

Anion Directed Synthesis of a Paddalane and Trisilver Tweezer Complex Based Upon Silver Coordination Chemistry

Paul D. Custer, Jered C. Garrison, Claire A. Tessier and Wiley J. Youngs

Department of Chemistry, The University of Akron, Akron, Ohio 44325, USA.

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General Considerations

All manipulations dealing with lithium reagents were carried out under an N₂ atmosphere. Phenylacetylene, AgBF₄ and Ag(O₃SCF₃) were purchased from Aldrich and used without further purification. 1,4,7-trimethyl-1,4,7-triazacyclononane (^{Me}3tacn) was synthesized according to literature procedures.¹⁻³ Dry THF was obtained from a PureSolv™ solvent purification system. ¹H and ¹³C NMR data were recorded on Varian's Gemini 300 MHz and Inova 400 MHz instruments. The spectra were referenced to the residual proton and ¹³C signals of the deuterated solvents. ¹⁹F NMR data was recorded on a Varian Gemini 300 MHz instrument and referenced to trifluoroacetic acid. Mass spectrometry data was collected on a Bruker Daltons (Billerica, MA) Esquire-LC mass spectrometer equipped with ESI. Infrared Spectra were collected as KBr films using a Bomem B-100 Infrared spectrometer.

X-ray Structure Determination Details

Crystals of **2**, **3** and **4** were coated in paratone oil and mounted on a CryoLoop™ and placed on the goniometer head under a stream of nitrogen cooled to 100K. The data was collected on a Bruker APEX CCD diffractometer with graphite-monochromated Mo K_α radiation ($\lambda = 0.71073$ Å). The unit cell was determined by using reflections from three different orientations. The data was integrated using SAINT.⁴ An empirical absorption correction and other corrections were applied to the data using multi-scan SADABS.⁴ Structure solution, refinement, and modeling were accomplished by using the Bruker SHELXTL package.^{4,5} The structure was determined by full-matrix least-squares refinement of F^2 and the selection of the appropriate atoms from the generated difference map. Hydrogen atom positions were calculated and U_{iso}(H) values were fixed according to a riding model.

¹ McAuley, A.; Norman, P. R.; Olubuyide, O. *Inorg. Chem.* **1984**, 1938-1943.

² Hay, R. W.; Norman, P. R. *J. Chem. Soc., Dalton* **1979** 1441-1445.

³ Wang, L.; Wang, C.; Bau, R.; Flood, T. C. *Organometallics* **1996**, 15, 491-498.

⁴ Bruker (1997). SMART (Version 5.625), SAINT (Version 6.22) and SHELXTL (Version 6.10)

⁵ Sheldrick, G. M. (1997). SHELX-97. University of Göttingen, Germany

(^{Me3}tacnRh(CCPPh)₃ (2). A 500 mL schlenk flask was charged with a stir bar, 1.978 g of ^{Me3}tacnRhCl₃ (5.20 mmol) and 100 mL of dry THF. A separate 100 mL schlenk flask was charged with 2.28 mL of phenylacetylene (20.75 mmol) and 50 mL dry THF. The flask containing the phenylacetylene was cooled to -78 °C, 13.0 mL of 2.0 M BuLi was added and stirred for 30 min. The mixture was transferred via canula to the flask, cooled to -78 °C, containing the ^{Me3}tacnRhCl₃. The reaction mixture was allowed to stir at RT for three days. The flask was opened to air and a solution of wet THF was added drop wise to quench any remaining lithium reagents. All volatiles were removed under vacuum. CH₂Cl₂ (150 mL) was added to the solid and the mixture was stirred for 30 min. The mixture was filtered to remove the LiCl. Evaporation of solvent yielded a brown solid which was washed with hexanes and dissolved in 150 mL of CH₂Cl₂. The solution was extracted with 5 x 200 mL of water. The organic phase was dried using MgSO₄ and filtered. The solvent was removed under vacuum to afford **2** as a light brown solid. Purification of the compound was achieved by dissolving **2** into a minimal amount of CH₂Cl₂ (~50 mL) and the addition of 250 mL of cold diethyl ether. The resulting white precipitate, **2**, was filtered. Yield: 1.958 g (3.39 mmol), 65%; Mp > 250 °C; ¹H NMR (300 MHz, d₆-DMSO) δ 2.92-3.28 (m, 12H, CH₂), 3.05 (s, 9H, CH₃), 7.02 - 7.26 (m, 15H, aromatic); ¹³C NMR (75 MHz, d₆-DMSO) δ = 51.26 (s, CH₃), 58.61 (s, CH₂), 98.52 (d, C_β, *J* = 9.4 Hz), 111.03 (d, C_α, *J* = 48.5 Hz), 124.28, 127.98, 129.27, 130.63 (aromatic carbons). IR (KBr): ν(CC) 2103 (s) cm⁻¹; ES-MS (8:2-DMSO:CH₃CN) calcd for [M]Na⁺: *m/z* 600.2, found 600.3; Anal. Calcd for C₃₃H₃₆N₃Rh: C, 68.63; H, 6.28; N, 7.28. Found: C, 68.62; H, 6.13; N, 7.22.

(^{Me3}tacnRh(CCPPh)₃AgBF₄)₂ (3). In a 100 mL round bottom flask was placed 0.060 g of **2** (0.104 mmol) and 60 mL of CH₃CN. AgBF₄ (0.146 g, 0.750 mmol) was added to the solution of **2** and stirred for 1 min. All volatiles were removed under reduced pressure. The resulting brown solid was washed with water and filtered. The solid was dissolved in 6 mL of CH₃CN and filtered. The resulting solution was allowed to slowly evaporate, in the dark, to give 0.0803 g of **3** as brown crystals. Analysis of **3** by x-ray diffraction studies reveals a non-coordinating acetonitrile molecule in the asymmetric unit. The weight of the acetonitrile was taken into account when calculating the yield. Yield: 0.078 g (0.101 mmol), 97%; Mp > 220 °C; ¹H NMR (300 MHz, d₆-DMSO) δ 3.10 (s, 18H, CH₃), 3.29 (s, 24H, CH₂), 7.06 (t, 12H, *J* = 6.9 Hz), 7.14 (t, 6H, *J* = 6.9 Hz), 7.32 (d, 12H, *J* = 6.9 Hz); ¹³C NMR (75 MHz, d₆-DMSO) δ 52.04 (s, CH₃), 59.87 (s, CH₂), 94.12 (d, C_α, *J* = 44.6 Hz), 103.95 (m, C_β), 124.10, 127.32, 127.94, 131.16 (aromatic carbons); IR (KBr): ν(CC) 2047 cm⁻¹; ES-MS (acetone) calcd for [M+BF₄]⁺ and [M]²⁺: *m/z* 1457.2 [M+BF₄]⁺ and 685.1 [M]²⁺, found 1457.2 and 686.1; Anal. Calcd for C₃₃H₃₆N₃RhAgBF₄: C, 51.33; H, 4.70; N, 5.44. Found: C, 50.57; H, 4.56; N, 5.63.

^{Me3}tacnRh(CCPh)₃ (Ag(O₃SCF₃))₃CH₃CN (4). A 100 mL schlenk flask was charged with 0.176 g of **2** (0.304 mmol) and a stir bar. Ag(O₃SCF₃) (0.318 g, 1.24 mmol) was added, under a nitrogen atmosphere, with the reaction flask shielded from light. The flask was opened to the atmosphere and 25 mL of acetonitrile was added to the flask. The solution was allowed to stir at RT for 2 h. The solution was concentrated to ~2 mL. The solution was allowed to slowly evaporate, in the dark, to yield brown crystals. The crystals were rinsed with cold water to remove impurities. Yield 0.373 g (0.268 mmol), 88%; ¹H NMR (300 MHz, d₆-DMSO) δ 3.10 (s, 9H, CH₃), 3.29 (s, 12H, CH₂), 7.06 (t, 6H, *J* = 7.2 Hz), 7.14 (t, 3H, *J* = 6.9 Hz), 7.32 (d, 6H, *J* = 7.2 Hz); ¹³C NMR (100 MHz, d₆-DMSO) δ 52.00 (s, CH₃), 59.83 (s, CH₂), 94.04 (d, C_α, *J* = 44.4 Hz), 103.93 (m, C_β), 124.05, 127.26, 127.87, 131.10 (aromatic carbons); ¹⁹F NMR (300 MHz, DMSO-d₆) δ 0.52 (s, CF₃).

File # 2 : CNRHCCPH

Mode = 2 (Mid-IR)

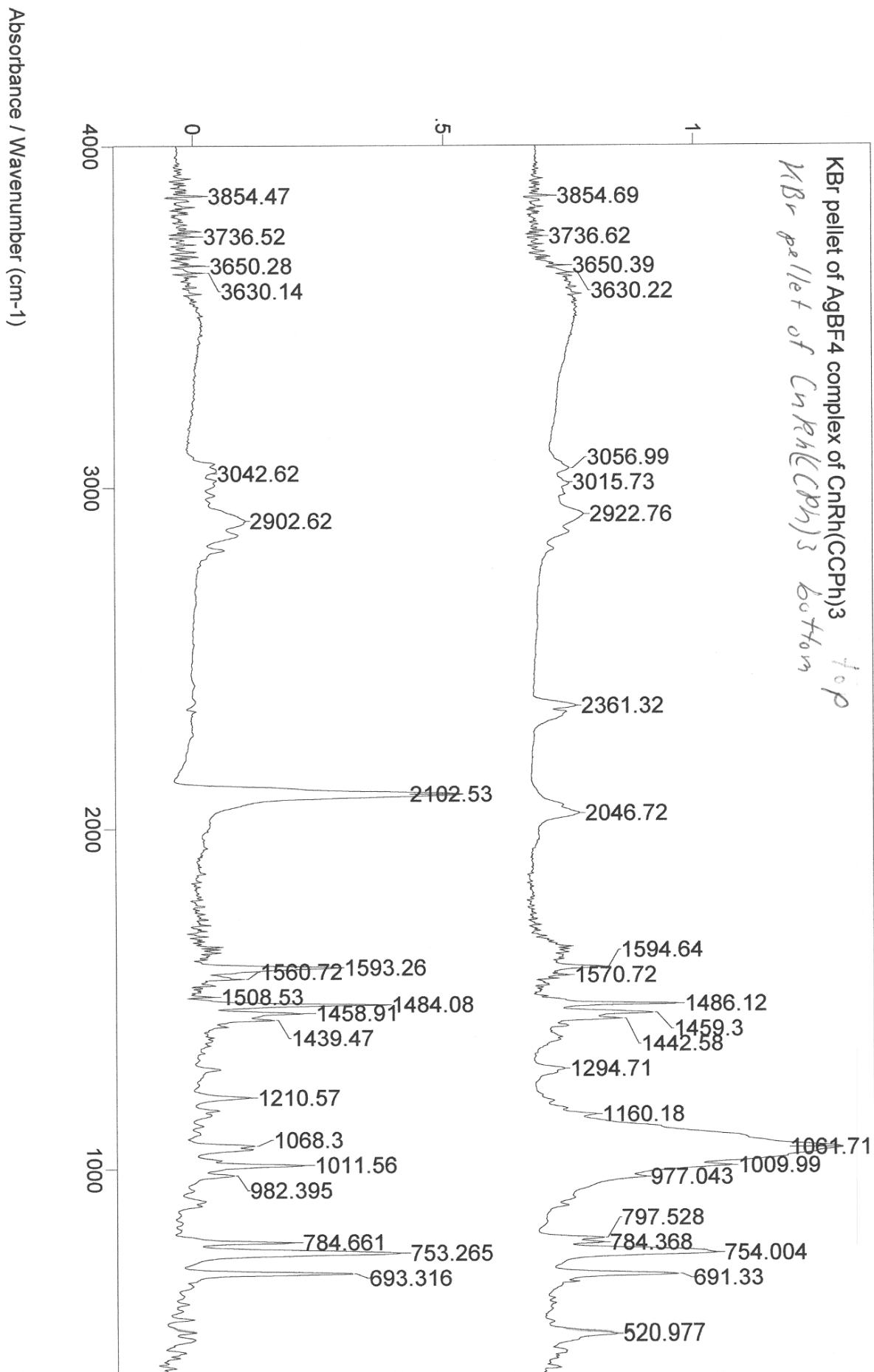
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Sample Description: E236 final P4

Scans = 8

Res = 4 cm⁻¹ 21 scans/min

Apod = Cosine



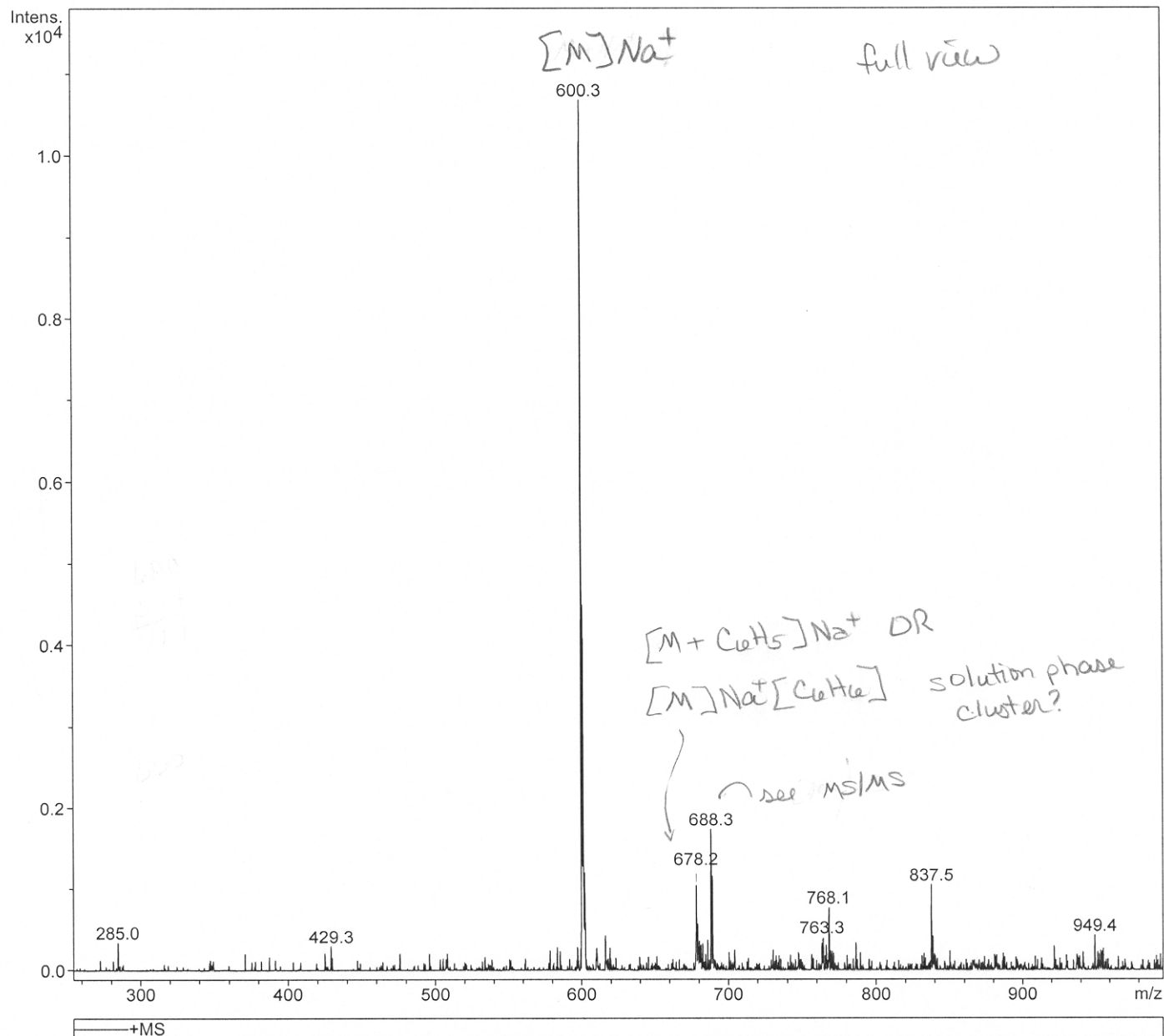
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Analysis Info

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Sample Name	research	Instrument	Esquire-LC_00143
Comment	Youngs/Paul Custer Paul 1 C33 H36 M3 Rh1 577 10-4M DMSO/CH3CN 8:2 120//5/8/150//600/20/100		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
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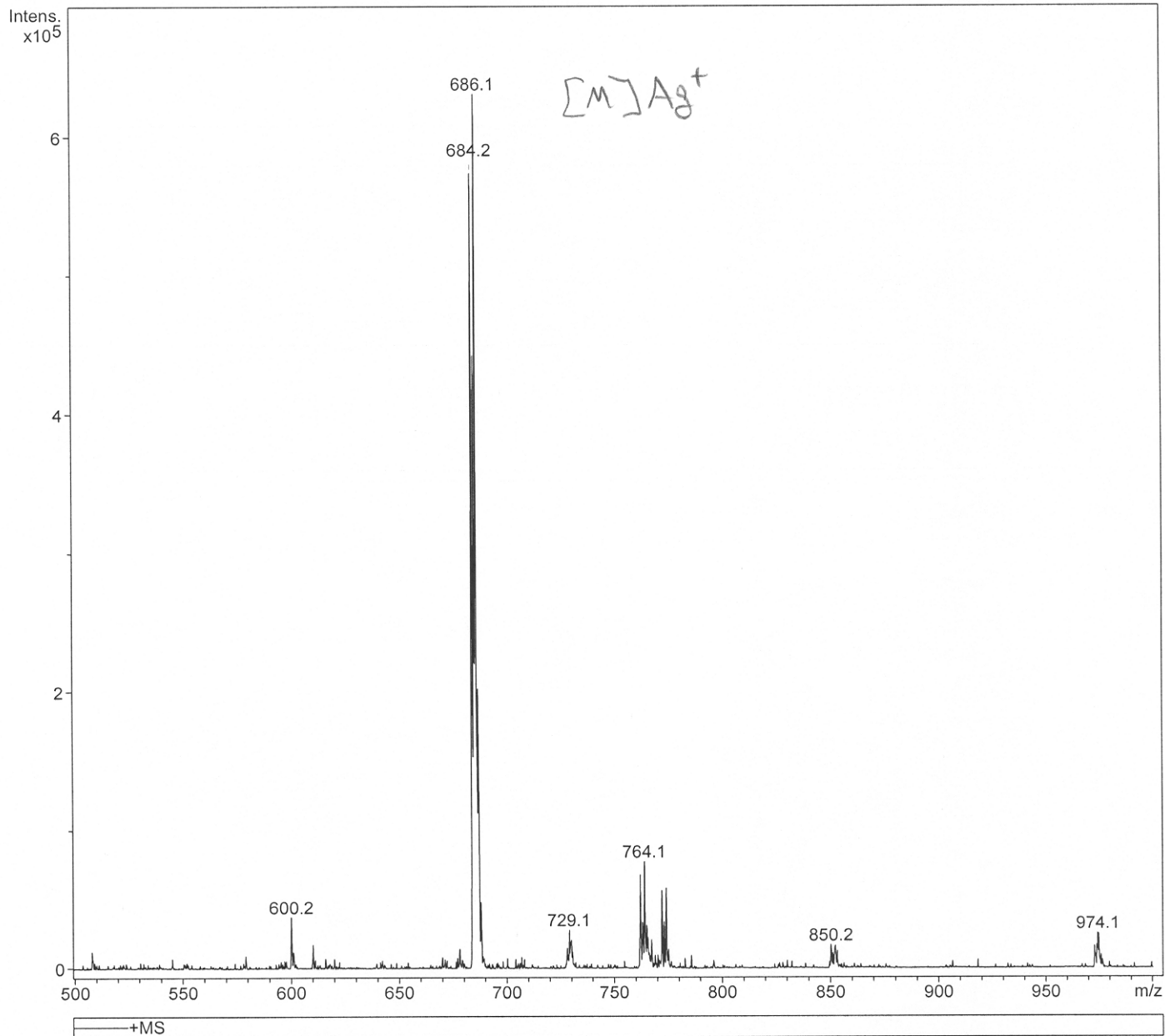
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Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
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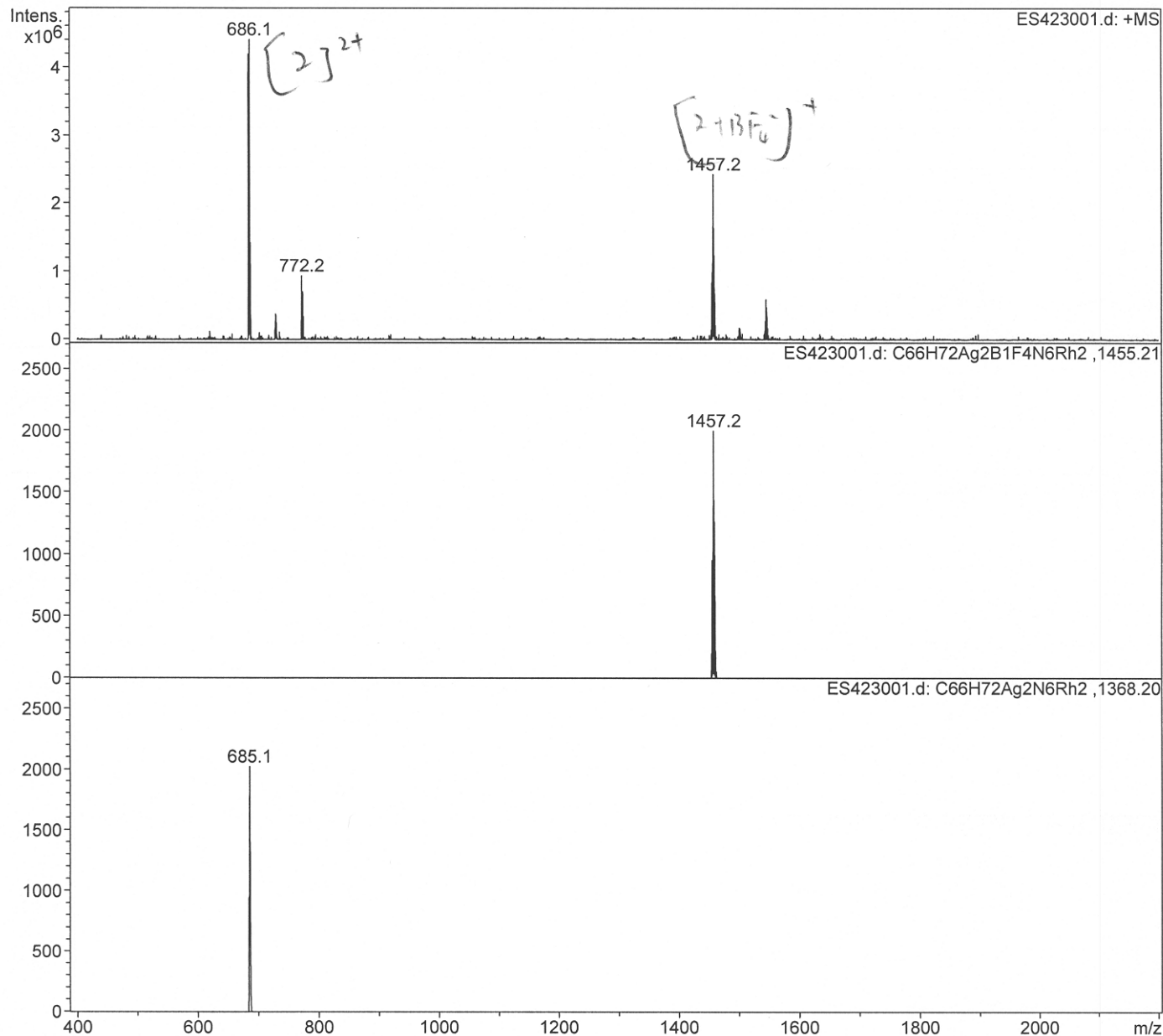
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Analysis Info

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Sample Name	research	Instrument	Esquire-LC_00143
Comment	Youngs/Paul Custer 007A in CH3CN C 33 H 35 N 3 Rh B F 4 Ag in acetone 10/8/300/1544/10/140 enhanced resolution		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Enhanced	Scan Begin	400.00 m/z	Scan End	2200.00 m/z
Capillary Exit	91.3 Volt	Skim 1	22.5 Volt	Trap Drive	107.5
Accumulation Time	123 μ s	Averages	30 Spectra	Auto MS/MS	Off



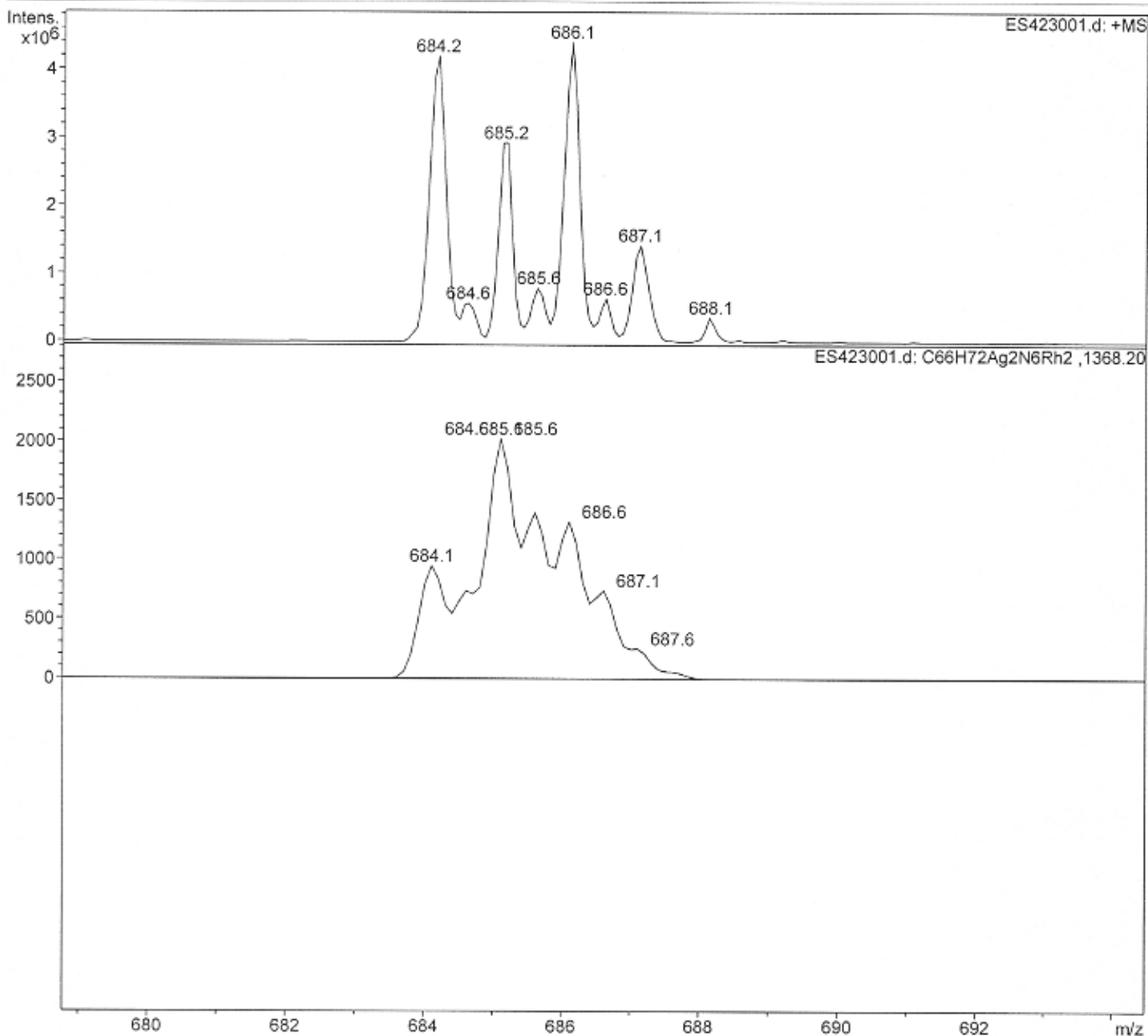
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Comment	Youngs/Paul Custer 007A in CH3CN C 33 H 35 N 3 Rh B F 4 Ag in acetone 10/8/300/1544/10/140 enhanced resolution		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Enhanced	Scan Begin	400.00 m/z	Scan End	2200.00 m/z
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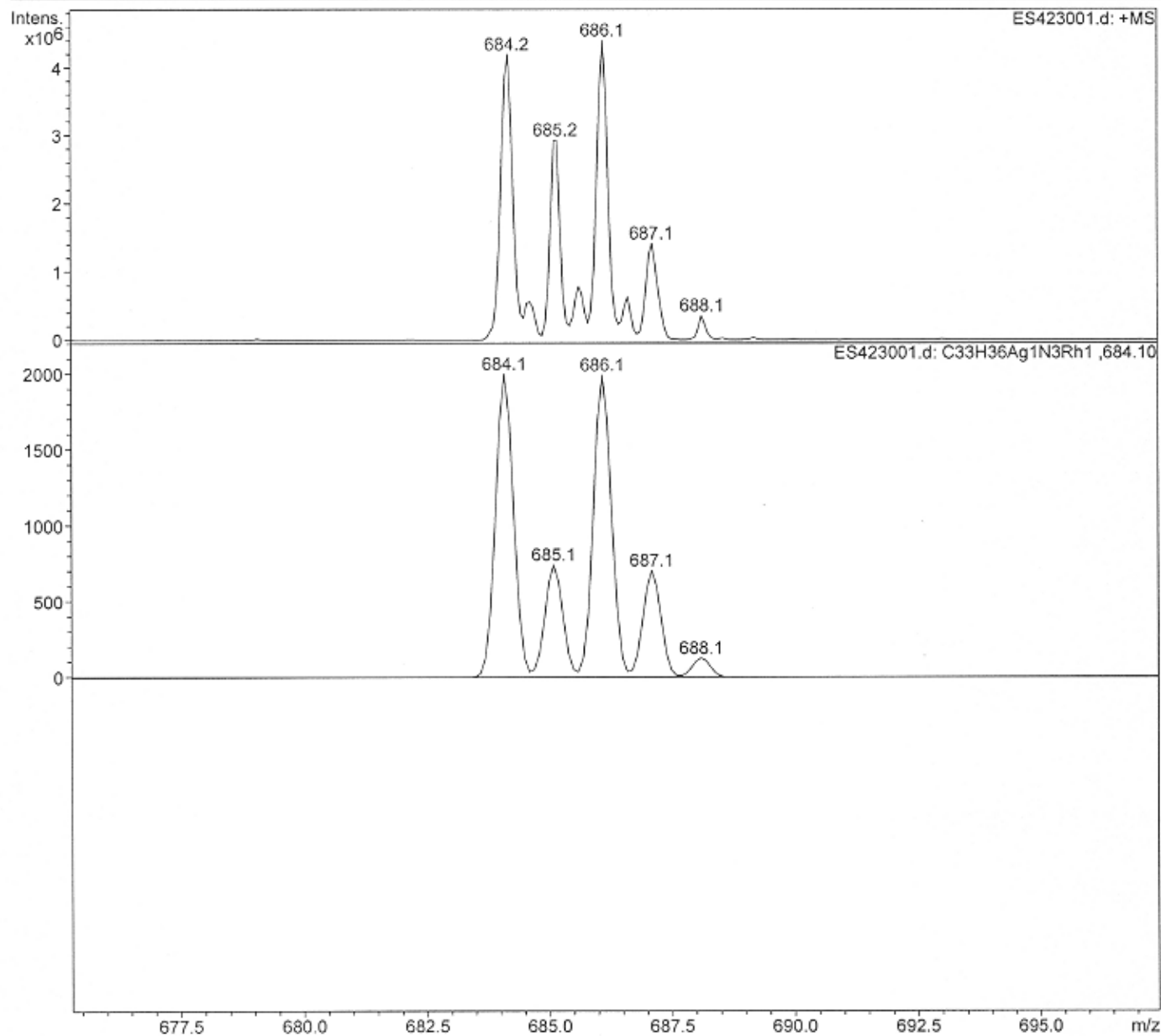
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Analysis Info

Analysis Name	ES423001.d	Acquisition Date	08/31/04 13:06:09
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Comment	Youngs/Paul Custer 007A in CH3CN C 33 H 35 N 3 Rh B F 4 Ag in acetone 10/8/300/1544/10/140 enhanced resolution		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Enhanced	Scan Begin	400.00 m/z	Scan End	2200.00 m/z
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X-ray Structure of 2 Asymmetric Unit

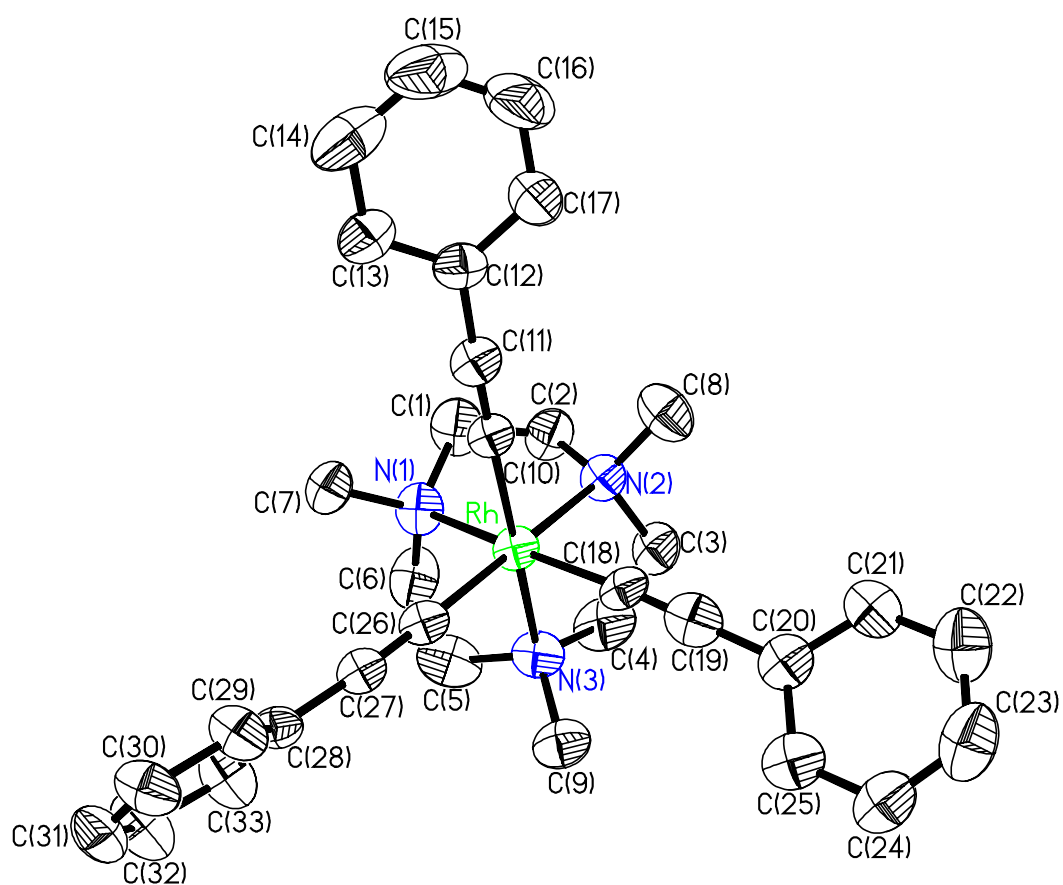


Table 1. Crystal data and structure refinement for **2**.

Identification code	2	
Empirical formula	C ₃₃ H ₃₆ N ₃ Rh	
Formula weight	577.56	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2 ₁ /c	
Unit cell dimensions	a = 11.4645(10) Å b = 17.7575(15) Å c = 13.7191(11) Å	$\alpha = 90^\circ$. $\beta = 92.6900(10)^\circ$. $\gamma = 90^\circ$.
Volume	2789.9(4) Å ³	
Z	4	
Density (calculated)	1.375 Mg/m ³	
Absorption coefficient	0.638 mm ⁻¹	
F(000)	1200	
Crystal size	0.13 x 0.08 x 0.03 mm ³	
Theta range for data collection	1.78 to 27.51°.	
Index ranges	-14 ≤ h ≤ 14, -22 ≤ k ≤ 22, -17 ≤ l ≤ 17	
Reflections collected	24233	
Independent reflections	6381 [R(int) = 0.0527]	
Completeness to theta = 27.51°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9811 and 0.8934	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	6381 / 0 / 337	
Goodness-of-fit on F ²	1.038	
Final R indices [I>2sigma(I)]	R1 = 0.0534, wR2 = 0.1277	
R indices (all data)	R1 = 0.0951, wR2 = 0.1456	
Largest diff. peak and hole	1.398 and -0.356 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Rh	5855(1)	6909(1)	9001(1)	37(1)
N(1)	5385(4)	5904(2)	8189(2)	49(1)
N(2)	4967(3)	7441(2)	7768(2)	43(1)
N(3)	7298(4)	6909(2)	8058(3)	49(1)
C(1)	4289(5)	6122(3)	7616(4)	67(2)
C(2)	4385(5)	6858(3)	7135(4)	60(1)
C(3)	5921(5)	7816(3)	7240(4)	56(1)
C(4)	6954(5)	7341(4)	7151(4)	73(2)
C(5)	7431(5)	6078(4)	7847(4)	70(2)
C(6)	6336(5)	5702(3)	7544(4)	67(2)
C(7)	5095(5)	5252(3)	8791(3)	52(1)
C(8)	4119(5)	8027(3)	8036(4)	66(2)
C(9)	8411(4)	7190(3)	8523(4)	59(1)
C(10)	4432(4)	6866(3)	9781(3)	37(1)
C(11)	3536(4)	6832(3)	10171(3)	46(1)
C(12)	2419(4)	6808(3)	10619(4)	47(1)
C(13)	2105(5)	6196(3)	11184(4)	58(1)
C(14)	1023(6)	6169(4)	11607(5)	85(2)
C(15)	262(6)	6741(5)	11448(7)	104(3)
C(16)	544(6)	7347(4)	10919(7)	109(3)
C(17)	1622(5)	7389(3)	10500(6)	80(2)
C(18)	6314(4)	7907(3)	9596(3)	40(1)
C(19)	6674(5)	8488(3)	9773(3)	53(1)
C(20)	7143(4)	9239(3)	10018(3)	50(1)
C(21)	6504(5)	9896(3)	9853(4)	56(1)
C(22)	6894(6)	10590(4)	10134(4)	74(2)
C(23)	7965(6)	10657(4)	10576(4)	77(2)
C(24)	8669(5)	10047(3)	10724(4)	64(2)
C(25)	8267(5)	9339(3)	10439(3)	59(1)
C(26)	6741(4)	6350(3)	10039(3)	38(1)
C(27)	7295(4)	5964(3)	10605(3)	39(1)

C(28)	7928(4)	5428(3)	11216(3)	41(1)
C(29)	7866(4)	5403(3)	12221(3)	52(1)
C(30)	8444(4)	4851(3)	12770(4)	59(1)
C(31)	9080(5)	4310(3)	12326(4)	61(1)
C(32)	9159(5)	4332(4)	11323(4)	74(2)
C(33)	8610(5)	4888(3)	10783(4)	62(2)

Table 3. Bond lengths [Å] and angles [°] for **2**.

<hr/>		C(8)-H(8A)	0.9800
Rh-C(26)	1.977(5)	C(8)-H(8B)	0.9800
Rh-C(10)	1.995(4)	C(8)-H(8C)	0.9800
Rh-C(18)	2.010(5)	C(9)-H(9A)	0.9800
Rh-N(3)	2.147(4)	C(9)-H(9B)	0.9800
Rh-N(2)	2.151(4)	C(9)-H(9C)	0.9800
Rh-N(1)	2.159(4)	C(10)-C(11)	1.181(6)
N(1)-C(7)	1.471(6)	C(11)-C(12)	1.446(7)
N(1)-C(6)	1.480(6)	C(12)-C(17)	1.381(7)
N(1)-C(1)	1.500(6)	C(12)-C(13)	1.393(7)
N(2)-C(8)	1.483(6)	C(13)-C(14)	1.395(8)
N(2)-C(2)	1.489(6)	C(13)-H(13)	0.9500
N(2)-C(3)	1.496(6)	C(14)-C(15)	1.350(10)
N(3)-C(9)	1.486(7)	C(14)-H(14)	0.9500
N(3)-C(4)	1.499(6)	C(15)-C(16)	1.347(11)
N(3)-C(5)	1.511(7)	C(15)-H(15)	0.9500
C(1)-C(2)	1.469(7)	C(16)-C(17)	1.389(9)
C(1)-H(1A)	0.9900	C(16)-H(16)	0.9500
C(1)-H(1B)	0.9900	C(17)-H(17)	0.9500
C(2)-H(2A)	0.9900	C(18)-C(19)	1.135(7)
C(2)-H(2B)	0.9900	C(19)-C(20)	1.470(8)
C(3)-C(4)	1.463(8)	C(20)-C(21)	1.390(7)
C(3)-H(3A)	0.9900	C(20)-C(25)	1.399(7)
C(3)-H(3B)	0.9900	C(21)-C(22)	1.359(8)
C(4)-H(4A)	0.9900	C(21)-H(21)	0.9500
C(4)-H(4B)	0.9900	C(22)-C(23)	1.350(9)
C(5)-C(6)	1.466(8)	C(22)-H(22)	0.9500
C(5)-H(5A)	0.9900	C(23)-C(24)	1.360(8)
C(5)-H(5B)	0.9900	C(23)-H(23)	0.9500
C(6)-H(6A)	0.9900	C(24)-C(25)	1.390(8)
C(6)-H(6B)	0.9900	C(24)-H(24)	0.9500
C(7)-H(7A)	0.9800	C(25)-H(25)	0.9500
C(7)-H(7B)	0.9800	C(26)-C(27)	1.197(6)
C(7)-H(7C)	0.9800	C(27)-C(28)	1.441(6)
		C(28)-C(29)	1.386(6)

C(28)-C(33)	1.387(7)	C(2)-N(2)-Rh	109.6(3)
C(29)-C(30)	1.384(7)	C(3)-N(2)-Rh	104.3(3)
C(29)-H(29)	0.9500	C(9)-N(3)-C(4)	111.8(4)
C(30)-C(31)	1.366(7)	C(9)-N(3)-C(5)	108.5(4)
C(30)-H(30)	0.9500	C(4)-N(3)-C(5)	111.5(4)
C(31)-C(32)	1.383(7)	C(9)-N(3)-Rh	114.3(3)
C(31)-H(31)	0.9500	C(4)-N(3)-Rh	108.6(3)
C(32)-C(33)	1.371(7)	C(5)-N(3)-Rh	101.7(3)
C(32)-H(32)	0.9500	C(2)-C(1)-N(1)	112.7(5)
C(33)-H(33)	0.9500	C(2)-C(1)-H(1A)	109.1
		N(1)-C(1)-H(1A)	109.1
C(26)-Rh-C(10)	90.00(17)	C(2)-C(1)-H(1B)	109.1
C(26)-Rh-C(18)	91.94(17)	N(1)-C(1)-H(1B)	109.1
C(10)-Rh-C(18)	91.16(18)	H(1A)-C(1)-H(1B)	107.8
C(26)-Rh-N(3)	92.84(16)	C(1)-C(2)-N(2)	113.4(4)
C(10)-Rh-N(3)	174.93(16)	C(1)-C(2)-H(2A)	108.9
C(18)-Rh-N(3)	92.95(17)	N(2)-C(2)-H(2A)	108.9
C(26)-Rh-N(2)	174.21(15)	C(1)-C(2)-H(2B)	108.9
C(10)-Rh-N(2)	94.00(16)	N(2)-C(2)-H(2B)	108.9
C(18)-Rh-N(2)	92.17(15)	H(2A)-C(2)-H(2B)	107.7
N(3)-Rh-N(2)	82.88(15)	C(4)-C(3)-N(2)	113.3(4)
C(26)-Rh-N(1)	93.69(16)	C(4)-C(3)-H(3A)	108.9
C(10)-Rh-N(1)	93.11(16)	N(2)-C(3)-H(3A)	108.9
C(18)-Rh-N(1)	172.93(15)	C(4)-C(3)-H(3B)	108.9
N(3)-Rh-N(1)	82.51(15)	N(2)-C(3)-H(3B)	108.9
N(2)-Rh-N(1)	81.92(14)	H(3A)-C(3)-H(3B)	107.7
C(7)-N(1)-C(6)	109.6(4)	C(3)-C(4)-N(3)	114.1(4)
C(7)-N(1)-C(1)	107.0(4)	C(3)-C(4)-H(4A)	108.7
C(6)-N(1)-C(1)	111.9(4)	N(3)-C(4)-H(4A)	108.7
C(7)-N(1)-Rh	114.8(3)	C(3)-C(4)-H(4B)	108.7
C(6)-N(1)-Rh	109.6(3)	N(3)-C(4)-H(4B)	108.7
C(1)-N(1)-Rh	103.9(3)	H(4A)-C(4)-H(4B)	107.6
C(8)-N(2)-C(2)	110.6(4)	C(6)-C(5)-N(3)	114.0(5)
C(8)-N(2)-C(3)	108.1(4)	C(6)-C(5)-H(5A)	108.8
C(2)-N(2)-C(3)	110.2(4)	N(3)-C(5)-H(5A)	108.8
C(8)-N(2)-Rh	113.9(3)	C(6)-C(5)-H(5B)	108.8

N(3)-C(5)-H(5B)	108.8	C(13)-C(14)-H(14)	120.4
H(5A)-C(5)-H(5B)	107.7	C(16)-C(15)-C(14)	121.2(7)
C(5)-C(6)-N(1)	111.4(4)	C(16)-C(15)-H(15)	119.4
C(5)-C(6)-H(6A)	109.3	C(14)-C(15)-H(15)	119.4
N(1)-C(6)-H(6A)	109.3	C(15)-C(16)-C(17)	120.4(7)
C(5)-C(6)-H(6B)	109.3	C(15)-C(16)-H(16)	119.8
N(1)-C(6)-H(6B)	109.3	C(17)-C(16)-H(16)	119.8
H(6A)-C(6)-H(6B)	108.0	C(12)-C(17)-C(16)	120.5(6)
N(1)-C(7)-H(7A)	109.5	C(12)-C(17)-H(17)	119.7
N(1)-C(7)-H(7B)	109.5	C(16)-C(17)-H(17)	119.7
H(7A)-C(7)-H(7B)	109.5	C(19)-C(18)-Rh	167.1(4)
N(1)-C(7)-H(7C)	109.5	C(18)-C(19)-C(20)	179.1(5)
H(7A)-C(7)-H(7C)	109.5	C(21)-C(20)-C(25)	115.4(5)
H(7B)-C(7)-H(7C)	109.5	C(21)-C(20)-C(19)	122.7(5)
N(2)-C(8)-H(8A)	109.5	C(25)-C(20)-C(19)	121.9(5)
N(2)-C(8)-H(8B)	109.5	C(22)-C(21)-C(20)	123.4(6)
H(8A)-C(8)-H(8B)	109.5	C(22)-C(21)-H(21)	118.3
N(2)-C(8)-H(8C)	109.5	C(20)-C(21)-H(21)	118.3
H(8A)-C(8)-H(8C)	109.5	C(23)-C(22)-C(21)	119.2(6)
H(8B)-C(8)-H(8C)	109.5	C(23)-C(22)-H(22)	120.4
N(3)-C(9)-H(9A)	109.5	C(21)-C(22)-H(22)	120.4
N(3)-C(9)-H(9B)	109.5	C(22)-C(23)-C(24)	121.1(6)
H(9A)-C(9)-H(9B)	109.5	C(22)-C(23)-H(23)	119.4
N(3)-C(9)-H(9C)	109.5	C(24)-C(23)-H(23)	119.4
H(9A)-C(9)-H(9C)	109.5	C(23)-C(24)-C(25)	119.5(6)
H(9B)-C(9)-H(9C)	109.5	C(23)-C(24)-H(24)	120.2
C(11)-C(10)-Rh	174.4(4)	C(25)-C(24)-H(24)	120.2
C(10)-C(11)-C(12)	177.8(5)	C(24)-C(25)-C(20)	121.2(6)
C(17)-C(12)-C(13)	117.5(5)	C(24)-C(25)-H(25)	119.4
C(17)-C(12)-C(11)	121.5(5)	C(20)-C(25)-H(25)	119.4
C(13)-C(12)-C(11)	121.0(5)	C(27)-C(26)-Rh	174.1(4)
C(12)-C(13)-C(14)	121.0(6)	C(26)-C(27)-C(28)	173.6(5)
C(12)-C(13)-H(13)	119.5	C(29)-C(28)-C(33)	117.4(4)
C(14)-C(13)-H(13)	119.5	C(29)-C(28)-C(27)	123.5(4)
C(15)-C(14)-C(13)	119.3(7)	C(33)-C(28)-C(27)	119.1(4)
C(15)-C(14)-H(14)	120.4	C(30)-C(29)-C(28)	121.2(5)

C(30)-C(29)-H(29)	119.4
C(28)-C(29)-H(29)	119.4
C(31)-C(30)-C(29)	120.4(5)
C(31)-C(30)-H(30)	119.8
C(29)-C(30)-H(30)	119.8
C(30)-C(31)-C(32)	119.0(5)
C(30)-C(31)-H(31)	120.5
C(32)-C(31)-H(31)	120.5
C(33)-C(32)-C(31)	120.5(5)

C(33)-C(32)-H(32)	119.7
C(31)-C(32)-H(32)	119.7
C(32)-C(33)-C(28)	121.4(5)
C(32)-C(33)-H(33)	119.3
C(28)-C(33)-H(33)	119.3

Symmetry transformations used to generate
equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Rh	44(1)	50(1)	18(1)	2(1)	6(1)	-2(1)
N(1)	67(3)	56(3)	23(2)	-2(2)	4(2)	0(2)
N(2)	52(2)	52(2)	26(2)	4(2)	1(2)	-2(2)
N(3)	51(2)	71(3)	26(2)	3(2)	8(2)	2(2)
C(1)	82(4)	70(4)	46(3)	-1(3)	-21(3)	-7(3)
C(2)	68(4)	62(3)	48(3)	5(3)	-16(3)	-14(3)
C(3)	68(4)	62(3)	37(3)	15(2)	5(2)	-4(3)
C(4)	73(4)	109(5)	39(3)	18(3)	15(3)	-13(4)
C(5)	66(4)	98(5)	47(3)	-20(3)	15(3)	18(3)
C(6)	97(5)	58(3)	48(3)	-7(3)	30(3)	-4(3)
C(7)	65(3)	54(3)	38(2)	1(2)	4(2)	-10(3)
C(8)	78(4)	77(4)	42(3)	10(3)	1(3)	22(3)
C(9)	53(3)	81(4)	43(3)	3(3)	17(2)	-8(3)
C(10)	40(2)	47(3)	25(2)	1(2)	1(2)	-3(2)
C(11)	50(3)	55(3)	32(2)	1(2)	3(2)	-5(2)
C(12)	42(3)	54(3)	45(3)	-11(2)	2(2)	-4(2)
C(13)	57(3)	62(3)	56(3)	-2(3)	10(2)	-10(3)
C(14)	80(5)	90(5)	88(5)	-24(4)	29(4)	-37(4)
C(15)	61(4)	86(6)	168(9)	-63(6)	34(5)	-16(4)
C(16)	61(4)	69(5)	198(10)	-38(6)	16(5)	17(4)
C(17)	56(4)	54(4)	128(6)	2(4)	-6(4)	5(3)
C(18)	40(2)	56(3)	25(2)	14(2)	-1(2)	-13(2)
C(19)	60(3)	74(4)	25(2)	8(2)	5(2)	5(3)
C(20)	60(3)	62(3)	28(2)	5(2)	7(2)	-5(3)
C(21)	64(3)	66(4)	39(3)	4(2)	10(2)	10(3)
C(22)	107(5)	65(4)	51(3)	7(3)	-2(3)	2(4)
C(23)	116(6)	66(4)	47(3)	8(3)	-2(3)	-20(4)
C(24)	73(4)	76(4)	41(3)	14(3)	-5(3)	-20(3)
C(25)	63(3)	79(4)	35(3)	7(3)	5(2)	-7(3)
C(26)	41(2)	53(3)	21(2)	-5(2)	7(2)	-5(2)
C(27)	42(2)	49(3)	27(2)	-5(2)	6(2)	-5(2)

C(28)	35(2)	53(3)	33(2)	-2(2)	-2(2)	-4(2)
C(29)	53(3)	69(3)	34(2)	3(2)	7(2)	14(3)
C(30)	53(3)	93(4)	33(2)	9(3)	5(2)	14(3)
C(31)	63(3)	66(3)	54(3)	7(3)	-13(3)	12(3)
C(32)	95(5)	75(4)	50(3)	-14(3)	-9(3)	31(3)
C(33)	85(4)	68(4)	31(2)	-8(2)	-2(2)	20(3)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **2**.

	x	y	z	U(eq)
H(1A)	4108	5733	7115	80
H(1B)	3633	6138	8060	80
H(2A)	3593	7036	6931	72
H(2B)	4831	6795	6540	72
H(3A)	5617	7960	6579	67
H(3B)	6154	8284	7592	67
H(4A)	7617	7663	6977	88
H(4B)	6801	6980	6610	88
H(5A)	7993	6016	7327	84
H(5B)	7766	5827	8441	84
H(6A)	6454	5150	7560	80
H(6B)	6109	5847	6865	80
H(7A)	4818	4838	8368	78
H(7B)	4482	5392	9231	78
H(7C)	5793	5090	9176	78
H(8A)	4508	8393	8476	98
H(8B)	3472	7791	8365	98
H(8C)	3816	8284	7446	98
H(9A)	8605	6897	9114	88
H(9B)	8324	7722	8696	88
H(9C)	9038	7137	8066	88
H(13)	2637	5790	11284	70
H(14)	824	5754	12002	102
H(15)	-487	6715	11714	125
H(16)	3	7749	10831	131
H(17)	1814	7820	10129	95
H(21)	5757	9859	9528	67
H(22)	6418	11021	10020	89
H(23)	8233	11139	10788	92
H(24)	9429	10105	11020	76

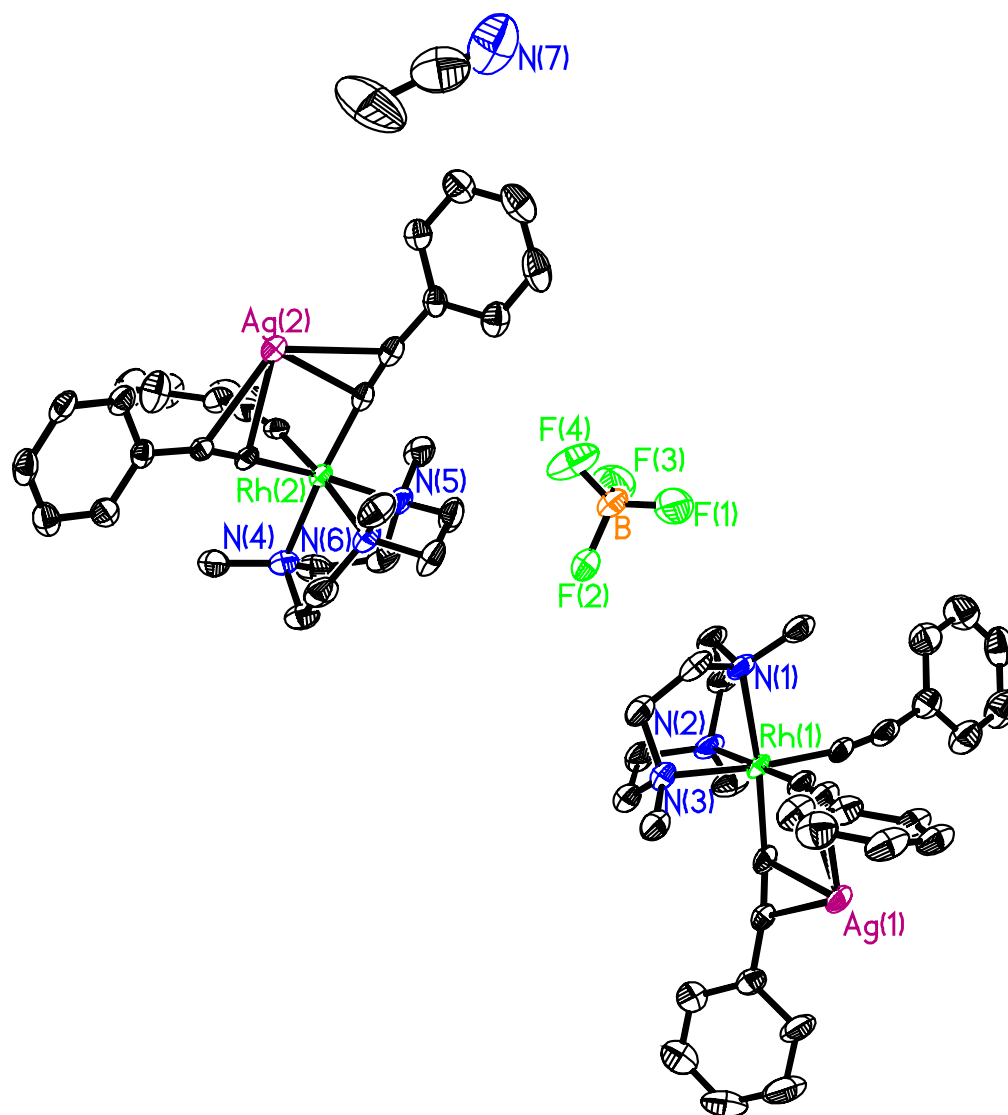
H(25)	8765	8915	10531	70
H(29)	7419	5770	12540	62
H(30)	8398	4849	13459	71
H(31)	9462	3925	12700	74
H(32)	9597	3958	11007	88
H(33)	8697	4905	10098	74

X-ray Structure of 3

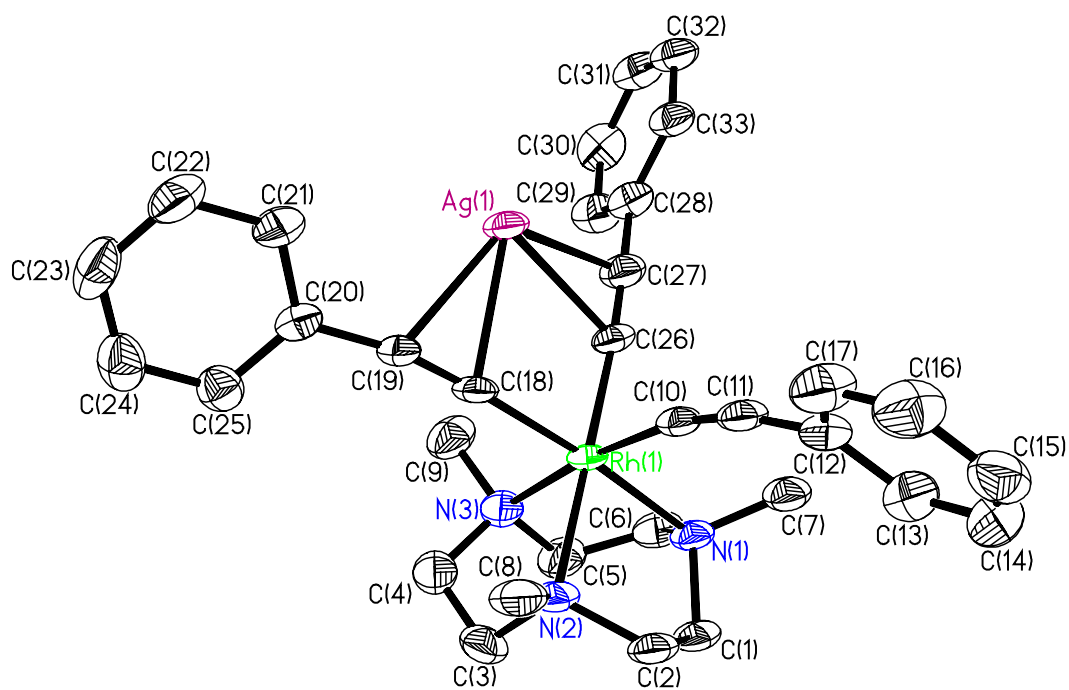
Important Information

This molecule had a disordered BF₄ molecule that could not be modeled. Platon's Squeeze routine was used to deal with the disorder. The program accounted for 82 electrons per cell. The number of electrons per cell of the disorder was 82.

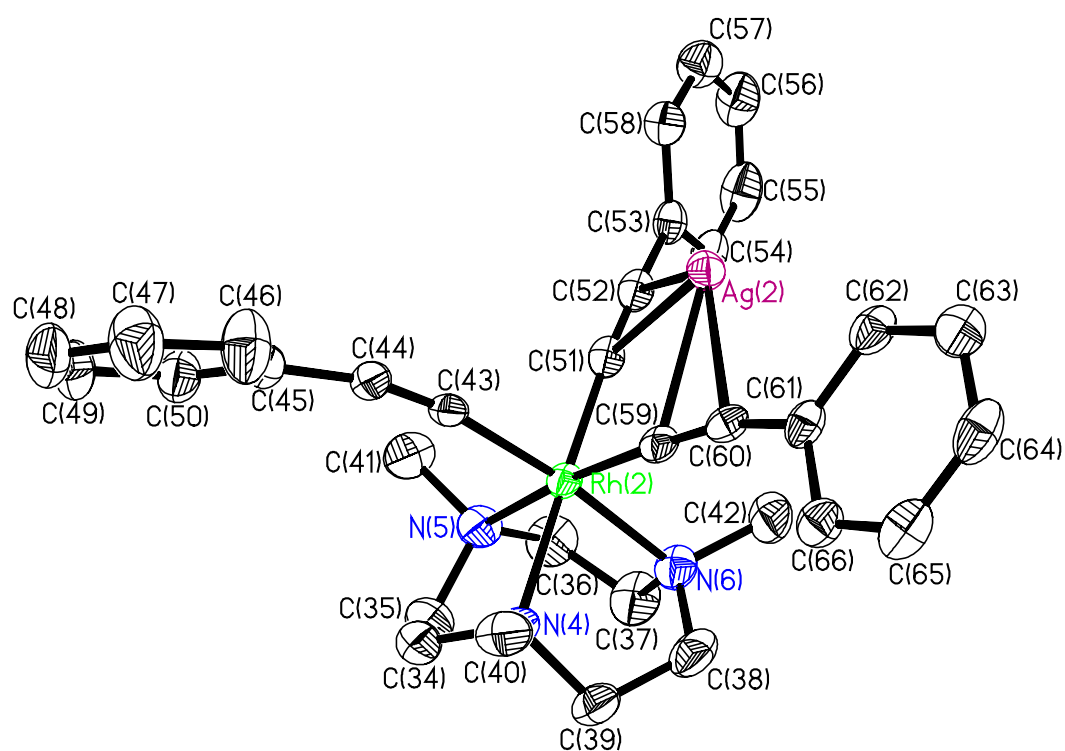
Asymmetric Unit



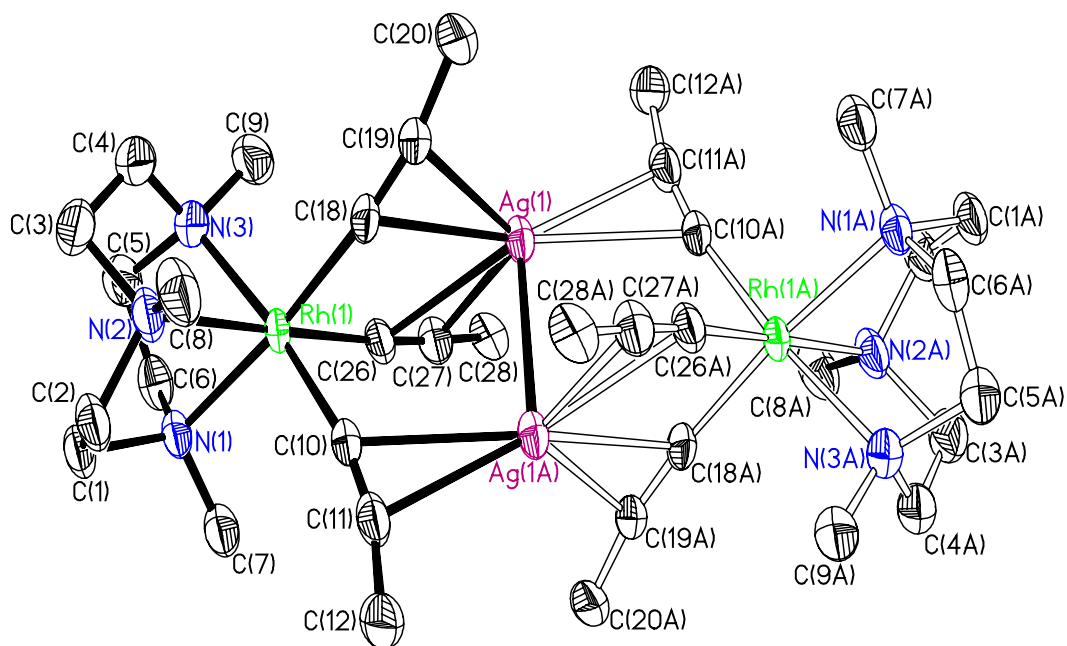
Asym Molecule 1



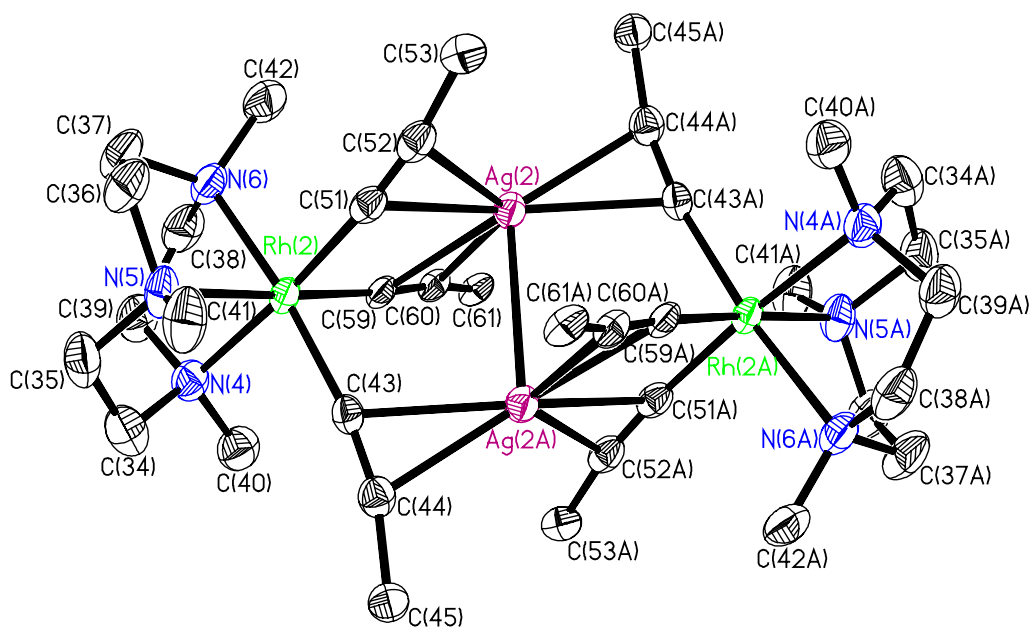
Asym Molecule 2



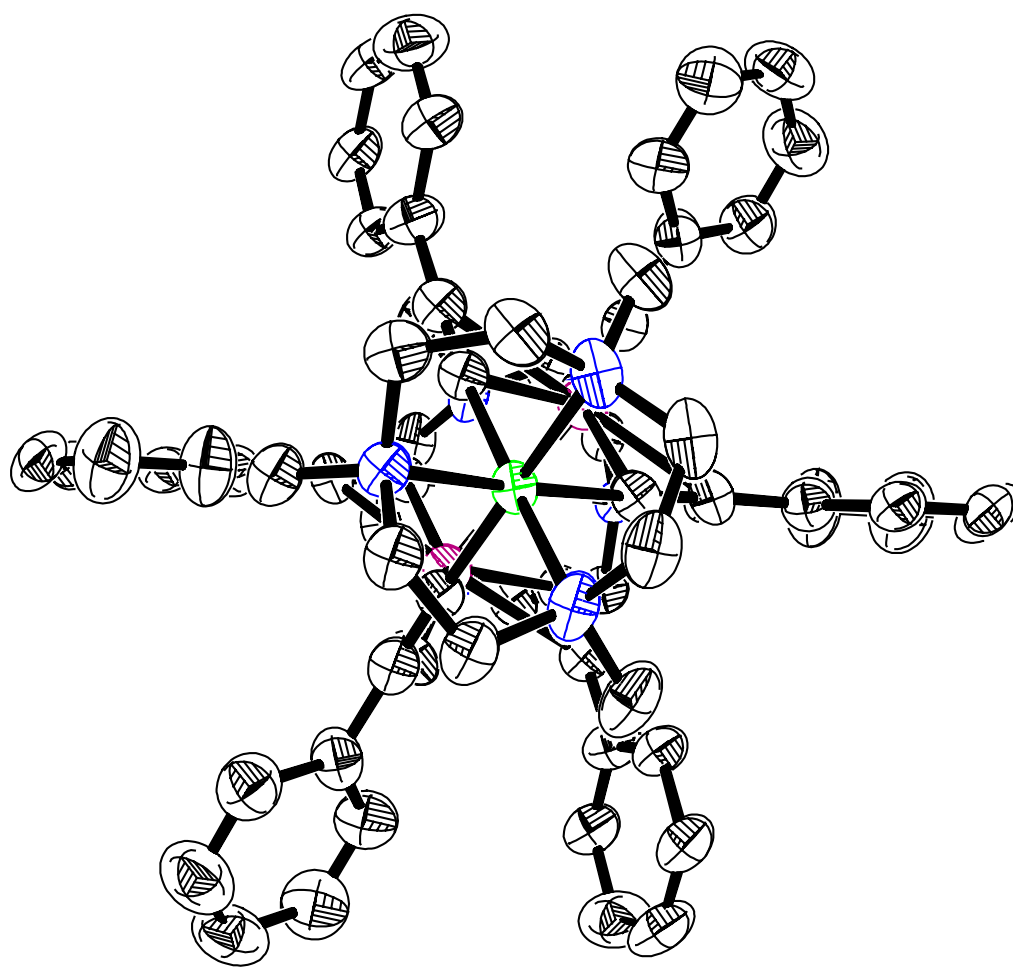
Molecule 1 –Ph



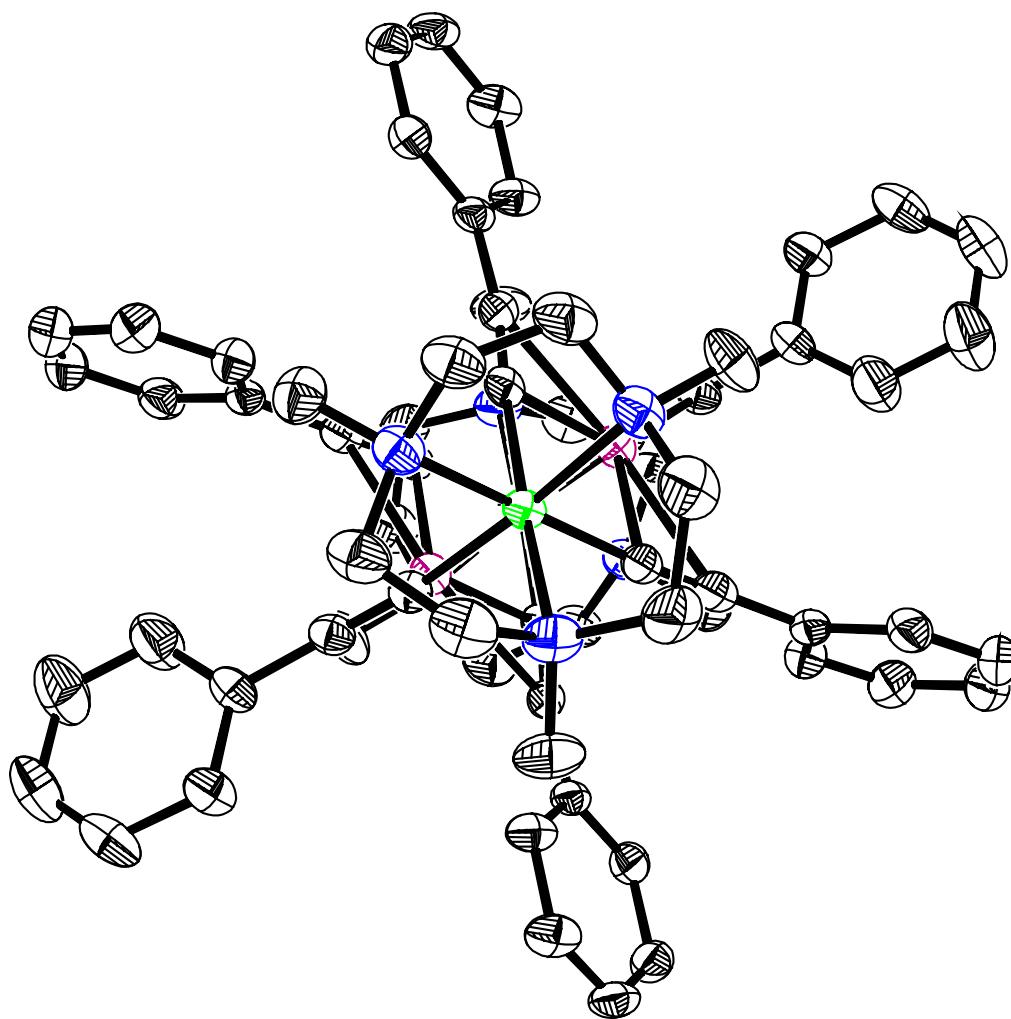
Molecule 2 –Ph



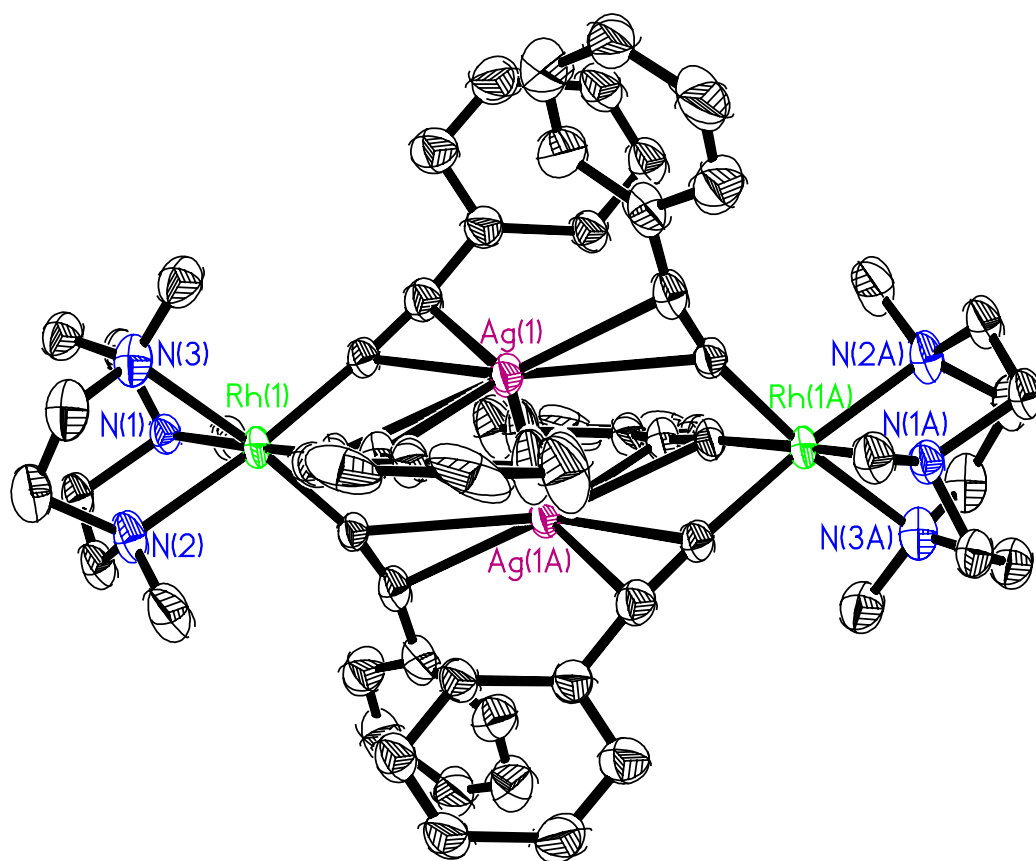
Molecule 1 Axis



Molecule 2 Axis



Molecule 1 Side



Molecule 2 Side

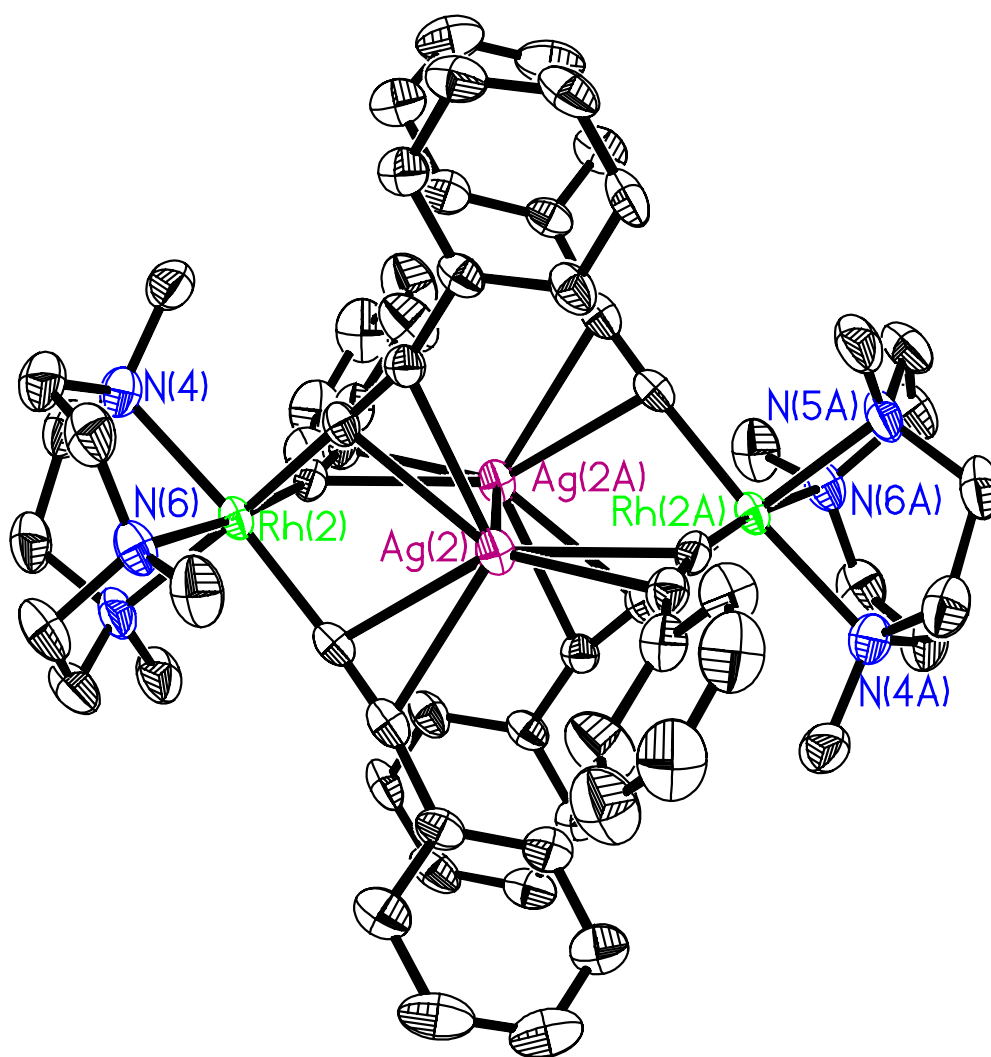


Table 1. Crystal data and structure refinement for **3**.

Identification code	3	
Empirical formula	C ₆₈ H ₇₅ Ag ₂ B ₂ F ₈ N ₇ Rh ₂	
Formula weight	1585.53	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 13.5520(11) Å b = 16.2471(13) Å c = 16.6517(14) Å	$\alpha = 77.6600(10)^\circ$. $\beta = 68.1070(10)^\circ$. $\gamma = 71.6640(10)^\circ$.
Volume	3209.8(5) Å ³	
Z	2	
Density (calculated)	1.640 Mg/m ³	
Absorption coefficient	1.177 mm ⁻¹	
F(000)	1596	
Crystal size	0.50 x 0.40 x 0.10 mm ³	
Theta range for data collection	1.33 to 27.50°.	
Index ranges	-17 ≤ h ≤ 17, -20 ≤ k ≤ 20, -21 ≤ l ≤ 21	
Reflections collected	27907	
Independent reflections	14346 [R(int) = 0.0297]	
Completeness to theta = 27.50°	97.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8914 and 0.5611	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	14346 / 0 / 765	
Goodness-of-fit on F ²	1.076	
Final R indices [I>2sigma(I)]	R1 = 0.0543, wR2 = 0.1338	
R indices (all data)	R1 = 0.0639, wR2 = 0.1385	
Largest diff. peak and hole	2.670 and -0.732 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
Ag(1)	8951(1)	9938(1)	10686(1)	36(1)
Ag(2)	4386(1)	5886(1)	5202(1)	28(1)
Rh(1)	9534(1)	8645(1)	9101(1)	31(1)
Rh(2)	6000(1)	4998(1)	6483(1)	24(1)
F(1)	8646(6)	8569(4)	5991(3)	113(2)
F(2)	8005(4)	7515(3)	6964(2)	76(1)
F(3)	9320(4)	7197(4)	5682(3)	82(1)
F(4)	7604(4)	7860(3)	5723(3)	82(1)
N(1)	9362(4)	8732(3)	7857(3)	34(1)
N(2)	10386(4)	7328(3)	8858(3)	42(1)
N(3)	8105(5)	8145(3)	9541(3)	46(1)
N(4)	5959(4)	4174(3)	7682(3)	34(1)
N(5)	7600(3)	4958(3)	6462(3)	36(1)
N(6)	5485(4)	6017(3)	7292(3)	35(1)
N(7)	4630(8)	9803(7)	1343(8)	114(3)
C(1)	10010(5)	7890(4)	7465(4)	41(1)
C(2)	10859(5)	7381(4)	7886(4)	42(1)
C(3)	9555(6)	6785(4)	9209(4)	51(2)
C(4)	8519(6)	7232(4)	9867(4)	55(2)
C(5)	7734(6)	8212(4)	8779(4)	49(2)
C(6)	8146(5)	8879(4)	8065(4)	45(1)
C(7)	9678(5)	9476(4)	7234(4)	42(1)
C(8)	11316(6)	6935(4)	9221(4)	53(2)
C(9)	7152(6)	8594(5)	10258(4)	53(2)
C(10)	10923(4)	9014(3)	8592(3)	31(1)
C(11)	11834(5)	9129(3)	8189(3)	39(1)
C(12)	12959(5)	9120(3)	7601(4)	40(1)
C(13)	13200(6)	9095(4)	6710(4)	53(2)
C(14)	14270(6)	9047(5)	6150(5)	60(2)
C(15)	15085(6)	9046(4)	6449(5)	56(2)
C(16)	14851(6)	9070(5)	7327(5)	62(2)

C(17)	13797(6)	9108(5)	7895(5)	54(2)
C(18)	9593(5)	8484(3)	10298(3)	35(1)
C(19)	9549(5)	8349(3)	11059(3)	37(1)
C(20)	9530(5)	8021(4)	11944(3)	36(1)
C(21)	9741(8)	8488(5)	12437(4)	66(2)
C(22)	9693(8)	8162(5)	13292(5)	73(2)
C(23)	9424(6)	7379(5)	13649(4)	58(2)
C(24)	9223(5)	6922(4)	13154(4)	51(2)
C(25)	9275(5)	7232(4)	12310(4)	40(1)
C(26)	8622(4)	9865(3)	9316(3)	33(1)
C(27)	8001(5)	10580(4)	9489(4)	41(1)
C(28)	7182(5)	11397(4)	9689(4)	42(1)
C(29)	6067(6)	11445(4)	9901(4)	51(2)
C(30)	5268(6)	12234(5)	10131(4)	54(2)
C(31)	5584(6)	12951(4)	10137(4)	51(2)
C(32)	6679(5)	12907(4)	9910(4)	45(1)
C(33)	7480(5)	12138(4)	9680(3)	38(1)
C(34)	7125(5)	3706(4)	7555(4)	45(1)
C(35)	7846(5)	4338(4)	7209(4)	46(1)
C(36)	7499(5)	5864(4)	6585(4)	47(1)
C(37)	6492(5)	6208(4)	7317(4)	49(2)
C(38)	4864(5)	5632(4)	8139(4)	45(1)
C(39)	5485(5)	4740(4)	8411(4)	43(1)
C(40)	5297(5)	3529(4)	7887(4)	43(1)
C(41)	8500(4)	4718(5)	5637(4)	48(2)
C(42)	4748(5)	6836(4)	7037(4)	48(2)
C(43)	6501(4)	3953(3)	5843(3)	26(1)
C(44)	6798(4)	3244(3)	5578(3)	29(1)
C(45)	7288(4)	2346(3)	5396(3)	33(1)
C(46)	6642(6)	1791(4)	5476(5)	57(2)
C(47)	7120(7)	919(5)	5383(6)	65(2)
C(48)	8236(6)	572(4)	5177(5)	59(2)
C(49)	8864(5)	1110(4)	5067(5)	55(2)
C(50)	8413(5)	1996(4)	5180(4)	42(1)
C(51)	6103(4)	5815(3)	5396(3)	27(1)
C(52)	6149(4)	6359(3)	4782(3)	30(1)

C(53)	6287(4)	7074(3)	4086(3)	29(1)
C(54)	6497(4)	7815(4)	4216(4)	39(1)
C(55)	6583(5)	8506(4)	3556(5)	52(2)
C(56)	6461(6)	8470(4)	2775(4)	53(2)
C(57)	6273(5)	7738(4)	2642(4)	45(1)
C(58)	6187(4)	7044(3)	3283(3)	36(1)
C(59)	4460(4)	5090(3)	6595(3)	25(1)
C(60)	3486(4)	5197(3)	6692(3)	25(1)
C(61)	2343(4)	5199(3)	7007(3)	27(1)
C(62)	1677(4)	5585(4)	6486(3)	33(1)
C(63)	562(4)	5616(4)	6818(4)	37(1)
C(64)	110(4)	5256(4)	7659(4)	40(1)
C(65)	763(5)	4861(4)	8174(4)	39(1)
C(66)	1875(4)	4839(3)	7854(3)	33(1)
C(67)	4086(10)	9375(8)	1804(8)	97(3)
C(68)	3330(11)	8842(9)	2424(8)	146(6)
B	8370(8)	7821(6)	6081(5)	58(2)

Table 3. Bond lengths [\AA] and angles [$^\circ$] for **3**.

Ag(1)-C(10)#1	2.363(4)	N(2)-C(8)	1.503(8)
Ag(1)-C(11)#1	2.377(5)	N(2)-C(3)	1.517(8)
Ag(1)-C(18)	2.387(5)	N(3)-C(4)	1.473(8)
Ag(1)-C(19)	2.466(5)	N(3)-C(9)	1.502(8)
Ag(1)-C(26)	2.515(5)	N(3)-C(5)	1.504(7)
Ag(1)-C(27)	2.627(6)	N(4)-C(34)	1.478(7)
Ag(1)-Ag(1)#1	2.9439(8)	N(4)-C(40)	1.494(7)
Ag(2)-C(44)#2	2.367(5)	N(4)-C(39)	1.502(7)
Ag(2)-C(43)#2	2.390(4)	N(5)-C(41)	1.486(7)
Ag(2)-C(59)	2.423(5)	N(5)-C(36)	1.486(7)
Ag(2)-C(51)	2.430(5)	N(5)-C(35)	1.496(7)
Ag(2)-C(60)	2.513(4)	N(6)-C(38)	1.470(7)
Ag(2)-C(52)	2.542(5)	N(6)-C(42)	1.477(7)
Ag(2)-Ag(2)#2	2.9134(7)	N(6)-C(37)	1.509(7)
Rh(1)-C(10)	1.981(5)	N(7)-C(67)	1.135(14)
Rh(1)-C(18)	1.983(5)	C(1)-C(2)	1.502(8)
Rh(1)-C(26)	2.004(5)	C(1)-H(1A)	0.9900
Rh(1)-N(2)	2.132(4)	C(1)-H(1B)	0.9900
Rh(1)-N(1)	2.140(4)	C(2)-H(2A)	0.9900
Rh(1)-N(3)	2.150(5)	C(2)-H(2B)	0.9900
Rh(2)-C(59)	1.984(5)	C(3)-C(4)	1.500(10)
Rh(2)-C(51)	1.989(5)	C(3)-H(3A)	0.9900
Rh(2)-C(43)	1.996(5)	C(3)-H(3B)	0.9900
Rh(2)-N(5)	2.137(4)	C(4)-H(4A)	0.9900
Rh(2)-N(6)	2.138(4)	C(4)-H(4B)	0.9900
Rh(2)-N(4)	2.146(4)	C(5)-C(6)	1.495(9)
F(1)-B	1.343(8)	C(5)-H(5A)	0.9900
F(2)-B	1.399(8)	C(5)-H(5B)	0.9900
F(3)-B	1.398(11)	C(6)-H(6A)	0.9900
F(4)-B	1.357(9)	C(6)-H(6B)	0.9900
N(1)-C(7)	1.477(7)	C(7)-H(7A)	0.9800
N(1)-C(6)	1.502(7)	C(7)-H(7B)	0.9800
N(1)-C(1)	1.505(7)	C(7)-H(7C)	0.9800
N(2)-C(2)	1.496(7)	C(8)-H(8A)	0.9800
		C(8)-H(8B)	0.9800
		C(8)-H(8C)	0.9800

C(9)-H(9A)	0.9800	C(29)-H(29)	0.9500
C(9)-H(9B)	0.9800	C(30)-C(31)	1.364(9)
C(9)-H(9C)	0.9800	C(30)-H(30)	0.9500
C(10)-C(11)	1.218(8)	C(31)-C(32)	1.370(9)
C(10)-Ag(1)#1	2.363(4)	C(31)-H(31)	0.9500
C(11)-C(12)	1.471(8)	C(32)-C(33)	1.389(8)
C(11)-Ag(1)#1	2.377(5)	C(32)-H(32)	0.9500
C(12)-C(17)	1.388(9)	C(33)-H(33)	0.9500
C(12)-C(13)	1.401(8)	C(34)-C(35)	1.516(9)
C(13)-C(14)	1.388(9)	C(34)-H(34A)	0.9900
C(13)-H(13)	0.9500	C(34)-H(34B)	0.9900
C(14)-C(15)	1.370(10)	C(35)-H(35A)	0.9900
C(14)-H(14)	0.9500	C(35)-H(35B)	0.9900
C(15)-C(16)	1.382(10)	C(36)-C(37)	1.494(9)
C(15)-H(15)	0.9500	C(36)-H(36A)	0.9900
C(16)-C(17)	1.378(9)	C(36)-H(36B)	0.9900
C(16)-H(16)	0.9500	C(37)-H(37A)	0.9900
C(17)-H(17)	0.9500	C(37)-H(37B)	0.9900
C(18)-C(19)	1.221(7)	C(38)-C(39)	1.499(9)
C(19)-C(20)	1.449(7)	C(38)-H(38A)	0.9900
C(20)-C(21)	1.382(8)	C(38)-H(38B)	0.9900
C(20)-C(25)	1.387(8)	C(39)-H(39A)	0.9900
C(21)-C(22)	1.394(9)	C(39)-H(39B)	0.9900
C(21)-H(21)	0.9500	C(40)-H(40A)	0.9800
C(22)-C(23)	1.384(11)	C(40)-H(40B)	0.9800
C(22)-H(22)	0.9500	C(40)-H(40C)	0.9800
C(23)-C(24)	1.363(10)	C(41)-H(41A)	0.9800
C(23)-H(23)	0.9500	C(41)-H(41B)	0.9800
C(24)-C(25)	1.371(8)	C(41)-H(41C)	0.9800
C(24)-H(24)	0.9500	C(42)-H(42A)	0.9800
C(25)-H(25)	0.9500	C(42)-H(42B)	0.9800
C(26)-C(27)	1.219(8)	C(42)-H(42C)	0.9800
C(27)-C(28)	1.446(8)	C(43)-C(44)	1.216(7)
C(28)-C(33)	1.380(8)	C(43)-Ag(2)#2	2.390(4)
C(28)-C(29)	1.398(9)	C(44)-C(45)	1.446(7)
C(29)-C(30)	1.410(9)	C(44)-Ag(2)#2	2.367(5)

C(45)-C(50)	1.381(7)	C(66)-H(66)	0.9500
C(45)-C(46)	1.399(8)	C(67)-C(68)	1.500(17)
C(46)-C(47)	1.375(9)	C(68)-H(68A)	0.9800
C(46)-H(46)	0.9500	C(68)-H(68B)	0.9800
C(47)-C(48)	1.373(10)	C(68)-H(68C)	0.9800
C(47)-H(47)	0.9500		
C(48)-C(49)	1.344(10)	C(10)#1-Ag(1)-C(11)#1	29.77(18)
C(48)-H(48)	0.9500	C(10)#1-Ag(1)-C(18)	148.43(19)
C(49)-C(50)	1.398(8)	C(11)#1-Ag(1)-C(18)	147.83(19)
C(49)-H(49)	0.9500	C(10)#1-Ag(1)-C(19)	123.89(18)
C(50)-H(50)	0.9500	C(11)#1-Ag(1)-C(19)	119.37(18)
C(51)-C(52)	1.197(7)	C(18)-Ag(1)-C(19)	29.08(17)
C(52)-C(53)	1.458(7)	C(10)#1-Ag(1)-C(26)	138.80(18)
C(53)-C(54)	1.398(7)	C(11)#1-Ag(1)-C(26)	136.62(19)
C(53)-C(58)	1.405(7)	C(18)-Ag(1)-C(26)	68.49(18)
C(54)-C(55)	1.394(9)	C(19)-Ag(1)-C(26)	96.38(17)
C(54)-H(54)	0.9500	C(10)#1-Ag(1)-C(27)	115.12(18)
C(55)-C(56)	1.386(10)	C(11)#1-Ag(1)-C(27)	109.50(19)
C(55)-H(55)	0.9500	C(18)-Ag(1)-C(27)	94.83(18)
C(56)-C(57)	1.369(9)	C(19)-Ag(1)-C(27)	120.89(17)
C(56)-H(56)	0.9500	C(26)-Ag(1)-C(27)	27.33(17)
C(57)-C(58)	1.379(8)	C(10)#1-Ag(1)-Ag(1)#1	90.30(13)
C(57)-H(57)	0.9500	C(11)#1-Ag(1)-Ag(1)#1	119.82(14)
C(58)-H(58)	0.9500	C(18)-Ag(1)-Ag(1)#1	80.85(13)
C(59)-C(60)	1.227(7)	C(19)-Ag(1)-Ag(1)#1	95.00(13)
C(60)-C(61)	1.439(6)	C(26)-Ag(1)-Ag(1)#1	76.66(12)
C(61)-C(66)	1.396(7)	C(27)-Ag(1)-Ag(1)#1	87.43(13)
C(61)-C(62)	1.398(7)	C(44)#2-Ag(2)-C(43)#2	29.61(16)
C(62)-C(63)	1.391(7)	C(44)#2-Ag(2)-C(59)	144.67(16)
C(62)-H(62)	0.9500	C(43)#2-Ag(2)-C(59)	140.86(15)
C(63)-C(64)	1.384(8)	C(44)#2-Ag(2)-C(51)	140.12(17)
C(63)-H(63)	0.9500	C(43)#2-Ag(2)-C(51)	144.88(16)
C(64)-C(65)	1.379(9)	C(59)-Ag(2)-C(51)	69.53(16)
C(64)-H(64)	0.9500	C(44)#2-Ag(2)-C(60)	116.55(16)
C(65)-C(66)	1.390(7)	C(43)#2-Ag(2)-C(60)	115.52(15)
C(65)-H(65)	0.9500	C(59)-Ag(2)-C(60)	28.71(15)

C(51)-Ag(2)-C(60)	97.65(16)	N(5)-Rh(2)-N(6)	83.17(17)
C(44)#2-Ag(2)-C(52)	112.75(17)	C(59)-Rh(2)-N(4)	95.16(18)
C(43)#2-Ag(2)-C(52)	120.79(16)	C(51)-Rh(2)-N(4)	175.30(17)
C(59)-Ag(2)-C(52)	96.63(16)	C(43)-Rh(2)-N(4)	90.58(17)
C(51)-Ag(2)-C(52)	27.74(16)	N(5)-Rh(2)-N(4)	82.67(17)
C(60)-Ag(2)-C(52)	123.68(16)	N(6)-Rh(2)-N(4)	83.09(17)
C(44)#2-Ag(2)-Ag(2)#2	120.30(12)	C(7)-N(1)-C(6)	106.8(4)
C(43)#2-Ag(2)-Ag(2)#2	90.83(11)	C(7)-N(1)-C(1)	109.9(4)
C(59)-Ag(2)-Ag(2)#2	75.80(10)	C(6)-N(1)-C(1)	112.0(4)
C(51)-Ag(2)-Ag(2)#2	79.83(11)	C(7)-N(1)-Rh(1)	115.2(3)
C(60)-Ag(2)-Ag(2)#2	85.91(10)	C(6)-N(1)-Rh(1)	103.6(3)
C(52)-Ag(2)-Ag(2)#2	91.88(11)	C(1)-N(1)-Rh(1)	109.2(3)
C(10)-Rh(1)-C(18)	92.3(2)	C(2)-N(2)-C(8)	108.2(5)
C(10)-Rh(1)-C(26)	92.5(2)	C(2)-N(2)-C(3)	111.2(4)
C(18)-Rh(1)-C(26)	87.6(2)	C(8)-N(2)-C(3)	110.5(5)
C(10)-Rh(1)-N(2)	91.7(2)	C(2)-N(2)-Rh(1)	103.6(3)
C(18)-Rh(1)-N(2)	94.69(19)	C(8)-N(2)-Rh(1)	114.8(3)
C(26)-Rh(1)-N(2)	175.1(2)	C(3)-N(2)-Rh(1)	108.4(4)
C(10)-Rh(1)-N(1)	93.11(18)	C(4)-N(3)-C(9)	109.4(5)
C(18)-Rh(1)-N(1)	174.17(19)	C(4)-N(3)-C(5)	112.0(4)
C(26)-Rh(1)-N(1)	94.26(18)	C(9)-N(3)-C(5)	108.9(5)
N(2)-Rh(1)-N(1)	83.02(16)	C(4)-N(3)-Rh(1)	103.0(4)
C(10)-Rh(1)-N(3)	173.49(18)	C(9)-N(3)-Rh(1)	115.0(3)
C(18)-Rh(1)-N(3)	92.0(2)	C(5)-N(3)-Rh(1)	108.7(3)
C(26)-Rh(1)-N(3)	92.6(2)	C(34)-N(4)-C(40)	109.5(5)
N(2)-Rh(1)-N(3)	83.0(2)	C(34)-N(4)-C(39)	112.1(4)
N(1)-Rh(1)-N(3)	82.44(17)	C(40)-N(4)-C(39)	108.5(4)
C(59)-Rh(2)-C(51)	88.30(19)	C(34)-N(4)-Rh(2)	104.1(3)
C(59)-Rh(2)-C(43)	89.33(18)	C(40)-N(4)-Rh(2)	114.1(3)
C(51)-Rh(2)-C(43)	92.63(18)	C(39)-N(4)-Rh(2)	108.6(3)
C(59)-Rh(2)-N(5)	174.90(17)	C(41)-N(5)-C(36)	109.2(5)
C(51)-Rh(2)-N(5)	93.61(18)	C(41)-N(5)-C(35)	109.8(5)
C(43)-Rh(2)-N(5)	95.30(18)	C(36)-N(5)-C(35)	110.2(4)
C(59)-Rh(2)-N(6)	92.00(17)	C(41)-N(5)-Rh(2)	114.6(3)
C(51)-Rh(2)-N(6)	93.64(18)	C(36)-N(5)-Rh(2)	103.9(3)
C(43)-Rh(2)-N(6)	173.62(18)	C(35)-N(5)-Rh(2)	109.0(3)

C(38)-N(6)-C(42)	108.3(5)	C(5)-C(6)-N(1)	110.0(5)
C(38)-N(6)-C(37)	112.2(5)	C(5)-C(6)-H(6A)	109.7
C(42)-N(6)-C(37)	109.5(5)	N(1)-C(6)-H(6A)	109.7
C(38)-N(6)-Rh(2)	102.5(3)	C(5)-C(6)-H(6B)	109.7
C(42)-N(6)-Rh(2)	115.4(3)	N(1)-C(6)-H(6B)	109.7
C(37)-N(6)-Rh(2)	108.8(3)	H(6A)-C(6)-H(6B)	108.2
C(2)-C(1)-N(1)	111.3(4)	N(1)-C(7)-H(7A)	109.5
C(2)-C(1)-H(1A)	109.4	N(1)-C(7)-H(7B)	109.5
N(1)-C(1)-H(1A)	109.4	H(7A)-C(7)-H(7B)	109.5
C(2)-C(1)-H(1B)	109.4	N(1)-C(7)-H(7C)	109.5
N(1)-C(1)-H(1B)	109.4	H(7A)-C(7)-H(7C)	109.5
H(1A)-C(1)-H(1B)	108.0	H(7B)-C(7)-H(7C)	109.5
N(2)-C(2)-C(1)	111.7(5)	N(2)-C(8)-H(8A)	109.5
N(2)-C(2)-H(2A)	109.3	N(2)-C(8)-H(8B)	109.5
C(1)-C(2)-H(2A)	109.3	H(8A)-C(8)-H(8B)	109.5
N(2)-C(2)-H(2B)	109.3	N(2)-C(8)-H(8C)	109.5
C(1)-C(2)-H(2B)	109.3	H(8A)-C(8)-H(8C)	109.5
H(2A)-C(2)-H(2B)	107.9	H(8B)-C(8)-H(8C)	109.5
C(4)-C(3)-N(2)	111.5(5)	N(3)-C(9)-H(9A)	109.5
C(4)-C(3)-H(3A)	109.3	N(3)-C(9)-H(9B)	109.5
N(2)-C(3)-H(3A)	109.3	H(9A)-C(9)-H(9B)	109.5
C(4)-C(3)-H(3B)	109.3	N(3)-C(9)-H(9C)	109.5
N(2)-C(3)-H(3B)	109.3	H(9A)-C(9)-H(9C)	109.5
H(3A)-C(3)-H(3B)	108.0	H(9B)-C(9)-H(9C)	109.5
N(3)-C(4)-C(3)	111.4(5)	C(11)-C(10)-Rh(1)	168.9(4)
N(3)-C(4)-H(4A)	109.3	C(11)-C(10)-Ag(1)#1	75.8(3)
C(3)-C(4)-H(4A)	109.3	Rh(1)-C(10)-Ag(1)#1	115.3(2)
N(3)-C(4)-H(4B)	109.3	C(10)-C(11)-C(12)	168.0(5)
C(3)-C(4)-H(4B)	109.3	C(10)-C(11)-Ag(1)#1	74.5(3)
H(4A)-C(4)-H(4B)	108.0	C(12)-C(11)-Ag(1)#1	117.5(4)
C(6)-C(5)-N(3)	111.7(5)	C(17)-C(12)-C(13)	118.8(6)
C(6)-C(5)-H(5A)	109.3	C(17)-C(12)-C(11)	122.7(5)
N(3)-C(5)-H(5A)	109.3	C(13)-C(12)-C(11)	118.5(6)
C(6)-C(5)-H(5B)	109.3	C(14)-C(13)-C(12)	119.3(7)
N(3)-C(5)-H(5B)	109.3	C(14)-C(13)-H(13)	120.4
H(5A)-C(5)-H(5B)	107.9	C(12)-C(13)-H(13)	120.4

C(15)-C(14)-C(13)	121.2(7)	C(27)-C(26)-Rh(1)	175.0(5)
C(15)-C(14)-H(14)	119.4	C(27)-C(26)-Ag(1)	81.4(3)
C(13)-C(14)-H(14)	119.4	Rh(1)-C(26)-Ag(1)	98.2(2)
C(14)-C(15)-C(16)	119.7(6)	C(26)-C(27)-C(28)	174.4(6)
C(14)-C(15)-H(15)	120.2	C(26)-C(27)-Ag(1)	71.2(4)
C(16)-C(15)-H(15)	120.2	C(28)-C(27)-Ag(1)	112.0(3)
C(17)-C(16)-C(15)	119.9(7)	C(33)-C(28)-C(29)	118.9(6)
C(17)-C(16)-H(16)	120.0	C(33)-C(28)-C(27)	121.1(5)
C(15)-C(16)-H(16)	120.0	C(29)-C(28)-C(27)	120.0(5)
C(16)-C(17)-C(12)	121.0(7)	C(28)-C(29)-C(30)	119.8(6)
C(16)-C(17)-H(17)	119.5	C(28)-C(29)-H(29)	120.1
C(12)-C(17)-H(17)	119.5	C(30)-C(29)-H(29)	120.1
C(19)-C(18)-Rh(1)	174.0(5)	C(31)-C(30)-C(29)	120.1(6)
C(19)-C(18)-Ag(1)	79.1(3)	C(31)-C(30)-H(30)	120.0
Rh(1)-C(18)-Ag(1)	103.1(2)	C(29)-C(30)-H(30)	120.0
C(18)-C(19)-C(20)	169.3(5)	C(30)-C(31)-C(32)	120.0(6)
C(18)-C(19)-Ag(1)	71.9(3)	C(30)-C(31)-H(31)	120.0
C(20)-C(19)-Ag(1)	118.6(3)	C(32)-C(31)-H(31)	120.0
C(21)-C(20)-C(25)	119.3(5)	C(31)-C(32)-C(33)	121.0(6)
C(21)-C(20)-C(19)	121.2(5)	C(31)-C(32)-H(32)	119.5
C(25)-C(20)-C(19)	119.5(5)	C(33)-C(32)-H(32)	119.5
C(20)-C(21)-C(22)	119.7(7)	C(28)-C(33)-C(32)	120.3(6)
C(20)-C(21)-H(21)	120.1	C(28)-C(33)-H(33)	119.9
C(22)-C(21)-H(21)	120.1	C(32)-C(33)-H(33)	119.9
C(23)-C(22)-C(21)	120.1(6)	N(4)-C(34)-C(35)	110.7(5)
C(23)-C(22)-H(22)	119.9	N(4)-C(34)-H(34A)	109.5
C(21)-C(22)-H(22)	119.9	C(35)-C(34)-H(34A)	109.5
C(24)-C(23)-C(22)	119.5(6)	N(4)-C(34)-H(34B)	109.5
C(24)-C(23)-H(23)	120.2	C(35)-C(34)-H(34B)	109.5
C(22)-C(23)-H(23)	120.2	H(34A)-C(34)-H(34B)	108.1
C(23)-C(24)-C(25)	121.0(6)	N(5)-C(35)-C(34)	112.0(4)
C(23)-C(24)-H(24)	119.5	N(5)-C(35)-H(35A)	109.2
C(25)-C(24)-H(24)	119.5	C(34)-C(35)-H(35A)	109.2
C(24)-C(25)-C(20)	120.4(6)	N(5)-C(35)-H(35B)	109.2
C(24)-C(25)-H(25)	119.8	C(34)-C(35)-H(35B)	109.2
C(20)-C(25)-H(25)	119.8	H(35A)-C(35)-H(35B)	107.9

N(5)-C(36)-C(37)	112.4(5)	N(6)-C(42)-H(42A)	109.5
N(5)-C(36)-H(36A)	109.1	N(6)-C(42)-H(42B)	109.5
C(37)-C(36)-H(36A)	109.1	H(42A)-C(42)-H(42B)	109.5
N(5)-C(36)-H(36B)	109.1	N(6)-C(42)-H(42C)	109.5
C(37)-C(36)-H(36B)	109.1	H(42A)-C(42)-H(42C)	109.5
H(36A)-C(36)-H(36B)	107.9	H(42B)-C(42)-H(42C)	109.5
C(36)-C(37)-N(6)	112.0(5)	C(44)-C(43)-Rh(2)	169.9(4)
C(36)-C(37)-H(37A)	109.2	C(44)-C(43)-Ag(2)#2	74.2(3)
N(6)-C(37)-H(37A)	109.2	Rh(2)-C(43)-Ag(2)#2	113.5(2)
C(36)-C(37)-H(37B)	109.2	C(43)-C(44)-C(45)	167.2(5)
N(6)-C(37)-H(37B)	109.2	C(43)-C(44)-Ag(2)#2	76.2(3)
H(37A)-C(37)-H(37B)	107.9	C(45)-C(44)-Ag(2)#2	116.6(3)
N(6)-C(38)-C(39)	113.0(5)	C(50)-C(45)-C(46)	117.7(5)
N(6)-C(38)-H(38A)	109.0	C(50)-C(45)-C(44)	120.8(5)
C(39)-C(38)-H(38A)	109.0	C(46)-C(45)-C(44)	121.5(5)
N(6)-C(38)-H(38B)	109.0	C(47)-C(46)-C(45)	120.2(6)
C(39)-C(38)-H(38B)	109.0	C(47)-C(46)-H(46)	119.9
H(38A)-C(38)-H(38B)	107.8	C(45)-C(46)-H(46)	119.9
C(38)-C(39)-N(4)	110.8(4)	C(48)-C(47)-C(46)	121.8(7)
C(38)-C(39)-H(39A)	109.5	C(48)-C(47)-H(47)	119.1
N(4)-C(39)-H(39A)	109.5	C(46)-C(47)-H(47)	119.1
C(38)-C(39)-H(39B)	109.5	C(49)-C(48)-C(47)	118.0(6)
N(4)-C(39)-H(39B)	109.5	C(49)-C(48)-H(48)	121.0
H(39A)-C(39)-H(39B)	108.1	C(47)-C(48)-H(48)	121.0
N(4)-C(40)-H(40A)	109.5	C(48)-C(49)-C(50)	122.2(6)
N(4)-C(40)-H(40B)	109.5	C(48)-C(49)-H(49)	118.9
H(40A)-C(40)-H(40B)	109.5	C(50)-C(49)-H(49)	118.9
N(4)-C(40)-H(40C)	109.5	C(45)-C(50)-C(49)	120.0(6)
H(40A)-C(40)-H(40C)	109.5	C(45)-C(50)-H(50)	120.0
H(40B)-C(40)-H(40C)	109.5	C(49)-C(50)-H(50)	120.0
N(5)-C(41)-H(41A)	109.5	C(52)-C(51)-Rh(2)	174.8(4)
N(5)-C(41)-H(41B)	109.5	C(52)-C(51)-Ag(2)	81.3(3)
H(41A)-C(41)-H(41B)	109.5	Rh(2)-C(51)-Ag(2)	100.28(18)
N(5)-C(41)-H(41C)	109.5	C(51)-C(52)-C(53)	171.7(5)
H(41A)-C(41)-H(41C)	109.5	C(51)-C(52)-Ag(2)	70.9(3)
H(41B)-C(41)-H(41C)	109.5	C(53)-C(52)-Ag(2)	116.0(3)

C(54)-C(53)-C(58)	118.7(5)	C(64)-C(63)-C(62)	120.2(5)
C(54)-C(53)-C(52)	120.1(5)	C(64)-C(63)-H(63)	119.9
C(58)-C(53)-C(52)	121.2(4)	C(62)-C(63)-H(63)	119.9
C(55)-C(54)-C(53)	119.3(6)	C(65)-C(64)-C(63)	120.3(5)
C(55)-C(54)-H(54)	120.4	C(65)-C(64)-H(64)	119.9
C(53)-C(54)-H(54)	120.4	C(63)-C(64)-H(64)	119.9
C(56)-C(55)-C(54)	121.1(6)	C(64)-C(65)-C(66)	120.0(5)
C(56)-C(55)-H(55)	119.4	C(64)-C(65)-H(65)	120.0
C(54)-C(55)-H(55)	119.4	C(66)-C(65)-H(65)	120.0
C(57)-C(56)-C(55)	119.6(6)	C(65)-C(66)-C(61)	120.4(5)
C(57)-C(56)-H(56)	120.2	C(65)-C(66)-H(66)	119.8
C(55)-C(56)-H(56)	120.2	C(61)-C(66)-H(66)	119.8
C(56)-C(57)-C(58)	120.6(6)	N(7)-C(67)-C(68)	177.7(13)
C(56)-C(57)-H(57)	119.7	C(67)-C(68)-H(68A)	109.5
C(58)-C(57)-H(57)	119.7	C(67)-C(68)-H(68B)	109.5
C(57)-C(58)-C(53)	120.8(5)	H(68A)-C(68)-H(68B)	109.5
C(57)-C(58)-H(58)	119.6	C(67)-C(68)-H(68C)	109.5
C(53)-C(58)-H(58)	119.6	H(68A)-C(68)-H(68C)	109.5
C(60)-C(59)-Rh(2)	175.7(4)	H(68B)-C(68)-H(68C)	109.5
C(60)-C(59)-Ag(2)	79.7(3)	F(1)-B-F(4)	115.8(8)
Rh(2)-C(59)-Ag(2)	100.67(18)	F(1)-B-F(3)	108.1(7)
C(59)-C(60)-C(61)	166.0(5)	F(4)-B-F(3)	107.1(6)
C(59)-C(60)-Ag(2)	71.6(3)	F(1)-B-F(2)	109.5(5)
C(61)-C(60)-Ag(2)	122.4(3)	F(4)-B-F(2)	109.0(7)
C(66)-C(61)-C(62)	119.2(4)	F(3)-B-F(2)	107.0(7)
C(66)-C(61)-C(60)	119.9(5)		
C(62)-C(61)-C(60)	120.9(4)	Symmetry transformations used to generate	
C(63)-C(62)-C(61)	119.9(5)	equivalent atoms:	
C(63)-C(62)-H(62)	120.0	#1 -x+2,-y+2,-z+2 #2 -x+1,-y+1,-z+1	
C(61)-C(62)-H(62)	120.0		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ag(1)	45(1)	34(1)	35(1)	-17(1)	-10(1)	-14(1)
Ag(2)	27(1)	28(1)	32(1)	-6(1)	-14(1)	-5(1)
Rh(1)	45(1)	28(1)	28(1)	-12(1)	-12(1)	-15(1)
Rh(2)	22(1)	27(1)	25(1)	-8(1)	-9(1)	-6(1)
F(1)	228(7)	88(4)	66(3)	21(3)	-64(4)	-101(4)
F(2)	120(4)	94(3)	47(2)	-9(2)	-21(2)	-77(3)
F(3)	71(3)	110(4)	68(3)	6(3)	-31(2)	-27(3)
F(4)	71(3)	114(4)	75(3)	-42(3)	-44(2)	2(3)
N(1)	45(3)	32(2)	33(2)	-14(2)	-17(2)	-10(2)
N(2)	71(3)	29(2)	35(2)	-11(2)	-24(2)	-13(2)
N(3)	69(3)	45(3)	37(2)	-9(2)	-16(2)	-32(3)
N(4)	37(2)	37(2)	28(2)	-9(2)	-11(2)	-6(2)
N(5)	26(2)	51(3)	40(2)	-10(2)	-16(2)	-12(2)
N(6)	36(2)	36(2)	41(2)	-15(2)	-16(2)	-9(2)
N(7)	88(7)	127(9)	140(9)	-51(7)	-31(6)	-29(6)
C(1)	59(4)	37(3)	36(3)	-17(2)	-19(3)	-12(3)
C(2)	60(4)	31(3)	39(3)	-17(2)	-19(3)	-6(3)
C(3)	88(5)	32(3)	49(3)	-3(2)	-33(3)	-24(3)
C(4)	91(5)	54(4)	40(3)	-4(3)	-23(3)	-43(4)
C(5)	56(4)	61(4)	44(3)	-17(3)	-17(3)	-28(3)
C(6)	54(4)	52(3)	46(3)	-19(3)	-26(3)	-15(3)
C(7)	60(4)	37(3)	40(3)	-12(2)	-24(3)	-11(3)
C(8)	84(5)	30(3)	49(3)	-15(3)	-36(3)	3(3)
C(9)	59(4)	72(4)	42(3)	-18(3)	-10(3)	-35(4)
C(10)	40(3)	30(2)	31(2)	-14(2)	-11(2)	-11(2)
C(11)	54(3)	32(3)	37(3)	-16(2)	-17(3)	-10(2)
C(12)	47(3)	28(3)	43(3)	-14(2)	-11(3)	-5(2)
C(13)	58(4)	52(4)	50(3)	-14(3)	-12(3)	-15(3)
C(14)	62(4)	54(4)	50(4)	-16(3)	-1(3)	-12(3)
C(15)	47(4)	39(3)	72(5)	-16(3)	-5(3)	-11(3)
C(16)	46(4)	68(5)	76(5)	-27(4)	-15(3)	-15(3)

C(17)	51(4)	59(4)	58(4)	-25(3)	-15(3)	-13(3)
C(18)	46(3)	26(2)	39(3)	-15(2)	-10(2)	-14(2)
C(19)	51(3)	31(3)	33(3)	-12(2)	-10(2)	-18(2)
C(20)	39(3)	36(3)	33(3)	-14(2)	-10(2)	-5(2)
C(21)	124(7)	54(4)	44(3)	-7(3)	-41(4)	-38(4)
C(22)	122(7)	62(5)	45(4)	-15(3)	-41(4)	-17(5)
C(23)	57(4)	62(4)	30(3)	-8(3)	-10(3)	12(3)
C(24)	46(3)	44(3)	50(3)	3(3)	-15(3)	0(3)
C(25)	40(3)	36(3)	43(3)	-4(2)	-15(2)	-6(2)
C(26)	39(3)	35(3)	31(2)	-16(2)	-11(2)	-10(2)
C(27)	49(3)	44(3)	38(3)	-16(2)	-14(3)	-15(3)
C(28)	45(3)	45(3)	38(3)	-15(2)	-16(2)	-6(3)
C(29)	57(4)	48(4)	55(4)	-14(3)	-26(3)	-8(3)
C(30)	45(3)	65(4)	54(4)	-11(3)	-20(3)	-8(3)
C(31)	61(4)	46(3)	49(3)	-18(3)	-31(3)	6(3)
C(32)	62(4)	38(3)	43(3)	-15(2)	-27(3)	-3(3)
C(33)	47(3)	37(3)	37(3)	-11(2)	-22(2)	-4(2)
C(34)	40(3)	52(4)	36(3)	-7(3)	-15(2)	0(3)
C(35)	39(3)	60(4)	49(3)	-9(3)	-26(3)	-6(3)
C(36)	40(3)	56(4)	58(4)	-12(3)	-18(3)	-24(3)
C(37)	47(3)	51(4)	65(4)	-23(3)	-24(3)	-18(3)
C(38)	46(3)	53(3)	40(3)	-24(3)	-10(3)	-9(3)
C(39)	44(3)	52(3)	31(3)	-15(2)	-11(2)	-6(3)
C(40)	54(4)	44(3)	33(3)	-3(2)	-14(3)	-17(3)
C(41)	26(3)	76(4)	45(3)	-15(3)	-11(2)	-15(3)
C(42)	56(4)	36(3)	59(4)	-24(3)	-28(3)	1(3)
C(43)	27(2)	30(2)	20(2)	-2(2)	-11(2)	-6(2)
C(44)	28(2)	34(3)	25(2)	-6(2)	-9(2)	-5(2)
C(45)	34(3)	31(3)	34(2)	-11(2)	-13(2)	0(2)
C(46)	45(4)	41(3)	89(5)	-20(3)	-23(4)	-5(3)
C(47)	71(5)	42(4)	95(6)	-19(4)	-42(4)	-6(3)
C(48)	69(5)	35(3)	73(5)	-20(3)	-33(4)	7(3)
C(49)	42(3)	49(4)	63(4)	-19(3)	-20(3)	14(3)
C(50)	36(3)	40(3)	47(3)	-12(2)	-12(2)	-2(2)
C(51)	22(2)	27(2)	35(2)	-10(2)	-9(2)	-8(2)
C(52)	23(2)	33(3)	39(3)	-14(2)	-10(2)	-6(2)

C(53)	21(2)	28(2)	37(3)	-7(2)	-5(2)	-7(2)
C(54)	34(3)	35(3)	47(3)	-16(2)	-6(2)	-9(2)
C(55)	47(3)	31(3)	67(4)	-8(3)	-1(3)	-14(3)
C(56)	55(4)	36(3)	51(4)	2(3)	-3(3)	-11(3)
C(57)	46(3)	43(3)	42(3)	-3(2)	-9(3)	-13(3)
C(58)	35(3)	33(3)	39(3)	-4(2)	-11(2)	-11(2)
C(59)	29(2)	24(2)	26(2)	-10(2)	-10(2)	-7(2)
C(60)	31(2)	26(2)	22(2)	-8(2)	-7(2)	-10(2)
C(61)	23(2)	28(2)	32(2)	-13(2)	-4(2)	-8(2)
C(62)	26(2)	44(3)	34(2)	-18(2)	-6(2)	-10(2)
C(63)	26(2)	52(3)	44(3)	-19(2)	-15(2)	-11(2)
C(64)	24(2)	47(3)	48(3)	-23(3)	1(2)	-14(2)
C(65)	38(3)	36(3)	40(3)	-12(2)	0(2)	-16(2)
C(66)	35(3)	29(2)	33(2)	-7(2)	-8(2)	-8(2)
C(67)	87(8)	104(9)	90(7)	-19(6)	-39(6)	6(6)
C(68)	126(10)	134(11)	116(10)	60(8)	-36(8)	-4(8)
B	92(6)	65(5)	44(4)	-4(3)	-32(4)	-45(5)

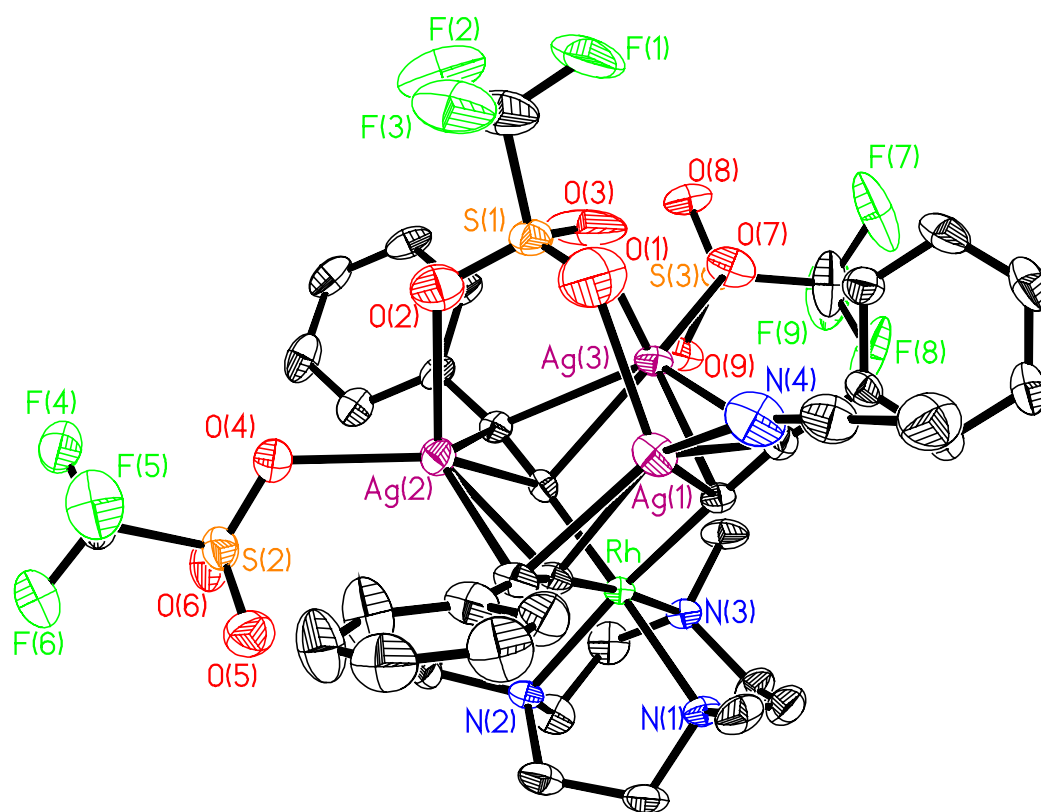
Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **3**.

	x	y	z	U(eq)
H(1A)	9503	7534	7539	49
H(1B)	10379	8019	6833	49
H(2A)	11160	6784	7707	50
H(2B)	11473	7663	7683	50
H(3A)	9375	6680	8722	61
H(3B)	9882	6212	9483	61
H(4A)	7949	6915	10004	66
H(4B)	8665	7216	10411	66
H(5A)	8007	7637	8553	59
H(5B)	6919	8369	8974	59
H(6A)	7756	9470	8247	55
H(6B)	7994	8839	7539	55
H(7A)	9521	9489	6702	63
H(7B)	10467	9412	7090	63
H(7C)	9257	10020	7495	63
H(8A)	11727	6365	9010	79
H(8B)	11018	6862	9858	79
H(8C)	11808	7321	9032	79
H(9A)	6549	8314	10431	80
H(9B)	6899	9210	10052	80
H(9C)	7385	8552	10760	80
H(13)	12639	9111	6492	64
H(14)	14439	9014	5549	72
H(15)	15808	9029	6054	67
H(16)	15416	9060	7539	74
H(17)	13642	9126	8497	65
H(21)	9918	9030	12194	79
H(22)	9846	8479	13630	87
H(23)	9380	7160	14235	69
H(24)	9045	6381	13398	61

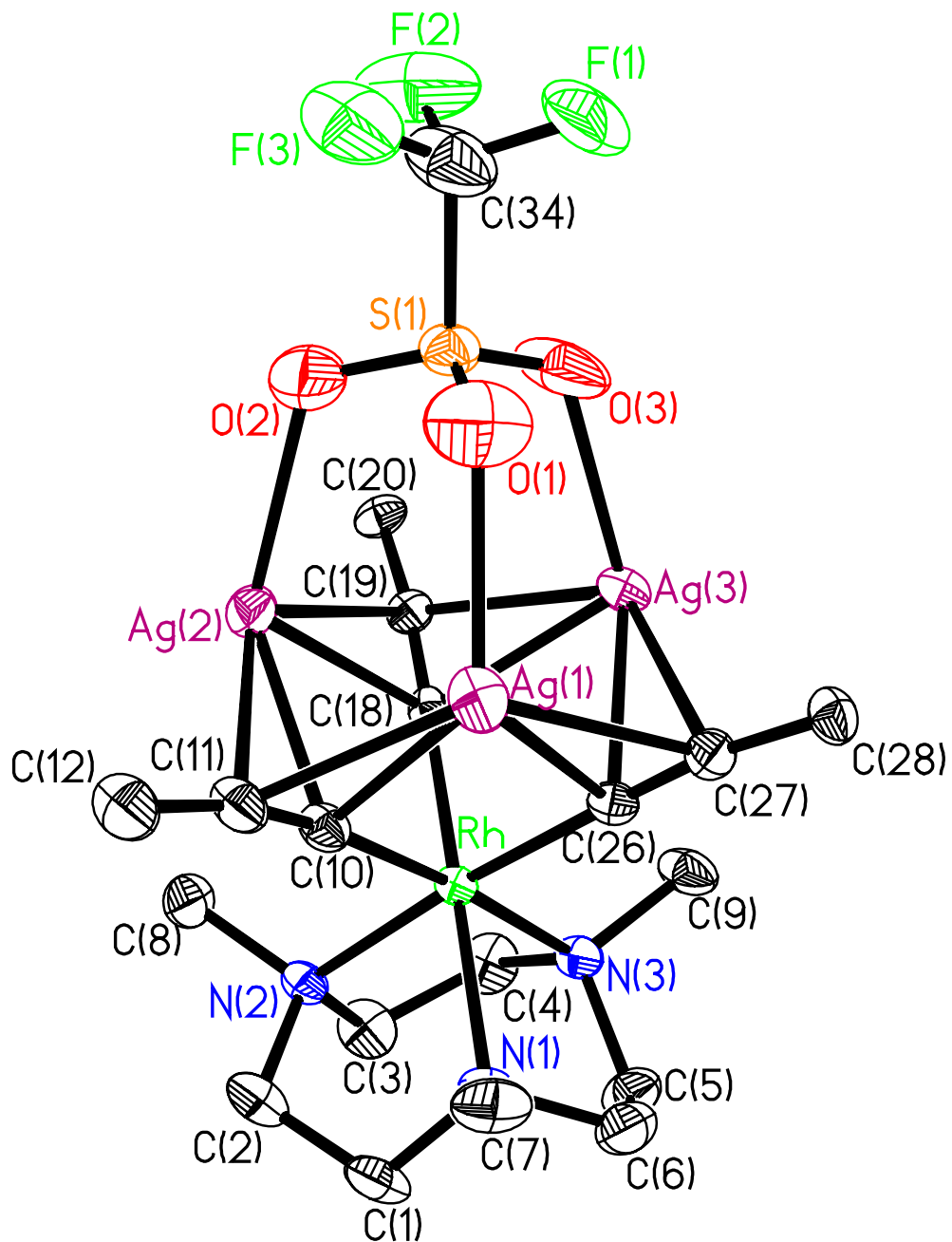
H(25)	9135	6904	11974	48
H(29)	5849	10948	9890	61
H(30)	4509	12266	10282	65
H(31)	5044	13480	10299	61
H(32)	6893	13411	9909	54
H(33)	8236	12121	9517	46
H(34A)	7370	3271	7139	54
H(34B)	7201	3391	8116	54
H(35A)	7729	4673	7683	56
H(35B)	8629	4005	7019	56
H(36A)	7475	6248	6041	56
H(36B)	8158	5877	6703	56
H(37A)	6366	6846	7286	59
H(37B)	6611	5941	7878	59
H(38A)	4164	5594	8110	54
H(38B)	4681	6020	8587	54
H(39A)	4983	4467	8923	51
H(39B)	6086	4790	8584	51
H(40A)	5556	3189	7394	64
H(40B)	4519	3839	7995	64
H(40C)	5381	3137	8407	64
H(41A)	8318	5109	5146	71
H(41B)	8585	4115	5562	71
H(41C)	9190	4773	5657	71
H(42A)	4102	6710	7008	72
H(42B)	5143	7095	6465	72
H(42C)	4512	7245	7469	72
H(46)	5871	2017	5594	69
H(47)	6665	547	5464	79
H(48)	8556	-32	5115	71
H(49)	9640	882	4907	66
H(50)	8879	2356	5107	50
H(54)	6581	7848	4750	47
H(55)	6727	9011	3642	62
H(56)	6507	8951	2335	64
H(57)	6201	7708	2103	54

H(58)	6058	6539	3181	43
H(62)	1987	5827	5906	40
H(63)	109	5884	6466	45
H(64)	-653	5281	7883	47
H(65)	452	4604	8747	47
H(66)	2319	4579	8215	39
H(68A)	2808	8810	2160	219
H(68B)	2924	9116	2965	219
H(68C)	3763	8252	2551	219

X-ray Structure of 4 Asymmetric Unit



Molecule



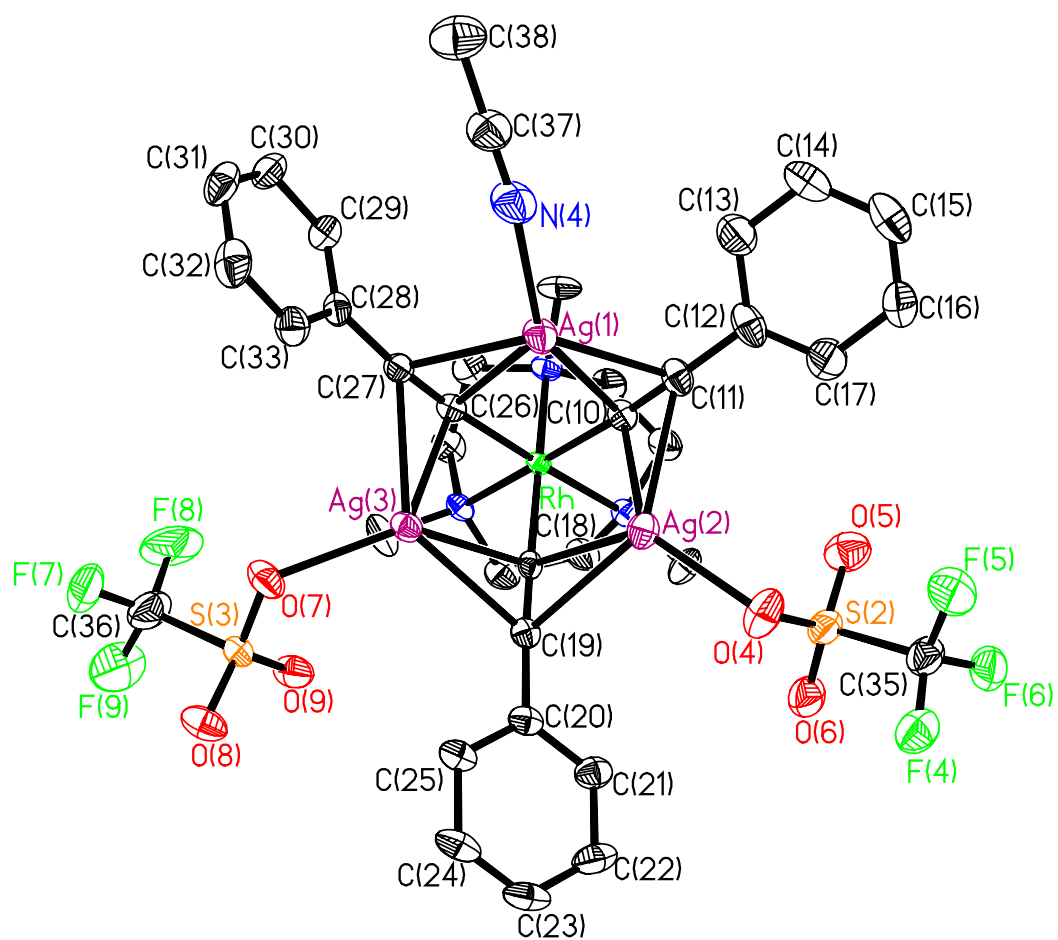


Table 1. Crystal data and structure refinement for **4**.

Identification code	4	
Empirical formula	C ₃₈ H ₃₉ Ag ₃ F ₉ N ₄ O ₉ Rh S ₃	
Formula weight	1389.43	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 21.9776(19) Å	$\alpha = 90^\circ$.
	b = 10.6829(9) Å	$\beta = 113.5970(10)^\circ$.
	c = 21.8444(19) Å	$\gamma = 90^\circ$.
Volume	4699.9(7) Å ³	
Z	4	
Density (calculated)	1.964 Mg/m ³	
Absorption coefficient	1.801 mm ⁻¹	
F(000)	2728	
Crystal size	0.25 x 0.15 x 0.07 mm ³	
Theta range for data collection	1.87 to 27.53°.	
Index ranges	-28 ≤ h ≤ 28, -13 ≤ k ≤ 13, -28 ≤ l ≤ 28	
Reflections collected	40140	
Independent reflections	10738 [R(int) = 0.0380]	
Completeness to theta = 27.53°	99.1 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8843 and 0.7942	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	10738 / 0 / 608	
Goodness-of-fit on F ²	1.099	
Final R indices [I > 2sigma(I)]	R1 = 0.0510, wR2 = 0.1213	
R indices (all data)	R1 = 0.0627, wR2 = 0.1270	
Largest diff. peak and hole	2.085 and -0.946 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ag(1)	3350(1)	4645(1)	-37(1)	31(1)
Ag(2)	2923(1)	4847(1)	1305(1)	28(1)
Ag(3)	1818(1)	6339(1)	-287(1)	25(1)
Rh	1901(1)	3241(1)	-39(1)	16(1)
S(1)	3320(1)	7494(1)	744(1)	31(1)
S(2)	3155(1)	3691(2)	2780(1)	33(1)
S(3)	415(1)	8021(1)	-997(1)	24(1)
F(1)	3757(3)	9559(5)	440(3)	87(2)
F(2)	3546(3)	9617(5)	1357(4)	90(2)
F(3)	4417(2)	8702(5)	1357(3)	76(2)
F(4)	3657(2)	4966(4)	3892(2)	47(1)
F(5)	4381(2)	4012(5)	3622(2)	59(1)
F(6)	3786(2)	2976(4)	4018(2)	50(1)
F(7)	98(3)	8937(6)	-2188(3)	89(2)
F(8)	8(3)	6902(6)	-2152(2)	88(2)
F(9)	-718(2)	8127(7)	-2025(2)	83(2)
O(1)	3662(3)	6800(6)	399(4)	76(2)
O(2)	3404(3)	6880(5)	1363(3)	56(1)
O(3)	2675(3)	7858(5)	294(3)	74(2)
O(4)	3242(3)	4794(4)	2434(2)	46(1)
O(5)	3364(3)	2538(5)	2579(3)	49(1)
O(6)	2536(2)	3625(5)	2844(2)	45(1)
O(7)	1085(2)	7855(4)	-953(2)	38(1)
O(8)	275(2)	9218(4)	-799(2)	36(1)
O(9)	160(2)	6981(4)	-761(2)	39(1)
N(1)	1960(2)	1619(4)	-574(2)	23(1)
N(2)	1672(2)	1909(4)	565(2)	21(1)
N(3)	870(2)	3087(4)	-649(2)	22(1)
N(4)	4069(3)	4823(6)	-514(3)	47(2)
C(1)	1889(3)	496(5)	-211(3)	35(1)
C(2)	2024(3)	758(5)	504(3)	34(1)

C(3)	936(3)	1715(6)	280(3)	33(1)
C(4)	569(3)	2747(6)	-170(3)	34(1)
C(5)	767(3)	2062(6)	-1156(3)	35(1)
C(6)	1387(3)	1747(6)	-1238(3)	36(1)
C(7)	2580(3)	1511(6)	-695(3)	36(1)
C(8)	1908(3)	2230(6)	1284(3)	33(1)
C(9)	546(3)	4249(5)	-1000(3)	35(1)
C(10)	2872(3)	3239(5)	534(3)	23(1)
C(11)	3475(3)	3113(5)	851(3)	29(1)
C(12)	4165(3)	2714(6)	1204(3)	37(1)
C(13)	4543(3)	2445(7)	821(4)	45(2)
C(14)	5190(4)	2006(8)	1148(4)	55(2)
C(15)	5455(3)	1841(7)	1839(4)	52(2)
C(16)	5100(4)	2100(9)	2190(4)	57(2)
C(17)	4434(4)	2557(9)	1866(4)	58(2)
C(18)	1770(2)	4714(5)	463(2)	19(1)
C(19)	1659(2)	5579(5)	779(3)	21(1)
C(20)	1463(2)	6443(5)	1178(3)	24(1)
C(21)	1427(3)	6018(6)	1771(3)	31(1)
C(22)	1234(3)	6825(7)	2151(3)	39(2)
C(23)	1064(3)	8063(6)	1942(3)	36(2)
C(24)	1093(3)	8479(6)	1363(3)	33(1)
C(25)	1297(3)	7675(5)	977(3)	27(1)
C(26)	2098(2)	4387(5)	-663(3)	21(1)
C(27)	2251(3)	5043(5)	-1040(3)	24(1)
C(28)	2443(3)	5639(5)	-1532(3)	25(1)
C(29)	2541(3)	4903(6)	-2006(3)	30(1)
C(30)	2766(3)	5444(7)	-2456(3)	36(1)
C(31)	2886(3)	6709(7)	-2438(3)	40(2)
C(32)	2787(3)	7448(6)	-1963(3)	41(2)
C(33)	2571(3)	6919(6)	-1508(3)	32(1)
C(34)	3797(4)	8901(8)	983(5)	60(2)
C(35)	3775(3)	3924(7)	3621(3)	39(2)
C(36)	-70(4)	7985(10)	-1885(3)	59(2)
C(37)	4303(3)	4719(7)	-893(4)	42(2)
C(38)	4591(4)	4556(8)	-1383(4)	57(2)

Table 3. Bond lengths [\AA] and angles [$^\circ$] for **4**.

Ag(1)-N(4)	2.220(6)	S(3)-O(7)	1.446(4)
Ag(1)-C(10)	2.443(5)	S(3)-C(36)	1.802(7)
Ag(1)-C(11)	2.468(6)	F(1)-C(34)	1.352(10)
Ag(1)-O(1)	2.482(7)	F(2)-C(34)	1.385(11)
Ag(1)-C(26)	2.551(5)	F(3)-C(34)	1.294(9)
Ag(1)-C(27)	2.565(5)	F(4)-C(35)	1.332(8)
Ag(2)-O(4)	2.279(4)	F(5)-C(35)	1.334(7)
Ag(2)-C(10)	2.378(5)	F(6)-C(35)	1.326(8)
Ag(2)-O(2)	2.397(5)	F(7)-C(36)	1.344(11)
Ag(2)-C(18)	2.467(5)	F(8)-C(36)	1.338(11)
Ag(2)-C(11)	2.619(6)	F(9)-C(36)	1.339(9)
Ag(2)-C(19)	2.662(5)	N(1)-C(1)	1.480(7)
Ag(3)-O(7)	2.337(4)	N(1)-C(7)	1.493(7)
Ag(3)-C(26)	2.409(5)	N(1)-C(6)	1.497(7)
Ag(3)-C(18)	2.418(5)	N(2)-C(8)	1.485(7)
Ag(3)-O(3)	2.424(5)	N(2)-C(2)	1.486(7)
Ag(3)-C(27)	2.605(5)	N(2)-C(3)	1.497(7)
Ag(3)-C(19)	2.617(5)	N(3)-C(9)	1.484(7)
Rh-C(10)	1.996(5)	N(3)-C(4)	1.491(7)
Rh-C(26)	2.004(5)	N(3)-C(5)	1.508(7)
Rh-C(18)	2.004(5)	N(4)-C(37)	1.143(9)
Rh-N(1)	2.123(4)	C(1)-C(2)	1.495(9)
Rh-N(3)	2.126(4)	C(1)-H(1A)	0.9900
Rh-N(2)	2.133(4)	C(1)-H(1B)	0.9900
S(1)-O(3)	1.417(5)	C(2)-H(2A)	0.9900
S(1)-O(2)	1.447(6)	C(2)-H(2B)	0.9900
S(1)-O(1)	1.462(6)	C(3)-C(4)	1.481(9)
S(1)-C(34)	1.788(8)	C(3)-H(3A)	0.9900
S(2)-O(6)	1.423(5)	C(3)-H(3B)	0.9900
S(2)-O(5)	1.443(5)	C(4)-H(4A)	0.9900
S(2)-O(4)	1.455(5)	C(4)-H(4B)	0.9900
S(2)-C(35)	1.813(7)	C(5)-C(6)	1.483(9)
S(3)-O(8)	1.423(4)	C(5)-H(5A)	0.9900
S(3)-O(9)	1.430(4)	C(5)-H(5B)	0.9900
		C(6)-H(6A)	0.9900
		C(6)-H(6B)	0.9900

C(7)-H(7A)	0.9800	C(27)-C(28)	1.450(8)
C(7)-H(7B)	0.9800	C(28)-C(29)	1.385(8)
C(7)-H(7C)	0.9800	C(28)-C(33)	1.393(8)
C(8)-H(8A)	0.9800	C(29)-C(30)	1.389(8)
C(8)-H(8B)	0.9800	C(29)-H(29)	0.9500
C(8)-H(8C)	0.9800	C(30)-C(31)	1.374(10)
C(9)-H(9A)	0.9800	C(30)-H(30)	0.9500
C(9)-H(9B)	0.9800	C(31)-C(32)	1.390(10)
C(9)-H(9C)	0.9800	C(31)-H(31)	0.9500
C(10)-C(11)	1.233(8)	C(32)-C(33)	1.382(9)
C(11)-C(12)	1.462(8)	C(32)-H(32)	0.9500
C(12)-C(17)	1.336(10)	C(33)-H(33)	0.9500
C(12)-C(13)	1.423(10)	C(37)-C(38)	1.456(10)
C(13)-C(14)	1.392(10)	C(38)-H(38A)	0.9800
C(13)-H(13)	0.9500	C(38)-H(38B)	0.9800
C(14)-C(15)	1.393(11)	C(38)-H(38C)	0.9800
C(14)-H(14)	0.9500		
C(15)-C(16)	1.326(11)	N(4)-Ag(1)-C(10)	145.5(2)
C(15)-H(15)	0.9500	N(4)-Ag(1)-C(11)	123.3(2)
C(16)-C(17)	1.431(10)	C(10)-Ag(1)-C(11)	29.09(18)
C(16)-H(16)	0.9500	N(4)-Ag(1)-O(1)	87.8(2)
C(17)-H(17)	0.9500	C(10)-Ag(1)-O(1)	118.9(2)
C(18)-C(19)	1.234(7)	C(11)-Ag(1)-O(1)	112.7(2)
C(19)-C(20)	1.449(7)	N(4)-Ag(1)-C(26)	125.1(2)
C(20)-C(25)	1.389(8)	C(10)-Ag(1)-C(26)	67.32(17)
C(20)-C(21)	1.404(8)	C(11)-Ag(1)-C(26)	96.10(17)
C(21)-C(22)	1.378(8)	O(1)-Ag(1)-C(26)	112.3(2)
C(21)-H(21)	0.9500	N(4)-Ag(1)-C(27)	100.9(2)
C(22)-C(23)	1.400(10)	C(10)-Ag(1)-C(27)	95.00(17)
C(22)-H(22)	0.9500	C(11)-Ag(1)-C(27)	123.48(18)
C(23)-C(24)	1.366(9)	O(1)-Ag(1)-C(27)	101.3(2)
C(23)-H(23)	0.9500	C(26)-Ag(1)-C(27)	27.74(17)
C(24)-C(25)	1.399(8)	O(4)-Ag(2)-C(10)	130.96(18)
C(24)-H(24)	0.9500	O(4)-Ag(2)-O(2)	91.55(18)
C(25)-H(25)	0.9500	C(10)-Ag(2)-O(2)	125.94(19)
C(26)-C(27)	1.227(8)	O(4)-Ag(2)-C(18)	125.78(18)

C(10)-Ag(2)-C(18)	71.78(18)	C(10)-Rh-N(2)	93.97(19)
O(2)-Ag(2)-C(18)	113.05(18)	C(26)-Rh-N(2)	175.47(19)
O(4)-Ag(2)-C(11)	113.80(19)	C(18)-Rh-N(2)	93.68(18)
C(10)-Ag(2)-C(11)	28.06(18)	N(1)-Rh-N(2)	82.97(17)
O(2)-Ag(2)-C(11)	113.54(19)	N(3)-Rh-N(2)	83.11(17)
C(18)-Ag(2)-C(11)	99.81(17)	O(3)-S(1)-O(2)	119.4(4)
O(4)-Ag(2)-C(19)	106.92(18)	O(3)-S(1)-O(1)	111.2(4)
C(10)-Ag(2)-C(19)	99.29(17)	O(2)-S(1)-O(1)	110.6(4)
O(2)-Ag(2)-C(19)	96.82(18)	O(3)-S(1)-C(34)	106.2(3)
C(18)-Ag(2)-C(19)	27.51(16)	O(2)-S(1)-C(34)	105.5(4)
C(11)-Ag(2)-C(19)	127.31(17)	O(1)-S(1)-C(34)	102.3(4)
O(7)-Ag(3)-C(26)	126.44(17)	O(6)-S(2)-O(5)	114.9(3)
O(7)-Ag(3)-C(18)	135.49(16)	O(6)-S(2)-O(4)	114.9(3)
C(26)-Ag(3)-C(18)	72.29(18)	O(5)-S(2)-O(4)	114.1(3)
O(7)-Ag(3)-O(3)	93.43(17)	O(6)-S(2)-C(35)	105.5(3)
C(26)-Ag(3)-O(3)	120.49(19)	O(5)-S(2)-C(35)	103.0(3)
C(18)-Ag(3)-O(3)	111.17(19)	O(4)-S(2)-C(35)	102.3(3)
O(7)-Ag(3)-C(27)	108.09(17)	O(8)-S(3)-O(9)	115.1(3)
C(26)-Ag(3)-C(27)	27.97(17)	O(8)-S(3)-O(7)	115.3(3)
C(18)-Ag(3)-C(27)	99.91(17)	O(9)-S(3)-O(7)	114.6(3)
O(3)-Ag(3)-C(27)	106.5(2)	O(8)-S(3)-C(36)	103.8(4)
O(7)-Ag(3)-C(19)	117.68(16)	O(9)-S(3)-C(36)	102.9(4)
C(26)-Ag(3)-C(19)	100.24(17)	O(7)-S(3)-C(36)	102.5(3)
C(18)-Ag(3)-C(19)	28.02(17)	S(1)-O(1)-Ag(1)	123.2(4)
O(3)-Ag(3)-C(19)	95.4(2)	S(1)-O(2)-Ag(2)	118.2(3)
C(27)-Ag(3)-C(19)	127.54(17)	S(1)-O(3)-Ag(3)	122.0(3)
C(10)-Rh-C(26)	87.6(2)	S(2)-O(4)-Ag(2)	122.4(3)
C(10)-Rh-C(18)	90.5(2)	S(3)-O(7)-Ag(3)	123.8(3)
C(26)-Rh-C(18)	90.5(2)	C(1)-N(1)-C(7)	109.4(5)
C(10)-Rh-N(1)	93.4(2)	C(1)-N(1)-C(6)	111.4(5)
C(26)-Rh-N(1)	92.71(19)	C(7)-N(1)-C(6)	108.0(5)
C(18)-Rh-N(1)	175.03(18)	C(1)-N(1)-Rh	108.9(3)
C(10)-Rh-N(3)	175.51(19)	C(7)-N(1)-Rh	114.9(3)
C(26)-Rh-N(3)	95.02(19)	C(6)-N(1)-Rh	104.2(3)
C(18)-Rh-N(3)	93.08(18)	C(8)-N(2)-C(2)	108.2(5)
N(1)-Rh-N(3)	82.91(17)	C(8)-N(2)-C(3)	109.5(4)

C(2)-N(2)-C(3)	111.3(5)	C(6)-C(5)-H(5A)	109.2
C(8)-N(2)-Rh	115.6(3)	N(3)-C(5)-H(5A)	109.2
C(2)-N(2)-Rh	103.7(3)	C(6)-C(5)-H(5B)	109.2
C(3)-N(2)-Rh	108.5(3)	N(3)-C(5)-H(5B)	109.2
C(9)-N(3)-C(4)	108.1(5)	H(5A)-C(5)-H(5B)	107.9
C(9)-N(3)-C(5)	109.2(4)	C(5)-C(6)-N(1)	111.1(5)
C(4)-N(3)-C(5)	111.1(5)	C(5)-C(6)-H(6A)	109.4
C(9)-N(3)-Rh	115.5(3)	N(1)-C(6)-H(6A)	109.4
C(4)-N(3)-Rh	103.9(3)	C(5)-C(6)-H(6B)	109.4
C(5)-N(3)-Rh	109.0(3)	N(1)-C(6)-H(6B)	109.4
C(37)-N(4)-Ag(1)	160.6(6)	H(6A)-C(6)-H(6B)	108.0
N(1)-C(1)-C(2)	112.8(5)	N(1)-C(7)-H(7A)	109.5
N(1)-C(1)-H(1A)	109.0	N(1)-C(7)-H(7B)	109.5
C(2)-C(1)-H(1A)	109.0	H(7A)-C(7)-H(7B)	109.5
N(1)-C(1)-H(1B)	109.0	N(1)-C(7)-H(7C)	109.5
C(2)-C(1)-H(1B)	109.0	H(7A)-C(7)-H(7C)	109.5
H(1A)-C(1)-H(1B)	107.8	H(7B)-C(7)-H(7C)	109.5
N(2)-C(2)-C(1)	110.9(5)	N(2)-C(8)-H(8A)	109.5
N(2)-C(2)-H(2A)	109.5	N(2)-C(8)-H(8B)	109.5
C(1)-C(2)-H(2A)	109.5	H(8A)-C(8)-H(8B)	109.5
N(2)-C(2)-H(2B)	109.5	N(2)-C(8)-H(8C)	109.5
C(1)-C(2)-H(2B)	109.5	H(8A)-C(8)-H(8C)	109.5
H(2A)-C(2)-H(2B)	108.1	H(8B)-C(8)-H(8C)	109.5
C(4)-C(3)-N(2)	112.6(5)	N(3)-C(9)-H(9A)	109.5
C(4)-C(3)-H(3A)	109.1	N(3)-C(9)-H(9B)	109.5
N(2)-C(3)-H(3A)	109.1	H(9A)-C(9)-H(9B)	109.5
C(4)-C(3)-H(3B)	109.1	N(3)-C(9)-H(9C)	109.5
N(2)-C(3)-H(3B)	109.1	H(9A)-C(9)-H(9C)	109.5
H(3A)-C(3)-H(3B)	107.8	H(9B)-C(9)-H(9C)	109.5
C(3)-C(4)-N(3)	111.7(5)	C(11)-C(10)-Rh	172.7(5)
C(3)-C(4)-H(4A)	109.3	C(11)-C(10)-Ag(2)	86.9(4)
N(3)-C(4)-H(4A)	109.3	Rh-C(10)-Ag(2)	100.3(2)
C(3)-C(4)-H(4B)	109.3	C(11)-C(10)-Ag(1)	76.6(4)
N(3)-C(4)-H(4B)	109.3	Rh-C(10)-Ag(1)	104.5(2)
H(4A)-C(4)-H(4B)	107.9	Ag(2)-C(10)-Ag(1)	90.56(18)
C(6)-C(5)-N(3)	112.2(5)	C(10)-C(11)-C(12)	169.3(6)

C(10)-C(11)-Ag(1)	74.3(4)	C(25)-C(20)-C(19)	121.1(5)
C(12)-C(11)-Ag(1)	112.0(4)	C(21)-C(20)-C(19)	119.3(5)
C(10)-C(11)-Ag(2)	65.0(4)	C(22)-C(21)-C(20)	119.9(6)
C(12)-C(11)-Ag(2)	123.1(4)	C(22)-C(21)-H(21)	120.0
Ag(1)-C(11)-Ag(2)	84.62(17)	C(20)-C(21)-H(21)	120.0
C(17)-C(12)-C(13)	120.0(6)	C(21)-C(22)-C(23)	120.1(6)
C(17)-C(12)-C(11)	121.6(6)	C(21)-C(22)-H(22)	120.0
C(13)-C(12)-C(11)	118.3(6)	C(23)-C(22)-H(22)	120.0
C(14)-C(13)-C(12)	118.9(7)	C(24)-C(23)-C(22)	120.2(6)
C(14)-C(13)-H(13)	120.6	C(24)-C(23)-H(23)	119.9
C(12)-C(13)-H(13)	120.6	C(22)-C(23)-H(23)	119.9
C(13)-C(14)-C(15)	119.8(7)	C(23)-C(24)-C(25)	120.3(6)
C(13)-C(14)-H(14)	120.1	C(23)-C(24)-H(24)	119.8
C(15)-C(14)-H(14)	120.1	C(25)-C(24)-H(24)	119.8
C(16)-C(15)-C(14)	120.8(7)	C(20)-C(25)-C(24)	119.8(6)
C(16)-C(15)-H(15)	119.6	C(20)-C(25)-H(25)	120.1
C(14)-C(15)-H(15)	119.6	C(24)-C(25)-H(25)	120.1
C(15)-C(16)-C(17)	120.4(7)	C(27)-C(26)-Rh	176.0(5)
C(15)-C(16)-H(16)	119.8	C(27)-C(26)-Ag(3)	84.9(4)
C(17)-C(16)-H(16)	119.8	Rh-C(26)-Ag(3)	98.2(2)
C(12)-C(17)-C(16)	120.1(7)	C(27)-C(26)-Ag(1)	76.7(3)
C(12)-C(17)-H(17)	120.0	Rh-C(26)-Ag(1)	100.5(2)
C(16)-C(17)-H(17)	120.0	Ag(3)-C(26)-Ag(1)	95.28(17)
C(19)-C(18)-Rh	176.0(4)	C(26)-C(27)-C(28)	171.2(6)
C(19)-C(18)-Ag(3)	85.0(4)	C(26)-C(27)-Ag(1)	75.5(3)
Rh-C(18)-Ag(3)	97.9(2)	C(28)-C(27)-Ag(1)	104.6(3)
C(19)-C(18)-Ag(2)	85.1(3)	C(26)-C(27)-Ag(3)	67.1(3)
Rh-C(18)-Ag(2)	97.13(19)	C(28)-C(27)-Ag(3)	121.5(4)
Ag(3)-C(18)-Ag(2)	98.81(17)	Ag(1)-C(27)-Ag(3)	90.36(17)
C(18)-C(19)-C(20)	170.4(5)	C(29)-C(28)-C(33)	119.7(5)
C(18)-C(19)-Ag(3)	67.0(3)	C(29)-C(28)-C(27)	119.0(5)
C(20)-C(19)-Ag(3)	121.0(4)	C(33)-C(28)-C(27)	121.2(5)
C(18)-C(19)-Ag(2)	67.4(3)	C(28)-C(29)-C(30)	119.9(6)
C(20)-C(19)-Ag(2)	116.0(3)	C(28)-C(29)-H(29)	120.0
Ag(3)-C(19)-Ag(2)	89.30(15)	C(30)-C(29)-H(29)	120.0
C(25)-C(20)-C(21)	119.6(5)	C(31)-C(30)-C(29)	120.6(6)

C(31)-C(30)-H(30)	119.7
C(29)-C(30)-H(30)	119.7
C(30)-C(31)-C(32)	119.6(6)
C(30)-C(31)-H(31)	120.2
C(32)-C(31)-H(31)	120.2
C(33)-C(32)-C(31)	120.4(6)
C(33)-C(32)-H(32)	119.8
C(31)-C(32)-H(32)	119.8
C(32)-C(33)-C(28)	119.8(6)
C(32)-C(33)-H(33)	120.1
C(28)-C(33)-H(33)	120.1
F(3)-C(34)-F(1)	108.8(7)
F(3)-C(34)-F(2)	106.5(8)
F(1)-C(34)-F(2)	109.2(7)
F(3)-C(34)-S(1)	113.1(6)
F(1)-C(34)-S(1)	110.9(6)
F(2)-C(34)-S(1)	108.1(5)
F(6)-C(35)-F(4)	107.7(5)
F(6)-C(35)-F(5)	107.1(6)
F(4)-C(35)-F(5)	108.2(6)
F(6)-C(35)-S(2)	111.5(5)
F(4)-C(35)-S(2)	111.3(5)
F(5)-C(35)-S(2)	110.9(4)
F(8)-C(36)-F(9)	108.1(8)
F(8)-C(36)-F(7)	109.3(6)
F(9)-C(36)-F(7)	106.2(7)
F(8)-C(36)-S(3)	111.3(6)
F(9)-C(36)-S(3)	111.1(5)
F(7)-C(36)-S(3)	110.6(6)
N(4)-C(37)-C(38)	178.4(8)
C(37)-C(38)-H(38A)	109.5
C(37)-C(38)-H(38B)	109.5
H(38A)-C(38)-H(38B)	109.5
C(37)-C(38)-H(38C)	109.5
H(38A)-C(38)-H(38C)	109.5
H(38B)-C(38)-H(38C)	109.5

Symmetry transformations used to generate
equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ag(1)	21(1)	36(1)	36(1)	6(1)	11(1)	-1(1)
Ag(2)	27(1)	30(1)	25(1)	-1(1)	6(1)	-4(1)
Ag(3)	26(1)	18(1)	32(1)	1(1)	13(1)	2(1)
Rh	13(1)	15(1)	20(1)	0(1)	8(1)	0(1)
S(1)	27(1)	28(1)	37(1)	-1(1)	11(1)	-7(1)
S(2)	37(1)	38(1)	27(1)	2(1)	15(1)	-1(1)
S(3)	23(1)	20(1)	29(1)	0(1)	10(1)	2(1)
F(1)	89(4)	54(3)	92(4)	30(3)	9(3)	-34(3)
F(2)	65(3)	56(3)	147(6)	-34(3)	42(4)	-10(3)
F(3)	29(2)	59(3)	119(4)	18(3)	9(3)	-10(2)
F(4)	48(2)	56(3)	36(2)	-5(2)	15(2)	-6(2)
F(5)	29(2)	89(4)	61(3)	6(3)	19(2)	-2(2)
F(6)	45(2)	63(3)	41(2)	15(2)	18(2)	9(2)
F(7)	85(4)	139(5)	63(3)	62(4)	49(3)	55(4)
F(8)	86(4)	131(5)	44(3)	-42(3)	24(3)	11(4)
F(9)	32(2)	160(6)	45(3)	-6(3)	1(2)	12(3)
O(1)	86(5)	69(4)	91(5)	-12(4)	56(4)	-12(4)
O(2)	58(3)	50(3)	55(3)	-2(3)	16(3)	-20(3)
O(3)	36(3)	29(3)	118(5)	15(3)	-11(3)	-7(2)
O(4)	62(3)	41(3)	29(2)	-2(2)	13(2)	-12(2)
O(5)	60(3)	42(3)	51(3)	-4(2)	30(3)	-2(2)
O(6)	33(2)	66(3)	33(2)	0(2)	9(2)	1(2)
O(7)	26(2)	35(2)	53(3)	9(2)	17(2)	9(2)
O(8)	30(2)	24(2)	59(3)	-9(2)	22(2)	-4(2)
O(9)	47(3)	26(2)	56(3)	4(2)	35(2)	2(2)
N(1)	23(2)	18(2)	31(2)	-3(2)	14(2)	-1(2)
N(2)	20(2)	17(2)	27(2)	3(2)	10(2)	-1(2)
N(3)	16(2)	19(2)	27(2)	-1(2)	6(2)	0(2)
N(4)	31(3)	51(4)	60(4)	7(3)	20(3)	-1(3)
C(1)	48(4)	16(3)	47(4)	3(2)	26(3)	1(2)

C(2)	40(3)	21(3)	46(4)	7(3)	22(3)	6(2)
C(3)	21(3)	37(3)	44(3)	4(3)	16(3)	-2(2)
C(4)	18(3)	44(4)	40(3)	0(3)	13(2)	-1(2)
C(5)	31(3)	35(3)	32(3)	-10(3)	5(3)	0(3)
C(6)	38(3)	34(3)	32(3)	-8(3)	10(3)	-2(3)
C(7)	41(3)	30(3)	51(4)	-12(3)	33(3)	-1(3)
C(8)	44(3)	32(3)	25(3)	3(2)	17(3)	-9(3)
C(9)	23(3)	21(3)	47(4)	3(3)	1(3)	0(2)
C(10)	20(2)	20(3)	28(3)	3(2)	10(2)	-2(2)
C(11)	26(3)	23(3)	37(3)	6(2)	10(2)	0(2)
C(12)	21(3)	32(3)	49(4)	6(3)	5(3)	-2(2)
C(13)	41(4)	46(4)	45(4)	-1(3)	13(3)	9(3)
C(14)	38(4)	62(5)	67(5)	3(4)	23(4)	18(4)
C(15)	28(3)	51(4)	65(5)	4(4)	6(3)	6(3)
C(16)	43(4)	84(6)	39(4)	16(4)	10(3)	19(4)
C(17)	35(4)	95(7)	45(4)	14(4)	18(3)	12(4)
C(18)	13(2)	24(3)	19(2)	4(2)	4(2)	-1(2)
C(19)	14(2)	24(3)	24(3)	-1(2)	6(2)	-4(2)
C(20)	15(2)	27(3)	28(3)	-8(2)	7(2)	-3(2)
C(21)	27(3)	36(3)	32(3)	-5(3)	14(2)	-3(2)
C(22)	30(3)	59(4)	33(3)	-15(3)	17(3)	-4(3)
C(23)	23(3)	41(4)	45(4)	-20(3)	15(3)	-1(3)
C(24)	23(3)	26(3)	51(4)	-13(3)	14(3)	-1(2)
C(25)	20(3)	25(3)	33(3)	-8(2)	9(2)	-2(2)
C(26)	20(2)	18(2)	25(3)	-1(2)	10(2)	-2(2)
C(27)	23(3)	24(3)	24(3)	-1(2)	9(2)	2(2)
C(28)	21(3)	26(3)	25(3)	6(2)	7(2)	1(2)
C(29)	33(3)	30(3)	27(3)	1(2)	13(2)	-2(2)
C(30)	32(3)	52(4)	24(3)	5(3)	13(2)	-3(3)
C(31)	29(3)	59(4)	29(3)	12(3)	9(3)	-10(3)
C(32)	37(3)	35(4)	47(4)	13(3)	14(3)	-8(3)
C(33)	35(3)	27(3)	33(3)	2(2)	11(3)	0(2)
C(34)	38(4)	41(4)	93(7)	11(4)	17(4)	-4(3)
C(35)	33(3)	53(4)	31(3)	2(3)	14(3)	-3(3)
C(36)	43(4)	104(7)	27(3)	2(4)	12(3)	16(4)
C(37)	36(4)	41(4)	55(4)	4(3)	23(3)	-5(3)

C(38)	58(5)	53(5)	71(5)	-6(4)	39(4)	-12(4)
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Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for **4**.

	x	y	z	U(eq)
H(1A)	2200	-158	-231	42
H(1B)	1432	164	-438	42
H(2A)	1875	38	694	41
H(2B)	2507	864	761	41
H(3A)	829	918	28	39
H(3B)	787	1646	650	39
H(4A)	569	3490	101	40
H(4B)	102	2489	-422	40
H(5A)	600	1304	-1013	42
H(5B)	426	2331	-1591	42
H(6A)	1486	2411	-1500	43
H(6B)	1326	951	-1488	43
H(7A)	2961	1401	-268	54
H(7B)	2641	2274	-914	54
H(7C)	2546	788	-983	54
H(8A)	1808	1539	1524	49
H(8B)	1684	2991	1335	49
H(8C)	2389	2372	1469	49
H(9A)	68	4103	-1241	52
H(9B)	737	4491	-1318	52
H(9C)	618	4922	-673	52
H(13)	4357	2565	351	55
H(14)	5449	1818	901	66
H(15)	5897	1541	2060	63
H(16)	5287	1980	2661	69
H(17)	4184	2749	2121	69
H(21)	1537	5175	1910	37
H(22)	1215	6541	2555	47
H(23)	928	8615	2204	43
H(24)	974	9318	1223	40

H(25)	1322	7970	577	32
H(29)	2455	4030	-2025	36
H(30)	2836	4936	-2778	43
H(31)	3037	7075	-2749	48
H(32)	2868	8323	-1950	49
H(33)	2509	7427	-1180	39
H(38A)	5022	4138	-1173	85
H(38B)	4651	5376	-1553	85
H(38C)	4294	4043	-1755	85
