Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information for

Synthesis and structure of a new phosphonium-1-indenylide (PHIN) ligand, 4,7-dimethyl-1-$C_9H_4PMePh_2$, and of new PHIN complexes of rhodium and iridium

Kourosh Purdavaie and Michael C. Baird,*
Department of Chemistry, Queen's University,
Kingston, ON K7L3N6, Canada

Peter H. M. Budzelaar*
Department of Chemistry, University of Manitoba,
Winnipeg, MB R3T 2N2, Canada

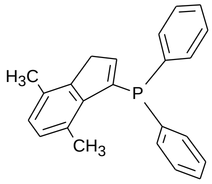
Table of contents

X-RAY STRUCTURE REPORT

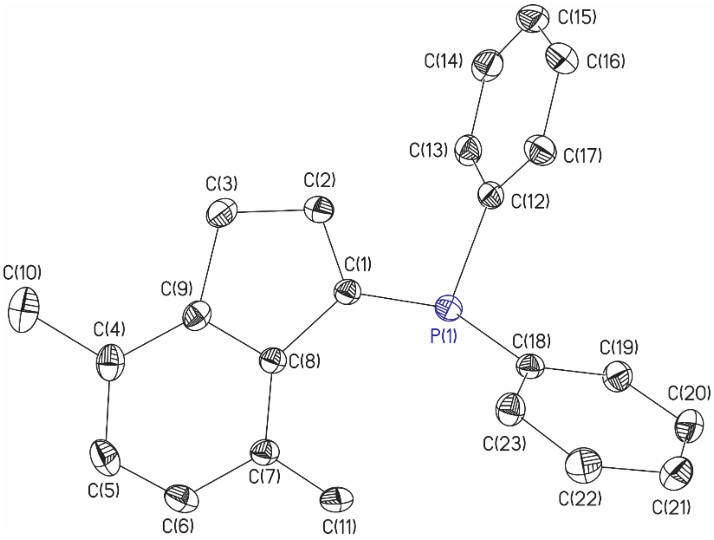
for

Diphenylphosphino-4,7-dimethylindene

Compound:



(1)



***XCD code:* mbkp_205**

Tuesday, September 22, 2015

Dr. Gabriele Schatte

(Instrumentation Manager – X-ray Diffraction Facility, Department of Chemistry, Queen’s University)

Submission Details

Submitted by: Kourosh Purdavaie

Sample Code: DMI

Principal Investigator: Prof. Mike Baird

Department: Chemistry, Queen's University

Data were collected and the crystal structure solved by:

Dr Gabriele Schatte (Instrumentation Manager – X-ray Diffraction Facility, Department of Chemistry, Queen's University)

Comments

Colorless and white crystals were submitted in two vial with a little bit solvent. Most of these crystals were multiple or twinned. A nice colourless rectangular crystal suitable for X-ray diffraction was retrieved from the sample.

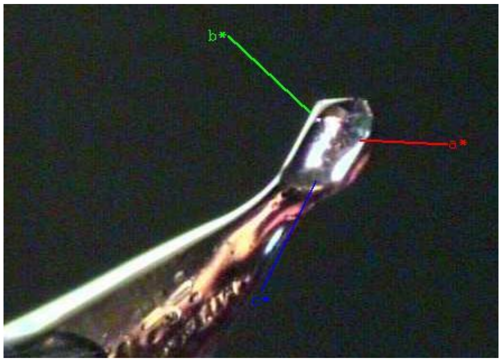
Experimental

Data Collection

A colourless rectangular-like crystal of (1) ($C_{23}H_{21}P_1$) having the approximate dimensions of 0.209 × 0.168 × 0.158 mm, coated with oil (Paratone 8277, Exxon), was collected onto the aperture of a mounted MicroMount^{TM} (diameter of the aperture: 100 microns; *MiTeGen* - Microtechnologies for Structural Genomics; USA) and quickly transferred to the cold stream of the Oxford Cryostream 700. The mounted Micromount^{TM} had previously been inserted into reusable magnetic goniometer base (B3S-R, *MiTeGen* - Microtechnologies for Structural Genomics; USA).

All measurements were made on a Bruker-AXS Smart Apex II 3-Circle diffractometer using graphite-monochromated Mo K_α radiation (λ = 0.71073 Å) at -93 °C (power: 50 kV × 30 mA, Bruker-AXS K780, diameter of the Monocap^{TM} collimator: 0.50 mm; frame size: 512 × 512).

The following picture shows the shape of the crystal:



An initial orientation matrix and cell was determined from three sets of 12 frames each using ω-scans (0.5° per frame, 10 s exposures per degree for each 6° in omega rotation[1] at *dx* = 63.00 mm)[2] [1]:

Set #	2 theta	Omega	phi
1	-28°	Start: 312°; End: 318°	0°
2	-28°	Start: 312°; End: 318°	120°
3	-28°	Start: 312°; End: 318°	240°

Data were measured using φ- and ω- scans [1] and seven sets of frames were collected (0.5° rotation per frame; exposure per frame: 15 s; *dx* = 63.117 mm):

Axis	dx/mm	2θ/°	ω/°	ϕ/°	χ/°	Width/°	Frames
Omega	63.112	-30.00	-232.50	30.00	54.74	0.50	450
Omega	63.112	0.00	-159.12	115.00	54.74	0.50	376
Omega	63.112	0.00	-163.07	-200.00	54.74	0.50	346

---

[1] Rotation in [°]: [# of frames] × [rotational angle]; *cf*. 0.5 °].

[2] dx: Detector distance.

Omega	63.112	10.00	-192.09	-195.00	54.74	0.50	365

A total of 1537 frames were collected. The frames were integrated with the Bruker SAINT [2] software package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 15913 reflections to a maximum θ angle of 27.21° (0.78 Å resolution). The final cell constants are based upon the refinement of the XYZ-centroids of 5343 reflections with $4.619° < 2\theta < 52.42°$:

a = 8.3278(4) Å	α = 90°
b = 17.6372(8) Å	β = 93.553(3)°
c = 12.0862(5) Å	γ = 90°
Volume	1771.80(14) Å^3

The space group was determined to be: $P2_1/c$ [No. 14].

Data Reduction

Data reduction was performed with the Bruker SAINT software [2], which corrects for beam inhomogeneity, possible crystal decay, Lorentz and polarisation effects. The linear absorption coefficient, μ, is 0.155 mm^{-1}. A multi-scan absorption correction was applied (Bruker SADABS,

[3]). The value for $wR_2(int)$ was 0.1350 before and 0.0456 after correction. The ratio of minimum to maximum transmission is 0.9421.

The resulting mean value $<E^2-1>$ for all data is 0.939 [expected 0.968 for centrosymmetric and 0.736 for non-centrosymmetric structures, respectively],[3] which corresponds to a centrosymmetric structure.

Using the program XPREP [4] an intensity statistics, showing the resolution dependence of *R(sigma)* and *R(int)*, was created for the reflection file:

```
================================================================================

INTENSITY STATISTICS FOR mbkp_205.hkl

Resolution  #Data #Theory %Complete Redundancy Mean I  Mean I/s R(int) R(sigma)

 Inf - 3.31      63     63    100.0     8.03     181.1   90.90  0.0178  0.0078
3.31 - 2.15     142    142    100.0     9.25      53.0   74.76  0.0199  0.0088
2.15 - 1.69     213    213    100.0     9.41      36.5   64.75  0.0220  0.0105
1.69 - 1.48     201    201    100.0     8.11      20.0   42.69  0.0314  0.0155
1.48 - 1.33     221    221    100.0     6.13      17.9   31.65  0.0357  0.0210
1.33 - 1.24     192    192    100.0     4.95      16.6   28.39  0.0383  0.0262
1.24 - 1.16     223    223    100.0     4.39      15.7   23.09  0.0476  0.0310
1.16 - 1.11     176    176    100.0     4.06      13.1   18.33  0.0429  0.0346
1.11 - 1.06     205    205    100.0     3.18       8.5   12.29  0.0570  0.0543
1.06 - 1.01     242    242    100.0     2.88       7.7   10.96  0.0636  0.0628
1.01 - 0.98     190    190    100.0     2.74       6.2    8.78  0.0875  0.0783
0.98 - 0.95     193    193    100.0     2.53       6.8    9.25  0.0744  0.0793
0.95 - 0.92     220    220    100.0     2.41       6.6    8.40  0.0736  0.0850
0.92 - 0.90     175    175    100.0     2.51       4.0    5.81  0.1028  0.1166
0.90 - 0.87     278    279     99.6     2.34       4.4    5.92  0.1152  0.1203
0.87 - 0.85     209    209    100.0     2.22       3.7    4.96  0.1259  0.1417
0.85 - 0.83     223    225     99.1     2.17       3.0    4.25  0.1326  0.1709
0.83 - 0.82     134    136     98.5     2.19       3.4    4.60  0.1121  0.1589
0.82 - 0.80     259    262     98.9     2.14       3.1    4.20  0.1384  0.1727
0.80 - 0.79     134    135     99.3     2.04       2.8    3.63  0.1672  0.1966
0.79 - 0.78     198    207     95.7     1.90       2.2    3.22  0.1713  0.2375
------------------------------------------------------------------------------
0.88 - 0.78    1246   1264     98.6     2.13       3.1    4.27  0.1344  0.1708
 Inf - 0.78    4091   4109     99.6     3.87      13.8   18.94  0.0299  0.0358

Merged [A],  lowest resolution = 17.64 Angstroms
------------------------------------------------------------------------------
```

NOTE: The R_{int} column gives a measure of whether symmetry equivalents have the same intensity. In the higher resolution shells (lower down the table), the R_{int} values get larger. The mean intensity and mean *I/sigma* tend to get smaller at higher resolution, while the R_{sigma} get larger. These variations with resolution happen because diffraction intensities tend to be weaker and more susceptible to noise at higher resolution. As a general rule, the data are usable so long as mean I/σ for a shell is ≥ 2 and the $R_{sigma} \leq 0.25$. Shells with mean $I/\sigma \leq 2$ contain very weak reflections.

```
================================================================================
```

---

[3] E: normalized structure factor.

This listing provides an indication of the resolution cutoff to be applied to the data.[4] A resolution cutoff was not applied. Of the 15913 reflections 433 reflections were rejected (SHELXL-2014 [6]).[5] The data (15480 reflections) were merged (all symmetry equivalents and Friedel opposites; $R_{\text{int}} = 0.0299$)[6] to provide 3945 data, of which all were unique ($R_{sigma} = 0.0307$) and 2994 observed ($I > 2\sigma(I)$) reflections (SHELXL-2014 [6]). The ranges of indices were $-10 \leq h \leq 10$, $-22 \leq k \leq 18$, $-15 \leq l \leq 15$ corresponding to a θ-range of 2.045 to 27.208.[7]

Structure Solution and Refinement

The structure was solved using direct methods in the space group $P2_1/c$ [No. 14] (SHELXT-2014 [5]) and refined by full-matrix least-squares method on F^2 with SHELXL-2014 [6] using ShelXle [8] as the graphical user interface (*GUI*).

The non-hydrogen atoms were refined anisotropically. Hydrogen atoms of the CH-, CH_2- and CH_3-groups were included at geometrically idealized positions (C-H bond distances 0.05/0.99/0.98 Å) and were not refined. The isotropic thermal parameters of these hydrogen atoms were fixed at 1.2 (CH-, CH_2-groups) and 1.5 times (CH_3-groups) that of the preceding carbon atom.

The final cycle of full-matrix least squares refinement using F^2 (SHELXL-2014, [5]) was based on 3945 reflections, 0 restraints, 219 variable parameters and converged (largest parameter shift was 0.001 times its esd) with an unweighted factor of $R_1 = 0.0429$ for $I > 2\sigma(I)$. The standard deviation of an observation of unit weight (*goodness-of-fit*) [7] was 1.041. The maximum and

---

[4] If the paper is destined for *Acta Cryst.*, there is a requirement that the data extend to 50° in 2θ for MoKα, i.e. a resolution of about 0.84 Å, so it may be advisable to cut there in borderline cases.

[5] Of the 433 rejected reflections 0 were presumably systematic absences and the remaining reflections were omitted because their F^2_o values were negative or their $\Delta F^2/esd$ were very high.

[6] $R_{\text{int}} = [\Sigma|F_o^2 - F_o^2(\text{mean})|]/[\Sigma|F_o^2|]$.

[7] The scattering angle θ defines the sphere in the reciprocal space outside of which data are not measured. With Mo radiation, this should be at least 25° and should not be less than 22°, the corresponding minimum lattice plane spacing, referred to as the "resolution" of the data, is 0.84 [10]. According to the guidelines given by the International Union of Crystallography, the diffractometer should be set so that a $\theta_{\max}$ of 25° for Mo radiation is reached. If the crystal diffracts *sufficiently well*, it is strongly recommended, that $\theta_{\max}$ is set to an even higher value.

minimum peaks in the final difference Fourier map corresponded to 0.310 and -0.296 $e^-/Å^3$, respectively.

Neutral atom scattering factors for non-hydrogen atoms and anomalous dispersion coefficients are contained in the SHELXTL [4] program library.

The plots for the crystal structure were generated using the program XP (part of the SHELXTL 6.14 [4] program library) and then imported into CorelDRAW™ X6 [9]. If not otherwise stated, the thermal ellipsoids in the molecular plots are shown at the 30% probability level.

References

[1] *APEX2 software package v2013.10-0,* Bruker AXS Inc.: Madison, WI, 2013.

[2] *Bruker SAINT v8.30A*: Part of the *APEX2 software package v2013.10-0*, Bruker AXS Inc.: Madison, WI, 2013.

[3] *Bruker SADABS v2012/1*: Part of the *APEX2 software package v2013.10-0*, Bruker AXS Inc.: Madison, WI, 2013.

[4] *SHELXTL (Version 6.14), XPREP (Version 2013/3), Program Library for Structure Solution and Molecular Graphics*; Bruker AXS, Inc.: Madison, WI, 2000-2013.

[5] G. M. Sheldrick, *SHELXT-2014, Program for the Solution of Crystal Structures;* University of Göttingen, Göttingen, Germany 2014.

[6] G. M. Sheldrick, *SHELXL-2014, Program for the Solution and Refining of Crystal Structures;* University of Göttingen, Göttingen, Germany 2014. G. M. Sheldrick, *Acta Cryst.* 2008, **A64**, 112–122.

Function minimized: $\Sigma w(|F_o|^2-|kF_c|^2)^2$; k: overall scale factor.

Refinement on F_o^2 for all reflections (all of these having $F_o^2 \geq -3\sigma(F_o^2)$). Weighted R-factors wR_2 and the values for *GooF* are based on F_o^2; conventional R-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. R-factors based on F_o^2 are statistically about twice as large as those based on F_o, and R-factors based on ALL data will be even larger.

[7] Standard deviation of an observation of unit weight (*goodness-of-fit* on F^2):

$GooF = \{\Sigma[w(F_o^2-F_c^2)^2]/(n - p)\}^{1/2}$

n: number of reflections, p: number of parameters

[8] *ShelXle v4.8.6 (2014)*: *A Qt graphical user interface for SHELXL*. C. B. Hübschle, G. M. Sheldrick and B. Dittrich, *J. Appl. Crystallogr.* **2011**, *44*, 1281-1284.

[9] *CorelDRAW^{TM} X6 v16.0.0.707*: Corel Corporation, Ottawa, Ontario, Canada, 2012.

[10] W. Massa, *Crystal Structure Determination*, Springer Verlag, Berlin, 2000.

Table 1. Crystal data and structure refinement for mbkp_205.

A. Crystal Data

Identification code	mbkp_205
Empirical formula	$C_{23}H_{21}P_1$
Formula weight	328.37
Crystal Color, Habit	colorless, rectangular-like
Crystal dimensions (mm)	0.209 × 0.168 × 0.158
Crystal system	monoclinic
Space group	$P2_1/c$ [No. 14]
Unit cell parameters[a]	
a (Å)	8.3278(4)
b (Å)	17.6372(8)
c (Å)	12.0862(5)
α (°)	90
β (°)	93.553(3)
γ (°)	90
V (Å^3)	1771.90(14)
Z[b]	4
F(000)	696
Density (ρ_{calcd})	1.231 Mg/m^3
Absorption coefficient (μ)	0.155 mm^{-1}

B. Data Collection and Refinement Conditions

Diffractometer	Bruker-AXS Smart Apex II diffractometer[c]
Radiation	monochromated Mo K_α
Wavelength (Mo K_α)	0.71073 Å
Temperature	-93(2) °C [180(2) K]
Scan type	φ- and ω-scans (0.5°/frame, 20 s exposure/frame, 4 sets)
Theta range for data collection	2.045 to 27.208°
Completeness to theta = 25.242°	99.8%
Reflections collected[d]	15480
Index ranges	$-10 \leq h \leq 10$, $-22 \leq k \leq 18$, $-15 \leq l \leq 15$
Independent reflections $[F_o^2 \geq -3\sigma(F_o^2)]$[e]	3945 $[R_{int} = 0.0299]$[f]
Observed reflections $[F_o^2 > 2\sigma(F_o^2)]$[g]	2994
Absorption correction method	multi-scan [SADABS][k]
Anomalous Dispersion	For all non-hydrogen atoms
Structure solution method	Direct methods (SHELXT-2014)[h]
Refinement method	Full-matrix least-squares on F^2 (SHELXL-2014)[i]
Function Minimized	$\Sigma w(\lvert F_o\rvert^2 - \lvert kF_c\rvert^2)^2$ (k: overall scale factor)
Weighing scheme, w $w = [\sigma(F_o^2) + (a\,P)^2 + (b\,P)]^{-1}$	$w = [\sigma(F_o^2) + (0.0449\,P)^2 + 0.5900\,P]^{-1}$
P-factor	$[\mathrm{Max}(F_o^2,0) + 2\,F_c^2]/3$
Data / restraints / parameters	3945 $[F_o^2 \geq -3\sigma(F_o^2)]$ / 0 / 219

Reflection (observed)/parameter ratio	14:1
Reflection (data)/parameter ratio	18:1
Goodness-of-fit[j] on F^2	1.041
$GooF = \{\Sigma[w(F_o^2-F_c^2)^2]/(n - p)\}^{1/2}$	
n: number of reflections, p: number of parameters	
Final R indices	
$R_1 = [\Sigma\|\|F_o\|-\|F_c\|\|]/[\Sigma\|F_o\|]$ for $[F_o^2 > 2\sigma(F_o^2)]$[i]	0.0429
$wR_2 = \{[\Sigma w(F_o^2-F_c^2)^2]/[\Sigma w(F_o^2)^2]\}^{1/2}$ [all data]	0.1077
Max. Shift/Error in Final Cycle	0.001
Largest difference peak and hole	0.310 and -0.296^{-}/Å^{3}
Transmission factor (min)	0.7023 [SADABS][k]
Transmission factor (max)	0.7455 [SADABS][k]

---

[a] Obtained from least-squares refinement of 5343 centered reflections with $2.31° < \theta < 27.21°$ (mosaicity: 0.34°).

[b] Z is the number of formula units per unit cell. Comparison of Z with the multiplicity of the general position n_a of the space group will then require that the asymmetric unit is Z/n_a times the formula unit. The asymmetric unit is the minimum group of atoms whose positions, together with those generated by the symmetry operations of the space group generate the complete contents of the unit cell.

[c] Bruker-AXS Smart Apex II: 3-circle diffractometer; sealed Mo X-ray tube; Apex II: type of detector.

[d] Number of reflections after truncation or rejection (before merging).

[e] The criterion for the independent or unique reflections [$F_o^2 \geq -3\sigma(F_o^2)$] was taken from:

G. M. Sheldrick, *SHELXL-2014, Program for the Solution of Crystal Structures;* University of Göttingen: Göttingen, Germany 2014.

[f] $R_{\text{int}} = [\Sigma|F_o^2 - F_o^2(\text{mean})|]/[\Sigma|F_o^2|]$

[g] The criterion for the observed reflections [$F_o^2 > 2\sigma(F_o^2)$] is equivalent to [$I > 2\sigma(I)$]: I is proportional to F_o^2.

[h] G. M. Sheldrick, *SHELXT-2014, Program for the Solution of Crystal Structures;* University of Göttingen, Göttingen, Germany 2014.

[i] G. M. Sheldrick, *SHELXL-2014, Program for the Solution of Crystal Structures;* University of Göttingen, Göttingen, Germany 2014. G. M. Sheldrick, *Acta Cryst.* 2008, **A64**, 112–122.

Function minimized: $\Sigma w(|\mathrm{F_o}|^2 - |k\mathrm{F_c}|^2)^2$; k: overall scale factor.

Refinement on F_o^2 for all reflections (all of these having $F_o^2 \geq -3\sigma(F_o^2)$). Weighted R-factors wR_2 and the values for *GooF* are based on F_o^2; conventional R-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. R-factors based on F_o^2 are statistically about twice as large as those based on F_o, and R-factors based on ALL data will be even larger.

[j] Standard deviation of an observation of unit weight (*goodness-of-fit* on F^2):

$$GooF = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n - p)\}^{1/2}$$

n: number of reflections, p: number of parameters

[k] *Bruker SADABS v2012/1*: Part of the *APEX2 software package v2013.10-0*, Bruker AXS Inc.: Madison, WI, 2013.

Table 2. Atomic coordinates (× 10^4), equivalent isotropic displacement parameters (Å^2 × 10^3) and site occupancy factors for mbkp_205.
U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
P(1)	3983(1)	2395(1)	6034(1)	28(1)
C(1)	4465(2)	1674(1)	5017(1)	26(1)
C(2)	5963(2)	1510(1)	4729(2)	31(1)
C(3)	6002(2)	850(1)	3965(2)	32(1)
C(4)	3592(2)	49(1)	3175(1)	32(1)
C(5)	1940(2)	-54(1)	3205(2)	38(1)
C(6)	1024(2)	408(1)	3846(1)	35(1)
C(7)	1681(2)	1003(1)	4483(1)	28(1)
C(8)	3347(2)	1115(1)	4461(1)	25(1)
C(9)	4264(2)	632(1)	3817(1)	27(1)
C(10)	4615(3)	-430(1)	2470(2)	45(1)
C(11)	606(2)	1483(1)	5156(2)	35(1)
C(12)	6020(2)	2746(1)	6408(1)	31(1)
C(13)	6788(2)	2447(1)	7366(2)	38(1)
C(14)	8389(2)	2611(1)	7646(2)	46(1)
C(15)	9223(2)	3089(1)	6991(2)	49(1)
C(16)	8471(2)	3408(1)	6055(2)	47(1)
C(17)	6875(2)	3239(1)	5764(2)	39(1)
C(18)	3134(2)	3171(1)	5182(1)	27(1)

C(19)	2837(2)	3853(1)	5720(2)	35(1)
C(20)	2134(2)	4456(1)	5147(2)	40(1)
C(21)	1671(2)	4389(1)	4033(2)	39(1)
C(22)	1942(2)	3716(1)	3499(2)	38(1)
C(23)	2680(2)	3113(1)	4057(1)	32(1)

Table 3a. Interatomic Distances [Å] for mbkp_205.

P(1)-C(18)	1.8294(17)
P(1)-C(1)	1.8301(17)
P(1)-C(12)	1.8358(17)
C(1)-C(2)	1.347(2)
C(1)-C(8)	1.487(2)
C(2)-C(3)	1.488(2)
C(3)-C(9)	1.498(2)
C(4)-C(9)	1.385(2)
C(4)-C(5)	1.390(2)
C(4)-C(10)	1.502(2)
C(5)-C(6)	1.386(3)
C(6)-C(7)	1.393(2)
C(7)-C(8)	1.403(2)
C(7)-C(11)	1.507(2)
C(8)-C(9)	1.410(2)
C(12)-C(13)	1.391(2)
C(12)-C(17)	1.392(2)
C(13)-C(14)	1.385(3)
C(14)-C(15)	1.373(3)
C(15)-C(16)	1.379(3)
C(16)-C(17)	1.386(3)
C(18)-C(23)	1.392(2)

C(18)-C(19)	1.396(2)
C(19)-C(20)	1.380(3)
C(20)-C(21)	1.383(3)
C(21)-C(22)	1.376(3)
C(22)-C(23)	1.384(2)

NOTE:

All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Table 3b. Interatomic Angles [°] for mbkp_205.

C(18)-P(1)-C(1)	103.59(7)
C(18)-P(1)-C(12)	101.82(8)
C(1)-P(1)-C(12)	99.36(7)
C(2)-C(1)-C(8)	107.76(14)
C(2)-C(1)-P(1)	124.55(13)
C(8)-C(1)-P(1)	127.37(11)
C(1)-C(2)-C(3)	112.59(15)
C(2)-C(3)-C(9)	102.55(13)
C(9)-C(4)-C(5)	116.68(15)
C(9)-C(4)-C(10)	120.75(16)
C(5)-C(4)-C(10)	122.56(17)
C(6)-C(5)-C(4)	121.14(17)
C(5)-C(6)-C(7)	122.76(16)
C(6)-C(7)-C(8)	116.77(15)
C(6)-C(7)-C(11)	119.67(15)
C(8)-C(7)-C(11)	123.56(15)
C(7)-C(8)-C(9)	119.71(15)
C(7)-C(8)-C(1)	132.55(15)
C(9)-C(8)-C(1)	107.74(13)
C(4)-C(9)-C(8)	122.93(15)
C(4)-C(9)-C(3)	127.72(15)
C(8)-C(9)-C(3)	109.34(14)
C(13)-C(12)-C(17)	118.48(16)
C(13)-C(12)-P(1)	116.57(13)

C(17)-C(12)-P(1)	124.76(14)
C(14)-C(13)-C(12)	120.67(19)
C(15)-C(14)-C(13)	120.04(19)
C(14)-C(15)-C(16)	120.21(18)
C(15)-C(16)-C(17)	119.95(19)
C(16)-C(17)-C(12)	120.59(18)
C(23)-C(18)-C(19)	118.24(16)
C(23)-C(18)-P(1)	124.53(13)
C(19)-C(18)-P(1)	117.11(13)
C(20)-C(19)-C(18)	120.84(17)
C(19)-C(20)-C(21)	120.40(17)
C(22)-C(21)-C(20)	119.15(18)
C(21)-C(22)-C(23)	121.01(17)
C(22)-C(23)-C(18)	120.34(16)

NOTE:

All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Table 4. Anisotropic displacement parameters (Å^2 × 10^3) for mbkp_205. The anisotropic displacement factor exponent takes the form:

$$[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$$

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1)	27(1)	30(1)	27(1)	0(1)	1(1)	-1(1)
C(1)	24(1)	25(1)	28(1)	4(1)	0(1)	0(1)
C(2)	25(1)	31(1)	38(1)	1(1)	1(1)	-2(1)
C(3)	27(1)	30(1)	39(1)	5(1)	7(1)	5(1)
C(4)	45(1)	23(1)	28(1)	5(1)	2(1)	2(1)
C(5)	48(1)	28(1)	36(1)	0(1)	-4(1)	-10(1)
C(6)	30(1)	37(1)	36(1)	6(1)	-4(1)	-7(1)
C(7)	26(1)	28(1)	29(1)	6(1)	0(1)	0(1)
C(8)	25(1)	24(1)	25(1)	6(1)	-1(1)	0(1)
C(9)	31(1)	23(1)	28(1)	7(1)	3(1)	4(1)
C(10)	64(1)	30(1)	42(1)	-3(1)	7(1)	6(1)
C(11)	23(1)	43(1)	40(1)	3(1)	2(1)	0(1)
C(12)	31(1)	28(1)	34(1)	-5(1)	-4(1)	0(1)
C(13)	43(1)	33(1)	36(1)	-5(1)	-7(1)	2(1)
C(14)	45(1)	47(1)	45(1)	-11(1)	-16(1)	10(1)
C(15)	30(1)	49(1)	67(2)	-21(1)	-10(1)	1(1)
C(16)	36(1)	40(1)	64(1)	-7(1)	2(1)	-8(1)
C(17)	35(1)	38(1)	45(1)	1(1)	-4(1)	-5(1)

C(18)	22(1)	30(1)	30(1)	-2(1)	3(1)	-1(1)
C(19)	37(1)	34(1)	34(1)	-8(1)	-2(1)	3(1)
C(20)	40(1)	30(1)	50(1)	-9(1)	-1(1)	5(1)
C(21)	37(1)	35(1)	45(1)	5(1)	-1(1)	5(1)
C(22)	43(1)	41(1)	30(1)	2(1)	-4(1)	0(1)
C(23)	37(1)	30(1)	30(1)	-3(1)	2(1)	2(1)

Table 5. Hydrogen coordinates (× 10^4) and isotropic displacement parameters (Å^2 × 10^3) for mbkp_205.

Atom	x	y	z	U(eq)
H(2)	6893	1786	4986	38
H(3A)	6659	432	4299	38
H(3B)	6433	996	3250	38
H(5)	1430	-448	2778	45
H(6)	-99	315	3852	41
H(10A)	5199	-102	1977	68
H(10B)	3928	-780	2025	68
H(10C)	5386	-720	2947	68
H(11A)	480	1986	4817	53
H(11B)	1089	1535	5913	53
H(11C)	-450	1241	5177	53
H(13)	6209	2128	7833	45
H(14)	8911	2393	8291	55
H(15)	10321	3200	7184	59
H(16)	9046	3744	5609	56
H(17)	6360	3461	5120	47
H(19)	3123	3902	6490	42
H(20)	1968	4921	5521	48
H(21)	1172	4802	3641	47
H(22)	1618	3666	2735	46
H(23)	2877	2656	3671	38

Table 6. Selected torsion angles [°] for mbkp_205.

C(18)-P(1)-C(1)-C(2)	105.46(15)
C(12)-P(1)-C(1)-C(2)	0.78(16)
C(18)-P(1)-C(1)-C(8)	-81.79(15)
C(12)-P(1)-C(1)-C(8)	173.53(14)
C(8)-C(1)-C(2)-C(3)	0.58(19)
P(1)-C(1)-C(2)-C(3)	174.53(12)
C(1)-C(2)-C(3)-C(9)	0.41(19)
C(9)-C(4)-C(5)-C(6)	0.1(3)
C(10)-C(4)-C(5)-C(6)	-178.57(17)
C(4)-C(5)-C(6)-C(7)	0.8(3)
C(5)-C(6)-C(7)-C(8)	-0.8(3)
C(5)-C(6)-C(7)-C(11)	179.79(16)
C(6)-C(7)-C(8)-C(9)	-0.2(2)
C(11)-C(7)-C(8)-C(9)	179.23(15)
C(6)-C(7)-C(8)-C(1)	-179.80(16)
C(11)-C(7)-C(8)-C(1)	-0.4(3)
C(2)-C(1)-C(8)-C(7)	178.27(17)
P(1)-C(1)-C(8)-C(7)	4.5(3)
C(2)-C(1)-C(8)-C(9)	-1.41(18)
P(1)-C(1)-C(8)-C(9)	-175.14(12)
C(5)-C(4)-C(9)-C(8)	-1.1(2)
C(10)-C(4)-C(9)-C(8)	177.62(15)

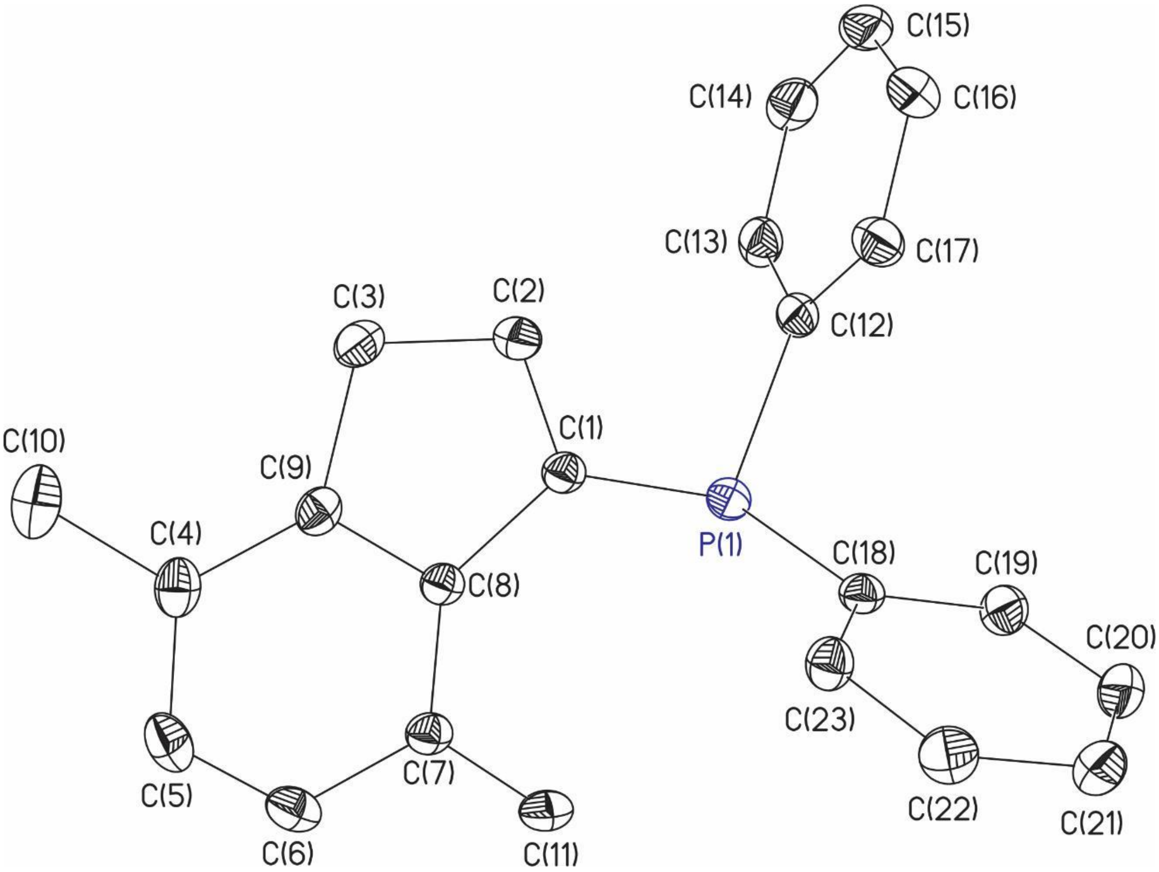
C(5)-C(4)-C(9)-C(3)	177.93(16)
C(10)-C(4)-C(9)-C(3)	-3.4(3)
C(7)-C(8)-C(9)-C(4)	1.1(2)
C(1)-C(8)-C(9)-C(4)	-179.14(15)
C(7)-C(8)-C(9)-C(3)	-178.05(14)
C(1)-C(8)-C(9)-C(3)	1.68(18)
C(2)-C(3)-C(9)-C(4)	179.57(16)
C(2)-C(3)-C(9)-C(8)	-1.30(18)
C(18)-P(1)-C(12)-C(13)	156.43(13)
C(1)-P(1)-C(12)-C(13)	-97.44(14)
C(18)-P(1)-C(12)-C(17)	-28.66(17)
C(1)-P(1)-C(12)-C(17)	77.48(17)
C(17)-C(12)-C(13)-C(14)	-2.7(3)
P(1)-C(12)-C(13)-C(14)	172.53(14)
C(12)-C(13)-C(14)-C(15)	1.7(3)
C(13)-C(14)-C(15)-C(16)	0.1(3)
C(14)-C(15)-C(16)-C(17)	-0.9(3)
C(15)-C(16)-C(17)-C(12)	-0.2(3)
C(13)-C(12)-C(17)-C(16)	1.9(3)
P(1)-C(12)-C(17)-C(16)	-172.90(15)
C(1)-P(1)-C(18)-C(23)	12.16(15)
C(12)-P(1)-C(18)-C(23)	114.96(14)
C(1)-P(1)-C(18)-C(19)	-171.84(12)
C(12)-P(1)-C(18)-C(19)	-69.04(14)
C(23)-C(18)-C(19)-C(20)	-0.9(2)

P(1)-C(18)-C(19)-C(20)	-177.21(14)
C(18)-C(19)-C(20)-C(21)	1.7(3)
C(19)-C(20)-C(21)-C(22)	-1.0(3)
C(20)-C(21)-C(22)-C(23)	-0.5(3)
C(21)-C(22)-C(23)-C(18)	1.3(3)
C(19)-C(18)-C(23)-C(22)	-0.6(2)
P(1)-C(18)-C(23)-C(22)	175.40(13)

NOTE:

All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Figure 1. Molecular ellipsoid plot of the major component in 4,7-dimethyl-1-$C_9H_4PMePh_2$ (II).



Chemistry Department, X-ray Lab

(Chernoff Hall, Room 107)

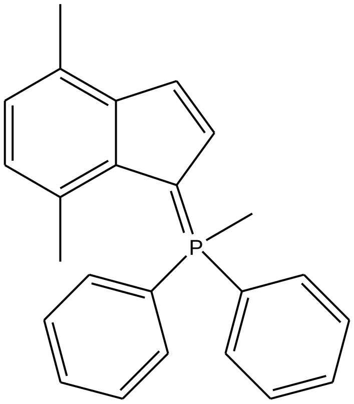
X-ray Crystallographic Analysis Report

By Dr. Ruiyao Wang

Tel. (613)533-6000x74474; email: rwang@chem.queensu.ca

PLEASE NOTE: This report was revised on April 30, 2014 by Dr Gabriele Schatte after changing the labelling scheme for the molecule upon request of Kourosk. The revisions are indicated in “blue”.

Sample ID:	mb42		
User:	**Kourosh**	**Supervisor:**	**Dr. Baird**
Date:	August 31, 2011 / revised April 30, 2014		



A crystal of the compound (yellow, block-shaped, size 0.30 x 0.25 x 0.20 mm) was mounted on a glass fiber with grease and cooled to -93 °C in a stream of nitrogen gas controlled with Cryostream Controller 700. Data collection was performed on a Bruker SMART APEX II X-ray diffractometer with graphite-monochromated Mo K_{α} radiation (λ = 0.71073 Å), operating at 50 kV and 30 mA over 2θ ranges of 4.38 ~ 51.98º. No significant decay was observed during the data collection.

Data were processed on a PC using the Bruker AXS Crystal Structure Analysis Package:[1] Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, V7.68A); data reduction: SAINT (Bruker, 2008); structure solution: XPREP (Bruker, 2008) and SHELXTL (Bruker, 2000-2008); structure refinement: SHELXTL; molecular graphics: SHELXTL; publication materials: SHELXTL. Neutral atom scattering factors were taken from Cromer and Waber.[2] The crystal is monoclinic space group *C*2/c, based on the systematic absences, *E* statistics and successful refinement of the structure. The structure was solved by direct methods. Full-matrix least-square refinements minimizing the function $\sum w\,(F_o^2 - F_c^2)^2$ were applied to the compound. All non-hydrogen atoms were refined anisotropically. All H atoms were placed in geometrically calculated positions, with C-H = 0.95 (aromatic), and 0.98(CH_3) Å, and refined as riding atoms, with Uiso(H) = 1.5UeqC(CH_3) or 1.2 UeqC(other C).

Convergence to final R_1 = 0.0341 for 3857 ($I > 2\sigma(I)$) independent reflections, and wR_2 = 0.0968 for all 4206 (R(int) = 0.0214) independent reflections, with 229 parameters and 0 restraints, were achieved.[3] The largest residual peak and hole to be 0.300 and –0.279 e/Å^3, respectively. Crystallographic data, atomic coordinates and equivalent isotropic displacement parameters, bond lengths and angles, anisotropic displacement parameters, hydrogen coordinates and isotropic displacement parameters, and torsion angles are given in Table 1 to 6. The molecular structure and the cell packing are shown in Figure 2.

The .CIF file, which comes with this report, contents the entire information about your structure. It is actually a .txt file. However, don't try anything to modify it unless you are familiar with it. You may be asked to deposit the .CIF file to Cambridge Crystallographic Data Centre (CCDC) before or when you submit your paper to a journal.

In case you have any concern about the data, drawings and the files of the report, please don't hesitate to contact me!

---

[1] Bruker AXS Crystal Structure Analysis Package:

Bruker (2000-2008). SHELXTL. Version 6.14. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2008). XPREP. Version 2008/2. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker. SAINT. Version 7.68A. Bruker AXS Inc., Madison,Wisconsin, USA.

Bruker (2008). APEX2. Bruker AXS Inc., Madison, Wisconsin, USA.

[2] Cromer, D. T.; Waber, J. T. *International Tables for X-ray Crystallography*; Kynoch Press: Birmingham, UK, 1974; Vol. 4, Table 2.2 A.

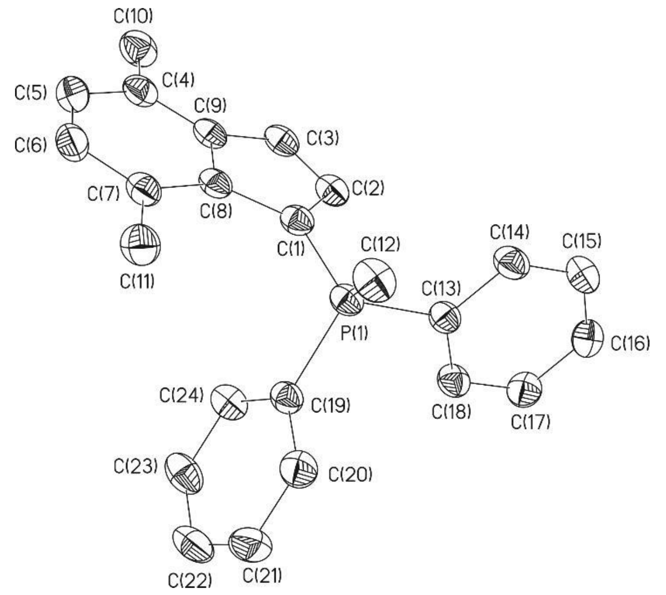
[3] $R_1 = [\Sigma||F_o|-|F_c||]/[\Sigma|F_o|]$ for $[F_o^2 > 2\sigma(F_o^2)]$

$wR_2 = \{[\Sigma w(F_o^2-F_c^2)^2]/[\Sigma w(F_o^2)^2]\}^{1/2}$ [all data]

($w = 1 / [\sigma^2(Fo^2) + (0.0544P)^2 + 2.679P]$, where $P = [\mathrm{Max}\ (Fo^2, 0) + 2Fc^2] / 3$)

Figure 2. Molecular Structure (ellipsoids for non-H atoms are shown at the 50% probability)

a)



b)

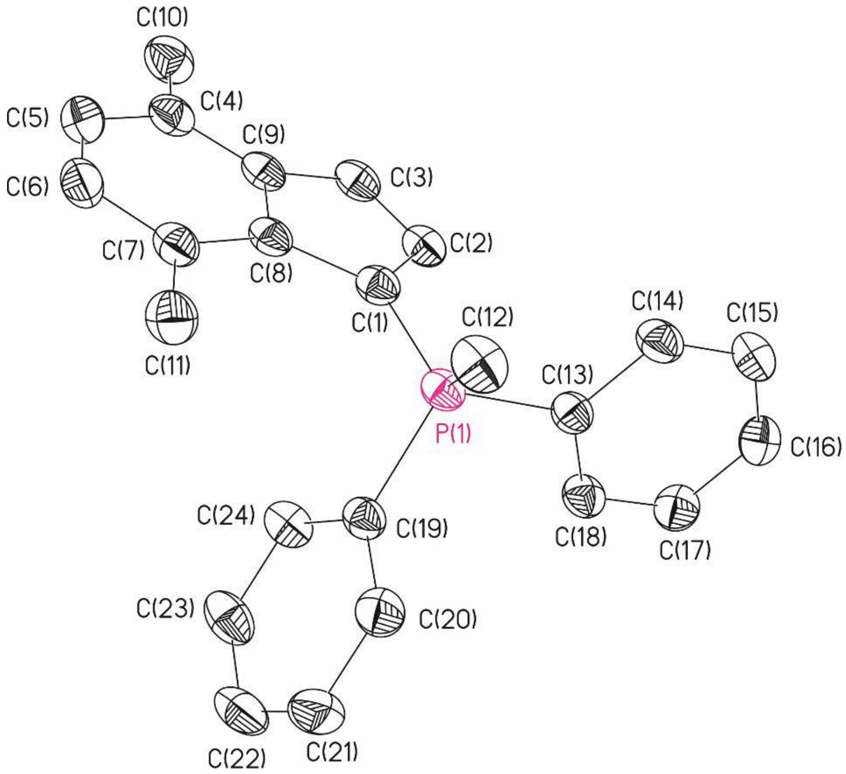
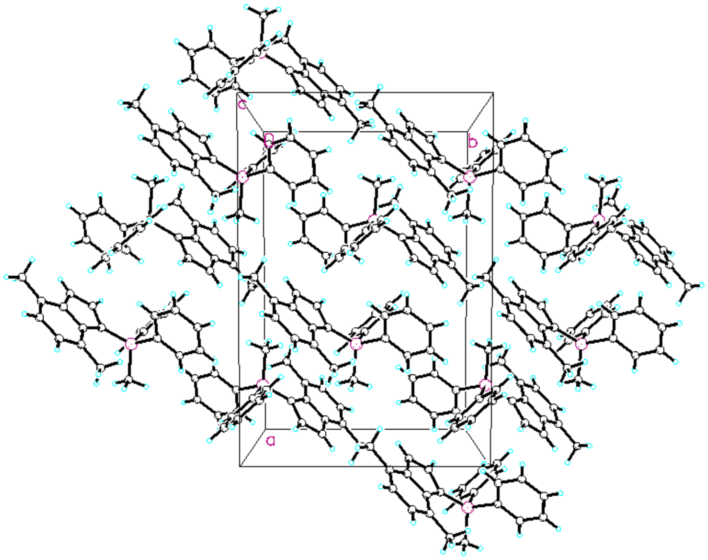


Figure 2. Unit cell packing

a)



b)

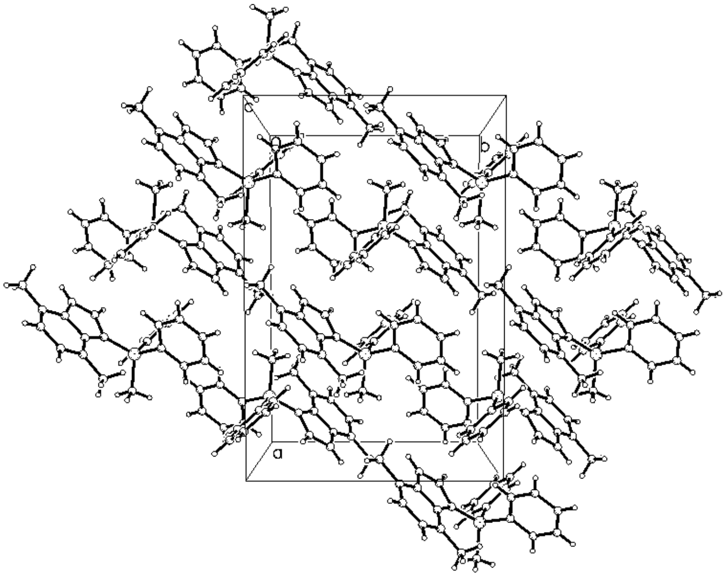


Table 7. Crystal data and structure refinement for mb42

Identification code	mb42	
Empirical formula	$C_{24}H_{23}P$	
Formula weight	342.39	
Temperature	180(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*C*2/*c*	
Unit cell dimensions	a = 16.6549(2) Å	α= 90°
	b = 11.23230(10) Å	β= 96.4639(4)°
	c = 20.2304(2) Å	γ = 90°
Volume	3760.50(7) $Å^3$	
Z	8	
Density (calculated)	1.210 Mg/m^3	
Absorption coefficient	0.149 mm^{-1}	
F(000)	1456	
Crystal size	0.30 x 0.25 x 0.20 mm^3	
Theta range for data collection	2.19 to 25.99°.	
Index ranges	-21<=h<=21, -14<=k<=14, -26<=l<=26	
Reflections collected	27446	
Independent reflections	4206 [R(int) = 0.0214]	
Completeness to theta = 25.99°	99.9 %	
Absorption correction	Multi-scan	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4206 / 0 / 229
Goodness-of-fit on F^2	1.024
Final R indices [I>2sigma(I)]	R1 = 0.0341
R indices (all data)	wR2 = 0.0968
Largest diff. peak and hole	0.300 and -0.279 e.Å^{-3}

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for mb42. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
P(1)	1866(1)	396(1)	195(1)	26(1)
C(1)	1358(1)	1655(1)	412(1)	28(1)
C(2)	719(1)	2155(1)	-41(1)	29(1)
C(3)	346(1)	3055(1)	264(1)	30(1)
C(4)	549(1)	3939(1)	1443(1)	34(1)
C(5)	1010(1)	3837(1)	2052(1)	38(1)
C(6)	1626(1)	2987(1)	2157(1)	36(1)
C(7)	1815(1)	2208(1)	1668(1)	31(1)
C(8)	1357(1)	2292(1)	1034(1)	27(1)
C(9)	724(1)	3167(1)	929(1)	29(1)
C(10)	-148(1)	4799(1)	1327(1)	44(1)
C(11)	2499(1)	1340(1)	1826(1)	38(1)
C(12)	2948(1)	526(1)	222(1)	37(1)
C(13)	1521(1)	36(1)	-658(1)	27(1)
C(14)	1817(1)	698(1)	-1163(1)	34(1)
C(15)	1555(1)	450(1)	-1824(1)	36(1)
C(16)	1010(1)	-465(1)	-1986(1)	35(1)
C(17)	718(1)	-1130(1)	-1488(1)	35(1)
C(18)	971(1)	-882(1)	-823(1)	31(1)
C(19)	1669(1)	-872(1)	705(1)	28(1)

C(20)	2182(1)	-1857(1)	770(1)	37(1)
C(21)	2004(1)	-2811(1)	1163(1)	44(1)
C(22)	1320(1)	-2787(1)	1493(1)	43(1)
C(23)	817(1)	-1807(1)	1439(1)	40(1)
C(24)	989(1)	-847(1)	1044(1)	33(1)

Table 9. Bond lengths [Å] and angles [°] for mb42.

P(1)-C(1)	1.7285(12)
P(1)-C(12)	1.8024(12)
P(1)-C(13)	1.8034(11)
P(1)-C(19)	1.8099(11)
C(1)-C(2)	1.4380(15)
C(1)-C(8)	1.4477(16)
C(2)-C(3)	1.3692(17)
C(3)-C(9)	1.4265(16)
C(4)-C(5)	1.3809(18)
C(4)-C(9)	1.4100(17)
C(4)-C(10)	1.5074(18)
C(5)-C(6)	1.3998(19)
C(6)-C(7)	1.3840(18)
C(7)-C(8)	1.4200(16)
C(7)-C(11)	1.5068(17)
C(8)-C(9)	1.4385(16)
C(13)-C(18)	1.3937(16)
C(13)-C(14)	1.3978(16)
C(14)-C(15)	1.3861(17)
C(15)-C(16)	1.3852(18)
C(16)-C(17)	1.3859(18)
C(17)-C(18)	1.3918(17)
C(19)-C(24)	1.3891(16)

C(19)-C(20)	1.3942(17)
C(20)-C(21)	1.3858(19)
C(21)-C(22)	1.384(2)
C(22)-C(23)	1.381(2)
C(23)-C(24)	1.3914(17)

C(1)-P(1)-C(12)	116.29(6)
C(1)-P(1)-C(13)	108.30(5)
C(12)-P(1)-C(13)	104.84(6)
C(1)-P(1)-C(19)	111.98(5)
C(12)-P(1)-C(19)	106.90(6)
C(13)-P(1)-C(19)	108.04(5)
C(2)-C(1)-C(8)	106.68(10)
C(2)-C(1)-P(1)	120.43(9)
C(8)-C(1)-P(1)	132.29(9)
C(3)-C(2)-C(1)	110.04(10)
C(2)-C(3)-C(9)	108.36(10)
C(5)-C(4)-C(9)	117.70(11)
C(5)-C(4)-C(10)	122.27(12)
C(9)-C(4)-C(10)	119.97(12)
C(4)-C(5)-C(6)	121.20(12)
C(7)-C(6)-C(5)	122.90(11)
C(6)-C(7)-C(8)	117.54(11)
C(6)-C(7)-C(11)	118.98(11)
C(8)-C(7)-C(11)	123.46(11)

C(7)-C(8)-C(9)	119.21(11)
C(7)-C(8)-C(1)	134.37(11)
C(9)-C(8)-C(1)	106.42(10)
C(4)-C(9)-C(3)	130.06(11)
C(4)-C(9)-C(8)	121.43(11)
C(3)-C(9)-C(8)	108.49(10)
C(18)-C(13)-C(14)	119.63(11)
C(18)-C(13)-P(1)	121.67(9)
C(14)-C(13)-P(1)	118.70(9)
C(15)-C(14)-C(13)	119.97(11)
C(16)-C(15)-C(14)	120.28(11)
C(15)-C(16)-C(17)	120.05(12)
C(16)-C(17)-C(18)	120.19(11)
C(17)-C(18)-C(13)	119.88(11)
C(24)-C(19)-C(20)	119.61(11)
C(24)-C(19)-P(1)	118.36(9)
C(20)-C(19)-P(1)	122.03(9)
C(21)-C(20)-C(19)	119.96(12)
C(22)-C(21)-C(20)	120.17(13)
C(23)-C(22)-C(21)	120.19(12)
C(22)-C(23)-C(24)	120.02(13)
C(19)-C(24)-C(23)	120.04(12)

Table 10. Anisotropic displacement parameters ($Å^2 x 10^3$) for mb42. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1)	23(1)	28(1)	27(1)	4(1)	1(1)	-4(1)
C(1)	26(1)	27(1)	28(1)	4(1)	-1(1)	-4(1)
C(2)	25(1)	33(1)	29(1)	6(1)	-2(1)	-6(1)
C(3)	23(1)	33(1)	34(1)	10(1)	1(1)	-3(1)
C(4)	34(1)	33(1)	37(1)	7(1)	12(1)	-3(1)
C(5)	47(1)	38(1)	32(1)	0(1)	12(1)	-4(1)
C(6)	42(1)	41(1)	26(1)	4(1)	2(1)	-7(1)
C(7)	31(1)	32(1)	29(1)	6(1)	0(1)	-6(1)
C(8)	26(1)	27(1)	29(1)	6(1)	2(1)	-6(1)
C(9)	25(1)	29(1)	32(1)	8(1)	6(1)	-5(1)
C(10)	46(1)	42(1)	47(1)	8(1)	16(1)	7(1)
C(11)	39(1)	40(1)	33(1)	4(1)	-8(1)	0(1)
C(12)	23(1)	46(1)	41(1)	4(1)	2(1)	-5(1)
C(13)	25(1)	29(1)	28(1)	2(1)	3(1)	-2(1)
C(14)	35(1)	32(1)	34(1)	4(1)	4(1)	-8(1)
C(15)	39(1)	41(1)	30(1)	7(1)	7(1)	-3(1)
C(16)	34(1)	42(1)	30(1)	-3(1)	3(1)	1(1)
C(17)	31(1)	37(1)	38(1)	-5(1)	4(1)	-7(1)
C(18)	28(1)	33(1)	33(1)	2(1)	7(1)	-5(1)
C(19)	29(1)	28(1)	25(1)	3(1)	-1(1)	-3(1)

C(20)	43(1)	35(1)	34(1)	3(1)	5(1)	6(1)
C(21)	59(1)	31(1)	42(1)	7(1)	-2(1)	7(1)
C(22)	52(1)	37(1)	36(1)	14(1)	-9(1)	-12(1)
C(23)	34(1)	50(1)	36(1)	13(1)	0(1)	-11(1)
C(24)	27(1)	36(1)	35(1)	7(1)	1(1)	-3(1)

Table 11. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for mb42.

	x	y	z	U(eq)
H(2)	577	1899	-486	35
H(3)	-90	3526	66	36
H(5)	908	4353	2405	46
H(6)	1929	2944	2584	44
H(10A)	-91	5276	930	66
H(10B)	-150	5326	1713	66
H(10C)	-657	4354	1263	66
H(11A)	2330	548	1662	57
H(11B)	2645	1306	2309	57
H(11C)	2967	1601	1611	57
H(12A)	3166	-199	39	56
H(12B)	3186	631	684	56
H(12C)	3080	1214	-43	56
H(14)	2197	1317	-1054	40
H(15)	1749	909	-2166	44
H(16)	837	-638	-2440	42
H(17)	345	-1756	-1600	42
H(18)	769	-1336	-482	37
H(20)	2653	-1874	545	45

H(21)	2352	-3483	1206	53
H(22)	1196	-3447	1757	51
H(23)	352	-1788	1672	48
H(24)	642	-173	1006	40

Table 12. Torsion angles [°] for mb42.

C(12)-P(1)-C(1)-C(2)	123.68(9)
C(13)-P(1)-C(1)-C(2)	6.01(10)
C(19)-P(1)-C(1)-C(2)	-113.02(9)
C(12)-P(1)-C(1)-C(8)	-66.50(12)
C(13)-P(1)-C(1)-C(8)	175.83(10)
C(19)-P(1)-C(1)-C(8)	56.80(12)
C(8)-C(1)-C(2)-C(3)	0.89(12)
P(1)-C(1)-C(2)-C(3)	173.04(8)
C(1)-C(2)-C(3)-C(9)	-0.54(13)
C(9)-C(4)-C(5)-C(6)	-0.64(18)
C(10)-C(4)-C(5)-C(6)	176.48(12)
C(4)-C(5)-C(6)-C(7)	0.25(19)
C(5)-C(6)-C(7)-C(8)	0.23(18)
C(5)-C(6)-C(7)-C(11)	178.83(12)
C(6)-C(7)-C(8)-C(9)	-0.28(15)
C(11)-C(7)-C(8)-C(9)	-178.82(10)
C(6)-C(7)-C(8)-C(1)	-179.28(12)
C(11)-C(7)-C(8)-C(1)	2.2(2)
C(2)-C(1)-C(8)-C(7)	178.21(12)
P(1)-C(1)-C(8)-C(7)	7.4(2)
C(2)-C(1)-C(8)-C(9)	-0.87(11)
P(1)-C(1)-C(8)-C(9)	-171.72(9)
C(5)-C(4)-C(9)-C(3)	178.79(11)

C(10)-C(4)-C(9)-C(3)	1.61(18)
C(5)-C(4)-C(9)-C(8)	0.57(16)
C(10)-C(4)-C(9)-C(8)	-176.61(11)
C(2)-C(3)-C(9)-C(4)	-178.43(11)
C(2)-C(3)-C(9)-C(8)	-0.03(12)
C(7)-C(8)-C(9)-C(4)	-0.12(16)
C(1)-C(8)-C(9)-C(4)	179.14(10)
C(7)-C(8)-C(9)-C(3)	-178.68(10)
C(1)-C(8)-C(9)-C(3)	0.57(12)
C(1)-P(1)-C(13)-C(18)	-102.33(10)
C(12)-P(1)-C(13)-C(18)	132.90(10)
C(19)-P(1)-C(13)-C(18)	19.16(11)
C(1)-P(1)-C(13)-C(14)	77.67(11)
C(12)-P(1)-C(13)-C(14)	-47.10(11)
C(19)-P(1)-C(13)-C(14)	-160.84(10)
C(18)-C(13)-C(14)-C(15)	0.77(19)
P(1)-C(13)-C(14)-C(15)	-179.23(10)
C(13)-C(14)-C(15)-C(16)	-1.0(2)
C(14)-C(15)-C(16)-C(17)	0.7(2)
C(15)-C(16)-C(17)-C(18)	-0.06(19)
C(16)-C(17)-C(18)-C(13)	-0.19(19)
C(14)-C(13)-C(18)-C(17)	-0.16(18)
P(1)-C(13)-C(18)-C(17)	179.84(9)
C(1)-P(1)-C(19)-C(24)	19.48(11)
C(12)-P(1)-C(19)-C(24)	147.94(10)

C(13)-P(1)-C(19)-C(24)	-99.70(10)
C(1)-P(1)-C(19)-C(20)	-159.84(10)
C(12)-P(1)-C(19)-C(20)	-31.39(12)
C(13)-P(1)-C(19)-C(20)	80.97(11)
C(24)-C(19)-C(20)-C(21)	1.06(19)
P(1)-C(19)-C(20)-C(21)	-179.62(10)
C(19)-C(20)-C(21)-C(22)	-0.2(2)
C(20)-C(21)-C(22)-C(23)	-0.8(2)
C(21)-C(22)-C(23)-C(24)	1.0(2)
C(20)-C(19)-C(24)-C(23)	-0.86(18)
P(1)-C(19)-C(24)-C(23)	179.80(9)
C(22)-C(23)-C(24)-C(19)	-0.17(19)

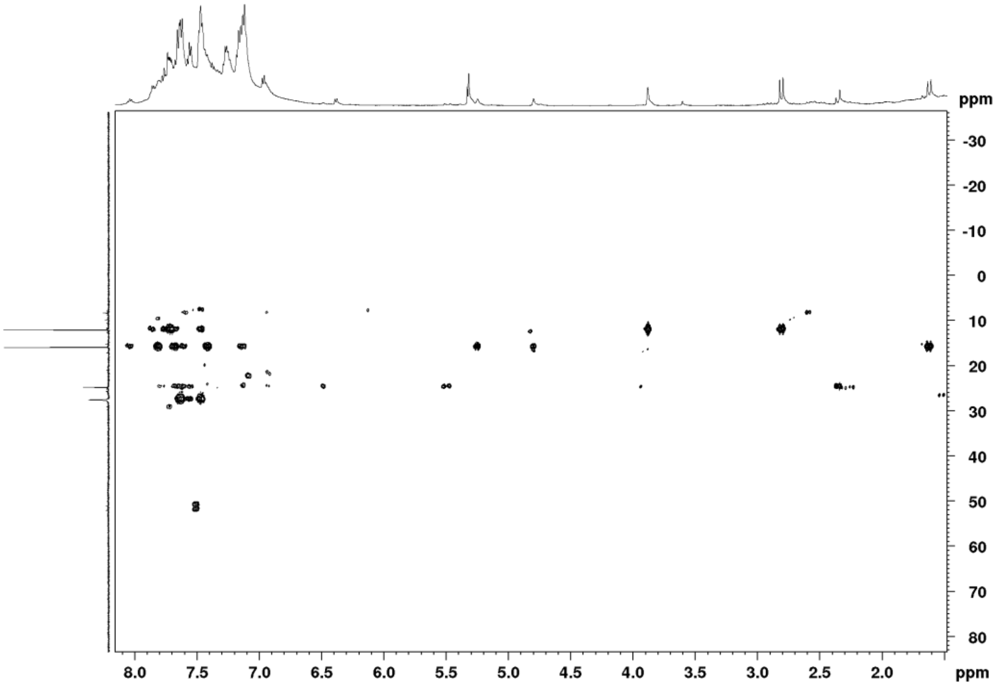


Figure 3. ^{1}H-^{31}P HMBC of $[(\eta^5$-**I**$)Rh(PPh_3)_2][BF_4]$ in CD_2Cl_2.

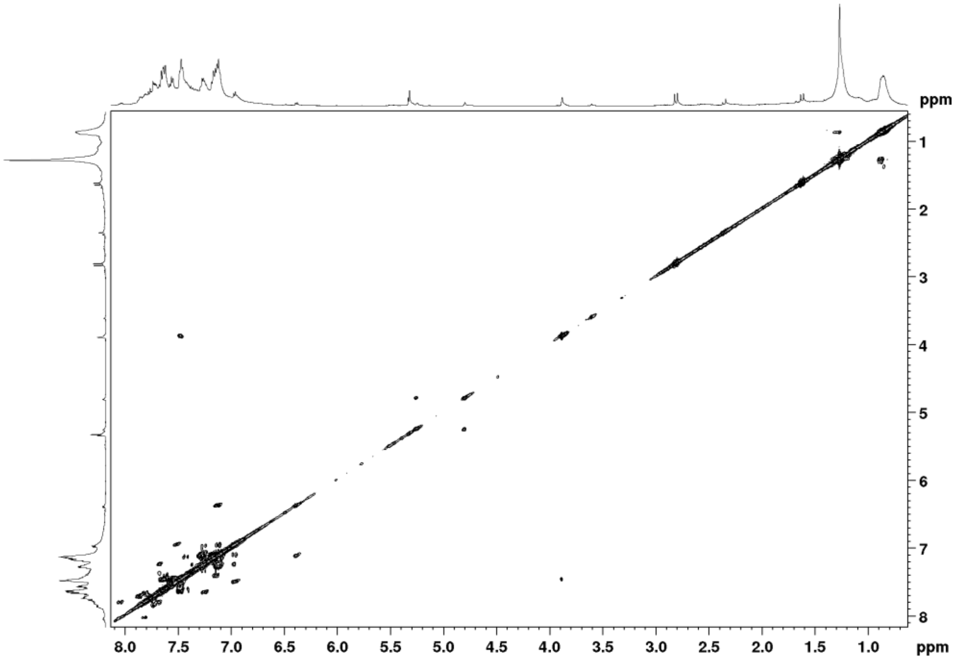


Figure 4. COSY spectrum of [(η^5-**I)**Rh(PPh$_3$)$_2$][BF$_4$] in CD$_2$Cl$_2$.

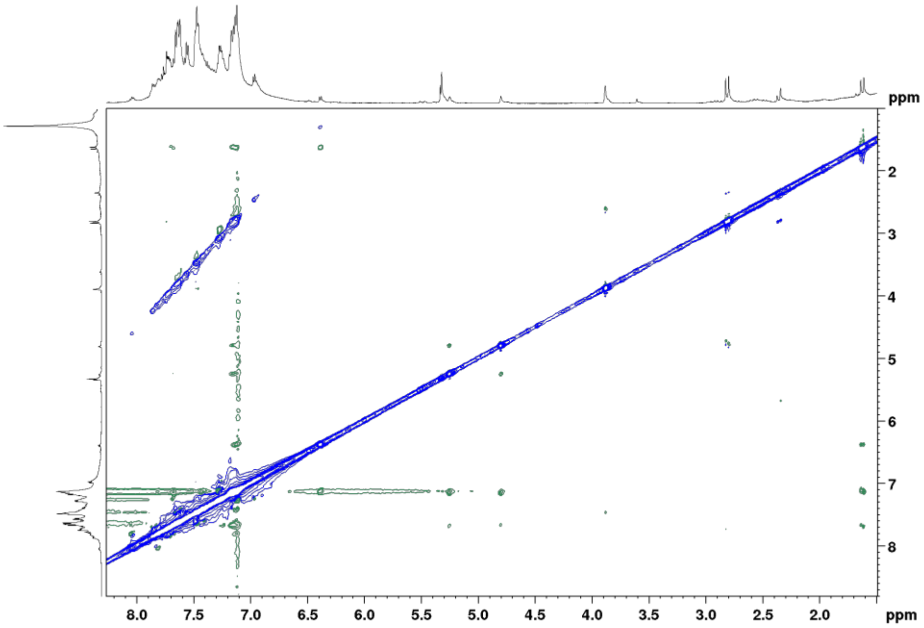


Figure 5. NOESY spectrum of [(η^5-**I**)Rh(PPh$_3$)$_2$][BF$_4$] in CD$_2$Cl$_2$.

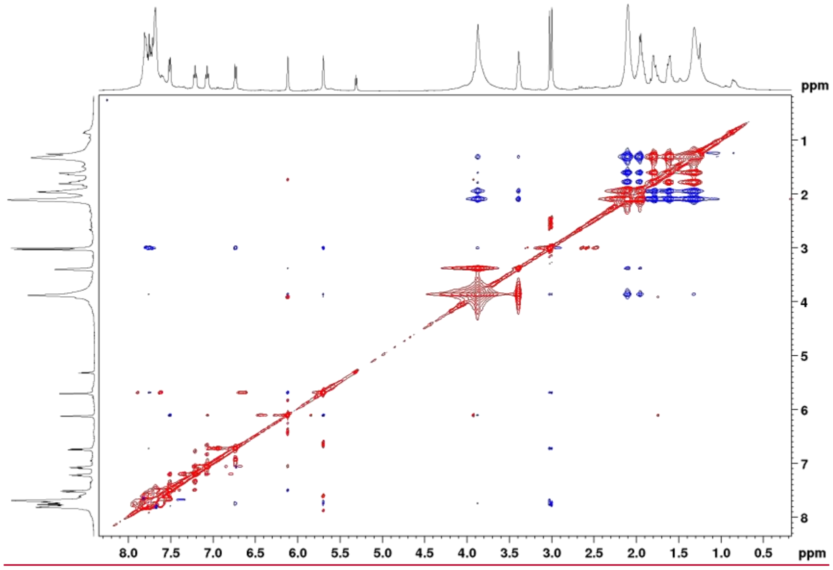


Figure 6. NOESY spectrum of the product of reaction between [Ir(COD)Cl]$_2$ and (**I**)

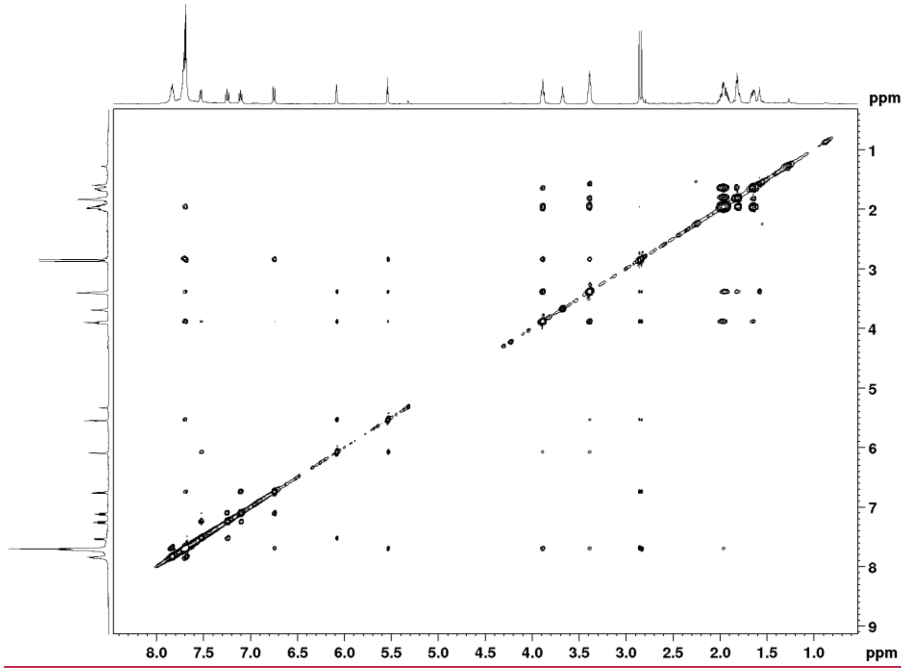


Figure 7. NOESY spectrum of [Ir(η^4-C_8H_{12})(η^5-1-$C_9H_6PMePh_2$)]BF_4.

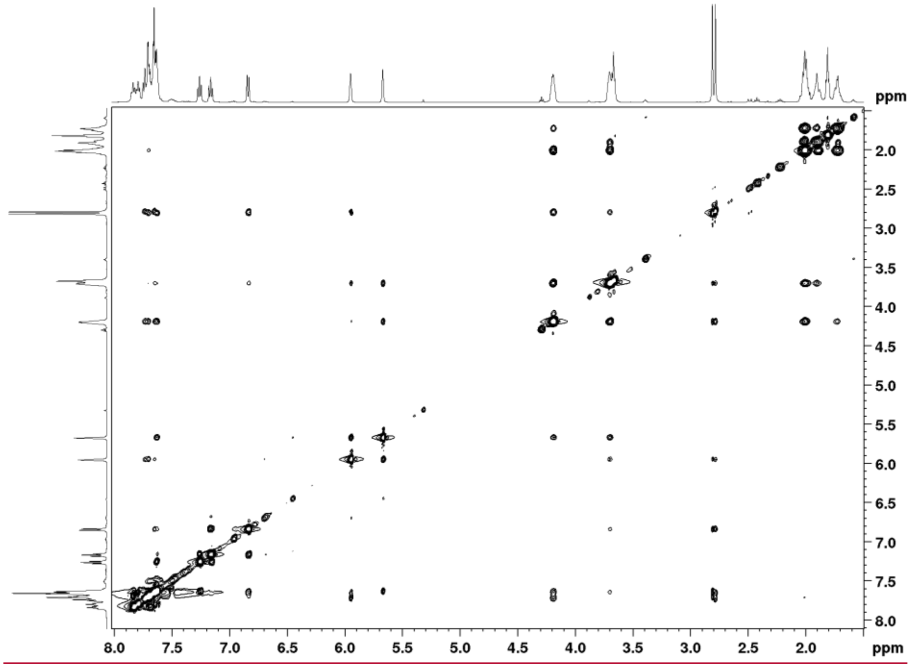


Figure 8. NOESY spectrum of [Rh(η^4-C_8H_{12})(η^5-1-$C_9H_6PMePh_2$)]BF_4.

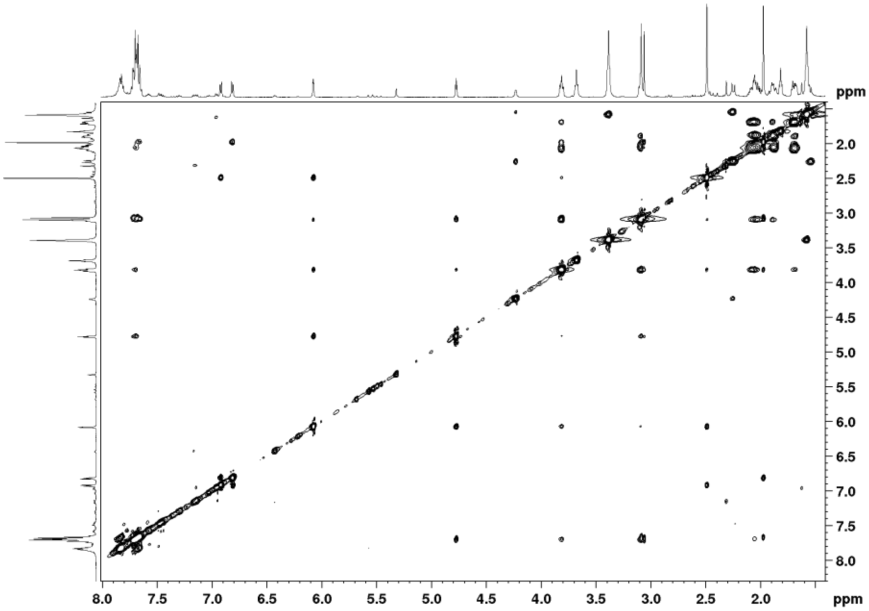


Figure 9. NOESY spectrum of [Ir(η^4-C_8H_{12})(η^5-4,7-dimethyl-1-$C_9H_4PMePh_2$)]BF_4.

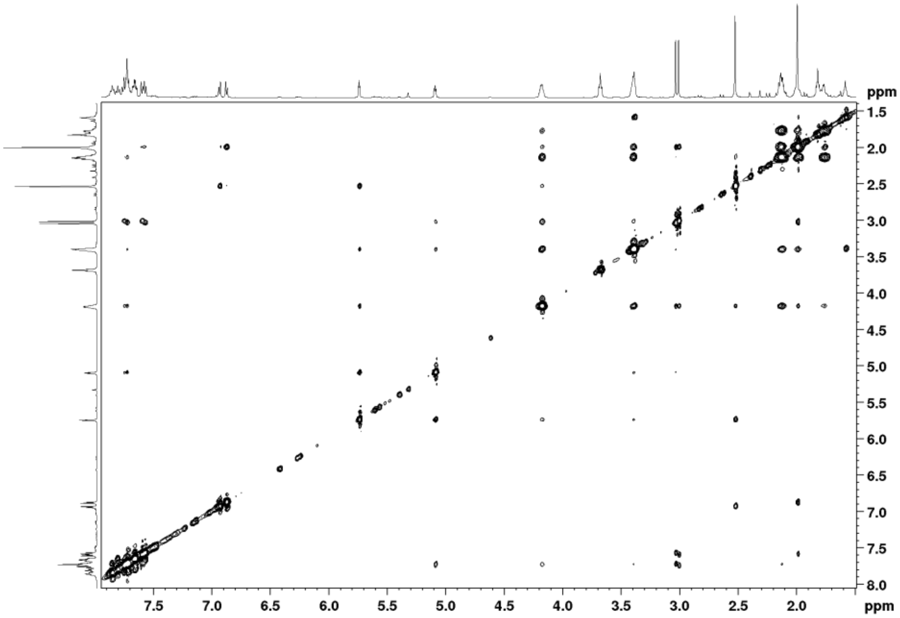


Figure 102. NOESY spectrum of [Rh(η^4-C_8H_{12})(η^5-4,7-dimethyl-1-$C_9H_4PMePh_2$)]BF_4.