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METAL π-COMPLEXES OF CYCLOOCTATETRAENES

III. * X-RAY CRYSTAL STRUCTURE OF THE 2:3 ADDUCT OF CYCLOOCTATETRAENE AND SILVER NITRATE

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Summary

X-ray analysis of $2C_8H_8 \cdot 3AgNO_3$ revealed a complex three-dimensional polymeric structure in which the two cyclootatetraene ligands partition the three silver(I) ions in the asymmetric unit. The coordination about each metal center is distorted trigonal bipyramidal, the ligand sites being filled by the olefinic π-bonds and the nitrato groups in monodentate and/or bidentate modes. Crystals are orthorhombic, space group *Pbcn*, with $a = 26.026(5)$, $b = 10.756(2)$, $c = 14.379(2)$ Å, and $Z = 8$. The structure was refined anisotropically to $R = 0.050$ for 2941 observed Mo-K_α data.

Introduction

It has been known for over three decades that cyclooctatetraene (C_8H_8, COT) reacts with aqueous silver nitrate to give three crystalline adducts of compositions $2COT \cdot AgNO_3$, $COT \cdot AgNO_3$, and $2COT \cdot 3AgNO_3$ [1]. These addition compounds have proved to be useful derivatives for the purification of COT, which can be conveniently regenerated from them. In 1958, Mathews and Lipscomb conducted an X-ray crystallographic analysis of the 1:1 adduct and provided the first example of π-interaction between a cyclic polyolefin and a heavy metal ion [2,3]. A recent reinvestigation of this structure using new Mo-K_α diffractometer data has rectified all the unsatisfactory aspects [4] of the previous work and elucidated the intricacies of all the silver(I)–ligand interactions and ligand distortions [5]. A parallel X-ray study of the 2:1 adduct has shown that it exists as discrete pentacoordinate $(C_8H_8)_2AgNO_3$ entities of C_2 molecular symmetry in the crystalline state [6]. In the present work, we prepared single crystals of the 2:3 adduct and carried out an X-ray analysis to determine the modes of coordination of the COT and nitrato groups around the presumably different silver(I) ions.

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* For part II see ref. 6.

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Experimental

Preparation of $2C_8H_8 \cdot 3AgNO_3$

A crystalline sample of the 1:1 adduct, $C_8H_8 \cdot AgNO_3$, was prepared according to the procedure previously described [5]. The supernatant liquid was removed by decantation, and the crystals were redissolved by the addition of a minimum amount of ethanol. Upon standing overnight, the solution deposited pale green crystals of the 2:3 adduct in the form of stout prisms.

Data collection

A single crystal cut to the shape of a cube (edge ~ 0.4 mm) was covered with petroleum jelly and sealed in a 0.5 mm diameter Lindemann glass capillary. Unit-cell dimensions were determined from a least-squares fit of seventeen high-angle reflections measured on a Nicolet R3m diffractometer system. Intensities were recorded in the θ–2θ mode with variable scan speeds, and three standards monitored every 125 reflections showed only random deviations within ±2% of their mean values. Besides Lorentz and polarization factors, absorption correction was applied using an empirical method based on a pseudo-ellipsoidal treatment of reflection intensity measurements at different azimuthal angles [7]. Details of the experimental procedure and structure refinement are summarized in Table 1.

Structure solution and refinement

The positions of the three silver(I) ions were determined by direct methods [7]. The remaining non-hydrogen atoms were located in successive difference Fourier

(Continued on p. 336)

TABLE 1

DATA COLLECTION AND PROCESSING PARAMETERS

Molecular formula	$2C_8H_8 \cdot 3AgNO_3$
Molecular weight	717.93
Cell constants	$a = 26.026(5)$, $b = 10.756(2)$, $c = 14.379(2)$Å, $V = 4025.2(9)$Å^3, $Z = 8$
Density (calcd.)	$\rho = 2.369(2)$ g cm^{-3}
Space group	*Pbcn* (No. 60)
Radiation	graphite-monochromatized Mo-K_α, $\lambda = 0.71069$ Å
Mean μr	0.73
Transmission factors	0.138 to 0.175
Scan type and speed	θ–2θ; 3.91 to 14.65 deg min^{-1}
Scan range	1° below $K\alpha_1$ to 1° above $K\alpha_2$
Background counting	stationary counts for 1/2 of scan time at each end of scan
Collection range	h, k, l; $2\theta_{max} = 60°$
Unique data measured	4379
Observed data with $\|F\| > 3\sigma(\|F\|)$	2941
Number of variables	283
Extinction parameter ϵ	6.2×10^{-7}
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.050
Weighting scheme	$w = [\sigma^2(\|F_o\|) + 0.0005\|F_o\|^2]^{-1}$
$R_w = (\Sigma w(\|F_o\|-\|F_c\|)^2/\Sigma w\|F_o\|^2)^{1/2}$	0.059
Goodness of fit	1.686

TABLE 2

FRACTIONAL ATOMIC COORDINATES AND THERMAL PARAMETERS[a] (Å^2 × 10^4 for Ag; × 10^3 for the other atoms)

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}	U_{eq}[b]
Ag(1)	0.19463(3)	0.24821(7)	0.42158(4)	683(4)	893(5)	484(3)	−144(4)	−39(3)	−35(4)	687(3)
Ag(2)	0.15185(2)	0.20808(7)	0.07855(4)	519(3)	785(4)	405(3)	0(3)	−69(3)	106(3)	570(2)
Ag(3)	−0.00354(3)	0.27567(7)	−0.12771(5)	606(4)	585(4)	815(5)	−20(4)	−206(4)	13(4)	669(2)
N(1)	0.1224(3)	0.2986(7)	0.5786(5)	54(4)	67(4)	62(4)	5(4)	13(4)	−1(4)	61(2)
O(1)	0.1683(3)	0.3305(8)	0.5746(5)	53(4)	117(6)	126(6)	−6(5)	10(4)	−17(4)	98(3)
O(2)	0.1047(3)	0.2368(7)	0.5146(5)	88(5)	123(6)	71(4)	−25(4)	−11(4)	−16(5)	94(3)
O(3)	0.0947(3)	0.3382(7)	0.6440(4)	84(5)	107(5)	61(3)	−3(4)	21(3)	−18(4)	84(3)
N(2)	0.2334(2)	0.0428(6)	−0.0118(4)	44(4)	68(4)	52(4)	−10(3)	−15(3)	9(3)	55(2)
O(4)	0.2260(2)	0.1581(5)	−0.0121(4)	51(3)	55(3)	75(4)	4(3)	−3(3)	5(3)	60(2)
O(5)	0.2037(3)	−0.0246(7)	0.0294(5)	69(4)	82(5)	118(5)	39(4)	−2(4)	1(4)	90(3)
O(6)	0.2702(2)	−0.0014(7)	−0.0547(5)	57(4)	117(5)	90(4)	−43(4)	−8(3)	30(4)	88(3)
N(3)	0	0.0290(8)	$-\frac{1}{4}$	45(5)	41(5)	50(5)	0	3(4)	0	45(3)
O(7)	0	−0.0820(9)	$-\frac{1}{4}$	99(9)	54(6)	265(17)	0	80(10)	0	140(7)
O(8)	−0.0292(2)	0.0840(6)	−0.1967(4)	71(4)	71(4)	71(4)	−23(3)	10(3)	−8(3)4	71(2)
N(4)	0	0.5279(12)	$-\frac{1}{4}$	46(6)	74(8)	80(8)	0	5(5)	0	67(4)
O(9)	0	0.6423(10)	$-\frac{1}{4}$	126(10)	75(7)	93(7)	0	−32(7)	0	98(5)
O(10)	0.0203(3)	0.4790(9)	−0.1844(6)	91(6)	125(7)	145(7)	76(6)	6(5)	13(5)	120(4)
C(1)	0.1552(4)	0.3848(11)	0.2744(7)	110(9)	95(8)	89(7)	−44(6)	−39(6)	43(7)	98(5)
C(2)	0.1209(4)	0.3040(13)	0.2918(6)	61(6)	230(15)	44(4)	−15(7)	0(4)	61(8)	112(6)
C(3)	0.1147(3)	0.1815(12)	0.2417(6)	45(5)	190(12)	53(5)	34(6)	−1(4)	−46(7)	96(5)
C(4)	0.1514(5)	0.0968(10)	0.2343(7)	115(10)	70(7)	64(6)	26(5)	−39(7)	−40(7)	83(4)
C(5)	0.2013(4)	0.1033(7)	0.2769(5)	103(7)	48(5)	58(5)	14(4)	−13(5)	14(5)	70(3)
C(6)	0.2357(3)	0.1885(8)	0.2626(5)	55(5)	93(6)	46(4)	−12(4)	−14(4)	22(5)	64(3)
C(7)	0.2305(4)	0.2941(8)	0.1974(6)	65(6)	82(6)	50(4)	−1(4)	4(4)	−30(5)	66(3)
C(8)	0.1946(4)	0.3804(8)	0.2027(6)	112(8)	50(5)	68(5)	0(4)	−24(6)	−20(6)	77(4)
C(9)	0.0809(3)	0.3698(7)	0.0296(5)	61(5)	45(4)	58(4)	−6(4)	−24(4)	−4(4)	55(3)
C(10)	0.1157(3)	0.3647(7)	−0.0370(5)	53(5)	45(4)	67(5)	12(4)	−13(4)	−21(4)	55(3)
C(11)	0.1102(3)	0.2962(9)	−0.1237(5)	52(5)	80(7)	43(4)	17(4)	1(3)	−1(4)	58(3)
C(12)	0.1011(3)	0.1753(9)	−0.1335(5)	45(5)	87(6)	41(4)	−10(4)	−9(3)	15(4)	58(3)

(table continued)

TABLE 2 (continued)

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}	U_{eq} [b]
C(13)	0.0960(3)	0.0876(7)	−0.0575(5)	57(5)	45(4)	73(5)	−11(4)	−23(4)	7(4)	58(3)
C(14)	0.0627(3)	0.0904(7)	0.0100(5)	62(5)	39(4)	63(5)	12(4)	−23(4)	−10(4)	55(3)
C(15)	0.0238(3)	0.1858(8)	0.0248(5)	44(4)	71(6)	49(4)	14(4)	−1(3)	−15(4)	55(3)
C(16)	0.0317(3)	0.3075(8)	0.0324(5)	56(5)	71(6)	41(3)	−6(4)	−2(3)	14(4)	56(3)

[a] Anisotropic temperature factor exponent takes the form: $-2\pi^2\Sigma\Sigma a_i^\star \cdot a_j^\star h_i h_j U_{ij}$. [b] U_{eq} is the equivalent isotropic temperature factor calculated as one-third of the trace of the orthogonalised U_{ij} matrix.

TABLE 3

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

(i)	*Coordination around Ag(1)*			
	Ag(1)–C(1)	2.773(10)	Ag(1)–C(2)	2.743(10)
	Ag(1)–C(5)	2.606(8)	Ag(1)–C(6)	2.605(7)
	Ag(1)–O(1)	2.469(8)	Ag(1)–O(2)	2.697(7)
	Ag(1)–O(4)^{I}	2.489(6)	Ag(1)–O(5)^{II}	2.872(7)
	Ag(1)–O(6)^{II}	2.894(8)		
(ii)	*Coordination around Ag(2)*			
	Ag(2)–C(3)	2.553(8)	Ag(2)–C(4)	2.539(10)
	Ag(2)–C(7)	2.823(9)	Ag(2)–C(8)	2.804(9)
	Ag(2)–C(9)	2.632(8)	Ag(2)–C(10)	2.546(8)
	Ag(2)–C(13)	2.760(8)	Ag(2)–C(14)	2.820(8)
	Ag(2)–O(4)	2.389(5)		
(iii)	*Coordination around Ag(3)*			
	Ag(3)–C(11)	2.969(8)	Ag(3)–C(12)	2.930(8)
	Ag(3)–C(15)	2.500(8)	Ag(3)–C(16)	2.503(7)
	Ag(3)–O(3)^{III}	2.478(7)	Ag(3)–O(8)	2.384(6)
	Ag(3)–O(10)	2.415(9)		
(iv)	*Nitrato groups*			
1	N(1)–O(1)	1.245(10)	O(1)–N(1)–O(2)	118.4(7)
	N(1)–O(2)	1.225(10)	O(1)–N(1)–O(3)	119.4(7)
	N(1)–O(3)	1.259(10)	O(2)–N(1)–O(3)	122.0(7)
2	N(2)–O(4)	1.256(9)	O(4)–N(2)–O(5)	119.5(6)
	N(2)–O(5)	1.214(9)	O(4)–N(2)–O(6)	120.0(7)
	N(2)–O(6)	1.233(9)	O(5)–N(2)–O(6)	120.5(7)
3	N(3)–O(7)	1.194(13)	O(7)–N(3)–O(8)	118.7(4)
	N(3)–O(8)	1.231(7)	O(8)–N(3)–O(8)^{IV}	122.6(9)
4	N(4)–O(9)	1.231(17)	O(9)–N(4)–O(10)	115.9(7)
	N(4)–O(10)	1.201(10)	O(10)–N(4)–O(10)^{IV}	128.1(14)
(v)	*Cyclooctatetraene molecules*			
1	C(1)–C(2)	1.272(16)	C(2)–C(3)	1.511(18)
	C(3)–C(4)	1.325(16)	C(4)–C(5)	1.437(16)
	C(5)–C(6)	1.298(12)	C(6)–C(7)	1.478(12)
	C(7)–C(8)	1.319(13)	C(8)–C(1)	1.454(14)
	C(8)–C(1)–C(2)	127.7(10)	C(1)–C(2)–C(3)	125.3(9)
	C(2)–C(3)–C(4)	124.1(9)	C(3)–C(4)–C(5)	125.8(10)
	C(4)–C(5)–C(6)	126.1(8)	C(5)–C(6)–C(7)	125.4(8)
	C(6)–C(7)–C(8)	124.7(8)	C(7)–C(8)–C(1)	124.3(9)
	C(8)–C(1)–C(2)–C(3)	1.8(18)	C(4)–C(5)–C(6)–C(7)	−1.6(14)
	C(1)–C(2)–C(3)–C(4)	56.1(16)	C(5)–C(6)–C(7)–C(8)	60.4(13)
	C(2)–C(3)–C(4)–C(5)	4.3(16)	C(6)–C(7)–C(8)–C(1)	0.8(15)
	C(3)–C(4)–C(5)–C(6)	−60.0(15)	C(7)–C(8)–C(1)–C(2)	−61.0(16)
2	C(9)–C(10)	1.318(11)	C(10)–C(11)	1.455(11)
	C(11)–C(12)	1.329(13)	C(12)–C(13)	1.449(11)
	C(13)–C(14)	1.302(11)	C(14)–C(15)	1.457(11)
	C(15)–C(16)	1.330(12)	C(16)–C(9)	1.445(12)
	C(16)–C(9)–C(10)	127.5(7)	C(9)–C(10)–C(11)	125.2(7)
	C(10)–C(11)–C(12)	127.1(7)	C(11)–C(12)–C(13)	125.0(7)
	C(12)–C(13)–C(14)	127.4(8)	C(13)–C(14)–C(15)	126.0(7)
	C(14)–C(15)–C(16)	126.8(8)	C(15)–C(16)–C(9)	126.2(8)
	C(16)–C(9)–C(10)–C(11)	1.8(14)	C(12)–C(13)–C(14)–C(15)	−1.7(14)
	C(9)–C(10)–C(11)–C(12)	−57.2(13)	C(13)–C(14)–C(15)–C(16)	−55.2(13)
	C(10)–C(11)–C(12)–C(13)	−1.5(14)	C(14)–C(15)–C(16)–C(9)	−1.5(13)
	C(11)–C(12)–C(13)–C(14)	59.4(13)	C(15)–C(16)–C(9)–C(10)	56.4(12)

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[a] Roman numerals as superscripts denote the following symmetry transformations: I. $\frac{1}{2}-x$, $\frac{1}{2}-y$, $\frac{1}{2}+z$; II. x, $-y$, $\frac{1}{2}+z$; III. $-x$, y, $\frac{1}{2}-z$; IV. $-x$, y, $1\frac{1}{2}-z$.

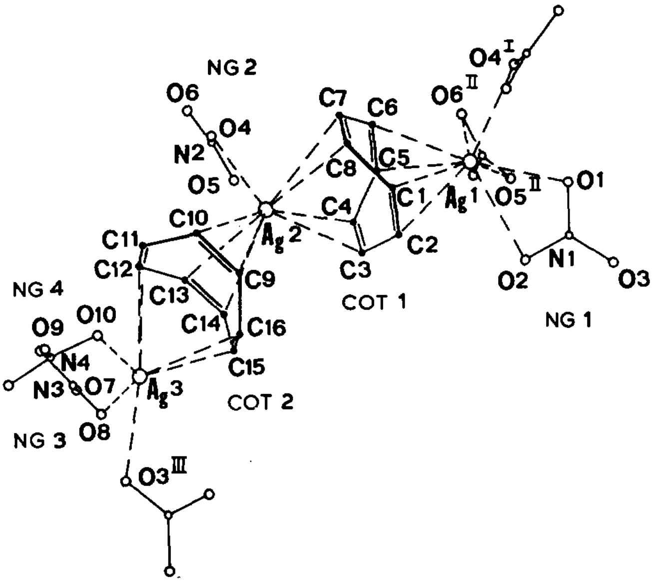

Fig. 1. A perspective view of the coordination geometries around the three silver(I) ions, showing the atom labelling scheme. Metal-ligand atom interactions are represented by broken lines. Numbering of the COT molecules and nitrato groups (NG) and the symmetry codes correspond to those shown in Table 3.

syntheses. Thermal parameters for all thirty-three non-hydrogen atoms in the asymmetric unit were refined anisotropically. The sixteen hydrogen atoms were generated on the bases of sp^2 hybridization of their respective parent carbon atoms,

TABLE 4

LEAST-SQUARES PLANES [a]

Plane	Atoms	l	m	n	d	rmsΔ (Å ×10^4)
1	C(1),C(2),C(5),C(6)	0.1709	0.0790	0.9821	4.9048	117
2	C(2),C(3),C(4),C(5)	0.3512	0.4296	−0.8320	−0.9881	128
3	C(6),C(7),C(8),C(1)	0.6222	0.5515	0.5773	6.9924	23
4	C(3),C(4),C(7),C(8)	0.1744	0.0816	0.9813	4.0837	60
5	C(8),C(1),C(2),C(3)	0.6224	−0.4381	0.6486	3.2536	52
6	C(4),C(5),C(6),C(7)	−0.3848	0.5549	0.7376	1.5431	46
Angles between planes: 1-2, 43.7°; 1-3, 44.5°; 4-5, 44.8°; 4-6, 45.4°						
7	C(9),C(10),C(13),C(14)	0.7201	−0.1831	0.6692	1.0835	102
8	C(10),C(11),C(12),C(13)	0.9854	−0.1691	−0.0194	2.3162	45
9	C(14),C(15),C(16),C(9)	0.0931	−0.1058	0.9900	0.1943	42
10	C(11),C(12),C(15),C(16)	0.7268	−0.1866	0.6610	0.3027	113
11	C(16),C(9),C(10),C(11)	0.4228	−0.7975	0.4304	−2.0910	53
12	C(12),C(13),C(14),C(15)	0.6462	0.5325	0.5467	1.6578	49
Angles between planes: 7-8, 43.3°; 7-9, 41.5°; 10-11, 42.2°; 10-12. 43.0°.						

[a] Plane equation is of the form $lX + mY + nZ = d$, where X, Y and Z are orthogonal coordinates in Å referred to the unit-cell axes a, b and c, respectively.

and were assigned fixed isotropic temperature factors. The final difference map showed residual extrema at 1.6 to $-1.2\ e\ \text{Å}^{-3}$, two of the three highest peaks being in the neighbourhood of the silver(I) ions.

All computations were performed on a Data General Nova 3/12 minicomputer with the SHELXTL program package [7,8]. Analytic expressions [9] of neutral-atom scattering factors [10] were employed, and anomalous dispersion corrections [11] were incorporated. In the blocked-cascade least-squares refinement procedure, an empirical isotropic extinction parameter ε in the expression [8]:

$$F_{\text{corr}} = F_c/\left(1 + \varepsilon F_c^2/\sin 2\theta\right)^{1/4}$$

was treated as an additional variable (Table 1).

The final positional and thermal parameters for the non-hydrogen atoms are presented in Table 2 in accordance with the atom labelling scheme in Fig. 1. * Bond lengths, bond angles, and torsion angles are listed in Table 3, and least-squares planes through selected sets of atoms in Table 4.

Results and discussion

Figure 1 shows the asymmetric unit, in which the two cyclooctatetraene molecules partition the three silver(I) ions. The configuration of the ligands about each metal center can be described, to a crude approximation, as trigonal bipyramidal. The Ag(1) ion is coordinated by one monodentate and two bidentate nitrato groups, and by two non-adjacent double bonds from COT molecule **1**; the centers of the C(5)–C(6) and O(1)–N(1)–O(2) ligands occupy the axial positions. The Ag(2) ion is coordinated by a monodentate nitrato group and four double bonds (two non-adjacent ones from each COT moiety), with the C(7)–C(8) and C(13)–C(14) olefinic ligands at the axial positions. The Ag(3) ion is coordinated by three monodentate nitrato groups, and by two double bonds from COT molecule **2**, the axial ligands being the C(11)–C(12) double bond and O(3)^{III}. The measured silver(I)–oxygen and silver(I)–olefin distances lie in the ranges 2.384–2.894 and 2.500–2.969 Å, respectively. Coordination of Ag(2) and Ag(3) involves monodentate nitrato groups and double bonds only, and in each instance the axial ligands lie farther away from the metal center than corresponding equatorial ligands of the same types. On the other hand, the reverse order of metal–ligand distances holds for the coordination around Ag(1), which also involves bidentate nitrato groups and exhibits the greatest deviation from idealized trigonal bipyramidal geometry.

The nitrato groups serve as bridges linking the silver (I) ions. Nitrato group **1** is coordinated to Ag(1) through O(1) and O(2), and to Ag(3) through O(3). In nitrato group **2**, O(5) and O(6) are linked to Ag(1), and O(4) to both Ag(1)^{V} ** and Ag(2). Nitrato groups **3** and **4** each have a N–O bond lying on the same crystallographic two-fold rotation axis and an off-axis oxygen atom coordinated to Ag(3); these two nitrato groups thus function as a double bridge between Ag(3) and Ag(3)^{IV}.

The two COT molecules are sandwiched in between the three silver(I) ions, with all double bonds partaking in metal-π bonding. The tub conformation of COT **2** is

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* Tables of hydrogen atomic coordinates and structure factors are available from the author upon request.

** Symmetry transformation: V. $\frac{1}{2}-x,\ \frac{1}{2}-y,\ -\frac{1}{2}+z$.

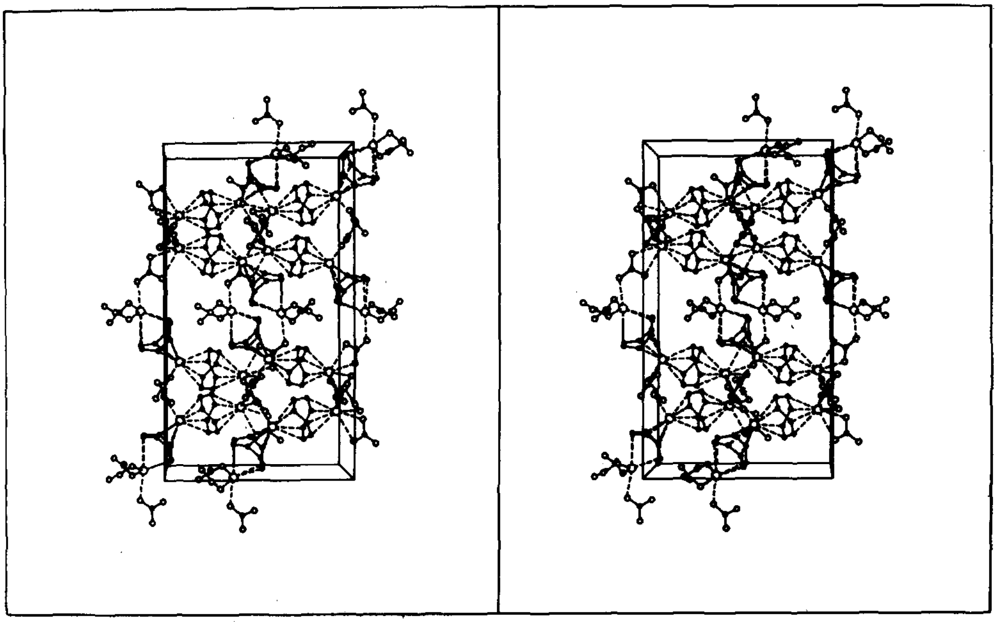

Fig. 2. Stereodrawing showing the packing arrangement in $2C_8H_8 \cdot 3AgNO_3$, with metal–ligand interactions represented by broken lines. The unit-cell origin lies at the lower left corner, with *a* pointing upwards, *b* towards the reader, and *c* from left to right.

close to that observed for free COT (bond angle $\theta = 126.1°$, torsion angle about C–C $\omega = 57.9°$, dihedral angle $\alpha = 43.2°$) [12], but COT **1** adopts a more folded configuration; this may be seen from the torsion angles listed in Table 3 and the dihedral angles shown in Table 4. In these two cyclic polyolefins, the averaged C–C single bond (1.461 Å) is virtually identical to those in the 2:1 [6] and 1:1 [5] adducts, but the averaged coordinated double bond (1.312 Å) is short and actually similar in length to the uncoordinated, rather than the coordinated, double bonds in these two adducts.

Figure 2 is a stereodrawing of the packing arrangement in the present $2COT \cdot 3AgNO_3$ adduct, in which the coordination polyhedra are interlinked into an intricate three-dimensional polymeric structure. The formation and relative stability of the three adducts can be rationalized in structural terms. Trigonal bipyramidal coordination of the silver(I) ion has emerged as an important common feature in all three crystal structures. The formation of discrete pentacoordinate $(C_8H_8)_2AgNO_3$ molecules is favored in the presence of a large excess of COT. A COT/$AgNO_3$ molar ratio between 1 and 2 tends to yield the 1:1 adduct with a chain-and-layer structure. Upon standing or recrystallization from ethanol, both the 2:1 and 1:1 adducts readily transform to the stable 2:3 adduct. The chemical transformation in this series is tantamount to the successive replacement of Van der Waals' interactions by much stronger ionic (metal–oxygen) and covalent (metal–olefin) bonds, and is therefore energetically favorable. The series of cyclooctatetraene-silver nitrate adducts thus provides a nice example of structural transition from a simple molecular crystal, through an intermediate chain-and-layer structure, to a complex three-dimensional polymeric lattice.

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