Table 1. *Atomic coordinates of non-H atoms* ($\times 10^4$) ($\times 10^5$ *for* Cu) *and equivalent isotropic thermal parameters* ($\times 10^4$) *with e.s.d.'s in parentheses*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq} (Å^2)
Cu	22269 (3)	18733 (5)	21683 (5)	425 (2)
N11	2361 (2)	2668 (2)	1203 (3)	418 (14)
N12	1562 (2)	2473 (3)	2108 (3)	422 (12)
C1	2780 (2)	2724 (4)	768 (4)	505 (18)
C2	2825 (2)	3315 (5)	160 (4)	588 (21)
C3	2431 (2)	3852 (4)	−4 (4)	530 (18)
C4	1992 (2)	3809 (4)	446 (3)	463 (16)
C5	1968 (2)	3212 (3)	1045 (3)	393 (14)
C6	1523 (2)	3102 (4)	1571 (3)	401 (14)
C7	1092 (2)	3611 (4)	1523 (4)	514 (19)
C8	689 (2)	3448 (4)	2036 (4)	533 (17)
C9	739 (2)	2808 (5)	2588 (4)	570 (20)
C10	1177 (2)	2323 (5)	2615 (4)	555 (20)
N21	2490 (2)	2578 (3)	3141 (3)	474 (14)
N22	2917 (2)	1379 (3)	2287 (3)	429 (14)
C21	2274 (3)	3230 (5)	3527 (4)	629 (22)
C22	2495 (3)	3655 (5)	4141 (4)	661 (24)
C23	2971 (3)	3402 (5)	4393 (5)	698 (26)
C24	3212 (3)	2731 (5)	4004 (4)	566 (19)
C25	2960 (2)	2345 (4)	3383 (3)	433 (15)
C26	3198 (2)	1650 (4)	2912 (3)	439 (15)
C27	3667 (2)	1272 (5)	3071 (5)	607 (22)
C28	3850 (3)	652 (4)	2581 (6)	705 (26)
C29	3577 (2)	375 (5)	1946 (5)	636 (21)
C210	3098 (2)	757 (4)	1834 (4)	556 (19)
S2	1878 (1)	103 (1)	2956 (1)	468 (4)
N2	1885 (2)	706 (3)	2169 (3)	466 (13)
O21	2350 (2)	−346 (3)	3069 (3)	713 (15)
O22	1701 (2)	600 (3)	3620 (3)	678 (17)
O23	1507 (2)	780 (4)	953 (3)	725 (18)
C31	1398 (2)	−591 (4)	2612 (3)	471 (15)
C32	1170 (3)	−1287 (5)	2995 (5)	667 (23)
C33	799 (3)	−1720 (4)	2572 (6)	776 (26)
C34	681 (3)	−1478 (5)	1822 (6)	821 (31)
C35	910 (2)	−806 (5)	1436 (5)	662 (21)
C36	1275 (2)	−348 (4)	1851 (3)	472 (16)
C37	1568 (2)	429 (4)	1601 (3)	454 (15)
Ow1	1638 (3)	5000 (0)	5000 (0)	783 (26)
Ow2	−971 (2)	5000 (0)	5000 (0)	796 (28)
Ow3	192 (3)	4785 (5)	3699 (4)	1043 (29)
S1	729 (1)	3086 (1)	5191 (1)	600 (5)
N1	213 (2)	3563 (4)	5429 (4)	721 (19)
O11	999 (2)	3570 (4)	4601 (4)	863 (21)
O12	1027 (2)	2863 (5)	5862 (4)	907 (23)
O13	−650 (2)	3302 (5)	5309 (4)	877 (21)
C11	449 (2)	2170 (4)	4750 (3)	519 (16)
C12	679 (3)	1478 (5)	4397 (5)	719 (25)
C13	355 (4)	883 (7)	4070 (7)	982 (41)
C14	−162 (4)	996 (7)	4050 (7)	1036 (44)
C15	−382 (3)	1713 (6)	4421 (6)	800 (30)
C16	−58 (2)	2290 (4)	4766 (4)	550 (19)
C17	−203 (2)	3118 (5)	5208 (4)	628 (22)

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

Cu—N11	2·082 (5)	Cu—N22	1·982 (5)
Cu—N12	1·986 (5)	Cu—N2	2·030 (5)
Cu—N21	2·098 (5)		
N22—Cu—N2	93·3 (2)	N12—Cu—N21	94·8 (2)
N21—Cu—N2	128·0 (2)	N11—Cu—N2	127·6 (2)
N21—Cu—N22	79·8 (2)	N11—Cu—N22	99·0 (2)
N12—Cu—N2	91·9 (2)	N11—Cu—N21	104·4 (2)
N12—Cu—N22	174·1 (2)	N11—Cu—N12	80·1 (2)

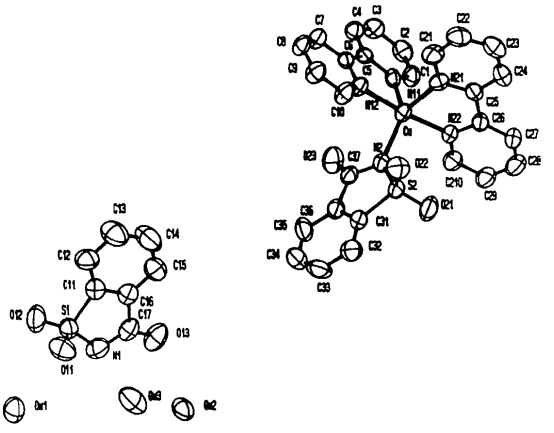

Fig. 1. View of the asymmetric unit showing atom numbering. Thermal ellipsoids are drawn at the 50% probability level.

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Bis(tetramethylphosphonium) Hexa-μ-bromo-tetrabromotetracuprate(II)

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Abstract. $2C_4H_{12}P^+.Br_{10}Cu_4^{2-}$, $M_r = 1235.5$, monoclinic, $P2_1/c$, $a = 6.416$ (2), $b = 20.214$ (6), $c = 11.236$ (3) Å, $\beta = 98.52$ (2)°, $V = 1455.4$ (7) Å^3, $Z = 2$, $D_x = 2.82$ Mg m^{-3}, $\mu = 16.6$ mm^{-1}, λ(Mo $K\alpha$) = 0.71069 Å, $F(000) = 1136$, $T = 295$ K, $R = 0.039$ for 1596 unique observed [$|F| \geq 3\sigma(F)$] reflections. The

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Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for* $(Me_4P)_2Cu_4Br_{10}$

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cu(1)	3587 (2)	3991 (1)	1912 (1)	41 (1)
Cu(2)	8055 (2)	4661 (1)	788 (1)	52 (1)
Br(1)	9366 (2)	5760 (1)	420 (1)	39 (1)
Br(2)	5248 (2)	5082 (1)	1792 (1)	42 (1)
Br(3)	6371 (2)	3611 (1)	831 (1)	53 (1)
Br(4)	966 (2)	4473 (1)	2952 (1)	46 (1)
Br(5)	2817 (2)	2909 (1)	2456 (1)	68 (1)
P	8441 (4)	1498 (1)	665 (3)	44 (1)
C(1)	6700 (16)	869 (5)	6 (10)	61 (5)
C(2)	8626 (18)	2116 (5)	−431 (10)	68 (5)
C(3)	10973 (15)	1164 (4)	1145 (10)	54 (4)
C(4)	7466 (17)	1823 (5)	1938 (10)	60 (5)

Table 2. *Bond distances* (Å) *and angles* (°) *for* $(Me_4P)_2Cu_4Br_{10}$

Cu(1)—Br(2)	2.481 (1)	Cu(2)—Br(1*B*)	2.447 (2)
Cu(1)—Br(3)	2.431 (2)	Cu(2)—Br(2*A*)	3.369 (2)
Cu(1)—Br(4)	2.396 (2)	Cu(2)—Br(4*A*)	2.864 (2)
Cu(1)—Br(5)	2.362 (2)	P—C(1)	1.789 (10)
Cu(1)—Br(1*A*)	3.041 (2)	P—C(1)	1.789 (10)
Cu(2)—Br(1)	2.453 (1)	P—C(2)	1.780 (11)
Cu(2)—Br(2)	2.421 (2)	P—C(3)	1.771 (10)
Cu(2)—Br(3)	2.405 (1)	P—C(4)	1.772 (12)

Br(2)—Cu(1)—Br(3)	84.6 (1)	Br(1)—Cu(2)—Br(4*A*)	94.0 (1)
Br(2)—Cu(1)—Br(4)	89.8 (1)	Br(2)—Cu(2)—Br(4*A*)	95.1 (1)
Br(3)—Cu(1)—Br(4)	174.3 (1)	Br(3)—Cu(2)—Br(4*A*)	95.8 (1)
Br(2)—Cu(1)—Br(5)	163.9 (1)	Br(1*B*)—Cu(2)—Br(4*A*)	90.8 (1)
Br(3)—Cu(1)—Br(5)	91.6 (1)	Br(2*A*)—Cu(2)—Br(4*A*)	178.0 (1)
Br(4)—Cu(1)—Br(5)	94.0 (1)	Cu(2)—Br(1)—Cu(1*A*)	96.5 (1)
Br(2)—Cu(1)—Br(1*A*)	91.2 (1)	Cu(2)—Br(1)—Cu(2*B*)	92.8 (1)
Br(3)—Cu(1)—Br(1*A*)	91.9 (1)	Cu(1*A*)—Br(1)—Cu(2*B*)	88.1 (1)
Br(4)—Cu(1)—Br(1*A*)	87.7 (1)	Cu(1*A*)—Br(1)—Br(1*B*)	93.3 (1)
Br(5)—Cu(1)—Br(1*A*)	104.6 (1)	Cu(1)—Br(2)—Cu(2)	93.6 (1)
Br(1)—Cu(2)—Br(2)	92.9 (1)	Cu(2)—Br(2)—Cu(2*C*)	94.0 (1)
Br(1)—Cu(2)—Br(3)	170.2 (1)	Cu(1)—Br(3)—Cu(2)	95.3 (1)
Br(2)—Cu(2)—Br(3)	86.5 (1)	Cu(1)—Br(4)—Cu(2*A*)	93.3 (1)
Br(1)—Cu(2)—Br(1*B*)	87.2 (1)	C(1)—P—C(2)	108.9 (5)
Br(2)—Cu(2)—Br(1*B*)	174.1 (1)	C(1)—P—C(3)	109.7 (5)
Br(3)—Cu(2)—Br(1*B*)	92.3 (1)	C(2)—P—C(3)	109.4 (5)
Br(1)—Cu(2)—Br(2*A*)	84.3 (1)	C(1)—P—C(4)	109.2 (5)
Br(2)—Cu(2)—Br(2*A*)	86.0 (1)	C(2)—P—C(4)	111.2 (5)
Br(3)—Cu(2)—Br(2*A*)	85.9 (1)	C(3)—P—C(4)	108.4 (5)
Br(1*B*)—Cu(2)—Br(2*A*)	88.2 (1)		

structure consists of quasi-planar bibridged $Cu_4Br_{10}^{2-}$ oligomers in which the Cu—Br distances average 2.425 Å with the terminal Cu—Br distances (3.362 and 3.396 Å) considerably shorter than the bridging Cu—Br distances (2.405–2.481 Å). The oligomers aggregate, with the formation of long, semi-coordinate bonds, distances ranging from 2.864 (2) to 3.369 (2) Å, to form stacks.

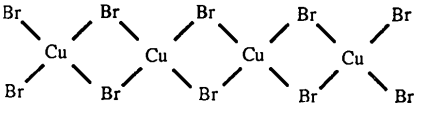
Experimental. Crystals were prepared by slow evaporation of a concentrated HBr solution containing a 1:2 ratio of $(CH_3)_4PBr$ to $CuBr_2$. Long, purple needles were obtained.

A crystal with dimensions of $0.25 \times 0.25 \times 0.50$ mm was selected for data collection on a Syntex $P2_1$ diffractometer with graphite monochromator, upgraded to Nicolet *P3F* specifications. Lattice constants from 25 reflections in the range $25 < 2\theta < 35°$. Data were collected with ω scans (0.9°); two check reflections monitored every 96 reflections (021 and 113) showed a systematic increase of 20% during the data collection; 2145 total reflections out to $2\theta = 45°$, 1884 unique with R(merge) = 0.041; hkl ranges, $0 \le h \le 6$, $0 \le k \le 21$, $-11 \le l \le 11$ (Campana, Shepard & Litchman, 1981). Empirical ψ-scan absorption corrections applied assuming an ellipsoidally shaped crystal (relative transmission factors range from 0.26 to 0.65).

The structure solution was based on the isomorphous $[(CH_3)_4N]_2Cu_4Cl_{10}$ salt (Halvorson, Grigereit & Willet, 1987). *SHELXTL* (Sheldrick, 1985) was used for all crystallographic calculations. A difference synthesis based on the Br, Cu and P positions yielded the C-atom positions. H atoms were constrained to ideal positions (C—H = 0.96 Å) and assigned isotropic thermal parameters of 0.08 $Å^2$.

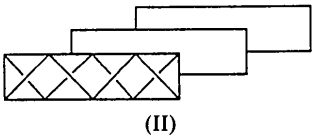
The final refinement resulted in $R = 0.039$ (3σ data set) and 0.048 (all data), $wR = 0.036$ [$F > 3\sigma(F)$] and 0.037 (all data), and $w = 1/[\sigma^2(F) + g(F)^2]$, with $g = 0.000$. 110 parameters refined. The goodness of fit was 1.80, $|\Delta/\sigma|$(max.) = 0.003. The largest peak on the final difference map was 0.7 e $Å^{-3}$ near Br(4), while the most negative excursion was −0.6 e $Å^{-3}$. Extinction corrections were made, $x = 0.0149$ (1). Atomic coordinates are listed in Table 1* and bond distances and angles are given in Table 2. A view of the stacks of $Cu_4Br_{10}^{2-}$ anions is shown in Fig. 1.

Related literature. The structure consists of quasi-planar bibridged $Cu_4Br_{10}^{2-}$ oligomers (I).



(I)

The oligomers aggregate, with the formation of long, semi-coordinate bonds to form stacks which can be represented as



(II)

* Lists of structure factors, anisotropic thermal parameters, data-collection parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54423 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

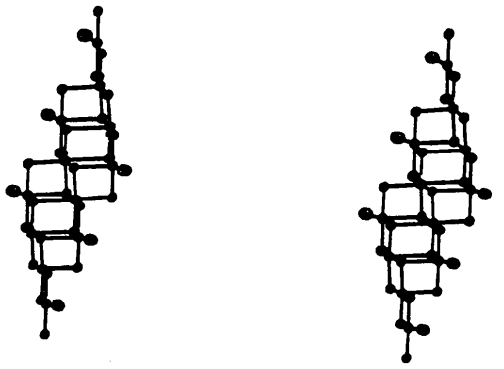


Fig. 1. Stereoscopic illustration of the stacking of $Cu_4Br_{10}^{2-}$ anions in $(Me_4P)_2Cu_4Br_{10}$.

This study extends the crystal chemistry of compounds in the series $(Me_4PcX)_n(MX_2)_m$, where Pc = N, P, As or Sb; M = divalent metal ion, and X = Cl, Br or I (Pressprich, Bond & Willett, 1991). For an $n:m$ ratio of 2:1, the Cu^{2+} salts assume the β-K_2SO_4 structure for Pc = N, X = Cl (Clay, Murray-Rust & Murray-Rust, 1975), Pc = N, X = Br (Hasebe, Mashiyama & Gesi, 1985), Pc = P, X = Cl (Pressprich, Bond, Willett & White, 1989) and Pc = P, X = Br (Madariaga, Alberdi & Zúñiga, 1990). The first three all exhibit incommensurate phases. For $n:m$ = 1:1, only $(Me_4N)CuCl_3$ is known (Weenk & Spek, 1976; Willett, Bond, Haije, Soonieus & Maaskant, 1988) and is notable for the existence of phases manifesting a cooperative, dynamic Jahn–Teller effect (Willett *et al.*, 1988; Haije & Maaskant, 1988) and one-dimensional ferromagnetic behavior at low temperature (Landee & Willett, 1979). For $n:m$ = 1:2, the above-cited $(Me_4N)_2Cu_4Cl_{10}$ salt is found, while $(Me_4P)Cu_2Cl_5$ assumes a structure with a complex two-dimensional Cu/Cl framework (Haije, Dobbelaar & Maaskant, 1986).

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Structure of Dicarbonyl(η^4-1,5-cyclooctadiene)(triphenylphosphine)ruthenium

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Abstract. $[Ru(\eta^4\text{-}C_8H_{12})(CO)_2\{P(C_6H_5)_3\}]$, M_r = 527·57, monoclinic, $P2_1/n$, a = 9·130 (6), b = 16·638 (13), c = 16·163 (14) Å, β = 99·2 (1)°, V = 2423 (8) Å^3, Z = 4, D_m = 1·43, D_x = 1·45 g cm^{-3}, Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 7·227 cm^{-1}, $F(000)$ = 1080, T = 295 K. R = 0·066 for 1867 reflections with $I > 3\sigma(I)$ in the $\pm h$, $+k$, $\pm l$ quadrant. The Ru atom is coordinated by seven atoms, six C atoms, and one P atom, located at distances varying from 1·8 to 2·3 Å. Some of the C atoms belonging to the cyclooctadiene molecule exhibit important anisotropic displacement coefficients.

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