Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

V—S(1)	2·361 (1)	V—S(2)	2·335 (1)
V—N(1)	2·151 (3)	V—N(2)	2·164 (3)
V—O(1)	1·596 (2)	S(1)—C(1)	1·816 (4)
S(2)—C(8)	1·813 (5)	N(1)—C(2)	1·489 (4)
N(1)—C(3)	1·479 (5)	N(1)—C(4)	1·482 (5)
N(2)—C(5)	1·461 (5)	N(2)—C(6)	1·491 (6)
N(2)—C(7)	1·471 (5)	C(1)—C(2)	1·496 (5)
C(4)—C(5)	1·262 (7)	C(7)—C(8)	1·481 (6)
S(1)—V—S(2)	91·6 (1)	S(1)—V—N(1)	83·0 (1)
S(1)—V—N(2)	149·2 (1)	S(1)—V—O(1)	107·3 (1)
S(2)—V—N(1)	138·1 (1)	S(2)—V—N(2)	83·9 (1)
S(2)—V—O(1)	112·3 (1)	N(1)—V—N(2)	80·4 (1)
N(1)—V—O(1)	109·0 (1)	N(2)—V—O(1)	102·6 (1)
V—S(1)—C(1)	100·3 (1)	V—S(2)—C(8)	100·1 (2)

Fig. 1. Molecular structure and crystallographic numbering scheme employed for [VO(C₈H₁₈N₂S₂)] (Johnson, 1971).

tering factors for neutral V atom corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149) and for the remaining atoms as incorporated in *SHELX76* (Sheldrick, 1976). All calculations on a Sun4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2* and numbering scheme used is shown in Fig. 1 which was drawn with *ORTEP* (Johnson, 1971) at 25% probability levels.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, mean-plane data and all interatomic parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53501 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The structure determination of the title compound forms part of a wider study of complexes of this type (Wilson *et al.*, 1988; Wedd & Spence, 1990).

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Structure of Di[(carbonyl)(chloro)(1,4,7-triazacyclononane)(triphenylphosphine)-osmium(II)] Tetrachlorozincate(II)

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Abstract. [OsCl(CO){P(C₆H₅)₃}(C₆H₁₅N₃)]₂[ZnCl₄], $M_r = 1497·51$, triclinic, $P\bar{1}$, $a = 16·000$ (4), $b = 17·262$ (5), $c = 10·432$ (3) Å, $\alpha = 94·52$ (2), $\beta = 92·22$ (2), $\gamma = 88·78$ (2)°, $V = 2870$ (3) Å³, $Z = 2$, $D_x = 1·7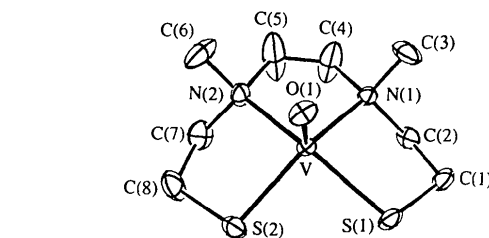3$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 52·25$ cm⁻¹, $F(000) = 1464$, $T = 296$ K, $R = 0·045$, 6364 unique observed reflections. The reaction of the dimer, {OsBr₂(CO)[P(C₆H₅)₃]₂}₂, with an equimolar amount of the cyclic amine 1,4,7-triazacyclononane (containing ZnCl₂ as an impurity) yielded a few clear crystals of the title compound as a minor product of the reaction. The structure is comprised of two

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chemically identical, chiral, monocationic osmium complexes and an associated $ZnCl_4^{2-}$ ion. The Os atoms in the complexes are six coordinate having bonds to Cl, C, P and three N atoms of the macro-cyclic ligand. The average Os—N bond length is 2·157 Å, and the average N—Os—N bond angle is 78·87°.

Experimental. The precursor compound, $\{OsBr_2(CO)[P(C_6H_5)_3]_2\}_2$ (I), prepared by refluxing di(tetra-*n*-butylammonium) hexabromoosmate(IV) with an eightfold excess triphenylphosphine in mixed acetic acid/acetic anhydride solution for 17 h. Several good crystals of the title compound (II) were obtained as a minor product of the reaction between (I) (0·1464 g, 0·08 mmol) and 1,4,7-triazacyclo-nonane (0·0217 g, 0·168 mmol, Aldrich) in 1,1,2-trichloroethane (*ca* 25 ml, MCB reagents), refluxed under N_2 for 40 h. In this preparation, zinc chloride was present as a minor impurity in the triaza ligand as supplied. An alternative preparation was devised in which (I) (0·3503 g, 0·198 mmol), 1,4,7-triazacyclononane (0·0504 g, 0·390 mmol), and $(NH_4)_2[ZnCl_4]$ (0·0483 g, 0·198 mmol, Alfa) were refluxed in 40 ml 1,1,2-trichloroethane. Solid $(NH_4)_2[ZnCl_4]$ was present during reflux. Crystals obtained by layering ethyl ether above a 1,1,2 tri-chloroethane solution of the compound.

(I)   (II)
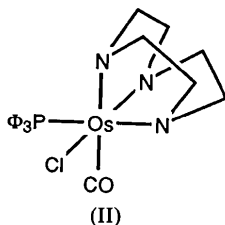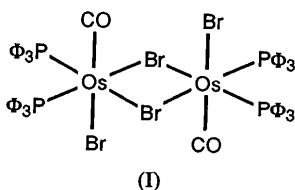
Crystal used for data collection 0·27 × 0·19 × 0·16 mm, colorless, transparent, fragment. Rigaku AFC5S diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω scans, scan speed 6° min⁻¹ (in ω), weak reflections $[I < 10·0\sigma(I)]$ rescanned (maximum of two rescans) and the counts accumulated to assure good counting statistics. Lattice parameters from least-squares fit of 25 strong reflections in 2θ range 38–42°. A total of 10093 reflections measured (h 0 to 19, k − 20 to 20, l − 12 to 12), 3729 reflections considered unobserved with $[I < 3\sigma(I)]$, data set comprised 6364 unique reflections, $(\sin\theta/\lambda)_{max} = 0·60$ Å⁻¹. Three standard reflections ($\overline{3}22$, $\overline{1}11$, $\overline{1}1\overline{2}$) changed by 0·4, 1·8 and − 1·0%, respectively; no decay correction applied. Data corrected for Lorentz, polarization and absorption (ψ scans, three reflections, transmission range 0·91–1·00). A Patterson synthesis provided the location of one of the Os atoms. The direct-methods phase-extension program *DIRDIF* (Beurskens, 1984) provided the loca-

Table 1. *Positional and equivalent isotropic thermal parameters for the non-H atoms with e.s.d.'s in parentheses*

$B_{eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33} + 2U_{12}\cos\gamma + 2U_{13}\cos\beta + 2U_{23}\cos\alpha).$

	x	y	z	B_{eq}(Å²)
Os(1)	0·15322 (3)	0·19666 (3)	0·34246 (4)	2·29 (2)
Os(2)	0·55013 (3)	0·26810 (3)	0·08040 (5)	2·80 (2)
Zn(1)	0·2287 (1)	0·0170 (1)	0·8028 (2)	3·56 (7)
Cl(1)	0·1667 (2)	0·2599 (2)	0·1425 (3)	2·7 (1)
Cl(2)	0·4692 (2)	0·3892 (2)	0·1128 (4)	3·7 (1)
Cl(3)	0·0929 (2)	0·0574 (2)	0·8078 (4)	4·1 (2)
Cl(4)	0·2924 (2)	0·0190 (2)	1·0030 (3)	4·0 (2)
Cl(5)	0·2943 (2)	0·1001 (3)	0·6827 (4)	5·4 (2)
Cl(6)	0·2428 (3)	−0·1084 (2)	0·7182 (5)	6·0 (2)
P(1)	0·1153 (2)	0·3169 (2)	0·4415 (3)	2·6 (1)
P(2)	0·6370 (2)	0·3200 (2)	−0·0607 (4)	3·1 (1)
O(1)	0·3282 (6)	0·2180 (6)	0·433 (1)	4·4 (5)
O(2)	0·6639 (8)	0·3072 (8)	0·305 (1)	6·4 (7)
N(1)	0·1210 (7)	0·1200 (6)	0·482 (1)	3·2 (5)
N(2)	0·1762 (6)	0·0848 (6)	0·238 (1)	3·2 (5)
N(3)	0·0276 (6)	0·1626 (6)	0·274 (1)	3·2 (5)
N(4)	0·4592 (6)	0·2179 (6)	0·195 (1)	3·6 (5)
N(5)	0·4579 (6)	0·2276 (7)	−0·069 (1)	3·6 (5)
N(6)	0·5849 (6)	0·1483 (6)	0·051 (1)	3·2 (5)
C(1)	0·1898 (8)	0·3967 (7)	0·447 (1)	3·0 (6)
C(2)	0·2553 (8)	0·3947 (8)	0·365 (1)	3·8 (7)
C(3)	0·3109 (9)	0·4554 (9)	0·369 (1)	4·3 (7)
C(4)	0·301 (1)	0·519 (1)	0·456 (2)	5·4 (8)
C(5)	0·236 (1)	0·522 (1)	0·535 (2)	7 (1)
C(6)	0·183 (1)	0·461 (1)	0·531 (2)	6 (1)
C(7)	0·0233 (7)	0·3550 (8)	0·356 (1)	3·3 (6)
C(8)	0·0301 (9)	0·409 (1)	0·267 (1)	4·5 (7)
C(9)	−0·037 (1)	0·427 (1)	0·188 (2)	6 (1)
C(10)	−0·112 (1)	0·387 (1)	0·198 (2)	7 (1)
C(11)	−0·120 (1)	0·335 (1)	0·284 (2)	6 (1)
C(12)	−0·0533 (8)	0·3200 (8)	0·364 (1)	4·0 (7)
C(13)	0·0845 (8)	0·3184 (7)	0·610 (1)	3·1 (6)
C(14)	0·0159 (9)	0·359 (1)	0·655 (1)	4·4 (7)
C(15)	−0·004 (1)	0·358 (1)	0·783 (2)	6 (1)
C(16)	0·045 (1)	0·313 (1)	0·865 (1)	6 (1)
C(17)	0·115 (1)	0·273 (1)	0·820 (1)	5·1 (8)
C(18)	0·134 (1)	0·2773 (8)	0·692 (1)	3·8 (6)
C(19)	−0·0165 (8)	0·1219 (8)	0·374 (1)	4·1 (7)
C(20)	0·0296 (8)	0·1285 (8)	0·502 (1)	3·8 (6)
C(21)	0·146 (1)	0·0386 (8)	0·443 (1)	4·0 (7)
C(22)	0·206 (1)	0·0331 (8)	0·337 (1)	4·4 (7)
C(23)	0·1029 (8)	0·0512 (8)	0·165 (1)	3·5 (6)
C(24)	0·0361 (9)	0·1148 (9)	0·146 (1)	4·1 (7)
C(25)	0·268 (1)	0·2099 (8)	0·404 (1)	3·8 (7)
C(26)	0·5799 (8)	0·3395 (8)	−0·210 (1)	3·3 (6)
C(27)	0·5387 (8)	0·4105 (8)	−0·221 (1)	3·8 (6)
C(28)	0·489 (1)	0·424 (1)	−0·328 (2)	5·3 (8)
C(29)	0·475 (1)	0·364 (1)	−0·421 (2)	7 (1)
C(30)	0·513 (1)	0·293 (1)	−0·411 (2)	7 (1)
C(31)	0·564 (1)	0·282 (1)	−0·306 (2)	5·5 (9)
C(32)	0·6858 (8)	0·4140 (8)	−0·014 (2)	3·9 (7)
C(33)	0·743 (1)	0·443 (1)	−0·090 (1)	5·1 (8)
C(34)	0·780 (1)	0·514 (1)	−0·056 (2)	7 (1)
C(35)	0·761 (1)	0·557 (1)	0·055 (2)	6 (1)
C(36)	0·704 (1)	0·529 (1)	0·132 (2)	7 (1)
C(37)	0·666 (1)	0·4571 (9)	0·099 (2)	5·4 (9)
C(38)	0·7278 (9)	0·2606 (8)	−0·111 (2)	4·1 (7)
C(39)	0·7709 (9)	0·221 (1)	−0·018 (2)	4·9 (8)
C(40)	0·842 (1)	0·178 (1)	−0·046 (2)	7 (1)
C(41)	0·874 (1)	0·175 (1)	−0·163 (3)	8 (1)
C(42)	0·832 (1)	0·215 (1)	−0·263 (2)	8 (1)
C(43)	0·762 (1)	0·258 (1)	−0·230 (2)	7 (1)
C(44)	0·3810 (8)	0·197 (1)	0·119 (1)	4·5 (7)
C(45)	0·3741 (8)	0·2343 (9)	−0·006 (1)	4·3 (7)
C(46)	0·4778 (8)	0·1483 (8)	−0·123 (1)	4·0 (6)
C(47)	0·5649 (8)	0·1224 (8)	−0·087 (1)	3·4 (6)
C(48)	0·5401 (9)	0·1001 (8)	0·142 (1)	4·2 (7)
C(49)	0·501 (1)	0·1494 (9)	0·250 (1)	4·4 (7)
C(50)	0·627 (1)	0·2940 (9)	0·227 (2)	3·9 (7)

tion of the second Os atom and six of the surrounding ligands. The remaining non-H atoms were found by least-squares/difference Fourier synthesis techniques. Full-matrix least-squares refinement was performed to minimize $\Sigma w(|F_o| - $

Table 2. *Selected bond distances (Å) and bond angles (°) with e.s.d.'s in parentheses*

Molecule I

Os(1)—Cl(1)	2·447 (3)	N(1)—C(20)	1·49 (2)
Os(1)—P(1)	2·324 (3)	N(1)—C(21)	1·48 (2)
Os(1)—N(1)	2·12 (1)	N(2)—C(22)	1·47 (2)
Os(1)—N2)	2·17 (1)	N(2)—C(23)	1·48 (1)
Os(1)—N(3)	2·18 (1)	N(3)—C(19)	1·51 (2)
Os(1)—C(25)	1·93 (2)	N(3)—C(24)	1·52 (2)
O(1)—C(25)	1·01 (2)	C(19)—C(20)	1·50 (2)
P(1)—C(1)	1·84 (1)	C(21)—C(22)	1·49 (2)
P(1)—C(7)	1·83 (1)	C(23)—C(24)	1·53 (2)
P(1)—C(13)	1·85 (1)		

Cl(1)—Os(1)—P(1)	88·3 (1)	C(1)—P(1)—C(7)	103·6 (6)
Cl(1)—Os(1)—N(1)	163·9 (3)	C(1)—P(1)—C(13)	102·6 (6)
Cl(1)—Os(1)—N(2)	89·5 (3)	C(7)—P(1)—C(13)	104·4 (6)
Cl(1)—Os(1)—N(3)	88·0 (3)	Os(1)—N(1)—C(20)	108·3 (8)
Cl(1)—Os(1)—C(25)	96·4 (4)	Os(1)—N(1)—C(21)	111·6 (8)
P(1)—Os(1)—N(1)	101·9 (3)	C(20)—N(1)—C(21)	112 (1)
P(1)—Os(1)—N(2)	174·0 (3)	Os(1)—N(2)—C(22)	105·3 (8)
P(1)—Os(1)—N(3)	96·7 (3)	Os(1)—N(2)—C(23)	115·0 (8)
P(1)—Os(1)—C(25)	91·0 (4)	C(22)—N(2)—C(23)	110 (1)
N(1)—Os(1)—N(2)	79·1 (4)	Os(1)—N(3)—C(19)	111·2 (8)
N(1)—Os(1)—N(3)	78·5 (4)	Os(1)—N(3)—C(24)	107·4 (7)
N(1)—Os(1)—C(25)	95·9 (5)	C(19)—N(3)—C(24)	114 (1)
N(2)—Os(1)—N(3)	77·6 (4)	N(3)—C(19)—C(20)	112 (1)
N(2)—Os(1)—C(25)	94·8 (5)	N(1)—C(20)—C(19)	109 (1)
N(3)—Os(1)—C(25)	171·2 (5)	N(1)—C(21)—C(22)	112 (1)
Os(1)—C(25)—O(1)	178 (1)	N(2)—C(22)—C(21)	109 (1)
Os(1)—P(1)—C(1)	118·9 (4)	N(2)—C(23)—C(24)	110 (1)
Os(1)—P(1)—C(7)	109·1 (4)	N(3)—C(24)—C(23)	108 (1)
Os(1)—P(1)—C(13)	116·6 (4)		

Molecule II

Os(2)—Cl(2)	2·443 (3)	N(4)—C(44)	1·49 (2)
Os(2)—P(2)	2·307 (4)	N(4)—C(49)	1·49 (2)
Os(2)—N(4)	2·15 (1)	N(5)—C(45)	1·52 (2)
Os(2)—N(5)	2·19 (1)	N(5)—C(46)	1·47 (2)
Os(2)—N(6)	2·13 (1)	N(6)—C(47)	1·50 (2)
Os(2)—C(50)	1·95 (2)	N(6)—C(48)	1·52 (2)
O(2)—C(50)	1·00 (2)	C(44)—C(45)	1·50 (2)
P(2)—C(26)	1·83 (1)	C(46)—C(47)	1·50 (2)
P(2)—C(32)	1·84 (1)	C(48)—C(49)	1·51 (2)
P(2)—C(38)	1·83 (2)		

Cl(2)—Os(2)—P(2)	92·7 (1)	C(26)—P(2)—C(32)	102·0 (6)
Cl(2)—Os(2)—N(4)	86·3 (3)	C(26)—P(2)—C(38)	105·1 (7)
Cl(2)—Os(2)—N(5)	89·0 (3)	C(32)—P(2)—C(38)	101·3 (6)
Cl(2)—Os(2)—N(6)	163·1 (3)	Os(2)—N(4)—C(44)	112·4 (9)
Cl(2)—Os(2)—C(50)	93·6 (4)	Os(2)—N(4)—C(49)	106·9 (8)
P(2)—Os(2)—N(4)	173·7 (3)	C(44)—N(4)—C(49)	112 (1)
P(2)—Os(2)—N(5)	94·5 (3)	Os(2)—N(5)—C(45)	105·6 (8)
P(2)—Os(2)—N(6)	100·0 (3)	Os(2)—N(5)—C(46)	111·6 (7)
P(2)—Os(2)—C(50)	92·1 (4)	C(45)—N(5)—C(46)	113 (1)
N(4)—Os(2)—N(5)	79·2 (4)	Os(2)—N(6)—C(47)	107·7 (8)
N(4)—Os(2)—N(6)	79·9 (4)	Os(2)—N(6)—C(48)	111·0 (8)
N(4)—Os(2)—C(50)	94·1 (5)	C(47)—N(6)—C(48)	112 (1)
N(5)—Os(2)—N(6)	78·9 (4)	N(4)—C(44)—C(45)	112 (1)
N(5)—Os(2)—C(50)	172·7 (5)	N(5)—C(45)—C(44)	109 (1)
N(6)—Os(2)—C(50)	97·0 (5)	N(5)—C(46)—C(47)	113 (1)
Os(2)—C(50)—O(2)	177 (2)	N(6)—C(47)—C(46)	110 (1)
Os(2)—P(2)—C(26)	110·8 (4)	N(6)—C(48)—C(49)	113 (1)
Os(2)—P(2)—C(32)	119·0 (5)	N(4)—C(49)—C(48)	108 (1)
Os(2)—P(2)—C(38)	116·8 (5)		

Convergence yielded $R = 0.045$, $wR = 0.056$, $S = 1.45$ and $(\Delta/\sigma)_{max} = 0.00$. Final difference synthesis produced $(\Delta\rho)_{max} = 1.98$ e Å^{-3} (at a seemingly random intermolecular position), and $(\Delta\rho)_{min} = -1.73$ e Å^{-3} [near the Os(2) position]. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148). Positional and equivalent isotropic thermal parameters are given in Table 1. Table 2 presents selected interatomic distances and angles.* Fig. 1 shows one of the two chiral osmium

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* Calculated and observed structure factors, anisotropic thermal parameters, calculated H-atom coordinates, distances and angles involving the phenyl rings and zinc tetrachloride, intramolecular distances and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53510 (76 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
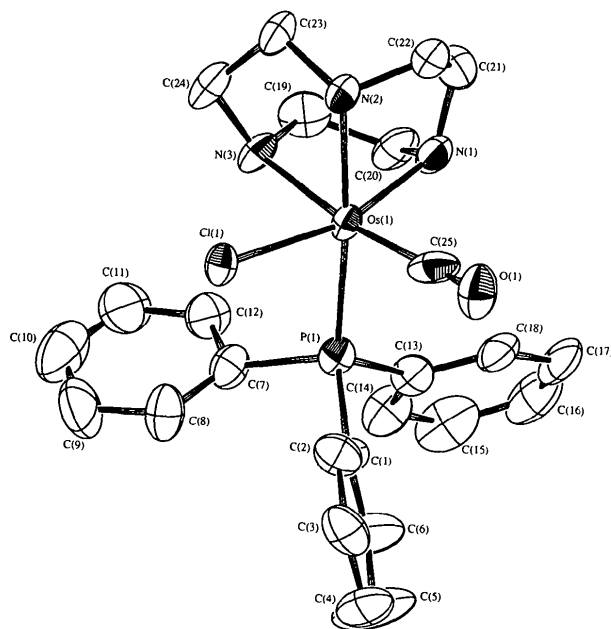

Fig. 1. Molecular configuration and atom-numbering scheme for the non-H atoms, thermal ellipsoids at the 50% probability level. H atoms omitted to improve clarity.

$|F_c|)^2$, where $w = 4F_o^2/\sigma^2(F_o^2)$ and $\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/Lp^2$ (S = scan rate, C = total integrated peak count, R = ratio of scan time to background counting time, B = total background count, Lp = Lorentz–polarization factor, and $p = 0.04$). After convergence phenyl, methylene and tetrahedral H atoms placed at idealized positions (C—H or N—H = 0·95 Å), and fixed (isotropic B's for H atoms calculated as $1·2 \times B_{eq}$ of associated C or N atom). Final stages of refinement performed with 622 variables including all non-H positional and anisotropic thermal parameters and one scale factor.
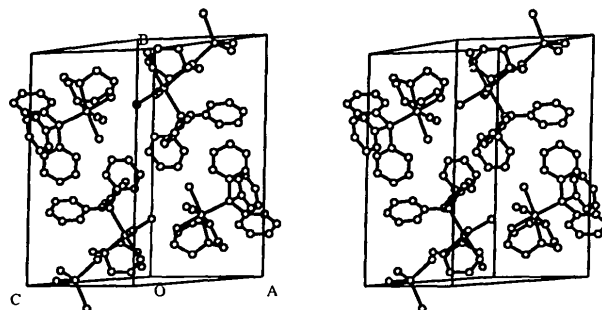

Fig. 2. Stereoscopic illustration of the molecular packing.

complexes while Fig. 2 illustrates the molecular packing and the chiral nature of the two complexes. All computer programs from the *TEXSAN* crystal-structure-analysis package (Molecular Structure Corporation, 1985).

Related literature. The title compound is the first reported osmium compound containing the 1,4,7-triazacyclononane macrocycle. Compounds of this ligand with ruthenium have been reported by Wieghardt, Herrmann, Koppen, Jibril & Huttner (1984).

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Structure of Dibromobis(triphenylarsine oxide)nickel(II), NiBr₂(Ph₃AsO)₂

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Abstract. $C_{36}H_{30}As_2Br_2NiO_2$, $M_r = 863.01$, orthorhombic, $Pca2_1$, $a = 18.826 (5)$, $b = 10.594 (6)$, $c = 17.402 (6)$ Å, $V = 3470 (4)$ Å^3, $Z = 4$, $D_x = 1.652$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0.71073$ Å, $\mu = 47.60$ cm^{-1}, $F(000) = 1704$, $T = 298$ K, $R = 0.059$ for 724 observed reflections. The Ni^{II} atom is coordinated to two Br ions [Br—Ni = 2.39 (1), 2.364 (8) Å] and to the O atoms of the (Ph₃AsO) groups [Ni—O = 1.94 (3), 2.00 (3) Å] in a distorted tetrahedral configuration [Br—Ni—Br = 123.1 (5)°].

Experimental. Synthesis of the title compound was performed as previously reported (Oliva, Castellano, Zukerman-Schpector & Massabni, 1984). The data collection and refinement parameters for the title compound are summarized in Table 1.

The structure was solved by Patterson and difference Fourier synthesis techniques. In the final cycles of blocked-matrix refinement only the heavy atoms As, Br and Ni were refined anisotropically; the phenyl rings were treated as rigid groups with individual isotropic temperature factors for the C atoms and a fixed isotropic temperature factor ($B = 4.7$ Å^2) for the H atoms. Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with corrections for anomalous dispersion from Cromer & Liberman (1970) and for H atoms from Stewart, Davidson & Simpson (1965). Programs used were *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

Final atomic coordinates are listed in Table 2,* selected interatomic distances and angles are given in Table 3. Fig. 1 is a stereoscopic projection of the complex.

Related literature. In a previous paper (Oliva, Castellano, Zukerman-Schpector & Massabni, 1984), the synthesis and the crystal structures of the two complexes NiBr₂.4(Ph₃AsO).8H₂O (green) and NiBr₂.4(Ph₃AsO).3/2(C₆H₅CH₃).H₂O (orange) obtained from the title compound (blue) by recrystalli-
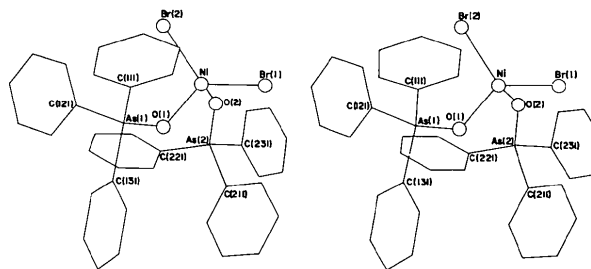

Fig. 1. Stereoscopic projection of the complex.