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from iucrdata.iucr.org

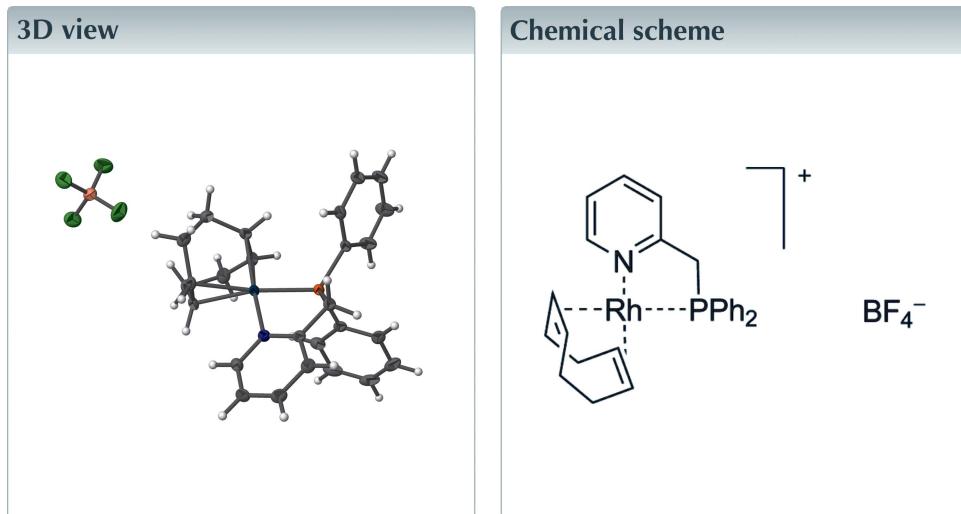
(η^2,η^2 -Cycloocta-1,5-diene)[2-(diphenylphosphanyl-methyl)pyridine- κ^2N,P]rhodium(I) tetrafluorido-borate 1,2-dichloroethane monosolvate

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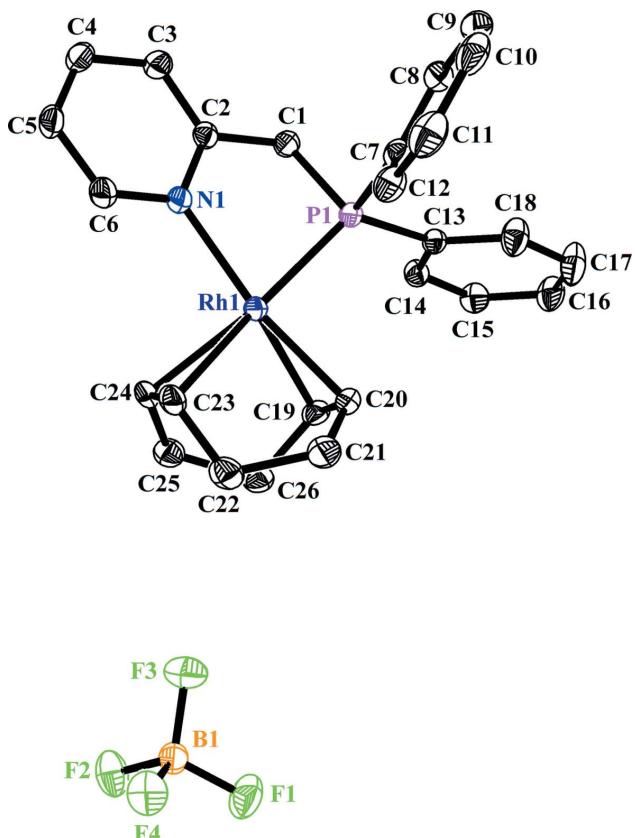
The title compound, $[\text{Rh}(\text{C}_8\text{H}_{12})(\text{C}_{18}\text{H}_{16}\text{NP})]\text{BF}_4$ has been prepared as a precatalyst for applications in rhodium-catalysed additions of carbocyclic acids to terminal alkynes leading to *anti*-Markovnikov Z-enol esters. Here the triclinic pseudopolymorph of the title compound is presented. In contrast to the earlier reported pseudopolymorph (orthorhombic space group) [Wei *et al.* (2013). *Chem. Eur. J.* **19**, 12067–12076], the triclinic polymorph contains half a molecule of dichloromethane as solvent in the asymmetric unit. The rhodium(I) atom exhibits a square-planar coordination. The estimated diffraction contribution of the disordered solvent (a half molecule of dichloroethane per asymmetric unit) was subtracted from the observed diffraction data using the SQUEEZE [Spek (2015). *Acta Cryst. C* **71**, 9–16] routine in PLATON. The given chemical formula and other crystal data do not take the solvent into account.



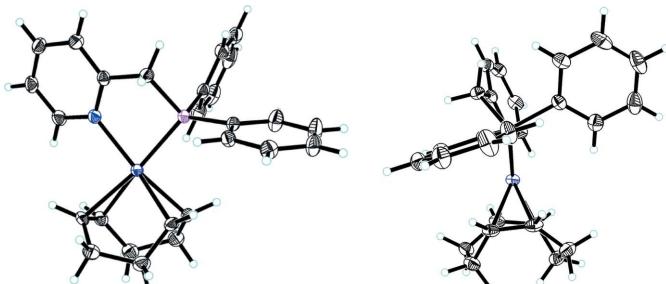
Structure description

The title compound was formed by the exchange of acetylacetone (acac) by dppmp in presence of HBF_4 starting from the precursor $[\text{Rh}(\text{acac})(\text{COD})]$ (Fennis *et al.*, 1990; Wei *et al.*, 2013). Two pseudo-polymorphs (triclinic, space group $P\bar{1}$ with dichloromethane as solvent; orthorhombic, space group $Pbca$, no solvent) of the title compound were observed, of which the triclinic one is presented here (Fig. 1).

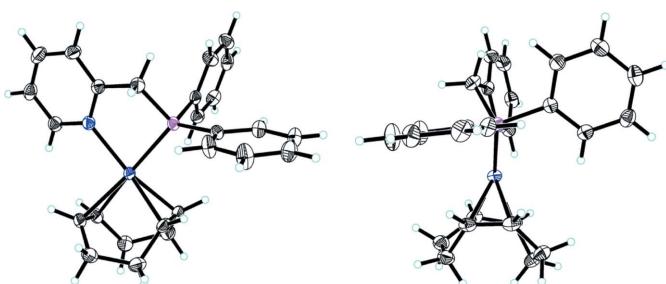
The main difference to the earlier reported pseudo polymorph (Wei *et al.*, 2013; CCDC 914750) is the half molecule of a disordered 1,2-dichloroethane solvate. On the other hand, there are only minor differences in the conformation of the complex cations (Figs. 2 and 3) and the bond lengths and angles are nearly equal (Table 1). An important structural feature is the square-planar coordination of the rhodium(I) atom. The dihedral

**Figure 1**

The molecular structure of the title compound, with atom labelling and displacement ellipsoids drawn at the 50% probability level. All H atoms have been omitted for clarity.

**Figure 2**

Front view (left) and side view (right) of triclinic polymorph of $[\text{Rh}(\text{COD})(\text{dppmp})]^+$ (ellipsoids drawn at the 50% probability level).

**Figure 3**

Front view (left) and side view (right) of pseudopolymorph of $[\text{Rh}(\text{COD})(\text{dppmp})]^+$ (ellipsoids drawn at the 50% probability level).

Table 1
Selected distances and angles (\AA , $^\circ$) of the two polymorphs of $[\text{Rh}(\text{COD})(\text{dppmp})]\text{BF}_4$.

C_M are the centroids of the COD double bonds.

| | Triclinic polymorph | Orthorhombic pseudopolymorph |
|------------------|----------------------|------------------------------|
| Rh—P | 2.2371 (4) | 2.2491 (5) |
| Rh—N | 2.1295 (14) | 2.1333 (17) |
| Rh— C_M | 2.019 (2), 2.140 (2) | 2.119 (2), 2.140 (2) |
| P—Rh—N | 80.58 (4) | 80.81 (5) |
| C_M —Rh— C_M | 86.48 (7) | 86.47 (8) |

Table 2
Experimental details.

| | |
|--|--|
| Crystal data | |
| Chemical formula | $[\text{Rh}(\text{C}_8\text{H}_{12})(\text{C}_{18}\text{H}_{16}\text{NP})]\text{BF}_4$ |
| M_r | 575.18 |
| Crystal system, space group | Triclinic, $P\bar{1}$ |
| Temperature (K) | 150 |
| a, b, c (\AA) | 8.7638 (2), 9.2286 (2), 16.7192 (3) |
| α, β, γ ($^\circ$) | 89.402 (1), 88.904 (1), 76.121 (1) |
| V (\AA^3) | 1312.47 (5) |
| Z | 2 |
| Radiation type | Mo $K\alpha$ |
| μ (mm^{-1}) | 0.75 |
| Crystal size (mm) | 0.46 \times 0.40 \times 0.21 |
| Data collection | |
| Diffractometer | Bruker APEXII CCD |
| Absorption correction | Multi-scan (SADABS; Bruker, 2014) |
| T_{\min}, T_{\max} | 0.639, 0.746 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 17088, 5176, 4973 |
| R_{int} | 0.017 |
| $(\sin \theta/\lambda)_{\max}$ (\AA^{-1}) | 0.617 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)], wR(F^2), S$ | 0.021, 0.054, 1.05 |
| No. of reflections | 5176 |
| No. of parameters | 307 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{\max}, \Delta\rho_{\min}$ ($\text{e } \text{\AA}^{-3}$) | 0.41, -0.46 |

Computer programs: APEX2 (Bruker, 2014), SAINT (Bruker, 2013), SHELXS97 (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015), XP in SHELXTL (Sheldrick, 2008), publCIF (Westrip, 2010) and PLATON (Spek, 2009).

angles between the P/Rh/N and $X/\text{Rh}/X$ (X = centroid of the double bond) planes are 4.7° (triclinic polymorph) and 2.8° (pseudo polymorph). The r.m.s. deviation of P/N/X/X to this plane is in both cases very small (0.0333 and 0.0474 \AA , respectively).

Synthesis and crystallization

A dry argon-flushed Schlenk tube was charged with dppmp (110 mg), Rh(COD)(acac) (124 mg) and THF (40 ml). The HBF_4 in water (40%) (50 μL) was added directly. After stir-

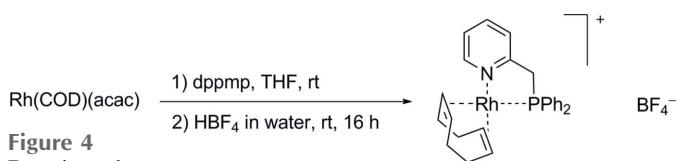


Figure 4
Reaction scheme.

ring for 16 h under room temperature, the solvent was removed under vacuum. Orange-coloured rystals suitable for X-ray crystallography were obtained by slow diffusion of diethyl ether into a dichloroethane solution. The reaction scheme is shown in Fig. 4.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The estimated diffraction contribution of the disordered solvent (a half molecule of dichloroethane per asymmetric unit) was subtracted from the observed diffraction data using SQUEEZE (Spek, 2015) in PLATON (Spek, 2009).

Acknowledgements

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References

- Bruker (2013). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2014). *APEX2* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fennis, P. J., Budzelaar, P. H. M., Frijns, J. H. G. & Orpen, A. G. (1990). *J. Organomet. Chem.* **393**, 287–298.
Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst. C* **71**, 3–8.
Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.
Spek, A. L. (2015). *Acta Cryst. C* **71**, 9–18.
Wei, S., Pedroni, J., Meissner, A., Lumbroso, A., Drexler, H.-J., Heller, D. & Breit, B. (2013). *Chem. Eur. J.* **19**, 12067–12076.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2016). **1**, x161318 [doi:10.1107/S2414314616013183]

(η^2,η^2 -Cycloocta-1,5-diene)[2-(diphenylphosphanyl methyl)pyridine- κ^2N,P]rhodium(I) tetrafluoridoborate 1,2-dichloroethane monosolvate

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Crystal data



$M_r = 575.18$

Triclinic, $P\bar{1}$

$a = 8.7638 (2)$ Å

$b = 9.2286 (2)$ Å

$c = 16.7192 (3)$ Å

$\alpha = 89.402 (1)^\circ$

$\beta = 88.904 (1)^\circ$

$\gamma = 76.121 (1)^\circ$

$V = 1312.47 (5)$ Å³

$Z = 2$

$F(000) = 584$

$D_x = 1.455 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9922 reflections

$\theta = 2.3\text{--}27.5^\circ$

$\mu = 0.75 \text{ mm}^{-1}$

$T = 150$ K

Part of block, orange

0.46 × 0.40 × 0.21 mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Detector resolution: 8.3333 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2014)

$T_{\min} = 0.639$, $T_{\max} = 0.746$

17088 measured reflections

5176 independent reflections

4973 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -10\text{--}10$

$k = -10\text{--}11$

$l = -20\text{--}20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.021$

$wR(F^2) = 0.054$

$S = 1.05$

5176 reflections

307 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 0.9042P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.41 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.46 \text{ e \AA}^{-3}$

Special details

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

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

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|---------------|--------------|----------------------------------|
| Rh1 | 0.06970 (2) | 0.24000 (2) | 0.32462 (2) | 0.01711 (5) |
| P1 | -0.00629 (5) | 0.06162 (5) | 0.25753 (2) | 0.01705 (9) |
| N1 | -0.09923 (16) | 0.19334 (16) | 0.40800 (8) | 0.0195 (3) |
| C1 | -0.0567 (2) | -0.04882 (18) | 0.34135 (10) | 0.0204 (3) |
| H1A | -0.1288 | -0.1094 | 0.3235 | 0.025* |
| H1B | 0.0395 | -0.1171 | 0.3622 | 0.025* |
| C2 | -0.13519 (19) | 0.05816 (18) | 0.40573 (10) | 0.0187 (3) |
| C3 | -0.2401 (2) | 0.0188 (2) | 0.46045 (10) | 0.0237 (4) |
| H3 | -0.2632 | -0.0765 | 0.4578 | 0.028* |
| C4 | -0.3105 (2) | 0.1194 (2) | 0.51872 (11) | 0.0264 (4) |
| H4 | -0.3804 | 0.0934 | 0.5573 | 0.032* |
| C5 | -0.2774 (2) | 0.2587 (2) | 0.51993 (11) | 0.0262 (4) |
| H5 | -0.3264 | 0.3305 | 0.5585 | 0.031* |
| C6 | -0.1723 (2) | 0.2917 (2) | 0.46427 (11) | 0.0246 (4) |
| H6 | -0.1502 | 0.3875 | 0.4655 | 0.030* |
| C7 | -0.18679 (19) | 0.12701 (19) | 0.20282 (10) | 0.0210 (3) |
| C8 | -0.2745 (2) | 0.0289 (2) | 0.17701 (11) | 0.0270 (4) |
| H8 | -0.2407 | -0.0748 | 0.1884 | 0.032* |
| C9 | -0.4113 (2) | 0.0836 (3) | 0.13474 (12) | 0.0353 (5) |
| H9 | -0.4709 | 0.0171 | 0.1169 | 0.042* |
| C10 | -0.4612 (2) | 0.2350 (3) | 0.11839 (12) | 0.0378 (5) |
| H10 | -0.5548 | 0.2717 | 0.0893 | 0.045* |
| C11 | -0.3758 (2) | 0.3324 (2) | 0.14405 (12) | 0.0364 (5) |
| H11 | -0.4109 | 0.4361 | 0.1327 | 0.044* |
| C12 | -0.2384 (2) | 0.2795 (2) | 0.18655 (11) | 0.0274 (4) |
| H12 | -0.1799 | 0.3470 | 0.2044 | 0.033* |
| C13 | 0.12691 (19) | -0.06447 (18) | 0.19045 (10) | 0.0191 (3) |
| C14 | 0.2673 (2) | -0.15094 (19) | 0.22032 (10) | 0.0217 (3) |
| H14 | 0.2877 | -0.1494 | 0.2759 | 0.026* |
| C15 | 0.3777 (2) | -0.2394 (2) | 0.16938 (11) | 0.0255 (4) |
| H15 | 0.4725 | -0.2989 | 0.1903 | 0.031* |
| C16 | 0.3497 (2) | -0.2407 (2) | 0.08854 (12) | 0.0303 (4) |
| H16 | 0.4253 | -0.3013 | 0.0538 | 0.036* |
| C17 | 0.2122 (2) | -0.1543 (3) | 0.05804 (12) | 0.0379 (5) |
| H17 | 0.1935 | -0.1550 | 0.0023 | 0.045* |
| C18 | 0.1007 (2) | -0.0661 (2) | 0.10865 (11) | 0.0312 (4) |
| H18 | 0.0063 | -0.0066 | 0.0873 | 0.037* |
| C19 | 0.0899 (2) | 0.46505 (19) | 0.36720 (11) | 0.0233 (4) |
| H19 | -0.0080 | 0.5251 | 0.3936 | 0.028* |
| C20 | 0.1787 (2) | 0.35865 (19) | 0.41578 (10) | 0.0223 (3) |
| H20 | 0.1353 | 0.3556 | 0.4714 | 0.027* |
| C21 | 0.3542 (2) | 0.2977 (2) | 0.40802 (11) | 0.0263 (4) |
| H21A | 0.3842 | 0.2037 | 0.4392 | 0.032* |
| H21B | 0.4075 | 0.3699 | 0.4316 | 0.032* |
| C22 | 0.4131 (2) | 0.2669 (2) | 0.32124 (11) | 0.0266 (4) |

| | | | | |
|------|--------------|--------------|--------------|------------|
| H22A | 0.4344 | 0.3592 | 0.2978 | 0.032* |
| H22B | 0.5133 | 0.1896 | 0.3212 | 0.032* |
| C23 | 0.2964 (2) | 0.21490 (19) | 0.26906 (11) | 0.0229 (4) |
| H23 | 0.3392 | 0.1138 | 0.2456 | 0.027* |
| C24 | 0.1833 (2) | 0.3075 (2) | 0.22174 (10) | 0.0239 (4) |
| H24 | 0.1619 | 0.2602 | 0.1709 | 0.029* |
| C25 | 0.1473 (2) | 0.4753 (2) | 0.21814 (11) | 0.0289 (4) |
| H25A | 0.0411 | 0.5134 | 0.1959 | 0.035* |
| H25B | 0.2237 | 0.5060 | 0.1813 | 0.035* |
| C26 | 0.1537 (2) | 0.5468 (2) | 0.30030 (11) | 0.0276 (4) |
| H26A | 0.2640 | 0.5474 | 0.3116 | 0.033* |
| H26B | 0.0919 | 0.6518 | 0.2987 | 0.033* |
| B1 | 0.6485 (2) | 0.6941 (3) | 0.33427 (13) | 0.0267 (4) |
| F1 | 0.74911 (15) | 0.65862 (16) | 0.26872 (7) | 0.0450 (3) |
| F2 | 0.73545 (15) | 0.67145 (14) | 0.40401 (7) | 0.0402 (3) |
| F3 | 0.53948 (16) | 0.60835 (16) | 0.33567 (8) | 0.0483 (3) |
| F4 | 0.57017 (14) | 0.84420 (14) | 0.32976 (8) | 0.0403 (3) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|--------------|---------------|
| Rh1 | 0.01708 (7) | 0.01760 (7) | 0.01754 (8) | -0.00597 (5) | 0.00292 (5) | -0.00431 (5) |
| P1 | 0.0170 (2) | 0.0176 (2) | 0.0168 (2) | -0.00476 (15) | 0.00187 (15) | -0.00293 (16) |
| N1 | 0.0178 (7) | 0.0226 (7) | 0.0184 (7) | -0.0051 (5) | 0.0011 (5) | -0.0036 (6) |
| C1 | 0.0221 (8) | 0.0196 (8) | 0.0202 (8) | -0.0063 (6) | 0.0022 (6) | -0.0014 (6) |
| C2 | 0.0165 (7) | 0.0219 (8) | 0.0170 (8) | -0.0031 (6) | -0.0020 (6) | -0.0009 (6) |
| C3 | 0.0241 (9) | 0.0269 (9) | 0.0214 (9) | -0.0085 (7) | 0.0000 (7) | 0.0014 (7) |
| C4 | 0.0223 (8) | 0.0374 (10) | 0.0200 (9) | -0.0086 (7) | 0.0030 (7) | -0.0006 (7) |
| C5 | 0.0235 (9) | 0.0330 (10) | 0.0207 (9) | -0.0043 (7) | 0.0037 (7) | -0.0094 (7) |
| C6 | 0.0238 (9) | 0.0252 (9) | 0.0250 (9) | -0.0061 (7) | 0.0025 (7) | -0.0079 (7) |
| C7 | 0.0170 (8) | 0.0270 (9) | 0.0178 (8) | -0.0032 (7) | 0.0032 (6) | -0.0029 (7) |
| C8 | 0.0230 (9) | 0.0354 (10) | 0.0235 (9) | -0.0091 (7) | 0.0027 (7) | -0.0049 (7) |
| C9 | 0.0217 (9) | 0.0618 (14) | 0.0253 (10) | -0.0154 (9) | 0.0023 (7) | -0.0063 (9) |
| C10 | 0.0193 (9) | 0.0660 (15) | 0.0219 (10) | 0.0015 (9) | 0.0026 (7) | 0.0023 (9) |
| C11 | 0.0301 (10) | 0.0407 (11) | 0.0302 (11) | 0.0067 (9) | 0.0049 (8) | 0.0058 (9) |
| C12 | 0.0255 (9) | 0.0283 (9) | 0.0256 (9) | -0.0016 (7) | 0.0044 (7) | -0.0009 (7) |
| C13 | 0.0197 (8) | 0.0186 (8) | 0.0194 (8) | -0.0059 (6) | 0.0042 (6) | -0.0047 (6) |
| C14 | 0.0253 (8) | 0.0206 (8) | 0.0197 (8) | -0.0066 (7) | 0.0004 (7) | -0.0023 (6) |
| C15 | 0.0234 (9) | 0.0228 (9) | 0.0279 (9) | -0.0012 (7) | 0.0007 (7) | -0.0016 (7) |
| C16 | 0.0279 (9) | 0.0327 (10) | 0.0261 (10) | 0.0006 (8) | 0.0066 (7) | -0.0077 (8) |
| C17 | 0.0328 (10) | 0.0549 (13) | 0.0189 (9) | 0.0033 (9) | 0.0010 (8) | -0.0062 (9) |
| C18 | 0.0246 (9) | 0.0419 (11) | 0.0215 (9) | 0.0029 (8) | -0.0006 (7) | -0.0021 (8) |
| C19 | 0.0248 (9) | 0.0198 (8) | 0.0258 (9) | -0.0061 (7) | 0.0031 (7) | -0.0094 (7) |
| C20 | 0.0236 (8) | 0.0257 (9) | 0.0203 (8) | -0.0106 (7) | 0.0008 (7) | -0.0088 (7) |
| C21 | 0.0217 (9) | 0.0311 (9) | 0.0272 (9) | -0.0084 (7) | -0.0032 (7) | 0.0001 (7) |
| C22 | 0.0197 (8) | 0.0290 (9) | 0.0317 (10) | -0.0070 (7) | 0.0033 (7) | -0.0043 (8) |
| C23 | 0.0207 (8) | 0.0235 (8) | 0.0257 (9) | -0.0083 (7) | 0.0091 (7) | -0.0062 (7) |
| C24 | 0.0283 (9) | 0.0283 (9) | 0.0187 (8) | -0.0140 (7) | 0.0067 (7) | -0.0048 (7) |

| | | | | | | |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| C25 | 0.0350 (10) | 0.0283 (9) | 0.0260 (10) | -0.0129 (8) | -0.0008 (8) | 0.0034 (7) |
| C26 | 0.0319 (10) | 0.0193 (8) | 0.0326 (10) | -0.0078 (7) | 0.0005 (8) | -0.0023 (7) |
| B1 | 0.0223 (10) | 0.0352 (11) | 0.0237 (10) | -0.0086 (8) | 0.0005 (8) | -0.0040 (8) |
| F1 | 0.0380 (7) | 0.0615 (8) | 0.0326 (7) | -0.0071 (6) | 0.0122 (5) | -0.0084 (6) |
| F2 | 0.0400 (7) | 0.0453 (7) | 0.0307 (6) | -0.0002 (5) | -0.0116 (5) | -0.0063 (5) |
| F3 | 0.0461 (8) | 0.0579 (8) | 0.0510 (8) | -0.0323 (7) | 0.0007 (6) | -0.0018 (7) |
| F4 | 0.0370 (7) | 0.0390 (7) | 0.0408 (7) | -0.0011 (5) | -0.0031 (5) | -0.0002 (5) |

Geometric parameters (\AA , $^{\circ}$)

| | | | |
|-------------|-------------|-------------|-------------|
| Rh1—N1 | 2.1295 (14) | C13—C14 | 1.394 (2) |
| Rh1—C24 | 2.1323 (17) | C14—C15 | 1.390 (2) |
| Rh1—C23 | 2.1395 (16) | C14—H14 | 0.9500 |
| Rh1—P1 | 2.2371 (4) | C15—C16 | 1.379 (3) |
| Rh1—C20 | 2.2423 (16) | C15—H15 | 0.9500 |
| Rh1—C19 | 2.2503 (17) | C16—C17 | 1.380 (3) |
| P1—C7 | 1.8135 (17) | C16—H16 | 0.9500 |
| P1—C13 | 1.8166 (16) | C17—C18 | 1.391 (3) |
| P1—C1 | 1.8331 (17) | C17—H17 | 0.9500 |
| N1—C6 | 1.354 (2) | C18—H18 | 0.9500 |
| N1—C2 | 1.359 (2) | C19—C20 | 1.368 (3) |
| C1—C2 | 1.508 (2) | C19—C26 | 1.515 (3) |
| C1—H1A | 0.9900 | C19—H19 | 1.0000 |
| C1—H1B | 0.9900 | C20—C21 | 1.509 (2) |
| C2—C3 | 1.391 (2) | C20—H20 | 1.0000 |
| C3—C4 | 1.383 (3) | C21—C22 | 1.537 (3) |
| C3—H3 | 0.9500 | C21—H21A | 0.9900 |
| C4—C5 | 1.384 (3) | C21—H21B | 0.9900 |
| C4—H4 | 0.9500 | C22—C23 | 1.521 (2) |
| C5—C6 | 1.380 (3) | C22—H22A | 0.9900 |
| C5—H5 | 0.9500 | C22—H22B | 0.9900 |
| C6—H6 | 0.9500 | C23—C24 | 1.396 (3) |
| C7—C8 | 1.397 (3) | C23—H23 | 1.0000 |
| C7—C12 | 1.397 (3) | C24—C25 | 1.506 (3) |
| C8—C9 | 1.388 (3) | C24—H24 | 1.0000 |
| C8—H8 | 0.9500 | C25—C26 | 1.538 (3) |
| C9—C10 | 1.386 (3) | C25—H25A | 0.9900 |
| C9—H9 | 0.9500 | C25—H25B | 0.9900 |
| C10—C11 | 1.376 (3) | C26—H26A | 0.9900 |
| C10—H10 | 0.9500 | C26—H26B | 0.9900 |
| C11—C12 | 1.392 (3) | B1—F3 | 1.379 (2) |
| C11—H11 | 0.9500 | B1—F1 | 1.385 (2) |
| C12—H12 | 0.9500 | B1—F2 | 1.392 (2) |
| C13—C18 | 1.392 (3) | B1—F4 | 1.393 (3) |
| N1—Rh1—C24 | | C13—C14—H14 | 119.8 |
| N1—Rh1—C23 | | C16—C15—C14 | 120.08 (17) |
| C24—Rh1—C23 | | C16—C15—H15 | 120.0 |

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| N1—Rh1—P1 | 80.58 (4) | C14—C15—H15 | 120.0 |
| C24—Rh1—P1 | 92.74 (5) | C15—C16—C17 | 120.07 (17) |
| C23—Rh1—P1 | 97.92 (5) | C15—C16—H16 | 120.0 |
| N1—Rh1—C20 | 93.09 (6) | C17—C16—H16 | 120.0 |
| C24—Rh1—C20 | 97.17 (7) | C16—C17—C18 | 120.23 (18) |
| C23—Rh1—C20 | 81.31 (7) | C16—C17—H17 | 119.9 |
| P1—Rh1—C20 | 162.34 (5) | C18—C17—H17 | 119.9 |
| N1—Rh1—C19 | 100.93 (6) | C17—C18—C13 | 120.29 (17) |
| C24—Rh1—C19 | 81.10 (7) | C17—C18—H18 | 119.9 |
| C23—Rh1—C19 | 87.72 (6) | C13—C18—H18 | 119.9 |
| P1—Rh1—C19 | 161.96 (5) | C20—C19—C26 | 125.25 (16) |
| C20—Rh1—C19 | 35.45 (7) | C20—C19—Rh1 | 71.96 (10) |
| C7—P1—C13 | 105.76 (8) | C26—C19—Rh1 | 110.01 (11) |
| C7—P1—C1 | 105.55 (8) | C20—C19—H19 | 113.9 |
| C13—P1—C1 | 107.98 (8) | C26—C19—H19 | 113.9 |
| C7—P1—Rh1 | 114.28 (6) | Rh1—C19—H19 | 113.9 |
| C13—P1—Rh1 | 121.83 (5) | C19—C20—C21 | 125.40 (16) |
| C1—P1—Rh1 | 100.07 (6) | C19—C20—Rh1 | 72.60 (10) |
| C6—N1—C2 | 117.88 (14) | C21—C20—Rh1 | 106.52 (11) |
| C6—N1—Rh1 | 123.16 (12) | C19—C20—H20 | 114.5 |
| C2—N1—Rh1 | 118.96 (11) | C21—C20—H20 | 114.5 |
| C2—C1—P1 | 107.80 (11) | Rh1—C20—H20 | 114.5 |
| C2—C1—H1A | 110.1 | C20—C21—C22 | 113.70 (15) |
| P1—C1—H1A | 110.1 | C20—C21—H21A | 108.8 |
| C2—C1—H1B | 110.1 | C22—C21—H21A | 108.8 |
| P1—C1—H1B | 110.1 | C20—C21—H21B | 108.8 |
| H1A—C1—H1B | 108.5 | C22—C21—H21B | 108.8 |
| N1—C2—C3 | 121.73 (15) | H21A—C21—H21B | 107.7 |
| N1—C2—C1 | 117.45 (14) | C23—C22—C21 | 113.02 (14) |
| C3—C2—C1 | 120.82 (15) | C23—C22—H22A | 109.0 |
| C4—C3—C2 | 119.49 (17) | C21—C22—H22A | 109.0 |
| C4—C3—H3 | 120.3 | C23—C22—H22B | 109.0 |
| C2—C3—H3 | 120.3 | C21—C22—H22B | 109.0 |
| C5—C4—C3 | 118.97 (16) | H22A—C22—H22B | 107.8 |
| C5—C4—H4 | 120.5 | C24—C23—C22 | 125.30 (16) |
| C3—C4—H4 | 120.5 | C24—C23—Rh1 | 70.65 (10) |
| C6—C5—C4 | 119.06 (16) | C22—C23—Rh1 | 113.35 (11) |
| C6—C5—H5 | 120.5 | C24—C23—H23 | 113.4 |
| C4—C5—H5 | 120.5 | C22—C23—H23 | 113.4 |
| N1—C6—C5 | 122.84 (17) | Rh1—C23—H23 | 113.4 |
| N1—C6—H6 | 118.6 | C23—C24—C25 | 126.26 (16) |
| C5—C6—H6 | 118.6 | C23—C24—Rh1 | 71.21 (10) |
| C8—C7—C12 | 119.70 (17) | C25—C24—Rh1 | 109.23 (12) |
| C8—C7—P1 | 121.64 (14) | C23—C24—H24 | 113.9 |
| C12—C7—P1 | 118.66 (14) | C25—C24—H24 | 113.9 |
| C9—C8—C7 | 119.72 (19) | Rh1—C24—H24 | 113.9 |
| C9—C8—H8 | 120.1 | C24—C25—C26 | 113.05 (15) |
| C7—C8—H8 | 120.1 | C24—C25—H25A | 109.0 |

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| C10—C9—C8 | 120.2 (2) | C26—C25—H25A | 109.0 |
| C10—C9—H9 | 119.9 | C24—C25—H25B | 109.0 |
| C8—C9—H9 | 119.9 | C26—C25—H25B | 109.0 |
| C11—C10—C9 | 120.35 (18) | H25A—C25—H25B | 107.8 |
| C11—C10—H10 | 119.8 | C19—C26—C25 | 112.51 (15) |
| C9—C10—H10 | 119.8 | C19—C26—H26A | 109.1 |
| C10—C11—C12 | 120.2 (2) | C25—C26—H26A | 109.1 |
| C10—C11—H11 | 119.9 | C19—C26—H26B | 109.1 |
| C12—C11—H11 | 119.9 | C25—C26—H26B | 109.1 |
| C11—C12—C7 | 119.78 (19) | H26A—C26—H26B | 107.8 |
| C11—C12—H12 | 120.1 | F3—B1—F1 | 110.61 (17) |
| C7—C12—H12 | 120.1 | F3—B1—F2 | 110.21 (17) |
| C18—C13—C14 | 118.88 (15) | F1—B1—F2 | 109.38 (16) |
| C18—C13—P1 | 121.84 (13) | F3—B1—F4 | 109.06 (16) |
| C14—C13—P1 | 118.95 (13) | F1—B1—F4 | 109.12 (17) |
| C15—C14—C13 | 120.44 (16) | F2—B1—F4 | 108.41 (16) |
| C15—C14—H14 | 119.8 | | |
