Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Chloropyridinebis[tricarbonyl(η^5-cyclopentadienyl)tungstenio]indium

Hiroshi Nakazawa,* Masumi Itazaki and Mari Owaribe

Department of Chemistry, Graduate School of Science, Osaka City University, Sumiyoshi-ku, Osaka 558-8585, Japan

Correspondence e-mail: nakazawa@sci.osaka-cu.ac.jp

Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.020 Å
R factor = 0.052
wR factor = 0.094
Data-to-parameter ratio = 18.0

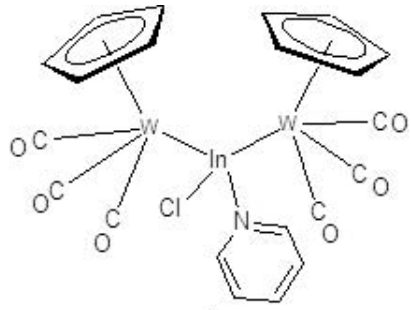
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 March 2005
Accepted 18 April 2005
Online 23 April 2005

In the title compound, [In{W(C_5H_5)(CO)$_3$}$_2$(C_5H_5N)Cl], the In atom is coordinated by two Cp(CO)$_3$W fragments, a Cl ligand and a pyridine molecule in a distorted tetrahedral environment. The In—W bond distances are 2.8445 (8) and 2.8716 (9) Å.

Comment

The crystal structure of the title compound, (I), shows that the In atom, with Cl and pyridine (py) substituents, bridges two Cp(CO)$_3$W fragments, resulting in a distorted tetrahedral geometry around the In atom (Fig. 1). Only three crystal structures have been reported to date for indium complexes with In—W bond(s) (Leiner & Scheer, 2002; Reger *et al.*, 1994; Rutsch *et al.*, 2002), and the present paper reports the first crystal structure of an In-bridged ditungsten complex.



(I)

The two In—W distances [2.8445 (8) and 2.8716 (9) Å] in (I) are comparable with those previously reported for [In{CpW(CO)$_3$}$_3$] [2.868 (2)–2.894 (2) Å; Leiner & Scheer, 2002], [HB(3,5-Me$_2$pz)$_3$]InW(CO)$_5$ [2.783 (2) Å; 3,5-Me$_2$pz = 3,5-dimethylpyrazolyl; Reger *et al.*, 1994] and [Ph_4P]$_2$-[W(CO)$_5$InCl$_3$] [2.7581 (1) Å; Rutsch *et al.*, 2002]. The In1—Cl1 bond distance of 2.460 (3) Å is close to those reported by Clarkson *et al.* (1991). The angles around the In atom are 127.38 (3)° (W1—In1—W2) and 95.0 (2)° (Cl1—In1—N1), and are similar to those in dichromium complexes bridged by an In atom having a Lewis base, *e.g.* [{Cp(CO)$_3$Cr}$_2$InCl(py)] (Clarkson *et al.*, 1991).

Experimental

To a solution of $InCl_3$ (332 mg, 1.5 mmol) in tetrahydrofuran (THF; 15 ml) was added a THF solution of K[Cp(CO)$_3$W] prepared by the reaction of [Cp(CO)$_3$W]$_2$ (1 g, 1.5 mmol) with $NaK_{2.8}$ (a liquid alloy, 1.6 ml) in THF (20 ml), and the mixture was stirred for 8 h (Hughes *et al.*, 1985). The solvent was removed under reduced pressure to give a yellow–orange oil, which was washed with water repeatedly and dried

in vacuo. The resulting material was dissolved in THF (30 ml), a small excess amount of pyridine was added and the solution was stirred for 1 h at room temperature. After removal of volatile materials under reduced pressure, the residue was washed with water and dried *in vacuo* to give (I) as a pale yellow solid (yield 1.18 g, 1.32 mmol, 88%). Single crystals of (I) were obtained by solvent diffusion at 253 K over a few days with a THF layer containing (I) and an overlayer of hexane. IR (THF, ν_{CO} cm^{-1}): 1988, 1963, 1887, 1874; ^{1}H NMR (400 MHz, $CDCl_3$, p.p.m.): δ 5.45 (*s*, Cp, 10 H), 7.54 (*m*, 2 H), 7.93 (*m*, 1 H), 8.87 (*d*, J = 4.4 Hz, 2 H); ^{13}C NMR (100.3 MHz, $CDCl_3$, p.p.m.): δ 88.7 (*s*, Cp), 125.6 (*s*, py), 140.1 (*s*, py), 148.7 (*s*, py), 217.2 (*s*, CO), 218.1 (*s*, CO).

Crystal data

$[InW_2(C_5H_5)_2Cl(C_5H_5N)(CO)_6]$	Z = 2
M_r = 895.33	D_x = 2.577 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 8.171 (2) Å	Cell parameters from 4130 reflections
b = 8.774 (2) Å	θ = 4.0–27.5°
c = 16.385 (4) Å	μ = 11.10 mm^{-1}
α = 93.826 (6)°	T = 203.2 K
β = 94.393 (6)°	Chip, pale yellow
γ = 98.725 (6)°	0.10 × 0.05 × 0.02 mm
V = 1154.0 (5) Å^3	

Data collection

Rigaku/MSC Mercury CCD diffractometer	5204 independent reflections
	3922 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.043
Absorption correction: multi-scan (Jacobson, 1998)	θ_{max} = 27.5°
	$h = -10 \rightarrow 9$
T_{min} = 0.523, T_{max} = 0.801	$k = -11 \rightarrow 9$
11 515 measured reflections	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0303P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.052	$(\Delta/\sigma)_{max}$ = -0.001
$wR(F^2)$ = 0.094	$\Delta\rho_{max}$ = 1.47 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −1.00 e Å^{-3}
5204 reflections	Extinction correction: none
289 parameters	
H-atom parameters not refined	

Table 1
Selected geometric parameters (Å, °).

W1—In1	2.8445 (8)	W2—C19	1.96 (1)
W1—C6	1.99 (1)	W2—C20	1.96 (1)
W1—C7	1.98 (1)	W2—C21	1.99 (2)
W1—C8	1.96 (1)	In1—Cl1	2.460 (3)
W2—In1	2.8716 (9)	In1—N1	2.303 (8)
In1—W1—C6	70.8 (3)	W1—In1—W2	127.38 (3)
In1—W1—C7	126.9 (3)	W1—In1—Cl1	108.04 (7)
In1—W1—C8	70.5 (3)	W1—In1—N1	105.5 (2)
In1—W2—C19	70.3 (3)	W2—In1—Cl1	109.50 (7)
In1—W2—C20	69.5 (3)	W2—In1—N1	106.5 (2)
In1—W2—C21	124.4 (4)	Cl1—In1—N1	95.0 (2)

H atoms were positioned with an ideal geometry, with a C—H distance of 0.95 Å and with U_{iso}(H) = U_{eq}(C), and were fixed in position in the further refinement, leading to final C—H distances in the range 0.96–0.98 Å.

Data collection: *CrystalClear* (Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2000); program(s) used to solve structure: *DIRDIF94* (Beurskens *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

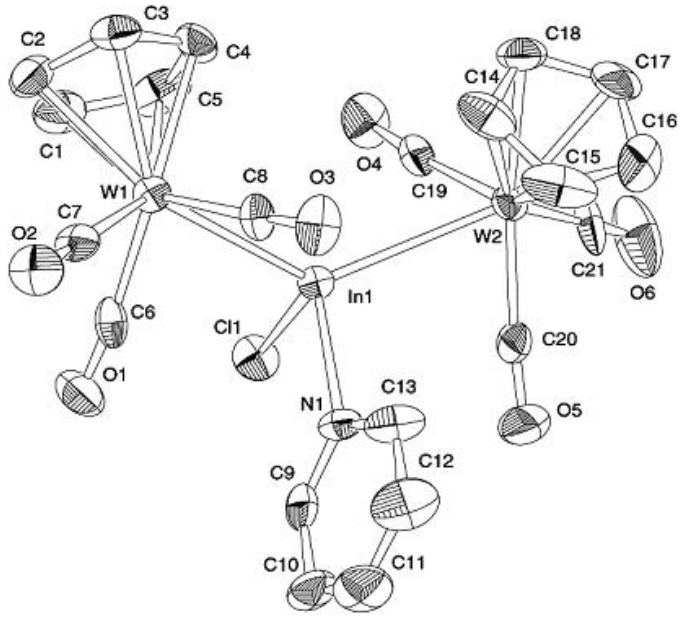


Figure 1
ORTEPII (Johnson, 1976) drawing of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

This work was supported by a Grant-in-Aid (No. 15205010) and by a Grant-in-Aid for Science Research on Priority Areas (No. 16033250, Reaction Control of Dynamic Complexes) from the Ministry of Education, Culture, Sports, Science and Technology, Japan, and by the Yamada Science Foundation.

References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., de Gelder, R., Israel, R. & Smits, J. M. M. (1994). *The DIRDIF94 Program System.* Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

Clarkson, L. M., Clegg, W., Hockless, D. C. R., Norman, N. C., Farrugia, L. J., Bott, S. G. & Atwood, J. L. (1991). *J. Chem. Soc. Dalton Trans.* pp. 2241–2252.

Hughes, R. P., Klaeui, W., Reisch, J. W. & Mueller, A. (1985). *Organometallics*, **4**, 1761–1766.

Jacobson, R. (1998). Private communication to the Rigaku Corporation.

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Leiner, E. & Scheer, M. (2002). *J. Organomet. Chem.* **646**, 247–254.

Molecular Structure Corporation (2000). *TEXSAN*. Version 1.11. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.

Reger, D. L., Mason, S. S., Rheingold, A. L., Haggerty, B. S. & Arnold, F. P. (1994). *Organometallics*, **13**, 5049–5053.

Rigaku (2001). *CrystalClear*. Version 1.3 SP1. Rigaku Corporation, Tokyo, Japan.

Rutsch, P., Renner, G., Huttner, G. & Sandhöfner, S. (2002). *Z. Naturforsch. B Chem. Sci.* **57**, 757–772.

Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.