0·01 Å in the present case; the dihedral angles between the CuS_2 and S_2C planes are 1·7 and 12·7° in the two determinations. By similar means, we have also prepared the related compounds $Cu(PPh_3)_2(SMe).CS_2$ and $Ag(PPh_3)_2(SPh).CS_2$; presumably the trithiocarbonate formulation applies to these compounds as well.

We thank Drs Fackler and Avdeef for making available information on the structure of $Cu(PPh_3)_2(S_2CSEt)$ in advance of publication.

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Osmium Tetroxide–9-Methylbenzanthracene–Bis(pyridine) Adduct (Toluene Solvate)

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Abstract. $C_{36}H_{24}N_2O_4Os$; monoclinic, $P2_1/c$; a = 11·285 (4), b = 32·507 (6), c = 8·044 (5) Å; β = 93·04° and $Z = 4$. The structure was refined to an R of 9·8%. Distortion from idealized octahedral geometry occurs around the Os atom; the aromatic parts of the molecule are folded in two parts about the saturated C ring.

Introduction. A single crystal of the title adduct (II) (Fig. 1) obtained from toluene was mounted on a PW 1100 four-circle automatic diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. 5506 reflexions were scanned in the θ–2θ mode above the background (3σ). No absorption corrections were made. The position of the Os atom was easily obtained from the Patterson map, and successive Fourier syntheses revealed step by step the whole molecule. An additional molecule of solvent (toluene) was also located in the asymmetric unit.

Refinement was carried out by full-matrix least squares with a modified version of *ORFLS* (Busing, Martin & Levy, 1962). The thermal parameters of the non-hydrogen atoms were anisotropic and the H atoms were situated at their theoretical positions (except those of benzanthracene and toluene methyl groups). The scattering factors were those given in *International Tables for X-ray Crystallography* (1974). The final R is 9·8%.*

The coordinates and thermal parameters for the heavy atoms are listed in Table 1.

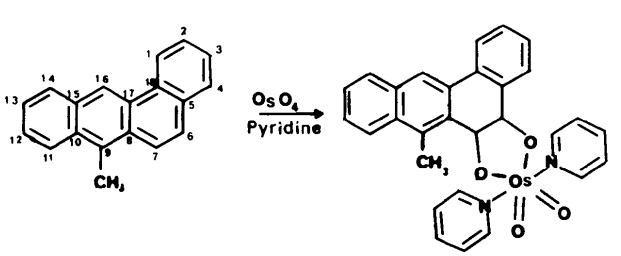

Fig. 1. Synthesis of the title adduct.

Discussion. The addition of osmium tetroxide to olefins gives an adduct which under smooth reductive conditions leads to a pure *cis*-diol (Criegee, 1936). This convenient synthesis can be extended, in some cases, to

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32230 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atomic positional and thermal* ($\times 10^4$) *parameters of the heavy atoms*

Toluene atoms are noted SV(1) to (7) and kept isotropic. Anisotropic thermal factors are of the form:

$$\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)].$$

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B
Os	1494 (1)	1102 (0)	2008 (1)	75 (1)	5 (0)	136 (1)	0 (0)	1 (1)	0 (0)	3·1
O=(1)	271 (9)	1115 (3)	3224 (9)	75 (9)	9 (1)	228 (20)	4 (3)	37 (11)	−5 (4)	4·4
O=(2)	2805 (9)	971 (3)	1163 (9)	74 (10)	10 (1)	168 (18)	2 (3)	16 (11)	−3 (4)	4·1
N′	2528 (9)	1046 (3)	4301 (9)	100 (12)	6 (1)	110 (17)	−1 (3)	44 (12)	1 (3)	3·4
C′(2)	1980 (10)	1023 (4)	5770 (11)	115 (20)	7 (1)	174 (26)	0 (4)	−4 (19)	−3 (5)	4·4
C′(3)	2643 (14)	1002 (4)	7265 (14)	181 (25)	7 (1)	170 (29)	1 (4)	15 (22)	2 (5)	5·6
C′(4)	3845 (12)	998 (5)	7284 (11)	162 (22)	10 (2)	184 (32)	2 (5)	−63 (23)	−5 (6)	5·8
C′(5)	4387 (17)	1019 (5)	5772 (16)	104 (18)	14 (2)	203 (32)	1 (5)	−21 (20)	13 (6)	5·5
C′(6)	3718 (11)	1047 (5)	4398 (17)	87 (17)	11 (2)	172 (30)	3 (5)	−18 (19)	−4 (6)	4·5
N″	1191 (9)	468 (4)	1950 (9)	81 (13)	16 (1)	42 (14)	5 (3)	−31 (12)	4 (4)	4·0
C″(2)	1759 (13)	190 (5)	1090 (11)	104 (19)	8 (1)	225 (32)	3 (4)	−19 (21)	−4 (6)	4·9
C″(3)	1555 (11)	−222 (5)	893 (12)	168 (24)	8 (2)	233 (40)	8 (5)	−36 (26)	12 (7)	6·1
C″(4)	623 (15)	−396 (5)	1672 (17)	207 (27)	6 (2)	248 (36)	−12 (5)	−70 (26)	7 (6)	6·6
C″(5)	−40 (12)	−128 (5)	2561 (10)	141 (21)	9 (2)	228 (34)	−9 (5)	10 (22)	11 (6)	5·6
C″(6)	216 (15)	285 (5)	2651 (13)	105 (17)	10 (2)	140 (26)	−1 (4)	11 (17)	−6 (5)	4·4
O–(1)	600 (9)	1212 (2)	−55 (11)	91 (10)	5 (1)	116 (14)	0 (2)	32 (10)	5 (3)	3·2
O–(2)	1896 (11)	1690 (3)	2014 (10)	113 (13)	8 (1)	310 (28)	−9 (3)	−87 (16)	4 (5)	5·9
C(1)	2833 (14)	2055 (5)	−3456 (13)	67 (15)	9 (2)	246 (41)	−3 (4)	34 (20)	10 (6)	4·5
C(2)	3974 (11)	2090 (5)	−2887 (12)	97 (17)	10 (2)	260 (38)	4 (4)	58 (21)	11 (6)	5·2
C(3)	4325 (14)	2049 (5)	−1250 (17)	75 (17)	10 (2)	371 (44)	−4 (4)	22 (22)	−1 (7)	5·9
C(4)	3438 (16)	1970 (5)	−76 (16)	90 (17)	9 (2)	240 (37)	−1 (4)	−21 (21)	−8 (6)	4·9
C(5)	2263 (11)	1946 (4)	−649 (15)	80 (15)	3 (1)	229 (32)	−3 (3)	17 (18)	5 (5)	3·8
C(6)	1338 (14)	1892 (4)	496 (18)	98 (15)	5 (1)	188 (26)	−3 (4)	14 (16)	3 (5)	4·0
C(7)	274 (14)	1647 (4)	−129 (14)	92 (15)	5 (1)	175 (25)	−4 (4)	−8 (15)	0 (5)	3·7
C(8)	−130 (12)	1762 (4)	−1830 (12)	80 (15)	5 (1)	156 (23)	2 (3)	14 (10)	2 (4)	3·4
C(9)	−1336 (16)	1720 (5)	−2342 (18)	87 (17)	8 (2)	177 (28)	5 (4)	−44 (18)	−1 (5)	4·2
C(10)	−1737 (14)	1851 (4)	−3930 (13)	72 (15)	7 (1)	165 (29)	1 (4)	−1 (17)	−1 (5)	3·6
C(11)	−2953 (15)	1851 (5)	−4485 (15)	105 (18)	10 (2)	159 (30)	1 (4)	−13 (19)	6 (5)	4·6
C(12)	−3306 (16)	2014 (5)	−5989 (17)	113 (19)	10 (2)	326 (38)	10 (5)	−32 (23)	10 (6)	6·1
C(13)	−2492 (11)	2174 (6)	−7033 (12)	132 (23)	12 (2)	198 (39)	3 (6)	−22 (25)	14 (7)	5·6
C(14)	−1350 (14)	2182 (5)	−6582 (15)	117 (22)	6 (1)	283 (40)	3 (4)	22 (24)	4 (6)	5·2
C(15)	−931 (15)	2020 (4)	−5022 (11)	92 (16)	5 (1)	211 (32)	−1 (4)	2 (19)	1 (5)	4·1
C(16)	295 (15)	2046 (4)	−4487 (18)	115 (17)	5 (1)	156 (25)	4 (4)	56 (17)	0 (4)	3·9
C(17)	681 (14)	1930 (4)	−2901 (12)	77 (14)	3 (1)	184 (28)	2 (3)	23 (16)	3 (4)	3·3
C(18)	1959 (14)	1973 (4)	−2305 (17)	82 (15)	4 (1)	246 (34)	−1 (3)	−2 (19)	7 (5)	4·1
Me(9)	−2208 (7)	1506 (3)	−1253 (6)	97 (9)	9 (1)	116 (17)	1 (4)	12 (13)	7 (3)	3·8

	x	y	z	B
SV(1)	−2708 (27)	4089 (9)	8142 (35)	8·70
SV(2)	−2359 (30)	4302 (11)	9494 (42)	7·19
SV(3)	−3633 (27)	4241 (9)	7171 (35)	9·60
SV(4)	−4166 (30)	4645 (10)	7315 (38)	8·47
SV(5)	−2906 (32)	4700 (11)	9775 (42)	7·49
SV(6)	−3741 (34)	4834 (11)	8668 (45)	9·02
SV(7)	−3830 (28)	4071 (10)	5901 (38)	11·13

aromatic compounds. The 6,7 double bond of 9-methylbenzanthracene (I) exhibits sufficient localization to react with peroxide reagents in the synthesis of enhanced carcinogenic epoxide derivatives (Marquard *et al.*, 1972). When this hydrocarbon (I) is submitted to the action of osmium tetroxide it shows poor reactivity, but the addition of pyridine readily accelerates the process towards a stable pyridine-containing adduct, II [Criegee, Marchand & Wannowius, 1942; Fieser & Fieser, 1967 (review of OsO_4 reactions); Silverston, Dansette & Jerina, 1976; Perrin-Roussel, Perrin & Jacquignon, 1974].

The X-ray study of this latter compound has been undertaken to determine which ligands of the Os atom are involved in the complexation of pyridines and the

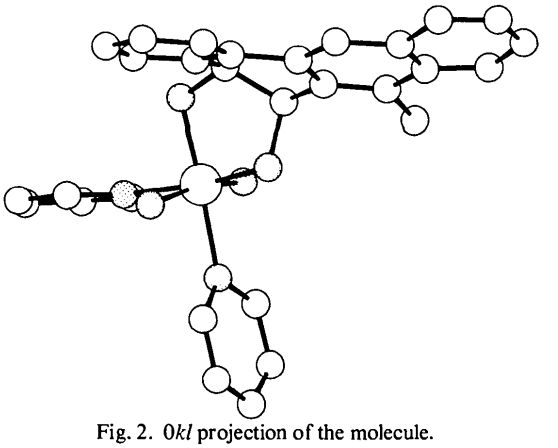

Fig. 2. 0*kl* projection of the molecule.

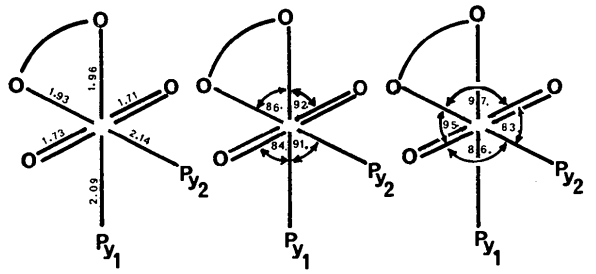

Fig. 3. Distances and angles around the Os atom.

spatial surrounding of this atom to obtain information about the activated complex of this addition.

The 0*kl* projection of the molecule displayed in Fig. 2 indicates that both pyridine rings are *trans* to the O–Os bonds of the benzanthracene moiety. Distances and angles around the Os atom are given in Fig. 3.

Both Figs. 2 and 3 reveal that important distortion from idealized octahedral geometry occurs around the Os atom.

As reported in other 6,7-dihydro compounds of benzanthracene (Glusker, Carrel, Zacharias & Harvey, 1974) the aromatic parts of the molecule are folded in two parts about the saturated *C* ring by an angle of 20°.

The toluene molecule was found to be more thermally agitated than the associated Os complex and some difficulties have been encountered in determining accurately the coordinates of its methyl group.

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Cinnzeylanine, A New Pentacyclic Diterpene Acetate from *Cinnamonum zeylanicum*

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Abstract. $C_{22}H_{34}O_8$, M_r = 426·51; orthorhombic, $P2_12_12_1$, a = 12·195 (2), b = 13·476 (2), c = 12·891 (1) Å, Z = 4, V = 2118·6 (5) Å^3, D_x = 1·35, D_m = 1·34 g cm^{-3} (flotation); λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 1·371 cm^{-1}. The structure was solved by the direct method and refined by block-diagonal least squares. The final R value was 0·067 for 2442 reflexions. The skeleton of the compound is very similar to that of ryanodine [Wiesner, *Advanc. Org. Chem.* (1972), **8**, 295–316].

Introduction. Cinnzeylanine is a new compound isolated from the dried bark of *Cinnamonum zeylanicum* Nees as an insect-growth regulator (Isogai, Suzuki, Tamura, Murakoshi, Ohashi & Sasada, 1976). Prismatic crystals were obtained by recrystallization from ethyl acetate–benzene. Intensity data up to $2\theta \leq 55°$ were collected with a crystal, 0·2 × 0·5 × 0·5 mm, on a Rigaku four-circle diffractometer and Mo $K\alpha$ radiation monochromatized by graphite; an ω–2θ scan at 4° (2θ) min^{-1} was employed. A Lorentz–polarization