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trans-Bis(4,4,4-trifluoro-1-phenyl-1,3-butanedionato-*O,O′*)bis(triphenylphosphine-*P*)nickel(II) at 173 K

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Key indicators

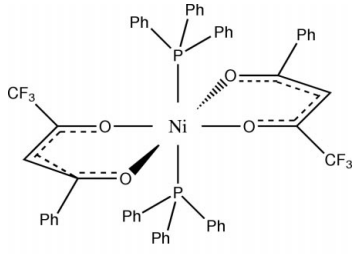
Single-crystal X-ray study
T = 171 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.109
Data-to-parameter ratio = 17.9

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

In the title compound, $[Ni(C_{15}H_6F_3O_2)_2(C_{18}H_{15}P)_2]$, the Ni atom occupies an inversion center and the complex has an all-*trans* configuration. The Ni—P distance is 2.5520 (5) Å.

Comment

In the title compound, (I), the Ni atom occupies an inversion center and the complex has an all-*trans* configuration. The Ni—P distance is 2.5520 (5) Å. The butanedionate ring is tilted, with the Ni atom 0.46 Å out of the plane of the ring. The 1-phenyl group is twisted 27.85 (9)° out of the same plane, and the *ortho*-phenyl H atom is 2.14 Å from the butanedionate H atom. The 1-phenyl group is also nearly parallel to one of the triphenylphosphine phenyl rings [dihedral angle = 8.30 (14)°] at a distance of about 3.7 Å.



(I)

Experimental

The title compound was synthesized by refluxing bis(4,4,4-trifluoro-1-phenyl-1,3-butanedionato-*O,O′*)nickel(II) (0.24 g) with two equivalents of triphenylphosphine (0.26 g) in 25 ml of $CHCl_3$. The resulting dark-green air-sensitive solution was allowed to cool in a tightly stoppered vial to produce crystals suitable for X-ray analysis. The dry solid was stable in air. The starting material bis(4,4,4-trifluoro-1-phenyl-1,3-butanedionato-*O,O′*)nickel(II) was synthesized in the following manner. To a solution of 4,4,4-trifluoro-1-phenyl-1,3-butanedione (Aldrich, 5.0 g) in about 30 ml ethanol, one equivalent of NaOH (0.9 g) dissolved in 10 ml water was added. To this mixture was slowly added, with stirring, a solution of $NiCl_2.6H_2O$ (2.7 g) dissolved in 30 ml water. The resulting precipitate was isolated by filtration and recrystallized from aqueous ethanol.

Crystal data

$[Ni(C_{15}H_6F_3O_2)_2(C_{18}H_{15}P)_2]$
M_r = 1013.55
Triclinic, $P\overline{1}$
a = 10.7917 (14) Å
b = 11.0583 (14) Å
c = 11.7156 (15) Å
α = 63.282 (2)°
β = 77.173 (2)°
γ = 71.153 (2)°
V = 1177.0 (3) Å^3
Z = 1
D_x = 1.430 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5700 reflections
θ = 2.2–28.3°
μ = 0.55 mm^{-1}
T = 171 (2) K
Irregular, green
0.40 × 0.26 × 0.12 mm

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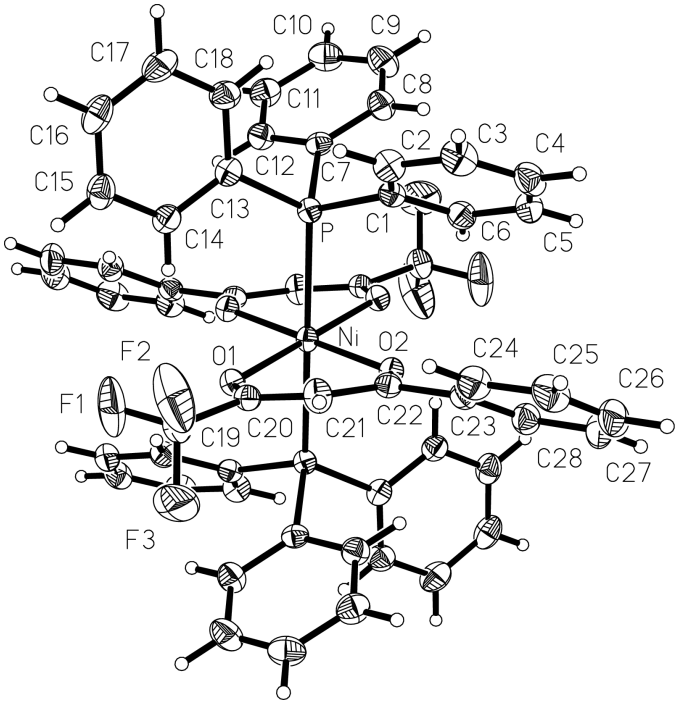

Figure 1
A drawing of the molecular structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Data collection

Bruker SMART CCD area-detector diffractometer	R_{int} = 0.027
ω scans	θ_{max} = 28.4°
13 372 measured reflections	$h = -14 \rightarrow 14$
5594 independent reflections	$k = -14 \rightarrow 14$
4643 reflections with $I > 2\sigma(I)$	$l = -15 \rightarrow 15$
	Intensity decay: <2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.9502P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.041	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.109	$\Delta\rho_{max}$ = 0.87 e Å^{-3}
S = 1.01	$\Delta\rho_{min}$ = −0.54 e Å^{-3}
5594 reflections	
313 parameters	
H-atom parameters constrained	

Table 1
Selected bond lengths (Å).

Ni—O1	2.0108 (13)	C19—F2	1.311 (3)
Ni—O2	2.0135 (13)	C19—F1	1.312 (3)
Ni—P	2.5520 (5)	C19—F3	1.319 (3)
P—C1	1.825 (2)	C19—C20	1.531 (3)
P—C13	1.827 (2)	C20—C21	1.381 (3)
P—C7	1.832 (2)	C21—C22	1.418 (3)
O1—C20	1.261 (2)	C22—C23	1.491 (3)
O2—C22	1.261 (2)		

An approximate sphere of reflections was collected to about 56° in 2θ (94.4% complete, average redundancy 2.4). Crystal stability was monitored by recollection of the first 50 frames after data collection was complete. No significant change in intensities was observed in 103 reflections.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1998). *SMART* (Version 6), *SAINT* (Version 6) and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.