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Poly[bis(benzimidazole-N^3)copper(II)-μ-acetylenedicarboxylato-O:O'-μ-aqua] and Poly[bis(benzimidazole-N^3)copper(II)-μ-aqua-μ-fumarato-O:O']

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Abstract

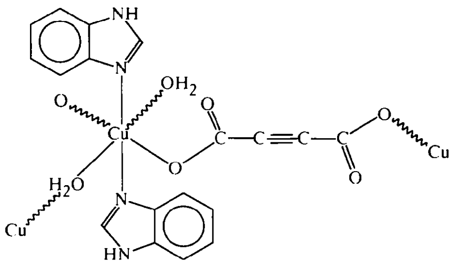
The title compounds, poly[bis(benzimidazole-N^3)-copper(II)-μ-acetylenedicarboxylato-O:O'-μ-aqua], [Cu(C_4O_4)($C_7H_6N_2$)$_2$(H_2O)], (I), and poly[bis(benzimidazole-N^3)copper(II)-μ-aqua-μ-fumarato-O:O'], [Cu($C_4H_2O_4$)($C_7H_6N_2$)$_2$(H_2O)], (II), have been synthesized and their crystal structures determined by single-crystal X-ray diffraction at room temperature. Structures (I) and (II) are very similar. In both structures, the centrosymmetric Cu atoms are bridged by the dicarboxylic acid ions in a bis-monodentate fashion, forming polymeric chains. The Cu···Cu intrachain distances are 9.546 (1) Å in (I) and 9.276 (1) Å in (II). These chains are connected by bridging H_2O molecules, which are located on the twofold axes by Cu–H_2O bonds of 2.593 (1) Å in (I) and 2.668 (1) Å in (II). The Cu···Cu distances in the –Cu–H_2O–Cu– chains are 5.090 (1) and 5.199 (1) Å in (I) and (II), respectively. The base of the elongated octahedron of the Cu^{II} atoms is formed by two short Cu—O and two short Cu—N bonds [Cu—O = 1.970 (1) and 1.946 (2) Å, and Cu—N = 2.005 (2) and 2.016 (2) Å in (I) and (II), respectively]. The coordination of the carboxylate groups and the hydrogen bonds are discussed. The isostructurality of the title compounds and of their previously investigated homologue, (III), has been analysed.

Comment

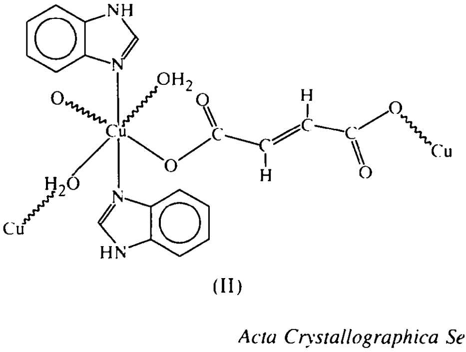
The Cu^{II} complexes of dicarboxylic acids are of interest because of their rich variety of structural features and their interesting magnetic properties. Most often, the Cu atoms are bridged by the dicarboxylic acid ions in a bis-bidentate fashion (Tosik *et al.*, 1995*a*), and rarely in the chelate-bridge tridentate fashion (Tosik *et al.*, 1995*b*) or the bis-bridge monodentate fashion (Tosik & Bukowska-Strzyżewska, 1992).

A knowledge of the structure is needed to allow an interpretation of the magnetic properties of polynuclear complexes with paramagnetic centres. It has been shown that two paramagnetic centres could interact through extended bridging ligands, even if these centres were relatively far away from each other (Verdaguer *et al.*, 1984).

The bridging acetylenedicarboxylato and fumarato ligands in the title compounds, (I) and (II), give a Cu···Cu separation of less than 10 Å, and the present μ-aqua Cu^{II} complexes provide an opportunity for analysing the interaction between neighbouring Cu^{II} centres through the apical water molecules. The magnetic properties of the title compounds will be investigated at a later date.



(I)



(II)

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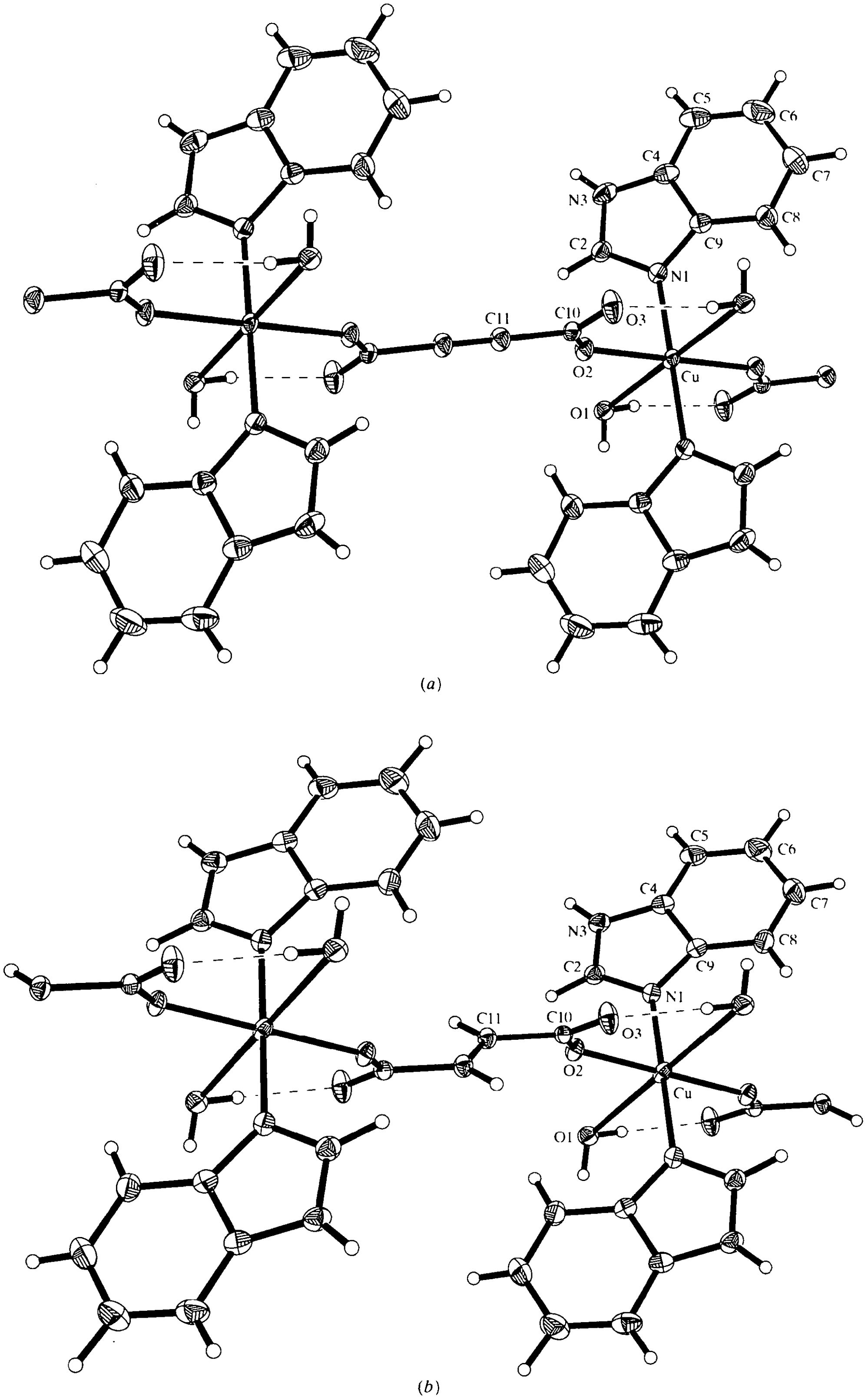

Fig. 1. The repeat unit of the polymeric structure of (*a*) (I) and (*b*) (II). Displacement ellipsoids are drawn at the 30% probability level.

The repeat units of the polymeric structures of (I) and (II) are given in Figs. 1(*a*) and 1(*b*), respectively. Each Cu atom and dicarboxylate ion is located on a symmetry centre. The Cu atoms are connected by bridging of the bidentate dicarboxylate ions to form polymeric chains, with Cu· · ·Cu distances of 9.546 (1) Å in (I) and 9.276 (1) Å in (II). The crystal packing shown in Fig. 2 illustrates the two-dimensional network of Cu^{II} complexes. The chains shown in Figs. 1(*a*) and 1(*b*) are connected by bridging H_2O molecules located on the twofold axes by Cu–H_2O bonds of 2.593 (1) and 2.668 (1) Å in (I) and (II), respectively. The Cu· · ·Cu distances in the –Cu–H_2O–Cu– chains are 5.090 (1) and 5.199 (1) Å in (I) and (II), respectively. In both structures, the base of the elongated octahedron of Cu^{II} ions is formed by two short Cu—O bonds to the two carboxylic ions and two Cu—N bonds, one from each benzimidazole molecule, with Cu—O = 1.970 (1) and 1.946 (2) Å, and Cu—N = 2.005 (2) and 2.016 (2) Å in (I) and (II), respectively. The present structures give a rare example of monodentate carboxylic groups displaying *syn* coordination. These groups are not in the ideal coplanar configuration with the Cu atom, instead giving Cu—O2—C10—O3 torsion angles of 11.3 (3)° in (I) and 14.6 (4)° in (II). The distances of the Cu atoms from the O2/O3/C10/C11 planes are 0.276 (3) and 0.436 (4) Å in (I) and (II), respectively. The uncoordinated O3 atom in both structures is the acceptor of the O1—H1· · ·O3 hydrogen bond inside the Cu^{II} coordination polyhedron, and of the N3—H3· · ·O3 bond between neighbouring Cu^{II} complexes (Fig. 2). Selected bond lengths of (I) and (II), and the geometry of the hydrogen bonds are given in Tables 2, 3, 5 and 6. Both carboxylate groups show a similar delocalization of π bonds, with C10—O2 = 1.254 (2) and 1.260 (3) Å, and C10—O3 = 1.228 (2) and 1.238 (3) Å in (I) and (II), respectively. The O2 atom, with the shorter C—O bond, forms a longer Cu—O bond [Cu—O2 = 1.970 (1) and 1.946 (2) Å in (I) and (II), respectively].

The title compounds, (I) and (II), and the previously investigated succinatobis(benzimidazole)copper(II) monohydrate, (III) (Tosik & Bukowska-Strzyżewska, 1992), despite the different hybridization of the central C atoms of the dicarboxylic acid ions, crystallize in the same *C*2/*c* space group, with very similar unit cells. Their isostructurality can be expressed by the numerical descriptors of isostructurality $I_i(n^*)$ and $I_i(n)$ proposed by Kálmán (Kálmán & Párkányi, 1997). The unit-cell similarity indices, Π [defined as $\Pi = |(a+b+c)/(a'+b'+c')-1|$, where *a*, *b*, *c* and *a*′, *b*′, *c*′ are the orthogonalized lattice parameters of the related crystals], show full cell similarity. For the crystal pairs (I)/(II), (I)/(III) and (II)/(III), the Π values are 0.0084, 0.0108 and 0.0025, respectively. The isostructurality index, $I_i(n) = [1-(\Sigma\Delta R_i^2/n)^{0.5}] \times 100$, and the molecular isometricity index, $I_i(n^*)$, indicate the average distance of the identical *n* atoms being compared. For $I_i(n)$, ΔR_i (the distances of identical atoms) are computed from the compared unit cells and for $I_i(n^*)$, from the best fit of the compared molecules. $I_i(n^*)$ and $I_i(n)$ values of 100% indicate full isometricity or isostructurality, and $I_i(n^*)$ and $I_i(n)$ of 0% indicate an average difference of 1 Å in the positions of the identical compared atoms. Fig. 3 gives $I_i(n^*)$ and $I_i(n)$ *versus* n^* or *n*, for the (I)/(II), (I)/(III) and (II)/(III) pairs which were analysed. Kálmán & Párkányi (1997) stress that, although molecular isometricity is a prerequisite for isostructurality, it does not guarantee a similar packing motif. For all the analysed pairs, we observe similarly high isometricity indices [for the (II)/(III), (I)/(III) and (I)/(II) pairs, $I_i(14^*)$ = 93.5, 90.5 and 90.1%, respectively], but

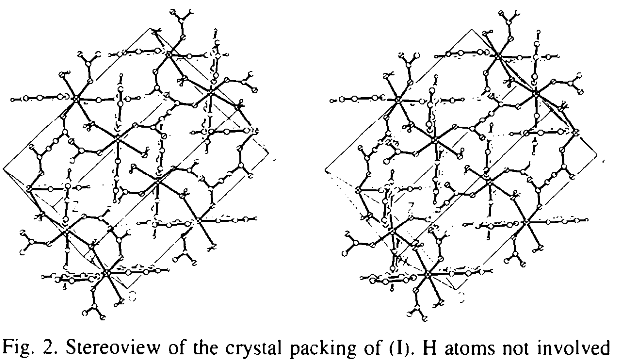

Fig. 2. Stereoview of the crystal packing of (I). H atoms not involved in the hydrogen bonds have been omitted.

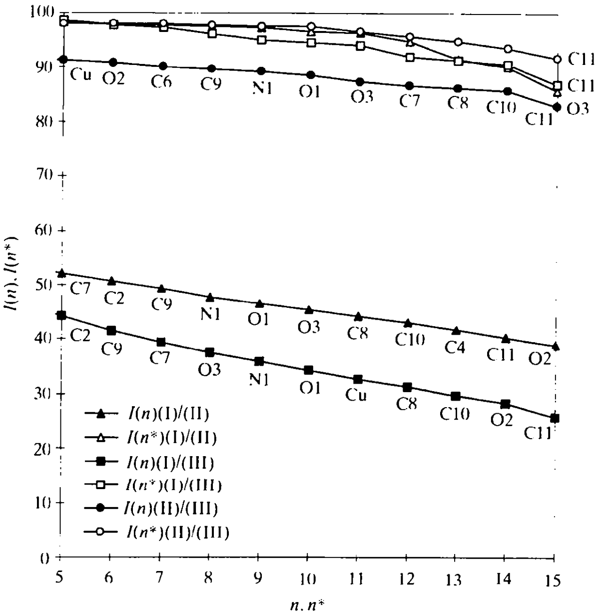

Fig. 3. Isostructurality and molecular isometricity indices; $I_i(n)$ and $I_i(n^*)$ are plotted *versus* *n* (n^*) for the three crystal pairs, (I)/(II), (I)/(III) and (II)/(III); *n* and n^* are the number of distances between the identical compared atoms in the unit cells and in the best fit of the molecules, respectively.

only the (II)/(III) pair has a large isostructurality index, with $I_i(15) = 83.0$ and $I_i(5) = 91.2\%$. For the (I)/(II) and (I)/(III) pairs, the values of $I_i(15)$ are 38.9 and 25.8%, respectively. Thus, for the three present compounds, we can observe a distinct lowering of the isostructurality, caused by the linear hybridization of the central C atoms of the dicarboxylic acid ions. The isostructural core of the compared compounds may be defined by the four shortest ΔR_i values. For the complexes analysed here, the best fit is observed for the external benzimidazole ligands, and not for the central Cu^{II} polyhedron.

Experimental

Benzimidazole (2 mmol) and the appropriate dicarboxylic acid (1 mmol), dissolved in a mixture of water (40 ml) and dimethyl sulfoxide (10 ml), were heated to boiling point and then mixed with $CuCl_2.2H_2O$ (1 mmol) dissolved in water (20 ml). Slow evaporation produced crystals suitable for X-ray diffraction after several days.

Compound (I)

Crystal data

$[Cu(C_4O_4)(C_7H_6N_2)_2(H_2O)]$
$M_r = 429.88$
Monoclinic
$C2/c$
$a = 12.167\ (1)$ Å
$b = 14.712\ (2)$ Å
$c = 10.180\ (1)$ Å
$\beta = 103.48\ (1)°$
$V = 1772.0\ (3)$ Å^3
$Z = 4$
$D_x = 1.611$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 48 reflections
$\theta = 4.3–24.7°$
$\mu = 1.27$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.75 × 0.25 × 0.15 mm
Blue

Data collection

Siemens *P*3 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.733$, $T_{max} = 0.827$
2009 measured reflections
1923 independent reflections
1679 reflections with $I > 2\sigma(I)$

$R_{int} = 0.009$
$\theta_{max} = 27°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 18$
$l = -13 \rightarrow 12$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.071$
$S = 1.080$
1923 reflections
157 parameters
All H-atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.033P)^2 + 1.824P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.248$ e Å^{-3}
$\Delta\rho_{min} = -0.286$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (I)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^ia^j\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu	0	1/2	0	0.02860 (11)
O1	0	0.53389 (14)	1/4	0.0354 (4)
O2	−0.09022 (11)	0.39349 (9)	0.02736 (14)	0.0354 (3)
O3	−0.13751 (14)	0.33452 (11)	−0.18049 (16)	0.0512 (4)
N1	0.14088 (13)	0.42555 (11)	0.05960 (16)	0.0320 (3)
C2	0.15342 (19)	0.36154 (14)	0.1529 (2)	0.0396 (4)
N3	0.25201 (17)	0.31748 (14)	0.1698 (2)	0.0470 (5)
C4	0.30904 (17)	0.35318 (15)	0.0804 (2)	0.0422 (5)
C5	0.4127 (2)	0.3315 (2)	0.0524 (3)	0.0600 (7)
C6	0.4441 (2)	0.3798 (2)	−0.0467 (4)	0.0710 (8)
C7	0.3759 (2)	0.4493 (2)	−0.1165 (3)	0.0659 (8)
C8	0.2738 (2)	0.47144 (18)	−0.0880 (3)	0.0480 (5)
C9	0.23981 (16)	0.42193 (13)	0.0117 (2)	0.0349 (4)
C10	−0.14280 (15)	0.34038 (12)	−0.0618 (2)	0.0305 (4)
C11	−0.21824 (17)	0.27642 (13)	−0.0151 (2)	0.0368 (4)

Table 2. *Selected geometric parameters (Å, °) for (I)*

Cu—O1	2.5934 (5)	N1—C2	1.321 (3)
Cu—O2	1.9699 (12)	N1—C9	1.401 (2)
Cu—N1	2.0052 (15)	N3—C2	1.339 (3)
O2—C10	1.254 (2)	N3—C4	1.370 (3)
O3—C10	1.228 (2)		
O1—Cu—O2	83.49 (5)	C2—N3—C4	107.86 (19)
O1—Cu—N1	90.21 (5)	N1—C2—N3	112.8 (2)
O2—Cu—N1	89.31 (6)	N3—C4—C5	132.1 (2)
Cu—O1—Cu^{i}	157.83 (9)	N3—C4—C9	105.72 (18)
Cu—O2—C10	126.90 (12)	N1—C9—C4	108.64 (18)
C2—N1—C9	105.00 (17)	O2—C10—O3	128.38 (17)
C2—N1—Cu	123.66 (14)	O3—C10—C11	116.98 (17)
C9—N1—Cu	131.18 (13)	O2—C10—C11	114.63 (17)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Table 3. *Hydrogen-bonding geometry (Å, °) for (I)*

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.79 (2)	2.00 (2)	2.757 (2)	161 (3)
N3—H3···O3^{ii}	0.78 (3)	2.10 (3)	2.859 (2)	163 (3)

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

Compound (II)

Crystal data

$[Cu(C_4H_2O_4)(C_7H_6N_2)_2(H_2O)]$
$M_r = 431.89$
Monoclinic
$C2/c$
$a = 12.941\ (2)$ Å
$b = 13.292\ (2)$ Å
$c = 10.398\ (2)$ Å
$\beta = 100.09\ (1)°$
$V = 1760.9\ (5)$ Å^3
$Z = 4$
$D_x = 1.629$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 30 reflections
$\theta = 4.0–13.0°$
$\mu = 1.28$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.70 × 0.25 × 0.15 mm
Blue

Data collection

Siemens *P*3 diffractometer
$\omega/2\theta$ scans

$R_{int} = 0.016$
$\theta_{max} = 27°$

Absorption correction:
ψ scan (North *et al.*, 1968)
$T_{\min} = 0.703$, $T_{\max} = 0.825$
2000 measured reflections
1917 independent reflections
1563 reflections with $I > 2\sigma(I)$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 16$
$l = -13 \rightarrow 13$
3 standard reflections every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.079$
$S = 1.063$
1917 reflections
161 parameters
All H-atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0319P)^2 + 3.0416P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.262$ e Å^{-3}
$\Delta\rho_{\min} = -0.347$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 4. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (II)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^ia^j\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu	0	1/2	0	0.02384 (13)
O1	0	0.5453 (2)	1/4	0.0319 (6)
O2	−0.07246 (13)	0.37887 (12)	0.04052 (16)	0.0270 (4)
O3	−0.14417 (16)	0.32996 (15)	−0.16071 (18)	0.0409 (5)
N1	0.13576 (15)	0.42477 (15)	0.05563 (19)	0.0253 (4)
N3	0.24643 (18)	0.31795 (17)	0.1727 (2)	0.0333 (5)
C2	0.1504 (2)	0.3601 (2)	0.1528 (3)	0.0313 (6)
C4	0.2998 (2)	0.35642 (19)	0.0806 (2)	0.0300 (5)
C5	0.4003 (2)	0.3389 (2)	0.0558 (3)	0.0433 (7)
C6	0.4280 (3)	0.3899 (3)	−0.0470 (4)	0.0542 (9)
C7	0.3590 (3)	0.4566 (3)	−0.1233 (4)	0.0521 (8)
C8	0.2601 (2)	0.4741 (2)	−0.0975 (3)	0.0387 (7)
C9	0.23025 (19)	0.42381 (18)	0.0061 (2)	0.0272 (5)
C10	−0.13374 (17)	0.32638 (17)	−0.0402 (2)	0.0235 (5)
C11	−0.20004 (19)	0.25624 (18)	0.0222 (2)	0.0262 (5)

Table 5. *Selected geometric parameters (Å, °) for (II)*

Cu—O1	2.6683 (7)	N1—C2	1.315 (3)
Cu—O2	1.9461 (15)	N1—C9	1.407 (3)
Cu—N1	2.016 (2)	N3—C2	1.346 (3)
O2—C10	1.260 (3)	N3—C4	1.373 (3)
O3—C10	1.238 (3)		
O1—Cu—O2	83.88 (7)	C2—N3—C4	107.6 (2)
O1—Cu—N1	88.92 (7)	N1—C2—N3	113.3 (2)
O2—Cu—N1	87.49 (7)	N3—C4—C5	131.9 (3)
Cu—O1—Cu^{i}	153.93 (12)	N3—C4—C9	105.7 (2)
C10—O2—Cu	125.81 (15)	N1—C9—C4	108.6 (2)
C2—N1—C9	104.9 (2)	O2—C10—O3	126.7 (2)
C2—N1—Cu	123.08 (18)	O2—C10—C11	113.6 (2)
C9—N1—Cu	131.97 (16)	O3—C10—C11	119.7 (2)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

Table 6. *Hydrogen-bonding geometry (Å, °) for (II)*

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.81 (3)	1.98 (4)	2.775 (2)	167 (4)
N3—H3···O3^{ii}	0.83 (3)	2.02 (3)	2.831 (3)	167 (3)

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

The title structures were solved by the conventional Patterson method and refined by full-matrix least-squares calculations. All non-H atoms were refined anisotropically. All H atoms were located from a difference synthesis and refined isotropically.

For both compounds, data collection: *P*3 software; cell refinement: *P*3 software; data reduction: *XDISK* in *SHELXTL/PC* (Sheldrick, 1990); program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997*b*); molecular graphics: *XP* in *SHELXTL/PC*; 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: KA1272). Services for accessing these data are described at the back of the journal.

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A Dinuclear Vanadyl–Oxalate Compound

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Abstract

The title compound, bis(tetraphenylphosphonium) μ-oxalato-$O^1,O^2{:}O^{1'},O^{2'}$-bis[aqua(oxalato-O^1,O^2)oxovanadium(IV)] octahydrate, $[(C_6H_5)_4P]_2[V_2O_2(C_2O_4)_3(H_2O)_2].8H_2O$, consists of an anionic vanadyl dimer linked by an oxalate ligand and additionally coordinated by two non-bridging oxalate and two water mol-