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Dicarbonylbis(triphenylphosphine)nickel(0): a redetermination at 90 K

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.001 Å
R factor = 0.024
wR factor = 0.075
Data-to-parameter ratio = 83.4

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

The structure of the title compound, $[Ni(C_{18}H_{15}P)_2(CO)_2]$, has been redetermined from high-resolution data collected at 90 K. The crystal structure reveals tetrahedral coordination geometry for nickel, with two triphenylphosphine and two carbonyl ligands. The Ni atom lies on a twofold rotation axis.

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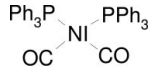
Comment

The crystal structure of the title compound, (I), has been published previously (Krüger & Tsay, 1974). In the present study, data were collected at low temperature, using a diffractometer equipped with an APEX2 CCD area detector.



(I)

As shown in Fig. 1, the structure of (I) is composed of neutral $[Ni(PPh_3)_2(CO)_2]$ complex molecules. The results show that the coordination around the Ni^0 atom, which lies on a twofold rotation axis, is distorted tetrahedral, composed of two triphenylphosphine P atoms [Ni—P1 = 2.2167 (1) Å] and two carbonyl C atoms [Ni—C19 = 1.7808 (4) Å]. The Ni^0 atom assumes a distorted tetrahedral configuration as in a series of other complexes: $[Ni(R_3P)_2(CO)_2]$, where R_3P is tricyclohexylphosphine (Del Pra *et al.*, 1981), diphenyl(2-pyridinyl)-phosphine (Wang *et al.*, 1989), tris(*o*-tolyloxy)phosphine

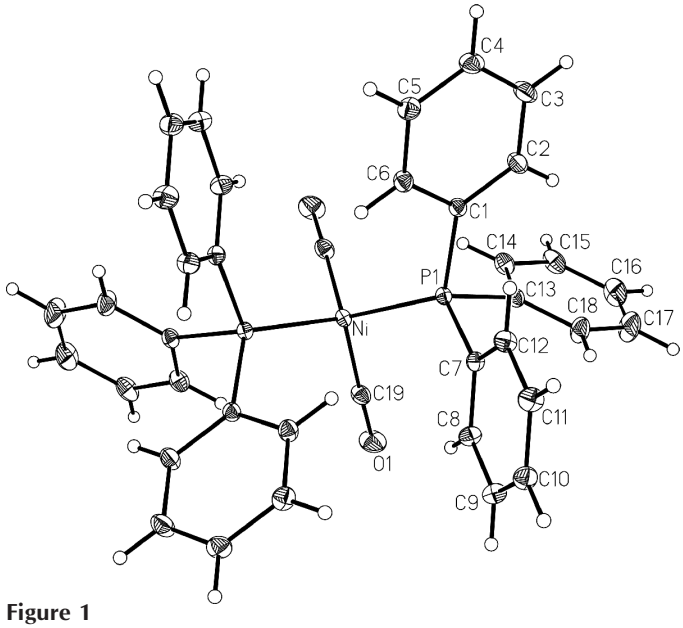


Figure 1
View of (I) (50% probability displacement ellipsoids).

(Meichel *et al.*, 2002), (4-hydroxybutyl)diphenylphosphine (Reinhard *et al.*, 2003) and 1,3,5-triaza-7-phosphaadamantane (Darensbourg *et al.*, 1999). The geometric parameters of the present structure agree well with those previously reported by Krüger & Tsay (1974), but with significantly improved precision. The precision of the Ni—P1 bond length (0.0001 Å), as well as the mean C—C bond length precision (0.0006 Å) for the low-temperature study, are better than the room-temperature data (0.001 and 0.004 Å, respectively). Similarly, the mean standard uncertainty for the C—C—C bond angles for the low-temperature data is 0.04°, whereas for the room-temperature data the mean standard deviation for the C—C—C bond angles is 0.2°.

Experimental

The title compound was obtained from a commercial source (Aldrich Ltd). Crystals suitable for structure determination were grown from a solution in 1,2-dimethoxyethane in a dry-box.

Crystal data

$[Ni(C_{18}H_{15}P)_2(CO)_2]$
$M_r = 639.25$
Monoclinic, $P2/c$
$a = 11.7267\ (4)$ Å
$b = 8.1512\ (2)$ Å
$c = 16.8433\ (5)$ Å
$\beta = 105.612\ (1)^\circ$
$V = 1550.60\ (8)$ Å^3
$Z = 2$
$D_x = 1.369$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7626 reflections
$\theta = 2.5$–49.3°
$\mu = 0.76$ mm^{-1}
$T = 90\ (1)$ K
Needle, colorless
0.20 × 0.05 × 0.04 mm

Data collection

Bruker SMART APEX2 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.863$, $T_{max} = 0.970$
99 172 measured reflections
16 260 independent reflections
13 619 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 50.0^\circ$
$h = -25 \rightarrow 24$
$k = -17 \rightarrow 17$
$l = -36 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.075$
$S = 1.04$
16 260 reflections
195 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 0.0217P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.76$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—C19	1.7808 (4)	O1—C19	1.1486 (5)
Ni—P1	2.2167 (1)		
C19^{i}—Ni—C19	113.47 (3)	C19—Ni—P1	103.849 (14)
C19—Ni—P1^{i}	109.58 (1)	P1^{i}—Ni—P1	116.842 (6)

Symmetry code: (i) $1-x, y, \frac{3}{2}-z$.

All H atoms were placed in calculated positions (C—H = 0.95 Å) and refined with a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *APEX*2 (Bruker, 2004); cell refinement: *APEX*2; data reduction: *SAINT-Plus* (Bruker, 2004); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *XL* in *SHELXTL* (Sheldrick, 2000); molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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