metal-organic papers

Decacarbonyl-1κ^3C,2κ^3C,3κ^4C-[μ-1-diphenylarsino-2-(diphenylphosphino)ethane-1:2κ^2As:P]-*triangulo*-triruthenium(0)

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean σ(C–C) = 0.008 Å

Disorder in main residue

R factor = 0.038

wR factor = 0.075

Data-to-parameter ratio = 18.0

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

The title compound, [Ru$_3$(C$_{26}$H$_{24}$AsP)(CO)$_{10}$], contains a triangle of bonded Ru atoms. The Ph$_2$P(CH$_2$)$_2$AsPh$_2$ ligand bridges an Ru—Ru bond, occupying equatorial positions on adjacent Ru atoms. There is disorder of the As and P atoms.

Comment

There have been extensive synthetic studies on mixed-ligand triruthenium cluster carbonyls (Bruce *et al.*, 1983, 1989). In particular, mixed-ligand cluster carbonyls with group 15 donor atom ligands are of special interest because of their catalytic activity. However, structure determinations of triruthenium clusters containing mixed group 15 ligands are rare. As part of our study of the substitution of transition metal carbonyl clusters with mixed ligand complexes, we have published several structures of triruthenium carbonyl clusters containing mixed P and As (Shawkataly *et al.*, 1998) and P and Sb ligands (Shawkataly *et al.*, 2004).

(I)
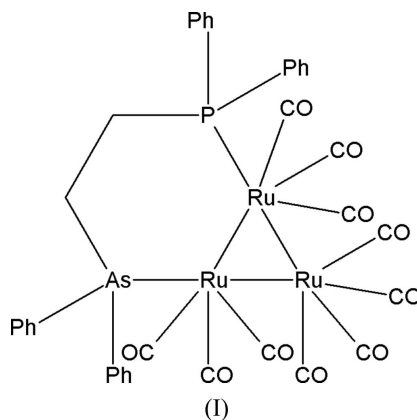
The structure of the title compound is similar to that of Ru$_3$(CO)$_{10}$[Ph$_2$P(CH$_2$)$_2$PPh$_2$] (Bruce *et al.*, 1982). The crystal structure of the discrete cluster (Fig. 1) reveals the presence of the *triangulo* Ru$_3$ framework found in similar species. The Ph$_2$P(CH$_2$)$_2$AsPh$_2$ ligand bridges an Ru—Ru bond, occupying equatorial sites on adjacent Ru atoms. Positional disorder of the As and P atoms corresponds to end-to-end disorder of the chelating ligand. Similar disorder involving P and As atoms is also present in the crystal structures of Ph$_2$P(CH$_2$)$_2$AsPh$_2$ (Shawkataly *et al.*, 2005) and Ru$_3$(CO)$_8$[Ph$_2$As(CH$_2$)$_2$AsPh$_2$][Ph$_2$P(CH$_2$)$_2$PPh$_2$] (Shawkataly *et al.*, 1998). The P—C, As—C, Ru—C, C—O and C—C bond lengths are typical of this type of organometallic compound (Cambridge Structural Database; November 2005 update; Allen, 2002).

Experimental

The title compound was synthesized from a mixture of $Ru_3(CO)_{12}$ and $Ph_2P(CH_2)_2AsPh_2$ (1:1 molar ratio) in dry tetrahydrofuran under N_2 with a catalytic amount of diphenylketyl radical (Bruce *et al.*, 1983). The product was purified by thin layer chromatography. Single crystals of the title compound were obtained by solvent diffusion from dichloromethane and methanol.

Crystal data

$[Ru_3(C_{26}H_{24}AsP)(CO)_{10}]$	$D_x = 1.832$ Mg m^{-3}
$M_r = 1025.65$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 49
$a = 12.7117$ (11) Å	reflections
$b = 14.3888$ (11) Å	$\theta = 6.7$–24.9°
$c = 20.4384$ (11) Å	$\mu = 2.19$ mm^{-1}
$\beta = 96.026$ (5)°	$T = 293$ (2) K
$V = 3717.7$ (5) Å^3	Prism, dark red
$Z = 4$	$0.44 \times 0.14 \times 0.1$ mm

Data collection

Siemens $P4$ diffractometer	$R_{int} = 0.042$
$\omega/2\theta$ scans	$\theta_{max} = 27.5°$
Absorption correction: ψ scans	$h = -1 \rightarrow 16$
($XSCANS$; Siemens, 1994)	$k = -1 \rightarrow 18$
$T_{min} = 0.701$, $T_{max} = 0.807$	$l = -26 \rightarrow 26$
10420 measured reflections	3 standard reflections
8514 independent reflections	frequency: 60 min
4541 reflections with $I > 2\sigma(I)$	intensity decay: 0.3%

Refinement

Refinement on F^2	H atoms treated by a mixture of
$R[F^2 > 2\sigma(F^2)] = 0.039$	independent and constrained
$wR(F^2) = 0.075$	refinement
$S = 0.80$	$w = 1/[\sigma^2(F_o^2) + (0.0098P)^2]$
8514 reflections	where $P = (F_o^2 + 2F_c^2)/3$
472 parameters	$(\Delta/\sigma)_{max} = 0.002$
	$\Delta\rho_{max} = 0.40$ e Å^{-3}
	$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ru3—Ru1	2.8331 (6)	As1/P1—C11	1.893 (5)
Ru3—Ru2	2.8512 (6)	As1/P1—C23	1.894 (5)
Ru1—Ru2	2.8532 (6)	P2/As2—C31	1.891 (5)
Ru1—As1/P1	2.3748 (8)	P2/As2—C25	1.892 (5)
Ru2—P2/As2	2.3796 (8)	P2/As2—C24	1.919 (5)
As1/P1—C17	1.865 (5)	C23—C24	1.536 (7)
As1/P1—Ru1—Ru2	102.06 (2)	C23—As1/P1—Ru1	118.17 (16)
P2/As2—Ru2—Ru1	101.31 (2)	C24—P2/As2—Ru2	117.59 (15)

The site-occupancy factors of As and P atoms were fixed at 0.5; the same atomic coordinates and displacement parameters were assumed. The methylene H atoms were located in a difference Fourier map and their positions were refined [C—H = 0.89 (5) – 0.99 (5) Å]. The phenyl H atoms were placed at calculated positions and refined using a riding model, with C—H distances of 0.93 Å. All H-atom U_{iso} parameters were fixed at $1.2U_{eq}(C)$.
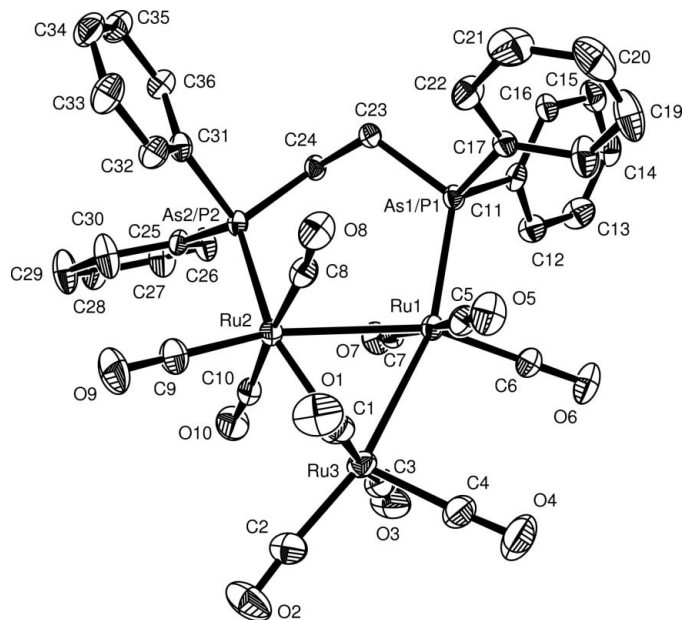
Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 25% probability level. H atoms have been omitted.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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