extensive network of hydrogen bonds. The N—H···I hydrogen bonds range from 3·676 to 3·820 Å and O—H···I from 3·490 to 3·522 Å. These distances are close to the sums of the van der Waals radii (Pauling, 1960): 3·65 and 3·55 Å for I···N and I···O, respectively. The N—H···O hydrogen bonds range from 2·989 to 3·080 Å.

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Refinement of the Structure of the Costunolide–Silver Nitrate Complex

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Abstract. The structure [Šorm, Suchý, Holub, Línek, Hadinec & Novák (1970). *Tetrahedron Lett.* pp. 1893–1896] of $C_{15}H_{20}O_2.2AgNO_3$, $M_r = 572{\cdot}1$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}495$ (2), $b = 11{\cdot}411$ (3), $c = 21{\cdot}436$ (3) Å, $Z = 4$, was refined to $R = 0{\cdot}043$. The coordinated C=C double bonds in the *trans,trans*-cyclodeca-1,5-diene ring of the complex are significantly longer than the C=C bonds in the uncomplexed molecule [Bovill, Cox, Cradwick, Guy, Sim & White (1976). *Acta Cryst.* B**32**, 3203–3209].

Introduction. Counter data were collected on a Hilger & Watts tape-controlled diffractometer, using Mo $K\alpha$ radiation. 2183 independent reflexions were obtained (each of them measured four times including left and right background) with an ω–2θ step scan for 2θ up to 60°. Very strong reflexions (more than 15 thousand counts s^{-1} in the peak) were remeasured with attenuators. One standard reflexion was measured after each 12 measurements. The specimen appeared to be stable to air, light and X-rays. Corrections were made for background (linear), standard-reflexion level and Lp. No absorption correction (for a needle-shaped specimen supposed to be cylindrical, $\mu R < 0{\cdot}6$) or extinction correction was made.

A local version (Novák, 1973) of *ORFLS* (Busing, Martin & Levy, 1962), allowing the use of anomalous-

Table 1. *Fractional coordinates of non-hydrogen atoms* ($\times 10^4$; *for* Ag *atoms* $\times 10^5$)

E.s.d.'s (in parentheses) refer to the last decimal place.

	x	*y*	*z*
Ag(1)	38894 (10)	79603 (6)	69744 (3)
Ag(2)	93357 (10)	5331 (5)	42840 (2)
N(1)	6118 (9)	2851 (6)	6691 (3)
N(2)	6382 (9)	6311 (6)	6438 (3)
O(1)	9366 (8)	2459 (4)	5206 (2)
O(2)	9551 (8)	4405 (5)	5100 (3)
O(3)	5327 (9)	3633 (5)	6983 (3)
O(4)	7032 (9)	2113 (6)	6977 (3)
O(5)	6013 (10)	2804 (6)	6104 (2)
O(6)	4741 (8)	6203 (5)	6343 (3)
O(7)	7447 (10)	5669 (7)	6177 (4)
O(8)	6870 (9)	7107 (5)	6787 (3)
C(1)	2280 (11)	8805 (6)	6167 (3)
C(2)	1149 (12)	7861 (6)	5874 (3)
C(3)	324 (13)	8318 (6)	5249 (3)
C(4)	9347 (10)	9447 (6)	5387 (3)
C(5)	304 (10)	472 (6)	5359 (3)
C(6)	9850 (10)	1584 (5)	5678 (3)
C(7)	1453 (10)	2123 (6)	6028 (3)
C(8)	1761 (12)	1723 (6)	6705 (3)
C(9)	2816 (10)	594 (6)	6797 (3)
C(10)	1823 (11)	9490 (6)	6665 (3)
C(11)	1056 (11)	3423 (6)	5942 (3)
C(12)	9930 (10)	3534 (7)	5388 (3)
C(13)	1414 (10)	4331 (6)	6310 (3)
C(14)	7545 (11)	9318 (7)	5660 (5)
C(15)	234 (13)	9291 (7)	7057 (4)

scattering corrections, was used for the least-squares refinement. Scattering factors and anomalous-scattering correction factors for Ag were taken from *International Tables for X-ray Crystallography* (1974). Cruickshank's weighting scheme, multiplied by a factor $0{\cdot}5[1 + \log^2_{10}(F_o/F_{\rm average})]$, was used. During the refinement 22 reflexions were discarded for which $\mathrm{abs}[\log_{10}(F_o/F_c)] > \log_{10} 2$. From the remaining 2161 reflexions, 192 weak reflexions had a variance $\sigma(I)$ from four independent measurements of >25%.

Table 2. *Fractional coordinates of* H *atoms* (×10³)

E.s.d.'s (in parentheses) refer to the last decimal place. (Coordinates of atoms marked by * are taken from a difference map.)

	x	y	z
H(1)	291 (10)	911 (6)	586 (3)
H(21)	17 (10)	773 (6)	621 (3)
H(22)	193 (9)	726 (6)	576 (3)
H(31)*	128	852	494
H(32)	948 (9)	785 (6)	508 (3)
H(5)	131 (9)	65 (6)	517 (3)
H(6)*	889	138	595
H(7)*	260	190	579
H(81)	248 (10)	237 (6)	690 (3)
H(82)	58 (10)	154 (6)	691 (3)
H(91)	365 (9)	74 (6)	651 (3)
H(92)	325 (9)	53 (6)	723 (3)
H(131)	82 (9)	507 (6)	615 (3)
H(132)	157 (9)	433 (6)	673 (3)
H(141)*	763	985	600
H(142)	702 (9)	863 (6)	577 (3)
H(143)	672 (9)	970 (6)	541 (3)
H(151)	994 (10)	841 (6)	707 (3)
H(152)	921 (9)	964 (6)	692 (3)
H(153)	54 (9)	952 (6)	748 (3)

Table 3. *Bond lengths and interatomic distances* (Ag–N) (Å)

E.s.d.'s (in parentheses) refer to the last decimal place.

Ag(1)–N(1)	2·864 (8)	C(9)–C(10)	1·490 (10)
Ag(1)–N(2)	2·890 (8)	C(10)–C(15)	1·475 (12)
Ag(2)–N(1)	3·091 (7)	C(11)–C(12)	1·462 (10)
Ag(2)–N(2)	3·029 (7)	C(11)–C(13)	1·330 (10)
N(1)–O(3)	1·241 (11)	C(1)–H(1)	0·88 (7)
N(1)–O(4)	1·247 (12)	C(2)–H(21)	1·04 (7)
N(1)–O(5)	1·262 (10)	C(2)–H(22)	0·93 (7)
N(2)–O(6)	1·253 (11)	C(3)–H(31)	1·00 (7)
N(2)–O(7)	1·219 (13)	C(3)–H(32)	0·90 (7)
N(2)–O(8)	1·232 (11)	C(5)–H(5)	0·88 (7)
O(1)–C(6)	1·467 (8)	C(6)–H(6)	0·95 (7)
O(1)–C(12)	1·355 (9)	C(7)–H(7)	1·03 (7)
O(2)–C(12)	1·204 (10)	C(8)–H(81)	1·01 (7)
C(1)–C(2)	1·508 (10)	C(8)–H(82)	1·01 (7)
C(1)–C(10)	1·367 (9)	C(9)–H(91)	0·89 (7)
C(2)–C(3)	1·565 (10)	C(9)–H(92)	0·99 (7)
C(3)–C(4)	1·511 (10)	C(13)–H(131)	1·01 (7)
C(4)–C(5)	1·373 (10)	C(13)–H(132)	0·91 (7)
C(4)–C(14)	1·479 (11)	C(14)–H(141)	0·95 (7)
C(5)–C(6)	1·481 (9)	C(14)–H(142)	0·91 (7)
C(6)–C(7)	1·544 (10)	C(14)–H(143)	0·93 (7)
C(7)–C(8)	1·539 (9)	C(15)–H(151)	1·03 (7)
C(7)–C(11)	1·524 (10)	C(15)–H(152)	0·91 (7)
C(8)–C(9)	1·524 (10)	C(15)–H(153)	0·97 (7)

The refinement yielded the atomic coordinates of all but four H atoms and the anisotropic thermal parameters of the non-hydrogen atoms. The value of $B_{\rm H} = 4{\cdot}0$ Å² was assigned to the H atoms. Four of them (marked in Table 2) did not refine well and their coordinates were taken from a difference map. For further computing (bond lengths, angles and torsion angles) they were assumed to have the greatest e.s.d.'s occurring for the H atoms refined.

Discussion. The positional parameters of the structure are given in Tables 1 and 2. The bond lengths (and important interatomic distances), bond angles of the complex and torsion angles of the non-hydrogen atoms

Table 4. *Bond angles* (°) *involving non-hydrogen atoms*

E.s.d.'s (in parentheses) refer to the last decimal place.

O(3)–N(1)–O(4)	120·0 (7)	O(1)–C(6)–C(7)	104·8 (5)
O(4)–N(1)–O(5)	119·7 (7)	C(5)–C(6)–C(7)	112·8 (6)
O(5)–N(1)–O(3)	120·3 (7)	C(6)–C(7)–C(8)	117·2 (6)
O(6)–N(2)–O(7)	120·6 (7)	C(6)–C(7)–C(11)	100·2 (5)
O(7)–N(2)–O(8)	121·8 (7)	C(8)–C(7)–C(11)	115·6 (5)
O(8)–N(2)–O(6)	117·6 (7)	C(7)–C(8)–C(9)	116·8 (6)
		C(8)–C(9)–C(10)	115·5 (6)
C(6)–O(1)–C(12)	109·9 (5)	C(1)–C(10)–C(9)	120·4 (7)
C(2)–C(1)–C(10)	126·4 (7)	C(1)–C(10)–C(15)	124·0 (7)
C(1)–C(2)–C(3)	109·9 (6)	C(9)–C(10)–C(15)	115·2 (6)
C(2)–C(3)–C(4)	107·9 (5)	C(7)–C(11)–C(12)	107·2 (6)
C(3)–C(4)–C(5)	117·7 (7)	C(7)–C(11)–C(13)	130·3 (6)
C(3)–C(4)–C(14)	115·8 (6)	C(12)–C(11)–C(13)	122·1 (7)
C(5)–C(4)–C(14)	125·4 (7)	O(1)–C(12)–C(11)	109·6 (6)
C(4)–C(5)–C(6)	126·1 (7)	O(2)–C(12)–C(11)	128·6 (7)
O(1)–C(6)–C(5)	108·8 (5)	O(1)–C(12)–O(2)	121·7 (6)

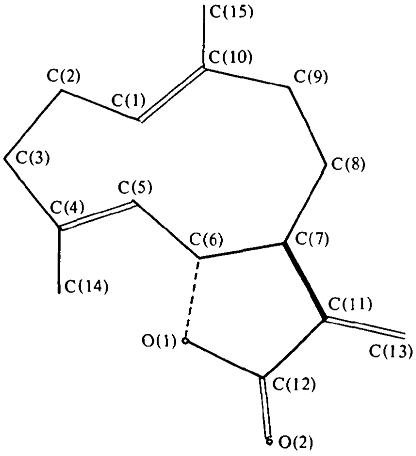

Fig. 1. Schematic representation of the organic part of the complex.

Table 5. *Torsion angles (°) of the non-hydrogen atoms of the organic part*

E.s.d.'s (in parentheses) refer to the last decimal place. The angle 1—2—3—4 is defined as positive if, when viewed along the 2—3 bond, atom 1 has to be rotated clockwise to eclipse atom 4.

1 2 3 4		1 2 3 4		1 2 3 4	
C(12)–O(1)–C(6)–C(5)	144·8 (6)	C(14)–C(4)–C(5)–C(6)	−10·1 (11)	C(6)–C(7)–C(11)–C(13)	−148·7 (8)
C(12)–O(1)–C(6)–C(7)	24·0 (7)	C(4)–C(5)–C(6)–O(1)	112·4 (7)	C(8)–C(7)–C(11)–C(12)	150·6 (6)
C(6)–O(1)–C(12)–O(2)	173·6 (7)	C(4)–C(5)–C(6)–C(7)	−131·8 (7)	C(8)–C(7)–C(11)–C(13)	−21·8 (12)
C(6)–O(1)–C(12)–C(11)	−8·5 (8)	O(1)–C(6)–C(7)–C(8)	−153·9 (6)	C(7)–C(8)–C(9)–C(10)	75·8 (8)
C(10)–C(1)–C(2)–C(3)	−103·5 (8)	O(1)–C(6)–C(7)–C(11)	−28·1 (6)	C(8)–C(9)–C(10)–C(1)	−112·2 (8)
C(2)–C(1)–C(10)–C(9)	162·9 (7)	C(5)–C(6)–C(7)–C(8)	87·9 (7)	C(8)–C(9)–C(10)–C(15)	60·8 (8)
C(2)–C(1)–C(10)–C(15)	−9·5 (12)	C(5)–C(6)–C(7)–C(11)	−146·2 (5)	C(7)–C(11)–C(12)–O(1)	−10·8 (8)
C(1)–C(2)–C(3)–C(4)	54·2 (8)	C(6)–C(7)–C(8)–C(9)	−84·5 (8)	C(7)–C(11)–C(12)–O(2)	167·0 (8)
C(2)–C(3)–C(4)–C(5)	−88·1 (8)	C(11)–C(7)–C(8)–C(9)	157·7 (6)	C(13)–C(11)–C(12)–O(1)	162·5 (7)
C(2)–C(3)–C(4)–C(14)	80·3 (8)	C(6)–C(7)–C(11)–C(12)	23·7 (7)	C(13)–C(11)–C(12)–O(2)	−19·9 (13)
C(3)–C(4)–C(5)–C(6)	157·0 (6)				

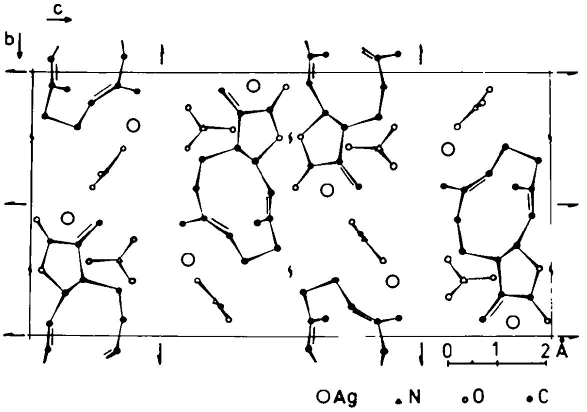

Fig. 2. The packing in the unit cell viewed down *a*. H atoms are omitted for clarity.

of the organic moiety are given in Tables 3–5.* Fig. 1 shows the organic part (costunolide) of the structure and the numbering of non-hydrogen atoms. H atoms are numbered systematically with respect to the C atoms to which they are bonded. For the relationship of the Ag^+ ions to the organic moiety and for the packing, see Fig. 2.

A comparison of our results with those of Bovill *et al.* (1976) concerning the geometric features of the substituted *trans,trans*-double bonds in the ten-membered ring is given in Table 6. In the presence of the Ag^+ ions the length of the double bond acquires a value approaching the C=C bond length in aromatic compounds as a result of the formation of an electron donor–acceptor complex. The comparison also shows

Table 6. *Comparison of the geometric parameters of the double bonds* C(4)–C(5) *and* C(1)–C(10)

E.s.d.'s (in parentheses) refer to the last decimal place.

(*a*) The C(4)–C(5) double bond.	This work	Previous work*
Bond lengths (Å)		
C(3)–C(4)	1·511 (9)	1·512 (3)
C(4)–C(5)	1·373 (10)	1·329 (2)
C(4)–C(14)	1·479 (11)	1·487 (3)
C(5)–C(6)	1·481 (9)	1·483 (2)
Bond angles (°)		
C(3)–C(4)–C(5)	117·7 (7)	117·9 (2)
C(3)–C(4)–C(14)	115·8 (6)	117·8 (2)
C(4)–C(5)–C(6)	126·1 (6)	126·1 (1)
C(5)–C(4)–C(14)	125·4 (6)	123·8 (2)
Torsion angle (°)		
C(3)–C(4)–C(5)–C(6)	157·0 (6)	155·9 (2)
(*b*) The C(1)–C(10) double bond		
Bond lengths (Å)		
C(1)–C(2)	1·508 (9)	1·485 (3)
C(1)–C(10)	1·367 (9)	1·326 (3)
C(9)–C(10)	1·490 (10)	1·500 (3)
C(10)–C(15)	1·475 (12)	1·496 (3)
Bond angles (°)		
C(1)–C(10)–C(9)	120·4 (7)	121·7 (1)
C(1)–C(10)–C(15)	124·0 (7)	123·8 (2)
C(2)–C(1)–C(10)	126·4 (7)	127·7 (2)
C(9)–C(10)–C(15)	115·2 (6)	114·5 (2)
Torsion angle (°)		
C(2)–C(1)–C(10)–C(9)	162·9 (7)	164·6 (2)

* Bovill *et al.* (1976).

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* Lists of structure factors, anisotropic thermal parameters of the non-hydrogen atoms, bond angles for triads containing hydrogen atoms and torsion angles for tetrads containing hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33784 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 7. *Position of the* Ag$^+$ *ions with respect to the* C=C *bonds, and angles between the planes*

M(1) and *M*(2) denote the mid-points of the double bonds C(1)–C(10) and C(4)–C(5) respectively. π(1) and π(2) are the best planes of C(2)–C(1)–C(10)–C(9) and C(3)–C(4)–C(5)–C(6) respectively.

Distances (Å)

Ag(1)–C(2)	3·130 (8)	Ag(2)–C(3)	3·349 (7)
–C(1)	2·319 (7)	–C(4)	2·669 (6)
–*M*(1)	2·273 (7)	–*M*(2)	2·452 (7)
–C(10)	2·425 (8)	–C(5)	2·416 (7)
–C(9)	3·134 (7)	–C(6)	3·243 (6)

Angles (°)

Ag(1)–C(1)–C(10)	77·6 (4)	Ag(2)–C(5)–C(4)	84·8 (3)
–*M*(1)–C(1)	85·3 (4)	–*M*(2)–C(5)	79·0 (4)
–C(10)–C(1)	69·0 (4)	–C(4)–C(5)	64·4 (4)

Angles of planes (°)

π(1)–[C(1)–C(10)–Ag(1)]	92·5 (7)
π(2)–[C(4)–C(5)–Ag(2)]	84·8 (6)

Distances from planes (Å)

Ag(1)–π(1)	2·359 (8)	Ag(2)–π(2)	2·539 (7)

that all other geometric parameters (bond lengths, bond and torsion angles) of the double C=C bonds do not exhibit significant differences. The positions of the Ag$^+$ ions with respect to the C=C bonds are characterized as shown in Table 7. The intraannular distance C(1)–C(4) is 2·857 (10) Å and suggests the presence of some slight electron interaction between these two atoms.

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Octacarbonyl-1,1-dinitrosyl-1-(trimethyl phosphite)-*triangulo*-triosmium

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Abstract. $C_{11}H_9N_2O_{13}Os_3P$, monoclinic, $P2_1/c$, a = 15·653 (5), b = 8·255 (7), c = 17·263 (5) Å, β = 105·15 (5)°, Z = 4, U = 2153 Å^3, D_x = 3·019 g cm^{-3}, μ(Mo $K\alpha$) = 171·4 cm^{-1}. The structure was refined to an R of 0·061 for 1369 unique diffractometer data. The structure is related to that of $Os_3(CO)_{12}$ by substitution of the four carbonyls on one Os atom by an equatorial trimethyl phosphite and two terminal nitrosyls.

Introduction. $Os_3(CO)_8(NO)_2P(OCH_3)_3$ was obtained from the reaction of trimethyl phosphite with $Os_3(CO)_9(NO)_2$, and crystallized as red elongated plates from hexane (Bhaduri, Johnson, Lewis, Watson & Zuccaro, 1977). The crystal structure has been determined to complement chemical and NMR studies of $Os_3(CO)_9(NO)_2$ and its reactions with a variety of ligands (Bhaduri, Johnson, Lewis, Watson & Zuccaro, 1978).

Layers h,0–9,l were collected on a Stoe two-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation. After application of Lp and empirical absorption corrections, equivalent reflexions were averaged to give 1369 reflexions with $F > 6\sigma(F)$ based on counting statistics. a, c and β were determined by a least-squares fit to median ω values for the zero-layer reflexions, and b from the μ angles of the 0k0 reflexions. The Os atoms were located by multisolution $\sum_2$ sign expansion, and the remaining atoms (except H) from difference syntheses. The structure was refined by the full-matrix least-squares method with complex neutral-atom scattering-factors and weights $w = [\sigma^2(F) + 0{\cdot}00136F_o^2]^{-1}$ to $R' = \sum w^{1/2}\Delta/\sum w^{1/2}|F_o| = 0{\cdot}0620$ and a corresponding unweighted R index of 0·0614. Interlayer scale factors were refined, so to avoid a nearly singular least-squares matrix the constraint $U_{22} = \frac{1}{2}(U_{11} + U_{33})$ was applied to the anisotropic Os atoms. The remaining atoms were isotropic; methyl H atoms were not included. Final positional and thermal