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Dibromobis(methyldiphenylphosphine)nickel(II)

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.022
wR factor = 0.057
Data-to-parameter ratio = 19.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The single-crystal structure of the title compound, $[NiBr_2(C_{13}H_{13}P)_2]$, was determined by X-ray diffraction at 100 K. The molecule has approximately tetrahedral geometry and exhibits twofold symmetry in the solid state.

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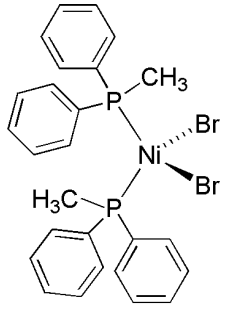
Comment

The title compound, (I), a versatile starting material for a range of nickel(II) complexes (Sturge *et al.*, 1992; McDonald *et al.*, 1992, 1993), crystallizes in the monoclinic space group $C2/c$ with four molecules in the unit cell.



(I)

The geometry of the compound is approximately tetrahedral and it exhibits crystallographic twofold symmetry. All the bond lengths and angles are in the range observed for similar compounds (Jarvis *et al.*, 1968; Kilbourn & Powell, 1970; Malm *et al.*, 1992). The Br—Ni—Br angle [128.52 (1)°] is the largest around the nickel center. The other angles are 100.42 (2)° for P—Ni—P, and 104.90 (1) and 107.39 (1)° for P—Ni—Br.

Experimental

The title nickel complex was prepared *via* the reaction of $NiBr_2$ with $PMePh_2$ in 1-butanol, as described in the literature by Hayter & Humiec (1965). Single crystals were grown from a solution in tetrahydrofuran.

Crystal data

$[NiBr_2(C_{13}H_{13}P)_2]$
M_r = 618.90
Monoclinic, $C2/c$
a = 14.7961 (10) Å
b = 12.4953 (8) Å
c = 14.5148 (10) Å
β = 110.0910 (10)°
V = 2520.2 (3) Å^3
Z = 4
D_x = 1.631 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5337 reflections
θ = 2.2–31.9°
μ = 4.08 mm^{-1}
T = 100 (2) K
Block, red
0.23 × 0.19 × 0.13 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
T_{min} = 0.450, T_{max} = 0.59
14798 measured reflections
3835 independent reflections
3529 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 30.5°
$h = -20 \rightarrow 20$
$k = -17 \rightarrow 17$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.057
S = 1.07
3835 reflections
193 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2 + 0.7837P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—Ni1	2.3425 (2)	P1—C1	1.8193 (12)
Ni1—P1	2.2791 (4)	P1—C13	1.8197 (14)
P1—C7	1.8114 (13)		
P1—Ni1—P1^{i}	100.415 (19)	C7—P1—C13	106.24 (6)
P1—Ni1—Br1^{i}	104.895 (9)	C1—P1—C13	103.49 (6)
P1^{i}—Ni1—Br1^{i}	107.391 (9)	C7—P1—Ni1	114.23 (4)
Br1^{i}—Ni1—Br1	128.515 (12)	C1—P1—Ni1	111.60 (4)
C7—P1—C1	105.96 (6)	C13—P1—Ni1	114.41 (5)

Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.

All H atoms were found in difference Fourier maps and were refined isotropically. The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000).

Data collection: *SMART* (Bruker, 1997–2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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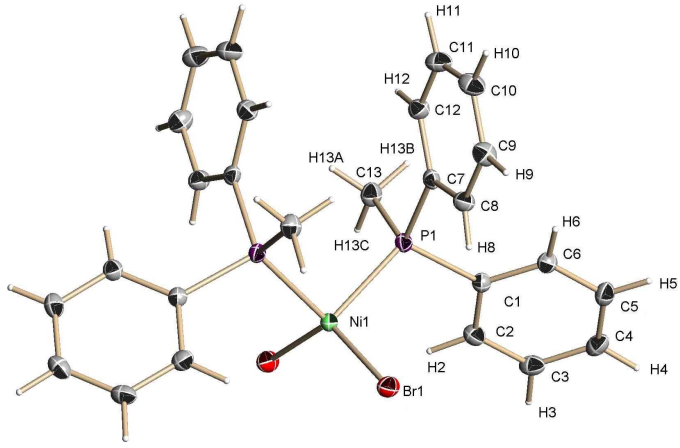

Figure 1
The structure of the title nickel complex, showing 50% probability displacement ellipsoids. Unlabeled atoms are related to the labeled atoms by $(1-x, y, \frac{1}{2}-z)$.

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