Crystal Structure of Bis(tetrapropylammonium)

Hexa-μ-bromo-*tetrahedro*-tetracuprate(I)

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The crystal structure of the title compound has been determined from single-crystal X-ray diffractometer data. $[N(C_3H_7)_4]_2[Cu_4Br_6]$ crystallizes in space group $P4_2/n$ with $a=15.708(5)$, $c=15.709(8)$ Å and $Z=4$. Full-matrix least-squares refinement of 167 structural parameters gave $R=0.062$ for 1168 observed independent reflections. The $[Cu_4Br_6]^{2-}$ anion is an aggregate composed of an octahedron of bromide ligands containing a tetrahedron of trigonal-planar coordinated copper(I) atoms. The orientation of the copper(I) tetrahedron is disordered, there being two equivalent copper(I) tetrahedra related by the centre of symmetry of the bromine octahedron. The copper(I) atoms have therefore been assigned occupancy 0.5 in four general eightfold sites. Cu–Br distances range from 2.373(5)–2.422(5) Å and Cu⋯Cu from 2.718(7)–2.750(7) Å. Two of the three crystallographically independent cations have $\bar{4}$ symmetry while the third has a disordered orientation with respect to a twofold axis.

In tetrabutylammonium dibromocuprate(I) the anion is a linear monomer,[1] whereas in the tetraethylammonium compound the anion is a centrosymmetric $[Cu_2Br_4]^{2-}$ dimer containing trigonal-planar coordinated copper(I).[2] As part of an investigation concerning the geometry assumed by halocuprate(I) ions in the solid state as a function of the size and effective positive charge on the cation, attempts were made to prepare tetrapropylammonium dibromocuprate(I). Crystals of bis(tetrapropylammonium) hexa-μ-bromo-*tetrahedro*-tetracuprate(I) were, however, obtained, the structure of which was also considered relevant to the investigation.

The bromocuprate(I) complex $[Cu_2Br_3]^-$ has been found as infinite double chains of edge-sharing Cu(I)–Br tetrahedra in $[C_6H_5N_2]$-$[Cu_2Br_3]^3$ and in $[(CH_3)_2N_2CHN_2(CH_3)_2]$-$[Cu_2Br_3]$,[4] but, to our knowledge, not previously as a discrete $[Cu_4Br_6]^{2-}$ aggregate. An analogous $[Cu_4I_6]^{2-}$ ion has been reported for methyltriphenylphosphonium hexaiodotetracuprate(I).[5]

EXPERIMENTAL

Bis(tetrapropylammonium) hexa-μ-bromo-*tetrahedro*-tetracuprate(I) was obtained in an attempt to prepare tetrapropylammonium dibromocuprate(I). Copper(I) bromide and tetrapropylammonium bromide (molar ratio 1:1) were dissolved in ethanol. Colourless prisms of $[N(C_3H_7)_4]_2[Cu_4Br_6]$ were deposited from the concentrated solution after a few days.

Rotation and Weissenberg photographs for crystals mounted along a and c showed $[N(C_3H_7)_4]_2[Cu_4Br_6]$ to be tetragonal, with Laue symmetry $4/m$, space group No. 86,[6a] $P4_2/n$. $[N(C_3H_7)_4]_2[Cu_4Br_6]$, $M_r=1106.3$, has $a=15.708(5)$ Å, $c=15.709(8)$ Å, $Z=4$, $D_c=1.91$ g cm^{-3} and $\mu(MoK\alpha)=8.88$ mm^{-1}. Diffracted intensities from a crystal, 0.23x0.14×0.10 mm, were measured at approximately 290 K for $2\theta \leqslant 50°$, on a Syntex $P2_1$ diffractometer, using graphite-monochromated MoKα radiation and the ω-2θ scan mode with a variable 2θ scan rate of 3.0–25.0 ° min.$^{-1}$ A 96-step profile was recorded for each reflection and the Lehmann and Larsen profile-analysis method[7] was used to calculate the intensities.[8] Of the 3417 independent reflections thus measured, 1169 had $I>3.0$ $\sigma(I)$ and were used to solve the structure. Correction was

made for Lorentz and polarisation effects but not for absorption. The unit-cell parameters were determined by least squares from diffractometer setting angles for 15 reflections.

STRUCTURE DETERMINATION AND REFINEMENT

The positions of the copper and bromine atoms were determined by direct methods (MULTAN 80).[9] It was apparent that the copper atoms were distributed over four general sites (8g), each copper atom being assigned occupancy 0.5. The tetrapropylammonium ions were located from successive electron density maps.[10] One of these groups was found to have a disordered orientation with respect to the twofold axis on which the nitrogen atom, N(3), is situated. The disorder

affects four carbon atoms, viz. C(7), C(8), C(10) and C(11), each of these occupying two general sites 8g [e.g. C(7) and C(7′)] with occupancy 0.5. Full-matrix least-squares refinement[10] of positional, isotropic thermal parameters for the carbon atoms of the disordered cation, and anisotropic thermal parameters for the remaining atoms gave $R=0.062$ (167 parameters; 1168 observed reflections, one reflection being considered to be affected by extinction being excluded from the final cycles of refinement). Atomic scattering factors were taken from the *International Tables for X-Ray Crystallography*[6b] and the F_o values were weighted according to $w=[\sigma^2(F_o)+0.0018F_o^2]^{-1}$. A final difference map showed a maximum electron density of 0.7 eÅ^{-3}. Hydrogen atoms were not included in the calculations. No attempt was made to refine occupation

Table 1. Fractional coordinates and equivalent isotropic thermal parameters (Å^2) for [N(C$_3$H$_7$)$_4$]$_2$[Cu$_4$Br$_6$]. B_{eq} is defined as $8\pi^2/3\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i\cdot\mathbf{a}_j$. For the carbon atoms of the disordered cation, C(7)−C(12), isotropic thermal parameters B (Å^2), where the temperature factor is $\exp\{-B(\sin^2\theta)/\lambda^2\}$, are given. Estimated standard deviations are within parentheses. An asterisk denotes an atom in 8g with occupancy 0.5.

| Atom	x	y	z	B_{eq} or B
Br(1)	0.1629(1)	0.0851(1)	0.4706(1)	5.26(5)
Br(2)	0.0874(1)	−0.1649(1)	0.5065(1)	6.21(6)
Br(3)	−0.0282(1)	−0.0205(1)	0.3152(1)	6.31(5)
Cu(1)*	0.0727(3)	−0.0318(3)	0.4301(3)	5.5(1)
Cu(2)*	−0.0164(3)	−0.0752(3)	0.5734(3)	4.9(1)
Cu(3)*	−0.0917(3)	0.0196(3)	0.4470(3)	4.8(1)
Cu(4)*	0.0316(3)	0.0893(3)	0.5492(3)	5.2(1)
N(1)	0.2500	0.2500	0.2500	4.0(5)
C(1)	0.2200(11)	0.1799(11)	0.1875(12)	4.7(4)
C(2)	0.1845(12)	0.1009(12)	0.2342(13)	5.8(5)
C(3)	0.1654(13)	0.0314(12)	0.1641(14)	6.7(5)
N(2)	−0.2500	−0.2500	0.2500	4.3(4)
C(4)	−0.1851(11)	−0.1998(11)	0.1961(11)	4.5(4)
C(5)	−0.2313(13)	−0.1380(14)	0.1331(13)	7.4(6)
C(6)	−0.1613(14)	−0.0950(14)	0.0809(13)	7.3(6)
N(3)	−0.2500	0.2500	0.2245(13)	4.6(5)
C(7)*	−0.293(2)	0.164(3)	0.255(2)	4.5(8)
C(7′)*	−0.242(3)	0.305(3)	0.301(3)	5.7(9)
C(8)*	−0.247(3)	0.113(3)	0.316(3)	8.0(12)
C(8′)*	−0.214(3)	0.400(3)	0.265(3)	8.2(14)
C(9)	−0.301(2)	0.045(2)	0.355(2)	9.0(6)
C(10)*	−0.298(3)	0.283(3)	0.150(3)	5.7(9)
C(10′)*	−0.155(3)	0.229(3)	0.192(3)	6.6(10)
C(11)*	−0.386(4)	0.313(4)	0.165(4)	8.6(14)
C(11′)*	−0.155(3)	0.158(3)	0.118(3)	7.6(12)
C(12)	−0.439(2)	0.358(2)	0.098(2)	9.7(7)
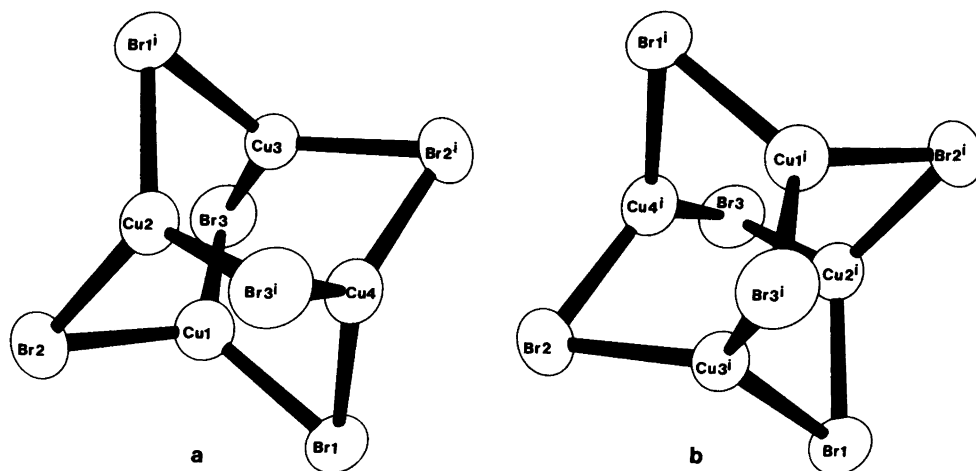
Fig. 1. The $[Cu_4Br_6]^{2-}$ ion showing the two orientations of the copper(I) tetrahedron. The superscript *i* denotes an atom in $-x,-y,1-z$. Thermal ellipsoids enclose 50 % probability.[15]

factors for the disordered atoms. Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1, distances and angles within the anion in Table 2, and those within the cations in Table 4. Structure factors and anisotropic thermal parameters may be obtained from the authors.

DISCUSSION

In $[N(C_3H_7)_4]_2[Cu_4Br_6]$ the anion is an aggregate composed of an octahedron of bromide ligands containing a tetrahedron of trigonal-planar coordinated copper(I) atoms. The copper(I) tetrahedron can assume either of two equivalent orientations related by inversion through the centre of the ligand octahedron which coincides with a crystallographic centre of symmetry (see Figs. 1a and b). Distances and angles within the anion are given in Table 2, the orientation being that of Fig. 1a. In the refinement the copper atoms were assigned site occupancies of 0.5, which corresponds to equal occurrence of anions with the orientation of Fig. 1a and with that of Fig. 1b. Preferential orientation of the anions was not investigated.

The $[Cu_4Br_6]^{2-}$ anion is essentially similar to the $[Cu_4I_6]^{2-}$ anion, determined in the methyltriphenylphosphonium compound,[5] which also shows similar disorder with respect to the

orientation of the copper(I) tetrahedron. In $[Cu_4Br_6]^{2-}$, the four copper(I) atoms, $Cu(1)-Cu(4)$, are all approximately trigonal-planar coordinated and lie 0.016(5), 0.015(5), 0.023(5) and 0.029(5) Å, respectively, from the planes through the three ligand atoms. Some features of the coordination geometries of discrete bromocuprate(I) and iodocuprate(I) ions containing trigonal-planar coordinated copper(I) are summarized in Table 3. In the cation radical salt of tetrathiotetracene (TTT) with dibromocuprate(I)[11] there are additional Cu−S contacts of 2.684 and 3.062 Å to neighbouring cation radicals such that the configuration of ligands about copper(I) is approximately trigonal bipyramidal.

There would appear to be less variation in the distances and angles tabulated in Table 3 for the $[Cu_4I_6]^{2-}$ and $[Cu_2I_4]^{2-}$ ions than is the case for their bromo counterparts. Perhaps the most striking discrepancy within the latter group of ions is the irregularity of the copper(I) coordination in bis(tetraethylammonium) di-μ-bromo-dibromodicuprate(I). As has been noted previously,[2] the $Br_b \cdots Br_b$ contact over the four-membered Cu−Br ring appears to assume a minimum value, the Cu$\cdots$Cu separation and the Cu−Br_b−Cu angle being larger and the Br_b−Cu−Br_b angle smaller than might perhaps have been expected. In $(TTT)_2[Cu_2Br_4]$,[11] it is conceivable that the steric requirements of the

Table 2. Interatomic distances (Å) and angles (°) within the $[Cu_4Br_6]^{2-}$ ion, the orientation being as in Fig. 1a. Estimated standard deviations are given in parentheses. The superscript (*i*) denotes an atom in $-x, -y, 1-z$.

Cu(1)−Br(1)	2.405(5)	Cu(3)−Br(1^i)	2.373(5)
Cu(1)−Br(2)	2.422(5)	Cu(3)−Br(2^i)	2.398(5)
Cu(1)−Br(3)	2.409(5)	Cu(3)−Br(3)	2.383(5)
Cu(2)−Br(1^i)	2.408(5)	Cu(4)−Br(1)	2.405(5)
Cu(2)−Br(2)	2.398(5)	Cu(4)−Br(2^i)	2.381(5)
Cu(2)−Br(3^i)	2.411(5)	Cu(4)−Br(3^i)	2.389(5)
Cu(1)···Cu(2)	2.738(7)	Cu(2)···Cu(3)	2.750(7)
Cu(1)···Cu(3)	2.719(7)	Cu(2)···Cu(4)	2.718(7)
Cu(1)···Cu(4)	2.744(7)	Cu(3)···Cu(4)	2.743(7)
Br(1)−Cu(1)−Br(2)	118.2(2)	Br(1^i)−Cu(3)−Br(2^i)	120.4(2)
Br(1)−Cu(1)−Br(3)	122.0(2)	Br(1^i)−Cu(3)−Br(3)	119.2(2)
Br(2)−Cu(1)−Br(3)	119.8(2)	Br(2^i)−Cu(3)−Br(3)	120.3(2)
Br(1^i)−Cu(2)−Br(2)	119.0(2)	Br(1)−Cu(4)−Br(2^i)	119.9(2)
Br(1^i)−Cu(2)−Br(3^i)	121.8(2)	Br(1)−Cu(4)−Br(3^i)	117.7(2)
Br(2)−Cu(2)−Br(3^i)	119.2(2)	Br(2^i)−Cu(4)−Br(3^i)	122.4(2)
Cu(1)−Br(1)−Cu(4)	69.6(2)	Cu(3)−Br(2^i)−Cu(4)	70.1(2)
Cu(2)−Br(1^i)−Cu(3)	70.2(2)	Cu(1)−Br(3)−Cu(3)	69.1(2)
Cu(1)−Br(2)−Cu(2)	69.2(2)	Cu(2)−Br(3^i)−Cu(4)	69.0(2)
Cu(2)···Cu(1)···Cu(3)	60.5(2)	Cu(1)···Cu(3)···Cu(2)	60.1(2)
Cu(2)···Cu(1)···Cu(4)	59.4(2)	Cu(1)···Cu(3)···Cu(4)	60.3(2)
Cu(3)···Cu(1)···Cu(4)	60.3(2)	Cu(2)···Cu(3)···Cu(4)	59.3(2)
Cu(1)···Cu(2)···Cu(3)	59.4(2)	Cu(1)···Cu(4)···Cu(2)	60.2(2)
Cu(1)···Cu(2)···Cu(4)	60.4(2)	Cu(1)···Cu(4)···Cu(3)	59.4(2)
Cu(3)···Cu(2)···Cu(4)	60.2(2)	Cu(2)···Cu(4)···Cu(3)	60.5(2)

Table 3. Copper(I) coordination geometry in some trigonal-planar coordinated bromocuprates(I) and iodocuprates(I). Distances are in Å and angles in °. A terminal halogen ligand is denoted X_t and a bridging ligand X_b.

Compound	Cu−X_t	Cu−X_b	Cu···Cu
$[N(C_3H_7)_4]_2[Cu_4Br_6]$	—	2.373(5)−2.422(5)	2.718(7)−2.750(7)
$[N(C_2H_5)_4]_2[Cu_2Br_4]$	2.319(2)	2.441(2)−2.454(2)	2.937(3)
$(TTT)_2[Cu_2Br_4]^a$	2.328(3)	2.472(3)−2.490(2)	2.660(3)
$[P(C_6H_5)_3(CH_3)]_2[Cu_4I_6]$	—	2.539(5)−2.638(5)	2.742(7)−2.757(7)
$[N(C_4H_9)_4]_2[Cu_2I_4]$	2.514(2)	2.566(2)−2.592(2)	2.726(4)
$[N(C_3H_7)_4]_2[Cu_2I_4]$	2.499(1)	2.571(1)−2.582(1)	2.698(2)
$[As(C_6H_5)_4]_2[Cu_2I_4]$	2.490(3)−2.491(3)	2.578(3)−2.610(3)	2.663(4)

Compound	Cu−X_b−Cu	X_b−Cu−X_b	X_b···X_b	Ref.
$[N(C_3H_7)_4]_2[Cu_4Br_6]$	69.0(2)−70.2(2)	117.2(2)−122.4(2)	4.102(3)−4.210(3)	Present work
$[N(C_2H_5)_4]_2[Cu_2Br_4]$	73.7(1)	106.3(1)	3.916(3)	2
$(TTT)_2[Cu_2Br_4]^a$	64.7(1)	115.4(1)	4.188(4)^b	11
$[P(C_6H_5)_3(CH_3)]_2[Cu_4I_6]$	63.9(2)−65.2(2)	117.9(2)−121.6(2)	4.38(2)−4.52(2)	5
$[N(C_4H_9)_4]_2[Cu_2I_4]$	63.8(1)	116.2(1)	4.380(3)	12
$[N(C_3H_7)_4]_2[Cu_2I_4]$	63.14(3)	116.86(3)	4.390(1)	13
$[As(C_6H_5)_4]_2[Cu_2I_4]$	61.4(1)−62.1(1)	114.2(1)−114.4(1)	4.360(3)	14

^{a} TTT=tetrathiotetracene; two additional Cu−S contacts, 2.684 and 3.062 Å to the tetrathiotetracene cation radicals. ^{b} Estimated.
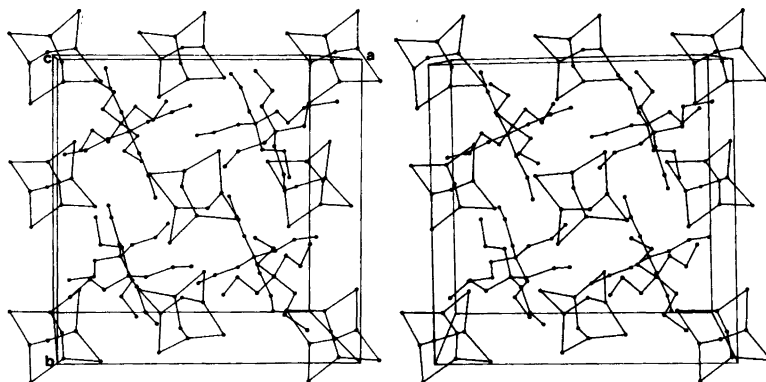
Fig. 2. Stereoscopic view [15] of the unit cell. For clarity all atoms are represented as spheres of radius 0.05 Å, and the anion and the disordered cation are each drawn in only one of the possible orientations.

interactions between copper(I) and the tetra-thiotetracene cation radicals is instrumental in the attainment of a shorter Cu···Cu contact and a larger $Br_b-Cu-Br_b$ angle. The coordination geometry of copper(I) in $[Cu_4Br_6]^{2-}$ is, however, also nearer the ideal trigonal-planar configuration and very similar to that found for $[Cu_4I_6]^{2-}$.

The non-bonded $Br_b \cdots Br_b$ contacts in $[Cu_4Br_6]^{2-}$ are longer than that over the four-membered ring in $[N(C_2H_5)_4]_2[Cu_2Br_4]$, while $Cu-Br$ distances appear to be slightly shorter than the $Cu-Br_b$ distances in both the $[Cu_2Br_4]^{2-}$ dimers (Table 3).

Bond lengths and angles within the tetrapropylammonium ions are given in Table 4. The two

Table 4. Bond lengths (Å) and angles (°) within the tetrapropylammonium ions in $[N(C_3H_7)_4]_2[Cu_4Br_6]$. Standard deviations are given in parentheses. Symmetry code: $(i):\frac{1}{2}-x,\frac{1}{2}-y,z$; $(ii): y,\frac{1}{2}-x,\frac{1}{2}-z$; $(iii): -x-\frac{1}{2},-y-\frac{1}{2},z$; $(iv): y,-x-\frac{1}{2},\frac{1}{2}-z$; $(v): -x-\frac{1}{2},\frac{1}{2}-y,z$. The other orientation of the disordered cation is obtained by rotation about the twofold axis through N(3), *i.e.* by applying the operation $-x-\frac{1}{2},\frac{1}{2}-y,z$ to the carbon atoms associated with N(3) cited below.

N(1)−C(1)	1.55(2)	N(2)−C(4)	1.54(2)
C(1)−C(2)	1.54(2)	C(4)−C(5)	1.56(3)
C(2)−C(3)	1.58(3)	C(5)−C(6)	1.53(3)
N(3)−C(7)	1.58(4)	N(3)−C(7′)	1.48(4)
C(7)−C(8)	1.43(6)	C(7′)−C(8′)	1.66(7)
C(8)−C(9)	1.50(6)	C(8′)−C(9ᵛ)	1.67(6)
N(3)−C(10)	1.49(4)	N(3)−C(10′)	1.61(5)
C(10)−C(11)	1.48(7)	C(10′)−C(11′)	1.61(7)
C(11)−C(12)	1.51(6)	C(11′)−C(12ᵛ)	1.53(6)
C(1)−N(1)−C(1ⁱ)	101(1)	C(4)−N(2)−C(4ⁱⁱⁱ)	113(1)
C(1)−N(1)−C(1ⁱⁱ)	114(1)	C(4)−N(2)−C(4ⁱᵛ)	108(1)
N(1)−C(1)−C(2)	112(1)	N(2)−C(4)−C(5)	111(1)
C(1)−C(2)−C(3)	107(1)	C(4)−C(5)−C(6)	106(2)
C(7)−N(3)−C(7′)	106(2)	C(7′)−N(3)−C(10)	119(2)
C(7)−N(3)−C(10)	109(2)	C(7′)−N(3)−C(10′)	107(2)
C(7)−N(3)−C(10′)	109(2)	C(10)−N(3)−C(10′)	107(3)
N(3)−C(7)−C(8)	118(3)	N(3)−C(7′)−C(8′)	106(3)
C(7)−C(8)−C(9)	113(4)	C(7′)−C(8′)−C(9ᵛ)	103(3)
N(3)−C(10)−C(11)	117(4)	N(3)−C(10′)−C(11′)	112(3)
C(10)−C(11)−C(12)	123(4)	C(10′)−C(11′)−C(12ᵛ)	105(4)

cations with $\bar{4}$ symmetry show no anomalous features, whereas the third cation is less well resolved owing to the disorder associated with its orientation. A stereoscopic view [15] of the unit cell is shown in Fig. 2, the anion and the disordered cation each having been drawn in one of the possible orientations for clarity. The shortest Cu$\cdots$C and Br$\cdots$C contacts are Cu(3)$\cdots$C(8)=3.52(5), Cu(3)$\cdots$C(9)=3.62(2), Cu(4)$\cdots$C(5)^i=3.53(2), Br(1)$\cdots$C(2)=3.74(2), Br(1)$\cdots$C(5)^i=3.87(2), Br(2)$\cdots$C(11')^{ii}=3.61(5), Br(2)$\cdots$C(10)^{iii}=3.81(4), Br(2)$\cdots$C(8)^{iv}= 3.84(5) and Br(3)$\cdots$C(8')^{iii}=3.86 Å. [Symmetry code: i: $-y,\frac{1}{2}+x,\frac{1}{2}+z$; ii: $y,-x-\frac{1}{2},\frac{1}{2}-z$; iii: $\frac{1}{2}-y,x,\frac{1}{2}-z$; iv: $-x,-y,1-z$]. It would thus seem likely that the disordered orientation of the third cation can be attributed to electrostatic interactions and efficient packing between cations and anions. The shortest Cu$\cdots$C distances in $[N(C_3H_7)_4]_2[Cu_4Br_6]$ are of the same order as the shortest such contact between the methyl group and $[Cu_4I_6]^{2-}$ in $[P(C_6H_5)_3(CH_3)]_2[Cu_4I_6]$, $i.e.$ 3.46(2) Å.[5]

In tetrabutylammonium dibromocuprate(I), copper(I) exhibits linear coordination geometry [1] whereas both $[N(C_3H_7)_4]_2[Cu_4Br_6]$ and $[N(C_2H_5)_4]_2[Cu_2Br_4]$ [2] contain trigonal-planar coordinated copper(I). There would appear to be a similar tendency towards increased coordination number of copper(I) with decreasing size of and increasing effective positive charge on the cation in tetraalkylammonium chlorocuprates(I).[1,16] In the tetraalkylammonium iodocuprates(I) investigated hitherto, there is a transition from trigonal-planar coordinated copper(I) in $[N(C_4H_9)_4]_2[Cu_2I_4]$ [12] and $[N(C_3H_7)_4]_2[Cu_2I_4]$ [13] to tetrahedral in $[N(C_2H_5)_4][Cu_2I_3]$.[17] As yet no discrete $[CuI_2]^-$ ion has been encountered in the solid state.

REFERENCES

1. Asplund, M., Jagner, S. and Nilsson, M. *Acta Chem. Scand. A 37* (1983) 57.
2. Asplund, M. and Jagner, S. *Acta Chem. Scand. A 38* (1984) 135.
3. Rømming, C. and Wærstad, K. *Chem. Commun.* (1965) 299.
4. Boehm, J. R., Balch, A. L., Bizot, K. F. and Enemark, J. H. *J. Am. Chem. Soc. 97* (1975) 501.
5. Bowmaker, G. A., Clark, G. R. and Yuen, D. K. P. *J. Chem. Soc. Dalton Trans.* (1976) 2329.
6. a. *International Tables for X-Ray Crystallography,* Kynoch Press, Birmingham 1952, Vol. 1, p. 176; b. *Ibid.* 1974, Vol. 4, p. 72.
7. Lehmann, M. S. and Larsen, F. K. *Acta Crystallogr. A 30* (1974) 580.
8. Lindqvist, O. and Ljungström, E. *J. Appl. Crystallogr. 12* (1979) 134.
9. Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J. P. and Woolfson, M. M. *MULTAN 80, A System of Computer Programs for the Automatic Solution of Crystal Structures from X-Ray Diffraction Data,* Univs. of York, England and Louvain, Belgium 1980.
10. Lindgren, O. *An Integrated Set of Crystallographic Programs,* In *On the Oxygen Coordination of Cerium in Some Sulfates and Chromates,* Thesis, Department of Inorganic Chemistry, Chalmers University of Technology and University of Göteborg, Göteborg 1977.
11. Shibaeva, R. P. and Kaminskii, V. F. *Kristallografiya 26* (1981) 332.
12. Asplund, M., Jagner, S. and Nilsson, M. *Acta Chem. Scand. A 36* (1982) 751.
13. Asplund, M. and Jagner, S. *Acta Chem. Scand. A 38* (1984) 411.
14. Asplund, M. and Jagner, S. *Acta Chem. Scand. A 38* (1984) 297.
15. Johnson, C. K. *ORTEP,* Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge 1965.
16. Asplund, M. and Jagner, S. *Acta Chem. Scand. A 38* (1984). *In press.*
17. Hartl, H. and Mahdjour-Hassan-Abadi, F. *Angew. Chem. 93* (1981) 804.