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Three-dimensional frameworks built from piperazine-2,5-dione and simple metal salts (*M* = Co, Ni, Cu and Ag)

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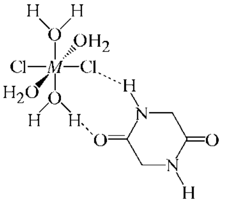
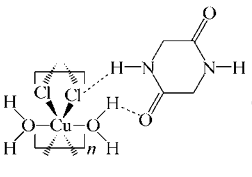
Compounds *trans*-tetraaquadichloridocobalt(II)–piperazine-2,5-dione (1/1), $[CoCl_2(H_2O)_4]\cdot C_4H_6N_2O_2$, (I), and *trans*-tetraaquadichloridonickel(II)–piperazine-2,5-dione (1/1), $[NiCl_2(H_2O)_4]\cdot C_4H_6N_2O_2$, (II), are isomorphous. In each structure, the metal complex and the piperazinedione unit both lie across centres of inversion in the space group $P2_1/n$. The $[MCl_2(H_2O)_4]$ units (*M* = Co or Ni) are linked by O—H···Cl hydrogen bonds into sheets of $R_2^2(8)$ and $R_4^2(12)$ rings, and these sheets are linked by the piperazinedione components *via* a combination of O—H···O and N—H···Cl hydrogen bonds into a three-dimensional framework. In *catena*-poly[[[*trans*-diaquacopper(II)]-di-μ-chlorido] piperazine-2,5-dione solvate], $\{[CuCl_2(H_2O)_2]\cdot C_4H_6N_2O_2\}_n$, (III), the metal ion and the piperazine unit both lie across centres of inversion in the space group $I2/a$. The coordination polymer forms chains of centrosymmetric $[CuCl_2(H_2O)_2]$ units running parallel to [010] and these are linked by the piperazinedione units into a three-dimensional framework structure. In poly[μ_3-nitrato-μ_2-piperazine-2,5-dione-silver(I)], $[Ag(NO_3)(C_4H_6N_2O_2)]_n$, (IV), the silver and nitrate ions lie on mirror planes in the space group *Pnma*, while the piperazinedione unit lies across a centre of inversion. The compound is a coordination polymer containing five-coordinate approximately square-pyramidal Ag, in which the ligating O atoms are derived from three different nitrate ligands and two different piperazinedione ligands. The ionic components form sheets in which each anion is coordinated to three different cations, and these sheets are linked into a three-dimensional framework by the organic ligands, each of which coordinates to two different Ag centres. The significance of this study lies in its demonstration of a wide variety of framework types built from a common and very simple organic component with simple metal salts.

Comment

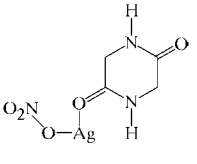
In the structure of piperazine-2,5-dione (diketopiperazine, DKP, the cyclic anhydride derived from glycine), the molecules lie across centres of inversion. They are linked into chains of $R_2^2(8)$ rings (Bernstein *et al.*, 1995) by means of N—H···O hydrogen bonds (Degeilh & Marsh, 1959; Sarangarajan *et al.*, 2005), and these chains are themselves linked into sheets by C—H···O hydrogen bonds (Sarangarajan *et al.*, 2005). In the hydrogen-bonded adducts formed by piperazine-2,5-dione with simple monocarboxylic acids, the same chain of $R_2^2(8)$ rings occurs, with the acid units pendant from it (Kartha *et al.*, 1981; Luo & Palmore, 2002), while with dicarboxylic acids or phenolic monocarboxyic acids these chains are linked into sheets (Luo & Palmore, 2002; Sarangarajan *et al.*, 2005). Unusually, the adduct formed with salicylic acid contains neither chains nor sheets but finite centrosymmetric aggregates of two acid molecules and one piperazinedione unit (Varughese & Kartha, 1982). Despite the substantial number of hydrogen-bonded adducts formed with organic acids, very little structural information is available for metal complexes of piperazine-2,5-dione. However, closely-related 1,4-dimethylpiperazine-2,5-dione forms a finite encapsulation complex with aluminium tris(2,6-diphenylphenoxide) (Ooi *et al.*, 1998), and the same amide forms a one-dimensional coordination polymer with Ph_2SnCl_2 (Kovala-Demertzi *et al.*, 1995). We report here the structures of four adducts, (I)–(IV) (Figs. 1–4), formed by piperazine-2.5-dione when it is cocrystallized from aqueous solutions containing simple salts of Co^{II}, Ni^{II}, Cu^{II} and Ag^{I}.



(I) *M*=Co
(II) *M*=Ni



(III)



(IV)

Compounds (I) and (II) are isomorphous. In these structures, the metal atom lies at a centre of inversion in the space group $P2_1/n$, selected as that at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$), and it is coordinated by two Cl and four water ligands. Within the selected asymmetric unit, the centrosymmetric piperidinedione component is linked to the neutral $[MCl_2(H_2O)_4]$ unit (*M* = Co or Ni) by a combination of O—H···O and N—H···Cl hydrogen bonds (Table 3), such that the organic component lies across the centre of inversion at ($\frac{1}{2}$, 0, 0) (Figs. 1 and 2). In each complex, the coordination of the metal atom has symmetry very close to D_{4h} (4/*mmm*), with the bond angles subtended at the metal atoms by pairs of *cis* ligands all within 3° of 90°. The *M*—O and *M*—Cl distances are slightly smaller in (II) than in (I) (Table 1).

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‡ Deceased. This paper is dedicated to the memory of T. R. Sarangarajan.

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All of the O—H and N—H bonds within the structures of (I) and (II) are involved in hydrogen bonding (Table 3) and the components are thereby linked into a three-dimensional framework structure. The $[M\mathrm{Cl}_2(\mathrm{H}_2\mathrm{O})_4]$ units are linked by the two independent O—H···Cl hydrogen bonds into a sheet parallel to $(10\overline{1})$ and containing both $R_2^2(8)$ and $R_4^2(12)$ rings (Fig. 5), and these inorganic sheets then linked by the organic components. This linking is most simply envisaged in terms of a chain running parallel to the [011] direction and containing alternating inorganic and organic units linked by hydrogen-bonded $R_2^2(8)$ rings (Fig. 6). The combination of $(10\overline{1})$ sheets and [011] chains suffices to generate the three-dimensional pillared layer structure, which could be regarded as an organic–inorganic hybrid structure. In nickel(II) chloride tetrahydrate, there are discrete $[\mathrm{NiCl}_2(\mathrm{H}_2\mathrm{O})_4]$ units, but of *cis* configuration and lying in general positions. These units are linked into a complex three-dimensional framework structure by both O—H···Cl and O—H···O hydrogen bonds (Ptasiewicz-Bak *et al.*, 1999), so that the hydrogen-bonded structure in the free hydrated chloride is quite different from that found here for the piperazinedione adduct, (II).

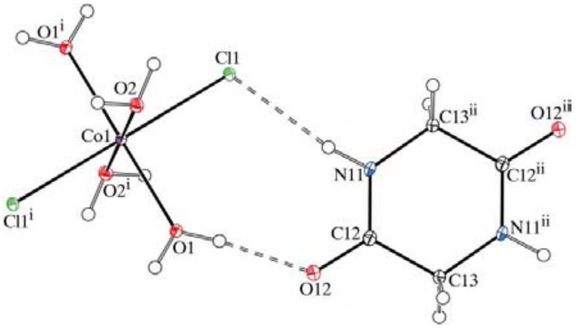

Figure 1
The independent molecular components in (I), showing the atom-labelling scheme and the hydrogen bonds within the selected asymmetric unit (dashed lines). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, -y, -z$.]

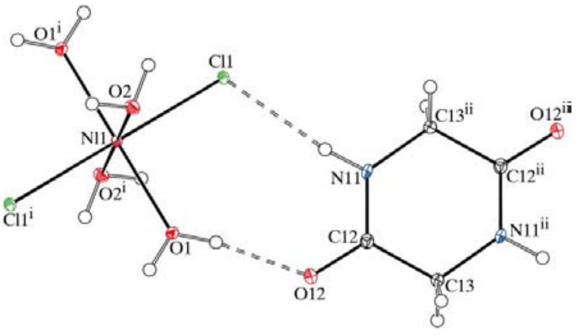

Figure 2
The independent molecular components in (II), showing the atom-labelling scheme and the hydrogen bonds within the selected asymmetric unit (dashed lines). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, -y, -z$.]

In the Cu complex, (III), the metal atom lies on a centre of inversion in the space group $I2/a$, selected as that at $(\frac{1}{4}, \frac{1}{4}, \frac{1}{4})$. It is coordinated by two water ligands and by four Cl ligands, each of which bridges two metal centres, so generating a chain of edge-fused octahedra of composition $[\mathrm{CuCl}_2(\mathrm{H}_2\mathrm{O})_2]_n$ running parallel to the [010] direction (Fig. 3). The piperazinedione component also lies across a centre of inversion, selected as that at $(\frac{1}{2}, 1, \frac{1}{2})$, so that within the selected asym-

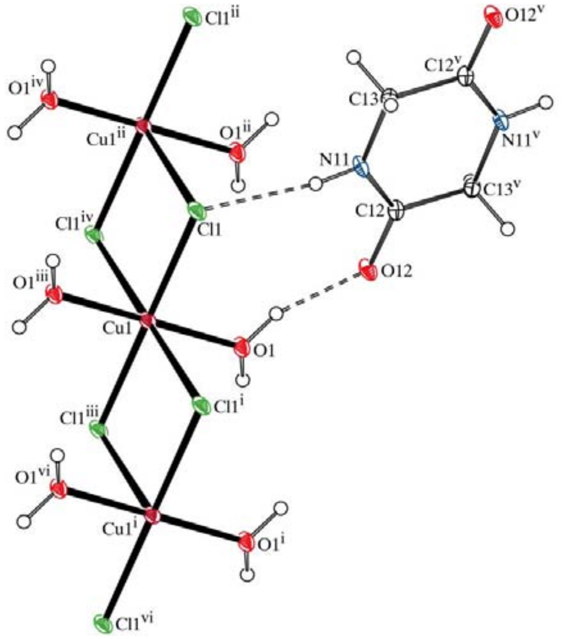

Figure 3
The independent components in (III), showing the atom-labelling scheme together with a portion of the $[\mathrm{CuCl}_2(\mathrm{H}_2\mathrm{O})_2]_n$ coordination polymer. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) $x, -1+y, z$; (ii) $x, 1+y, z$; (iii) $\frac{1}{2}-x, \frac{1}{2}-y, \frac{1}{2}-z$; (iv) $\frac{1}{2}-x, \frac{3}{2}-y, \frac{1}{2}-z$; (v) $1-x, 2-y, 1-z$; (vi) $\frac{1}{2}-x, -\frac{1}{2}-y, \frac{1}{2}-z$.]

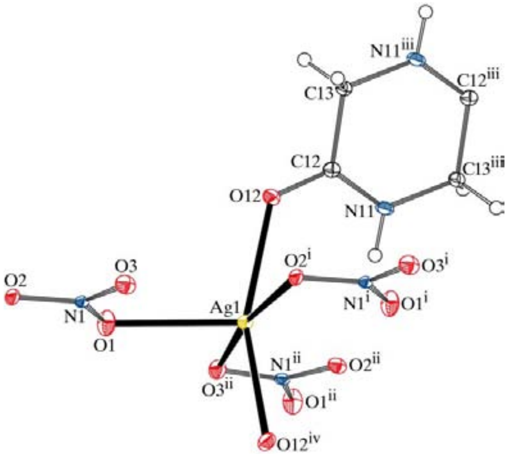

Figure 4
The independent components of adduct (IV), showing the atom-labelling scheme and the five-coordination of the Ag atom. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry codes: (i) $x, \frac{1}{4}, -1+z$; (ii) $-\frac{1}{2}+x, \frac{1}{4}, \frac{3}{2}-z$; (iii) $1-x, 1-y, -z$; (iv) $x, \frac{1}{2}-y, z$.]

metric unit the components are linked by O—H···O and N—H···Cl hydrogen bonds (Table 3), as in compounds (I) and (II). Within the coordination polymer chain, the centrosymmetric Cu_2Cl_2 units contain two significantly different Cu—Cl distances (Table 1). A similar chain, with Cu—Cl distances of 2.2724 (13) and 3.1537 (14) Å, is present in benzimidazolium tetrachlorocuprate monohydrate [Cambridge Structural Database (Allen, 2002) refcode FUTRUH; Bukowska-Strzyżewska & Skoweranda, 1987]. More frequently, however, $[CuCl_2(H_2O)_2]$ units are found as isolated square-planar units, as in NPYOCU (Williams *et al.*, 1971) and TPPOCU (Dunaj-Jurco *et al.*, 1979), or as square-planar units weakly coordinated by two further axial ligands, as in UGAYUW (Giantsidis *et al.*, 2002). The hydrogen bonds in (III) link the reference Cu atom at $(\frac{1}{4}, \frac{1}{4}, \frac{1}{4})$, *via* the piperazinedione units centred at $(0, 0, \frac{1}{2})$, $(0, -\frac{1}{2}, 0)$, $(\frac{1}{2}, \frac{1}{2}, 0)$ and $(\frac{1}{2}, 1, \frac{1}{2})$, respectively, to the Cu atoms at $(-\frac{1}{4}, -\frac{1}{4}, \frac{3}{4})$, $(-\frac{1}{4}, -\frac{5}{4}, -\frac{1}{4})$, $(\frac{3}{4}, \frac{3}{4}, -\frac{1}{4})$ and $(\frac{3}{4}, \frac{7}{4}, \frac{3}{4})$, so forming a sheet parallel to $(2\bar{1}\bar{1})$ (Fig. 7). In this manner, the reference coordination polymer chain along $(\frac{1}{4}, y, \frac{1}{4})$, is directly linked to the four chains along $(-\frac{1}{4}, y, -\frac{1}{4})$, $(-\frac{1}{4}, y, \frac{3}{4})$, $(\frac{3}{4}, y, -\frac{1}{4})$ and $(\frac{3}{4}, y, \frac{3}{4})$, thereby generating a three-dimensional framework structure.

Complex (IV), derived from $Ag(NO_3)$, is a coordination polymer, $[Ag(NO_3)(DKP)]_n$, containing five-coordinate Ag. The compound crystallizes in the space group *Pnma*, and the Ag^+ ions and all the atoms of the nitrate anions lie on mirror planes, while the DKP units lie across centres of inversion. For the selected asymmetric unit, in which DKP atom O12 is coordinated to Ag (Fig. 4), the DKP unit lies across the centre of inversion at $(\frac{1}{2}, \frac{1}{2}, 0)$, while the Ag^+ and nitrate ions lie on the mirror plane at $y = \frac{1}{4}$. The constitution of (IV) is most readily analysed in terms of the two-dimensional substructure built from Ag^+ and NO_3^- units only. These layers are linked by the DKP units acting as bridging ligands between pairs of Ag^+ ions in different layers. Within the ionic layer, the Ag^+ ion at $(x, \frac{1}{4},$

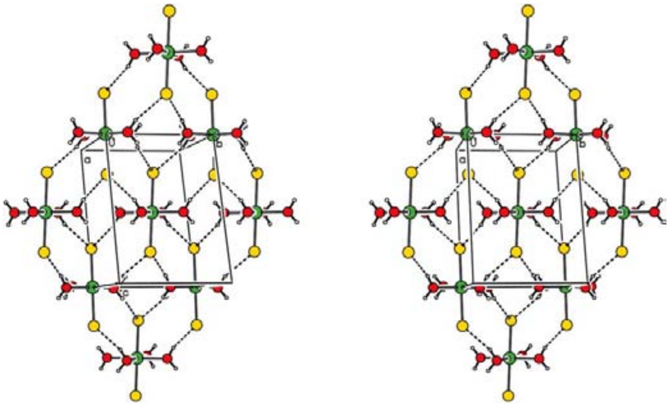

Figure 5
A stereoview of part of the crystal structure of (I), showing the formation of a hydrogen-bonded sheet built from $[CoCl_2(H_2O)_4]$ units only.

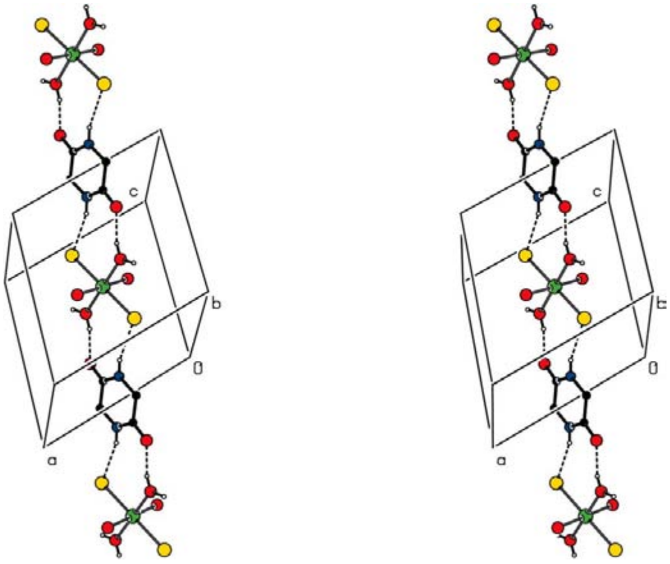

Figure 6
A stereoview of part of the crystal structure of (I), showing the formation of a hydrogen-bonded chain built from alternating $[CoCl_2(H_2O)_4]$ and piperazinedione units. For the sake of clarity, H atoms bonded to C atoms have been omitted.

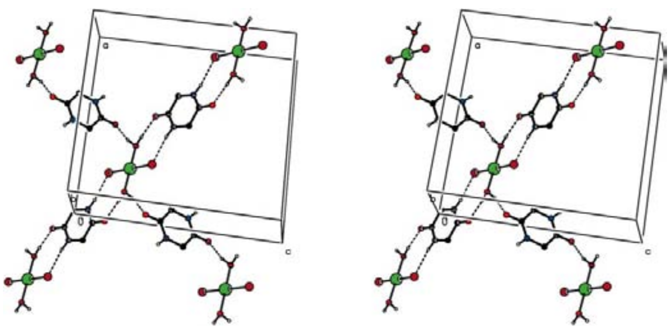

Figure 7
A stereoview of part of the crystal structure of (III), showing the formation of a hydrogen-bonded sheet parallel to $(2\bar{1}\bar{1})$ formed from $[CuCl_2(H_2O)_2]$ and piperazinedione units.

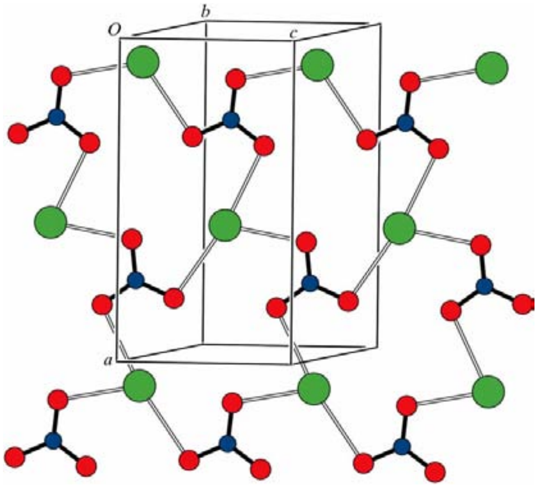

Figure 8
Part of the crystal structure of (IV), showing the formation of a layer parallel to (010) built from silver and nitrate ions only.

z) is coordinated by O atoms from three different nitrate ions, namely atom O1 in the anion at $(x, \frac{1}{4}, z)$, atom O2 in the anion at $(x, \frac{1}{4}, -1 + z)$ and atom O3 in the anion at $(-\frac{1}{2} + x, \frac{1}{4}, \frac{3}{2} - z)$ (Table 2). Propagation of these interactions then leads to the formation of an (010) sheet built from a single type of 12-membered ring (Fig. 8).

In addition to the three O atoms from within the ionic layer, the Ag^+ ion at $(x, \frac{1}{4}, z)$ is also coordinated by two O atoms from two different DKP ligands, namely those at (x, y, z) and $(x, \frac{1}{2} - y, z)$. The coordination polyhedron around the Ag^+ ion is best described as a distorted square pyramid (Fig. 4) in which two of the basal Ag—O distances are significantly longer than the other pair (Table 2). The two independent O—Ag—O angles in the basal plane have values close to 90°, while the angles subtended at Ag by the axial O and one of the basal O sites range from 74.94 (6) to 114.11 (6)°. The atoms of type O12 at (x, y, z) and $(x, \frac{1}{2} - y, z)$ form part of the DKP units centred at $(\frac{1}{2}, \frac{1}{2}, 0)$ and $(\frac{1}{2}, 0, 0)$, respectively, so that propagation of this Ag—O interaction by reflection and inversion generates a chain running parallel to the [010] direction (Fig. 9) which serves to link all of the (010) layers into a three-dimensional coordination polymer. The three-dimensional framework is reinforced by a single N—H···O hydrogen bond.

Thus, in each of compounds (I) and (II) there is a finite $[M\text{Cl}_2(\text{H}_2\text{O})_4]$ fragment, in compound (III) there is a one-dimensional coordination polymer, $[\text{CuCl}_2(\text{H}_2\text{O})_2]_n$, and in compound (IV) there is a three-dimensional coordination polymer, $[\text{Ag}(\text{NO}_3)(\text{DKP})]_n$. In (I) and (II), the formation of the three-dimensional framework is dependent upon the actions of multiple hydrogen bonds, while in compound (IV) the framework is formed by the three-dimensional coordination polymer, to the formation of which the single hydrogen bond is incidental. The three-dimensional framework in (III) is of a hybrid type, depending upon hydrogen bonds to link the one-dimensional coordination polymer chains. The organic piperazine-2,5-dione component may thus prove to be a very versatile building block for crystal structure design and construction.

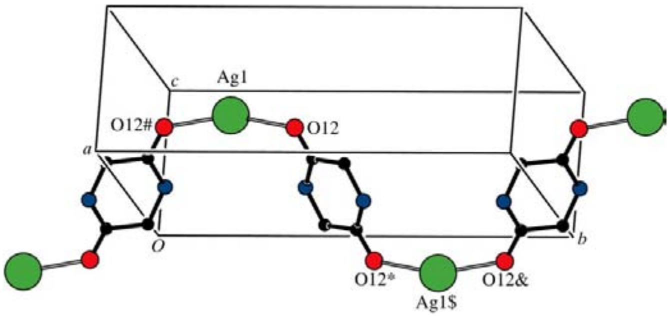

Figure 9
Part of the crystal structure of (IV), showing the formation of a chain parallel to [010] consisting of cations and DKP units only. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*), a hash (#), a dollar sign ($) or an ampersand (&) are at the symmetry positions $(1 - x, 1 - y, -z)$, $(x, \frac{1}{2} - y, z)$, $(x, \frac{3}{4}, z)$ and $(1 - x, \frac{1}{2} + y, z)$, respectively.

Experimental

Compounds (I)–(IV) were prepared by dissolving in water equimolar quantities of piperazine-2,5-dione and, respectively, cobalt(II) chloride hexahydrate, nickel(II) chloride tetrahydrate, copper(II) chloride dihydrate or silver nitrate. The solutions were set aside to crystallize, providing crystals of (I)–(IV) suitable for single-crystal X-ray diffraction analysis.

Compound (I)

Crystal data

$[\text{CoCl}_2(\text{H}_2\text{O})_4]\cdot\text{C}_4\text{H}_6\text{N}_2\text{O}_2$
$M_r = 316.00$
Monoclinic, $P2_1/n$
$a = 9.5326\ (4)$ Å
$b = 6.6602\ (2)$ Å
$c = 10.0167\ (4)$ Å
$\beta = 114.1660\ (17)°$
$V = 580.22\ (4)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 1.95$ mm^{-1}
$T = 120\ (2)$ K
0.40 × 0.20 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.509$, $T_{\max} = 0.876$
7527 measured reflections
1330 independent reflections
1189 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.053$
$S = 1.05$
1330 reflections
71 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}

Compound (II)

Crystal data

$[\text{NiCl}_2(\text{H}_2\text{O})_4]\cdot\text{C}_4\text{H}_6\text{N}_2\text{O}_2$
$M_r = 315.78$
Monoclinic, $P2_1/n$
$a = 9.4844\ (4)$ Å
$b = 6.6616\ (3)$ Å
$c = 9.9975\ (4)$ Å
$\beta = 114.6290\ (18)°$
$V = 574.19\ (4)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 2.17$ mm^{-1}
$T = 120\ (2)$ K
0.40 × 0.30 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.468$, $T_{\max} = 0.649$
6275 measured reflections
1315 independent reflections
1218 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$
$wR(F^2) = 0.046$
$S = 1.06$
1315 reflections
71 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

Compound (III)

Crystal data

$[\text{CuCl}_2(\text{H}_2\text{O})_2]\cdot\text{C}_4\text{H}_6\text{N}_2\text{O}_2$
$M_r = 284.58$
Monoclinic, $I2/a$
$a = 15.224\ (3)$ Å
$b = 3.9694\ (8)$ Å
$c = 15.444\ (4)$ Å
$\beta = 94.73\ (3)°$
$V = 930.1\ (4)$ Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 2.91$ mm^{-1}
$T = 120\ (2)$ K
0.16 × 0.04 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.654$, $T_{\max} = 0.918$
4485 measured reflections
1059 independent reflections
787 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.0$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.164$
$S = 1.09$
1059 reflections
62 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 1.58$ e Å^{-3}
$\Delta\rho_{\min} = -1.45$ e Å^{-3}

Compound (IV)

Crystal data

$[Ag(NO_3)(C_4H_6N_2O_2)]$
$M_r = 567.98$
Orthorhombic, *Pnma*
$a = 9.62760$ (10) Å
$b = 14.7180$ (2) Å
$c = 5.2468$ (4) Å
$V = 743.47$ (6) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 2.71$ mm^{-1}
$T = 120$ (2) K
0.42 × 0.22 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan *SORTAV* (Blessing, 1995, 1997)
$T_{\min} = 0.389$, $T_{\max} = 0.722$
7016 measured reflections
888 independent reflections
826 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$
$wR(F^2) = 0.044$
$S = 1.08$
888 reflections
68 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}

Table 1
Selected bond distances (Å) for compounds (I)–(III).

	(I), *M* = Co	(II), *M* = Ni	(III), *M* = Cu
M—O1	2.0361 (9)	2.0287 (9)	1.942 (4)
M—O2	2.1113 (9)	2.0869 (9)	
M—Cl	2.4562 (3)	2.3971 (3)	2.2502 (15)
M—Cl^{i}			3.1640 (18)

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

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

Ag1—O1	2.4907 (18)	Ag1—O3^{ii}	2.6522 (18)
Ag1—O2^{i}	2.6653 (17)	Ag1—O12	2.3627 (13)
O1—Ag1—O2^{i}	114.11 (6)	O2^{i}—Ag1—O3^{ii}	170.95 (6)
O1—Ag1—O3^{ii}	74.94 (6)	O2^{i}—Ag1—O12	82.36 (3)
O1—Ag1—O12	100.67 (3)	O3^{ii}—Ag1—O12	96.24 (3)
O12—Ag1—O12^{iii}	157.45 (7)		

Symmetry codes: (i) $x, y, z-1$; (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, -y+\frac{1}{2}, z$.

For compounds (I), (II) and (IV), the space groups were uniquely assigned from the systematic absences as $P2_1/n$, $P2_1/n$ and *Pnma*, respectively. For compound (III), the systematic absences permitted *Cc* and *C*2/*c* as possible space groups; *C*2/*c* was selected and confirmed by the refinement. Because of the very large value of β in *C*2/*c* [132.180 (3)°], the alternative setting *I*2/*a* was adopted prior to the final refinement. Compound (III) was refined as a nonmerohedral twin, resulting in twin fractions of 0.469 and 0.531.

For each of (I)–(IV), all H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.99 Å, N—H = 0.88 Å and O—H = 0.84 or 0.90 Å, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(C,N) or $1.5U_{\rm eq}$(O).

Table 3
Hydrogen-bond parameters (Å, °) for compounds (I)–(IV).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
(I)				
O1—H1*A*···Cl^{i}	0.84	2.35	3.1426 (10)	158
O1—H1*B*···O12	0.84	1.86	2.6901 (14)	167
O2—H2*A*···Cl^{ii}	0.84	2.42	3.2302 (10)	163
O2—H2*B*···O12^{iii}	0.84	1.96	2.7834 (14)	168
N11—H11···Cl1	0.88	2.38	3.2326 (12)	164
(II)				
O1—H1*A*···Cl^{i}	0.84	2.37	3.1508 (10)	155
O1—H1*B*···O12	0.84	1.87	2.6941 (13)	167
O2—H2*A*···Cl^{ii}	0.84	2.45	3.2596 (9)	163
O2—H2*B*···O12^{iii}	0.84	1.96	2.7906 (13)	168
N11—H11···Cl1	0.88	2.39	3.2419 (11)	163
(III)				
N11—H11···Cl1	0.88	2.31	3.174 (5)	168
O1—H1*A*···O2	0.90	1.87	2.736 (7)	162
O1—H1*B*···O12^{iv}	0.90	1.85	2.735 (7)	167
(IV)				
N11—H11···O2^{v}	0.88	2.13	3.0004 (18)	173

Symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$; (iii) $x, 1+y, z$; (iv) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (v) $-\frac{1}{2}+x, \frac{1}{2}-y, \frac{3}{2}-z$.

For all compounds, data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3165). Services for accessing these data are described at the back of the journal.

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