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A Sulfur Mimic of 1,1-Bis(diphenylphosphino)methane: A New Ligand Opens Up

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Extended Experimental Section

General Considerations. All procedures and manipulations were performed under an argon or nitrogen atmosphere using standard Schlenk-line and glove box techniques unless stated otherwise. All solvents were degassed and dried using standard procedures prior to all manipulations and reactions unless stated otherwise. Deuterated solvents were purchased from Cambridge Isotope Laboratories or Sigma Aldrich, degassed, and dried over activated molecular sieves prior to use. All other reagents were purchased from commercial sources and utilized without further purification. The ESI-MS data was collected on an AB/Sciex QStar mass spectrometer with an ESI source, the EI-MS data was collected on a Waters GC ToF mass spectrometer with an EI/CI source, and the DART-MS data was collected on a JOEL AccuTOF-DART mass spectrometer with a DART-ion source (no solvent is required). NMR spectra were recorded at ambient temperature and pressure using a Varian Gemini 400 MHz spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, 376 MHz for ^{19}F, and 161 MHz for ^{31}P) or an Agilent DD2-600 MHz spectrometer (600 MHz for ^{1}H, 151 MHz for ^{13}C, 564 MHz for ^{19}F, and 243 MHz for ^{31}P) unless stated otherwise. The ^{1}H and ^{13}C NMR were measured relative to partially deuterated solvent peaks but are reported relative to tetramethylsilane (TMS). All ^{31}P chemical shifts were measured relative to 85% phosphoric acid as an external reference. The elemental analyses were performed at the Department of Chemistry, University of Toronto, on a Perkin-Elmer 2400 CHN elemental analyzer. Some complexes gave inconsistent carbon analyses but acceptable hydrogen contents; we attribute this as a combustion problem due to the triflate counter-ion. Single-crystal X-ray diffraction data were collected using a Nonius Kappa-CCD or Bruker Kappa APEX DUO diffractometer with Mo Kα radiation ($\lambda = 0.710$ Å) or Cu Kα radiation ($\lambda = 2.29$ Å). The CCD data were integrated and scaled using the Denzo-SMN package. The structures were solved and refined using SHELXTL V6.1. Refinement was by full-matrix least squares on F^2 using all data.

Synthesis of compound 1 ($Ph_2PP(S)Ph_2$). A solution of Li_2S (0.050 g, 1.1 mmol) in CH_3CN (5 mL) was prepared, and $ClPPh_2$ (0.470 g, 2.12 mmol) in CH_3CN (2 mL) was added. The cloudy solution was stirred overnight, and the solvent removed under reduced pressure. The white powder was dissolved in toluene (~ 7 mL), filtered, and stored in a freezer (-33°C) for several days (3-4). Crystals suitable for single crystal X-ray diffraction studies were isolated from the toluene solution by filtration, and dried overnight. Yield: 87.4% (0.375 g). ^{1}H NMR (400 MHz, THF-d_8) δ: 8.14 (ddt, 4H, Ar-CH, *J* = 12.6, 8.1, 1.1 Hz), 7.69-7.63 (m, 4H, Ar-CH), 7.61-7.51 (m, 6H, Ar-CH), 7.49-7.43 (m, 2H, Ar-CH), and 7.39-7.33 (m, 4H, Ar-CH) ppm. ^{31}P{^{1}H} NMR (161 MHz, THF-d_8) δ: 41.71 (d, *J* = 247.0 Hz), and -16.21 (d, *J* = 247.0 Hz) ppm. ^{13}C{^{1}H} NMR (100 MHz, THF-d_8) δ: 138.41 (dd, Ar-CH, *J* = 19.9, 7.2 Hz), 137.40 (dd, C-P, *J* = 68.8, 14.4 Hz), 135.62 (dd, Ar-CH, *J* = 9.1, 6.8 Hz), 134.53 (dd, Ar-CH, *J* = 3.0, 1.2 Hz), 133.12 (dd, Ar-CH, *J* = 2.2, 0.6 Hz), 131.89 (dd, C-P, *J* = 59.6, 12.9 Hz), 131.65(d, Ar-CH, *J* = 11.7 Hz), and 131.49 (dd, Ar-CH, *J* = 7.5, 1.1 Hz) ppm. Anal. Calcd for [$C_{24}H_{20}P_2S$]: C, 71.61; H, 5.01. Found: C, 71.61; H, 5.40. MS (DART, THF; m/z$^+$): 403.1 [$C_{24}H_{21}P_2S$]$^+$.

Synthesis of compound 2 ($Cy_2PP(S)Cy_2$). A solution of Li_2S (0.050 g, 1.1 mmol) in CH_3CN (5 mL) was prepared, and $ClPCy_2$ (0.507 g, 2.12 mmol) in CH_3CN (2 mL) was added. The cloudy solution was stirred for 2 days, and the precipitate was isolated by filtration. The white powder was dissolved in toluene (~ 7 mL), filtered, and the solvent was removed. Yield: 69.6% (0.323 g). ^{1}H NMR (600 MHz, THF-d_8) δ: 2.25-2.17 (m, 2H, P-CH), 2.15-2.01 (m, 6H, CH_2), 1.98-1.88 (m, 4H, P-CH and CH_2), 1.87-1.73 (m, 8H, CH_2), 1.71-1.62 (m, 4H, CH_2), and 1.54-1.18 (m, 20H, CH_2) ppm. ^{31}P{^{1}H} NMR (242 MHz, THF-d_8) δ: 59.08 (d, *J* = 301.7 Hz), and -14.02 (d, *J* = 301.7 Hz) ppm. ^{13}C{^{1}H} NMR (100 MHz, THF-d_8) δ: 39.72 (dd, P-CH, *J* = 34.7, 8.2 Hz), 31.23 (d, CH_2, *J* = 20.4, 6.2 Hz), 31.13 (dd, CH_2, *J* = 24.0, 3.6 Hz), 38.59 (t, CH_2, *J* = 6.8 Hz), 25.75 (ddd, CH_2, *J* = 32.4, 4.8, 3.6 Hz), 25.71 (d, CH_2, *J* = 14.0 Hz), 25.38-25.13 (m, CH_2), 25.15 (d, CH_2, *J* = 12.1 Hz), and 24.21 (dd, CH_2, *J* = 11.5, 1.4 Hz) ppm. Anal. Calcd for $[C_{24}H_{44}P_2S]$: C, 67.57; H, 10.40. Found: C, 67.65; H, 10.73. MS (DART, DCM; m/z$^+$): 427.3 $[C_{24}H_{45}P_2S]^+$.

Synthesis of compound 3 (RuCp*(Ph_2PSPPh_2)Cl). A solution of **1** (0.106 g, 0.263 mmol) in THF (2 mL) was prepared, and RuCp*(cod)Cl (0.100 g, 0.263 mmol) in THF (5 mL) was added. The light orange solution was heated to 40°C and stirred overnight. The resulting deep orange solution was cooled to room temperature and the solvent removed under reduced pressure. Hexanes (~8 mL) was added to the orange residue, and stirred for 15 min. The product was collected as a yellow powder, washed with hexanes (5 mL), and dried overnight. Yield: 92.3% (0.164 g). Crystals suitable for single crystal X-ray diffraction studies grown from the slow diffusion of pentane into a THF solution of complex **2**. ^{1}H NMR (600 MHz, THF-d_8) δ: 7.71-7.66 (m, 8H, Ar-CH), 7.54-7.49 (m, 8H, Ar-CH), 7.43-7.38 (m, 10H, Ar-CH), 7.32-7.29 (m, 10H, Ar-CH), and 1.44 (t, 15H, Ar-CH_3, *J* = 2.4 Hz) ppm. ^{31}P{^{1}H} NMR (242 MHz, THF-d_8) δ: 39.24 (s) ppm. ^{13}C{^{1}H} NMR (100 MHz, THF-d_8) δ: 141.87-141.48 (m, C-P), 140.79-140.44 (m, C-P), 132.35 (t, Ar-CH, *J* = 7.0 Hz), 131.06 (t, Ar-CH, *J* = 6.3 Hz), 139.65 (s, Ar-CH), 128.88 (s, Ar-CH), 127.06 (t, Ar-CH, *J* = 5.4 Hz), 126.99 (t, Ar-CH, *J* = 5.1 Hz), 95.59 (t, C-CH_3, *J* = 2.4 Hz), and 8.39 (s, Ar-CH_3) ppm. Anal. Calcd for $[C_{34}H_{35}ClP_2lRuS]0.25[C_6H_6]$: C, 61.46; H, 5.30. Found: C, 61.14; H, 5.13. MS (ESI$^+$, THF; m/z$^+$): 639.1 $[C_{34}H_{35}P_2RuS]^+$, 674.1 $[C_{34}H_{35}ClP_2RuS]^+$.

Synthesis of compound 4 (RuCp*(Cy_2PSPCy_2)Cl). A solution of **2** (0.050 g, 0.12 mmol) in THF (2 mL) was prepared, and RuCp*(cod)Cl (0.045 g, 0.12 mmol) in THF (3 mL) was added. The light orange solution was heated to 40°C and stirred overnight. The resulting deep orange solution was cooled to room temperature and the solvent removed under reduced pressure. Hexanes (~6 mL) was added to the orange residue, and stirred for 15 min. The product was cooled in a freezer (-33°C) for 2 hours, collected as a yellow powder, washed with hexanes (2 mL), and dried overnight. Yield: 56 % (0.046 g). Crystals suitable for single crystal X-ray diffraction studies grown from the slow diffusion of pentane into a THF solution of complex **2**. ^{1}H NMR (600 MHz, THF-d_8) δ: 2.97-2.87 (m, 2H, P-CH), 2.45-2.26 (m, 2H, CH_2), 2.19-2.13 (m, 2H, CH_2), 2.09 (tt, 2H, P-CH, *J* = 11.8, 2.3 Hz), 1.96-1.82 (m, 6H, CH_2), 1.76-1.69 (m, 8H, CH_2), 1.67 (t, 15H, CH_3, *J* = 1.9 Hz), and 1.64-1.13 (m, 22H, CH_2) ppm. ^{31}P{^{1}H} NMR (242 MHz, THF-d_8) δ: 67.15 (s) ppm. ^{13}C{^{1}H} NMR (100 MHz, THF-d_8) δ: 87.26 (t, C-CH_3, *J* = 2.8 Hz), 41.38 (t, C-P, *J* = 3.4 Hz), 39.97 (t, C-P, *J* = 6.4 Hz), 29.94 (s, CH_2), 29.19 (t, CH_2, *J* = 2.6 Hz), 28.61 (d, CH_2, *J* = 9.0 Hz), 27.32 (t, CH_2, *J* = 6.1 Hz), 27.07 (dt, CH_2, *J* = 9.7, 4.7 Hz), 26.08 (d, CH_2, *J* = 19.6 Hz), and 10.34 (s, Ar-CH_3) ppm. Anal. Calcd for $[C_{34}H_{59}ClP_2lRuS]$: C, 58.47; H, 8.52. Found: C, 58.39; H, 8.22. MS (ESI$^+$, THF; m/z$^+$): 662.3 $[C_{34}H_{59}P_2RuS]^+$.

Synthesis of compound 5 ([RuCp*(Ph_2PSPPh_2)$HPPh_2$][OTf]). A solution of RuCp*(cod)Cl (0.020 g, 0.039 mmol) in THF (2 mL) was prepared, and AgOTf (0.014 g, 0.054 mmol) in THF (2 mL) was added. After 30 min. the dark orange solution was filtered and $HPPh_2$ (0.010 g, 0.054 mmol) in THF (2 mL) was added dropwise. The solution was stirred for 5 min. and then **1** (0.022 g, 0.054 mmol) in THF (2 mL) was added. The resulting yellow solution was stirred for 7 days, and the solvent removed under reduced pressure. Crystals suitable for single crystal X-ray diffraction studies were isolated from a mixture of MeOH and ether stored in a freezer (-33°C), and dried overnight. Yield: 45 % (0.023 g). **In THF:** ^{1}H NMR (400 MHz, THF-d_8) δ: 7.88-7.81 (m, 4H, Ar-CH), 7.73-7.61 (m, 16H, Ar-CH), 7.34-7.28 (m, 2H, Ar-CH), 7.33 (dt, P-H, *J* = 367.1, 8.4 Hz), 7.22-7.16 (m, 4H, Ar-CH), 7.16-7.09 (m, 4H, Ar-CH), and 1.72 (q, 15H, Ar-CH_3, *J* = 2.1 Hz) ppm. ^{31}P{^{1}H} NMR (161 MHz, THF-d_8) δ: 32.36 (d, *J* = 35.1 Hz), and 29.37 (dd, *J* = 36.0, 34.7 Hz) ppm. ^{13}C{^{1}H} NMR (100 MHz, THF-d_8) δ: 136.89-136.56 (m, C-P), 134.63 (td, C-P, *J* = 21.1, 3.1 Hz), 133.31 (d, Ar-CH, *J* = 9.8 Hz), 132.13 (t, Ar-CH, *J* = 6.0 Hz), 131.04 (d, C-P, *J* = 38.0 Hz), 130.85-130.73 (m, Ar-CH), 129.21-129.08 (m, Ar-CH), 128.23 (t, Ar-CH, *J* = 5.1Hz), 127.92 (d, Ar-CH, *J* = 10.0 Hz), 95.86-95.79 (m, C-CH_3), and 9.42 (s, Ar-CH_3) ppm. **In MeOH:** ^{1}H NMR (400 MHz, MeOH-d_4) δ: 7.64-7.56 (m, 4H, Ar-CH), 7.55-7.39 (m, 16H, Ar-CH), 7.23-7.17 (m, 2H, Ar-CH), 7.05-6.99 (m, 4H, Ar-CH), 6.87(ddd, P-H, *J* = 356.1, 11.5, 7.8 Hz), 6.82-6.75, and 1.54 (q, 15H, Ar-CH_3, *J* = 2.2 Hz) ppm. ^{31}P{^{1}H} NMR (161 MHz, MeOH-d_4) δ: 36.28 (dd, *J* = 37.0, 33.3 Hz), and 34.37 (d, *J* = 35.5 Hz) ppm. ^{13}C{^{1}H} NMR (100 MHz, MeOH-d_4) δ136.38-135.99 (m, C-P), 134.48-133.97 (m, C-P), 133.05 (d, Ar-CH, *J* = 9.7 Hz), 133.02 (d, Ar-CH, *J* = 9.5 Hz), 131.90 (t, Ar-CH, *J* = 6.1 Hz), 130.93 (d, Ar-CH, *J* = 14.4 Hz), 130.53 (t, Ar-CH, *J* = 5.6 Hz), 130.46 (d, C-P, *J* = 45.9 Hz), 129.56 (d, Ar-CH, *J* = 2.1 Hz), 128.88 (t, Ar-CH, *J* = 5.1 Hz), 128.18 (t, Ar-CH, *J* = 5.2Hz), 127.98 (d, Ar-CH, *J* = 9.7 Hz), 95.80 (d, C-CH_3, *J* = 1.3 Hz), and 9.19 (s, Ar-CH_3) ppm. Anal. Calcd for [$C_{47}H_{46}F_3O_3P_3RuS_2$][$C_4H_{10}O$]: C, 58.44; H, 5.39. Found: C, 57.42; H, 5.54. MS (ESI$^+$, THF; m/z$^+$): 639.1 [$C_{34}H_{35}P_2RuS$]$^+$, 825.2 [$C_{46}H_{46}P_3RuS$]$^+$.

Crystal Structure and Discussion of 1

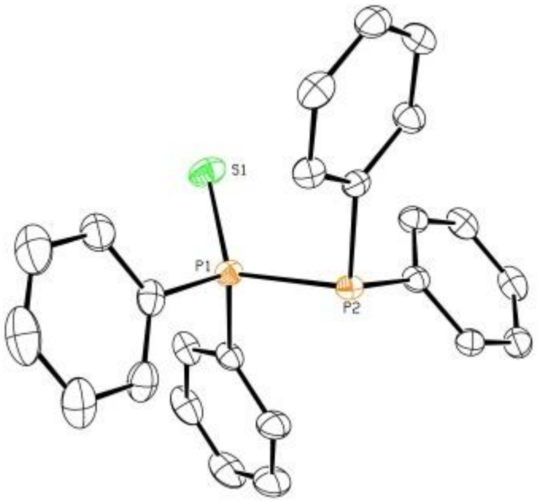

Figure S1. ORTEP3 representation (thermal ellipsoids at 50% probability; all the hydrogens are omitted for clarity) and atom numbering for **1**.

Crystals of **1** were grown from a solution of **1** in toluene that was cooled in a freezer (-33°C) for several days. The structure was almost identical to the structure reported by Aluri *et al.*, but **1** was generated using a different synthetic method, and the crystallization conditions were also not the same as those published previously.[2]

Analysis of the Formation of S1.

The alternative synthetic scheme developed to generate **5** utilized RuCp*(cod)Cl (cod 1,5-cyclooctadiene) as a practical metal precursor. Initially, the chloride ligand was abstracted with AgOTf and the resulting AgCl was filtered away. Presumably, a solvated [RuCp*(cod)(THF)]$^+$ species was generated in solution, (see Figure S2). One equivalent of diphenylphosphine was then added to the solution dropwise. A 31P NMR spectrum of the reaction mixture revealed two species in solution, at 31.6, **SA**, and 30.5 ppm, **S1**, both with a large P-H coupling, 374 and 347 Hz, respectively (see Figure S2). The bisthioether ligand precursor **1** was subsequently added to the reaction mixture, but only **SA** reacted further to give the desired product **5**, while **SA** persisted in solution with unreacted **1**. Based on these observations it is believed that **SA** is the desired RuCp*(cod)($PHPh_2$) intermediate. The nature of **S1** was not understood until crystals were grown from the crude reaction mixture, which gave a cationic ruthenium complex [RuCp*($HPPh_2$)$_3$][OTf], **S1**. The structure was consistent with the NMR data as well as our proposed mechanism for the formation of the bisthioether ligands; the diphosphine monosulfide **1** should not be a strong enough ligand to displace a diphenylphosphine and therefore **S1** should be inert to ligand substitution under these conditions.

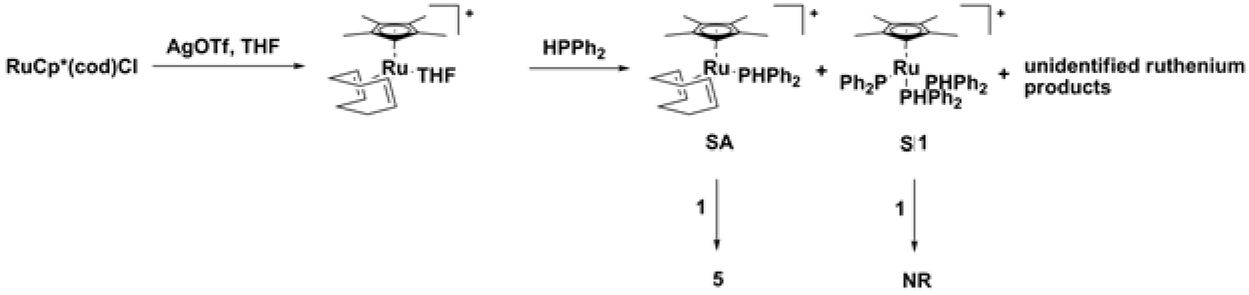

Figure S2. Synthesis of **5** and **S1** as well as other proposed intermediates.

Crystal Structure and Discussion of S1

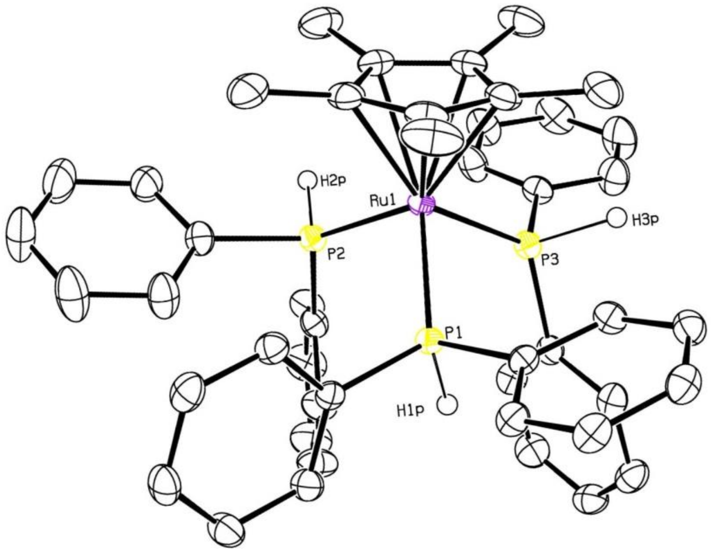

Figure S3. ORTEP3 representation and atom numbering for **S1** (thermal ellipsoids at 50% probability; the solvent molecule, counter-ion, and most of the hydrogens are omitted for clarity).

During the synthesis of **5**, crystals of **S1** suitable for XRD studies were grown from the slow diffusion of ether into a sample of the reaction mixture. The ruthenium centre displayed a piano stool structure with three *cis* diphenylphosphine ligands *trans* to the Cp* moiety (see Figure S3). All three Ru-P bond lengths, Ru(1)-P(1), Ru(1)-P(2), and Ru(1)-P(3), were within the normal range for neutral phosphine donors, 2.301(2), 2.302(2), and 2.302(2) Å, respectively, while all the P-Ru-P bond angles, P(1)-Ru(1)-P(2), P(1)-Ru(1)-P(3), and P(2)-Ru(1)-P(3), were close to the ideal angle of 90°, 96.86(6), 90.08(6), and 96.11(6)°, respectively.[1]

Table S1. Selected Bond Lengths and Angles for 1.

Bond Lengths (Å)	
P(1)-P(2)	2.226(2)
P(1)-S(1)	1.953(2)
Bond Angles (deg)	
P(1)-S(1)-P(2)	118.35(6)

Table S1. Selected Bond Lengths and Angles for 2.

Bond Lengths (Å)	
P(1)-P(2)	2.225(4)
P(1)-S(1)	1.972(2)
Bond Angles (deg)	
P(1)-S(1)-P(2)	116.47(9)
C(1)-P(1)-C(13)	113.2(4)
C(7)-P(1)-C(19)	103.7(4)

Table S2. Selected Bond Lengths and Angles for 3.

Bond Lengths (Å)	
Ru(1)-P(1)	2.288(4)
Ru(1)-P(2)	2.278(4)
P(1)-S(1)	2.126(6)
P(2)-S(1)	2.145(6)
Bond Angles (deg)	
P(1)-Ru(1)-P(2)	75.8(2)
P(1)-Ru(1)-Cl(1)	89.0(2)
P(2)-Ru(1)-Cl(1)	88.0(2)
P(1)-S(1)-P(2)	82.1(2)

Table S3. Selected Bond Lengths and Angles for 4.

Bond Lengths (Å)	
Ru(1)-P(1)	2.315(2)
Ru(1)-P(2)	2.302(2)
P(1)-S(1)	2.136(4)
P(2)-S(1)	2.126(4)
Bond Angles (deg)	
P(1)-Ru(1)-P(2)	74.71(6)
P(1)-Ru(1)-Cl(1)	88.32(6)
P(2)-Ru(1)-Cl(1)	87.03(6)
P(1)-S(1)-P(2)	82.19(9)

Table S4. Selected Bond Lengths and Angles for 5.

Bond Lengths (Å)	
Ru(1)-P(1)	2.299(2)
Ru(1)-P(2)	2.306(2)
Ru(1)-P(3)	2.334(2)
P(1)-S(1)	2.123(4)
P(2)-S(1)	2.125(4)
Bond Angles (deg)	
P(1)-Ru(1)-P(2)	74.90(6)
P(1)-Ru(1)-P(3)	102.14(6)
P(2)-Ru(1)-P(3)	89.60(6)
P(1)-S(1)-P(2)	82.46(9)

Table S6. Selected Crystal Data, Data Collection, and Refinement Parameters for Compounds 1, 2, and 3.

	1	**2**	**$3{\cdot}C_4H_8O{\cdot}0.5C_5H_{12}$**
empirical formula	$C_{24}H_{20}P_2S$	$C_{24}H_{44}P_2S$	$C_{40.50}H_{49}ClOP_2RuS$
FW	402.40	426.59	782.32
lattice type	Monoclinic	Monoclinic	Monoclinic
space group	$P2_1/c$	$P2_1/c$	$P2_1/c$
T, K	147(2)	147(2)	147(2)
a, Å	12.900(12)	10.690(3)	28.8822(9)
b, Å	13.621(17)	21.438(6)	17.6795(6)
c, Å	16.021(2)	11.299(3)	15.4946(5)
α, deg	90	90	90
β, deg	91.450(3)	110.251(6)	105.449(1)
γ, deg	90	90	90
V, $Å^3$	2026.7(4)	2429(1)	7626.0(4)
Z	4	4	8
ρ_{calc}/Mg m^{-3}	1.319	1.166	1.363
μ(Mo/Cu, Kα) mm^{-1}	0.324	0.273	5.502
F(000)	840	936	3256
cryst size, mm^3	0.300x0.260x0.200	0.33x0.25x0.15	0.28x0.17x0.04
range θ collected, deg	1.963 to 27.518	1.90 to 27.56	1.59 to 66.71
reflns collected/unique	4646/36723	5579/22191	13049/55571
abs cor	Semi-empirical from equivalents		
Max and min transmn coeff.	0.7456 and 0.6705	0.7456 and 0.6413	0.7528 and 0.5469
goodness of fit	1.042	1.029	1.138
$R_1(I>2\sigma(I))^a$	0.0287	0.0433	0.0471
wR_2 (all data)^a	0.0788	0.1171	0.1210
peak and hole, e $Å^{-3}$	0.339 and -0.204	0.637 to -0.277	1.206 to -0.834

[a] Definition of R indices: $R_1 = \Sigma(F_O - F_C)/\Sigma(F_O)$; $wR_2 = [\Sigma[w(F_O^2 - F_C^2)^2]/[\Sigma[w(F_O^2)^2]]^{1/2}$

Table S7. Selected Crystal Data, Data Collection, and Refinement Parameters for Compounds 5 and S1.

	4	**5·$C_4H_{10}O$**	**S1· $C_4H_{10}O$**
empirical formula	$C_{34}H_{59}ClP_2RuS$	$C_{51}H_{56}F_3O_4P_3RuS_2$	$C_{51}H_{58}F_3O_4P_3RuS$
FW	698.33	1048.05	1018.01
lattice type	Monoclinic	Monoclinic	Monoclinic
space group	$P2_1/n$	$P2_1/n$	$P2_1/n$
T, K	147(2)	230(2)	147(2)
a, Å	9.5887(7)	11.9425(10)	12.0971(12)
b, Å	20.4899(14)	19.3668(17)	19.4201(19)
c, Å	17.4167(12)	21.7142(18)	20.657(2)
α, deg	90	90	90
β, deg	95.800(2)	95.243(2)	94.114(3)
γ, deg	90	90	90
V, $Å^3$	3404.1(4)	5001.2(7)	4840.4(8)
Z	4	4	4
ρ_{calc}/Mg m^{-3}	1.363	1.392	1.397
μ(Mo/Cu, Kα) mm^{-1}	0.717	0.547	0.521
F(000)	1480	2168	2112
cryst size, mm^3	0.135x0.120x0.050	0.200x0.100x0.060	0.260x0.250x0.110
range θ collected, deg	1.539 to 27.497	1.877 to 27.540	1.894 to 27.552
reflns collected/unique	7762/61189	11508/43900	11171/129010
abs cor	Semi-empirical from equivalents		
Max and min transmn coeff.	0.7456 and 0.6830	0.7456 and 0.6355	0.7456 and 0.6672
goodness of fit	1.038	1.011	1.032
R_1(I>2σ(I))[a]	0.0334	0.0376	0.0308
wR_2 (all data)[a]	0.0786	0.0968	0.0805
peak and hole, e $Å^{-3}$	1.482 and -0.481	1.132 and -0.543	1.126 and -0.601

[a] Definition of R indices: $R_1 = \Sigma(F_O - F_C)/\Sigma(F_O)$; $wR_2 = [\Sigma[w(F_O^2 - F_C^2)^2]/[\Sigma[w(F_O^2)^2]]^{1/2}$

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2. Aluri, B. R.; Peitz, S.; Wöhl, A.; Peulecke, N.; Müller, B. H.; Spannenberg, A.; Rosenthal, U. *Acta Crystallogr*. *E* **2009**, *65*, o404.