

Supplementary File for “Transient chirality in a distal-substituted resorcinarene metal complex”

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1 General Procedures

All chemicals used were bought from Merck or Aldrich. Tetrahydrofuran, pentane, diethylether and toluene were dried over sodium wire/sand and distilled under nitrogen with benzophenone as an indicator. Dichloromethane and acetonitrile were distilled over calcium hydride under nitrogen. Other reagents were purified according to standard procedures.^{1, 2} The molarity of *n*BuLi was determined using a method as described in the literature.³

All reactions were performed under anhydrous conditions and nitrogen or argon atmosphere, unless stated otherwise. Low temperature reactions were performed in a Dewar using dry ice in acetone (−78 °C)

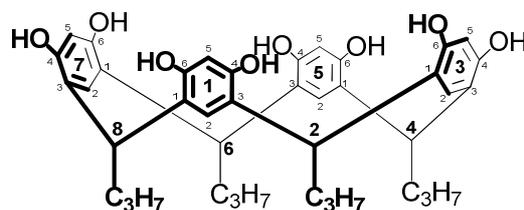
All ¹H and ¹³C nuclear magnetic resonance spectra were obtained using a 300 MHz Varian VNMRS (75 MHz for ¹³C), a 400 MHz Varian Unity Inova (100 MHz for ¹³C) or a 600 MHz Varian Unity Inova (150 MHz for ¹³C). Chloroform-*d* and was used as standard solvent, unless otherwise stated. Chemical shifts (δ) were recorded using the residual chloroform peaks (δ 7.26 in ¹H NMR and δ 77.0 in ¹³C NMR) or the residual DMSO peaks (δ 2.50 in ¹H NMR and δ 39.5 in ¹³C NMR) in DMSO-*d*₆, as reference. All chemical shifts are reported in ppm and all resorcinarene spectra were obtained at 25°C, unless otherwise stated.

All chromatography was performed using either (or a combination of) petrol ether, ethyl acetate, methanol and dichloromethane. Thin layer chromatography (tlc) was carried out on aluminium backed Merck silica gel 60 F₂₅₄ plates. Visualization was achieved with UV lamp, iodine vapour or by spraying with a Cerium Ammonium Molybdate solution (CAM) and then heating. All column chromatography was carried out with Merck silica gel 60 (particle size 0.040-0.063 mm).

Melting points were obtained using a Gallenkamp Melting Point Apparatus and are uncorrected. Infrared spectra were obtained using a Nexus Thermo-Nicolet FT-IR instrument using thin film solutions of chloroform or dichloromethane on NaCl plates, or using the ATR. High resolution mass spectrometry was performed by the CAF (Central Analytical Facility) Institute at Stellenbosch University using a Waters API Q-TOF Ultima spectrometer. Routine mass spectrometry was performed using a Waters API Quattro Micro spectrometer. In both cases ESI+ was used as ionisation method.

2 Compounds

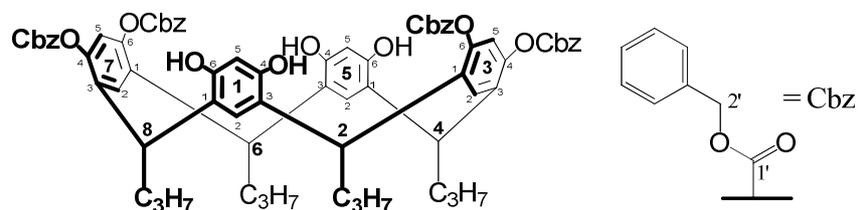
1⁴,1⁶,3⁴,3⁶,5⁴,5⁶,7⁴,7⁶-Octahydroxy-2,4,6,8-tetrapropyl-1,3,5,7(1,3)-tetrabenzenacyclooctaphane (1)⁴



Resorcinol (8.26 g, 75 mmol) was dissolved in dry dichloromethane (250 ml). The solution was cooled to 0 °C and butanal (6.76 ml, 75 mmol) was added. Boron trifluoride diethyl ether complex (19.4 ml, 153 mmol) was added slowly over a period of 30 minutes, via a syringe pump, to the solution and the reaction was allowed to warm to room temperature, and left stirring for 26 hours. The solution formed a red colour, with a light pink precipitate. The resulting precipitate was filtered off and washed with dichloromethane to yield a light pink product. The precipitate was dried on high vacuum for 12 hours to leave the tetrapropyl resorcinarene **1** (8.97 g, 73%).

mp >350 °C(dec) (from Water/Ethanol); δ_{H} (400 MHz; DMSO-D₆; 25 °C) δ 0.89 (12H, t, *J* 7.3, -(CH₂)₂CH₃), 1.19 (8H, s, *J* 7.3, -CH₂CH₂CH₃), 2.08 (8H, m, -CH₂CH₂CH₃), 4.22 (4H, t, *J* 7.9, H-2,4,6,8), 6.14 (4H, s, H-1²,3²,5²,7²), 7.24 (4H, s, H-1⁵,3⁵,5⁵,7⁵) and 8.92 (8H, s, Ar-OH)

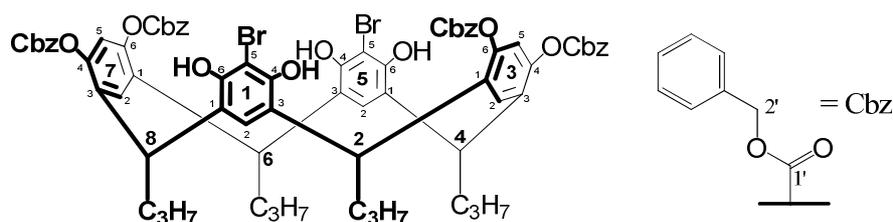
3⁴,3⁶,7⁴,7⁶-Tetra(benzyloxycarbonyloxy)-1⁴,1⁶,5⁴,5⁶-tetrahydroxy-2,4,6,8-tetrapropyl-1,3,5,7(1,3)-tetrabenzenacyclooctaphane (2)



Resorcinarene **1** (1.98 g, 3 mmol) was dissolved in dry acetonitrile (30 ml) at room temperature and to this mixture was added triethylamine (1.68 ml, 12 mmol) and the resulting pink suspension was stirred for 20 minutes after which benzyl chloroformate (1.70 ml, 12 mmol) was added. The solution turned light red/orange within 5 minutes and the reaction was left stirring for 48 hours at room temperature. The resulting light pink precipitate was collected via filtration and washed with cold portions of acetonitrile. The precipitate was taken up in 1M HCl (25 ml) and extracted with dichloromethane (4×30 ml), the organic phases combined and dried over anhydrous magnesium sulphate. The drying agent was filtered off and the solvent removed under reduced pressure to leave a fine white solid, the tetraCBz resorcinarene **2** (0.94 g, 27%).

mp 170 °C (from dichloromethane/petroleum ether); *R_f* = 0.13 (ethyl acetate/petroleum ether, 2:3); ν_{max} (film)/cm⁻¹ 3426, 2956, 2871, 1759, 1620, 1493, 1267, 1224, 911 and 736; δ_{H} (400 MHz; CDCl₃; 25 °C) δ 0.89 (12H, t, *J* 7.3, -(CH₂)₂CH₃), 1.13–1.22 (4H, m, -CH₂CH₂CH₃), 1.29–1.40 (4H, m, -CH₂CH₂CH₃), 1.74–1.83 (4H, m, -CH₂CH₂CH₃), 1.92–2.00 (4H, m, -CH₂CH₂CH₃), 4.33 (4H, br t, *J*, H-2,4,6,8), 5.21–5.27 (8H, m, H-2'), 5.94 (2H, s, H-1⁵,5⁵), 6.67 (2H, br s, H-3⁵,7⁵), 6.71 (4H, s, Ar-OH), 6.92 (2H, s, H-1²,5²), 7.12 (2H, s, H-3²,5²) and 7.3–7.36 (20H, m, Ph); δ_{C} (100 MHz; CDCl₃; 25 °C): δ 14.1 (-(CH₂)₂CH₃), 20.9 (-CH₂CH₂CH₃), 35.0 (C-2,4,6,8), 37.4 (-CH₂CH₂CH₃), 70.9 (C-2'), 102.9 (C-1⁵,5⁵), 115.1 (C-1²,5²), 118.6 (C-3²,7²), 124.1 (C-3¹,3³,7¹,7³), 127.9 (C-3⁵,7⁵), 128.6 (Ph), 134.5 (Ph), 136.2 (C-1¹,1³,5¹,5³), 146.0 (C-3⁴,3⁶,7⁴,7⁶), 153.6 (C-1') and 153.9 (C-1⁴,1⁶,5⁴,5⁶); *m/z* (ESI+) 1193.4918 (M+H. C₇₂H₇₃O₁₆ requires 1193.4899), 1211 (M+H₂O, 100%) and 1193 (M, 22).

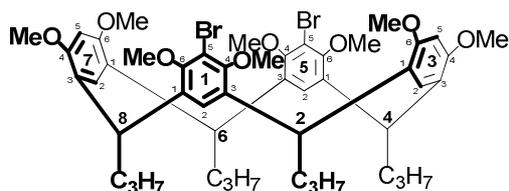
3^{4,3^{6,7^{4,7⁶}}-Tetra(benzyloxycarbonyloxy)-1^{5,5}-dibromo-1^{4,1^{6,5^{4,5⁶}}-tetrahydroxy-2,4,6,8-tetrapropyl-1,3,5,7(1,3)-tetrabenzenacyclooctaphane (3)}}



Resorcinarene **2** (2.04 g, 1.7 mmol) was dissolved in dry dichloromethane (85.5 ml) and cooled to $-78\text{ }^{\circ}\text{C}$. To this solution was added, via a dropping funnel, a solution of 1M bromine in acetic acid (6 ml, 6 mmol). The reaction was stirred for 30 minutes after which another equivalent of the 1M bromine in acetic acid (1.7 ml, 1.7 mmol) was added and the reaction was stirred for another 30 minutes until completion (monitored with tlc). The reaction mixture was quenched with a 10% sodium thiosulphate (w/v, 60 ml) solution and warmed to room temperature. The reaction mixture was neutralized with a 10% sodium carbonate (w/v, 100 ml) solution, carried over to a separatory funnel and the organic phase separated. The water layer was extracted with dichloromethane (3x60 ml), the organic phases combined and dried over anhydrous magnesium sulphate. The drying agent was filtered off and the solvent removed under reduced pressure to leave yellow-orange foam (2.31 g, >100%). The product was purified using flash column chromatography (silica gel eluting with ethyl acetate: petroleum ether 3:17 followed by ethyl acetate: petroleum ether 1:4). The resulting product was recrystallized in petroleum ether/dichloromethane to yield fine pale white crystals of dibromo-tetraCBz-resorcinarene **3** (1.85 g, 80%).

mp $220\text{ }^{\circ}\text{C}$ (from dichloromethane/petroleum ether); $R_f = 0.56$ (ethyl acetate/petroleum ether, 2:3); $\nu_{\text{max}}(\text{ATR})/\text{cm}^{-1}$ 3508, 3454, 2956, 2871, 1765, 1744, 1610, 1214, 971 and 696; $\delta_{\text{H}}(400\text{ MHz; CDCl}_3; 25\text{ }^{\circ}\text{C})$ δ 0.88 (12H, t, J 7.3, $-(\text{CH}_2)_2\text{CH}_3$), 1.14–1.24 (4H, m, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 1.28–1.37 (4H, m, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 1.71–1.80 (4H, m, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 1.88–1.97 (4H, m, $-\text{CH}_2\text{CH}_2\text{CH}_6$), 4.33 (4H, t, J 7.6, H-2,4,6,8), 5.22 (4H, s, Ar-OH), 5.30 (8H, m, H-2'), 6.53 (2H, s, H-3^{5,7⁵}), 7.08 (2H, s, H-1^{2,5²}), 7.11 (2H, s, H-3^{2,7²}) and 7.34–7.47 (20H, m, Ph); $\delta_{\text{C}}(100\text{ MHz; CDCl}_3; 25\text{ }^{\circ}\text{C})$: δ 13.9 ($-(\text{CH}_2)_2\text{CH}_3$), 20.7 ($-\text{CH}_2\text{CH}_2\text{CH}_3$), 35.8 (C-2,4,6,8), 37.4 ($-\text{CH}_2\text{CH}_2\text{CH}_3$), 70.5 (C-2'), 100.6 (C-1^{5,5}), 115.4 (C-3^{5,7⁵}), 120.2 (C-3^{1,3^{3,7^{1,7⁵}}), 123.6 (C-1^{2,5²}), 126.8 (C-3^{2,7²}), 128.6 (Ph), 128.7 (Ph), 128.8 (Ph), 134.8 (Ph), 135.5 (C-1^{1,3^{1,5^{1,5³}}), 146.4 (C-3^{4,3^{6,7^{4,7⁶}}), 149.2 (C-1^{4,1^{6,5^{4,5⁶}}) and 153.8 (C-1'); m/z (ESI+) 1366.3375 (M+NH₄. C₇₂H₇₄O₁₆NBr₂ requires 1366.3374), 1368.33 (M+NH₄, 100%).}}}}

**1⁵,5⁵-Dibromo-1⁴,1⁶,3⁴,3⁶,5⁴,5⁶,7⁴,7⁶-octamethoxy-2,4,6,8-tetrapropyl-1,3,5,7(1,3)-
tetrabenzenacyclooctaphane (4)**

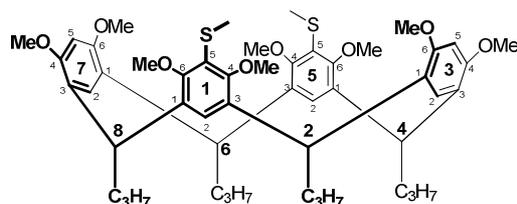


To a mixture of resorcinarene **3** (1.80 g, 1.3 mmol) in ethanol/THF (1:1 v/v, 75 ml) was added Pd/C (10%, 0.138 g, 0.13 mmol) and a hydrogen-filled balloon connected. The mixture was stirred at room temperature in a waterbath for 24 hours after which another equivalent of Pd/C (10%, 0.138 g, 0.13 mmol) was added and stirred until completion as monitored by tlc (24 hours). The mixture was filtered over a Celite plug and washed with ethanol and THF. The solvent was removed under reduced pressure to leave a solid (1.22 g, >100%).

The solid and potassium carbonate (3.31 g, 24 mmol) were suspended in dry acetonitrile (40 ml) and dimethyl sulphate (2.29 ml, 24 mmol) was added. The solution was heated to reflux for 42 hours. The reaction was stopped and cooled to room temperature. The solvent was removed under reduced pressure and the resulting solid added to H₂O (50 ml) and extracted with dichloromethane (3×50 ml). The organic layers were combined and dried over anhydrous magnesium sulphate. The drying agent was filtered off and the solvent removed under reduced pressure to leave an orange solid. The product was purified using flash column chromatography (silica gel eluting with dichloromethane followed by dichloromethane: methanol 99:1). The resulting product was recrystallized in boiling ethanol to yield white crystals (0.94 g, 75% over 2 steps).

mp 209–210 °C (from ethanol/dichloromethane); R_f = 0.62 (ethyl acetate/petroleum ether, 2:3); ν_{\max} (ATR)/cm⁻¹ 2933, 2871, 1613, 1582, 1237, 1081 and 1037; δ_{H} (400 MHz; CDCl₃; 25 °C) δ 0.93 (12H, t, J 7.3, $-(\text{CH}_2)_2\text{CH}_3$), 1.34 (8H, m, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 1.82 (8H, m, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 3.60 (12H, s, Ar-OCH₃), 3.68 (12H, s, Ar-OCH₃), 4.51 (4H, t, J 7.4, H-2,4,6,8), 6.30 (2H, s, H-3⁵,7⁵), 6.55 (2H, s, H-1²,5²) and 6.75 (2H, s, H-3²,7²); δ_{C} (100 MHz; CDCl₃; 25 °C): δ 14.2 ($-(\text{CH}_2)_2\text{CH}_3$), 21.2 ($-\text{CH}_2\text{CH}_2\text{CH}_3$), 36.5 (C-2,4,6,8), 37.5 ($-\text{CH}_2\text{CH}_2\text{CH}_3$), 55.6 (Ar-OCH₃), 60.3 (Ar-OCH₃), 96.3 (C-3⁵,7⁵), 112.5 (C-1⁵,5⁵), 124.7 (C-3¹,3³,7¹,7⁵), 125.0 (C-1²,5²), 126.4 (C-3²,7²), 136.1 (C-1¹,1³,5¹,5³), 153.6 (C-1⁴,1⁶,5⁴,5⁶) and 156.1 (C-3⁴,3⁶,7⁴,7⁶); m/z (ESI+) 942.316 (M+NH₄⁺, C₄₈H₆₆O₁₆NBr₂ requires 942.3155), 944.3 (M+NH₄⁺, 100%).

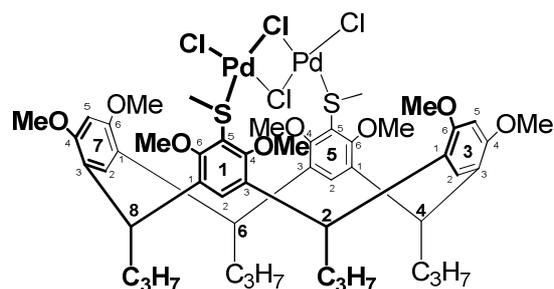
**1^{4,1⁶,3⁴,3⁶,5⁴,5⁶,7⁴,7⁶}-Octamethoxy-1^{5,5}-dimethylthiyl-2,4,6,8-tetrapropyl-1,3,5,7(1,3)-
tetrabenzenacyclooctaphane (5)**



Resorcinarene **4** (350 mg, 0.38 mmol) was dissolved in tetrahydrofuran (19 ml). After cooling *n*BuLi (0.84 ml, 0.83 mmol, 0.99 M solution in hexane) was added. The reaction was quenched with dimethyl disulfide (0.15 ml, 1.66 mmol), warmed to room temperature and stirred overnight. H₂O (15 ml) was added and the product extracted into ethyl acetate (3×30 ml). The organic phases were combined and dried over anhydrous magnesium sulphate. The drying agent was filtered off and the solvent removed under reduced pressure to leave a white solid. Final purification was achieved by flash column chromatography (silica gel eluting with ethyl acetate: petroleum ether 3:22 followed by ethyl acetate: petroleum ether 3:17 followed by ethyl acetate: petroleum ether 1:3) to leave a white solid. Final purification was achieved by recrystallisation in ethanol/dichloromethane to yield the dithioether **5** (228 mg, 70%).

mp 208–209 °C (from ethanol/dichloromethane); *R*_f = 0.64 (ethyl acetate/petroleum ether, 2:3); *v*_{max}(ATR)/cm⁻¹ 2952, 2930, 2869, 1611, 1582, 1409, 1296, 1010, 915 and 812; δ_H(300 MHz; CDCl₃; 25 °C) δ 0.93 (12H, t, *J* 7.4, -(CH₂)₂CH₃), 1.29–1.37 (8H, m, -CH₂CH₂CH₃), 1.78–1.84 (8H, m, -CH₂CH₂CH₃), 2.33 (6H, s, -SCH₃), 3.59 (12H, s, Ar-OCH₃), 3.64 (12H, s, Ar-OCH₃), 4.54 (4H, t, *J* 7.6, H-2,4,6,8), 6.35 (2H, s, H-3⁵,7⁵), 6.69 (2H, s, H-1²,5²) and 6.71 (2H, s, H-3²,7²); δ_C(75.5 MHz; CDCl₃; 25 °C): δ 14.2 (-(CH₂)₂CH₃), 18.1 (-CH₂CH₂CH₃), 21.2 (Ar-SCH₃), 35.9 (C-2,4,6,8), 37.7 (-CH₂CH₂CH₃), 55.7 (Ar-OCH₃), 60.4 (Ar-OCH₃), 96.3 (C-3⁵,7⁵), 122.9 (C-1⁵,5⁵), 125.6 (C-3¹,3³,7¹,7⁵), 128.9 (C-1²,5²), 126.3 (C-3²,7²), 134.6 (C-1¹,1³,5¹,5³), 155.8 (C-1⁴,1⁶,5⁴,5⁶) and 157.2 (C-3⁴,3⁶,7⁴,7⁶); *m/z* (ESI+) 878.4676 (M+NH₄⁺, C₅₀H₇₂O₈NS₂ requires 878.4699), 878.47 (M+NH₄⁺, 100%).

S,S'-{1⁴,1⁶,3⁴,3⁶,5⁴,5⁶,7⁴,7⁶-octamethoxy-1⁵,5⁵-dimethylthiyl-2,4,6,8-tetrapropyl-1,3,5,7(1,3)-tetrabenzenacyclooctaphane}-bis[palladium chloride(μ₂-chlorine)₂] (6)



To resorcinarene **5** (75 mg, 0.087 mmol) dissolved in dry dichloromethane (3 ml) was added palladium(II) chloride (30.5 mg, 0.174 mmol) and the resulting brown reaction stirred for 24 hours at room temperature. The solution was filtered through Celite and carefully washed with dichloromethane. The solvent was evaporated and the brown solid was re-dissolved in dichloromethane, layered with pentane and placed in a refrigerator at $-15\text{ }^{\circ}\text{C}$. After a few days small brown crystals formed and were collected by filtration and dried to give the dipalladium complex **6** (74 mg, 70%).

mp $239\text{--}243\text{ }^{\circ}\text{C}$ (dec) (from dichloromethane/pentane); $\nu_{\text{max}}(\text{ATR})/\text{cm}^{-1}$ 2949, 2869, 1610, 1581, 1288, 1160, 966 and 816; $\delta_{\text{H}}(400\text{ MHz}; \text{CDCl}_3; 50\text{ }^{\circ}\text{C})$ δ 0.88 (12H, t, J 7.3, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 1.16–1.27 (8H, m, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 1.82 (8H, q, J 7.5, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 2.55 (6H, s, $-\text{SCH}_3$), 3.60 (12H, br s, $\text{Ar}-\text{OCH}_3$), 4.00 (12H, s, $\text{Ar}-\text{OCH}_3$), 4.67 (4H, t, J 7.5, H-2,4,6,8), 6.35 (2H, s, H-3²,7²), 6.61 (2H, s, H-3⁵,7⁵) and 7.57 (2H, s, H-1²,5²); $\delta_{\text{C}}(100\text{ MHz}; \text{CDCl}_3; 50\text{ }^{\circ}\text{C})$: δ 13.9 ($-\text{CH}_2\text{CH}_2\text{CH}_3$), 21.0 ($-\text{CH}_2\text{CH}_2\text{CH}_3$), 23.6 ($-\text{SCH}_3$), 35.7 (C-2,4,6,8), 38.2 ($-\text{CH}_2\text{CH}_2\text{CH}_3$), 55.7–55.9 ($\text{Ar}-\text{OCH}_3$), 62.1, 95.3 (C-3⁵,7⁵), 115.9 (C-1¹,3¹,5¹,5³), 125.8–125.9 (C-3²,7²), 126.8 (C-3¹,3³,7¹,7⁵), 132.6 (C-1²,5²), 155.6 (C-3⁴,3⁶,7⁴,7⁶) and 157.8 (C-1⁴,1⁶,5⁴,5⁶);

3 Crystal Structure

A single crystal was covered in a small amount of paratone oil and mounted on a glass fibre. X-ray intensity data were collected at 100 K on a Bruker SMART APEX CCD with 1.75 kW graphite monochromated Mo radiation. The detector to crystal distance was 60 mm. Data was collected by omega scans. The data were scaled and reduced using the *APEXII* software suite. Unit cell dimensions were refined on all data and the space group was assigned on the basis of systematic absences and intensity statistics. The structure was solved and refined using *SHELX97*.⁵ Hydrogen atoms are placed in calculated positions and included in the model during later stages of the refinement. The program *X-SEED*,⁶ an interface to *SHELX*, was used during the structure solution and refinements.

**S,S'-{1⁴,1⁶,3⁴,3⁶,5⁴,5⁶,7⁴,7⁶-octamethoxy-1⁵,5⁵-dimethylthiyl-2,4,6,8-tetrapropyl-1,3,5,7(1,3)-
tetrabenzenacyclooctaphane}-bis[palladium chloride(μ_2 -chlorine)₂] (6)**

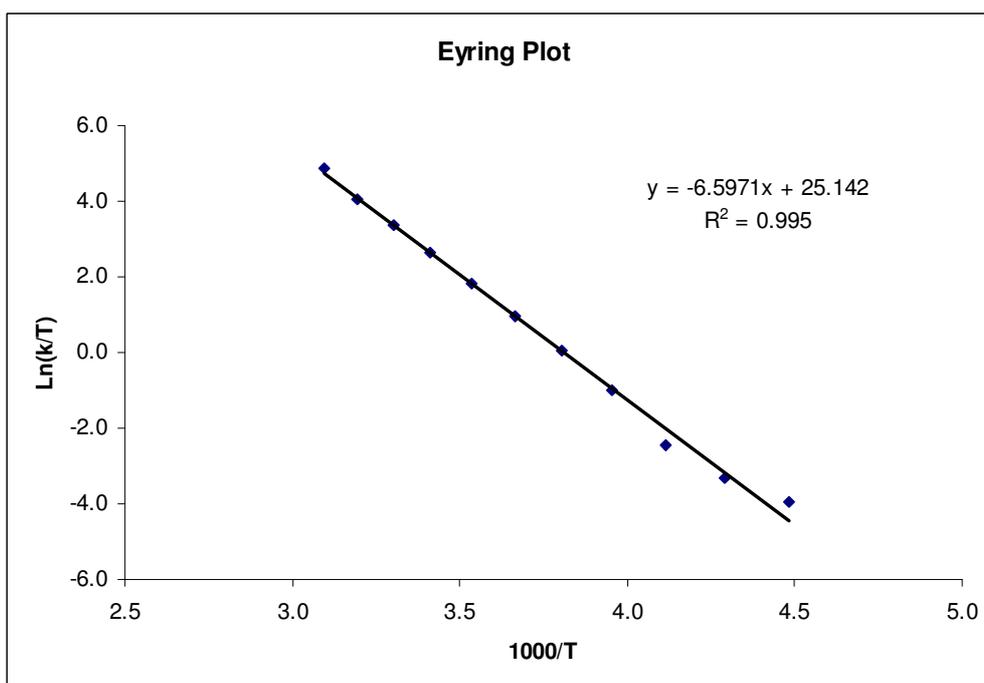
Empirical formula	C _{51.50} H ₇₁ Cl ₇ O ₈ Pd ₂ S ₂	
Formula weight	1343.15	
Temperature (K)	173(2)	
Wavelength (Å)	0.71073	
Crystal system	triclinic	
Space group	P-1	
Unit cell dimensions (Å, °)	$a = 13.4931(11)$	$\alpha = 81.372(1)$
	$b = 14.4968(12)$	$\beta = 84.347(1)$
	$c = 15.5364(12)$	$\gamma = 70.855(1)$
Volume (Å ³)	2834.5(4)	
Z	2	
Calculated density (g cm ⁻³)	1.574	
Absorption coefficient (mm ⁻¹)	1.088	
F_{000}	1374	
Crystal size (mm ³)	0.23 × 0.21 × 0.15	
θ range for data collection (°)	1.50 to 28.15	
Miller index ranges	$-17 \leq h \leq 16, -19 \leq k \leq 19, -19 \leq l \leq 19$	
Reflections collected	32160	
Independent reflections	12633 [$R_{\text{int}} = 0.0321$]	
Completeness to θ_{max} (%)	91.0	
Max. and min. transmission	0.8538 and 0.7879	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12633 / 0 / 686	
Goodness-of-fit on F^2	1.026	
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0359, wR2 = 0.0783$	
R indices (all data)	$R1 = 0.0549, wR2 = 0.0867$	
Largest diff. peak and hole (e Å ⁻³)	0.797 and -1.035	

4 Thermodynamic calculations

T (K)	k (s ⁻¹)	ΔG
323	42000	50.7
313	18000	51.3
303	8900	51.3
293	4100	51.5
283	1760	51.6
273	719	51.7
263	280	51.8
253	95	52.0
243	21	53.0
233	8.5	52.4
223	4.2	51.4
average ΔG		51.9
standard deviation		0.6

$$\Delta G = RT \left[\ln \left(\frac{k_b T}{h} \right) - \ln(k_{rate}) \right]$$

$$\ln \left(\frac{k_{rate}}{T} \right) = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} + \ln \left(\frac{k_b}{h} \right)$$



$$\frac{\Delta H}{R} = -slope$$

$$\Delta H = 6.5971 \times 8.31451$$

$$\Delta H = 54.9 \text{ kJ/mol}$$

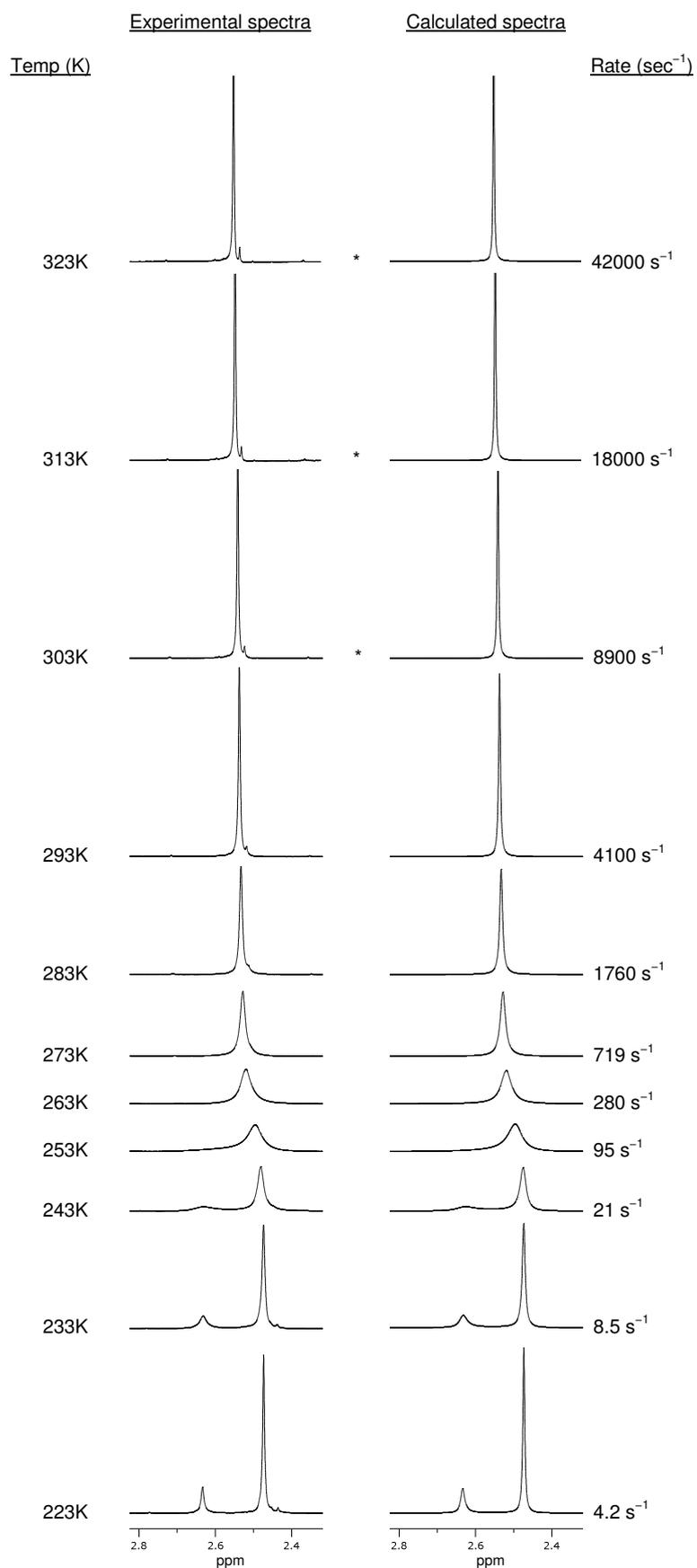
$$\frac{\Delta S}{R} + \ln \left(\frac{k_b}{h} \right) = 25.142$$

$$\Delta S = (25.142 - 23.760) \times 8.31451$$

$$\Delta S = 11.5 \text{ J/mol/K}$$

Graph was plotted in MS Excel 2003 and errors were determined using standard formulae

5 **Figure S1 – Comparison of experimental and calculated spectra**



*These spectra have been cropped in order to fit this figure

6 **References**

1. K. Fairfull-Smith, P. M. J. Redon, J. W. Haycock and N. H. Williams, *Tetrahedron Lett.*, 2007, **48**, 1317-1319.
2. A. I. Vogel, B. S. Furniss, A. J. Hannaford, V. Rogers, P. W. G. Smith and A. R. Tatchell, *Vogel's Textbook of Practical Organic Chemistry*, 5 edn., Longman Scientific & Technical, London, 1989.
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