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Dalton Transactions - Full Article

Transition Metal Complexes of Antimony Centered Ligands based upon Acenaphthyl

Scaffolds. Coordination Non-Innocent or Not?

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Supporting Information

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Figure S1b. ${}^{13}C{}^{1}H$ -NMR spectrum (CDCl₃) of 1.



Figure S1c. ${}^{31}P{}^{1}H$ -NMR spectrum (CDCl₃) of 1.



Figure S2a. ¹H-NMR spectrum (CDCl₃) of 2.



Figure S2b. ${}^{13}C{}^{1}H$ -NMR spectrum (CDCl₃) of 2.





Figure S2c. ${}^{31}P{}^{1}H$ -NMR spectrum (CDCl₃) of 2.



Figure S3b. ${}^{13}C{}^{1}H$ -NMR spectrum (CDCl₃) of **3** (poor solubility, acquisition time 48 h).



Figure S3b. ${}^{31}P{}^{1}H$ -NMR spectrum (CDCl₃) of **3** (poor solubility, acquisition time 12 h).



Figure S4a. ¹H-NMR spectrum (CDCl₃) of 4.



Figure S4b. $^{13}C\{^{1}H\}$ -NMR spectrum (CDCl₃) of 4.





Figure S4c. ${}^{31}P{}^{1}H$ -NMR spectrum (CDCl₃) of 4.



Figure S5a. ¹H-NMR spectrum (CDCl₃) of 5.



Figure S5b. ${}^{13}C{}^{1}H$ -NMR spectrum (CDCl₃) of 5 (poor solubility, acquisition time 72 h).



Figure S5c. ${}^{31}P{}^{1}H$ -NMR spectrum (CDCl₃) of 5.



Figure S6a. ¹H-NMR spectrum (THF-d₈) of 6.



Figure S6b. ¹³C{¹H}-NMR spectrum (THF-d₈) of **6** (poor solubility, acquisition time 24 h, decomposition in CDCl₃ and CD₂Cl₂).



Figure S6c. ${}^{31}P{}^{1}H$ -NMR spectrum (THF-d₈) of 6.



Figure S6d. UV/Vis spectrum (THF) of 6.

Image: 1 to the second lengths and AnglesBond Lengths and AnglesSb(1)-C(10) $2.187(2)$ $2.201(2)$ Sb(1)-C(1)/C(70) $2.632(1)$ $2.192(2)$ Sb(1)-C(40) $2.181(2)$ $2.203(3)$ C(10)-Sb(1)-C(40) $93.14(9)$ $96.4(2)$ C(10)-Sb(1)-C(1)/C(70) $91.68(7)$ $92.6(2)$ Sb(1)-C(1)-C(1)/C(70) $92.28(7)$ $94.8(4)$ peri Region DistancesSb(1)-P(1) $2.774(1)$ $3.171(1)$ Sb(1)-P(2) $3.266(1)$ $3.229(1)$ Sb(1)-P(3) $2.26(2)$ $126.7(2)$ Sb(1)-C(10)-C(19) $121.7(2)$ $125.3(3)$ Sb(1)-C(10)-C(19) $126.9(2)$ $126.7(2)$ Sb(1)-C(70)-C(79) $124.3(3)$ $129.1(3)$ C(10)-C(19)-C(18) $126.9(3)$ $129.1(3)$ C(10)-C(19)-C(18) $126.9(2)$ $121.1(2)$ P(1)-C(18)-C(19) $115.1(2)$ $120.9(2)$ P(1)-C(18)-C(19) $115.1(2)$ $120.5(3)$ Σ of bay angles $363.7(7)$ $373.7(8)$ Splay angles $363.7(7)$ $375.3(7)$ Splay angles $363.7(7)$ $15.3(7)$ P(1) $0.271(1)$ $0.016(1)$ P(2) <t< th=""><th></th><th>4</th><th>•</th></t<>		4	•	
Bond Lengths and AnglesSb(1)-C(10)2.187(2)2.201(2)Sb(1)-C(10)/C(70)2.632(1)2.192(2)Sb(1)-C(40)2.181(2)2.203(3)C(10)-Sb(1)-C(40)93.14(9)96.4(2)C(10)-Sb(1)-C(1)/C(70)91.68(7)92.6(2)C(40)-Sb(1)-C(1)/C(70)92.28(7)94.8(4)peri Region DistancesSb(1)-P(1)2.774(1)3.171(1)Sb(1)-P(2)3.266(1)3.229(1)Sb(1)-P(3)2.774(1)3.171(1)Sb(1)-C(10)-C(19)121.7(2)125.3(3)Sb(1)-C(10)-C(19)121.7(2)125.3(3)Sb(1)-C(70)-C(79)124.3(3)126.9(3)C(10)-C(19)-C(18)126.9(3)129.1(3)C(10)-C(79)-C(78)128.9(2)128.9(2)P(1)-C(18)-C(19)115.1(2)120.9(2)P(1)-C(78)-C(79)2.05(3)2.05(3)S of bay angles37.7(7)15.3(7)14(6)16.9(8)373.7(8)Splay anglea3.7(7)15.3(7)14(6)16.9(8)13.7(8)P(1)0.271(1)0.016(1)P(1)0.271(1)0.016(1)P(1)0.242(1)0.019(1)0.462(1)0.400(1)0.462(1)0.510(1)0.014(1)P(3)0.2487(1)0.652(1)Central Acenaphthyl Ring Torsion AnglesC: (13)-(14)-(19)-(18)178.9(3)178.6(3)C: (13)-(14)-(19)-(18)178.9(3)-175.6(3)C: (13)-(14)-(19)-(18)178.1(3)-177.6(3)<		1	2	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		Bond Lengths and	Angles	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Sb(1)-C(10)	2.187(2)	2.201(2)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Sb(1)-Cl(1)/C(70)	2.632(1)	2.192(2)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Sb(1)-C(40)	2.181(2)	2.203(3)	
$\begin{array}{ccccc} C(10)-Sb(1)-Cl(1)/C(70) & 91.68(7) & 92.6(2) \\ C(40)-Sb(1)-Cl(1)/C(70) & 92.28(7) & 94.8(4) \\ & peri Region Distances \\ \\ Sb(1)-P(1) & 2.774(1) & 3.171(1) \\ Sb(1)-P(2) & 3.266(1) & 3.229(1) \\ Sb(1)-P(2) & 3.266(1) & 3.243(1) \\ & peri Region Bond Angles \\ \\ Sb(1)-C(10)-C(19) & 121.7(2) & 125.3(3) \\ Sb(1)-C(40)-C(49) & 126.8(2) & 126.7(2) \\ Sb(1)-C(70)-C(79) & 124.3(3) \\ C(10)-C(19)-C(18) & 126.9(3) & 129.1(3) \\ C(40)-C(49)-C(48) & 129.4(2) & 129.1(3) \\ C(70)-C(79)-C(78) & 128.9(2) \\ P(1)-C(18)-C(19) & 115.1(2) & 120.9(2) \\ P(1)-C(48)-C(49) & 117.8(2) & 121.1(3) \\ P(1)-C(78)-C(79) & 363.7(7) & 375.3(7) \\ & 374.0(6) & 376.9(8) \\ & & 373.7(8) \\ \\ Splay angles & 363.7(7) & 15.3(7) \\ 14(6) & 16.9(8) \\ & & 13.7(8) \\ \hline P(1) & 0.271(1) & 0.016(1) \\ P(2) & 0.510(1) & 0.014(1) \\ P(3) & 0.287(1) \\ Sb(1) & 0.242(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.019(1) \\ 0.462(1) & 0.118(13) & -177.6(3) \\ C: (13)-(14)-(19)-(10) & 178.1(3) & -177.6(3) \\ C: (45)-(44)-(49)-(40) & -175.6(3) & -175.4(3) \\ C: (45)-(44)-(49)-(40) & -175.6(3) & -175.4(3) \\ C: (75)-(74)-(79)-(70) & 175.4(3) \\ C: (75)-(74)-(79)-(70$	C(10)-Sb(1)-C(40)	93.14(9)	96.4(2)	
$\begin{array}{cccc} C(40)-Sb(1)-Cl(1)/C(70) & 92.28(7) & 94.8(4) \\ & peri Region Distances \\ Sb(1)-P(1) & 2.774(1) & 3.171(1) \\ Sb(1)-P(2) & 3.266(1) & 3.229(1) \\ Sb(1)-P(3) & & & & & & & & & & & & & & & & & & &$	C(10)-Sb(1)-Cl(1)/C(70)	91.68(7)	92.6(2)	
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	C(40)-Sb(1)-Cl(1)/C(70)	92.28(7)	94.8(4)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		peri Region Dista	inces	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Sb(1) - P(1)	2.774(1)	3.171(1)	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Sb(1)-P(2)	3.266(1)	3.229(1)	
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	Sb(1) - P(3)		3.243(1)	
$\begin{array}{c cccc} F(1) = 0 & F(1) $		peri Region Bond Angles		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Sb(1)-C(10)-C(19)	121 7(2)	125 3(3)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Sb(1) - C(40) - C(49)	126.8(2)	126.7(2)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Sb(1) - C(70) - C(79)	120.0(2)	120.7(2) 124.3(3)	
$\begin{array}{cccc} C(10) & C(17) & C(18) & 125.7(3) & 125.7(3) \\ C(40)-C(49)-C(48) & 129.4(2) & 129.1(3) \\ C(70)-C(79)-C(78) & 128.9(2) \\ P(1)-C(18)-C(19) & 115.1(2) & 120.9(2) \\ P(1)-C(48)-C(49) & 117.8(2) & 121.1(3) \\ P(1)-C(78)-C(79) & 120.5(3) \\ \Sigma \ of \ bay \ angles & 363.7(7) & 375.3(7) \\ Splay \ angles & 363.7(7) & 375.3(7) \\ 374.0(6) & 376.9(8) \\ 373.7(8) & 374.0(6) & 376.9(8) \\ 373.7(8) & 374.0(6) & 376.9(8) \\ 373.7(8) & 374.0(6) & 376.9(8) \\ 373.7(8) & 374.0(6) & 376.9(8) \\ 373.7(8) & 374.0(6) & 376.9(8) \\ 13.7(8) & 0.247(1) & 0.016(1) \\ P(2) & 0.510(1) & 0.014(1) \\ P(3) & 0.242(1) & 0.019(1) \\ 0.462(1) & 0.040(1) \\ 0.462(1) & 0.040(1) \\ 0.462(1) & 0.040(1) \\ 0.652(1) \\ \hline \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \$	C(10) - C(19) - C(18)	126 9(3)	129.1(3)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(10) = C(10) = C(10)	120.9(5) 129.4(2)	129.1(3) 129.1(3)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(70) - C(70) - C(78)	127.7(2)	129.1(3) 128.0(2)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	P(1) = C(18) = C(10)	115 1(2)	120.9(2) 120.9(2)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	P(1) = C(48) = C(40)	113.1(2) 117.9(2)	120.9(2) 121.1(2)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	P(1) = C(48) = C(49)	117.8(2)	121.1(3) 120.5(2)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	P(1) = C(78) = C(79)		120.5(3)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Σ of bay angles	363.7(7)	3/5.3(7)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		374.0(6)	3/6.9(8)	
Splay anglea $3.7(7)$ $15.3(7)$ 14(6)16.9(8)13.7(8)Out-of-Plane DisplacementP(1)0.271(1)P(2)0.510(1)P(3)0.287(1)Sb(1)0.242(1)0.462(1)0.019(1)0.462(1)0.040(1)0.652(1)Central Acenaphthyl Ring Torsion AnglesC: (13)-(14)-(19)-(18)178.9(3)C: (43)-(44)-(49)-(48)-173.6(2)-173.6(2)-179.9(4)C: (73)-(74)-(79)-(78)173.4(3)C: (15)-(14)-(19)-(10)178.1(3)C: (45)-(44)-(49)-(40)-175.6(3)C: (75)-(74)-(79)-(70)176.3(5)			3/3./(8)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Splay angle ^a	3.7(7)	15.3(7)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		14(6)	16.9(8)	
$\begin{array}{c cccc} Out-of-Plane \ Displacement \\ P(1) & 0.271(1) & 0.016(1) \\ P(2) & 0.510(1) & 0.014(1) \\ P(3) & 0.287(1) \\ Sb(1) & 0.242(1) & 0.019(1) \\ 0.462(1) & 0.040(1) \\ 0.652(1) \\ \hline \\ Central \ Acenaphthyl \ Ring \ Torsion \ Angles \\ C: \ (13)-(14)-(19)-(18) & 178.9(3) & 178.6(3) \\ C: \ (43)-(44)-(49)-(48) & -173.6(2) & -179.9(4) \\ C: \ (73)-(74)-(79)-(78) & 173.4(3) \\ C: \ (15)-(14)-(19)-(10) & 178.1(3) & -177.6(3) \\ C: \ (45)-(44)-(49)-(40) & -175.6(3) & -175.4(3) \\ C: \ (75)-(74)-(79)-(70) & 176.3(5) \\ \hline \end{array}$			13.7(8)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		Out-of-Plane Displacement		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	P(1)	0.271(1)	0.016(1)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	P(2)	0.510(1)	0.014(1)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	P(3)		0.287(1)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Sb(1)	0.242(1)	0.019(1)	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		0.462(1)	0.040(1)	
$\begin{array}{c c} Central \ Acenaphthyl \ Ring \ Torsion \ Angles \\ \hline C: (13)-(14)-(19)-(18) & 178.9(3) & 178.6(3) \\ C: (43)-(44)-(49)-(48) & -173.6(2) & -179.9(4) \\ C: (73)-(74)-(79)-(78) & 173.4(3) \\ C: (15)-(14)-(19)-(10) & 178.1(3) & -177.6(3) \\ C: (45)-(44)-(49)-(40) & -175.6(3) & -175.4(3) \\ C: (75)-(74)-(79)-(70) & 176.3(5) \\ \hline \end{array}$			0.652(1)	
C: $(13)-(14)-(19)-(18)$ 178.9(3)178.6(3)C: $(43)-(44)-(49)-(48)$ $-173.6(2)$ $-179.9(4)$ C: $(73)-(74)-(79)-(78)$ 178.1(3) $-177.6(3)$ C: $(15)-(14)-(19)-(10)$ 178.1(3) $-177.6(3)$ C: $(45)-(44)-(49)-(40)$ $-175.6(3)$ $-175.4(3)$ C: $(75)-(74)-(79)-(70)$ 176.3(5)		Central Acenaphthyl Ring Torsion Angles		
C: $(43)-(44)-(49)-(48)$ $-173.6(2)$ $-179.9(4)$ C: $(73)-(74)-(79)-(78)$ $173.4(3)$ C: $(15)-(14)-(19)-(10)$ $178.1(3)$ $-177.6(3)$ C: $(45)-(44)-(49)-(40)$ $-175.6(3)$ $-175.4(3)$ C: $(75)-(74)-(79)-(70)$ $176.3(5)$	C: (13)–(14)–(19)–(18)	178.9(3)	178.6(3)	
C: $(73)-(74)-(79)-(78)$ 173.4(3)C: $(15)-(14)-(19)-(10)$ 178.1(3)C: $(45)-(44)-(49)-(40)$ -175.6(3)C: $(75)-(74)-(79)-(70)$ 176.3(5)	C: (43) - (44) - (49) - (48)	-173.6(2)	-179.9(4)	
C: $(15)-(14)-(19)-(10)$ 178.1(3)-177.6(3)C: $(45)-(44)-(49)-(40)$ -175.6(3)-175.4(3)C: $(75)-(74)-(79)-(70)$ 176.3(5)	C: (73)–(74)–(79)–(78)		173.4(3)	
$\begin{array}{c} (12) & (12) & (12) \\ C: (45)-(44)-(49)-(40) & -175.6(3) \\ C: (75)-(74)-(79)-(70) & 176.3(5) \\ \end{array}$	C: (15)-(14)-(19)-(10)	178 1(3)	-177 6(3)	
C: (75)-(74)-(70) 175.4(5) 176.3(5) 176.3(5)	$C^{-}(45)-(44)-(49)-(40)$	-175 6(3)	-1754(3)	
	$C^{-}(75)-(74)-(79)-(70)$		176 3(5)	

Table S1.Selected interatomic distances (Å) and angles (°) of 1 and 2.

^a Splay angle: sum of the three bay region angles – 360.



Figure S7. AIM topologies, NCI *iso*-surfaces (s = 0.5), and ELI-D *iso*-surfaces ($\gamma = 1.3$) of **1** (left column) and **2** (right column).



Figure S8. AIM topology, NCI *iso*-surface ($\mathbf{s} = 0.5$), ELI-D *iso*-surface ($\gamma = 1.3$; inset shows magnification around the metal center at $\gamma = 1.25$), and ELI-D distribution mapped on the Sb–Pt ELI-D basin in **5**.



Figure S9 AIM topology, NCI *iso*-surface ($\mathbf{s} = 0.5$), ELI-D *iso*-surface ($\gamma = 1.3$; inset shows magnification around the metal center at $\gamma = 1.25$), and ELI-D distribution mapped on the Sb-Rh ELI-D basin in **6**.