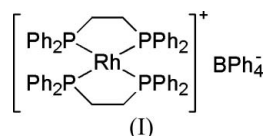


Bis[bis(diphenylphosphino)ethane]rhodium(I)  
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## Key indicators

Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.031  
 $wR$  factor = 0.074  
Data-to-parameter ratio = 19.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.In the title compound,  $[\text{Rh}(\text{C}_{26}\text{H}_{24})_2](\text{C}_{24}\text{H}_{20}\text{B})$ , both the Rh and the B atom lie on twofold axes of symmetry. Selected geometrical parameters are  $\text{Rh}-\text{P} = 2.3076(5)$  and  $2.3114(5)$  Å, and  $\text{P}-\text{Rh}-\text{P} = 82.43(2)$  and  $98.11(2)^\circ$ .

## Comment

The lipophilic bis-chelated 1:2  $M^I$  diphosphine complexes  $[M(\text{dppe})_2]X$  [where  $M = \text{Au}, \text{Ag}$  and  $\text{Cu}$ ,  $\text{dppe} = \text{Ph}_2\text{P}(\text{CH}_2)_2\text{PPh}_2$  and  $X$  is a non-coordinating anion] exhibit antitumour activity against a range of tumour models [see Berners-Price *et al.* (1990); Berners-Price, Bowen, Galettis *et al.* (1999); Berners-Price, Bowen, Hambley & Healy (1999) for examples]. Since the dppe ligand is itself cytotoxic (Mirabelli *et al.*, 1987), albeit less potent than  $[M(\text{dppe})_2]X$ , it has been suggested that the phosphine is the antitumour agent and the role of the metal may largely be to protect the phosphine and deliver it to target sites. Conversely, the  $\text{Rh}^I$  complex  $[\text{RhCl}(\text{PPh}_3)_2\text{dppe}]$  and the complexes  $[M'\text{Cl}_2(\text{dppe})]$ , where  $M'$  is  $\text{Pt}^{II}$ ,  $\text{Pd}^{II}$  and  $\text{Ni}^{II}$ , are all inactive. In this work, we sought to test the antitumor activity and various propositions for explaining this activity by examination of the  $[M(\text{dppe})_2]X$  class of compounds. We report here the preparation and structure of  $[\text{Rh}(\text{dppe})_2][\text{BPh}_4]$ , (I). Though initial tests on compound (I) seem to indicate that the complex is inactive, we will communicate the biological data elsewhere.The structure of (I) does not show any unexpected features. The coordination around the Rh atom is approximately square planar, the distortion being due to the twofold crystallographic symmetry. This distortion can be seen from the dihedral angle of  $12.67(2)^\circ$  between the planes defined by  $\text{P1}/\text{Rh}/\text{P2}$  and  $\text{P1}'/\text{Rh}/\text{P2}'$  [symmetry code: (i)  $1 - x, y, \frac{1}{2} - z$ ]. In each phosphine the two aliphatic C atoms bridging the two phosphorus atoms are not equally positioned on either side of the  $\text{P}/\text{Rh}/\text{P}$  plane. The dominant feature of the five-membered chelate ring is the staggered conformation about the  $\text{C}-\text{C}$  bond. The bite angle of the dppe ligands in the title compound is  $82.43(2)^\circ$ . This value compares well with other cationic rhodium-dppe complexes reported in literature.

## Experimental

 $[\text{Rh}(\text{NBD})\text{Cl}]_2$  (NBD = norbornadiene) was kindly provided by Dr A. Muller (University of the Free State). All other chemicals were

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used as received. A solution of 1,2-bis(diphenylphosphino)ethane (0.50 g, 1.25 mmol) in dichloromethane (5 ml) was slowly added to a solution of [Rh(NBD)Cl]<sub>2</sub> (0.145 g, 0.31 mmol) in dichloromethane (5 ml). After stirring for 1 h a solution of NaBPh<sub>4</sub> (0.22 g, 0.64 mmol) in ethanol (5 ml) was added. The resulting mixture was stirred for 4 h and filtered. The volatiles were removed *in vacuo*. The residue was taken up in acetone, filtered, and the solvent was removed *in vacuo* to give the title compound as a yellow powder. Recrystallization from dichloromethane afforded crystals suitable for X-ray diffraction. All analytical data were in agreement with the literature (Faraone, 1975).

Crystal data

[Rh(C<sub>26</sub>H<sub>24</sub>P<sub>2</sub>)<sub>2</sub>](C<sub>24</sub>H<sub>20</sub>B)  
*M<sub>r</sub>* = 1218.9  
 Monoclinic, C2/c  
*a* = 17.010 (2) Å  
*b* = 16.560 (2) Å  
*c* = 22.142 (3) Å  
 $\beta$  = 102.642 (2)°  
*V* = 6086.0 (13) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.33 Mg m<sup>-3</sup>  
 Mo K $\alpha$  radiation  
 Cell parameters from 977 reflections  
 $\theta$  = 2.6–28.2°  
 $\mu$  = 0.43 mm<sup>-1</sup>  
*T* = 173 (2) K  
 Cuboid, yellow  
 0.2 × 0.2 × 0.19 mm

Data collection

Bruker SMART CCD 1K diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 1998)  
*T<sub>min</sub>* = 0.919, *T<sub>max</sub>* = 0.923  
 20334 measured reflections

7320 independent reflections  
 5664 reflections with *I* > 2 $\sigma$ (*I*)  
*R<sub>int</sub>* = 0.037  
 $\theta_{max}$  = 28°  
*h* = -19 → 22  
*k* = -19 → 21  
*l* = -29 → 27

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.031  
*wR*(*F*<sup>2</sup>) = 0.074  
*S* = 1.04  
 7320 reflections  
 371 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0346P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.39 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.37 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

|                       |             |  |             |
|-----------------------|-------------|--|-------------|
| Rh–P2                 | 2.3076 (5)  | P2–C2                                  | 1.8410 (18) |
| Rh–P1                 | 2.3114 (5)  | B–C61                                  | 1.644 (3)   |
| P1–C11                | 1.8326 (19) | B–C51                                  | 1.648 (3)   |
| P1–C21                | 1.8358 (18) | C1–C2                                  | 1.527 (2)   |
| P1–C1                 | 1.8484 (18) |  |             |
|                       |             |  |             |
| P2–Rh–P2 <sup>i</sup> | 169.75 (3)  | P1 <sup>i</sup> –Rh–P1                 | 173.98 (3)  |
| P2–Rh–P1 <sup>i</sup> | 98.110 (18) | C61 <sup>ii</sup> –B–C61               | 115.1 (2)   |
| P2–Rh–P1              | 82.432 (18) | C61 <sup>ii</sup> –B–C51 <sup>ii</sup> | 103.31 (9)  |
|                       |             |  |             |
| P1–C1–C2–P2           | -49.41 (15) |  |             |

Symmetry codes: (i) -*x* + 1, *y*, -*z* + ½; (ii) -*x* + 2, *y*, -*z* + ½.

The aromatic and methylene H atoms were placed in geometrically idealized positions (C–H = 0.95 and 0.99 Å, respectively) and constrained to ride on their parent atoms with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C).

Data collection: SMART-NT (Bruker, 1998); cell refinement: SAINT-Plus (Bruker, 1999); data reduction: SAINT-Plus and XPREP (Bruker, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: DIAMOND (Brandenburg & Berndt, 2001); software used to prepare material for publication: WinGX (Farrugia, 1999).

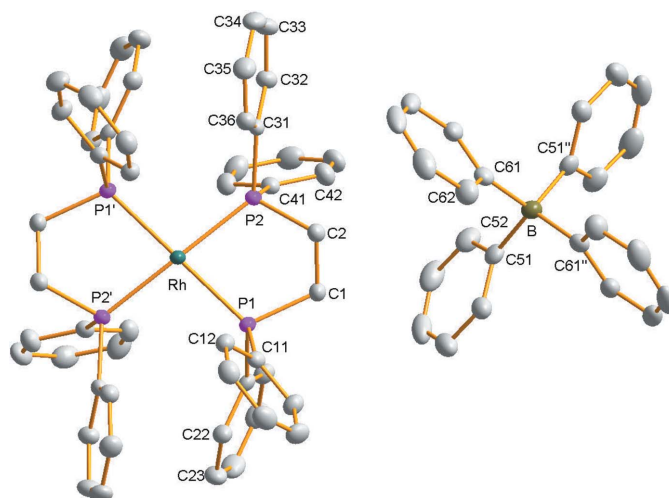


Figure 1

The structure of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity. For the aromatic C atoms, the first digit indicates ring number and the second digit indicates the position of the atom in the ring. [Symmetry codes: (i) 1 - *x*, *y*, ½ - *z*; (ii) 2 - *x*, *y*, ½ - *z*.]

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