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Tetraammonium bis(metforminium) di-μ_6-oxido-tetra-μ_3-oxido-tetradeca-μ_2-oxido-octaoxidodeca-vanadium(V) hexahydrate

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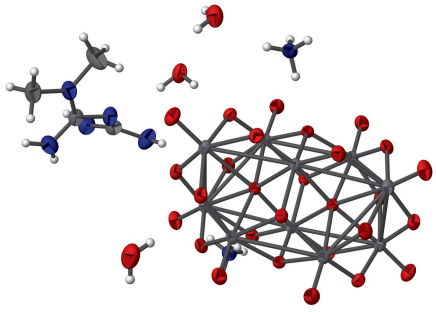
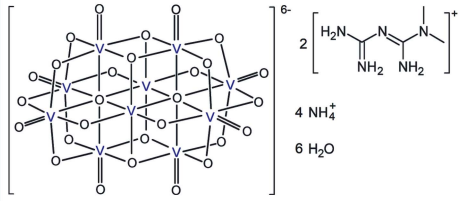
The title compound, $(NH_4)_4(C_4H_{12}N_5)_2[V_{10}O_{28}]\cdot 6H_2O$, crystallizes with the decavanadate anion placed on an inversion centre in space group $P\overline{1}$. This anion is surrounded by a first shell of ammonium cations and water molecules, forming efficient N—H···O and O—H···O hydrogen bonds. A second shell includes metforminium monocations with a twisted geometry, also forming numerous intermolecular hydrogen bonds. The complex three-dimensional network of non-covalent interactions affords a crystal structure in which the cations and anions are densely packed.

3D view



Chemical scheme



Structure description

Metformin hydrochloride (Metf·HCl: 1,1-dimethylbiguanide hydrochloride; Niranjana Devi *et al.*, 2017) is the first-line therapy for type 2 diabetes. On the other hand, some anionic or cationic vanadium species, such as vanadate and vanadyl, have also been shown to be useful in the treatment of human diabetes (Domingo & Gómez, 2016). Based on this background, several groups belonging to the Autonomous University of Puebla are involved in the synthesis of compounds including both metformin and oxidovanadate derivatives, with the hope of achieving synergistic effects (Sánchez-Lombardo *et al.*, 2014). The associated chemical crystallography is rather complex, because due to its basic character metformin can be found in various states of protonation (neutral, cationic or dicationic forms), while the degree of condensation for the vanadate moiety strongly depends on the pH of the reaction medium. Finally, most of these compounds are crystallized with a number of water molecules, which is unpredictable. The compound

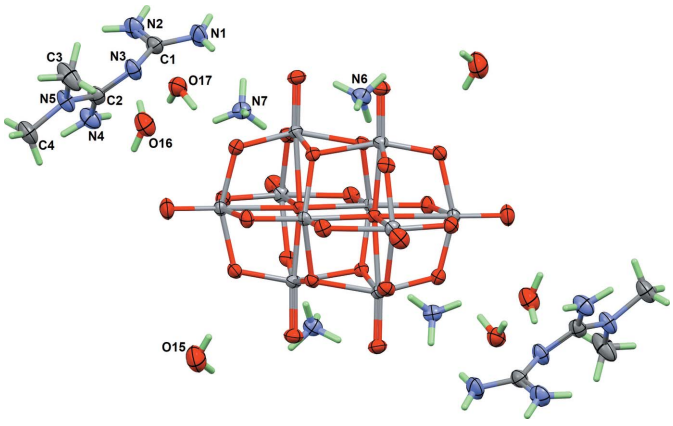


Figure 1
The molecular entities in the structure of the title compound, with displacement ellipsoids for non-H atoms at the 50% probability level. Cations and water molecules in the asymmetric unit are labelled.

reported here includes one $(V_{10}O_{28})^{6-}$ anion, four ammonium cations, two metforminium(1+) cations $HMetf^{+}$, and six water molecules (Fig. 1).

The $(V_{10}O_{28})^{6-}$ anion is situated on an inversion centre in space group $P\overline{1}$, and approaches the expected D_{2h} symmetry, which has been extensively reported (Bošnjaković-Pavlović *et al.*, 2011). The negative charges are balanced by four NH_4^+ and two $HMetf^+$ cations. The high resolution of the measured diffraction data (d_{min} = 0.56 Å) unequivocally establishes that there is no protonation of the decavanadate. The $HMetf^+$ monocation has its charge located mainly on N2. Furthermore, this cation is characterized by a dihedral angle of 54.85 (5)° between planes C2–C4/N3–N5 and C1/N1–N3. This twisted geometry is observed in several other compounds of metforminium(1+). Indeed, metformin and its cations $HMetf^+$ and H_2Metf^{2+} are highly flexible entities: the twist angle for 93 structures recovered from the CSD (Groom *et al.*, 2016) varies from 1 to 85°.

In the crystal structure, anions and cations are well distributed, in such a way that the repulsive Coulombic forces between the highly charged anions are minimized. The decavanadate anion, the cations, and the crystal water molecules engage in an extensive network of hydrogen bonds (Table 1). All N—H and O—H groups present in the asymmetric unit serve as donor groups. The two strongest hydrogen bonds are formed between the anion and one ammonium [N6—H6*A*···O8^{v}; symmetry code: (v) $-x+1, -y+2, -z+1$], as well as between the anion and a water molecule (O17—H17*A*···O13; Fig. 2). As a consequence of the large number of hydrogen bonds, ions and molecules are packed in an efficient way (Fig. 3), as reflected in the quite high Kitaigorodskii packing index of 0.743 (Kitaigorodskii, 1965; Spek, 2009). The mean atomic volume for non-H atoms is 16.5 Å^3 for the title compound, similar to those calculated for previously reported structures in this series (Sánchez-Lombardo *et al.*, 2014). This indicates that in this family of ionic compounds, the lattice energy can be optimized through the inclusion of a suitable number of water molecules.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O10^{i}	0.818 (19)	2.080 (19)	2.8981 (12)	178 (2)
N1—H1*B*···O6	0.76 (2)	2.71 (2)	3.4507 (15)	163.9 (19)
N2—H2*A*···O12^{ii}	0.854 (18)	2.112 (18)	2.9457 (12)	165.4 (17)
N2—H2*B*···O15^{iii}	0.849 (19)	2.072 (19)	2.9164 (16)	172.9 (17)
N4—H4*D*···O9^{iii}	0.912 (19)	2.423 (19)	3.2993 (14)	161.1 (16)
N4—H4*E*···O16^{iv}	0.736 (19)	2.269 (19)	2.9686 (16)	159.2 (19)
N6—H6*A*···O8^{v}	0.882 (19)	1.865 (19)	2.7463 (13)	176.7 (17)
N6—H6*B*···O17^{i}	0.874 (18)	1.921 (19)	2.7871 (13)	170.7 (17)
N6—H6*C*···O7	0.808 (19)	1.990 (19)	2.7922 (11)	172.2 (18)
N6—H6*D*···O2^{i}	0.873 (19)	2.074 (19)	2.8541 (13)	148.3 (16)
N7—H7*A*···O16	0.835 (18)	2.083 (18)	2.8810 (14)	159.8 (17)
N7—H7*B*···O4^{vi}	0.880 (18)	2.056 (18)	2.8627 (12)	152.1 (16)
N7—H7*C*···O1^{vii}	0.843 (19)	2.072 (19)	2.9050 (12)	169.7 (17)
N7—H7*D*···O11^{viii}	0.873 (18)	1.928 (18)	2.7957 (12)	172.1 (16)
O15—H15*A*···O12	0.81 (2)	2.38 (2)	3.1833 (16)	171 (2)
O15—H15*B*···O17^{ix}	0.78 (2)	2.03 (2)	2.8046 (18)	177 (3)
O16—H16*A*···O15^{vi}	0.80 (2)	2.05 (2)	2.8477 (18)	176 (2)
O16—H16*B*···O5^{x}	0.83 (2)	2.23 (2)	2.8937 (13)	137 (2)
O17—H17*A*···O13	0.85 (1)	1.87 (2)	2.7130 (12)	172 (2)
O17—H17*B*···N3	0.82 (2)	2.07 (2)	2.8830 (15)	170 (2)

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

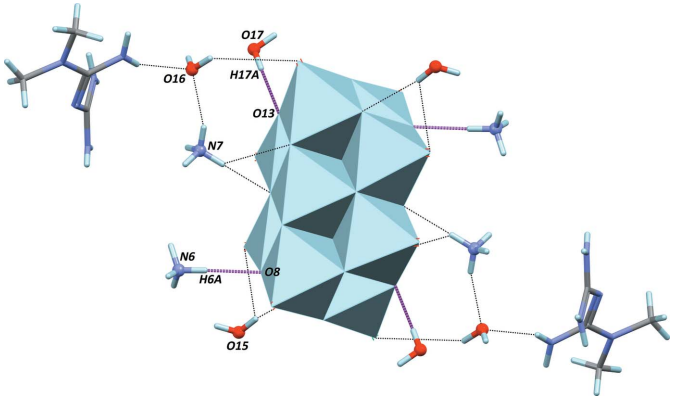


Figure 2
Main interactions between the $(V_{10}O_{28})^{6-}$ anion (polyhedral representation) and the cations and water molecules. The strongest hydrogen bonds are represented as magenta dashed bonds (entries 7 and 19 in Table 1), while secondary hydrogen bonds are represented with thin black dashed lines.

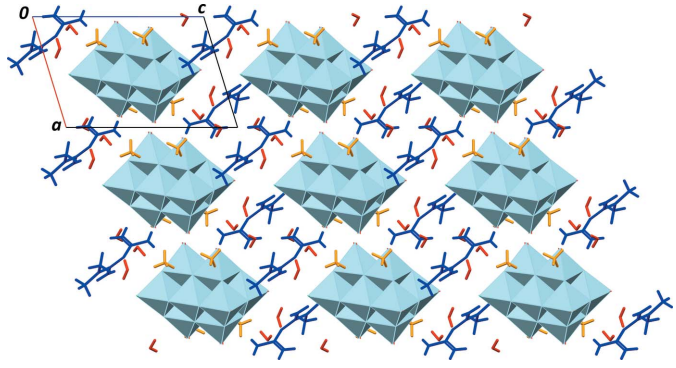


Figure 3
Part of the crystal structure of the title salt, viewed along [010]. Colour code: pale-blue polyhedra: $(V_{10}O_{28})^{6-}$ anions; orange: ammonium; blue: metforminium(1+); red: water.

Table 2
Experimental details.

Crystal data	
Chemical formula	$(NH_4)_4(C_4H_{12}N_5)_2[V_{10}O_{28}]\cdot 6H_2O$
M_r	1398.03
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	295
a, b, c (Å)	9.7965 (2), 10.1010 (2), 13.0974 (3)
α, β, γ (°)	81.081 (2), 70.906 (2), 63.321 (2)
V (Å^3)	1094.30 (5)
Z	1
Radiation type	Ag $K\alpha$, λ = 0.56083 Å
μ (mm^{-1})	1.10
Crystal size (mm)	0.26 × 0.26 × 0.19
Data collection	
Diffractometer	Stoe Stadivari
Absorption correction	Multi-scan (*X-AREA*; Stoe & Cie, 2020)
T_{min}, T_{max}	0.426, 0.907
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	94693, 11269, 9224
R_{int}	0.030
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.851
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.025, 0.078, 1.06
No. of reflections	11269
No. of parameters	361
No. of restraints	9
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.47, −0.95

Computer programs: *X-AREA* (Stoe & Cie, 2020), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

Synthesis and crystallization

Good-quality single crystals of the title compound were obtained during the reaction between ammonium metavanadate (NH_4VO_3, 1.117 g, 9.5 mmol; Pérez-Benítez & Bernès, 2018) and metformin hydrochloride (Metf·HCl, 0.497 g, 3 mmol; Niranjana Devi *et al.*, 2017) in 100 ml of distilled water and 6 ml of acetic acid 5% *v*/*v*. In a typical procedure, the ammonium metavanadate was dissolved by heating in a water bath and then metformin hydrochloride was added and stirred until its dissolution. The water bath was removed and once the mixture cooled down to room temperature, the acetic acid was added. The homogeneous solution was slowly evaporated during several days at ambient conditions, which allowed the separation of reaction by-products by fractional crystallization, being the main products $[H_2Metf]_3(V_{10}O_{28})\cdot 8H_2O$ and $[H_2Metf]_2[NH_4]_2(V_{10}O_{28})\cdot 10H_2O$ (CCDC-993916 and 993917, with yields of *ca* 53 and 24%, respectively; Sánchez-Lombardo *et al.*, 2014) and the title compound, $[HMetf]_2[NH_4]_4(V_{10}O_{28})\cdot 6H_2O$ (*ca*. 5% yield). These yields are poorly reproducible, and no powder diffraction was performed on the solid phases obtained by fractional crystallization to check their purity. Therefore, we cannot rule out the presence of other crystallized compounds in this reaction.

Refinement

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

Acknowledgements

APB thanks Rosa Elena Arroyo-Carmona for carrying out the fractional crystallization of the title compound. X-ray data were collected remotely, during the current pandemic, as part of a course. We thank the *Comisión para el seguimiento y evaluación de la pandemia COVID-19* (BUAP, Puebla), who allowed one of us to switch the diffractometer on and off.

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full crystallographic data

IUCrData (2021). **6**, x210634 [https://doi.org/10.1107/S2414314621006349]

Tetraammonium bis(metforminium) di-μ_6-oxido-tetra-μ_3-oxido-tetradeca-μ_2-oxido-octaoxidodecavanadium(V) hexahydrate

J. Alberto Polito-Lucas, José A. Núñez-Ávila, Sylvain Bernès and Aarón Pérez-Benítez

Tetraammonium bis(metforminium) di-μ_6-oxido-tetra-μ_3-oxido-tetradeca-μ_2-oxido-octaoxidodecavanadium(V) hexahydrate

Crystal data

$(NH_4)_4(C_4H_{12}N_5)_2[V_{10}O_{28}]\cdot 6H_2O$
M_r = 1398.03
Triclinic, $P\overline{1}$
a = 9.7965 (2) Å
b = 10.1010 (2) Å
c = 13.0974 (3) Å
α = 81.081 (2)°
β = 70.906 (2)°
γ = 63.321 (2)°
V = 1094.30 (5) Å^3
Z = 1
$F(000)$ = 700
D_x = 2.121 Mg m^{-3}
Ag $K\alpha$ radiation, λ = 0.56083 Å
Cell parameters from 131899 reflections
θ = 2.2–33.7°
μ = 1.10 mm^{-1}
T = 295 K
Tetrahedron, gold
0.26 × 0.26 × 0.19 mm

Data collection

Stoe Stadivari diffractometer
Radiation source: Sealed X-ray tube, Axo Astix-f Microfocus source
Graded multilayer mirror monochromator
Detector resolution: 5.81 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (X-AREA; Stoe & Cie, 2020)
T_{min} = 0.426, T_{max} = 0.907
94693 measured reflections
11269 independent reflections
9224 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 28.5°, θ_{min} = 2.2°
h = −16→16
k = −17→17
l = −22→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.078
S = 1.06
11269 reflections
361 parameters
9 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
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.0305P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.95 e Å^{-3}
Extinction correction: SHELXL-2018/3 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0048 (9)

Special details

Refinement. All H atoms, with exception of the methyl groups in the $HMetf^{+}$ cation, were refined with free coordinates and isotropic displacement parameters calculated as $U_{iso}(H) = 1.2$ or $1.5\times U_{eq}$(carrier atom). The geometry for the three water molecules was restrained, with target bond lengths O—H = 0.85 (2) Å and H···H separations of 1.34 (2) Å. Methyl H atoms were included using a riding model with $U_{iso}(H) = 1.5\times U_{eq}$(carrier atom).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
V1	0.39655 (2)	0.84689 (2)	0.38300 (2)	0.02229 (4)
V2	0.22257 (2)	0.64431 (2)	0.44890 (2)	0.02011 (3)
V3	0.44412 (2)	0.63696 (2)	0.58069 (2)	0.01697 (3)
V4	0.72567 (2)	0.57810 (2)	0.36227 (2)	0.01973 (3)
V5	0.50248 (2)	0.59053 (2)	0.23433 (2)	0.02221 (4)
O1	0.33595 (10)	1.02437 (8)	0.37493 (7)	0.03200 (16)
O2	0.04039 (9)	0.66456 (10)	0.48094 (7)	0.02993 (15)
O3	0.43043 (9)	0.62643 (8)	0.71288 (5)	0.02402 (13)
O4	0.90793 (9)	0.55609 (9)	0.33301 (7)	0.02955 (15)
O5	0.52329 (11)	0.57067 (11)	0.11007 (6)	0.03517 (18)
O6	0.20204 (8)	0.83200 (8)	0.43086 (6)	0.02483 (13)
O7	0.38555 (9)	0.81825 (7)	0.54386 (6)	0.02400 (13)
O8	0.61636 (8)	0.77845 (7)	0.36354 (6)	0.02343 (12)
O9	0.43593 (9)	0.79045 (8)	0.24684 (6)	0.02459 (13)
O10	0.24430 (7)	0.63107 (7)	0.59608 (5)	0.01873 (11)
O11	0.66939 (8)	0.57599 (7)	0.52504 (5)	0.01927 (11)
O12	0.71111 (8)	0.55367 (8)	0.23235 (5)	0.02325 (12)
O13	0.29624 (8)	0.60661 (8)	0.30286 (5)	0.02357 (12)
O14	0.52088 (7)	0.40515 (7)	0.58287 (5)	0.01779 (10)
C1	0.06586 (13)	1.15154 (12)	0.16742 (9)	0.03002 (19)
C2	0.26523 (13)	1.04792 (11)	0.00500 (8)	0.02878 (18)
C3	0.2491 (2)	0.8359 (2)	−0.04832 (12)	0.0567 (5)
H3A	0.301998	0.759308	−0.002811	0.085*
H3B	0.268909	0.794033	−0.115518	0.085*
H3C	0.136312	0.880514	−0.012978	0.085*
C4	0.42436 (17)	0.94509 (16)	−0.17351 (10)	0.0408 (3)
H4A	0.377248	1.032108	−0.214239	0.061*
H4B	0.453653	0.858445	−0.212180	0.061*
H4C	0.517801	0.943143	−0.163104	0.061*
N1	0.02667 (15)	1.12792 (15)	0.27388 (9)	0.0428 (3)
H1A	−0.050 (2)	1.197 (2)	0.3093 (15)	0.051*
H1B	0.078 (2)	1.055 (2)	0.2967 (16)	0.051*
N2	−0.00914 (13)	1.28620 (12)	0.13003 (9)	0.0373 (2)
H2A	−0.089 (2)	1.355 (2)	0.1705 (14)	0.045*
H2B	0.000 (2)	1.299 (2)	0.0630 (15)	0.045*
N3	0.17233 (12)	1.03435 (10)	0.10395 (7)	0.03244 (19)
N4	0.32345 (14)	1.14908 (13)	−0.02036 (10)	0.0388 (2)
H4D	0.381 (2)	1.154 (2)	−0.0901 (15)	0.047*
H4E	0.301 (2)	1.201 (2)	0.0224 (15)	0.047*

N5	0.30971 (13)	0.94745 (11)	−0.06891 (7)	0.03350 (19)
N6	0.18645 (12)	1.10848 (11)	0.61533 (8)	0.02925 (17)
H6A	0.252 (2)	1.143 (2)	0.6196 (14)	0.044*
H6B	0.110 (2)	1.130 (2)	0.6764 (15)	0.044*
H6C	0.238 (2)	1.022 (2)	0.5989 (14)	0.044*
H6D	0.143 (2)	1.156 (2)	0.5644 (15)	0.044*
N7	0.22214 (11)	0.34134 (10)	0.33362 (8)	0.02681 (15)
H7A	0.258 (2)	0.3611 (19)	0.2689 (15)	0.040*
H7B	0.117 (2)	0.3902 (19)	0.3535 (13)	0.040*
H7C	0.247 (2)	0.250 (2)	0.3412 (14)	0.040*
H7D	0.265 (2)	0.3602 (19)	0.3754 (14)	0.040*
O15	0.99461 (17)	0.64089 (15)	0.09649 (9)	0.0543 (3)
H15A	0.924 (2)	0.621 (3)	0.1371 (18)	0.081*
H15B	1.021 (3)	0.683 (3)	0.1260 (19)	0.081*
O16	0.28519 (14)	0.39198 (12)	0.10371 (8)	0.0451 (2)
H16A	0.204 (2)	0.464 (2)	0.1036 (18)	0.068*
H16B	0.365 (2)	0.405 (2)	0.0658 (17)	0.068*
O17	0.07918 (11)	0.80032 (11)	0.20277 (7)	0.03818 (19)
H17A	0.152 (2)	0.7348 (19)	0.2284 (15)	0.057*
H17B	0.114 (2)	0.8621 (19)	0.1780 (16)	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.02379 (7)	0.01451 (6)	0.02375 (7)	−0.00547 (5)	−0.00600 (5)	0.00227 (4)
V2	0.01597 (6)	0.02161 (7)	0.02045 (6)	−0.00631 (5)	−0.00604 (5)	0.00221 (5)
V3	0.01774 (6)	0.01375 (6)	0.01733 (6)	−0.00493 (4)	−0.00428 (4)	−0.00203 (4)
V4	0.01655 (6)	0.01890 (6)	0.02113 (6)	−0.00737 (5)	−0.00352 (5)	0.00193 (4)
V5	0.02394 (7)	0.02343 (7)	0.01623 (6)	−0.00812 (5)	−0.00540 (5)	0.00068 (5)
O1	0.0359 (4)	0.0162 (3)	0.0350 (4)	−0.0073 (3)	−0.0061 (3)	0.0027 (2)
O2	0.0196 (3)	0.0363 (4)	0.0325 (4)	−0.0114 (3)	−0.0096 (3)	0.0059 (3)
O3	0.0284 (3)	0.0222 (3)	0.0195 (3)	−0.0088 (2)	−0.0060 (2)	−0.0034 (2)
O4	0.0198 (3)	0.0333 (4)	0.0332 (4)	−0.0122 (3)	−0.0063 (3)	0.0058 (3)
O5	0.0416 (4)	0.0430 (5)	0.0189 (3)	−0.0167 (4)	−0.0084 (3)	−0.0002 (3)
O6	0.0202 (3)	0.0191 (3)	0.0278 (3)	−0.0038 (2)	−0.0057 (2)	0.0022 (2)
O7	0.0275 (3)	0.0148 (3)	0.0256 (3)	−0.0063 (2)	−0.0059 (2)	−0.0018 (2)
O8	0.0232 (3)	0.0187 (3)	0.0275 (3)	−0.0099 (2)	−0.0063 (2)	0.0027 (2)
O9	0.0254 (3)	0.0218 (3)	0.0227 (3)	−0.0078 (2)	−0.0077 (2)	0.0043 (2)
O10	0.0164 (2)	0.0165 (2)	0.0186 (2)	−0.00421 (19)	−0.00313 (19)	−0.00076 (18)
O11	0.0192 (3)	0.0188 (3)	0.0201 (3)	−0.0082 (2)	−0.0060 (2)	−0.00041 (19)
O12	0.0210 (3)	0.0247 (3)	0.0184 (3)	−0.0073 (2)	−0.0024 (2)	−0.0001 (2)
O13	0.0234 (3)	0.0266 (3)	0.0214 (3)	−0.0103 (2)	−0.0088 (2)	0.0018 (2)
O14	0.0169 (2)	0.0156 (2)	0.0186 (2)	−0.0056 (2)	−0.00443 (19)	−0.00012 (18)
C1	0.0266 (4)	0.0305 (5)	0.0278 (4)	−0.0078 (4)	−0.0042 (3)	−0.0085 (3)
C2	0.0271 (4)	0.0238 (4)	0.0266 (4)	−0.0051 (3)	−0.0037 (3)	−0.0035 (3)
C3	0.0736 (11)	0.0555 (9)	0.0407 (7)	−0.0432 (8)	0.0149 (7)	−0.0195 (6)
C4	0.0440 (6)	0.0412 (6)	0.0261 (5)	−0.0177 (5)	0.0048 (4)	−0.0050 (4)
N1	0.0390 (5)	0.0437 (6)	0.0251 (4)	−0.0017 (5)	−0.0030 (4)	−0.0092 (4)

N2	0.0353 (5)	0.0281 (4)	0.0343 (5)	−0.0034 (4)	−0.0039 (4)	−0.0079 (3)
N3	0.0342 (4)	0.0260 (4)	0.0234 (4)	−0.0058 (3)	0.0004 (3)	−0.0048 (3)
N4	0.0403 (5)	0.0346 (5)	0.0367 (5)	−0.0174 (4)	0.0002 (4)	−0.0080 (4)
N5	0.0390 (5)	0.0300 (4)	0.0237 (4)	−0.0148 (4)	0.0032 (3)	−0.0059 (3)
N6	0.0284 (4)	0.0227 (4)	0.0323 (4)	−0.0077 (3)	−0.0070 (3)	−0.0029 (3)
N7	0.0254 (4)	0.0237 (4)	0.0315 (4)	−0.0102 (3)	−0.0087 (3)	−0.0008 (3)
O15	0.0633 (7)	0.0591 (7)	0.0337 (5)	−0.0251 (6)	−0.0099 (5)	0.0053 (4)
O16	0.0503 (6)	0.0430 (5)	0.0348 (4)	−0.0206 (4)	−0.0042 (4)	0.0031 (4)
O17	0.0328 (4)	0.0406 (5)	0.0318 (4)	−0.0107 (4)	−0.0091 (3)	0.0082 (3)

Geometric parameters (Å, º)

V1—O1	1.6137 (7)	V5—O14^{i}	2.3350 (6)
V1—O9	1.8226 (7)	C1—N2	1.3271 (16)
V1—O6	1.8689 (8)	C1—N1	1.3332 (15)
V1—O8	1.8811 (7)	C1—N3	1.3451 (13)
V1—O7	2.0554 (7)	C2—N4	1.3318 (16)
V1—O14^{i}	2.3173 (6)	C2—N5	1.3374 (13)
V1—V5	3.0663 (2)	C2—N3	1.3460 (14)
V1—V2	3.0755 (2)	C3—N5	1.4474 (18)
V1—V3	3.1011 (2)	C3—H3A	0.9600
V1—V4	3.1042 (2)	C3—H3B	0.9600
V2—O2	1.6161 (8)	C3—H3C	0.9600
V2—O6	1.8001 (7)	C4—N5	1.4554 (14)
V2—O13	1.8440 (7)	C4—H4A	0.9600
V2—O10	1.9854 (7)	C4—H4B	0.9600
V2—O11^{i}	2.0185 (7)	C4—H4C	0.9600
V2—O14^{i}	2.2343 (6)	N1—H1A	0.818 (19)
V2—V4^{i}	3.0815 (2)	N1—H1B	0.76 (2)
V3—O3	1.6825 (7)	N2—H2A	0.854 (18)
V3—O7	1.6968 (7)	N2—H2B	0.849 (19)
V3—O11	1.9098 (7)	N4—H4D	0.912 (19)
V3—O10	1.9284 (7)	N4—H4E	0.736 (19)
V3—O14	2.1121 (6)	N6—H6A	0.882 (19)
V3—O14^{i}	2.1349 (6)	N6—H6B	0.874 (18)
V3—V5^{i}	3.0737 (2)	N6—H6C	0.808 (19)
V4—O4	1.6145 (7)	N6—H6D	0.873 (19)
V4—O8	1.8153 (7)	N7—H7A	0.835 (18)
V4—O12	1.8158 (7)	N7—H7B	0.880 (18)
V4—O10^{i}	2.0094 (7)	N7—H7C	0.843 (19)
V4—O11	2.0192 (6)	N7—H7D	0.873 (18)
V4—O14^{i}	2.2168 (6)	O15—H15A	0.812 (16)
V4—V5	3.1128 (2)	O15—H15B	0.778 (16)
V5—O5	1.6055 (8)	O16—H16A	0.804 (15)
V5—O9	1.8385 (7)	O16—H16B	0.830 (15)
V5—O13	1.8649 (7)	O17—H17A	0.850 (14)
V5—O12	1.8978 (7)	O17—H17B	0.820 (15)
V5—O3^{i}	2.0607 (7)		

O1—V1—O9	104.84 (4)	O12—V4—V1	84.23 (2)
O1—V1—O6	101.04 (4)	O10^{i}—V4—V1	124.447 (19)
O9—V1—O6	92.16 (3)	O11—V4—V1	87.655 (19)
O1—V1—O8	102.51 (4)	O14^{i}—V4—V1	48.162 (16)
O9—V1—O8	91.10 (3)	V2^{i}—V4—V1	120.184 (6)
O6—V1—O8	154.52 (3)	O4—V4—V5	136.16 (3)
O1—V1—O7	98.71 (4)	O8—V4—V5	84.21 (2)
O9—V1—O7	156.44 (3)	O12—V4—V5	33.88 (2)
O6—V1—O7	83.85 (3)	O10^{i}—V4—V5	87.47 (2)
O8—V1—O7	83.12 (3)	O11—V4—V5	124.45 (2)
O1—V1—O14^{i}	172.87 (4)	O14^{i}—V4—V5	48.462 (16)
O9—V1—O14^{i}	82.28 (3)	V2^{i}—V4—V5	119.451 (6)
O6—V1—O14^{i}	78.35 (3)	V1—V4—V5	59.105 (6)
O8—V1—O14^{i}	77.07 (3)	O5—V5—O9	104.08 (4)
O7—V1—O14^{i}	74.17 (2)	O5—V5—O13	102.38 (4)
O1—V1—V5	138.11 (3)	O9—V5—O13	91.70 (3)
O9—V1—V5	33.28 (2)	O5—V5—O12	102.89 (4)
O6—V1—V5	84.42 (2)	O9—V5—O12	90.47 (3)
O8—V1—V5	84.55 (2)	O13—V5—O12	153.29 (3)
O7—V1—V5	123.18 (2)	O5—V5—O3^{i}	100.16 (4)
O14^{i}—V1—V5	49.017 (16)	O9—V5—O3^{i}	155.75 (3)
O1—V1—V2	133.39 (3)	O13—V5—O3^{i}	83.71 (3)
O9—V1—V2	83.40 (3)	O12—V5—O3^{i}	83.45 (3)
O6—V1—V2	32.35 (2)	O5—V5—O14^{i}	174.46 (4)
O8—V1—V2	123.42 (2)	O9—V5—O14^{i}	81.46 (3)
O7—V1—V2	80.88 (2)	O13—V5—O14^{i}	77.01 (3)
O14^{i}—V1—V2	46.362 (16)	O12—V5—O14^{i}	77.00 (3)
V5—V1—V2	61.310 (6)	O3^{i}—V5—O14^{i}	74.30 (2)
O1—V1—V3	129.40 (3)	O5—V5—V1	137.01 (4)
O9—V1—V3	125.76 (2)	O9—V5—V1	32.96 (2)
O6—V1—V3	79.55 (2)	O13—V5—V1	83.39 (2)
O8—V1—V3	78.07 (2)	O12—V5—V1	84.06 (2)
O7—V1—V3	30.690 (19)	O3^{i}—V5—V1	122.825 (19)
O14^{i}—V1—V3	43.479 (15)	O14^{i}—V5—V1	48.521 (15)
V5—V1—V3	92.496 (6)	O5—V5—V3^{i}	131.12 (4)
V2—V1—V3	61.458 (5)	O9—V5—V3^{i}	124.81 (2)
O1—V1—V4	134.70 (3)	O13—V5—V3^{i}	78.32 (2)
O9—V1—V4	81.46 (2)	O12—V5—V3^{i}	78.62 (2)
O6—V1—V4	123.80 (2)	O3^{i}—V5—V3^{i}	30.960 (18)
O8—V1—V4	32.23 (2)	O14^{i}—V5—V3^{i}	43.346 (15)
O7—V1—V4	81.57 (2)	V1—V5—V3^{i}	91.865 (6)
O14^{i}—V1—V4	45.455 (15)	O5—V5—V4	135.05 (4)
V5—V1—V4	60.588 (6)	O9—V5—V4	80.99 (2)
V2—V1—V4	91.644 (6)	O13—V5—V4	122.29 (2)
V3—V1—V4	61.402 (5)	O12—V5—V4	32.23 (2)
O2—V2—O6	103.19 (4)	O3^{i}—V5—V4	81.44 (2)
O2—V2—O13	102.93 (4)	O14^{i}—V5—V4	45.285 (16)

O6—V2—O13	94.22 (3)	V1—V5—V4	60.308 (5)
O2—V2—O10	99.24 (4)	V3^{i}—V5—V4	61.383 (5)
O6—V2—O10	92.09 (3)	V3—O3—V5^{i}	109.98 (3)
O13—V2—O10	154.87 (3)	V2—O6—V1	113.89 (4)
O2—V2—O11^{i}	98.97 (4)	V3—O7—V1	111.12 (3)
O6—V2—O11^{i}	156.53 (3)	V4—O8—V1	114.22 (4)
O13—V2—O11^{i}	88.24 (3)	V1—O9—V5	113.76 (4)
O10—V2—O11^{i}	76.68 (3)	V3—O10—V2	107.49 (3)
O2—V2—O14^{i}	173.66 (3)	V3—O10—V4^{i}	106.61 (3)
O6—V2—O14^{i}	82.01 (3)	V2—O10—V4^{i}	100.95 (3)
O13—V2—O14^{i}	80.07 (3)	V3—O11—V2^{i}	107.71 (3)
O10—V2—O14^{i}	76.76 (3)	V3—O11—V4	107.45 (3)
O11^{i}—V2—O14^{i}	75.42 (3)	V2^{i}—O11—V4	99.49 (3)
O2—V2—V1	136.83 (3)	V4—O12—V5	113.89 (3)
O6—V2—V1	33.75 (2)	V2—O13—V5	115.19 (4)
O13—V2—V1	83.44 (2)	V3—O14—V3^{i}	102.04 (3)
O10—V2—V1	88.47 (2)	V3—O14—V4^{i}	93.65 (2)
O11^{i}—V2—V1	124.06 (2)	V3^{i}—O14—V4^{i}	93.42 (2)
O14^{i}—V2—V1	48.643 (16)	V3—O14—V2^{i}	93.73 (2)
O2—V2—V4^{i}	89.10 (3)	V3^{i}—O14—V2^{i}	92.47 (2)
O6—V2—V4^{i}	131.89 (3)	V4^{i}—O14—V2^{i}	169.39 (3)
O13—V2—V4^{i}	128.50 (2)	V3—O14—V1^{i}	169.74 (3)
O10—V2—V4^{i}	39.806 (19)	V3^{i}—O14—V1^{i}	88.20 (2)
O11^{i}—V2—V4^{i}	40.263 (18)	V4^{i}—O14—V1^{i}	86.38 (2)
O14^{i}—V2—V4^{i}	84.694 (17)	V2^{i}—O14—V1^{i}	84.99 (2)
V1—V2—V4^{i}	120.428 (6)	V3—O14—V5^{i}	87.30 (2)
O3—V3—O7	107.11 (4)	V3^{i}—O14—V5^{i}	170.66 (3)
O3—V3—O11	97.73 (3)	V4^{i}—O14—V5^{i}	86.25 (2)
O7—V3—O11	97.83 (3)	V2^{i}—O14—V5^{i}	86.50 (2)
O3—V3—O10	97.47 (3)	V1^{i}—O14—V5^{i}	82.46 (2)
O7—V3—O10	96.04 (3)	N2—C1—N1	118.43 (10)
O11—V3—O10	155.39 (3)	N2—C1—N3	123.86 (10)
O3—V3—O14	88.41 (3)	N1—C1—N3	117.57 (11)
O7—V3—O14	164.44 (3)	N4—C2—N5	118.72 (10)
O11—V3—O14	80.64 (3)	N4—C2—N3	123.49 (10)
O10—V3—O14	80.55 (3)	N5—C2—N3	117.62 (10)
O3—V3—O14^{i}	166.38 (3)	N5—C3—H3A	109.5
O7—V3—O14^{i}	86.51 (3)	N5—C3—H3B	109.5
O11—V3—O14^{i}	80.29 (3)	H3A—C3—H3B	109.5
O10—V3—O14^{i}	80.39 (3)	N5—C3—H3C	109.5
O14—V3—O14^{i}	77.96 (3)	H3A—C3—H3C	109.5
O3—V3—V5^{i}	39.06 (2)	H3B—C3—H3C	109.5
O7—V3—V5^{i}	146.17 (2)	N5—C4—H4A	109.5
O11—V3—V5^{i}	89.66 (2)	N5—C4—H4B	109.5
O10—V3—V5^{i}	90.04 (2)	H4A—C4—H4B	109.5
O14—V3—V5^{i}	49.358 (17)	N5—C4—H4C	109.5
O14^{i}—V3—V5^{i}	127.319 (17)	H4A—C4—H4C	109.5
O3—V3—V1	145.30 (3)	H4B—C4—H4C	109.5

O7—V3—V1	38.19 (2)	C1—N1—H1A	115.0 (14)
O11—V3—V1	89.69 (2)	C1—N1—H1B	119.0 (15)
O10—V3—V1	88.75 (2)	H1A—N1—H1B	126 (2)
O14—V3—V1	126.279 (18)	C1—N2—H2A	122.6 (12)
O14^{i}—V3—V1	48.321 (17)	C1—N2—H2B	119.7 (12)
V5^{i}—V3—V1	175.629 (6)	H2A—N2—H2B	114.9 (17)
O4—V4—O8	102.13 (4)	C1—N3—C2	122.59 (10)
O4—V4—O12	102.49 (4)	C2—N4—H4D	118.1 (12)
O8—V4—O12	95.66 (3)	C2—N4—H4E	117.1 (15)
O4—V4—O10^{i}	99.74 (4)	H4D—N4—H4E	124.7 (19)
O8—V4—O10^{i}	155.62 (3)	C2—N5—C3	122.03 (10)
O12—V4—O10^{i}	89.99 (3)	C2—N5—C4	120.63 (11)
O4—V4—O11	99.12 (4)	C3—N5—C4	117.34 (10)
O8—V4—O11	89.83 (3)	H6A—N6—H6B	106.4 (16)
O12—V4—O11	156.02 (3)	H6A—N6—H6C	108.3 (17)
O10^{i}—V4—O11	76.13 (3)	H6B—N6—H6C	117.5 (17)
O4—V4—O14^{i}	174.28 (3)	H6A—N6—H6D	109.4 (17)
O8—V4—O14^{i}	81.05 (3)	H6B—N6—H6D	107.1 (16)
O12—V4—O14^{i}	81.78 (3)	H6C—N6—H6D	108.0 (17)
O10^{i}—V4—O14^{i}	76.30 (2)	H7A—N7—H7B	109.0 (16)
O11—V4—O14^{i}	76.03 (3)	H7A—N7—H7C	109.8 (16)
O4—V4—V2^{i}	89.60 (3)	H7B—N7—H7C	109.4 (16)
O8—V4—V2^{i}	130.07 (2)	H7A—N7—H7D	112.0 (16)
O12—V4—V2^{i}	129.23 (2)	H7B—N7—H7D	111.8 (15)
O10^{i}—V4—V2^{i}	39.240 (19)	H7C—N7—H7D	104.8 (16)
O11—V4—V2^{i}	40.246 (19)	H15A—O15—H15B	112 (2)
O14^{i}—V4—V2^{i}	84.733 (17)	H16A—O16—H16B	112 (2)
O4—V4—V1	135.49 (3)	H17A—O17—H17B	102.5 (18)
O8—V4—V1	33.55 (2)		

O7—V3—O3—V5^{i}	179.48 (4)	V3—V1—O9—V5	2.25 (5)
O11—V3—O3—V5^{i}	−79.82 (4)	V4—V1—O9—V5	47.45 (3)
O10—V3—O3—V5^{i}	80.77 (4)	O5—V5—O9—V1	178.29 (5)
O14—V3—O3—V5^{i}	0.51 (4)	O13—V5—O9—V1	75.08 (4)
O14^{i}—V3—O3—V5^{i}	0.80 (16)	O12—V5—O9—V1	−78.29 (4)
V1—V3—O3—V5^{i}	179.443 (13)	O3^{i}—V5—O9—V1	−3.32 (11)
O2—V2—O6—V1	−175.89 (4)	O14^{i}—V5—O9—V1	−1.51 (4)
O13—V2—O6—V1	−71.55 (4)	V3^{i}—V5—O9—V1	−1.85 (5)
O10—V2—O6—V1	84.11 (4)	V4—V5—O9—V1	−47.36 (3)
O11^{i}—V2—O6—V1	23.77 (10)	O4—V4—O12—V5	−174.54 (4)
O14^{i}—V2—O6—V1	7.80 (4)	O8—V4—O12—V5	−70.76 (4)
V4^{i}—V2—O6—V1	83.24 (4)	O10^{i}—V4—O12—V5	85.48 (4)
O1—V1—O6—V2	179.64 (4)	O11—V4—O12—V5	31.67 (9)
O9—V1—O6—V2	74.06 (4)	O14^{i}—V4—O12—V5	9.32 (4)
O8—V1—O6—V2	−23.07 (10)	V2^{i}—V4—O12—V5	85.57 (4)
O7—V1—O6—V2	−82.65 (4)	V1—V4—O12—V5	−39.17 (3)
O14^{i}—V1—O6—V2	−7.60 (4)	O5—V5—O12—V4	176.70 (5)
V5—V1—O6—V2	41.65 (4)	O9—V5—O12—V4	72.14 (4)

V3—V1—O6—V2	−51.93 (3)	O13—V5—O12—V4	−22.59 (9)
V4—V1—O6—V2	−7.11 (5)	O3^{i}—V5—O12—V4	−84.33 (4)
O3—V3—O7—V1	−179.96 (4)	O14^{i}—V5—O12—V4	−8.99 (3)
O11—V3—O7—V1	79.41 (4)	V1—V5—O12—V4	39.76 (3)
O10—V3—O7—V1	−80.21 (4)	V3^{i}—V5—O12—V4	−53.35 (3)
O14—V3—O7—V1	−3.81 (14)	O2—V2—O13—V5	175.65 (4)
O14^{i}—V3—O7—V1	−0.27 (4)	O6—V2—O13—V5	71.08 (4)
V5^{i}—V3—O7—V1	−179.378 (13)	O10—V2—O13—V5	−33.02 (10)
O4—V4—O8—V1	174.69 (4)	O11^{i}—V2—O13—V5	−85.55 (4)
O12—V4—O8—V1	70.60 (4)	O14^{i}—V2—O13—V5	−10.04 (4)
O10^{i}—V4—O8—V1	−32.00 (10)	V1—V2—O13—V5	39.04 (3)
O11—V4—O8—V1	−86.02 (4)	V4^{i}—V2—O13—V5	−85.02 (4)
O14^{i}—V4—O8—V1	−10.14 (4)	O5—V5—O13—V2	−175.94 (5)
V2^{i}—V4—O8—V1	−85.43 (4)	O9—V5—O13—V2	−71.13 (4)
V5—V4—O8—V1	38.66 (3)	O12—V5—O13—V2	23.31 (10)
O1—V1—O8—V4	−177.47 (4)	O3^{i}—V5—O13—V2	85.00 (4)
O9—V1—O8—V4	−72.00 (4)	O14^{i}—V5—O13—V2	9.71 (4)
O6—V1—O8—V4	25.37 (10)	V1—V5—O13—V2	−39.18 (3)
O7—V1—O8—V4	85.10 (4)	V3^{i}—V5—O13—V2	54.11 (3)
O14^{i}—V1—O8—V4	9.83 (3)	V4—V5—O13—V2	9.28 (5)
V5—V1—O8—V4	−39.33 (3)	N2—C1—N3—C2	30.11 (19)
V2—V1—O8—V4	10.82 (5)	N1—C1—N3—C2	−154.27 (13)
V3—V1—O8—V4	54.39 (3)	N4—C2—N3—C1	34.71 (19)
O1—V1—O9—V5	−178.43 (4)	N5—C2—N3—C1	−150.09 (12)
O6—V1—O9—V5	−76.42 (4)	N4—C2—N5—C3	−178.69 (15)
O8—V1—O9—V5	78.31 (4)	N3—C2—N5—C3	5.88 (19)
O7—V1—O9—V5	3.13 (11)	N4—C2—N5—C4	2.39 (18)
O14^{i}—V1—O9—V5	1.52 (4)	N3—C2—N5—C4	−173.04 (12)
V2—V1—O9—V5	−45.22 (4)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O10^{ii}	0.818 (19)	2.080 (19)	2.8981 (12)	178 (2)
N1—H1*B*···O6	0.76 (2)	2.71 (2)	3.4507 (15)	163.9 (19)
N2—H2*A*···O12^{iii}	0.854 (18)	2.112 (18)	2.9457 (12)	165.4 (17)
N2—H2*B*···O15^{iv}	0.849 (19)	2.072 (19)	2.9164 (16)	172.9 (17)
N4—H4*D*···O9^{iv}	0.912 (19)	2.423 (19)	3.2993 (14)	161.1 (16)
N4—H4*E*···O16^{v}	0.736 (19)	2.269 (19)	2.9686 (16)	159.2 (19)
N6—H6*A*···O8^{vi}	0.882 (19)	1.865 (19)	2.7463 (13)	176.7 (17)
N6—H6*B*···O17^{ii}	0.874 (18)	1.921 (19)	2.7871 (13)	170.7 (17)
N6—H6*C*···O7	0.808 (19)	1.990 (19)	2.7922 (11)	172.2 (18)
N6—H6*D*···O2^{ii}	0.873 (19)	2.074 (19)	2.8541 (13)	148.3 (16)
N7—H7*A*···O16	0.835 (18)	2.083 (18)	2.8810 (14)	159.8 (17)
N7—H7*B*···O2^{vii}	0.880 (18)	2.367 (17)	2.9194 (12)	121.1 (14)
N7—H7*B*···O4^{viii}	0.880 (18)	2.056 (18)	2.8627 (12)	152.1 (16)

N7—H7*C*···O1^{ix}	0.843 (19)	2.072 (19)	2.9050 (12)	169.7 (17)
N7—H7*D*···O11^{i}	0.873 (18)	1.928 (18)	2.7957 (12)	172.1 (16)
O15—H15*A*···O4	0.81 (2)	2.52 (2)	3.0266 (14)	122 (2)
O15—H15*A*···O12	0.81 (2)	2.38 (2)	3.1833 (16)	171 (2)
O15—H15*B*···O17^{x}	0.78 (2)	2.03 (2)	2.8046 (18)	177 (3)
O16—H16*A*···O15^{viii}	0.80 (2)	2.05 (2)	2.8477 (18)	176 (2)
O16—H16*B*···O5^{xi}	0.83 (2)	2.23 (2)	2.8937 (13)	137 (2)
O17—H17*A*···O13	0.85 (1)	1.87 (2)	2.7130 (12)	172 (2)
O17—H17*B*···N3	0.82 (2)	2.07 (2)	2.8830 (15)	170 (2)

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