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$Cu_{3}V_{10}O_{28} \cdot 24H_{2}O$ and $CuNa_{4}V_{10}O_{28} \cdot 23H_{2}O$

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The crystal structures of tricopper decavanadate tetracosahydrate, (I), and copper tetrasodium decavanadate tricosahydrate, (II), have been determined by single-crystal X-ray diffraction. Both compounds exhibit a catenary structure consisting of $[V_{10}O_{28}]^{6-}$ anions linked by Cu^{2+} cations in (I) or by Na⁺ cations in (II). Compound (II) also contains a polymeric linear array of edge-sharing $[Na(OH_2)_6]^+$ and [Cu- $(OH_2)_6]^{2+}$ octahedra. In both compounds, the $[V_{10}O_{28}]^{6-}$ ions lie about inversion centres and the Cu^{2+} ions in (I) also lie about inversion centers.

Comment

Crystallization of polyoxometalates with metal cations often gives rise to (i) the linkage of polyxometalate anions into one-, two- or three-dimensional networks and/or (ii) the arrangement of metal cations into oligo/polycationic assemblies (*e.g.* Hedman, 1977), many of which are observed only in the solid state. The formation of these oligo/polymeric entities could be applied to the design of new materials with properties specific to the structure, especially when some of the constituent cations or anions possess microscopic features (spins, dipoles *etc.*) that would lead to macroscopic properties when appropriately aligned. Thus, we began an investigation of threedimensional assemblies involving the decavanadate anion, $[V_{10}O_{28}]^{6-}$, and Cu^{2+} , which is a transition metal cation with a d^9 electron configuration. From this $Cu^{2+}-[V_{10}O_{28}]^{6-}$ system, we obtained crystals of tricopper decavanadate tetracosahydrate, (I), and copper tetrasodium decavanadate tricosahydrate, (II), the structures of which are reported here.

From solutions containing only Cu²⁺ as cationic species, the decavanadate anion crystallizes with only Cu²⁺ cations to give (I). A unit cell of (I) consists of a $[V_{10}O_{28}]^{6-}$ anion and three Cu^{2+} cations, all of which are located at the $\overline{1}$ sites, and water molecules of crystallization. As shown in Fig. 1, the metaloxygen framework of the decavanadate anion is identical to that reported by Evans (1966). Each Cu^{2+} cation is ligated by six O atoms to complete an octahedral coordination, elongated in one direction as the result of the Jahn-Teller effect. The equatorial Cu-O distances range from 1.9296 (14) to 2.0166 (14) Å and the axial distances from 2.2918 (14) to 2.3272 (14) Å (Table 1). The coordination sphere of atom Cu1 consists of four water O atoms as the equatorial ligands and two O atoms from different decavanadate anions as the axial ligands. As a result, each Cu1 atom links two decavanadate anions in a one-dimensional $\{[Cu(H_2O)_4]^{2+} \cdots [V_{10}O_{28}]^{6-} \cdots \}_{\infty}$ chain structure running along the [111] direction, as illustrated in Fig. 2. In contrast, atoms Cu2 and Cu3 are fully hydrated by



Figure 1

A view of an asymmetric unit of (I) and selected neighbors. Displacement ellipsoids are shown at the 50% probability level. The elongated Cu···O contacts are shown as broken lines. [Symmetry codes: (i) 1 - x, 1 - y, 1 - z; (ii) -x, -y, -z; (iii) -x, 1 - y, -z; (iv) 1 + x, 1 + y, 1 + z; (v) -x, -y, 1 - z.]



Figure 2

Packing diagram of (I), projected on to the *bc* plane. Hatched octahedra represent CuO_6 moieties. Open circles denote water O atoms that do not coordinate to Cu^{2+} .

six water molecules, completing discrete octahedra without sharing any ligands with other cations. These aqua ligands accept hydrogen bonds from the aqua ligands of atom Cu1 [O16-H16B···O22 and O15-H15A···O19(-x, 1-y, -z); Table 2] and donate hydrogen bonds to the surface O atoms of the decavanadate anion.

From solutions containing both Cu²⁺ and Na⁺ as cationic species, the decavanadate anion crystallizes with both Cu²⁺ and Na⁺ cations to give a double salt, (II). The unit cell of (II) includes a $[V_{10}O_{28}]^{6-}$ anion, a Cu²⁺ cation, four Na⁺ cations and water molecules. The decavanadate anion, the Cu²⁺ cation and two of the three independent Na⁺ cations (Na2 and Na3) are located at the inversion centers. As shown in Fig. 3, the molecular structure of the decavanadate anion in (II) has a metal–oxygen framework identical to that reported by Evans (1966). The Cu²⁺ cation and two of the three independent Na⁺ cations (Na1 and Na2) are octahedrally coordinated by six water O atoms. These cations share aqua ligands to form an edge-sharing linear polycationic array of octahedra, *viz.* {[Na1-(H₂O)₂]⁺(μ -H₂O)₂[Cu1(H₂O)₂]²⁺(μ -H₂O)₂[Na1(H₂O)₂]⁺(μ -

 $H_2O_2[Na2(H_2O_2)^+(\mu-H_2O_2)_{\infty}]_{\infty}$, running along the [011] direction, as illustrated in Fig. 4. The Cu²⁺ cation in (II) also exhibits a Jahn-Teller distortion, with equatorial and axial Cu-O distances of 1.9247 (15)-2.0420 (15) and 2.3896 (18) Å, respectively (Table 3). The water molecules at the axial sites also coordinate to adjacent Na⁺ cations, participating in the linkage of the catenary polycation. These longer Cu-O bonds, together with the O15-H15B...O18 and O19-H19B···O22 hydrogen bonds (Table 4), may stabilize the infinite chain. The remaining Na⁺ cation, Na3, shares aqua ligands with this array to connect adjacent arrays into a two-dimensional polycationic network, as shown in Fig. 4. Terminal O atoms of the decavanadate anions also coordinate to this Na cation, to form a one-dimensional $\{[Na(H_2O)_4]^+\cdots$ $[V_{10}O_{28}]^{6-}\cdots\}_\infty$ chain structure running along the [111] direction. The decavanadate anions link the polycationic sheet structure into a three-dimensional network.

A similar catenary structure, viz. $\{[Na(H_2O)_4]^+\cdots$ $[V_{10}O_{28}]^{6-}\cdots\}_{\infty}$, has been observed in (HMTA-H···H₂O)- $(HMTA-CH_2OH)[H_3V_{10}O_{28}[Na(H_2O)_4]]\cdot 4H_2O$ (Duraisamy et al., 2000; HMTA is hexamethylenetetramine), where hydrogen bonds from the aqua ligands of the Na⁺ cations to O atoms of the decavanadate anions support the chain structure. In contrast, the aqua ligands in the $\{[Cu(H_2O)_4]^{2+}\cdots$ $[V_{10}O_{28}]^{6-}\cdots\}_{\infty}$ chain of (I) and the $\{[Na(H_2O)_4]^+\cdots$ $[V_{10}O_{28}]^{6-}\cdots\}_{\infty}$ chain of (II) do not form hydrogen bonds to the decavanadate anions. The absence of such hydrogen bonds results in an almost linear Cu/Na \cdots O-V linkage [170.50 (9)° for Cu1 \cdots O3–V3 for (I) and 169.40 (9)° for Na3 \cdots O3–V3 for (II)] as opposed to the bent $Na \cdots O - V$ linkage (141.5°) in $(HMTA-H···H_2O)(HMTA-CH_2OH)[H_3V_{10}O_{28}[Na(H_2O)_4]]$ 4H₂O. A discrete oligocationic structure, {[Na(H₂O)₃][Ni- $(H_2O)_6][Na(H_2O)_3]\}^{4\scriptscriptstyle +},$ has recently been reported in [Ni-



Figure 3

A view of an asymmetric unit and selected neighbors of (II). Displacement ellipsoids are shown at the 50% probability level. The elongated Cu···O contacts are shown as broken lines. [Symmetry codes: (i) 1 - x, 1 - y, 1 - z; (ii) -x, -y, -z; (iii) -x, 1 - y, -z; (iv) 1 + x, 1 + y, 1 + z; (vi) -x, -y, -1 - z; (vii) x, 1 + y, 1 + z.]



Figure 4

Packing diagram of (II), projected on to the bc plane. Hatched and shaded octahedra represent CuO₆ and NaO₆ moieties, respectively. Open circles represent water O atoms that do not coordinate to any metal cations.

 $(H_2O)_6]_2[Na(H_2O)_3]_2[V_{10}O_{28}]\cdot 4H_2O$ obtained from a similar Na⁺-Ni²⁺- $[V_{10}O_{28}]^{6-}$ system (Higami *et al.*, 2002). The difference in the M^{2+} -O distances {2.053–2.107 Å for Ni²⁺ in [Ni(H₂O)₆]_2[Na(H₂O)₃]_2[V₁₀O₂₈]\cdot 4H₂O *versus* 1.925–2.390 Å for Cu²⁺ in (II)} may lead to different poly/oligocationic structures. Zheng *et al.* (2001) demonstrated that the decavanadate anion works as a template for the self-assembly of Cu²⁺ complexes with organic ligands. Compound (II) exemplifies that the decavanadate anion also acts as a template for the self-assembly of purely inorganic cations.

Experimental

A decavanadate solution was prepared according to the method described by Jahr & Preuss (1965). V₂O₅ (1.48 g) was dissolved in aqueous H_2O_2 [30% aqueous H_2O_2 (20 ml) diluted by water (200 ml)], and the solution was stirred vigorously for 12 h to remove excess H₂O₂. An aqueous solution (20 ml) containing Cu(CH₃-COO)₂·H₂O (1.28 g) was added to this solution and the pH was adjusted to 2.98 by the addition of CH₃COOH. The reaction solution was concentrated to a total volume of 175 ml by slow evaporation at room temperature, and the solubility of the target compound was reduced via vapor-phase transfer of acetone into this solution. Orange prismatic crystals of (I) were obtained after 2-5 d. To an aqueous solution (50 ml) containing NaVO₃ (0.78 g), Cu(CH₃₋ COO)₂·H₂O (0.512 g in 20 ml water) was added, followed by an adjustment of the pH to 3.39 with CH₃COOH. Following vapor-phase diffusion of acetone into this mixture, orange prismatic crystals of (II) precipitated after 1-2 d.

Compound (I)

Crystal data Cu₃V₁₀O₂₈·24H₂O Z = 1M = 1580.40 $D_{\rm r} = 2.562 {\rm Mg} {\rm m}^{-3}$ Triclinic, P1 Mo $K\alpha$ radiation a = 8.8567 (5) ÅCell parameters from 5734 b = 10.7189 (6) Å reflections c = 11.5492(7) Å $\theta = 1.9 - 27.5^{\circ}$ $\mu = 3.83 \text{ mm}^{-1}$ $\alpha = 104.662 (2)^{\circ}$ $\beta = 95.579(3)^{\circ}$ T = 93 (2) K $\gamma = 102.064 (3)^{\circ}$ Plate, orange $V = 1024.14 (10) \text{ Å}^3$ $0.36 \times 0.23 \times 0.12 \text{ mm}$

Data collection Rigaku R-AXIS RAPID 4688 independent reflections diffractometer 4486 reflections with $I > 2\sigma(I)$ Oscillation ω scans $R_{\rm int} = 0.020$ Absorption correction: numerical $\theta_{\rm max} = 27.5^{\circ}$ $h=-11 \rightarrow 11$ (Higashi, 1999) $T_{\min} = 0.359, T_{\max} = 0.687$ $k = -13 \rightarrow 13$ $l = -14 \rightarrow 14$ 9090 measured reflections Refinement Refinement on F^2 $w = 1/[\sigma^2(F_a^2) + (0.0468P)^2$ + 0.0054P] $R[F^2 > 2\sigma(F^2)] = 0.022$ $wR(F^2) = 0.077$ where $P = (F_{a}^{2} + 2F_{c}^{2})/3$ S = 1.34 $(\Delta/\sigma)_{\rm max} = 0.001$ $\Delta \rho_{\rm max} = 1.23 \text{ e} \text{ } \text{\AA}^{-3}$ 4688 reflections $\Delta \rho_{\rm min} = -0.97 \ {\rm e} \ {\rm \AA}^{-3}$ 395 parameters Extinction correction: SHELXL97 All H-atom parameters refined Extinction coefficient: 0.0433 (12)

Table 1

Selected interatomic distances (Å) for (I).

V1-01	1.6054 (13)	V4-O9	1.8587 (13)
V1-O8	1.8193 (13)	V4-011	1.8825 (13)
V1-O9	1.8238 (13)	V4-O6	2.0526 (13)
V1-O12	1.9972 (13)	V4-O14	2.2916 (13)
V1-O13 ⁱ	2.0016 (13)	V5-O6	1.6833 (13)
V1-O14	2.2331 (12)	V5-O5 ⁱ	1.6930 (14)
V2-O2	1.6050 (13)	V5-O12	1.8930 (13)
V2-O11	1.8067 (13)	V5-O13	1.9375 (13)
V2-O10	1.8333 (13)	V5-O14	2.1122 (13)
V2-O13	1.9927 (13)	V5-O14 ⁱ	2.1299 (13)
V2-O12 ⁱ	2.0211 (13)	Cu1-O15	1.9296 (14)
V2-O14	2.2379 (12)	Cu1-O16	1.9690 (14)
V3-O3	1.6041 (14)	Cu1-O3	2.3254 (13)
V3-O7	1.8218 (13)	Cu2-O17	1.9323 (14)
V3-O8	1.8719 (14)	Cu2-O18	2.0166 (14)
V3-O10	1.8903 (13)	Cu2-O19	2.2918 (14)
V3-O5	2.0349 (13)	Cu3-O20	1.9624 (14)
V3-O14	2.3021 (13)	Cu3-O21	2.0018 (15)
V4-O4	1.5992 (13)	Cu3-O22	2.3272 (14)
V4-07	1.8360 (13)		

Symmetry code: (i) 1 - x, 1 - y, 1 - z.

Table 2

Hydrogen-bonding geometry (Å, °) for (I).

$D - H \cdot \cdot \cdot A$	D-H	$H \cdot \cdot \cdot A$	$D \cdot \cdot \cdot A$	$D - H \cdot \cdot \cdot A$
O15−H15A····O19 ⁱⁱⁱ	0.83 (3)	1.85 (3)	2.640 (2)	160 (3)
$O15-H15B\cdots O24^{viii}$	0.69 (3)	2.02 (3)	2.694 (2)	165 (3)
$O16-H16A\cdots O23^{ix}$	0.72 (3)	2.00 (3)	2.713 (2)	169 (3)
O16−H16B···O22	0.65 (3)	2.23 (4)	2.881 (2)	173 (4)
O17−H17A···O10	0.72(3)	1.94 (3)	2.653 (2)	167 (3)
$O17-H17B\cdots O4^{viii}$	0.72 (3)	2.04 (3)	2.726 (2)	161 (3)
$O18-H18A\cdots O24^{x}$	0.75 (3)	2.08 (3)	2.823 (2)	168 (3)
$O18-H18B\cdots O11^{x}$	0.72 (3)	2.04 (3)	2.7623 (19)	176 (3)
$O19-H19A\cdots O23^{viii}$	0.73 (3)	2.02 (3)	2.729 (2)	165 (3)
$O19-H19B\cdots O26^{i}$	0.85 (3)	1.84 (3)	2.685 (2)	172 (3)
$O20-H20A\cdots O26^{xi}$	0.74 (4)	2.15 (3)	2.805 (2)	148 (3)
$O20-H20A\cdots O1^{xi}$	0.74 (4)	2.64 (3)	3.095 (2)	122 (3)
O20−H20B···O8	0.73 (3)	2.09 (3)	2.778 (2)	159 (3)
$O21-H21A\cdots O13^{i}$	0.69 (4)	2.01 (4)	2.6868 (19)	170 (4)
$O21 - H21B \cdots O25$	0.81 (4)	2.09 (4)	2.894 (2)	175 (3)
$O22-H22A\cdots O9^{viii}$	0.72 (4)	2.07 (4)	2.786 (2)	172 (3)
O22−H22 <i>B</i> ···O5	0.73 (3)	2.05 (3)	2.777 (2)	176 (3)
$O23-H23A\cdots O10^{x}$	0.72 (3)	2.12 (4)	2.820 (2)	166 (3)
O23−H23 <i>B</i> ···O2	0.72 (3)	2.05 (4)	2.759 (2)	170 (4)
$O24-H24A\cdots O25^{xii}$	0.86 (3)	2.00 (3)	2.838 (2)	167 (3)
$O24 - H24B \cdots O7$	0.78 (4)	2.08 (4)	2.807 (2)	156 (3)
O25−H25A···O1	0.60(3)	2.17 (4)	2.761 (2)	169 (4)
$O25-H25B\cdots O9^{xi}$	0.79 (4)	2.27 (4)	3.014 (2)	156 (4)
O26−H26A···O12	0.76 (4)	2.04 (4)	2.780 (2)	167 (4)
$O26-H26B\cdots O6^{xiii}$	0.75 (3)	2.22 (3)	2.956 (2)	168 (3)

Symmetry codes: (i) 1 - x, 1 - y, 1 - z; (ii) -x, 1 - y, -z; (vii) x - 1, y, z; (ix) x - 1, y - 1, z; (x) 1 - x, 1 - y, -z; (xi) 1 - x, -y, 1 - z; (xii) x, y, z - 1; (xiii) 2 - x, 1 - y, 1 - z.

Compound (II)

Crystal data

$CuNa_4V_{10}O_{28}\cdot 23H_2O$	Z = 1
$M_r = 1527.27$	$D_x = 2.352 \text{ Mg m}^{-3}$
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 8.9829 (6) Å	Cell parameters from 4086
b = 10.7777 (5) Å	reflections
c = 11.9761 (9) Å	$\theta = 1.8-27.4^{\circ}$
$\alpha = 104.979 \ (4)^{\circ}$	$\mu = 2.72 \text{ mm}^{-1}$
$\beta = 99.028 \ (2)^{\circ}$	T = 93 (2) K
$\gamma = 99.963 \ (3)^{\circ}$	Pillar, orange
$V = 1078.12 (12) \text{ Å}^3$	$0.20\times0.14\times0.07~\mathrm{mm}$

Table 3

Selected interatomic distances (Å) for (II).

1.6148 (14)	V4-014	2.2922 (14)
1.8152 (14)	V5–O5 ⁱ	1.6865 (14)
1.8298 (14)	V5-O6	1.6936 (14)
1.9879 (14)	V5-O12	1.9060 (14)
2.0049 (14)	V5-O13	1.9248 (14)
2.2417 (13)	V5-O14 ⁱ	2.1079 (13)
1.6223 (14)	V5-O14	2.1282 (13)
1.7969 (14)	Cu1-O15	1.9247 (15)
1.8239 (14)	Cu1-O16	2.0420 (15)
1.9909 (13)	Cu1-O17	2.3896 (18)
2.0302 (14)	Na1-O20	2.3511 (17)
2.2436 (13)	Na1-O21	2.3565 (17)
1.6014 (14)	Na1-O18	2.3603 (17)
1.8253 (14)	Na1-O19	2.4239 (18)
1.8655 (14)	Na1-O17	2.4553 (18)
1.8755 (14)	Na1-O16	2.4842 (17)
2.0525 (14)	Na2-O21	2.3778 (15)
2.2988 (13)	Na2-O22	2.3888 (15)
1.6029 (14)	Na2-O20	2.3899 (16)
1.8331 (14)	Na3-O3	2.2900 (14)
1.8655 (14)	Na3-O19	2.3765 (17)
1.9024 (14)	Na3-O23	2.4543 (17)
2.0286 (14)		
	$\begin{array}{c} 1.6148 (14) \\ 1.8152 (14) \\ 1.8298 (14) \\ 1.9879 (14) \\ 2.0049 (14) \\ 2.2417 (13) \\ 1.6223 (14) \\ 1.7969 (14) \\ 1.8239 (14) \\ 1.9099 (13) \\ 2.0302 (14) \\ 2.0302 (14) \\ 2.2436 (13) \\ 1.6014 (14) \\ 1.8253 (14) \\ 1.8655 (14) \\ 1.8755 (14) \\ 2.0525 (14) \\ 2.0525 (14) \\ 2.0525 (14) \\ 2.0525 (14) \\ 2.0525 (14) \\ 1.8331 (14) \\ 1.8655 (14) \\ 1.8029 (14) \\ 1.8055 (14) \\ 1.9024 (14) \\ 2.0286 (14) \end{array}$	$\begin{array}{llllllllllllllllllllllllllllllllllll$

Symmetry code: (i) 1 - x, 1 - y, 1 - z.

Table 4

H	ĺyd	rogen-	bonding	geometry	(A, °]) for	(II).
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$D - H \cdot \cdot \cdot A$	D-H	$H \cdot \cdot \cdot A$	$D \cdot \cdot \cdot A$	$D - \mathbf{H} \cdot \cdot \cdot A$
$O15-H15A\cdots O10^{iii}$	0.78 (4)	1.83 (4)	2.600 (2)	170 (3)
O15−H15B···O18	0.71(3)	2.06 (3)	2.743 (2)	164 (3)
O16−H16A···O23	0.74 (3)	1.94 (3)	2.683 (2)	175 (3)
$O16-H16B\cdots O2^{iii}$	0.88 (4)	1.94 (4)	2.820 (2)	174 (3)
$O17 - H17A \cdots O25A$	0.67 (3)	2.01 (3)	2.661 (4)	164 (3)
$O17 - H17A \cdots O25B$	0.67 (3)	2.37 (3)	3.013 (4)	160 (3)
$O17 - H17B \cdot \cdot \cdot O11^{x}$	0.79 (4)	2.02 (4)	2.811 (2)	174 (3)
$O18-H18A\cdots O12^{xiv}$	0.73 (4)	2.01(4)	2.729 (2)	170 (3)
$O18-H18B\cdots O6^{x}$	0.77(3)	2.06 (3)	2.819 (2)	168 (3)
O19−H19A···O25B	0.77(3)	1.94 (4)	2.691 (4)	165 (3)
O19−H19A···O25A	0.77 (3)	2.37 (4)	3.076 (4)	154 (3)
O19−H19B···O22	0.80(4)	2.14 (4)	2.922 (2)	168 (3)
$O20-H20A\cdots O1^{xiv}$	0.68 (3)	2.13 (3)	2.794 (2)	166 (3)
$O20-H20B\cdots O8^{ii}$	0.76 (4)	2.04 (4)	2.778 (2)	165 (4)
$O21-H21A\cdots O13^{x}$	0.76 (3)	1.99 (3)	2.738 (2)	169 (3)
$O21 - H21B \cdots O24^{xii}$	0.83 (4)	2.12 (4)	2.941 (2)	171 (3)
$O22-H22A\cdots O5^{ii}$	0.79 (3)	2.10(3)	2.884 (2)	173 (3)
$O22-H22B\cdots O9^{xv}$	0.76 (4)	2.09 (4)	2.844 (2)	173 (3)
$O23-H23A\cdots O26^{viii}$	0.76 (4)	2.13 (4)	2.883 (5)	171 (4)
$O23-H23A\cdots O26^{ii}$	0.76 (4)	2.28 (4)	3.015 (5)	162 (4)
$O23-H23B\cdots O4^{viii}$	0.71 (5)	2.09 (5)	2.752 (2)	154 (5)
$O23-H23B\cdots O15^{iii}$	0.71 (5)	2.65 (4)	3.137 (2)	127 (4)
$O24-H24A\cdots O1$	0.72 (5)	2.25 (5)	2.918 (3)	154 (4)
$O24-H24B\cdots O9^{xi}$	0.64 (5)	2.59 (4)	3.118 (2)	143 (5)
$O26-H26A\cdots O7$	0.80(7)	2.04 (7)	2.798 (5)	158 (6)
$O26-H26B\cdots O24^{xii}$	0.67 (7)	2.34 (8)	2.980 (4)	161 (8)

Symmetry codes: (ii) -x, -y, -z; (iii) -x, 1-y, -z; (viii) x-1, y, z; (x) 1-x, 1-y, -z; (xi) 1-x, -y, 1-z; (xi) x, y, z-1; (xiv) x-1, y, z-1; (xv) 1-x, -y, -z.

Data collection

Rigaku R-AXIS RAPID diffractometer Oscillation ω scans Absorption correction: multi-scan (Higashi, 1995) $T_{min} = 0.666, T_{max} = 0.827$ 12 066 measured reflections 4887 independent reflections

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.028$ $wR(F^2) = 0.068$ S = 1.244887 reflections 395 parameters All H-atom parameters refined $w = 1/[\sigma^2(F_o^2) + (0.0307P)^2 + 0.881P]$ where $P = (F_o^2 + 2F_o^2)/3$ 4594 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.029$ $\theta_{\text{max}} = 27.4^{\circ}$ $h = -11 \rightarrow 10$ $k = -13 \rightarrow 13$ $l = -15 \rightarrow 15$

 $\begin{array}{l} (\Delta/\sigma)_{\rm max} = 0.001 \\ \Delta\rho_{\rm max} = 0.47 \ {\rm e} \ {\rm \AA}^{-3} \\ \Delta\rho_{\rm min} = -0.54 \ {\rm e} \ {\rm \AA}^{-3} \\ {\rm Extinction \ correction: \ SHELXL97} \\ {\rm Extinction \ coefficient: \ 0.0126 \ (5)} \end{array}$

In (II), the O25 water molecule is disordered over two sites, *viz*. O25A and O25B, with site occupancies of 0.53 and 0.47, respectively. Because of the correlation between the site-occupancy factors and the displacement parameters, these atoms were refined with isotropic displacement parameters and fixed site occupancies. The H atoms attached to atoms O25A and O25B could not be located. Atom O26 is located close to a center of symmetry, and thus the occupancy of this atom was fixed at 0.5.

For both compounds, data collection: *PROCESS-AUTO* (Rigaku Corporation, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ATOMS* (Dowty, 1999) and *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BC1014). Services for accessing these data are described at the back of the journal.

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