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Bis(triphenylphosphine)iminium tricarbonyl-cyclopentadienylmolybdate

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Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.090
Data-to-parameter ratio = 17.8

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

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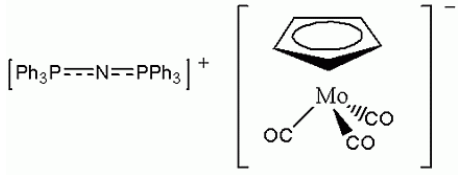
The crystal structure of bis(triphenylphosphine)iminium cyclopentadienyltricarbonylmolybdate, [PPN][Mo(CO)$_3$(η^5-C$_5$H$_5$)] (PPN$^+$ = [(PPh$_3$)$_2$N]$^+$, C$_{36}$H$_{30}$NP$_2$), is reported. The anion is configured in the 'piano-stool' arrangement while the cation adopts a bent configuration about the P—N—P bond. An 'expanded-phenyl-embrace' supramolecular motif is noted in the packing of the PPN$^+$ cation.

Comment

Though a commonly used metal carbonyl reagent, the structure of [PPN][Mo(CO)$_3$(η^5-C$_5$H$_5$)], (I), has not been previously reported.



(I)

The anion adopts the classic piano-stool-type arrangement common for Cp*ML*$_3$ systems. The average Mo—C$_{CO}$ bond length [1.930 (3) Å] is slightly larger than that reported for the tetrabutylammonium salt [1.909 (9) Å] while the average C$_{CO}$—Mo—C$_{CO}$ angle [87 (2)°] and Mo—C$_{Cp}$ distance [2.37 (2) Å] are similar to those reported [88.1(3)° and 2.37 (1) Å respectively; Crotty *et al.*, 1977]. The PPN$^+$ cation adopts the typical bent configuration about the P—N—P bond [142.62 (14)°].

A characteristic of triphenylphosphine-related systems is the observance of 'phenyl embraces' as a crystal packing motif (Scudder & Dance, 1998). These embraces involve intermolecular phenyl attractions (both edge–face and offset face–face attractions) forming extended networks through the crystal lattice. This type of packing motif is noted in the structure of [PPN][Mo(CO)$_3$(η^5-C$_5$H$_5$)]. The non-bonded P—P distance of 6.8606 (8) Å and N—P—P angle of 72.41 (7)° are characteristic of an 'expanded-phenyl-embrace' crystal packing motif in a compound containing PPN$^+$ (Lewis & Dance, 2000).

Experimental

[Mo(CO)$_3$(η^5-C$_5$H$_5$)]$_2$ (0.2 g, 0.41 mmol) was reduced in tetrahydrofuran (20 ml) over a 1% Na/Hg amalgam for 2–3 h. A dichloromethane solution (5 ml) of [PPN]Cl (0.47 g, 0.82 mmol) was added and the reaction mixture stirred for an additional 30 min. The pale

yellow–orange solution was transferred by syringe and solvent removed under vacuum. Recrystallization by addition of diethyl ether to a dichloromethane solution of the title compound produced a pale yellow powder. Crystals suitable for crystallographic analysis were obtained by liquid–liquid diffusion under a nitrogen atmosphere of diethyl ether and light petroleum spirits into a dichloromethane solution of the compound

Crystal data

$C_{36}H_{30}NP_2{\cdot}C_8H_5MoO_3$	D_x = 1.401 Mg m^{-3}
M_r = 783.61	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 7126 reflections
a = 14.0385 (2) Å	θ = 1.5–27.5°
b = 19.1269 (2) Å	μ = 0.48 mm^{-1}
c = 13.8322 (1) Å	T = 203 (2) K
β = 90.728 (1)°	Prism, yellow
V = 3713.83 (7) Å^3	0.26 × 0.24 × 0.20 mm
Z = 4	

Data collection

Siemens SMART CCD diffractometer	8177 independent reflections
	6724 reflections with $I > 2\sigma(I)$
Multiscan scans	R_{int} = 0.025
Absorption correction: empirical (Blessing, 1995)	θ_{max} = 27.5°
	$h = -18 \rightarrow 18$
T_{min} = 0.885, T_{max} = 0.910	$k = 0 \rightarrow 24$
22556 measured reflections	$l = 0 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0305P)^2 + 3.4046P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.037	
$wR(F^2)$ = 0.090	
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
8177 reflections	$\Delta\rho_{max}$ = 0.52 e Å^{-3}
460 parameters	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
H-atom parameters constrained	

H atoms were placed in calculated positions, with U_{iso} values 1.2 times the U_{iso} values of the atoms to which they are attached.

Data collection: *SMART* (Siemens, 1994); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SADABS* (Blessing, 1995); program(s) used to solve structure: *SHELXS*-97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

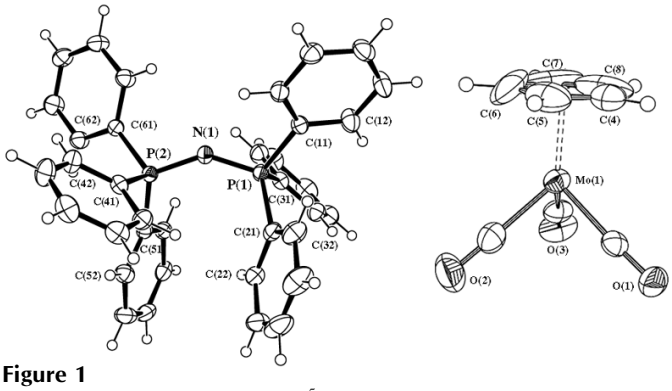


Figure 1
Structure of [PPN][Mo(CO)$_3$(η^5–C$_5$H$_5$)] showing the atom-labelling scheme. Ellipsoids are drawn at the 50% probability level (Farrugia, 1997)

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