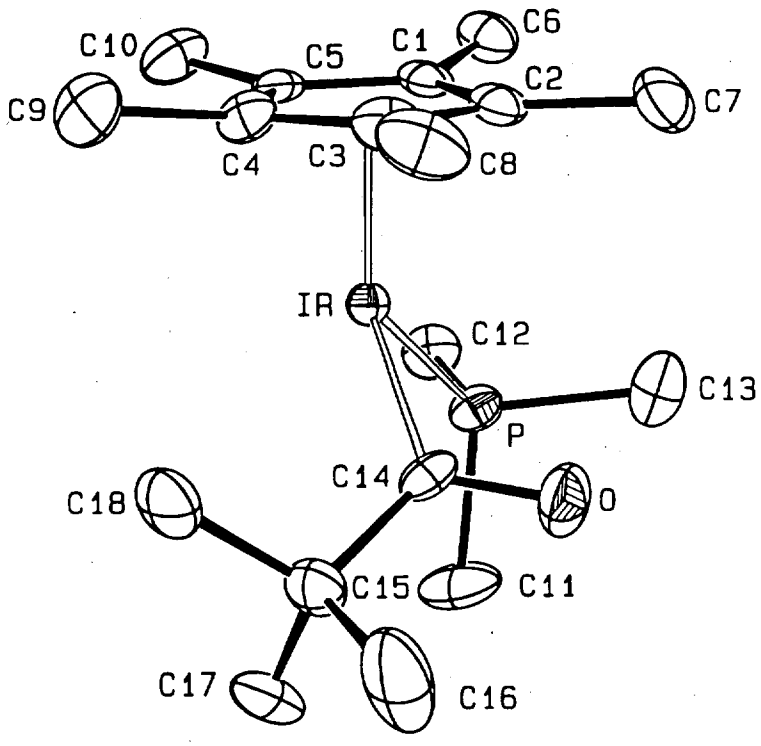
Deprotonation of the Transition Metal Hydride $(\eta^5\text{-}C_5Me_5)(PMe_3)IrH_2$. Synthesis and Chemistry of the Strongly Basic Lithium Iridate $(\eta^5\text{-}C_5Me_5)(PMe_3)Ir(H)(Li)$

Thomas H. Peterson, Jeffery T. Golden and Robert G. Bergman*

Contribution from the Department of Chemistry, University of California, Berkeley, CA 94720

Supplementary Material



Structure of **7a**

Table S-1: Crystal and Data Collection Parameters for **7a**.
Empirical formula: Ir P O C_{18} H_{34}

(A) Crystal Parameters at T = -101° C [a,b]

a = 13.906(3) Å	Space Group: $P2_1/n$
b = 10.343(2) Å	Formula weight = 489.6 amu
c = 14.475(2) Å	Z = 4
α = 90.0°	d(calc) = 1.65 g cm^{-3}
ß = 109.200(14)°	μ(calc) = 68.4 cm^{-1}
γ = 90.0°	

V = 1966.1(12) $Å^3$
Size: 0.22 x 0.30 x 0.40 mm

(B) Data Measurement Parameters

Radiation: Mo Kα (γ = 0.71073Å)

Monochromator: Highly-oriented graphite (2θ = 12.2°)
Detector: Crystal scintillation counter, with PHA.
Reflections measured: + H, + K, +/-L

2θ range: 3 -> 45 deg

Scan Type: θ-2θ

Scan width: Δθ = 0.90 + 0.35 tanθ

Scan speed: 5.49 (θ, deg/min)

Background: Measured over 0.25*(Δθ) added to each end of the scan.

Vert. aperture = 4.0 mm Horiz. aperture = 2.0 + 1.0 tanθ mm
No. of reflections collected: 2732
No. of unique reflections: 2555
highest and lowest peaks in difference Fourier: 1.02/-0.36 electrons $Å^{-3}$

(C) Data reduction and refinement
Intensity standards: (338),(273),(732); measured every 1 hour of x-ray exposure time. Over the data collection period no net decrease in intensity was observed.

Orientation: Three reflections were checked after every 250 measurements. Crystal orientation was redetermined if any of the reflections were offset by more than 0.10 degree from their predicted positions. Reorientation was performed once during data collection.

R = 3.02%	wR = 3.66%
R_{all} = 4.0%	GOF = 1.78

a] Unit cell parameters and their esd's were derived by a least-squares fit to the setting angles of the unresolved Mo Kα components of 24 reflections with 2_ between 26° and 32°.

[b] In this and all subsequent tables the esd's of all parameters are given in parentheses, right-justified to the least significant digit(s) of the reported value.

Table S-2: Table of Positional Parameters and Their Estimated Standard Deviations for **7a**.

Atom	x	y	z	B(Å^2)
Ir	0.04618(1)	0.01015(1)	0.30512(1)	1.255(6)
P	0.1902(2)	-0.0644(2)	0.4116(2)	1.94(5)
O	0.1767(4)	0.2292(5)	0.3272(4)	3.0(1)
C1	0.0256(6)	-0.1400(8)	0.1835(5)	1.9(2)
C2	0.0481(6)	-0.0174(7)	0.1493(5)	2.0(2)
C3	-0.0327(6)	0.0678(8)	0.1459(6)	2.2(2)
C4	-0.1028(6)	0.0014(7)	0.1824(6)	1.9(2)
C5	-0.0676(5)	-0.1284(7)	0.2042(5)	1.6(2)
C6	0.0810(6)	-0.2635(8)	0.1852(6)	2.5(2)
C7	0.1362(7)	0.0127(8)	0.1145(6)	3.0(2)
C8	-0.0419(7)	0.2021(9)	0.1045(6)	3.3(2)
C9	-0.2064(6)	0.0482(9)	0.1779(6)	3.1(2)
C10	-0.1250(6)	-0.2344(9)	0.2338(6)	2.9(2)
C11	0.2403(7)	0.0150(8)	0.5286(7)	3.6(2)
C12	0.1879(6)	-0.2325(8)	0.4480(6)	2.6(2)
C13	0.3006(7)	-0.0611(9)	.3691(7)	3.5(2)
C14	0.0989(6)	0.1929(7)	0.3429(6)	1.8(2)
C15	0.0453(6)	0.2962(7)	0.3856(6)	2.2(2)
C16	0.0801(7)	0.4306(9)	0.3668(8)	4.9(2)
C17	0.0739(7)	0.2730(9)	0.4943(7)	3.5(2)
C18	-0.0695(6)	0.2902(8)	0.3403(6)	2.9(2)

Table S-3: Table of Positional Parameters and Their Estimated Standard Deviations for **7a**.

Atom	x	y	z	B(Å^2)
H6A	0.05509(1)	-0.30463(1)	0.12371(1)	3.2*
H6B	0.15144(1)	-0.24611(1)	0.19994(1)	3.2*
H6C	0.07168(1)	-0.31836(1)	0.23461(1)	3.2*
H7A	0.11867(1)	-0.00728(1)	0.04698(1)	3.9*
H7B	0.15308(1)	0.10186(1)	0.12441(1)	3.9*
H7C	0.19398(1)	0.03765(1)	0.15044(1)	3.9*
H8A	-0.07848(1)	0.19924(1)	0.03647(1)	4.3*
H8B	-0.07806(1)	0.25484(1)	0.13614(1)	4.3*
H8C	0.02366(1)	0.23711(1)	0.11529(1)	4.3*
H9A	-0.25422(1)	0.03050(1)	0.11565(1)	4.0*
H9B	-0.22644(1)	0.00514(1)	0.22701(1)	4.0*
H9C	-0.20395(1)	0.13881(1)	0.18999(1)	4.0*
H10A	-0.16763(1)	-0.27760(1)	0.17716(1)	3.8*
H10B	-0.07857(1)	-0.29429(1)	0.27469(1)	3.8*
H10C	0.16615(1)	-0.19826(1)	0.26857(1)	3.8*
H11A	0.30196(1)	-0.02641(1)	0.56612(1)	4.7*
H11B	0.25465(1)	0.10313(1)	0.51838(1)	4.7*
H11C	0.19252(1)	0.01098(1)	0.56179(1)	4.7*
H12A	0.25236(1)	-0.25525(1)	0.49351(1)	3.3*
H12B	0.13687(1)	-0.24364(1)	0.47796(1)	3.3*
H12C	0.17318(1)	-0.28661(1)	0.39218(1)	3.3*
H13A	0.35843(1)	-0.09421(1)	0.41870(1)	4.4*
H13B	0.28690(1)	0.11227(1)	0.31179(1)	4.4*
H13C	0.31350(1)	0.02568(1)	0.35465(1)	4.4*
H16A	0.04664(1)	0.49363(1)	0.39363(1)	6.3*
H16B	0.15162(1)	0.43784(1)	0.39808(1)	6.3*
H16C	0.06361(1)	0.44438(1)	0.29894(1)	6.3*
H17A	0.04143(1)	0.33582(1)	0.52221(1)	4.5*
H17B	0.05211(1)	0.18902(1)	0.50583(1)	4.5*
H17C	0.14550(1)	0.27954(1)	0.52421(1)	4.5*
H18A	-0.09922(1)	0.35545(1)	0.36799(1)	3.8*
H18B	-0.08792(1)	0.30411(1)	0.27138(1)	3.8*
H18C	-0.09289(1)	0.20784(1)	0.35188(1)	3.8*

Starred atoms were included with isotropic thermal parameters. The thermal parameter given for anisotropically refined atoms is the isotropic equivalent thermal parameter defined as:
(4/3) [a^2ß(1,1) + b^2ß(2,2) + c^2ß(3,3) + ab(cosγ)ß(1,2) + ac(cosß)ß(1,3) + bc(cosα)ß(2,3)]
where a,b,c are real cell parameters, and ß(i,j) are anisotropic betas.

Table S-4: Anisotropic Thermal Parameters for **7a.**

Name	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)	Beqv
Ir	1.31(1)	1.22(1)	1.24(1)	-0.03(1)	0.422(8)	0.03(1)	1.255(6)
P	1.87(8)	.58(8)	2.13(8)	-0.09(7)	0.31(6)	-0.02(7)	1.94(5)
O	2.6(2)	2.1(2)	5.0(3)	-0.7(2)	2.2(2)	-0.6(2)	3.0(1)
C1	2.1(3)	2.4(3)	0.9(3)	0.1(3)	0.2(2)	-0.3(3)	1.9(2)
C2	2.4(3)	2.3(3)	1.1(3)	-0.3(3)	0.5(2)	-0.2(3)	2.0(2)
C3	3.0(3)	1.7(3)	1.6(3)	0.3(3)	0.3(3)	0.3(3)	2.2(2)
C4	1.5(3)	1.8(3)	1.8(3)	0.3(3)	-0.1(2)	0.2(3)	1.9(2)
C5	1.9(3)	1.7(3)	1.0(3)	-0.7(2)	0.3(2)	-0.4(2)	1.6(2)
C6	2.8(3)	2.8(4)	.8(3)	0.4(3)	0.6(3)	0.0(3)	2.5(2)
C7	4.0(4)	3.2(4)	2.6(3)	-0.8(3)	2.0(2)	-0.4(3)	3.0(2)
C8	5.0(4)	3.2(4)	1.6(4)	0.5(4)	0.8(3)	0.7(3)	3.3(2)
C9	1.9(3)	4.2(4)	2.7(4)	1.2(3)	-0.0(3)	0.6(4)	3.1(2)
C10	2.7(4)	2.8(4)	2.8(4)	-0.9(3)	0.3(3)	-0.1(3)	2.9(2)
C11	3.1(4)	3.3(4)	2.9(4)	0.3(4)	-1.2(3)	-1.0(4)	3.6(2)
C12	2.7(3)	2.4(4)	2.4(4)	0.3(3)	0.4(3)	0.3(3)	2.6(2)
C13	2.5(3)	3.4(4)	4.7(4)	-0.2(3)	1.4(3)	0.4(4)	3.5(2)
C14	1.3(3)	2.0(3)	1.6(3)	-0.2(3)	-0.3(3)	0.2(3)	1.8(2)
C15	2.5(3)	1.7(3)	2.5(3)	0.1(3)	1.0(3)	-0.4(3)	2.2(2)
C16	6.1(4)	2.2(4)	8.7(5)	-0.2(3)	5.5(3)	-0.8(4)	4.9(2)
C17	3.8(4)	3.6(4)	3.3(4)	-0.0(3)	1.4(3)	-2.0(3)	3.5(2)
C18	2.6(3)	3.0(4)	3.2(4)	1.0(3)	1.1(3)	-0.6(3)	2.9(2)

The form of the anisotropic temperature factor is:
$\exp[-0.25\{h^2a^2B(1,1) + k^2b^2B(2,2) + l^2c^2B(3,3) + 2hkabB(1,2) + 2hlacB(1,3) + 2klbcB(2,3)\}]$ where a, b, and c are reciprocal lattice constants.

Table S-5: Intramolecular Distances for **7a**.

ATOM 1	ATOM 2	DISTANCE
Ir	P	2.225(2)
Ir	C1	2.294(8)
Ir	C2	2.282(8)
Ir	C3	2.281(8)
Ir	C4	2.244(8
Ir	C5	2.274(7)
Ir	C14	2.036(8)
Ir	CP	1.924
P	C11	1.803(9)
P	C12	1.820(9)
P	C13	1.834(10)
C14	O	1.235(9)
C14	C15	1.544(11)
C15	C16	1.525(13)
C15	C17	1.509(13)
C15	C18	1.515(12)
C1	C2	1.433(11)
C1	C5	1.430(11)
C1	C6	1.487(11)
C2	C3	1.416(12)
C2	C7	1.503(12)
C3	C4	1.428(12)
C3	C8	1.502(12)
C4	C5	1.427(11)
C4	C9	1.500(12)
C5	C10	1.499(12)

Table S-6: Intramolecular Angles for **7a**.

ATOM 1	ATOM 2	ATOM 3	ANGLE
Cp	IR	P	130.12
Cp	IR	C14	123.88
P	IR	C14	88.45(23)
Ir	P	C11	118.3(3)
Ir	P	C12	115.7(3)
Ir	P	C13	115.0(3)
C11	P	C12	101.7(4)
C11	P	C13	101.5(5)
C12	P	C13	102.2(4)
Ir	C14	O	119.6(6)
Ir	C14	C15	124.8(6)
O	C14	C15	115.5(7)
C14	C15	C16	109.7(7)
C14	C15	C17	108.1(7)
C14	C15	C18	112.1(7)
C16	C15	C17	109.9(8)
C16	C15	C18	108.1(8)
C17	C15	C18	108.9(7)
C2	C1	C5	108.3(7)
C2	C1	C6	126.6(8)
C5	C1	C6	124.7(7)
C1	C2	C3	107.6(7)
C1	C2	C7	126.4(7)
C3	C2	C7	125.8(7)
C2	C3	C4	108.6(7)
C2	C3	C8	123.5(8)
C4	C3	C8	127.9(8)
C3	C4	C5	107.9(7)
C3	C4	C9	126.2(7)
C5	C4	C9	124.6(7)
C1	C5	C4	107.6(7)
C1	C5	C10	127.1(7)
C4	C5	C10	125.1(7)