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[1,2-Bis(diphenylphosphino)ethane]dibromonickel(II) tetrahydrofuran solvate

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Key indicators

Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.007 Å
R factor = 0.052
wR factor = 0.138
Data-to-parameter ratio = 16.6

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

The title complex, [Ni(dppe)Br_2]·THF, where dppe is bis(diphenylphosphino)ethane ($C_{26}H_{24}P_2$) and THF is tetrahydrofuran (C_4H_8O), consists of a square-planar Ni center coordinated by the chelating phosphine ligand and by two *cis*–Br atoms. One molecule of THF is included in the asymmetric unit.

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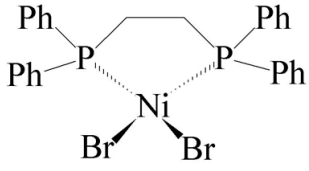
Comment

In the course of our studies, the formation of Ni(dppe)Br_2 was observed as a by-product for the reaction of Ni(dppe)$_2$ with *N*-bromophthalimide. Even though this compound is well known as a good starting product, only the CH_2Cl_2 adduct has been previously characterized using X-ray crystallography (Rahn *et al.*, 1989). The structure of the THF adduct, (I) (Fig. 1), is



(I)

isostructural with the one previously reported for the CH_2Cl_2 analog, having a slightly larger volume (about 20 Å^3), presumably because of the larger volume of THF compared to CH_2Cl_2. The THF adduct adopts a pseudo-square planar geometry with a P—Ni—P bite angle of 89.97 (5)° and an angle of 93.93 (4)° between the Br atoms. We note that the Ni—P bond lengths [2.1573 (14) and 2.1603 (14) Å] are also

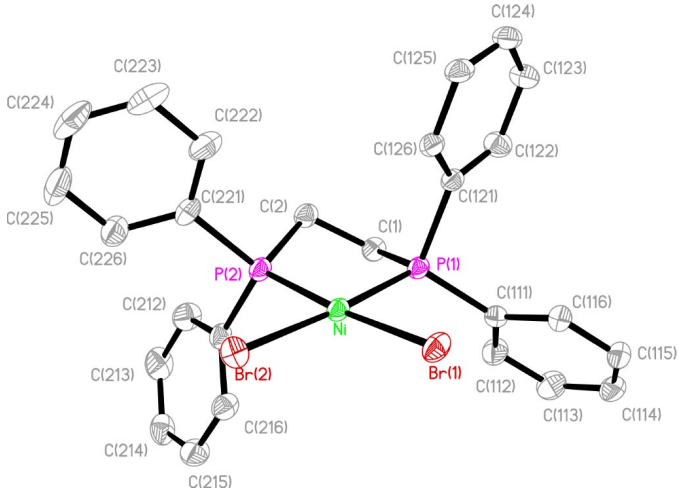


Figure 1
SHELXTL (Bruker, 1997) drawing of the title molecule, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

slightly longer in the THF adduct than the corresponding distances in the CH_2Cl_2 adduct [2.141 (1) and 2.156 (1) Å], but both Ni—Br bonds are very similar. In this structure, there is a stacking interaction between the THF ring and the C121—C126 phenyl ring, as shown in Fig. 2.

Experimental

Using Schlenk techniques, THF (10 ml) was added to a mixture of $Ni(dppe)_2$ (40 mg, 0.047 mmol) and *N*-bromophthalimide (13 mg, 0.058 mmol) and stirred for 5 min. A large excess of hexanes was then added to the solution, resulting, after 12 h, in the formation of dark-red crystals. The filtrate was removed and the solid dried under N_2.

Crystal data

$[NiBr_2(C_{26}H_{24}P_2)]\cdot C_4H_8O$
M_r = 689.03
Monoclinic, $P2_1/c$
a = 11.709 (5) Å
b = 14.551 (4) Å
c = 17.558 (6) Å
β = 107.38 (3)°
V = 2854.9 (17) Å^3
Z = 4
D_x = 1.603 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 20.0–21.0°
μ = 5.48 mm^{-1}
T = 220 (2) K
Block, dark red
0.57 × 0.15 × 0.12 mm

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: by integration (*ABSORP* in *NRCVAX*; Gabe *et al.*, 1989)
T_{min} = 0.234, T_{max} = 0.573
6651 measured reflections
5425 independent reflections
4582 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 70.0°
$h = -14 \rightarrow 14$
$k = -17 \rightarrow 17$
$l = -21 \rightarrow 21$
5 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.138
S = 1.08
5425 reflections
326 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1052P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.80 e Å^{-3}
$\Delta\rho_{min}$ = −1.12 e Å^{-3}
Extinction correction: *SHELXL*96 (Sheldrick, 1996)
Extinction coefficient: 0.00115 (13)

Table 1
Selected geometric parameters (Å, °).

Ni—P2	2.1573 (14)	Ni—Br1	2.3212 (12)
Ni—P1	2.1603 (14)	Ni—Br2	2.3419 (12)
P2—Ni—P1	86.97 (5)	P2—Ni—Br2	88.92 (5)
P2—Ni—Br1	175.13 (4)	P1—Ni—Br2	175.46 (4)
P1—Ni—Br1	90.02 (5)	Br1—Ni—Br2	93.95 (4)

The space group was confirmed by the *PLATON* program (Spek, 1995). Data reduction was performed using a locally modified version of the *NRC*-2 program (Ahmed *et al.*, 1973). The structure was solved by direct methods using *SHELXS*97 (Sheldrick, 1997) and difmap synthesis using *SHELXTL* (Bruker, 1997) and *SHELXL*96 (Sheldrick, 1996). H atoms were constrained to ride on the attached atoms; *SHELXL*96 defaults, C—H = 0.94–0.98 Å. The isotropic displacement parameters, U_{iso}, were set to values 20% higher than those of the attached atoms. A final verification of possible voids was performed using the *VOID* routine of the *PLATON* program (Spek, 1995).

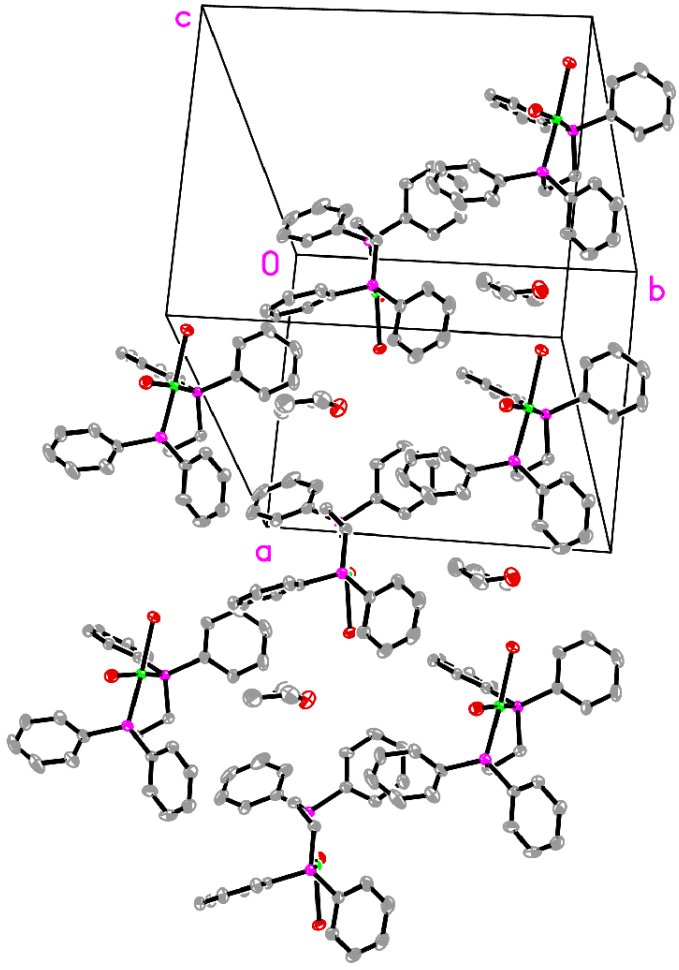


Figure 2
SHELXTL (Bruker, 1997) drawing of the unit-cell contents showing the interaction between THF and phenyl rings. Ellipsoids are drawn at the 30% probability level.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *NRC*-2 and *NRC*-2*A* (Ahmed *et al.*, 1973); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996); molecular graphics: *SHELXTL* (Bruker, 1997).

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