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Supplementary material: Organotin(IV) trifluoromethanesulfonate chemistry: isolation and characterization of novel 1,10-phenanthroline-based derivatives

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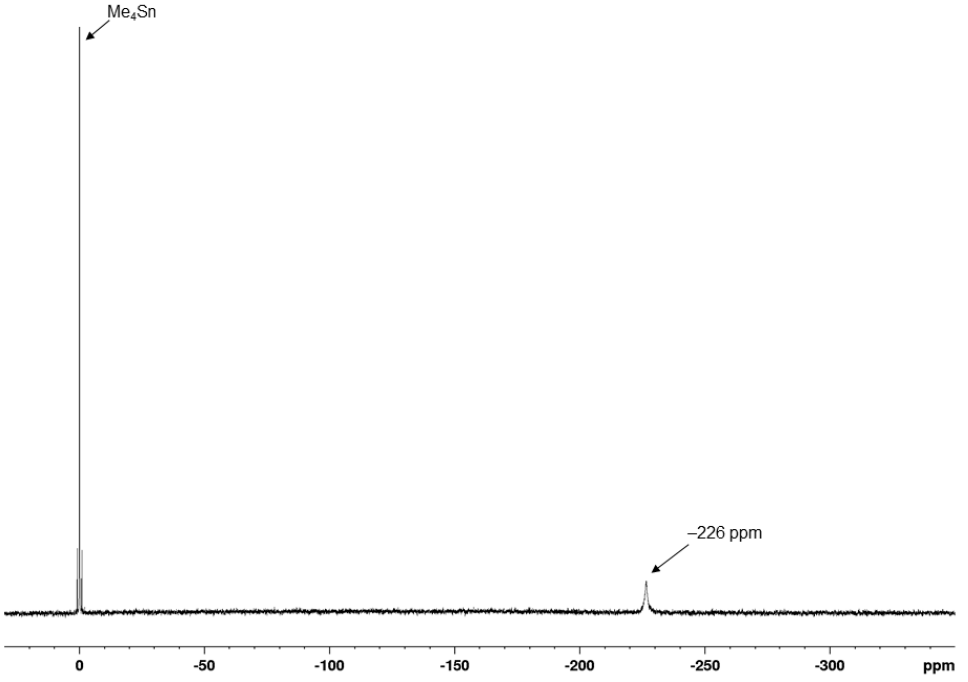
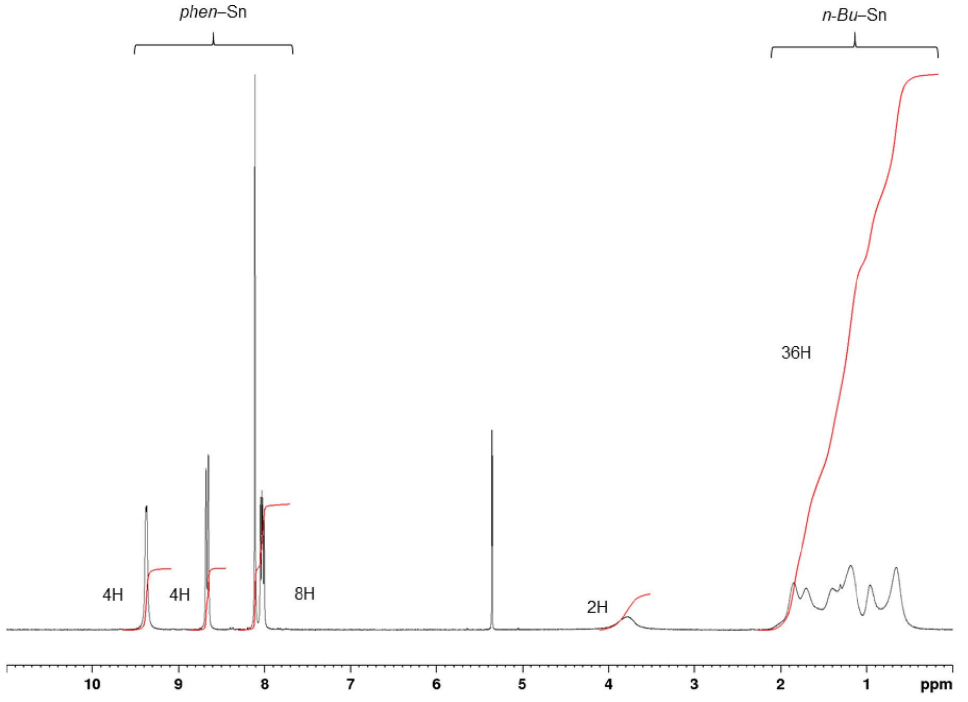
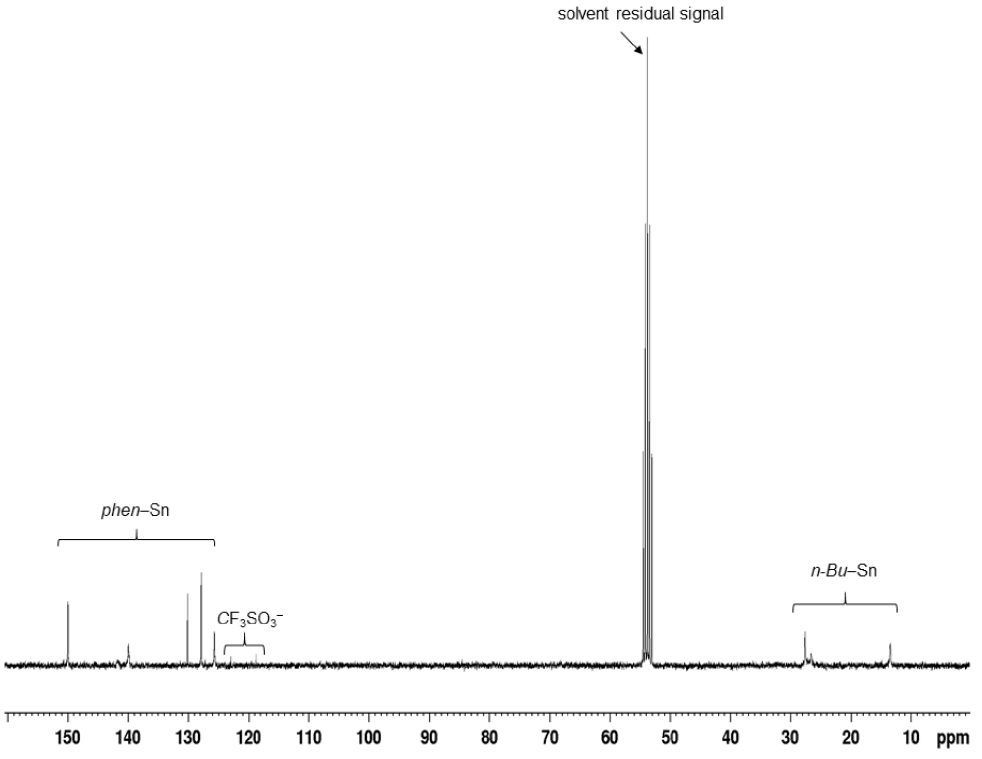
1. Spectroscopic data



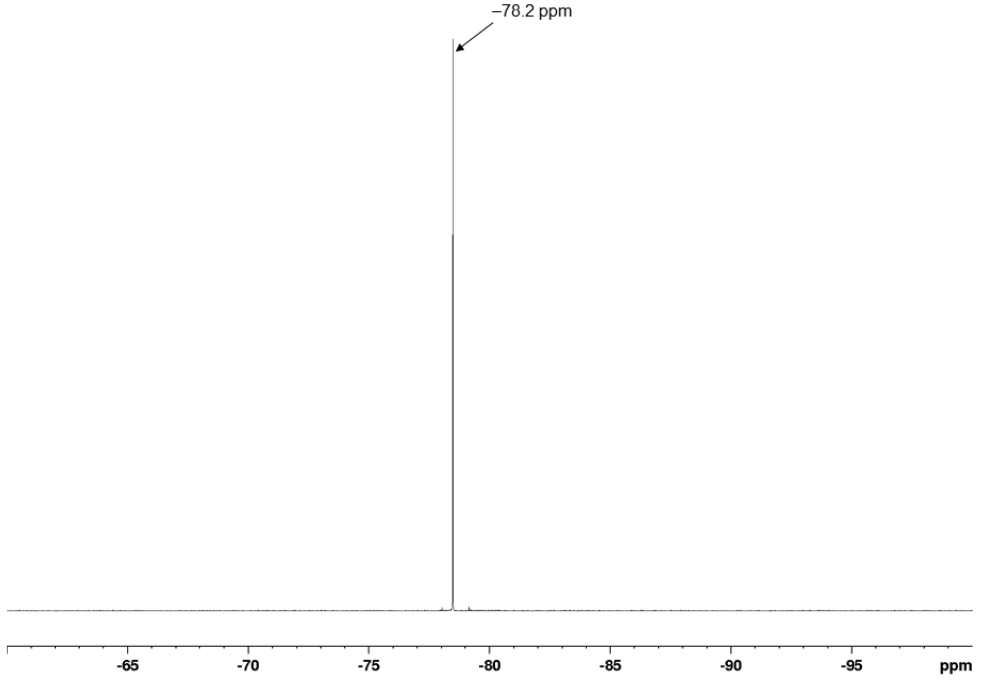
Supplementary Figure S1. $^{119}Sn\{^{1}H\}$ NMR spectrum of **5** (186 MHz, CD_3CN, 298 K).



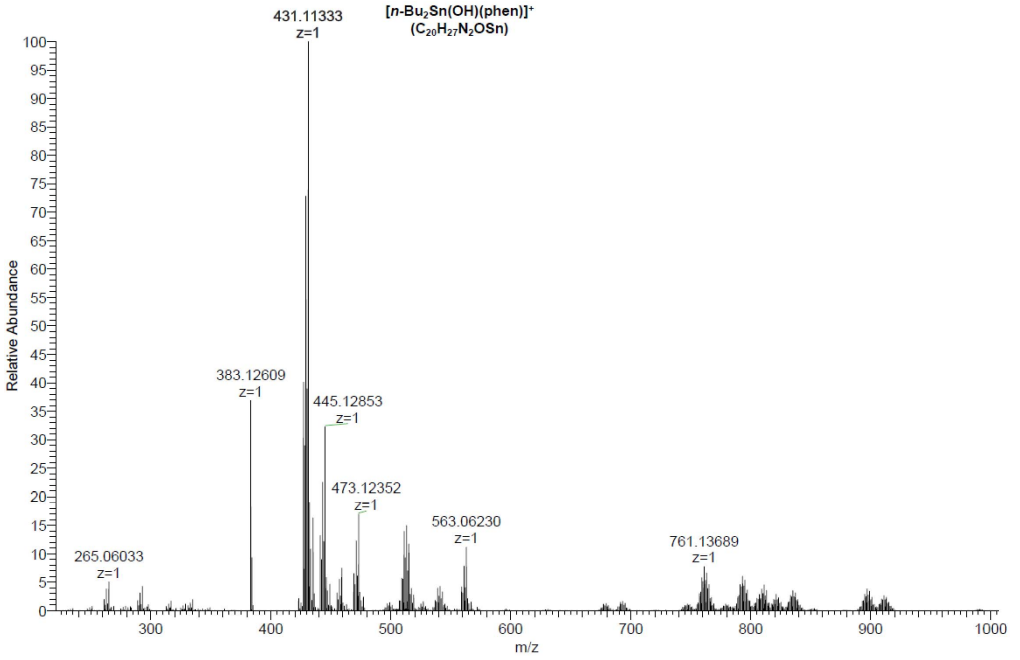
Supplementary Figure S2. ^{1}H NMR spectrum of **5** (300 MHz, CD_2Cl_2, 301.5 K).



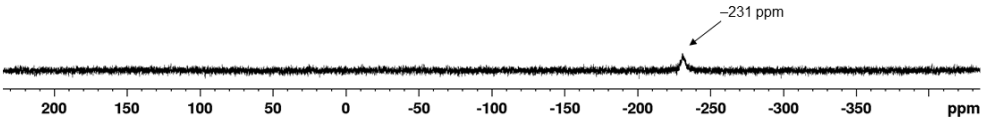
Supplementary Figure S3. $^{13}C\{^{1}H\}$ NMR spectrum of **5** (75 MHz, CD_2Cl_2, 300 K).



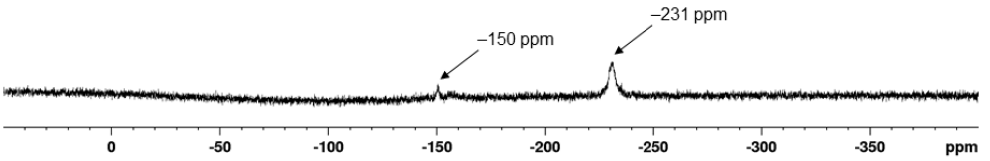
Supplementary Figure S4. ^{19}F NMR spectrum of **5** (282 MHz, CD_2Cl_2, 301 K).



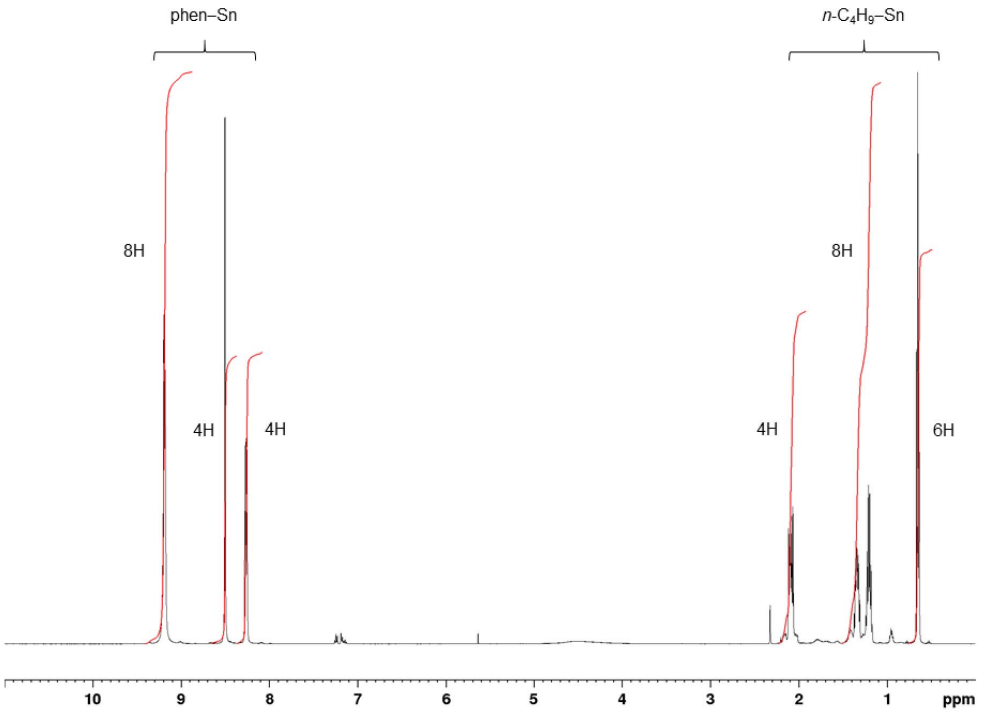
Supplementary Figure S5. ESI-HRMS spectrum of **5** from a mixture of dichloromethane/methanol.



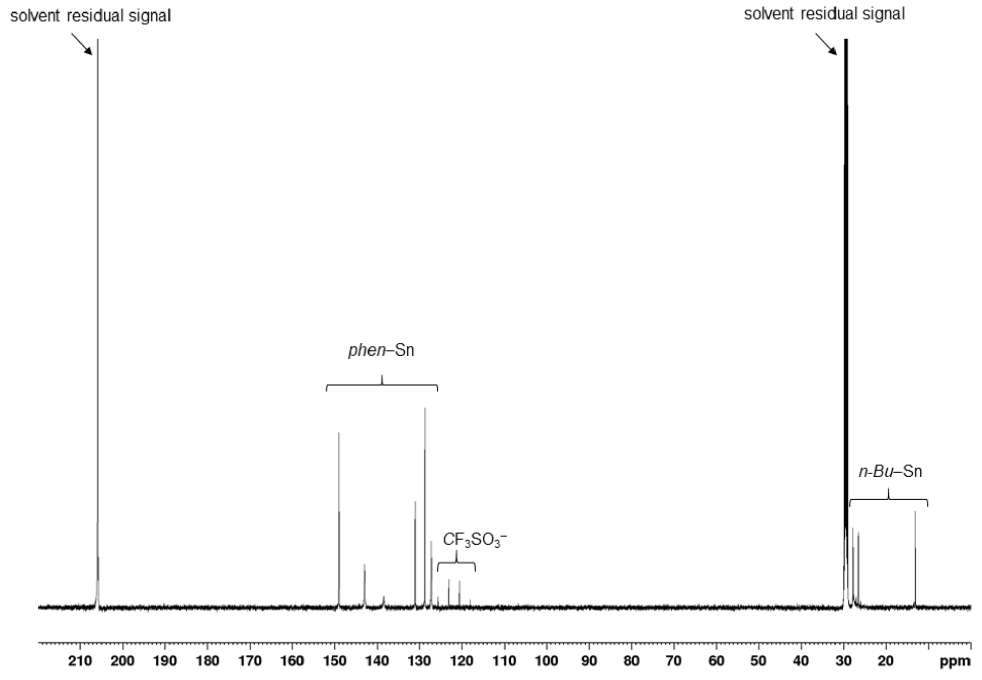
Supplementary Figure S6. ^{119}Sn{^{1}H} NMR spectrum of **6** (186 MHz, aceton-d_6, 300 K).



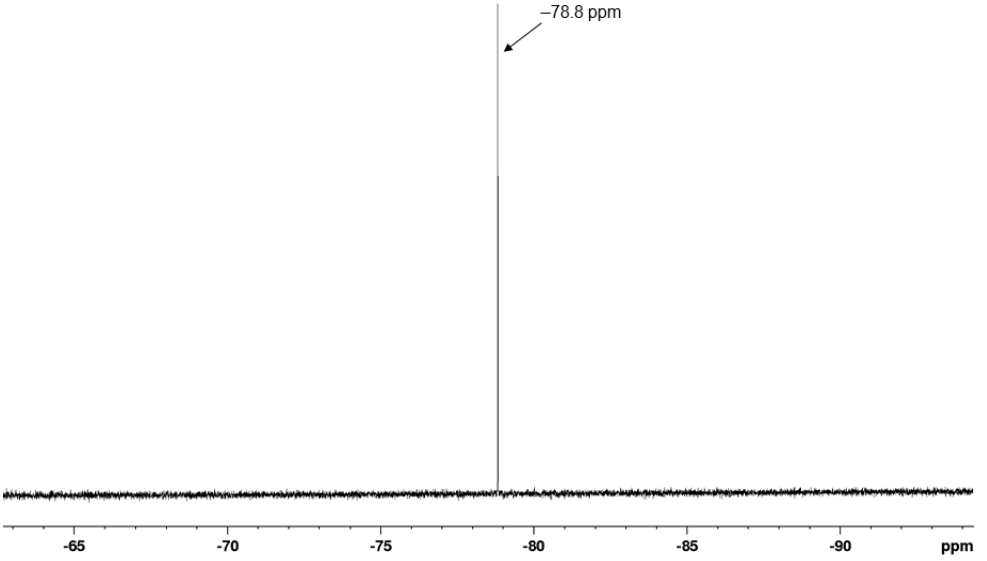
Supplementary Figure S7. Mixture of **5** and **6**. ^{119}Sn{^{1}H} NMR spectrum in aceton-d_6 (186 MHz, 300 K).



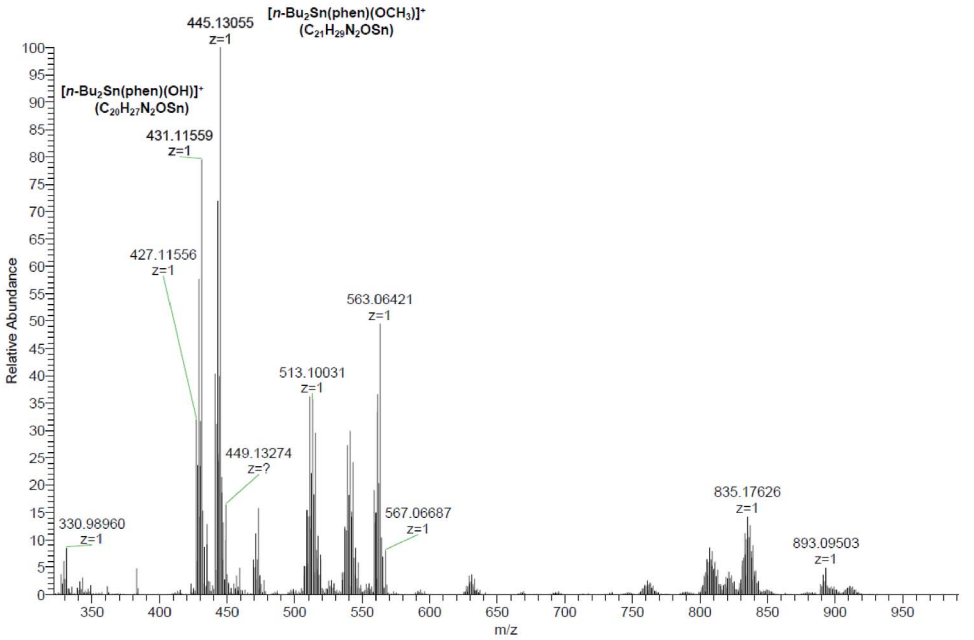
Supplementary Figure S8. ^{1}H NMR spectrum of **6** (500 MHz, aceton-d_6, 298 K).



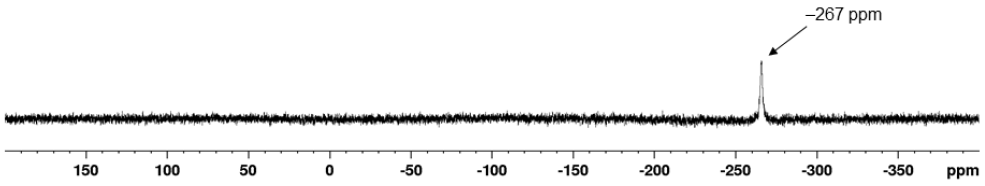
Supplementary Figure S9. ^{13}C{^{1}H} NMR spectrum of **6** (125 MHz, aceton-d_6, 298 K).



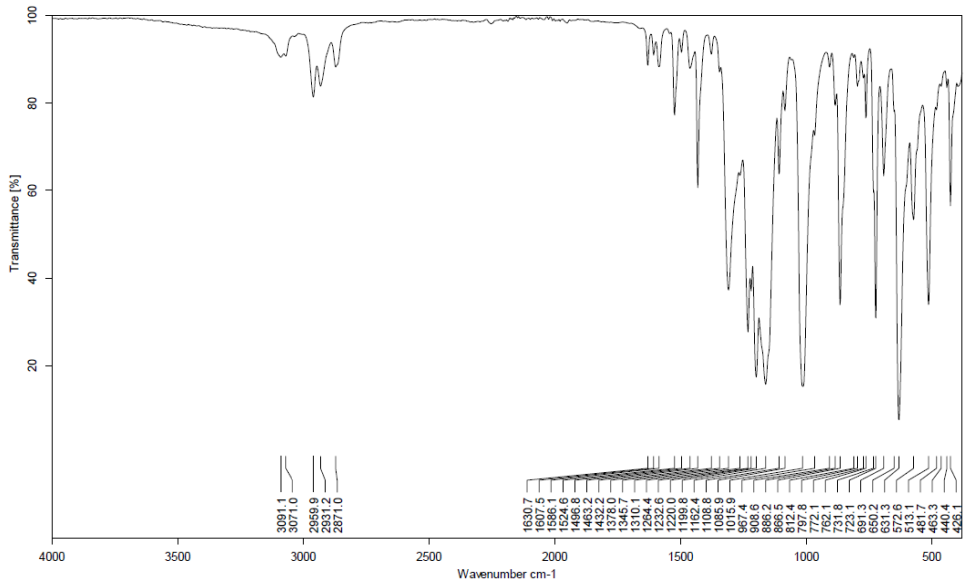
Supplementary Figure S10. ^{19}F NMR spectrum of **6** (470 MHz, aceton-d_6, 298 K).



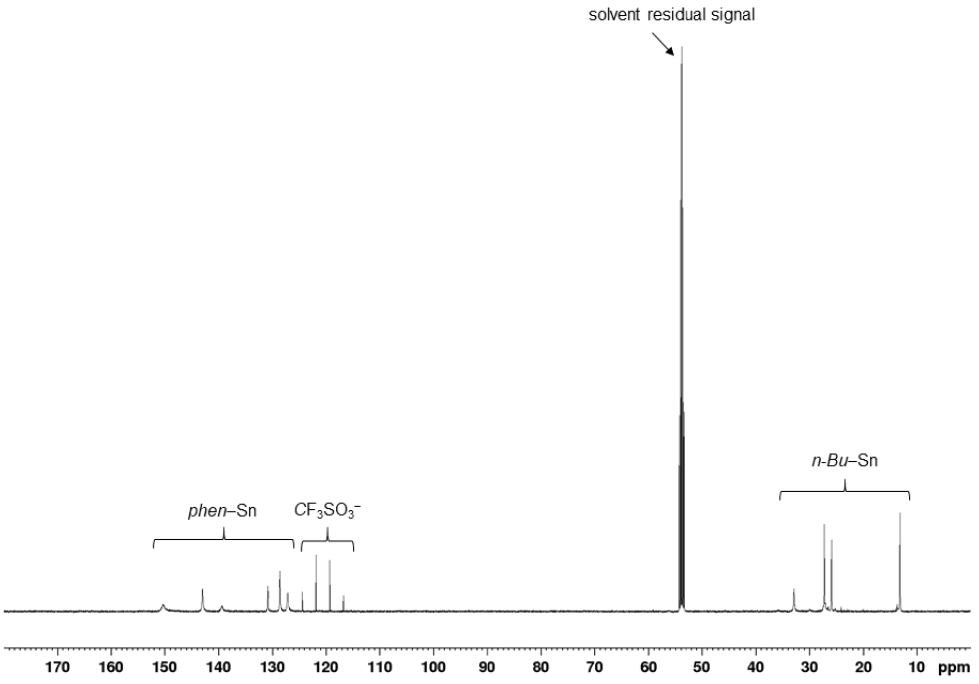
Supplementary Figure S11. ESI-HRMS spectrum of **6** in a mixture of dichloromethane/methanol.



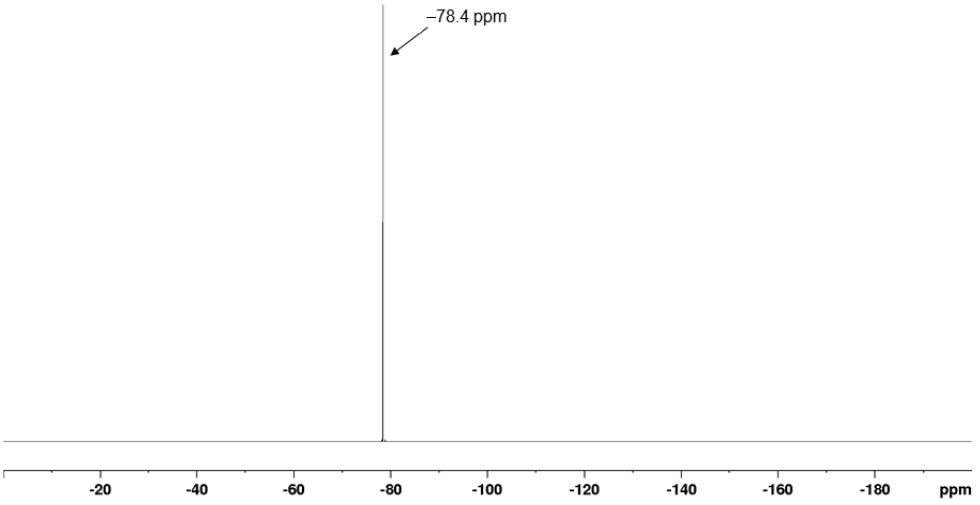
Supplementary Figure S12. $^{119}Sn\{^{1}H\}$ NMR spectrum of **3** (149 MHz, CD_3CN, 298 K).



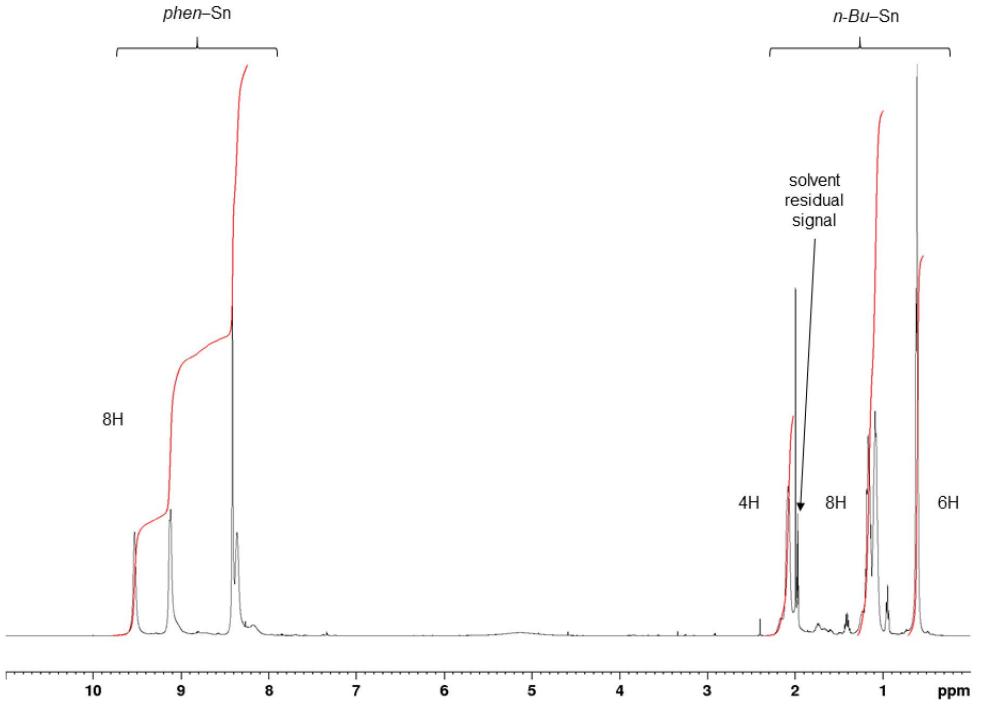
Supplementary Figure S13. FT-IR (ATR) spectrum of **3**.



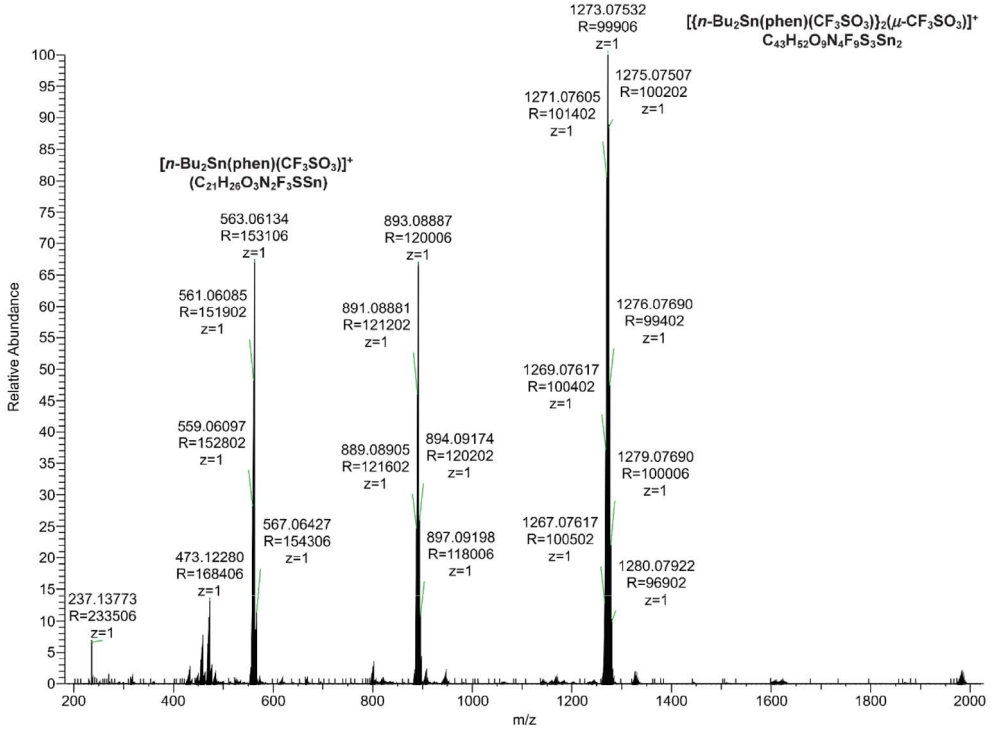
Supplementary Figure S14. $^{13}C\{^{1}H\}$ NMR spectrum of **3** (125 MHz, CD_2Cl_2, 298 K).



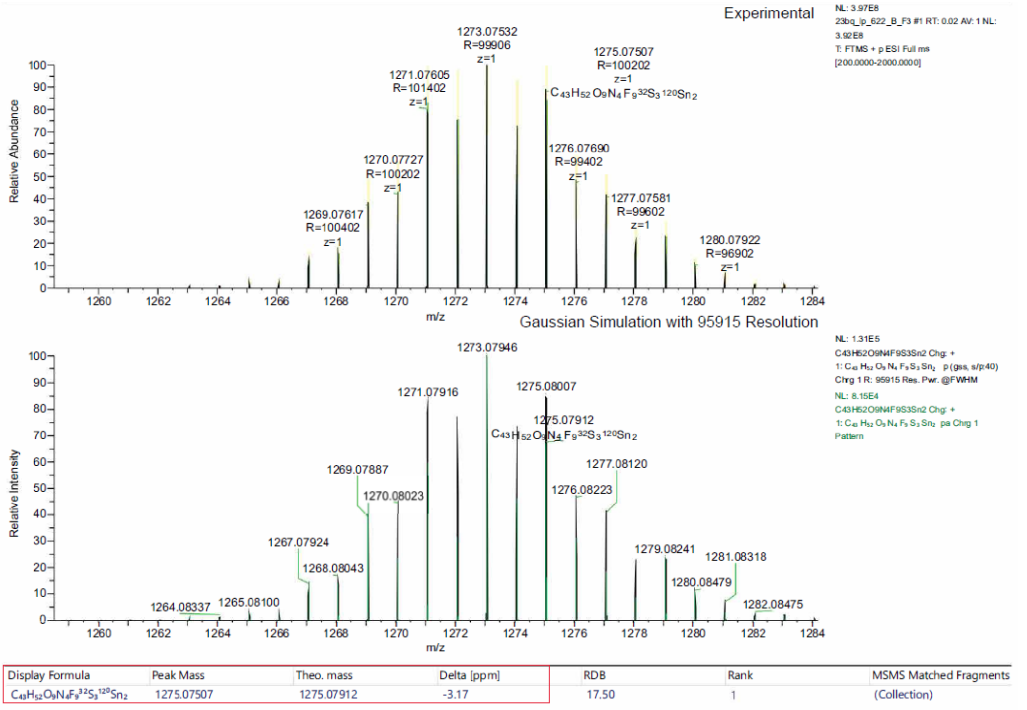
Supplementary Figure S15. ^{19}F NMR spectrum of **3** (470 MHz, CH_2Cl_2, 298 K).



Supplementary Figure S16. ^{1}H NMR spectrum of **3** (500 MHz, CD_3CN, 298 K).

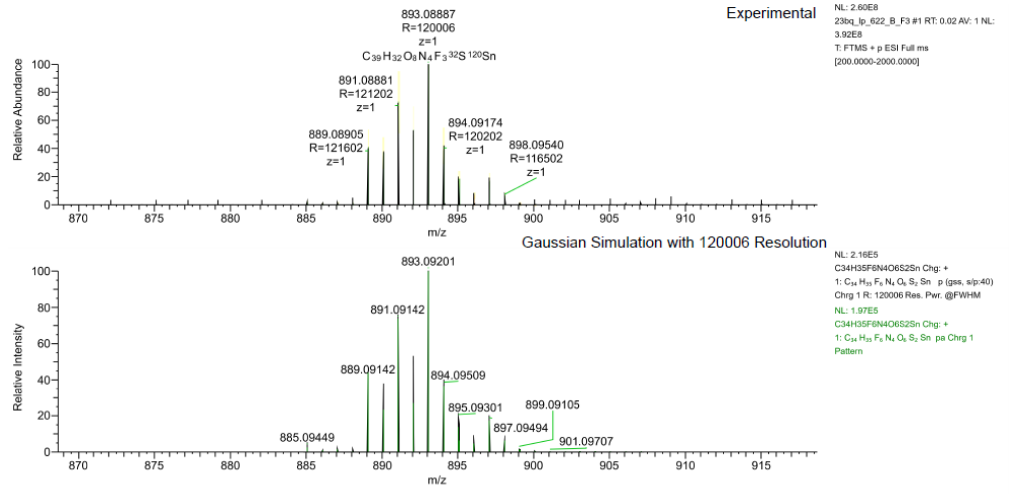


Supplementary Figure S17. ESI-HRMS spectrum of **3** in a mixture of acetonitrile/dichloromethane.



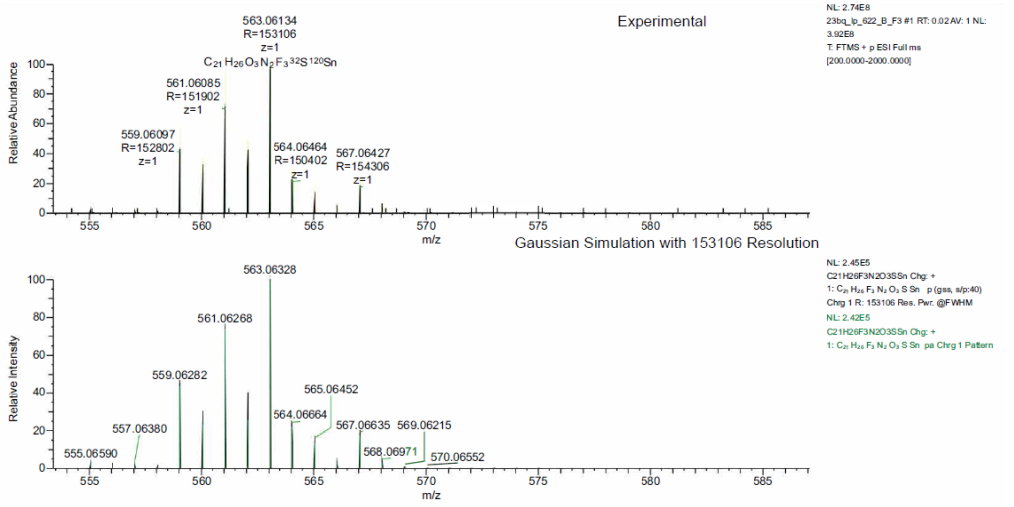
Display Formula	Peak Mass	Theo. mass	Delta [ppm]	RDB	Rank	MSMS Matched Fragments
$C_{43}H_{52}O_9N_4F_9{}^{32}S_3{}^{120}Sn_2$	1275.07507	1275.07912	-3.17	17.50	1	(Collection)

Supplementary Figure S18. Experimental (top) and simulated (bottom) ESI-HRMS isotopic distributions for the cluster at m/z = 1275.07507 Da.



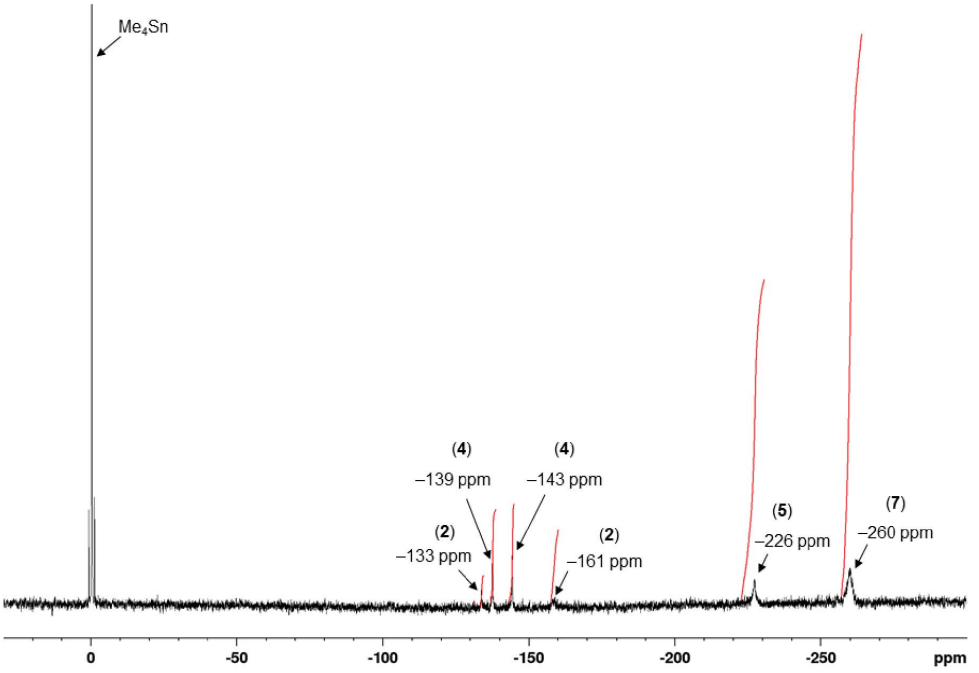
Display Formula	Peak Mass	Theo. mass	Delta [ppm]	RDB	Rank	MSMS Matched Fragments
$C_{37}H_{31}O_8N_4F_6{}^{32}S^{120}Sn$	893.08887	893.08850	0.41	22.50	3	(Collection)
$C_{34}H_{32}O_7N_4F_7{}^{32}S^{120}Sn$	893.08887	893.08964	-0.87	18.50	1	(Collection)
$C_{31}H_{33}O_6N_4F_8{}^{32}S^{120}Sn$	893.08887	893.09078	-2.15	14.50	2	(Collection)
$C_{39}H_{32}O_8N_4F_3{}^{32}S^{120}Sn$	893.08887	893.09094	-2.32	25.50	4	(Collection)
$C_{34}H_{35}O_9N_4F_6{}^{32}S_2{}^{120}Sn$	893.08887	893.09187	-3.36	17.50	6	(Collection)
$C_{28}H_{34}O_9N_4F_9{}^{32}S^{120}Sn$	893.08887	893.09193	-3.43	10.50	7	(Collection)
$C_{36}H_{33}O_9N_4F_4{}^{32}S^{120}Sn$	893.08887	893.09209	-3.60	21.50	5	(Collection)
$C_{31}H_{36}O_7N_4F_7{}^{32}S_2{}^{120}Sn$	893.08887	893.09301	-4.64	13.50	8	(Collection)
$C_{33}H_{34}O_{10}N_4F_5{}^{32}S^{120}Sn$	893.08887	893.09323	-4.88	17.50	9	(Collection)

Supplementary Figure S19. Experimental (top) and simulated (bottom) ESI-HRMS isotopic distributions for the cluster at m/z = 893.0887 Da.



Display Formula	Peak Mass	Theo. mass	Delta [ppm]	RDB	Rank	MSMS Matched Fragments
$C_{14}H_{28}O_2N_4F_5{}^{32}S_2{}^{120}Sn$	563.06134	563.05903	4.10	1.50	8	(Collection)
$C_{16}H_{26}O_5N_4F_3{}^{32}S{}^{120}Sn$	563.06134	563.05925	3.71	5.50	5	(Collection)
$C_{21}H_{26}O_3N_2F_3{}^{32}S{}^{120}Sn$	563.06134	563.06327	-3.43	9.50	6	(Collection)
$C_{19}H_{28}N_2F_5{}^{32}S_2{}^{120}Sn$	563.06134	563.06305	-3.04	5.50	4	(Collection)
$C_{22}H_{24}N_2F_5{}^{32}S{}^{120}Sn$	563.06134	563.05968	2.94	10.50	3	(Collection)
$C_{13}H_{27}O_6N_4F_4{}^{32}S{}^{120}Sn$	563.06134	563.06039	1.69	1.50	7	(Collection)
$C_{16}H_{26}O_2N_2F_7{}^{32}S{}^{120}Sn$	563.06134	563.06197	-1.12	2.50	2	(Collection)
$C_{19}H_{25}ON_2F_6{}^{32}S{}^{120}Sn$	563.06134	563.06083	0.91	6.50	1	(Collection)

Supplementary Figure S20. Experimental (top) and simulated (bottom) ESI-HRMS isotopic distributions for the cluster at $m/z = 563.0613$ Da.

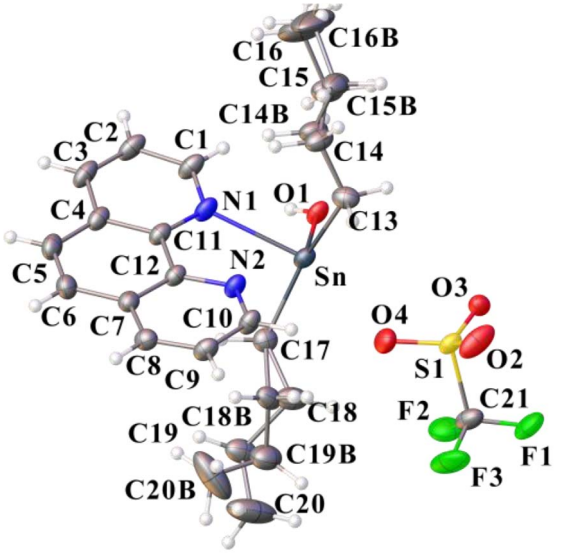


Supplementary Figure S21. $^{119}Sn\{^1H\}$ NMR spectrum of **7** (186 MHz, CD_3CN, 298 K).

2. Crystallographic data

2.1. *Complex 5:* $R_1 = 2.57\%$

2.1.1. *Crystal data and experimental*



Experimental. Single clear light colourless prism-shaped crystals of compound **5** recrystallized by slow evaporation. A suitable crystal with dimensions $0.25 \times 0.25 \times 0.25$ mm^3 was selected and mounted on a mylar loop oil on a Bruker Kappa Apex II diffractometer. The crystal was kept at a steady $T = 200$ K during data collection. The structure was solved with the ShelXS-97 [1] solution program using dual methods and by using Olex2 1.5 [2] as the graphical interface. The model was refined with ShelXL 2018/3 [3] using full matrix least squares minimization on F^2.

Crystal data. $C_{42}H_{54}F_6N_4O_8S_2Sn_2$, $M_r = 1158.39$, monoclinic, $C2/c$ (No. 15), $a = 23.4720(5)$ Å, $b = 11.6253(2)$ Å, $c = 18.7908(4)$ Å, $\beta = 109.4520(10)°$, $\alpha = \gamma = 90°$, $V = 4834.76(17)$ Å^3, $T = 200$ K, $Z = 4$, $Z' = 0.5$, μ(Mo K$_\alpha$) = 1.195, 11504 reflections measured, 5924 unique ($R_{int} = 0.0132$) which were used in all calculations. The final wR_2 was 0.0637 (all data) and R_1 was 0.0257 ($I \geq 2\sigma(I)$).

Compound	Compound **5**
CCDC	2254272
Formula	$C_{42}H_{54}F_6N_4O_8S_2Sn_2$
$D_{calc.}$/g·cm^{-3}	1.591
μ/mm^{-1}	1.195
Formula Weight	1158.39
Colour	clear light colourless
Shape	prism-shaped
Size/mm^3	$0.25 \times 0.25 \times 0.25$
T/K	200
Crystal System	monoclinic
Space Group	$C2/c$
a/Å	23.4720(5)
b/Å	11.6253(2)
c/Å	18.7908(4)
α/°	90
β/°	109.4520(10)
γ/°	90
V/Å^3	4834.76(17)
Z	4
Z'	0.5
Wavelength/Å	0.71073
Radiation type	Mo K$_\alpha$
Θ_{min}/°	3.429
Θ_{max}/°	28.285
Measured Refl's.	11504
Indep't Refl's	5924
Refl's $I \geq 2\sigma(I)$	5282
R_{int}	0.0132
Parameters	347
Restraints	0
Largest Peak	0.432
Deepest Hole	−0.605
GooF	1.074
wR_2 (all data)	0.0637
wR_2	0.0597
R_1 (all data)	0.0312
R_1	0.0257

2.1.2. *Structure quality indicators*

A clear light colorless prism-shaped-shaped crystal with dimensions 0.25 × 0.25 × 0.25 mm^3 was mounted on a mylar loop oil. Data were collected using a Bruker Kappa Apex II diffractometer operating at T = 200 K. Data were measured using ϕ and ω scans with Mo K_α radiation. The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program Apex3 [4]. The maximum resolution that was achieved was Θ = 28.285° (0.75 Å). The unit cell was refined using Saint V8.40B (Bruker, 2016) on 25194 reflections, 219% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using Saint V8.40B [5]. The final completeness is 98.10% out to 28.285° in Θ. A multi-scan absorption correction was performed [6]. The absorption coefficient μ of this material is 1.195 mm^{-1} at this wavelength (λ = 0.71073 Å) and the minimum and maximum transmissions are 0.382 and 1.822.

The structure was solved and the space group $C2/c$ (#15) determined by the ShelXS-97 [1] structure solution program using using dual methods and refined by full matrix least squares minimisation on F^2 using version 2018/3 of ShelXL 2018/3 [3]. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. The two *n*-butyl ligand linked to the Sn atom were found disordered over two positions with occupation factors respectively equal to 10.65:10.35 and 10.59:10.41.

Supplementary Table S1. Fractional atomic coordinates (×10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for compound **5**

Atom	x	y	z	U_{eq}
C17	7960.5(10)	1551(2)	1508.3(14)	46.9(5)
C18	8647(6)	1636(10)	1863(6)	65(2)
C19	8806(5)	485(9)	2445(7)	65(2)
C20	9447(4)	498(8)	2959(7)	141(5)
C18B	8564(8)	1603(15)	2096(8)	52(3)
C19B	8970(8)	615(17)	2298(12)	79(4)
C20B	8985(10)	600(12)	3061(7)	142(6)
Sn	7522.6(2)	3013.8(2)	881.9(2)	36.15(5)
N1	6687.6(8)	2782.0(16)	1351.3(11)	43.0(4)
N2	7732.5(8)	3765.3(16)	2267.7(10)	40.8(4)
O1	6958.9(6)	2209.5(13)	−86.9(8)	40.3(3)
C1	6144.4(9)	2428(2)	907.5(14)	48.8(5)
C2	5695.6(10)	2038(2)	1182.5(16)	53.7(6)
C3	5813.1(11)	1994.4(19)	1943.9(17)	52.2(6)
C4	6375.1(10)	2386.3(18)	2437.2(14)	43.5(5)
C5	6518.7(11)	2399(2)	3241.0(15)	51.6(6)
C6	7048.1(12)	2833(2)	3690.7(15)	51.0(5)
C7	7477.3(10)	3317.7(18)	3383.7(12)	40.1(4)
C8	8013.1(10)	3849(2)	3827.9(12)	43.7(5)
C9	8386.6(10)	4334(2)	3493.8(13)	45.2(5)
C10	8233.9(9)	4270(2)	2709.2(13)	44.1(5)
C11	7354.3(9)	3298.4(17)	2598.0(12)	36.9(4)
C12	6796.5(9)	2801.2(17)	2112.6(13)	38.9(4)
C13	7372.3(11)	4791(2)	605.1(14)	50.3(5)
C14	6826(2)	5403(4)	577(3)	55.0(10)
C15	6303(2)	5154(4)	−140(3)	73.7(14)
C16	5730(6)	5808(13)	−195(10)	137(5)
C14B	6624(4)	4907(8)	322(6)	57.7(19)
C15B	6445(4)	6136(7)	140(6)	73(3)
C16B	5775(13)	6330(20)	−180(30)	179(17)
C21	9813.0(11)	3847(3)	1135.9(14)	57.5(6)
F1	10110.8(7)	4098.1(17)	665.7(10)	75.3(5)
F2	9638.7(9)	2752.2(16)	1008.0(12)	83.5(5)
F3	10200.4(8)	3936(3)	1824.5(10)	107.6(8)
S1	9166.1(2)	4791.3(5)	979.3(3)	39.59(11)
O2	9434.1(9)	5898.2(18)	1165.5(15)	80.0(7)
O3	8837.1(7)	4616.1(14)	189.9(8)	43.8(3)
O4	8858.0(8)	4354.7(19)	1465.1(10)	61.6(5)

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Supplementary Table S2. Anisotropic displacement parameters ($\times 10^4$) for compound **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + \cdots + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C17	47.8(12)	39.3(11)	51.6(12)	−6.6(10)	13.7(10)	−6.7(9)
C18	53(4)	45(3)	81(7)	3(5)	2(4)	1(3)
C19	58(5)	45(3)	77(6)	6(3)	1(3)	−6(3)
C20	86(5)	96(6)	179(10)	45(6)	−38(6)	4(5)
C18B	47(5)	46(4)	54(6)	10(5)	4(4)	−11(3)
C19B	67(9)	67(8)	96(9)	20(6)	17(6)	8(6)
C20B	240(19)	99(9)	63(6)	19(6)	20(10)	−47(12)
Sn	28.08(7)	39.03(8)	40.56(8)	−10.70(6)	10.38(5)	−4.87(5)
N1	30.0(8)	48.8(10)	52.3(10)	−18.7(8)	16.4(8)	−11.6(7)
N2	32.1(8)	47.6(10)	44.5(9)	−12.9(8)	15.5(7)	−10.0(7)
O1	28.4(6)	48.0(8)	45.4(8)	−16.3(6)	13.5(6)	−12.9(6)
C1	32.0(10)	55.6(13)	60.0(14)	−24.9(11)	16.8(10)	−11.3(9)
C2	32.4(10)	53.8(13)	77.8(17)	−26.4(12)	22.1(11)	−12.5(9)
C3	39.1(11)	43.0(12)	84.7(18)	−17.6(12)	34.4(12)	−12.3(9)
C4	38.4(10)	33.3(10)	65.5(14)	−6.6(10)	26.2(10)	−5.8(8)
C5	53.5(13)	42.4(12)	68.6(15)	5.7(11)	33.5(12)	−5.1(10)
C6	53.8(13)	51.3(13)	52.0(13)	9.3(11)	22.9(11)	−1.1(11)
C7	39.4(10)	36.0(10)	46.9(11)	0.8(8)	16.9(9)	1.5(8)
C8	42.1(11)	47.4(12)	40.0(11)	−4.1(9)	11.5(9)	2.2(9)
C9	34.3(10)	51.2(12)	47.4(12)	−15.3(10)	10.3(9)	−5.7(9)
C10	34.8(10)	53.2(12)	46.8(11)	−15.2(10)	16.7(9)	−12.2(9)
C11	31.9(9)	34.2(9)	46.7(11)	−8.2(8)	15.9(8)	−4.0(7)
C12	31.9(9)	34.8(10)	53.6(12)	−10.8(9)	18.9(9)	−5.3(8)
C13	52.9(13)	43.0(12)	48.8(12)	−6.4(10)	8.6(10)	6.9(10)
C14	40(2)	45(2)	78(3)	−6(2)	16(2)	2.1(17)
C15	44(2)	64(3)	98(4)	13(3)	3(2)	−5(2)
C16	54(4)	168(14)	160(10)	2(10)	−2(5)	34(7)
C14B	35(4)	55(5)	75(6)	3(4)	8(4)	5(4)
C15B	55(4)	56(5)	112(7)	38(5)	32(5)	17(4)
C16B	80(13)	160(20)	310(40)	120(30)	79(19)	75(15)
C21	36.6(11)	82.7(19)	47.2(12)	−12.7(13)	6.0(10)	10.0(12)
F1	42.4(8)	109.1(14)	81.2(11)	−17.8(10)	29.8(8)	7.0(8)
F2	74.3(12)	66.7(11)	105.7(15)	10.1(10)	24.7(11)	26.4(9)
F3	52.0(10)	193(3)	56.5(10)	−19.6(13)	−10.3(8)	37.6(13)
S1	26.6(2)	47.3(3)	44.5(3)	−11.5(2)	11.28(19)	−4.65(19)
O2	50.8(10)	64.2(12)	131.8(19)	−54.2(13)	39.5(12)	−23.2(9)
O3	35.6(7)	51.7(9)	40.8(8)	4.3(7)	8.3(6)	−3.8(6)
O4	53.0(10)	89.7(14)	49.8(9)	6.1(9)	27.2(8)	10.1(10)

Supplementary Table S3. Bond lengths in Å for compound **5**

Atom	Atom	Length (Å)
C17	C18	1.527(13)
C17	C18B	1.480(18)
C17	Sn	2.128(2)
C18	C19	1.690(15)
C19	C20	1.492(13)
C18B	C19B	1.46(3)
C19B	C20B	1.42(2)
Sn	N1	2.4171(17)
Sn	O1	2.0820(13)
Sn	O1[1]	2.2343(14)
Sn	C13	2.131(2)
N1	C1	1.335(3)
N1	C12	1.367(3)
N2	C10	1.330(3)
N2	C11	1.354(3)
C1	C2	1.395(3)
C2	C3	1.365(4)
C3	C4	1.412(3)
C4	C5	1.434(4)
C4	C12	1.409(3)
C5	C6	1.347(4)
C6	C7	1.433(3)
C7	C8	1.400(3)
C7	C11	1.407(3)
C8	C9	1.359(3)
C9	C10	1.398(3)
C11	C12	1.444(3)
C13	C14	1.451(5)
C13	C14B	1.662(8)
C14	C15	1.518(6)
C15	C16	1.519(15)
C14B	C15B	1.496(11)
C15B	C16B	1.50(3)
C21	F1	1.329(3)
C21	F2	1.334(3)
C21	F3	1.315(3)
C21	S1	1.816(3)
S1	O2	1.4239(19)
S1	O3	1.4406(15)
S1	O4	1.4331(18)

[1] $3/2-x,1/2-y,-z$.

Supplementary Table S4. Bond Angles in ° for compound **5**

Atom	Atom	Atom	Angle (°)
C18	C17	Sn	115.8(4)
C18B	C17	Sn	123.1(7)
C17	C18	C19	101.8(7)
C20	C19	C18	111.7(8)
C19B	C18B	C17	122.8(15)
C20B	C19B	C18B	93.8(15)
C17	Sn	N1	90.58(8)
C17	Sn	O1[1]	90.38(8)
C17	Sn	C13	156.93(9)
O1	Sn	C17	100.02(7)
O1[1]	Sn	N1	156.85(5)
O1	Sn	N1	84.98(6)
O1	Sn	O1[1]	72.08(6)
O1	Sn	C13	102.51(8)
C13	Sn	N1	96.24(8)
C13	Sn	O1[1]	91.85(8)
C1	N1	Sn	121.88(15)
C1	N1	C12	117.75(19)
C12	N1	Sn	119.23(13)
C10	N2	C11	118.04(18)
Sn	O1	Sn[1]	107.92(6)
N1	C1	C2	123.4(2)
C3	C2	C1	119.1(2)
C2	C3	C4	119.7(2)
C3	C4	C5	122.4(2)
C12	C4	C3	117.6(2)
C12	C4	C5	120.0(2)
C6	C5	C4	120.6(2)
C5	C6	C7	121.4(2)
C8	C7	C6	123.1(2)
C8	C7	C11	117.53(19)
C11	C7	C6	119.4(2)
C9	C8	C7	119.8(2)
C8	C9	C10	119.1(2)
N2	C10	C9	122.9(2)
N2	C11	C7	122.53(18)
N2	C11	C12	117.76(19)
C7	C11	C12	119.68(19)

Continued on next page

Supplementary Table S4. (Continued.)

Atom	Atom	Atom	Angle (°)
N1	C12	C4	122.27(19)
N1	C12	C11	118.80(18)
C4	C12	C11	118.9(2)
C14	C13	Sn	123.8(2)
C14B	C13	Sn	103.2(3)
C13	C14	C15	112.5(4)
C14	C15	C16	113.6(7)
C15B	C14B	C13	109.7(7)
C14B	C15B	C16B	114.1(13)
F1	C21	F2	106.7(2)
F1	C21	S1	110.9(2)
F2	C21	S1	110.95(18)
F3	C21	F1	107.0(2)
F3	C21	F2	108.8(3)
F3	C21	S1	112.27(19)
O2	S1	C21	103.25(13)
O2	S1	O3	114.98(13)
O2	S1	O4	116.03(13)
O3	S1	C21	102.36(10)
O4	S1	C21	104.76(13)
O4	S1	O3	113.25(10)

[1] $3/2-x,1/2-y,-z$.

Supplementary Table S5. Torsion Angles in ° for compound **5**

Atom	Atom	Atom	Atom	Angle (°)
C17	C18	C19	C20	−170.1(9)
C17	C18B	C19B	C20B	−119.3(13)
Sn	C17	C18	C19	168.5(5)
Sn	C17	C18B	C19B	−153.5(9)
Sn	N1	C1	C2	165.05(18)
Sn	N1	C12	C4	−163.33(16)
Sn	N1	C12	C11	18.7(3)
Sn	C13	C14	C15	−77.8(4)
Sn	C13	C14B	C15B	177.3(7)
N1	C1	C2	C3	−0.9(4)
N2	C11	C12	N1	2.6(3)
N2	C11	C12	C4	−175.38(19)
C1	N1	C12	C4	4.7(3)
C1	N1	C12	C11	−173.2(2)
C1	C2	C3	C4	2.5(4)
C2	C3	C4	C5	177.9(2)
C2	C3	C4	C12	−0.5(3)
C3	C4	C5	C6	−176.6(2)
C3	C4	C12	N1	−3.2(3)
C3	C4	C12	C11	174.78(19)
C4	C5	C6	C7	1.4(4)
C5	C4	C12	N1	178.3(2)
C5	C4	C12	C11	−3.7(3)
C5	C6	C7	C8	175.4(2)
C5	C6	C7	C11	−2.6(3)
C6	C7	C8	C9	−177.2(2)
C6	C7	C11	N2	178.4(2)
C6	C7	C11	C12	0.6(3)
C7	C8	C9	C10	−1.4(3)
C7	C11	C12	N1	−179.46(19)
C7	C11	C12	C4	2.5(3)
C8	C7	C11	N2	0.2(3)
C8	C7	C11	C12	−177.56(19)
C8	C9	C10	N2	0.8(4)
C10	N2	C11	C7	−0.8(3)
C10	N2	C11	C12	177.0(2)
C11	N2	C10	C9	0.3(3)
C11	C7	C8	C9	0.9(3)
C12	N1	C1	C2	−2.6(4)
C12	C4	C5	C6	1.8(4)
C13	C14	C15	C16	−178.5(8)
C13	C14B	C15B	C16B	176(2)
F1	C21	S1	O2	−63.6(2)
F1	C21	S1	O3	56.1(2)
F1	C21	S1	O4	174.46(18)
F2	C21	S1	O2	178.0(2)
F2	C21	S1	O3	−62.3(2)
F2	C21	S1	O4	56.1(2)
F3	C21	S1	O2	56.0(3)
F3	C21	S1	O3	175.7(2)
F3	C21	S1	O4	−65.9(3)

Supplementary Table S6. Hydrogen fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for compound **5**

Atom	x	y	z	U_{eq}
H17A	7788.98	1409.37	1915.58	56
H17B	7863.62	873.83	1169.52	56
H18A	8769.12	2362.96	2146.81	78
H18B	8846.87	1577.98	1476.28	78
H19A	8534.1	483.59	2750.59	79
H19B	8730.64	−228.65	2140.65	79
H20A	9713.05	672.13	2667.93	211
H20B	9551.38	−257.47	3199.26	211
H20C	9496.07	1086.53	3348.56	211
H18C	8788.93	2226.04	1946.21	62
H18D	8500.5	1861.22	2565.9	62
H19C	8791.62	−90.64	2017.24	95
H19D	9370.37	770.26	2249.4	95
H20D	9051.65	1382.07	3267.29	213
H20E	9313.06	98.87	3359.31	213
H20F	8598.67	309.09	3082.52	213
H1	6734.15	1631.14	−90.67	48
H1A	6059.1	2443.27	376.56	59
H2	5313.21	1805.07	844.38	64
H3	5518.14	1702.04	2140.86	63
H5	6237.89	2097.99	3458.22	62
H6	7138.93	2816.87	4222.51	61
H8	8115.28	3870.64	4361.27	52
H9	8746.69	4710.77	3789.38	54
H10	8500.47	4602.84	2481.39	53
H13A	7712.27	5212.46	968.3	60
H13B	7417.16	4888.09	103.09	60
H14A	6909.1	6240.04	609.97	66
H14B	6710.2	5181.04	1018.28	66
H15A	6216.05	4319.03	−165.7	88
H15B	6425.69	5354.89	−580.73	88
H16A	5602.32	5607.29	235.79	205
H16B	5410.34	5601.05	−665.15	205
H16C	5807.73	6636.39	−190.07	205
H14C	6441.58	4423.32	−130.49	69
H14D	6473.46	4632.47	725.66	69
H15C	6631.79	6419.03	−227.53	88
H15D	6609.52	6599.56	605.93	88
H16D	5574.19	5868.98	107.34	269
H16E	5627.38	6087.51	−709.06	269
H16F	5686.18	7142.25	−141.71	269

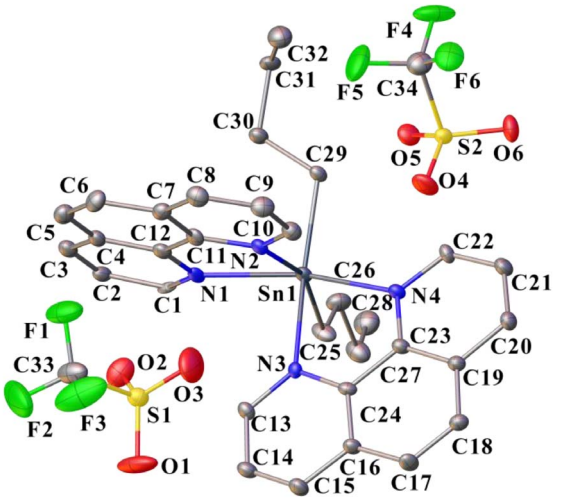
U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Supplementary Table S7. Atomic Occupancies for all atoms that are not fully occupied in compound **5**

Atom	Occupancy
C18	0.59
H18A	0.59
H18B	0.59
C19	0.59
H19A	0.59
H19B	0.59
C20	0.59
H20A	0.59
H20B	0.59
H20C	0.59
C18B	0.41
H18C	0.41
H18D	0.41
C19B	0.41
H19C	0.41
H19D	0.41
C20B	0.41
H20D	0.41
H20E	0.41
H20F	0.41
C14	0.65
H14A	0.65
H14B	0.65
C15	0.65
H15A	0.65
H15B	0.65
C16	0.65
H16A	0.65
H16B	0.65
H16C	0.65
C14B	0.35
H14C	0.35
H14D	0.35
C15B	0.35
H15C	0.35
H15D	0.35
C16B	0.35
H16D	0.35
H16E	0.35
H16F	0.35

2.2. *Complex 6:* $R_1 = 7.79\%$

2.2.1. *Crystal data and experimental*



Experimental. Single clear light colorless prism-shaped crystals of compound **6** recrystallized from toluene by slow evaporation. A suitable crystal with dimensions $0.35 \times 0.18 \times 0.05$ mm^3 was selected and mounted on a mylar loop oil on a Bruker Kappa Apex II diffractometer. The crystal was kept at a steady $T = 115$ K during data collection. The structure was solved with the ShelXS-97 [1] solution program using dual methods and by using Olex2 1.5 [2] as the graphical interface. The model was refined with ShelXL 2018/3 [3] using full matrix least squares minimization on F^2.

Crystal data. $C_{41}H_{42}F_6N_4O_6S_2Sn$, $M_r = 983.59$, triclinic, $P-1$ (No. 2), $a = 12.780(5)$ Å, $b = 13.023(5)$ Å, $c = 13.150(5)$ Å, $\alpha = 92.118(5)°$, $\beta = 95.363(5)°$, $\gamma = 107.171(5)°$, $V = 2077.1(14)$ Å^3, $T = 115$ K, $Z = 2$, $Z' = 1$, μ(Mo K$_\alpha$) = 0.795, 7097 reflections measured, 7097 unique (R_{int} = .) which were used in all calculations. The final wR_2 was 0.1922 (all data) and R_1 was 0.0779 ($I \geq 2\sigma(I)$).

Compound	Compound **6**
CCDC	2254273
Formula	$C_{41}H_{42}F_6N_4O_6S_2Sn$
$D_{calc.}$/g·cm^{-3}	1.573
μ/mm^{-1}	0.795
Formula Weight	983.59
Colour	clear light colourless
Shape	prism-shaped
Size/mm^3	$0.35 \times 0.18 \times 0.05$
T/K	115
Crystal System	triclinic
Space Group	$P-1$
a/Å	12.780(5)
b/Å	13.023(5)
c/Å	13.150(5)
α/°	92.118(5)
β/°	95.363(5)
γ/°	107.171(5)
V/Å^3	2077.1(14)
Z	2
Z'	1
Wavelength/Å	0.71073
Radiation type	Mo K$_\alpha$
Θ_{min}/°	1.559
Θ_{max}/°	24.999
Measured Refl's.	7097
Indep't Refl's	7097
Refl's $I \geq 2\sigma(I)$	6047
R_{int}	.
Parameters	545
Restraints	505
Largest Peak	1.363
Deepest Hole	−0.838
GooF	1.097
wR_2 (all data)	0.1922
wR_2	0.1736
R_1 (all data)	0.0999
R_1	0.0779

2.2.2. *Structure quality indicators*

A clear light colorless prism-shaped-shaped crystal with dimensions $0.35 \times 0.18 \times 0.05$ mm^3 was mounted on a mylar loop oil. Data were collected

using a Bruker Kappa Apex II diffractometer operating at T = 115 K. Data were measured using ϕ and ω scans with Mo K_α radiation. The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program Apex3 [4]. The maximum resolution that was achieved was Θ = 24.999°(0.84 Å). The unit cell was refined using Saint V8.40B [5] on 62925 reflections, 87% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using Saint V8.40B [5]. The final completeness is 96.90% out to 24.999° in Θ. A multi-scan absorption correction was performed [6]. The absorption coefficient μ of this material is 0.795 mm^{-1} at this wavelength (λ = 0.71073Å) and the minimum and maximum transmissions are 0.798 and 1.049.

The structure was solved and the space group $P-1$ (#2) determined by the ShelXS-97 [1] structure solution program using using dual methods and refined by full matrix least squares minimisation on F^2 using version 2018/3 of ShelXL 2018/3 [3]. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. Twin/Basf refinement type was used to determine absolute configuration from anomalous scattering using the Flack method [7]. The structure display a merohedral twinning and the twin law was found by using twinning implemented in Olex2 [2]. The use of twin law (1 0.587 0.203 0 −1 0 0 0 −1) with a population of 0.832(2)/0.168(2) reduced the R_1 (for $I > 2\sigma$ (I)) from 12.64% to 7.79%.

Supplementary Table S8. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for Compound **6**

Atom	x	y	z	U_{eq}
Sn1	5154.6(6)	2173.2(5)	1907.6(5)	21.35(18)
C25	6823(8)	2097(10)	1975(8)	36(2)
C26	7309(9)	1925(10)	1013(8)	40(3)
C27	8545(9)	2009(11)	1218(9)	44(3)
C28	9007(10)	1734(11)	287(10)	51(3)
C29	4324(8)	2211(7)	406(7)	23.3(19)
C30	3560(9)	1129(8)	−51(7)	29(2)
C31	2894(9)	1217(9)	−1077(7)	34(2)
C32	1983(10)	1721(10)	−925(9)	47(3)
N1	4340(7)	410(6)	2154(5)	26.0(16)
N2	3408(6)	2013(6)	2466(5)	22.1(15)
C1	4792(7)	−346(7)	1981(6)	18.2(16)
C2	4190(8)	−1453(8)	1910(7)	27.7(19)
C3	3092(9)	−1738(8)	2071(7)	31(2)
C4	2598(8)	−950(8)	2320(7)	27.4(18)
C5	1476(9)	−1194(9)	2521(8)	35(2)
C6	1026(9)	−393(9)	2688(8)	34(2)
C7	1640(8)	709(8)	2686(7)	28.3(19)
C8	1195(9)	1575(9)	2821(8)	35(2)
C9	1876(9)	2615(9)	2762(8)	36(2)
C10	2952(8)	2805(8)	2579(7)	30(2)
C11	2750(8)	971(8)	2521(7)	25.7(18)
C12	3243(8)	130(8)	2338(7)	26.7(18)
N3	5528(6)	2470(6)	3664(5)	19.3(14)
N4	5584(6)	3978(6)	2285(5)	21.7(15)
C13	5584(8)	1724(8)	4330(7)	25.3(19)
C14	5889(8)	1996(8)	5382(7)	30(2)
C15	6135(9)	3040(8)	5749(7)	32(2)
C16	6118(8)	3860(8)	5070(7)	25.4(19)
C17	6415(9)	4975(8)	5390(7)	29(2)
C18	6412(8)	5726(8)	4707(7)	28(2)
C19	6144(8)	5410(8)	3628(7)	25.4(19)
C20	6160(8)	6166(8)	2877(8)	30(2)
C21	5944(9)	5803(8)	1858(8)	33(2)
C22	5667(8)	4697(7)	1579(7)	26(2)
C23	5823(8)	4317(8)	3302(7)	24.3(18)
C24	5809(7)	3524(8)	4040(7)	22.8(18)
S2	2991(2)	5233.6(19)	1030.0(17)	24.9(5)
O4	2876(7)	5263(7)	2103(5)	48(2)
O5	3309(6)	4342(6)	643(6)	37.7(17)
O6	3571(6)	6254(5)	674(6)	41.3(18)
C34	1589(9)	4988(9)	435(9)	47(2)
F4	1565(8)	4951(8)	−575(6)	85(3)
F5	921(6)	4078(7)	708(8)	86(3)
F6	1177(6)	5773(6)	696(6)	60(2)
S1	3099(2)	740(2)	5520.3(19)	32.5(6)
O1	3737(8)	858(11)	6491(6)	89(4)
O2	3331(7)	79(7)	4748(7)	54(2)
O3	2926(10)	1700(7)	5141(10)	86(3)
C33	1727(9)	−36(11)	5794(8)	57(3)
F1	1001(6)	−241(8)	4995(6)	81(3)
F2	1744(9)	−971(9)	6154(10)	127(4)
F3	1396(7)	461(11)	6528(8)	124(4)
C35	568(11)	6409(11)	4639(10)	52(3)
C36	−260(11)	6913(11)	4619(10)	54(3)
C37	−963(11)	6871(11)	3698(11)	57(3)
C38	−867(11)	6305(11)	2860(10)	55(3)
C39	−59(11)	5796(11)	2856(10)	55(3)
C40	668(11)	5877(11)	3716(10)	52(3)
C41	1330(12)	6470(14)	5582(10)	72(4)

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Supplementary Table S9. Anisotropic displacement parameters ($\times 10^4$) for compound **6**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}\times U_{11}+\cdots+2hka^*\times b^*\times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sn1	27.9(3)	23.8(3)	13.3(3)	1.8(2)	3.9(2)	8.8(3)
C25	24(5)	56(7)	36(5)	17(5)	6(4)	22(5)
C26	43(6)	46(7)	28(5)	−6(5)	−4(4)	14(5)
C27	37(6)	57(8)	45(6)	17(6)	9(5)	19(6)
C28	39(7)	58(8)	53(7)	4(6)	7(5)	11(6)
C29	34(5)	22(4)	17(4)	2(3)	3(3)	12(4)
C30	38(5)	30(5)	19(4)	2(4)	0(4)	11(4)
C31	44(6)	34(6)	20(5)	−3(4)	−9(4)	9(5)
C32	43(7)	50(7)	46(7)	5(6)	0(5)	12(5)
N1	46(4)	20(4)	11(3)	−1(3)	−1(3)	9(3)
N2	29(4)	25(3)	14(3)	5(3)	4(3)	9(3)
C1	7(4)	35(4)	10(4)	−4(3)	2(3)	2(3)
C2	38(5)	24(4)	22(5)	−2(4)	−6(4)	13(4)
C3	44(5)	29(5)	20(5)	3(4)	0(4)	10(4)
C4	38(4)	25(4)	18(4)	4(4)	0(4)	7(3)
C5	41(5)	34(5)	26(5)	−2(4)	2(4)	6(4)
C6	27(5)	37(4)	34(6)	−1(4)	10(4)	1(4)
C7	31(4)	33(4)	19(4)	1(4)	4(4)	5(3)
C8	37(5)	43(5)	28(5)	3(4)	9(4)	15(4)
C9	36(5)	42(5)	35(6)	−2(5)	3(4)	20(4)
C10	35(5)	32(5)	25(5)	3(4)	5(4)	13(4)
C11	34(4)	29(4)	14(4)	5(4)	8(4)	8(3)
C12	35(4)	31(4)	14(4)	4(4)	1(4)	10(3)
N3	20(4)	18(3)	19(3)	3(2)	5(3)	3(3)
N4	28(4)	24(4)	15(3)	2(3)	2(3)	9(3)
C13	32(5)	26(5)	20(4)	6(3)	7(4)	10(4)
C14	38(6)	34(4)	21(4)	6(4)	0(4)	14(4)
C15	43(6)	33(4)	19(4)	6(3)	2(4)	11(4)
C16	34(5)	25(4)	17(4)	0(3)	0(4)	10(4)
C17	45(6)	26(4)	16(4)	−4(3)	2(4)	13(4)
C18	30(5)	29(5)	22(4)	−2(3)	−1(4)	7(4)
C19	28(5)	25(4)	21(4)	2(3)	−2(4)	7(4)
C20	36(6)	22(5)	30(4)	5(3)	1(4)	8(4)
C21	43(6)	28(4)	28(4)	8(4)	−2(4)	12(4)
C22	32(5)	22(4)	26(4)	9(3)	7(4)	9(4)
C23	29(5)	26(4)	19(3)	2(3)	0(3)	11(4)
C24	23(5)	30(4)	18(4)	3(3)	−1(3)	11(4)
S2	29.9(13)	25.5(12)	19.6(11)	0.5(9)	1.1(9)	9.5(10)
O4	76(6)	55(5)	17(4)	7(3)	6(3)	24(5)
O5	45(4)	34(4)	40(4)	2(3)	3(3)	23(4)
O6	48(5)	25(4)	52(5)	12(3)	18(4)	7(3)
C34	42(6)	50(6)	52(5)	1(5)	0(5)	19(4)
F4	96(7)	120(8)	45(4)	−22(4)	−38(4)	58(6)
F5	42(4)	54(5)	148(9)	6(5)	2(5)	−7(4)
F6	37(4)	66(5)	87(6)	6(4)	8(4)	30(4)
S1	36.9(15)	36.6(15)	25.3(13)	0.5(11)	4.6(11)	12.9(12)
O1	46(5)	170(12)	27(4)	13(5)	−8(4)	1(6)
O2	46(5)	57(5)	61(5)	−14(4)	17(4)	15(4)
O3	105(9)	37(5)	120(9)	11(5)	−1(7)	28(5)
C33	36(6)	93(8)	34(5)	9(5)	7(4)	6(6)
F1	43(4)	134(8)	52(4)	5(5)	−5(3)	7(5)
F2	92(8)	131(8)	141(10)	96(7)	13(7)	−4(6)
F3	46(5)	230(12)	77(6)	−54(7)	23(5)	15(7)
C35	44(7)	59(8)	47(6)	−11(5)	2(5)	8(6)
C36	49(7)	63(8)	49(6)	−25(6)	10(5)	15(6)
C37	45(7)	61(8)	65(7)	5(6)	12(5)	11(6)
C38	47(7)	58(8)	51(6)	4(6)	−7(6)	7(6)
C39	64(8)	61(8)	36(6)	−2(6)	−1(5)	15(6)
C40	58(8)	51(7)	48(6)	−4(5)	−2(5)	22(6)
C41	44(8)	111(13)	46(7)	4(8)	−3(6)	2(8)

Supplementary Table S10. Bond Lengths in Å for compound **6**

Atom	Atom	Length (Å)
Sn1	C25	2.158(9)
Sn1	C29	2.162(9)
Sn1	N1	2.273(8)
Sn1	N2	2.369(8)
Sn1	N3	2.309(7)
Sn1	N4	2.275(8)
C25	C26	1.498(14)
C26	C27	1.548(15)
C27	C28	1.486(16)
C29	C30	1.522(13)
C30	C31	1.552(13)
C31	C32	1.520(15)
N1	C1	1.304(11)
N1	C12	1.388(13)
N2	C10	1.336(12)
N2	C11	1.377(12)

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Supplementary Table S10. (Continued.)

Atom	Atom	Length (Å)
C1	C2	1.414(13)
C2	C3	1.380(15)
C3	C4	1.400(14)
C4	C5	1.427(15)
C4	C12	1.402(14)
C5	C6	1.351(15)
C6	C7	1.418(14)
C7	C8	1.418(14)
C7	C11	1.398(14)
C8	C9	1.387(15)
C9	C10	1.369(14)
C11	C12	1.439(13)
N3	C13	1.344(11)
N3	C24	1.372(12)
N4	C22	1.332(11)
N4	C23	1.369(11)
C13	C14	1.404(13)
C14	C15	1.360(14)
C15	C16	1.421(13)
C16	C17	1.425(13)
C16	C24	1.391(13)
C17	C18	1.352(13)
C18	C19	1.443(13)
C19	C20	1.418(13)
C19	C23	1.399(13)
C20	C21	1.379(14)
C21	C22	1.404(14)
C23	C24	1.441(12)
S2	O4	1.433(7)
S2	O5	1.430(7)
S2	O6	1.437(7)
S2	C34	1.818(11)
C34	F4	1.325(13)
C34	F5	1.324(13)
C34	F6	1.329(12)
S1	O1	1.427(8)
S1	O2	1.415(8)
S1	O3	1.431(9)
S1	C33	1.823(11)
C33	F1	1.299(12)
C33	F2	1.328(14)
C33	F3	1.312(13)
C35	C36	1.398(18)
C35	C40	1.410(17)
C35	C41	1.488(18)
C36	C37	1.428(19)
C37	C38	1.339(19)
C38	C39	1.383(18)
C39	C40	1.375(17)

Supplementary Table S11. Bond Angles in ° for compound **6**

Atom	Atom	Atom	Angle (°)
C25	Sn1	C29	116.9(4)
C25	Sn1	N1	97.0(4)
C25	Sn1	N2	157.9(3)
C25	Sn1	N3	84.3(3)
C25	Sn1	N4	96.9(4)
C29	Sn1	N1	98.0(3)
C29	Sn1	N2	84.2(3)
C29	Sn1	N3	156.9(3)
C29	Sn1	N4	94.6(3)
N1	Sn1	N2	71.9(3)
N1	Sn1	N3	87.7(3)
N1	Sn1	N4	154.5(3)
N3	Sn1	N2	76.3(3)
N4	Sn1	N2	87.5(3)
N4	Sn1	N3	72.6(3)
C26	C25	Sn1	120.4(7)
C25	C26	C27	112.2(9)
C28	C27	C26	112.7(10)
C30	C29	Sn1	114.9(6)
C29	C30	C31	112.9(8)
C32	C31	C30	112.4(9)
C1	N1	Sn1	123.3(6)
C1	N1	C12	119.4(8)
C12	N1	Sn1	116.5(6)
C10	N2	Sn1	126.5(6)
C10	N2	C11	117.8(8)
C11	N2	Sn1	114.9(6)

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Supplementary Table S11. (Continued.)

Atom	Atom	Atom	Angle (°)
N1	C1	C2	122.6(8)
C3	C2	C1	118.4(9)
C2	C3	C4	120.6(10)
C3	C4	C5	123.3(9)
C3	C4	C12	117.4(9)
C12	C4	C5	119.4(9)
C6	C5	C4	120.3(10)
C5	C6	C7	122.2(10)
C6	C7	C8	124.2(9)
C11	C7	C6	118.7(9)
C11	C7	C8	117.1(9)
C9	C8	C7	118.3(10)
C10	C9	C8	121.1(10)
N2	C10	C9	122.5(10)
N2	C11	C7	123.3(9)
N2	C11	C12	116.7(8)
C7	C11	C12	120.0(9)
N1	C12	C4	121.6(9)
N1	C12	C11	118.9(9)
C4	C12	C11	119.5(9)
C13	N3	Sn1	126.1(6)
C13	N3	C24	117.8(8)
C24	N3	Sn1	115.9(5)
C22	N4	Sn1	123.6(6)
C22	N4	C23	120.0(8)
C23	N4	Sn1	116.3(6)
N3	C13	C14	121.9(9)
C15	C14	C13	119.7(9)
C14	C15	C16	120.4(9)
C15	C16	C17	123.6(8)
C24	C16	C15	116.3(9)
C24	C16	C17	120.1(8)
C18	C17	C16	121.2(9)
C17	C18	C19	120.2(9)
C20	C19	C18	122.5(9)
C23	C19	C18	119.4(8)
C23	C19	C20	118.0(9)
C21	C20	C19	118.9(9)
C20	C21	C22	120.2(9)
N4	C22	C21	121.1(9)
N4	C23	C19	121.6(8)
N4	C23	C24	118.5(8)
C19	C23	C24	119.7(8)
N3	C24	C16	123.9(8)
N3	C24	C23	116.8(8)
C16	C24	C23	119.3(9)
O4	S2	O6	114.2(5)
O4	S2	C34	103.4(5)
O5	S2	O4	115.3(5)
O5	S2	O6	114.5(5)
O5	S2	C34	104.0(5)
O6	S2	C34	103.4(5)
F4	C34	S2	110.5(8)
F4	C34	F6	106.5(9)
F5	C34	S2	111.7(8)
F5	C34	F4	109.3(11)
F5	C34	F6	107.1(9)
F6	C34	S2	111.6(8)
O1	S1	O3	116.4(8)
O1	S1	C33	102.8(5)
O2	S1	O1	116.6(7)
O2	S1	O3	112.5(7)
O2	S1	C33	102.9(5)
O3	S1	C33	102.9(6)
F1	C33	S1	113.0(8)
F1	C33	F2	107.6(11)
F1	C33	F3	109.7(11)
F2	C33	S1	110.6(9)
F3	C33	S1	110.6(9)
F3	C33	F2	104.9(12)
C36	C35	C40	116.9(12)
C36	C35	C41	121.3(12)
C40	C35	C41	121.8(13)
C35	C36	C37	120.4(12)
C38	C37	C36	120.0(13)
C37	C38	C39	120.8(13)
C40	C39	C38	120.1(13)
C39	C40	C35	121.5(13)

Supplementary Table S12. Torsion Angles in ° for compound **6**

Atom	Atom	Atom	Atom	Angle (°)
Sn1	C25	C26	C27	174.3(8)
Sn1	C29	C30	C31	174.2(7)
Sn1	N1	C1	C2	−165.2(6)
Sn1	N1	C12	C4	168.4(7)
Sn1	N1	C12	C11	−9.7(10)
Sn1	N2	C10	C9	170.2(7)
Sn1	N2	C11	C7	−170.2(7)
Sn1	N2	C11	C12	6.2(10)
Sn1	N3	C13	C14	175.7(7)
Sn1	N3	C24	C16	−176.4(7)
Sn1	N3	C24	C23	0.9(10)
Sn1	N4	C22	C21	−179.4(7)
Sn1	N4	C23	C19	177.3(7)
Sn1	N4	C23	C24	1.4(11)
C25	C26	C27	C28	174.4(11)
C29	C30	C31	C32	−73.2(12)
N1	C1	C2	C3	−2.6(13)
N2	C11	C12	N1	2.1(12)
N2	C11	C12	C4	−176.1(8)
C1	N1	C12	C4	−1.5(13)
C1	N1	C12	C11	−179.6(8)
C1	C2	C3	C4	−1.3(14)
C2	C3	C4	C5	−178.4(9)
C2	C3	C4	C12	3.6(13)
C3	C4	C5	C6	−175.4(10)
C3	C4	C12	N1	−2.3(13)
C3	C4	C12	C11	175.8(8)
C4	C5	C6	C7	−0.9(16)
C5	C4	C12	N1	179.6(8)
C5	C4	C12	C11	−2.3(13)
C5	C6	C7	C8	177.7(10)
C5	C6	C7	C11	−1.0(15)
C6	C7	C8	C9	−177.5(10)
C6	C7	C11	N2	177.5(9)
C6	C7	C11	C12	1.2(13)
C7	C8	C9	C10	−0.1(16)
C7	C11	C12	N1	178.6(8)
C7	C11	C12	C4	0.4(13)
C8	C7	C11	N2	−1.3(14)
C8	C7	C11	C12	−177.6(9)
C8	C9	C10	N2	−1.2(16)
C10	N2	C11	C7	0.1(13)
C10	N2	C11	C12	176.5(8)
C11	N2	C10	C9	1.1(14)
C11	C7	C8	C9	1.3(14)
C12	N1	C1	C2	4.0(12)
C12	C4	C5	C6	2.6(14)
N3	C13	C14	C15	0.2(15)
N4	C23	C24	N3	−1.5(13)
N4	C23	C24	C16	175.9(9)
C13	N3	C24	C16	−1.7(13)
C13	N3	C24	C23	175.5(8)
C13	C14	C15	C16	−2.0(16)
C14	C15	C16	C17	−177.0(10)
C14	C15	C16	C24	1.9(15)
C15	C16	C17	C18	178.2(10)
C15	C16	C24	N3	0.0(14)
C15	C16	C24	C23	−177.2(9)
C16	C17	C18	C19	−2.2(15)
C17	C16	C24	N3	178.9(9)
C17	C16	C24	C23	1.8(14)
C17	C18	C19	C20	−178.1(10)
C17	C18	C19	C23	3.8(15)
C18	C19	C20	C21	176.9(10)
C18	C19	C23	N4	−178.5(9)
C18	C19	C23	C24	−2.6(14)
C19	C20	C21	C22	2.8(16)
C19	C23	C24	N3	−177.5(8)
C19	C23	C24	C16	−0.1(14)
C20	C19	C23	N4	3.4(14)
C20	C19	C23	C24	179.2(9)
C20	C21	C22	N4	1.4(16)
C22	N4	C23	C19	0.7(14)
C22	N4	C23	C24	−175.2(8)
C23	N4	C22	C21	−3.1(14)
C23	C19	C20	C21	−5.0(15)
C24	N3	C13	C14	1.6(13)
C24	C16	C17	C18	−0.6(15)
O4	S2	C34	F4	179.3(8)
O4	S2	C34	F5	−58.8(10)
O4	S2	C34	F6	61.0(9)

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Supplementary Table S12. (Continued.)

Atom	Atom	Atom	Atom	Angle (°)
O5	S2	C34	F4	−59.9(9)
O5	S2	C34	F5	62.0(9)
O5	S2	C34	F6	−178.2(8)
O6	S2	C34	F4	60.0(9)
O6	S2	C34	F5	−178.1(8)
O6	S2	C34	F6	−58.3(9)
O1	S1	C33	F1	−179.8(11)
O1	S1	C33	F2	−59.0(11)
O1	S1	C33	F3	56.7(12)
O2	S1	C33	F1	−58.2(11)
O2	S1	C33	F2	62.5(10)
O2	S1	C33	F3	178.3(10)
O3	S1	C33	F1	58.9(12)
O3	S1	C33	F2	179.7(10)
O3	S1	C33	F3	−64.6(11)
C35	C36	C37	C38	3(2)
C36	C35	C40	C39	−4(2)
C36	C37	C38	C39	−3(2)
C37	C38	C39	C40	−1(2)
C38	C39	C40	C35	4(2)
C40	C35	C36	C37	0(2)
C41	C35	C36	C37	178.2(13)
C41	C35	C40	C39	178.0(14)

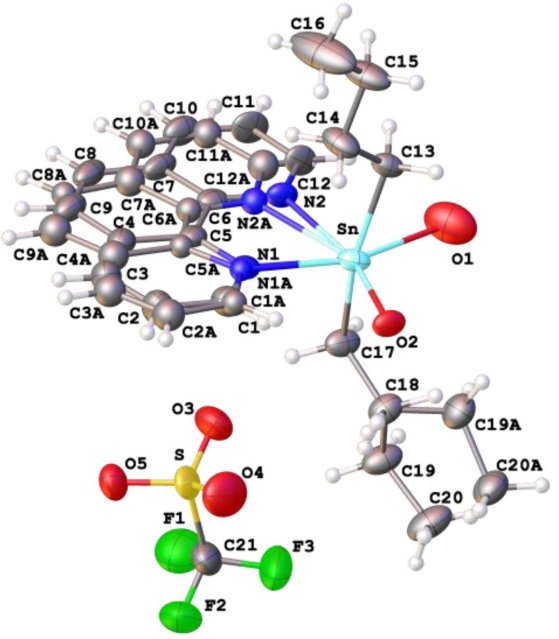
Supplementary Table S13. Hydrogen fractional atomic coordinates ($\times 10^4$) and Equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for compound **6**

Atom	x	y	z	U_{eq}
H25A	7307.17	2778.41	2331.42	43
H25B	6872.99	1511.31	2417.28	43
H26A	7222.95	2469.67	532.09	48
H26B	6899.38	1204.62	683.21	48
H27A	8963.39	2752.03	1484.58	53
H27B	8637.7	1516.34	1751.88	53
H28A	8967.07	2252.13	−226.58	76
H28B	8582.11	1006.81	7.5	76
H28C	9777.79	1762.73	463.93	76
H29A	3889.67	2724.38	448.97	28
H29B	4887.42	2490.75	−65.23	28
H30A	3038.92	811.73	446.23	35
H30B	4002.22	635.63	−167.75	35
H31A	3401.86	1657.04	−1528.61	41
H31B	2565.73	488.4	−1422.37	41
H32A	1564.77	1726.61	−1587.35	71
H32B	2308.21	2461.05	−627.37	71
H32C	1489.75	1299.62	−462.78	71
H1	5554.83	−145.99	1898.94	22
H2	4532.7	−1986.53	1755.13	33
H3	2669.03	−2476.62	2012.81	38
H5	1042.29	−1923.32	2537.81	42
H6	274.4	−574.63	2809.91	41
H8	449.07	1446.24	2949.15	42
H9	1592.23	3205.79	2850.03	43
H10	3391.37	3527.16	2531	36
H13	5411.39	992.61	4082.33	30
H14	5923.85	1453.25	5835.8	36
H15	6318.6	3221.65	6462.64	38
H17	6617.52	5195.03	6096.07	34
H18	6587.63	6462.78	4938.46	33
H20	6315.99	6910.56	3072.37	36
H21	5983.33	6303.06	1344.13	40
H22	5535.25	4457.44	874.55	31
H36	−354.79	7285.21	5221.75	65
H37	−1499.38	7245.41	3680.89	69
H38	−1360.97	6252.91	2259.96	66
H39	−5.75	5390.39	2258.14	66
H40	1251.66	5567.04	3687.74	62
H41A	2043.16	6998.24	5515.69	108
H41B	1433.23	5761.23	5675.38	108
H41C	1016.68	6688.95	6174.91	108

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

2.3. *Complex 7:* $R_1 = 8.42\%$

2.3.1. *Crystal data and experimental*



Experimental. Single colorless plate-shaped crystals of compound **7** were recrystallized from a mixture of toluene and dichloromethane by solvent layering. A suitable crystal (0.37 × 0.22 × 0.12) was selected and mounted on a mylar loop with grease on a Nonius Kappa Apex II diffractometer. The crystal was kept at $T = 115$ K during data collection. Using Olex2 [2], the structure was solved with the ShelXT [8] structure solution program, using the Direct Methods solution method. The model was refined with version of XL [9] using Least Squares minimization.

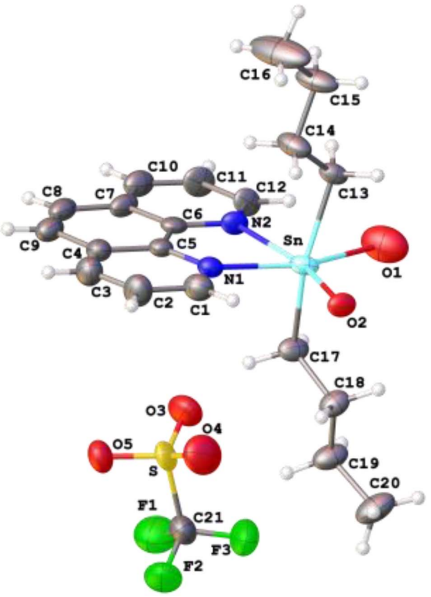
Crystal data. $C_{21}H_{26}F_3N_2O_5SSn$, $M_r = 594.19$, monoclinic, $C2/c$ (No. 15), $a = 19.9562(17)$ Å, $b = 20.5503(16)$ Å, $c = 13.9765(15)$ Å, $\beta = 97.318(9)°$, $\alpha = \gamma = 90°$, $V = 5685.2(9)$ Å^3, $T = 115$ K, $Z = 8$, $Z' = 1$, μ(Mo K$_\alpha$) = 1.021, 85172 reflections measured, 6517 unique ($R_{int} = 0.0488$) which were used in all calculations. The final wR_2 was 0.2007 (all data) and R_1 was 0.0842 ($I > 2(I)$).

Compound	Compound **7**
CCDC	2254274
Formula	$C_{21}H_{26}F_3N_2O_5SSn$
$D_{calc.}$/g·cm^{-3}	1.388
μ/mm^{-1}	1.021
Formula Weight	594.19
Colour	colourless
Shape	plate
Max Size/mm	0.37
Mid Size/mm	0.22
Min Size/mm	0.12
T/K	115
Crystal System	monoclinic
Space Group	$C2/c$
a/Å	19.9562(17)
b/Å	20.5503(16)
c/Å	13.9765(15)
α/°	90
β/°	97.318(9)
γ/°	90
V/Å^3	5685.2(9)
Z	8
Z'	1
Θ_{min}/°	2.939
Θ_{max}/°	27.575
Measured Refl.	85172
Independent Refl.	6517
Reflections Used	5091
R_{int}	0.0488
Parameters	375
Restraints	405
Largest Peak	2.379
Deepest Hole	−1.751
GooF	1.111
wR_2 (all data)	0.2007
wR_2	0.1933
R_1 (all data)	0.1043
R_1	0.0842

Structure quality indicators. A colorless plate-shaped crystal with dimensions 0.37 × 0.22 × 0.12 was mounted on a mylar loop with grease. Data were collected using a Nonius Kappa Apex II diffractometer equipped with an Oxford Cryosystems low-temperature apparatus operating at T = 115 K. Data were measured using ϕ and ω scans using Mo K_α radiation. The total number of runs and images was based on the strategy calculation from the program Apex2 [10]. The actually achieved resolution was Θ = 27.575.

Data reduction was performed using the software Sadabs [11] which corrects for Lorentz polarization. The final completeness is 99.40 out to 27.575 in Θ. The absorption coefficient (μ) of this material is 1.021 and the minimum and maximum transmissions are 0.6727 and 0.7456.

The structure was solved in the space group $C2/c$ (#15) by Direct Methods using the ShelXT [8] structure solution program and refined by Least Squares using version of XL [9]. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model.



Supplementary Figure S22. The crystal was found disordered, probably because it was recorded at 115 K, hence near a transition phase. The space group was then not easy to be found and several space groups was attempted: $P21$, $A2/n$, $C2/n$ and finally $C2/c$. The phenanthroline group was found disordered over two positions with occupation factors respectively equal to 10.69:10.31. The minor components of theses disordered group were isotropically refined with Same restraints. Two carbon atoms of the *n*-butyl group were also find disordered with occupation factors respectively equal to 10.54:10.46 and were anisotropically refined. Hydrogen atoms of the two oxygen atoms link to the tin were not located but by distance comparison (Sn1–O1 = 2.512 Å, Sn–O2 = 2.142 Å), O1 was founded to be linked to two hydrogen atoms whereas O1 to only one hydrogen atom, which compensates the $(CF_3SO_3)^-$ charge. A solvent accessible voids of 334 $Å^3$ was found but unfortunately, it was not possible to model it satisfactorily by either a toluene or a dichloromethane molecule (recrystallization solvent). The Squeeze routine [12] in Platon was used and the *hkl* intensities were modified accordingly [12].

Supplementary Table S14. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for compound **7**

Atom	x	y	z	U_{eq}
Sn	7602.2(3)	1498.1(2)	3326.8(4)	33.08(17)
O1	6993(4)	427(4)	3266(7)	80(2)
O2	7686(3)	1963(3)	4709(3)	34.1(11)
N1	8155(7)	2322(6)	2782(7)	31(2)
C1	8389(8)	2826(6)	3311(11)	43(3)
C2	8718(8)	3358(6)	2938(10)	50(3)
C3	8806(7)	3352(6)	1995(10)	47(3)
C4	8565(6)	2838(6)	1404(8)	40(2)
C5	8247(6)	2315(5)	1831(7)	30.5(19)
C9	8646(7)	2806(7)	416(9)	47(3)
C8	8388(6)	2281(6)	−129(8)	46(2)
C6	8003(5)	1769(6)	1270(6)	30.7(19)
C7	8078(6)	1750(6)	272(7)	40(2)
C10	7840(7)	1207(7)	−237(9)	46(3)
C11	7539(7)	717(6)	197(7)	44(3)
C12	7474(6)	761(6)	1188(6)	36(2)
N2	7704(5)	1274(5)	1709(6)	32.8(18)
N1A	8275(14)	2422(11)	2928(12)	20(6)
C1A	8504(15)	2867(12)	3584(15)	25(6)
C2A	8841(14)	3436(12)	3353(17)	40(7)
C3A	8947(13)	3533(11)	2423(16)	40(6)
C4A	8720(14)	3087(11)	1707(14)	36(7)
C5A	8380(12)	2528(9)	1993(12)	21(5)
C9A	8798(13)	3153(11)	725(14)	40(6)
C8A	8573(12)	2675(10)	66(13)	24(5)
C6A	8145(11)	2051(9)	1300(11)	25(5)
C7A	8260(13)	2105(10)	321(13)	36(6)
C10A	8043(12)	1608(10)	−314(14)	32(5)
C11A	7734(15)	1065(12)	−14(15)	33(7)
C12A	7623(13)	1048(11)	969(14)	37(6)
N2A	7826(11)	1517(9)	1593(12)	27(5)
C13	8426(4)	861(3)	3787(5)	30.1(14)
C14	9105(4)	1189(4)	3873(7)	45(2)
C15	9678(4)	721(4)	4212(8)	54(2)
C16	10355(5)	1039(6)	4330(14)	111(6)
C17	6624(4)	1847(4)	2795(5)	36.4(16)
C18	6180(4)	2051(4)	3550(6)	37.6(16)
C19	5488(7)	2199(9)	3126(11)	46(4)
C20	5063(8)	2369(10)	3936(13)	58(5)
C19A	5876(9)	1592(10)	4106(14)	45(4)
C20A	5325(10)	1891(11)	4689(15)	51(5)
C21	6089(6)	4198(5)	2906(8)	61(2)
F1	5790(4)	4108(4)	2025(6)	92(2)
F2	5974(4)	4803(3)	3127(6)	94(2)
F3	5789(4)	3829(4)	3509(6)	98(2)
S	6971.4(13)	4008.1(11)	2984.0(15)	49.5(6)
O3	6980(4)	3343(3)	2605(6)	73(2)
O4	7209(5)	4058(5)	3970(5)	85(2)
O5	7224(3)	4460(3)	2363(4)	51.2(15)

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Supplementary Table S15. Anisotropic displacement parameters ($\times 10^4$) compound **7**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + \cdots + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Sn	33.0(3)	31.9(3)	33.5(3)	−3.6(2)	0.80(18)	12.9(2)
O1	71(5)	61(4)	102(6)	−15(4)	−10(4)	1(3)
O2	40(3)	45(3)	17(2)	−4(2)	2.3(19)	12(2)
N1	32(6)	32(4)	29(4)	2(3)	5(3)	11(3)
C1	44(7)	39(5)	46(6)	0(5)	6(5)	5(4)
C2	65(8)	39(5)	50(6)	0(4)	17(5)	−1(5)
C3	55(7)	41(5)	49(5)	6(4)	18(5)	5(5)
C4	40(5)	39(5)	43(4)	11(4)	16(4)	14(4)
C5	29(5)	34(4)	30(4)	6(3)	7(3)	14(3)
C9	42(6)	64(6)	37(5)	12(4)	15(4)	21(5)
C8	40(5)	68(6)	33(5)	15(4)	13(4)	26(4)
C6	27(4)	38(4)	27(4)	4(3)	2(3)	18(3)
C7	39(5)	62(5)	20(4)	4(3)	8(3)	20(4)
C10	49(7)	66(6)	23(5)	3(4)	6(4)	20(5)
C11	65(7)	54(6)	13(4)	−11(4)	3(4)	12(5)
C12	55(6)	42(5)	11(4)	−4(3)	1(4)	9(4)
N2	43(5)	36(4)	20(3)	−5(3)	5(3)	11(3)
C13	35(3)	27(3)	26(3)	−4(3)	−4(2)	9(2)
C14	32(3)	31(4)	67(6)	2(4)	−8(3)	5(3)
C15	32(4)	36(4)	87(7)	−11(4)	−17(4)	10(3)
C16	34(5)	55(6)	232(19)	−16(9)	−22(6)	3(4)
C17	34(3)	50(4)	23(3)	−3(3)	−3(3)	13(3)
C18	27(3)	52(4)	34(4)	−6(3)	2(3)	−1(3)
C19	30(5)	69(10)	37(7)	−8(6)	1(4)	11(5)
C20	34(7)	88(13)	52(8)	−17(9)	11(6)	0(7)
C19A	35(7)	53(8)	47(8)	0(6)	8(6)	−7(6)
C20A	39(8)	64(12)	54(10)	5(9)	15(8)	3(8)
C21	72(5)	52(4)	64(5)	27(4)	27(4)	22(3)
F1	73(4)	106(5)	98(4)	11(4)	10(3)	19(4)
F2	118(6)	64(3)	111(5)	18(3)	60(4)	41(3)
F3	88(5)	96(5)	121(6)	57(4)	57(4)	24(4)
S	68.1(14)	49.0(12)	33.9(10)	23.6(9)	16.3(10)	18.7(10)
O3	89(5)	40(3)	96(5)	13(3)	35(4)	18(3)
O4	111(6)	108(6)	38(3)	15(3)	12(3)	32(5)
O5	69(4)	44(3)	42(3)	15(3)	14(3)	7(3)

Supplementary Table S16. Bond Lengths in Å for compound **7**

Atom	Atom	Length (Å)
Sn	O1	2.512(8)
Sn	O2	2.142(5)
Sn	N1	2.208(11)
Sn	N2	2.342(8)
Sn	N1A	2.431(17)
Sn	N2A	2.520(17)
Sn	C13	2.136(7)
Sn	C17	2.123(7)
N1	C1	1.324(14)
N1	C5	1.365(11)
C1	C2	1.409(13)
C2	C3	1.351(16)
C3	C4	1.388(16)
C4	C5	1.417(13)
C4	C9	1.412(14)
C5	C6	1.419(17)
C9	C8	1.38(2)
C8	C7	1.406(14)
C6	C7	1.424(11)
C6	N2	1.363(13)
C7	C10	1.374(16)
C10	C11	1.355(17)
C11	C12	1.409(11)
C12	N2	1.330(12)
N1A	C1A	1.333(16)
N1A	C5A	1.367(15)
C1A	C2A	1.407(16)
C2A	C3A	1.36(2)
C3A	C4A	1.390(19)
C4A	C5A	1.417(16)
C4A	C9A	1.407(17)
C5A	C6A	1.42(2)
C9A	C8A	1.38(2)
C8A	C7A	1.395(18)
C6A	C7A	1.420(15)
C6A	N2A	1.356(16)
C7A	C10A	1.386(18)
C10A	C11A	1.37(2)
C11A	C12A	1.420(17)
C12A	N2A	1.329(16)
C13	C14	1.503(11)
C14	C15	1.523(11)
C15	C16	1.491(13)
C17	C18	1.521(10)
C18	C19	1.464(16)
C18	C19A	1.408(19)
C19	C20	1.54(2)
C19A	C20A	1.57(3)
C21	F1	1.311(13)
C21	F2	1.309(12)
C21	F3	1.330(11)
C21	S	1.794(11)
S	O3	1.466(7)
S	O4	1.403(8)
S	O5	1.408(6)

Supplementary Table S17. Bond Angles in ° for compound **7**

Atom	Atom	Atom	Angle (°)
O1	Sn	N2A	97.2(4)
O2	Sn	O1	113.8(3)
O2	Sn	N1	89.1(3)
O2	Sn	N2	162.2(3)
O2	Sn	N1A	82.9(4)
O2	Sn	N2A	149.0(4)
N1	Sn	O1	157.1(3)
N1	Sn	N2	73.1(4)
N2	Sn	O1	84.1(3)
N1A	Sn	O1	163.0(5)
N1A	Sn	N2A	66.2(6)
C13	Sn	O1	79.9(3)
C13	Sn	O2	91.9(2)
C13	Sn	N1	100.1(4)
C13	Sn	N2	90.7(3)
C13	Sn	N1A	97.0(8)
C13	Sn	N2A	94.3(5)

Continued on next page

Supplementary Table S17. (Continued.)

Atom	Atom	Atom	Angle (°)
C17	Sn	O1	82.2(3)
C17	Sn	O2	97.4(2)
C17	Sn	N1	95.6(4)
C17	Sn	N2	85.2(3)
C17	Sn	N1A	99.6(8)
C17	Sn	N2A	85.7(5)
C17	Sn	C13	161.9(3)
C1	N1	Sn	124.3(8)
C1	N1	C5	118.1(10)
C5	N1	Sn	117.6(8)
N1	C1	C2	123.2(13)
C3	C2	C1	118.6(13)
C2	C3	C4	120.6(11)
C3	C4	C5	117.8(10)
C3	C4	C9	122.9(11)
C9	C4	C5	119.3(11)
N1	C5	C4	121.7(10)
N1	C5	C6	117.8(9)
C4	C5	C6	120.5(8)
C8	C9	C4	119.6(11)
C9	C8	C7	122.6(10)
C5	C6	C7	119.4(10)
N2	C6	C5	118.8(7)
N2	C6	C7	121.8(10)
C8	C7	C6	118.4(11)
C10	C7	C8	124.4(10)
C10	C7	C6	117.2(11)
C11	C10	C7	121.0(10)
C10	C11	C12	119.6(11)
N2	C12	C11	121.3(11)
C6	N2	Sn	112.6(6)
C12	N2	Sn	128.2(8)
C12	N2	C6	119.1(9)
C1A	N1A	Sn	121.9(12)
C1A	N1A	C5A	117.9(15)
C5A	N1A	Sn	120.0(11)
N1A	C1A	C2A	123.0(17)
C3A	C2A	C1A	118.6(18)
C2A	C3A	C4A	121.1(17)
C3A	C4A	C5A	117.0(14)
C3A	C4A	C9A	124.9(16)
C9A	C4A	C5A	118.1(16)
N1A	C5A	C4A	122.5(14)
N1A	C5A	C6A	117.8(12)
C6A	C5A	C4A	119.8(12)
C8A	C9A	C4A	121.0(16)
C9A	C8A	C7A	123.0(14)
C5A	C6A	C7A	121.6(13)
N2A	C6A	C5A	118.7(11)
N2A	C6A	C7A	119.7(14)
C8A	C7A	C6A	116.5(15)
C10A	C7A	C8A	124.7(15)
C10A	C7A	C6A	118.8(15)
C11A	C10A	C7A	121.5(16)
C10A	C11A	C12A	116.7(17)
N2A	C12A	C11A	123.0(18)
C6A	N2A	Sn	116.8(10)
C12A	N2A	Sn	122.9(13)
C12A	N2A	C6A	120.3(15)
C14	C13	Sn	113.6(5)
C13	C14	C15	112.0(7)
C16	C15	C14	113.1(8)
C18	C17	Sn	116.2(5)
C19	C18	C17	112.3(8)
C19A	C18	C17	121.8(10)
C18	C19	C20	109.3(12)
C18	C19A	C20A	113.8(15)
F1	C21	F3	109.1(11)
F1	C21	S	110.5(7)
F2	C21	F1	106.5(9)
F2	C21	F3	106.6(9)
F2	C21	S	113.1(9)
F3	C21	S	110.8(7)
O3	S	C21	103.7(5)
O4	S	C21	104.5(5)
O4	S	O3	113.9(5)
O4	S	O5	117.0(5)
O5	S	C21	104.2(4)
O5	S	O3	111.7(4)

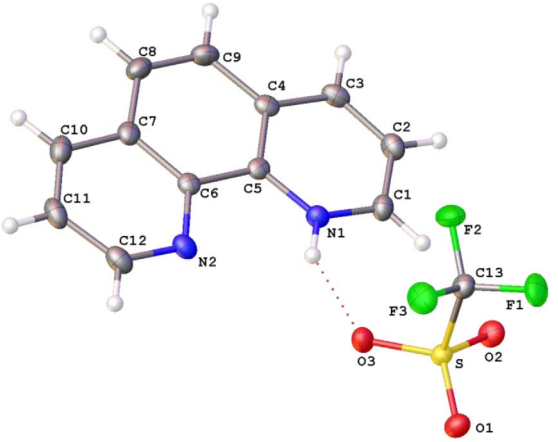
Supplementary Table S18. Hydrogen fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for compound **7**

Atom	x	y	z	U_{eq}
H1	8333	2830	3976	51
H2	8875	3713	3340	60
H3	9034	3702	1734	57
H9	8876	3144	128	56
H8	8422	2278	−801	55
H10	7886	1175	−903	55
H11	7372	346	−163	53
H12	7262	416	1489	43
H1A	8437	2796	4237	30
H2A	8991	3746	3838	48
H3A	9180	3912	2260	48
H9A	9007	3532	512	48
H8A	8635	2737	−591	28
H10A	8111	1645	−972	39
H11A	7599	717	−442	40
H12A	7393	684	1191	44
H13A	8360	674	4421	36
H13B	8422	497	3322	36
H14A	9111	1554	4336	54
H14B	9176	1371	3238	54
H15A	9596	528	4836	65
H15B	9677	362	3739	65
H16A	10455	1199	3704	166
H16B	10700	722	4584	166
H16C	10355	1404	4781	166
H17A	6676	2224	2370	44
H17B	6385	1502	2390	44
H18A	6171	1698	4028	45
H18B	6379	2441	3893	45
H18C	6456	2342	4008	45
H18D	5811	2321	3215	45
H19A	5290	1818	2760	55
H19B	5489	2570	2675	55
H20A	4656	2604	3663	86
H20B	5329	2643	4417	86
H20C	4933	1968	4243	86
H19C	6232	1385	4565	54
H19D	5664	1248	3673	54
H20D	4954	2069	4239	77
H20E	5528	2239	5110	77
H20F	5151	1550	5082	77

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

2.4. *Salt **phenHOTF**: R_1 = 3.96%*

2.4.1. *Crystal data and experimental*



Experimental. Single clear light colorless prism crystals of salt **phenHOTf** recrystallized from a mixture of dichloromethane and toluene by slow evaporation. A suitable crystal with dimensions 0.10 × 0.08 × 0.05 mm^3 was selected and mounted on a mylar loop oil on a Bruker Kappa Apex II diffractometer. The crystal was kept at a steady T = 115 K during data collection. The structure was solved with the ShelXT 2018/2 [8] solution program using dual methods and by using Olex2 [2] as the graphical interface. The model was refined with ShelXL 2018/3 [3] using full matrix least squares minimisation on F^2.

Crystal data. $C_{13}H_9F_3N_2O_3S$, M_r = 330.28, monoclinic, $P2_1/c$ (No. 14), a = 6.8543(3) Å, b = 23.6000(9) Å, c = 8.1856(3) Å, β = 97.931(3)°, $\alpha = \gamma$ = 90°, V = 1311.45(9) Å^3, T = 115 K, Z = 4, Z' = 1, μ(Mo K$_{\alpha 1}$) = 0.298, 22021 reflections measured, 3004 unique (R_{int} = 0.0504) which were used in all calculations. The final wR_2 was 0.0909 (all data) and R_1 was 0.0396 ($I \geq 2\sigma(I)$).

Compound CCDC	Compound **phenHOTf**
Formula	$C_{13}H_9F_3N_2O_3S$
$D_{calc.}$/ g·cm^{-3}	1.673
μ/mm^{-1}	0.298
Formula Weight	330.28
Colour	clear light colourless
Shape	prism
Size/mm^3	0.10 × 0.08 × 0.05
T/K	115
Crystal System	monoclinic
Space Group	$P2_1/c$
a/Å	6.8543(3)
b/Å	23.6000(9)
c/Å	8.1856(3)
α/°	90
β/°	97.931(3)
γ/°	90
V/Å^3	1311.45(9)
Z	4
Z'	1
Wavelength/Å	0.71073
Radiation type	Mo Kα_1
Θ_{min}/°	2.656
Θ_{max}/°	27.522
Measured Refl's.	22021
Indep't Refl's	3004
Refl's $I \geq 2\sigma(I)$	2422
R_{int}	0.0504
Parameters	199
Restraints	0
Largest Peak	0.420
Deepest Hole	−0.445
GooF	1.029
wR_2 (all data)	0.0909
wR_2	0.0837
R_1 (all data)	0.0568
R_1	0.0396

A clear light colorless prism-shaped crystal with dimensions 0.10 × 0.08 × 0.05 mm^3 was mounted on a mylar loop oil. Data were collected using a Bruker

Supplementary Table S19. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for salt **phenHOTf**

Atom	x	y	z	U_{eq}
C13	1938(3)	3599.7(8)	9903(2)	19.6(4)
F1	1977.6(19)	3325.0(5)	11333.9(14)	29.2(3)
F2	3769.4(17)	3600.8(5)	9519.7(16)	28.7(3)
F3	1438.1(19)	4137.6(5)	10151.1(16)	28.7(3)
O1	−1649(2)	3319.7(6)	8875.9(18)	22.0(3)
O2	973(2)	2700.8(6)	8203.8(18)	21.9(3)
O3	439(2)	3621.0(6)	6863.9(17)	21.5(3)
S	219.0(7)	3269.1(2)	8280.0(6)	15.76(12)
C1	4119(3)	2960.4(8)	5658(2)	19.6(4)
C2	5803(3)	2699.3(8)	5247(2)	21.0(4)
C3	7014(3)	2991.5(8)	4346(2)	20.0(4)
C4	6534(3)	3547.1(8)	3801(2)	16.8(4)
C5	4802(3)	3791.0(8)	4222(2)	15.4(4)
C6	4181(3)	4347.2(8)	3652(2)	15.9(4)
C7	5375(3)	4642.5(8)	2668(2)	18.9(4)
C8	7154(3)	4389.4(9)	2279(3)	22.9(4)
C9	7720(3)	3866.8(9)	2824(3)	22.5(4)
C10	4721(3)	5183.2(9)	2106(3)	24.6(5)
C11	2996(3)	5391.9(9)	2524(3)	24.7(5)
C12	1920(3)	5062.8(9)	3517(3)	23.3(4)
N1	3679(2)	3487.4(7)	5149.9(19)	16.2(3)
N2	2469(2)	4552.2(7)	4081(2)	19.0(3)

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Kappa Apex II diffractometer equipped with an Oxford Cryosystems low-temperature device operating at T = 115 K. Data were measured using ϕ and ω scans using MoKα_1 radiation. The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program Apex3 [4]. The maximum resolution that was achieved was Θ = 27.522°(0.77 Å).

The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program Apex3 [4]. The unit cell was refined using SainT [5], on 5201 reflections, 24% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using SainT [5]. The final completeness is 100.00% out to 27.522° in Θ. A multi-scan absorption correction was performed using SadabS-2016/2 [11] was used for absorption correction. wR_2(int) was 0.0548 before and 0.0486 after correction. The ratio of minimum to maximum transmission is 0.8822. The absorption coefficient μ of this material is 0.298 mm^{-1} at this wavelength (λ = 0.71073 Å) and the minimum and maximum transmissions are 0.658 and 0.746.

The structure was solved and the space group $P2_1/c$ (#14) determined by the ShelXT 2018/2 [8] structure solution program using using dual methods and refined by full matrix least squares minimisation on F^2 using version 2018/3 of ShelXL 2018/3 [3]. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model.

Supplementary Table S20. Anisotropic Displacement Parameters ($\times 10^4$) for salt **phenHOTf**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + \cdots + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C13	21.9(10)	15.9(10)	20.9(10)	−0.2(8)	2.0(8)	−3.0(8)
F1	39.6(8)	28.7(7)	17.4(6)	3.7(5)	−3.6(5)	−4.1(6)
F2	17.9(6)	27.6(7)	39.8(8)	2.4(6)	1.7(5)	−4.2(5)
F3	35.6(7)	15.9(6)	33.0(7)	−7.1(5)	−0.8(6)	−1.8(5)
O1	20.1(7)	20.0(7)	26.7(8)	0.3(6)	6.1(6)	−1.2(6)
O2	25.2(7)	14.4(7)	26.4(8)	−3.5(6)	4.6(6)	−0.4(6)
O3	22.0(7)	23.7(7)	18.9(7)	3.9(6)	3.6(6)	2.6(6)
S	16.4(2)	14.4(2)	16.5(2)	−0.93(18)	2.50(17)	−1.58(18)
C1	24.9(10)	17.2(10)	16.0(10)	0.6(8)	0.8(8)	−2.4(8)
C2	24.9(10)	15.4(9)	21.5(10)	−1.5(8)	−0.8(8)	1.9(8)
C3	17.9(10)	21.9(10)	19.2(10)	−7.1(8)	−0.8(8)	3.9(8)
C4	16.3(9)	19.6(10)	13.5(9)	−5.3(7)	−1.7(7)	−0.4(7)
C5	16.9(9)	18.0(10)	10.6(9)	−3.8(7)	−0.6(7)	−3.0(7)
C6	17.7(9)	15.8(9)	13.0(9)	−3.7(7)	−1.8(7)	−2.0(7)
C7	20.3(10)	20.2(10)	14.6(9)	−0.5(8)	−2.9(7)	−3.8(8)
C8	20.0(10)	28.0(11)	20.8(10)	0.9(8)	3.0(8)	−7.3(8)
C9	16.0(9)	29.1(11)	22.6(11)	−2.8(9)	3.9(8)	−0.5(8)
C10	31.1(12)	22.4(11)	18.9(10)	3.3(8)	−1.5(9)	−6.7(9)
C11	32.3(12)	17.3(10)	21.4(11)	0.9(8)	−7.7(9)	1.6(9)
C12	24.9(11)	22.5(11)	20.7(11)	−3.4(8)	−3.5(8)	4.7(8)
N1	16.9(8)	15.6(8)	16.0(8)	−3.0(6)	2.3(6)	1.4(6)
N2	20.5(8)	19.0(8)	16.4(8)	−2.7(7)	−1.4(6)	2.8(7)

Supplementary Table S21. Bond Lengths in Å for salt **phenHOTf**

Atom	Atom	Length (Å)
C13	F1	1.336(2)
C13	F2	1.335(2)
C13	F3	1.337(2)
C13	S	1.825(2)
O1	S	1.4367(14)
O2	S	1.4417(14)
O3	S	1.4505(14)
C1	C2	1.390(3)
C1	N1	1.333(2)
C2	C3	1.370(3)
C3	C4	1.409(3)
C4	C5	1.404(3)
C4	C9	1.432(3)
C5	C6	1.438(3)
C5	N1	1.358(2)
C6	C7	1.410(3)
C6	N2	1.359(2)
C7	C8	1.433(3)
C7	C10	1.408(3)
C8	C9	1.350(3)
C10	C11	1.367(3)
C11	C12	1.405(3)
C12	N2	1.326(3)

Supplementary Table S22. Bond Angles in ° for salt **phenHOTf**

Atom	Atom	Atom	Angle (°)
F1	C13	F3	107.57(16)
F1	C13	S	111.34(13)
F2	C13	F1	107.49(16)
F2	C13	F3	107.62(15)
F2	C13	S	111.31(14)
F3	C13	S	111.31(14)
O1	S	C13	103.50(9)
O1	S	O2	115.68(8)
O1	S	O3	114.80(8)
O2	S	C13	103.31(9)
O2	S	O3	114.88(8)
O3	S	C13	102.07(9)
N1	C1	C2	119.87(18)
C3	C2	C1	119.49(18)
C2	C3	C4	120.39(18)
C3	C4	C9	122.75(18)
C5	C4	C3	118.21(18)
C5	C4	C9	119.03(17)
C4	C5	C6	121.16(17)
N1	C5	C4	118.97(17)
N1	C5	C6	119.84(17)
C7	C6	C5	117.86(17)
N2	C6	C5	117.94(17)
N2	C6	C7	124.20(18)
C6	C7	C8	120.01(18)
C10	C7	C6	116.75(18)
C10	C7	C8	123.23(19)
C9	C8	C7	121.45(19)
C8	C9	C4	120.46(18)
C11	C10	C7	119.50(19)
C10	C11	C12	119.14(19)
N2	C12	C11	123.9(2)
C1	N1	C5	123.04(17)
C12	N2	C6	116.54(18)

Supplementary Table S23. Torsion Angles in ° for salt **phenHOTf**

Atom	Atom	Atom	Atom	Angle (°)
F1	C13	S	O1	61.58(15)
F1	C13	S	O2	−59.40(15)
F1	C13	S	O3	−178.91(13)
F2	C13	S	O1	−178.50(13)
F2	C13	S	O2	60.52(15)
F2	C13	S	O3	−59.00(15)
F3	C13	S	O1	−58.44(15)
F3	C13	S	O2	−179.42(13)
F3	C13	S	O3	61.07(15)
C1	C2	C3	C4	−1.7(3)
C2	C1	N1	C5	0.4(3)

Continued on next page

Supplementary Table S23. (Continued.)

Atom	Atom	Atom	Atom	Angle (°)
C2	C3	C4	C5	0.6(3)
C2	C3	C4	C9	−178.49(19)
C3	C4	C5	C6	−177.47(17)
C3	C4	C5	N1	0.9(3)
C3	C4	C9	C8	177.38(19)
C4	C5	C6	C7	−0.5(3)
C4	C5	C6	N2	178.77(17)
C4	C5	N1	C1	−1.4(3)
C5	C4	C9	C8	−1.7(3)
C5	C6	C7	C8	−0.6(3)
C5	C6	C7	C10	179.15(17)
C5	C6	N2	C12	−179.04(17)
C6	C5	N1	C1	176.95(17)
C6	C7	C8	C9	0.5(3)
C6	C7	C10	C11	−0.2(3)
C7	C6	N2	C12	0.2(3)
C7	C8	C9	C4	0.6(3)
C7	C10	C11	C12	0.4(3)
C8	C7	C10	C11	179.50(19)
C9	C4	C5	C6	1.7(3)
C9	C4	C5	N1	−179.95(17)
C10	C7	C8	C9	−179.18(19)
C10	C11	C12	N2	−0.3(3)
C11	C12	N2	C6	0.0(3)
N1	C1	C2	C3	1.2(3)
N1	C5	C6	C7	−178.89(17)
N1	C5	C6	N2	0.4(3)
N2	C6	C7	C8	−179.84(18)
N2	C6	C7	C10	−0.1(3)

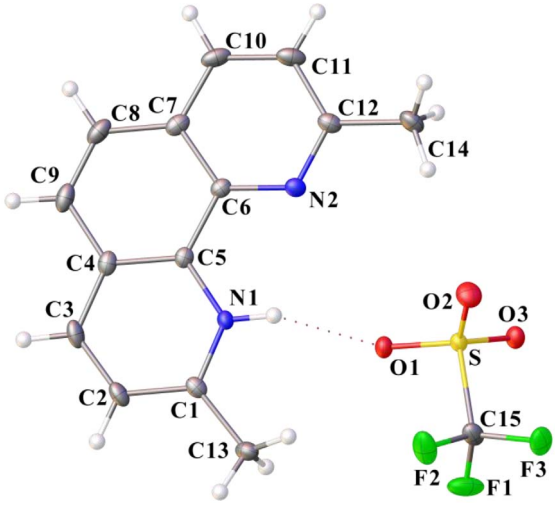
Supplementary Table S24. Hydrogen fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for salt **phenHOTf**

Atom	x	y	z	U_{eq}
H1	3284.17	2763.6	6299.88	23
H2	6111.64	2321.24	5587.51	25
H3	8185.14	2818.49	4086.95	24
H8	7953.59	4593.65	1622.27	28
H9	8913.37	3710.13	2558.11	27
H10	5471.7	5401.18	1442.32	29
H11	2532.05	5754.8	2148.69	30
H12	727.98	5214.93	3800.8	28
H1A	2610.56	3646	5428.65	19

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

2.5. *Salt dmphenHOTf:* $R_1 = 3.12\%$

2.5.1. *Crystal data and experimental*



Experimental. Single clear light colourless prism-shaped crystals of salt **dmphenHOTf** were used as supplied. A suitable crystal with dimensions 0.50 × 0.20 × 0.13 mm^3 was selected and mounted on a mylar loop with grease on a Bruker D8 Venture triumph Mo diffractometer. The crystal was kept at a steady T = 100 K during data collection. The structure was solved with the ShelXT [8] solution program using dual methods and by using Olex2 1.5 [2] as the graphical interface. The model was refined with XL [3] using full matrix least squares minimisation on F^2.

Crystal data. $C_{15}H_{13}F_3N_2O_3S$, M_r = 358.33, orthorhombic, *Pna*2$_1$ (No. 33), a = 7.2497(3) Å, b = 18.3555(5) Å, c = 11.5922(8) Å, $\alpha = \beta = \gamma = 90$ °, V = 1542.60(13) Å^3, T = 100 K, Z = 4, Z' = 1, μ (Mo K$_\alpha$) = 0.260, 15961 reflections measured, 3528 unique (R_{int} = 0.0305) which were used in all calculations. The final wR_2 was 0.0779 (all data) and R_1 was 0.0312 ($I \geq 2\sigma(I)$).

Compound	Compound **dmphenHOTf**
CCDC	2254276
Formula	$C_{15}H_{13}F_3N_2O_3S$
$D_{calc.}$/g·cm^{-3}	1.543
μ/mm^{-1}	0.260
Formula Weight	358.33
Colour	clear light colourless
Shape	prism-shaped
Size/mm^3	0.50 × 0.20 × 0.13
T/K	100
Crystal System	orthorhombic
Flack Parameter	0.02(2)
Hooft Parameter	0.03(2)
Space Group	*Pna*2$_1$
a/Å	7.2497(3)
b/Å	18.3555(5)
c/Å	11.5922(8)
α/°	90
β/°	90
γ/°	90
V/Å^3	1542.60(13)
Z	4
Z'	1
Wavelength/Å	0.71073
Radiation type	Mo Kα
Θ_{min}/°	3.495
Θ_{max}/°	27.540
Measured Refl's.	15961
Indep't Refl's	3528
Refl's $I \geq 2\sigma$(I)	3145
R_{int}	0.0305
Parameters	219
Restraints	1
Largest Peak	0.276
Deepest Hole	−0.181
GooF	1.025
wR_2 (all data)	0.0779
wR_2	0.0743
R_1 (all data)	0.0388
R_1	0.0312

2.5.2. *Structure quality indicators*

A clear light colorless prism-shaped-shaped crystal with dimensions 0.50 × 0.20 × 0.13 mm^3 was mounted on a mylar loop with grease. Data were collected using a Bruker D8 Venture triumph Mo diffractometer equipped with an Oxford Cryosystems low-temperature device operating at T = 100 K. Data were measured using ϕ and ω scans' with Mo K$_\alpha$ radiation. The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program Apex3 [4]. The maximum resolution that was achieved was Θ = 27.540°(0.77 Å).

The unit cell was refined using SainT [5] on 9120 reflections, 57% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using SainT [5]. The final completeness is 99.70% out to 27.540° in Θ. SadabS-2014/3 [11] was used for absorption correction. wR_2(int) was 0.0932 before and 0.0514 after correction. The ratio of minimum to maximum transmission is 0.9234. The $\lambda/2$ correction factor is 0.00150. The absorption coefficient μ of this material is 0.260 mm^{-1} at this wavelength (λ = 0.71073 Å) and the minimum and maximum transmissions are 0.689 and 0.746.

The structure was solved and the space group $Pna2_1$ (#33) determined by the ShelXT [8] structure solution program using using dual methods and refined by full matrix least squares minimization on F^2 using version 2019/1 of ShelXL [3]. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model.

The Flack parameter was refined to 0.02(2). Determination of absolute structure using Bayesian statistics on Bijvoet differences using the Olex2 results in 0.03(2). Note: The Flack parameter is used to determine chirality of the crystal studied, the value should be near 0, a value of 1 means that the stereochemistry is wrong and the model should be inverted. A value of 0.5 means that the crystal consists of a racemic mixture of the two enantiomers.

Supplementary Table S25. Fractional atomic coordinates (×10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for salt **dmphenHOTf**

Atom	x	y	z	U_{eq}
S	3516.5(7)	4054.5(3)	6698.8(5)	20.64(15)
O1	3706(2)	3417.7(9)	5972.3(16)	25.8(4)
F2	2237(3)	3190.9(9)	8320.1(16)	50.9(5)
F3	3480(3)	4175.8(10)	8961.8(14)	43.3(5)
O3	5058(2)	4545.5(8)	6685.1(18)	26.9(4)
F1	5196(3)	3313.0(11)	8325.6(16)	54.2(6)
O2	1745(2)	4397.9(10)	6682(2)	36.5(4)
N1	2366(3)	2274.3(10)	4448.0(17)	17.5(4)
N2	1730(3)	3579.5(11)	3410.7(18)	20.8(4)
C5	1754(3)	2284.5(13)	3329(2)	19.2(5)
C6	1445(3)	2968.5(13)	2779(2)	19.6(5)
C4	1440(3)	1625.1(14)	2759(2)	23.8(5)
C3	1756(4)	974.8(13)	3374(3)	28.3(6)
C11	921(3)	4266.8(14)	1728(3)	30.5(6)
C10	614(4)	3650.3(15)	1097(2)	30.8(6)
C1	2672(3)	1663.7(13)	5045(2)	21.6(5)
C12	1467(3)	4216.2(14)	2894(3)	24.8(5)
C7	868(3)	2965.4(14)	1616(2)	24.9(5)
C2	2348(4)	996.0(13)	4489(3)	26.4(5)
C9	833(4)	1642.8(15)	1583(2)	30.2(6)
C13	3380(4)	1723.9(14)	6243(3)	27.7(6)
C8	567(4)	2285.9(16)	1040(2)	31.0(6)
C14	1717(4)	4892.4(14)	3604(3)	32.1(6)
C15	3613(4)	3661.7(14)	8151(2)	30.7(6)

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

Supplementary Table S26. Anisotropic displacement parameters ($\times 10^4$) for salt **dmphenHOTf**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2} \times U_{11} + \cdots + 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S	24.1(3)	17.7(2)	20.1(3)	−0.6(3)	1.1(3)	−0.7(2)
O1	31.8(10)	21.7(8)	23.9(10)	−3.6(7)	−0.1(8)	−3.1(7)
F2	79.9(15)	35.8(9)	36.9(10)	−0.8(8)	24.0(10)	−22.1(10)
F3	69.3(14)	39.1(9)	21.5(8)	−7.3(7)	9.9(8)	−7.6(9)
O3	31.1(9)	24.0(8)	25.5(8)	−1.0(8)	1.6(9)	−6.6(7)
F1	71.0(14)	61.5(12)	30.1(9)	16.6(8)	2.4(9)	27.2(10)
O2	29.8(10)	35.8(10)	43.8(11)	−2.3(11)	2.0(10)	7.4(8)
N1	14.7(10)	16.3(9)	21.5(9)	−2.2(8)	1.8(8)	−0.2(7)
N2	19.0(10)	23.5(10)	19.8(10)	2.3(9)	5.6(8)	1.7(8)
C5	13.5(12)	24.6(11)	19.6(11)	−3.0(10)	5.6(9)	−0.5(9)
C6	12.8(11)	27.6(12)	18.4(11)	0.0(10)	3.9(9)	−0.2(9)
C4	15.5(12)	29.6(12)	26.3(13)	−7.8(11)	6.2(10)	−3.3(10)
C3	26.5(14)	20.3(12)	38.1(16)	−10.2(12)	13.4(12)	−2.5(10)
C11	27.2(12)	37.2(13)	27.0(12)	13.5(14)	9.5(14)	8.9(10)
C10	21.9(14)	52.6(17)	18.0(12)	7.5(12)	3.7(11)	7.1(13)
C1	15.9(12)	21.4(12)	27.6(13)	1.8(10)	8.6(10)	1.7(10)
C12	20.9(12)	25.7(12)	27.7(12)	5.9(11)	7.0(11)	3.5(10)
C7	14.9(10)	40.6(14)	19.2(11)	−1.2(12)	4.2(12)	1.5(9)
C2	24.9(13)	16.8(11)	37.4(14)	−1.3(10)	10.1(12)	2.0(10)
C9	21.3(12)	39.9(14)	29.5(14)	−15.5(13)	5.3(12)	−6.1(10)
C13	26.9(14)	26.0(13)	30.1(13)	7.2(11)	−2.4(11)	−0.3(10)
C8	21.5(13)	51.5(16)	20.0(13)	−8.1(12)	1.4(10)	−2.9(12)
C14	39.9(17)	22.0(13)	34.4(15)	7.6(11)	8.1(11)	1.2(12)
C15	46.4(17)	24.5(12)	21.1(13)	0.9(10)	9.0(11)	−0.3(12)

Supplementary Table S27. Bond Lengths in Å for salt **dmphenHOTf**

Atom	Atom	Length (Å)
S	O1	1.4472(17)
S	O3	1.4359(17)
S	O2	1.4306(19)
S	C15	1.832(3)
F2	C15	1.334(3)
F3	C15	1.336(3)
F1	C15	1.329(3)
N1	C5	1.371(3)
N1	C1	1.336(3)
N2	C6	1.355(3)
N2	C12	1.327(3)
C5	C6	1.426(3)
C5	C4	1.397(3)
C6	C7	1.412(4)
C4	C3	1.409(4)
C4	C9	1.433(4)
C3	C2	1.363(4)
C11	C10	1.366(4)
C11	C12	1.412(4)
C10	C7	1.406(4)
C1	C2	1.404(3)
C1	C13	1.485(4)
C12	C14	1.500(4)
C7	C8	1.432(4)
C9	C8	1.352(4)

Supplementary Table S28. Bond Angles in ° for salt **dmphenHOTf**

Atom	Atom	Atom	Angle (°)
O1	S	C15	102.31(11)
O3	S	O1	115.26(11)
O3	S	C15	103.14(12)
O2	S	O1	115.68(12)
O2	S	O3	114.96(11)
O2	S	C15	102.71(14)
C1	N1	C5	123.7(2)
C12	N2	C6	117.6(2)
N1	C5	C6	119.1(2)
N1	C5	C4	119.2(2)
C4	C5	C6	121.7(2)
N2	C6	C5	117.6(2)
N2	C6	C7	124.4(2)
C7	C6	C5	118.0(2)
C5	C4	C3	117.9(2)
C5	C4	C9	118.7(2)
C3	C4	C9	123.4(2)
C2	C3	C4	120.5(2)
C10	C11	C12	120.3(2)
C11	C10	C7	119.3(2)
N1	C1	C2	117.8(2)
N1	C1	C13	118.7(2)
C2	C1	C13	123.5(2)
N2	C12	C11	122.0(2)
N2	C12	C14	117.6(2)
C11	C12	C14	120.3(2)
C6	C7	C8	119.6(2)
C10	C7	C6	116.4(2)
C10	C7	C8	124.0(2)
C3	C2	C1	120.8(2)
C8	C9	C4	120.5(2)
C9	C8	C7	121.4(2)
F2	C15	S	111.2(2)
F2	C15	F3	107.5(2)
F3	C15	S	111.46(18)
F1	C15	S	111.25(18)
F1	C15	F2	108.1(2)
F1	C15	F3	107.2(2)

Supplementary Table S29. Torsion Angles in ° for salt **dmphenHOTf**

Atom	Atom	Atom	Atom	Angle (°)
O1	S	C15	F2	−60.9(2)
O1	S	C15	F3	179.27(19)
O1	S	C15	F1	59.7(2)
O3	S	C15	F2	179.18(18)
O3	S	C15	F3	59.3(2)

Continued on next page

Supplementary Table S29. (Continued.)

Atom	Atom	Atom	Atom	Angle (°)
O3	S	C15	F1	−60.3(2)
O2	S	C15	F2	59.4(2)
O2	S	C15	F3	−60.5(2)
O2	S	C15	F1	179.95(19)
N1	C5	C6	N2	−1.7(3)
N1	C5	C6	C7	178.2(2)
N1	C5	C4	C3	0.5(3)
N1	C5	C4	C9	−178.9(2)
N1	C1	C2	C3	0.5(3)
N2	C6	C7	C10	1.2(3)
N2	C6	C7	C8	−178.5(2)
C5	N1	C1	C2	0.1(3)
C5	N1	C1	C13	178.5(2)
C5	C6	C7	C10	−178.7(2)
C5	C6	C7	C8	1.6(3)
C5	C4	C3	C2	0.1(4)
C5	C4	C9	C8	−0.2(4)
C6	N2	C12	C11	−0.4(3)
C6	N2	C12	C14	177.9(2)
C6	C5	C4	C3	−179.1(2)
C6	C5	C4	C9	1.5(3)
C6	C7	C8	C9	−0.3(4)
C4	C5	C6	N2	177.9(2)
C4	C5	C6	C7	−2.2(3)
C4	C3	C2	C1	−0.6(4)
C4	C9	C8	C7	−0.4(4)
C3	C4	C9	C8	−179.6(2)
C11	C10	C7	C6	−0.3(3)
C11	C10	C7	C8	179.4(2)
C10	C11	C12	N2	1.2(4)
C10	C11	C12	C14	−177.0(2)
C10	C7	C8	C9	−180.0(2)
C1	N1	C5	C6	179.0(2)
C1	N1	C5	C4	−0.6(3)
C12	N2	C6	C5	179.1(2)
C12	N2	C6	C7	−0.8(3)
C12	C11	C10	C7	−0.8(4)
C9	C4	C3	C2	179.4(2)
C13	C1	C2	C3	−177.8(3)

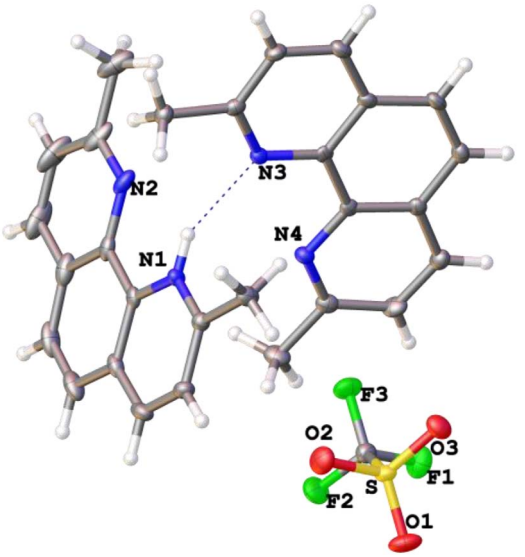
Supplementary Table S30. Hydrogen Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters (Å^2 × 10^3) for salt **dmphenHOTf**

Atom	x	y	z	U_{eq}
H1	2568.27	2693.95	4792.59	21
H3	1555.13	518.34	3008.95	34
H11	767.72	4731.61	1380.05	37
H10	231.44	3683.78	314.62	37
H2	2545	553.46	4896.2	32
H9	616.76	1199.45	1182.84	36
H13A	4719.74	1793.08	6226.38	41
H13B	3086.64	1277.19	6668.48	41
H13C	2799.45	2141.22	6626.01	41
H8	171.82	2286.94	258.23	37
H14A	1833.7	4759.41	4419.29	48
H14B	645.33	5211.67	3502.41	48
H14C	2833.97	5148.01	3353.5	48

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij}.

2.6. *Salt dmphenHOTf.dmphen* $R_1 = 5.89\%$

2.6.1. *Crystal data and experimental*



Experimental. Single colorless prism-shaped crystals of salt **dmphenHOTf·dmphen** were obtained by recrystallization. A suitable crystal ($0.37 \times 0.08 \times 0.07$ mm^3) was selected and mounted on a mylar loop with grease on a Bruker D8 Venture triumph Mo diffractometer. The crystal was kept at $T = 100$ K during data collection. Using Olex2 [2], the structure was solved with the ShelXT structure solution program [8], using the direct methods solution method. The model was refined with version of ShelXL [3] using Least Squares minimization.

Crystal data. $C_{29}H_{25}F_3N_4O_3S$, $M_r = 566.59$, monoclinic, $P2_1/n$ (No. 14), $a = 9.4592(10)$ Å, $b = 13.8145(14)$ Å, $c = 20.028(2)$ Å, $\beta = 97.216(3)°$, $\alpha = \gamma = 90°$, $V = 2596.5(5)$ Å^3, $T = 100$ K, $Z = 4$, $Z' = 1$, μ (Mo K_α) = 0.187, 54934 reflections measured, 5938 unique ($R_{int} = 0.0808$) which were used in all calculations. The final wR_2 was 0.1071 (all data) and R_1 was 0.0589 ($I > 2(I)$).

Compound	**dmphenHOTf-dmphen**
CCDC	2254277
Formula	$C_{29}H_{25}F_3N_4O_3S$
$D_{calc.}$/ g·cm^{-3}	1.449
μ/mm^{-1}	0.187
Formula Weight	566.59
Colour	colourless
Shape	prism
Max Size/mm	0.37
Mid Size/mm	0.08
Min Size/mm	0.07
T/K	100
Crystal System	monoclinic
Space Group	$P2_1/n$
a/Å	9.4592(10)
b/Å	13.8145(14)
c/Å	20.028(2)
α/°	90
β/°	97.216(3)
γ/°	90
V/Å^3	2596.5(5)
Z	4
Z'	1
Θ_{min}/°	2.915
Θ_{max}/°	27.538
Measured Refl.	54934
Independent Refl.	5938
Reflections Used	4281
R_{int}	0.0808
Parameters	365
Restraints	0
Largest Peak	0.308
Deepest Hole	−0.419
GooF	1.167
wR_2 (all data)	0.1071
wR_2	0.0983
R_1 (all data)	0.0946
R_1	0.0589

Experimental extended. A colorless prism-shaped crystal with dimensions 0.37 × 0.08 × 0.07 mm^3 was mounted on a mylar loop with grease. Data were collected using a Bruker D8 Venture triumph Mo diffractometer equipped with an Oxford Cryosystems low-temperature apparatus operating at T = 100 K. The actually achieved resolution was Θ = 27.538. Cell parameters were retrieved using the SAINT software [5] and refined using SAINT [5] on 9997 reflections, 18 of the observed reflections. Data reduction was performed using the SAINT [5] software which corrects for Lorentz polarization. The final completeness is 99.70 out to 27.538 in Θ. The absorption coefficient (MU) of this material is 0.187 and the minimum and maximum transmissions are 0.6946 and 0.7456.

The structure was solved by direct methods using the SHELXT [8] structure solution program and refined by Least Squares using version of SHELXL [3]. The structure was solved in the space group P2$_1$/n (#14). All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model. An hydrogene atom was split in half on the two N1 and N3 atoms. The coordinates of the hydrogens bonded to the nitrogen atoms were refined with a set of restrains.

Supplementary Table S31. Fractional atomic coordinates (×10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for **dmphenHOTf·dmphen**

Atom	x	y	z	U_{eq}
C27	2621(3)	7849.5(18)	3600.0(12)	18.6(5)
C15	1435(3)	7290.2(17)	3849.4(12)	15.6(5)
C19	394(2)	6754.6(16)	4788.2(12)	13.4(5)
C20	403(2)	6742.5(16)	5510.4(12)	13.3(5)
C26	1352(3)	7310.1(18)	6551.7(12)	17.8(5)
C28	2428(3)	7942(2)	6961.4(13)	23.9(6)
C25	392(3)	6745.9(19)	6866.2(13)	22.5(6)
C16	391(3)	6805.8(18)	3415.3(12)	19.1(5)
C17	−643(3)	6287.3(18)	3673.3(12)	18.7(5)
C18	−668(2)	6243.0(17)	4374.0(12)	15.0(5)
C23	−1689(2)	5674.8(18)	4674.3(12)	17.7(5)
C22	−1638(2)	5619.3(17)	5348.0(12)	17.1(5)
C21	−606(2)	6160.0(17)	5783.0(12)	14.8(5)
C24	−571(3)	6164.3(18)	6489.7(12)	19.5(5)
N3	1412(2)	7271.1(14)	4514.2(10)	13.6(4)
N4	1368(2)	7307.1(14)	5888.7(10)	15.3(4)
C4	5356(3)	8938.4(18)	6171.5(12)	19.1(5)
C2	5942(3)	7263.1(18)	6032.2(12)	18.2(5)
C1	4836(3)	7193.4(17)	5502.5(12)	15.5(5)
C5	4268(2)	8831.9(17)	5628.4(12)	15.2(5)
C6	3377(3)	9645.0(17)	5402.4(13)	19.0(5)
C12	1592(3)	10278(2)	4641.6(13)	29.5(7)
C14	501(3)	10140(2)	4038.6(14)	37.2(8)
C11	1774(4)	11197(2)	4953.5(15)	40.4(9)
C10	2757(4)	11320(2)	5502.3(15)	37.4(8)
C7	3606(3)	10531.9(18)	5749.0(14)	26.7(6)
C8	4676(3)	10603(2)	6314.9(14)	30.2(7)
C9	5529(3)	9848(2)	6515.9(14)	27.6(7)
C3	6202(3)	8120.3(19)	6363.5(13)	21.6(6)
C13	4509(3)	6258.8(17)	5134.5(13)	20.4(6)
N1	4022(2)	7960.5(14)	5312.3(10)	14.8(4)
N2	2369(2)	9517.3(15)	4859(1)	20.8(5)
C29	4449(3)	5330.0(18)	7146.2(13)	20.0(5)
O2	6362.4(19)	4717.9(14)	6452.4(9)	29.2(5)
O3	4360.9(19)	3656.4(13)	6573.9(9)	24.9(4)
O1	6180(2)	3931.9(14)	7523.9(9)	30.1(5)
F2	5304.3(17)	6011.8(11)	7444.6(8)	32.1(4)
F3	3691.0(16)	5736.5(11)	6608.2(7)	28.6(4)
F1	3530.9(17)	5085.6(11)	7571.3(8)	31.5(4)
S	5456.5(6)	4285.4(5)	6898.0(3)	16.91(14)

U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{ij}.

Supplementary Table S32. Anisotropic displacement parameters ($\times 10^4$) **dmphenHOTf·dmphen**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[a^{*2} \times U_{11} + \cdots 2hka^* \times b^* \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C27	18.4(13)	19.4(13)	18.4(13)	2.3(10)	3.8(10)	−0.5(10)
C15	16.2(12)	15.4(12)	14.9(12)	1.2(10)	0.9(10)	4.4(10)
C19	12.5(12)	9.2(11)	17.9(12)	2.6(9)	−0.3(10)	3.9(9)
C20	13.0(12)	10.0(11)	16.7(12)	0.7(9)	1(1)	3.8(9)
C26	16.2(12)	17.8(13)	19.1(13)	−2.1(10)	0.8(10)	4.6(10)
C28	22.6(14)	30.6(15)	18.0(13)	−7.0(11)	0.1(11)	0.3(12)
C25	23.6(14)	31.5(15)	12.9(12)	0.8(11)	4.7(11)	2.3(12)
C16	22.1(14)	21.9(13)	12.6(12)	1.6(10)	−0.3(10)	0.6(11)
C17	15.8(13)	17.3(13)	21.3(13)	−2.8(11)	−3.9(10)	−0.5(10)
C18	11.8(12)	15.2(12)	16.9(12)	0.3(10)	−2.2(10)	3(1)
C23	12.1(12)	16.5(12)	22.9(13)	−1.9(11)	−3.7(10)	−1.1(10)
C22	12.0(12)	15.9(13)	23.5(13)	1.4(10)	3(1)	0.1(10)
C21	10.8(12)	14.2(12)	19.0(13)	1.5(10)	0.9(10)	2.9(9)
C24	17.0(13)	22.4(14)	19.5(13)	4.6(11)	3.8(11)	1.2(11)
N3	14(1)	11.8(10)	14.3(10)	−0.9(8)	−0.5(8)	0.6(8)
N4	13.7(10)	16.9(11)	14.8(10)	−1.6(8)	0.2(8)	0.1(8)
C4	19.2(13)	19.7(13)	19.5(13)	−4.7(10)	6.9(11)	−8(1)
C2	15.4(12)	21.1(13)	17.5(12)	2.5(10)	−0.3(10)	1.9(10)
C1	14.8(12)	15.7(12)	16.4(12)	0.5(10)	3.1(10)	0.1(10)
C5	14.4(12)	14.0(12)	18.7(12)	−0.3(10)	7.4(10)	−3.5(10)
C6	22.1(14)	13.6(12)	23.6(14)	2.5(10)	12.4(11)	−1.2(10)
C12	41.0(17)	30.4(16)	21.3(14)	11.9(12)	20.3(13)	17.9(13)
C14	44.6(19)	45.1(19)	24.5(15)	14.0(14)	14.0(14)	27.6(15)
C11	69(2)	24.6(16)	32.8(18)	12.6(14)	26.1(17)	27.3(16)
C10	68(2)	13.7(14)	36.7(18)	2.8(12)	30.6(17)	10.1(14)
C7	37.2(16)	15.8(14)	31.8(15)	−0.8(11)	22.8(13)	−0.5(12)
C8	38.8(17)	19.8(14)	36.2(16)	−13.8(12)	21.3(14)	−12.7(13)
C9	30.0(16)	28.8(16)	25.9(15)	−13.3(12)	11.1(12)	−14.4(13)
C3	15.2(13)	31.8(15)	16.9(13)	−0.8(11)	−1.6(11)	−4.7(11)
C13	23.5(14)	13.6(13)	22.7(13)	−2.5(10)	−2.9(11)	1.6(10)
N1	13.9(10)	11.8(10)	18.1(10)	−0.1(8)	−0.7(8)	−0.4(8)
N2	27.0(12)	17.8(11)	19.6(11)	7.4(9)	11(1)	7.3(9)
C29	20.7(13)	18.9(13)	19.9(13)	−0.3(11)	0.6(11)	−1.4(11)
O2	24.2(10)	36.1(11)	29.5(11)	4.5(9)	12.4(8)	1.2(9)
O3	23.1(10)	22.3(10)	28.4(10)	−6.9(8)	−0.5(8)	−1.4(8)
O1	32.9(11)	37.3(12)	18.3(10)	2.9(8)	−4.1(8)	13.8(9)
F2	38.4(10)	23.7(8)	32.3(9)	−8.4(7)	−2.4(8)	−7.6(7)
F3	31.8(9)	24.0(8)	28.2(8)	4.0(7)	−3.5(7)	9.1(7)
F1	34.2(9)	30.2(9)	34.0(9)	0.5(7)	19.5(8)	4.1(7)
S	15.6(3)	21.2(3)	13.6(3)	1.9(3)	0.8(2)	3.1(3)

Supplementary Table S33. Bond Lengths in Å for **dmphenHOTf·dmphen**

Atom	Atom	Length (Å)
C27	C15	1.498(3)
C15	C16	1.401(3)
C15	N3	1.335(3)
C19	C20	1.445(3)
C19	C18	1.410(3)
C19	N3	1.367(3)
C20	C21	1.410(3)
C20	N4	1.357(3)
C26	C28	1.504(3)
C26	C25	1.404(4)
C26	N4	1.330(3)
C25	C24	1.368(4)
C16	C17	1.365(3)
C17	C18	1.408(3)
C18	C23	1.434(3)
C23	C22	1.346(3)
C22	C21	1.434(3)
C21	C24	1.412(3)
C4	C5	1.408(3)
C4	C9	1.432(3)
C4	C3	1.410(4)
C2	C1	1.397(3)
C2	C3	1.365(4)
C1	C13	1.500(3)
C1	N1	1.336(3)
C5	C6	1.442(3)
C5	N1	1.366(3)
C6	C7	1.411(4)
C6	N2	1.366(3)
C12	C14	1.499(4)
C12	C11	1.415(4)
C12	N2	1.324(3)
C11	C10	1.359(5)
C10	C7	1.405(4)
C7	C8	1.425(4)
C8	C9	1.349(4)
C29	F2	1.333(3)
C29	F3	1.340(3)
C29	F1	1.334(3)
C29	S	1.832(3)
O2	S	1.4423(19)
O3	S	1.4426(18)
O1	S	1.4364(18)

Supplementary Table S34. Bond Angles in ° for **dmphenHOTf·dmphen**

Atom	Atom	Atom	Angle (°)
C16	C15	C27	122.6(2)
N3	C15	C27	116.7(2)
N3	C15	C16	120.7(2)
C18	C19	C20	119.8(2)
N3	C19	C20	119.4(2)
N3	C19	C18	120.7(2)
C21	C20	C19	118.3(2)
N4	C20	C19	118.1(2)
N4	C20	C21	123.5(2)
C25	C26	C28	120.6(2)
N4	C26	C28	117.1(2)
N4	C26	C25	122.4(2)
C24	C25	C26	120.1(2)
C17	C16	C15	119.9(2)
C16	C17	C18	120.2(2)
C19	C18	C23	119.7(2)
C17	C18	C19	117.7(2)
C17	C18	C23	122.6(2)
C22	C23	C18	120.7(2)
C23	C22	C21	121.0(2)
C20	C21	C22	120.3(2)
C20	C21	C24	117.0(2)
C24	C21	C22	122.7(2)
C25	C24	C21	119.1(2)
C15	N3	C19	120.8(2)
C26	N4	C20	117.9(2)
C5	C4	C9	119.5(2)
C5	C4	C3	117.5(2)
C3	C4	C9	123.0(2)
C3	C2	C1	120.1(2)
C2	C1	C13	121.4(2)
N1	C1	C2	120.8(2)
N1	C1	C13	117.8(2)
C4	C5	C6	120.2(2)

Continued on next page

Supplementary Table S34. (Continued.)

Atom	Atom	Atom	Angle (°)
N1	C5	C4	121.0(2)
N1	C5	C6	118.8(2)
C7	C6	C5	118.3(2)
N2	C6	C5	118.1(2)
N2	C6	C7	123.6(2)
C11	C12	C14	120.2(3)
N2	C12	C14	117.5(3)
N2	C12	C11	122.3(3)
C10	C11	C12	120.2(3)
C11	C10	C7	119.4(3)
C6	C7	C8	120.0(2)
C10	C7	C6	117.0(3)
C10	C7	C8	122.9(3)
C9	C8	C7	121.5(2)
C8	C9	C4	120.4(3)
C2	C3	C4	120.1(2)
C1	N1	C5	120.6(2)
C12	N2	C6	117.6(2)
F2	C29	F3	107.3(2)
F2	C29	F1	107.4(2)
F2	C29	S	111.80(18)
F3	C29	S	110.85(17)
F1	C29	F3	107.1(2)
F1	C29	S	112.11(17)
O2	S	C29	102.19(12)
O2	S	O3	114.84(11)
O3	S	C29	103.17(11)
O1	S	C29	103.78(11)
O1	S	O2	115.20(12)
O1	S	O3	115.13(12)

Supplementary Table S35. Hydrogen fractional atomic coordinates ($\times 10^4$) and Equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for **dmphenHOTf·dmphen**

Atom	x	y	z	U_{eq}
H27A	3534	7540	3758	28
H27B	2622	8514	3772	28
H27C	2482	7861	3107	28
H28A	2453	8578	6747	36
H28B	3371	7641	6989	36
H28C	2161	8019	7416	36
H25	411	6768	7341	27
H16	402	6838	2942	23
H17	−1347	5955	3379	22
H23	−2408	5334	4395	21
H22	−2296	5216	5538	20
H24	−1206	5769	6701	23
H3	2059	7595	4779	16
H2	6515	6713	6162	22
H14A	866	10403	3639	56
H14B	−378	10479	4109	56
H14C	302	9448	3973	56
H11	1206	11728	4779	48
H10	2871	11933	5717	45
H8	4791	11196	6556	36
H9	6249	9919	6888	33
H3A	6954	8166	6724	26
H13A	3476	6155	5068	31
H13B	4972	5724	5399	31
H13C	4864	6287	4696	31
H1	3323	7904	4981	18

U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{ij}.

An hydrogene atom was split in half on the two N1 AND N3 atoms. The coordinates of the hydrogens bonded to the nitrogen atoms were refined with a set of restrains.

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