

O(1)—Mo(1)—CP(1)	108.5	O(2)—Mo(2)—CP(2)	126.9
O(2)—Mo(1)—N(1)	115.2 (2)	N(2)—Mo(2)—C(28)	88.0 (2)
O(2)—Mo(1)—C(21)	78.6 (2)	N(2)—Mo(2)—CP(2)	117.4
O(2)—Mo(1)—CP(1)	125.9	C(28)—Mo(2)—CP(2)	104.6
N(1)—Mo(1)—C(21)	86.7 (2)	Mo(1)—O(1)—Mo(2)	111.5 (1)
N(1)—Mo(1)—CP(1)	118.8	Mo(1)—O(2)—Mo(2)	113.9 (1)
C(21)—Mo(1)—CP(1)	106.0	Mo(1)—N(1)—O(3)	168.6 (4)
O(1)—Mo(2)—O(2)	67.3 (1)	Mo(2)—N(2)—O(4)	168.1 (4)
O(1)—Mo(2)—N(2)	92.8 (2)	Mo(1)—C(21)—C(22)	115.3 (4)
O(1)—Mo(2)—CP(2)	107.6	Mo(2)—C(28)—C(29)	116.5 (3)

† CP(1) and CP(2) are the unweighted centroids of the C(1)–C(5) and C(11)–C(15) rings, respectively.

The H atoms were fixed in calculated positions (O—H 0.87 and C—H 0.98 Å, with displacement parameters 1.2 times greater than those of the parent atoms). The orientations of the hydroxy and methyl H atoms were based on difference map positions. Some of the Cp* methyl C atoms exhibit markedly anisotropic displacement parameters. This is consistent with a combination of moderate molecular libration and some oscillation of the Cp* ligands about the metal-ring centroid vectors and is a feature often noted for Cp* complexes.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *TEXSAN*.

The authors are grateful to the Natural Sciences and Engineering Research Council of Canada for support of this work in the form of grants to PL. They also thank Professor R. C. Thompson of the Department of Chemistry, University of British Columbia, for his assistance with the magnetic susceptibility measurements (see supplementary material).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including intermolecular contacts and torsion angles, together with analytical, IR, ¹H NMR, mass spectral and magnetic susceptibility data, and a discussion of the spectral data have been deposited with the IUCr (Reference: FG1147). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Faller, J. W., Chase, K. J. & Mazzieri, M. R. (1995). *Inorg. Chim. Acta*, **229**, 39–45.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Legzdins, P., Lundmark, P. J. & Rettig, S. J. (1993). *Organometallics*, **12**, 3545–3552.
- Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1985, 1992). *TEXSAN. Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. C. (1968). *Acta Cryst.* **A24**, 351–359.
- Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O. & Watson, D. G. (1989). *J. Chem. Soc. Dalton Trans.* pp. S1–S83.

Acta Cryst. (1996). **C52**, 1115–1117

mer-{Bis[dimethyl(diphenylphosphino-methyl)silyl]amine-*N,P,P'*}dihydrido(iodo)iridium(III)

MICHAEL D. FRYZUK, PATRICIA A. MACNEIL, STEVEN J. RETTIG* AND MARTIN STEPHAN

Department of Chemistry, University of British Columbia, 2036 Main Mall, Vancouver, BC, Canada V6T 1Z1. E-mail: xtal@xray1.chem.ubc.ca

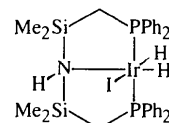
(Received 7 September 1995; accepted 7 December 1995)

Abstract

The [Ir(H)₂I(C₃₀H₃₇NP₂Si₂)] molecule has irregular octahedral coordination geometry, with the tridentate amine NH(SiMe₂CH₂PPh₂)₂ ligand meridionally coordinated. The amine N—H bond is *syn* to the Ir—I linkage as a consequence of intramolecular N—H···I hydrogen bonding. Important bond lengths include Ir—I 2.7862 (4), Ir—P 2.285 (1) (× 2), Ir—N 2.339 (4), and Ir—H 1.43 (4) and 1.46 (5) Å.

Comment

The geometrical parameters of [Ir(H)₂I{NH(SiMe₂CH₂-PPh₂)₂}] (I), are very similar to those reported (Fryzuk, MacNeil & Rettig, 1987) for the closely related compounds [Ir(Me)(H)I{NH[SiMe₂CH₂P(Pr)₂]₂}], [Rh(H)₂I{NH[SiMe₂CH₂P(Pr)₂]₂}], [Ir(H)₂Cl{NH(SiMe₂-CH₂PPh₂)₂}.toluene and [Ir(Me)I₂{NH(SiMe₂CH₂-PPh₂)₂}.benzene (Fryzuk *et al.*, 1992). Each of these



(I)

structures has slightly irregular octahedral coordination geometry with the tridentate NH(SiMe₂CH₂PR₂)₂ ligand meridionally coordinated to the metal atom with the halide ligand positioned *syn* to the amine N—H bond. This arrangement, as well as the stereoselectivity of the reactions leading to the hydride complexes, results from the formation of a weak intramolecular N—H···halide hydrogen bond. In the title compound, this weak N—H···I interaction has the geometry H···I 2.95 (5), N···I 3.536 (4) Å and N—H···I 135 (4)°. The H···I distance is significantly shorter than the sum of the van der Waals radii (3.35 Å; Bondi, 1964).

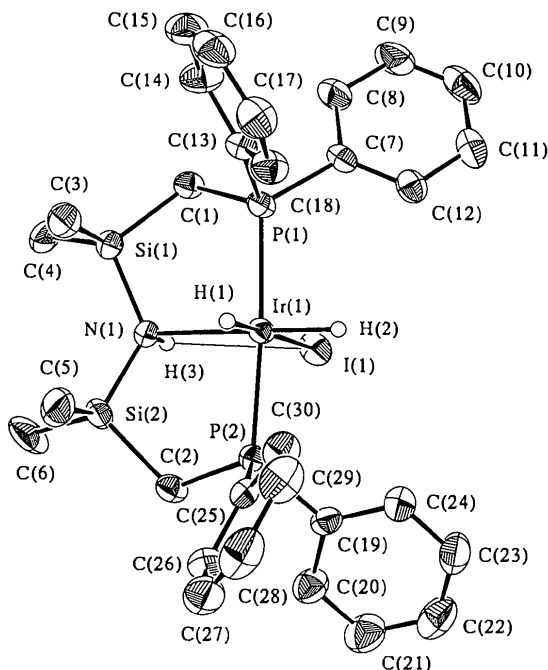


Fig. 1. Perspective view of the title molecule (33% probability ellipsoids are shown for the non-H atoms).

Experimental

The synthesis and physical properties of the title compound are reported elsewhere (Fryzuk, MacNeil & Rettig, 1987). Crystals were obtained by recrystallization from toluene and were mounted in a nitrogen-filled glass capillary.

Crystal data

[Ir(H)₂I(C₃₀H₃₇NP₂Si₂)]

$M_r = 850.89$

Monoclinic

$P2_1/c$

$a = 17.892(1) \text{ \AA}$

$b = 11.115(3) \text{ \AA}$

$c = 18.279(1) \text{ \AA}$

$\beta = 114.431(4)^\circ$

$V = 3309.8(7) \text{ \AA}^3$

$Z = 4$

$D_x = 1.707 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.7107 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 14.4 - 17.4^\circ$

$\mu = 5.15 \text{ mm}^{-1}$

$T = 294 \text{ K}$

Prism

$0.38 \times 0.30 \times 0.22 \text{ mm}$

Colorless

Data collection

Rigaku AFC-6S diffractometer

$\omega/2\theta$ scans

Absorption correction:

ψ scan (North, Phillips & Mathews, 1968)

$T_{\min} = 0.823$, $T_{\max} = 1.000$

10 396 measured reflections

10 107 independent reflections

5055 observed reflections

$[I > 3\sigma(I)]$

$R_{\text{int}} = 0.03$

$\theta_{\max} = 30^\circ$

$h = 0 \rightarrow 25$

$k = 0 \rightarrow 15$

$l = -25 \rightarrow 23$

3 standard reflections

monitored every 200

reflections

intensity decay: none

Refinement

Refinement on F

$R = 0.027$

$wR = 0.022$

$S = 1.320$

5055 reflections

347 parameters

$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.50 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.68 \text{ e \AA}^{-3}$

Extinction correction:

Zachariasen (1967) type

II, Gaussian isotropic

Extinction coefficient:

$0.299(15) \times 10^{-7}$

Atomic scattering factors

from *International Tables*

for X-ray Crystallography

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for H atoms: $U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^*$ for all others.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}/U_{\text{iso}}$
Ir(1)	0.25254 (1)	0.11385 (2)	0.35291 (1)	0.03751 (5)
I(1)	0.35713 (2)	0.13220 (3)	0.51534 (2)	0.0589 (1)
P(1)	0.19004 (7)	0.2928 (1)	0.35382 (8)	0.0409 (3)
P(2)	0.31079 (7)	-0.0626 (1)	0.33892 (8)	0.0410 (3)
Si(1)	0.31711 (8)	0.3662 (1)	0.29610 (8)	0.0465 (4)
Si(2)	0.39387 (9)	0.1204 (1)	0.27694 (9)	0.0543 (4)
N(1)	0.3528 (2)	0.2182 (4)	0.3274 (2)	0.041 (1)
C(1)	0.2640 (3)	0.4111 (4)	0.3616 (3)	0.045 (1)
C(2)	0.4061 (3)	-0.0256 (4)	0.3312 (3)	0.046 (1)
C(3)	0.2459 (3)	0.3671 (5)	0.1891 (3)	0.064 (2)
C(4)	0.4041 (3)	0.4706 (5)	0.3137 (3)	0.070 (2)
C(5)	0.3241 (4)	0.1006 (5)	0.1707 (3)	0.083 (2)
C(6)	0.4956 (4)	0.1764 (6)	0.2862 (5)	0.105 (3)
C(7)	0.1531 (3)	0.3248 (5)	0.4317 (3)	0.046 (1)
C(8)	0.1447 (4)	0.4423 (5)	0.4522 (3)	0.068 (2)
C(9)	0.1114 (4)	0.4669 (6)	0.5070 (4)	0.084 (3)
C(10)	0.0854 (4)	0.3742 (7)	0.5396 (3)	0.083 (2)
C(11)	0.0928 (3)	0.2569 (6)	0.5191 (3)	0.067 (2)
C(12)	0.1277 (3)	0.2321 (5)	0.4662 (3)	0.053 (2)
C(13)	0.0974 (3)	0.3283 (4)	0.2632 (3)	0.044 (1)
C(14)	0.0772 (3)	0.4465 (5)	0.2350 (3)	0.068 (2)
C(15)	0.0063 (4)	0.4704 (5)	0.1674 (4)	0.072 (2)
C(16)	-0.0459 (3)	0.3795 (6)	0.1286 (3)	0.071 (2)
C(17)	-0.0282 (3)	0.2650 (6)	0.1567 (3)	0.068 (2)
C(18)	0.0439 (3)	0.2383 (5)	0.2239 (3)	0.057 (2)
C(19)	0.3380 (3)	-0.1808 (4)	0.4152 (3)	0.048 (1)
C(20)	0.4102 (3)	-0.2432 (5)	0.4389 (3)	0.066 (2)
C(21)	0.4278 (4)	-0.3376 (6)	0.4922 (4)	0.087 (2)
C(22)	0.3731 (5)	-0.3698 (6)	0.5232 (4)	0.088 (2)
C(23)	0.3018 (4)	-0.3063 (6)	0.5012 (4)	0.076 (2)
C(24)	0.2849 (4)	-0.2115 (5)	0.4484 (3)	0.067 (2)
C(25)	0.2491 (3)	-0.1521 (4)	0.2508 (3)	0.044 (1)
C(26)	0.2825 (3)	-0.2292 (5)	0.2124 (3)	0.062 (2)
C(27)	0.2336 (5)	-0.3004 (5)	0.1491 (4)	0.078 (2)
C(28)	0.1496 (5)	-0.2964 (6)	0.1218 (4)	0.086 (2)
C(29)	0.1150 (4)	-0.2208 (6)	0.1587 (4)	0.075 (2)
C(30)	0.1643 (3)	-0.1489 (5)	0.2225 (3)	0.059 (2)
H(1)	0.202 (3)	0.107 (4)	0.268 (3)	0.08 (1)
H(2)	0.194 (3)	0.050 (4)	0.376 (3)	0.08 (1)
H(3)	0.381 (3)	0.214 (5)	0.373 (3)	0.06 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Ir(1)—I(1)	2.7862 (4)	P(2)—C(19)	1.829 (5)
Ir(1)—P(1)	2.285 (1)	P(2)—C(25)	1.826 (5)
Ir(1)—P(2)	2.285 (1)	Si(1)—N(1)	1.771 (4)
Ir(1)—N(1)	2.339 (4)	Si(1)—C(1)	1.878 (4)
Ir(1)—H(1)	1.43 (4)	Si(1)—C(3)	1.840 (5)
Ir(1)—H(2)	1.46 (5)	Si(1)—C(4)	1.860 (5)
N(1)—H(3)	0.77 (4)	Si(2)—N(1)	1.771 (4)
P(1)—C(1)	1.830 (4)	Si(2)—C(2)	1.867 (5)
P(1)—C(7)	1.835 (4)	Si(2)—C(5)	1.836 (6)
P(1)—C(13)	1.838 (5)	Si(2)—C(6)	1.863 (6)
P(2)—C(2)	1.816 (4)		

I(1)—Ir(1)—P(1)	92.50 (3)	C(2)—P(2)—C(19)	105.1 (2)
I(1)—Ir(1)—P(2)	92.62 (3)	C(2)—P(2)—C(25)	107.4 (2)
I(1)—Ir(1)—N(1)	86.77 (10)	C(19)—P(2)—C(25)	98.9 (2)
P(1)—Ir(1)—P(2)	174.46 (4)	N(1)—Si(1)—C(1)	104.5 (2)
P(1)—Ir(1)—N(1)	89.3 (1)	N(1)—Si(1)—C(3)	110.3 (2)
P(2)—Ir(1)—N(1)	88.9 (1)	N(1)—Si(1)—C(4)	111.1 (2)
I(1)—Ir(1)—H(1)	176 (1)	C(1)—Si(1)—C(3)	111.4 (2)
I(1)—Ir(1)—H(2)	88 (1)	C(1)—Si(1)—C(4)	109.4 (2)
P(1)—Ir(1)—H(1)	87 (1)	C(3)—Si(1)—C(4)	110.0 (3)
P(1)—Ir(1)—H(2)	90 (1)	N(1)—Si(2)—C(2)	103.9 (2)
P(2)—Ir(1)—H(1)	87 (1)	N(1)—Si(2)—C(5)	112.2 (2)
P(2)—Ir(1)—H(2)	91 (1)	N(1)—Si(2)—C(6)	110.4 (2)
N(1)—Ir(1)—H(1)	90 (1)	C(2)—Si(2)—C(5)	109.7 (2)
N(1)—Ir(1)—H(2)	174 (1)	C(2)—Si(2)—C(6)	110.3 (3)
H(1)—Ir(1)—H(2)	95 (2)	C(5)—Si(2)—C(6)	110.3 (3)
Ir(1)—P(1)—C(1)	106.6 (1)	Ir(1)—N(1)—Si(1)	108.7 (2)
Ir(1)—P(1)—C(7)	120.6 (2)	Ir(1)—N(1)—Si(2)	108.5 (2)
Ir(1)—P(1)—C(13)	116.2 (2)	Si(1)—N(1)—Si(2)	125.0 (2)
C(1)—P(1)—C(7)	106.3 (2)	Ir(1)—N(1)—H(3)	87 (3)
C(1)—P(1)—C(13)	105.6 (2)	Si(1)—N(1)—H(3)	113 (3)
C(7)—P(1)—C(13)	100.2 (2)	Si(2)—N(1)—H(3)	107 (3)
Ir(1)—P(2)—C(2)	107.5 (2)	P(1)—C(1)—Si(1)	107.4 (2)
Ir(1)—P(2)—C(19)	121.4 (2)	P(2)—C(2)—Si(2)	109.3 (2)
Ir(1)—P(2)—C(25)	115.4 (2)		

The metal hydride and amine H atoms were refined with isotropic displacement parameters. The remaining H atoms were fixed in calculated positions (methyl groups staggered, with C—H = 0.98 Å and displacement parameters 20% larger than those of the parent atoms).

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

We thank the Natural Sciences and Engineering Research Council Canada for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including least-squares-planes data, torsion angles and selected non-bonded contacts < 3.80 Å, have been deposited with the IUCr (Reference: FG1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Fryzuk, M. D., Huang, L., McManus, N. T., Paglia, P., Rettig, S. J. & White, G. (1992). *Organometallics*, **11**, 2979–2990.
- Fryzuk, M. D., MacNeil, P. A. & Rettig, S. J. (1987). *J. Am. Chem. Soc.* **109**, 2803–2812.
- Molecular Structure Corporation (1988). *MSCIAFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Molecular Structure Corporation (1992). *TEXSAN. Single Crystal Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Acta Cryst. (1996). **C52**, 1117–1119

Chlorotris(triphenylphosphine)copper(I) Tetrahydrofuran Solvate

TOM F. CARLSON,^a JOHN P. FACKLER JR^a AND ROMAN A. KRESIŃSKI^{b*}

^aDepartment of Chemistry, Texas A&M University, College Station, Texas 77843, USA, and ^bSchool of Sciences, Staffordshire University, College Road, Stoke-on-Trent ST4 2DE, England. E-mail: sctrak@staffs.ac.uk

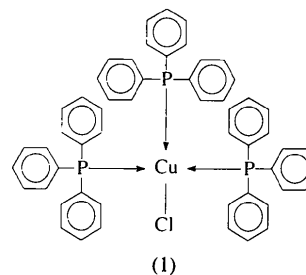
(Received 28 April 1995; accepted 14 September 1995)

Abstract

The structure of [CuCl{P(C₆H₅)₃}₃].C₄H₈O consists of discrete [CuCl{P(C₆H₅)₃}₃] molecules containing pseudo-tetrahedrally coordinated copper, together with one disordered tetrahydrofuran solvent molecule per molecule of complex.

Comment

The structure of [CuCl{P(C₆H₅)₃}₃], (1), has been determined previously in its unsolvated form [(2); Gill *et al.*, 1976], as a 1:3 solvate with tetrahydrofuran [(3); Foltg, Huffman, Mahoney, Stryker & Caulton, 1987] and as a 1:1 acetone solvate [(4); Barron *et al.*, 1987].



One of these previously determined structures, (2), exhibits very nearly tetrahedral coordination geometry [angles 108.41 (7)–110.51 (6)°, with the Cu—Cl distances [mean 2.335 (2) Å] shorter than the Cu—P distances [mean 2.351 (1) Å]. In structures (3) and (4), the Cu—P distances are shorter than the Cu—Cl distances [2.3183 (17) and 2.3469 (27) Å, respectively, for (3); 2.326 (4) and 2.349 (6) Å for (4)] and a concomitant increase of the P—Cu—P angles [115.00 (4) and 115.0 (2)°, respectively, for (3) and (4)] relative to those in (2) is exhibited. This may be considered to be a consequence of the increased mutual steric repulsion of the triphenylphosphine ligands in (3) and (4) relative to (2). However, in the structure described herein, (1), the Cu—Cl bond length [2.324 (2) Å] is shorter than the Cu—P