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METAL π-COMPLEXES OF CYCLOOCTATETRAENES

IV *. SYNTHESIS AND CRYSTAL STRUCTURE OF BENZOCYCLOOCTATETRAENESILVER(I) PERCHLORATE: AN EXAMPLE OF ARENE AND OLEFIN LIGANDS COORDINATED SIMULTANEOUSLY TO A SILVER(I) ION

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Summary

A 1:1 adduct of benzocyclooctatetraene and silver perchlorate, $C_{12}H_{10} \cdot AgClO_4$, **1**, has been isolated and characterized by X-ray crystallography. Complex **1** crystallizes in space group $P2_1/c$ with $Z = 4$, $a = 8.045(2)$, $b = 17.412(4)$, $c = 8.501(1)$ Å, β 91.13(2)°, and $V = 1190.6(4)$ Å^3. The structure has been refined to $R_F = 0.051$ and $R_{wF} = 0.072$ for 2165 observed Mo-K_α data and 154 variables. The perchlorate group exhibits two unequally populated orientations about the central chlorine atom. The three moieties in compound **1** are interconnected by ionic (metal–oxygen) and covalent (metal–olefin and metal–aromatic) bonds to form layers parallel to the *bc* plane. The coordination about the silver ion may be described as distorted trigonal bipyramidal, the ligand sites being filled by two non-adjacent double bonds of a benzocyclooctatetraene molecule, one aromatic C–C bond of a neighbouring benzocyclooctatetraene molecule, and two oxygen atoms belonging to different perchlorate groups. This is the first reported instance of simultaneous donor π-bonding from aromatic and olefinic ligands to a silver ion.

Introduction

The interaction of olefins with silver salts in solution has been thoroughly studied and frequently reviewed [2,3,4]. The complexes formed by alicyclic di- and oligo-

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olefins with $AgNO_3$, $AgClO_4$, and $AgBF_4$ are generally more stable than those of the corresponding aliphatic olefins and often isolable as crystalline solids. In the various adducts hitherto investigated by X-ray crystallography, the ligands have been found to have a distorted trigonal bipyramidal, distorted tetrahedral, or trigonal planar (rarely) arrangement about the silver ion [4]. Typical silver-trigonal carbon distances involving the coordinated double bond lie in the range 2.3–2.6 Å, but longer contacts representing weaker interactions are also found in several instances [1,5,6,7].

The existence of donor-acceptor complexes between aromatic hydrocarbons and silver salts was known over six decades ago [8], but the first structural study on benzene · $AgClO_4$ was carried out nearly forty years later [9]. From 1963 onwards [10], Amma and coworkers conducted a systematic study of the crystal and molecular structures of a number of metal ion-aromatic complexes covering a wide range of stoichiometries [11,12]. Of the many aromatic molecules scrutinized, acenaphthylene and indene presented opportunities for observing silver–arene interactions as well as silver–olefin interactions with the localized double bond in the five-membered ring. As it turned out, the silver ion in acenaphthylene · $AgClO_4$ does not bind to the double bond in the five-membered ring, but rather to the same portions of the fused six-membered rings as in acenaphthene · $AgClO_4$ [13]. X-ray analysis of the indene-silver perchlorate adduct [14] showed that it consists of discrete (indene · $AgClO_4)_2$ dimeric units, with one silver ion bound only to the olefin portions of two indene molecules and the other only to the aromatic rings. The silver–aromatic and silver–olefin interactions are thus mutually exclusive in this indene complex.

As part of a systematic study of the synthesis and structural chemistry of benzannelated eight-membered carbocycles, we have prepared a 1:1 adduct of benzocyclooctatetraene and silver perchlorate, $C_{12}H_{10} \cdot AgClO_4$, **1**, and established its structure by X-ray crystallography. To our knowledge this is the first reported example in which the silver ion π-bonds simultaneously to both aromatic and olefinic ligands.

Experimental

Preparation of benzocyclooctatetraene-silver perchlorate, 1

Benzocyclooctatetraene, **2**, m.p. 44–45°C, was synthesized according to the literature method [15] and recrystallized from ethanol. Equimolar quantities (4 mmol) of reagent grade $AgClO_4$ (Fisher) and compound **2** were each dissolved in a minimum amount of distilled toluene. The two solutions were then mixed thoroughly in a loosely stoppered Erlenmeyer flask, and transparent rectangular plates of **1** were deposited after a few days. Compound **1** decomposes fairly quickly in air and crystals must be freshly prepared prior to diffraction use.

X-ray data collection and structure determination

A selected crystal of **1** (0.28 × 0.36 × 0.40 mm) was covered with petroleum jelly, lodged in a 0.5 mm diameter Lindemann glass capillary and sealed with epoxy resin. It was then centered on a Nicolet R3m automatic diffractometer, and determination of the crystal class, orientation matrix, and accurate unit cell parameters were performed in a standard manner [16]. Intensity data were collected at 22°C using graphite-monochromatized Mo-K_α radiation, a scintillation counter, and a pulse height analyser (see details summarized in Table 1). The crystal remained stable

TABLE 1

DATA COLLECTION AND PROCESSING PARAMETERS

Molecular formula	$C_{12}H_{10}\cdot AgClO_4$
Molecular weight	361.53
Cell constants	$a = 8.045(2)$, $b = 17.412(4)$, $c = 8.501(1)$ Å, $\beta = 91.13(2)°$, $V = 1190.6(4)$ Å^3, $Z = 4$
Density (calcd.)	2.017 g cm^{-3}
Space group	$P2_1/c$ (C^5_{2h}, No.14)
Radiation	graphite-monochromatized Mo-K_α, $\lambda = 0.71069$ Å
Absorption coefficient	19.04 cm^{-1}
Mean μr	0.324
Transmission factors	0.381–0.433
Scan type and speed	θ–2θ; 2.55–8.37 deg min^{-1}
Scan range	1° below $K_{\alpha 1}$ to 1° above $K_{\alpha 2}$
Background counting	stationary counts for one-half of scan time at each end of scan
Collection range	$h, k, \pm l$; $2\theta_{max} = 54°$
Unique data measured	2498
Observed data with $\|F\| > 3\sigma(\|F\|)$	2165
Number of variables	154
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.051
Weighting scheme	$w = [\sigma^2(\|F\|)+0.0005\|F\|^2]^{-1}$
$R_w = \{\Sigma w(\|F_o\|-\|F_c\|)^2/\Sigma w\|F_o\|^2\}^{1/2}$	0.072
Goodness of fit	2.345

throughout the diffraction experiment as three standard reflections monitored every 125 data measurements showed only random deviations within ± 1.5% of their mean values. The systematic absences: $h0l$ with l odd and $0k0$ with k odd indicated space group $P2_1/c$. Absorption correction was applied using an empirical method based on a pseudoellipsoidal treatment of reflection intensity measurements at different azimuthal (ψ) angles [17,18,19]. Redundant and equivalent reflection data were averaged and converted to unscaled $|F_o|$ values following corrections for Lorentz and polarization factors.

Normalized structure factors were generated from $|F_o|$ values and the statistical distributions of the $|E|$ values were consistent with those expected for a centrosymmetric crystal. An initial attempt to solve the structure by direct methods yielded the positions of the silver and chlorine atoms and six of the twelve carbon atoms in the asymmetric unit. The remaining carbon and oxygen atoms were located in successive difference Fourier syntheses, and it soon became apparent that the perchlorate group has two preferred orientations of unequal population centered at the chlorine atom. In the structure refinement, the silver, chlorine, and carbon atoms were varied anisotropically. The eight highest electron-density peaks lying 1.35–1.50 Å from the chlorine atom were fitted to the vertices of two regular tetrahedra, interatomic distance constraints of Cl–O = 1.42(1) and O ··· O = $2(2/3)^{1/2} \times 1.42(1)$ Å being applied by the method of observational equations [20]. The site occupancy factors of the tetrahedral groups O(1)–O(4) and O(5)–O(8) were varied as g and $(1-g)$, respectively, and all oxygen atoms were tied to the same isotropic thermal parameter. The ten hydrogen atoms were generated on the basis of sp^2 hybridization of their

(Continued on p. 418)

TABLE 2

POSITIONAL AND THERMAL PARAMETERS[a] (Å^2) FOR BENZOCYCLOOCTATETRAENE-SILVER PERCHLORATE 1:1 COMPLEX, 1

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}	U_{eq}
Ag	0.09553(5)	0.12951(3)	0.30208(6)	0.0346(3)	0.0649(4)	0.0577(3)	0.0133(2)	0.0027(2)	0.0004(2)	0.0524(2)
C(1)	−0.1357(7)	0.1566(3)	0.4928(7)	0.043(3)	0.039(3)	0.045(3)	−0.006(2)	0.003(2)	−0.003(2)	0.042(2)
C(2)	−0.1782(7)	0.2026(3)	0.3719(8)	0.046(2)	0.033(3)	0.070(4)	0.009(3)	0.011(3)	−0.001(2)	0.050(2)
C(3)	−0.2970(7)	0.1850(4)	0.2467(8)	0.042(3)	0.047(3)	0.069(4)	0.025(3)	0.003(3)	0.005(3)	0.053(2)
C(4)	−0.2929(8)	0.1268(4)	0.1504(8)	0.038(3)	0.076(5)	0.057(4)	0.031(3)	−0.008(3)	−0.003(3)	0.057(2)
C(5)	−0.1677(7)	0.0648(4)	0.1500(7)	0.043(3)	0.071(4)	0.037(3)	−0.001(3)	−0.001(2)	−0.001(3)	0.050(2)
C(6)	−0.1258(6)	0.0206(3)	0.2709(6)	0.038(3)	0.045(3)	0.038(3)	−0.003(2)	0.002(2)	0.005(2)	0.040(2)
C(7)	−0.2063(6)	0.0188(3)	0.4274(6)	0.030(2)	0.034(2)	0.035(2)	0.004(2)	−0.002(2)	0.003(2)	0.033(1)
C(8)	−0.2106(6)	0.0811(3)	0.5292(6)	0.029(2)	0.042(3)	0.036(2)	0.003(2)	−0.001(2)	0.002(2)	0.036(1)
C(9)	−0.2848(7)	0.0723(4)	0.6760(7)	0.043(3)	0.049(3)	0.041(3)	−0.004(2)	−0.002(2)	0.006(2)	0.044(2)
C(10)	−0.3580(7)	0.0050(4)	0.7190(7)	0.037(3)	0.065(4)	0.048(3)	0.013(3)	0.013(2)	0.004(3)	0.050(2)
C(11)	−0.3562(6)	−0.0578(3)	0.6179(7)	0.032(2)	0.043(3)	0.060(3)	0.017(3)	−0.005(2)	−0.007(2)	0.045(2)
C(12)	−0.2772(6)	−0.0518(3)	0.4745(7)	0.032(2)	0.037(3)	0.052(3)	0.001(2)	−0.010(2)	−0.001(2)	0.040(2)
Cl	0.25151(18)	0.31575(8)	0.43629(16)	0.0568(8)	0.0420(7)	0.0418(7)	−0.0055(6)	0.0006(6)	−0.0080(6)	0.0469(4)

Atom	x	y	z	site occupancy	U_{iso}	Atom	x	y	z	U_{iso}
O(1)	0.2530(11)	0.2355(4)	0.4562(11)	0.642(6)	0.093(1)	H(1)	−0.0491	0.1748	0.5628	0.060
O(2)	0.2676(12)	0.3523(5)	0.5862(9)	0.642(6)	0.093(1)	H(2)	−0.1241	0.2516	0.3676	0.060
O(3)	0.1024(10)	0.3411(6)	0.3605(11)	0.642(6)	0.093(1)	H(3)	−0.3880	0.2202	0.2331	0.060
O(4)	0.3927(10)	0.3396(5)	0.3468(10)	0.642(6)	0.093(1)	H(4)	−0.3797	0.1242	0.0714	0.060
O(5)	0.1591(17)	0.3024(8)	0.5778(13)	0.358(6)	0.093(1)	H(5)	−0.1122	0.0553	0.0530	0.060
O(6)	0.3563(17)	0.3787(6)	0.4507(18)	0.358(6)	0.093(1)	H(6)	−0.0338	−0.0138	0.2574	0.060
O(7)	0.1317(15)	0.3281(9)	0.3093(14)	0.358(6)	0.093(1)	H(9)	−0.2824	0.1147	0.7480	0.060
O(8)	0.3359(18)	0.2464(6)	0.4010(17)	0.358(6)	0.093(1)	H(10)	−0.4124	0.0020	0.8183	0.060
						H(11)	−0.4075	−0.1052	0.6479	0.060
						H(12)	−0.2706	−0.0955	0.4060	0.060

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[a] The anisotropic temperature factor takes the form: $\exp[-2\pi^2 \sum_i \sum_j U_{ij} h_i h_j a_i^{\star} \cdot a_j^{\star}]$; U_{eq} is the equivalent isotropic thermal parameter calculated as one-third of the trace of the U matrix; the isotropic temperature factor takes the form: $\exp[-8\pi^2 U_{iso} \sin^2\theta/\lambda^2]$.

TABLE 3

BOND LENGTHS (Å), BOND ANGLES (deg), AND TORSION ANGLES (deg)[a,b]

(i) Coordination around Ag^I ion			
Ag–C(1)	2.536(6)	Ag–C(2)	2.622(6)
Ag–C(5)	2.705(6)	Ag–C(6)	2.612(6)
Ag–C(11′)	2.522(5)	Ag–C(12′)	2.732(5)
Ag–O(1)	2.581(8)	Ag–O(2*)	2.342(8)
Ag–O(5*)	2.311(12)		
O(1)–Ag–C(5)	155.8(2)	O(1)–Ag–C(6)	153.8(2)
O(1)–Ag–C(1)	84.3(2)	O(1)–Ag–C(2)	86.8(2)
O(1)–Ag–C(11′)	79.6(2)	O(1)–Ag–C(12′)	75.6(2)
O(1)–Ag–O(2*)	90.5(3)	O(1)–Ag–O(5*)	86.4(4)
C(5)–Ag–C(1)	78.8(2)	C(6)–Ag–C(1)	72.1(2)
C(5)–Ag–C(2)	70.0(2)	C(6)–Ag–C(2)	78.5(2)
C(5)–Ag–C(11′)	124.1(2)	C(6)–Ag–C(11′)	103.2(2)
C(5)–Ag–C(12′)	121.9(2)	C(6)–Ag–C(12′)	93.7(2)
C(5)–Ag–O(2*)	98.7(2)	C(6)–Ag–O(2*)	115.5(2)
C(5)–Ag–O(5*)	90.2(3)	C(6)–Ag–O(5*)	116.8(3)
C(1)–Ag–C(11′)	122.4(2)	C(2)–Ag–C(11′)	151.3(2)
C(1)–Ag–C(12′)	92.1(2)	C(2)–Ag–C(12′)	121.5(2)
C(1)–AG–O(2*)	158.2(3)	C(2)–Ag–O(2*)	128.6(3)
C(1)–Ag–O(5*)	127.5(4)	C(2)–Ag–O(5*)	98.0(4)
C(11′)–Ag–O(2*)	77.1(3)	C(12′)–Ag–O(2*)	107.2(2)
C(11′)–Ag–O(5*)	106.3(4)	C(12′)–Ag–O(5*)	134.7(3)
(ii) Disordered ClO_4^- groups			
Cl–O(1)	1.408(7)	Cl–O(5)	1.445(12)
Cl–O(2)	1.428(8)	Cl–O(6)	1.387(12)
Cl–O(3)	1.421(8)	Cl–O(7)	1.449(12)
Cl–O(4)	1.441(8)	Cl–O(8)	1.420(12)
O(1)–Cl–O(2)	109.6(5)	O(5)–Cl–O(6)	112.0(8)
O(1)–Cl–O(3)	111.6(5)	O(5)–Cl–O(7)	107.4(7)
O(1)–Cl–O(4)	110.2(5)	O(5)–Cl–O(8)	107.2(8)
O(2)–Cl–O(3)	109.1(5)	O(6)–Cl–O(7)	110.1(8)
O(2)–Cl–O(4)	106.5(5)	O(6)–Cl–O(8)	113.5(8)
O(3)–Cl–O(4)	109.8(5)	O(7)–Cl–O(8)	106.4(8)
(iii) Benzocyclooctatetraene molecule			
C(8)–C(9)	1.403(7)[1.389(2)]	C(7)–C(12)	1.416(7)[1.396(2)]
C(9)–C(10)	1.363(9)[1.378(3)]	C(11)–C(12)	1.390(8)[1.371(2)]
C(10)–C(11)	1.391(9)[1.376(3)]	C(7)–C(8)	1.388(7)[1.399(2)]
C(1)–C(8)	1.482(8)[1.481(2)]	C(6)–C(7)	1.491(7)[1.476(2)]
C(1)–C(2)	1.341(8)[1.330(3)]	C(5)–C(6)	1.323(8)[1.321(3)]
C(2)–C(3)	1.449(9)[1.445(3)]	C(4)–C(5)	1.478(9)[1.453(3)]
C(3)–C(4)	1.304(9)[1.316(3)]		
C(7)–C(8)–C(9)	119.0(5)[118.4(2)]	C(8)–C(7)–C(12)	119.1(5)[119.0(1)]
C(8)–C(9)–C(10)	121.7(5)[122.0(2)]	C(7)–C(12)–C(11)	120.4(5)[121.3(2)]
C(9)–C(10)–C(11)	120.0(5)[119.3(2)]	C(10)–C(11)–C(12)	119.6(5)[120.0(2)]
C(1)–C(8)–C(9)	117.7(5)[116.9(1)]	C(6)–C(7)–C(12)	117.0(5)[117.7(2)]
C(7)–C(8)–C(1)	123.3(5)[124.0(1)]	C(6)–C(7)–C(8)	123.8(5)[123.8(1)]
C(8)–C(1)–C(2)	126.2(5)[127.4(2)]	C(5)–C(6)–C(7)	126.7(5)[127.7(2)]
C(1)–C(2)–C(3)	126.3(5)[127.8(2)]	C(4)–C(5)–C(6)	125.9(6)[127.3(2)]
C(2)–C(3)–C(4)	126.9(6)[126.7(2)]	C(3)–C(4)–C(5)	126.5(6)[126.5(2)]
C(6)–C(7)–C(8)–C(1)	0.2(8)[1.4(3)]	C(2)–C(3)–C(4)–C(5)	1.5(12)[−0.1(3)]
C(7)–C(8)–C(1)–C(2)	61.1(8)[57.8(3)]	C(3)–C(4)–C(5)–C(6)	54.6(10)[55.9(3)]
C(8)–C(1)–C(2)–C(3)	−4.8(10)[−4.1(3)]	C(4)–C(5)–C(6)–C(7)	6.2(10)[3.3(3)]
C(1)–C(2)–C(3)–C(4)	−57.4(10)[−55.0(3)]	C(5)–C(6)–C(7)–C(8)	−62.1(8)[−59.4(3)]

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[a] Symmetry transformations: ′ $-x, -y, 1-z$; * $x, \frac{1}{2}-y, -\frac{1}{2}+z$. [b] Corresponding values for uncomplexed benzocyclooctatetraene [32] are enclosed in square brackets for comparison.

respective parent carbon atoms, and were assigned a fixed isotropic temperature factor of 0.06 Å^2. The final difference map showed residual extrema at 1.08 to -1.03 e Å^{-3}, with the three highest peaks lying in the neighbourhood of the silver and chlorine atoms.

All computations were performed on a Data General Corporation Nova 3/12 minicomputer with the SHELXTL program package [17,21]. Analytic expressions [22] of neutral-atom scattering factors [23] were employed, and anomalous dispersion corrections [24] were incorporated. Blocked-cascade least-squares refinement [21,25] of 154 parameters converged to the R indices listed in Table 1.

Table 2 lists the final positional and thermal parameters for all atoms in the asymmetric unit. Bond lengths, bond angles, and torsion angles are given in Table 3. A table of observed and calculated structure factor is available from the authors upon request.

Results and discussion

Figure 1 shows an ORTEP plot with labelling of all atoms in the asymmetric unit. The perchlorate group exhibits orientational disorder, and linkages between oxygen atoms belonging to the less populated orientation and other atoms are indicated by broken bonds. With the perchlorate group in its principal orientation, the configuration of the ligands about the metal center may be described as distorted trigonal bipyramidal *. The silver ion is coordinated by two non-adjacent double bonds of a benzocyclooctatetraene molecule, one aromatic C–C bond from a neighbouring benzocyclooctatetraene molecule, and two oxygen atoms belonging to different perchlorate groups; atom O(1) and the center of the C(5)–C(6) olefinic ligand occupy the axial positions of the trigonal bipyramid. From a structural point of view, complex **1** is more closely related to the cyclooctatetraene adducts of silver nitrate [1,26,27], in which the metal ions have pentagonal bipyramidal coordination, than silver-aromatic complexes with coordination numbers of three [12,14] or four [13,28,29,30,31] for the metal ion.

The silver–olefin bonds [2.536(6)–2.705(6) Å] lie close to the upper end of the range 2.3–2.6 Å [4] of commonly observed values. The silver ion is asymmetrically located relative to the aromatic ligand, with a 'short' bond at 2.522(5) Å and a significantly longer one at 2.732(5) Å, in accord with the well-established asymmetry in metal–carbon distances characteristic of most metal ion-aromatic complexes regardless of stoichiometry, stereochemistry, nature of anion, and crystal packing [14,28]. It is interesting to note that the silver–olefin and silver–aromatic interactions are approximately equal in magnitude in the present complex. In contrast to this, binding of one silver species to the olefin part of the indene molecule [Ag–C = 2.36(2), 2.46(2) Å] in indenesilver perchlorate [14] is much stronger than that between the other silver ion and the aromatic portion [Ag–C = 2.47(2), 2.76(2) Å]. It would appear, therefore, that the generalization that olefins are stronger π bases than aromatics in silver perchlorate complexes [14] needs modification when both types of ligands coordinate to the same metal center. The Ag–O(1) distance of

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* The silver ion would be four-coordinate if only the energetically less favored orientation of the perchlorate group is considered.

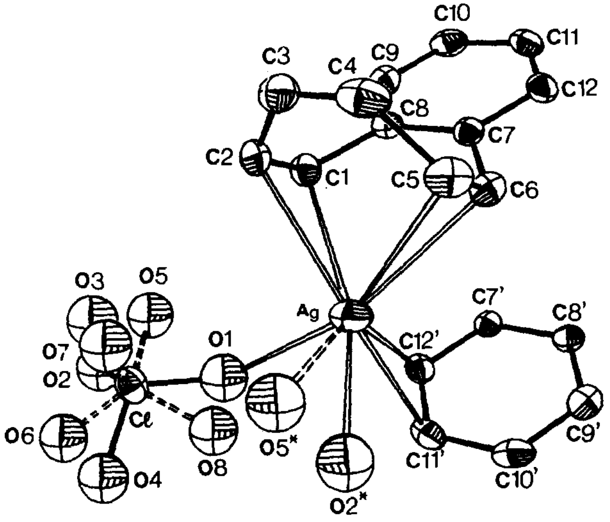

Fig. 1. A perspective view of the coordination around a silver ion in $C_{12}H_{10}\cdot AgClO_4$, **1**, showing the atom labelling scheme and thermal ellipsoids at the 30% probability level. Metal–ligand atom interactions are represented by open bonds, and linkages involving oxygen atoms of the less populated orientation of the perchlorate ion by broken bonds.

2.581(8) Å is intermediate in the range 2.44 to 2.66 Å [14] for several silver perchlorate-aromatic complexes, but the Ag–O(2*) and Ag–O(5*) interactions [2.342(8) and 2.311(12) Å, respectively] are among the shortest of such distances known and undoubtedly play a major role in stabilizing the crystal packing [12].

The benzocyclooctatetraene moiety, bound to a silver ion via two non-adjacent

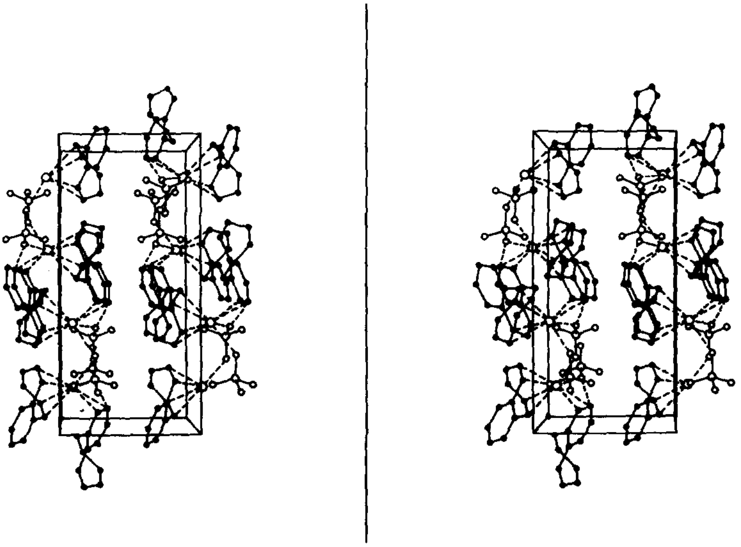

Fig. 2. Stereo drawing showing the packing arrangement in **1**, with metal–ligand interactions represented by dotted lines. The less favored orientation of the two-fold disordered perchlorate group has been omitted for the sake of clarity. The origin of the unit cell lies at the lower left corner, with *a* pointing from left to right, *b* upwards, and *c* towards the reader.

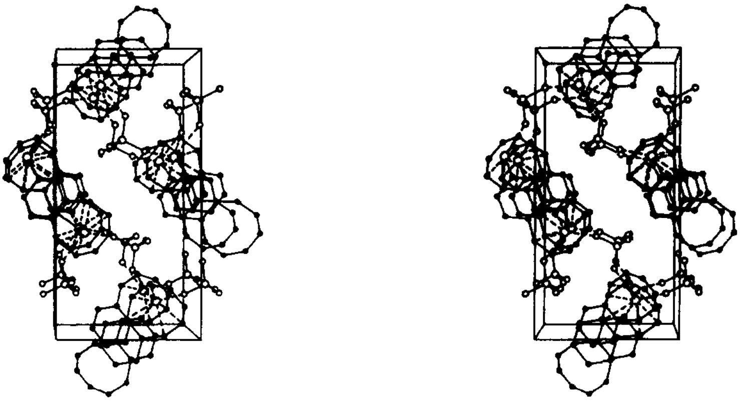

Fig. 3. Stereo view of the crystal structure of **1** normal to the layers. The unit-cell origin lies at the upper left corner, with *a* towards the reader, *b* downwards, and *c* from left to right.

olefinic ligands and to another via the aromatic C(11)–C(12) bond, has dimensions similar to those of the uncomplexed molecule [32], except that the eight-membered ring is somewhat flattened and bonds involving C(11) and C(12) are slightly lengthened (Table 3).

The silver and perchlorate ions generated by the *c* glide are linked alternately to form zigzag chains propagating in the *c* direction. The benzocyclooctatetraene molecules are stacked one above the other in columns parallel to *c*, each molecule being bound to two silver ions belonging to neighbouring chains related by a 2_1 screw axis. The crystal structure (Fig. 2) therefore consists of zigzag chains and columns cross-linked into infinitely extended layers parallel to the *bc* plane, the interaction between layers being of the Van der Waals type. Figure 3 shows another view of the structure normal to the layers.

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