   | 88.30 (6) | H8B—C8—H8C | 109.5 |
| O9—V4—V2i   | 130.12 (5) | C5—N6—H6A | 121.6 (18) |
| O13—V4—V2i  | 128.48 (5) | C5—N6—H6B | 116 (2) |
| O6—V4—V2i   | 39.63 (4) | H6A—N6—H6B | 122 (3) |
| O11i—V4—V2i | 40.24 (4) | C5—N7—H7A | 122 (2) |
| O14—V4—V2i  | 84.39 (3) | C5—N7—H7B | 123 (2) |
| O4—V4—V3    | 135.96 (6) | H7A—N7—H7B | 115 (3) |
| O9—V4—V3    | 84.25 (5) | C5—N8—C6 | 118.98 (18) |
| O13—V4—V3   | 32.81 (5) | C6—N9—H9A | 120 (2) |
| O6—V4—V3    | 124.78 (4) | C6—N9—H9B | 126 (2) |
| O11i—V4—V3  | 85.92 (4) | H9A—N9—H9B | 112 (3) |
| O14—V4—V3   | 48.45 (3) | C6—N10—C8 | 120.82 (19) |
| V2i—V4—V3   | 119.12 (14) | C6—N10—C7 | 120.68 (19) |
| O4—V4—V5    | 138.15 (6) | C8—N10—C7 | 118.08 (17) |
| O9—V4—V5    | 33.81 (5) | N11—C9—N12 | 119.9 (2) |
| O13—V4—V5   | 83.95 (5) | N11—C9—N13 | 119.58 (19) |
| O6—V4—V5    | 87.98 (4) | N12—C9—N13 | 120.6 (2) |
| O11i—V4—V5  | 123.03 (4) | O15—C10—N14 | 123.2 (2) |
| O14—V4—V5   | 47.94 (3) | O15—C10—N13 | 122.1 (2) |
| V2i—V4—V5   | 119.587 (14) | N14—C10—N13 | 114.7 (2) |
| V3—V4—V5    | 59.228 (12) | C9—N11—H11A | 121.0 (19) |
| O5—V5—O8    | 103.84 (8) | C9—N11—H11B | 124 (2) |
| O5—V5—O7    | 102.08 (8) | H11A—N11—H11B | 116 (3) |
| O8—V5—O7    | 92.75 (7) | C9—N12—H12A | 115 (2) |
| O5—V5—O9    | 101.85 (8) | C9—N12—H12B | 119 (2) |
| O8—V5—O9    | 91.20 (7) | H12A—N12—H12B | 126 (3) |
| O7—V5—O9    | 154.03 (7) | C9—N13—C10 | 124.73 (18) |
| O5—V5—O10   | 99.67 (7) | C9—N13—C10 | 115.2 (19) |
| O8—V5—O10   | 156.41 (6) | C10—N13—C10 | 120.0 (19) |
| O7—V5—O10   | 84.18 (6) | C10—N14—H14A | 115 (2) |
| O9—V5—O10   | 82.01 (6) | C10—N14—H14B | 123 (3) |
| O5—V5—O14   | 173.78 (7) | H14A—N14—H14B | 122 (3) |
| O8—V5—O14   | 82.35 (5) | H16A—O16—H16B | 111 (3) |
| O7—V5—O14   | 77.92 (5) | H17A—O17—H17B | 99 (3) |
| Bond                  | Angle (°) | Error 1 | Bond                  | Angle (°) | Error 1 |
|----------------------|----------|---------|----------------------|----------|---------|
| O10—V1—O1—V3i       | −179.43  | 7       | O7—V2—O12—V3        | −70.42    | 9       |
| O11—V1—O1—V3i       | −78.38   | 8       | O6′—V2—O12—V3       | 84.95     | 8       |
| O6—V1—O1—V3i        | 82.74    | 7       | O11—V2—O12—V3       | 28.32     | 19      |
| O14—V1—O1—V3i       | 4.8      | 3       | O14—V2—O12—V3       | 8.53      | 7       |
| O14′—V1—O1—V3i      | 1.73     | 7       | V4′—V2—O12—V3       | 83.39     | 9       |
| V5—V1—O1—V3i        | −178.31  | 3       | V5—V2—O12—V3        | −40.10    | 7       |
| O2—V2—O7—V5         | 174.91   | 8       | O3—V3—O12—V2        | 176.91    | 9       |
| O12—V2—O7—V5        | 68.39    | 9       | O13—V3—O12—V2       | −22.2     | 2       |
| O6—V2—O7—V5         | −33.07   | 19      | O8—V3—O12—V2        | 73.38     | 9       |
| O11—V2—O7—V5        | −86.17   | 8       | O1′—V3—O12—V2       | −84.07    | 8       |
| O14—V2—O7—V5        | −10.96   | 7       | O14—V3—O12—V2       | −8.37     | 7       |
| V4—V2—O7—V5         | −85.57   | 8       | V5—V3—O12—V2        | 40.66     | 7       |
| O5—V5—O7—V2         | −175.65  | 9       | V1′—V3—O12—V2       | −52.71    | 7       |
| O8—V5—O7—V2         | −70.87   | 9       | V4—V3—O12—V2        | −7.28     | 11      |
| O9—V5—O7—V2         | 27.55    | 19      | O3—V3—O13—V4        | −175.67   | 9       |
| O10—V5—O7—V2        | 85.67    | 8       | O8—V3—O13—V4        | −71.43    | 9       |
| O14—V5—O7—V2        | 10.70    | 7       | O12—V3—O13—V4       | 23.5      | 2       |
| V1—V5—O7—V2         | 54.41    | 7       | O1′—V3—O13—V4       | 84.84     | 8       |
| V3—V5—O7—V2         | −37.93   | 7       | O14—V3—O13—V4       | 9.73      | 7       |
| V4—V5—O7—V2         | 11.29    | 10      | V5—V3—O13—V4        | −38.87    | 7       |
| O5—V5—O8—V3         | −178.80  | 9       | V1′—V3—O13—V4       | 53.86     | 7       |
| O7—V5—O8—V3         | 78.04    | 9       | O4—V4—O13—V3        | 176.40    | 9       |
| O9—V5—O8—V3         | −76.28   | 8       | O9—V4—O13—V3        | 70.03     | 9       |
| O10—V5—O8—V3        | −3.7     | 2       | O6—V4—O13—V3        | −33.6     | 2       |
| O14—V5—O8—V3        | 0.62     | 7       | O11′—V4—O13—V3      | −85.2     | 8       |
| V1—V5—O8—V3         | 0.33     | 11      | O14—V4—O13—V3       | −10.01    | 7       |
| V2—V5—O8—V3         | 47.35    | 7       | V2′—V4—O13—V3       | −85.08    | 9       |
| V4—V5—O8—V3         | −45.74   | 7       | V5—V4—O13—V3        | 38.28     | 7       |
| O3—V3—O8—V5         | −179.37  | 9       | N1—C1—N3—C2         | 27.6      | 4       |
| O13—V3—O8—V5        | 77.35    | 8       | N2—C1—N3—C2         | −156.9    | 3       |
| O12—V3—O8—V5        | −77.32   | 8       | N4—C2—N3—C1         | 40.9      | 4       |
| O1′—V3—O8—V5        | −6.7     | 2       | N5—C2—N3—C1         | −146.1    | 2       |
| O14—V3—O8—V5        | −0.62    | 7       | N4—C2—N5—C3         | −173.7    | 2       |
| V1′—V3—O8—V5        | −1.73    | 11      | N3—C2—N5—C3         | 13.1      | 4       |
| V4—V3—O8—V5         | 46.10    | 7       | N4—C2—N5—C4         | 2.3       | 4       |
| O4—V4—O9—V5         | −175.39  | 8       | N3—C2—N5—C4         | −170.9    | 3       |
| O13—V4—O9—V5        | −70.10   | 8       | N6—C5—N8—C6         | −160.5    | 2       |
| O6—V4—O9—V5         | 85.61    | 8       | N7—C5—N8—C6         | 22.6      | 3       |
| O11′—V4—O9—V5       | 28.06    | 18      | N9—C6—N8—C5         | 44.9      | 3       |
| O14—V4—O9—V5        | 9.47     | 7       | N10—C6—N8—C5        | −141.8    | 2       |
| V2′—V4—O9—V5        | 84.38    | 8       | N9—C6—N10—C8        | −3.0      | 3       |
| V3—V4—O9—V5         | −39.32   | 6       | N8—C6—N10—C8        | −176.5    | 2       |
| O1—V1—O10—V5        | 178.99   | 7       | N9—C6—N10—C7        | −175.4    | 2       |
| O11—V1—O10—V5       | 79.26    | 7       | N8—C6—N10—C7        | 11.1      | 3       |
| O6—V1—O10—V5        | −83.36   | 7       | N11—C9—N13—C10      | 179.5     | 2       |
| O14—V1—O10—V5       | −2.03    | 7       | N12—C9—N13—C10      | −1.6      | 4       |
| O14′—V1—O10—V5      | −6.3     | 3       | O15—C10—N13—C9      | 0.5       | 4       |
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Symmetry code: (i) \(-x, -y+1, -z+1\); (ii) \(-x, -y+2, -z+1\); (iii) \(x-1, y, z\); (iv) \(x, -y+1, -z\); (v) \(x+1, y, z\); (vi) \(-x+1, -y+2, -z+1\); (vii) \(-x-1, -y+1, -z+1\).