Journal of Organometallic Chemistry 693 (2008) 3711–3714

Contents lists available at ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

Spanning $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ metalloligands with 1,4-dimercurated durene

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ARTICLE INFO

Article history:
Received 24 April 2008
Received in revised form 2 September 2008
Accepted 2 September 2008
Available online 10 September 2008

Keywords:
Platinum complexes
Organomercury complexes
Sulfide complexes
Crystal structure

ABSTRACT

Reaction of the metalloligand $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ with 0.5 mol equivalents of durene-1,4-bis(mercuric acetate) $[AcOHgC_6Me_4HgOAc]$ in methanol gives the polynuclear complex $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)]^{2+}$, isolated as its PF_6^- and BPh_4^- salts. Positive-ion ESI mass spectra indicate that $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)]^{2+}$ undergoes fragmentation by successive loss of PPh_3 ligands, while the ESI mass spectrum of the BPh_4^- salt showed additional ions $[Pt_2(\mu\text{-}S)_2(PPh_3)_4(HgC_6Me_4HgPh)]^+$ and $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgPh]^+$ as a result of phenyl transfer from BPh_4^- to Hg. A single-crystal X-ray structure determination on $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)](BPh_4)_2$ shows that the cation crystallises on a centre of symmetry, with structural features that are comparable to those of the previously characterised complex $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgPh]BPh_4$.

1. Introduction

The ability of the platinum(II) sulfido complex $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ **1** to act as a metalloligand is well established [1]. Closely related analogues with alternative phosphine ligands are known to behave in the same way [2–5]. Through such reactions, larger homo- and hetero-metallic sulfido-bridged aggregates of higher nuclearities can easily be assembled. We are interested in developing systematic methods for assembling larger assemblies containing $\{Pt_2S_2\}$ cores as a basic building block, because such assemblies are relatively rare [2,6,7], and reasoned that the use of organomercury complexes could offer a strategy to achieve this goal. A range of organomercury complexes has previously been reacted with $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$, using electrospray ionisation mass spectrometry (ESI MS) as a powerful tool to screen reactions for the successful formation of (cationic) products [8]. Isolated complexes include the simple adducts $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgPh]BPh_4$ **2** [8], $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgEt]PF_6$ [8], $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgFc]PF_6$ [9] $[Fc = Fe(\eta^5\text{-}C_5H_5)(\eta^5\text{-}C_5H_4)]$, the dimercurated ferrocene derivative $[Pt_2(\mu\text{-}S)_2(PPh_3)_4Hg_2Fc^*](PF_6)_2$ [9] $[Fc^* = Fe(\eta^5\text{-}C_5H_4)_2]$ and the organomercury adducts of the monobenzylated $\{Pt_2S_2\}$ core, $[Pt_2(\mu\text{-}SCH_2Ph)(\mu\text{-}SHgR)(PPh_3)_4](PF_6)_2$ (R = Ph, Fc).[10] In this paper, we describe the reactivity of $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ towards dimercurated durene (1,2,4,5-tetramethylbenzene), $AcOHgC_6Me_4HgOAc$ **3**, which leads to the formation of hexametallic assemblies of two $\{Pt_2S_2\}$ units spanned by the dimercurated arene moiety $[HgC_6Me_4Hg]^{2+}$. The availability of a wide range of poly-mercurated organic [11,12] and organometallic [13,14] compounds suggests that the assembly of even larger $\{Pt_2S_2\}$-Hg aggregates might ultimately be possible by this methodology.

2. Results and discussion

The reaction of a methanol suspension of $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ **1** with 0.5 mol equivalents of durene-1,4-bis(mercuric acetate) **3** leads to fairly rapid formation of a bright yellow solution which was shown by positive-ion ESI MS to contain almost exclusively the cation $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)]^{2+}$ [observed m/z 1769.754, calculated m/z 1769.755]. This complex contains two $\{Pt_2S_2\}$ metalloligands bridged by the $[p\text{-}HgC_6Me_4Hg]^{2+}$ fragment. The product was readily isolated from solution as its hexafluorophosphate salt **4a** by addition of excess NH_4PF_6 to the filtered reaction solution. The positive-ion ESI mass spectrum of isolated **4a** in CH_2Cl_2–CH_3OH showed exclusively $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)]^{2+}$ at low cone voltages (up to 50 V), but as the cone voltage is increased, successive loss of PPh_3 ligands occurs to give the series of dications $[M\text{-}\mathbf{n}PPh_3]^{2+}$ (**n**, m/z: 0, 1769.5; **1**, 1638.5; **2**, 1507.5; **3**, 1376.5; **4**, 1245.5) at a cone voltage of 90 V. The complex shows a single resonance in its $^{31}P\text{-}\{^1H\}$ NMR spectrum, showing coupling to ^{195}Pt of 3038 Hz, which is essentially the same as that observed for $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgPh]PF_6$ [8].

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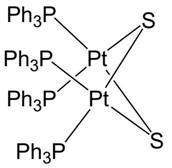
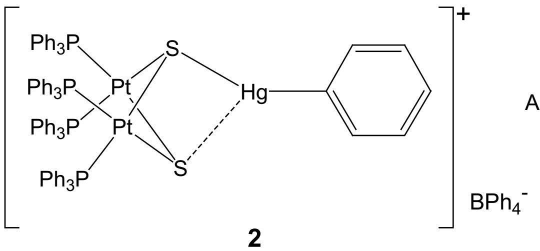
doi:10.1016/j.jorganchem.2008.09.013



1



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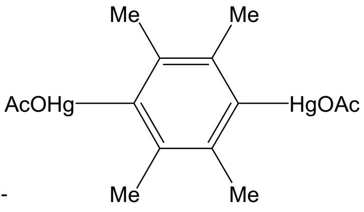

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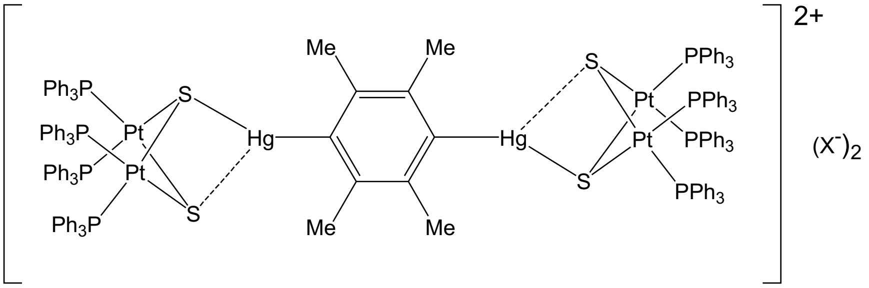
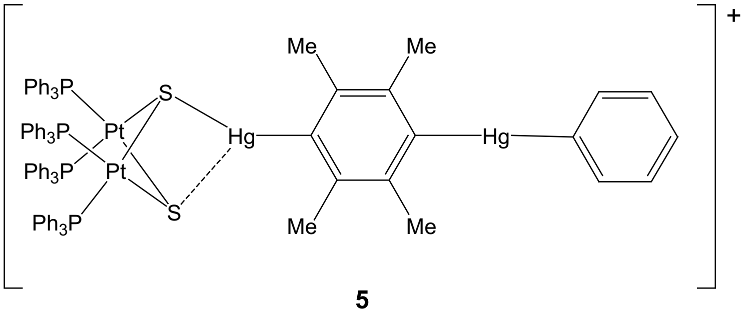


4a, X = PF_6
4b, X = BPh_4



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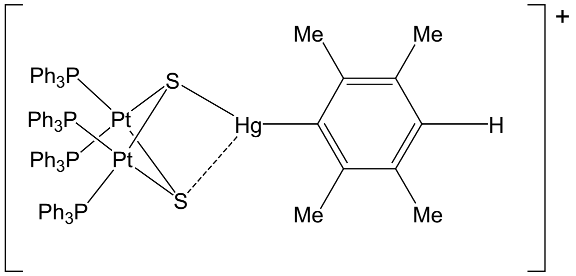

6

The $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)]^{2+}$ cation can also be isolated as its tetraphenylborate salt **4b**, by addition of excess $NaBPh_4$ to the reaction solution. However in this case, the isolated solid product was found to contain additional minor species when analysed by ESI MS. One species at *m/z* 2113 (*ca.* 10% relative intensity) was not observed at all in the reaction solution. Exact mass measurement indicates that this species is the *phenyl*-mercury adduct $[Pt_2(\mu\text{-}S)_2(PPh_3)_4(HgC_6Me_4HgPh)]^+$ **5** (observed *m/z* 2113.326, calculated *m/z* 2113.311). A number of other very minor species were observed in isolated **4b**, including the monocationic phenyl derivative $[Pt_2(\mu\text{-}S)(\mu\text{-}SHgPh)(PPh_3)_4]^+$ **2** (observed *m/z* 1781.247, calculated *m/z* 1781.248). The source of the phenyl group is undoubtedly the BPh_4^- ion, since the phenylating ability of tetraphenylborate is well known [15–18], including phenylation of mercury species, primarily for analytical purposes [19–22]. Consistent with this, the reaction of $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ with one equivalent of $Hg(OAc)_2$ followed by precipitation with $NaBPh_4$ gave a yellow product that showed **2** (*m/z* 1781, 100% relative intensity) together with the known species [23,24] $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2Hg]^{2+}$ (*m/z* 1603.5, 85% relative intensity) as the only significant species. The isolated BPh_4^- salt **4b** also yielded an ion identified as the mono-mercurated species $[Pt_2(\mu\text{-}S)_2(PPh_3)_4Hg(C_6Me_4H)]^+$ **6** (observed *m/z* 1837.307, calculated *m/z* 1837.310); this species was observed as a trace component of the reaction solution before precipitation, but as a more significant component of the tetraphenylborate-precipitated solid. This species could arise from traces of mono-mercurated durene in the starting material, or from Hg–C bond cleavage in the product mediated by BPh_4^-. Recrystallisation of **4b** from dichloromethane-diethyl ether furnished a relatively pure sample of the compound.

In order to characterise the $\{Pt_2S_2\}$–Hg binding in this polymetallic system, and to compare it with the mono-mercury complex $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgPh]^+$ **2** [8], an X-ray structural determination of **4b** was carried out; the structure of the cation is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. The cation of complex **4b** crystallises on a centre of symmetry, so only half of the cation is unique. Overall, the structural features are similar to those of $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgPh]^+$ **2**, with the mercury in each complex being strongly coordinated to one sulfur and weakly coordinated to the other, resulting in a 'T-shaped' coordination geometry. Thus, in **4b** the Hg–S(1) and Hg–S(2) bond distances are 2.3990(7) and 2.9473(7) Å, with an S(1)–Hg(1)–C(1) bond angle of 173.49(9)°, compared to the corresponding Hg–S bond distances of 2.4709(12) and 2.9286(12) Å, and an S–Hg–C bond angle of 174.59(16)° in **2**. The asymmetric mercury binding is, not unexpectedly, transmitted to the $\{Pt_2S_2\}$ core of **4b**, with sulfur S(2) [which is more weakly coordinated to Hg] forming the shorter Pt–S bonds [Pt(1)–S(2) 2.3232(7) and Pt(2)–S(2) 2.3519(7) Å, compared with Pt(1)–S(1) 2.3675(7) and Pt(2)–S(1) 2.3797(7) Å]. Consequently, the Pt–P bonds *trans* to S(2) are slightly longer than those *trans* to S(1); for example Pt(1)–P(1) [*trans* to S(2)] is 2.2939(7) Å, while Pt(1)–P(2) [*trans* to S(1)] is 2.2661(7) Å. This indicates that sulfur S(2) displays a stronger *trans*-influence at the Pt centre than S(1). The same trend is observed in the phenyl derivative **2**.

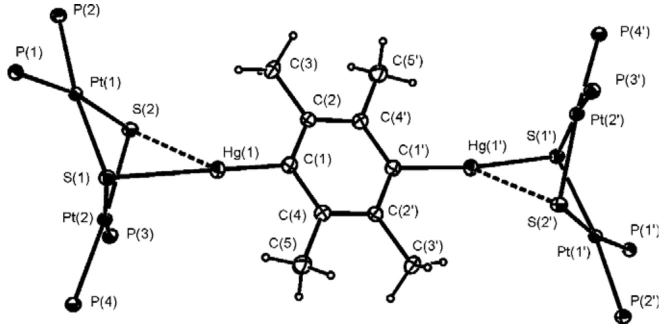

Fig. 1. Molecular structure of the cation of **4b** · $4CH_2Cl_2$ with the atom numbering scheme. Phenyl rings of the triphenylphosphine ligands have been omitted for clarity.

Table 1
Selected bond lengths (Å) and bond angles (°) for **4b** · $4CH_2Cl_2$

Pt(1)–P(1)	2.2939(7)	Pt(1)–P(2)	2.2661(7)
Pt(2)–P(3)	2.2767(7)	Pt(2)–P(4)	2.2913(8)
Pt(1)–S(1)	2.3675(7)	Pt(1)–S(2)	2.3232(7)
Pt(2)–S(1)	2.3797(7)	Pt(2)–S(2)	2.3519(7)
Hg(1)–C(1)	2.080(3)	Hg(1)–S(1)	2.3990(7)
Hg(1)–S(2)	2.9473(7)	C(1)–C(4)	1.399(4)
C(1)–C(2)	1.402(4)	C(2)–C(4′)	1.406(4)
C(2)–C(3)	1.515(4)	C(4)–C(5)	1.509(4)
P(1)–Pt(1)–P(2)	98.84(3)	P(1)–Pt(1)–S(1)	87.60(2)
P(2)–Pt(1)–S(2)	90.62(3)	S(1)–Pt(1)–S(2)	83.31(2)
P(3)–Pt(2)–P(4)	102.29(3)	P(3)–Pt(2)–S(2)	83.66(3)
P(4)–Pt(2)–S(1)	91.65(3)	S(1)–Pt(2)–S(2)	82.44(2)
Pt(1)–S(1)–Pt(2)	86.16(2)	Pt(1)–S(2)–Pt(2)	87.82(2)
Pt(1)–S(1)–Hg(1)	94.56(3)	Pt(1)–S(2)–Hg(1)	82.36(2)
Pt(2)–S(1)–Hg(1)	79.97(2)	Pt(2)–S(2)–Hg(1)	69.794(18)
S(1)–Hg(1)–S(2)	70.50(2)	S(1)–Hg(1)–C(1)	173.49(9)
S(2)–Hg(1)–C(1)	112.13(8)	Hg(1)–C(1)–C(2)	119.4(2)
Hg(1)–C(1)–C(4)	119.3(2)		

The Hg···Pt non-bonded distances in **4b** [3.0707(1) and 3.5020(1) Å] can be compared with 3.078 and 3.515 Å in **2**, indicating that in both complexes the Hg-arene group is tilted towards one of the $Pt(PPh_3)_2$ groups. The fold angle [angle between the two S–Pt–S planes] of the $\{Pt_2S_2\}$ butterfly of **4b** [133.4°] is very similar to the corresponding angle of 132.9° in **2**. The durene ring is also twisted at an angle of 46.6° to the Hg–S(1)–S(2) plane, presumably to minimise steric interactions.

3. Experimental

$[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ **1** was prepared by the literature procedure from *cis*-$[PtCl_2(PPh_3)_2]$ and $Na_2S \cdot 9H_2O$ in benzene suspension, [25] while the organomercury compound $1,4\text{-}AcOHgC_6Me_4HgOAc$ **3** was prepared by mercuration of 1,2,4,5-tetramethylbenzene (durene, Aldrich) with $Hg(OAc)_2$, as described in the literature [26]. Sodium tetraphenylborate (BDH) and ammonium hexafluorophosphate (Aldrich) were used as supplied. Reactions were carried out in LR grade methanol, without exclusion of light, air or moisture.

Routine ESI mass spectra were recorded in positive-ion mode on a VG Platform II instrument using a methanol mobile phase. Isolated products were dissolved in a couple of drops of dichloromethane prior to dilution to *ca.* 0.1 mg mL^{-1} with methanol. High resolution mass measurements were made on a Bruker Daltonics MicrOTOF instrument, calibrated using a sodium formate solution. $^{31}P\text{-}\{^1H\}$ NMR spectra were recorded on a Bruker AC300P instrument at 121.5 MHz in $CDCl_3$ solution, and spectra were referenced relative to external H_3PO_4. Elemental microanalyses were performed by the Campbell Microanalytical Laboratory, Department of Chemistry, University of Otago, New Zealand.

3.1. Synthesis of $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)](PF_6)_2$ 4a

A suspension of $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ **1** (300 mg, 0.200 mmol) and **3** (65 mg, 0.100 mmol) in methanol (30 mL) was stirred at room temperature for 2 h, giving a bright yellow solution. ESI MS showed a single peak due to the product cation $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)]^{2+}$. After filtration to remove a trace of insoluble grey matter, solid NH_4PF_6 (200 mg, 1.23 mmol) was added to the stirred filtrate, giving a yellow precipitate. Water (20 mL) was added to assist the precipitation, the product isolated by filtration, washed successively with water (2 × 20 mL) and diethyl ether (2 × 20 mL), and dried *in vacuo* to give **4a** (295 mg, 77%) as a bright yellow solid. Found: C, 47.7; H, 3.6. $C_{154}H_{132}F_{12}Hg_2P_{10}Pt_4S_4$ requires C, 48.3; H, 3.5%. $^{31}P\text{-}\{^1H\}$ NMR $[(CD_3)_2SO]$, δ 20.1 [s, ^1J(PtP) 3038 Hz]. ^1H NMR, δ 7.78–7.08 (m, Ph) and 2.57 (s, CH_3).

3.2. Synthesis of $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)](BPh_4)_2$ 4b

Following the procedure for **4a**, $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ **1** (300 mg, 0.200 mmol) with **3** (65 mg, 0.100 mmol) were reacted. To the resulting yellow solution after filtration was added solid $NaBPh_4$ (200 mg, 0.585 mmol), the product filtered, washed successively with methanol (20 mL), water (20 mL), methanol (20 mL) and diethyl ether (20 mL) and dried *in vacuo* to give **4b** (305 mg, 73%) as a yellow powder. Found: C, 57.2; H, 4.1%. $C_{202}H_{172}B_2Hg_2P_8Pt_4S_4$ requires C, 58.1; H, 4.2. $^{31}P\text{-}\{^1H\}$ NMR $[(CD_3)_2SO]$, δ 21.3 [s, ^1J(PtP) 3038 Hz]. ^1H NMR, δ 7.46–6.85 (m, Ph) and 2.54 (s, CH_3).

3.3. Reaction of $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ with $Hg(OAc)_2$ and $NaBPh_4$

Mercury(II) acetate (21.6 mg, 0.068 mmol) was added to a stirred suspension of $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ (102 mg, 0.068 mmol) in

Table 2
Crystal, collection and refinement data for **4b** · $4CH_2Cl_2$

Crystal data		
Empirical formula		$C_{206}H_{180}B_2Cl_8Hg_2P_8Pt_4S_4$
Formula weight		4518.26
Crystal system		Monoclinic
Space group		$P2_1/c$
Unit cell dimensions		
a (Å)		13.5909(1)
b (Å)		40.2714(4)
c (Å)		16.3746(2)
β (°)		95.575(1)
V (Å^3)		8919.83(16)
Z		2
D_{calc} (g cm^{-3})		1.682
Data collection		
Crystal size (mm)		0.28 × 0.26 × 0.26
Radiation, wavelength (Å)		Mo Kα, $\lambda = 0.71073$
Temperature (K)		89(2)
θ Range for data collection (°)		1.81–32.62
Reflections collected		180006
Independent reflections		32403 [$R_{int} = 0.0407$]
Absorption coefficient (mm^{-1})		5.133
$T_{max,min}$		1.00, 0.69
$F(000)$		4436
Structure analysis and refinement		
Solution by:		Direct methods
Refinement method		Full-matrix least-squares on F^2
Data/restraints/parameters		32403/0/1056
Goodness-of-fit on F^2		1.095
Final R indices [$I > 2\sigma(I)$]	R_1	0.0328
	wR_2	0.0662
R indices (all data)	R_1	0.0465
	wR_2	0.0697
Largest difference peak (e Å^{-3})		3.122
Largest difference hole (e Å^{-3})		−1.449

methanol (20 mL), whereupon the reactants rapidly dissolved to give a clear yellow solution. After stirring for 1 h, $NaBPh_4$ (200 mg, 0.585 mmol) was added giving a yellow precipitate that was isolated by filtration, washed with water (10 mL) and methanol (10 mL) and dried *in vacuo* to give a yellow powder (105 mg). ESI MS analysis showed $[Pt_2(\mu\text{-}S)_2(PPh_3)_4HgPh]^+$ **2** (m/z 1781, 100%) and $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2Hg]^{2+}$ (m/z 1603.5, 85%).

3.4. X-ray structure determination of $[\{Pt_2(\mu\text{-}S)_2(PPh_3)_4\}_2(\mu\text{-}1,4\text{-}C_6Me_4Hg_2)](BPh_4)_2$ **4b**·$4CH_2Cl_2$

Bright yellow crystals suitable for an X-ray structural study were obtained by vapour diffusion of diethyl ether into a dichloromethane solution of the complex at room temperature. Intensity data were obtained on a Bruker Apex II CCD diffractometer. Crystal, collection and refinement data are given in Table 2. The complex crystallises with four molecules of CH_2Cl_2 per cation.

Acknowledgements

We thank the University of Waikato (UW) and the National University of Singapore (NUS) for financial support of this work, including Summer Research Scholarships (NEC and RAL). We also thank Dr. Tania Groutso (University of Auckland) for collection of the X-ray data set.

Appendix A. Supplementary material

CCDC 685996 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2008.09.013.

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