Table 2. *Selected bond distances* (Å), *bond angles* (°), *and their e.s.d.'s*

	(I)	(II)
Os–Br,Cl(1)	2·495 (2)	2·340 (5)
Os–Br,Cl(2)	2·490 (2)	2·334 (6)
Os–Br,Cl(3)	2·486 (2)	2·330 (5)
P–C(1)	1·79 (2)	1·82 (3)
P–C(7)	1·80 (2)	1·85 (3)
P–C(13)	1·78 (2)	1·91 (4)
P–H(1)	1·5 (1)	
Br,Cl(3)–Os–Br,Cl(2)	90·89 (6)	90·6 (2)
Br,Cl(3)–Os–Br,Cl(1)	90·25 (6)	90·3 (2)
Br,Cl(2)–Os–Br,Cl(1)	89·36 (6)	89·5 (2)
C(13)–P–C(1)	111·9 (8)	115 (1)
C(13)–P–C(7)	109 (1)	111 (2)
C(1)–P–C(7)	114·3 (9)	111 (1)
H(1)–P–C(1)	106 (6)	
H(1)–P–C(7)	116 (6)	
H(1)–P–C(13)	100 (6)	

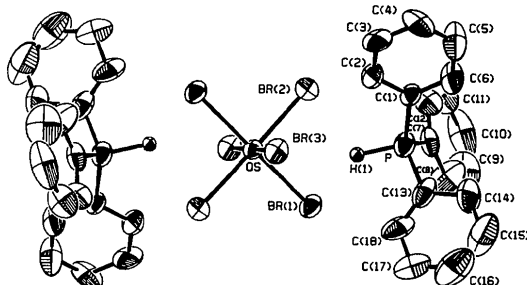Fig. 2. Stereoview of the molecular packing.

Fig. 1. Molecular structure and numbering scheme for compound (I); thermal ellipsoids at the 50% probability level.

It is interesting to note that the *a* and *c* axial dimensions of (I) are significantly larger than those of (II) while the *b* dimension remains essentially constant. Examination of the molecular packing, as shown by Fig. 2, indicates that the larger $OsBr_6^{2-}$ ions are responsible for the lattice expansion in the *a* and *c* directions; however,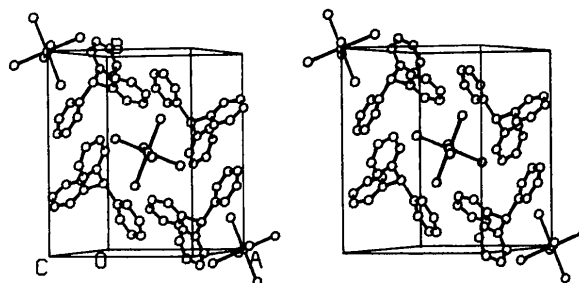 the *b* dimension appears to be controlled by the end-to-end packing of the phosphonium ions and thus remains constant.

Hexahaloosmium ions in both lattices are essentially octahedral, exhibiting only slight differences in bond distances and angles. Bond distances and angles for the triphenylphosphonium ions are regular with the exception of one C–P–C angle in each compound [114·3 (9)° for (I), 115 (1)° for (II)], which deviates significantly from the expected tetrahedral geometry.

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Structure of Bis(2-acetylcyclohexanonato)copper(II)

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Abstract. [Cu(C$_8$H$_{11}$O$_2$)$_2$], $M_r = 341·9$, orthorhombic, *Pbca*, $a = 15·273$ (7), $b = 6·369$ (2), $c = 15·687$ (6) Å, $V = 1526$ (1) Å^3, $Z = 4$, $D_x = 1·486$ g cm^{-3}, λ(Mo Kα) = 0·71069 Å, $\mu = 14·45$ cm^{-1}, $F(000) = 716$, $T = 293$ K, $R = 0·0516$ for 862 observed reflections. The complex exhibits a square planar coordination around the metal atom. The ligand acts as a bidentate monoanion. The chelate ring is nearly planar while the

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cyclohexane ring adopts a half-chair conformation. The Cu—O distances are: Cu—O(1) 1·896 (4) and Cu—O(2) 1·914 (3) Å. The C—O distances are: C(1)—O(1) 1·287 (6) and C(7)—O(2) 1·283 (6) Å.

Introduction. The structure of metal β-diketonate complexes is still a matter of study owing to the diversity of the coordination modes and stereochemistries involved in these compounds (Kawaguchi, 1986) and to their adduct-forming ability (Gatteschi, Laugier, Rey & Zanchini, 1987).

2-Acetylcyclohexanone forms metal complexes in which one of the O atoms of the β-diketone [O(1)] connects the chelate and cyclohexane rings generating a bicyclic system. The structure of the copper derivative of this diketone has been determined in order to observe the influence of the bicyclic structure on the bond parameters of the complex.

Experimental. Crystals by slow evaporation of an MeOH–CH$_2$Cl$_2$ solution. Crystal dimensions (mm): 0·17 × 0·3 × 0·4. Cell dimensions from angular measurements of 25 strong reflections in the range 5 < 2θ < 20°. Nicolet $R3m$ four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation; 2θ scan with variable scan rate from 4 to 30° min^{-1} and a scan width of 1·0°; 3 < 2θ < 50°; $h\,0\rightarrow18$; $k\,0\rightarrow7$; $l\,0\rightarrow18$. Two monitor reflections with constant intensity in each case. 1354 measured reflections with 862 unique [$I > 2\cdot5\sigma(I)$].

Lorentz and polarization corrections. Structure solved by Patterson heavy-atom method followed by Fourier synthesis. Metal atom at center of symmetry. Refinement by full-matrix least squares on F, anisotropic thermal parameters for all non-H atoms. H atoms in fixed idealized positions, $U = 0\cdot06$ Å^2; weighting scheme $[\sigma^2|F_o| + G|F_o|^2]^{-1}$, where σ is the e.s.d. based on counting statistics and G an adjustable variable; final $G = 0\cdot0015$; max. $\Delta\rho$ in final difference maps < 0·6 e Å^{-3}; max. $\Delta/\sigma = 0\cdot00$; $R = 0\cdot0493$; $wR = 0\cdot0516$; $S = 1\cdot148$. Solution, refinement and plots using $SHELXTL$ (Sheldrick, 1981) on a Nova computer, anomalous-scattering-factor values from $International\ Tables\ for\ X$-ray Crystallography (1974).

Discussion. Final positional parameters are shown in Table 1.* The molecular structure of the complex including the atom-numbering scheme is illustrated in Fig. 1. Associated bond parameters for the structure are presented in Table 2.

The metal atom sits on a crystallographic center of symmetry displaying square-planar coordination geometry. The ligand is coordinated as a bidentate monoanionic ligand with the chelate ring system in a nearly planar arrangement. Similar behavior has been observed in other copper diketonate complexes (Hon, Pfluger & Belford, 1966).

Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2 × 10^3)

	x	y	z	U_{eq}*
Cu	0	0	0	48 (1)
O(1)	150 (3)	−1591 (5)	−1011 (2)	59 (1)
O(2)	809 (2)	−1732 (5)	611 (2)	57 (1)
C(1)	642 (3)	−3210 (7)	−1119 (3)	45 (2)
C(2)	1134 (3)	−4209 (7)	−492 (3)	41 (2)
C(3)	1640 (4)	−6203 (8)	−709 (3)	56 (2)
C(4)	1391 (5)	−7168 (10)	−1562 (4)	78 (2)
C(5)	1354 (4)	−5555 (10)	−2253 (3)	68 (2)
C(6)	651 (4)	−3953 (9)	−2032 (3)	58 (2)
C(7)	1191 (3)	−3395 (8)	336 (3)	44 (2)
C(8)	1757 (4)	−4458 (8)	1001 (3)	61 (2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond angles* (°) *and bond lengths* (Å)

E.s.d.'s in the least significant digits are given in parentheses.

O(1)—Cu—O(2)	91·9 (1)	O(1)—Cu—O(1a)	180·0
O(2)—Cu—O(1a)	88·1 (1)	O(1)—Cu—O(2a)	88·1 (1)
O(2)—Cu—O(2a)	180·0	O(1a)—Cu—O(2a)	91·9 (1)
Cu—O(1)—C(1)	127·6 (3)	Cu—O(2)—C(7)	127·0 (3)
O(1)—C(1)—C(2)	126·0 (4)	O(1)—C(1)—C(6)	112·5 (4)
C(2)—C(1)—C(6)	121·6 (4)	C(1)—C(2)—C(3)	119·7 (4)
C(1)—C(2)—C(7)	121·3 (4)	C(3)—C(2)—C(7)	118·9 (4)
C(2)—C(3)—C(4)	114·0 (5)	C(3)—C(4)—C(5)	111·7 (5)
C(4)—C(5)—C(6)	108·8 (5)	C(1)—C(6)—C(5)	115·7 (4)
O(2)—C(7)—C(2)	126·0 (5)	O(2)—C(7)—C(8)	113·4 (4)
C(2)—C(7)—C(8)	120·5 (4)		

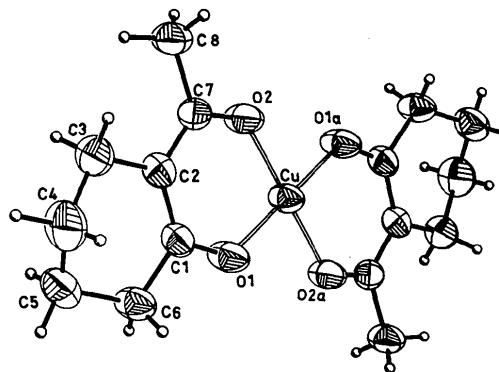|			
Cu—O(1)	1·896 (4)	Cu—O(2)	1·914 (3)
Cu—O(1a)	1·896 (4)	Cu—O(2a)	1·914 (3)
O(1)—C(1)	1·287 (6)	O(2)—C(7)	1·283 (6)
C(1)—C(2)	1·392 (7)	C(1)—C(6)	1·507 (7)
C(2)—C(3)	1·525 (7)	C(2)—C(7)	1·401 (7)
C(3)—C(4)	1·520 (8)	C(4)—C(5)	1·494 (8)
C(5)—C(6)	1·522 (8)	C(7)—C(8)	1·514 (7)

Fig. 1. The molecular conformation of [Cu(C$_8$H$_{11}$O$_2$)$_2$] showing atom numbering. The thermal ellipsoids are drawn at the 50% probability level.

The Cu—O bond lengths are of the same order of magnitude as those found in other similar copper complexes (Carmichael, Steinrauf & Belford, 1965; Hon, Pfluger & Belford, 1966). The C—O bonds have been reported to show some important differences depending on whether the group bonded to the C atom is capable of conjugation with the chelate ring. Nevertheless, the values observed in this case are similar to those described in cases where alkyl groups are bonded to the C atom and both values are essentially the same.

The remaining bonds of the chelate ring, C(1)—C(2) and C(2)—C(7), have bond lengths of 1·392 (7) and 1·401 (7) Å, significantly shorter than the other C—C distances in the complex (av. 1·513 Å) suggesting an increase in the order of the bond. This must be caused by an electronic delocalization within the chelate ring. The unsaturated character of the C(1)—C(2) bond is further supported by the half-chair conformation of the cyclohexane ring, similar to that observed in cyclohexene (Eliel, Allinger, Angyol & Morrison, 1967).

There are no short intermolecular interactions between the C(3) and Cu atoms like those described for copper acetylacetonate [Dahl; unpublished results referred to by Robertson & Truter (1967)], copper ethylacetylacetonate (Hall, McKinnon & Waters, 1966) and copper 1-phenyl-1,3-butanedionate (Hon, Pfluger & Belford, 1966). It has been proposed that the presence of an H atom on the γ-C atom is necessary for such an interaction to take place (Robertson & Truter, 1967).

The complex reported herein is almost completely planar, with the atoms forming the chelate ring and C(3) and C(6) showing a maximum deviation of 0·10 Å from the plane while atoms C(4) and C(5) deviate about equally in opposite directions from the plane.

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Structure of Bis[*N,N*-bis(1-pyrazolylmethyl)amine]nickel(II) Bis(tetrafluoroborate)

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Abstract. [Ni(C$_8$H$_{11}$N$_5$)$_2$](BF$_4$)$_2$, $M_r = 586\cdot71$, orthorhombic, $P2_12_12_1$, $a = 10\cdot255$ (1), $b = 13\cdot734$ (2), $c = 17\cdot995$ (1) Å, $V = 2534\cdot5$ (5) Å^3, $Z = 4$, $D_x = 1\cdot538$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 18\cdot8$ cm^{-1}, $F(000) = 1192$, $T = 295$ K, $R = 0\cdot076$ for 1845 observed reflections with $I > 2\cdot5\sigma(I)$. The BF$_4$ anions show some disorder. The Ni^{II} ion is surrounded by four azole nitrogens at 2·02 (1) to 2·07 (1) Å and two amine nitrogens at 2·17 (1) and 2·182 (9) Å in an almost octahedral geometry. The two ligands are in a *fac* arrangement. The cation has non-crystallographic twofold axial symmetry. The ligands are weakly hydrogen bonded through the H atoms on the amine nitrogens to the fluoroborate anions with N···F distances of 2·82 (1) and 2·97 (1) Å.

Introduction. Several transition-metal compounds of tris(3,5-dimethyl-1-pyrazolylmethyl)amine have been

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