Table 5. *Measured and calculated Bijvoet ratios*

hkl	$100\dfrac{\Delta F}{F_{calc}}$	$100\dfrac{\Delta F}{F_{obs}}$	$\dfrac{\Delta F_{obs}}{\sigma(\Delta F)}$
1 2 2	+28	+21	6·7
1 2 8	+11	+16	5·4
1 3 6	+14	+33	4·7
1 3 7	+23	+22	6·3
1 3 9	+5	+12	4·0
2 2 8	+14	+17	4·8
2 2 10	+10	+19	5·3
2 3 7	+23	+30	7·4
3 3 7	+8	+12	3·9

Least-squares plane (C) indicates the five O atoms are fairly planar. The Rb^+ cation is displaced 0·94 Å above the plane (C) due to the inability of the ligand to enlarge its cavity enough to fit the cation. The S atom is situated 1·54 Å below the mean plane (C) toward the Rb^+ cation of an adjacent ligand.

The absolute configuration was determined by the Bijvoet ratios, $\Delta F/F = (F_{hkl} - F_{h\bar{k}\bar{l}})/0\cdot5(F_{hkl} + F_{h\bar{k}\bar{l}})$. Nine Friedel pairs were chosen from the 265 measured because they were regarded as statistically significant, where $\Delta F > 3\cdot8\sigma(\Delta F)$. The term $3\cdot8\sigma(\Delta F)$ was chosen from consideration of 105 equivalent (calculated F's are equal) Friedel pairs; none of the 105 showed a ΔF greater than $3\cdot8\sigma(\Delta F)$. Table 5 compares the measured and calculated Bijvoet ratios. Comparison of the signs shows the absolute configuration to be that reported in Table 1.

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Hydrated Silver Nitrate Complex of 1,4,7,10,13-Pentaoxa-16-thiacyclooctadecane

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Abstract. $C_{12}H_{24}O_5S \cdot AgNO_3 \cdot H_2O$, $M_r = 468\cdot28$, monoclinic, $P2_1/c$, $a = 9\cdot856$ (4), $b = 19\cdot923$ (6), $c = 9\cdot310$ (2) Å, $\beta = 92\cdot11$ (3)°, $V = 1826\cdot9$ Å^3, $Z = 4$, $D_x = 1\cdot70$, $D_m = 1\cdot71$ Mg m^{-3} (flotation in CCl_4 and $CHBr_3$), $F(000) = 960$, colorless crystals, m.p. 360 K. The structure was solved by the heavy-atom method and refined to $R = 0\cdot078$ ($R_w = 0\cdot070$) for a total of 3203 independent reflections. The Ag^+ cation sits in the cavity of the ligand and coordinates to the five O atoms and the S atom. It also coordinates to an S atom from an adjacent ligand. The Ag–S contact distances indicate covalent character in the Ag–S interaction. Each nitrate anion hydrogen bonds to two water molecules and does not interact with an Ag^+ cation.

Introduction. The complex was prepared by allowing the solvent to evaporate from an equimolar salt–ligand mixture dissolved in aqueous methanol. The crystal was shaped to a sphere ($d = 0\cdot25$ mm) with a Nonius crystal spherizer. Preliminary crystallographic data obtained on a General Electric XRD-5 diffractometer were used to determine the space group. Systematic absences ($0k0$, $k = 2n + 1$ and $h0l$, $l = 2n + 1$) indicated the unambiguous space group $P2_1/c$. The

space-group selection was substantiated by the systematic extinctions observed during data collection and later by the locations of the Ag—Ag vectors in the Patterson map. Intensity data were collected using an Enraf–Nonius CAD-4 automated diffractometer employing graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). Accurate lattice parameters were determined by a least-squares refinement of 25 2θ values. Diffraction data to a maximum 2θ of 50° ($\sin\theta/\lambda = 0.595$ Å^{-1}) were recorded using the $\omega/2\theta$ scan mode. Of the 3316 independent reflections measured, 113 were systematically extinct and 989 were considered unobserved as $I < 2\sigma(I)$. The scan rate varied from 2° to 20° min^{-1} with a scan range of $(0.70 + 0.35 \tan\theta)°$. The variable scan rate allows for rapid data collection for intense reflections where a fast scan rate is used and assures good counting statistics for weak reflections where a slow scan rate is used. Moving-crystal, moving-counter background counts were taken at each end of the scan range. The ratio of the scan time to the background counting time was 2. Three standards measured periodically showed no significant change. No absorption corrections were made as μ(Mo $K\alpha$) = 1.23 mm^{-1}. The structure was solved by the heavy-atom method and refined using a full-matrix least-squares procedure (Busing, Martin & Levy, 1962). The large thermal parameters of the ligand atoms and the deviations of the C—C and C—O bond lengths from the accepted values indicated that the ligand was disordered. Two positions for each of the ligand atoms, with the exception of the C(6) and O(10) atoms, were resolved. The positional and thermal parameters of the non-H atoms were refined with experimental weights (Stout & Jensen, 1968). The thermal parameters of the Ag$^+$, S(1A), S(1B), C(6), O(10), O(HOH) and nitrate atoms were refined anisotropically; the remainder of the atoms were refined isotropically. The occupancies for the disordered atoms were set at 0.5 based on the relative peak height on the Fourier map. These values were not refined. H atoms were not included in the model. The positional and isotropic thermal parameters of the atoms are listed in Table 1.* The final R was 0.078, while the weighted R was 0.070. The 'goodness of fit', $[\sum w(\Delta F)^2/(n - m)]^{1/2}$, was 1.65 for $m = 211$ and $n = 3203$. Atomic-scattering-factor tables were taken from Cromer & Waber (1965). Anomalous-dispersion corrections for Ag and S were taken from Cromer & Liberman (1970).

Discussion. The conformation and atom numbering of molecule A are shown in Fig. 1. The conformation of

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35983 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates* ($\times 10^4$) *and isotropic* U *values* ($\times 10^3$) *for the non*-H *atoms with e.s.d.'s in parentheses*

$\bar{U}$ is the average of the three eigenvalues of each anisotropic vibration tensor. It is listed for all atoms refined anisotropically, namely Ag$^+$, N, O(N1), O(N2), O(N3), O(HOH), S(1A), C(6), O(10) and S(1B). The remainder of the atoms were refined isotropically and U is listed for them.

	x	y	z	$U/\bar{U}$ (Å^2)
Ag$^+$	1661 (0)	2139 (0)	4575 (0)	55 (1)
N	2936 (5)	9175 (3)	1116 (7)	76 (4)
O(N1)	2323 (7)	8933 (4)	114 (7)	162 (6)
O(N2)	2918 (7)	8950 (3)	2325 (7)	140 (5)
O(N3)	3642 (7)	9655 (4)	920 (8)	152 (6)
O(HOH)	6084 (5)	10182 (3)	2194 (5)	108 (4)
S(1A)	804 (14)	2738 (8)	2253 (19)	47 (4)
C(2A)	−912 (14)	2521 (8)	1925 (15)	73 (3)
C(3A)	−1722 (14)	2381 (8)	3121 (17)	82 (4)
O(4A)	−1233 (10)	1896 (5)	4014 (10)	53 (2)
C(5A)	−1497 (23)	1160 (12)	4009 (24)	107 (8)
C(6)	−512 (9)	788 (5)	3212 (13)	142 (9)
O(7A)	765 (10)	853 (5)	3955 (11)	64 (3)
C(8A)	1879 (22)	565 (11)	3297 (24)	95 (8)
C(9A)	3175 (16)	575 (8)	4393 (16)	61 (4)
O(10)	3415 (4)	1247 (2)	4496 (5)	80 (3)
C(11A)	4815 (21)	1443 (12)	4999 (23)	94 (7)
C(12A)	5116 (24)	2115 (11)	4898 (28)	102 (8)
O(13A)	4102 (9)	2415 (5)	5964 (12)	73 (3)
C(14A)	4328 (14)	3153 (7)	6134 (16)	76 (4)
C(15A)	3768 (14)	3579 (7)	4959 (15)	68 (3)
O(16A)	2364 (11)	3439 (5)	4890 (12)	75 (3)
C(17A)	1972 (18)	3846 (8)	3592 (21)	99 (5)
C(18A)	652 (15)	3604 (7)	2894 (15)	79 (4)
S(1B)	1113 (13)	2861 (8)	2300 (19)	43 (4)
C(2B)	−725 (13)	2926 (6)	2428 (14)	67 (3)
C(3B)	−1355 (17)	2202 (8)	2373 (19)	82 (4)
O(4B)	−949 (12)	1845 (6)	3663 (13)	81 (4)
C(5B)	−1525 (18)	1232 (9)	3514 (21)	78 (5)
O(7B)	625 (12)	780 (6)	4396 (14)	89 (4)
C(8B)	1674 (18)	450 (9)	3609 (20)	71 (5)
C(9B)	2757 (20)	487 (9)	4315 (20)	79 (6)
C(11B)	4635 (15)	1257 (8)	5219 (16)	57 (4)
C(12B)	5082 (19)	1960 (11)	5284 (22)	81 (6)
O(13B)	4212 (13)	2466 (7)	5296 (15)	101 (4)
C(14B)	4496 (21)	3139 (11)	5154 (24)	117 (6)
C(15B)	3488 (18)	3556 (9)	5692 (20)	97 (5)
O(16B)	2065 (10)	3496 (5)	5287 (11)	66 (3)
C(17B)	1369 (12)	3910 (6)	4298 (13)	58 (3)
C(18B)	1556 (12)	3731 (6)	2712 (13)	57 (3)

molecule B is approximately related to that of molecule A by a pseudo-mirror plane determined by the S(1), O(10) and Ag atoms. Bond lengths, angles and torsion angles are shown in Table 2. The 'abnormal' bond lengths and angles indicate that the C and O atomic positions of the ligand are considerably uncertain. The bond lengths, angles and torsion angles involving the C(6) and O(10) atoms are particularly suspect since attempts at resolving these two atoms were unsuccessful. The reported C(6) and O(10) atomic positions appear to be the averages of their atomic positions for the two conformations. Fig. 2 is a stereoscopic view of the unit cell.
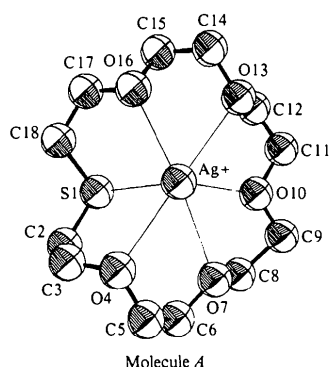
Fig. 1 *ORTEP* (Johnson, 1965) drawing of $C_{12}H_{24}O_5S.AgNO_3.H_2O$ with the atoms drawn as spheres of radius 0·3 Å, showing atom numbering. The nitrate anion and water of hydration are omitted.
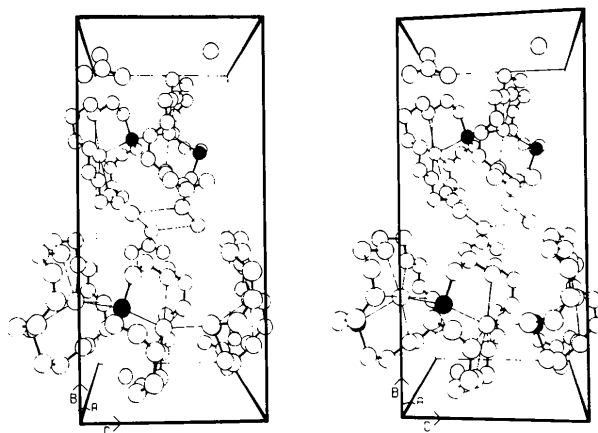

Fig. 2. Stereoscopic view of the crystal structure viewed along **a**.

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

1	2	3	4	1–2	1–2–3	1–2–3–4
S(1A)	C(2A)	C(3A)	O(4A)	1·761 (19)	118·4 (12)	−55·5 (18)
C(2A)	C(3A)	O(4A)	C(5A)	1·422 (21)	115·1 (12)	−91·8 (19)
C(3A)	O(4A)	C(5A)	C(6A)	1·351 (18)	130·0 (13)	92·8 (20)
O(4A)	C(5A)	C(6A)	O(7A)	1·490 (26)	112·7 (16)	65·8 (17)
C(5A)	C(6A)	O(7A)	C(8A)	1·448 (26)	107·6 (12)	−175·7 (16)
C(6A)	O(7A)	C(8A)	C(9A)	1·419 (14)	116·4 (12)	−170·4 (12)
O(7A)	C(8A)	C(9A)	O(10A)	1·400 (25)	109·6 (16)	−67·1 (16)
C(8A)	C(9A)	O(10A)	C(11A)	1·606 (27)	100·8 (12)	−159·0 (13)
C(9A)	O(10A)	C(11A)	C(12A)	1·362 (16)	115·8 (11)	170·7 (17)
O(10A)	C(11A)	C(12A)	O(13A)	1·493 (21)	115·6 (18)	64·0 (21)
C(11A)	C(12A)	O(13A)	C(14A)	1·376 (33)	100·7 (18)	175·1 (15)
C(12A)	O(13A)	C(14A)	C(15A)	1·553 (27)	110·4 (12)	78·2 (16)
O(13A)	C(14A)	C(15A)	O(16A)	1·493 (18)	115·9 (12)	58·4 (15)
C(14A)	C(15A)	O(16A)	C(17A)	1·476 (20)	105·0 (11)	−174·9 (12)
C(15A)	O(16A)	C(17A)	C(18A)	1·411 (18)	98·8 (11)	158·2 (13)
O(16A)	C(17A)	C(18A)	S(1A)	1·494 (22)	111·5 (13)	−62·4 (16)
C(17A)	C(18A)	S(1A)	C(2A)	1·512 (23)	111·2 (11)	166·0 (12)
C(18A)	S(1A)	C(2A)	C(3A)	1·833 (21)	101·5 (10)	−75·2 (15)
S(1B)	C(2B)	C(3B)	O(4B)	1·825 (18)	109·0 (10)	68·0 (15)
C(2B)	C(3B)	O(4B)	C(5B)	1·569 (20)	109·2 (12)	−179·5 (13)
C(3B)	O(4B)	C(5B)	C(6B)	1·439 (21)	104·9 (13)	106·0 (15)
O(4B)	C(5B)	C(6B)	O(7B)	1·352 (22)	107·3 (14)	58·1 (17)
C(5B)	C(6B)	O(7B)	C(8B)	1·372 (21)	112·1 (12)	−163·5 (13)
C(6B)	O(7B)	C(8B)	C(9B)	1·542 (16)	99·0 (11)	169·8 (15)
O(7B)	C(8B)	C(9B)	O(10B)	1·448 (22)	108·8 (16)	−67·6 (19)
C(8B)	C(9B)	O(10B)	C(11B)	1·235 (26)	115·9 (15)	−176·9 (16)
C(9B)	O(10B)	C(11B)	C(12B)	1·654 (19)	113·6 (10)	179·8 (12)
O(10B)	C(11B)	C(12B)	O(13B)	1·357 (15)	107·0 (12)	29·5 (23)
C(11B)	C(12B)	O(13B)	C(14B)	1·469 (26)	122·2 (16)	−170·2 (18)
C(12B)	O(13B)	C(14B)	C(15B)	1·325 (25)	127·4 (16)	−159·3 (18)
O(13B)	C(14B)	C(15B)	O(16B)	1·376 (26)	113·1 (17)	−52·1 (25)
C(14B)	C(15B)	O(16B)	C(17B)	1·401 (28)	123·5 (16)	−101·4 (20)
C(15B)	O(16B)	C(17B)	C(18B)	1·445 (20)	125·0 (11)	80·5 (15)
O(16B)	C(17B)	C(18B)	S(1B)	1·396 (15)	115·0 (10)	55·2 (13)
C(17B)	C(18B)	S(1B)	C(2B)	1·537 (17)	112·7 (10)	71·3 (12)
C(18B)	S(1B)	C(2B)	C(3B)	1·826 (19)	98·6 (9)	−170·4 (11)
O(N1)	N	O(N2)		1·194 (9)	123·5 (7)	
O(N2)	N	O(N3)		1·212 (9)	117·7 (7)	
O(N3)	N	O(N1)		1·201 (10)	118·8 (7)	

Table 3. *Ag⁺-ion contact distances (Å)*

Ag⁺···S(1A)	2·584 (17)	Ag⁺···S(1B)	2·600 (17)
Ag⁺···O(4A)	2·921 (10)	Ag⁺···O(4B)	2·742 (12)
Ag⁺···O(7A)	2·764 (10)	Ag⁺···O(7B)	2·897 (13)
Ag⁺···O(10A)	2·482 (4)	Ag⁺···O(10B)	2·482 (4)
Ag⁺···O(13A)	2·745 (10)	Ag⁺···O(13B)	2·660 (13)
Ag⁺···O(16A)	2·694 (11)	Ag⁺···O(16B)	2·809 (10)
Ag⁺···S(1A')	2·673 (18)	Ag⁺···S(1B')	2·613 (18)

Table 3 shows the Ag⁺-cation coordination contact distances in the complex. Each Ag⁺ cation coordinates to two S atoms. In addition to the intramolecular Ag–S bond, there is also an Ag⁺ interaction with an S of an adjacent ligand. The S–Ag contact distances of 2·58 and 2·60 Å (intramolecular) and 2·67 and 2·61 Å (intermolecular) are about 0·5 Å shorter than the sum of the corresponding ionic and van der Waals radii of 3·11 Å (Pauling, 1960) and indicate covalent character

in the interaction. The Ag–O(10) distance of 2·48 Å is similar to the Ag–O distances found in AgNO₂ (Long & Marsh, 1962), AgNO₃ (Meyer, Rimsky & Chevalier, 1978; Gibbons & Trotter, 1971; Lindley & Woodward, 1966), AgClO₂ (Cooper & Marsh, 1961), and AgClO₄(dioxan)₃ (Prosen & Trueblood, 1956) and indicates negligible covalent character in the bond. The remainder of the Ag–O distances (2·66 to 2·92 Å) indicate weak cation–dipole interactions. The close interaction of the S(1) and O(10) atoms with the Ag⁺ cation gives the ligand a conformation similar to that of the PdCl₂ complex of 1,4,10,13-tetraoxa-7,16-dithiacyclooctadecane (Metz, Moras & Weiss, 1974).

The nitrate anion does not interact with the Ag⁺ cation. The shortest nitrate–Ag distance is 5·33 Å. The N–O distances are shorter than those reported for other structures of nitrates (Gibbons & Trotter, 1971; Meyer *et al.*, 1978; Cherin, Hamilton & Post, 1967). The short N–O bond distances are probably an artificial effect of thermal motion due to rotational oscillation of the nitrate (Gibbons & Trotter, 1971). The water O–nitrate oxygen distances [O(HOH)–O(N3), 2·844 (9) Å and O(HOH)–O'(N3), 2·940 (9) Å] suggest the existence of a network of hydrogen bonds linking water molecules to nitrate groups. The O(N3)–O(HOH)–O'(N3) angle is 75°. Unfortunately the presence of the network could not be confirmed as the H atoms could not be located in the Fourier difference maps.

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Struktur von *catena*-μ-(Hexamethylentetramin-N,N')-[tetra-μ-acetato-dikupfer(II)]

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Abstract. [{[Cu(CH₃COO)₂]₂(C₆H₁₂N₄)}ₙ], orthorhombic, *Cmma*, $a = 15 \cdot 616$ (4), $b = 15 \cdot 459$ (5), $c = 8 \cdot 066$ (5) Å, $Z = 4$, $D_m = 1 \cdot 70$ (5), $D_c = 1 \cdot 72$ Mg m⁻³, $\mu(\text{Mo } K\alpha) = 2 \cdot 3$ mm⁻¹. The final R value for 1528 independent data is $0 \cdot 045$. The compound consists of polymeric chains formed by [Cu(CH₃COO)₂]₂ moieties analogous to those in cupric acetate hydrate, bridged by hexamethylenetetramine molecules.

Einleitung. Hexamethylentetramin (I) wird in der Anorganischen Chemie häufig als Fällungsmittel für Hydroxide, sowohl im qualitativ-analytischen Trennungsgang als auch für präparative Zwecke, benutzt. Beim Vermischen konzentrierter wässriger Lösungen von Kupfer(II)-acetat und (I) wurde die Bildung eines grünen, feinkristallinen Niederschlages beobachtet, bei dem es sich seinem Aussehen nach nicht um ein Hydroxid handeln konnte. Es wurde vermutet, dass es sich um eine 'Additionsverbindung' mit (I) handeln könnte; derartige 'Additionsverbindungen' von (I) mit Schwermetallsalzen sind in der Literatur beschrieben (Altpeter, 1931), sie sind bisher jedoch kaum näher untersucht worden.

Grössere, säulenförmige Kristalle von bis zu 5 mm Länge konnten durch Eindiffundieren von Kupfer(II)-

acetatlösung in eine wässrige Lösung von (I) erhalten werden.

Ein Kristall von 0,13 × 0,24 × 0,33 mm Grösse wurde für die Strukturbestimmung ausgewählt. Für die Gitterkonstantenbestimmung und die Intensitätsmessung wurde ein Vierkreisdiffraktometer Syntex *P2₁* verwendet; die Gitterparameter wurden aus den Winkelmessungen für 15 Reflexe ermittelt und nach der Methode der kleinsten Fehlerquadrate verfeinert.

Der Kristall erwies sich als orthorhombisch, die systematischen Auslöschungen der Reflexe *hkl* für $h + k = 2n + 1$ und *hk0* für $h = 2n + 1$ und $k = 2n + 1$ entsprachen den Raumgruppen *Cmma* (Nr. 67) und *Cm2a* bzw. *Cm2b* (Nichtstandardaufstellungen von *Abm2*, Nr. 39). Die |*E*|-Statistik und die Abwesenheit eines piezoelektrischen Effektes (Bunn, 1961) gaben den Ausschlag für die Wahl der zentrosymmetrischen Raumgruppe *Cmma*. Die erfolgreiche Strukturbestimmung bestätigte die Richtigkeit dieser Annahme, Versuche zur Lösung der Struktur in den anderen Raumgruppen schlugen fehl.

Unter Verwendung monochromatischer Mo *Kα*-Strahlung (Graphitmonochromator) wurden die Intensitätsdaten von 1528 unabhängigen Reflexen mit $2\theta_{max} = 55°$ nach der ω–2θ-Methode mit Abtastgeschwindig-