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

$$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
In	0.0000	0.0000	0.1402 (1)	2.12 (4)
Nb(1)	0.0000	0.5000	0.0796 (1)	0.71 (2)
Nb(2)	0.0000	0.5000	0.2500	0.63 (3)
Se(1)	0.1259 (1)	0.3151 (1)	0.16147 (7)	1.00 (2)
Se(2)	0.2249 (2)	0.4836 (2)	0.0000	0.62 (3)
Se(3)	−0.1075 (2)	0.3003 (2)	0.0000	1.30 (4)
Se(4)	−0.2148 (1)	0.4529 (1)	0.17180 (8)	1.11 (2)

Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

Nb($1^{\rm i}$)—Nb($1^{\rm ii}$)	3.037 (3)	Nb(1)—Nb(2)	3.248 (2)
Nb(1)—Se(1)	2.627 (2)	Nb(1)—Se(2)	2.617 (2)
Nb(1)—Se(3)	2.627 (2)	Nb(1)—Se(4)	2.721 (2)
Nb(2)—Se(1)	2.705 (1)	Nb(2)—Se(4)	2.558 (1)
Se(1)—Se($4^{\rm iii}$)	2.356 (2)	Se(2)—Se($3^{\rm iii}$)	2.325 (2)
In—Se(1)	3.233 (1)		
Nb(1)—Nb(2)—Nb($1^{\rm i}$)	180.00	Nb(2)—Nb($1^{\rm i}$)—Nb($1^{\rm ii}$)	180.00
Se(1)—Nb(1)—Se($1^{\rm iii}$)	107.15 (8)	Se(1)—Nb(1)—Se(2)	86.42 (4)
Se(1)—Nb(1)—Se($2^{\rm iii}$)	138.74 (4)	Se(1)—Nb(1)—Se(3)	92.32 (4)
Se(1)—Nb(1)—Se($3^{\rm i(1)ii}$)	130.24 (4)	Se(1)—Nb(1)—Se(4)	81.12 (6)
Se(1)—Nb(1)—Se($4^{\rm iii}$)	52.23 (4)	Se(2)—Nb(1)—Se($2^{\rm iii}$)	109.05 (8)
Se(2)—Nb(1)—Se(3)	86.34 (6)	Se(2)—Nb(1)—Se($3^{\rm iii}$)	52.62 (5)
Se(2)—Nb(1)—Se(4)	116.15 (5)	Se(2)—Nb(1)—Se($4^{\rm iii}$)	77.21 (4)
Se(3)—Nb(1)—Se($3^{\rm iii}$)	109.37 (7)	Se(3)—Nb(1)—Se(4)	88.13 (4)
Se(3)—Nb(1)—Se($4^{\rm iii}$)	141.18 (4)	Se(4)—Nb(1)—Se($4^{\rm iii}$)	99.57 (7)
Se(1)—Nb(2)—Se($1^{\rm iii}$)	102.78 (4)	Se(1)—Nb2—Se($1^{\rm i}$)	79.70 (4)
Se(1)—Nb(2)—Se($1^{\rm iv}$)	163.26 (3)	Se(1)—Nb(2)—Se(4)	82.70 (4)
Se(1)—Nb(2)—Se($4^{\rm iii}$)	53.09 (3)	Se(1)—Nb(2)—Se($4^{\rm i}$)	143.12 (3)
Se(1)—Nb(2)—Se($4^{\rm iv}$)	85.84 (4)	Se(4)—Nb(2)—Se($4^{\rm iii}$)	108.68 (4)
Se(4)—Nb(2)—Se($4^{\rm i}$)	128.02 (4)	Se(4)—Nb(2)—Se($4^{\rm iv}$)	93.66 (4)

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

The crystals of $\mathrm{InNb_3(Se_2)_6}$ show metallic lustre and are stable in air. A microanalysis of single crystals was made with a Jeol scanning electron microscope. The experimental values in molar ratio are 1.1:2.7:11.7 (In:Nb:Se). The density was measured by the micropycnometer method in toluene.

The data were collected using scan rates of 5–16° min^{-1} and a scan width of $(1.5+0.35\tan\theta)°$. Refinement was by full-matrix least-squares methods. Programs used to solve structure: *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Other programs used: *SDP* (Frenz, 1978). Calculations performed on a VAX 785 computer.

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Lists of structure factors, anisotropic thermal parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55752 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1022]

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Structure of 1-Benzhydryl-4-(2-benzoylethyl)piperazinium Tetrachlorocuprate(II) Hydrate

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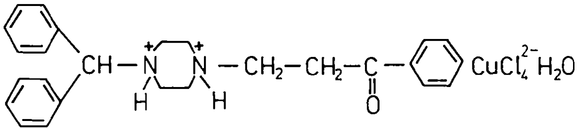
Abstract

The structure consists of bulky $\mathrm{Ph_2CH(C_4H_{10}N_2)CH_2CH_2(CO)Ph^{2+}}$ cations extended along the c axis, isolated $[\mathrm{CuCl_4}]^{2-}$ anions and a disordered water molecule scattered in a channel running parallel to the a axis through the centre of the unit cell. Both the tertiary-amine-group

H atoms participate in hydrogen bonding with the water molecule and with two Cl atoms. A peculiarity of this structure is the presence of a short intermolecular O···C contact of 3.036(6) Å.

Comment

Various substituted piperazines exhibit antitumour activity (Larionov, 1962; Huang, 1982). A new copper complex (1) of 1-benzhydryl-4-(2-benzoylethyl)piperazine (bhbep) was recently prepared and characterized by Tcholakova, Parvanova, Pankova, Natova & Vasileva (1990). The present study confirms both its composition and structure. The powder pattern has been evaluated and submitted to the PDF (powder diffraction file) database (Maciček, 1992).



(1)

The piperazine fragment in the bhbepH$_2^{2+}$ cation is diprotonated and has a chair conformation (Boeyens, 1978) characterized by the puckering parameters Q = 0.548 Å, Θ = 0.894 and φ = 202.8° (Cremer & Pople, 1975). The C—N distances are consistent with the observed mean for C—N bonds in Me_3NH^+ ions [1.502(15) Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987] although the average of the two *exo* C—N distances [1.511(4) Å] differs considerably from that for the four *endo* ones [1.493(3) Å]. The N1—C3—C2—C1(=O)—C1*a* linkage is planar to within 0.15 Å and has an unfolded conformation of usual geometry (*cf.* Allen *et al.*, 1987). As expected, the C1—C_{phenyl} distance of 1.488(7) Å is shorter than the average of the two C8—C_{phenyl} distances [1.520(5) Å]. Phenyl rings *b* and *c* form a dihedral angle of 68.1(3)° and ring *a* is tilted relative to the $NC_2C(O)C$ plane by 23.4(8)°.

The tetrachlorocuprate anion has a flattened tetrahedral geometry. The Cu—Cl distances range from 2.162(2) (Cl4) to 2.276(2) Å (Cl1). Cl1 participates in a rather strong H bond with H2 [N2—H2$_{calc}$ 0.95, H2···Cl1 2.196, N2···Cl1 3.102(4) Å, N2—H2···Cl1 159.1°]. The average Cu—Cl distance of 2.238(51) Å resembles the conventional mean bond value [2.248(32) Å; Orpen, Brammer, Allen, Kennard, Watson & Taylor, 1989]. The Cl—Cu—Cl angles are within the range 99.74(8)-118.85(7)°.

The keto O atom forms an unusually short intermolecular contact [O···C6 $(1-x, -y, 1-z)$ 3.036(6) Å] and two longer ones [O···C7 $(1-x, y, 1-z)$ 3.209(6) Å, O···C3*c* $(-x, -y, 1-z)$ 3.392(8) Å]. There are no intermolecular C···C distances shorter than 3.5 Å. The plausibility of a correct assignment of the electron-density peaks in the channel down the *a* axis at $(x,\frac{1}{2},\frac{1}{2})$ to a disordered water molecule is supported by close contacts of the O*w* atoms with H-bond acceptors [Cl2, Cl3; O···Cl 2.58-3.14 Å] and an H-bond donor, N1 [O···N 2.66-2.93 Å].

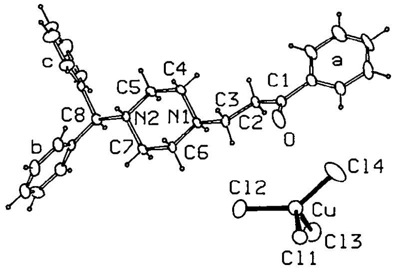


Fig. 1. The bhbepH$_2^{2+}$ and CuCl$_4^{2-}$ ions with the atom-numbering scheme. Thermal ellipsoids are drawn at the 30% probability level; H atoms are represented by spheres of arbitrary size.

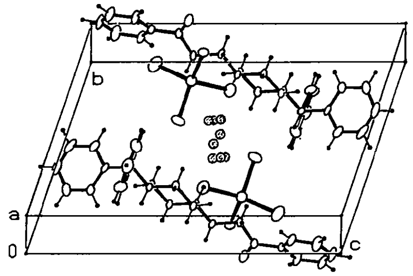


Fig. 2. Projection of the unit-cell content onto the *bc* plane. The disordered water molecules are dotted.

Experimental

Crystal data

$C_{26}H_{30}N_2O^{2+}.CuCl_4^{2-}.H_2O$
M_r = 609.90
Triclinic
$P\bar{1}$
a = 8.880 (1) Å
b = 10.594 (1) Å
c = 16.387 (2) Å
α = 71.20 (1)°
β = 89.85 (1)°
γ = 77.74 (1)°
V = 1422.5 (3) Å^3
Z = 2

D_x = 1.424 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 22 reflections
θ = 17.96-19.62°
μ = 1.16 mm^{-1}
T = 292 K
Approximately prismatic
0.2 × 0.2 × 0.13 mm
Orange

Data collection

Enraf-Nonius CAD-4 diffractometer
Continuous $\omega/2\theta$ scan profiles
Absorption correction: none
5369 measured reflections
5013 independent reflections
3475 observed reflections [$I>\sigma(I)$]

R_{int} = 0.019
θ_{max} = 25°
$h = 0 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity variation: 0.7%

Refinement

Refinement on *F*
Final $R = 0.098$
$wR = 0.076$
$S = 3.089$
3475 reflections
323 parameters
H atoms positioned geometrically and not refined (C—H, N—H = 0.95 Å)
$w = 1/[k_i\sigma^2(F)+(0.001F)^2]$ where $k_i = \{10.0, \sigma(I)/(0.01, F^2)_{\max}\}_{\max}$ for $I<3\sigma(I)$, else $k_i = 1$
$(\Delta/\sigma)_{\max} = 0.014$
$\Delta\rho_{\max} = 0.77$ e Å^{-3}
$\Delta\rho_{\min} = -1.22$ e Å^{-3}
Atomic scattering factors from *SDP*/*PDP* (Enraf-Nonius, 1985)

Final *R* factors are high as a result of poor crystal quality, disorder and the inclusion of data down to $I/\sigma(I) = 1$.

Data collection: CAD-4 (Enraf–Nonius, 1988). Cell refinement: reduced cell by *BLAF* (Macíček & Yordanov, 1992). Data reduction: *SDP*/*PDP* and *BAYES* (Blessing, 1987). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP*/*PDP*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *KAPPA* (Macíček, 1992; unpublished).

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

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cu	0.2105 (2)	−0.1413 (1)	0.34609 (9)	0.0580 (4)
Cl1	0.0254 (3)	0.0435 (2)	0.3466 (2)	0.0503 (8)
Cl2	0.3128 (4)	−0.2102 (3)	0.4830 (2)	0.080 (1)
Cl3	0.1373 (4)	−0.3320 (3)	0.3434 (2)	0.080 (1)
Cl4	0.2798 (5)	−0.0678 (4)	0.2157 (2)	0.113 (2)
O	0.4589 (8)	−0.1063 (7)	0.7130 (4)	0.065 (3)
N1	0.2561 (7)	0.2131 (6)	0.4923 (4)	0.026 (2)
N2	0.0867 (7)	0.3307 (6)	0.3218 (4)	0.025 (2)
C1	0.363 (1)	−0.0193 (9)	0.7274 (5)	0.035 (3)
C2	0.288 (1)	0.1086 (8)	0.6552 (5)	0.030 (3)
C3	0.328 (1)	0.0913 (8)	0.5698 (5)	0.031 (3)
C4	0.0850 (9)	0.2346 (8)	0.4816 (5)	0.030 (3)
C5	0.0142 (9)	0.3507 (8)	0.4005 (5)	0.032 (3)
C6	0.3274 (9)	0.1948 (8)	0.4130 (5)	0.028 (3)
C7	0.2581 (9)	0.3102 (8)	0.3334 (5)	0.029 (3)
C8	0.0202 (9)	0.4490 (8)	0.2412 (5)	0.028 (3)
C1*a*	0.321 (1)	−0.0309 (8)	0.8171 (5)	0.035 (3)
C2*a*	0.182 (1)	0.036 (1)	0.8355 (6)	0.055 (4)
C3*a*	0.152 (1)	0.020 (1)	0.9197 (6)	0.081 (5)
C4*a*	0.254 (1)	−0.057 (1)	0.9848 (6)	0.078 (5)
C5*a*	0.393 (1)	−0.125 (1)	0.9673 (6)	0.076 (5)
C6*a*	0.425 (1)	−0.115 (1)	0.8844 (6)	0.058 (4)
C1*b*	0.0966 (9)	0.4295 (9)	0.1613 (5)	0.032 (3)
C2*b*	0.108 (1)	0.3121 (9)	0.1399 (5)	0.043 (3)
C3*b*	0.173 (1)	0.300 (1)	0.0652 (6)	0.056 (4)
C4*b*	0.226 (1)	0.406 (1)	0.0135 (6)	0.067 (4)
C5*b*	0.215 (1)	0.521 (1)	0.0317 (6)	0.068 (4)
C6*b*	0.151 (1)	0.533 (1)	0.1065 (6)	0.049 (3)
C1*c*	−0.1546 (9)	0.4793 (8)	0.2323 (5)	0.031 (3)
C2*c*	−0.239 (1)	0.3909 (9)	0.2240 (6)	0.045 (3)
C3*c*	−0.395 (1)	0.427 (1)	0.2107 (7)	0.060 (4)
C4*c*	−0.469 (1)	0.550 (1)	0.2079 (6)	0.067 (4)
C5*c*	−0.389 (1)	0.642 (1)	0.2161 (6)	0.067 (4)
C6*c*	−0.232 (1)	0.608 (1)	0.2277 (6)	0.051 (3)
O*w*1*	0.265 (2)	0.449 (2)	0.523 (1)	0.038
O*w*2*	0.359 (2)	0.426 (2)	0.502 (1)	0.038
O*w*3*	0.338 (2)	0.436 (2)	0.539 (1)	0.038
O*w*4*	0.295 (2)	0.517 (1)	0.4862 (9)	0.038

*Disordered, partially occupied water oxygen sites with occupancies O*w*1 0.34(1), O*w*2 0.27(1), O*w*3 0.33(1) and O*w*4 0.39(1). $U_{\rm iso}$ was arbitrarily set at 0.038 Å^2 for these sites.

Table 2. *Interatomic distances* (Å) *and angles* (°)

Cu—Cl1	2.276 (2)	N2—C7	1.495 (6)
Cu—Cl2	2.251 (2)	N2—C8	1.512 (6)
Cu—Cl3	2.263 (2)	C1—C2	1.505 (7)
Cu—Cl4	2.162 (2)	C1—C1*a*	1.488 (7)
O—C1	1.199 (6)	C2—C3	1.502 (6)
N1—C3	1.511 (6)	C4—C5	1.515 (6)
N1—C4	1.489 (6)	C6—C7	1.494 (6)
N1—C6	1.493 (5)	C8—C1*b*	1.526 (7)
N2—C5	1.495 (6)	C8—C1*c*	1.514 (7)
Cl1—Cu—Cl2	99.74 (8)	O—C1—C2	120.5 (5)
Cl1—Cu—Cl3	118.85 (7)	O—C1—C1*a*	121.2 (5)
Cl1—Cu—Cl4	100.01 (8)	C2—C1—C1*a*	118.2 (5)
Cl2—Cu—Cl3	100.86 (8)	C1—C2—C3	109.6 (4)
Cl2—Cu—Cl4	139.2 (1)	N1—C3—C2	114.2 (4)
Cl3—Cu—Cl4	100.2 (1)	N1—C4—C5	113.0 (4)
C3—N1—C4	112.9 (4)	N2—C5—C4	111.3 (4)
C3—N1—C6	109.0 (4)	N1—C6—C7	111.8 (4)
C4—N1—C6	110.1 (4)	N2—C7—C6	112.8 (4)
C5—N2—C7	109.8 (4)	N2—C8—C1*b*	111.2 (4)
C5—N2—C8	111.4 (4)	N2—C8—C1*c*	112.3 (4)
C7—N2—C8	111.4 (4)	C1*b*—C8—C1*c*	113.8 (4)
C4—N1—C3—C2	−68.0 (9)	C7—N2—C8—C1*c*	−175.1 (7)
C6—N1—C3—C2	169.2 (7)	C1*a*—C1—C2—C3	−171.9 (8)
C8—N2—C7—C6	179.4 (7)	C2—C1—C1*a*—C2*a*	27 (1)
C5—N2—C8—C1*b*	178.9 (7)	C1—C2—C3—N1	179.0 (7)
C5—N2—C8—C1*c*	−52.2 (9)	N2—C8—C1*b*—C2*b*	52 (1)
C7—N2—C8—C1*b*	56.0 (9)	N2—C8—C1*c*—C2*c*	−62 (1)

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55831 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1015]

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