

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).

This research was supported in part by the Research Corporation.

References

- BEURSKENS, P. T. (1984). *DIRDIF*. Tech. Rep. 1984/1. Crystallography Laboratory, Toernooiveld, 6525 Ed. Nijmegen, The Netherlands.
- Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX77381, USA.
- WIEGHARDT, K., HERRMANN, W., KOPPEN, M., JIBRIL, I. & HUTTNER, G. (1984). *Z. Naturforsch. Teil B*, **39**, 1335–1343.

Acta Cryst. (1991). **C47**, 654–655

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

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(Received 4 June 1990; accepted 17 August 1990)

Abstract. C₃₆H₃₀As₂Br₂NiO₂, *M_r* = 863.01, orthorhombic, *Pca*2₁, *a* = 18.826 (5), *b* = 10.594 (6), *c* = 17.402 (6) Å, *V* = 3470 (4) Å³, *Z* = 4, *D_x* = 1.652 g cm⁻³, λ(Mo *Kα*) = 0.71073 Å, μ = 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 Å²) 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 *SHELX76* (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-

* Lists of structure factors, anisotropic thermal parameters, a complete list of C-atom parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53493 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

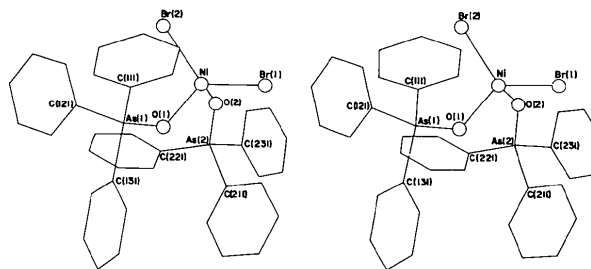


Fig. 1. Stereoscopic projection of the complex.

Table 1. *Crystallographic summary for NiBr₂(Ph₃AsO)₂*

Data collection ^{a,b}	
Mode	$\theta-2\theta$
Scan rate ($^{\circ}\text{min}^{-1}$)	1.9–6.7
θ range ($^{\circ}$)	0–22
Range of hkl	$0 \leq h \leq 19, 0 \leq k \leq 11, 0 \leq l \leq 18$
Unique reflections	2209
Crystal dimensions approx. (mm)	$0.08 \times 0.18 \times 0.30$
Structure refinement ^{c,d}	
Reflections used [$I > 2\sigma(I)$]	724
Number of variables	126
R, wR	0.059, 0.061
S	0.75
Max. shift/e.s.d.	0.1
Max., min. density in difference map ($\text{e}\ \text{\AA}^{-3}$)	0.55, –0.53

(a) Unit-cell parameters by least-squares refinement of the setting angles of 23 reflections with $10 < \theta < 22^{\circ}$.

(b) Enraf–Nonius CAD-4 diffractometer with graphite monochromator was used. Standard reflections showed no significant variation.

(c) The intensities were corrected for L_p and for absorption: min. and max. values 0.85–1.02 (Walker & Stuart, 1983).

(d) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = \sigma^2(|F_o|) + 0.0075|F_o|^2$.

Table 2. *Fractional atomic coordinates and isotropic temperature factors (\AA^2) (only two C atoms for each rigid phenyl ring are given)*

	x	y	z	B_{iso}
Br(1)	0.4470 (3)	0.6576 (5)	0.4263 (7)	5.8 (2)
Br(2)	0.4620 (3)	1.0272 (5)	0.3441 (7)	5.1 (2)
Ni	0.4762 (3)	0.8070 (6)	0.3285 (7)	3.7 (2)
As(1)	0.6208 (2)	0.7067 (5)	0.2423 (7)	3.7 (2)
As(2)	0.3572 (2)	0.8114 (5)	0.1875 (7)	3.3 (2)
O(1)	0.577 (2)	0.783 (3)	0.310 (2)	5.0 (8)
O(2)	0.430 (2)	0.756 (3)	0.230 (2)	5.7 (9)
C(111)	0.281 (1)	0.840 (3)	0.256 (2)	4 (1)
C(112)	0.221 (1)	0.908 (3)	0.231 (2)	5 (1)
C(121)	0.371 (1)	0.963 (2)	0.133 (2)	2.4 (9)
C(122)	0.365 (1)	1.081 (2)	0.168 (2)	6 (1)
C(131)	0.333 (2)	0.682 (3)	0.115 (2)	5 (1)
C(132)	0.264 (2)	0.665 (3)	0.087 (2)	5 (1)
C(211)	0.583 (1)	0.546 (2)	0.226 (2)	6 (1)
C(212)	0.561 (1)	0.475 (2)	0.289 (2)	7 (1)
C(221)	0.630 (2)	0.803 (3)	0.152 (2)	4 (1)
C(222)	0.692 (2)	0.803 (3)	0.108 (2)	5 (1)
C(231)	0.713 (1)	0.673 (3)	0.277 (2)	4 (1)
C(232)	0.752 (1)	0.565 (3)	0.261 (2)	5 (1)

Acta Cryst. (1991). **C47**, 655–657

Structure of a Bis{[(2,3- $\eta, \kappa P'$)-1,2-diphospha-2-propene]nickel}

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(Received 30 July 1990; accepted 31 August 1990)

Abstract. Bis[μ -(3,4- $\eta, \kappa P^5$)-5-chloro-2,2,6,6-tetramethyl-3-phenyl-4,5-diphospha-2-silahept-3-ene]bis[carbonylnickel(0)], $\text{C}_{30}\text{H}_{46}\text{Cl}_2\text{Ni}_2\text{O}_2\text{P}_4\text{Si}_2$, $M_r = 10108.2701/91/030655-03\text{S}03.00$

Table 3. *Selected interatomic distances (\AA) and angles ($^{\circ}$)*

Br(1)—Ni	2.39 (1)	As(1)—C(221)	1.88 (3)
Br(2)—Ni	2.364 (8)	As(1)—C(231)	1.87 (2)
Ni—O(1)	1.94 (3)	As(2)—O(2)	1.66 (3)
Ni—O(2)	2.00 (3)	As(2)—C(111)	1.89 (3)
As(1)—O(1)	1.65 (3)	As(2)—C(121)	1.88 (3)
As(1)—C(211)	1.87 (3)	As(2)—C(131)	1.92 (3)
Br(1)—Ni—Br(2)	123.1 (5)	C(211)—As(1)—C(231)	103 (1)
Br(1)—Ni—O(1)	105 (1)	C(221)—As(1)—C(231)	107 (1)
Br(1)—Ni—O(2)	109 (1)	O(2)—As(2)—C(111)	114 (1)
Br(2)—Ni—O(1)	105 (1)	O(2)—As(2)—C(121)	114 (1)
Br(2)—Ni—O(2)	108 (1)	O(2)—As(2)—C(131)	104 (1)
O(1)—Ni—O(2)	104 (1)	C(111)—As(2)—C(121)	107 (1)
O(1)—As(1)—C(211)	111 (1)	C(111)—As(2)—C(131)	110 (1)
O(1)—As(1)—C(221)	112 (1)	C(121)—As(2)—C(131)	108 (1)
O(1)—As(1)—C(231)	109 (1)	Ni—O(1)—As(1)	132 (1)
C(211)—As(1)—C(221)	114 (1)	Ni—O(2)—As(2)	130 (1)

zation were reported. At that time it was not possible to produce single crystals of the blue complex. The present crystals were of poor diffracting quality and did not allow measurements of data with a resolution good enough to perform anisotropic refinements for all the non-H atoms and for accurate calculation of distances and angles. Nevertheless, the main aim of the present study could still be achieved, namely the unambiguous determination of the (somewhat distorted) tetrahedral coordination around the Ni ion.

This work has received partial support from CAPES, CNPq, FAPESP and FINEP.

References

- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- OLIVA, G., CASTELLANO, E. E., ZUKERMAN-SCHPECTOR, J. & MASSABNI, A. C. (1984). *Inorg. Chim. Acta*, **89**, 9–17.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

807.0, monoclinic, $P2_1/c$, $a = 18.418 (4)$, $b = 10.697 (3)$, $c = 21.080 (7)$ \AA , $\beta = 102.32 (2)^{\circ}$, $V = 4057 \text{\AA}^3$, $Z = 4$, $D_x = 1.32 \text{ Mg m}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71073 \text{ \AA}$, $\mu = 0.35 \text{ mm}^{-1}$, $T = 293 \text{ K}$.

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