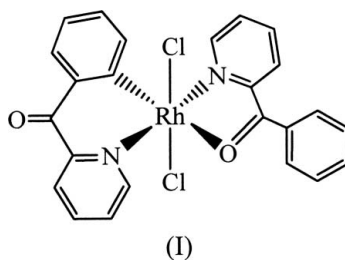


Mei Ching Tseng, Shao Pin
Wang, Yu Cheng Yu, Shi Yuan
Sheu and Wen Liang Huang*Department of Chemistry, National Cheng-Kung
University, Tainan 70101, TaiwanCorrespondence e-mail:
wlhuang@mail.ncku.edu.tw

Key indicators

Single-crystal X-ray study
 $T = 200$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.030
 wR factor = 0.090
Data-to-parameter ratio = 13.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*trans*-(2-Benzoylpyridine- $\kappa^2\text{N},\text{O}$)dichloro-
[2-(pyridine-2-carbonyl)phenyl- $\kappa^2\text{C}^1,\text{N}$]-
rhodium(III)The title organometallic complex, *trans*-[Rh(C₁₂H₈NO)-Cl₂(C₁₂H₉NO)], which was obtained from the reaction of rhodium(III) chloride and 2-benzoylpyridine, features an Rh^{III} atom coordinated by two N, one O, one C and two Cl atoms in a distorted octahedral environment.Received 19 October 2005
Accepted 26 October 2005
Online 31 October 2005

Comment

Several studies of the cyclometallation of *ortho*-metallated ligands such as anionic ppy (ppyH is 2-phenylpyridine) have been reported. For example, by simply mixing rhodium(III) chloride trihydrate with 2-pyridyl phenone (Hbzpy) in 2-methoxyethanol for 4 d at room temperature, de Geest & Steel (1995) synthesized Rh(Hbzpy)(bzpy)Cl₂ (bzpy is 2-pyridyl-2-phenonide). They reported that the complex had a five-membered *N,O* metallacycle involving the chelated Hbzpy ligand and a six-membered *N,C* metallacycle involving the chelated bzpy ligand, on the basis of NMR chemical-shift analysis. The authors claimed that they had isolated the *cis*-isomer out of the six possible diastereoisomers. We have prepared the title orthometallated Rh^{III} complex, (I), containing 2-pyridyl phenone, in order to ascertain the stereochemistry.Complex (I) exhibits a pseudo-octahedral geometry at the Rh^{III} centre, with two Cl ligands in a *trans* orientation. The pyridyl N atom of the Hbzpy ligand is *trans* to the N atom of the bzpy ligand. Cyclometallation leads to a boat conformation, with atoms Rh and C19 above the N2–C20–C18–C13 plane (Fig. 1). The pyridyl ring of the Hbzpy ligand and the phenyl ring of the bzpy ligand are mutually stacked [C1–N1–C13–C14 = 53.9 (2)°] (Table 1). The upfield shift of H(C1)/H(C14) and the nuclear Overhauser effect of these H atoms were also observed in the NMR spectrum. On this basis, we suggest that the NMR results alone are not sufficient to determine a *trans/cis* conformation. The five-membered chelate ring deviates slightly from planarity [N1–C5–C6–O1 = 17.4 (3)°] and is inclined to the phenyl plane [C5–C6–C7–C8 = 46.2 (4)°]. There are no short intermolecular contacts.

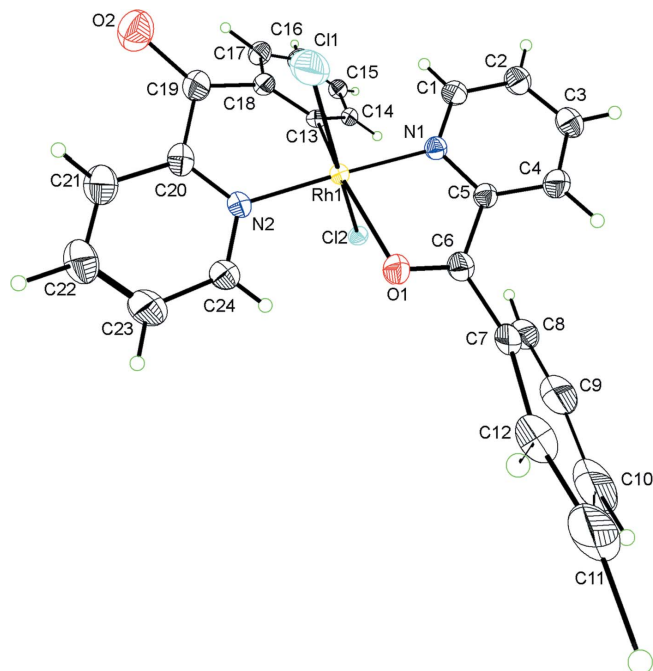


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

Experimental

The title complex was synthesized by heating a mixture of rhodium(III) chloride trihydrate (0.132 g, 0.5 mmol) and 2-pyridyl phenone (0.549 g, 3.0 mmol) in an oven at 423 K for 15 h. After cooling, the product was washed with methanol and orange single crystals were isolated (yield 78%). Details of the NMR analysis are available in the archived CIF.

Crystal data

[Rh(C₁₂H₈NO)Cl₂(C₁₂H₉NO)]
M_r = 539.21
 Monoclinic, *P*₂₁/*n*
a = 13.5492 (2) Å
b = 8.25150 (10) Å
c = 19.2229 (3) Å
 β = 96.5210 (10)°
V = 2135.24 (5) Å³
Z = 4

D_x = 1.677 Mg m⁻³
 Mo *K* α radiation
 Cell parameters from 18110 reflections
 θ = 2.0–25.4°
 μ = 1.08 mm⁻¹
T = 200 (2) K
 Prism, orange
 0.45 × 0.24 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.729, *T_{max}* = 0.918
 20293 measured reflections

3894 independent reflections
 3560 reflections with *I* > 2 σ (*I*)
R_{int} = 0.050
 θ_{\max} = 25.3°
h = -16 → 16
k = -9 → 9
l = -23 → 23

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.030
wR(*F*²) = 0.090
S = 1.13
 3894 reflections
 281 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.8456P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 0.90 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -1.18 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick, 1997)
 Extinction coefficient: 0.0129 (7)

Table 1

Selected geometric parameters (Å, °).

Rh1—Cl1	2.3246 (7)	Rh1—N2	2.028 (2)
Rh1—Cl2	2.3454 (6)	Rh1—Cl3	1.981 (3)
Rh1—O1	2.2831 (18)	O1—C6	1.229 (3)
Rh1—N1	2.042 (2)	O2—C19	1.217 (3)
Cl1—Rh1—Cl2	175.00 (2)	N2—Rh1—O1	98.96 (8)
O1—Rh1—Cl1	92.30 (5)	N2—Rh1—N1	174.74 (8)
O1—Rh1—Cl2	82.70 (5)	Cl3—Rh1—Cl1	93.29 (7)
N1—Rh1—Cl1	88.61 (6)	Cl3—Rh1—Cl2	91.70 (7)
N1—Rh1—Cl2	90.42 (6)	Cl3—Rh1—O1	171.09 (8)
N1—Rh1—O1	76.29 (7)	Cl3—Rh1—N1	96.90 (10)
N2—Rh1—Cl1	89.32 (6)	Cl3—Rh1—N2	88.05 (10)
N2—Rh1—Cl2	91.22 (6)		
Rh1—O1—C6—C5	28.0 (3)	Cl1—N1—Cl3—Cl4	53.9 (2)
Rh1—N1—C5—C6	-6.0 (3)	N1—C5—C6—O1	-17.4 (3)
Rh1—N2—C20—C19	8.8 (3)	C5—C6—C7—C8	-46.2 (4)
Rh1—Cl3—C18—C19	-13.3 (4)	C17—C18—C19—O2	-29.9 (4)
O1—Rh1—N2—C24	-28.5 (2)	O2—C19—C20—C21	30.3 (4)
N1—Rh1—Cl3—Cl4	40.3 (2)		

All H atoms bonded to C atoms were placed in calculated positions, with C—H = 0.96 Å, and treated as riding atoms, with *U*_{iso}(H) = 1.2*U*_{eq}(C). The final difference Fourier map had a large hole at about 1 Å from Rh.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); 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).

Financial support from the National Science Council of the Republic of China is acknowledged.

References

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Geest, D. J. de & Steel, P. J. (1995). *Aust. J. Chem.* **48**, 1573–1585.
 Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
 Sheldrick, G. M. (1985). *SHELXS86*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.