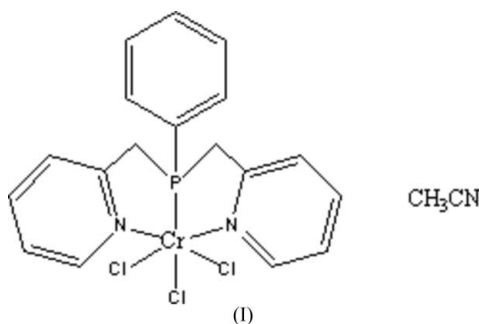


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## Key indicators

Single-crystal X-ray study  
 $T = 110$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.039  
 $wR$  factor = 0.086  
Data-to-parameter ratio = 21.6For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.[Bis(2-methyl-1-pyridyl)phenylphosphine- $\kappa^2N,P,N'$ ]-  
trichlorochromium(III) acetonitrile solvateIn the title molecular structure,  $[\text{CrCl}_3\{\text{PPh}(2\text{-CH}_2\text{C}_5\text{H}_4\text{N})_2\}]\text{-CH}_3\text{CN}$ , the  $\text{Cr}^{\text{III}}$  atom exhibits an octahedral coordination geometry, with a tridentate ligand capping one face of the octahedron and three  $\text{Cl}^-$  ions occupying the opposite face.Received 17 August 2006  
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## Comment

The synthesis of tricarbonyl compounds of tungsten, molybdenum, and chromium, displaying an octahedral geometry with a tridentate ligand has been reported (Lindner *et al.*, 1983). Bis(2-methyl-1-pyridyl)phenylphosphine, which has been demonstrated to be a flexible ligand, possesses a natural bite angle that is ideally suited for capping the face of an octahedron. Taking also into account the intrinsic electronic properties of phosphine ligands, bis(2-methyl-1-pyridyl)phenylphosphine has been found to coordinate either as a bidentate or tridentate ligand. This ligand coordination flexibility can potentially allow the synthesis of di- and polynuclear complexes that are of great interest in many fields, such as catalysis, bioinorganic chemistry and materials sciences (Gavrilova & Bosnich, 2004). In this study, we present one of the coordination compounds involving this tridentate ligand with chromium chloride in an octahedral geometry.The  $\text{Cr}^{\text{III}}$  atom in (I) is six-coordinated by three  $\text{Cl}^-$  ions, two N atoms and one P atom (Fig. 1). The *cis* angles about the Cr atom reveal a distorted octahedral coordination environment (Table 1). The *cis* P–Cr–N and N–Cr–N angles are more acute than those involving the Cl atoms, which is evidence of the constraining effect of the ligand coordination to the metal center. The Cl1–Cr1–P1 angle is nearly linear and the Cr–N, Cr–Cl and Cr–P distances cover a relatively small range.

## Experimental

Compound (I) was obtained by mixing  $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$  (0.06 g, 0.4 mmol) and  $\text{PPh}(2\text{-CH}_2\text{C}_5\text{H}_4\text{N})_2$  (0.117 g, 0.4 mmol) in 20 ml

acetonitrile. The mixture was stirred for 24 h at room temperature. The green precipitate obtained from the reaction, partially soluble in acetonitrile, was separated from the solvent and washed three times with dichloromethane for purification purposes. Diffraction-quality crystals were obtained by slow diffusion of diethyl ether into a concentrated acetonitrile solution of (I) in the presence of air.

### Crystal data

[CrCl<sub>3</sub>(C<sub>18</sub>H<sub>17</sub>N<sub>2</sub>P)]·C<sub>2</sub>H<sub>3</sub>N  
*M<sub>r</sub>* = 491.71  
 Monoclinic, *P*2<sub>1</sub>/*n*  
*a* = 11.0206 (12) Å  
*b* = 16.1145 (16) Å  
*c* = 13.0390 (14) Å  
 $\beta$  = 107.081 (4)°  
*V* = 2213.5 (4) Å<sup>3</sup>

*Z* = 4  
*D<sub>x</sub>* = 1.476 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 $\mu$  = 0.96 mm<sup>-1</sup>  
*T* = 110 (2) K  
 Block, green  
 0.27 × 0.19 × 0.16 mm

### Data collection

Bruker APEX X8 diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.697, *T<sub>max</sub>* = 0.859

21619 measured reflections  
 5487 independent reflections  
 4230 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.044  
 $\theta_{\max}$  = 28.3°

### Refinement

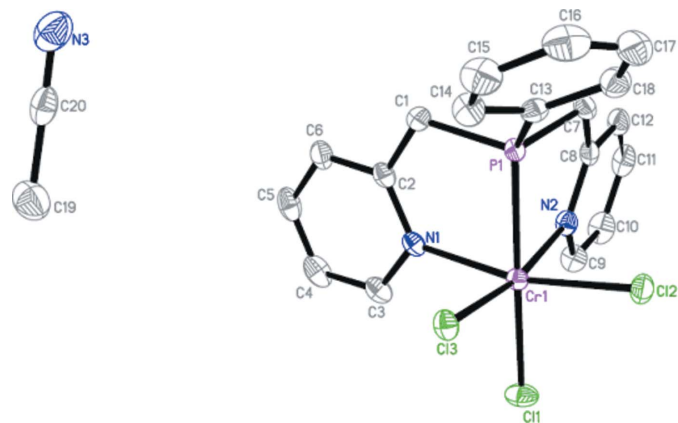
Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.039  
*wR* (*F*<sup>2</sup>) = 0.086  
*S* = 1.03  
 5487 reflections  
 254 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.906P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.46 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.40 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

Cr1—N1	2.1352 (18)	Cr1—Cl3	2.3197 (6)
Cr1—N2	2.1447 (18)	Cr1—Cl1	2.3358 (7)
Cr1—Cl2	2.3171 (7)	Cr1—P1	2.3502 (7)
N1—Cr1—N2	85.21 (7)	Cl3—Cr1—Cl1	94.81 (2)
N1—Cr1—Cl2	166.73 (5)	N1—Cr1—P1	81.22 (5)
N2—Cr1—Cl2	89.18 (5)	N2—Cr1—P1	80.06 (5)
N1—Cr1—Cl3	88.68 (5)	Cl2—Cr1—P1	85.98 (2)
N2—Cr1—Cl3	169.74 (5)	Cl3—Cr1—P1	90.87 (2)
Cl2—Cr1—Cl3	95.01 (2)	Cl1—Cr1—P1	173.86 (3)
N1—Cr1—Cl1	96.59 (5)	Cl3—P1—Cr1	126.34 (8)
N2—Cr1—Cl1	94.07 (5)	C1—P1—Cr1	103.12 (7)
Cl2—Cr1—Cl1	95.79 (2)	C7—P1—Cr1	102.11 (8)



**Figure 1**

A view of the molecular structure of (I); displacement ellipsoids are drawn at the 50% probability level and H atoms are not shown.

All H atoms were included in calculated positions (C—H = 0.93–0.97 Å) and refined in the riding-model approximation, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>iso</sub>(C) or 1.5*U*<sub>eq</sub>(methyl C).

Data collection: APEX2 (Bruker, 2003); cell refinement: SAINT-Plus (Bruker, 2003); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Sheldrick, 2000); software used to prepare material for publication: SHELXTL.

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### References

- Bruker (2003). APEX2 (Version 1.0-28) and SAINT-Plus (Version 6.25). Bruker AXS Inc., Madison, Wisconsin, USA.
- Gavrilova, A. L. & Bosnich, B. (2004). *Chem. Rev.* **104**, 349–384.
- Lindner, E., Rauleder, H. & Hiller, W. (1983). *Z. Naturforsch. Teil B*, **4**, 417–425.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Sheldrick, G. M. (2000). SHELXTL. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.