

Supporting Information

Does an Axial Propeller Shape on a Dirhodium(III) Core Affect Equatorial Ligand Chirality?

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Rahway, New Jersey 07065*

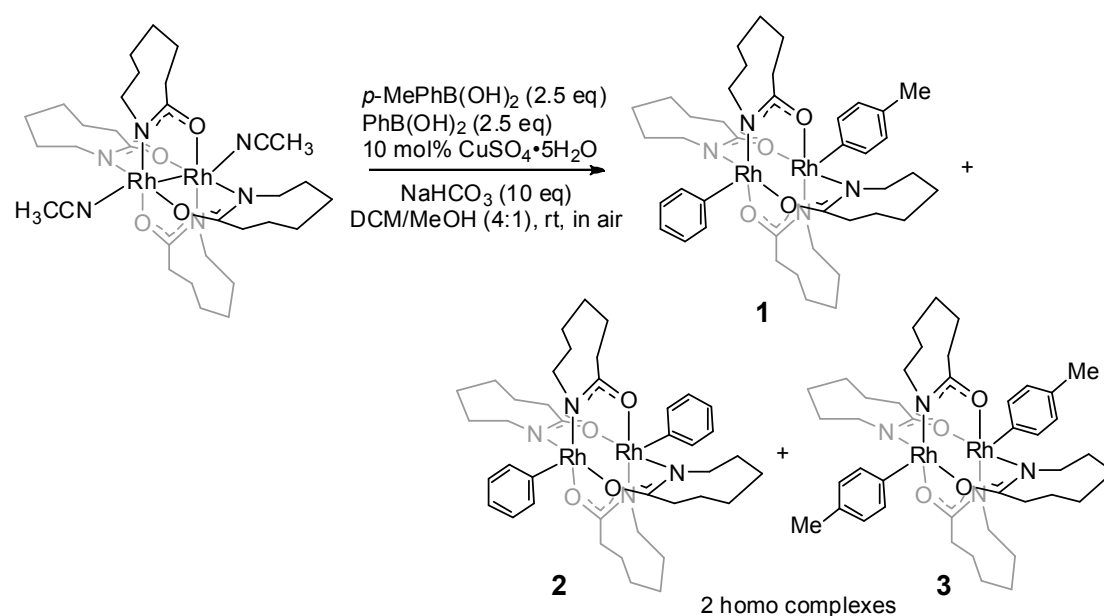
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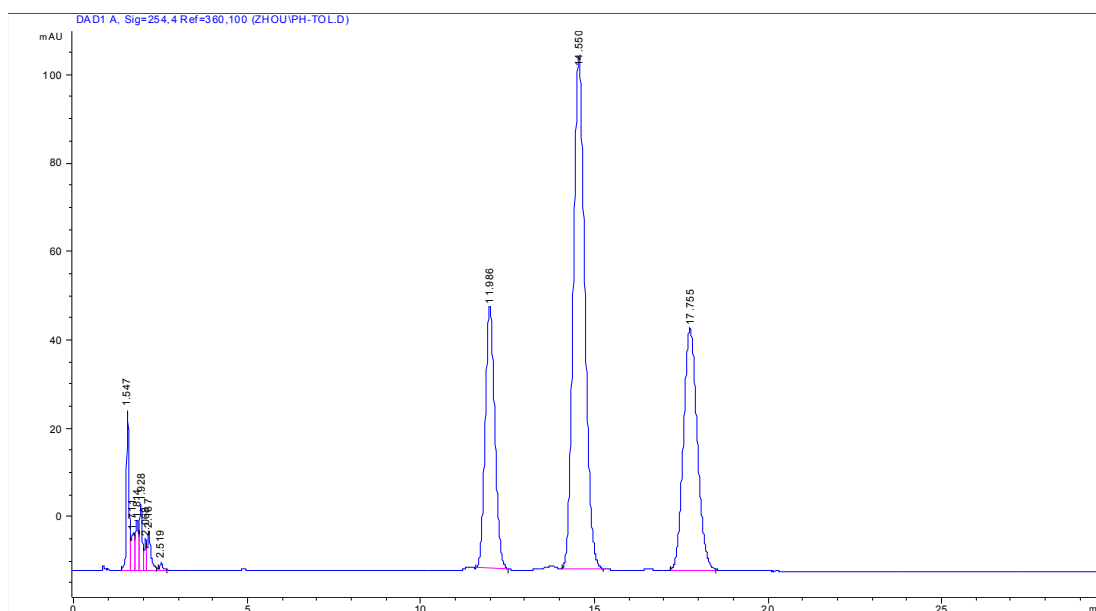
[†]University of Maryland

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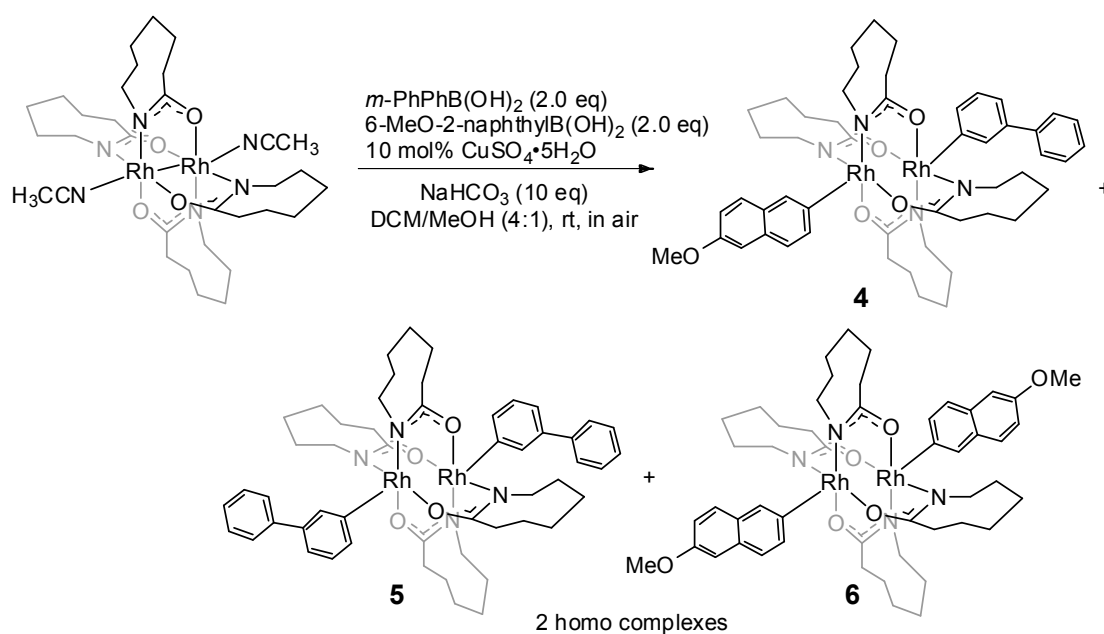
General. Reactions were performed in oven-dried (140 °C) or flame-dried glassware under an atmosphere of dry N₂. Dichloromethane (DCM) was passed through a solvent column prior to use and was not distilled. Methanol was not distilled. Thin layer chromatography (TLC) was carried out using EM Science silica gel 60 F₂₅₄ plates. The developed chromatogram was analyzed by UV lamp (254 nm). Liquid chromatography was performed using flash chromatography of the indicated system on silica gel (230-400 mesh). Arylboronic acids were purchased from Aldrich and used as received. ¹H NMR and ¹³C NMR spectra were recorded in CDCl₃ or CD₂Cl₂ on a Bruker Avance 400 MHz spectrometer. Chemical shifts were reported in ppm with the solvent signals as reference, and coupling constants (*J*) were given in Hertz. IR spectra were recorded on a Jasco FTIR 4100 spectrometer. Mass spectra were obtained with a JEOL AccuTOF-CS spectrometer.



Procedure for Synthesis of 1+2+3: Rh₂(ACO)₄(CH₃CN)₂ (48.0 mg, 0.06 mmol), phenylboronic acid (18.3 mg, 2.50 equiv), *p*-methylphenylboronic acid (20.4 mg, 2.50 equiv) and NaHCO₃ (50.4 mg, 10.0 equiv) were dissolved in 6.00 mL of CH₂Cl₂. Copper sulfate (10.0 mol%) in 1.50 mL of MeOH was added to the solution, and the resulting purple mixture was stirred under air at room temperature for 12 h, during which the solution turned greenish brown. After filtration of NaHCO₃ and evaporation of the solvent under reduced pressure, the crude product was purified by column chromatography on silica gel using CH₂Cl₂ as the eluent to give compound 1 as a greenish brown solid and mixture of three dirhodium complexes in 38.0% yield (20.0 mg). Column chromatography could not separate these complexes. HPLC analysis on C-18 column (CH₃CN/iPrOH 87:13, 1.0 mL/min, 254 nm UV-vis) shows three peaks, which include two homo compounds and one hetero compound. Calculation based on HPLC and ¹H NMR analysis shows the molar ratio of three compounds is: PhPh (**2**) : PhTol (**1**) : TolTol (**3**) = 22 : 48 : 26. A HPLC chromatograph of the mixture is shown below: retention times of PhPh (**2**), PhTol (**1**), and TolTol (**3**) are 12.0 min, 14.6 min, and 17.8 min, respectively.

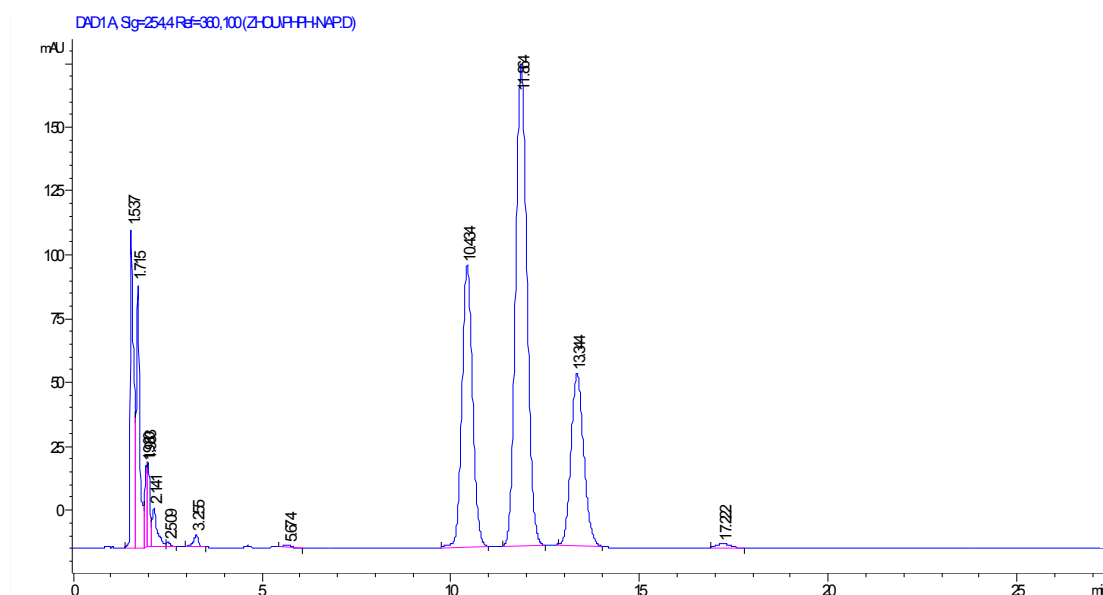


1+2+3. ^1H NMR (400 MHz, CD_2Cl_2): δ 7.47-7.50 (comp, 2H), 7.33 (d, 2H, $J = 8.0$ Hz), 7.09-7.11 (comp, 3H), 6.94 (d, 2H, $J = 8.0$ Hz), 3.15-3.26 (comp, 8H), 2.41 (s, 3H), 2.24-2.31 (comp, 8H), 1.22-1.58 (comp, 32H); ^{13}C NMR (150 MHz, CDCl_3): δ 181.9, 136.5, 136.0, 132.8, 126.9, 125.8, 123.6, 53.3, 50.1, 33.4, 32.6, 28.0, 26.1, 25.1; HRMS (ESI) for $\text{C}_{41}\text{H}_{61}\text{N}_4\text{O}_4\text{Rh}_2$ $[\text{M}+\text{H}]^+$ calcd: 879.2803; found: 879.1859; other two homo complexes: HRMS (ESI) for $\text{C}_{40}\text{H}_{59}\text{N}_4\text{O}_4\text{Rh}_2$ $[\text{M}+\text{H}]^+$ calcd: 865.2646; found: 865.1860; HRMS (ESI) for $\text{C}_{42}\text{H}_{63}\text{N}_4\text{O}_4\text{Rh}_2$ $[\text{M}+\text{H}]^+$ calcd: 893.2959; found: 893.2034; IR (neat): 2923, 2854, 1658, 1633, 1579, 1454 cm^{-1} . See X-ray experimental for crystallographic data for **1**.

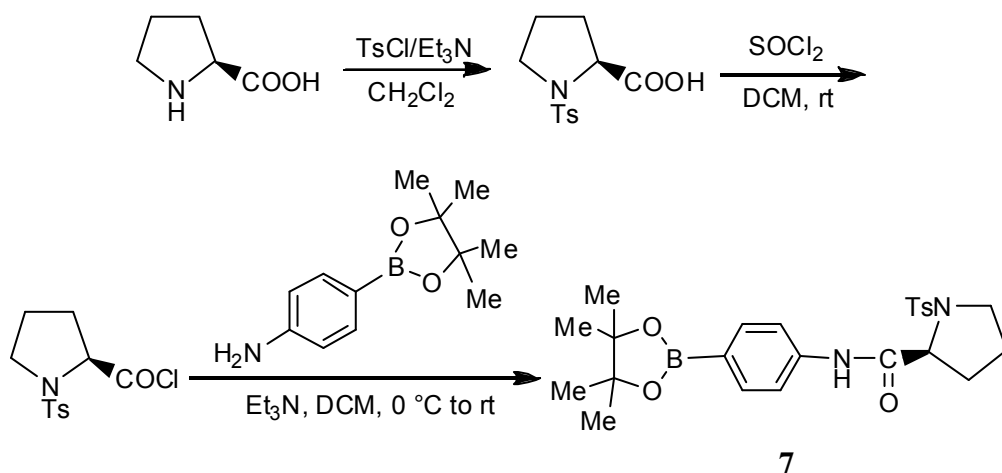


Procedure for Synthesis of 4+5+6. $\text{Rh}_2(\text{ACO})_4(\text{CH}_3\text{CN})_2$ (48.0 mg, 0.06 mmol),

3-biphenylboronic acid (24.0 mg, 2.00 equiv), 6-methoxy-2-naphthaleneboronic acid (24.0 mg, 2.00 equiv) and NaHCO₃ (50.4 mg, 10.0 equiv) were dissolved in 6.00 mL of CH₂Cl₂. Copper sulfate (10.0 mol%) in 1.50 mL of MeOH was added to the solution, and the resulting purple mixture was stirred under air at room temperature for 12 h, during which the solution turned yellowish brown. After filtration of NaHCO₃ and evaporation of the solvent under reduced pressure, the crude product was purified by preparative TLC on silica gel using CH₂Cl₂ as the eluent to give compound **4** as yellowish brown solid in 9.8% yield. HPLC analysis of reaction mixture on a C-18 column (CH₃CN/iPrOH 87:13, 1.0 mL/min, 254 nm UV-vis) shows three peaks, which include two homo compounds and one hetero compound. A HPLC chromatograph of the mixture is shown below: retention time of NaphthNaphth (**6**) : *m*BiPhNaphth (**4**): *m*BiPhmBiPh (**5**) are 10.4 min, 11.9 min, and 13.3 min, respectively. The ratio of three peaks is 22 : 41 : 17.

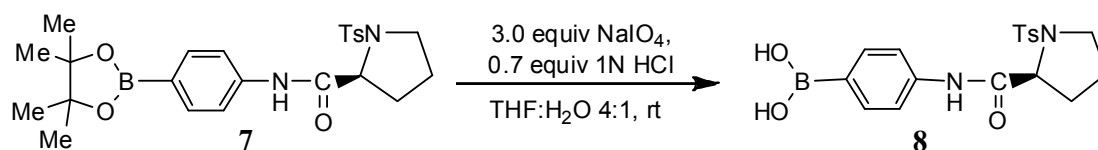


4. ¹H NMR (400 MHz, CD₂Cl₂): δ 7.90 (s, 1H), 7.72-7.76 (comp, 4H), 7.63-7.66 (comp, 1H), 7.54-7.57 (comp, 2H), 7.46-7.50 (comp, 2H), 7.37-7.42 (comp, 2H), 7.21-7.26 (comp, 2H), 7.11-7.14 (m, 1H), 3.98 (s, 3H), 3.25-3.38 (comp, 8H), 2.35-2.41 (comp, 8H), 1.27-1.66 (comp, 32H); ¹³C NMR (150 MHz, CD₂Cl₂): δ 182.1, 157.7, 156.6, 146.7, 141.9, 140.4, 138.7, 135.6, 135.0, 133.5, 132.5, 129.5, 128.4, 128.2, 127.0, 126.6, 125.8, 125.3, 123.2, 122.8, 118.9, 117.1, 105.3, 55.1, 50.2, 33.3, 32.7, 28.0, 26.0, 25.1; HRMS (ESI) for C₅₁H₆₇N₄O₅Rh₂ [M+H]⁺ calcd: 1021.3222; found: 1021.3059; IR (neat): 2923, 2854, 1648, 1572, 1541, 1454 cm⁻¹. See X-ray experimental for crystallographic data on **4**.



Procedure for Synthesis of 7. L-proline (1.00 g) and *p*-toluenesulfonyl chloride (1.70 g, 1.02 equiv) were dissolved in 30.0 mL of dry DCM, and triethylamine (3.00 mL, 2.50 equiv) was then added dropwise. The resulting mixture was stirred at room temperature for 16 h. Dilute hydrochloric acid (0.50 M) was added to the mixture till pH = 3. The mixture was washed with brine, then extracted with DCM. The organic layer was dried over anhydrous magnesium sulfate. After evaporation of DCM, *N*-Ts-L-proline was obtained as white solid in 78% yield and used for the next step without further purification. The white solid (1.83 g) was dissolved in 10.0 mL of dry DCM, thionyl chloride (1.00 mL, 2.00 equiv) was added, and the resulting mixture was stirred under nitrogen atmosphere at room temperature for 16 h. The solvent was then evaporated under reduced pressure to give a yellow viscous liquid which was then dissolved in 30.0 mL of dry DCM. 4-Aminophenylboronic acid pinacol ester (0.60 g) was added, followed by triethylamine (3.00 mL). The resulting mixture was stirred at 0 °C for 1 h. After evaporation of the solvent under reduced pressure, the crude product was purified by flash column chromatography on silica gel using 2:1(v/v) hexanes:ethyl acetate as eluent to give pure **7** as a pale yellow solid in 88% yield (1.13 g). D-proline and racemic DL-proline derivatives were prepared using the same procedure.

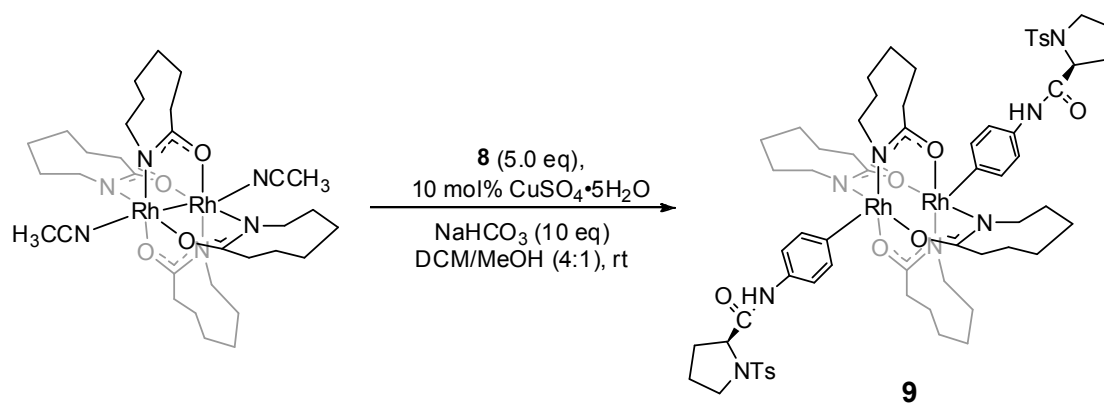
7. ^1H NMR (400 MHz, CDCl_3): δ 8.83 (s, 1H), 7.71-7.76 (dd, 4H, J = 12.0, 8.0 Hz), 7.56-7.58 (d, 2H, J = 8.0 Hz), 7.32-7.34 (d, 2H, J = 8.0 Hz), 4.13-4.16 (m, 1H), 3.55-3.60 (m, 1H), 3.18-3.24 (m, 1H), 2.40 (s, 3H), 2.27-2.32 (m, 1H), 1.48-1.82 (comp, 3H), 1.30 (s, 12H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.1, 144.7, 140.1, 135.7, 132.5, 130.1, 127.9, 118.7, 83.7, 63.0, 50.1, 29.5, 24.8, 24.4, 21.6; HRMS (ESI) for $\text{C}_{24}\text{H}_{32}\text{BN}_2\text{O}_5\text{S}$ $[\text{M}+\text{H}]^+$ calcd: 471.2125; found: 471.2039; IR (neat): 3265, 2989, 2972, 1681, 1598, 1538, 1396, 1342 cm^{-1} .



Procedure for Synthesis of 8. Compound **7** (470 mg, 1.00 mmol) and sodium periodate (642 mg, 3.00 equiv) were stirred in 8.00 mL of a 4:1 mixture of THF and

water for 1 h. Aqueous hydrochloric acid (1.00 M, 0.70 ml) was then added to the suspension; and the resulting yellow mixture was stirred at ambient temperature for 6 h, then diluted with water and extracted with ethyl acetate. The combined organic layer was washed with water and brine, then dried over sodium sulfate. After evaporation of solvent, **8** was obtained as light yellow solid in 92% yield (360 mg). D-proline and racemic DL-proline derivatives were prepared using the same procedure.

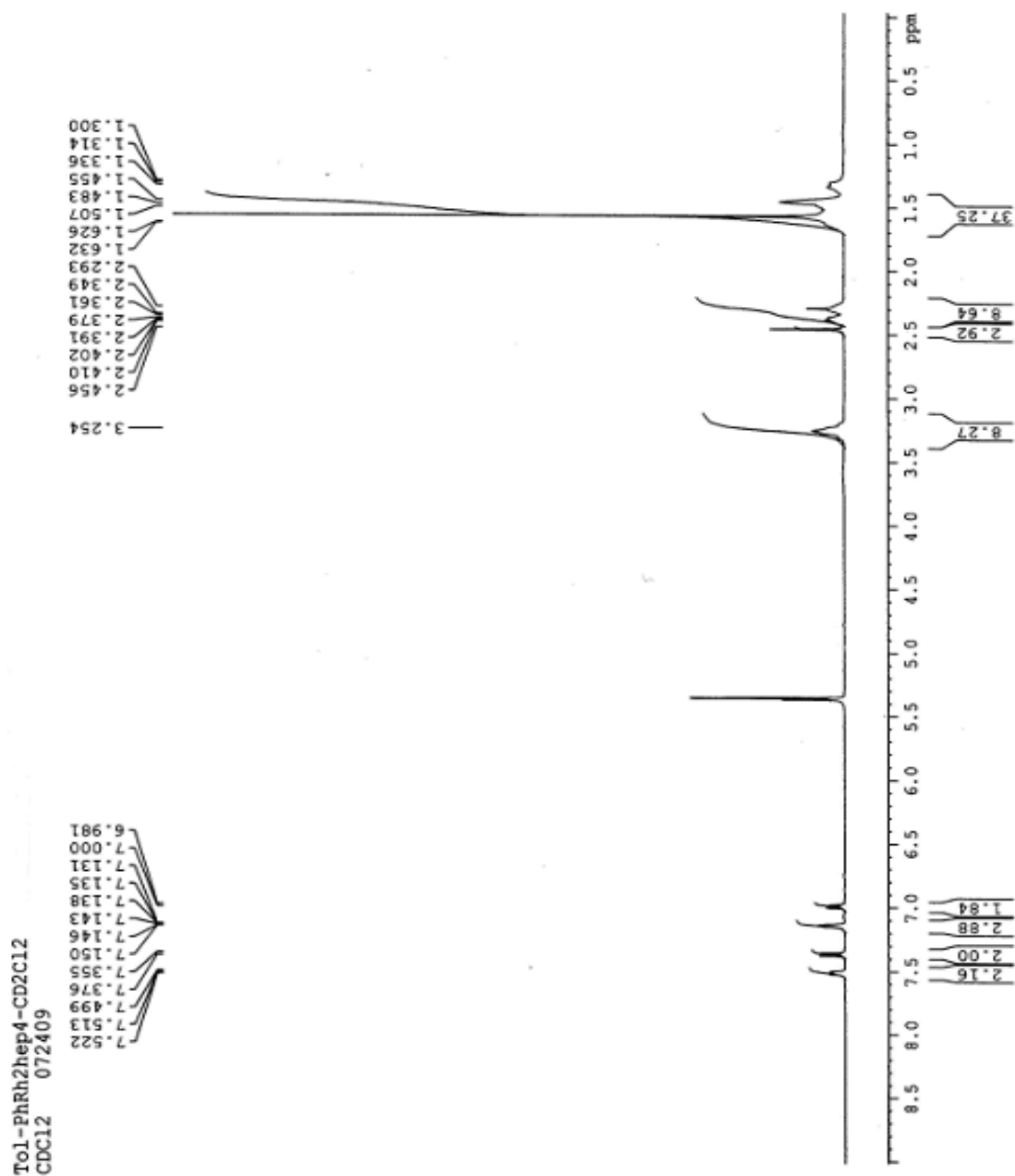
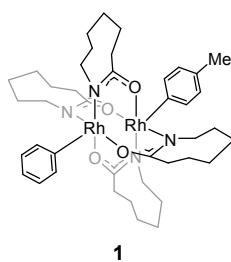
8. ^1H NMR (400 MHz, CDCl_3): δ 8.94 (s, 1H), 8.17 (d, 2H, $J = 8.0$ Hz), 7.70-7.76 (dd, 4H, $J = 16.0, 8.0$ Hz), 7.36 (d, 2H, $J = 8.0$ Hz), 4.19-4.22 (m, 1H), 3.57-3.65 (m, 1H), 3.22-3.28 (m, 1H), 2.42 (s, 3H), 2.25-2.35 (m, 1H), 1.81-1.84 (m, 1H), 1.67-1.80 (comp, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ 171.2, 144.7, 141.3, 136.7, 134.9, 132.5, 130.1, 127.9, 119.0, 63.0, 60.4, 24.5, 21.6, 21.0; HRMS (ESI) for $\text{C}_{18}\text{H}_{22}\text{BN}_2\text{O}_5\text{S}$ $[\text{M}+1]^+$ calcd: 389.1342; found: 389.1316; IR (neat): 3378, 3338, 2974, 2954, 1686, 1595, 1532, 1327 cm^{-1} .

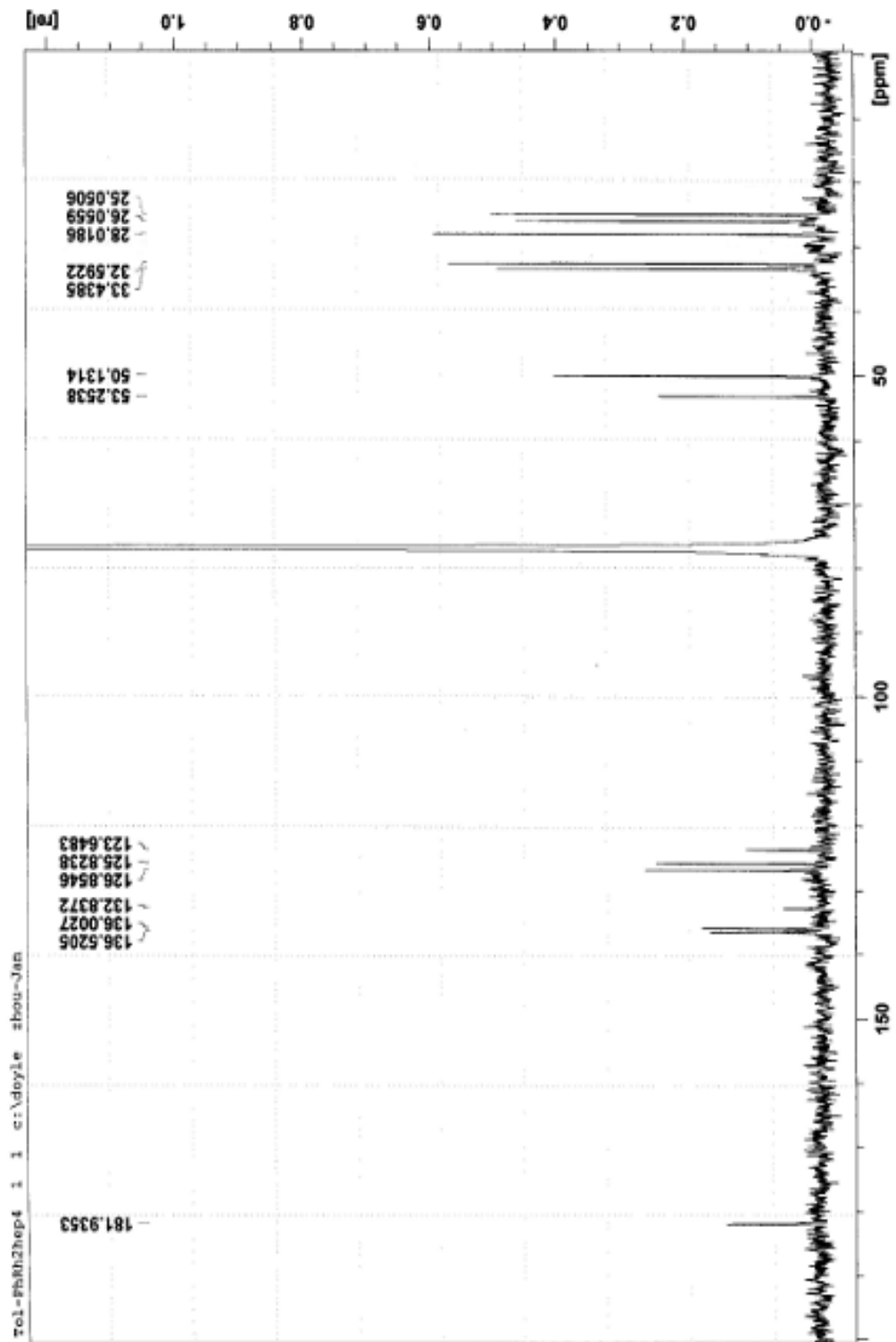


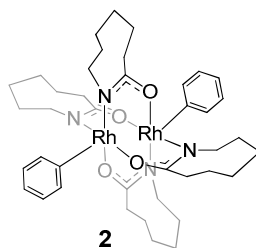
Procedure for Synthesis of 9. $\text{Rh}_2(\text{ACO})_4(\text{CH}_3\text{CN})_2$ (48.0 mg, 0.06 mmol), compound **8** (116 mg, 5.00 equiv) and NaHCO_3 (50.4 mg, 10.0 equiv) were dissolved in 6.00 mL of CH_2Cl_2 . Copper sulfate (10.0 mol%) in 1.50 mL of MeOH was then added to the above solution, and the resulting purple mixture was stirred under air at room temperature for 12 h, during which the solution turned yellowish brown. After filtration of NaHCO_3 and evaporation of the solvent under reduced pressure, the crude product was purified by column chromatography on silica gel using 1:2 hexanes:ethyl acetate as the eluent to give **9** as a yellowish brown solid in 40% yield (34.0 mg). D-proline and racemic DL-proline derivatives were prepared using the same procedure.

9. ^1H NMR (400 MHz, CDCl_3): δ 8.72 (s, 2H), 7.73-7.78 (comp, 4H), 7.34-7.48 (comp, 8H), 6.85-6.87 (comp, 4H), 4.14-4.22 (comp, 2H), 3.78 (s, 4H), 3.58-3.63 (comp, 2H), 3.19-3.25 (comp, 4H), 2.44 (s, 6H), 2.26-2.34 (comp, 4H), 1.34-1.81 (comp, 14H); ^{13}C NMR (150 MHz, CDCl_3): δ 182.0, 168.5, 156.5, 144.4, 136.3, 132.6, 130.5, 129.9, 128.8, 127.8, 121.6, 119.9, 117.7, 114.0, 62.8, 55.4, 50.0, 33.4, 32.6, 29.6, 28.0, 26.0, 25.0, 24.3, 21.5; HRMS (ESI) for $\text{C}_{64}\text{H}_{86}\text{N}_8\text{NaO}_{10}\text{Rh}_2\text{S}_2$ $[\text{M}+\text{Na}]^+$ calcd: 1419.3916; found: 1419.3378; IR (neat): 3342, 2927, 1677, 1597, 1581, 1510, 1342, 1158 cm^{-1} .

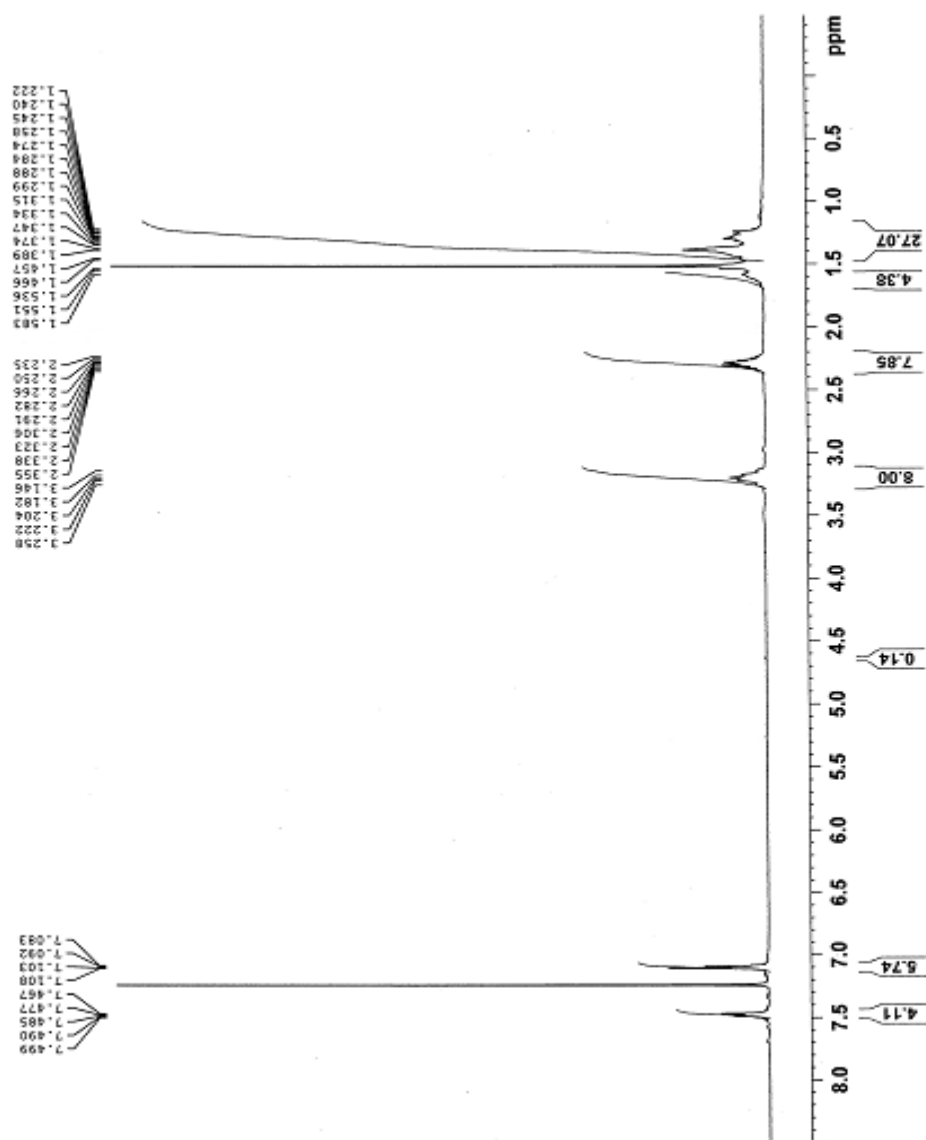
NMR Spectra

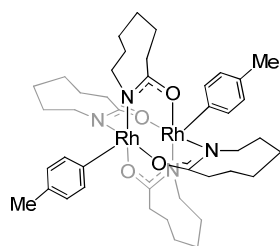




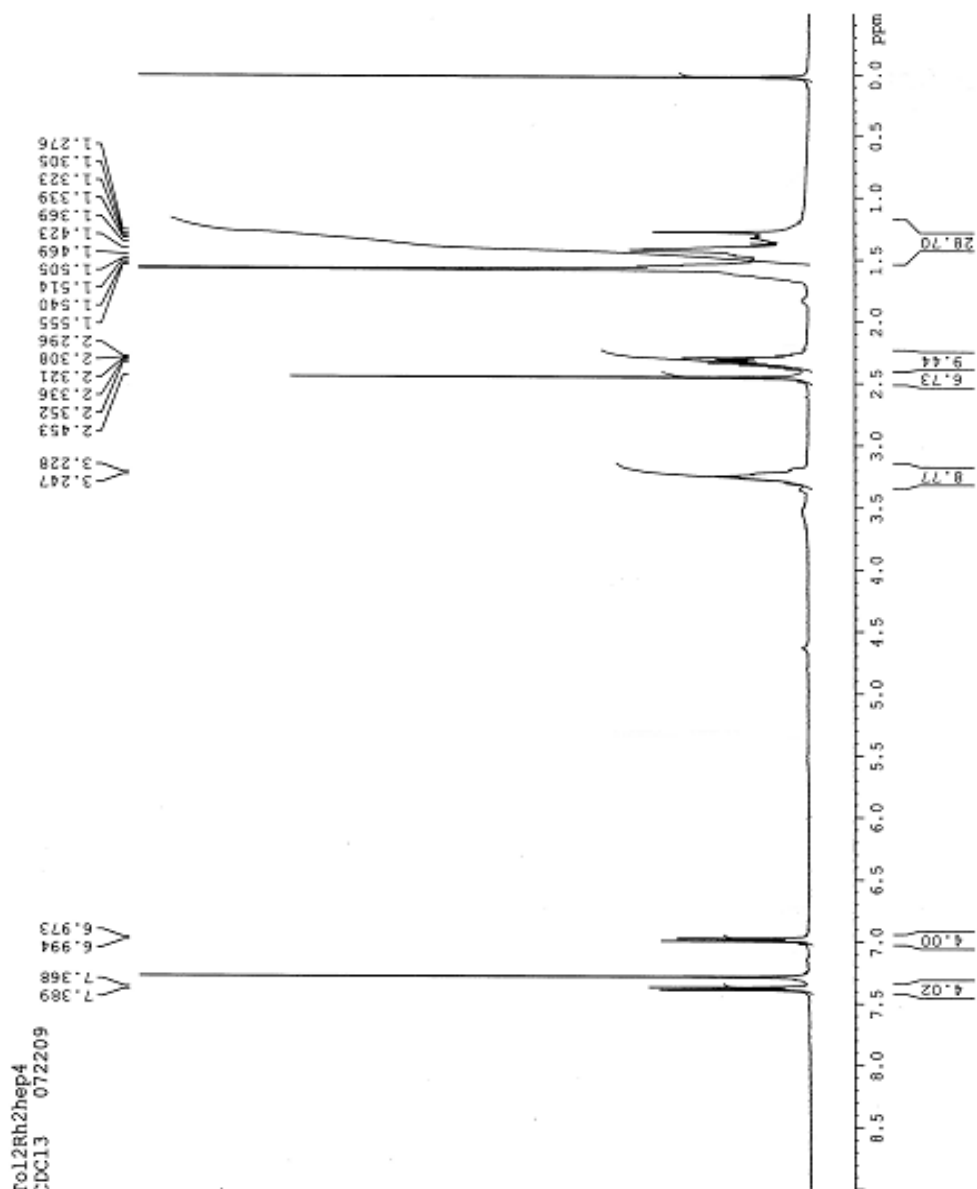


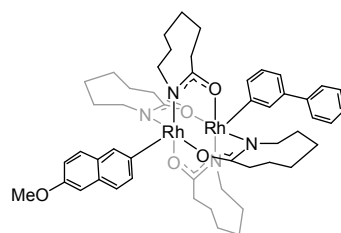
Ph2Rh2hep4 pure
CDC13 110908



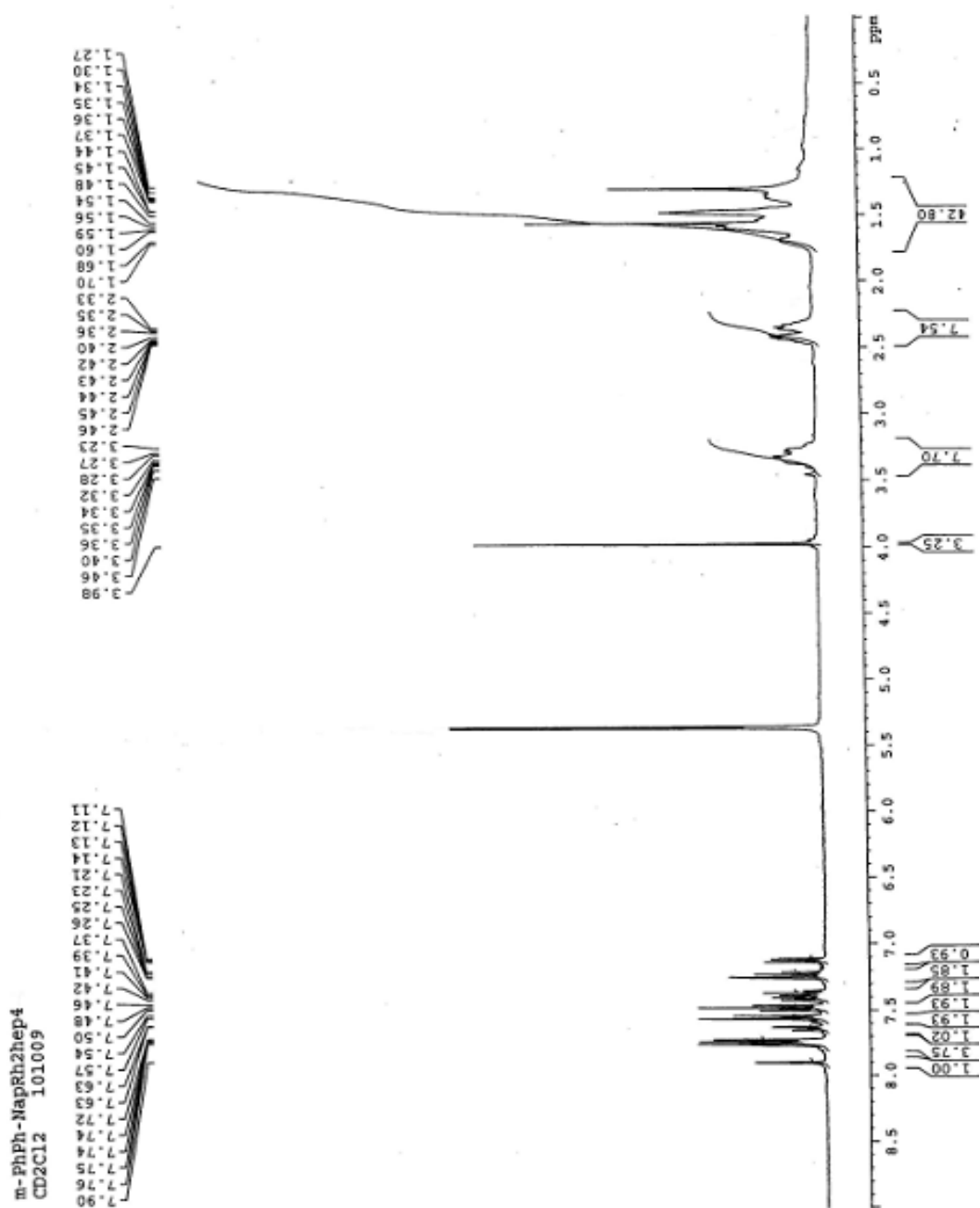


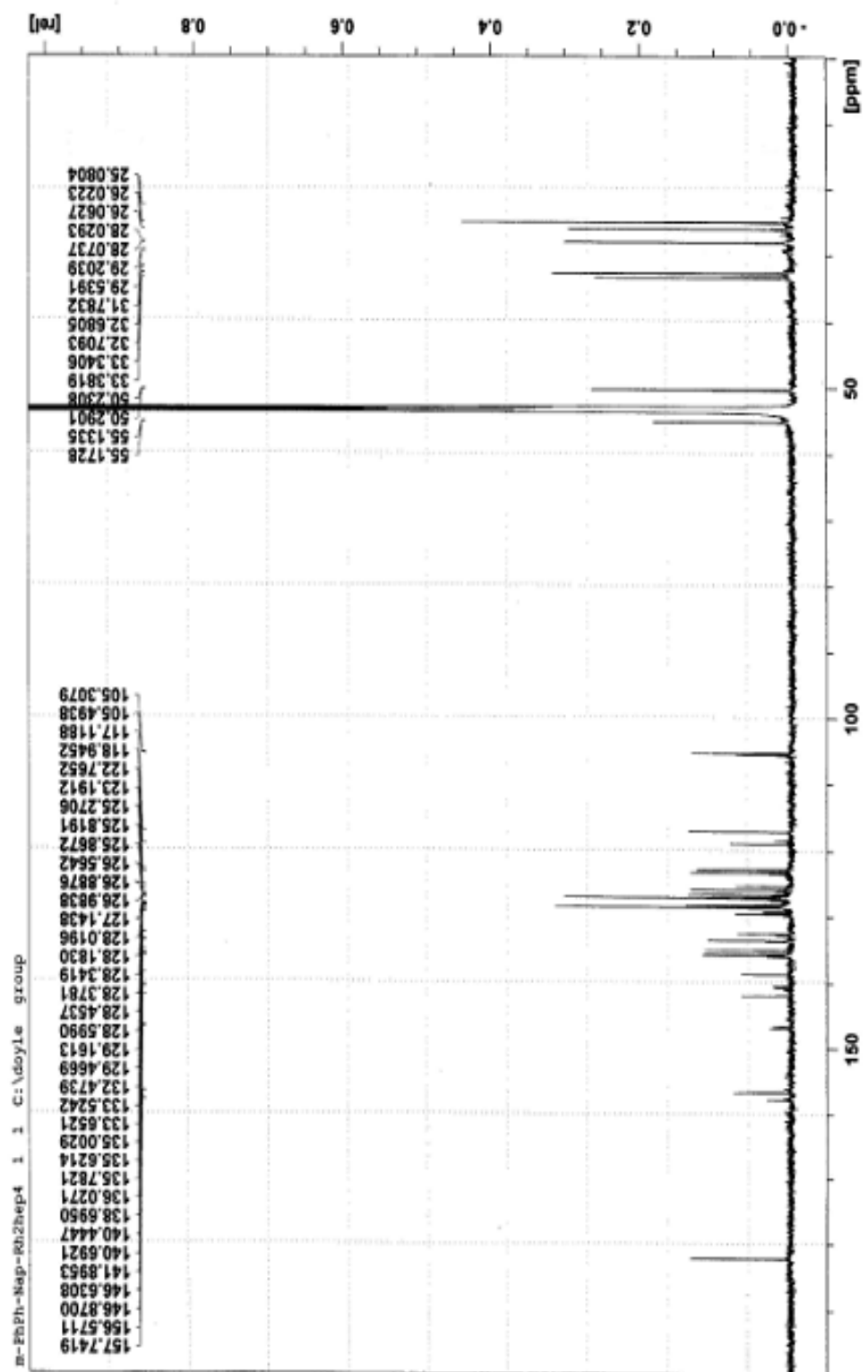
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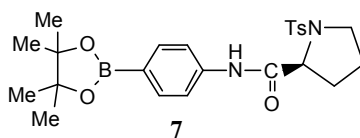




4

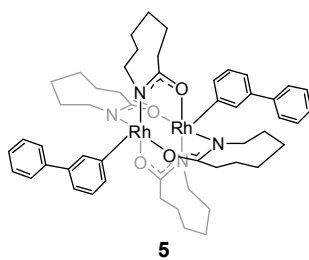
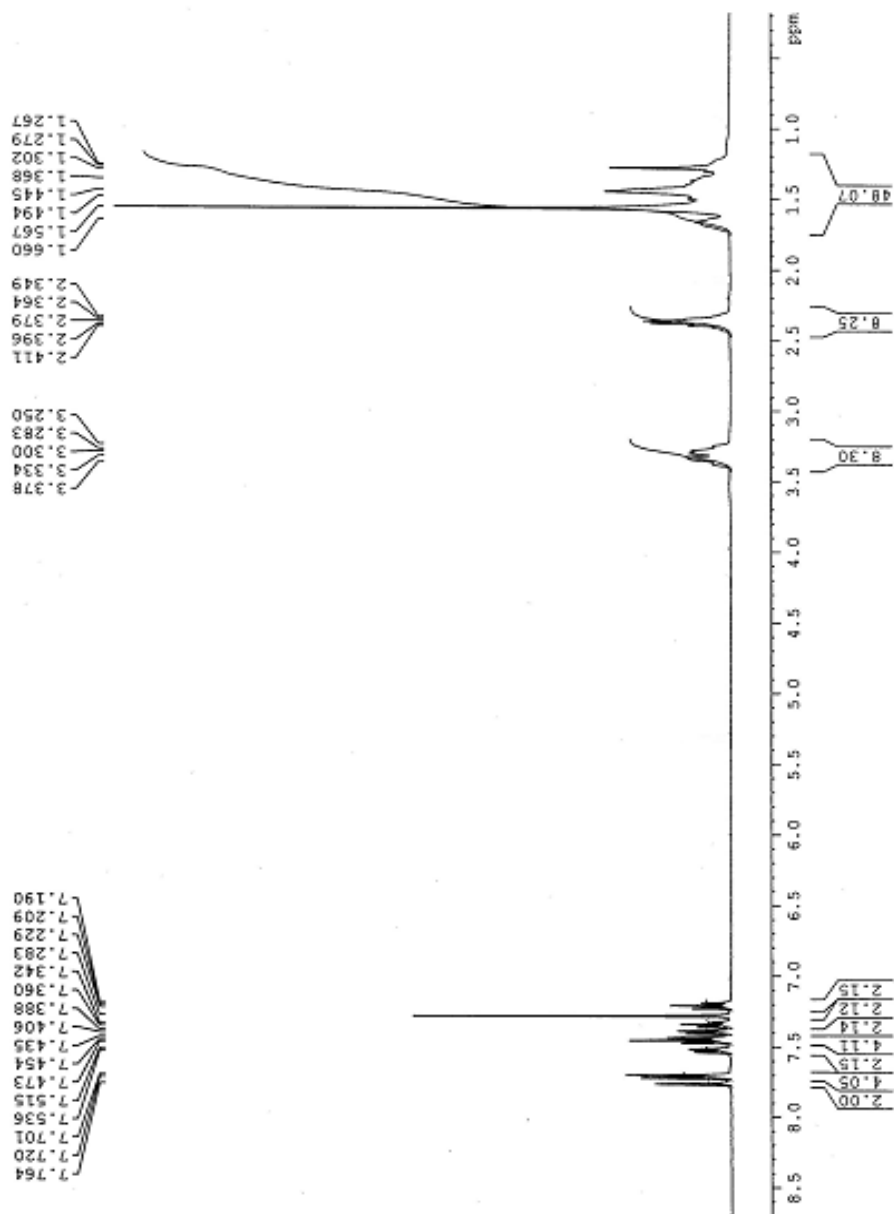


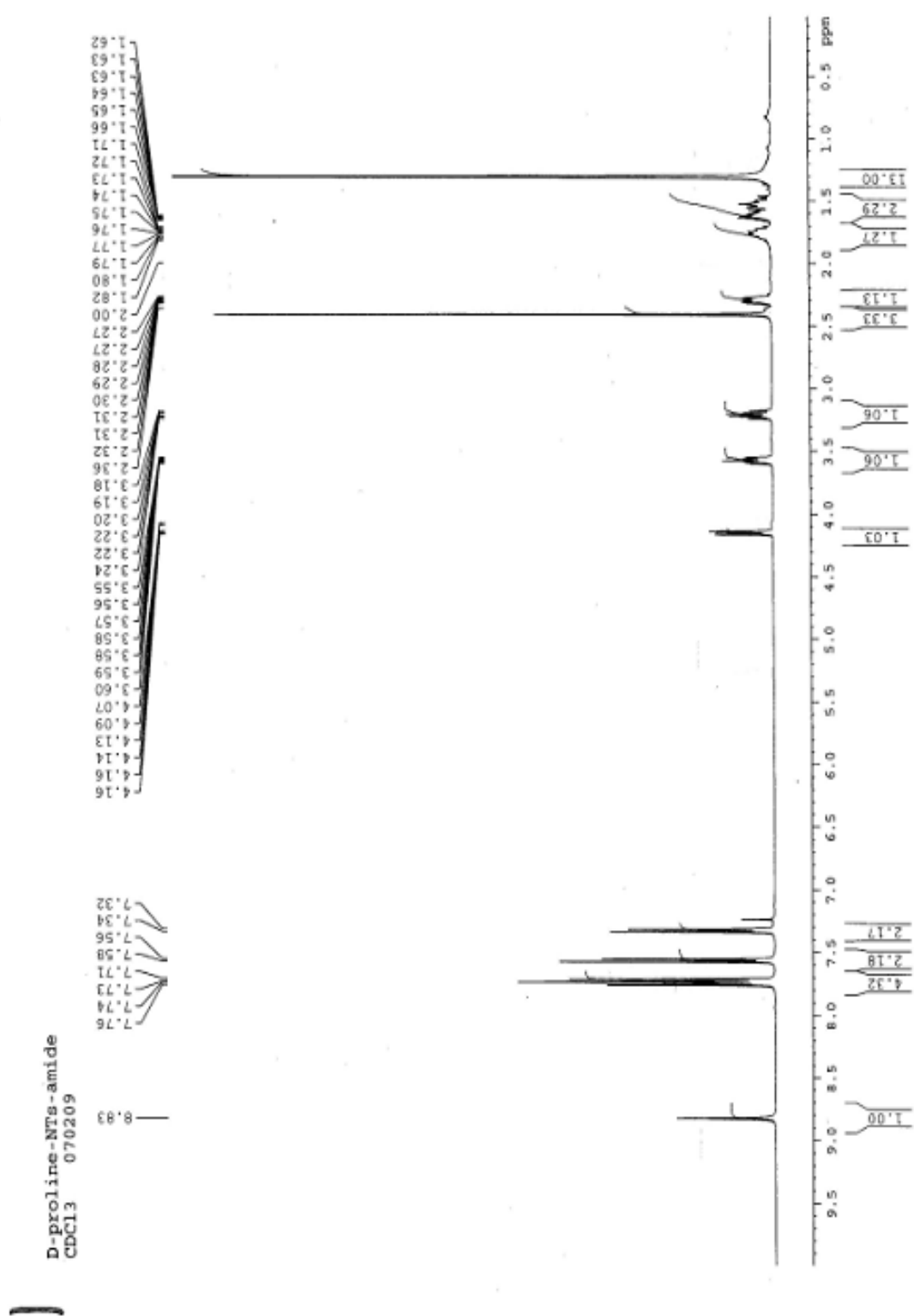


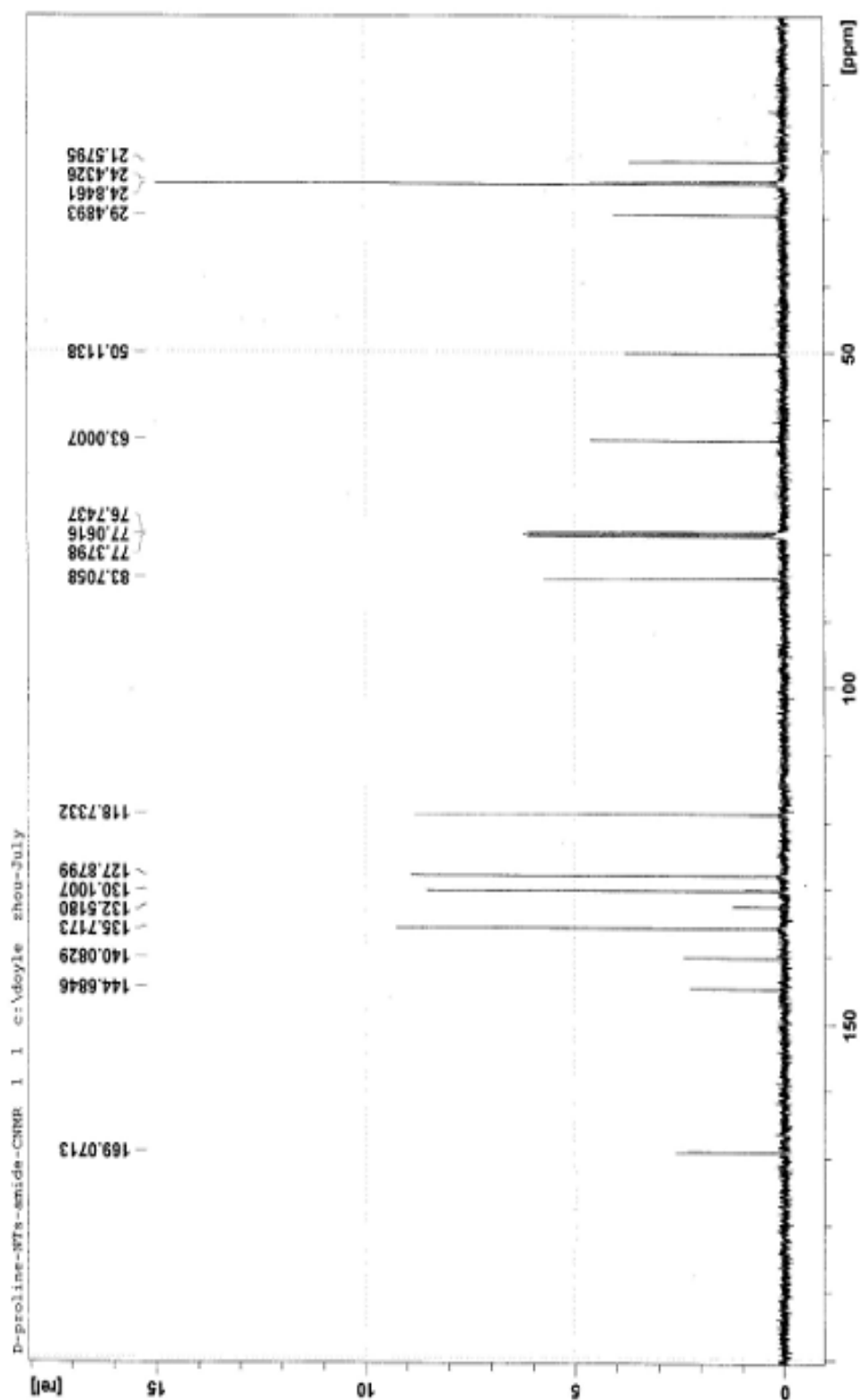


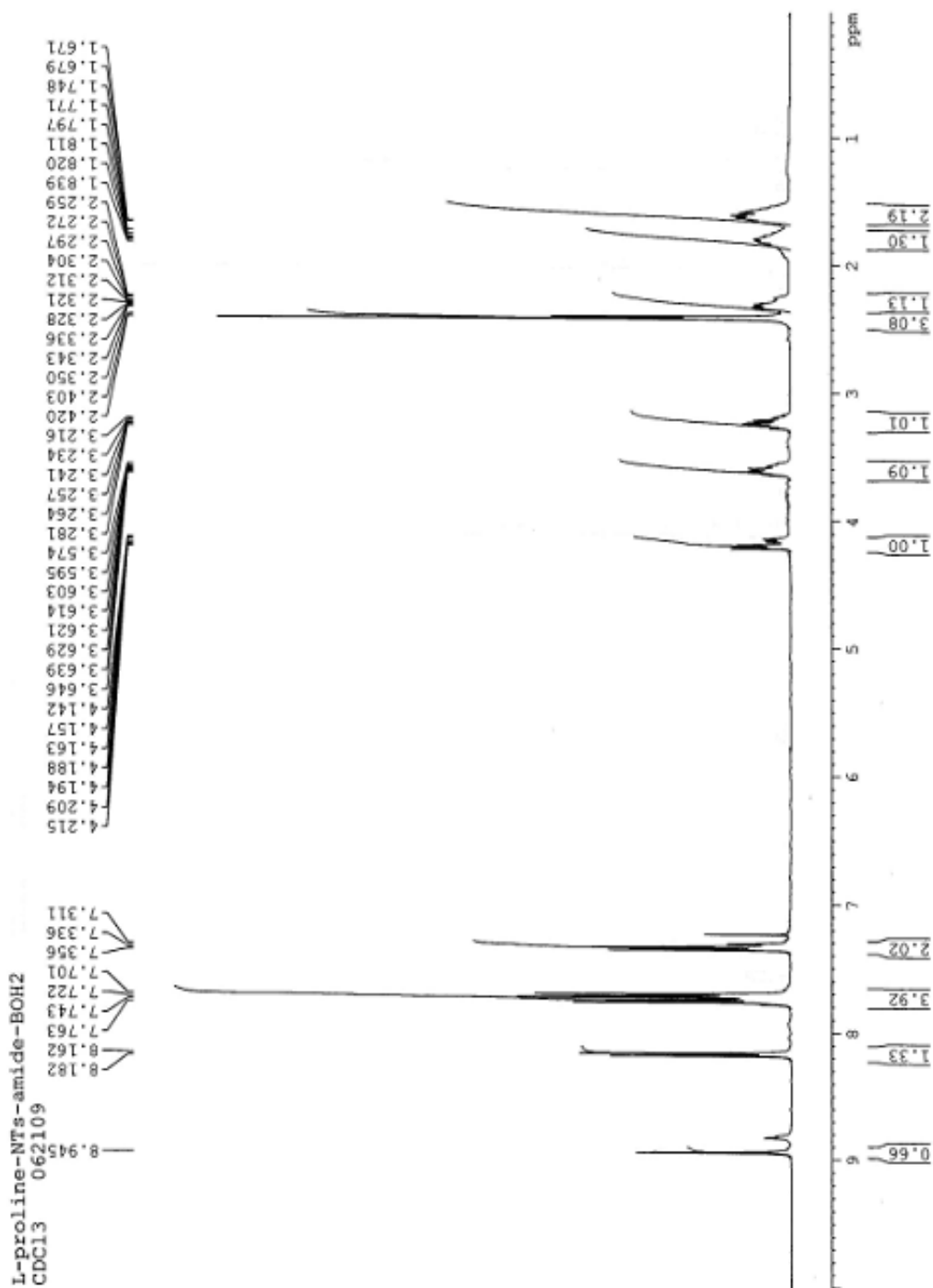
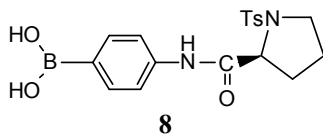
S13

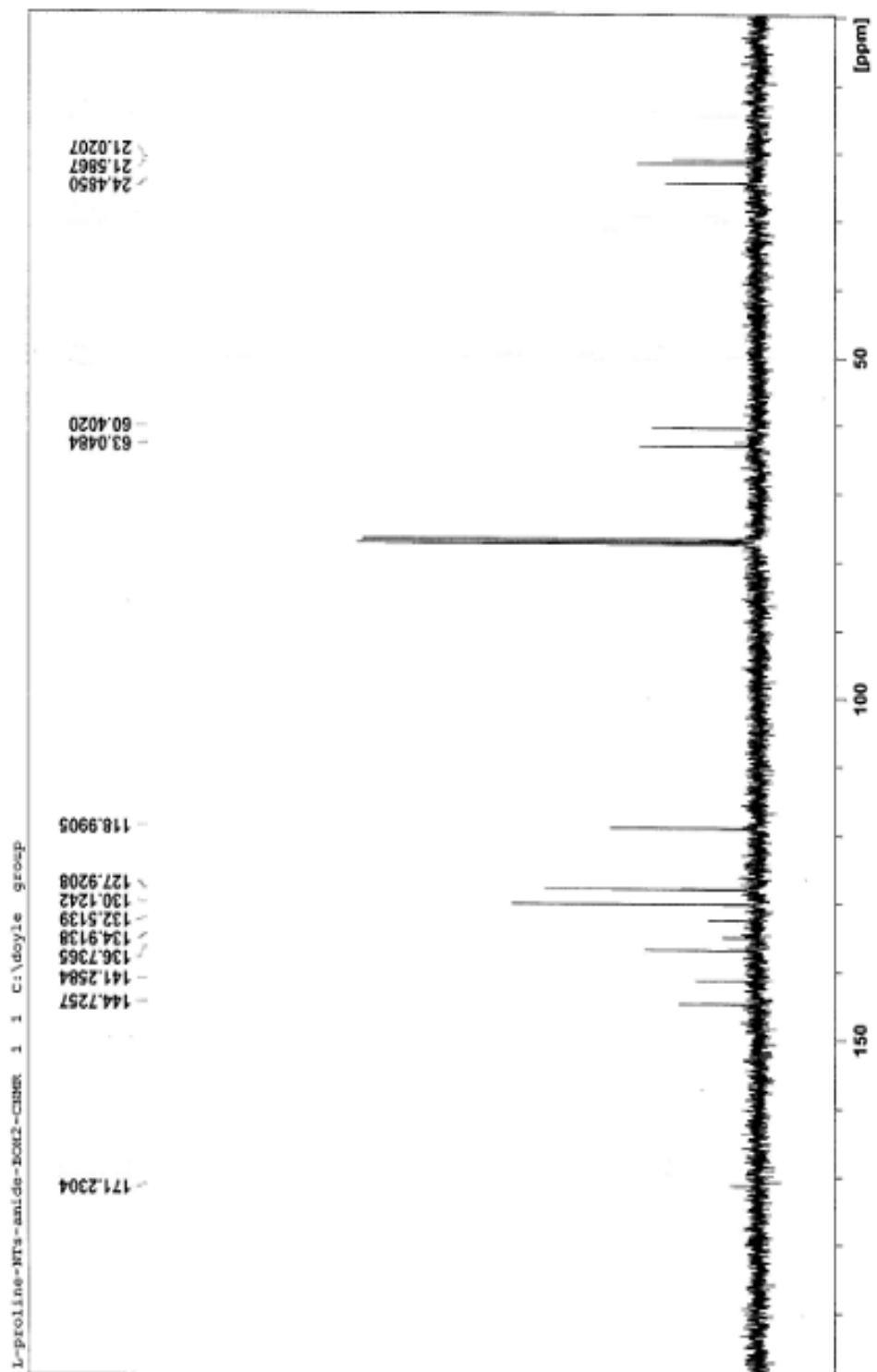
¹H NMR spectrum (CDCl₃) of compound 5. The spectrum shows peaks in the aromatic region (7.64-7.19 ppm) and aliphatic region (1.26-1.60 ppm). Integration values are provided for several peaks.



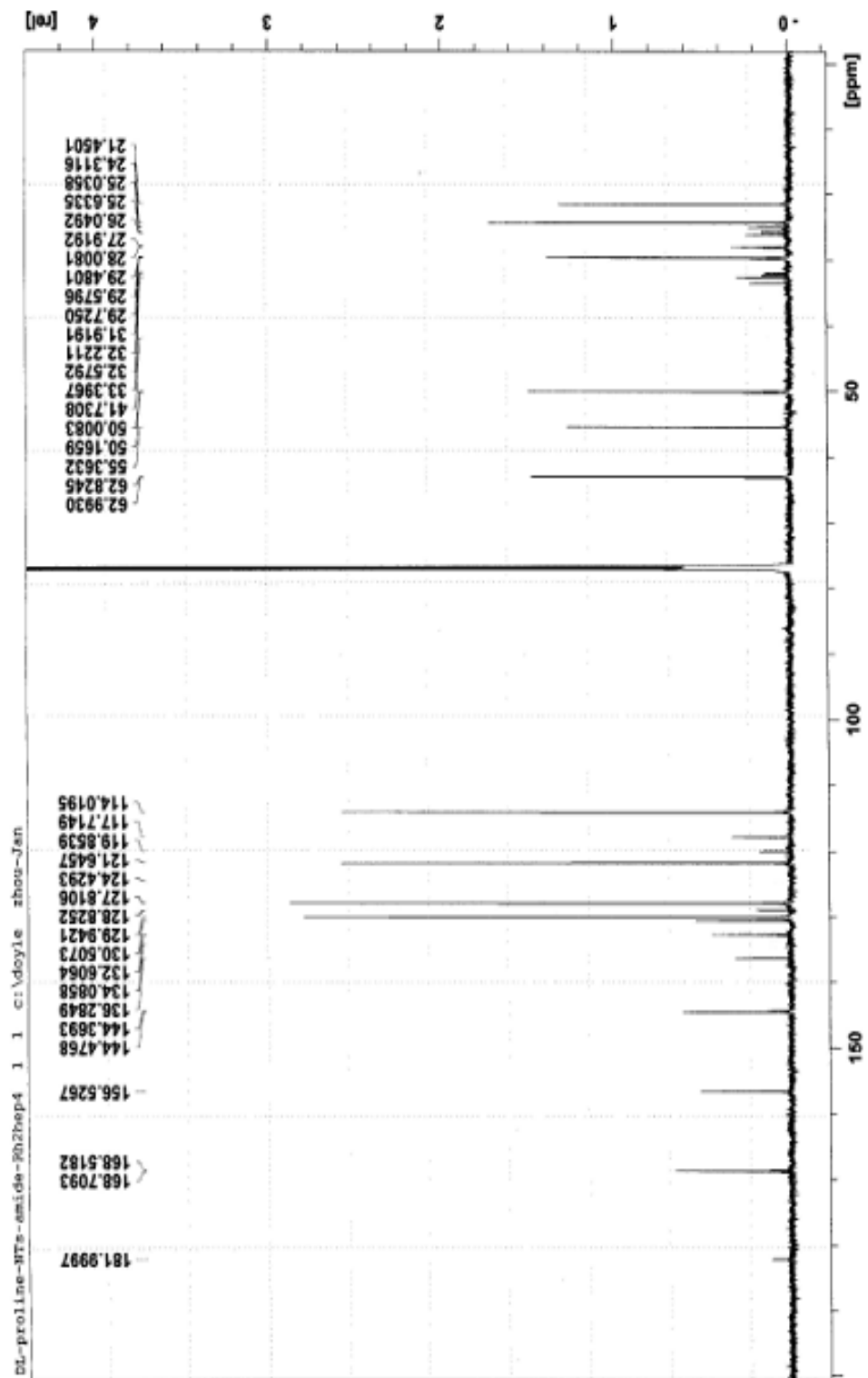




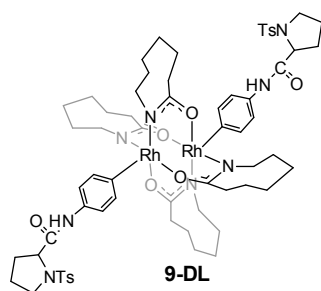




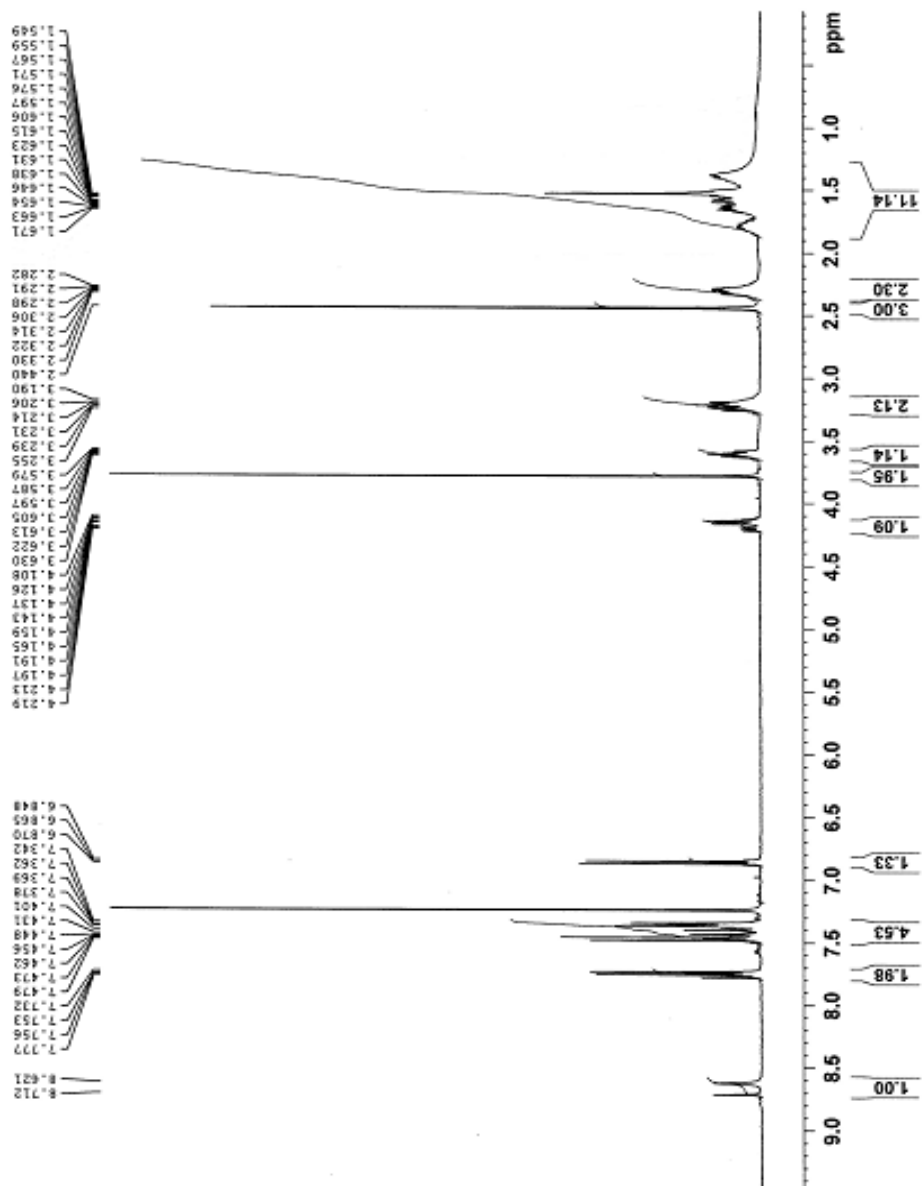




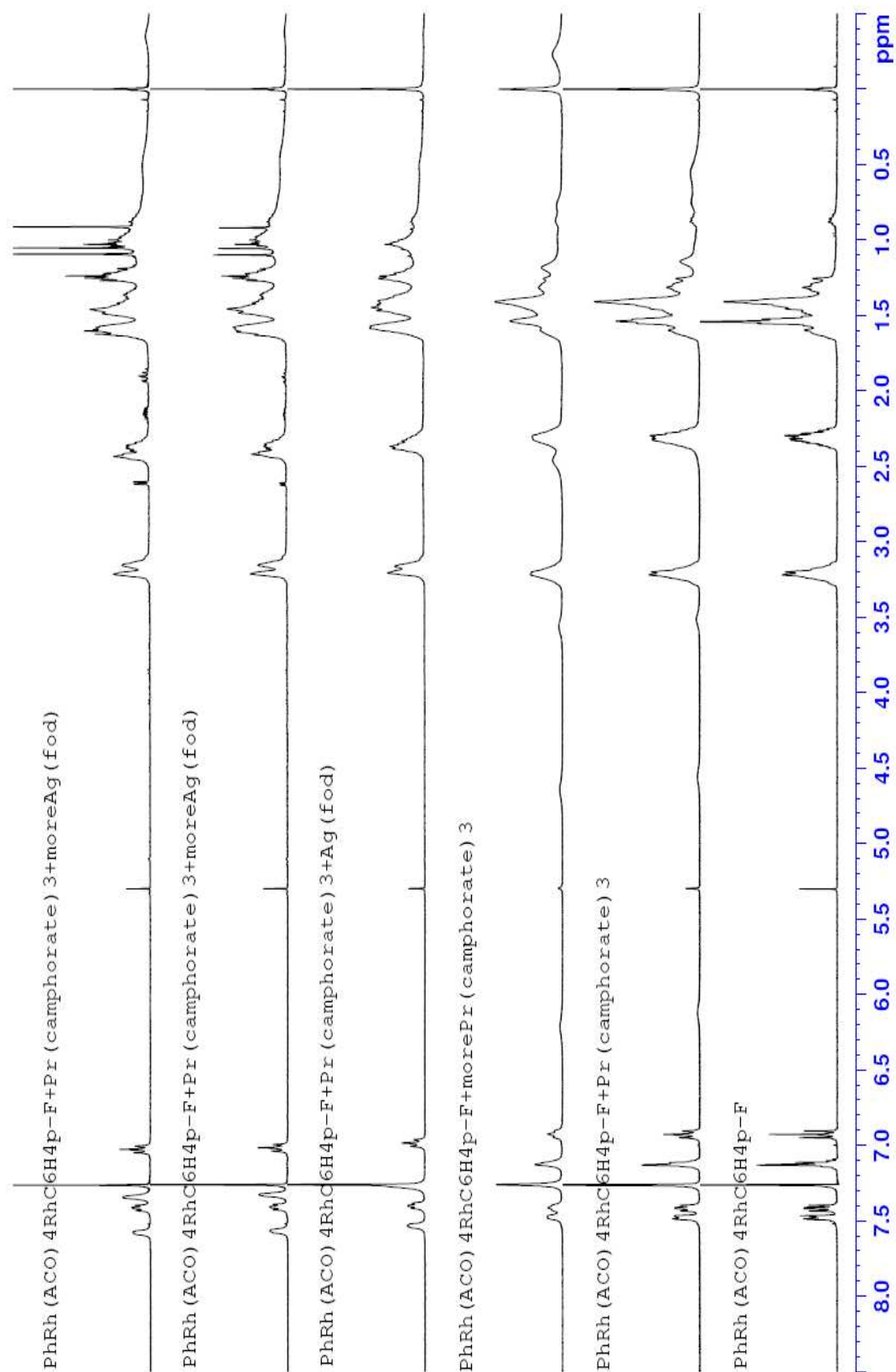




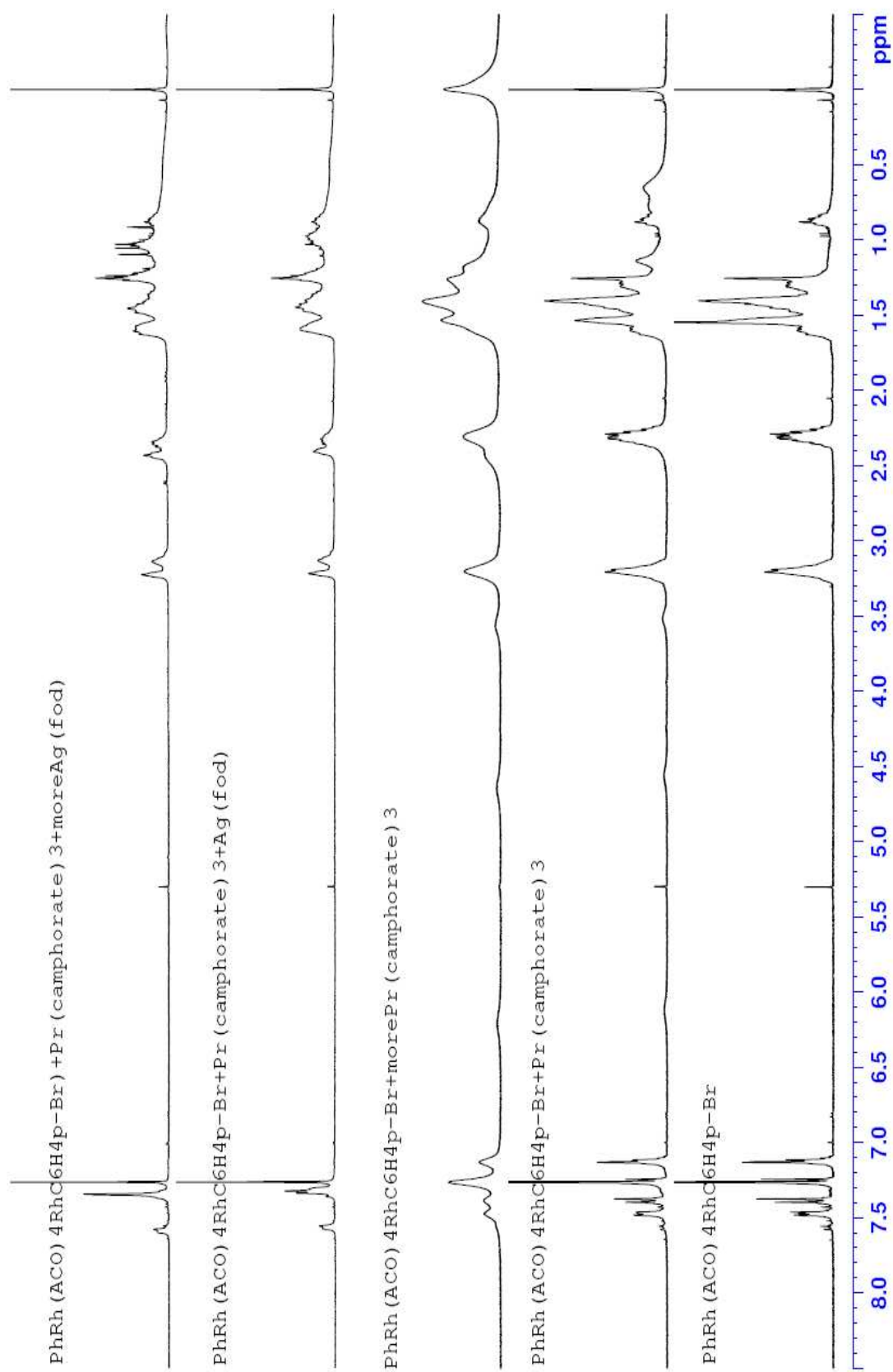
DL-proline-NTs-amide-Rh₂hep4-old CDCl₃



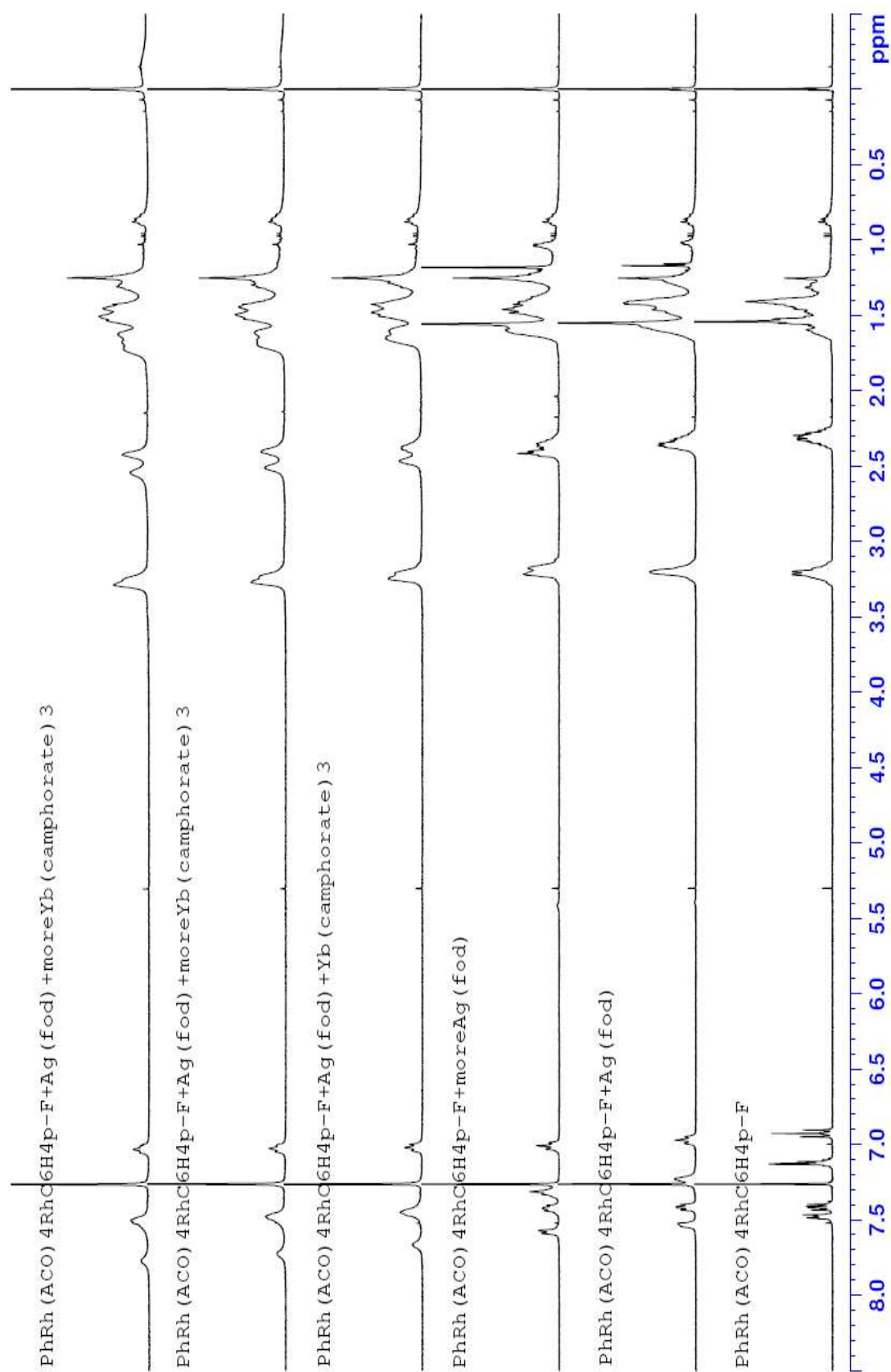
NMR Spectra of Dirhodium Complexes with Chiral Shift Reagents
(4-FC₆H₄)Rh(ACO)₄RhPh (10) + 13a + 14



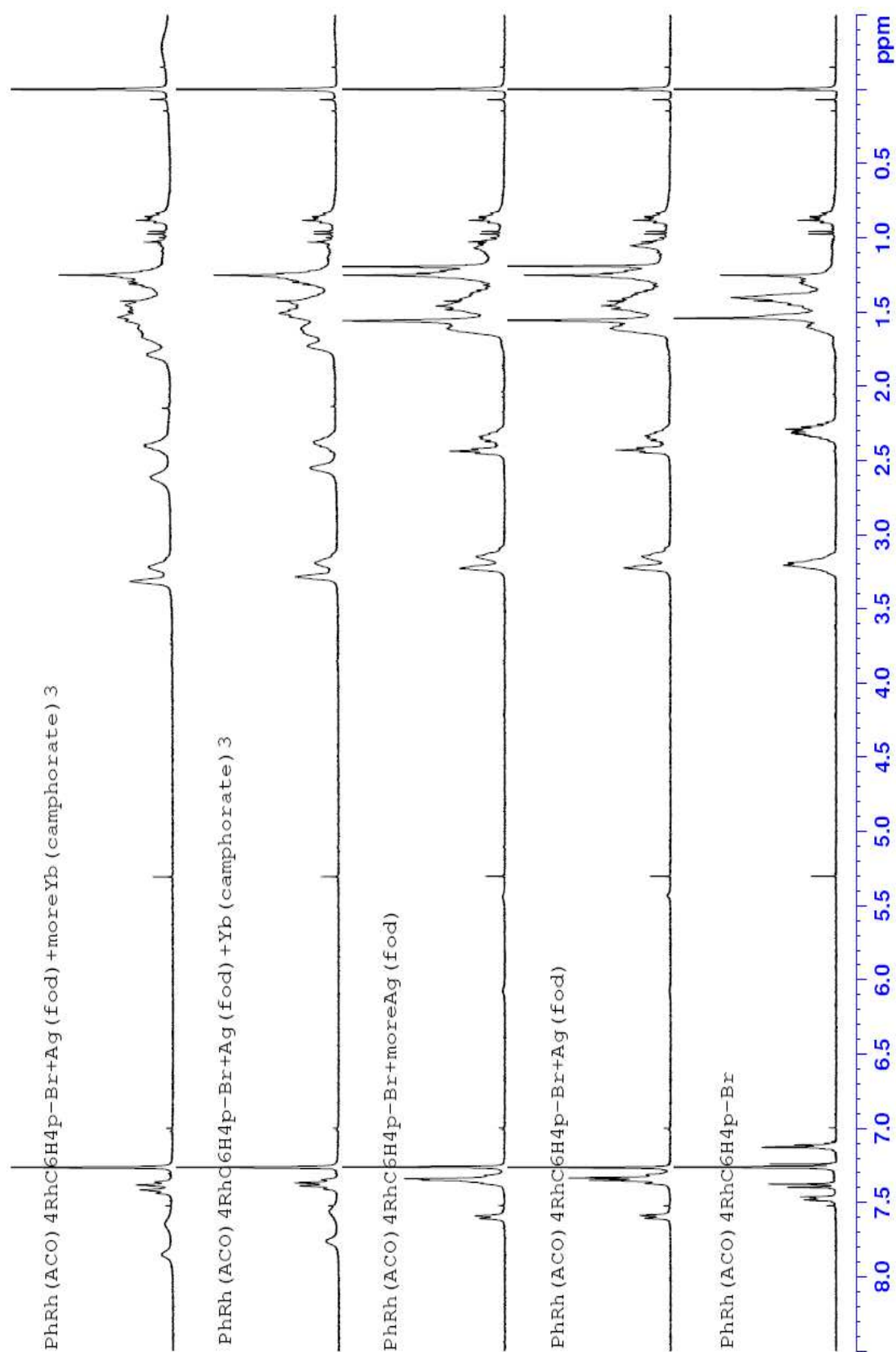
(4-BrC₆H₄)Rh(ACO)₄RhPh (11) + 13a + 14



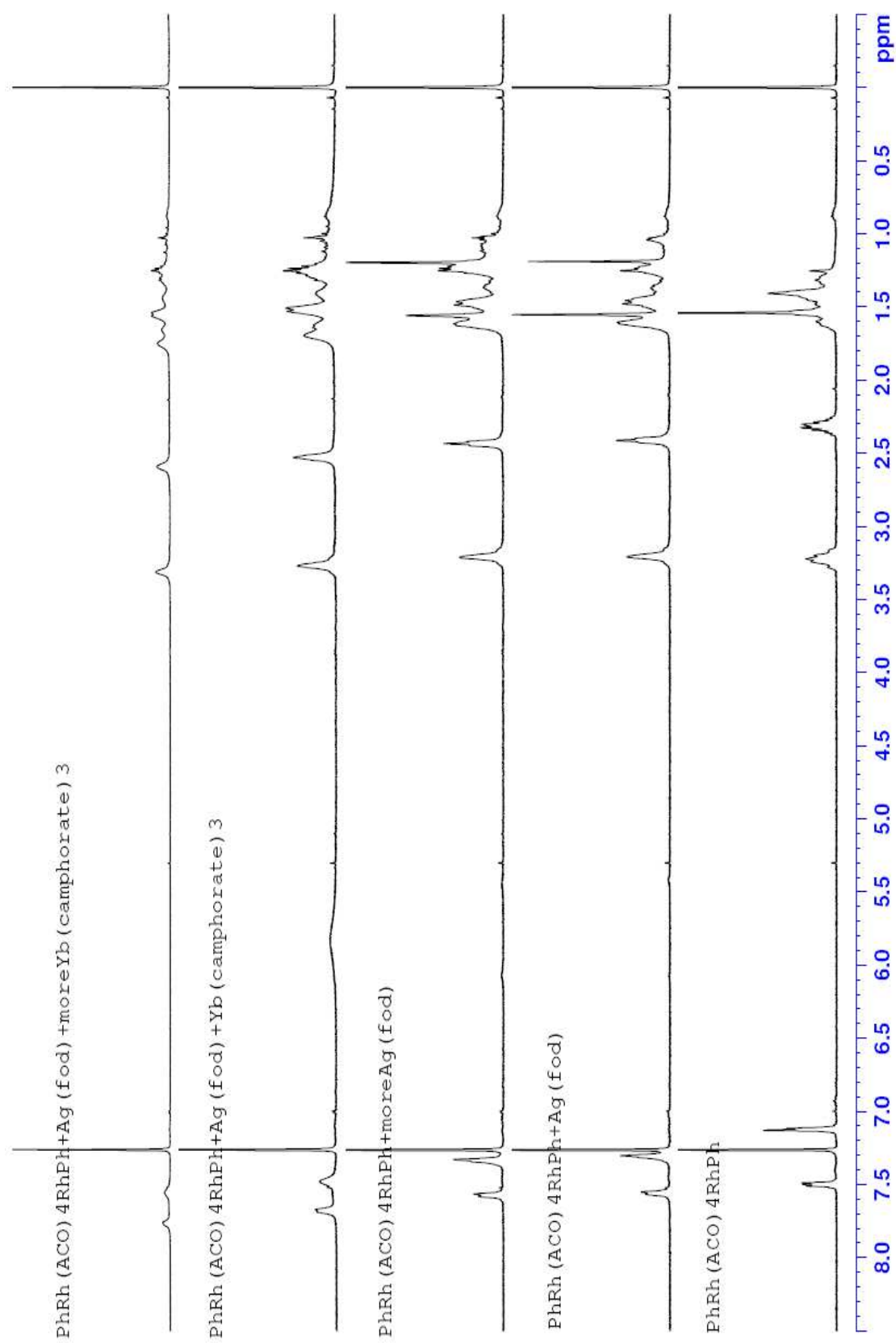
(4-FC₆H₄)Rh(ACO)₄RhPh (**10**) + **13b** + **14**



(4-BrC₆H₄)Rh(ACO)₄RhPh (11) + 13b + 14



PhRh(ACO)₄RhPh (11) + 13b + 14



X-Ray data for 1

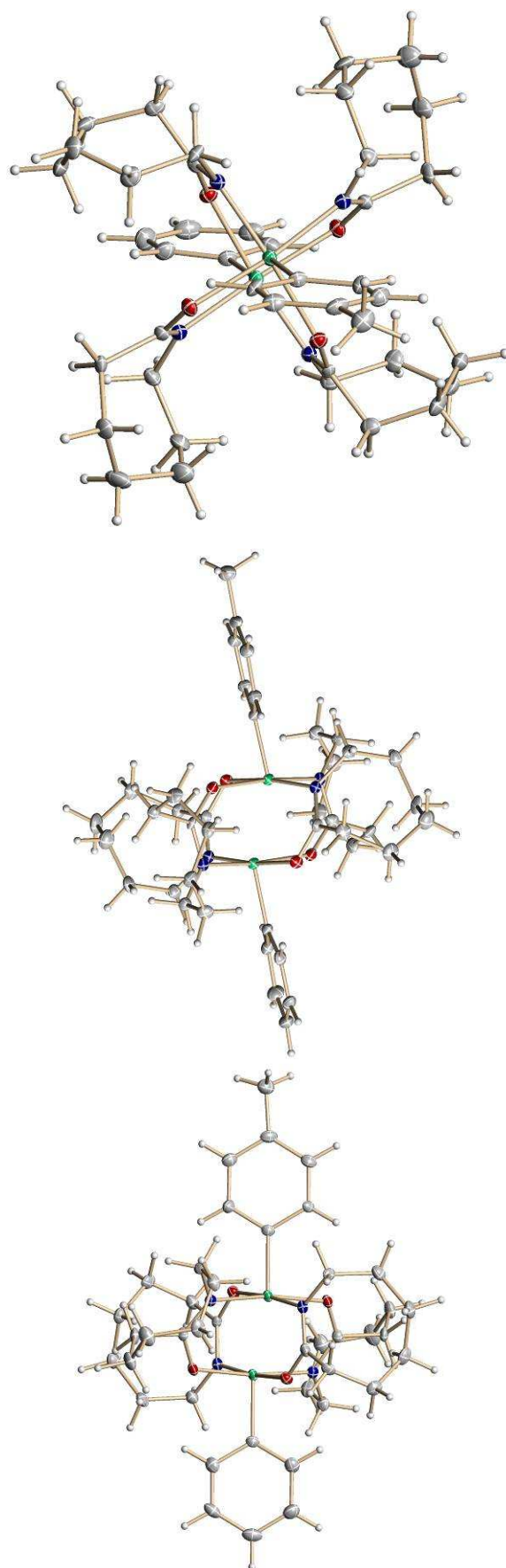


Figure S1. A view of complex **1**. Anisotropic atomic displacement ellipsoids for the non-hydrogen atoms are shown at the 30% probability level. Hydrogen atoms are displayed with an arbitrarily small radius.

A yellow plate of $_{41}\text{H}_{60}\text{N}_4\text{O}_4\text{Rh}_2$, approximate dimensions $0.017 \times 0.135 \times 0.26 \text{ mm}^3$, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 200(2) K on a three-circle diffractometer system equipped with Bruker Smart Apex II CCD area detector using a graphite monochromator and a $\text{MoK}\alpha$ fine-focus sealed tube ($\lambda = 0.71073 \text{ \AA}$). The detector was placed at a distance of 5.00 cm from the crystal.

A total of 1795 frames were collected with a scan width of -0.5° in ω and an exposure time of 60 sec/frame using Apex2 (Bruker, 2005). The total data collection time was 32.9 hours. The frames were integrated with Apex2 software package using a narrow-frame integration algorithm. The integration of the data using a Monoclinic unit cell yielded a total of 20763 reflections to a maximum θ angle of 25.00° , of which 6681 were independent (completeness = 99.7%, $R_{\text{int}} = 4.05\%$, $R_{\text{sig}} = 4.26\%$) and 5800 were greater than $2\sigma(I)$. The final cell dimensions of $a = 8.8987(10) \text{ \AA}$, $b = 20.4220(22) \text{ \AA}$, $c = 11.1073(12) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 109.1585(16)^\circ$, $\gamma = 90^\circ$, $V = 1906.7(4) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of 10349 reflections with $2.2 < \theta < 28.2^\circ$ using Apex2. Analysis of the data showed 0 % decay during data collection. Data were corrected for absorption effects with the Semi-empirical from equivalents method using SADABS (Sheldrick, 1996). The minimum and maximum transmission coefficients were 0.791 and 0.985.

The structure was solved and refined using the SHELXS-97 (Sheldrick, 1990) and SHELXL-97 (Sheldrick, 1997) software in the space group $P2_1$ with $Z = 2$ for the formula unit $_{41}\text{H}_{60}\text{N}_4\text{O}_4\text{Rh}_2$. The final anisotropic full-matrix least-squares refinement on F^2 with 402 variables converged at $R_1 = 4.52\%$ for the observed data and $wR_2 = 9.98\%$ for all data. The goodness-of-fit was 1.000. The largest peak on the final difference map was $1.420 \text{ e/\AA}^3$ and the largest hole was $-0.991 \text{ e/\AA}^3$. On the basis of the final model, the calculated density was 1.531 g/cm^3 and $F(000)$, 912 e.

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

Empirical formula	C41 H60 N4 O4 Rh2	
Formula weight	878.75	
Temperature	200(2) K	
Wavelength	0.71073 $\text{\AA}$	
Crystal size	$0.26 \times 0.135 \times 0.017 \text{ mm}^3$	
Crystal habit	yellow plate	
Crystal system	Monoclinic	
Space group	$P2_1$	
Unit cell dimensions	$a = 8.8987(10) \text{ \AA}$	$\alpha = 90^\circ$
	$b = 20.4220(22) \text{ \AA}$	$\beta = 109.1585(16)^\circ$
	$c = 11.1073(12) \text{ \AA}$	$\gamma = 90^\circ$
Volume	$1906.7(4) \text{ \AA}^3$	
Z	2	
Density, ρ_{calc}	1.531 g/cm^3	
Absorption coefficient, μ	0.912 mm^{-1}	
$F(000)$	912 e	
Diffractometer	Bruker Smart Apex II CCD area detector	
Radiation source	fine-focus sealed tube, $\text{MoK}\alpha$	
Detector distance	5.00 cm	
Detector resolution	11.198 pixels/mm	
Total frames	1795	
Frame size	512 pixels	

Frame width	-0.5°
Exposure per frame	60 sec
Total measurement time	32.9 hours
Data collection method	ω and ϕ scans
θ range for data collection	1.94 to 25.00°
Index ranges	$-10 \leq h \leq 10$, $-24 \leq k \leq 24$, $-13 \leq l \leq 13$
Reflections collected	20763
Independent reflections	6681
Observed reflection, $I > 2\sigma(I)$	5800
Coverage of independent reflections	99.7 %
Variation in check reflections	0 %
Absorption correction	Semi-empirical from equivalents SADABS (Sheldrick, 1996)
Max. and min. transmission	0.985 and 0.791
Structure solution technique	direct
Structure solution program	SHELXS-97 (Sheldrick, 1990)
Refinement technique	Full-matrix least-squares on F^2
Refinement program	SHELXL-97 (Sheldrick, 1997)
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$
Data / restraints / parameters	6681 / 448 / 402
Goodness-of-fit on F^2	0.982
$\Delta/\sigma_{\max}$	0.001
Final R indices:	
R_1 , $I > 2\sigma(I)$	0.0452
wR_2 , all data	0.0998
R_{int}	0.0405
R_{sig}	0.0426
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.002P)^2 + 14.4P]$, $P = [\max(F_o^2, 0) + 2F_o^2]/3$
Absolute structure parameter	0.51(8)
Largest diff. peak and hole	1.420 and -0.991 $\text{e}/\text{\AA}^3$

$$R_1 = \Sigma |F_o| - |F_c| / \Sigma |F_o|, \quad wR_2 = [\Sigma w(F_o^2 - F_c^2)^2 / \Sigma w(F_o^2)]^{1/2}$$

Table S2. Atomic coordinates and equivalent* isotropic atomic displacement parameters ($\text{\AA}^2$) for **1**.

Atom	x/a	y/b	z/c	U_{eq}
Rh1	0.47652(9)	0.55555(2)	0.29411(5)	0.01838(9)
C1	0.3479(11)	0.6249(5)	0.3404(7)	0.0219(8)
C2	0.3776(12)	0.6436(5)	0.4676(7)	0.029(3)
C3	0.2867(13)	0.6913(6)	0.4968(8)	0.035(3)
C4	0.1640(14)	0.7228(6)	0.4020(9)	0.040(3)
C5	0.1321(13)	0.7027(6)	0.2784(8)	0.040(3)
C6	0.2189(12)	0.6524(6)	0.2482(8)	0.036(3)
N1	0.6995(10)	0.5900(4)	0.3624(7)	0.0202(7)
O1	0.7434(10)	0.4924(4)	0.2804(6)	0.0231(6)
C11	0.7923(9)	0.5480(5)	0.3350(7)	0.0195(7)
C12	0.9689(10)	0.5591(7)	0.3743(8)	0.031(2)
C13	1.0518(13)	0.5156(7)	0.4886(9)	0.037(3)
C14	0.9890(15)	0.5274(5)	0.5990(10)	0.033(2)
C15	0.9831(12)	0.5996(6)	0.6366(9)	0.032(2)
C16	0.8210(13)	0.6328(6)	0.5863(8)	0.033(3)

C17	0.7547(13)	0.6470(5)	0.4438(8)	0.0252(9)
N2	0.4541(12)	0.5922(4)	0.1154(7)	0.0227(7)
O2	0.4761(10)	0.4882(4)	0.0404(6)	0.0230(6)
C21	0.4540(12)	0.5496(5)	0.0263(7)	0.0211(8)
C22	0.4112(11)	0.5706(5)	-0.1123(7)	0.025(2)
C23	0.5480(13)	0.5825(6)	-0.1616(9)	0.037(3)
C24	0.6352(17)	0.6472(7)	-0.1139(9)	0.054(3)
C25	0.7208(13)	0.6491(7)	0.0307(9)	0.0426(11)
C26	0.6322(13)	0.6849(6)	0.1050(10)	0.036(3)
C27	0.4643(13)	0.6620(5)	0.0919(10)	0.029(2)
Rh1A	0.52154(9)	0.44449(2)	0.21410(5)	0.01838(9)
C1A	0.6542(11)	0.3765(5)	0.1710(7)	0.0219(8)
C2A	0.6183(11)	0.3558(5)	0.0439(7)	0.023(2)
C3A	0.7070(13)	0.3082(5)	0.0124(8)	0.032(3)
C4A	0.8326(13)	0.2772(6)	0.1054(9)	0.036(3)
C5A	0.8671(13)	0.2973(5)	0.2298(8)	0.036(3)
C6A	0.7805(11)	0.3468(5)	0.2624(7)	0.026(2)
C7A	0.9354(11)	0.2241(4)	0.0788(9)	0.046(2)
N1A	0.2991(10)	0.4108(4)	0.1480(7)	0.0202(7)
O1A	0.2522(9)	0.5076(4)	0.2286(6)	0.0231(6)
C11A	0.2004(9)	0.4527(5)	0.1715(7)	0.0195(7)
C12A	0.0257(9)	0.4415(7)	0.1350(7)	0.026(2)
C13A	-0.0688(13)	0.4791(6)	0.0142(9)	0.031(2)
C14A	-0.0106(16)	0.4699(6)	-0.0971(10)	0.040(3)
C15A	0.0139(15)	0.3996(6)	-0.1336(10)	0.044(3)
C16A	0.1810(14)	0.3701(7)	-0.0768(8)	0.037(3)
C17A	0.2419(13)	0.3527(5)	0.0661(8)	0.0252(9)
N2A	0.5402(12)	0.4087(4)	0.3917(7)	0.0227(7)
O2A	0.5237(10)	0.5112(4)	0.4700(6)	0.0230(6)
C21A	0.5517(12)	0.4497(5)	0.4863(7)	0.0211(8)
C22A	0.5962(12)	0.4290(6)	0.6262(7)	0.032(3)
C23A	0.4557(15)	0.4163(7)	0.6720(9)	0.043(3)
C24A	0.3625(16)	0.3533(7)	0.6258(9)	0.051(3)
C25A	0.2805(13)	0.3508(7)	0.4806(9)	0.0426(11)
C26A	0.3648(13)	0.3132(6)	0.4044(10)	0.035(3)
C27A	0.5276(13)	0.3382(5)	0.4127(10)	0.031(2)

* U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table S3. Anisotropic atomic displacement parameters* ($\text{\AA}^2$) for **1**.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Rh1	0.02021(15)	0.02191(15)	0.01376(13)	-0.00074(14)	0.00657(10)	0.00018(13)

C1	0.0209(18)	0.0226(19)	0.025(2)	-0.0033(15)	0.0121(16)	-0.0029(14)
C2	0.032(6)	0.029(6)	0.028(4)	0.002(4)	0.013(4)	-0.004(4)
C3	0.037(6)	0.037(6)	0.040(4)	-0.015(4)	0.024(4)	-0.008(4)
C4	0.037(6)	0.025(6)	0.067(5)	-0.008(4)	0.031(5)	0.001(4)
C5	0.027(6)	0.049(7)	0.048(4)	0.005(5)	0.017(4)	0.010(5)
C6	0.029(6)	0.046(6)	0.036(4)	-0.004(4)	0.016(4)	-0.002(5)
N1	0.0200(15)	0.0242(16)	0.0153(16)	-0.0004(12)	0.0044(13)	-0.0033(12)
O1	0.0205(13)	0.0277(14)	0.0233(14)	-0.0048(11)	0.0101(11)	-0.0020(11)
C11	0.0180(17)	0.0254(18)	0.0157(16)	0.0009(15)	0.0062(13)	0.0000(15)
C12	0.034(4)	0.034(5)	0.028(4)	-0.007(4)	0.015(4)	-0.006(4)
C13	0.018(5)	0.044(6)	0.042(5)	-0.001(4)	0.002(4)	0.008(4)
C14	0.018(5)	0.045(5)	0.030(4)	0.009(4)	0.002(4)	0.000(4)
C15	0.018(5)	0.049(6)	0.026(4)	-0.005(4)	0.001(4)	-0.006(4)
C16	0.031(5)	0.040(6)	0.023(4)	-0.011(4)	0.002(4)	-0.005(4)
C17	0.0203(19)	0.0269(19)	0.026(2)	-0.0046(16)	0.0048(16)	-0.0026(15)
N2	0.0237(17)	0.0248(16)	0.0202(16)	0.0008(13)	0.0081(13)	-0.0015(13)
O2	0.0306(14)	0.0250(13)	0.0129(13)	-0.0013(11)	0.0066(11)	-0.0003(11)
C21	0.0202(18)	0.0270(18)	0.0163(18)	0.0024(16)	0.0063(15)	-0.0017(16)
C22	0.023(5)	0.035(6)	0.013(3)	0.006(3)	0.001(3)	0.001(4)
C23	0.037(6)	0.057(6)	0.016(4)	0.000(4)	0.009(4)	-0.004(4)
C24	0.066(7)	0.067(7)	0.039(4)	0.005(4)	0.032(5)	-0.023(5)
C25	0.029(2)	0.062(3)	0.037(2)	-0.003(2)	0.0111(19)	-0.015(2)
C26	0.042(6)	0.033(6)	0.035(5)	0.014(4)	0.016(5)	-0.004(4)
C27	0.035(5)	0.028(4)	0.026(4)	0.006(4)	0.013(4)	0.000(5)
Rh1A	0.02021(15)	0.02191(15)	0.01376(13)	-0.00074(14)	0.00657(10)	0.00018(13)
C1A	0.0209(18)	0.0226(19)	0.025(2)	-0.0033(15)	0.0121(16)	-0.0029(14)
C2A	0.018(5)	0.027(5)	0.021(3)	-0.006(3)	0.005(3)	-0.002(4)
C3A	0.033(5)	0.028(6)	0.038(4)	-0.013(4)	0.017(4)	-0.009(4)
C4A	0.025(5)	0.035(6)	0.056(5)	-0.008(4)	0.022(4)	-0.001(4)
C5A	0.033(6)	0.030(6)	0.044(4)	0.000(4)	0.011(4)	0.002(4)
C6A	0.018(5)	0.029(5)	0.024(4)	0.000(3)	0.000(3)	0.000(4)
C7A	0.038(5)	0.045(5)	0.053(5)	-0.010(4)	0.011(4)	0.001(4)
N1A	0.0200(15)	0.0242(16)	0.0153(16)	-0.0004(12)	0.0044(13)	-0.0033(12)
O1A	0.0205(13)	0.0277(14)	0.0233(14)	-0.0048(11)	0.0101(11)	-0.0020(11)
C11A	0.0180(17)	0.0254(18)	0.0157(16)	0.0009(15)	0.0062(13)	0.0000(15)
C12A	0.019(4)	0.032(5)	0.031(4)	-0.007(4)	0.015(3)	0.000(4)
C13A	0.017(5)	0.034(5)	0.040(4)	-0.001(4)	0.008(3)	-0.006(4)
C14A	0.025(5)	0.053(6)	0.035(4)	0.009(4)	0.001(4)	0.001(4)
C15A	0.052(6)	0.052(6)	0.023(4)	-0.003(4)	0.005(4)	-0.003(5)
C16A	0.039(6)	0.045(6)	0.027(4)	-0.012(4)	0.012(4)	-0.005(5)
C17A	0.0203(19)	0.0269(19)	0.026(2)	-0.0046(16)	0.0048(16)	-0.0026(15)
N2A	0.0237(17)	0.0248(16)	0.0202(16)	0.0008(13)	0.0081(13)	-0.0015(13)
O2A	0.0306(14)	0.0250(13)	0.0129(13)	-0.0013(11)	0.0066(11)	-0.0003(11)
C21A	0.0202(18)	0.0270(18)	0.0163(18)	0.0024(16)	0.0063(15)	-0.0017(16)

C22A	0.038(5)	0.038(6)	0.015(3)	0.003(3)	-0.001(4)	-0.003(4)
C23A	0.056(7)	0.057(6)	0.018(4)	0.002(4)	0.016(4)	-0.005(5)
C24A	0.052(6)	0.073(7)	0.035(4)	-0.005(5)	0.025(5)	-0.013(5)
C25A	0.029(2)	0.062(3)	0.037(2)	-0.003(2)	0.0111(19)	-0.015(2)
C26A	0.041(6)	0.034(6)	0.026(4)	-0.002(4)	0.008(4)	-0.015(4)
C27A	0.041(5)	0.026(4)	0.028(4)	0.005(4)	0.014(4)	0.004(5)

* The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2a^{*2}U_{11} + \dots + 2hka^*b^*U_{12}]$

Table S4. Hydrogen atom coordinates and isotropic atomic displacement parameters ($\text{\AA}^2$) for **1**.

Atom	x/a	y/b	z/c	U_{iso}
H2	0.4608	0.6233	0.5336	0.035
H3	0.3074	0.7033	0.5834	0.042
H4	0.1046	0.7571	0.4227	0.048
H5	0.0495	0.7234	0.2125	0.048
H6	0.1892	0.6367	0.1630	0.043
H12A	0.9934	0.6057	0.3972	0.037
H12B	1.0077	0.5484	0.3026	0.037
H13A	1.0353	0.4691	0.4624	0.044
H13B	1.1676	0.5244	0.5173	0.044
H14A	1.0568	0.5031	0.6743	0.039
H14B	0.8803	0.5090	0.5759	0.039
H15A	1.0209	0.6025	0.7307	0.039
H15B	1.0588	0.6246	0.6059	0.039
H16A	0.8276	0.6748	0.6323	0.040
H16B	0.7432	0.6049	0.6089	0.040
H17A	0.6649	0.6780	0.4285	0.030
H17B	0.8384	0.6690	0.4180	0.030
H22A	0.3420	0.5365	-0.1661	0.030
H22B	0.3477	0.6113	-0.1239	0.030
H23A	0.5072	0.5830	-0.2559	0.044
H23B	0.6247	0.5459	-0.1345	0.044
H24A	0.5572	0.6835	-0.1376	0.065
H24B	0.7142	0.6546	-0.1578	0.065
H25A	0.7401	0.6036	0.0627	0.051
H25B	0.8258	0.6703	0.0472	0.051
H26A	0.6259	0.7316	0.0800	0.043
H26B	0.6969	0.6825	0.1964	0.043
H27A	0.4260	0.6868	0.1526	0.035
H27B	0.3929	0.6722	0.0048	0.035
H2A	0.5316	0.3750	-0.0208	0.027
H3A	0.6828	0.2959	-0.0744	0.038

H5A	0.9517	0.2769	0.2947	0.043
H6A	0.8085	0.3605	0.3487	0.031
H7A	1.0397	0.2243	0.1456	0.070
H7B	0.9487	0.2320	-0.0041	0.070
H7C	0.8844	0.1815	0.0777	0.070
H12C	-0.0109	0.4553	0.2062	0.031
H12D	0.0038	0.3941	0.1207	0.031
H13C	-0.1814	0.4651	-0.0113	0.037
H13D	-0.0651	0.5264	0.0349	0.037
H14C	0.0918	0.4934	-0.0785	0.048
H14D	-0.0876	0.4913	-0.1723	0.048
H15C	-0.0162	0.3975	-0.2276	0.053
H15D	-0.0612	0.3712	-0.1087	0.053
H16C	0.2574	0.4014	-0.0927	0.044
H16D	0.1846	0.3298	-0.1251	0.044
H17C	0.1550	0.3317	0.0894	0.030
H17D	0.3298	0.3206	0.0821	0.030
H22C	0.6612	0.3886	0.6388	0.039
H22D	0.6632	0.4636	0.6801	0.039
H23C	0.3811	0.4537	0.6450	0.051
H23D	0.4951	0.4158	0.7663	0.051
H24C	0.2807	0.3487	0.6679	0.061
H24D	0.4359	0.3156	0.6520	0.061
H25C	0.1736	0.3314	0.4634	0.051
H25D	0.2652	0.3964	0.4484	0.051
H26C	0.3755	0.2671	0.4333	0.042
H26D	0.2967	0.3137	0.3138	0.042
H27C	0.6025	0.3272	0.4981	0.037
H27D	0.5630	0.3143	0.3493	0.037

Table S5. Bond lengths (Å) and angles (°) for **1**.

Rh1-C1	1.991(8)	Rh1-N1	2.005(9)	Rh1-O2A	2.068(6)
Rh1-N2	2.068(7)	Rh1-O1A	2.126(8)	Rh1-Rh1A	2.5156(6)
C1-C6	1.382(7)	C1-C2	1.403(7)	C2-C3	1.372(7)
C3-C4	1.399(8)	C4-C5	1.369(8)	C5-C6	1.392(8)
N1-C11	1.294(9)	N1-C17	1.457(9)	O1-C11	1.294(9)
O1-Rh1A	2.108(8)	C11-C12	1.504(9)	C12-C13	1.527(11)
C13-C14	1.525(10)	C14-C15	1.538(11)	C15-C16	1.524(10)
C16-C17	1.524(9)	N2-C21	1.317(9)	N2-C27	1.458(9)
O2-C21	1.270(9)	O2-Rh1A	2.043(6)	C21-C22	1.522(8)
C22-C23	1.509(10)	C23-C24	1.534(11)	C24-C25	1.536(11)
C25-C26	1.505(11)	C26-C27	1.526(10)	Rh1A-C1A	1.980(8)

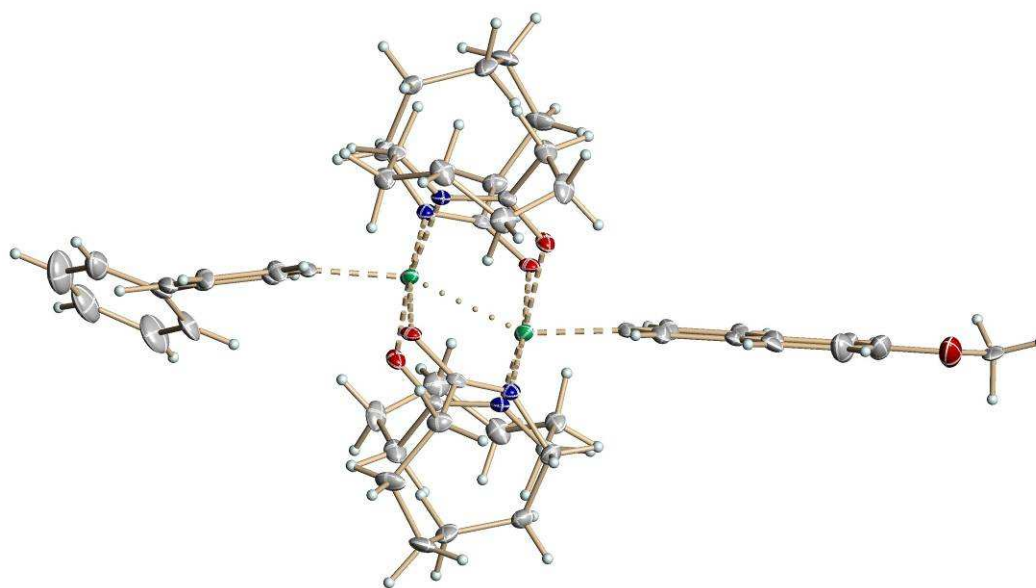
Rh1A-N1A	1.994(9)	Rh1A-N2A	2.059(7)	C1A-C6A	1.382(7)
C1A-C2A	1.407(7)	C2A-C3A	1.368(7)	C3A-C4A	1.400(8)
C4A-C5A	1.376(8)	C4A-C7A	1.509(12)	C5A-C6A	1.390(7)
N1A-C11A	1.312(9)	N1A-C17A	1.480(9)	O1A-C11A	1.296(9)
C11A-C12A	1.490(9)	C12A-C13A	1.534(10)	C13A-C14A	1.501(10)
C14A-C15A	1.527(11)	C15A-C16A	1.535(11)	C16A-C17A	1.542(10)
N2A-C21A	1.321(8)	N2A-C27A	1.468(9)	O2A-C21A	1.281(9)
C21A-C22A	1.533(8)	C22A-C23A	1.518(10)	C23A-C24A	1.527(11)
C24A-C25A	1.537(10)	C25A-C26A	1.512(11)	C26A-C27A	1.510(10)
C1-Rh1-N1	103.5(4)	C1-Rh1-O2A	91.3(3)	N1-Rh1-O2A	85.8(3)
C1-Rh1-N2	96.2(3)	N1-Rh1-N2	90.5(3)	O2A-Rh1-N2	172.2(3)
C1-Rh1-O1A	82.0(4)	N1-Rh1-O1A	173.0(4)	O2A-Rh1-O1A	89.9(3)
N2-Rh1-O1A	93.1(3)	C6-C1-C2	118.1(6)	C6-C1-Rh1	120.4(5)
C2-C1-Rh1	121.3(6)	C3-C2-C1	120.1(7)	C2-C3-C4	121.5(7)
C5-C4-C3	118.1(7)	C4-C5-C6	120.9(7)	C1-C6-C5	120.9(7)
C11-N1-C17	124.3(8)	C11-N1-Rh1	107.9(6)	C17-N1-Rh1	127.3(6)
C11-O1-Rh1A	134.7(6)	O1-C11-N1	123.3(8)	O1-C11-C12	115.3(8)
N1-C11-C12	121.2(8)	C11-C12-C13	109.1(8)	C14-C13-C12	111.8(8)
C13-C14-C15	115.1(9)	C16-C15-C14	116.1(9)	C15-C16-C17	117.5(8)
N1-C17-C16	115.4(8)	C21-N2-C27	119.7(7)	C21-N2-Rh1	117.3(6)
C27-N2-Rh1	122.2(6)	C21-O2-Rh1A	121.5(5)	O2-C21-N2	126.6(7)
O2-C21-C22	112.3(7)	N2-C21-C22	120.8(8)	C23-C22-C21	116.6(8)
C22-C23-C24	113.0(9)	C23-C24-C25	113.8(9)	C26-C25-C24	114.6(10)
C25-C26-C27	118.1(9)	N2-C27-C26	113.5(9)	C1A-Rh1A-N1A	105.5(4)
C1A-Rh1A-O2	91.6(3)	N1A-Rh1A-O2	86.5(3)	C1A-Rh1A-N2A	96.6(4)
N1A-Rh1A-N2A	89.3(3)	O2-Rh1A-N2A	171.5(4)	C1A-Rh1A-O1	81.1(4)
N1A-Rh1A-O1	172.5(4)	O2-Rh1A-O1	90.0(3)	N2A-Rh1A-O1	93.3(3)
C6A-C1A-C2A	117.9(6)	C6A-C1A-Rh1A	122.4(5)	C2A-C1A-Rh1A	119.8(6)
C3A-C2A-C1A	120.7(6)	C2A-C3A-C4A	121.3(7)	C5A-C4A-C3A	117.9(7)
C5A-C4A-C7A	117.4(8)	C3A-C4A-C7A	124.7(7)	C4A-C5A-C6A	121.2(7)
C1A-C6A-C5A	121.0(6)	C11A-N1A-C17A	121.8(8)	C11A-N1A-Rh1A	110.5(6)
C17A-N1A-Rh1A	127.0(6)	C11A-O1A-Rh1	135.2(6)	O1A-C11A-N1A	120.5(8)
O1A-C11A-C12A	116.0(8)	N1A-C11A-C12A	123.5(8)	C11A-C12A-C13A	112.6(8)
C14A-C13A-C12A	114.8(8)	C13A-C14A-C15A	117.0(9)	C14A-C15A-C16A	117.6(10)
C15A-C16A-C17A	118.7(9)	N1A-C17A-C16A	112.4(8)	C21A-N2A-C27A	119.1(7)
C21A-N2A-Rh1A	119.8(6)	C27A-N2A-Rh1A	120.9(6)	C21A-O2A-Rh1	122.2(5)
O2A-C21A-N2A	123.3(7)	O2A-C21A-C22A	112.8(7)	N2A-C21A-C22A	123.9(8)
C23A-C22A-C21A	114.8(8)	C22A-C23A-C24A	116.5(9)	C23A-C24A-C25A	113.5(10)
C26A-C25A-C24A	116.9(10)	C27A-C26A-C25A	115.9(8)	N2A-C27A-C26A	116.3(9)

Table S6. Torsion angles (°) for **1**.

N1-Rh1-C1-C6	126.4(10)	O2A-Rh1-C1-C6	-147.6(10)
N2-Rh1-C1-C6	34.4(10)	O1A-Rh1-C1-C6	-57.9(10)
N1-Rh1-C1-C2	-58.4(10)	O2A-Rh1-C1-C2	27.6(10)
N2-Rh1-C1-C2	-150.4(10)	O1A-Rh1-C1-C2	117.3(10)
C6-C1-C2-C3	-4.4(18)	Rh1-C1-C2-C3	-179.7(9)
C1-C2-C3-C4	-0.6(19)	C2-C3-C4-C5	3(2)
C3-C4-C5-C6	0(2)	C2-C1-C6-C5	7.3(18)
Rh1-C1-C6-C5	-177.4(9)	C4-C5-C6-C1	-5(2)
C1-Rh1-N1-C11	-179.2(6)	O2A-Rh1-N1-C11	90.5(6)
N2-Rh1-N1-C11	-82.7(6)	C1-Rh1-N1-C17	8.7(8)
O2A-Rh1-N1-C17	-81.7(7)	N2-Rh1-N1-C17	105.2(7)
Rh1A-O1-C11-N1	0.1(13)	Rh1A-O1-C11-C12	175.6(6)
C17-N1-C11-O1	168.3(8)	Rh1-N1-C11-O1	-4.2(10)
C17-N1-C11-C12	-7.1(13)	Rh1-N1-C11-C12	-179.5(6)
O1-C11-C12-C13	-72.0(10)	N1-C11-C12-C13	103.7(11)
C11-C12-C13-C14	-56.2(13)	C12-C13-C14-C15	-50.0(14)
C13-C14-C15-C16	100.1(11)	C14-C15-C16-C17	-70.4(13)
C11-N1-C17-C16	-75.8(12)	Rh1-N1-C17-C16	95.1(10)
C15-C16-C17-N1	72.5(13)	C1-Rh1-N2-C21	-148.7(8)
N1-Rh1-N2-C21	107.6(8)	O1A-Rh1-N2-C21	-66.4(8)
C1-Rh1-N2-C27	41.3(9)	N1-Rh1-N2-C27	-62.4(8)
O1A-Rh1-N2-C27	123.6(8)	Rh1A-O2-C21-N2	-1.2(15)
Rh1A-O2-C21-C22	-176.1(6)	C27-N2-C21-O2	165.6(10)
Rh1-N2-C21-O2	-4.6(15)	C27-N2-C21-C22	-19.9(15)
Rh1-N2-C21-C22	169.9(7)	O2-C21-C22-C23	-84.7(12)
N2-C21-C22-C23	100.1(12)	C21-C22-C23-C24	-75.1(13)
C22-C23-C24-C25	65.4(14)	C23-C24-C25-C26	-99.8(14)
C24-C25-C26-C27	56.8(14)	C21-N2-C27-C26	-78.1(12)
Rh1-N2-C27-C26	91.7(9)	C25-C26-C27-N2	52.7(13)
C21-O2-Rh1A-C1A	-148.7(9)	C21-O2-Rh1A-N1A	105.9(9)
C21-O2-Rh1A-O1	-67.6(8)	C11-O1-Rh1A-C1A	-179.1(8)
C11-O1-Rh1A-O2	89.3(8)	C11-O1-Rh1A-N2A	-82.9(8)
N1A-Rh1A-C1A-C6A	-123.2(10)	O2-Rh1A-C1A-C6A	150.0(10)
N2A-Rh1A-C1A-C6A	-32.0(10)	O1-Rh1A-C1A-C6A	60.3(10)
N1A-Rh1A-C1A-C2A	55.2(10)	O2-Rh1A-C1A-C2A	-31.5(9)
N2A-Rh1A-C1A-C2A	146.4(9)	O1-Rh1A-C1A-C2A	-121.2(9)
C6A-C1A-C2A-C3A	-0.5(17)	Rh1A-C1A-C2A-C3A	-179.0(9)
C1A-C2A-C3A-C4A	2.0(19)	C2A-C3A-C4A-C5A	-2(2)
C2A-C3A-C4A-C7A	179.6(11)	C3A-C4A-C5A-C6A	0(2)
C7A-C4A-C5A-C6A	178.7(11)	C2A-C1A-C6A-C5A	-1.2(18)
Rh1A-C1A-C6A-C5A	177.2(9)	C4A-C5A-C6A-C1A	1.6(19)
C1A-Rh1A-N1A-C11A	-179.8(6)	O2-Rh1A-N1A-C11A	-89.2(6)
N2A-Rh1A-N1A-C11A	83.5(6)	C1A-Rh1A-N1A-C17A	-8.9(8)
O2-Rh1A-N1A-C17A	81.8(7)	N2A-Rh1A-N1A-C17A	-105.6(7)

C1-Rh1-O1A-C11A	177.5(9)	O2A-Rh1-O1A-C11A	-91.2(8)
N2-Rh1-O1A-C11A	81.6(8)	Rh1-O1A-C11A-N1A	3.9(13)
Rh1-O1A-C11A-C12A	-175.6(6)	C17A-N1A-C11A-O1A	-170.5(8)
Rh1A-N1A-C11A-O1A	1.0(10)	C17A-N1A-C11A-C12A	9.0(13)
Rh1A-N1A-C11A-C12A	-179.5(6)	O1A-C11A-C12A-C13A	78.3(10)
N1A-C11A-C12A-C13A	-101.3(11)	C11A-C12A-C13A-C14A	50.9(14)
C12A-C13A-C14A-C15A	50.5(16)	C13A-C14A-C15A-C16A	-93.6(13)
C14A-C15A-C16A-C17A	71.6(15)	C11A-N1A-C17A-C16A	76.8(11)
Rh1A-N1A-C17A-C16A	-93.2(10)	C15A-C16A-C17A-N1A	-79.1(14)
C1A-Rh1A-N2A-C21A	142.4(9)	N1A-Rh1A-N2A-C21A	-112.1(9)
O1-Rh1A-N2A-C21A	61.0(9)	C1A-Rh1A-N2A-C27A	-42.7(9)
N1A-Rh1A-N2A-C27A	62.8(8)	O1-Rh1A-N2A-C27A	-124.2(8)
C1-Rh1-O2A-C21A	152.3(9)	N1-Rh1-O2A-C21A	-104.2(9)
O1A-Rh1-O2A-C21A	70.3(8)	Rh1-O2A-C21A-N2A	-5.7(14)
Rh1-O2A-C21A-C22A	175.5(6)	C27A-N2A-C21A-O2A	-162.0(10)
Rh1A-N2A-C21A-O2A	12.9(14)	C27A-N2A-C21A-C22A	16.6(15)
Rh1A-N2A-C21A-C22A	-168.4(7)	O2A-C21A-C22A-C23A	83.7(12)
N2A-C21A-C22A-C23A	-95.1(12)	C21A-C22A-C23A-C24A	73.9(13)
C22A-C23A-C24A-C25A	-63.6(15)	C23A-C24A-C25A-C26A	98.6(14)
C24A-C25A-C26A-C27A	-61.1(15)	C21A-N2A-C27A-C26A	80.4(12)
Rh1A-N2A-C27A-C26A	-94.4(9)	C25A-C26A-C27A-N2A	-49.4(13)

X-Ray data for 4



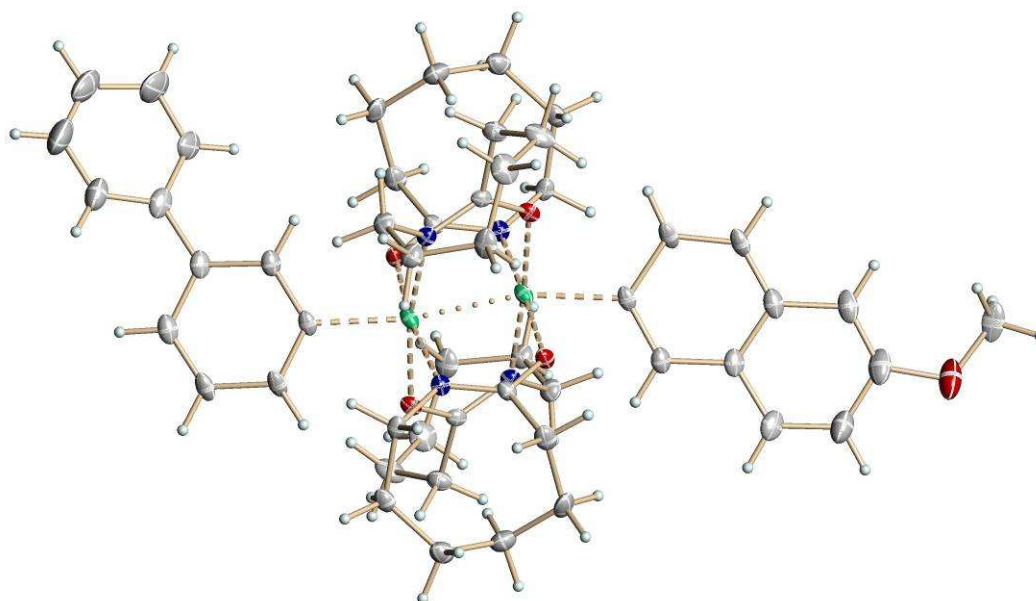


Figure S2. View of complex **4**. Ellipsoids for the non-hydrogen atoms are shown at the 30% probability level. Hydrogen atoms are displayed with an arbitrarily small radius.

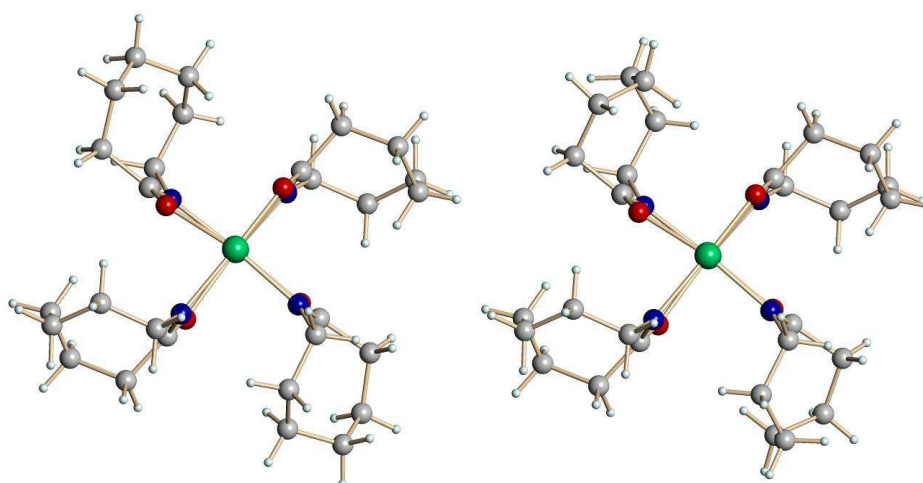


Figure S3. A view of **4** along Rh-Rh pair showing propeller configuration of ligands

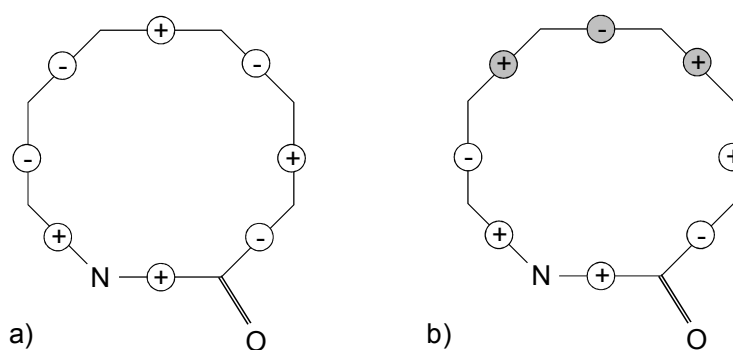


Figure S4. Alternative conformations of ligands: a) signs of torsion angles in molecule **1** (molecule **2** has all signs opposite); b) signs of torsion angles in molecules **3** and **1a** (molecules **4** and **2a** have all signs opposite).

Brief structure description. This crystal structure is made of Rh₂ dimeric complexes and

dichloromethane solvent. This structure determination has confirmed that Rh₂ complex consists of a pair of Rh atoms at 2.5292(7) Å from each other with four chelating 1-aza-2-cyclooctanoate (HACO) ligands and two different apical ligands which are diphenyl (DP) and methoxynaphthalene (NP). It crystallizes in monoclinic non-centrosymmetric space group *Pc* and shows merohedral twinning in a 3:2 ratio between components which are related by a center of symmetry to each other. This structure is heavily disordered due to superposition of complexes with four different conformations. In addition to this two of the four HACO ligands have two alternative conformations.

Figure S2 shows general view of the complex in 2 perpendicular orientations. Four chelating HP ligands coordinate Rh dimer and form propeller-like complex depicted in Figure S3. The major component (73.9(8) % of all complexes) shown in Figure S3, left has 2 pairs of HACO ligands with different conformations. Ligands of the same conformation are opposite to each other. The minor component (26.1(8) % of all complexes) has all ligands in the same conformation. The conformation or distribution of torsion angles in HACO ligands is shown in Figure S4.

As can be seen from Figure S2 both apical ligands are tilted from Rh-Rh axis towards O atoms of HP ligands. It was found out that the apical ligands can swap with each other while central part [Rh₂(HACO)₄] remains the same that yields two conformations of the Rh complex. This was observed during structure refinement as superposition of DP and NP ligands in both apical positions in 0.773(4) : 0.227(4) ratio. Merohedral twinning or reflection in *c*-glide plane produces the inverse or mirror images of two previous conformants with opposite orientations of HACO ligands, e.g counter-clockwise vs. clockwise.

Crystallographic Determination

A yellow needle of C₅₂H₆₈Cl₂N₄O₅Rh₂, approximate dimensions 0.02× 0.05× 0.355 mm³, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 200(2) K on a three-circle diffractometer system equipped with Bruker Smart Apex II CCD area detector using a graphite monochromator and a MoK α fine-focus sealed tube (λ = 0.71073 Å). The detector was placed at a distance of 5.000 cm from the crystal.

A total of 1556 frames were collected with a scan width of -0.30° in ω and an exposure time of 53 sec/frame using Apex2 (Bruker, 2005). The total data collection time was 25.5 hours. The frames were integrated with Apex2 software package using a narrow-frame integration algorithm. The integration of the data using a Monoclinic unit cell yielded a total of 16606 reflections to a maximum θ angle of 25.00°, of which 8623 were independent (completeness = 100.0%, R_{int} = 3.03%, R_{sig} = 4.95%) and 6784 were greater than 2 σ (I). The final cell dimensions of a = 12.7859(15) Å, b = 19.152(2) Å, c = 10.3475(12) Å, α = 90°, β = 99.6295(18)°, γ = 90°, V = 2498.1(5) Å³, are based upon the refinement of the XYZ-centroids of 4854 reflections with 2.3 < θ < 27.1° using Apex2. Analysis of the data showed 0 % decay during data collection. Data were corrected for absorption effects with the Semi-empirical from equivalents method using SADABS (Sheldrick, 1996). The minimum and maximum transmission coefficients were 0.881 and 0.984.

The structure was solved and refined using the SHELXS-97 (Sheldrick, 1990) and SHELXL-97 (Sheldrick, 1997) software in the space group *Pc* with Z = 2 for the formula unit C₅₂H₆₈Cl₂N₄O₅Rh₂. The final anisotropic full-matrix least-squares refinement on F^2 with 533 variables converged at R_1 =5.85 % for the observed data and wR_2 =11.95 % for all data. The goodness-of-fit was 1.000. The largest peak on the final difference map was 0.948 e⁻/Å³ and the largest hole was -1.135 e⁻/Å³. On the

basis of the final model, the calculated density was 1.470 g/cm³ and F(000), 1144 e⁻.

Table S7. Crystal data and structure refinement for **4**.

Empirical formula	C ₅₂ H ₆₈ Cl ₂ N ₄ O ₅ Rh ₂
Formula weight	1105.82
Temperature	200(2) K
Wavelength	0.71073 Å
Crystal size	0.355 × 0.05 × 0.02 mm ³
Crystal habit	yellow needle
Crystal system	Monoclinic
Space group	Pc
Unit cell dimensions	a = 12.7859(15) Å α = 90° b = 19.152(2) Å β = 99.6295(18)° c = 10.3475(12) Å γ = 90°
Volume	2498.1(5) Å ³
Z	2
Density, ρ _{calc}	1.470 g/cm ³
Absorption coefficient, μ	0.818 mm ⁻¹
F(000)	1144 e ⁻
Diffractionmeter	Bruker Smart Apex II CCD area detector
Radiation source	fine-focus sealed tube, MoKα
Detector distance	5.000 cm
Detector resolution	11.198 pixels/mm
Total frames	1556
Frame size	512 pixels
Frame width	-0.30°
Exposure per frame	53 sec
Total measurement time	25.5 hours
Data collection method	ω scans
θ range for data collection	1.93 to 25.00°
Index ranges	-15 ≤ h ≤ 15, -22 ≤ k ≤ 22, -12 ≤ l ≤ 12
Reflections collected	16606
Independent reflections	8623
Observed reflection, I>2σ(I)	6784
Coverage of independent reflections	100.0 %
Variation in check reflections	0 %
Absorption correction	Semi-empirical from equivalents SADABS (Sheldrick, 1996)
Max. and min. transmission	0.984 and 0.881
Structure solution technique	direct
Structure solution program	SHELXS-97 (Sheldrick, 1990)
Refinement technique	Full-matrix least-squares on F ²
Refinement program	SHELXL-97 (Sheldrick, 1997)
Function minimized	Σw(F _o ² - F _c ²) ²
Data / restraints / parameters	8623 / 487 / 533
Goodness-of-fit on F ²	0.993
Δ/σ _{max}	0.001
Final R indices:	
R ₁ , I>2σ(I)	0.0585
wR ₂ , all data	0.1195
R _{int}	0.0303
R _{sig}	0.0495
Weighting scheme	w = 1/[σ ² (F _o ²)+(0.001P) ² +19.1P], P = [max(F _o ² ,0)+2F _o ²]/3
Absolute structure parameter	0.38(7)
Largest diff. peak and hole	0.948 and -1.135 e ⁻ /Å ³

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|, \quad wR_2 = [\Sigma w(F_o^2 - F_c^2)^2 / \Sigma w(F_o^2)^2]^{1/2}$$

Table S8. Atomic coordinates and equivalent* isotropic atomic displacement parameters ($\text{\AA}^2$) for 4.

Atom	x/a	y/b	z/c	U_{eq}
Rh1	0.4645(4)	0.50396(4)	0.3833(5)	0.0292(3)
Rh2	0.5385(4)	0.50251(4)	0.6258(5)	0.0293(3)
O1	0.4373(6)	0.6062(3)	0.4121(7)	0.0277(8)
C11	0.4493(7)	0.6343(3)	0.5267(7)	0.0257(10)
C12	0.4074(7)	0.7084(3)	0.5219(9)	0.0386(12)
C13	0.2900(7)	0.7170(7)	0.5217(11)	0.050(3)
C14	0.2572(9)	0.7056(6)	0.6574(12)	0.052(2)
C15	0.2749(8)	0.6326(6)	0.7118(12)	0.048(2)
C16	0.3762(7)	0.6235(5)	0.8085(8)	0.0387(12)
C17	0.4812(7)	0.6357(4)	0.7618(8)	0.0373(12)
N1	0.4907(6)	0.6023(3)	0.6361(7)	0.0282(8)
C12A	0.4074(7)	0.7084(3)	0.5219(9)	0.0386(12)
C13A	0.2882(7)	0.701(2)	0.500(3)	0.050(3)
C14A	0.2466(9)	0.6548(16)	0.6031(17)	0.052(2)
C15A	0.290(2)	0.6723(15)	0.7439(16)	0.048(2)
C16A	0.3762(7)	0.6235(5)	0.8085(8)	0.0387(12)
O2	0.5862(6)	0.4013(3)	0.5923(7)	0.0277(8)
C21	0.5677(7)	0.3721(3)	0.4787(8)	0.0257(10)
C22	0.6002(7)	0.2959(3)	0.4846(9)	0.0386(12)
C23	0.7150(8)	0.2807(7)	0.4771(12)	0.050(3)
C24	0.7433(10)	0.2908(6)	0.3388(12)	0.052(2)
C25	0.7346(8)	0.3648(6)	0.2877(12)	0.048(2)
C26	0.6331(7)	0.3801(5)	0.1947(8)	0.0387(12)
C27	0.5285(7)	0.3701(4)	0.2437(8)	0.0373(12)
N2	0.5231(6)	0.4037(3)	0.3703(7)	0.0282(8)
C22A	0.6002(7)	0.2959(3)	0.4846(9)	0.0386(12)
C23A	0.7195(8)	0.296(2)	0.496(4)	0.050(3)
C24A	0.7616(9)	0.3458(16)	0.3992(17)	0.052(2)
C25A	0.721(2)	0.3320(17)	0.2560(17)	0.048(2)
C26A	0.6331(7)	0.3801(5)	0.1947(8)	0.0387(12)
O3	0.3513(6)	0.4710(3)	0.4844(7)	0.0322(7)
C31	0.3447(6)	0.4554(4)	0.6045(7)	0.0272(9)
C32	0.2398(6)	0.4258(4)	0.6230(9)	0.0448(14)
C33	0.2312(8)	0.3481(4)	0.5849(10)	0.0517(16)
C34	0.3217(8)	0.3043(4)	0.6536(10)	0.0482(14)
C35	0.3469(8)	0.3122(4)	0.8024(9)	0.0484(15)
C36	0.4377(7)	0.3610(4)	0.8537(9)	0.0398(13)
C37	0.4222(7)	0.4399(3)	0.8348(7)	0.0306(12)
N3	0.4230(6)	0.4644(3)	0.7005(7)	0.0278(8)
O4	0.6688(6)	0.5349(3)	0.5251(7)	0.0322(7)

C41	0.6757(6)	0.5505(4)	0.4052(7)	0.0272(9)
C42	0.7797(6)	0.5817(4)	0.3873(9)	0.0448(14)
C43	0.7858(8)	0.6593(4)	0.4263(11)	0.0517(16)
C44	0.6939(8)	0.7021(4)	0.3580(10)	0.0482(14)
C45	0.6709(8)	0.6955(4)	0.2088(10)	0.0484(15)
C46	0.5827(7)	0.6455(4)	0.1543(9)	0.0398(13)
C47	0.6001(7)	0.5668(4)	0.1749(7)	0.0306(12)
N4	0.5971(5)	0.5426(3)	0.3090(7)	0.0278(8)
C51	0.6638(3)	0.5089(4)	0.7715(4)	0.0310(9)
C52	0.6951(4)	0.4492(7)	0.8602(8)	0.0400(12)
C53	0.7774(7)	0.4600(7)	0.9670(10)	0.0427(12)
C54	0.8300(7)	0.5212(6)	0.9947(10)	0.0413(13)
C55	0.8005(6)	0.5771(5)	0.9114(9)	0.0361(12)
C56	0.7173(8)	0.5711(7)	0.7998(11)	0.0367(11)
C57	0.8585(8)	0.6403(6)	0.9449(11)	0.0447(16)
C58	0.9375(9)	0.6452(8)	1.0516(14)	0.0653(15)
C59	0.9650(8)	0.5883(7)	1.1329(11)	0.064(2)
C60	0.9144(7)	0.5278(7)	1.1075(10)	0.0517(19)
O61	1.0472(7)	0.6039(6)	1.2360(10)	0.083(3)
C62	1.0797(14)	0.5505(11)	1.326(2)	0.063(3)
C71	0.3528(4)	0.5025(4)	0.2233(5)	0.0310(9)
C72	0.3336(7)	0.5604(6)	0.1537(8)	0.0400(12)
C73	0.2415(9)	0.5637(6)	0.0556(11)	0.0427(12)
C74	0.1751(9)	0.5092(4)	0.0336(11)	0.0413(13)
C75	0.1957(5)	0.4474(3)	0.1080(6)	0.0361(12)
C76	0.2863(7)	0.4457(7)	0.2032(10)	0.0367(11)
C77	0.1247(6)	0.3891(4)	0.0847(8)	0.0447(16)
C78	0.0706(8)	0.3706(7)	-0.0343(16)	0.0653(15)
C79	0.0048(10)	0.3162(8)	-0.0546(17)	0.104(5)
C80	-0.0099(9)	0.2788(7)	0.0403(13)	0.089(4)
C81	0.0423(9)	0.2914(7)	0.1761(18)	0.088(4)
C82	0.1074(8)	0.3464(5)	0.1913(14)	0.057(3)
C51A	0.3528(4)	0.5025(4)	0.2233(5)	0.0310(9)
C52A	0.3146(7)	0.5598(9)	0.1316(12)	0.0400(12)
C53A	0.2294(10)	0.5452(10)	0.0303(13)	0.0427(12)
C54A	0.1796(9)	0.4825(9)	0.0105(13)	0.0413(13)
C55A	0.2156(9)	0.4289(7)	0.0966(15)	0.0361(12)
C56A	0.3022(11)	0.4387(9)	0.2031(16)	0.0367(11)
C57A	0.1606(15)	0.3640(9)	0.071(2)	0.0447(16)
C58A	0.0782(16)	0.3554(12)	-0.030(3)	0.0653(15)
C59A	0.0442(13)	0.4101(13)	-0.115(2)	0.064(2)
C60A	0.0919(11)	0.4720(12)	-0.0970(15)	0.0517(19)
O61A	-0.0405(15)	0.3908(15)	-0.212(2)	0.083(3)
C62A	-0.097(3)	0.4448(19)	-0.283(3)	0.063(3)

C71A	0.6638(3)	0.5089(4)	0.7715(4)	0.0310(9)
C72A	0.6778(10)	0.4525(9)	0.8461(14)	0.0400(12)
C73A	0.7658(16)	0.4500(11)	0.950(2)	0.0427(12)
C74A	0.8335(14)	0.5037(10)	0.9727(19)	0.0413(13)
C75A	0.8184(6)	0.5639(6)	0.8930(12)	0.0361(12)
C76A	0.7318(8)	0.5649(8)	0.7920(14)	0.0367(11)
C77A	0.8909(9)	0.6215(8)	0.9169(19)	0.0447(16)
C78A	0.930(2)	0.6473(16)	1.037(3)	0.0653(15)
C79A	0.998(2)	0.7009(17)	1.058(3)	0.104(5)
C80A	1.0281(17)	0.7303(12)	0.963(4)	0.089(4)
C81A	0.993(2)	0.7089(14)	0.825(3)	0.088(4)
C82A	0.926(2)	0.6552(13)	0.810(3)	0.057(3)
C1	0.919(2)	0.4763(6)	0.6808(19)	0.093(5)
Cl1	1.0019(5)	0.5456(3)	0.6851(7)	0.0882(13)
Cl2	0.9488(5)	0.4076(3)	0.5900(8)	0.1034(16)
C1A	0.099(6)	0.541(2)	0.325(10)	0.093(5)
Cl1A	0.0169(15)	0.4719(10)	0.331(2)	0.0882(13)
Cl2A	0.0661(15)	0.6135(10)	0.405(3)	0.1034(16)

* U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table S9. Anisotropic atomic displacement parameters* ($\text{\AA}^2$) for **4**.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Rh1	0.0335(6)	0.0339(6)	0.0205(6)	0.0009(3)	0.0054(4)	0.0158(4)
Rh2	0.0354(6)	0.0301(6)	0.0223(6)	0.0011(3)	0.0048(5)	0.0124(3)
O1	0.027(2)	0.0329(16)	0.0274(14)	0.0061(13)	0.0153(14)	0.0043(15)
C11	0.020(2)	0.027(2)	0.033(2)	0.0030(16)	0.0137(19)	-0.0003(19)
C12	0.047(3)	0.034(3)	0.039(3)	0.005(2)	0.017(2)	0.003(2)
C13	0.053(3)	0.044(6)	0.060(4)	0.012(4)	0.029(3)	0.025(3)
C14	0.036(4)	0.070(5)	0.057(5)	-0.004(4)	0.025(4)	0.019(4)
C15	0.045(4)	0.078(6)	0.025(4)	-0.003(4)	0.015(3)	-0.005(4)
C16	0.042(3)	0.043(3)	0.033(3)	-0.009(2)	0.011(2)	0.000(2)
C17	0.044(3)	0.038(3)	0.030(2)	-0.009(2)	0.006(2)	0.000(2)
N1	0.027(2)	0.0327(17)	0.0270(16)	-0.0003(15)	0.0103(15)	-0.0025(15)
C12A	0.047(3)	0.034(3)	0.039(3)	0.005(2)	0.017(2)	0.003(2)
C13A	0.053(3)	0.044(6)	0.060(4)	0.012(4)	0.029(3)	0.025(3)
C14A	0.036(4)	0.070(5)	0.057(5)	-0.004(4)	0.025(4)	0.019(4)
C15A	0.045(4)	0.078(6)	0.025(4)	-0.003(4)	0.015(3)	-0.005(4)
C16A	0.042(3)	0.043(3)	0.033(3)	-0.009(2)	0.011(2)	0.000(2)
O2	0.027(2)	0.0329(16)	0.0274(14)	0.0061(13)	0.0153(14)	0.0043(15)
C21	0.020(2)	0.027(2)	0.033(2)	0.0030(16)	0.0137(19)	-0.0003(19)
C22	0.047(3)	0.034(3)	0.039(3)	0.005(2)	0.017(2)	0.003(2)

C23	0.053(3)	0.044(6)	0.060(4)	0.012(4)	0.029(3)	0.025(3)
C24	0.036(4)	0.070(5)	0.057(5)	-0.004(4)	0.025(4)	0.019(4)
C25	0.045(4)	0.078(6)	0.025(4)	-0.003(4)	0.015(3)	-0.005(4)
C26	0.042(3)	0.043(3)	0.033(3)	-0.009(2)	0.011(2)	0.000(2)
C27	0.044(3)	0.038(3)	0.030(2)	-0.009(2)	0.006(2)	0.000(2)
N2	0.027(2)	0.0327(17)	0.0270(16)	-0.0003(15)	0.0103(15)	-0.0025(15)
C22A	0.047(3)	0.034(3)	0.039(3)	0.005(2)	0.017(2)	0.003(2)
C23A	0.053(3)	0.044(6)	0.060(4)	0.012(4)	0.029(3)	0.025(3)
C24A	0.036(4)	0.070(5)	0.057(5)	-0.004(4)	0.025(4)	0.019(4)
C25A	0.045(4)	0.078(6)	0.025(4)	-0.003(4)	0.015(3)	-0.005(4)
C26A	0.042(3)	0.043(3)	0.033(3)	-0.009(2)	0.011(2)	0.000(2)
O3	0.0230(16)	0.0428(19)	0.0314(15)	0.0091(15)	0.0066(12)	-0.0022(15)
C31	0.0248(19)	0.031(3)	0.0274(19)	0.0041(19)	0.0086(15)	0.0062(18)
C32	0.033(3)	0.063(4)	0.040(3)	0.016(3)	0.011(2)	-0.008(3)
C33	0.038(3)	0.065(4)	0.049(3)	0.008(3)	-0.003(3)	-0.028(3)
C34	0.049(4)	0.040(3)	0.057(3)	-0.003(3)	0.013(3)	-0.012(3)
C35	0.040(3)	0.047(3)	0.058(3)	0.015(3)	0.008(3)	-0.003(3)
C36	0.037(3)	0.052(3)	0.032(3)	0.013(2)	0.009(2)	0.001(3)
C37	0.024(3)	0.048(3)	0.022(2)	-0.001(2)	0.010(2)	-0.008(2)
N3	0.0244(19)	0.035(2)	0.0261(16)	-0.0013(17)	0.0099(13)	0.0034(17)
O4	0.0230(16)	0.0428(19)	0.0314(15)	0.0091(15)	0.0066(12)	-0.0022(15)
C41	0.0248(19)	0.031(3)	0.0274(19)	0.0041(19)	0.0086(15)	0.0062(18)
C42	0.033(3)	0.063(4)	0.040(3)	0.016(3)	0.011(2)	-0.008(3)
C43	0.038(3)	0.065(4)	0.049(3)	0.008(3)	-0.003(3)	-0.028(3)
C44	0.049(4)	0.040(3)	0.057(3)	-0.003(3)	0.013(3)	-0.012(3)
C45	0.040(3)	0.047(3)	0.058(3)	0.015(3)	0.008(3)	-0.003(3)
C46	0.037(3)	0.052(3)	0.032(3)	0.013(2)	0.009(2)	0.001(3)
C47	0.024(3)	0.048(3)	0.022(2)	-0.001(2)	0.010(2)	-0.008(2)
N4	0.0244(19)	0.035(2)	0.0261(16)	-0.0013(17)	0.0099(13)	0.0034(17)
C51	0.023(2)	0.053(2)	0.019(2)	0.0061(18)	0.0096(15)	0.0060(17)
C52	0.025(2)	0.068(2)	0.029(3)	0.022(2)	0.0093(17)	0.002(2)
C53	0.031(3)	0.072(3)	0.026(2)	0.017(2)	0.0071(16)	0.0071(19)
C54	0.027(2)	0.069(3)	0.029(3)	0.000(2)	0.0094(16)	0.013(2)
C55	0.020(2)	0.058(3)	0.033(2)	-0.0063(19)	0.0102(16)	0.009(2)
C56	0.024(2)	0.055(2)	0.032(2)	0.0016(19)	0.0080(14)	0.0046(18)
C57	0.020(3)	0.062(3)	0.054(3)	-0.017(2)	0.012(2)	0.008(2)
C58	0.049(3)	0.074(3)	0.068(3)	-0.022(2)	-0.006(2)	0.001(3)
C59	0.057(6)	0.082(4)	0.048(5)	-0.028(4)	-0.005(4)	0.018(4)
C60	0.043(5)	0.079(4)	0.031(4)	-0.009(3)	0.002(3)	0.018(4)
O61	0.066(5)	0.102(6)	0.069(5)	-0.039(4)	-0.019(3)	0.023(4)
C62	0.033(8)	0.135(10)	0.026(7)	-0.021(5)	0.020(6)	-0.006(7)
C71	0.023(2)	0.053(2)	0.019(2)	0.0061(18)	0.0096(15)	0.0060(17)
C72	0.025(2)	0.068(2)	0.029(3)	0.022(2)	0.0093(17)	0.002(2)
C73	0.031(3)	0.072(3)	0.026(2)	0.017(2)	0.0071(16)	0.0071(19)

C74	0.027(2)	0.069(3)	0.029(3)	0.000(2)	0.0094(16)	0.013(2)
C75	0.020(2)	0.058(3)	0.033(2)	-0.0063(19)	0.0102(16)	0.009(2)
C76	0.024(2)	0.055(2)	0.032(2)	0.0016(19)	0.0080(14)	0.0046(18)
C77	0.020(3)	0.062(3)	0.054(3)	-0.017(2)	0.012(2)	0.008(2)
C78	0.049(3)	0.074(3)	0.068(3)	-0.022(2)	-0.006(2)	0.001(3)
C79	0.079(10)	0.100(10)	0.115(6)	-0.019(7)	-0.030(8)	-0.027(7)
C80	0.039(7)	0.077(8)	0.140(9)	-0.017(6)	-0.012(7)	-0.014(6)
C81	0.039(8)	0.095(9)	0.124(7)	0.008(7)	-0.001(7)	-0.029(6)
C82	0.019(6)	0.068(7)	0.080(4)	0.007(4)	-0.002(5)	-0.004(4)
C51A	0.023(2)	0.053(2)	0.019(2)	0.0061(18)	0.0096(15)	0.0060(17)
C52A	0.025(2)	0.068(2)	0.029(3)	0.022(2)	0.0093(17)	0.002(2)
C53A	0.031(3)	0.072(3)	0.026(2)	0.017(2)	0.0071(16)	0.0071(19)
C54A	0.027(2)	0.069(3)	0.029(3)	0.000(2)	0.0094(16)	0.013(2)
C55A	0.020(2)	0.058(3)	0.033(2)	-0.0063(19)	0.0102(16)	0.009(2)
C56A	0.024(2)	0.055(2)	0.032(2)	0.0016(19)	0.0080(14)	0.0046(18)
C57A	0.020(3)	0.062(3)	0.054(3)	-0.017(2)	0.012(2)	0.008(2)
C58A	0.049(3)	0.074(3)	0.068(3)	-0.022(2)	-0.006(2)	0.001(3)
C59A	0.057(6)	0.082(4)	0.048(5)	-0.028(4)	-0.005(4)	0.018(4)
C60A	0.043(5)	0.079(4)	0.031(4)	-0.009(3)	0.002(3)	0.018(4)
O61A	0.066(5)	0.102(6)	0.069(5)	-0.039(4)	-0.019(3)	0.023(4)
C62A	0.033(8)	0.135(10)	0.026(7)	-0.021(5)	0.020(6)	-0.006(7)
C71A	0.023(2)	0.053(2)	0.019(2)	0.0061(18)	0.0096(15)	0.0060(17)
C72A	0.025(2)	0.068(2)	0.029(3)	0.022(2)	0.0093(17)	0.002(2)
C73A	0.031(3)	0.072(3)	0.026(2)	0.017(2)	0.0071(16)	0.0071(19)
C74A	0.027(2)	0.069(3)	0.029(3)	0.000(2)	0.0094(16)	0.013(2)
C75A	0.020(2)	0.058(3)	0.033(2)	-0.0063(19)	0.0102(16)	0.009(2)
C76A	0.024(2)	0.055(2)	0.032(2)	0.0016(19)	0.0080(14)	0.0046(18)
C77A	0.020(3)	0.062(3)	0.054(3)	-0.017(2)	0.012(2)	0.008(2)
C78A	0.049(3)	0.074(3)	0.068(3)	-0.022(2)	-0.006(2)	0.001(3)
C79A	0.079(10)	0.100(10)	0.115(6)	-0.019(7)	-0.030(8)	-0.027(7)
C80A	0.039(7)	0.077(8)	0.140(9)	-0.017(6)	-0.012(7)	-0.014(6)
C81A	0.039(8)	0.095(9)	0.124(7)	0.008(7)	-0.001(7)	-0.029(6)
C82A	0.019(6)	0.068(7)	0.080(4)	0.007(4)	-0.002(5)	-0.004(4)
C1	0.116(16)	0.108(11)	0.057(10)	0.039(8)	0.022(11)	-0.003(9)
Cl1	0.057(2)	0.138(4)	0.068(2)	0.002(3)	0.0054(19)	-0.001(2)
Cl2	0.047(2)	0.133(4)	0.133(4)	-0.010(3)	0.021(2)	-0.024(2)
C1A	0.116(16)	0.108(11)	0.057(10)	0.039(8)	0.022(11)	-0.003(9)
Cl1A	0.057(2)	0.138(4)	0.068(2)	0.002(3)	0.0054(19)	-0.001(2)
Cl2A	0.047(2)	0.133(4)	0.133(4)	-0.010(3)	0.021(2)	-0.024(2)

* The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2hka^*b^*U_{12}]$

Table S10. Hydrogen atom coordinates and isotropic atomic displacement parameters ($\text{\AA}^2$) for **4**.

Atom	x/a	y/b	z/c	U_{iso}
H12A	0.4462	0.7340	0.5983	0.046
H12B	0.4247	0.7310	0.4421	0.046
H13A	0.2509	0.6833	0.4587	0.060
H13B	0.2685	0.7646	0.4906	0.060
H14A	0.2975	0.7387	0.7204	0.063
H14B	0.1810	0.7172	0.6507	0.063
H15A	0.2144	0.6198	0.7554	0.058
H15B	0.2757	0.5998	0.6380	0.058
H16A	0.3769	0.5753	0.8431	0.046
H16B	0.3732	0.6554	0.8829	0.046
H17A	0.4916	0.6866	0.7534	0.045
H17B	0.5391	0.6182	0.8297	0.045
H12C	0.4330	0.7330	0.6053	0.046
H12D	0.4304	0.7347	0.4492	0.046
H13C	0.2646	0.6804	0.4120	0.060
H13D	0.2561	0.7478	0.5007	0.060
H14C	0.1684	0.6589	0.5901	0.063
H14D	0.2638	0.6054	0.5870	0.063
H15C	0.3194	0.7203	0.7476	0.058
H15D	0.2315	0.6717	0.7949	0.058
H16C	0.3534	0.5746	0.7892	0.046
H16D	0.3862	0.6300	0.9046	0.046
H22A	0.5843	0.2760	0.5674	0.046
H22B	0.5554	0.2711	0.4116	0.046
H23A	0.7311	0.2320	0.5054	0.060
H23B	0.7605	0.3118	0.5392	0.060
H24A	0.8170	0.2745	0.3402	0.063
H24B	0.6961	0.2608	0.2766	0.063
H25A	0.7400	0.3972	0.3631	0.058
H25B	0.7953	0.3743	0.2422	0.058
H26A	0.6319	0.3502	0.1164	0.046
H26B	0.6363	0.4291	0.1654	0.046
H27A	0.4706	0.3889	0.1772	0.045
H27B	0.5157	0.3194	0.2519	0.045
H22C	0.5786	0.2728	0.5616	0.046
H22D	0.5667	0.2711	0.4043	0.046
H23C	0.7430	0.2476	0.4796	0.060
H23D	0.7514	0.3086	0.5863	0.060
H24C	0.7422	0.3942	0.4196	0.063
H24D	0.8400	0.3430	0.4142	0.063

H25C	0.7809	0.3363	0.2070	0.058
H25D	0.6950	0.2832	0.2463	0.058
H26C	0.6210	0.3730	0.0986	0.046
H26D	0.6568	0.4289	0.2119	0.046
H32A	0.1821	0.4522	0.5684	0.054
H32B	0.2313	0.4311	0.7159	0.054
H33A	0.1638	0.3293	0.6057	0.062
H33B	0.2286	0.3440	0.4890	0.062
H34A	0.3862	0.3162	0.6170	0.058
H34B	0.3053	0.2546	0.6329	0.058
H35A	0.2824	0.3292	0.8335	0.058
H35B	0.3636	0.2655	0.8412	0.058
H36A	0.4990	0.3473	0.8122	0.048
H36B	0.4579	0.3522	0.9488	0.048
H37A	0.3538	0.4532	0.8606	0.037
H37B	0.4792	0.4643	0.8945	0.037
H42A	0.7886	0.5770	0.2944	0.054
H42B	0.8382	0.5559	0.4417	0.054
H43A	0.7883	0.6630	0.5222	0.062
H43B	0.8526	0.6792	0.4058	0.062
H44A	0.7080	0.7518	0.3806	0.058
H44B	0.6294	0.6883	0.3929	0.058
H45A	0.6526	0.7424	0.1713	0.058
H45B	0.7367	0.6803	0.1784	0.058
H46A	0.5651	0.6538	0.0588	0.048
H46B	0.5194	0.6585	0.1924	0.048
H47A	0.5448	0.5416	0.1139	0.037
H47B	0.6697	0.5543	0.1516	0.037
H52	0.6610	0.4051	0.8456	0.048
H53	0.7975	0.4216	1.0240	0.051
H56	0.6990	0.6104	0.7447	0.044
H57	0.8417	0.6802	0.8911	0.054
H58	0.9738	0.6882	1.0703	0.078
H60	0.9333	0.4888	1.1633	0.062
H62A	1.0285	0.5458	1.3859	0.094
H62B	1.1497	0.5618	1.3757	0.094
H62C	1.0837	0.5065	1.2785	0.094
H72	0.3805	0.5991	0.1690	0.048
H73	0.2268	0.6050	0.0050	0.051
H74	0.1138	0.5121	-0.0323	0.050
H76	0.3024	0.4049	0.2549	0.044
H78	0.0803	0.3980	-0.1079	0.078
H79	-0.0309	0.3059	-0.1406	0.124
H80	-0.0571	0.2404	0.0236	0.106

H81	0.0306	0.2628	0.2473	0.106
H82	0.1436	0.3573	0.2768	0.068
H52A	0.3463	0.6047	0.1408	0.048
H53A	0.2048	0.5820	-0.0286	0.051
H56A	0.3249	0.4009	0.2603	0.044
H57A	0.1819	0.3254	0.1274	0.054
H58A	0.0439	0.3114	-0.0435	0.078
H60A	0.0685	0.5095	-0.1549	0.062
H62D	-0.1213	0.4779	-0.2223	0.094
H62E	-0.1581	0.4254	-0.3415	0.094
H62F	-0.0506	0.4690	-0.3350	0.094
H72A	0.6300	0.4143	0.8305	0.048
H73A	0.7767	0.4097	1.0044	0.051
H74A	0.8920	0.5013	1.0426	0.050
H76A	0.7194	0.6046	0.7366	0.044
H78A	0.9083	0.6260	1.1111	0.078
H79A	1.0223	0.7167	1.1448	0.124
H80A	1.0761	0.7683	0.9797	0.106
H81A	1.0171	0.7316	0.7540	0.106
H82A	0.9007	0.6389	0.7235	0.068
H1A	0.8459	0.4921	0.6455	0.111
H1B	0.9192	0.4601	0.7718	0.111
H1C	0.0987	0.5532	0.2323	0.111
H1D	0.1716	0.5270	0.3639	0.111

Table S11. Bond lengths (Å) and angles (°) for **4**.

Rh1-C71	1.997(6)	Rh1-O1	2.020(5)	Rh1-O3	2.024(5)
Rh1-N2	2.074(5)	Rh1-N4	2.110(5)	Rh1-Rh2	2.5292(7)
Rh2-N3	1.922(5)	Rh2-C51	2.012(6)	Rh2-N1	2.015(5)
Rh2-O2	2.079(5)	Rh2-O4	2.197(5)	Rh2-C31	2.611(6)
O1-C11	1.287(5)	C11-N1	1.318(6)	C11-C12	1.515(6)
C12-C13	1.510(7)	C13-C14	1.547(10)	C14-C15	1.510(9)
C15-C16	1.508(8)	C16-C17	1.519(7)	C17-N1	1.472(6)
O2-C21	1.287(5)	C21-N2	1.317(6)	C21-C22	1.515(6)
C22-C23	1.510(7)	C23-C24	1.546(10)	C24-C25	1.510(9)
C25-C26	1.508(8)	C26-C27	1.520(7)	C27-N2	1.472(6)
O3-C31	1.294(5)	C31-N3	1.298(6)	C31-C32	1.497(7)
C32-C33	1.539(8)	C33-C34	1.508(8)	C34-C35	1.527(8)
C35-C36	1.515(8)	C36-C37	1.533(7)	C37-N3	1.469(6)
O4-C41	1.294(5)	C41-N4	1.299(6)	C41-C42	1.497(7)
C42-C43	1.540(8)	C43-C44	1.508(8)	C44-C45	1.527(8)
C45-C46	1.515(8)	C46-C47	1.533(7)	C47-N4	1.470(6)

C51-C56	1.381(14)	C51-C52	1.479(13)	C52-C53	1.407(12)
C53-C54	1.358(15)	C54-C55	1.388(15)	C54-C60	1.456(15)
C55-C57	1.431(13)	C55-C56	1.438(14)	C57-C58	1.369(17)
C58-C59	1.39(2)	C59-C60	1.332(16)	C59-O61	1.399(13)
O61-C62	1.40(2)	C71-C72	1.322(14)	C71-C76	1.375(12)
C72-C73	1.422(14)	C73-C74	1.341(14)	C74-C75	1.412(10)
C75-C76	1.390(10)	C75-C77	1.434(12)	C77-C78	1.355(12)
C77-C82	1.421(10)	C78-C79	1.333(18)	C79-C80	1.255(19)
C80-C81	1.471(19)	C81-C82	1.334(15)	C1-C11	1.696(13)
C1-C12	1.698(14)				

C71-Rh1-O1	91.1(3)	C71-Rh1-O3	87.1(2)	O1-Rh1-O3	94.1(3)
C71-Rh1-N2	98.6(3)	O1-Rh1-N2	168.6(3)	O3-Rh1-N2	92.4(3)
C71-Rh1-N4	102.1(2)	O1-Rh1-N4	83.2(2)	O3-Rh1-N4	170.4(3)
N2-Rh1-N4	88.9(3)	C71-Rh1-Rh2	156.79(18)	O1-Rh1-Rh2	84.79(15)
O3-Rh1-Rh2	70.44(15)	N2-Rh1-Rh2	88.55(16)	N4-Rh1-Rh2	100.08(15)
N3-Rh2-C51	106.9(2)	N3-Rh2-N1	94.5(3)	C51-Rh2-N1	96.5(3)
N3-Rh2-O2	88.9(3)	C51-Rh2-O2	87.8(3)	N1-Rh2-O2	173.5(3)
N3-Rh2-O4	173.1(3)	C51-Rh2-O4	76.5(2)	N1-Rh2-O4	91.1(3)
O2-Rh2-O4	85.2(2)	N3-Rh2-Rh1	102.59(17)	C51-Rh2-Rh1	149.54(17)
N1-Rh2-Rh1	88.62(17)	O2-Rh2-Rh1	85.21(15)	O4-Rh2-Rh1	73.39(14)
N3-Rh2-C31	28.43(16)	C51-Rh2-C31	135.2(2)	N1-Rh2-C31	92.3(3)
O2-Rh2-C31	88.0(2)	O4-Rh2-C31	147.3(2)	Rh1-Rh2-C31	74.20(13)
C11-O1-Rh1	122.9(4)	O1-C11-N1	124.5(4)	O1-C11-C12	111.9(4)
N1-C11-C12	123.5(4)	C13-C12-C11	116.6(6)	C12-C13-C14	113.8(6)
C15-C14-C13	115.0(7)	C16-C15-C14	114.4(6)	C15-C16-C17	118.6(5)
N1-C17-C16	114.9(4)	C11-N1-C17	118.5(4)	C11-N1-Rh2	118.4(4)
C17-N1-Rh2	122.0(4)	C21-O2-Rh2	122.9(4)	O2-C21-N2	124.4(4)
O2-C21-C22	112.0(4)	N2-C21-C22	123.6(4)	C23-C22-C21	116.5(6)
C22-C23-C24	113.8(6)	C25-C24-C23	115.2(7)	C26-C25-C24	114.3(6)
C25-C26-C27	118.3(5)	N2-C27-C26	114.6(4)	C21-N2-C27	118.6(4)
C31-O3-Rh1	137.3(4)	O3-C31-N3	122.6(5)	O3-C31-C32	114.4(4)
N3-C31-C32	123.0(4)	O3-C31-Rh2	77.8(3)	N3-C31-Rh2	44.8(3)
C32-C31-Rh2	167.8(4)	C31-C32-C33	111.1(5)	C34-C33-C32	113.6(5)
C33-C34-C35	115.8(5)	C36-C35-C34	115.8(5)	C35-C36-C37	119.2(5)
N3-C37-C36	114.5(4)	C31-N3-C37	123.7(4)	C31-N3-Rh2	106.8(4)
C37-N3-Rh2	129.0(4)	C41-O4-Rh2	134.3(4)	O4-C41-N4	122.7(5)
O4-C41-C42	114.6(4)	N4-C41-C42	122.7(4)	C41-C42-C43	111.1(5)
C44-C43-C42	113.6(5)	C43-C44-C45	115.6(5)	C46-C45-C44	115.7(5)
C45-C46-C47	119.3(5)	N4-C47-C46	114.5(4)	C41-N4-C47	123.2(4)
C41-N4-Rh1	109.1(4)	C47-N4-Rh1	127.1(4)	C56-C51-C52	117.6(7)
C56-C51-Rh2	121.0(7)	C52-C51-Rh2	121.2(5)	C53-C52-C51	117.6(10)
C54-C53-C52	124.8(11)	C53-C54-C55	117.5(10)	C53-C54-C60	121.3(10)
C55-C54-C60	121.3(10)	C54-C55-C57	115.2(9)	C54-C55-C56	121.7(10)

C57-C55-C56	123.1(10)	C51-C56-C55	120.9(11)	C58-C57-C55	122.4(12)
C57-C58-C59	120.9(13)	C60-C59-C58	119.9(11)	C60-C59-O61	127.9(12)
C58-C59-O61	112.2(12)	C59-C60-C54	120.3(11)	C62-O61-C59	116.9(11)
C72-C71-C76	121.8(8)	C72-C71-Rh1	118.8(6)	C76-C71-Rh1	118.3(7)
C71-C72-C73	118.9(10)	C74-C73-C72	120.8(11)	C73-C74-C75	120.3(10)
C76-C75-C74	117.6(8)	C76-C75-C77	122.0(6)	C74-C75-C77	120.4(6)
C71-C76-C75	120.6(11)	C78-C77-C82	115.7(10)	C78-C77-C75	124.6(8)
C82-C77-C75	119.7(6)	C79-C78-C77	124.1(16)	C80-C79-C78	119.7(16)
C79-C80-C81	123.4(15)	C82-C81-C80	114.6(15)	C81-C82-C77	122.5(13)
Cl1-C1-Cl2	115.0(12)				

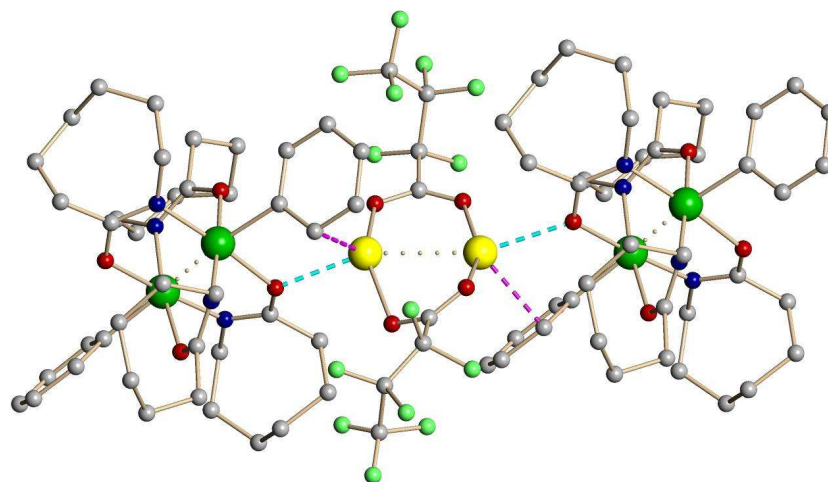
Table S12. Torsion angles (°) for **4**.

C71-Rh1-Rh2-N3	19.2(6)	O1-Rh1-Rh2-N3	99.8(3)
O3-Rh1-Rh2-N3	3.6(3)	N2-Rh1-Rh2-N3	-89.4(3)
N4-Rh1-Rh2-N3	-178.1(3)	C71-Rh1-Rh2-C51	-175.6(7)
O1-Rh1-Rh2-C51	-95.0(5)	O3-Rh1-Rh2-C51	168.8(5)
N2-Rh1-Rh2-C51	75.8(5)	N4-Rh1-Rh2-C51	-12.8(5)
C71-Rh1-Rh2-N1	-75.1(6)	O1-Rh1-Rh2-N1	5.5(3)
O3-Rh1-Rh2-N1	-90.7(3)	N2-Rh1-Rh2-N1	176.3(3)
N4-Rh1-Rh2-N1	87.7(3)	C71-Rh1-Rh2-O2	107.0(6)
O1-Rh1-Rh2-O2	-172.4(3)	O3-Rh1-Rh2-O2	91.4(3)
N2-Rh1-Rh2-O2	-1.7(3)	N4-Rh1-Rh2-O2	-90.3(2)
C71-Rh1-Rh2-O4	-166.6(6)	O1-Rh1-Rh2-O4	-86.0(2)
O3-Rh1-Rh2-O4	177.8(3)	N2-Rh1-Rh2-O4	84.7(3)
N4-Rh1-Rh2-O4	-3.9(3)	C71-Rh1-Rh2-C31	17.7(6)
O1-Rh1-Rh2-C31	98.3(2)	O3-Rh1-Rh2-C31	2.1(3)
N2-Rh1-Rh2-C31	-90.9(3)	N4-Rh1-Rh2-C31	-179.5(3)
C71-Rh1-O1-C11	148.8(6)	O3-Rh1-O1-C11	61.6(6)
N2-Rh1-O1-C11	-62.8(15)	N4-Rh1-O1-C11	-109.2(6)
Rh2-Rh1-O1-C11	-8.4(6)	Rh1-O1-C11-N1	7.5(11)
Rh1-O1-C11-C12	-171.1(4)	O1-C11-C12-C13	83.9(8)
N1-C11-C12-C13	-94.7(9)	C11-C12-C13-C14	76.2(11)
C12-C13-C14-C15	-64.3(13)	C13-C14-C15-C16	97.7(8)
C14-C15-C16-C17	-62.2(11)	C15-C16-C17-N1	-46.5(10)
O1-C11-N1-C17	-168.6(7)	C12-C11-N1-C17	9.8(10)
O1-C11-N1-Rh2	-0.3(11)	C12-C11-N1-Rh2	178.1(5)
C16-C17-N1-C11	82.4(8)	C16-C17-N1-Rh2	-85.5(7)
N3-Rh2-N1-C11	-107.2(6)	C51-Rh2-N1-C11	145.2(6)
O4-Rh2-N1-C11	68.7(6)	Rh1-Rh2-N1-C11	-4.7(6)
C31-Rh2-N1-C11	-78.8(6)	N3-Rh2-N1-C17	60.6(5)
C51-Rh2-N1-C17	-46.9(5)	O4-Rh2-N1-C17	-123.5(5)
Rh1-Rh2-N1-C17	163.2(5)	C31-Rh2-N1-C17	89.0(5)

N3-Rh2-O2-C21	102.2(6)	C51-Rh2-O2-C21	-150.9(6)
O4-Rh2-O2-C21	-74.3(6)	Rh1-Rh2-O2-C21	-0.6(6)
C31-Rh2-O2-C21	73.7(6)	Rh2-O2-C21-N2	4.5(11)
Rh2-O2-C21-C22	-173.8(4)	O2-C21-C22-C23	-85.3(9)
N2-C21-C22-C23	96.5(8)	C21-C22-C23-C24	-73.5(12)
C22-C23-C24-C25	64.9(13)	C23-C24-C25-C26	-99.0(8)
C24-C25-C26-C27	59.2(11)	C25-C26-C27-N2	49.8(10)
O2-C21-N2-C27	166.2(7)	C22-C21-N2-C27	-15.8(10)
O2-C21-N2-Rh1	-6.4(11)	C22-C21-N2-Rh1	171.6(5)
C26-C27-N2-C21	-80.1(8)	C26-C27-N2-Rh1	92.2(7)
C71-Rh1-N2-C21	-153.4(6)	O1-Rh1-N2-C21	58.6(15)
O3-Rh1-N2-C21	-65.9(6)	N4-Rh1-N2-C21	104.6(6)
Rh2-Rh1-N2-C21	4.5(6)	C71-Rh1-N2-C27	34.4(5)
O1-Rh1-N2-C27	-113.7(13)	O3-Rh1-N2-C27	121.8(5)
N4-Rh1-N2-C27	-67.7(5)	Rh2-Rh1-N2-C27	-167.8(5)
C71-Rh1-O3-C31	180.0(9)	O1-Rh1-O3-C31	-89.1(8)
N2-Rh1-O3-C31	81.5(8)	Rh2-Rh1-O3-C31	-6.1(8)
Rh1-O3-C31-N3	5.9(13)	Rh1-O3-C31-C32	-173.9(6)
Rh1-O3-C31-Rh2	5.7(7)	N3-Rh2-C31-O3	179.8(8)
C51-Rh2-C31-O3	-173.7(4)	N1-Rh2-C31-O3	84.7(5)
O2-Rh2-C31-O3	-88.8(5)	O4-Rh2-C31-O3	-10.9(6)
Rh1-Rh2-C31-O3	-3.2(4)	C51-Rh2-C31-N3	6.5(7)
N1-Rh2-C31-N3	-95.1(6)	O2-Rh2-C31-N3	91.4(5)
O4-Rh2-C31-N3	169.3(5)	Rh1-Rh2-C31-N3	177.0(6)
N3-Rh2-C31-C32	-2(2)	C51-Rh2-C31-C32	5(3)
N1-Rh2-C31-C32	-97(2)	O2-Rh2-C31-C32	89(2)
O4-Rh2-C31-C32	167(2)	Rh1-Rh2-C31-C32	175(2)
O3-C31-C32-C33	79.2(9)	N3-C31-C32-C33	-100.5(8)
Rh2-C31-C32-C33	-99(2)	C31-C32-C33-C34	53.6(9)
C32-C33-C34-C35	50.6(10)	C33-C34-C35-C36	-98.2(7)
C34-C35-C36-C37	71.9(9)	C35-C36-C37-N3	-74.6(7)
O3-C31-N3-C37	-172.7(7)	C32-C31-N3-C37	7.0(11)
Rh2-C31-N3-C37	-172.5(9)	O3-C31-N3-Rh2	-0.2(9)
C32-C31-N3-Rh2	179.5(6)	C36-C37-N3-C31	74.8(8)
C36-C37-N3-Rh2	-95.9(7)	C51-Rh2-N3-C31	-175.2(5)
N1-Rh2-N3-C31	86.6(5)	O2-Rh2-N3-C31	-87.8(5)
Rh1-Rh2-N3-C31	-3.0(5)	C51-Rh2-N3-C37	-3.3(7)
N1-Rh2-N3-C37	-101.5(6)	O2-Rh2-N3-C37	84.1(6)
Rh1-Rh2-N3-C37	169.0(5)	C31-Rh2-N3-C37	171.9(10)
N3-Rh2-O4-C41	62(2)	C51-Rh2-O4-C41	-177.4(8)
N1-Rh2-O4-C41	-81.0(8)	O2-Rh2-O4-C41	93.7(8)
Rh1-Rh2-O4-C41	7.3(7)	C31-Rh2-O4-C41	15.0(10)
Rh2-O4-C41-N4	-7.2(12)	Rh2-O4-C41-C42	169.9(5)
O4-C41-C42-C43	-78.5(9)	N4-C41-C42-C43	98.6(8)

C41-C42-C43-C44	-53.5(9)	C42-C43-C44-C45	-51.8(10)
C43-C44-C45-C46	98.2(7)	C44-C45-C46-C47	-69.9(9)
C45-C46-C47-N4	73.9(7)	O4-C41-N4-C47	173.5(7)
C42-C41-N4-C47	-3.4(11)	O4-C41-N4-Rh1	1.1(9)
C42-C41-N4-Rh1	-175.8(6)	C46-C47-N4-C41	-77.9(8)
C46-C47-N4-Rh1	93.1(7)	C71-Rh1-N4-C41	176.2(5)
O1-Rh1-N4-C41	86.5(5)	N2-Rh1-N4-C41	-85.3(5)
Rh2-Rh1-N4-C41	3.1(5)	C71-Rh1-N4-C47	4.2(6)
O1-Rh1-N4-C47	-85.5(6)	N2-Rh1-N4-C47	102.7(6)
Rh2-Rh1-N4-C47	-168.9(5)	N3-Rh2-C51-C56	-125.8(3)
N1-Rh2-C51-C56	-29.1(3)	O2-Rh2-C51-C56	146.0(3)
O4-Rh2-C51-C56	60.4(3)	Rh1-Rh2-C51-C56	69.3(5)
C31-Rh2-C51-C56	-129.0(4)	N3-Rh2-C51-C52	48.1(4)
N1-Rh2-C51-C52	144.9(3)	O2-Rh2-C51-C52	-40.0(3)
O4-Rh2-C51-C52	-125.6(3)	Rh1-Rh2-C51-C52	-116.8(4)
C31-Rh2-C51-C52	44.9(5)	C56-C51-C52-C53	-0.03(5)
Rh2-C51-C52-C53	-174.2(3)	C51-C52-C53-C54	0.00(6)
C52-C53-C54-C55	0.03(12)	C52-C53-C54-C60	180.00(7)
C53-C54-C55-C57	-179.92(9)	C60-C54-C55-C57	0.10(16)
C53-C54-C55-C56	-0.03(16)	C60-C54-C55-C56	179.99(10)
C52-C51-C56-C55	0.02(12)	Rh2-C51-C56-C55	174.2(3)
C54-C55-C56-C51	0.00(16)	C57-C55-C56-C51	179.89(9)
C54-C55-C57-C58	-0.16(16)	C56-C55-C57-C58	179.95(11)
C55-C57-C58-C59	0.14(17)	C57-C58-C59-C60	-0.06(17)
C57-C58-C59-O61	179.99(10)	C58-C59-C60-C54	0.01(17)
O61-C59-C60-C54	179.95(11)	C53-C54-C60-C59	179.99(10)
C55-C54-C60-C59	-0.03(17)	C60-C59-O61-C62	-1.3(9)
C58-C59-O61-C62	178.7(9)	O1-Rh1-C71-C72	29.5(4)
O3-Rh1-C71-C72	123.5(4)	N2-Rh1-C71-C72	-144.5(4)
N4-Rh1-C71-C72	-53.8(4)	Rh2-Rh1-C71-C72	108.8(6)
O1-Rh1-C71-C76	-138.9(3)	O3-Rh1-C71-C76	-44.9(3)
N2-Rh1-C71-C76	47.1(3)	N4-Rh1-C71-C76	137.8(3)
Rh2-Rh1-C71-C76	-59.6(7)	C76-C71-C72-C73	0.03(6)
Rh1-C71-C72-C73	-167.9(4)	C71-C72-C73-C74	-0.03(6)
C72-C73-C74-C75	0.01(13)	C73-C74-C75-C76	0.01(17)
C73-C74-C75-C77	179.90(9)	C72-C71-C76-C75	-0.01(13)
Rh1-C71-C76-C75	168.0(4)	C74-C75-C76-C71	-0.01(17)
C77-C75-C76-C71	-179.90(9)	C76-C75-C77-C78	-145.5(5)
C74-C75-C77-C78	34.6(5)	C76-C75-C77-C82	34.5(5)
C74-C75-C77-C82	-145.4(5)	C82-C77-C78-C79	0.00(6)
C75-C77-C78-C79	-179.99(6)	C77-C78-C79-C80	-0.01(7)
C78-C79-C80-C81	0.04(15)	C79-C80-C81-C82	-0.05(19)
C80-C81-C82-C77	0.04(19)	C78-C77-C82-C81	-0.01(15)
C75-C77-C82-C81	179.98(11)		

X-Ray data for 12



A green-red needle-like specimen of $C_{46}H_{58}Ag_2F_{14}N_4O_8Rh_2$, approximate dimensions 0.05 mm x 0.06 mm x 0.20 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker SMART Apex 2 CCD system equipped with a graphite monochromator and a MoK_{α} fine focus sealed tube ($\lambda = 0.71073 \text{ \AA}$). Data collection temperature was 150°K.

The total exposure time was 19.62 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame algorithm. The integration of the data using a triclinic unit cell yielded a total of 31039 reflections to a maximum θ angle of 27.50° ($0.77 \text{ \AA}$ resolution), of which 12068 were independent (average redundancy 2.572, completeness = 99.8%, $R_{\text{int}} = 2.48\%$, $R_{\text{sig}} = 3.22\%$) and 9907 (82.09%) were greater than $2\sigma(F^2)$. The final cell constants of $a = 11.9673(11) \text{ \AA}$, $b = 14.4165(13) \text{ \AA}$, $c = 17.0517(16) \text{ \AA}$, $\alpha = 89.008(2)^\circ$, $\beta = 74.2910(10)^\circ$, $\gamma = 68.7950(10)^\circ$, volume = $2629.6(4) \text{ \AA}^3$, are based upon the refinement of the XYZ-centroids of 9962 reflections above $20 \sigma(I)$ with $4.983^\circ < 2\theta < 62.89^\circ$. Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.914. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.8500 and 0.9300.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group P -1, with $Z = 2$ for the formula unit, $C_{46}H_{58}Ag_2F_{14}N_4O_8Rh_2$. The final anisotropic full-matrix least-squares refinement on F^2 with 798 variables converged at $R1 = 3.07\%$, for the observed data and $wR2 = 5.82\%$ for all data. The goodness-of-fit was 1.001. The largest peak in the final difference electron density synthesis was $0.637 \text{ e}/\text{\AA}^3$ and the largest hole was $-0.461 \text{ e}/\text{\AA}^3$ with an RMS deviation of $0.088 \text{ e}/\text{\AA}^3$. On the basis of the final model, the calculated density was $1.872 \text{ g}/\text{cm}^3$ and $F(000)$, 1472 e^- .

The structure shows strong disorder of one of two $C_3F_7COO^-$ ions coordinating Ag atoms. The multiple conformation of the ion are superimposed onto each other. The major conformation of the ion was found and refined in anisotropic approximation including occupation factors. The other conformations were modeled by refining occupation factors of multiple C and F atoms. The total count of electrons of disordered model corresponds to whole C_3F_7 group.

Table S13. Sample and crystal data for 12.

Chemical formula	C ₄₆ H ₅₈ Ag ₂ F ₁₄ N ₄ O ₈ Rh ₂
Formula weight	1482.52
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal size	0.05 x 0.06 x 0.20 mm
Crystal habit	green-red needle
Crystal system	triclinic
Space group	P -1
Unit cell dimensions	a = 11.9673(11) Å α = 89.008(2)° b = 14.4165(13) Å β = 74.2910(10)° c = 17.0517(16) Å γ = 68.7950(10)°
Volume	2629.6(4) Å ³
Z	2
Density (calculated)	1.872 Mg/cm ³
Absorption coefficient	1.455 mm ⁻¹
F(000)	1472
Diffractometer	Bruker Smart Apex 2 CCD
Radiation source	fine focus sealed tube, MoK _α
Theta range for data collection	2.05 to 27.50°
Index ranges	-15 ≤ h ≤ 15, -18 ≤ k ≤ 18, -22 ≤ l ≤ 22
Reflections collected	31039
Independent reflections	12068 [R(int) = 0.0248]
Coverage of independent reflections	99.8%
Absorption correction	multi-scan
Max. and min. transmission	0.9300 and 0.8500
Structure solution technique	direct methods
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Refinement method	Full-matrix least-squares on F ²
Refinement program	SHELXL-97 (Sheldrick, 2008)
Function minimized	Σ w(F _o ² - F _c ²) ²
Data / restraints / parameters	12068 / 135 / 798
Goodness-of-fit on F ²	1.001
Δ/σ _{max}	0.002
Final R indices	9907 data; I > 2σ(I) R1 = 0.0307, wR2 = 0.0554 all data R1 = 0.0421, wR2 = 0.0582
Weighting scheme	w=1/[σ ² (F _o ²)+(0.01P) ² +4.72P], P=(max(F _o ² ,0)+2F _c ²)/3
Largest diff. peak and hole	0.637 and -0.461 eÅ ⁻³
R.M.S. deviation from mean	0.088 eÅ ⁻³

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

$$R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$$

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

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

$$w = 1 / [\sigma(F_o^2) + (aP)^2 + bP] \text{ where } P \text{ is } [2F_c^2 + \text{Max}(F_o^2, 0)] / 3$$

Table S14. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å²) for **12**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	U _{eq}
Rh1	0.575877(19)	0.760355(15)	0.684187(13)	0.01678(5)
Rh2	0.39126(19)	0.782027(16)	0.804932(13)	0.01769(5)
C11	0.7234(2)	0.7914(2)	0.61746(17)	0.61746(17)
C12	0.7381(3)	0.8775(2)	0.8775(2)	0.0223(6)

C13	0.8414(3)	0.8999(2)	0.5982(2)	0.0290(7)
C14	0.9272(3)	0.8391(3)	0.5302(2)	0.0335(8)
C15	0.9109(3)	0.7556(2)	0.5050(2)	0.0316(7)
C16	0.8105(3)	0.7311(2)	0.54869(19)	0.0266(6)
C21	0.2320(3)	0.7703(2)	0.87209(17)	0.0224(6)
C22	0.1208(3)	0.8520(2)	0.88152(18)	0.0244(6)
C23	0.0085(3)	0.8498(3)	0.9328(2)	0.0330(7)
C24	0.0079(3)	0.7670(3)	0.9736(2)	0.0371(8)
C25	0.1176(3)	0.6843(3)	0.9622(2)	0.0344(8)
C26	0.2294(3)	0.6849(2)	0.91094(19)	0.0290(7)
O30	0.30865(17)	0.79954(15)	0.70893(12)	0.0223(4)
C31	0.3658(3)	0.7794(2)	0.63090(17)	0.0215(6)
C32	0.2789(3)	0.7975(2)	0.57730(18)	0.0282(7)
C33	0.2250(3)	0.9089(2)	0.5651(2)	0.0316(7)
C34	0.3223(3)	0.9563(2)	0.53667(19)	0.0291(7)
C35	0.4272(3)	0.9038(2)	0.45854(19)	0.0293(7)
C36	0.5493(3)	0.8311(2)	0.47204(19)	0.0290(7)
C37	0.5478(3)	0.7359(2)	0.51213(17)	0.0247(6)
N30	0.4868(2)	0.75237(17)	0.60117(14)	0.0198(5)
O40	0.45552(18)	0.63008(15)	0.77883(13)	0.0274(5)
C41	0.5671(3)	0.5747(2)	0.73705(19)	0.0258(6)
C42	0.5935(3)	0.4644(2)	0.7432(2)	0.0431(9)
C43	0.5452(4)	0.4252(3)	0.6815(3)	0.0623(13)
C44	0.5973(4)	0.4451(3)	0.5931(3)	0.0587(12)
C45	0.7400(4)	0.4094(3)	0.5630(3)	0.0506(10)
C46	0.7927(3)	0.4906(2)	0.5655(2)	0.0356(8)
C47	0.7754(3)	0.5409(2)	0.6483(2)	0.0275(7)
N40	0.6468(2)	0.60908(17)	0.68988(15)	0.0223(5)
O50	0.64500(17)	0.77695(14)	0.78209(12)	0.0207(4)
C51	0.5992(3)	0.76716(19)	0.85866(17)	0.0197(6)
C52	0.6830(3)	0.7628(2)	0.91182(19)	0.0276(7)
C53	0.7796(4)	0.6562(4)	0.9062(8)	0.0372(13)
C54	0.7270(4)	0.5722(3)	0.9198(3)	0.0369(11)
C55	0.6231(4)	0.5893(3)	0.0000(3)	0.0377(11)
C56	0.4904(4)	0.6319(3)	0.9921(3)	0.0365(15)
C57	0.4440(3)	0.7408(2)	0.97255(19)	0.0300(7)
N50	0.4893(2)	0.76149(17)	0.88734(14)	0.0207(5)
C52A	0.6830(3)	0.7628(2)	0.91182(19)	0.0276(7)
C53A	0.7914(11)	0.6632(12)	0.909(3)	0.0372(13)
C54A	0.7336(12)	0.5983(9)	0.9669(9)	0.0369(11)
C55A	0.6459(11)	0.5659(9)	0.9329(9)	0.0377(11)
C56A	0.5060(11)	0.6262(7)	0.9681(16)	0.0365(15)
C57A	0.4440(3)	0.7408(2)	0.97255(19)	0.0300(7)
O60	0.47695(16)	0.91373(13)	0.69040(11)	0.0186(4)

C61	0.3919(2)	0.9684(2)	0.75312(17)	0.0186(6)
C62	0.3505(3)	0.0794(2)	0.74257(18)	0.0226(6)
C63	0.4315(3)	0.1304(2)	0.76492(18)	0.0271(7)
C64	0.4160(3)	0.1444(2)	0.85636(19)	0.0292(7)
C65	0.4592(3)	0.0458(2)	0.89622(18)	0.0259(6)
C66	0.3556(3)	0.0112(2)	0.94324(18)	0.0250(6)
C67	0.2720(3)	0.9962(2)	0.89525(17)	0.0225(6)
N60	0.3425(2)	0.93139(17)	0.81862(14)	0.0185(5)
Ag1	0.10300(2)	0.780111(19)	0.752243(16)	0.03147(6)
Ag2	0.86214(2)	0.785677(18)	0.737423(16)	0.02953(6)
O71	0.1630(2)	0.61985(18)	0.70975(17)	0.0453(6)
O72	0.9764(2)	0.62540(17)	0.70382(16)	0.0410(6)
C71	0.0883(3)	0.5845(2)	0.69891(19)	0.0277(7)
C72	0.1417(3)	0.4697(3)	0.6773(2)	0.0360(8)
F71	0.26696(19)	0.43387(16)	0.64416(14)	0.0523(6)
F72	0.0933(2)	0.44626(16)	0.61995(14)	0.0528(6)
C73	0.1127(3)	0.4086(2)	0.7491(2)	0.0400(8)
F73	0.1688(2)	0.31039(15)	0.72226(16)	0.0611(7)
F74	0.98795(19)	0.42962(16)	0.77365(15)	0.0521(6)
C74	0.1512(4)	0.4234(3)	0.8234(3)	0.0475(9)
F75	0.2718(2)	0.4048(2)	0.80619(16)	0.0662(7)
F76	0.0885(2)	0.51551(18)	0.85913(14)	0.0620(6)
F77	0.1247(3)	0.3618(2)	0.87930(17)	0.0765(8)
O81	0.9874(2)	0.94516(19)	0.74428(17)	0.0477(7)
O82	0.8010(2)	0.93806(17)	0.80660(15)	0.0390(6)
C81	0.8758(3)	0.9795(3)	0.7841(3)	0.0430(9)
C82	0.8250(7)	0.0873(5)	0.8310(5)	0.055(3)
F81	0.8119(14)	0.0887(6)	0.9110(5)	0.076(4)
F82	0.7083(7)	0.1411(5)	0.8137(10)	0.088(4)
C83	0.897(3)	0.1512(11)	0.8027(14)	0.074(3)
F83	0.7919(5)	0.2593(3)	0.8403(3)	0.144(3)
F84	0.9997(12)	0.1251(9)	0.8234(10)	0.111(5)
C84	0.9423(12)	0.1724(7)	0.7169(7)	0.096(3)
F85	0.9285(8)	0.2606(5)	0.7044(6)	0.112(3)
F86	0.8753(19)	0.1287(6)	0.6790(8)	0.114(4)
F87	0.0530(12)	0.1215(9)	0.6921(11)	0.113(5)
C82A	0.8254(11)	0.0972(8)	0.7751(9)	0.04
C83A	0.862(5)	0.156(3)	0.822(3)	0.04
C83B	0.8319(17)	0.1664(13)	0.7754(11)	0.04
C84A	0.9963(12)	0.1633(10)	0.7711(9)	0.04
C84B	0.897(2)	0.2414(15)	0.8164(13)	0.04
F81A	0.8748(17)	0.1058(6)	0.8918(7)	0.05
F81B	0.928(2)	0.0949(9)	0.8765(8)	0.05
F81C	0.0661(14)	0.0790(14)	0.7669(12)	0.05

F81D	0.6941(14)	0.1232(12)	0.8567(16)	0.05
F81E	0.8081(19)	0.1335(11)	0.7048(10)	0.05
F81F	0.0086(15)	0.2238(13)	0.8210(9)	0.05
F81G	0.0494(19)	0.108(2)	0.745(2)	0.05
F81H	0.9609(19)	0.1088(12)	0.6584(10)	0.05
F81I	0.966(2)	0.2601(17)	0.8444(12)	0.05
F81J	0.721(2)	0.1066(13)	0.8895(15)	0.05
F81K	0.733(2)	0.1454(15)	0.7426(15)	0.05
F81L	0.885(3)	0.2801(19)	0.747(2)	0.05
F81M	0.750(2)	0.196(2)	0.7248(15)	0.05

Table S15. Bond lengths (Å) and angles (°) for **12**.

Rh1-N30	2.016(2)	Rh1-C11	2.020(3)	Rh1-N40	2.045(2)
Rh1-O60	2.0852(18)	Rh1-O50	2.1056(19)	Rh1-Rh2	2.5055(3)
Rh2-C21	2.004(3)	Rh2-N60	2.017(2)	Rh2-N50	2.017(2)
Rh2-O40	2.054(2)	Rh2-O30	2.0982(19)	C11-C16	1.392(4)
C11-C12	1.400(4)	C12-C13	1.404(4)	C12-Ag2#2	2.532(3)
C12-H12	0.95	C13-C14	1.381(5)	C13-H13	0.95
C14-C15	1.380(5)	C14-H14	0.95	C15-C16	1.392(4)
C15-H15	0.95	C16-H16	0.95	C21-C22	1.393(4)
C21-C26	1.395(4)	C22-C23	1.402(4)	C22-Ag1	2.544(3)
C22-H22	0.95	C23-C24	1.372(5)	C23-H23	0.95
C24-C25	1.386(5)	C24-H24	0.95	C25-C26	1.390(4)
C25-H25	0.95	C26-H26	0.95	O30-C31	1.304(3)
O30-Ag1	2.4876(18)	C31-N30	1.306(3)	C31-C32	1.515(4)
C32-C33	1.533(4)	C32-H32A	0.99	C32-H32B	0.99
C33-C34	1.522(4)	C33-H33A	0.99	C33-H33B	0.99
C34-C35	1.538(4)	C34-H34A	0.99	C34-H34B	0.99
C35-C36	1.533(4)	C35-H35A	0.99	C35-H35B	0.99
C36-C37	1.527(4)	C36-H36A	0.99	C36-H36B	0.99
C37-N30	1.477(4)	C37-H37A	0.99	C37-H37B	0.99
O40-C41	1.289(3)	C41-N40	1.309(4)	C41-C42	1.512(4)
C42-C43	1.532(6)	C42-H42A	0.99	C42-H42B	0.99
C43-C44	1.532(6)	C43-H43A	0.99	C43-H43B	0.99
C44-C45	1.531(5)	C44-H44A	0.99	C44-H44B	0.99
C45-C46	1.523(5)	C45-H45A	0.99	C45-H45B	0.99
C46-C47	1.527(4)	C46-H46A	0.99	C46-H46B	0.99
C47-N40	1.475(4)	C47-H47A	0.99	C47-H47B	0.99
O50-C51	1.299(3)	O50-Ag2#2	2.5514(18)	C51-N50	1.305(3)
C51-C52	1.508(4)	C52-C53	1.539(5)	C52-H52A	0.99
C52-H52B	0.99	C53-C54	1.542(7)	C53-H53A	0.99
C53-H53B	0.99	C54-C55	1.532(6)	C54-H54A	0.99

C54-H54B	0.99	C55-C56	1.525(6)	C55-H55A	0.99
C55-H55B	0.99	C56-C57	1.528(5)	C56-H56A	0.99
C56-H56B	0.99	C57-N50	1.473(4)	C57-H57A	0.99
C57-H57B	0.99	C53A-C54A	1.545(11)	C53A-H53C	0.99
C53A-H53D	0.99	C54A-C55A	1.532(10)	C54A-H54C	0.99
C54A-H54D	0.99	C55A-C56A	1.530(10)	C55A-H55C	0.99
C55A-H55D	0.99	C56A-H56C	0.99	C56A-H56D	0.99
O60-C61	1.289(3)	C61-N60	1.316(3)	C61-C62	1.517(4)
C62-C63	1.534(4)	C62-H62A	0.99	C62-H62B	0.99
C63-C64	1.528(4)	C63-H63A	0.99	C63-H63B	0.99
C64-C65	1.541(4)	C64-H64A	0.99	C64-H64B	0.99
C65-C66	1.524(4)	C65-H65A	0.99	C65-H65B	0.99
C66-C67	1.525(4)	C66-H66A	0.99	C66-H66B	0.99
C67-N60	1.472(3)	C67-H67A	0.99	C67-H67B	0.99
Ag1-O71	2.226(2)	Ag1-O81	2.303(3)	Ag1-Ag2	2.9335(4)
Ag2-O72	2.207(2)	Ag2-O82	2.283(2)	Ag2-C12#1	2.532(3)
Ag2-O50#1	2.5513(18)	O71-C71	1.231(4)	O72-C71	1.232(4)
C71-C72	1.553(4)	C72-F71	1.351(4)	C72-F72	1.366(4)
C72-C73	1.532(5)	C73-F73	1.354(4)	C73-F74	1.355(4)
C73-C74	1.502(5)	C74-F75	1.317(4)	C74-F76	1.326(4)
C74-F77	1.345(4)	O81-C81	1.237(4)	O82-C81	1.225(4)
C81-C82	1.588(8)	C81-C82A	1.602(12)	C82-F81	1.330(12)
C82-F82	1.440(11)	C82-C83	1.47(2)	C83-F84	1.29(2)
C83-C84	1.48(3)	C83-F83	1.61(2)	C84-F87	1.214(13)
C84-F85	1.245(10)	C84-F86	1.456(16)		
N30-Rh1-C11	103.15(10)	N30-Rh1-N40	93.97(9)	C11-Rh1-N40	104.23(10)
N30-Rh1-O60	84.22(8)	C11-Rh1-O60	84.93(9)	N40-Rh1-O60	170.83(8)
N30-Rh1-O50	172.34(8)	C11-Rh1-O50	83.16(9)	N40-Rh1-O50	88.59(8)
O60-Rh1-O50	92.11(7)	N30-Rh1-Rh2	94.34(6)	C11-Rh1-Rh2	155.03(8)
N40-Rh1-Rh2	92.01(7)	O60-Rh1-Rh2	79.19(5)	O50-Rh1-Rh2	78.34(5)
C21-Rh2-N60	99.87(10)	C21-Rh2-N50	101.65(11)	N60-Rh2-N50	91.61(9)
C21-Rh2-O40	86.05(10)	N60-Rh2-O40	173.50(8)	N50-Rh2-O40	89.81(9)
C21-Rh2-O30	84.53(10)	N60-Rh2-O30	89.31(8)	N50-Rh2-O30	173.49(8)
O40-Rh2-O30	88.58(8)	C21-Rh2-Rh1	159.01(8)	N60-Rh2-Rh1	93.05(6)
N50-Rh2-Rh1	94.40(7)	O40-Rh2-Rh1	80.52(5)	O30-Rh2-Rh1	79.12(5)
C16-C11-C12	119.0(3)	C16-C11-Rh1	122.8(2)	C12-C11-Rh1	118.2(2)
C11-C12-C13	119.6(3)	C11-C12-Ag2#2	92.48(18)	C13-C12-Ag2#2	89.27(18)
C11-C12-H12	120.2	C13-C12-H12	120.2	Ag2#2-C12-H12	88.3
C14-C13-C12	120.7(3)	C14-C13-H13	119.7	C12-C13-H13	119.7
C15-C14-C13	119.6(3)	C15-C14-H14	120.2	C13-C14-H14	120.2
C14-C15-C16	120.6(3)	C14-C15-H15	119.7	C16-C15-H15	119.7
C11-C16-C15	120.6(3)	C11-C16-H16	119.7	C15-C16-H16	119.7
C22-C21-C26	119.4(3)	C22-C21-Rh2	118.2(2)	C26-C21-Rh2	122.4(2)

C21-C22-C23	120.1(3)	C21-C22-Ag1	87.65(18)	C23-C22-Ag1	95.90(19)
C21-C22-H22	120.0	C23-C22-H22	120.0	Ag1-C22-H22	86.5
C24-C23-C22	120.1(3)	C24-C23-H23	120.0	C22-C23-H23	120.0
C23-C24-C25	120.0(3)	C23-C24-H24	120.0	C25-C24-H24	120.0
C24-C25-C26	120.6(3)	C24-C25-H25	119.7	C26-C25-H25	119.7
C25-C26-C21	119.8(3)	C25-C26-H26	120.1	C21-C26-H26	120.1
C31-O30-Rh2	127.09(17)	C31-O30-Ag1	115.01(16)	Rh2-O30-Ag1	111.74(8)
O30-C31-N30	123.0(3)	O30-C31-C32	114.1(2)	N30-C31-C32	122.8(3)
C31-C32-C33	110.4(2)	C31-C32-H32A	109.6	C33-C32-H32A	109.6
C31-C32-H32B	109.6	C33-C32-H32B	109.6	H32A-C32-H32B	108.1
C34-C33-C32	114.4(2)	C34-C33-H33A	108.6	C32-C33-H33A	108.6
C34-C33-H33B	108.6	C32-C33-H33B	108.6	H33A-C33-H33B	107.6
C33-C34-C35	115.0(3)	C33-C34-H34A	108.5	C35-C34-H34A	108.5
C33-C34-H34B	108.5	C35-C34-H34B	108.5	H34A-C34-H34B	107.5
C36-C35-C34	115.6(3)	C36-C35-H35A	108.4	C34-C35-H35A	108.4
C36-C35-H35B	108.4	C34-C35-H35B	108.4	H35A-C35-H35B	107.5
C37-C36-C35	118.1(3)	C37-C36-H36A	107.8	C35-C36-H36A	107.8
C37-C36-H36B	107.8	C35-C36-H36B	107.8	H36A-C36-H36B	107.1
N30-C37-C36	113.8(2)	N30-C37-H37A	108.8	C36-C37-H37A	108.8
N30-C37-H37B	108.8	C36-C37-H37B	108.8	H37A-C37-H37B	107.7
C31-N30-C37	121.6(2)	C31-N30-Rh1	114.41(19)	C37-N30-Rh1	123.09(17)
C41-O40-Rh2	125.49(17)	O40-C41-N40	124.0(3)	O40-C41-C42	112.7(3)
N40-C41-C42	123.1(3)	C41-C42-C43	110.1(3)	C41-C42-H42A	109.6
C43-C42-H42A	109.6	C41-C42-H42B	109.6	C43-C42-H42B	109.6
H42A-C42-H42B	108.2	C44-C43-C42	113.7(3)	C44-C43-H43A	108.8
C42-C43-H43A	108.8	C44-C43-H43B	108.8	C42-C43-H43B	108.8
H43A-C43-H43B	107.7	C45-C44-C43	114.7(4)	C45-C44-H44A	108.6
C43-C44-H44A	108.6	C45-C44-H44B	108.6	C43-C44-H44B	108.6
H44A-C44-H44B	107.6	C46-C45-C44	114.8(3)	C46-C45-H45A	108.6
C44-C45-H45A	108.6	C46-C45-H45B	108.6	C44-C45-H45B	108.6
H45A-C45-H45B	107.5	C45-C46-C47	118.9(3)	C45-C46-H46A	107.6
C47-C46-H46A	107.6	C45-C46-H46B	107.6	C47-C46-H46B	107.6
H46A-C46-H46B	107.0	N40-C47-C46	115.2(2)	N40-C47-H47A	108.5
C46-C47-H47A	108.5	N40-C47-H47B	108.5	C46-C47-H47B	108.5
H47A-C47-H47B	107.5	C41-N40-C47	120.4(2)	C41-N40-Rh1	113.75(19)
C47-N40-Rh1	125.83(18)	C51-O50-Rh1	127.08(16)	C51-O50-Ag2#2	118.03(16)
Rh1-O50-Ag2#2	113.88(8)	O50-C51-N50	122.6(3)	O50-C51-C52	114.8(2)
N50-C51-C52	122.6(3)	C51-C52-C53	110.7(5)	C51-C52-H52A	109.5
C53-C52-H52A	109.5	C51-C52-H52B	109.5	C53-C52-H52B	109.5
H52A-C52-H52B	108.1	C52-C53-C54	116.0(4)	C52-C53-H53A	108.3
C54-C53-H53A	108.3	C52-C53-H53B	108.3	C54-C53-H53B	108.3
H53A-C53-H53B	107.4	C55-C54-C53	113.7(6)	C55-C54-H54A	108.8
C53-C54-H54A	108.8	C55-C54-H54B	108.8	C53-C54-H54B	108.8
H54A-C54-H54B	107.7	C56-C55-C54	115.2(4)	C56-C55-H55A	108.5

C54-C55-H55A	108.5	C56-C55-H55B	108.5	C54-C55-H55B	108.5
H55A-C55-H55B	107.5	C55-C56-C57	116.0(4)	C55-C56-H56A	108.3
C57-C56-H56A	108.3	C55-C56-H56B	108.3	C57-C56-H56B	108.3
H56A-C56-H56B	107.4	N50-C57-C56	117.2(3)	N50-C57-H57A	108.0
C56-C57-H57A	108.0	N50-C57-H57B	108.0	C56-C57-H57B	108.0
H57A-C57-H57B	107.3	C51-N50-C57	121.8(2)	C51-N50-Rh2	114.11(19)
C57-N50-Rh2	124.08(18)	C54A-C53A-H53C	110.5	C54A-C53A-H53D	110.5
H53C-C53A-H53D	108.7	C55A-C54A-C53A	112.2(15)	C55A-C54A-H54C	109.2
C53A-C54A-H54C	109.2	C55A-C54A-H54D	109.2	C53A-C54A-H54D	109.2
H54C-C54A-H54D	107.9	C56A-C55A-C54A	116.2(12)	C56A-C55A-H55C	108.2
C54A-C55A-H55C	108.2	C56A-C55A-H55D	108.2	C54A-C55A-H55D	108.2
H55C-C55A-H55D	107.4	C55A-C56A-H56C	106.1	C55A-C56A-H56D	106.1
H56C-C56A-H56D	106.3	C61-O60-Rh1	126.03(17)	O60-C61-N60	123.2(2)
O60-C61-C62	113.4(2)	N60-C61-C62	123.3(2)	C61-C62-C63	114.5(2)
C61-C62-H62A	108.6	C63-C62-H62A	108.6	C61-C62-H62B	108.6
C63-C62-H62B	108.6	H62A-C62-H62B	107.6	C64-C63-C62	115.1(2)
C64-C63-H63A	108.5	C62-C63-H63A	108.5	C64-C63-H63B	108.5
C62-C63-H63B	108.5	H63A-C63-H63B	107.5	C63-C64-C65	114.0(2)
C63-C64-H64A	108.7	C65-C64-H64A	108.7	C63-C64-H64B	108.7
C65-C64-H64B	108.7	H64A-C64-H64B	107.6	C66-C65-C64	115.6(3)
C66-C65-H65A	108.4	C64-C65-H65A	108.4	C66-C65-H65B	108.4
C64-C65-H65B	108.4	H65A-C65-H65B	107.4	C65-C66-C67	116.4(2)
C65-C66-H66A	108.2	C67-C66-H66A	108.2	C65-C66-H66B	108.2
C67-C66-H66B	108.2	H66A-C66-H66B	107.4	N60-C67-C66	113.2(2)
N60-C67-H67A	108.9	C66-C67-H67A	108.9	N60-C67-H67B	108.9
C66-C67-H67B	108.9	H67A-C67-H67B	107.8	C61-N60-C67	119.9(2)
C61-N60-Rh2	113.69(18)	C67-N60-Rh2	126.04(18)	O71-Ag1-O81	149.10(10)
O71-Ag1-O30	99.65(7)	O81-Ag1-O30	95.05(7)	O71-Ag1-C22	127.68(10)
O81-Ag1-C22	82.24(10)	O30-Ag1-C22	75.78(8)	O71-Ag1-Ag2	82.24(6)
O81-Ag1-Ag2	73.83(6)	O30-Ag1-Ag2	156.65(5)	C22-Ag1-Ag2	121.50(6)
O72-Ag2-O82	155.16(9)	O72-Ag2-C12#1	117.49(9)	O82-Ag2-C12#1	86.44(9)
O72-Ag2-O50#1	99.54(7)	O82-Ag2-O50#1	94.54(7)	C12#1-Ag2-O50#1	70.71(7)
O72-Ag2-Ag1	79.59(6)	O82-Ag2-Ag1	79.66(6)	C12#1-Ag2-Ag1	129.60(6)
O50#1-Ag2-Ag1	157.77(4)	C71-O71-Ag1	121.6(2)	C71-O72-Ag2	126.0(2)
O71-C71-O72	130.4(3)	O71-C71-C72	115.5(3)	O72-C71-C72	114.1(3)
F71-C72-F72	106.1(3)	F71-C72-C73	107.5(3)	F72-C72-C73	105.7(3)
F71-C72-C71	111.7(3)	F72-C72-C71	110.3(3)	C73-C72-C71	115.0(3)
F73-C73-F74	106.9(3)	F73-C73-C74	107.7(3)	F74-C73-C74	107.4(3)
F73-C73-C72	108.6(3)	F74-C73-C72	108.7(3)	C74-C73-C72	117.1(3)
F75-C74-F76	108.8(3)	F75-C74-F77	107.5(3)	F76-C74-F77	106.4(3)
F75-C74-C73	112.4(3)	F76-C74-C73	111.3(3)	F77-C74-C73	110.1(3)
C81-O81-Ag1	118.5(2)	C81-O82-Ag2	113.5(2)	O82-C81-O81	130.4(3)
O82-C81-C82	111.2(4)	O81-C81-C82	116.9(4)	O82-C81-C82A	119.4(5)
O81-C81-C82A	106.9(5)	C82-C81-C82A	35.0(5)	F81-C82-F82	111.7(10)

F81-C82-C83	103.6(11)	F82-C82-C83	102.9(11)	F81-C82-C81	115.3(7)
F82-C82-C81	105.5(6)	C83-C82-C81	117.3(11)	F84-C83-C82	113.8(13)
F84-C83-C84	100.(2)	C82-C83-C84	126.3(12)	F84-C83-F83	117.0(12)
C82-C83-F83	100.2(18)	C84-C83-F83	100.1(9)	F87-C84-F85	108.2(12)
F87-C84-F86	107.9(13)	F85-C84-F86	116.1(10)	F87-C84-C83	107.6(15)
F85-C84-C83	117.2(11)	F86-C84-C83	99.1(11)		

Table S16. Torsion angles (°) for **12**.

N30-Rh1-Rh2-C21	29.0(3)	C11-Rh1-Rh2-C21	163.7(3)
N40-Rh1-Rh2-C21	-65.1(3)	O60-Rh1-Rh2-C21	112.3(2)
O50-Rh1-Rh2-C21	-153.2(2)	N30-Rh1-Rh2-N60	-99.14(9)
C11-Rh1-Rh2-N60	35.6(2)	N40-Rh1-Rh2-N60	166.73(9)
O60-Rh1-Rh2-N60	-15.87(8)	O50-Rh1-Rh2-N60	78.58(8)
N30-Rh1-Rh2-N50	169.01(9)	C11-Rh1-Rh2-N50	-56.3(2)
N40-Rh1-Rh2-N50	74.88(9)	O60-Rh1-Rh2-N50	-107.72(8)
O50-Rh1-Rh2-N50	-13.27(8)	N30-Rh1-Rh2-O40	79.94(9)
C11-Rh1-Rh2-O40	-145.4(2)	N40-Rh1-Rh2-O40	-14.20(9)
O60-Rh1-Rh2-O40	163.20(8)	O50-Rh1-Rh2-O40	-102.35(8)
N30-Rh1-Rh2-O30	-10.43(8)	C11-Rh1-Rh2-O30	124.3(2)
N40-Rh1-Rh2-O30	-104.57(9)	O60-Rh1-Rh2-O30	72.83(7)
O50-Rh1-Rh2-O30	167.28(8)	N30-Rh1-C11-C16	-57.0(3)
N40-Rh1-C11-C16	40.6(3)	O60-Rh1-C11-C16	-139.9(3)
O50-Rh1-C11-C16	127.4(3)	Rh2-Rh1-C11-C16	169.68(16)
N30-Rh1-C11-C12	123.1(2)	N40-Rh1-C11-C12	-139.2(2)
O60-Rh1-C11-C12	40.3(2)	O50-Rh1-C11-C12	-52.4(2)
Rh2-Rh1-C11-C12	-10.2(4)	C16-C11-C12-C13	-1.5(4)
Rh1-C11-C12-C13	178.4(2)	C16-C11-C12-Ag2#2	-92.1(3)
Rh1-C11-C12-Ag2#2	87.79(18)	C11-C12-C13-C14	1.4(4)
Ag2#2-C12-C13-C14	93.9(3)	C12-C13-C14-C15	0.0(5)
C13-C14-C15-C16	-1.4(5)	C12-C11-C16-C15	0.1(4)
Rh1-C11-C16-C15	-179.7(2)	C14-C15-C16-C11	1.3(5)
N60-Rh2-C21-C22	26.0(2)	N50-Rh2-C21-C22	119.7(2)
O40-Rh2-C21-C22	-151.3(2)	O30-Rh2-C21-C22	-62.4(2)
Rh1-Rh2-C21-C22	-101.2(3)	N60-Rh2-C21-C26	-152.4(2)
N50-Rh2-C21-C26	-58.7(3)	O40-Rh2-C21-C26	30.3(3)
O30-Rh2-C21-C26	119.3(3)	Rh1-Rh2-C21-C26	80.4(4)
C26-C21-C22-C23	3.1(4)	Rh2-C21-C22-C23	-175.3(2)
C26-C21-C22-Ag1	-92.3(3)	Rh2-C21-C22-Ag1	89.23(18)
C21-C22-C23-C24	-0.3(5)	Ag1-C22-C23-C24	90.4(3)
C22-C23-C24-C25	-1.9(5)	C23-C24-C25-C26	1.3(5)
C24-C25-C26-C21	1.6(5)	C22-C21-C26-C25	-3.7(5)
Rh2-C21-C26-C25	174.6(2)	C21-Rh2-O30-C31	-154.6(2)
N60-Rh2-O30-C31	105.4(2)	N50-Rh2-O30-C31	7.3(8)

O40-Rh2-O30-C31	-68.5(2)	Rh1-Rh2-O30-C31	12.2(2)
C21-Rh2-O30-Ag1	-3.94(11)	N60-Rh2-O30-Ag1	-103.93(9)
N50-Rh2-O30-Ag1	158.0(7)	O40-Rh2-O30-Ag1	82.22(9)
Rh1-Rh2-O30-Ag1	162.84(8)	Rh2-O30-C31-N30	-6.0(4)
Ag1-O30-C31-N30	-155.9(2)	Rh2-O30-C31-C32	178.24(17)
Ag1-O30-C31-C32	28.4(3)	O30-C31-C32-C33	80.2(3)
N30-C31-C32-C33	-95.5(3)	C31-C32-C33-C34	51.2(3)
C32-C33-C34-C35	54.6(4)	C33-C34-C35-C36	-97.8(3)
C34-C35-C36-C37	67.0(4)	C35-C36-C37-N30	-75.2(3)
O30-C31-N30-C37	-176.3(2)	C32-C31-N30-C37	-1.0(4)
O30-C31-N30-Rh1	-7.1(3)	C32-C31-N30-Rh1	168.3(2)
C36-C37-N30-C31	84.6(3)	C36-C37-N30-Rh1	-83.7(3)
C11-Rh1-N30-C31	-149.3(2)	N40-Rh1-N30-C31	105.1(2)
O60-Rh1-N30-C31	-65.89(19)	O50-Rh1-N30-C31	-4.2(7)
Rh2-Rh1-N30-C31	12.77(19)	C11-Rh1-N30-C37	19.8(2)
N40-Rh1-N30-C37	-85.8(2)	O60-Rh1-N30-C37	103.2(2)
O50-Rh1-N30-C37	164.8(5)	Rh2-Rh1-N30-C37	-178.2(2)
C21-Rh2-O40-C41	-175.7(3)	N60-Rh2-O40-C41	28.6(9)
N50-Rh2-O40-C41	-74.1(2)	O30-Rh2-O40-C41	99.6(2)
Rh1-Rh2-O40-C41	20.4(2)	Rh2-O40-C41-N40	-16.3(4)
Rh2-O40-C41-C42	168.0(2)	O40-C41-C42-C43	83.8(4)
N40-C41-C42-C43	-92.0(4)	C41-C42-C43-C44	55.4(4)
C42-C43-C44-C45	52.7(4)	C43-C44-C45-C46	-99.1(4)
C44-C45-C46-C47	65.3(5)	C45-C46-C47-N40	-71.6(4)
O40-C41-N40-C47	176.1(3)	C42-C41-N40-C47	-8.6(5)
O40-C41-N40-Rh1	-2.4(4)	C42-C41-N40-Rh1	172.9(3)
C46-C47-N40-C41	87.0(4)	C46-C47-N40-Rh1	-94.7(3)
N30-Rh1-N40-C41	-80.8(2)	C11-Rh1-N40-C41	174.5(2)
O60-Rh1-N40-C41	-2.6(7)	O50-Rh1-N40-C41	91.9(2)
Rh2-Rh1-N40-C41	13.6(2)	N30-Rh1-N40-C47	100.8(2)
C11-Rh1-N40-C47	-3.9(3)	O60-Rh1-N40-C47	179.1(9)
O50-Rh1-N40-C47	-86.5(2)	Rh2-Rh1-N40-C47	-164.8(2)
N30-Rh1-O50-C51	34.9(7)	C11-Rh1-O50-C51	-179.3(2)
N40-Rh1-O50-C51	-74.8(2)	O60-Rh1-O50-C51	96.0(2)
Rh2-Rh1-O50-C51	17.5(2)	N30-Rh1-O50-Ag2#2	-156.9(6)
C11-Rh1-O50-Ag2#2	-11.10(10)	N40-Rh1-O50-Ag2#2	93.41(9)
O60-Rh1-O50-Ag2#2	-95.73(8)	Rh2-Rh1-O50-Ag2#2	-174.24(8)
Rh1-O50-C51-N50	-11.8(4)	Ag2#2-O50-C51-N50	-179.63(19)
Rh1-O50-C51-C52	168.12(18)	Ag2#2-O50-C51-C52	0.3(3)
O50-C51-C52-C53	-83.1(4)	N50-C51-C52-C53	96.8(4)
C51-C52-C53-C54	-50.7(10)	C52-C53-C54-C55	-53.9(10)
C53-C54-C55-C56	99.1(6)	C54-C55-C56-C57	-70.7(6)
C55-C56-C57-N50	77.3(5)	O50-C51-N50-C57	174.1(2)
C52-C51-N50-C57	-5.9(4)	O50-C51-N50-Rh2	-5.4(3)

C52-C51-N50-Rh2	174.7(2)	C56-C57-N50-C51	-80.5(4)
C56-C57-N50-Rh2	98.8(3)	C21-Rh2-N50-C51	-179.0(2)
N60-Rh2-N50-C51	-78.6(2)	O40-Rh2-N50-C51	95.05(19)
O30-Rh2-N50-C51	19.4(8)	Rh1-Rh2-N50-C51	14.57(19)
C21-Rh2-N50-C57	1.6(2)	N60-Rh2-N50-C57	102.0(2)
O40-Rh2-N50-C57	-84.4(2)	O30-Rh2-N50-C57	-160.0(6)
Rh1-Rh2-N50-C57	-164.8(2)	C53A-C54A-C55A-C56A	-99.9(15)
N30-Rh1-O60-C61	114.7(2)	C11-Rh1-O60-C61	-141.5(2)
N40-Rh1-O60-C61	35.7(6)	O50-Rh1-O60-C61	-58.5(2)
Rh2-Rh1-O60-C61	19.19(19)	Rh1-O60-C61-N60	-10.7(4)
Rh1-O60-C61-C62	171.76(16)	O60-C61-C62-C63	-85.6(3)
N60-C61-C62-C63	96.8(3)	C61-C62-C63-C64	-72.4(3)
C62-C63-C64-C65	65.1(3)	C63-C64-C65-C66	-100.2(3)
C64-C65-C66-C67	57.4(3)	C65-C66-C67-N60	52.1(3)
O60-C61-N60-C67	163.8(2)	C62-C61-N60-C67	-18.9(4)
O60-C61-N60-Rh2	-9.3(3)	C62-C61-N60-Rh2	168.0(2)
C66-C67-N60-C61	-80.7(3)	C66-C67-N60-Rh2	91.5(3)
C21-Rh2-N60-C61	-144.82(19)	N50-Rh2-N60-C61	113.07(19)
O40-Rh2-N60-C61	10.5(9)	O30-Rh2-N60-C61	-60.49(19)
Rh1-Rh2-N60-C61	18.58(18)	C21-Rh2-N60-C67	42.5(2)
N50-Rh2-N60-C67	-59.6(2)	O40-Rh2-N60-C67	-162.1(7)
O30-Rh2-N60-C67	126.9(2)	Rh1-Rh2-N60-C67	-154.1(2)
C31-O30-Ag1-O71	58.7(2)	Rh2-O30-Ag1-O71	-95.80(10)
C31-O30-Ag1-O81	-94.1(2)	Rh2-O30-Ag1-O81	111.48(10)
C31-O30-Ag1-C22	-174.7(2)	Rh2-O30-Ag1-C22	30.82(10)
C31-O30-Ag1-Ag2	-34.1(3)	Rh2-O30-Ag1-Ag2	171.44(4)
C21-C22-Ag1-O71	34.3(2)	C23-C22-Ag1-O71	-85.7(2)
C21-C22-Ag1-O81	-154.27(18)	C23-C22-Ag1-O81	85.7(2)
C21-C22-Ag1-O30	-57.02(16)	C23-C22-Ag1-O30	-177.0(2)
C21-C22-Ag1-Ag2	140.13(14)	C23-C22-Ag1-Ag2	20.2(2)
O71-Ag1-Ag2-O72	2.13(10)	O81-Ag1-Ag2-O72	162.38(10)
O30-Ag1-Ag2-O72	98.51(14)	C22-Ag1-Ag2-O72	-127.66(11)
O71-Ag1-Ag2-O82	168.39(10)	O81-Ag1-Ag2-O82	-31.36(9)
O30-Ag1-Ag2-O82	-95.22(13)	C22-Ag1-Ag2-O82	38.60(10)
O71-Ag1-Ag2-C12#1	-115.07(11)	O81-Ag1-Ag2-C12#1	45.18(11)
O30-Ag1-Ag2-C12#1	-18.68(15)	C22-Ag1-Ag2-C12#1	115.14(12)
O71-Ag1-Ag2-O50#1	91.89(14)	O81-Ag1-Ag2-O50#1	-107.86(14)
O30-Ag1-Ag2-O50#1	-171.72(16)	C22-Ag1-Ag2-O50#1	-37.90(14)
O81-Ag1-O71-C71	-43.0(4)	O30-Ag1-O71-C71	-160.3(3)
C22-Ag1-O71-C71	120.3(3)	Ag2-Ag1-O71-C71	-3.8(3)
O82-Ag2-O72-C71	-35.5(4)	C12#1-Ag2-O72-C71	127.7(3)
O50#1-Ag2-O72-C71	-159.2(3)	Ag1-Ag2-O72-C71	-1.7(3)
Ag1-O71-C71-O72	4.2(5)	Ag1-O71-C71-C72	-174.3(2)
Ag2-O72-C71-O71	-0.9(5)	Ag2-O72-C71-C72	177.6(2)

O71-C71-C72-F71	-23.6(4)	O72-C71-C72-F71	157.7(3)
O71-C71-C72-F72	-141.3(3)	O72-C71-C72-F72	40.0(4)
O71-C71-C72-C73	99.3(4)	O72-C71-C72-C73	-79.4(4)
F71-C72-C73-F73	-52.5(4)	F72-C72-C73-F73	60.4(3)
C71-C72-C73-F73	-177.6(3)	F71-C72-C73-F74	-168.5(3)
F72-C72-C73-F74	-55.6(3)	C71-C72-C73-F74	66.4(4)
F71-C72-C73-C74	69.6(4)	F72-C72-C73-C74	-177.5(3)
C71-C72-C73-C74	-55.5(4)	F73-C73-C74-F75	65.1(4)
F74-C73-C74-F75	180.0(3)	C72-C73-C74-F75	-57.4(4)
F73-C73-C74-F76	-172.5(3)	F74-C73-C74-F76	-57.7(4)
C72-C73-C74-F76	64.9(4)	F73-C73-C74-F77	-54.7(4)
F74-C73-C74-F77	60.1(4)	C72-C73-C74-F77	-177.3(3)
O71-Ag1-O81-C81	83.7(3)	O30-Ag1-O81-C81	-157.9(3)
C22-Ag1-O81-C81	-83.0(3)	Ag2-Ag1-O81-C81	43.0(3)
O72-Ag2-O82-C81	72.1(4)	C12#1-Ag2-O82-C81	-93.0(3)
O50#1-Ag2-O82-C81	-163.3(3)	Ag1-Ag2-O82-C81	38.3(3)
Ag2-O82-C81-O81	-17.6(6)	Ag2-O82-C81-C82	177.0(4)
Ag2-O82-C81-C82A	139.1(6)	Ag1-O81-C81-O82	-27.8(6)
Ag1-O81-C81-C82	136.9(4)	Ag1-O81-C81-C82A	173.3(5)
O82-C81-C82-F81	65.4(10)	O81-C81-C82-F81	-102.2(9)
C82A-C81-C82-F81	176.7(14)	O82-C81-C82-F82	-58.3(9)
O81-C81-C82-F82	134.1(8)	C82A-C81-C82-F82	53.0(11)
O82-C81-C82-C83	-172.1(9)	O81-C81-C82-C83	20.4(12)
C82A-C81-C82-C83	-60.8(11)	F81-C82-C83-F84	54.(2)
F82-C82-C83-F84	170.2(17)	C81-C82-C83-F84	-74.6(19)
F81-C82-C83-C84	177.4(18)	F82-C82-C83-C84	-66.(2)
C81-C82-C83-C84	49.(2)	F81-C82-C83-F83	-71.9(11)
F82-C82-C83-F83	44.6(11)	C81-C82-C83-F83	159.8(7)
F84-C83-C84-F87	27.1(15)	C82-C83-C84-F87	-102.(2)
F83-C83-C84-F87	147.0(13)	F84-C83-C84-F85	-95.0(16)
C82-C83-C84-F85	135.6(17)	F83-C83-C84-F85	24.9(18)
F84-C83-C84-F86	139.3(11)	C82-C83-C84-F86	10.(2)
F83-C83-C84-F86	-100.9(11)		

Table S17. Anisotropic atomic displacement parameters* ($\text{\AA}^2$) for **12**.

Atom	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
Rh1	0.01328(10)	0.01898(10)	0.01775(11)	-0.00177(8)	-0.00307(8)	-0.00642(8)
Rh2	0.01458(10)	0.01984(11)	0.01853(12)	-0.00068(8)	-0.00262(8)	-0.00764(8)
C11	0.0154(13)	0.0279(15)	0.0225(15)	0.0051(12)	-0.0071(11)	-0.0096(12)
C12	0.0177(14)	0.0258(15)	0.0238(16)	0.0034(12)	-0.0075(12)	-0.0074(12)

C13	0.0277(16)	0.0307(16)	0.0370(19)	0.0123(14)	-0.0143(14)	-0.0170(14)
C14	0.0204(15)	0.0441(19)	0.0371(19)	0.0113(15)	-0.0053(14)	-0.0155(14)
C15	0.0169(15)	0.0386(18)	0.0288(18)	-0.0009(14)	0.0024(13)	-0.0044(13)
C16	0.0203(15)	0.0291(16)	0.0272(17)	-0.0018(13)	-0.0029(12)	-0.0079(12)
C21	0.0193(14)	0.0289(15)	0.0204(15)	-0.0017(12)	-0.0027(11)	-0.0124(12)
C22	0.0211(14)	0.0290(15)	0.0225(16)	-0.0036(12)	-0.0030(12)	-0.0104(12)
C23	0.0179(15)	0.0395(18)	0.0353(19)	-0.0053(15)	-0.0005(13)	-0.0083(14)
C24	0.0267(17)	0.053(2)	0.0306(19)	0.0009(16)	0.0048(14)	-0.0226(16)
C25	0.0345(18)	0.0425(19)	0.0320(19)	0.0099(15)	-0.0067(15)	-0.0230(16)
C26	0.0237(15)	0.0324(17)	0.0316(18)	0.0053(14)	-0.0059(13)	-0.0129(13)
O30	0.0182(10)	0.0318(11)	0.0203(11)	-0.0005(8)	-0.0050(8)	-0.0135(9)
C31	0.0234(15)	0.0224(14)	0.0217(15)	-0.0009(12)	-0.0059(12)	-0.0122(12)
C32	0.0248(16)	0.0455(19)	0.0208(16)	-0.0007(14)	-0.0078(13)	-0.0195(14)
C33	0.0207(15)	0.0443(19)	0.0272(17)	0.0025(14)	-0.0098(13)	-0.0069(14)
C34	0.0268(16)	0.0322(17)	0.0266(17)	0.0022(13)	-0.0109(13)	-0.0065(13)
C35	0.0278(16)	0.0354(17)	0.0252(17)	0.0051(13)	-0.0090(13)	-0.0113(14)
C36	0.0250(16)	0.0349(17)	0.0230(16)	0.0024(13)	-0.0029(13)	-0.0092(13)
C37	0.0203(14)	0.0299(16)	0.0204(15)	-0.0060(12)	-0.0046(12)	-0.0058(12)
N30	0.0186(12)	0.0235(12)	0.0179(12)	-0.0032(10)	-0.0038(9)	-0.0090(10)
O40	0.0217(11)	0.0230(10)	0.0342(12)	-0.0027(9)	0.0000(9)	-0.0101(9)
C41	0.0228(15)	0.0234(15)	0.0322(17)	-0.0022(13)	-0.0075(13)	-0.0097(12)
C42	0.0313(18)	0.0233(16)	0.062(3)	-0.0014(16)	0.0067(17)	-0.0094(14)
C43	0.044(2)	0.036(2)	0.101(4)	-0.021(2)	0.003(2)	-0.0245(19)
C44	0.048(2)	0.049(2)	0.084(3)	-0.029(2)	-0.019(2)	-0.021(2)
C45	0.048(2)	0.039(2)	0.057(3)	-0.0243(19)	-0.0029(19)	-0.0152(18)
C46	0.0284(17)	0.0270(16)	0.041(2)	-0.0100(14)	-0.0020(15)	-0.0033(14)
C47	0.0178(14)	0.0219(15)	0.0387(19)	-0.0007(13)	-0.0063(13)	-0.0036(12)
N40	0.0193(12)	0.0200(12)	0.0268(14)	-0.0024(10)	-0.0064(10)	-0.0064(10)
O50	0.0190(10)	0.0258(10)	0.0201(11)	0.0005(8)	-0.0068(8)	-0.0104(8)
C51	0.0203(14)	0.0164(13)	0.0222(15)	-0.0012(11)	-0.0068(12)	-0.0059(11)
C52	0.0267(16)	0.0366(17)	0.0256(17)	0.0003(13)	-0.0100(13)	-0.0168(14)
C53	0.0252(19)	0.055(2)	0.037(2)	0.017(2)	-0.017(2)	-0.0161(17)
C54	0.038(2)	0.025(2)	0.041(3)	0.008(2)	-0.014(2)	-0.0018(18)
C55	0.043(2)	0.031(2)	0.045(3)	0.020(2)	-0.020(2)	-0.0164(19)
C56	0.036(2)	0.050(2)	0.034(4)	0.028(2)	-0.014(2)	-0.0250(18)
C57	0.0295(17)	0.0410(18)	0.0246(17)	0.0097(14)	-0.0099(13)	-0.0175(14)
N50	0.0199(12)	0.0203(12)	0.0221(13)	0.0028(10)	-0.0069(10)	-0.0072(10)
C52A	0.0267(16)	0.0366(17)	0.0256(17)	0.0003(13)	-0.0100(13)	-0.0168(14)
C53A	0.0252(19)	0.055(2)	0.037(2)	0.017(2)	-0.017(2)	-0.0161(17)
C54A	0.038(2)	0.025(2)	0.041(3)	0.008(2)	-0.014(2)	-0.0018(18)
C55A	0.043(2)	0.031(2)	0.045(3)	0.020(2)	-0.020(2)	-0.0164(19)
C56A	0.036(2)	0.050(2)	0.034(4)	0.028(2)	-0.014(2)	-0.0250(18)
C57A	0.0295(17)	0.0410(18)	0.0246(17)	0.0097(14)	-0.0099(13)	-0.0175(14)
O60	0.0160(9)	0.0211(10)	0.0179(10)	0.0000(8)	-0.0043(8)	-0.0061(8)

C61	0.0152(13)	0.0212(13)	0.0200(14)	-0.0023(11)	-0.0068(11)	-0.0058(11)
C62	0.0244(15)	0.0213(14)	0.0193(15)	0.0014(11)	-0.0074(12)	-0.0043(12)
C63	0.0334(17)	0.0195(14)	0.0268(17)	0.0010(12)	-0.0056(13)	-0.0099(13)
C64	0.0329(17)	0.0269(16)	0.0295(17)	-0.0014(13)	-0.0091(14)	-0.0127(14)
C65	0.0292(16)	0.0289(16)	0.0244(16)	-0.0001(13)	-0.0126(13)	-0.0123(13)
C66	0.0312(16)	0.0247(15)	0.0194(15)	-0.0015(12)	-0.0077(13)	-0.0102(13)
C67	0.0205(14)	0.0238(14)	0.0182(15)	-0.0038(11)	-0.0005(11)	-0.0058(12)
N60	0.0157(11)	0.0217(12)	0.0180(12)	-0.0018(9)	-0.0043(9)	-0.0070(9)
Ag1	0.02219(12)	0.03885(14)	0.03567(14)	-0.00352(11)	-0.00518(10)	-0.01586(10)
Ag2	0.02169(12)	0.03072(12)	0.03870(15)	0.00587(10)	-0.01248(10)	-0.00991(10)
O71	0.0279(13)	0.0393(14)	0.0713(19)	-0.0060(13)	-0.0172(12)	-0.0126(11)
O72	0.0310(13)	0.0322(13)	0.0637(18)	-0.0009(12)	-0.0217(12)	-0.0098(10)
C71	0.0257(16)	0.0303(16)	0.0249(16)	0.0016(13)	-0.0051(13)	-0.0095(13)
C72	0.0267(17)	0.0357(18)	0.042(2)	-0.0034(15)	-0.0084(15)	-0.0077(14)
F71	0.0335(11)	0.0494(13)	0.0558(14)	-0.0118(11)	0.0039(10)	-0.0055(10)
F72	0.0582(14)	0.0509(13)	0.0500(14)	-0.0129(11)	-0.0196(11)	-0.0173(11)
C73	0.0293(17)	0.0249(16)	0.057(2)	0.0013(15)	-0.0083(16)	-0.0026(14)
F73	0.0616(15)	0.0280(11)	0.0860(19)	-0.0024(11)	-0.0220(13)	-0.0067(10)
F74	0.0369(12)	0.0478(13)	0.0724(16)	0.0106(11)	-0.0132(11)	-0.0186(10)
C74	0.044(2)	0.040(2)	0.054(2)	0.0088(18)	-0.0149(19)	-0.0102(17)
F75	0.0447(14)	0.0838(18)	0.0721(18)	0.0106(14)	-0.0266(13)	-0.0187(13)
F76	0.0725(17)	0.0531(14)	0.0498(15)	-0.0035(11)	-0.0216(13)	-0.0076(12)
F77	0.087(2)	0.0736(18)	0.0716(18)	0.0370(15)	-0.0273(15)	-0.0307(15)
O81	0.0291(13)	0.0460(15)	0.0640(18)	0.0151(13)	-0.0061(12)	-0.0149(12)
O82	0.0305(13)	0.0411(14)	0.0449(15)	-0.0056(11)	-0.0049(11)	-0.0164(11)
C81	0.034(2)	0.0326(18)	0.061(3)	0.0030(17)	-0.0127(18)	-0.0110(15)
C82	0.052(4)	0.048(4)	0.053(5)	-0.004(3)	0.006(3)	-0.021(3)
F81	0.118(9)	0.056(5)	0.049(4)	-0.005(3)	-0.002(5)	-0.041(5)
F82	0.061(4)	0.053(3)	0.124(9)	-0.006(4)	-0.019(5)	0.004(2)
C83	0.085(7)	0.064(5)	0.079(6)	0.000(4)	-0.012(6)	-0.043(5)
F83	0.162(5)	0.067(3)	0.150(4)	0.011(2)	0.026(3)	-0.032(3)
F84	0.107(6)	0.115(8)	0.138(9)	0.008(6)	-0.057(7)	-0.057(5)
C84	0.112(6)	0.066(5)	0.095(6)	0.021(4)	-0.018(4)	-0.024(4)
F85	0.138(5)	0.072(4)	0.114(6)	0.019(4)	0.009(5)	-0.058(3)
F86	0.139(10)	0.127(6)	0.102(6)	0.028(4)	-0.076(7)	-0.049(6)
F87	0.102(6)	0.110(7)	0.111(9)	0.014(6)	-0.004(5)	-0.041(5)

* The anisotropic atomic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U_{11} + \dots + 2hka^*b^*U_{12}]$

Table S18. Hydrogen atom coordinates and isotropic atomic displacement parameters ($\text{\AA}^2$) for **12**.

Atom	x/a	y/b	z/c	U_{iso}
H12	0.6785	0.9204	0.6880	0.027
H13	0.8525	0.9574	0.6156	0.035
H14	0.9969	0.8547	0.5009	0.04
H15	0.9687	0.7145	0.4575	0.038
H16	0.8014	0.6726	0.5314	0.032
H22	0.1211	0.9092	0.8531	0.029
H23	-0.0672	0.9057	0.9394	0.04
H24	-0.0678	0.7664	1.0097	0.044
H25	0.1163	0.6266	0.9898	0.041
H26	0.3039	0.6273	0.9024	0.035
H32A	0.2100	0.7746	0.6032	0.034
H32B	0.3254	0.7587	0.5235	0.034
H33A	0.1775	0.9175	0.5244	0.038
H33B	0.1653	0.9451	0.6174	0.038
H34A	0.3605	0.9571	0.5813	0.035
H34B	0.2794	1.0265	0.5271	0.035
H35A	0.3959	0.8666	0.4271	0.035
H35B	0.4461	0.9556	0.4245	0.035
H36A	0.6132	0.8113	0.4182	0.035
H36B	0.5776	0.8681	0.5060	0.035
H37A	0.5038	0.7051	0.4858	0.03
H37B	0.6350	0.6880	0.5020	0.03
H42A	0.5520	0.4535	0.7993	0.052
H42B	0.6846	0.4275	0.7321	0.052
H43A	0.5677	0.3523	0.6843	0.075
H43B	0.4529	0.4568	0.6973	0.075
H44A	0.5686	0.4117	0.5567	0.07
H44B	0.5618	0.5178	0.5883	0.07
H45A	0.7756	0.3565	0.5969	0.061
H45B	0.7681	0.3791	0.5060	0.061
H46A	0.7550	0.5435	0.5323	0.043
H46B	0.8835	0.4613	0.5378	0.043
H47A	0.8026	0.4883	0.6847	0.033
H47B	0.8311	0.5791	0.6404	0.033
H52A	0.6317	0.7840	0.9693	0.033
H52B	0.7268	0.8094	0.8941	0.033
H53A	0.8251	0.6542	0.9472	0.045
H53B	0.8412	0.6418	0.8515	0.045
H54A	0.6939	0.5661	0.8736	0.044
H54B	0.7961	0.5082	0.9194	0.044
H55A	0.6358	0.5249	1.0245	0.045

H55B	0.6315	0.6355	1.0384	0.045
H56A	0.4328	0.6256	1.0440	0.044
H56B	0.4854	0.5903	0.9486	0.044
H57A	0.3515	0.7662	0.9878	0.036
H57B	0.4675	0.7798	1.0081	0.036
H52C	0.6299	0.7813	0.9691	0.033
H52D	0.7190	0.8147	0.8965	0.033
H53C	0.8569	0.6746	0.9285	0.045
H53D	0.8293	0.6302	0.8530	0.045
H54C	0.8016	0.5381	0.9752	0.044
H54D	0.6864	0.6366	1.0209	0.044
H55C	0.6685	0.5697	0.8730	0.045
H55D	0.6614	0.4951	0.9427	0.045
H56C	0.4826	0.6080	1.0248	0.044
H56D	0.4634	0.5993	0.9372	0.044
H57C	0.3517	0.7632	0.9904	0.036
H57D	0.4712	0.7736	1.0102	0.036
H62A	0.2633	1.1129	0.7770	0.027
H62B	0.3510	1.0891	0.6849	0.027
H63A	0.5204	1.0903	0.7372	0.033
H63B	0.4115	1.1968	0.7432	0.033
H64A	0.3268	1.1826	0.8846	0.035
H64B	0.4643	1.1846	0.8646	0.035
H65A	0.5184	0.9926	0.8528	0.031
H65B	0.5055	1.0536	0.9342	0.031
H66A	0.3021	1.0608	0.9905	0.03
H66B	0.3946	0.9473	0.9653	0.03
H67A	0.2152	0.9664	0.9300	0.027
H67B	0.2198	1.0621	0.8823	0.027

Table S19. Data collection details for **12**.

Axis	dx/mm	2 θ /°	ω /°	ϕ /°	χ /°	Width/°	Frames	Time/s
Omega	50.328	-31.50	-31.50	60.00	54.71	0.30	610	30.00
Omega	50.328	-31.50	-31.50	180.00	54.71	0.30	610	30.00
Omega	50.328	-31.50	-31.50	300.00	54.71	0.30	610	30.00
Phi	50.328	-31.50	-211.50	0.00	54.71	0.30	524	30.00