Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.

Self-association of diphenylpnictoginic acids in solution and solid state: covalent vs hydrogen bonding

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Supporting Information

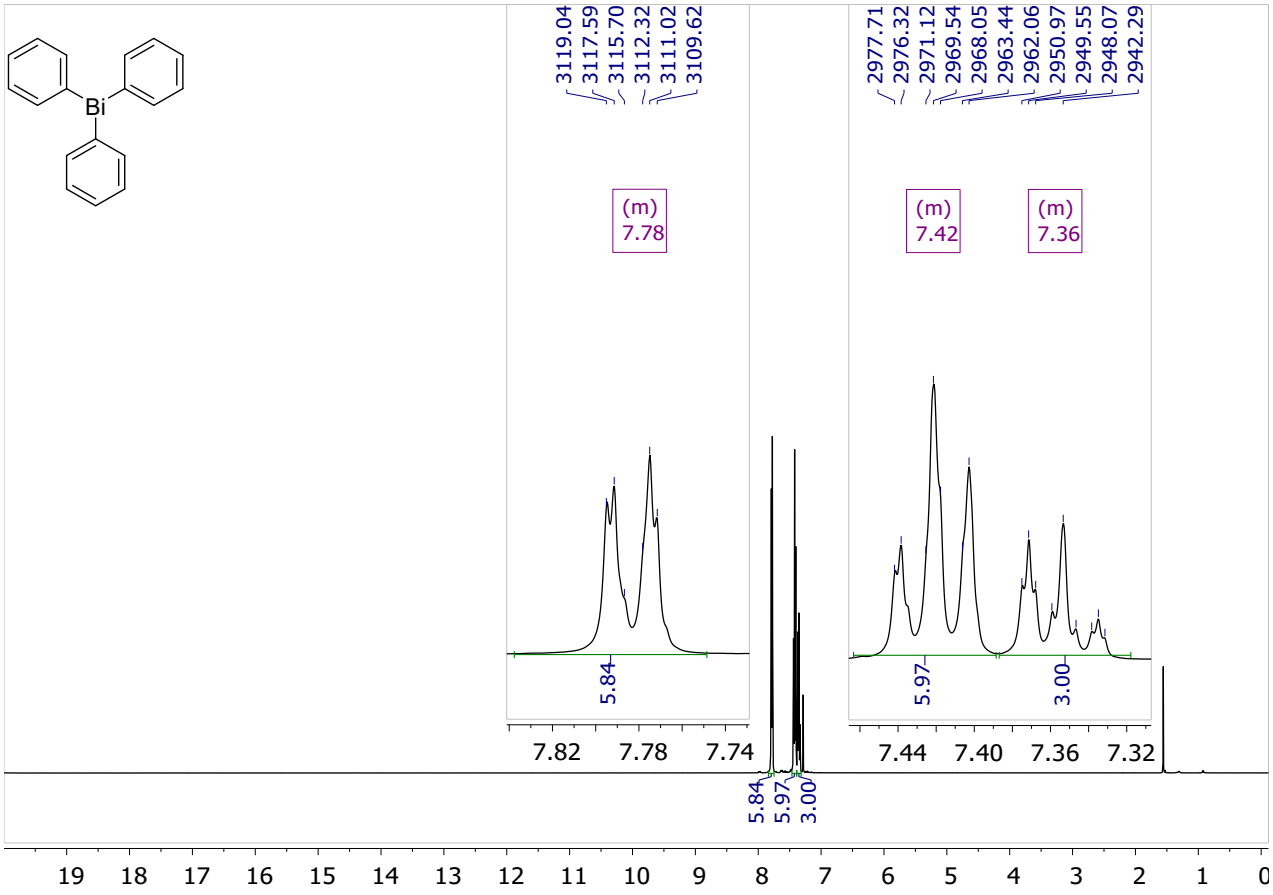

Figure S1: ^{1}H NMR spectrum of **2** ($CDCl_3$, 400 MHz).

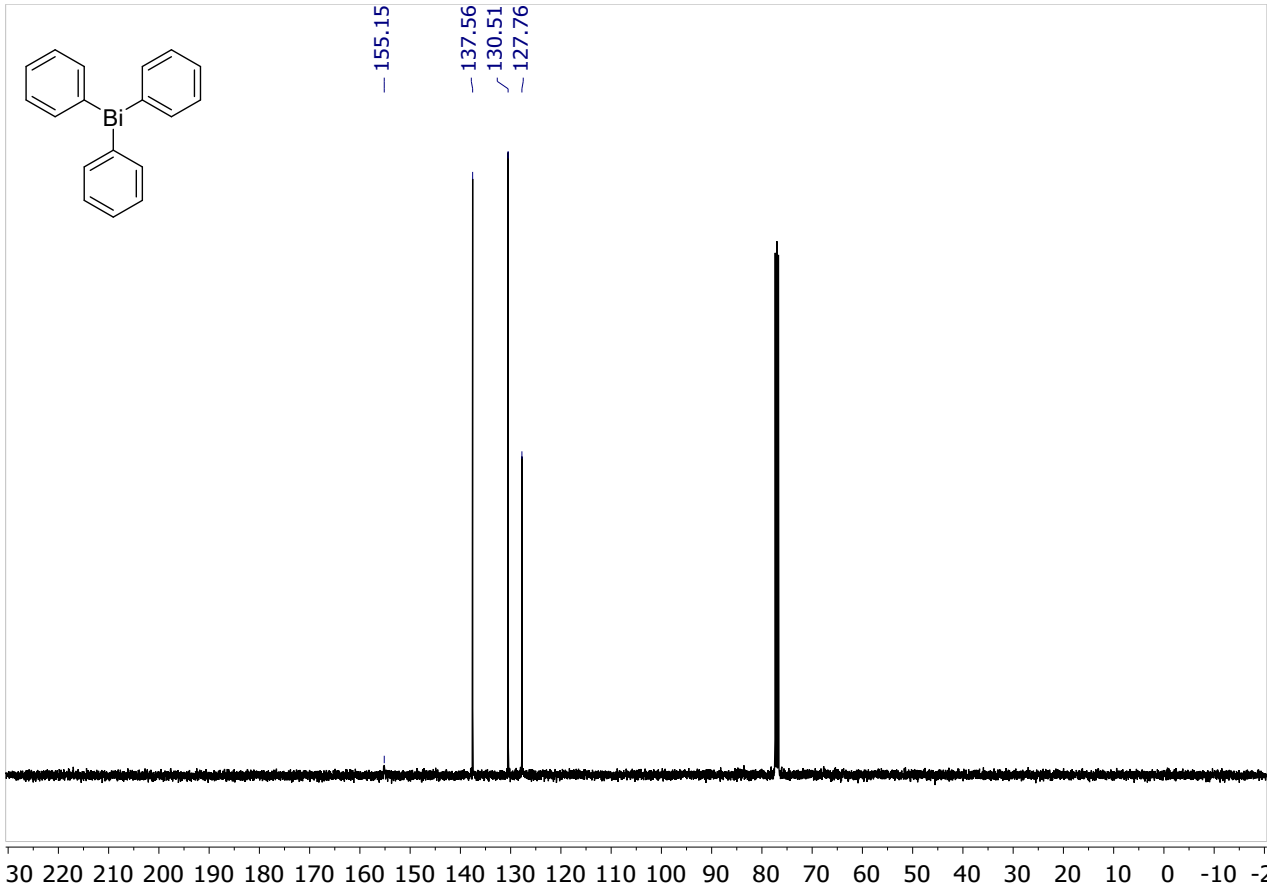

Figure S2: $^{13}C\{^{1}H\}$ NMR spectrum of **2** ($CDCl_3$, 100 MHz).

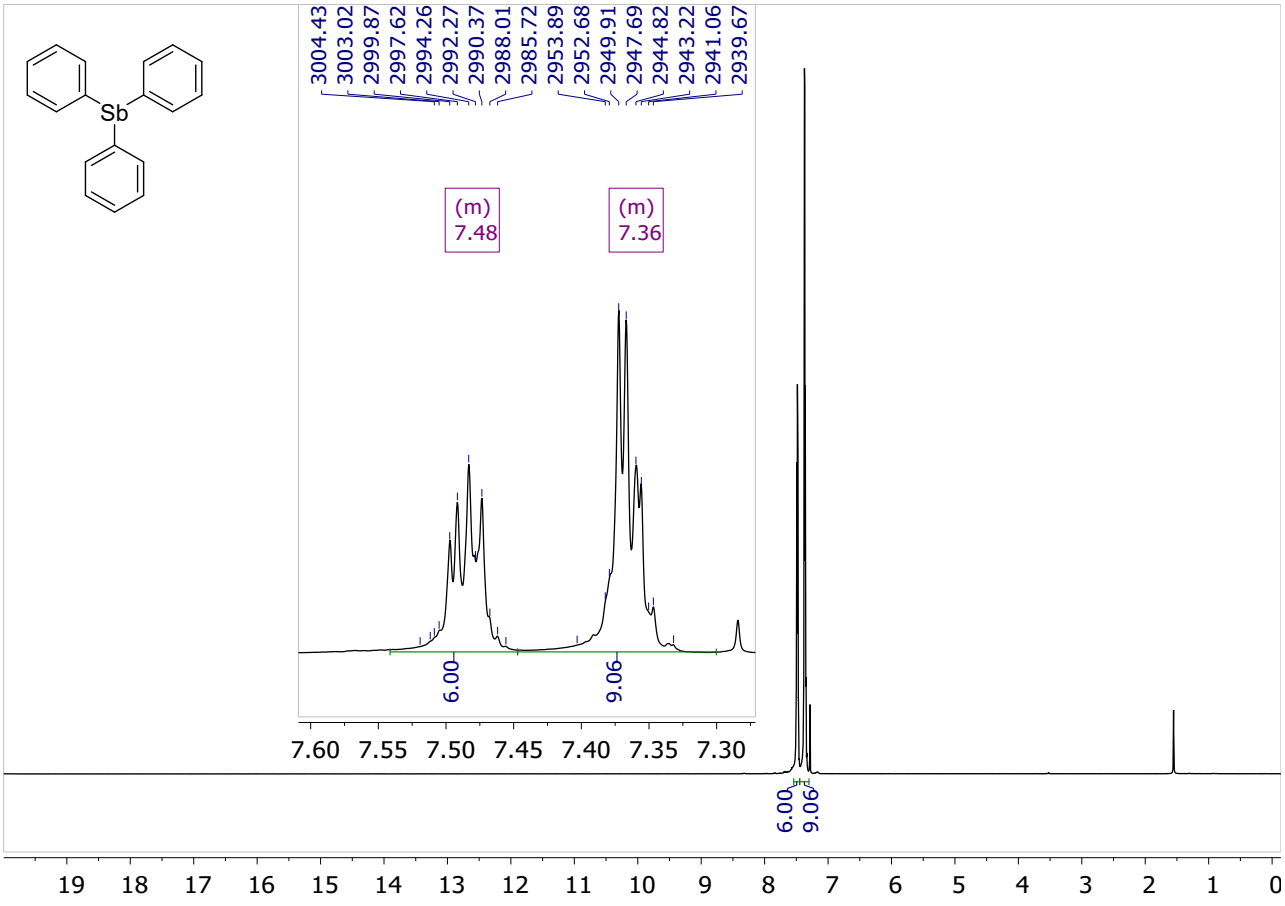

Figure S3: ^{1}H NMR spectrum of **3** ($CDCl_3$, 400 MHz).

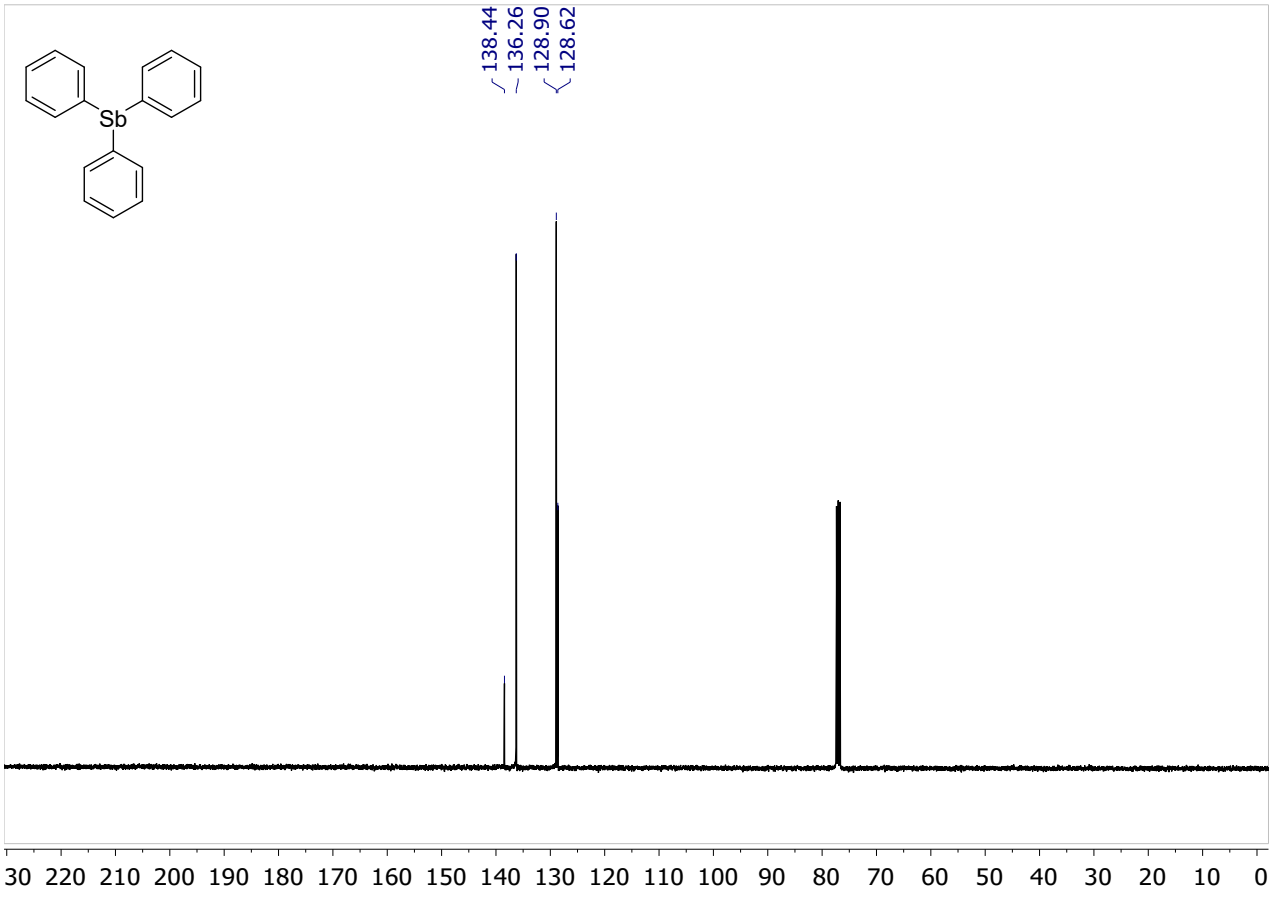

Figure S4: $^{13}C\{^{1}H\}$ NMR spectrum of **3** ($CDCl_3$, 100 MHz).

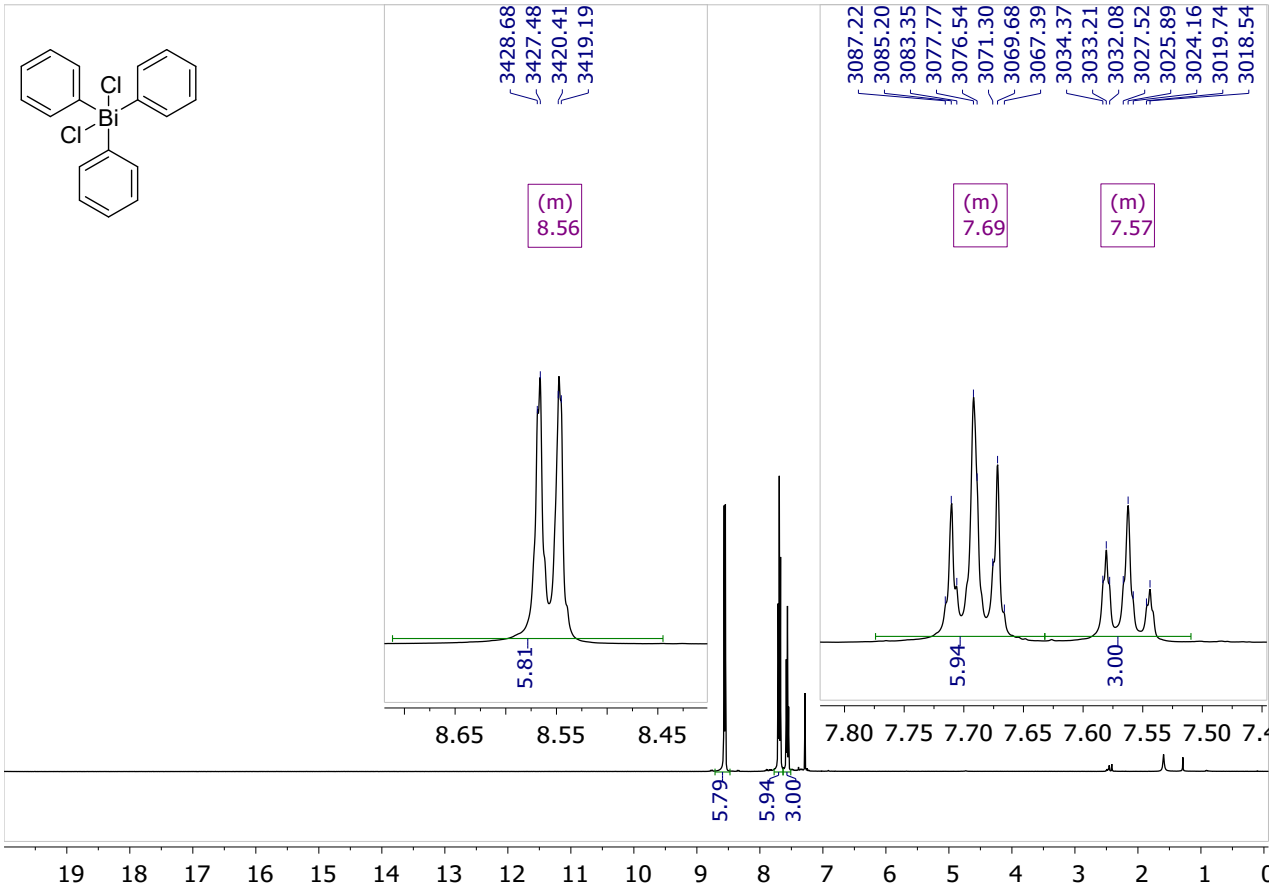

Figure S5: ^{1}H NMR spectrum of **6** ($CDCl_3$, 400 MHz).

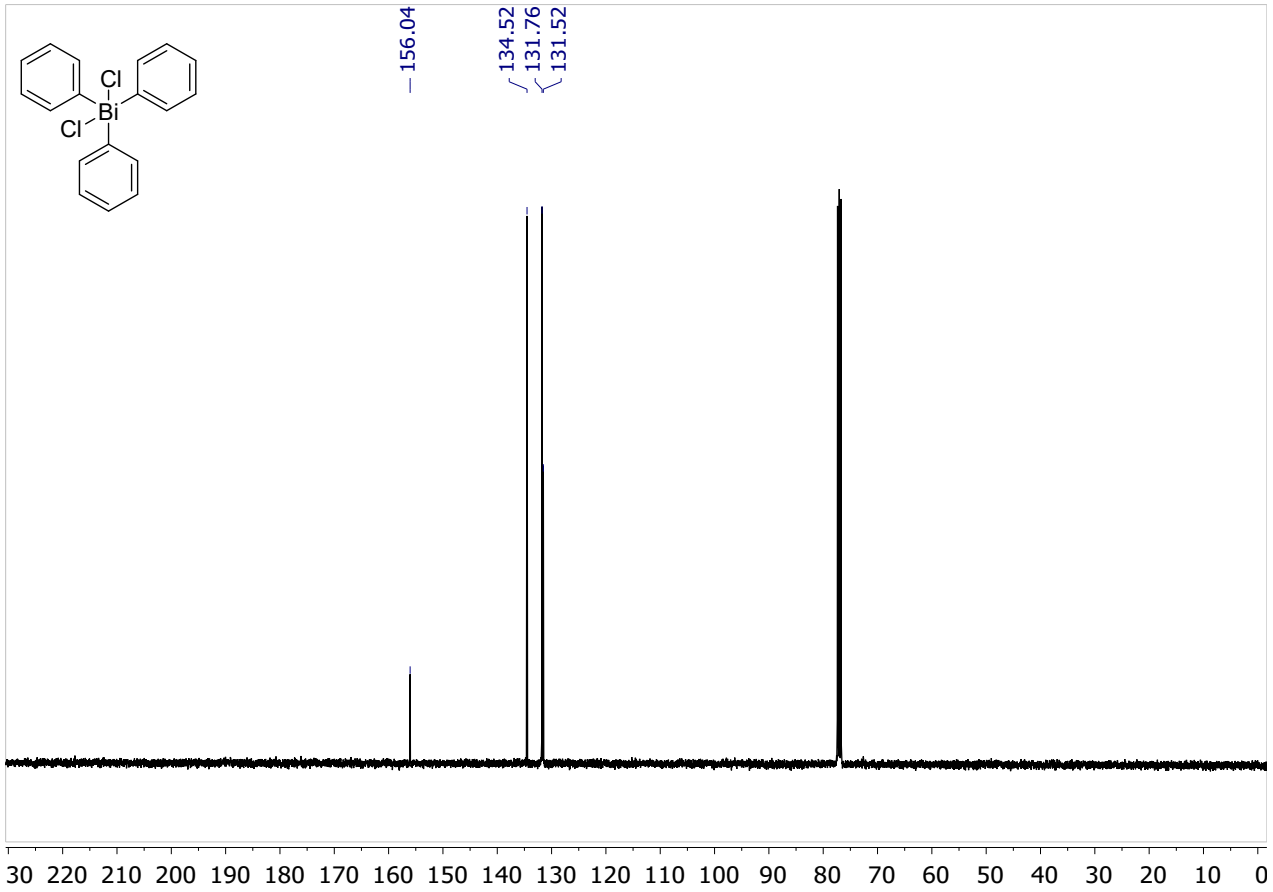

Figure S6: $^{13}C\{^{1}H\}$ NMR spectrum of **6** ($CDCl_3$, 100 MHz).

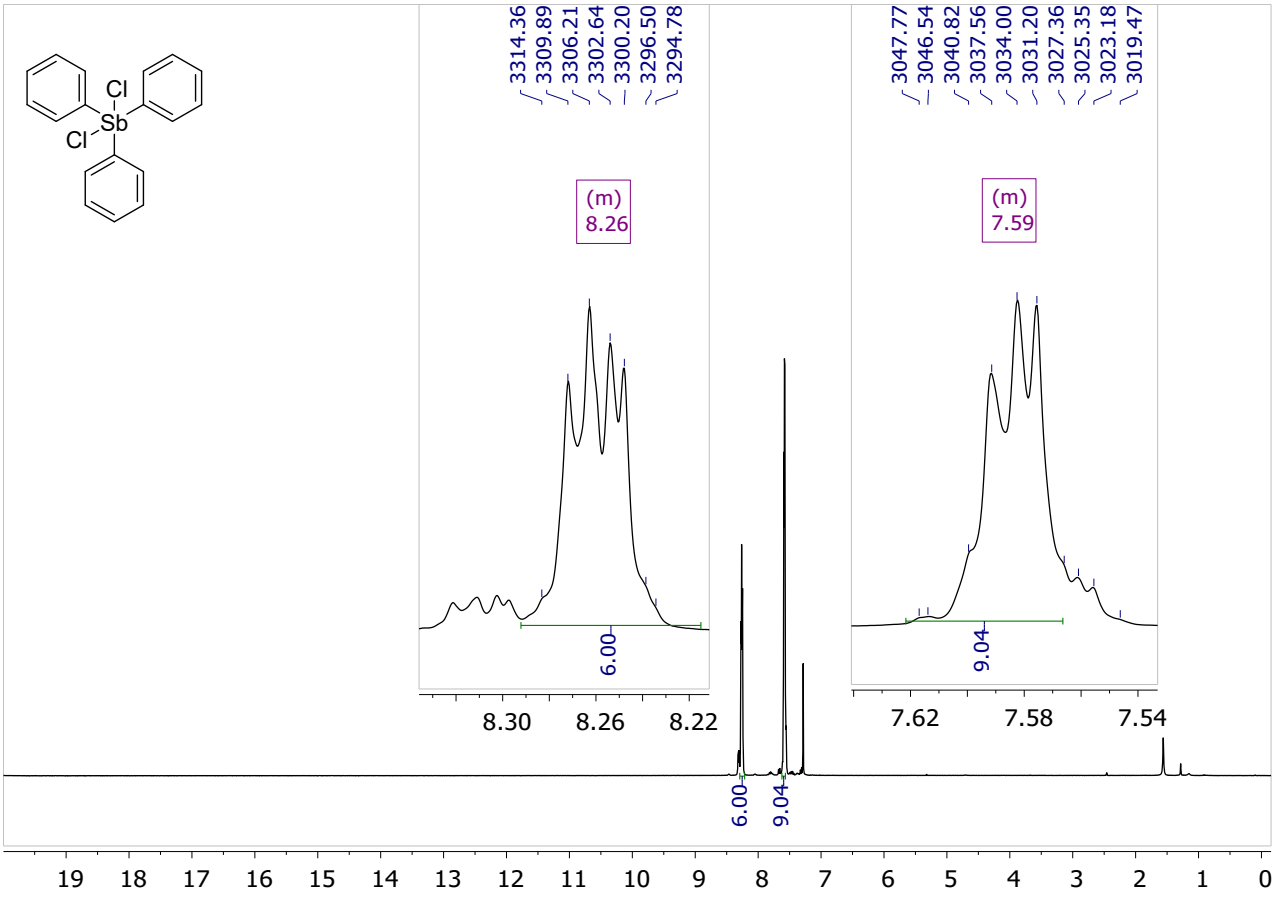

Figure S7: ^{1}H NMR spectrum of **7** ($CDCl_3$, 400 MHz).

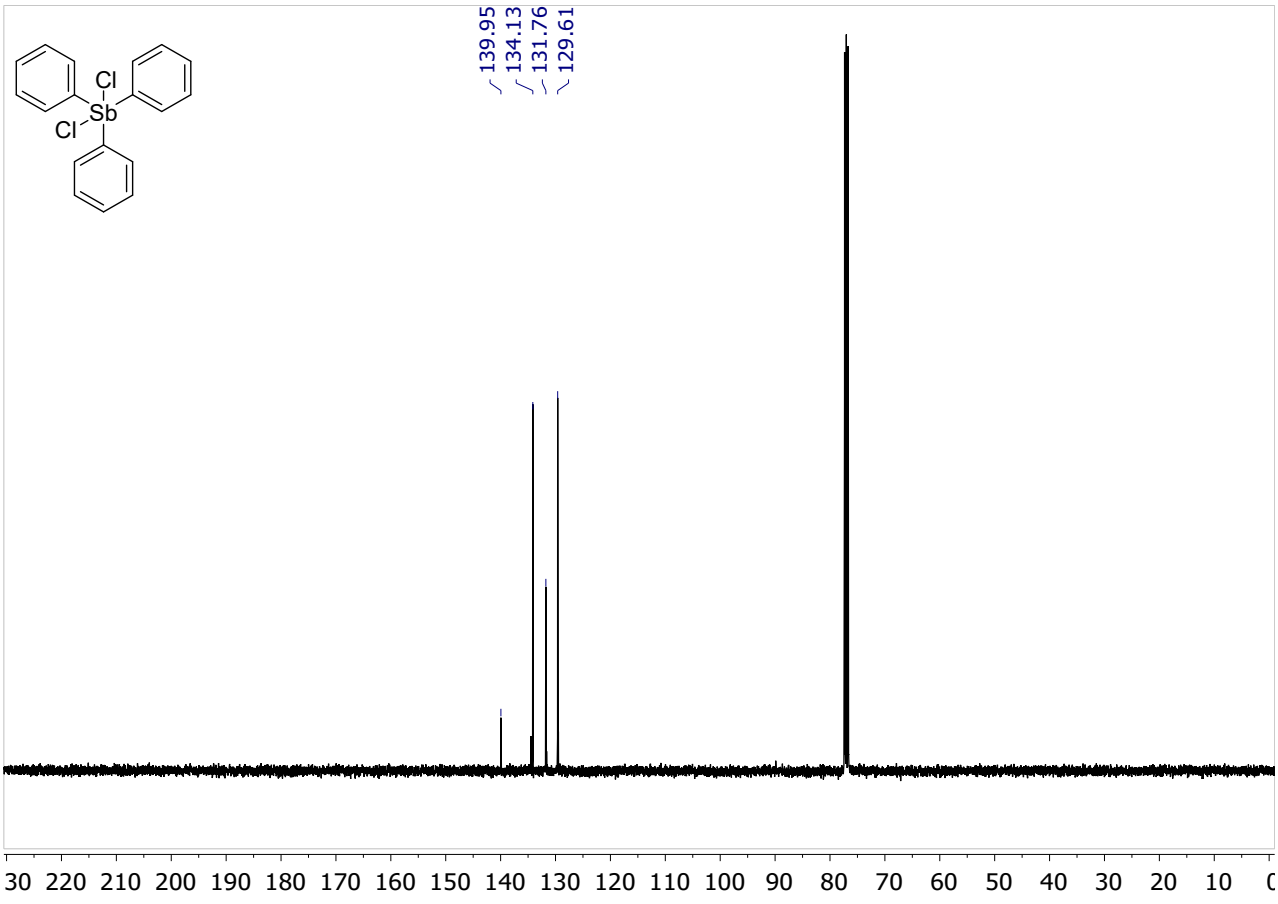

Figure S8: $^{13}C\{^{1}H\}$ NMR spectrum of **7** ($CDCl_3$, 100 MHz).

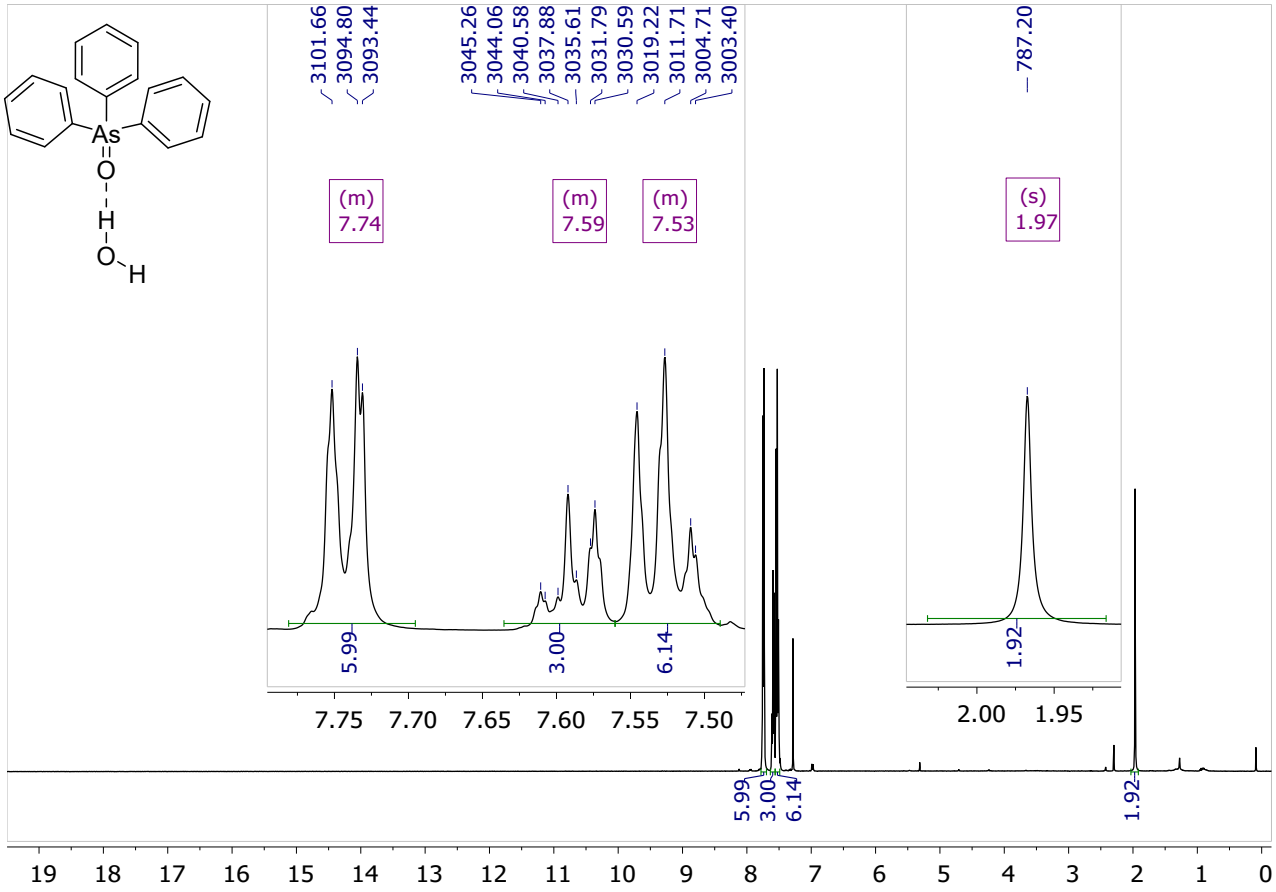

Figure S9: ^{1}H NMR spectrum of **8**·H_2O ($CDCl_3$, 400 MHz).

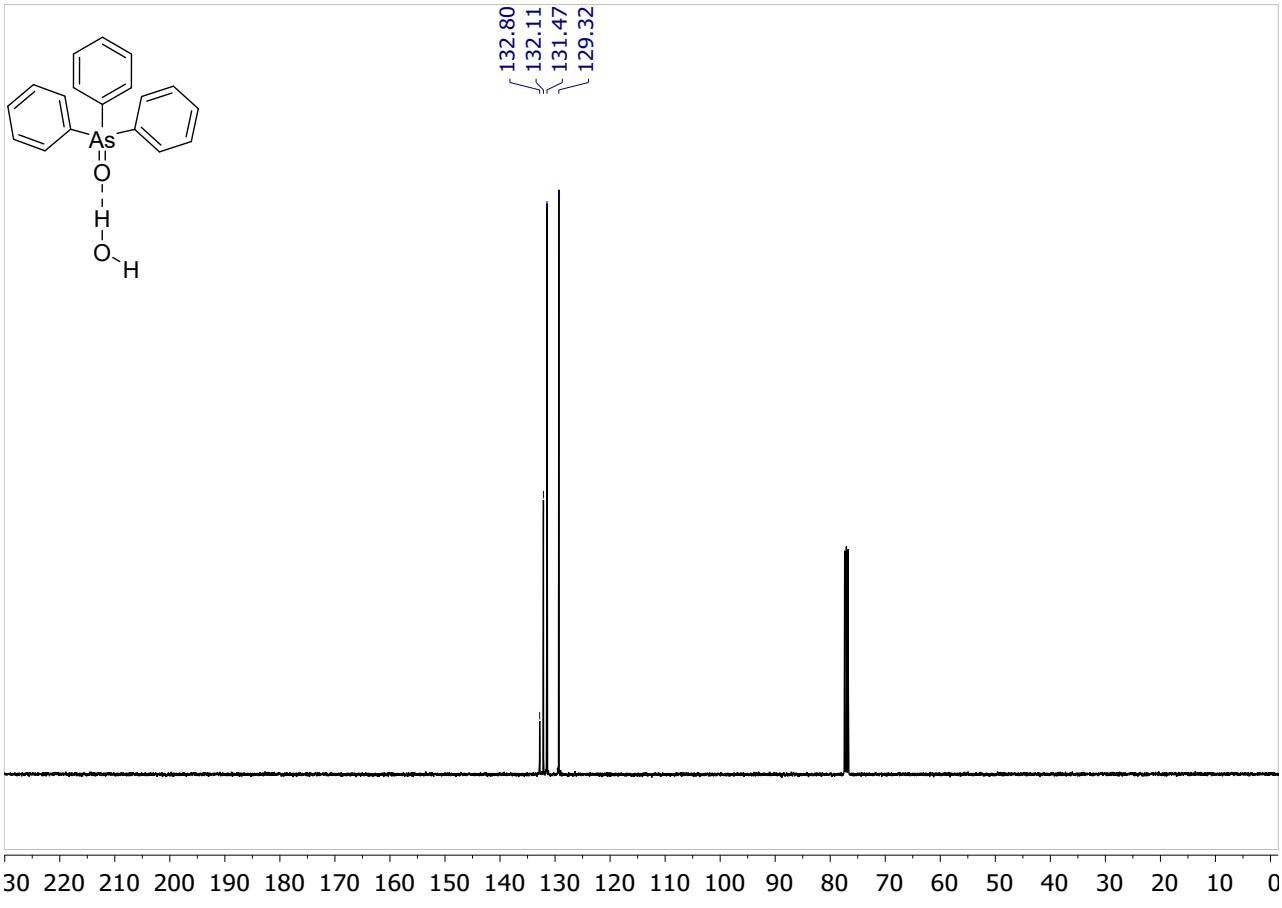

Figure S10: $^{13}C\{^{1}H\}$ NMR spectrum of **8**·H_2O ($CDCl_3$, 100 MHz).

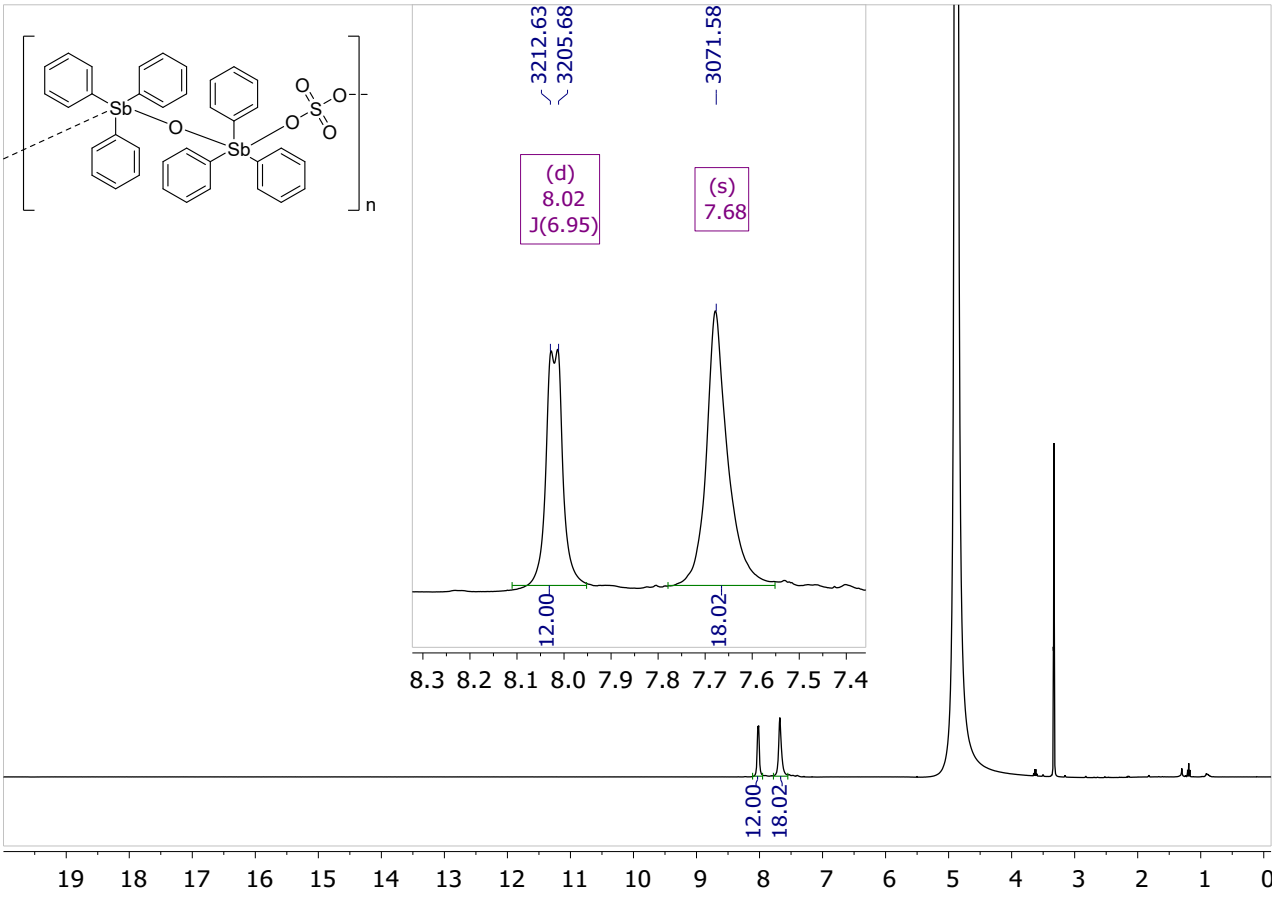

Figure S11: ^{1}H NMR spectrum of **12** (CD_3OH, 400 MHz).

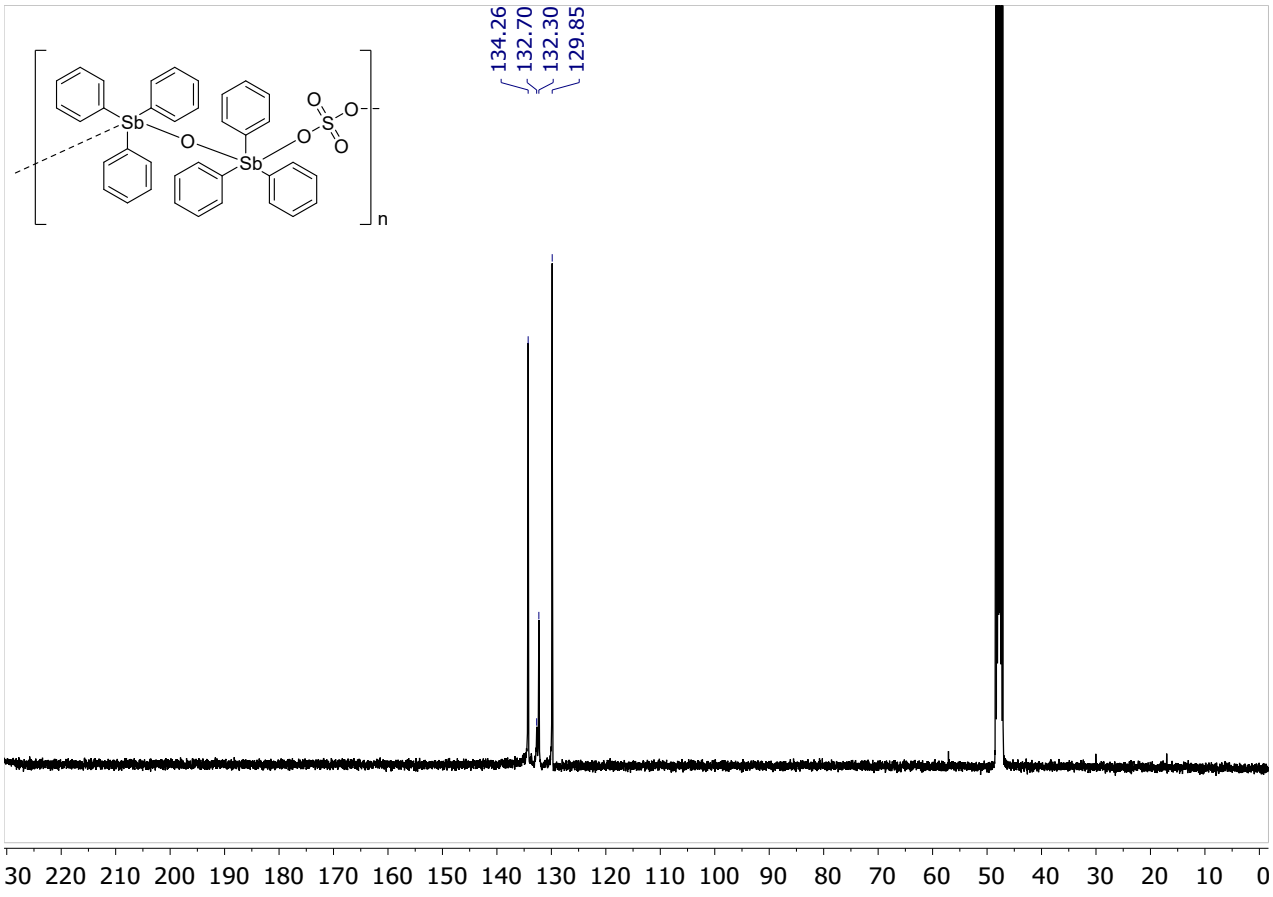

Figure S12: $^{13}C\{^{1}H\}$ NMR spectrum of **12** (CD_3OH, 100 MHz).

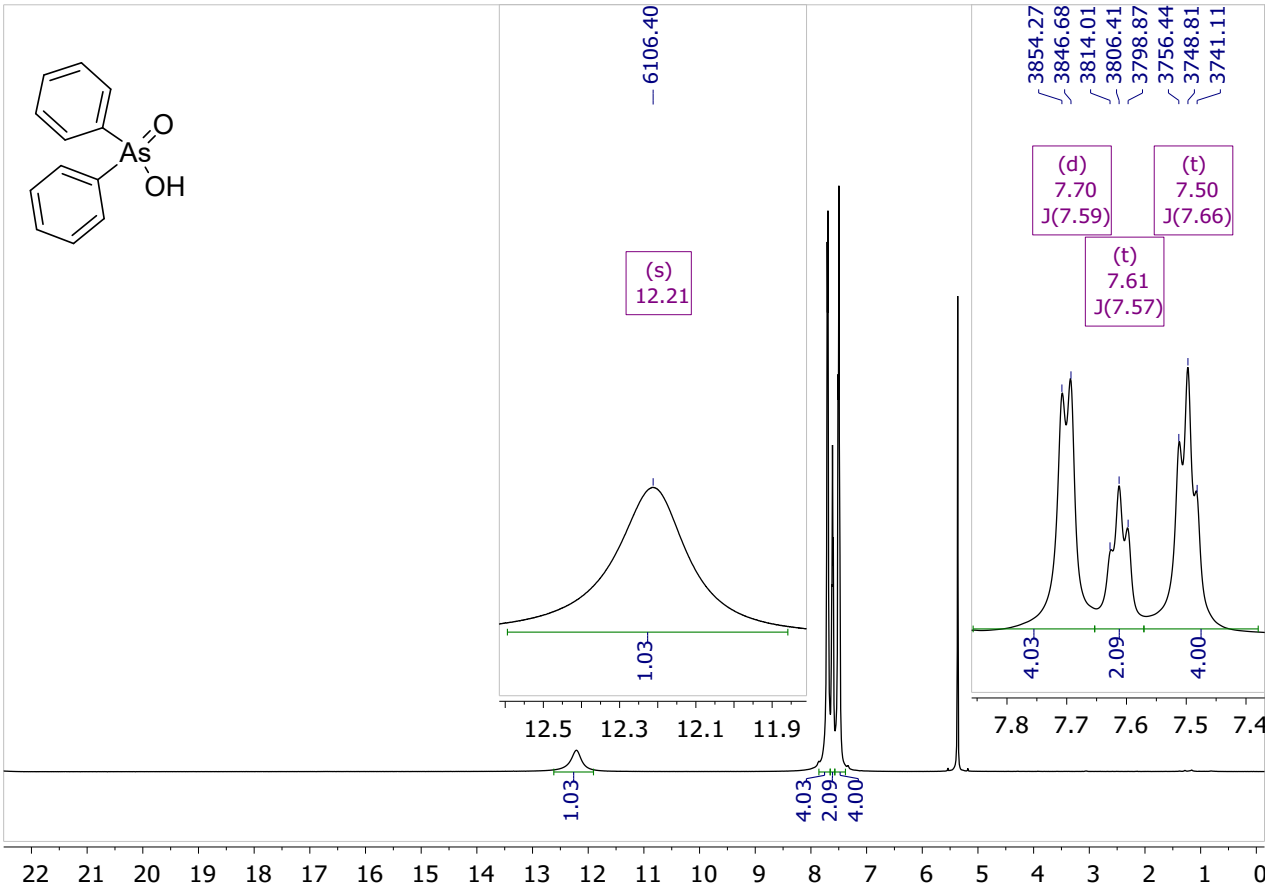

Figure S13: ^{1}H NMR spectrum of **14** (CD_2Cl_2, 500 MHz, 180K).

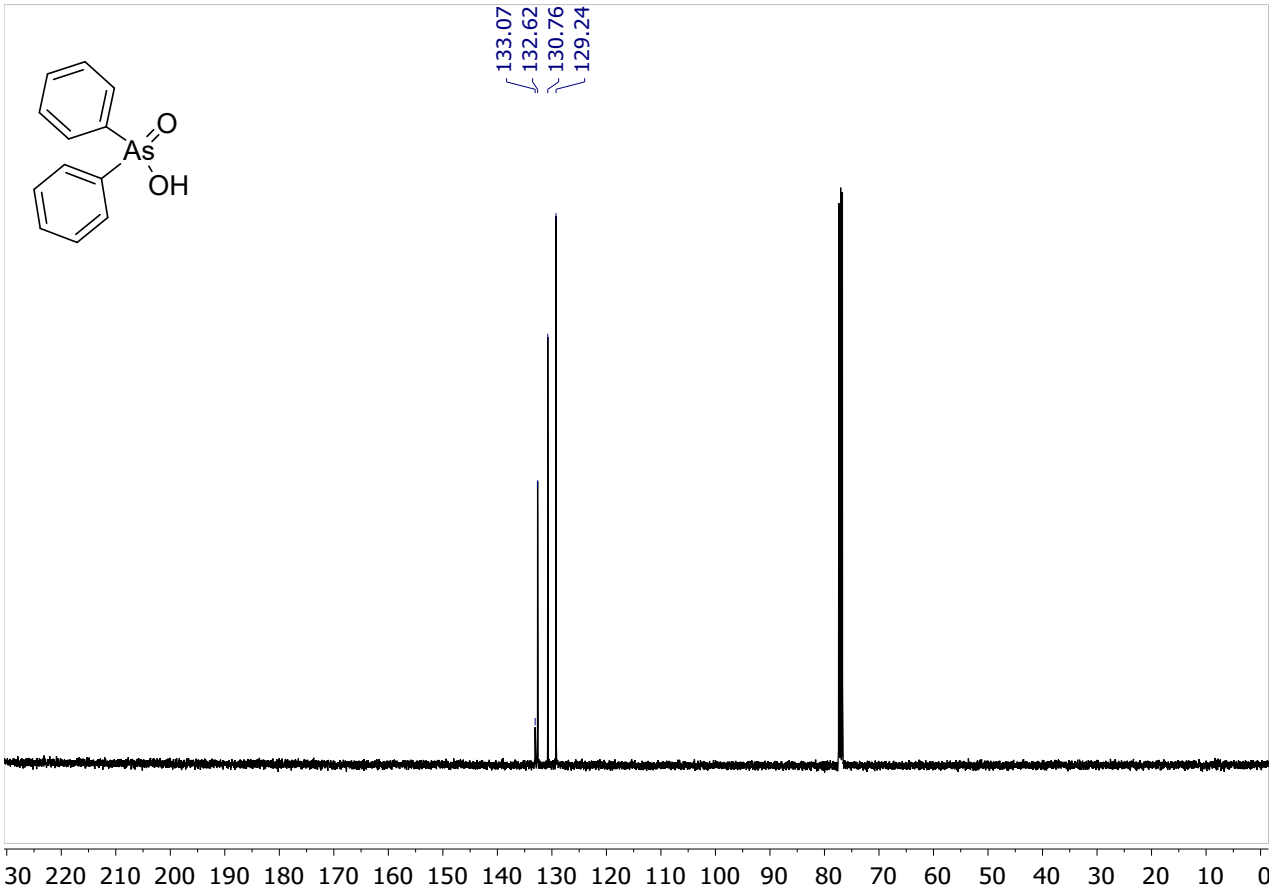

Figure S14: $^{13}C\{^{1}H\}$ NMR spectrum of **14** ($CDCl_3$, 100 MHz).

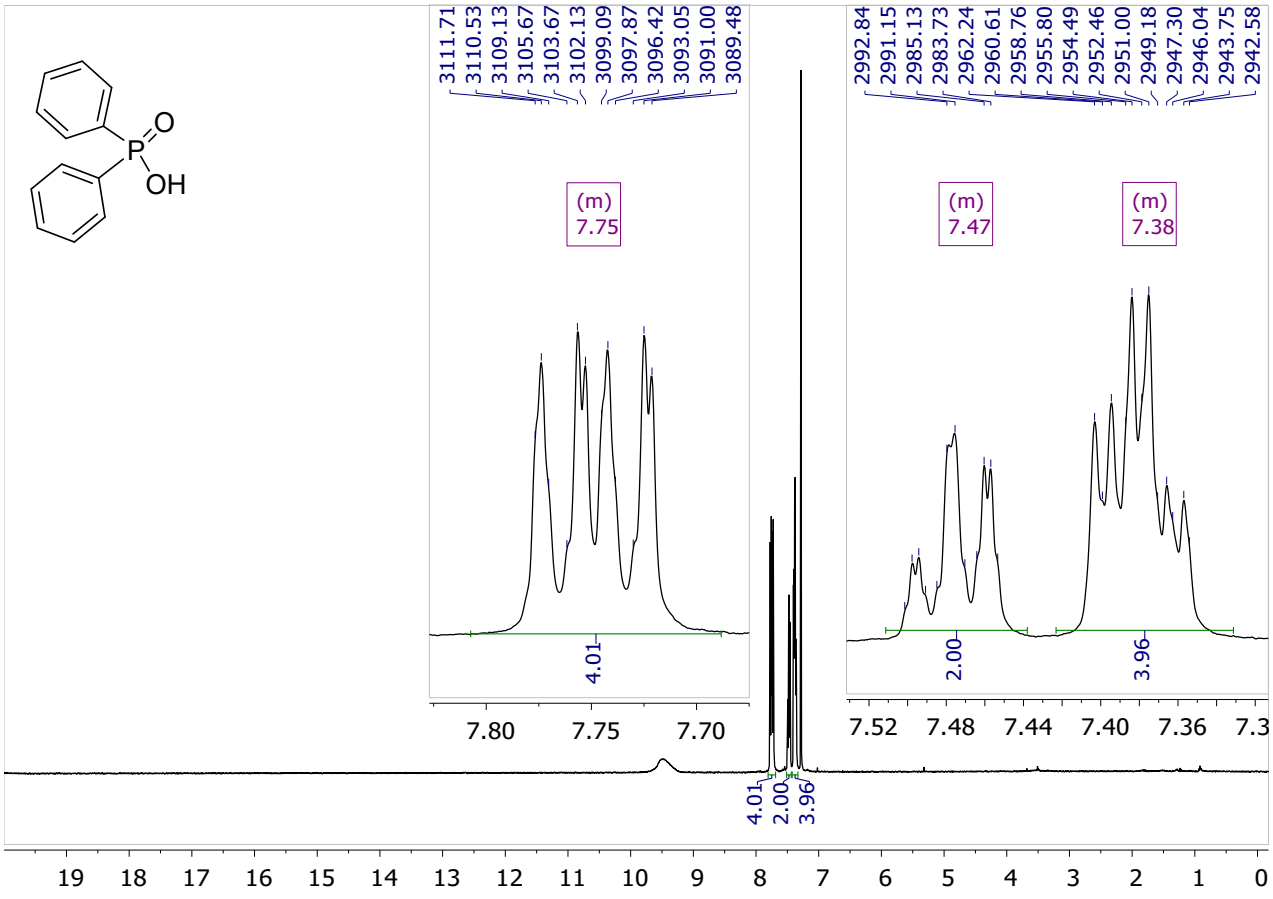

Figure S15: ^{1}H NMR spectrum of **15** ($CDCl_3$, 400 MHz).

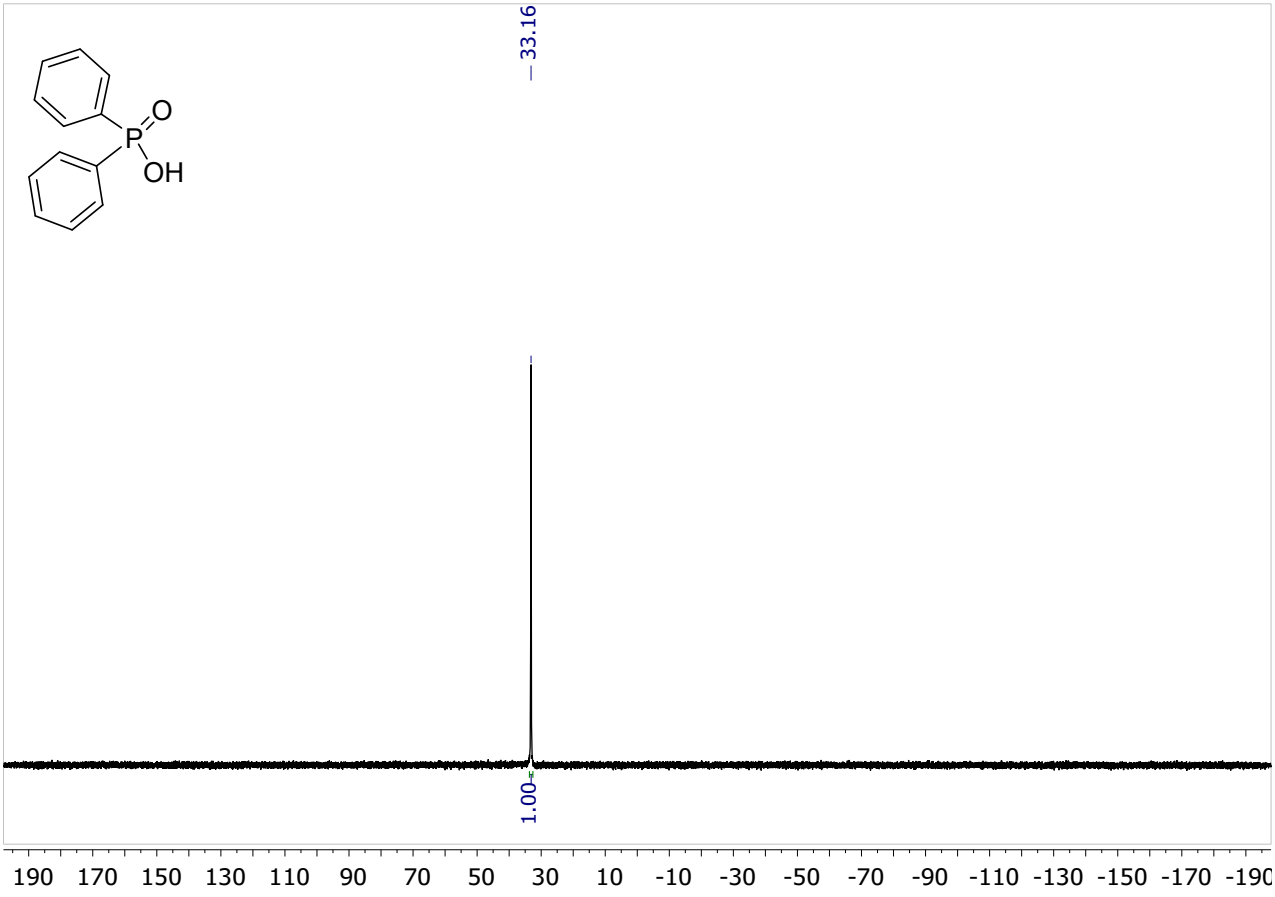

Figure S16: $^{31}P\{^{1}H\}$ NMR spectrum of **15** ($CDCl_3$, 162 MHz).

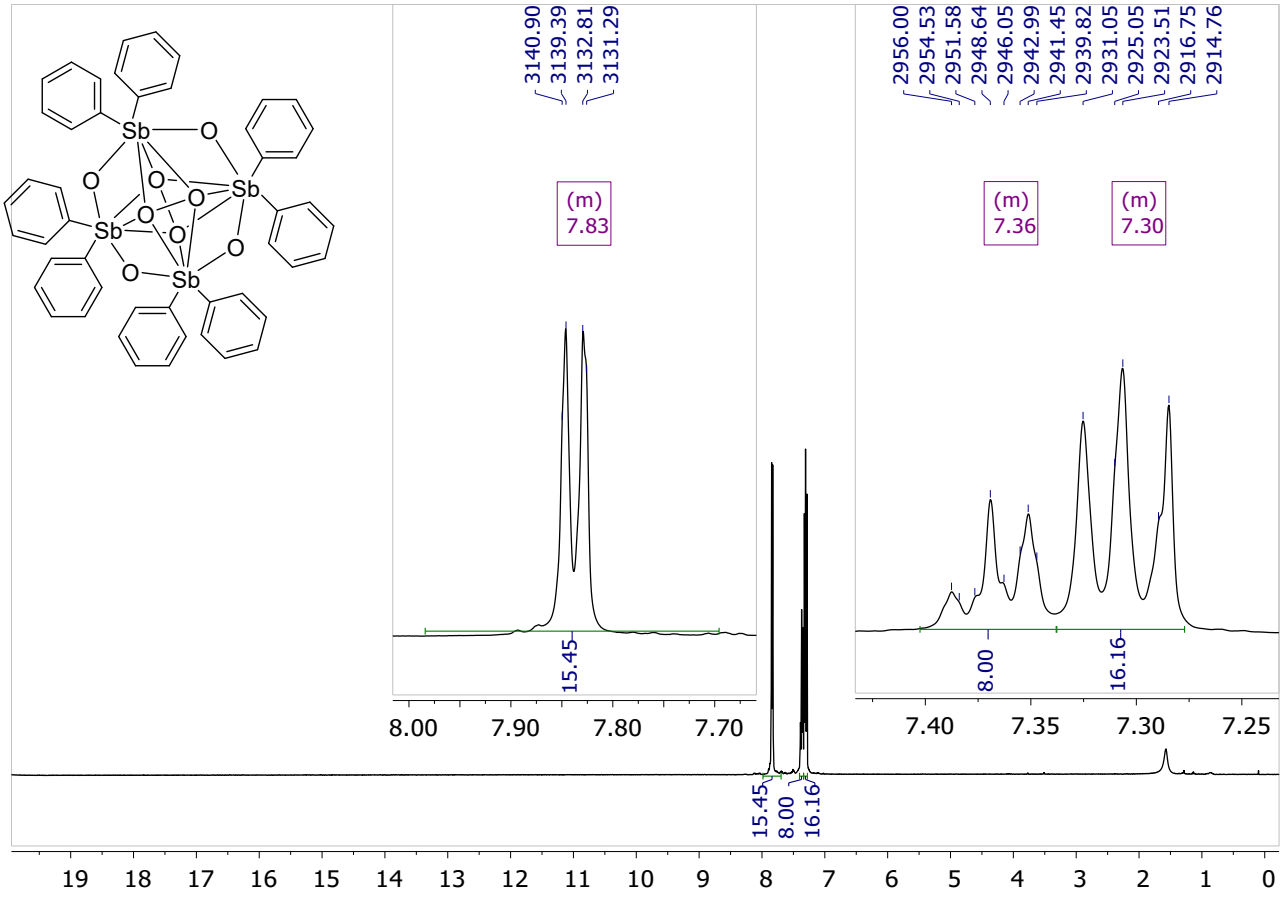

Figure S17: ^{1}H NMR spectrum of **17** ($CDCl_3$, 400 MHz).

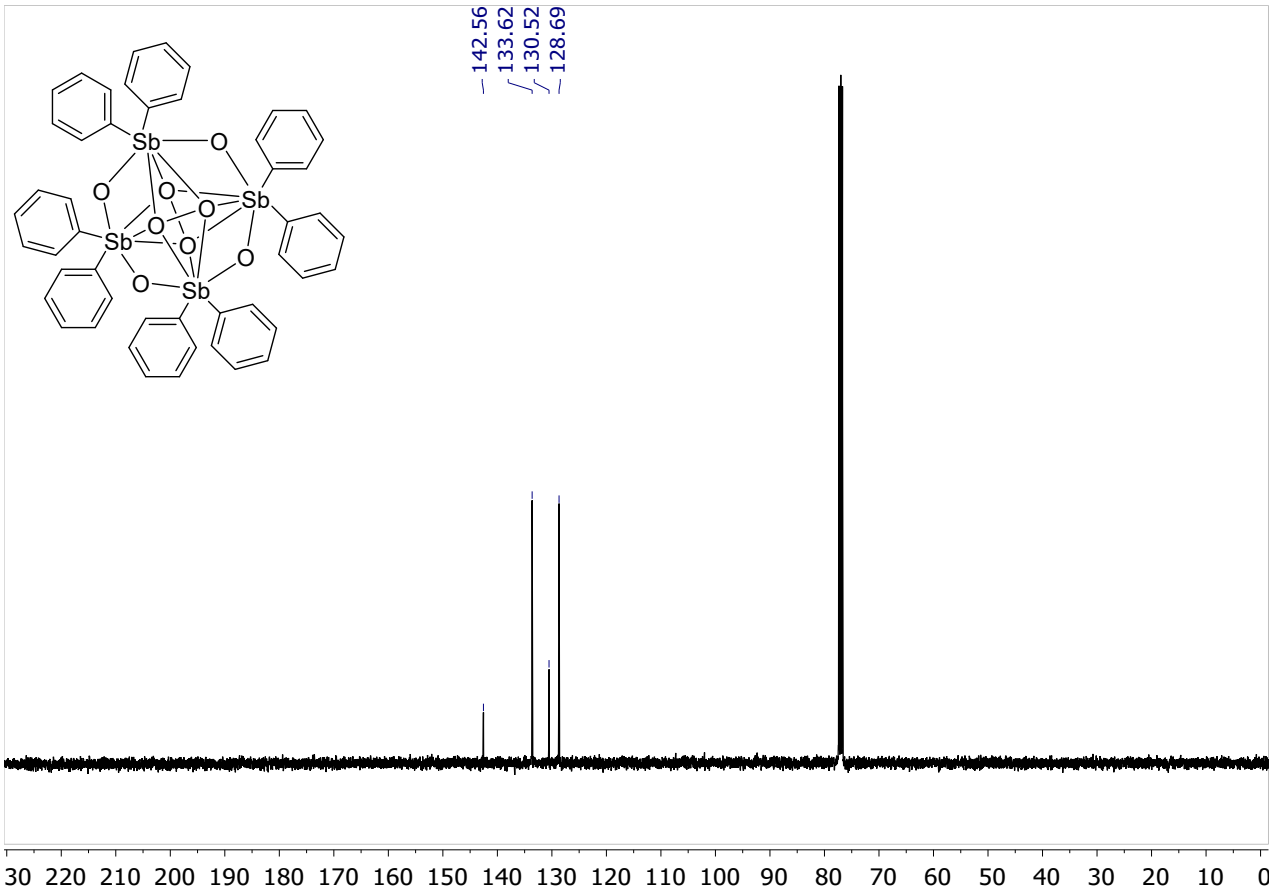

Figure S18: $^{13}C\{^{1}H\}$ NMR spectrum of **17** ($CDCl_3$, 100 MHz).

Table S1. Crystal data and structure refinement for (**8**·H_2O)$_2$, **7-A**, **7-B**, **12**, **6**

Identification code	(**8**·H_2O)$_2$	**7-A**	**7-B**	**12**	**6**
CCDC code	2114680	2114672	2114679	2114681	2114673
Empirical formula	$C_{18}H_{17}AsO_2$	$C_{18}H_{15}Cl_2Sb$	$C_{18}H_{15}Cl_2Sb$	$C_{36}H_{32}O_6SSb_2$	$C_{18}H_{15}BiCl_2$
Formula weight	340.23	423.95	423.95	836.17	511.18
Temperature, K	100(1)	100.0(3)	100.00(10)	99.9(5)	100.00(2)
Crystal system	monoclinic	monoclinic	orthorhombic	monoclinic	orthorhombic
Space group	$P2_1/n$	$P2_1/c$	Pbca	$P2_1/c$	$P2_12_12_1$
a, Å	9.4650(2)	11.12210(10)	13.01340(10)	12.0623(2)	9.1006(3)
b, Å	16.2211(3)	9.41070(10)	23.6265(2)	14.7844(2)	17.0456(4)
c, Å	11.0562(3)	15.8718(2)	43.3575(4)	18.0122(2)	22.1478(5)
α, °	90	90	90	90	90
β, °	113.710(3)	95.1810(10)	90	101.6130(10)	90
γ, °	90	90	90	90	90
Volume, Å^3	1554.21(7)	1654.46(3)	13330.7(2)	3146.43(8)	3435.68(16)
Z	4	4	32	4	8
ρ_{calc}, g/cm^3	1.454	1.702	1.690	1.765	1.977
μ, mm^{-1}	2.188	16.104	15.989	14.626	10.565
F(000)	696.0	832.0	6656.0	1656.0	1920.0
Crystal size, mm^3	0.38 × 0.26 × 0.2	0.08 × 0.06 × 0.04	0.14 × 0.12 × 0.08	0.07 × 0.04 × 0.02	0.18 × 0.15 × 0.13
Radiation	Mo Kα (λ = 0.71073)	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Mo Kα (λ = 0.71073)
2Θ range for data collection, °	5.33 to 58.688	7.982 to 134.984	7.756 to 131.998	7.482 to 134.978	5.122 to 51.998
Index ranges	-12 ≤ h ≤ 12, -22 ≤ k ≤ 20, -9 ≤ l ≤ 15	-13 ≤ h ≤ 11, -11 ≤ k ≤ 11, -19 ≤ l ≤ 18	-15 ≤ h ≤ 10, -27 ≤ k ≤ 27, -51 ≤ l ≤ 51	-14 ≤ h ≤ 14, -16 ≤ k ≤ 17, -21 ≤ l ≤ 21	-11 ≤ h ≤ 11, -17 ≤ k ≤ 21, -27 ≤ l ≤ 27
Reflections collected	7334	19558	55699	21886	18050
Independent reflections	3598 [R_{int} = 0.0374, R_{sigma} = 0.0570]	2985 [R_{int} = 0.0452, R_{sigma} = 0.0294]	11598 [R_{int} = 0.0366, R_{sigma} = 0.0248]	5645 [R_{int} = 0.0326, R_{sigma} = 0.0292]	6672 [R_{int} = 0.0330, R_{sigma} = 0.0385]
Data/restraints/ parameters	3598/0/193	2985/0/190	11598/0/757	5645/0/409	6672/0/379
Goodness-of-fit on F^2	1.047	1.037	1.071	1.036	0.991
Final R indexes [I≥2σ (I)]	R_1 = 0.0369, wR_2 = 0.0864	R_1 = 0.0397, wR_2 = 0.0974	R_1 = 0.0240, wR_2 = 0.0600	R_1 = 0.0238, wR_2 = 0.0609	R_1 = 0.0203, wR_2 = 0.0361
Final R indexes [all data]	R_1 = 0.0474, wR_2 = 0.0935	R_1 = 0.0421, wR_2 = 0.0995	R_1 = 0.0267, wR_2 = 0.0618	R_1 = 0.0264, wR_2 = 0.0622	R_1 = 0.0245, wR_2 = 0.0370
Largest diff. peak/hole, e·Å^{-3}	0.95/-0.56	2.63/-1.79	0.91/-0.69	0.47/-1.06	0.83/-0.62
Flack parameter	-	-	-	-	-0.031(4)

Table S2. Crystal data and structure refinement for $\mathbf{8}_2\cdot(4\text{-}MeC_6H_5SO_2NH_2)_3$, **15**, **14-A**, **14-B**, **17**

Identification code	$\mathbf{8}_2\cdot(4\text{-}MeC_6H_5SO_2NH_2)_3$	**15**	**14-A**	**14-B**	**17**
CCDC code	2114676	2114677	2114675	2114674	2114678
Empirical formula	$C_{57}H_{57}As_2N_3O_8S_3$	$C_{12}H_{11}O_2P$	$C_{12}H_{11}AsO_2$	$C_{12}H_{11}AsO_2$	$C_{50}H_{42}Cl_6O_8Sb_4$
Formula weight	1158.07	218.18	262.13	262.13	1470.53
Temperature, K	100.1(3)	100.0(3)	100.00(10)	100(2)	100.0(2)
Crystal system	trigonal	monoclinic	monoclinic	monoclinic	monoclinic
Space group	R-3	$P2_1/c$	$P2_1/c$	$P2_1/c$	C2/c
a, Å	18.7709(2)	11.4435(3)	11.4506(4)	16.0104(3)	22.9804(2)
b, Å	18.7709(2)	5.9428(2)	6.0251(2)	22.3412(6)	13.64820(10)
c, Å	26.6661(3)	15.6087(4)	15.7851(4)	6.16130(10)	35.9388(3)
α, °	90	90	90	90	90
β, °	90	100.199(2)	99.389(3)	90.009(2)	112.5390(10)
γ, °	120	90	90	90	90
Volume, $Å^3$	8136.9(2)	1044.72(5)	1074.44(6)	2203.84(8)	10410.92(16)
Z	6	4	4	8	8
ρ_{calc}, g/cm^3	1.418	1.387	1.620	1.580	1.876
μ, mm^{-1}	3.068	2.134	4.089	3.987	19.548
F(000)	3588.0	456.0	528.0	1056.0	5696.0
Crystal size, mm^3	0.16 × 0.12 × 0.1	0.1 × 0.06 × 0.04	0.1 × 0.06 × 0.03	0.16 × 0.06 × 0.05	0.22 × 0.2 × 0.08
Radiation	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)
2Θ range for data collection, °	6.368 to 134.944	7.85 to 134.95	7.826 to 154.5	5.52 to 131.95	5.324 to 136.224
Index ranges	-19 ≤ h ≤ 13, -18 ≤ k ≤ 22, -30 ≤ l ≤ 31	-13 ≤ h ≤ 13, -6 ≤ k ≤ 7, -18 ≤ l ≤ 18	-14 ≤ h ≤ 14, -7 ≤ k ≤ 7, -14 ≤ l ≤ 19	-18 ≤ h ≤ 12, -24 ≤ k ≤ 26, -7 ≤ l ≤ 7	-27 ≤ h ≤ 27, -16 ≤ k ≤ 16, -39 ≤ l ≤ 43
Reflections collected	5621	4553	11093	16170	28825
Independent reflections	3183 [R_{int} = 0.0688, R_{sigma} = 0.0555]	1876 [R_{int} = 0.0264, R_{sigma} = 0.0336]	2233 [R_{int} = 0.0398, R_{sigma} = 0.0297]	3801 [R_{int} = 0.0358, R_{sigma} = 0.0312]	9385 [R_{int} = 0.0469, R_{sigma} = 0.0386]
Data/restraints/ parameters	3183/0/227	1876/0/137	2233/0/140	3801/0/279	9385/0/607
Goodness-of-fit on F^2	1.083	1.045	1.194	1.036	1.053
Final R indexes [I≥2σ (I)]	R_1 = 0.0575, wR_2 = 0.1591	R_1 = 0.0344, wR_2 = 0.0880	R_1 = 0.0295, wR_2 = 0.0813	R_1 = 0.0308, wR_2 = 0.0834	R_1 = 0.0348, wR_2 = 0.0902
Final R indexes [all data]	R_1 = 0.0593, wR_2 = 0.1629	R_1 = 0.0388, wR_2 = 0.0912	R_1 = 0.0311, wR_2 = 0.0821	R_1 = 0.0343, wR_2 = 0.0864	R_1 = 0.0363, wR_2 = 0.0914
Largest diff. peak/hole, $e\cdot Å^{-3}$	1.28/-0.90	0.38/-0.34	0.47/-0.74	0.56/-0.48	1.52/-1.44
Flack parameter	-	-	-	-	-