# $[(^{i}Pr_3P)_6Rh_6H_{12}]^{2+}$: A High-Hydride Content Octahedron that Bridges the Gap Between Late and Early-Transition Metal Clusters.

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**Supporting Information**

Experimental Details for **1a** and **1b** (including mass spec data)
Ball and stick diagram of the gross structural features of $[(^{i}Pr)_3PH][CB_{11}Me_{11}H]$.
Experimental details for the in situ observation of dihydrogen species
X-ray data collection, refinement details and bond lengths and angles for **1a** and **1b**
Details of HYDEX analysis

## Experimental

**General.** All manipulations were performed under an inert atmosphere of argon, using standard Schlenk-line and glove box techniques. Glassware was dried in an oven at 130°C overnight and flamed with a blowtorch, under vacuum, three times before use. $CH_2Cl_2$, $C_6H_5F$ and pentane were distilled from $CaH_2$ and pentane from sodium. $CD_2Cl_2$ was distilled under vacuum from $CaH_2$. Microanalyses were by Mr. Alan Carver (University of Bath Microanalytical Service). Ag[*closo*-1-H-$CB_{11}Me_{11}$] (1) was prepared using literature procedures. [($^iPr_3P)_2$Rh(nbd)]($CB_{11}Me_{11}H$) or [($^iPr_3P)_2$Rh(nbd)][B($Ar_F)_4$] were prepared by an adaptation of the published route using Ag[*closo*-1-H-$CB_{11}Me_{11}$], K[B($Ar_F)_4$] (2) and $^iPr_3P$. **(3)**

(1) Prepared by an adaptation of the method given in King, B. T.; Janousek, Z.; Gruner, B.; Trammell, M.; Noll, B. C.; Michl, J. *J. Am. Chem. Soc.* **1996**, *118*, 3313]; J. Michl, personal communication, 2002
(2) Buschmann, W. E.; Miller, J. S. *Inorg. Synth.* **2002**, 33, 85
(3) Rifat, A.; Laing, V. E.; Kocoik-Köhn, G.; Mahon, M. F.; Ruggiero, G. D. and Weller, A. S. *J. Organomet. Chem*. **2003**, 680, 127

**NMR spectroscopy.** $^1H$, $^{11}B$ and $^{31}P$ NMR spectra were recorded on a Bruker Advance 300 MHz or Varian 400 MHz (Bath) FT-NMR spectrometers. Residual protio solvent was used as reference for $^1H$ NMR spectra ($CD_2Cl_2$: □= 5.33). $^{11}B$ and $^{31}P$ NMR spectra were referenced against $BF_3.OEt_2$ (external) and 85% $H_3PO_4$ (external) respectively. Coupling constants are quoted in Hertz.

**[$Rh_6(^iPr_3P)_6H_{12}$][$CB_{11}Me_{11}H]_2$ 1a**

A Young's tube was charged with (0.030 g, $3.7x10^{-5}$ mol) [($^iPr_3P)_2$Rh(nbd)($CB_{11}Me_{11}H$)] and dissolved in $C_6H_5F$ (5ml). This was fully degassed by three freeze-pump-thaw cycles then backfilled immediately with $H_2$ at 4 atmospheres pressure (77K warming to room temperature) resulting in a colour change from orange to yellow. Stirring for four days at 40°C yielded a further gradual darkening of the solution to dark brown. NMR analysis of the reaction mixture by $^{31}P\{^1H\}$ NMR spectroscopy showed three products formed in an approximate 1:1:1 ratio based on phosphorus. Two are identified as **1a** and [$HP^iPr_3$][$CB_{11}Me_{11}H$]. The third component shows no hydride signal in the $^1H$ NMR spectrum and a single doublet in the $^{31}P\{^1H\}$ NMR spectrum [□ 60.4 ppm, *J*(RhP) 110 Hz] under slight pressure of $H_2$. Attempts to identify this third component have so far failed, but studies are ongoing. The solvent was removed *in-vacuo* and re-dissolving the residue in $CH_2Cl_2$ and layering with pentane yielded black [$Rh_6(^iPr_3P)_6H_{12}$][$CB_{11}Me_{11}H]_2$ **1a** and colourless crystals of co-product [$HP^iPr_3$][$CB_{11}Me_{11}H$]. Yield [$Rh_6(^iPr_3P)_6H_{12}$][$CB_{11}Me_{11}H]_2$ (0.016 g, 20% based on Rh). **1b** was prepared in an identical manner starting from [($^iPr_3P)_2$Rh(nbd)][B($Ar_F)_4$].

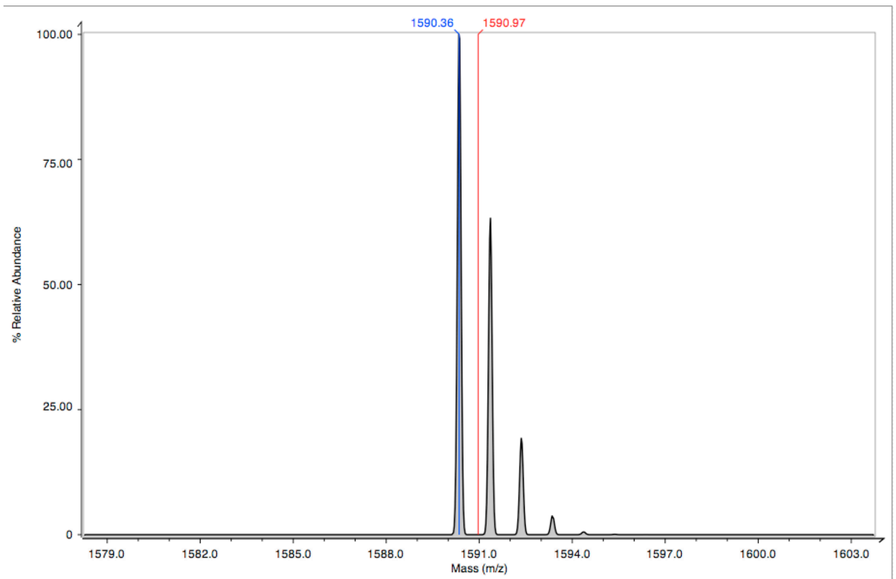
***NMR Data (all in $CD_2Cl_2$ at 298K).***

**[$Rh_6(^iPr_3P)_6H_{12}$][$CB_{11}Me_{11}H]_2$ 1a: $^1H$ (400MHz, 5 sec delay between acquisitions, 1000 scans)** 2.31 (br m 18H), 1.28 (m, 108H), 1.16 (s, 2H $cage_{C-H}$), -0.17 (s, 30H), -0.43 (s, 30H), -0.54 (s, 6H), -21.44 (br s, 12H, relative to $^iPr_3$ groups, integration consistent over 4 independently synthesized samples). **$^{11}B$ (96 MHz)** 0.17 (s, 1B), -7.89 (s, 5B), -11.14 (s, 5B). **$^{31}P\{^1H\}$ (121MHz)** 110.5 (d, 140Hz).

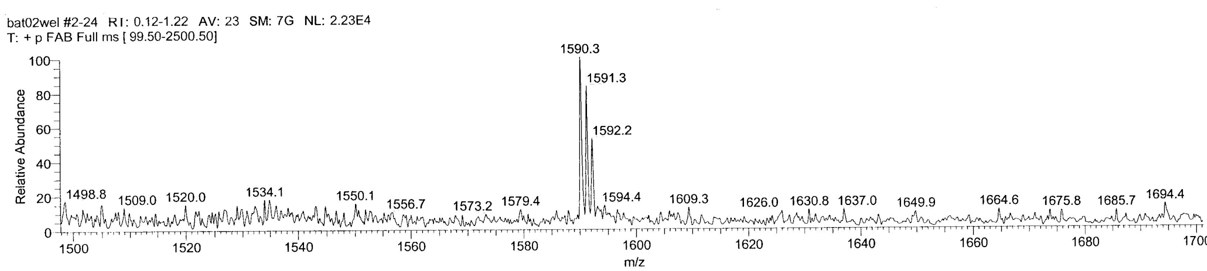
$C_{78}H_{206}B_{22}P_6Rh_6$ requires %C 42.9 %H 9.43, found %C 43.6 %H 8.86

**$[Rh_6(^iPr_3P)_6H_{12}][BAr_F]_2$ lb: $^1$H (400MHz, 400MHz, 5 sec delay between acquisitions, 1000 scans)** 7.63 (s, 16H, ortho arene) 7.47 (s, 8H, para arene), 2.35 (m, 18H, $^I$Pr bridgehead) 1.26 (m, 108H, $CH_3$), -21.40 (br, s, 12H, relative to $^iPr_3$ groups). **$^{11}$B** (128MHz) –5.9 (s) **$^{31}$P{$^1$H}:** (121MHz) 110.5 (d, 140Hz).
$C_{82}H_{78}B_2F_{48}P_6Rh_6$ requires %C 35.2% %H 2.81%, found %C 34.9 %H 3.29

Mass Spec (FAB+, NOBA matrix): 1590.3 $[Rh_6(^iPr_3P)_6H_{12}]^{+\cdot}$ showing the correct isotope pattern for $Rh_6C_{54}H_{138}P_6$ (see below)




*Calculated for $[RhH_2P(C_3H_7)_3]_6^+$*




*Observed for **1a***

NMR data for the $[^iPr_3PH]^+$ cation are consistent with that reported by Torres *et al.* (*J. Am. Chem. Soc.* **1999**, *121*, 10632-10633) for $[^iPr_3PH]BF_4$. The gross structure was confirmed by a single crystal X-ray diffraction study, but due the poor quality of crystals the data refinement was poor ($wR_1$ = 10.6%) and thus not reported in full here. Figure S1 shows a ball and stick plot for $[HP^iPr_3][CB_{11}Me_{11}H]$.

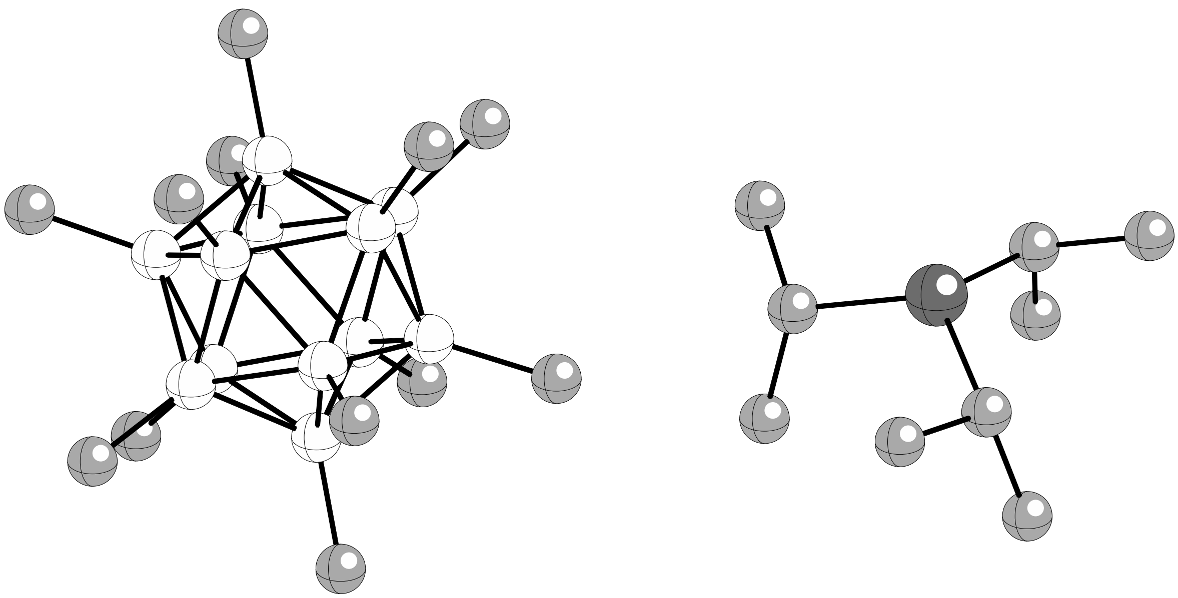


**Figure S1:** Ball and stick diagram of the solid-state structure of $[(^iPr)_3PH][CB_{11}Me_{11}H]$. The hydrogen atoms are not shown. The cage anion is disordered and the cage carbon atom was not located.

**$[(^iPr_3P)_2Rh(H_2)_xH_2][Y]$: (x = 1 or 2 and Y = 1-H-$CB_{11}(CH_3)_{11}$ or $B(Ar_F)_4$)**
A typical experiment consisted of charging a Young's NMR tube with 0.015g of $[(^iPr_3P)_2RhNBD][Y]$, addition of 0.3ml of $CD_2Cl_2$ generated an orange solution. The sample was freeze/pumped/thawed three times before being backfilled with $H_2$ at 77K (~ 4 atm $H_2$ at 298 K). On thawing, the solution rapidly changes colour from orange to pale yellow. Attempts to isolate solid material repeatedly failed. $^1H$ NMR (400MHz, $CD_2Cl_2$, 298K for Y = $B(Ar_F)_4$) □7.73 (s, 8H, ortho arene), 7.57 (s, 4H, para arene), 2.31 (m, 6H, $^iPr$ bridgehead) , 1.25 [dd 36H, $CH_3$, 7, 15 Hz], -8.62 (v. br 3.6H relative to $^iPr$ protons, $(H_2)$/H, $T_1$ 106 ms). $^{31}P\{^1H\}$ NMR (162MHz, $CD_2Cl_2$, 298K) □60.4 [d, *J*(RhP) 107Hz]. $^{11}B$ NMR (128MHz, $CD_2Cl_2$, **298**K) □–5.9 (s).

**$[(^iPr_3P)_2Rh(H_2)H_2][BAr_F]$:**
$^1H$ NMR (400MHz, $CD_2Cl_2$, **190K**) □7.61 (s, 8H, ortho arene), 7.46 (s, 4H, para arene), 2.08, (m, 6H, $^IPr$ bridgehead), 1.02 (m, 36H, $CH_3$), -0.28 (br 2H (□$^2$-$H_2$), $T_1$ 94 ms), -12.66 (s br, 1H, $T_1$ 238 ms), -22.42 (s br, 1H, $T_1$ 234). $^{31}P\{^1H\}$ (162MHz, $CD_2Cl_2$, 190K) 62.1 [d, *J*(RhP) 100Hz]

**$[(^iPr_3P)_2Rh(H_2)_2H_2][B(Ar_F)_4]$:**
$^1H$ NMR (400MHz, $CD_2Cl_2$, **190K)** □7.61 (s, 8H, ortho arene), 7.46 (s, 4H, para arene), 2.08, (m, 6H, $^IPr$ bridgehead), 1.02 (m, 36H, $CH_3$), -2.06 (v. br 4H (□$^2$-$H_2$), $T_1$ 43 ms), -14.23 (s br, 2H, $T_1$ 360 ms), $^{31}P\{^1H\}$ (162MHz, $CD_2Cl_2$, **190K**) □68.4 [d, *J*(RhP) 92Hz].

At 190 K the ratio $[(^iPr_3P)_2Rh(H_2)H_2][BAr_F]$:$[(^iPr_3P)_2Rh(H_2)_2H_2][BAr_F]$ is 4:1. Assignment of the hydrides in each complex comes from $T_1$ measurements* and comparison of chemical shifts with the previously reported complexes $[(R_3P)_2Ir(H_2)_xH_2][B(Ar_F)_4]$ (x = 1, 2).[(4)]

* $T_1$ measurements were made at 190K using the software provided with a Bruker Advance 400 spectrometer using the standard inversion-recovery-delay method (180°□□90°) method. The calculation of relaxation times was made using a nonlinear three-parameter fitting routine. In each experiment a waiting period of 5 times longer than the expected relaxation time and 11 variable delays were used.


(4) (a) Cooper, A. C.; Caulton, K. G. *Inorg Chem.* **1998**, 37, 5938. (b) Cooper, A. C.; Eisenstein, O.; Caulton, K. G. *New J. Chem.* **1998**, 307. (c) Crabtree, R. H.; Lavin, M. *Chem. Commun.* **1985**, 1661

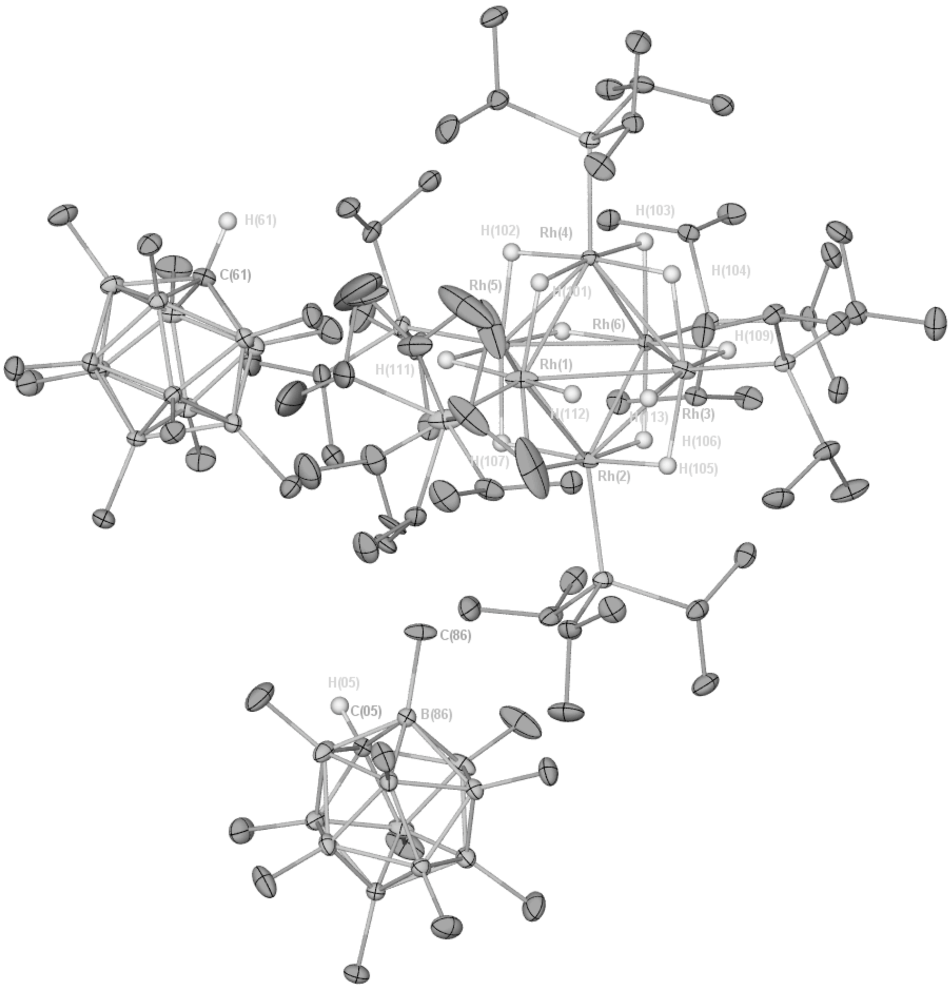



**Figure S2**: $[Rh_6(^{i}Pr_3P)_6H_{12}][CB_{11}Me_{11}H]_2$, **1a**, showing atom labelling scheme and the disorder associated with P1 and H12/H13 for 1a. Thermal ellipsoids are shown at the 50 % probability level. The asymmetric unit in this structure consists of 1 cationic cluster, 2 anionic carboranes, and 3 area of diffuse solvent. Equal disorder of the isopropyl groups associated with the phosphine P1 in the cluster was readily modelled between the atoms with labels without suffixes and those labelled with suffix 'A'. The C-H moiety in one of the anions was over 2 sites. In particular C05, B86 and C86 (and pendant hydrogens) share occupancy with C06, B85 and C85 respectively. The solvent regions were diffuse and ultimately 'mopped-up' using partial carbons with site occupancy (C901-909). At this stage of the convergence the bridging hydride atoms at the cluster core were located, without prejudice, in a difference Fourier electron density map using data up to a 2θ maximum of 60°. All hydrides were treated as having full occupancy, with the exceptions of H112 and H113. Bridging distances from each hydride to the core pair of bonded rhodium atoms were constrained to be similar during the final least squares refinement. The metal-hydride distances involving H112 and H113 were also constrained to be similar to each other. This course of action was chosen largely to aid the refinement giving the shortcomings of X-ray data in the determination of these small species. Assignment of the site occupancy concerning H112 and H113 was made on chemical grounds as opposed to anything structural. That is to say, making H112 and H113 both full occupancy would give the cluster 13 hydrides and thus be paramagnetic, contrary to the diamagnetism observe in the NMR spectra. Treatment of these hydrides as having full occupancies did not have a decisive effect of the refinement.

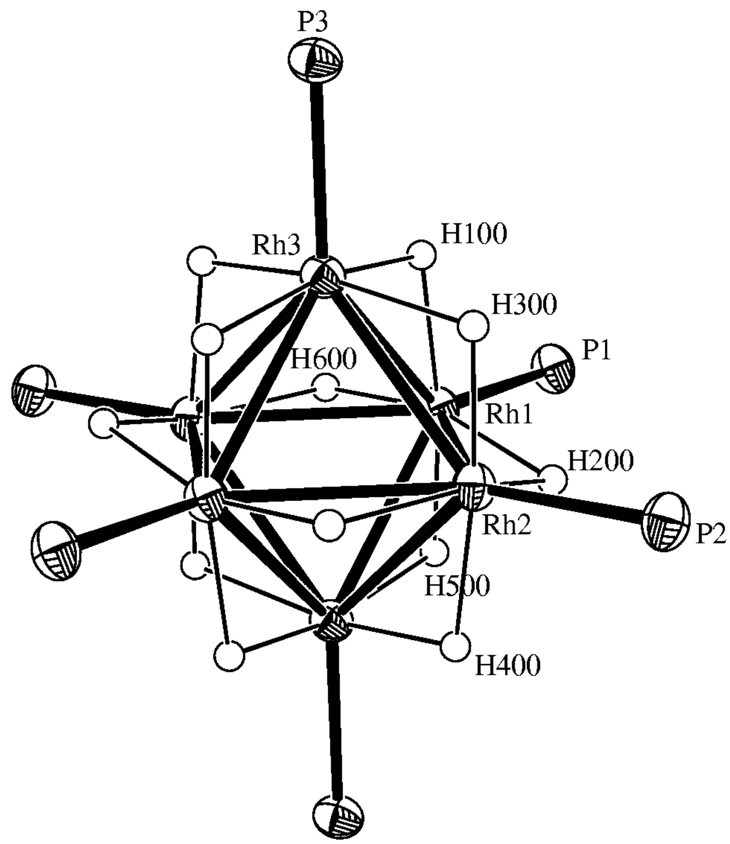



**Figure S3:** $[Rh_6(^iPr_3P)_6H_{12}][B(Ar_F)_4]_2$, **1b**, showing atom labelling scheme. Only dicationic portion shown, with $[B(Ar_F)_4]$ anions and one molecule of solvent ($C_6H_5F$) not shown. $^IPr$ groups on the cluster phosphine ligands have also been omitted for clarity. The molecule is centrosymetric, with half of the octahedron (and one anion) generated crystallographically. Thermal ellipsoids are shown at the 50 % probability level. The $[Rh_6]^{2+}$ dication sits on a center of inversion. All six bridging hydrogens in the asymmetric unit could be located in the Fourier map due to the good quality of the high angle (60°) data. They have a site occupation factor of 1 and could be refined isotropically. In the unique anion four of the eight attached $CF_3$ groups show rotational disorder. The complex crystallizes with a half solvent molecule of fluorobenzene in the asymmetrical unit. The Fluorine in this solvent molecule is disordered in the ratio of 1:1 and due to the molecule sitting on a center of inversion it occupies four different positions.

**Table S1.** Crystal data and structure refinement for **1a**.

| | |
|---|---|
| Identification code | Compound 1 |
| Empirical formula | C82.50 H206 B22 P6 Rh6 |
| Formula weight | 2239.57 |
| Temperature | 150(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| Space group | $P2_1/n$ |
| Unit cell dimensions | a = 21.50300(10)Å $\alpha$ = 90° |
| | b = 25.48900(10)Å $\beta$ = 103.30° |
| | c = 21.82500(10)Å $\gamma$= 90° |
| Volume | 11641.32(9) $Å^3$ |
| Z | 4 |
| Density (calculated) | 1.278 $Mg/m^3$ |
| Absorption coefficient | 0.949 $mm^{-1}$ |
| F(000) | 4684 |
| Crystal size | 0.43 x 0.15 x 0.13 mm |
| Theta range for data collection | 3.60 to 30.08° |
| Index ranges | -30<=h<=29; -35<=k<=35; -30<=l<=30 |
| Reflections collected | 205546 |
| Independent reflections | 33956 [R(int) = 0.0662] |
| Reflections observed (>2$\sigma$) | 23910 |
| Data Completeness | 0.993 |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.92 and 0.86 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 33956 / 12 / 1251 |
| Goodness-of-fit on $F^2$ | 1.034 |
| Final R indices [I>2$\sigma$(I)] | $R_1$ = 0.0480 $wR_2$ = 0.1007 |
| R indices (all data) | $R_1$ = 0.0832 $wR_2$ = 0.1163 |
| Largest diff. peak and hole | 1.629 and -2.201 $eÅ^{-3}$ |

**Table S2.** Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$ x $10^3$) for 1.U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| Atom | x | y | z | U(eq) |
|---|---|---|---|---|
| | | | | |
| Rh(1) | 5405(1) | 3006(1) | 3530(1) | 40(1) |
| Rh(2) | 6631(1) | 3272(1) | 3420(1) | 28(1) |
| Rh(3) | 6173(1) | 2298(1) | 2844(1) | 29(1) |
| Rh(4) | 5966(1) | 2030(1) | 4020(1) | 22(1) |
| Rh(5) | 6435(1) | 2952(1) | 4563(1) | 24(1) |
| Rh(6) | 7144(1) | 2360(1) | 3932(1) | 21(1) |
| P(1) | 4534(1) | 3514(1) | 3322(1) | 51(1) |
| P(2) | 6998(1) | 3932(1) | 2911(1) | 28(1) |
| P(3) | 5961(1) | 1810(1) | 1962(1) | 26(1) |
| P(4) | 5513(1) | 1313(1) | 4345(1) | 26(1) |
| P(5) | 6606(1) | 3286(1) | 5541(1) | 28(1) |
| P(6) | 8130(1) | 2022(1) | 4205(1) | 23(1) |
| C(1) | 4282(4) | 3720(3) | 2447(4) | 47(2) |
| C(2) | 4413(4) | 3335(4) | 1940(4) | 36(2) |
| C(3) | 3570(5) | 3908(4) | 2281(5) | 63(3) |
| C(4) | 3872(4) | 3172(4) | 3502(6) | 68(3) |
| C(05) | 3638(4) | 5735(3) | 1634(4) | 27(2) |
| C(5) | 3638(12) | 2694(11) | 3131(16) | 115(11) |
| C(06) | 3248(5) | 5329(4) | 1053(5) | 31(3) |
| C(6) | 3948(6) | 3125(7) | 4260(8) | 70(4) |
| C(7) | 4700(4) | 4071(4) | 3774(5) | 54(2) |
| C(8) | 5184(14) | 4401(6) | 3616(15) | 48(5) |
| C(9) | 4096(6) | 4385(5) | 3820(6) | 79(4) |
| C(10) | 6323(2) | 4327(2) | 2451(2) | 38(1) |
| C(11) | 6506(3) | 4839(2) | 2168(3) | 67(2) |
| C(12) | 5891(2) | 3997(2) | 1944(2) | 48(1) |
| C(13) | 7474(2) | 3734(2) | 2339(2) | 35(1) |
| C(14) | 7855(2) | 3228(2) | 2514(2) | 40(1) |
| C(15) | 7915(2) | 4163(2) | 2179(2) | 51(1) |
| C(16) | 7503(2) | 4403(2) | 3455(2) | 38(1) |
| C(17) | 8089(2) | 4132(2) | 3861(2) | 48(1) |
| C(18) | 7115(2) | 4684(2) | 3864(2) | 49(1) |
| C(19) | 6071(2) | 2153(2) | 1252(2) | 38(1) |
| C(20) | 6766(2) | 2330(2) | 1325(2) | 66(2) |
| C(21) | 5618(2) | 2615(2) | 1070(2) | 54(1) |
| C(22) | 5145(2) | 1523(2) | 1760(2) | 32(1) |
| C(23) | 4964(2) | 1290(2) | 1095(2) | 36(1) |
| C(24) | 4616(2) | 1889(2) | 1861(2) | 53(1) |
| C(25) | 6521(2) | 1243(2) | 2050(2) | 36(1) |
| C(26) | 6355(2) | 833(2) | 2496(2) | 47(1) |
| C(27) | 6613(2) | 973(2) | 1444(2) | 56(1) |
| C(28) | 4917(2) | 990(1) | 3706(2) | 34(1) |
| C(29) | 4640(2) | 477(2) | 3898(2) | 48(1) |
| C(30) | 4388(2) | 1357(2) | 3373(2) | 52(1) |
| C(31) | 5171(2) | 1448(2) | 5035(2) | 35(1) |
| C(32) | 5033(2) | 970(2) | 5412(2) | 50(1) |

| | | | | |
|---|---|---|---|---|
| C(33) | 4584(2) | 1804(2) | 4875(2) | 55(1) |
| C(34) | 6113(2) | 797(1) | 4635(2) | 35(1) |
| C(35) | 6624(2) | 994(2) | 5199(2) | 44(1) |
| C(36) | 6424(2) | 593(2) | 4117(2) | 43(1) |
| C(37) | 7468(2) | 3384(2) | 5888(2) | 38(1) |
| C(38) | 7655(2) | 3451(2) | 6610(2) | 62(1) |
| C(39) | 7755(2) | 3823(2) | 5567(2) | 46(1) |
| C(40) | 6145(2) | 3893(2) | 5587(2) | 39(1) |
| C(41) | 6246(2) | 4326(2) | 5143(2) | 47(1) |
| C(42) | 6212(3) | 4116(2) | 6252(2) | 69(2) |
| C(43) | 6339(2) | 2839(2) | 6094(2) | 34(1) |
| C(44) | 6694(2) | 2314(2) | 6153(2) | 41(1) |
| C(45) | 5614(2) | 2756(2) | 5918(2) | 43(1) |
| C(46) | 8239(2) | 1597(1) | 4911(2) | 28(1) |
| C(47) | 8897(2) | 1338(2) | 5120(2) | 43(1) |
| C(48) | 8044(2) | 1854(2) | 5469(2) | 37(1) |
| C(49) | 8727(2) | 2558(1) | 4318(2) | 30(1) |
| C(50) | 9423(2) | 2383(2) | 4366(2) | 44(1) |
| C(51) | 8688(2) | 2923(2) | 4859(2) | 40(1) |
| C(52) | 8355(2) | 1593(2) | 3611(2) | 32(1) |
| C(53) | 8340(2) | 1883(2) | 2995(2) | 42(1) |
| C(54) | 7946(2) | 1096(2) | 3496(2) | 41(1) |
| C(61) | 983(2) | 3495(1) | 3547(2) | 32(1) |
| C(62) | 2102(2) | 3338(2) | 3061(3) | 57(1) |
| C(63) | 571(2) | 3231(2) | 2234(2) | 43(1) |
| C(64) | -383(2) | 3452(2) | 3218(2) | 40(1) |
| C(65) | 543(2) | 3712(2) | 4664(2) | 50(1) |
| C(66) | 2090(2) | 3643(2) | 4572(2) | 60(1) |
| C(67) | 1379(3) | 4888(2) | 4597(2) | 55(1) |
| C(68) | -200(2) | 4769(2) | 3730(2) | 45(1) |
| C(69) | -179(2) | 4463(2) | 2191(2) | 40(1) |
| C(70) | 1430(2) | 4378(2) | 2078(2) | 41(1) |
| C(71) | 958(2) | 5338(1) | 3031(2) | 40(1) |
| C(81) | 4483(2) | 4966(2) | 1114(4) | 86(2) |
| C(82) | 3214(2) | 5051(2) | -203(2) | 45(1) |
| C(83) | 4437(2) | 5983(2) | 13(3) | 61(1) |
| C(84) | 4915(2) | 6209(2) | 1645(3) | 68(2) |
| C(85) | 4075(6) | 5352(4) | 2287(5) | 72(3) |
| C(86) | 2948(4) | 4767(3) | 1333(4) | 42(2) |
| C(87) | 1942(2) | 5593(2) | 293(2) | 48(1) |
| C(88) | 2850(3) | 6374(2) | -485(2) | 56(1) |
| C(89) | 3902(2) | 7106(2) | 663(2) | 42(1) |
| C(90) | 3652(3) | 6746(2) | 2134(2) | 55(1) |
| C(91) | 2453(3) | 5807(2) | 1897(2) | 68(2) |
| C(92) | 2364(2) | 6862(2) | 829(3) | 57(1) |
| C(107) | 2377(2) | 4662(2) | 3555(2) | 50(1) |
| C(901) | -1735(12) | 1819(10) | 1010(11) | 163(8) |
| C(902) | -1733(7) | 2224(6) | 551(7) | 93(4) |
| C(903) | -1491(7) | 2563(6) | 1026(7) | 95(4) |
| C(904) | -975(5) | 2723(5) | 1414(5) | 58(2) |
| C(905) | -949(7) | 2444(6) | 1589(7) | 91(4) |
| C(906) | 6038(6) | 5778(5) | 3138(6) | 71(3) |
| C(907) | 6186(9) | 5947(8) | 3442(9) | 115(6) |
| C(908) | 4365(5) | 4163(4) | 5577(5) | 60(3) |

| | | | | |
|---|---|---|---|---|
| C(909) | 4512(10) | 4079(8) | 6084(10) | 135(6) |
| B(62) | 1573(2) | 3724(2) | 3206(2) | 34(1) |
| B(63) | 763(2) | 3664(2) | 2767(2) | 29(1) |
| B(64) | 258(2) | 3784(2) | 3292(2) | 27(1) |
| B(65) | 745(2) | 3921(2) | 4050(2) | 34(1) |
| B(66) | 1563(2) | 3882(2) | 4002(2) | 38(1) |
| B(67) | 1185(2) | 4502(2) | 4002(2) | 33(1) |
| B(68) | 372(2) | 4442(2) | 3562(2) | 29(1) |
| B(69) | 388(2) | 4281(2) | 2768(2) | 26(1) |
| B(70) | 1207(2) | 4241(2) | 2712(2) | 28(1) |
| B(71) | 1698(2) | 4380(2) | 3476(2) | 34(1) |
| B(72) | 964(2) | 4728(1) | 3203(2) | 27(1) |
| B(81) | 3976(2) | 5433(2) | 1004(2) | 39(1) |
| B(82) | 3323(2) | 5480(2) | 344(2) | 29(1) |
| B(83) | 3946(2) | 5957(2) | 465(2) | 33(1) |
| B(84) | 4197(2) | 6080(2) | 1282(2) | 34(1) |
| B(85) | 3786(5) | 5650(4) | 1624(5) | 32(2) |
| B(86) | 3141(6) | 5339(4) | 1120(5) | 27(3) |
| B(87) | 2662(2) | 5758(2) | 602(2) | 29(1) |
| B(88) | 3134(2) | 6156(2) | 215(2) | 29(1) |
| B(89) | 3673(2) | 6526(2) | 796(2) | 28(1) |
| B(90) | 3539(2) | 6357(2) | 1541(2) | 35(1) |
| B(91) | 2911(2) | 5880(2) | 1421(2) | 39(1) |
| B(92) | 2879(2) | 6405(2) | 881(2) | 31(1) |
| C(1A) | 4675(4) | 4222(3) | 3137(3) | 35(2) |
| C(2A) | 5207(12) | 4545(6) | 3578(12) | 37(3) |
| C(3A) | 4083(5) | 4580(4) | 2959(5) | 58(3) |
| C(4A) | 3875(5) | 3354(4) | 2724(8) | 88(5) |
| C(5A) | 4090(9) | 3398(7) | 2107(8) | 139(10) |
| C(6A) | 3655(10) | 2762(10) | 2817(15) | 121(13) |
| C(7A) | 4120(7) | 3661(4) | 4068(7) | 85(4) |
| C(8A) | 4324(13) | 4044(7) | 4482(8) | 163(10) |
| C(9A) | 4026(18) | 2999(15) | 4190(30) | 380(40) |

**Table S3.** Bond lengths [Å] and angles [°] for **1**.

| | | | |
|---|---|---|---|
| Rh(1)-H(108) | 1.79(4) | Rh(1)-H(101) | 1.84(4) |
| Rh(1)-H(111) | 1.85(4) | Rh(1)-P(1) | 2.2359(11) |
| Rh(1)-Rh(5) | 2.7762(4) | Rh(1)-Rh(2) | 2.7868(4) |
| Rh(1)-Rh(4) | 2.8606(4) | Rh(1)-Rh(3) | 3.0597(5) |
| Rh(2)-H(108) | 1.79(4) | Rh(2)-H(106) | 1.81(3) |
| Rh(2)-H(105) | 1.83(4) | Rh(2)-H(107) | 1.86(4) |
| Rh(2)-P(2) | 2.2556(9) | Rh(2)-Rh(6) | 2.7024(3) |
| Rh(2)-Rh(5) | 2.7479(4) | Rh(2)-Rh(3) | 2.8547(4) |
| Rh(3)-H(104) | 1.80(4) | Rh(3)-H(105) | 1.84(4) |
| Rh(3)-H(109) | 1.85(4) | Rh(3)-P(3) | 2.2484(9) |
| Rh(3)-Rh(6) | 2.7826(3) | Rh(3)-Rh(4) | 2.7878(4) |
| Rh(4)-H(102) | 1.77(3) | Rh(4)-H(104) | 1.80(4) |
| Rh(4)-H(101) | 1.82(4) | Rh(4)-H(103) | 1.91(5) |
| Rh(4)-P(4) | 2.2612(9) | Rh(4)-Rh(6) | 2.7181(3) |
| Rh(4)-Rh(5) | 2.7193(3) | Rh(5)-H(110) | 1.76(3) |
| Rh(5)-H(102) | 1.78(3) | Rh(5)-H(111) | 1.84(4) |
| Rh(5)-H(107) | 1.84(4) | Rh(5)-P(5) | 2.2475(9) |
| Rh(5)-Rh(6) | 2.7329(3) | Rh(6)-H(110) | 1.76(3) |
| Rh(6)-H(106) | 1.83(3) | Rh(6)-H(109) | 1.83(4) |
| Rh(6)-H(103) | 1.90(5) | Rh(6)-P(6) | 2.2374(8) |
| P(1)-C(7) | 1.719(10) | P(1)-C(4A) | 1.740(10) |
| P(1)-C(4) | 1.790(9) | P(1)-C(1A) | 1.889(8) |
| P(1)-C(1) | 1.935(9) | P(1)-C(7A) | 2.062(10) |
| P(2)-C(16) | 1.853(4) | P(2)-C(13) | 1.858(4) |
| P(2)-C(10) | 1.860(4) | P(3)-C(19) | 1.841(4) |
| P(3)-C(22) | 1.858(3) | P(3)-C(25) | 1.862(4) |
| P(4)-C(34) | 1.849(4) | P(4)-C(28) | 1.855(4) |
| P(4)-C(31) | 1.856(4) | P(5)-C(43) | 1.844(4) |
| P(5)-C(37) | 1.849(4) | P(5)-C(40) | 1.853(4) |
| P(6)-C(52) | 1.843(4) | P(6)-C(49) | 1.852(3) |
| P(6)-C(46) | 1.854(3) | C(1)-C(2) | 1.553(13) |
| C(1)-C(3) | 1.563(12) | C(4)-C(5) | 1.49(3) |
| C(4)-C(6) | 1.63(2) | C(05)-B(91) | 1.569(10) |
| C(05)-B(90) | 1.605(10) | C(05)-B(86) | 1.693(14) |
| C(05)-B(84) | 1.796(9) | C(05)-B(81) | 1.864(10) |
| C(06)-B(81) | 1.616(11) | C(06)-B(82) | 1.639(11) |
| C(06)-B(85) | 1.704(14) | C(06)-B(87) | 1.784(11) |
| C(06)-B(91) | 1.846(11) | C(7)-C(8) | 1.44(2) |
| C(7)-C(9) | 1.547(13) | C(10)-C(12) | 1.523(6) |
| C(10)-C(11) | 1.535(6) | C(13)-C(14) | 1.527(6) |
| C(13)-C(15) | 1.540(5) | C(16)-C(17) | 1.531(6) |
| C(16)-C(18) | 1.533(6) | C(19)-C(21) | 1.522(6) |
| C(19)-C(20) | 1.535(6) | C(22)-C(24) | 1.529(6) |
| C(22)-C(23) | 1.532(5) | C(25)-C(26) | 1.527(6) |
| C(25)-C(27) | 1.544(6) | C(28)-C(30) | 1.521(6) |
| C(28)-C(29) | 1.534(5) | C(31)-C(33) | 1.527(6) |
| C(31)-C(32) | 1.538(5) | C(34)-C(36) | 1.530(5) |
| C(34)-C(35) | 1.534(6) | C(37)-C(39) | 1.523(6) |
| C(37)-C(38) | 1.545(5) | C(40)-C(41) | 1.517(6) |
| C(40)-C(42) | 1.533(6) | C(43)-C(45) | 1.532(5) |
| C(43)-C(44) | 1.532(5) | C(46)-C(48) | 1.522(5) |
| C(46)-C(47) | 1.533(5) | C(49)-C(51) | 1.520(5) |

| | | | |
|---|---|---|---|
| C(49)-C(50) | 1.542(5) | C(52)-C(53) | 1.528(5) |
| C(52)-C(54) | 1.530(5) | C(61)-B(64) | 1.698(5) |
| C(61)-B(65) | 1.703(6) | C(61)-B(63) | 1.712(5) |
| C(61)-B(62) | 1.713(6) | C(61)-B(66) | 1.715(5) |
| C(62)-B(62) | 1.590(6) | C(63)-B(63) | 1.590(5) |
| C(64)-B(64) | 1.592(5) | C(65)-B(65) | 1.592(6) |
| C(66)-B(66) | 1.598(6) | C(67)-B(67) | 1.605(6) |
| C(68)-B(68) | 1.596(5) | C(69)-B(69) | 1.607(5) |
| C(70)-B(70) | 1.603(5) | C(71)-B(72) | 1.601(5) |
| C(81)-B(81) | 1.595(6) | C(82)-B(82) | 1.596(5) |
| C(83)-B(83) | 1.603(6) | C(84)-B(84) | 1.599(6) |
| C(85)-B(85) | 1.625(14) | C(86)-B(86) | 1.614(13) |
| C(87)-B(87) | 1.595(5) | C(88)-B(88) | 1.607(6) |
| C(89)-B(89) | 1.605(5) | C(90)-B(90) | 1.604(6) |
| C(91)-B(91) | 1.598(6) | C(92)-B(92) | 1.594(6) |
| C(107)-B(71) | 1.600(5) | C(901)-C(902) | 1.44(3) |
| C(901)-C(903) | 1.97(3) | C(902)-C(903) | 1.358(19) |
| C(903)-C(904) | 1.297(18) | C(903)-C(905) | 1.52(2) |
| C(904)-C(905) | 0.802(16) | C(906)-C(907) | 0.793(19) |
| C(907)-C(909)#1 | 2.01(3) | C(908)-C(909) | 1.10(2) |
| C(909)-C(907)#1 | 2.01(3) | B(62)-B(70) | 1.767(6) |
| B(62)-B(71) | 1.771(6) | B(62)-B(66) | 1.788(7) |
| B(62)-B(63) | 1.790(6) | B(63)-B(69) | 1.768(6) |
| B(63)-B(70) | 1.771(6) | B(63)-B(64) | 1.777(6) |
| B(64)-B(69) | 1.773(5) | B(64)-B(65) | 1.777(6) |
| B(64)-B(68) | 1.778(6) | B(65)-B(68) | 1.774(6) |
| B(65)-B(67) | 1.775(6) | B(65)-B(66) | 1.788(6) |
| B(66)-B(67) | 1.778(6) | B(66)-B(71) | 1.780(6) |
| B(67)-B(72) | 1.793(6) | B(67)-B(71) | 1.794(6) |
| B(67)-B(68) | 1.797(6) | B(68)-B(69) | 1.789(5) |
| B(68)-B(72) | 1.794(5) | B(69)-B(72) | 1.785(5) |
| B(69)-B(70) | 1.796(5) | B(70)-B(71) | 1.792(6) |
| B(70)-B(72) | 1.796(5) | B(71)-B(72) | 1.790(6) |
| B(81)-B(85) | 1.600(12) | B(81)-B(82) | 1.768(6) |
| B(81)-B(83) | 1.772(6) | B(81)-B(84) | 1.785(6) |
| B(81)-B(86) | 1.884(13) | B(82)-B(88) | 1.777(6) |
| B(82)-B(83) | 1.783(6) | B(82)-B(87) | 1.791(5) |
| B(82)-B(86) | 1.860(13) | B(83)-B(84) | 1.770(6) |
| B(83)-B(89) | 1.780(6) | B(83)-B(88) | 1.781(6) |
| B(84)-B(85) | 1.685(11) | B(84)-B(89) | 1.771(6) |
| B(84)-B(90) | 1.785(7) | B(85)-B(90) | 1.875(12) |
| B(85)-B(91) | 1.923(12) | B(86)-B(91) | 1.651(13) |
| B(86)-B(87) | 1.717(12) | B(87)-B(91) | 1.773(6) |
| B(87)-B(88) | 1.779(6) | B(87)-B(92) | 1.783(6) |
| B(88)-B(89) | 1.779(6) | B(88)-B(92) | 1.784(6) |
| B(89)-B(90) | 1.769(6) | B(89)-B(92) | 1.786(6) |
| B(90)-B(92) | 1.778(6) | B(90)-B(91) | 1.790(6) |
| B(91)-B(92) | 1.774(6) | C(1A)-C(3A) | 1.540(11) |
| C(1A)-C(2A) | 1.55(3) | C(4A)-C(5A) | 1.53(2) |
| C(4A)-C(6A) | 1.61(3) | C(7A)-C(8A) | 1.33(2) |
| C(7A)-C(9A) | 1.73(4) | | |
| | | | |
| H(108)-Rh(1)-H(101) | 162(3) | H(108)-Rh(1)-H(111) | 91(3) |
| H(101)-Rh(1)-H(111) | 92(3) | H(108)-Rh(1)-P(1) | 88.3(17) |

| | | | |
|---|---|---|---|
| H(101)-Rh(1)-P(1) | 109.4(17) | H(111)-Rh(1)-P(1) | 92.0(13) |
| H(108)-Rh(1)-Rh(5) | 85(2) | H(101)-Rh(1)-Rh(5) | 86.5(18) |
| H(111)-Rh(1)-Rh(5) | 40.9(13) | P(1)-Rh(1)-Rh(5) | 131.88(4) |
| H(108)-Rh(1)-Rh(2) | 39.0(17) | H(101)-Rh(1)-Rh(2) | 123.2(17) |
| H(111)-Rh(1)-Rh(2) | 88.5(14) | P(1)-Rh(1)-Rh(2) | 127.27(3) |
| Rh(5)-Rh(1)-Rh(2) | 59.202(9) | H(108)-Rh(1)-Rh(4) | 124.3(17) |
| H(101)-Rh(1)-Rh(4) | 38.5(17) | H(111)-Rh(1)-Rh(4) | 84.9(15) |
| P(1)-Rh(1)-Rh(4) | 147.26(3) | Rh(5)-Rh(1)-Rh(4) | 57.663(9) |
| Rh(2)-Rh(1)-Rh(4) | 85.297(11) | H(108)-Rh(1)-Rh(3) | 85.6(19) |
| H(101)-Rh(1)-Rh(3) | 78(2) | H(111)-Rh(1)-Rh(3) | 127.8(13) |
| P(1)-Rh(1)-Rh(3) | 139.75(4) | Rh(5)-Rh(1)-Rh(3) | 87.067(11) |
| Rh(2)-Rh(1)-Rh(3) | 58.231(10) | Rh(4)-Rh(1)-Rh(3) | 56.061(9) |
| H(108)-Rh(2)-H(106) | 174(2) | H(108)-Rh(2)-H(105) | 92(3) |
| H(106)-Rh(2)-H(105) | 90(2) | H(108)-Rh(2)-H(107) | 84(3) |
| H(106)-Rh(2)-H(107) | 94(2) | H(105)-Rh(2)-H(107) | 172(2) |
| H(108)-Rh(2)-P(2) | 93.9(17) | H(106)-Rh(2)-P(2) | 91.7(12) |
| H(105)-Rh(2)-P(2) | 84.8(14) | H(107)-Rh(2)-P(2) | 102.5(13) |
| H(108)-Rh(2)-Rh(6) | 132.2(17) | H(106)-Rh(2)-Rh(6) | 42.3(12) |
| H(105)-Rh(2)-Rh(6) | 88.3(16) | H(107)-Rh(2)-Rh(6) | 89.5(15) |
| P(2)-Rh(2)-Rh(6) | 133.56(3) | H(108)-Rh(2)-Rh(5) | 85.4(19) |
| H(106)-Rh(2)-Rh(5) | 89.6(13) | H(105)-Rh(2)-Rh(5) | 130.9(14) |
| H(107)-Rh(2)-Rh(5) | 41.9(13) | P(2)-Rh(2)-Rh(5) | 144.33(3) |
| Rh(6)-Rh(2)-Rh(5) | 60.181(9) | H(108)-Rh(2)-Rh(1) | 38.9(17) |
| H(106)-Rh(2)-Rh(1) | 135.7(12) | H(105)-Rh(2)-Rh(1) | 88.0(17) |
| H(107)-Rh(2)-Rh(1) | 84.0(15) | P(2)-Rh(2)-Rh(1) | 132.04(3) |
| Rh(6)-Rh(2)-Rh(1) | 93.377(11) | Rh(5)-Rh(2)-Rh(1) | 60.207(10) |
| H(108)-Rh(2)-Rh(3) | 92(2) | H(106)-Rh(2)-Rh(3) | 85.5(14) |
| H(105)-Rh(2)-Rh(3) | 39.2(14) | H(107)-Rh(2)-Rh(3) | 133.7(13) |
| P(2)-Rh(2)-Rh(3) | 123.81(3) | Rh(6)-Rh(2)-Rh(3) | 60.021(9) |
| Rh(5)-Rh(2)-Rh(3) | 91.825(11) | Rh(1)-Rh(2)-Rh(3) | 65.676(11) |
| H(104)-Rh(3)-H(105) | 163(2) | H(104)-Rh(3)-H(109) | 89(3) |
| H(105)-Rh(3)-H(109) | 94(3) | H(104)-Rh(3)-P(3) | 88.3(13) |
| H(105)-Rh(3)-P(3) | 108.2(14) | H(109)-Rh(3)-P(3) | 96.4(15) |
| H(104)-Rh(3)-Rh(6) | 85.3(15) | H(105)-Rh(3)-Rh(6) | 85.7(16) |
| H(109)-Rh(3)-Rh(6) | 40.6(15) | P(3)-Rh(3)-Rh(6) | 136.60(3) |
| H(104)-Rh(3)-Rh(4) | 39.2(13) | H(105)-Rh(3)-Rh(4) | 124.1(14) |
| H(109)-Rh(3)-Rh(4) | 85.1(17) | P(3)-Rh(3)-Rh(4) | 127.50(2) |
| Rh(6)-Rh(3)-Rh(4) | 58.412(9) | H(104)-Rh(3)-Rh(2) | 124.6(13) |
| H(105)-Rh(3)-Rh(2) | 39.0(14) | H(109)-Rh(3)-Rh(2) | 86.4(16) |
| P(3)-Rh(3)-Rh(2) | 147.09(3) | Rh(6)-Rh(3)-Rh(2) | 57.273(9) |
| Rh(4)-Rh(3)-Rh(2) | 85.392(10) | H(104)-Rh(3)-Rh(1) | 84.9(15) |
| H(105)-Rh(3)-Rh(1) | 79.9(17) | H(109)-Rh(3)-Rh(1) | 126.8(15) |
| P(3)-Rh(3)-Rh(1) | 135.97(3) | Rh(6)-Rh(3)-Rh(1) | 86.134(10) |
| Rh(4)-Rh(3)-Rh(1) | 58.354(9) | Rh(2)-Rh(3)-Rh(1) | 56.094(9) |
| H(102)-Rh(4)-H(104) | 172.9(19) | H(102)-Rh(4)-H(101) | 93(3) |
| H(104)-Rh(4)-H(101) | 88(3) | H(102)-Rh(4)-H(103) | 88(3) |
| H(104)-Rh(4)-H(103) | 90(3) | H(101)-Rh(4)-H(103) | 174(2) |
| H(102)-Rh(4)-P(4) | 95.0(12) | H(104)-Rh(4)-P(4) | 91.9(14) |
| H(101)-Rh(4)-P(4) | 91.5(17) | H(103)-Rh(4)-P(4) | 94.2(15) |
| H(102)-Rh(4)-Rh(6) | 86.5(13) | H(104)-Rh(4)-Rh(6) | 87.3(15) |
| H(101)-Rh(4)-Rh(6) | 130.0(17) | H(103)-Rh(4)-Rh(6) | 44.3(15) |
| P(4)-Rh(4)-Rh(6) | 138.42(2) | H(102)-Rh(4)-Rh(5) | 40.0(12) |
| H(104)-Rh(4)-Rh(5) | 133.2(14) | H(101)-Rh(4)-Rh(5) | 88.6(18) |
| H(103)-Rh(4)-Rh(5) | 88.3(18) | P(4)-Rh(4)-Rh(5) | 134.88(3) |

| | | | |
|---|---|---|---|
| Rh(6)-Rh(4)-Rh(5) | 60.347(9) | H(102)-Rh(4)-Rh(3) | 133.9(12) |
| H(104)-Rh(4)-Rh(3) | 39.3(14) | H(101)-Rh(4)-Rh(3) | 86(2) |
| H(103)-Rh(4)-Rh(3) | 89.1(19) | P(4)-Rh(4)-Rh(3) | 131.12(3) |
| Rh(6)-Rh(4)-Rh(3) | 60.699(9) | Rh(5)-Rh(4)-Rh(3) | 93.911(10) |
| H(102)-Rh(4)-Rh(1) | 85.4(13) | H(104)-Rh(4)-Rh(1) | 91.2(15) |
| H(101)-Rh(4)-Rh(1) | 38.9(17) | H(103)-Rh(4)-Rh(1) | 135.5(15) |
| P(4)-Rh(4)-Rh(1) | 130.15(2) | Rh(6)-Rh(4)-Rh(1) | 91.422(10) |
| Rh(5)-Rh(4)-Rh(1) | 59.611(10) | Rh(3)-Rh(4)-Rh(1) | 65.585(11) |
| H(110)-Rh(5)-H(102) | 89(2) | H(110)-Rh(5)-H(111) | 173.4(18) |
| H(102)-Rh(5)-H(111) | 90(2) | H(110)-Rh(5)-H(107) | 92(2) |
| H(102)-Rh(5)-H(107) | 170.8(17) | H(111)-Rh(5)-H(107) | 88(2) |
| H(110)-Rh(5)-P(5) | 95.1(13) | H(102)-Rh(5)-P(5) | 95.1(12) |
| H(111)-Rh(5)-P(5) | 91.5(13) | H(107)-Rh(5)-P(5) | 94.0(13) |
| H(110)-Rh(5)-Rh(4) | 85.9(15) | H(102)-Rh(5)-Rh(4) | 39.7(12) |
| H(111)-Rh(5)-Rh(4) | 89.4(15) | H(107)-Rh(5)-Rh(4) | 131.2(13) |
| P(5)-Rh(5)-Rh(4) | 134.76(3) | H(110)-Rh(5)-Rh(6) | 39.2(13) |
| H(102)-Rh(5)-Rh(6) | 85.8(13) | H(111)-Rh(5)-Rh(6) | 134.2(13) |
| H(107)-Rh(5)-Rh(6) | 89.0(15) | P(5)-Rh(5)-Rh(6) | 134.31(3) |
| Rh(4)-Rh(5)-Rh(6) | 59.804(9) | H(110)-Rh(5)-Rh(2) | 85.3(15) |
| H(102)-Rh(5)-Rh(2) | 128.5(12) | H(111)-Rh(5)-Rh(2) | 90.0(14) |
| H(107)-Rh(5)-Rh(2) | 42.5(13) | P(5)-Rh(5)-Rh(2) | 136.38(3) |
| Rh(4)-Rh(5)-Rh(2) | 88.837(10) | Rh(6)-Rh(5)-Rh(2) | 59.084(9) |
| H(110)-Rh(5)-Rh(1) | 132.1(13) | H(102)-Rh(5)-Rh(1) | 87.9(13) |
| H(111)-Rh(5)-Rh(1) | 41.2(13) | H(107)-Rh(5)-Rh(1) | 84.7(15) |
| P(5)-Rh(5)-Rh(1) | 132.74(3) | Rh(4)-Rh(5)-Rh(1) | 62.726(10) |
| Rh(6)-Rh(5)-Rh(1) | 92.949(12) | Rh(2)-Rh(5)-Rh(1) | 60.591(10) |
| H(110)-Rh(6)-H(106) | 92(2) | H(110)-Rh(6)-H(109) | 173(2) |
| H(106)-Rh(6)-H(109) | 91(2) | H(110)-Rh(6)-H(103) | 88(3) |
| H(106)-Rh(6)-H(103) | 176(2) | H(109)-Rh(6)-H(103) | 89(3) |
| H(110)-Rh(6)-P(6) | 93.9(13) | H(106)-Rh(6)-P(6) | 93.8(11) |
| H(109)-Rh(6)-P(6) | 92.2(15) | H(103)-Rh(6)-P(6) | 90.0(15) |
| H(110)-Rh(6)-Rh(2) | 86.7(15) | H(106)-Rh(6)-Rh(2) | 41.8(11) |
| H(109)-Rh(6)-Rh(2) | 91.4(17) | H(103)-Rh(6)-Rh(2) | 134.4(15) |
| P(6)-Rh(6)-Rh(2) | 135.49(2) | H(110)-Rh(6)-Rh(4) | 85.9(15) |
| H(106)-Rh(6)-Rh(4) | 131.5(11) | H(109)-Rh(6)-Rh(4) | 87.4(17) |
| H(103)-Rh(6)-Rh(4) | 44.7(15) | P(6)-Rh(6)-Rh(4) | 134.67(2) |
| Rh(2)-Rh(6)-Rh(4) | 89.810(10) | H(110)-Rh(6)-Rh(5) | 39.2(13) |
| H(106)-Rh(6)-Rh(5) | 89.7(13) | H(109)-Rh(6)-Rh(5) | 134.6(15) |
| H(103)-Rh(6)-Rh(5) | 88.2(19) | P(6)-Rh(6)-Rh(5) | 133.06(2) |
| Rh(2)-Rh(6)-Rh(5) | 60.735(10) | Rh(4)-Rh(6)-Rh(5) | 59.849(9) |
| H(110)-Rh(6)-Rh(3) | 132.9(13) | H(106)-Rh(6)-Rh(3) | 87.3(14) |
| H(109)-Rh(6)-Rh(3) | 41.0(15) | H(103)-Rh(6)-Rh(3) | 89.6(19) |
| P(6)-Rh(6)-Rh(3) | 133.17(2) | Rh(2)-Rh(6)-Rh(3) | 62.706(9) |
| Rh(4)-Rh(6)-Rh(3) | 60.889(9) | Rh(5)-Rh(6)-Rh(3) | 93.726(11) |
| C(7)-P(1)-C(4A) | 130.5(5) | C(7)-P(1)-C(4) | 110.3(6) |
| C(4A)-P(1)-C(4) | 59.9(7) | C(7)-P(1)-C(1A) | 46.4(4) |
| C(4A)-P(1)-C(1A) | 101.9(5) | C(4)-P(1)-C(1A) | 133.4(4) |
| C(7)-P(1)-C(1) | 108.4(4) | C(4A)-P(1)-C(1) | 47.8(6) |
| C(4)-P(1)-C(1) | 107.0(5) | C(1A)-P(1)-C(1) | 63.3(4) |
| C(7)-P(1)-C(7A) | 57.8(5) | C(4A)-P(1)-C(7A) | 102.1(7) |
| C(4)-P(1)-C(7A) | 53.5(6) | C(1A)-P(1)-C(7A) | 96.6(4) |
| C(1)-P(1)-C(7A) | 131.0(5) | C(7)-P(1)-Rh(1) | 108.0(3) |
| C(4A)-P(1)-Rh(1) | 121.1(4) | C(4)-P(1)-Rh(1) | 110.2(3) |
| C(1A)-P(1)-Rh(1) | 115.5(2) | C(1)-P(1)-Rh(1) | 113.0(3) |

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|---|---|---|---|
| C(7A)-P(1)-Rh(1) | 116.0(4) | C(16)-P(2)-C(13) | 105.96(17) |
| C(16)-P(2)-C(10) | 105.32(19) | C(13)-P(2)-C(10) | 105.47(18) |
| C(16)-P(2)-Rh(2) | 112.81(12) | C(13)-P(2)-Rh(2) | 115.92(13) |
| C(10)-P(2)-Rh(2) | 110.56(13) | C(19)-P(3)-C(22) | 106.47(16) |
| C(19)-P(3)-C(25) | 104.37(18) | C(22)-P(3)-C(25) | 105.78(18) |
| C(19)-P(3)-Rh(3) | 114.72(14) | C(22)-P(3)-Rh(3) | 114.60(12) |
| C(25)-P(3)-Rh(3) | 110.03(12) | C(34)-P(4)-C(28) | 104.44(17) |
| C(34)-P(4)-C(31) | 103.29(17) | C(28)-P(4)-C(31) | 111.05(17) |
| C(34)-P(4)-Rh(4) | 111.40(12) | C(28)-P(4)-Rh(4) | 113.33(12) |
| C(31)-P(4)-Rh(4) | 112.56(12) | C(43)-P(5)-C(37) | 104.47(18) |
| C(43)-P(5)-C(40) | 103.22(17) | C(37)-P(5)-C(40) | 111.29(19) |
| C(43)-P(5)-Rh(5) | 112.31(13) | C(37)-P(5)-Rh(5) | 111.71(12) |
| C(40)-P(5)-Rh(5) | 113.21(13) | C(52)-P(6)-C(49) | 104.12(17) |
| C(52)-P(6)-C(46) | 103.60(16) | C(49)-P(6)-C(46) | 111.66(16) |
| C(52)-P(6)-Rh(6) | 115.28(11) | C(49)-P(6)-Rh(6) | 109.67(11) |
| C(46)-P(6)-Rh(6) | 112.17(11) | C(2)-C(1)-C(3) | 110.9(8) |
| C(2)-C(1)-P(1) | 118.4(5) | C(3)-C(1)-P(1) | 110.4(7) |
| C(5)-C(4)-C(6) | 115.9(14) | C(5)-C(4)-P(1) | 117.7(11) |
| C(6)-C(4)-P(1) | 111.1(9) | B(91)-C(05)-B(90) | 68.7(4) |
| B(91)-C(05)-B(86) | 60.7(5) | B(90)-C(05)-B(86) | 117.4(7) |
| B(91)-C(05)-B(84) | 118.5(5) | B(90)-C(05)-B(84) | 63.0(4) |
| B(86)-C(05)-B(84) | 113.0(6) | B(91)-C(05)-B(81) | 114.2(5) |
| B(90)-C(05)-B(81) | 112.3(5) | B(86)-C(05)-B(81) | 63.8(5) |
| B(84)-C(05)-B(81) | 58.4(3) | B(81)-C(06)-B(82) | 65.8(5) |
| B(81)-C(06)-B(85) | 57.6(5) | B(82)-C(06)-B(85) | 112.3(7) |
| B(81)-C(06)-B(87) | 115.8(6) | B(82)-C(06)-B(87) | 62.9(4) |
| B(85)-C(06)-B(87) | 112.6(7) | B(81)-C(06)-B(91) | 112.8(6) |
| B(82)-C(06)-B(91) | 111.5(6) | B(85)-C(06)-B(91) | 65.4(5) |
| B(87)-C(06)-B(91) | 58.4(4) | C(8)-C(7)-C(9) | 112.1(12) |
| C(8)-C(7)-P(1) | 114.3(13) | C(9)-C(7)-P(1) | 113.4(8) |
| C(12)-C(10)-C(11) | 110.3(4) | C(12)-C(10)-P(2) | 111.3(3) |
| C(11)-C(10)-P(2) | 115.9(3) | C(14)-C(13)-C(15) | 109.5(3) |
| C(14)-C(13)-P(2) | 114.1(3) | C(15)-C(13)-P(2) | 114.9(3) |
| C(17)-C(16)-C(18) | 111.1(3) | C(17)-C(16)-P(2) | 111.1(3) |
| C(18)-C(16)-P(2) | 111.1(3) | C(21)-C(19)-C(20) | 110.4(4) |
| C(21)-C(19)-P(3) | 112.9(3) | C(20)-C(19)-P(3) | 111.3(3) |
| C(24)-C(22)-C(23) | 109.7(3) | C(24)-C(22)-P(3) | 114.7(3) |
| C(23)-C(22)-P(3) | 113.5(2) | C(26)-C(25)-C(27) | 109.5(4) |
| C(26)-C(25)-P(3) | 110.8(3) | C(27)-C(25)-P(3) | 117.8(3) |
| C(30)-C(28)-C(29) | 111.0(3) | C(30)-C(28)-P(4) | 113.4(3) |
| C(29)-C(28)-P(4) | 114.6(3) | C(33)-C(31)-C(32) | 110.1(3) |
| C(33)-C(31)-P(4) | 112.7(3) | C(32)-C(31)-P(4) | 116.6(3) |
| C(36)-C(34)-C(35) | 110.6(3) | C(36)-C(34)-P(4) | 112.2(3) |
| C(35)-C(34)-P(4) | 110.9(3) | C(39)-C(37)-C(38) | 110.4(3) |
| C(39)-C(37)-P(5) | 112.7(3) | C(38)-C(37)-P(5) | 115.7(3) |
| C(41)-C(40)-C(42) | 109.8(4) | C(41)-C(40)-P(5) | 114.5(3) |
| C(42)-C(40)-P(5) | 115.2(3) | C(45)-C(43)-C(44) | 111.1(3) |
| C(45)-C(43)-P(5) | 111.9(3) | C(44)-C(43)-P(5) | 111.8(2) |
| C(48)-C(46)-C(47) | 110.6(3) | C(48)-C(46)-P(6) | 114.1(2) |
| C(47)-C(46)-P(6) | 115.5(3) | C(51)-C(49)-C(50) | 110.5(3) |
| C(51)-C(49)-P(6) | 113.2(3) | C(50)-C(49)-P(6) | 115.4(3) |
| C(53)-C(52)-C(54) | 111.0(3) | C(53)-C(52)-P(6) | 112.2(3) |
| C(54)-C(52)-P(6) | 111.9(3) | B(64)-C(61)-B(65) | 63.0(2) |
| B(64)-C(61)-B(63) | 62.8(2) | B(65)-C(61)-B(63) | 115.1(3) |

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|---|---|---|---|
| B(64)-C(61)-B(62) | 115.3(3) | B(65)-C(61)-B(62) | 115.2(3) |
| B(63)-C(61)-B(62) | 63.0(2) | B(64)-C(61)-B(66) | 115.6(3) |
| B(65)-C(61)-B(66) | 63.1(3) | B(63)-C(61)-B(66) | 115.4(3) |
| B(62)-C(61)-B(66) | 62.9(3) | C(902)-C(901)-C(903) | 43.7(10) |
| C(903)-C(902)-C(901) | 89.3(15) | C(904)-C(903)-C(902) | 145.0(15) |
| C(904)-C(903)-C(905) | 31.9(7) | C(902)-C(903)-C(905) | 125.4(14) |
| C(904)-C(903)-C(901) | 120.0(13) | C(902)-C(903)-C(901) | 47.1(10) |
| C(905)-C(903)-C(901) | 88.5(12) | C(905)-C(904)-C(903) | 89.3(17) |
| C(904)-C(905)-C(903) | 58.8(14) | C(906)-C(907)-C(909)#1 | 102(2) |
| C(908)-C(909)-C(907)#1 | 116.6(17) | C(62)-B(62)-C(61) | 120.7(3) |
| C(62)-B(62)-B(70) | 125.3(4) | C(61)-B(62)-B(70) | 104.4(3) |
| C(62)-B(62)-B(71) | 126.1(3) | C(61)-B(62)-B(71) | 104.4(3) |
| B(70)-B(62)-B(71) | 60.9(2) | C(62)-B(62)-B(66) | 120.1(4) |
| C(61)-B(62)-B(66) | 58.6(2) | B(70)-B(62)-B(66) | 108.8(3) |
| B(71)-B(62)-B(66) | 60.0(2) | C(62)-B(62)-B(63) | 119.5(3) |
| C(61)-B(62)-B(63) | 58.5(2) | B(70)-B(62)-B(63) | 59.7(2) |
| B(71)-B(62)-B(63) | 108.3(3) | B(66)-B(62)-B(63) | 108.1(3) |
| C(63)-B(63)-C(61) | 121.2(3) | C(63)-B(63)-B(69) | 125.3(3) |
| C(61)-B(63)-B(69) | 104.1(3) | C(63)-B(63)-B(70) | 125.4(3) |
| C(61)-B(63)-B(70) | 104.3(3) | B(69)-B(63)-B(70) | 61.0(2) |
| C(63)-B(63)-B(64) | 119.8(3) | C(61)-B(63)-B(64) | 58.2(2) |
| B(69)-B(63)-B(64) | 60.0(2) | B(70)-B(63)-B(64) | 108.8(3) |
| C(63)-B(63)-B(62) | 120.5(3) | C(61)-B(63)-B(62) | 58.5(2) |
| B(69)-B(63)-B(62) | 108.1(3) | B(70)-B(63)-B(62) | 59.5(2) |
| B(64)-B(63)-B(62) | 107.8(3) | C(64)-B(64)-C(61) | 120.8(3) |
| C(64)-B(64)-B(69) | 125.2(3) | C(61)-B(64)-B(69) | 104.5(3) |
| C(64)-B(64)-B(63) | 119.2(3) | C(61)-B(64)-B(63) | 59.0(2) |
| B(69)-B(64)-B(63) | 59.7(2) | C(64)-B(64)-B(65) | 120.7(3) |
| C(61)-B(64)-B(65) | 58.7(2) | B(69)-B(64)-B(65) | 108.2(3) |
| B(63)-B(64)-B(65) | 108.4(3) | C(64)-B(64)-B(68) | 125.8(3) |
| C(61)-B(64)-B(68) | 104.6(3) | B(69)-B(64)-B(68) | 60.5(2) |
| B(63)-B(64)-B(68) | 108.5(3) | B(65)-B(64)-B(68) | 59.9(2) |
| C(65)-B(65)-C(61) | 120.7(3) | C(65)-B(65)-B(68) | 125.6(4) |
| C(61)-B(65)-B(68) | 104.6(3) | C(65)-B(65)-B(67) | 125.5(3) |
| C(61)-B(65)-B(67) | 104.5(3) | B(68)-B(65)-B(67) | 60.8(2) |
| C(65)-B(65)-B(64) | 120.1(3) | C(61)-B(65)-B(64) | 58.4(2) |
| B(68)-B(65)-B(64) | 60.1(2) | B(67)-B(65)-B(64) | 108.6(3) |
| C(65)-B(65)-B(66) | 119.5(3) | C(61)-B(65)-B(66) | 58.8(2) |
| B(68)-B(65)-B(66) | 108.7(3) | B(67)-B(65)-B(66) | 59.9(2) |
| B(64)-B(65)-B(66) | 108.2(3) | C(66)-B(66)-C(61) | 121.6(3) |
| C(66)-B(66)-B(67) | 125.4(4) | C(61)-B(66)-B(67) | 103.9(3) |
| C(66)-B(66)-B(71) | 125.5(3) | C(61)-B(66)-B(71) | 103.9(3) |
| B(67)-B(66)-B(71) | 60.5(2) | C(66)-B(66)-B(62) | 120.4(4) |
| C(61)-B(66)-B(62) | 58.5(2) | B(67)-B(66)-B(62) | 107.9(3) |
| B(71)-B(66)-B(62) | 59.5(2) | C(66)-B(66)-B(65) | 120.5(4) |
| C(61)-B(66)-B(65) | 58.2(2) | B(67)-B(66)-B(65) | 59.7(2) |
| B(71)-B(66)-B(65) | 107.9(3) | B(62)-B(66)-B(65) | 107.5(3) |
| C(67)-B(67)-B(65) | 120.5(3) | C(67)-B(67)-B(66) | 120.8(3) |
| B(65)-B(67)-B(66) | 60.4(2) | C(67)-B(67)-B(72) | 123.5(3) |
| B(65)-B(67)-B(72) | 107.3(3) | B(66)-B(67)-B(72) | 107.7(3) |
| C(67)-B(67)-B(71) | 122.8(3) | B(65)-B(67)-B(71) | 107.9(3) |
| B(66)-B(67)-B(71) | 59.8(2) | B(72)-B(67)-B(71) | 59.9(2) |
| C(67)-B(67)-B(68) | 121.6(4) | B(65)-B(67)-B(68) | 59.5(2) |
| B(66)-B(67)-B(68) | 108.2(3) | B(72)-B(67)-B(68) | 60.0(2) |

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|---|---|---|---|
| B(71)-B(67)-B(68) | 108.0(3) | C(68)-B(68)-B(65) | 121.3(3) |
| C(68)-B(68)-B(64) | 120.9(3) | B(65)-B(68)-B(64) | 60.0(2) |
| C(68)-B(68)-B(69) | 122.0(3) | B(65)-B(68)-B(69) | 107.6(3) |
| B(64)-B(68)-B(69) | 59.6(2) | C(68)-B(68)-B(72) | 123.3(3) |
| B(65)-B(68)-B(72) | 107.3(3) | B(64)-B(68)-B(72) | 107.2(3) |
| B(69)-B(68)-B(72) | 59.8(2) | C(68)-B(68)-B(67) | 122.6(3) |
| B(65)-B(68)-B(67) | 59.6(2) | B(64)-B(68)-B(67) | 107.6(3) |
| B(69)-B(68)-B(67) | 107.7(3) | B(72)-B(68)-B(67) | 59.9(2) |
| C(69)-B(69)-B(63) | 121.2(3) | C(69)-B(69)-B(64) | 120.5(3) |
| B(63)-B(69)-B(64) | 60.3(2) | C(69)-B(69)-B(72) | 122.8(3) |
| B(63)-B(69)-B(72) | 107.9(3) | B(64)-B(69)-B(72) | 107.9(3) |
| C(69)-B(69)-B(68) | 120.9(3) | B(63)-B(69)-B(68) | 108.4(3) |
| B(64)-B(69)-B(68) | 59.9(2) | B(72)-B(69)-B(68) | 60.3(2) |
| C(69)-B(69)-B(70) | 122.8(3) | B(63)-B(69)-B(70) | 59.6(2) |
| B(64)-B(69)-B(70) | 107.8(3) | B(72)-B(69)-B(70) | 60.2(2) |
| B(68)-B(69)-B(70) | 108.5(3) | C(70)-B(70)-B(62) | 120.6(3) |
| C(70)-B(70)-B(63) | 120.9(3) | B(62)-B(70)-B(63) | 60.8(2) |
| C(70)-B(70)-B(71) | 122.0(3) | B(62)-B(70)-B(71) | 59.7(2) |
| B(63)-B(70)-B(71) | 108.2(3) | C(70)-B(70)-B(72) | 123.4(3) |
| B(62)-B(70)-B(72) | 107.5(3) | B(63)-B(70)-B(72) | 107.3(3) |
| B(71)-B(70)-B(72) | 59.8(2) | C(70)-B(70)-B(69) | 122.6(3) |
| B(62)-B(70)-B(69) | 107.9(3) | B(63)-B(70)-B(69) | 59.4(2) |
| B(71)-B(70)-B(69) | 107.6(3) | B(72)-B(70)-B(69) | 59.6(2) |
| C(107)-B(71)-B(62) | 121.6(3) | C(107)-B(71)-B(66) | 122.1(3) |
| B(62)-B(71)-B(66) | 60.5(3) | C(107)-B(71)-B(72) | 121.8(3) |
| B(62)-B(71)-B(72) | 107.6(3) | B(66)-B(71)-B(72) | 107.7(3) |
| C(107)-B(71)-B(70) | 121.1(3) | B(62)-B(71)-B(70) | 59.5(2) |
| B(66)-B(71)-B(70) | 108.1(3) | B(72)-B(71)-B(70) | 60.2(2) |
| C(107)-B(71)-B(67) | 122.2(3) | B(62)-B(71)-B(67) | 108.0(3) |
| B(66)-B(71)-B(67) | 59.7(3) | B(72)-B(71)-B(67) | 60.0(2) |
| B(70)-B(71)-B(67) | 108.2(3) | C(71)-B(72)-B(69) | 121.8(3) |
| C(71)-B(72)-B(71) | 121.2(3) | B(69)-B(72)-B(71) | 108.2(3) |
| C(71)-B(72)-B(67) | 121.8(3) | B(69)-B(72)-B(67) | 108.0(3) |
| B(71)-B(72)-B(67) | 60.1(2) | C(71)-B(72)-B(68) | 122.0(3) |
| B(69)-B(72)-B(68) | 60.0(2) | B(71)-B(72)-B(68) | 108.3(3) |
| B(67)-B(72)-B(68) | 60.1(2) | C(71)-B(72)-B(70) | 121.3(3) |
| B(69)-B(72)-B(70) | 60.2(2) | B(71)-B(72)-B(70) | 60.0(2) |
| B(67)-B(72)-B(70) | 108.1(3) | B(68)-B(72)-B(70) | 108.3(3) |
| C(81)-B(81)-B(85) | 115.4(6) | C(81)-B(81)-C(06) | 120.5(5) |
| B(85)-B(81)-C(06) | 64.0(6) | C(81)-B(81)-B(82) | 124.1(4) |
| B(85)-B(81)-B(82) | 110.9(5) | C(06)-B(81)-B(82) | 57.7(4) |
| C(81)-B(81)-B(83) | 125.6(4) | B(85)-B(81)-B(83) | 108.9(5) |
| C(06)-B(81)-B(83) | 106.3(5) | B(82)-B(81)-B(83) | 60.5(2) |
| C(81)-B(81)-B(84) | 121.3(3) | B(85)-B(81)-B(84) | 59.4(4) |
| C(06)-B(81)-B(84) | 108.3(5) | B(82)-B(81)-B(84) | 108.2(3) |
| B(83)-B(81)-B(84) | 59.7(2) | C(81)-B(81)-C(05) | 124.5(5) |
| B(85)-B(81)-C(05) | 9.6(5) | C(06)-B(81)-C(05) | 58.0(5) |
| B(82)-B(81)-C(05) | 101.6(4) | B(83)-B(81)-C(05) | 102.5(4) |
| B(84)-B(81)-C(05) | 58.9(3) | C(81)-B(81)-B(86) | 121.6(5) |
| B(85)-B(81)-B(86) | 59.6(5) | C(06)-B(81)-B(86) | 4.4(6) |
| B(82)-B(81)-B(86) | 61.1(4) | B(83)-B(81)-B(86) | 107.2(4) |
| B(84)-B(81)-B(86) | 105.0(4) | C(05)-B(81)-B(86) | 53.7(4) |
| C(82)-B(82)-C(06) | 121.0(5) | C(82)-B(82)-B(81) | 121.0(3) |
| C(06)-B(82)-B(81) | 56.5(4) | C(82)-B(82)-B(88) | 123.8(3) |

| | | | |
|---|---|---|---|
| C(06)-B(82)-B(88) | 108.0(4) | B(81)-B(82)-B(88) | 107.8(3) |
| C(82)-B(82)-B(83) | 123.6(3) | C(06)-B(82)-B(83) | 104.8(4) |
| B(81)-B(82)-B(83) | 59.9(2) | B(88)-B(82)-B(83) | 60.0(2) |
| C(82)-B(82)-B(87) | 121.0(3) | C(06)-B(82)-B(87) | 62.5(4) |
| B(81)-B(82)-B(87) | 108.1(3) | B(88)-B(82)-B(87) | 59.8(2) |
| B(83)-B(82)-B(87) | 108.0(3) | C(82)-B(82)-B(86) | 121.5(5) |
| C(06)-B(82)-B(86) | 6.6(6) | B(81)-B(82)-B(86) | 62.5(4) |
| B(88)-B(82)-B(86) | 104.1(4) | B(83)-B(82)-B(86) | 107.8(4) |
| B(87)-B(82)-B(86) | 56.1(4) | C(83)-B(83)-B(84) | 121.5(3) |
| C(83)-B(83)-B(81) | 120.8(4) | B(84)-B(83)-B(81) | 60.5(2) |
| C(83)-B(83)-B(89) | 122.8(3) | B(84)-B(83)-B(89) | 59.9(2) |
| B(81)-B(83)-B(89) | 108.0(3) | C(83)-B(83)-B(88) | 122.6(4) |
| B(84)-B(83)-B(88) | 107.9(3) | B(81)-B(83)-B(88) | 107.5(3) |
| B(89)-B(83)-B(88) | 60.0(2) | C(83)-B(83)-B(82) | 121.1(3) |
| B(84)-B(83)-B(82) | 108.3(3) | B(81)-B(83)-B(82) | 59.7(2) |
| B(89)-B(83)-B(82) | 107.9(3) | B(88)-B(83)-B(82) | 59.8(2) |
| C(84)-B(84)-B(85) | 117.3(5) | C(84)-B(84)-B(83) | 124.8(4) |
| B(85)-B(84)-B(83) | 105.2(4) | C(84)-B(84)-B(89) | 125.0(3) |
| B(85)-B(84)-B(89) | 110.9(5) | B(83)-B(84)-B(89) | 60.4(2) |
| C(84)-B(84)-B(90) | 120.5(4) | B(85)-B(84)-B(90) | 65.3(4) |
| B(83)-B(84)-B(90) | 108.1(3) | B(89)-B(84)-B(90) | 59.7(2) |
| C(84)-B(84)-B(81) | 120.7(3) | B(85)-B(84)-B(81) | 54.8(4) |
| B(83)-B(84)-B(81) | 59.8(2) | B(89)-B(84)-B(81) | 107.8(3) |
| B(90)-B(84)-B(81) | 107.8(3) | C(84)-B(84)-C(05) | 123.6(4) |
| B(85)-B(84)-C(05) | 12.3(4) | B(83)-B(84)-C(05) | 105.4(4) |
| B(89)-B(84)-C(05) | 100.0(4) | B(90)-B(84)-C(05) | 53.3(3) |
| B(81)-B(84)-C(05) | 62.7(4) | B(81)-B(85)-C(85) | 118.1(9) |
| B(81)-B(85)-B(84) | 65.8(5) | C(85)-B(85)-B(84) | 124.5(8) |
| B(81)-B(85)-C(06) | 58.4(5) | C(85)-B(85)-C(06) | 118.8(8) |
| B(84)-B(85)-C(06) | 109.0(7) | B(81)-B(85)-B(90) | 111.9(6) |
| C(85)-B(85)-B(90) | 125.2(8) | B(84)-B(85)-B(90) | 59.9(4) |
| C(06)-B(85)-B(90) | 105.1(7) | B(81)-B(85)-B(91) | 109.7(6) |
| C(85)-B(85)-B(91) | 120.0(8) | B(84)-B(85)-B(91) | 106.5(6) |
| C(06)-B(85)-B(91) | 60.9(5) | B(90)-B(85)-B(91) | 56.2(4) |
| C(86)-B(86)-B(91) | 121.3(8) | C(86)-B(86)-C(05) | 120.9(8) |
| B(91)-B(86)-C(05) | 55.9(5) | C(86)-B(86)-B(87) | 126.6(8) |
| B(91)-B(86)-B(87) | 63.5(5) | C(05)-B(86)-B(87) | 104.9(7) |
| C(86)-B(86)-B(82) | 124.0(8) | B(91)-B(86)-B(82) | 110.2(6) |
| C(05)-B(86)-B(82) | 104.8(7) | B(87)-B(86)-B(82) | 59.9(4) |
| C(86)-B(86)-B(81) | 118.0(7) | B(91)-B(86)-B(81) | 109.3(6) |
| C(05)-B(86)-B(81) | 62.5(5) | B(87)-B(86)-B(81) | 106.2(6) |
| B(82)-B(86)-B(81) | 56.4(4) | C(87)-B(87)-B(86) | 119.4(5) |
| C(87)-B(87)-B(91) | 120.7(3) | B(86)-B(87)-B(91) | 56.4(4) |
| C(87)-B(87)-B(88) | 123.6(3) | B(86)-B(87)-B(88) | 110.2(5) |
| B(91)-B(87)-B(88) | 107.8(3) | C(87)-B(87)-B(92) | 122.7(3) |
| B(86)-B(87)-B(92) | 106.2(5) | B(91)-B(87)-B(92) | 59.8(2) |
| B(88)-B(87)-B(92) | 60.1(2) | C(87)-B(87)-C(06) | 124.4(5) |
| B(86)-B(87)-C(06) | 9.6(6) | B(91)-B(87)-C(06) | 62.5(4) |
| B(88)-B(87)-C(06) | 101.8(4) | B(92)-B(87)-C(06) | 106.4(4) |
| C(87)-B(87)-B(82) | 121.8(3) | B(86)-B(87)-B(82) | 64.0(5) |
| B(91)-B(87)-B(82) | 108.0(3) | B(88)-B(87)-B(82) | 59.7(2) |
| B(92)-B(87)-B(82) | 108.0(3) | C(06)-B(87)-B(82) | 54.6(4) |
| C(88)-B(88)-B(82) | 120.6(3) | C(88)-B(88)-B(87) | 121.0(3) |
| B(82)-B(88)-B(87) | 60.5(2) | C(88)-B(88)-B(89) | 122.4(3) |

| | | | |
|---|---|---|---|
| B(82)-B(88)-B(89) | 108.2(3) | B(87)-B(88)-B(89) | 108.2(3) |
| C(88)-B(88)-B(83) | 121.4(3) | B(82)-B(88)-B(83) | 60.1(2) |
| B(87)-B(88)-B(83) | 108.6(3) | B(89)-B(88)-B(83) | 60.0(2) |
| C(88)-B(88)-B(92) | 122.0(3) | B(82)-B(88)-B(92) | 108.5(3) |
| B(87)-B(88)-B(92) | 60.0(2) | B(89)-B(88)-B(92) | 60.1(2) |
| B(83)-B(88)-B(92) | 108.4(3) | C(89)-B(89)-B(90) | 120.8(3) |
| C(89)-B(89)-B(84) | 121.3(3) | B(90)-B(89)-B(84) | 60.5(3) |
| C(89)-B(89)-B(88) | 122.4(3) | B(90)-B(89)-B(88) | 108.0(3) |
| B(84)-B(89)-B(88) | 107.9(3) | C(89)-B(89)-B(83) | 122.2(3) |
| B(90)-B(89)-B(83) | 108.3(3) | B(84)-B(89)-B(83) | 59.8(2) |
| B(88)-B(89)-B(83) | 60.0(2) | C(89)-B(89)-B(92) | 121.2(3) |
| B(90)-B(89)-B(92) | 60.0(2) | B(84)-B(89)-B(92) | 108.6(3) |
| B(88)-B(89)-B(92) | 60.1(2) | B(83)-B(89)-B(92) | 108.3(3) |
| C(90)-B(90)-C(05) | 121.2(4) | C(90)-B(90)-B(89) | 124.3(3) |
| C(05)-B(90)-B(89) | 108.1(4) | C(90)-B(90)-B(92) | 123.7(4) |
| C(05)-B(90)-B(92) | 103.2(4) | B(89)-B(90)-B(92) | 60.4(2) |
| C(90)-B(90)-B(84) | 121.0(4) | C(05)-B(90)-B(84) | 63.7(4) |
| B(89)-B(90)-B(84) | 59.8(2) | B(92)-B(90)-B(84) | 108.3(3) |
| C(90)-B(90)-B(91) | 120.5(3) | C(05)-B(90)-B(91) | 54.7(4) |
| B(89)-B(90)-B(91) | 107.9(3) | B(92)-B(90)-B(91) | 59.6(2) |
| B(84)-B(90)-B(91) | 108.1(3) | C(90)-B(90)-B(85) | 122.0(5) |
| C(05)-B(90)-B(85) | 9.3(5) | B(89)-B(90)-B(85) | 102.7(4) |
| B(92)-B(90)-B(85) | 107.5(4) | B(84)-B(90)-B(85) | 54.8(4) |
| B(91)-B(90)-B(85) | 63.2(4) | C(05)-B(91)-C(91) | 120.1(5) |
| C(05)-B(91)-B(86) | 63.4(5) | C(91)-B(91)-B(86) | 116.6(5) |
| C(05)-B(91)-B(87) | 107.9(4) | C(91)-B(91)-B(87) | 123.2(4) |
| B(86)-B(91)-B(87) | 60.1(4) | C(05)-B(91)-B(92) | 104.9(4) |
| C(91)-B(91)-B(92) | 125.7(4) | B(86)-B(91)-B(92) | 109.6(5) |
| B(87)-B(91)-B(92) | 60.4(2) | C(05)-B(91)-B(90) | 56.6(4) |
| C(91)-B(91)-B(90) | 122.7(3) | B(86)-B(91)-B(90) | 109.9(5) |
| B(87)-B(91)-B(90) | 108.0(3) | B(92)-B(91)-B(90) | 59.9(2) |
| C(05)-B(91)-C(06) | 59.1(5) | C(91)-B(91)-C(06) | 123.8(5) |
| B(86)-B(91)-C(06) | 7.5(6) | B(87)-B(91)-C(06) | 59.0(4) |
| B(92)-B(91)-C(06) | 104.1(4) | B(90)-B(91)-C(06) | 102.8(4) |
| C(05)-B(91)-B(85) | 5.4(5) | C(91)-B(91)-B(85) | 122.1(5) |
| B(86)-B(91)-B(85) | 58.0(5) | B(87)-B(91)-B(85) | 103.4(4) |
| B(92)-B(91)-B(85) | 105.6(4) | B(90)-B(91)-B(85) | 60.5(4) |
| C(06)-B(91)-B(85) | 53.7(5) | C(92)-B(92)-B(91) | 121.3(4) |
| C(92)-B(92)-B(90) | 121.3(3) | B(91)-B(92)-B(90) | 60.5(2) |
| C(92)-B(92)-B(87) | 121.9(3) | B(91)-B(92)-B(87) | 59.8(2) |
| B(90)-B(92)-B(87) | 108.1(3) | C(92)-B(92)-B(88) | 122.7(4) |
| B(91)-B(92)-B(88) | 107.5(3) | B(90)-B(92)-B(88) | 107.4(3) |
| B(87)-B(92)-B(88) | 59.8(2) | C(92)-B(92)-B(89) | 122.1(3) |
| B(91)-B(92)-B(89) | 107.9(3) | B(90)-B(92)-B(89) | 59.5(2) |
| B(87)-B(92)-B(89) | 107.8(3) | B(88)-B(92)-B(89) | 59.8(2) |
| C(3A)-C(1A)-C(2A) | 106.8(9) | C(3A)-C(1A)-P(1) | 117.2(6) |
| C(2A)-C(1A)-P(1) | 120.4(8) | C(5A)-C(4A)-C(6A) | 109.9(15) |
| C(5A)-C(4A)-P(1) | 106.6(9) | C(6A)-C(4A)-P(1) | 109.8(12) |
| C(8A)-C(7A)-C(9A) | 130(2) | C(8A)-C(7A)-P(1) | 122.4(11) |
| C(9A)-C(7A)-P(1) | 91.9(13) | | |

Symmetry transformations used to generate equivalent atoms:
#1 -x+1--y+1--z+1----

**Table S4**. Crystal data and structure refinement for **1b.**

| | |
|---|---|
| Identification code | 1b |
| Empirical formula | C124 H167 B2 F48 P6 Rh6 |
| Formula weight | 3394.48 |
| Temperature | 150(2) K |
| Wavelength | 0.71073 A |
| Crystal system | triclinic |
| Space group | P$\bar{1}$ |
| Unit cell dimensions | a = 13.33700(10) A alpha= 73.2250(10) |
| | b = 17.4180(2) A beta = 68.2810(10) |
| | c = 17.8320(2) A gamma = 88.4600(10) |
| Volume | 3669.70(7) A^3 |
| Z, Calculated density | 1, 1.536 Mg/m^3 |
| Absorption coefficient | 0.829 mm^-1 |
| F(000) | 1713 |
| Crystal size | 0.60 x 0.40 x 0.10 mm |
| Theta range for data collection | 4.69 to 30.04 deg. |
| Limiting indices | -18<=h<=18, -24<=k<=24, -25<=l<=25 |
| Reflections collected / unique | 70877 / 21102 [R(int) = 0.0492] |
| Completeness to theta = 30.04 | 98.1 % |
| Max. and min. transmission | 0.9217 and 0.6362 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 21102 / 0 / 1033 |
| Goodness-of-fit on F^2 | 1.027 |
| Final R indices [I>2sigma(I)] | R1 = 0.0351, wR2 = 0.0866 |
| R indices (all data) | R1 = 0.0477, wR2 = 0.0948 |
| Largest diff. peak and hole | 1.002 and -1.138 e.A^-3 |

Table S5. Bond lengths [A] and angle [deg] for 1b.

| | |
|---|---|
| Rh(1)-H(600) | 1.59(4) |
| Rh(1)-H(500) | 1.66(3) |
| Rh(1)-H(100) | 1.82(3) |
| Rh(1)-H(200) | 2.04(3) |
| Rh(1)-P(1) | 2.2461(6) |
| Rh(1)-Rh(3)#1 | 2.7009(2) |
| Rh(1)-Rh(2) | 2.7142(2) |
| Rh(1)-Rh(3) | 2.7209(2) |
| Rh(1)-Rh(2)#1 | 2.7346(2) |
| Rh(2)-H(200) | 1.38(3) |
| Rh(2)-H(300) | 1.70(3) |
| Rh(2)-H(400) | 1.85(4) |
| Rh(2)-H(600)#1 | 1.93(4) |
| Rh(2)-P(2) | 2.2450(6) |
| Rh(2)-Rh(3)#1 | 2.7106(2) |
| Rh(2)-Rh(3) | 2.7303(2) |
| Rh(2)-Rh(1)#1 | 2.7346(2) |
| Rh(3)-H(400)#1 | 1.64(4) |
| Rh(3)-H(100) | 1.66(3) |
| Rh(3)-H(500)#1 | 1.86(3) |
| Rh(3)-H(300) | 1.96(3) |
| Rh(3)-P(3) | 2.2444(6) |
| Rh(3)-Rh(1)#1 | 2.7009(2) |
| Rh(3)-Rh(2)#1 | 2.7106(2) |
| P(1)-C(1) | 1.843(3) |
| P(1)-C(4) | 1.849(3) |
| P(1)-C(7) | 1.858(3) |
| P(2)-C(16) | 1.854(2) |
| P(2)-C(13) | 1.855(3) |
| P(2)-C(10) | 1.855(3) |
| P(3)-C(19) | 1.842(3) |
| P(3)-C(22) | 1.847(2) |
| P(3)-C(25) | 1.854(2) |
| C(1)-C(2) | 1.536(4) |
| C(1)-C(3) | 1.538(4) |
| C(4)-C(6) | 1.524(4) |
| C(4)-C(5) | 1.531(4) |
| C(7)-C(9) | 1.533(4) |
| C(7)-C(8) | 1.534(3) |
| C(10)-C(12) | 1.524(4) |
| C(10)-C(11) | 1.533(4) |
| C(13)-C(14) | 1.533(4) |
| C(13)-C(15) | 1.536(4) |
| C(16)-C(18) | 1.525(4) |
| C(16)-C(17) | 1.526(4) |
| C(19)-C(21) | 1.527(4) |
| C(19)-C(20) | 1.533(4) |
| C(22)-C(23) | 1.526(4) |
| C(22)-C(24) | 1.526(4) |
| C(25)-C(26) | 1.527(4) |
| C(25)-C(27) | 1.540(4) |
| B(1)-C(52) | 1.636(3) |

| | |
|---|---|
| B(1)-C(36) | 1.640(4) |
| B(1)-C(44) | 1.643(4) |
| B(1)-C(28) | 1.645(3) |
| C(28)-C(33) | 1.395(3) |
| C(28)-C(29) | 1.404(3) |
| C(29)-C(30) | 1.381(3) |
| C(30)-C(31) | 1.386(3) |
| C(30)-C(34) | 1.499(3) |
| C(31)-C(32) | 1.388(3) |
| C(32)-C(33) | 1.391(3) |
| C(32)-C(35) | 1.501(3) |
| C(34)-F(3) | 1.313(4) |
| C(34)-F(1) | 1.320(3) |
| C(34)-F(2) | 1.338(3) |
| C(35)-F(4) | 1.322(3) |
| C(35)-F(5) | 1.333(3) |
| C(35)-F(6) | 1.343(3) |
| C(36)-C(37) | 1.394(3) |
| C(36)-C(41) | 1.408(4) |
| C(37)-C(38) | 1.394(4) |
| C(38)-C(39) | 1.378(4) |
| C(38)-C(42) | 1.510(4) |
| C(39)-C(40) | 1.392(4) |
| C(40)-C(41) | 1.385(4) |
| C(40)-C(43) | 1.504(4) |
| C(42)-F(7) | 1.173(12) |
| C(42)-F(8) | 1.286(11) |
| C(42)-F(9A) | 1.289(12) |
| C(42)-F(9) | 1.324(9) |
| C(42)-F(8A) | 1.352(10) |
| C(42)-F(7A) | 1.444(10) |
| C(43)-F(12) | 1.308(5) |
| C(43)-F(10) | 1.320(4) |
| C(43)-F(11) | 1.331(6) |
| C(44)-C(45) | 1.401(3) |
| C(44)-C(49) | 1.402(3) |
| C(45)-C(46) | 1.392(3) |
| C(46)-C(47) | 1.384(4) |
| C(46)-C(50) | 1.495(4) |
| C(47)-C(48) | 1.383(4) |
| C(48)-C(49) | 1.392(3) |
| C(48)-C(51) | 1.500(4) |
| C(50)-F(14A) | 1.259(8) |
| C(50)-F(13) | 1.282(11) |
| C(50)-F(15A) | 1.290(12) |
| C(50)-F(15) | 1.335(11) |
| C(50)-F(13A) | 1.350(13) |
| C(50)-F(14) | 1.396(9) |
| C(51)-F(17) | 1.274(13) |
| C(51)-F(18A) | 1.281(18) |
| C(51)-F(16) | 1.301(9) |
| C(51)-F(17A) | 1.338(14) |
| C(51)-F(18) | 1.36(2) |
| C(51)-F(16A) | 1.387(9) |
| C(52)-C(57) | 1.397(4) |
| C(52)-C(53) | 1.398(3) |

| | |
|---|---|
| C(53)-C(54) | 1.392(4) |
| C(54)-C(55) | 1.382(4) |
| C(54)-C(58) | 1.498(5) |
| C(55)-C(56) | 1.368(4) |
| C(56)-C(57) | 1.392(4) |
| C(56)-C(59) | 1.514(5) |
| C(58)-F(19A) | 1.16(2) |
| C(58)-F(21A) | 1.231(8) |
| C(58)-F(20) | 1.292(15) |
| C(58)-F(20A) | 1.327(18) |
| C(58)-F(19) | 1.360(18) |
| C(58)-F(21) | 1.392(10) |
| C(59)-F(23A) | 1.222(15) |
| C(59)-F(22A) | 1.256(15) |
| C(59)-F(24) | 1.257(9) |
| C(59)-F(22) | 1.33(2) |
| C(59)-F(23) | 1.357(16) |
| C(59)-F(24A) | 1.378(14) |
| C(60)-C(61) | 1.254(11) |
| C(60)-F(30) | 1.337(16) |
| C(60)-C(62)#2 | 1.467(13) |
| F(30A)-C(61) | 1.39(2) |
| C(61)-C(62) | 1.331(15) |
| C(62)-C(60)#2 | 1.467(13) |

| | |
|---|---|
| H(600)-Rh(1)-H(500) | 88.1(18) |
| H(600)-Rh(1)-H(100) | 90.6(17) |
| H(500)-Rh(1)-H(100) | 169.1(16) |
| H(600)-Rh(1)-H(200) | 160.5(16) |
| H(500)-Rh(1)-H(200) | 82.0(14) |
| H(100)-Rh(1)-H(200) | 96.0(14) |
| H(600)-Rh(1)-P(1) | 89.4(13) |
| H(500)-Rh(1)-P(1) | 89.9(11) |
| H(100)-Rh(1)-P(1) | 100.9(11) |
| H(200)-Rh(1)-P(1) | 107.3(8) |
| H(600)-Rh(1)-Rh(3)#1 | 88.5(13) |
| H(500)-Rh(1)-Rh(3)#1 | 42.7(11) |
| H(100)-Rh(1)-Rh(3)#1 | 126.4(11) |
| H(200)-Rh(1)-Rh(3)#1 | 72.8(8) |
| P(1)-Rh(1)-Rh(3)#1 | 132.657(17) |
| H(600)-Rh(1)-Rh(2) | 134.1(13) |
| H(500)-Rh(1)-Rh(2) | 88.9(12) |
| H(100)-Rh(1)-Rh(2) | 84.2(11) |
| H(200)-Rh(1)-Rh(2) | 29.6(8) |
| P(1)-Rh(1)-Rh(2) | 136.395(17) |
| Rh(3)#1-Rh(1)-Rh(2) | 60.074(6) |
| H(600)-Rh(1)-Rh(3) | 89.7(14) |
| H(500)-Rh(1)-Rh(3) | 132.6(11) |
| H(100)-Rh(1)-Rh(3) | 36.6(11) |
| H(200)-Rh(1)-Rh(3) | 84.8(8) |
| P(1)-Rh(1)-Rh(3) | 137.449(17) |
| Rh(3)#1-Rh(1)-Rh(3) | 89.832(7) |

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| Rh(2)-Rh(1)-Rh(3) | 60.309(6) |
| H(600)-Rh(1)-Rh(2)#1 | 43.8(13) |
| H(500)-Rh(1)-Rh(2)#1 | 88.1(12) |
| H(100)-Rh(1)-Rh(2)#1 | 83.5(11) |
| H(200)-Rh(1)-Rh(2)#1 | 118.7(8) |
| P(1)-Rh(1)-Rh(2)#1 | 133.121(17) |
| Rh(3)#1-Rh(1)-Rh(2)#1 | 60.301(6) |
| Rh(2)-Rh(1)-Rh(2)#1 | 90.399(7) |
| Rh(3)-Rh(1)-Rh(2)#1 | 59.585(6) |
| H(200)-Rh(2)-H(300) | 102.3(17) |
| H(200)-Rh(2)-H(400) | 76.8(17) |
| H(300)-Rh(2)-H(400) | 171.2(17) |
| H(200)-Rh(2)-H(600)#1 | 165.5(17) |
| H(300)-Rh(2)-H(600)#1 | 88.5(16) |
| H(400)-Rh(2)-H(600)#1 | 91.0(16) |
| H(200)-Rh(2)-P(2) | 85.9(13) |
| H(300)-Rh(2)-P(2) | 88.7(12) |
| H(400)-Rh(2)-P(2) | 99.9(12) |
| H(600)#1-Rh(2)-P(2) | 104.3(11) |
| H(200)-Rh(2)-Rh(3)#1 | 82.0(12) |
| H(300)-Rh(2)-Rh(3)#1 | 134.9(12) |
| H(400)-Rh(2)-Rh(3)#1 | 36.4(12) |
| H(600)#1-Rh(2)-Rh(3)#1 | 83.5(11) |
| P(2)-Rh(2)-Rh(3)#1 | 136.304(17) |
| H(200)-Rh(2)-Rh(1) | 47.1(13) |
| H(300)-Rh(2)-Rh(1) | 90.2(11) |
| H(400)-Rh(2)-Rh(1) | 82.7(12) |
| H(600)#1-Rh(2)-Rh(1) | 124.3(11) |
| P(2)-Rh(2)-Rh(1) | 131.388(17) |
| Rh(3)#1-Rh(2)-Rh(1) | 59.720(6) |
| H(200)-Rh(2)-Rh(3) | 99.2(13) |
| H(300)-Rh(2)-Rh(3) | 45.4(12) |
| H(400)-Rh(2)-Rh(3) | 125.8(12) |
| H(600)#1-Rh(2)-Rh(3) | 81.3(11) |
| P(2)-Rh(2)-Rh(3) | 134.036(17) |
| Rh(3)#1-Rh(2)-Rh(3) | 89.433(7) |
| Rh(1)-Rh(2)-Rh(3) | 59.967(6) |
| H(200)-Rh(2)-Rh(1)#1 | 134.3(13) |
| H(300)-Rh(2)-Rh(1)#1 | 89.7(12) |
| H(400)-Rh(2)-Rh(1)#1 | 85.0(12) |
| H(600)#1-Rh(2)-Rh(1)#1 | 34.7(11) |
| P(2)-Rh(2)-Rh(1)#1 | 138.978(17) |
| Rh(3)#1-Rh(2)-Rh(1)#1 | 59.957(6) |
| Rh(1)-Rh(2)-Rh(1)#1 | 89.601(7) |
| Rh(3)-Rh(2)-Rh(1)#1 | 59.239(6) |
| H(400)#1-Rh(3)-H(100) | 91.5(18) |
| H(400)#1-Rh(3)-H(500)#1 | 88.8(17) |
| H(100)-Rh(3)-H(500)#1 | 168.2(16) |
| H(400)#1-Rh(3)-H(300) | 170.9(17) |
| H(100)-Rh(3)-H(300) | 89.2(16) |
| H(500)#1-Rh(3)-H(300) | 88.7(14) |
| H(400)#1-Rh(3)-P(3) | 89.9(13) |
| H(100)-Rh(3)-P(3) | 92.1(12) |
| H(500)#1-Rh(3)-P(3) | 99.7(10) |
| H(300)-Rh(3)-P(3) | 99.2(10) |
| H(400)#1-Rh(3)-Rh(1)#1 | 87.0(13) |

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| H(100)-Rh(3)-Rh(1)#1 | 130.9(12) |
| H(500)#1-Rh(3)-Rh(1)#1 | 37.3(10) |
| H(300)-Rh(3)-Rh(1)#1 | 85.7(10) |
| P(3)-Rh(3)-Rh(1)#1 | 136.923(17) |
| H(400)#1-Rh(3)-Rh(2)#1 | 42.1(13) |
| H(100)-Rh(3)-Rh(2)#1 | 87.2(12) |
| H(500)#1-Rh(3)-Rh(2)#1 | 85.1(10) |
| H(300)-Rh(3)-Rh(2)#1 | 128.9(10) |
| P(3)-Rh(3)-Rh(2)#1 | 131.845(17) |
| Rh(1)#1-Rh(3)-Rh(2)#1 | 60.206(6) |
| H(400)#1-Rh(3)-Rh(1) | 89.5(13) |
| H(100)-Rh(3)-Rh(1) | 40.7(12) |
| H(500)#1-Rh(3)-Rh(1) | 127.5(10) |
| H(300)-Rh(3)-Rh(1) | 85.0(10) |
| P(3)-Rh(3)-Rh(1) | 132.788(17) |
| Rh(1)#1-Rh(3)-Rh(1) | 90.168(7) |
| Rh(2)#1-Rh(3)-Rh(1) | 60.458(6) |
| H(400)#1-Rh(3)-Rh(2) | 132.6(13) |
| H(100)-Rh(3)-Rh(2) | 86.6(12) |
| H(500)#1-Rh(3)-Rh(2) | 84.5(10) |
| H(300)-Rh(3)-Rh(2) | 38.4(10) |
| P(3)-Rh(3)-Rh(2) | 137.497(17) |
| Rh(1)#1-Rh(3)-Rh(2) | 60.460(6) |
| Rh(2)#1-Rh(3)-Rh(2) | 90.567(7) |
| Rh(1)-Rh(3)-Rh(2) | 59.724(6) |
| C(1)-P(1)-C(4) | 104.34(13) |
| C(1)-P(1)-C(7) | 104.29(12) |
| C(4)-P(1)-C(7) | 110.64(12) |
| C(1)-P(1)-Rh(1) | 112.93(8) |
| C(4)-P(1)-Rh(1) | 113.63(8) |
| C(7)-P(1)-Rh(1) | 110.49(8) |
| C(16)-P(2)-C(13) | 103.40(12) |
| C(16)-P(2)-C(10) | 104.56(12) |
| C(13)-P(2)-C(10) | 111.31(13) |
| C(16)-P(2)-Rh(2) | 111.10(8) |
| C(13)-P(2)-Rh(2) | 113.15(8) |
| C(10)-P(2)-Rh(2) | 112.59(9) |
| C(19)-P(3)-C(22) | 104.43(12) |
| C(19)-P(3)-C(25) | 103.41(12) |
| C(22)-P(3)-C(25) | 111.71(12) |
| C(19)-P(3)-Rh(3) | 112.60(8) |
| C(22)-P(3)-Rh(3) | 111.60(8) |
| C(25)-P(3)-Rh(3) | 112.54(8) |
| C(2)-C(1)-C(3) | 110.9(2) |
| C(2)-C(1)-P(1) | 111.11(18) |
| C(3)-C(1)-P(1) | 111.80(19) |
| C(6)-C(4)-C(5) | 109.9(2) |
| C(6)-C(4)-P(1) | 114.09(19) |
| C(5)-C(4)-P(1) | 114.6(2) |
| C(9)-C(7)-C(8) | 109.9(2) |
| C(9)-C(7)-P(1) | 112.93(18) |
| C(8)-C(7)-P(1) | 116.3(2) |
| C(12)-C(10)-C(11) | 109.8(3) |
| C(12)-C(10)-P(2) | 115.6(2) |
| C(11)-C(10)-P(2) | 114.38(18) |
| C(14)-C(13)-C(15) | 110.3(2) |

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| C(14)-C(13)-P(2) | 113.0(2) |
| C(15)-C(13)-P(2) | 116.4(2) |
| C(18)-C(16)-C(17) | 110.7(2) |
| C(18)-C(16)-P(2) | 111.96(19) |
| C(17)-C(16)-P(2) | 110.96(17) |
| C(21)-C(19)-C(20) | 110.5(3) |
| C(21)-C(19)-P(3) | 111.41(18) |
| C(20)-C(19)-P(3) | 112.17(18) |
| C(23)-C(22)-C(24) | 109.4(3) |
| C(23)-C(22)-P(3) | 114.07(19) |
| C(24)-C(22)-P(3) | 115.75(19) |
| C(26)-C(25)-C(27) | 110.8(2) |
| C(26)-C(25)-P(3) | 113.06(18) |
| C(27)-C(25)-P(3) | 115.77(19) |
| C(52)-B(1)-C(36) | 105.05(19) |
| C(52)-B(1)-C(44) | 112.87(19) |
| C(36)-B(1)-C(44) | 113.70(19) |
| C(52)-B(1)-C(28) | 112.97(19) |
| C(36)-B(1)-C(28) | 109.38(19) |
| C(44)-B(1)-C(28) | 103.05(18) |
| C(33)-C(28)-C(29) | 115.7(2) |
| C(33)-C(28)-B(1) | 124.3(2) |
| C(29)-C(28)-B(1) | 119.9(2) |
| C(30)-C(29)-C(28) | 122.4(2) |
| C(29)-C(30)-C(31) | 121.2(2) |
| C(29)-C(30)-C(34) | 118.4(2) |
| C(31)-C(30)-C(34) | 120.4(2) |
| C(30)-C(31)-C(32) | 117.4(2) |
| C(31)-C(32)-C(33) | 121.3(2) |
| C(31)-C(32)-C(35) | 120.3(2) |
| C(33)-C(32)-C(35) | 118.4(2) |
| C(32)-C(33)-C(28) | 121.9(2) |
| F(3)-C(34)-F(1) | 106.7(3) |
| F(3)-C(34)-F(2) | 106.3(3) |
| F(1)-C(34)-F(2) | 106.3(2) |
| F(3)-C(34)-C(30) | 112.4(2) |
| F(1)-C(34)-C(30) | 113.1(2) |
| F(2)-C(34)-C(30) | 111.6(2) |
| F(4)-C(35)-F(5) | 106.8(2) |
| F(4)-C(35)-F(6) | 107.5(2) |
| F(5)-C(35)-F(6) | 104.1(2) |
| F(4)-C(35)-C(32) | 113.5(2) |
| F(5)-C(35)-C(32) | 112.6(2) |
| F(6)-C(35)-C(32) | 111.8(2) |
| C(37)-C(36)-C(41) | 115.9(2) |
| C(37)-C(36)-B(1) | 124.1(2) |
| C(41)-C(36)-B(1) | 119.9(2) |
| C(36)-C(37)-C(38) | 122.1(2) |
| C(39)-C(38)-C(37) | 121.2(2) |
| C(39)-C(38)-C(42) | 119.7(3) |
| C(37)-C(38)-C(42) | 119.1(3) |
| C(38)-C(39)-C(40) | 117.9(3) |
| C(41)-C(40)-C(39) | 121.0(3) |
| C(41)-C(40)-C(43) | 118.9(3) |
| C(39)-C(40)-C(43) | 120.0(3) |
| C(40)-C(41)-C(36) | 122.0(2) |

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| F(7)-C(42)-F(8) | 104.3(9) |
| F(7)-C(42)-F(9A) | 91.7(11) |
| F(8)-C(42)-F(9A) | 118.2(9) |
| F(7)-C(42)-F(9) | 110.7(9) |
| F(8)-C(42)-F(9) | 104.6(8) |
| F(9A)-C(42)-F(9) | 20.5(11) |
| F(7)-C(42)-F(8A) | 109.4(9) |
| F(8)-C(42)-F(8A) | 7.9(10) |
| F(9A)-C(42)-F(8A) | 111.4(9) |
| F(9)-C(42)-F(8A) | 96.9(8) |
| F(7)-C(42)-F(7A) | 9.5(10) |
| F(8)-C(42)-F(7A) | 97.9(6) |
| F(9A)-C(42)-F(7A) | 100.9(9) |
| F(9)-C(42)-F(7A) | 119.5(7) |
| F(8A)-C(42)-F(7A) | 103.7(6) |
| F(7)-C(42)-C(38) | 113.4(7) |
| F(8)-C(42)-C(38) | 112.4(6) |
| F(9A)-C(42)-C(38) | 114.4(6) |
| F(9)-C(42)-C(38) | 110.9(5) |
| F(8A)-C(42)-C(38) | 114.3(6) |
| F(7A)-C(42)-C(38) | 110.6(5) |
| F(12)-C(43)-F(10) | 107.5(3) |
| F(12)-C(43)-F(11) | 105.7(4) |
| F(10)-C(43)-F(11) | 105.0(4) |
| F(12)-C(43)-C(40) | 112.5(3) |
| F(10)-C(43)-C(40) | 113.5(3) |
| F(11)-C(43)-C(40) | 112.1(3) |
| C(45)-C(44)-C(49) | 115.3(2) |
| C(45)-C(44)-B(1) | 121.7(2) |
| C(49)-C(44)-B(1) | 122.5(2) |
| C(46)-C(45)-C(44) | 122.4(2) |
| C(47)-C(46)-C(45) | 120.9(2) |
| C(47)-C(46)-C(50) | 120.1(2) |
| C(45)-C(46)-C(50) | 118.9(2) |
| C(48)-C(47)-C(46) | 117.8(2) |
| C(47)-C(48)-C(49) | 121.1(2) |
| C(47)-C(48)-C(51) | 119.4(2) |
| C(49)-C(48)-C(51) | 119.4(2) |
| C(48)-C(49)-C(44) | 122.3(2) |
| F(14A)-C(50)-F(13) | 82.2(9) |
| F(14A)-C(50)-F(15A) | 111.7(8) |
| F(13)-C(50)-F(15A) | 114.5(8) |
| F(14A)-C(50)-F(15) | 122.0(7) |
| F(13)-C(50)-F(15) | 103.2(8) |
| F(15A)-C(50)-F(15) | 15.2(10) |
| F(14A)-C(50)-F(13A) | 97.5(8) |
| F(13)-C(50)-F(13A) | 15.4(14) |
| F(15A)-C(50)-F(13A) | 106.7(9) |
| F(15)-C(50)-F(13A) | 93.4(8) |
| F(14A)-C(50)-F(14) | 30.4(6) |
| F(13)-C(50)-F(14) | 111.2(10) |
| F(15A)-C(50)-F(14) | 90.9(9) |
| F(15)-C(50)-F(14) | 104.9(8) |
| F(13A)-C(50)-F(14) | 125.9(9) |
| F(14A)-C(50)-C(46) | 115.9(4) |
| F(13)-C(50)-C(46) | 114.3(6) |

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| F(15A)-C(50)-C(46) | 114.3(6) |
| F(15)-C(50)-C(46) | 113.6(6) |
| F(13A)-C(50)-C(46) | 108.9(7) |
| F(14)-C(50)-C(46) | 109.1(4) |
| F(17)-C(51)-F(18A) | 114.9(9) |
| F(17)-C(51)-F(16) | 112.2(9) |
| F(18A)-C(51)-F(16) | 80.6(10) |
| F(17)-C(51)-F(17A) | 11.7(13) |
| F(18A)-C(51)-F(17A) | 108.6(10) |
| F(16)-C(51)-F(17A) | 122.0(8) |
| F(17)-C(51)-F(18) | 102.1(10) |
| F(18A)-C(51)-F(18) | 21.8(15) |
| F(16)-C(51)-F(18) | 102.0(8) |
| F(17A)-C(51)-F(18) | 93.2(10) |
| F(17)-C(51)-F(16A) | 88.7(8) |
| F(18A)-C(51)-F(16A) | 109.5(9) |
| F(16)-C(51)-F(16A) | 31.3(5) |
| F(17A)-C(51)-F(16A) | 100.2(8) |
| F(18)-C(51)-F(16A) | 129.0(8) |
| F(17)-C(51)-C(48) | 115.0(6) |
| F(18A)-C(51)-C(48) | 115.2(7) |
| F(16)-C(51)-C(48) | 114.5(5) |
| F(17A)-C(51)-C(48) | 112.0(6) |
| F(18)-C(51)-C(48) | 109.4(8) |
| F(16A)-C(51)-C(48) | 110.3(4) |
| C(57)-C(52)-C(53) | 114.8(2) |
| C(57)-C(52)-B(1) | 122.3(2) |
| C(53)-C(52)-B(1) | 122.5(2) |
| C(54)-C(53)-C(52) | 122.5(2) |
| C(55)-C(54)-C(53) | 121.0(3) |
| C(55)-C(54)-C(58) | 119.1(3) |
| C(53)-C(54)-C(58) | 120.0(3) |
| C(56)-C(55)-C(54) | 117.9(3) |
| C(55)-C(56)-C(57) | 121.1(3) |
| C(55)-C(56)-C(59) | 119.7(3) |
| C(57)-C(56)-C(59) | 119.1(3) |
| C(56)-C(57)-C(52) | 122.7(3) |
| F(19A)-C(58)-F(21A) | 115.2(12) |
| F(19A)-C(58)-F(20) | 115.2(13) |
| F(21A)-C(58)-F(20) | 74.1(12) |
| F(19A)-C(58)-F(20A) | 101.3(13) |
| F(21A)-C(58)-F(20A) | 97.5(13) |
| F(20)-C(58)-F(20A) | 23.8(18) |
| F(19A)-C(58)-F(19) | 12.7(16) |
| F(21A)-C(58)-F(19) | 123.5(9) |
| F(20)-C(58)-F(19) | 106.7(12) |
| F(20A)-C(58)-F(19) | 90.3(12) |
| F(19A)-C(58)-F(21) | 86.3(12) |
| F(21A)-C(58)-F(21) | 41.2(9) |
| F(20)-C(58)-F(21) | 113.1(13) |
| F(20A)-C(58)-F(21) | 134.0(11) |
| F(19)-C(58)-F(21) | 98.5(10) |
| F(19A)-C(58)-C(54) | 115.9(10) |
| F(21A)-C(58)-C(54) | 116.0(5) |
| F(20)-C(58)-C(54) | 113.8(8) |
| F(20A)-C(58)-C(54) | 107.5(10) |

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| F(19)-C(58)-C(54) | 114.4(7) |
| F(21)-C(58)-C(54) | 109.4(5) |
| F(23A)-C(59)-F(22A) | 115.9(15) |
| F(23A)-C(59)-F(24) | 124.3(9) |
| F(22A)-C(59)-F(24) | 59.1(13) |
| F(23A)-C(59)-F(22) | 30(2) |
| F(22A)-C(59)-F(22) | 137.7(12) |
| F(24)-C(59)-F(22) | 111.8(13) |
| F(23A)-C(59)-F(23) | 73.0(14) |
| F(22A)-C(59)-F(23) | 51.3(14) |
| F(24)-C(59)-F(23) | 105.8(12) |
| F(22)-C(59)-F(23) | 103.1(12) |
| F(23A)-C(59)-F(24A) | 102.3(14) |
| F(22A)-C(59)-F(24A) | 100.2(16) |
| F(24)-C(59)-F(24A) | 41.6(13) |
| F(22)-C(59)-F(24A) | 77.2(11) |
| F(23)-C(59)-F(24A) | 138.0(8) |
| F(23A)-C(59)-C(56) | 116.4(8) |
| F(22A)-C(59)-C(56) | 111.3(8) |
| F(24)-C(59)-C(56) | 115.5(6) |
| F(22)-C(59)-C(56) | 109.3(10) |
| F(23)-C(59)-C(56) | 110.6(7) |
| F(24A)-C(59)-C(56) | 108.6(7) |
| C(61)-C(60)-F(30) | 122.6(16) |
| C(61)-C(60)-C(62)#2 | 117.3(10) |
| F(30)-C(60)-C(62)#2 | 119.9(12) |
| C(60)-C(61)-C(62) | 120.8(14) |
| C(60)-C(61)-F(30A) | 129.9(18) |
| C(62)-C(61)-F(30A) | 109.1(12) |
| C(61)-C(62)-C(60)#2 | 121.9(9) |

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y,-z+2    #2 -x+1,-y+1,-z+1

## Analysis of hydride positions in 1a and 1b using HYDEX:

Using HYDEX [5] the best fit for the 12 hydrides confirmed from $^{1}H$ NMR experiments in **1a** and **1b** is with 12 edge bridging hydrides. This gives a low potential energy minimum and there are no short H...H contacts. For **1a** one of the hydrides (H12/H13) has been assigned as terminal split with 50% occupancy over two sites, from HYDEX it models happily as a single edge bridging hydride. Figure S4 shows the structure with the 12 hydrides in calculated positions. Notable is that it is very similar to both the experimentally observed X-ray structures apart from the two half occupancy terminal hydrides in **1a**. Attempts to fit 8 face capping hydrides and then the other four hydrides as terminal did not work. Placing the hydrides as 12 terminal hydrides results in the experimentally observed structure with 12 edge bridging hydrides.

(5) Orpen, A. G; *Dalton Trans*., **1980**, 2509

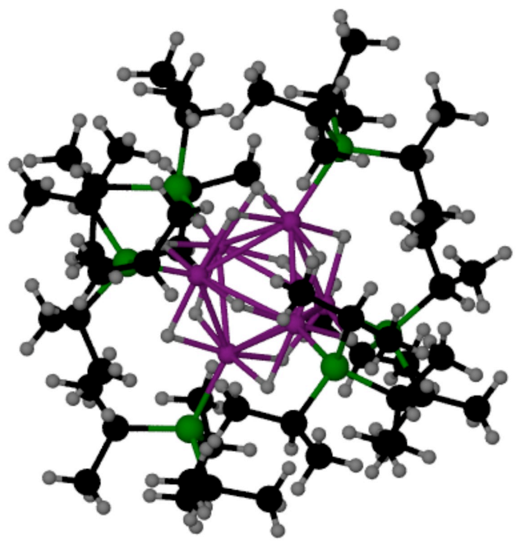

**Figure S4**. Result of HYDEX analysis of the twelve hydrides on clusters **1a** and **1b** showing the (experimentally also observed) 12 bridging hydride positions.