

Electronic Supplementary Information

Transmetallation of Bis(6-diphenylphosphinoxy-acenaphth-5-yl)mercury with Tin Tetrachloride, Antimony Trichloride and Bismuth Trichloride

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NMR spectra

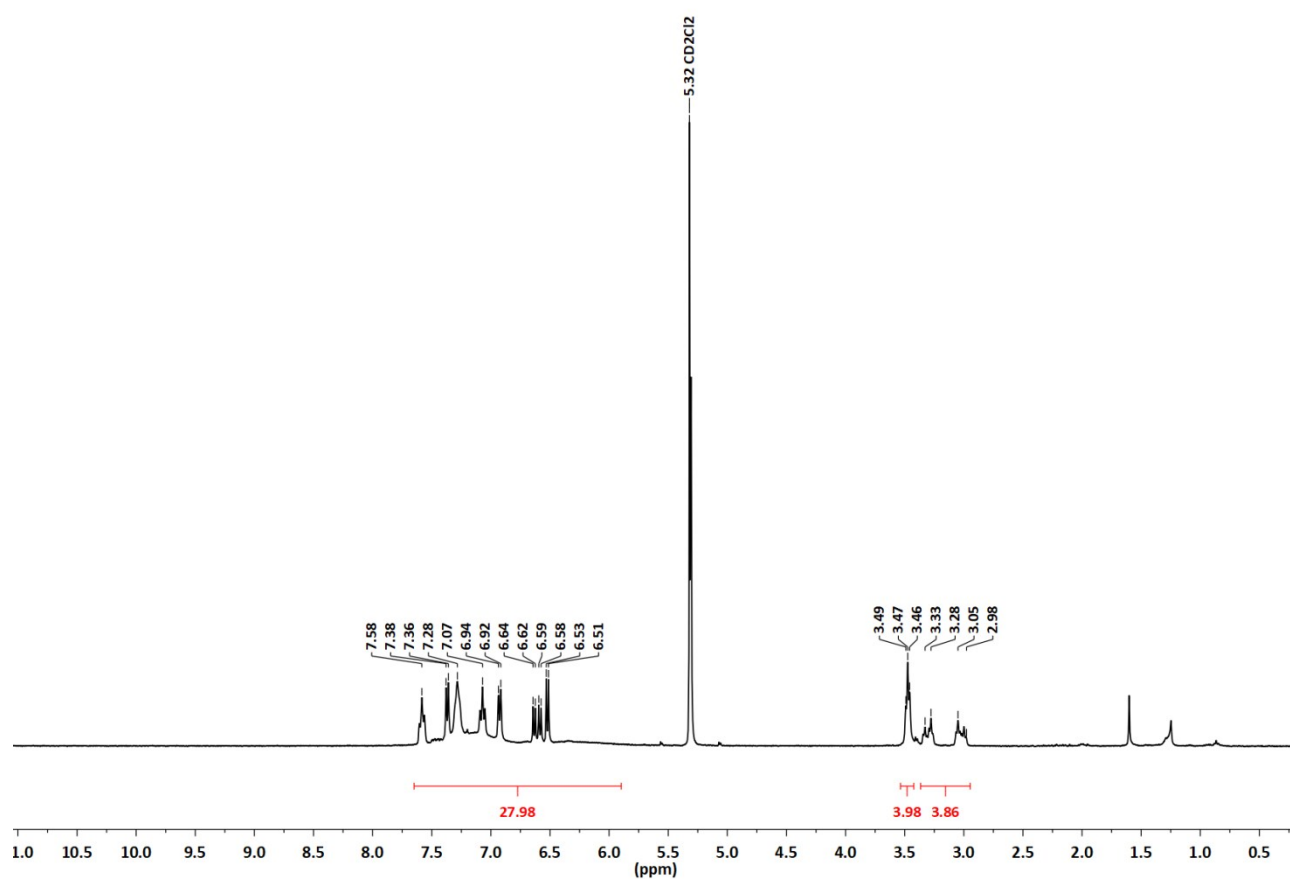


Figure S1. ¹H NMR (360 MHz, CD₂Cl₂) spectrum of **1a**.

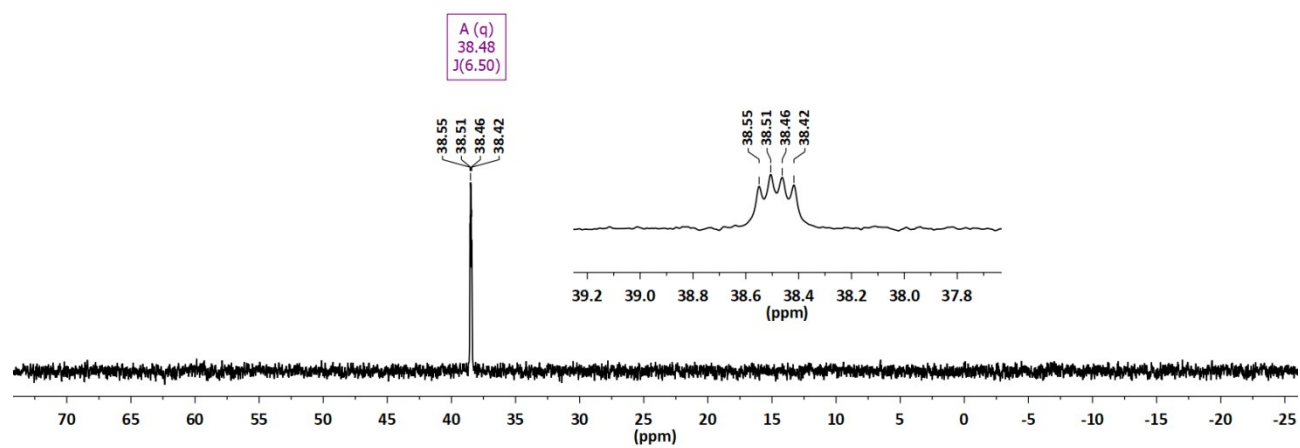


Figure S2. ³¹P NMR (146 MHz, CD₂Cl₂) spectrum of **1a**.

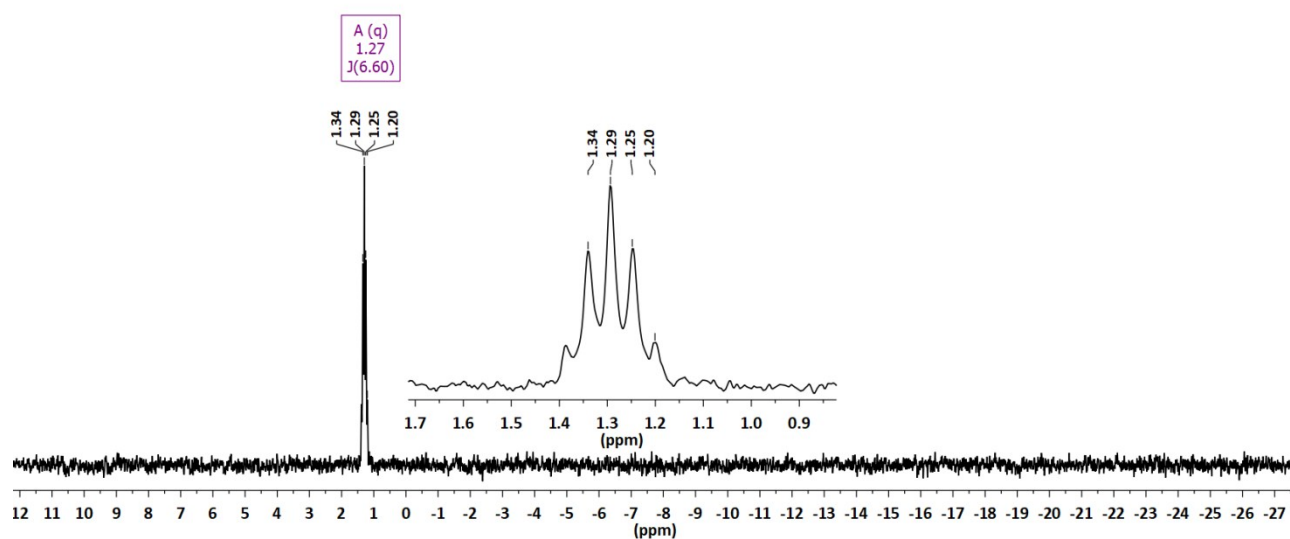


Figure S3. ^7Li NMR (140 MHz, CD_2Cl_2) spectrum of **1a**.

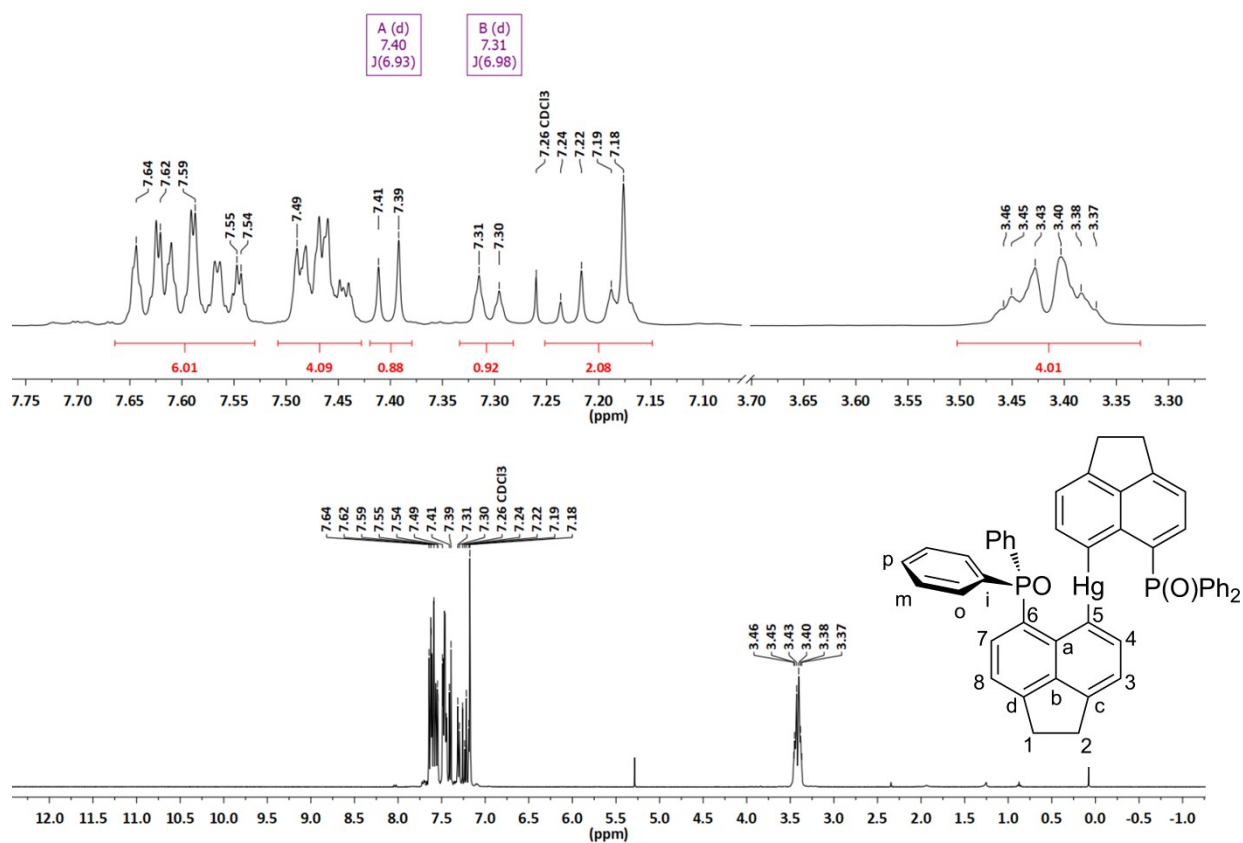


Figure S4. ^1H NMR (360 MHz, CDCl_3) spectrum of **3**.

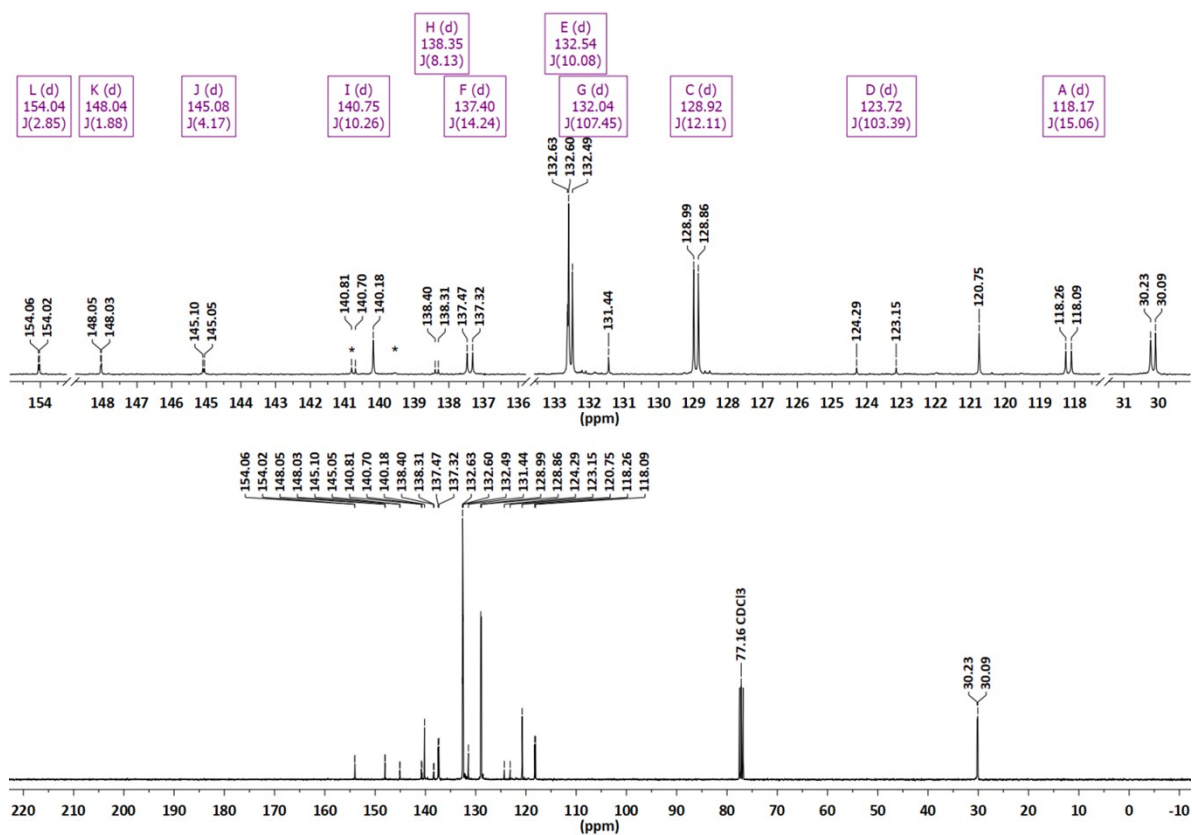


Figure S5. $^{13}\text{C}\{^1\text{H}\}$ NMR (91 MHz, CDCl_3) spectrum of **3** (* denotes ^{199}Hg satellites).

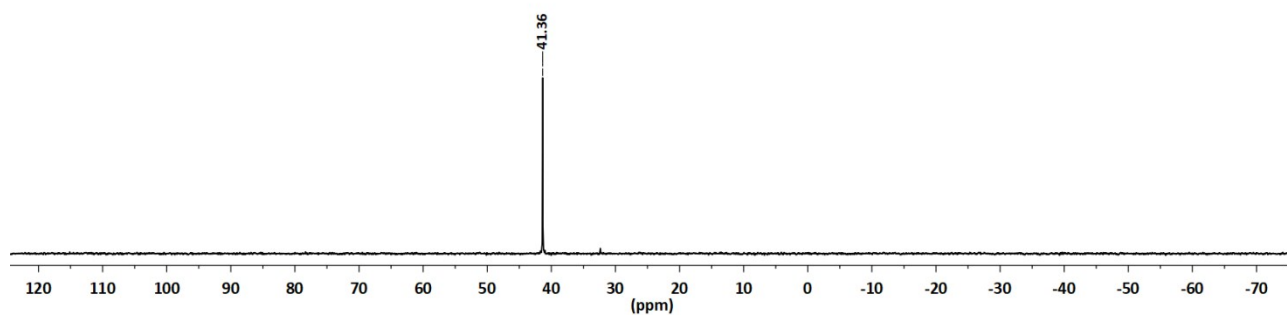


Figure S6. $^{31}\text{P}\{^1\text{H}\}$ NMR (146 MHz, CDCl_3) spectrum of **3**.

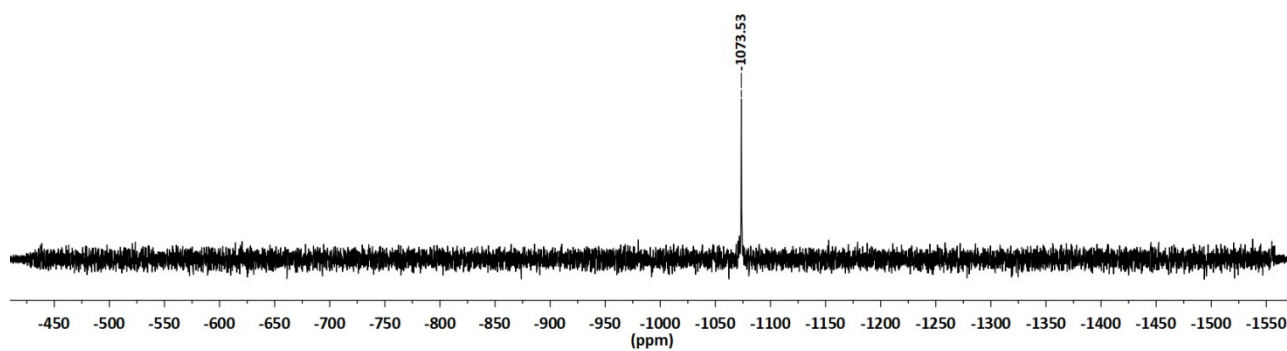
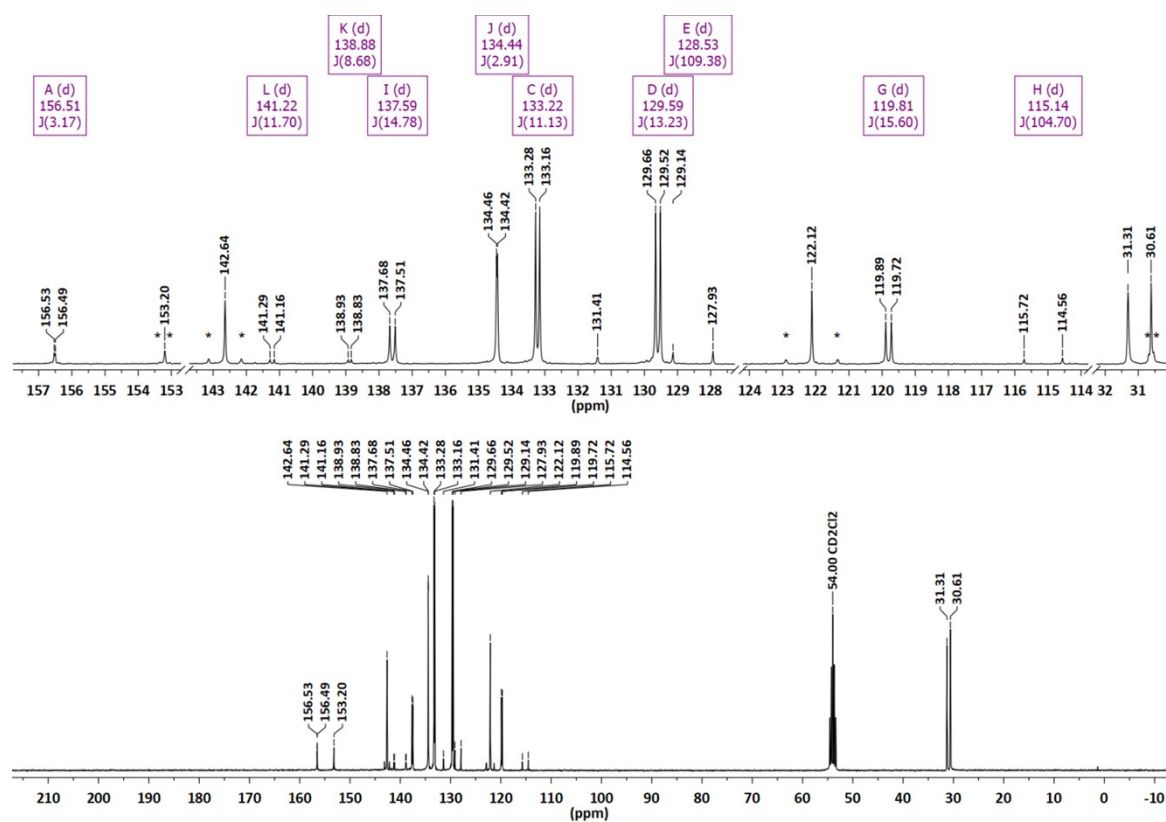
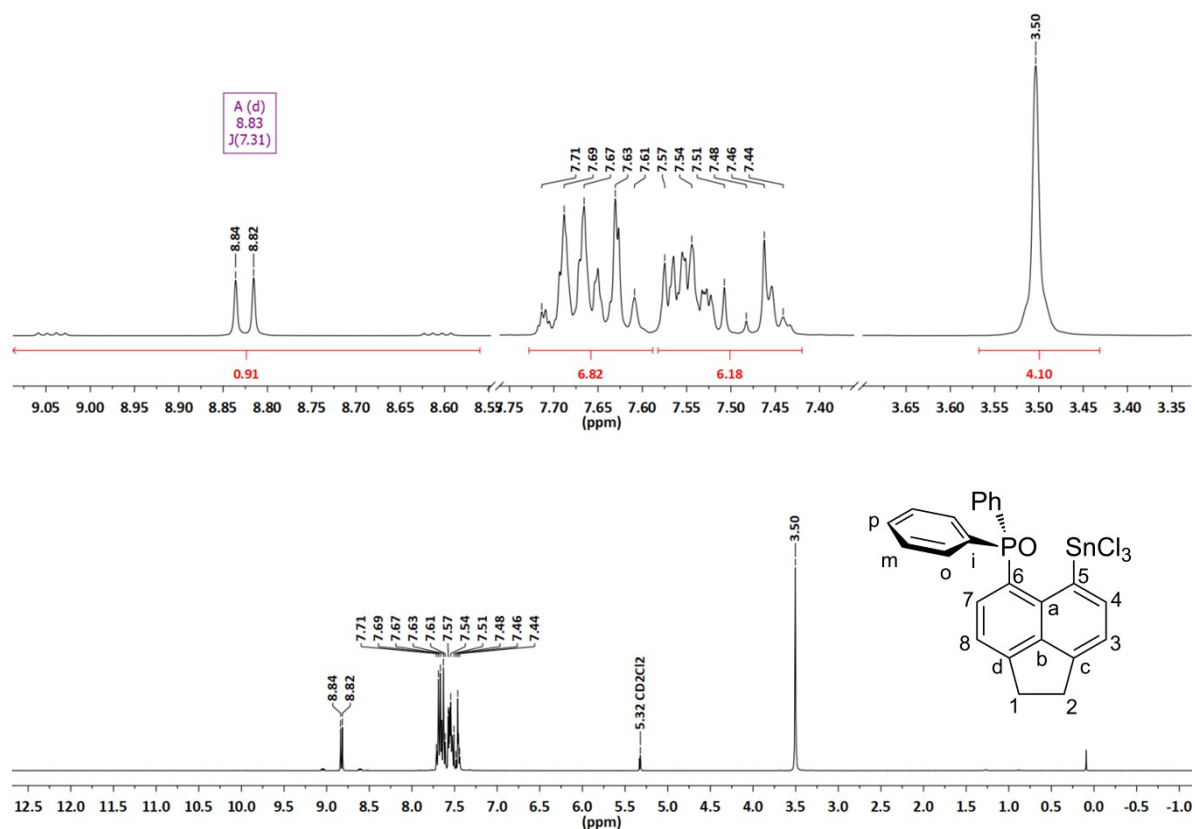


Figure S7. $^{199}\text{Hg}\{^1\text{H}\}$ NMR (65 MHz, CDCl_3) spectrum of **3**.



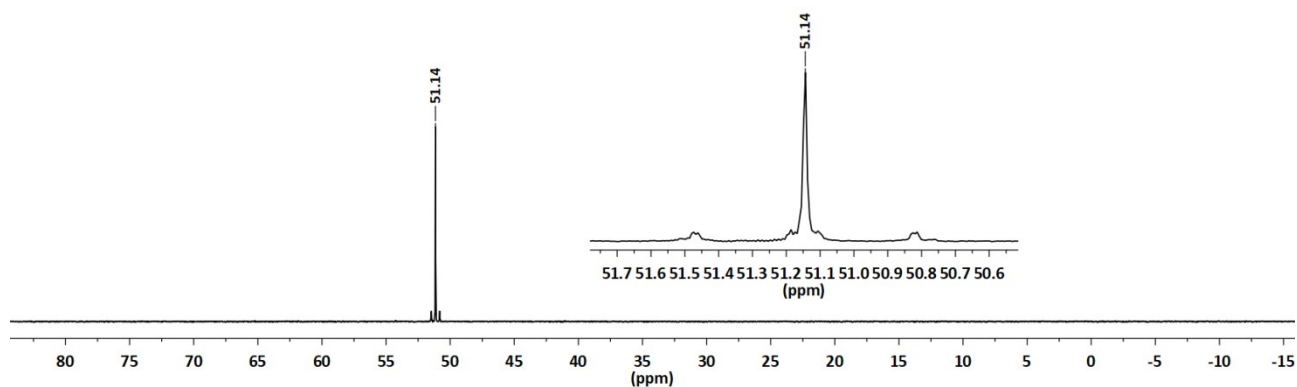


Figure S10. $^{31}\text{P}\{^1\text{H}\}$ NMR (146 MHz, CD_2Cl_2) spectrum of **4**.

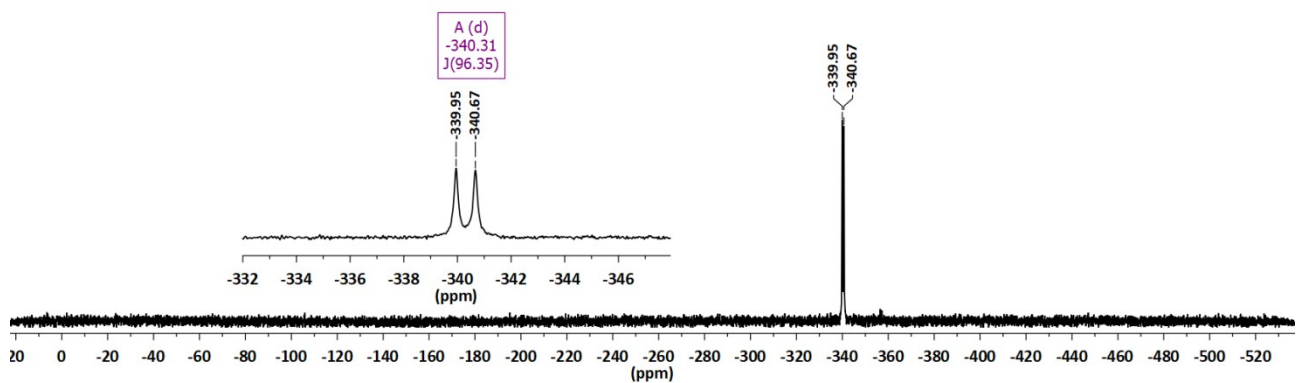


Figure S11. $^{119}\text{Sn}\{^1\text{H}\}$ NMR (134 MHz, CD_2Cl_2) spectrum of **4**.

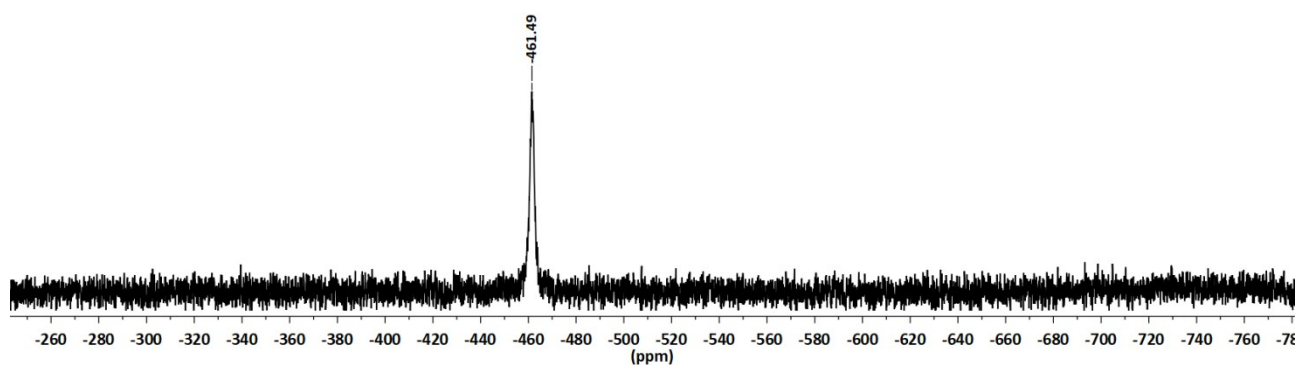


Figure S12. $^{119}\text{Sn}\{^1\text{H}\}$ NMR (134 MHz, $\text{CD}_2\text{Cl}_2 + \text{THF}$ ca. 10:1, v/v) spectrum of **4**·THF.

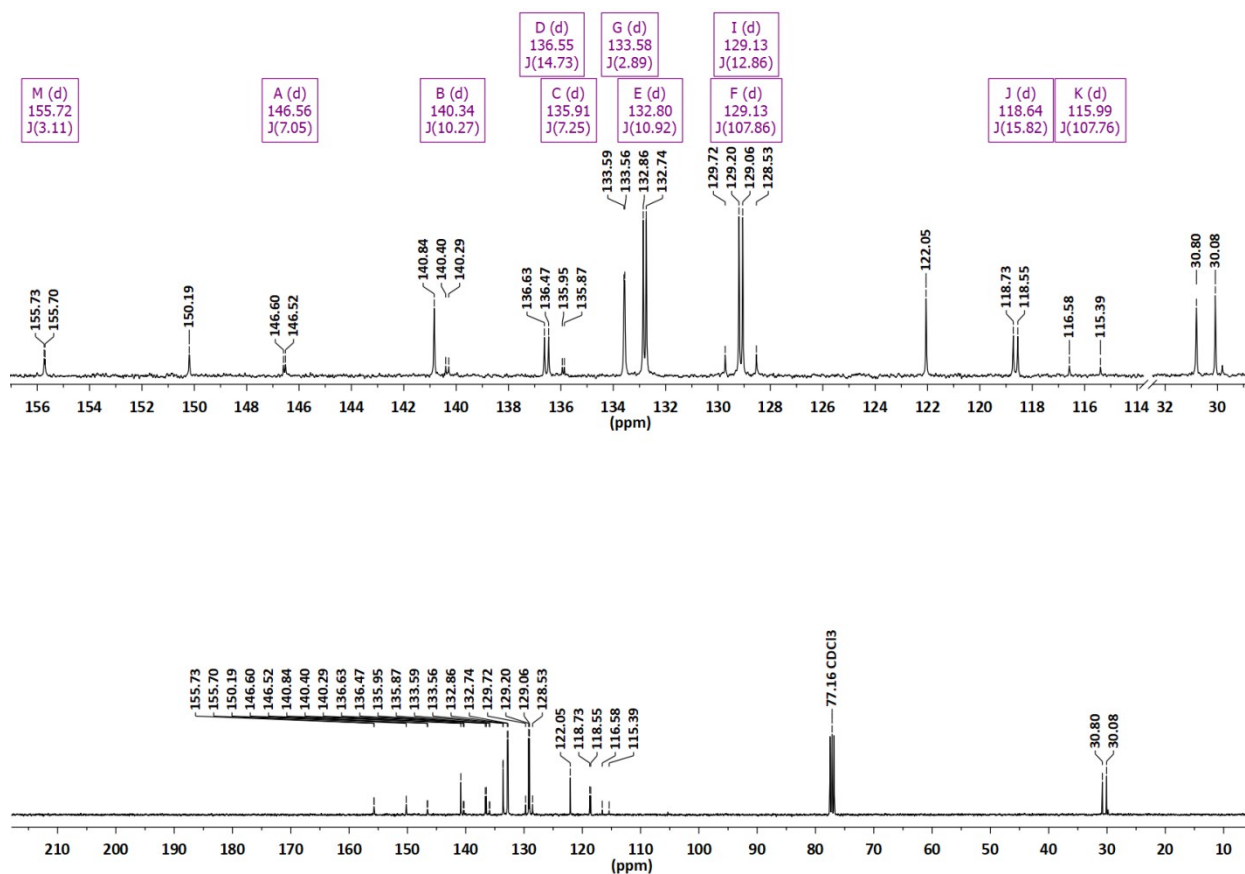
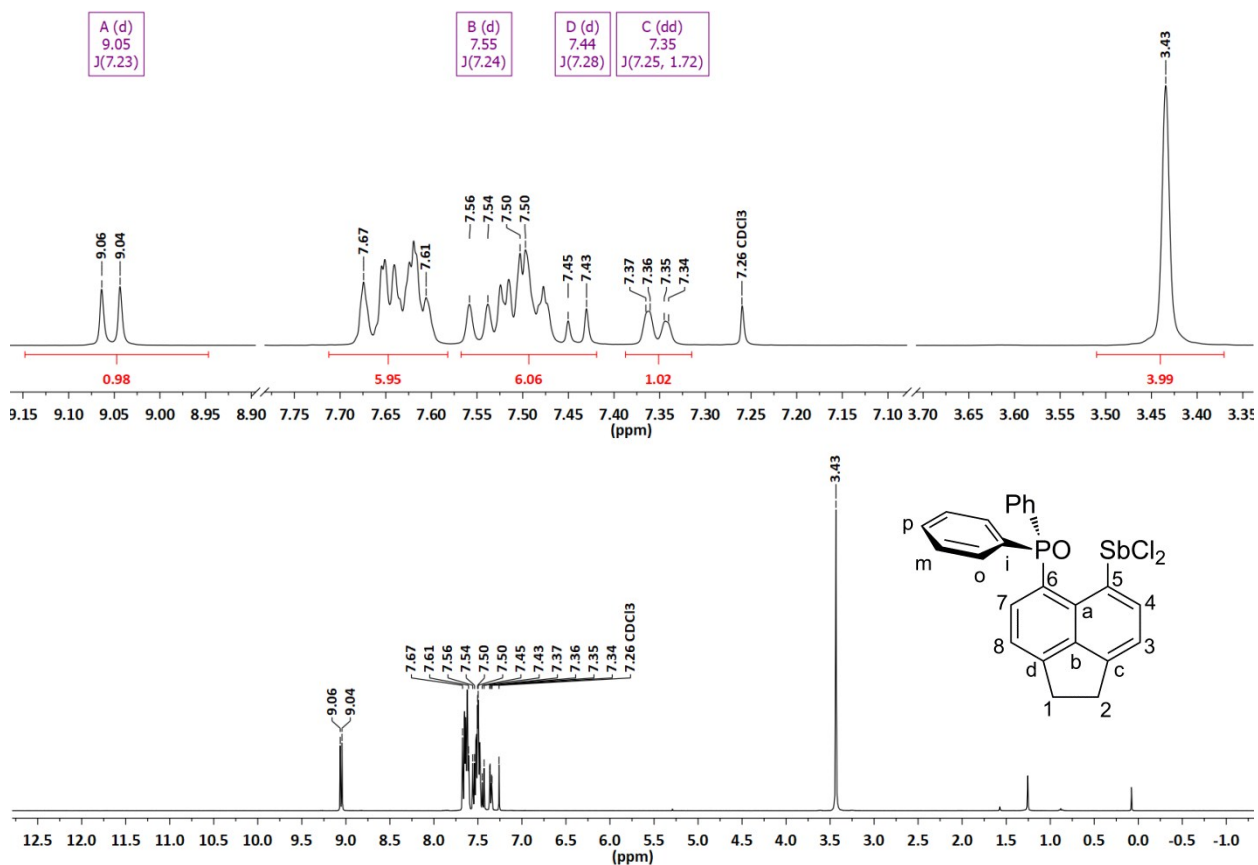


Figure S14. $^{13}\text{C}\{^1\text{H}\}$ NMR (91 MHz, CDCl_3) spectrum of **5.**

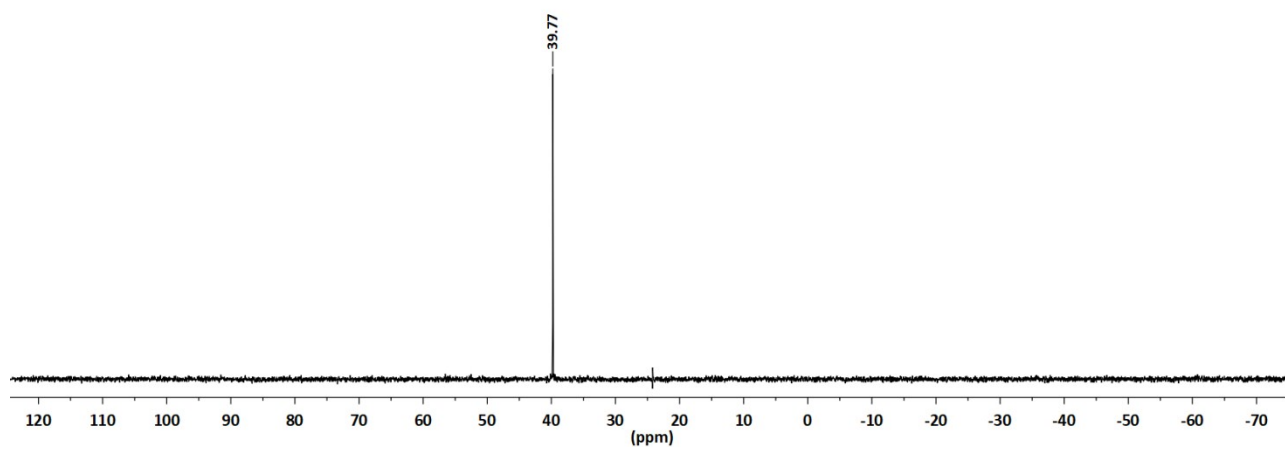


Figure S15. $^{31}\text{P}\{^1\text{H}\}$ NMR (146 MHz, CDCl_3) spectrum of **5**.

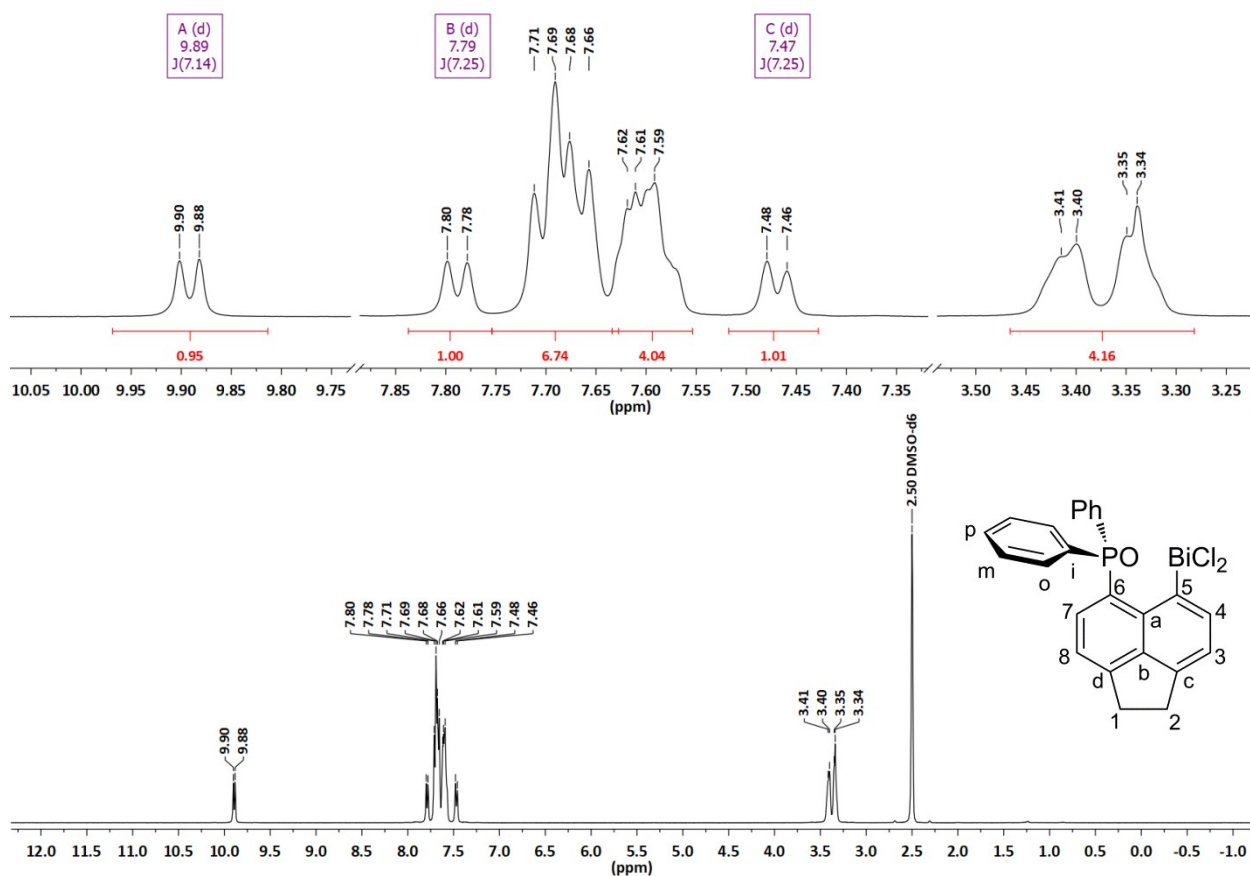


Figure S16. ^1H NMR (360 MHz, DMSO- d_6) spectrum of 6.

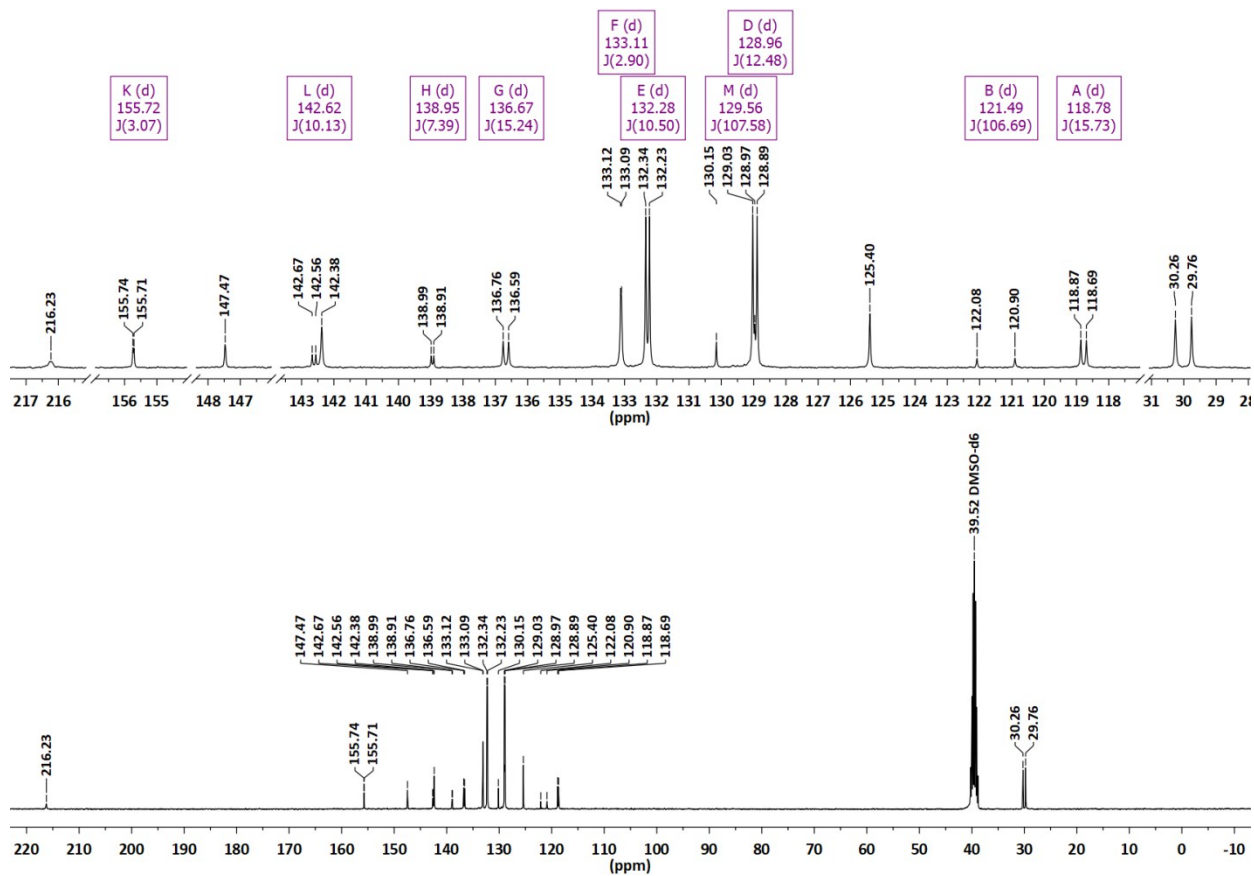


Figure S17. $^{13}\text{C}\{^1\text{H}\}$ NMR (91 MHz, DMSO- d_6) spectrum of 6.

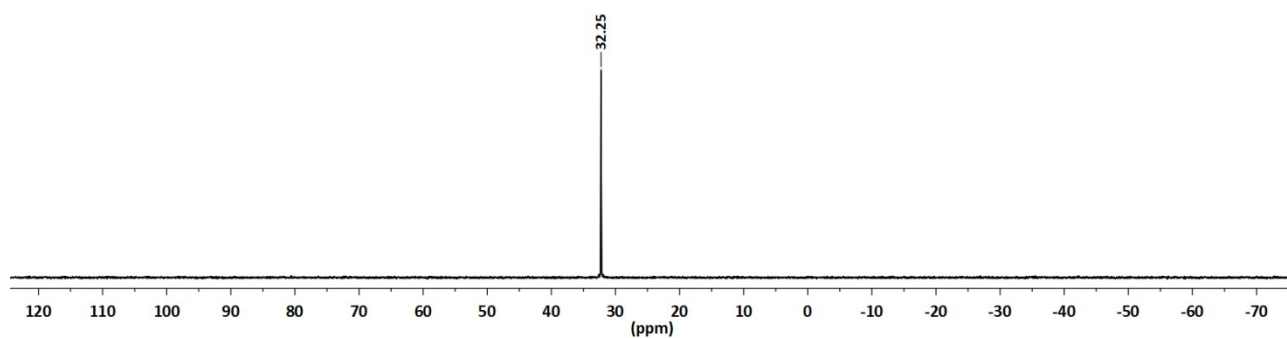


Figure S18. $^{31}\text{P}\{^1\text{H}\}$ NMR (146 MHz, $\text{DMSO-}d_6$) spectrum of **6**.

Computational data

Table S1. Topological and integrated bond properties from AIM and ELI-D.

	contact	d [Å]	$\rho(\mathbf{r})$ [eÅ ⁻³]	$\nabla^2\rho(\mathbf{r})$ [eÅ ⁻⁵]	ϵ	$G/\rho(\mathbf{r})$ [a.u.]	$H/\rho(\mathbf{r})$ [a.u.]	N_{ELI} [e]	V_{ELI} [Å ³]	γ_{ELI}	γ_{ELI}'
3	PO	1.498	1.59	26.9	0.01	2.11	-0.93	1.60	2.0	1.45	
5 ·THF	PO	1.508	1.55	25.2	0.04	2.05	-0.91	1.54	1.9	1.47	
6 ·THF	PO	1.509	1.55	25.2	0.01	2.05	-0.91	1.54	1.8	1.47	
5	PO	1.510	1.55	25.1	0.02	2.05	-0.92	1.56	1.9	1.46	
4 ·THF	PO	1.513	1.53	24.7	0.03	2.04	-0.90	1.51	1.7	1.48	
6	PO	1.513	1.54	24.7	0.01	2.04	-0.91	1.55	1.8	1.47	
4	PO	1.514	1.52	24.6	0.03	2.03	-0.90	1.49	1.7	1.47	
all	PO	1.509	1.55	25.2	0.02	2.05	-0.91	1.54	1.84	1.47	
3	HgC	2.076	0.89	2.7	0.04	0.68	-0.46	1.88	6.9	1.62	
4	SnC	2.164	0.70	2.2	0.10	0.65	-0.42	2.33	11.1	1.74	
4 ·THF	SnC	2.184	0.68	2.1	0.07	0.63	-0.42	2.23	8.3	1.77	
5 ·THF	SbC	2.204	0.71	0.6	0.06	0.53	-0.47	2.33	8.1	1.78	
5	SbC	2.209	0.70	0.7	0.07	0.53	-0.46	2.31	8.6	1.77	
6 ·THF	BiC	2.300	0.64	1.3	0.07	0.54	-0.39	2.13	8.6	1.74	
6	BiC	2.308	0.63	1.3	0.08	0.53	-0.39	2.13	9.0	1.73	
all	EC	2.206	0.71	1.6	0.07	0.58	-0.43	2.19	8.7	1.74	
4 ·THF	SnO1	2.198	0.44	6.4	0.01	1.12	-0.11	6.22	15.9	1.62	
4	SnO1	2.215	0.43	6.1	0.01	1.10	-0.11	5.50	14.6	1.62	
								0.74	1.0		1.54
sum	SnO	2.207	0.43	6.2	0.01	1.11	-0.11	6.22	15.7	1.62	1.54
5 ·THF	SbO1	2.413	0.32	3.0	0.06	0.77	-0.11	6.14	16.8	1.62	
6 ·THF	BiO1	2.444	0.32	3.9	0.02	0.90	-0.05	6.15	17.5	1.61	
5	SbO1	2.446	0.30	2.8	0.04	0.75	-0.11	6.12	16.9	1.61	
6	BiO1	2.464	0.31	3.8	0.01	0.88	-0.04	6.13	17.5	1.61	
sum	Sb/Bi-O	2.442	0.31	3.4	0.03	0.83	-0.08	6.13	17.2	1.61	
3	HgO	2.626	0.22	2.9	0.12	0.92	0.02	6.11	18.2	1.60	
4 ·THF	SnO2	2.667	0.17	1.6	0.08	0.69	-0.03	4.93	11.4	1.75	
6 ·THF	BiO2	2.850	0.15	1.5	0.15	0.69	0.02	5.02	12.3	1.74	
5 ·THF	SbO2	2.952	0.12	1.0	0.01	0.60	-0.01	4.99	12.4	1.74	

For all bonds, $\rho(\mathbf{r})_{\text{bcp}}$ is the electron density at the bond critical point, $\nabla^2\rho(\mathbf{r})_{\text{bcp}}$ is the corresponding Laplacian, ϵ is the bond ellipticity, $G/\rho(\mathbf{r})_{\text{bcp}}$ and $H/\rho(\mathbf{r})_{\text{bcp}}$ are the kinetic and total energy density over $\rho(\mathbf{r})_{\text{bcp}}$ ratios, N_{ELI} and V_{ELI} are electron populations and volumes of related ELI-D basins, γ_{ELI} is the ELI-D value at the attractor position, no. refers to the number of averaged bonds. E = Sn, Sb, Hg, Bi. O2 = O_{THF}.

Table S2. Topological and integrated bond properties from AIM and ELI-D.

	contact	d [Å]	$\rho(\mathbf{r})$ [eÅ ⁻³]	$\nabla^2\rho(\mathbf{r})$ [eÅ ⁻⁵]	ϵ	$G/\rho(\mathbf{r})$ [a.u.]	$H/\rho(\mathbf{r})$ [a.u.]	N_{ELI} [e]	V_{ELI} [Å ³]	γ_{ELI}	γ_{ELI}^*
3	HgCl	2.315	0.68	4.8	0.01	0.85	-0.36	7.58	38.1	1.65	
4	SnCl3	2.354	0.58	3.9	0.06	0.81	-0.34	7.76	37.6	1.67	
4 ·THF	SnCl2	2.355	0.58	3.9	0.07	0.81	-0.34	7.77	37.4	1.67	
4 ·THF	SnCl2	2.371	0.57	3.5	0.03	0.78	-0.35	7.10	34.4	1.67	
								0.66	1.1		1.45
5	SbCl2	2.377	0.59	2.5	0.06	0.69	-0.38	7.32	35.9	1.66	
								0.44	0.6		1.43
4 ·THF	SnCl2	2.391	0.53	3.7	0.03	0.80	-0.32	7.22	36.9	1.65	
								0.54	0.8		1.46
5 ·THF	SbCl2	2.401	0.56	2.5	0.05	0.68	-0.37	7.34	36.3	1.65	
4	SnCl1	2.408	0.52	3.4	0.01	0.78	-0.32	7.75	36.7	1.65	
4 ·THF	SnCl1	2.421	0.51	3.3	0.00	0.77	-0.32	7.35	35.7	1.65	
								0.41	0.6		1.46
5	SbCl1	2.465	0.50	2.3	0.03	0.66	-0.33	7.74	37.0	1.64	
6	BiCl2	2.485	0.52	3.0	0.03	0.71	-0.30	7.62	37.6	1.65	
5 ·THF	SbCl1	2.495	0.47	2.1	0.04	0.63	-0.32	7.76	36.6	1.63	
								0.42	0.6		1.44
5 ·THF	BiCl2	2.532	0.47	2.9	0.05	0.69	-0.27	7.63	37.8	1.64	
6	BiCl1	2.568	0.44	2.9	0.02	0.71	-0.25	7.61	37.9	1.62	
5 ·THF	BiCl1	2.592	0.42	2.8	0.03	0.70	-0.24	7.63	37.7	1.62	
all	ECl	2.435	0.53	3.2	0.03	0.74	-0.32	7.71	37.2	1.65	1.45
5	LP(Sb)							2.21	18.6	1.93	
5 ·THF	LP(Sb)							2.16	16.7	1.96	
6	LP(Bi)							1.93	16.9	1.53	
6 ·THF	LP(Bi)							1.69	13.6	1.56	

For all bonds, $\rho(\mathbf{r})_{\text{bcp}}$ is the electron density at the bond critical point, $\nabla^2\rho(\mathbf{r})_{\text{bcp}}$ is the corresponding Laplacian, ϵ is the bond ellipticity, $G/\rho(\mathbf{r})_{\text{bcp}}$ and $H/\rho(\mathbf{r})_{\text{bcp}}$ are the kinetic and total energy density over $\rho(\mathbf{r})_{\text{bcp}}$ ratios, N_{ELI} and V_{ELI} are electron populations and volumes of related ELI-D basins, γ_{ELI} is the ELI-D value at the attractor position, no. refers to the number of averaged bonds. E = Sn, Sb, Hg, Bi.

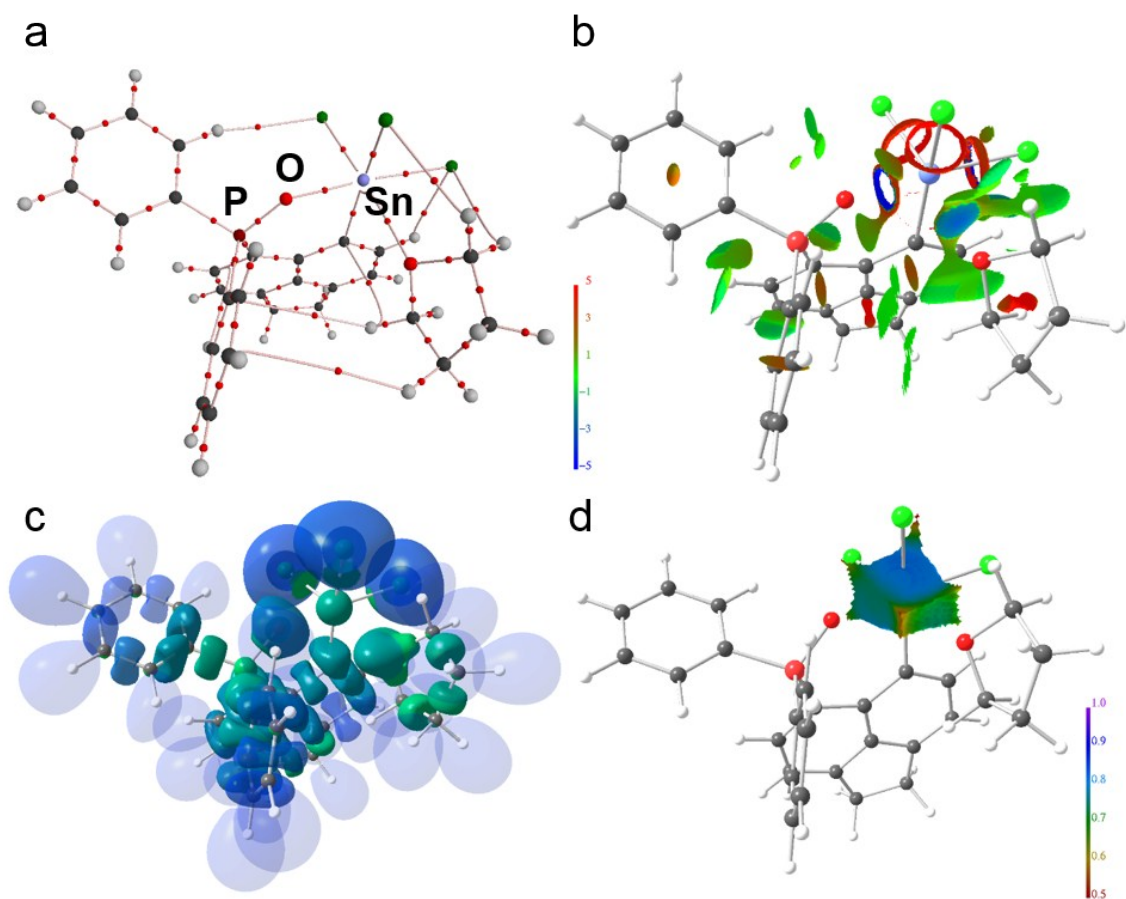


Figure S19. RSBI analysis of 4·THF. (a) AIM bond paths motif, (b) NCI *iso*-surface at $s(\mathbf{r}) = 0.5$, (c) ELI-D localization domain representation at *iso*-value of 1.3, (d) ELI-D distribution mapped on the Sn-P ELI-D basin.

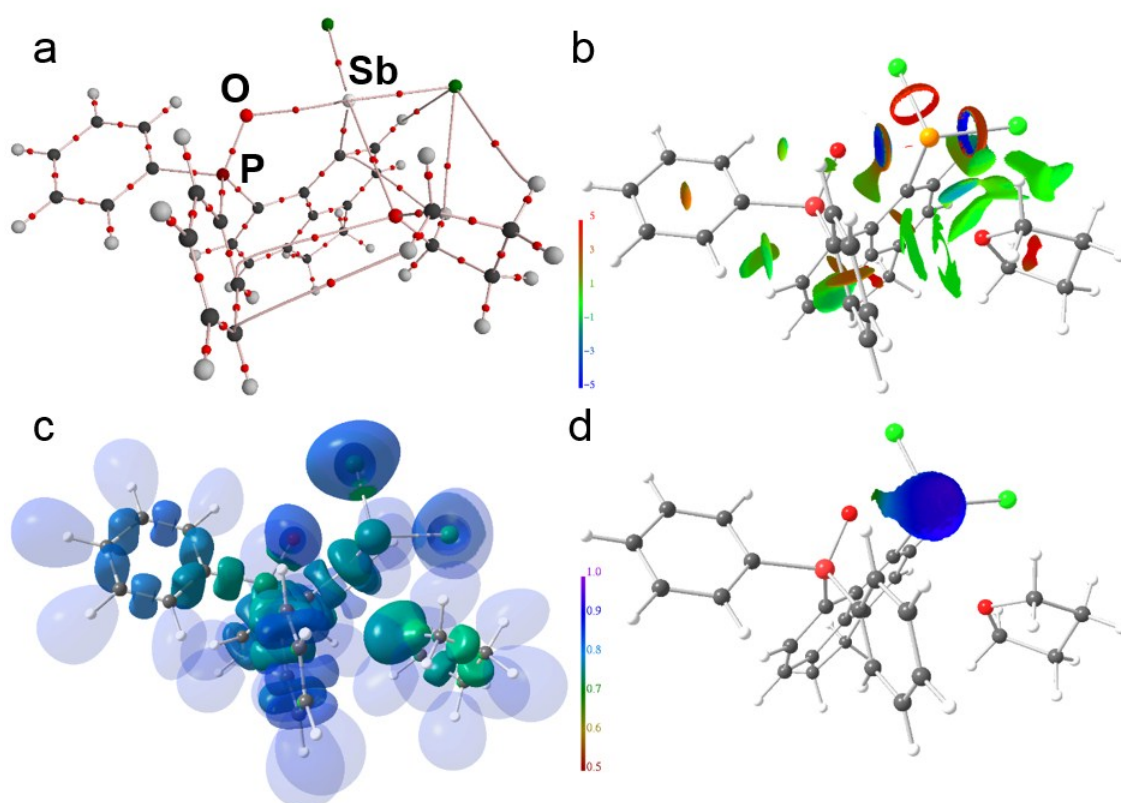


Figure S20. RSBI analysis of 5·THF (a) AIM bond paths motif, (b) NCI *iso*-surface at $s(\mathbf{r}) = 0.5$, (c) ELI-D localization domain representation at *iso*-value of 1.3, (d) ELI-D distribution mapped on the Sn-P ELI-D basin.

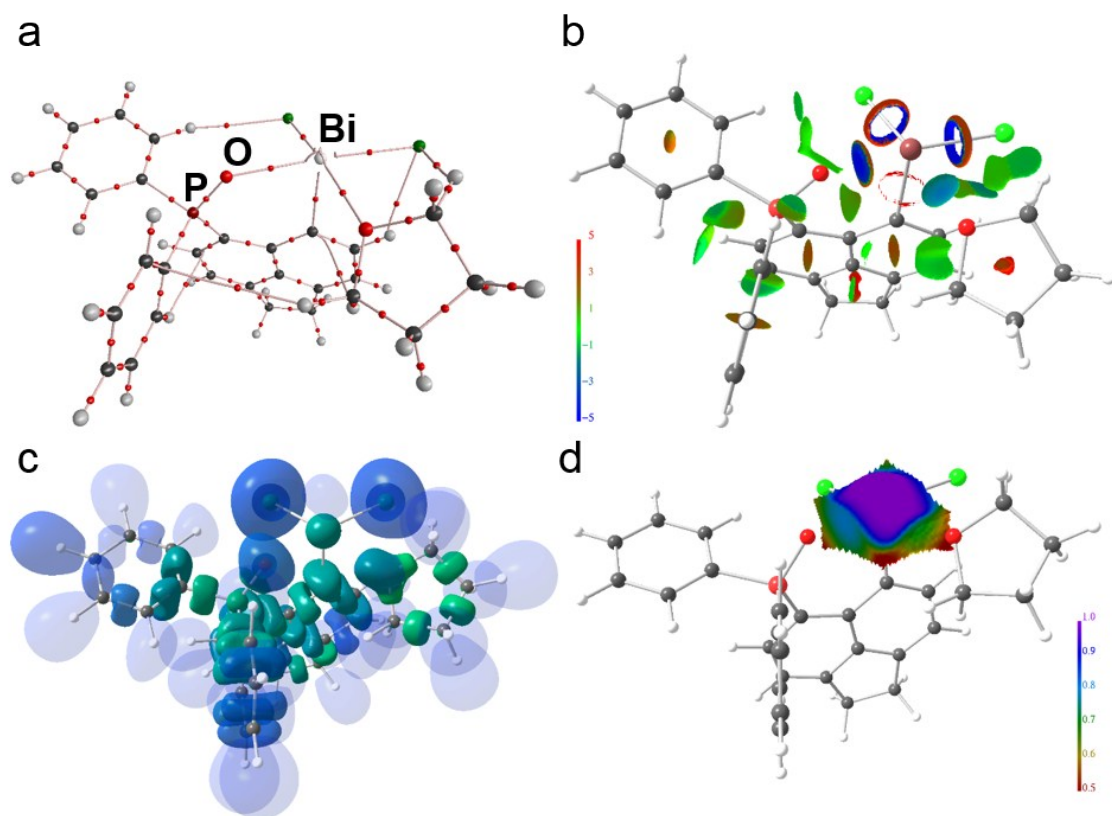


Figure S21. RSBI analysis of 6·THF (a) AIM bond paths motif, (b) NCI *iso*-surface at $s(\mathbf{r}) = 0.5$, (c) ELI-D localization domain representation at *iso*-value of 1.3, (d) ELI-D distribution mapped on the Sn-P ELI-D basin.

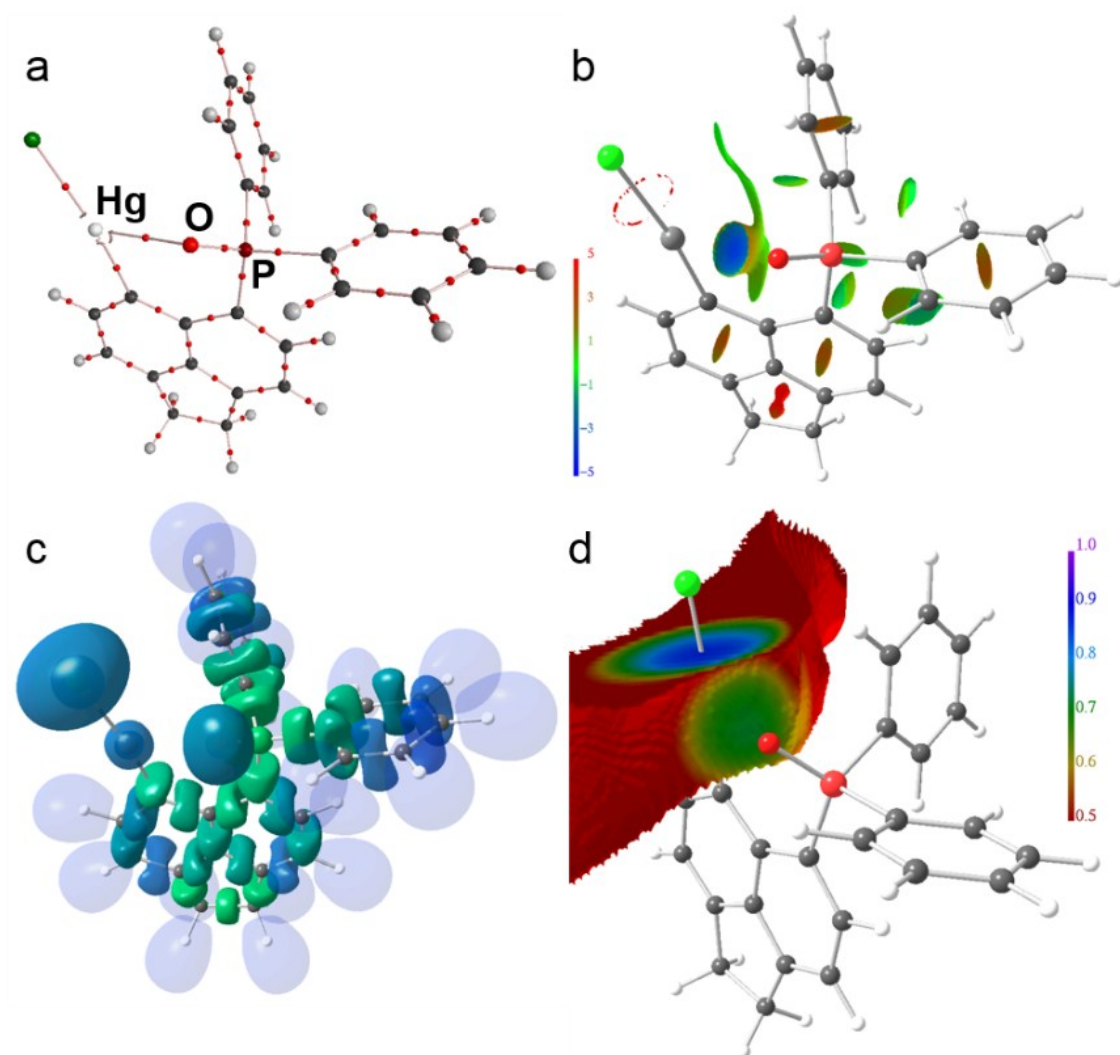


Figure S22 RSBI analysis of **3**. (a) AIM bond paths motif, (b) NCI *iso*-surface at $s(\mathbf{r}) = 0.5$, (c) ELI-D localization domain representation at *iso*-value of 1.3, (d) ELI-D distribution mapped on the Sn-P ELI-D basin.

Crystallographic data

Table S3. Crystal data and structure refinement

	1a ·2CH ₂ Cl ₂	3 ·THF	4
Formula	C ₉₈ H ₇₆ Cl ₅ Hg ₂ LiO ₄ P ₄	C ₂₄ H ₁₈ ClHgOP	C ₂₄ H ₁₈ Cl ₃ OPSn
Formula weight, g mol ⁻¹	2026.84	589.39	578.39
Crystal system	Triclinic	Monoclinic	Triclinic
Crystal size, mm	0.08 × 0.08 × 0.08	0.07 × 0.05 × 0.05	0.06 × 0.05 × 0.04
Space group	P $\bar{1}$	P2 ₁ /n	P $\bar{1}$
<i>a</i> , Å	14.2748(8)	10.8014(4)	8.9934(4)
<i>b</i> , Å	15.6725(8)	10.3777(4)	9.5252(4)
<i>c</i> , Å	21.2164(11)	18.5406(8)	14.9658(6)
α , °	105.246(2)	90	102.200(1)
β , °	98.704(2)	105.655(1)	98.392(1)
γ , °	94.464(2)	90	113.284(1)
<i>V</i> , Å ³	4492.2(4)	2001.2(1)	1112.79(8)
<i>Z</i>	2	4	2
ρ_{calcd} , Mg m ⁻³	1.498	1.956	1.726
μ (Mo <i>K</i> α), mm ⁻¹	3.684	7.917	1.595
<i>F</i> (000)	2008	1128	572
θ range, deg	2.60 to 28.28	2.27 to 30.57	2.44 to 31.61
Index ranges	-19 ≤ <i>h</i> ≤ 19	-15 ≤ <i>h</i> ≤ 15	-13 ≤ <i>h</i> ≤ 13
	-20 ≤ <i>k</i> ≤ 20	-14 ≤ <i>k</i> ≤ 14	-13 ≤ <i>k</i> ≤ 14
	-28 ≤ <i>l</i> ≤ 25	-26 ≤ <i>l</i> ≤ 26	-22 ≤ <i>l</i> ≤ 22
No. of reflns collected	126062	76418	43017
Completeness to θ_{max}	99.9%	99.8%	99.4%
No. indep. Reflns	22272	6128	7411
No. obsd reflns with (<i>I</i> > 2 σ (<i>I</i>))	18428	5850	6826
No. refined params	1027	253	271
GooF (<i>F</i> ²)	1.078	1.176	1.062
<i>R</i> ₁ (<i>F</i>) (<i>I</i> > 2 σ (<i>I</i>))	0.0544	0.0198	0.0246
<i>wR</i> ₂ (<i>F</i> ²) (all data)	0.1216	0.0516	0.0573
Largest diff peak/hole, e Å ⁻³	4.052 / -3.526	0.651 / -2.109	0.636 / -1.399
CCDC number	1897901	1897902	1897903

Table S3. cont

4·THF	5	5·THF	6
C ₃₂ H ₃₄ Cl ₃ O ₃ PSn	C ₂₄ H ₁₈ Cl ₂ OPSb	C ₂₈ H ₂₆ Cl ₂ O ₂ PSb	C ₂₅ H ₂₀ BiCl ₄ OP
722.60	546.00	618.11	718.16
Triclinic	Monoclinic	Triclinic	Triclinic
0.08 × 0.06 × 0.04	0.07 × 0.06 × 0.04	0.08 × 0.07 × 0.07	0.08 × 0.07 × 0.05
$\bar{1}$	P2 ₁ /n	$\bar{1}$	$\bar{1}$
8.8921(3)	8.6794(3)	9.2077(3)	9.1561(3)
10.4750(4)	12.6557(5)	9.6481(4)	11.7354(4)
16.9858(7)	19.5559(8)	15.3566(6)	12.0175(5)
85.291(1)	90	97.547(1)	90.800(1)
84.161(1)	97.497(1)	97.893(1)	105.168(1)
72.333(1)	90	110.393(1)	95.897(1)
1497.5(1)	2129.73(14)	1242.80(8)	1238.58(8)
2	4	2	2
1.603	1.703	1.652	1.926
1.207	1.635	1.414	7.630
732	1080	620	688
2.32 to 30.58	2.28 to 28.35	2.30 to 28.42	2.32 to 35.63
−12 ≤ h ≤ 12	−12 ≤ h ≤ 12	−12 ≤ h ≤ 11	−15 ≤ h ≤ 15
−14 ≤ k ≤ 14	−17 ≤ k ≤ 18	−12 ≤ k ≤ 12	−19 ≤ k ≤ 19
−24 ≤ l ≤ 24	−27 ≤ l ≤ 27	−20 ≤ l ≤ 20	−19 ≤ l ≤ 18
48875	98732	73343	63344
99.6%	99.8%	99.2%	99.9%
9171	6549	6215	11415
8229	6038	5770	10581
361	262	307	289
1.068	1.244	1.064	1.126
0.0257	0.0214	0.0233	0.0247
0.0546	0.0610	0.0589	0.0577
0.740 / −0.801	0.559 / −1.204	0.567 / −1.294	2.568 / −3.007
1897904	1897905	1897906	1897907