

## Tris(tricarbonyl(triphenylphosphine)ruthenium

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## Key indicators

Single-crystal X-ray study

$T = 173$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å

$R$  factor = 0.042

$wR$  factor = 0.106

Data-to-parameter ratio = 18.1

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

The title compound,  $[\text{Ru}(\text{CO})_3(\text{PPh}_3)]_3$ , has three ruthenium centres, each in an octahedral environment with distortion from ideal geometry. The compound contains the characteristic triangular  $\text{Ru}_3$  core, with the phosphorus ligands each occupying an equatorial position at one end of an  $\text{Ru}-\text{Ru}$  vector.

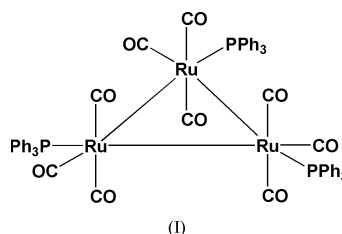
Received 13 March 2002

Accepted 18 March 2002

Online 28 March 2002

## Comment

The substitution reactions and crystal structures of mono- and disubstituted dodecacarbonyltriruthenium with phosphorus ligands have been reported (Bruce, Liddell, Hughes, Skelton & White, 1988; Bruce, Liddell, Hughes, Patrick *et al.*, 1988); the title compound, the third member of this series, is the trisubstituted complex, (I).



The molecular structure of (I) is shown in Fig. 1. The three triphenylphosphine ligands take up positions that are as far apart as possible, each occupying an equatorial site. As found in the previously reported structures, the  $\text{Ru}-\text{CO}_{\text{ax}}$  distances are longer than the  $\text{Ru}-\text{CO}_{\text{eq}}$ , average values being 1.933 and 1.879 Å, respectively. The  $\text{Ru}-\text{P}$  distances are typical of these organometallic compounds, with an average bond length of 2.340 Å, and the metal-metal bond distances are similarly unremarkable (average length 2.873 Å).

## Experimental

Crystals of the title compound were obtained, *via* a serendipitous route, upon reaction of  $\text{Ru}_3(\text{CO})_{12}$  with triphenylphosphine and 2,2'-biphenol, refluxing in toluene for 1 h. The resulting deep-red reaction mixture was purified by column chromatography (alumina,  $\text{CH}_2\text{Cl}_2$ /hexane). The title compound was isolated as the major fraction and was identified by FAB mass spectrometry [ $\text{MS}^+$  1342], and recrystallized from hot  $\text{CHCl}_3$ /hexane.

## Crystal data

$[\text{Ru}(\text{C}_{18}\text{H}_{15}\text{P})(\text{CO})_3]_3$

$M_r = 1342.11$

Monoclinic,  $P2_1/n$

$a = 14.174$  (2) Å

$b = 21.640$  (4) Å

$c = 18.239$  (3) Å

$\beta = 92.533$  (17)°

$V = 5589.0$  (17) Å<sup>3</sup>

$Z = 4$

$D_x = 1.595$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

Cell parameters from 1013

reflections

$\theta = 2.9-26.7^\circ$

$\mu = 0.94$  mm<sup>-1</sup>

$T = 173$  (2) K

Needle, dark red

$0.4 \times 0.1 \times 0.1$  mm

## Data collection

Bruker CCD area-detector  
 diffractometer  
 $\omega$  rotation with narrow frames  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.808$ ,  $T_{\max} = 0.909$   
 35892 measured reflections

12757 independent reflections  
 8075 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.072$   
 $\theta_{\text{max}} = 27.5^\circ$   
 $h = -18 \rightarrow 18$   
 $k = -27 \rightarrow 28$   
 $l = -16 \rightarrow 23$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.106$   
 $S = 0.96$   
 12757 reflections  
 703 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0463P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.55 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.57 \text{ e } \text{\AA}^{-3}$

Table 1

 Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Ru1—C10	1.880 (5)	Ru2—C22	1.943 (5)
Ru1—C11	1.921 (5)	Ru2—P2	2.3304 (13)
Ru1—C12	1.959 (5)	Ru2—Ru3	2.8675 (7)
Ru1—P1	2.3387 (13)	Ru3—C30	1.879 (5)
Ru1—Ru2	2.8623 (6)	Ru3—C31	1.929 (5)
Ru1—Ru3	2.8886 (7)	Ru3—C32	1.939 (5)
Ru2—C20	1.877 (5)	Ru3—P3	2.3511 (13)
Ru2—C21	1.912 (5)		
C10—Ru1—C11	93.6 (2)	C21—Ru2—C22	166.22 (19)
C10—Ru1—C12	98.00 (19)	Ru1—Ru2—Ru3	60.549 (16)
C11—Ru1—C12	168.0 (2)	C30—Ru3—C31	88.5 (2)
Ru2—Ru1—Ru3	59.815 (16)	C30—Ru3—C32	99.0 (2)
C20—Ru2—C21	93.0 (2)	C31—Ru3—C32	171.1 (2)
C20—Ru2—C22	100.7 (2)	Ru2—Ru3—Ru1	59.636 (17)

All H atoms were constrained to ideal geometries and assigned isotropic displacement parameters 1.2 times that of their adjacent C atom.

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

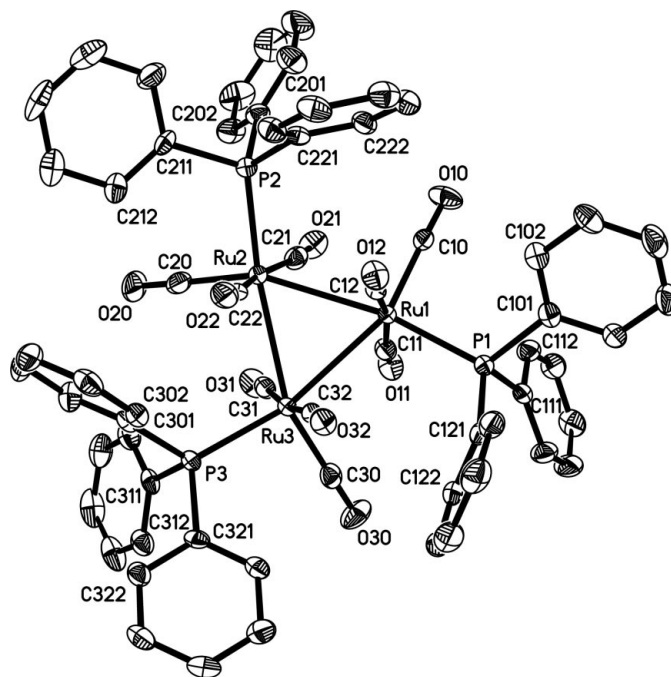


Figure 1

A view of the molecular structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

We thank the University of Bristol for financial support (RHL). We thank the EPSRC National Mass Spectrometry Service Centre, Swansea, for the mass spectrum.

## References

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