1157 measured reflections
1001 independent reflections

2 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.074$
$S = 1.223$
1002 reflections
53 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 1.5167P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.669$ e Å^{-3}
$\Delta\rho_{min} = -0.597$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.01410 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

$U_{eq} = (1/3)\sum_i\sum_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Pd	0	0	0	0.0204 (2)
Br1	0.03489 (8)	0.20516 (8)	0.23012 (8)	0.0269 (2)
Br2	0.26806 (7)	−0.04122 (9)	0.05017 (9)	0.0315 (2)
N1	0.2267 (7)	0.5322 (7)	0.0661 (8)	0.036 (2)
C2	0.3627 (8)	0.4393 (10)	0.1389 (10)	0.037 (2)
C3	0.4611 (8)	0.4192 (8)	0.0147 (9)	0.029 (2)

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

Pd—Br1	2.4422 (9)	N1—C2	1.467 (10)
Pd—Br2	2.4529 (9)	C2—C3	1.513 (11)
Pd···Br1^{i}	3.2712 (10)	C3—C3^{ii}	1.520 (10)
Br1—Pd—Br2^{iii}	89.49 (3)	N1—C2—C3	112.4 (6)
Br1—Pd—Br2	90.51 (3)	C2—C3—C3^{ii}	113.3 (6)
N1—C2—C3—C3^{ii}	73.4 (8)	C2—C3—C3^{ii}—C2^{ii}	180.0 (6)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Br1^{i}	0.89	2.71	3.513 (7)	150
N1—H1B···Br1^{ii}	0.89	2.57	3.455 (6)	175
N1—H1C···Br2^{iii}	0.89	2.59	3.422 (6)	156

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

Unit-cell parameters were first determined from automatic indexing with *DICVOL*91 (Boultif & Louër, 1991) of a powder X-ray diffraction pattern recorded with a Siemens D-500 diffractometer. The cell parameters thus obtained have been confirmed by the calculation from single-crystal X-ray diffraction measurements of 25 reflections. A half sphere of reflections was recorded and the intensity data were reduced using a local program giving a *hkl* file based on F^2. The structure was solved by the Patterson method and from subsequent difference Fourier map calculations. All non-H atoms were refined by full-matrix least squares with anisotropic displacement parameters. H atoms were idealized using the standard procedure of *SHELXL*93 (Sheldrick, 1993). H-atom isotropic displacement parameters were set at $1.5U_{eq}$ of the attached C or N atom.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *NONIUS* (unpublished). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ORTEX* (McArdle, 1993). Software used to prepare material for publication: *PLATON* (Spek, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1029). Services for accessing these data are described at the back of the journal.

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catena-Poly[diamminecopper(II)-μ-acetato-*O*:*O*'] Tetrafluoroborate

LEROY CRONIN,[a] MIRIAM HARPER,[a] PAUL H. WALTON[a] AND CHRIS LINDSAY[b]

[a]*Department of Chemistry, University of York, Heslington, York YO10 5DD, England, and* [b]*ICI Polymer Research Centre, Wilton, Middlesbrough, Cleveland TS90 8JE, England. E-mail: phw2@york.ac.uk*

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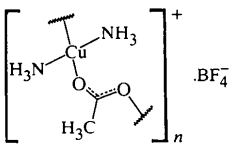
Abstract

The crystal of the title compound, $[Cu(C_2H_3O_2)(NH_3)_2]BF_4$, contains infinite chains in which the acetate groups bridge pairs of Cu^{II} ions using both *syn* and *anti* coordination modes.

Comment

As part of a general study of copper(II) salts in polymerization reactions, we have synthesized a new copper(II) complex in which bridging acetate groups form an infinite chain. Ferrari *et al.* (1972) have reported a similar chain structure with bromide acting as counterion. The geometry around each Cu atom in the title compound, (I), is square planar, with the ammine ligands *trans* to each other. The coordination sphere is completed by the O atoms of two symmetry-related acetate groups.



(I)

Discrete copper complexes containing *trans*-ammine and monodentate acetate groups have been described by Bukowska-Strzyzewska (1963) and Simonov *et al.* (1963).

In the title compound, (I) (Fig. 1), there is an axial interaction between the F1 atom of the tetrafluoroborate anion above the copper(II) ion [F1···Cu1 = 2.495 (3) Å], which thereby attains overall square-pyramidal coordination.

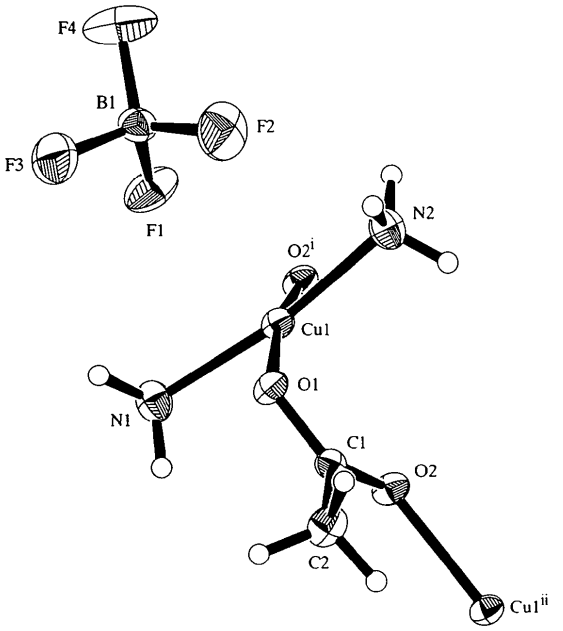


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids.

Experimental

A solution of 4-biphenyl cyanate in ethanol was added to a solution of copper(II) bis(tetrafluoroborate) in ethanol. An immediate colour change from pale blue to green was observed. On addition of a solution of ammonium acetate in ethanol, the solution turned deep green. After being left overnight at ambient temperature, the solution yielded royal-blue crystals.

Crystal data

$[Cu(C_2H_3O_2)(NH_3)_2]BF_4$	Mo $K\alpha$ radiation
M_r = 243.46	λ = 0.71069 Å
Monoclinic	Cell parameters from 20 reflections
$P2_1/c$	θ = 6.98–8.87°
a = 8.0862 (19) Å	μ = 2.701 mm^{-1}
b = 12.576 (4) Å	T = 293 (2) K
c = 8.186 (2) Å	Plate
β = 98.441 (19)°	0.6 × 0.3 × 0.1 mm
V = 823.4 (4) $Å^3$	Blue
Z = 4	
D_x = 1.976 Mg m^{-3}	
D_m not measured	

Data collection

Rigaku AFC-6*S* diffractometer	1271 reflections with $I > 2\sigma(I)$
ω–2θ scans	R_{int} = 0.030
Absorption correction:	θ_{max} = 25.10°
ψ scans (North *et al.*, 1968)	h = 0 → 9
T_{min} = 0.400, T_{max} = 0.763	k = 0 → 14
1456 measured reflections	l = −9 → 9
1451 independent reflections	3 standard reflections
	every 150 reflections
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = 0.001
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$\Delta\rho_{max}$ = 0.63 e $Å^{-3}$
$wR(F^2)$ = 0.106	$\Delta\rho_{min}$ = −0.39 e $Å^{-3}$
S = 1.053	Extinction correction: none
1451 reflections	Scattering factors from *International Tables for Crystallography* (Vol. C)
111 parameters	
H atoms riding	
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 1.3063P]$ where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Selected geometric parameters (Å, °)*

Cu1—N2	1.977 (4)	Cu1—O1	1.987 (3)
Cu1—N1	1.980 (4)	Cu1—O2^i	1.991 (3)
N2—Cu1—N1	171.63 (16)	N1—Cu1—O2^i	90.39 (13)
N2—Cu1—O1	90.88 (14)	O1—Cu1—O2^i	169.37 (10)
N1—Cu1—O1	89.05 (14)	C1—O1—Cu1	112.3 (2)
N2—Cu1—O2^i	91.21 (13)	C1—O2—Cu1^{ii}	125.8 (2)

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

Data collection: *TEXSAN* (Molecular Structure Corporation, 1994). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SHELXS*96 (Sheldrick, 1996*a*). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996*b*). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *SHELXL*96.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1344). Services for accessing these data are described at the back of the journal.

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Potassium Tartronate and Ammonium Tartronate

JUN-ICHIRO TAKA AND SETSUO KASHINO

Department of Chemistry, Faculty of Science, Okayama University, Tsushima, Okayama 700, Japan. E-mail: kashinos@cc.okayama-u.ac.jp

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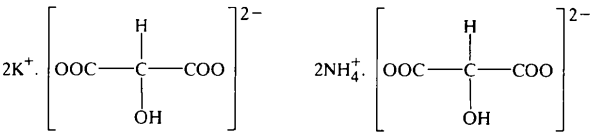
Abstract

Potassium tartronate (dipotassium hydroxypropanedioate, $2K^+.C_3H_2O_5^{2-}$), (I), crystallizes in the monoclinic space group $P2_1/n$, with $Z = 4$, and ammonium tartronate (diammonium hydroxypropanedioate, $2NH_4^+.C_3H_2O_5^{2-}$), (II), crystallizes in orthorhombic space group $Pna2_1$, with $Z = 4$. There are two symmetry-independent cations in both (I) and (II). The coordination number of the K^+ ion in (I) is seven, and the number of hydrogen bonds around the NH_4^+ ion in (II) is four. This difference arises from the fact that the hydrogen bonding governs the number of O atoms around NH_4^+ in (II), in contrast to the electrostatic interactions in (I).

Comment

Potassium hydrogen tartronate, (III) (Kroon & Kanters, 1982; Kanters & Kroon, 1976; Roelofsen *et al.*, 1978), and ammonium hydrogen tartronate, (IV) (Taka *et al.*, 1998), crystallize in the same space group, $P2_1/c$, and the crystal structures of (III) and (IV) are similar to each other. The differences in these structures are interpreted in terms of the nature of the interactions around the cations (Taka *et al.*, 1998), *i.e.* electrostatic for K^+ and hydrogen bonding for NH_4^+. The K^+ ion in (III) is coordinated by eight O atoms, while the NH_4^+ ion in (IV) forms six N—H···O hydrogen bonds, including two bifurcated hydrogen bonds. The present study was undertaken in order to obtain information on the interactions between the cation and the O atoms of the anion for the title salts of tartronic acid, *i.e.* potassium tartronate, (I), and ammonium tartronate, (II).



(I) (II)

The asymmetric unit is composed of two cations and one tartronate anion in the crystals of both (I) and (II) (Fig. 1). The hydroxy group in (I) forms an intramolecular hydrogen bond [O1—H1O 0.70 (3), H1O···O4 2.07 (3), O1···O4 2.586 (3) Å and O1—H1O···O4 131 (4)°], while that in (II) forms an intermolecular hydrogen bond [O1—H1O 0.80 (3), H1O···O5^{iv} 2.17 (3), O1···O5^{iv} 2.921 (2) Å and O1—H1O···O5^{iv}

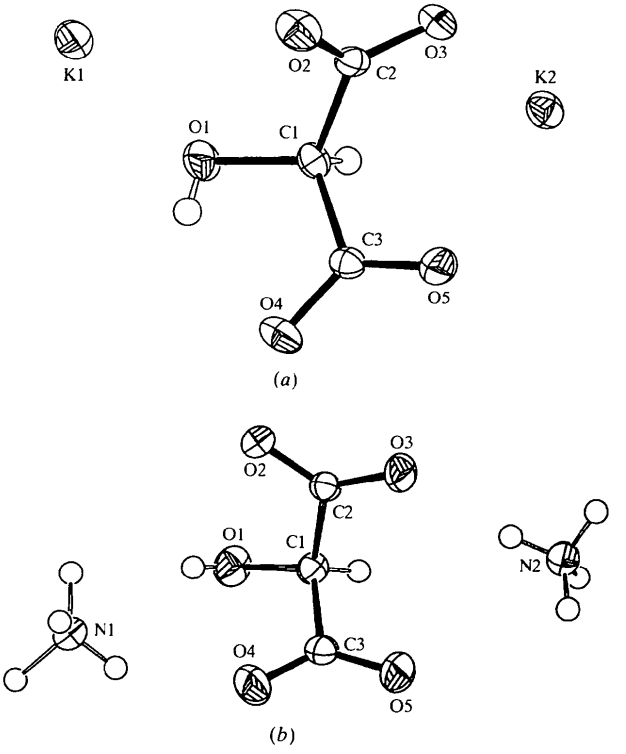


Fig. 1. *ORTEP*II (Johnson, 1976) representations of (*a*) potassium tartronate, (I), and (*b*) ammonium tartronate, (II), with the atomic numbering of the asymmetric units. Displacement ellipsoids are shown at the 50% probability level for non-H atoms and H atoms are drawn as spheres with $B_{iso} = 1.0$ Å^2.