

(Friauf, 1927). The Pt–Pt distances (2.645 Å) are significantly shorter than in metallic platinum (2.774 Å).

Related literature. For a tentative phase diagram of the Na–Pt system, based on data given by Loebich & Raub (1981), see Moffatt (1984). The composition of the cubic 1:2 phase was erroneously given as Na₂Pt in Massalski (1986). The present single-crystal study confirms the MgCu₂-type structure for NaPt₂, derived earlier from inconclusive powder diffraction data (Nash, Boyden & Wittig, 1960; Loebich & Raub, 1981).

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Structure of 2,3,4- μ_3 -Bromo-1,2,3;1,3,4;1,2,4-tri- μ_3 -sulfido-tris[(triphenylphosphine)copper](oxomolybdenum)(3 Cu–Mo)

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Abstract. [MoCu₃Br(O)S₃{P(C₆H₅)₃}₃], $M_r = 1265$, triclinic, $P\bar{1}$, $a = 11.804$ (5), $b = 13.049$ (4), $c = 20.406$ (3) Å, $\alpha = 74.84$ (2), $\beta = 84.41$ (2), $\gamma = 63.85$ (3)°, $V = 2722.7$ Å³, $Z = 2$, $D_x = 1.54$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 22.6$ cm⁻¹, $F(000) = 1268$, $T = 296$ K, $R = 0.064$ for 2890 unique observed reflections with $I > 3\sigma(I)$. The Mo atom is tetrahedrally coordinated by three μ_3 -S atoms and an O atom, and all three Cu atoms are also tetrahedrally coordinated by two μ_3 -S atoms, a P atom and a Br atom. The bond lengths of Cu– μ_3 -Br (av. 2.791 Å) are much longer than Mo– μ_3 -S (av. 2.256 Å) and Cu– μ_3 -S (av. 2.284 Å), so that the cubane-like core (MoCu₃-S₃Br) is distorted. There are three metal–metal bonds with the following bond lengths: Mo–Cu(1) 2.708, Mo–Cu(2) 2.705, Mo–Cu(3) 2.737 Å.

Experimental. Crystals of the title compound were prepared by allowing [NMe₄]₂[Mo₂O₂S(S₂)₄].CH₃CN to react with CuBr and PPh₃ (Wu, Lu, Zhu, Wu & Lu, 1987). Crystal 0.25 × 0.20 × 0.25 mm. Data were collected on a CAD-4 κ -geometry diffractometer using Mo $K\alpha$ radiation. $\omega/2\theta$ scan, scan speed varied from 3 to 5° min⁻¹ (in ω), the scan width was 0.60° +

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0.35° tan θ . Cell constants were obtained by least-squares fit to 25 diffractions' maxima. The intensities were corrected for absorption using empirical scan data (maximum and minimum transmission factors 1.00 and 0.83 respectively) and Lorentz and polarization factors to give a total of 9398 intensities, up to a maximum 2θ of 50° ($-14 < h < 14$, $-15 < k < 15$, $0 \leq l < 24$). Max. $(\sin\theta)/\lambda = 0.59$ Å⁻¹, 5991 reflections with $I < 3\sigma(I)$ were considered unobserved. 2890 reflections with $I > 3\sigma(I)$ and max. $(\sin\theta)/\lambda = 0.50$ Å⁻¹ were used

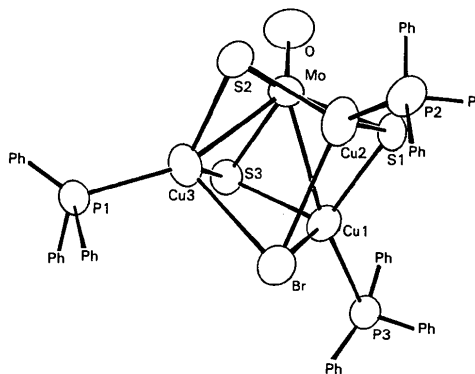


Fig. 1. The configuration of the cluster core of [(MoCu₃S₃Br)(O)(PPh₃)₃].

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Table 1. Atomic coordinates and equivalent isotropic thermal parameters

$$B_{eq} = \frac{4}{3} \sum_i \sum_j B_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Mo	0.1470 (2)	0.5005 (2)	0.1747 (1)	3.46 (5)
Br	0.4471 (2)	0.3150 (2)	0.3071 (1)	4.02 (7)
Cu(1)	0.2878 (3)	0.5465 (2)	0.2499 (2)	4.06 (8)
Cu(2)	0.2043 (3)	0.3357 (2)	0.2941 (2)	4.45 (9)
Cu(3)	0.3935 (3)	0.3424 (2)	0.1699 (2)	4.79 (9)
S(1)	0.1013 (5)	0.5353 (5)	0.2785 (3)	3.4 (2)
S(2)	0.2213 (6)	0.3035 (5)	0.1884 (3)	4.0 (2)
S(3)	0.3100 (6)	0.5448 (5)	0.1379 (3)	3.8 (2)
O	0.017 (2)	0.578 (2)	0.122 (1)	6.7 (6)
P(1)	0.5642 (6)	0.2364 (5)	0.1200 (4)	4.2 (2)
P(2)	0.1682 (6)	0.2161 (5)	0.3840 (3)	3.4 (2)
P(3)	0.3525 (5)	0.6522 (5)	0.2913 (3)	3.2 (2)
C(11)	0.520 (2)	0.616 (2)	0.279 (1)	3.1 (6)
C(12)	0.573 (2)	0.569 (2)	0.226 (1)	4.5 (7)
C(13)	0.703 (2)	0.559 (2)	0.208 (2)	6.0 (9)
C(14)	0.768 (2)	0.584 (2)	0.249 (1)	5.4 (8)
C(15)	0.710 (2)	0.626 (2)	0.304 (2)	5.6 (8)
C(16)	0.587 (2)	0.643 (2)	0.322 (1)	4.6 (7)
C(21)	0.335 (2)	0.638 (2)	0.381 (1)	3.9 (6)
C(22)	0.364 (2)	0.531 (2)	0.422 (1)	4.1 (6)
C(23)	0.359 (2)	0.511 (2)	0.495 (1)	4.8 (7)
C(24)	0.313 (3)	0.609 (2)	0.525 (2)	6.2 (9)
C(25)	0.282 (2)	0.716 (2)	0.484 (1)	6.3 (8)
C(26)	0.295 (2)	0.736 (2)	0.411 (1)	5.2 (7)
C(31)	0.266 (2)	0.812 (2)	0.253 (1)	3.5 (6)
C(32)	0.134 (3)	0.855 (2)	0.246 (2)	7 (1)
C(33)	0.330 (3)	0.882 (2)	0.237 (1)	6.0 (8)
C(34)	0.255 (3)	1.007 (2)	0.213 (1)	6.7 (9)
C(35)	0.127 (3)	1.049 (2)	0.206 (2)	9 (1)
C(36)	0.066 (3)	0.979 (3)	0.223 (2)	9 (1)
C(41)	0.122 (2)	0.275 (2)	0.459 (1)	3.1 (6)
C(42)	0.177 (2)	0.346 (2)	0.470 (1)	3.5 (6)
C(43)	0.148 (2)	0.385 (2)	0.529 (1)	4.4 (7)
C(44)	0.066 (2)	0.359 (2)	0.575 (1)	5.5 (8)
C(45)	0.008 (2)	0.291 (2)	0.563 (1)	5.8 (9)
C(46)	0.038 (2)	0.248 (2)	0.505 (1)	4.6 (7)
C(51)	0.301 (2)	0.074 (2)	0.413 (1)	4.0 (6)
C(52)	0.389 (2)	0.029 (2)	0.364 (1)	5.7 (8)
C(53)	0.500 (2)	-0.085 (2)	0.387 (2)	6.8 (9)
C(54)	0.508 (3)	-0.139 (2)	0.451 (2)	6.3 (9)
C(55)	0.431 (2)	0.100 (2)	0.501 (2)	6.1 (9)
C(56)	0.322 (2)	0.014 (2)	0.480 (1)	5.2 (8)
C(61)	0.040 (2)	0.181 (2)	0.370 (1)	3.6 (6)
C(62)	-0.064 (2)	0.278 (2)	0.334 (1)	5.7 (8)
C(63)	-0.170 (3)	0.263 (2)	0.325 (1)	6.2 (9)
C(64)	-0.171 (2)	0.148 (2)	0.353 (1)	6.3 (9)
C(65)	-0.065 (2)	0.056 (2)	0.389 (1)	5.9 (8)
C(66)	0.040 (2)	0.073 (2)	0.396 (1)	5.2 (7)
C(71)	0.659 (2)	0.085 (2)	0.169 (1)	4.4 (7)
C(72)	0.721 (3)	-0.003 (2)	0.136 (2)	8 (1)
C(73)	0.799 (3)	-0.124 (3)	0.172 (2)	9 (1)
C(74)	0.679 (3)	0.060 (2)	0.238 (1)	6.2 (9)
C(75)	0.755 (3)	-0.057 (3)	0.275 (1)	7 (1)
C(76)	0.813 (3)	-0.146 (2)	0.241 (2)	6.6 (9)
C(81)	0.525 (2)	0.221 (2)	0.043 (1)	4.9 (8)
C(82)	0.595 (3)	0.224 (2)	-0.018 (1)	6.4 (9)
C(83)	0.567 (3)	0.202 (2)	-0.076 (2)	8 (1)
C(84)	0.389 (3)	0.175 (2)	-0.019 (2)	9 (1)
C(85)	0.425 (3)	0.196 (2)	0.041 (1)	6.4 (9)
C(86)	0.707 (3)	0.477 (2)	0.042 (1)	6 (1)
C(91)	0.679 (2)	0.296 (2)	0.098 (1)	4.9 (8)
C(92)	0.629 (2)	0.419 (2)	0.069 (1)	6.5 (8)
C(93)	0.464 (3)	0.181 (3)	-0.077 (1)	8 (1)
C(94)	0.834 (3)	0.407 (3)	0.053 (2)	9 (1)
C(95)	0.884 (3)	0.283 (3)	0.083 (3)	17 (2)
C(96)	0.799 (3)	0.234 (3)	0.102 (3)	13 (2)

in the refinement. Three standard reflections were measured periodically, only random deviations.

The structure was solved by direct methods using *MULTAN*11/82 (Main *et al.*, 1982). Five heavy atoms (Mo, Cu, Br) were located in the *E* map. The remaining non-H atoms were located in the succeeding difference Fourier syntheses (H atoms were placed in geometrically calculated positions with C—H 0.96 Å, but

Table 2. Selected bond lengths (Å) and bond angles (°)

Mo—Cu(1)	2.708 (2)	Cu(2)—S(2)	2.276 (3)
Mo—Cu(2)	2.705 (2)	Cu(2)—P(2)	2.218 (3)
Mo—Cu(3)	2.737 (2)	Cu(3)—S(2)	2.279 (3)
Mo—S(1)	2.248 (3)	Cu(3)—S(3)	2.303 (4)
Mo—S(2)	2.267 (3)	Cu(3)—P(1)	2.211 (3)
Mo—S(3)	2.253 (3)	P(1)—C(71)	1.85 (1)
Mo—O	1.709 (8)	P(1)—C(81)	1.78 (1)
Br—Cu(1)	2.757 (2)	P(1)—C(91)	1.80 (1)
Br—Cu(2)	2.790 (2)	P(2)—C(41)	1.81 (2)
Br—Cu(3)	2.826 (2)	P(2)—C(51)	1.81 (2)
Cu(1)—S(1)	2.279 (3)	P(2)—C(61)	1.82 (1)
Cu(1)—S(3)	2.280 (3)	P(3)—C(11)	1.83 (2)
Cu(1)—P(3)	2.207 (3)	P(3)—C(21)	1.79 (1)
Cu(2)—S(1)	2.284 (3)	P(3)—C(31)	1.85 (1)
Cu(1)—Mo—Cu(2)	72.58 (5)	Mo—Cu(3)—Br	94.11 (6)
Cu(1)—Mo—Cu(3)	72.09 (5)	Br—Cu(3)—S(2)	97.16 (9)
Cu(2)—Mo—Cu(3)	72.57 (5)	Br—Cu(3)—S(3)	97.21 (9)
S(1)—Mo—S(2)	106.9 (2)	Br—Cu(3)—P(1)	110.7 (2)
S(1)—Mo—S(3)	106.7 (2)	S(2)—Cu(3)—S(3)	104.2 (4)
S(1)—Mo—O	110.9 (3)	S(2)—Cu(3)—P(1)	122.7 (1)
S(2)—Mo—S(3)	106.3 (2)	S(3)—Cu(3)—P(1)	119.7 (1)
S(2)—Mo—O	112.1 (3)	Mo—S(1)—Cu(1)	73.48 (9)
S(3)—Mo—O	113.5 (3)	Mo—S(1)—Cu(2)	73.30 (9)
Cu(1)—Br—Cu(2)	70.58 (5)	Cu(1)—S(1)—Cu(2)	89.3 (2)
Cu(1)—Br—Cu(3)	70.04 (5)	Mo—S(2)—Cu(2)	73.09 (9)
Cu(2)—Br—Cu(3)	70.00 (5)	Mo—S(2)—Cu(3)	74.0 (1)
Mo—Cu(1)—Br	96.36 (6)	Cu(2)—S(2)—Cu(3)	90.1 (1)
Br—Cu(1)—S(1)	97.84 (8)	Mo—S(3)—Cu(1)	73.4 (1)
Br—Cu(1)—S(3)	99.73 (9)	Mo—S(3)—Cu(3)	73.9 (1)
Br—Cu(1)—P(3)	105.51 (9)	Cu(1)—S(3)—Cu(2)	88.7 (2)
S(1)—Cu(1)—S(3)	104.7 (2)	C(71)—P(1)—C(81)	105.1 (5)
S(1)—Cu(1)—P(3)	123.0 (1)	C(71)—P(1)—C(91)	102.8 (5)
S(3)—Cu(1)—P(3)	120.9 (1)	C(81)—P(1)—C(91)	104.6 (6)
Mo—Cu(2)—Br	95.66 (6)	C(41)—P(2)—C(51)	104.5 (5)
Br—Cu(2)—S(1)	96.81 (8)	C(41)—P(2)—C(61)	104.1 (5)
Br—Cu(2)—S(2)	98.24 (9)	C(51)—P(2)—C(61)	104.7 (5)
Br—Cu(2)—P(2)	108.60 (9)	C(11)—P(3)—C(21)	103.7 (5)
S(1)—Cu(2)—S(2)	105.4 (1)	C(11)—P(3)—C(31)	106.0 (5)
S(1)—Cu(2)—P(2)	121.0 (1)	C(21)—P(3)—C(31)	104.4 (5)
S(2)—Cu(2)—P(2)	121.5 (1)		

not included in the refinement). The structure was refined by full-matrix least-squares techniques with anisotropic thermal parameters for all non-H atoms (595 variables). Final $R = 0.064$, $wR = 0.086$ and $S = 6.32$, the function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = 4F_o^2/\sigma^2(F_o^2)$, $\sigma^2(F_o^2) = [\sigma_o^2(F_o^2) + (0.07F_o^2)^2]$ where $\sigma_o^2(F_o^2)$ is the standard deviation based on counting statistics. $(\Delta/\sigma)_{\max} = 0.22$. In final difference electron density synthesis max. height less than $1.5 e \text{ \AA}^{-3}$. All calculations were performed on a VAX 785 computer using *SDP* (Frenz, 1978), the scattering factors were taken from Cromer & Waber (1974). *ORTEPII* (Johnson, 1976) was used to produce the molecular configuration shown in Fig. 1. The atom coordinates and equivalent isotropic thermal parameters are listed in Table 1, important bond lengths and bond angles are given in Table 2.*

Related literature. μ_3 -Chloro-tri- μ_3 -sulfido-tris[(tri-phenylphosphine)copper](oxomolybdenum)(3 *Cu—Mo*) was prepared by allowing MoOS_3^{2-} to react with PPh_3 and $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (Muller, Bogue & Schimanski, 1980).

*Lists of H-atom coordinates, full bond lengths and angles, structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51720 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

This crystal belongs to the orthorhombic system with space group $P2_12_12_1$.

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Magnesium Galactarate Dihydrate

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Abstract. Mg²⁺.C₆H₈O₈²⁻.2H₂O, $M_r = 268.46$, monoclinic, Cc , $a = 7.605$ (1), $b = 8.785$ (2), $c = 16.404$ (2) Å, $\beta = 92.56$ (1)°, $V = 1094.9$ Å³, $Z = 8$, $D_x = 1.63$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 6.17$ cm⁻¹, $F(000) = 276$, $T = 293$ K, $R = 0.052$ for 862 observed reflections and 105 parameters refined. The galactarate ion is centrosymmetrical and the Mg²⁺ ion is six-coordinated (octahedral). Mg-O distances are in the range 2.003 (3) to 2.117 (2) Å.

Experimental. The sample was prepared from MgCl₂ and disodium galactarate and crystallized from water. Crystal tabular, 0.15 × 0.06 × 0.09 mm, Enraf-Nonius CAD-4F diffractometer, Ni-filtered Cu $K\alpha$; cell parameters from 22 θ measurements in the range $22 < \theta < 40^\circ$; reflections measured for half the sphere of reflection to $2\theta = 140^\circ$ for ranges of h , k and l of -9 to 9, 0 to 10 and -20 to 20 respectively; 1535 reflections measured plus 628 with $[F < 3\sigma(F)]$; inten-

sity of 223 reflection measured 54 times: average count of 777.4 with a standard deviation (of the distribution) = 10.7 (1.4%) and no significant trend: no absorption correction; data merged using *SHELX76* (Sheldrick, 1976) to give 862 unique reflections with $R_{\text{int}} = 0.05$; h , k , l range -9 to 9, 0 to 10 and 0 to 20; one reflection with a high F_c/F_o ratio (002), possibly due to extinction, removed; structure solved by direct methods with *SHELXS86* (Sheldrick, 1985), non-H atoms refined by least squares (F magnitudes) with anisotropic thermal parameters; all six H atoms found from difference Fourier syntheses, refined with isotropic thermal parameters; $R = 0.052$, $wR = 0.06$; for final cycle maximum shift/e.s.d. = 0.245, average = 0.040; $w = [\sigma^2(F) + 0.005349F^2]^{-1}$; 105 parameters;

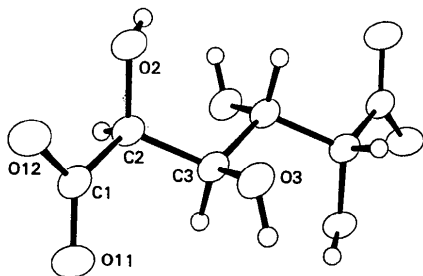


Fig. 1. x -axis projection of the anion showing the numbering scheme. Drawn using *ORTEP* (Johnson, 1965).

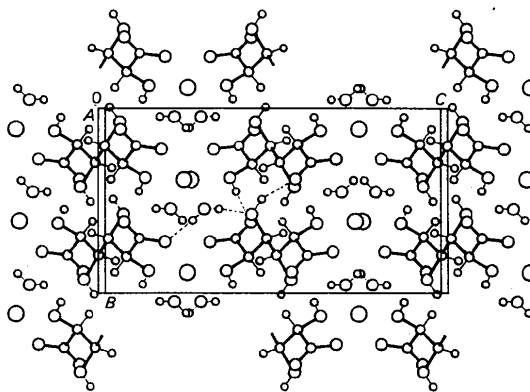


Fig. 2. Diagram showing the packing in the unit cell. Drawn using *PLUTO* (Motherwell & Clegg, 1978).