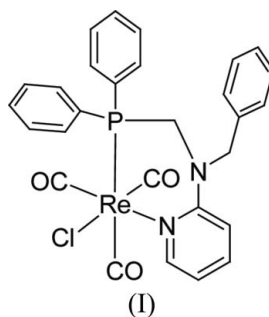


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nkpengli@mail.nankai.edu.cn**Key indicators**Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å  
 $R$  factor = 0.035  
 $wR$  factor = 0.069  
Data-to-parameter ratio = 16.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**{2-[*N*-Benzyl-*N*-(diphenylphosphinomethyl)-amino]pyridine}tricarbonylchlororhenium(I)**

In the mononuclear title compound,  $[\text{ReCl}(\text{C}_{25}\text{H}_{23}\text{N}_2\text{P})(\text{CO})_3]$ , the  $\text{Re}^{\text{I}}$  atom is six-coordinated by N and P atoms of a large-bite *P,N*-bidentate ligand, three carbonyl ligands and one Cl atom in a slightly distorted octahedral geometry. The *P,N*-bidentate ligand and the  $\text{Re}^{\text{I}}$  atom form a six-membered ring.

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Over the past few decades, tertiary phosphines have attracted considerable interest, especially as important ligands in coordination chemistry (Zhang & Cheng, 1996). To obtain cluster compounds, rhenium carbonyl complexes are used as starting materials along with hydrogen and hydrides (Saillant *et al.*, 1970; Bau *et al.*, 1967). Previously, we have reported the crystal structures of some heterobimetallic  $\text{Pt}^{\text{II}}-\text{M}^{\text{I}}$  ( $M = \text{Cu}, \text{Ag}$ ) macrocyclic complexes with a large-bite *P,N*-bidentate ligand, including 2-[*N*-benzyl-*N*-(diphenylphosphinomethyl)amino]pyridine (Li *et al.*, 2003). Now we report here the structure of the title compound, (I), a new  $\text{Re}^{\text{I}}$  complex with the above-cited ligand.



As shown in Fig. 1, the  $\text{Re}^{\text{I}}$  atom is six-coordinated by N and P atoms of the large-bite *P,N*-bidentate ligand, three carbonyl ligands and one Cl atom in a slightly distorted octahedral geometry. The P and N atoms and two carbonyl ligands are in the equatorial plane, and the P and N atoms are in *cis* positions. The third carbonyl ligand and the Cl atom are in *trans* positions with respect to the equatorial plane. The three *trans* angles at the  $\text{Re}^{\text{I}}$  centre are close to  $180^\circ$ . All other angles subtended at the  $\text{Re}^{\text{I}}$  atom are close to  $90^\circ$ , ranging from  $84.82(11)$  to  $95.20(17)^\circ$  (Table 1), indicating a slightly distorted octahedral geometry for atom Re1. The  $\text{Re}-\text{C}$  bond lengths lie in the range  $1.887(6)$ – $1.950(6)$  Å. A six-membered ring is formed by the *P,N*-bidentate ligand and the  $\text{Re}^{\text{I}}$  atom; the ring is in a half-chair form. There are no hydrogen bonds in the crystal structure.

## Experimental

The 2-[*N*-benzyl-*N*-(diphenylphosphinomethyl)amino]pyridine ligand was prepared according to the literature method of Li *et al.* (2003). Complex (I) was obtained from the treatment of the above ligand (0.764 g, 20 mmol) with  $\text{Re}(\text{CO})_5\text{Cl}$  (0.362 g, 10 mmol) in tetrahydrofuran (yield 0.867 g, 63%; m.p. 401–403 K). Single crystals of (I) were obtained by slow diffusion of diethyl ether into a dichloromethane solution.

### Crystal data

$[\text{ReCl}(\text{C}_{25}\text{H}_{23}\text{N}_2\text{P})(\text{CO})_3]$   
 $M_r = 688.10$   
 Monoclinic,  $P2_1/n$   
 $a = 10.671$  (5) Å  
 $b = 20.789$  (10) Å  
 $c = 12.097$  (6) Å  
 $\beta = 100.204$  (7)°  
 $V = 2641$  (2) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.731$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 4.80$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Block, colourless  
 $0.32 \times 0.28 \times 0.24$  mm

### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.210$ ,  $T_{\max} = 0.316$

12176 measured reflections  
 5310 independent reflections  
 3899 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.038$   
 $\theta_{\text{max}} = 26.4^\circ$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.069$   
 $S = 1.00$   
 5310 reflections  
 325 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0261P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.82$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.85$  e Å<sup>-3</sup>

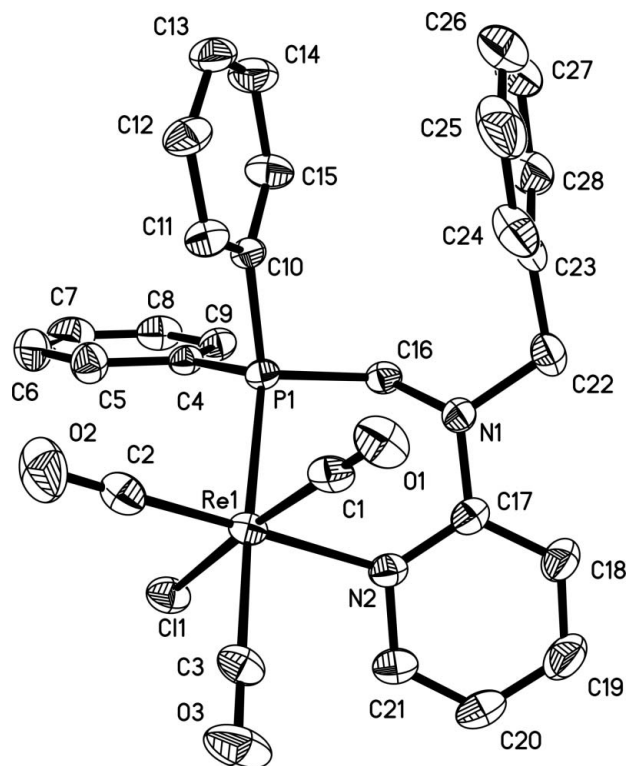
**Table 1**

Selected geometric parameters (Å, °).

Re1–C2	1.887 (6)	Re1–N2	2.253 (4)
Re1–C1	1.931 (6)	Re1–P1	2.4321 (15)
Re1–C3	1.950 (6)	Re1–Cl1	2.4687 (16)
C2–Re1–C1	90.9 (2)	C3–Re1–P1	176.77 (16)
C2–Re1–C3	87.9 (2)	N2–Re1–P1	85.60 (10)
C1–Re1–C3	89.9 (2)	C2–Re1–Cl1	95.09 (17)
C2–Re1–N2	179.2 (2)	C1–Re1–Cl1	173.97 (15)
C1–Re1–N2	89.15 (18)	C3–Re1–Cl1	90.48 (16)
C3–Re1–N2	91.33 (19)	N2–Re1–Cl1	84.82 (11)
C2–Re1–P1	95.20 (17)	P1–Re1–Cl1	88.24 (6)
C1–Re1–P1	91.02 (14)		

H atoms were positioned geometrically, with C–H = 0.93 or 0.97 Å, and were constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve



**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1999); software used to prepare material for publication: SHELXTL.

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