# SUPPORTING INFORMATION

## Unravelling Structure - Luminescence Relationship in Two Dimensional Antimony(III) Doped Cadmium (II) Halide Hybrids

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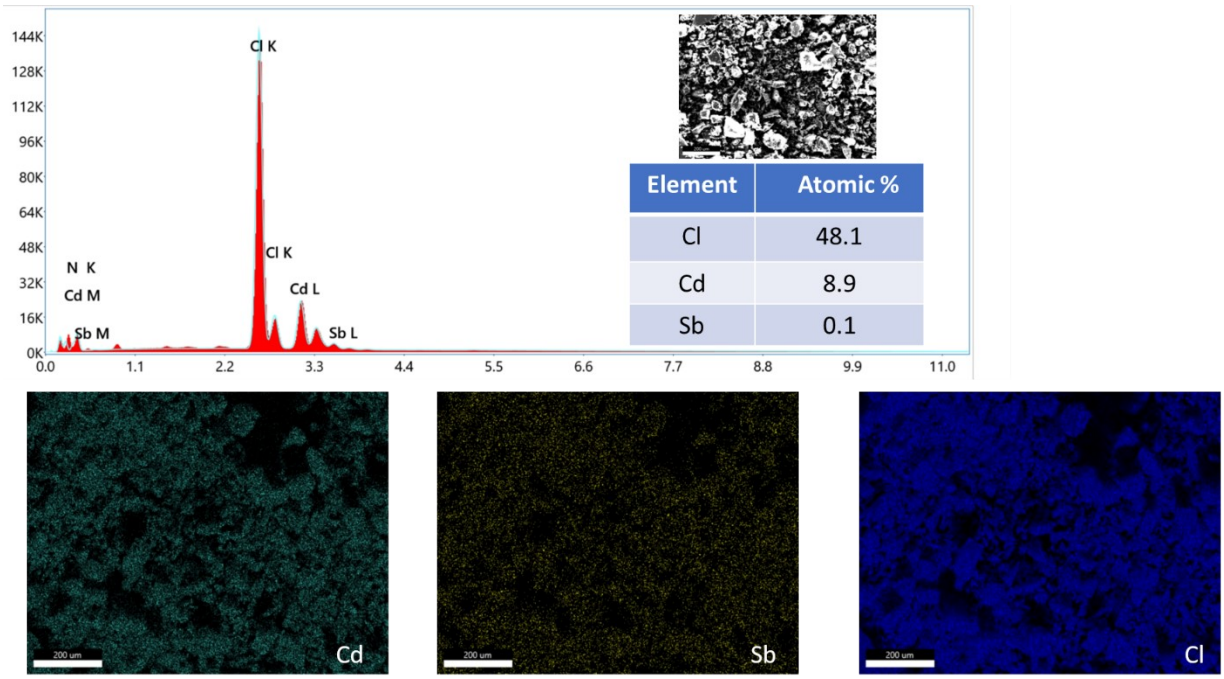


Figure S1. SEM EDS analysis of Sb:DETACdCl hybrid.

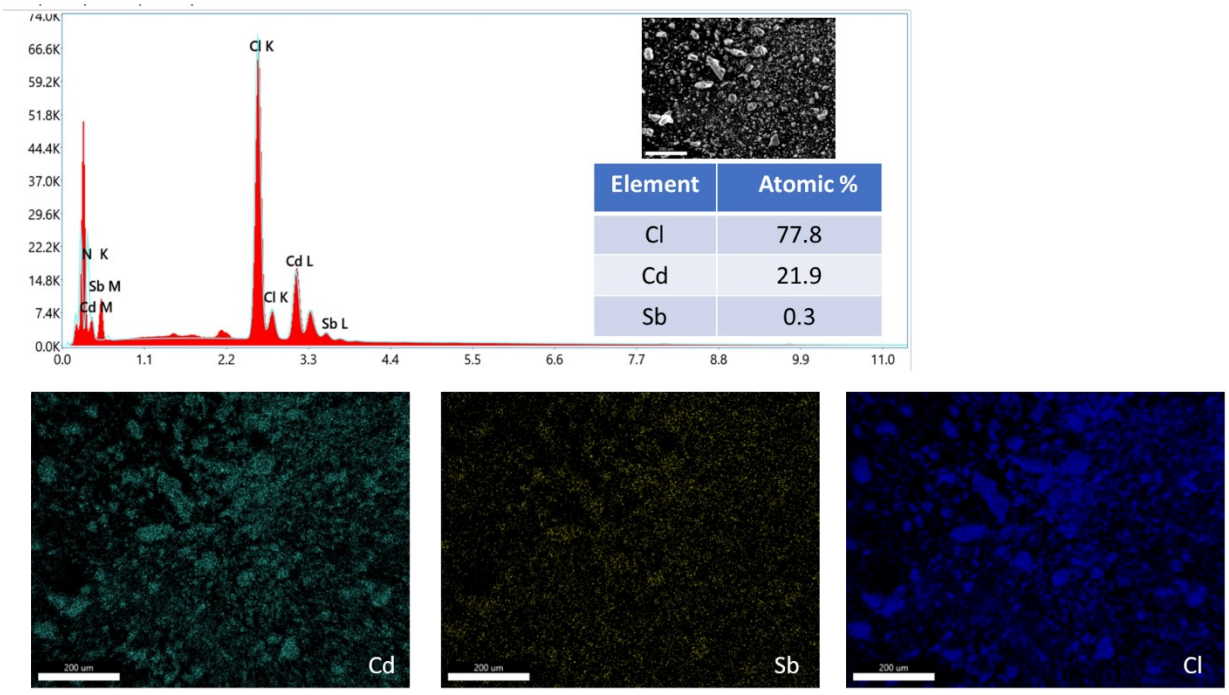


Figure S2. SEM EDS analysis of Sb:PDACdCl hybrid.

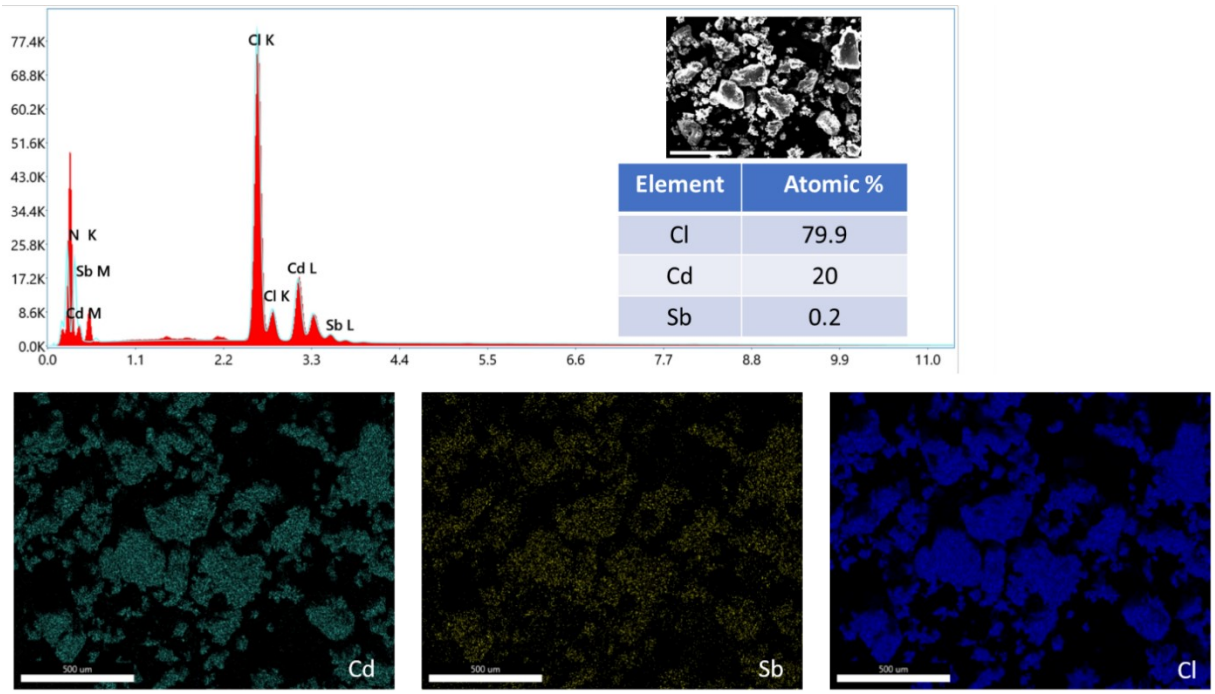


Figure S3. SEM EDS analysis of Sb:DABCdCl hybrid.

Table S1. ICP Analysis of doped hybrids (Sb:DETACdCl, Sb:PDACdCl, Sb:DABCdCl)

| Compound | Sb feed percentage | Sb percentage from ICP |
|---|---|---|
| Sb: PDACdCl | 10% | 0.0008 |
| | 30% | 0.0062 |
| | 50% | 0.0110 |
| Sb: DETACdCl | 10% | 0.0028 |
| | 30% | 0.0033 |
| | 50% | 0.0365 |
| Sb: DABCdCl | 10% | 0.0027 |
| | 30% | 0.0167 |
| | 50% | 0.0063 |

Table S2: Comparison of structural and optical properties of host hybrids.

| Compound | Space group | Octahedral distortion (d) | Bond angle variance ($deg^2$) | M-Cl-M (deg) | $\Phi_{tilt\ (deg)}$ | PL Max (nm) | PLE Peak positions (nm) | PLQY(%) |
|---|---|---|---|---|---|---|---|---|
| DETACdCl<br>C4 H16 N3 Cd Cl5 | Orthorhombic<br>*Pnma* | $8.51x10^{-5}$ | 2.1785 | 153.48 | 11.25 | 490 | 274, 330 , 380 | 3.5 |
| PDACdCl<br>C5 H16 N2 Cd Cl4 | Orthorhombic<br>*Imma* | $9.22x10^{-4}$ | 1.4491 | 168.40 | 12.49 | 480 | 398 | 0.5 |
| DABCdCl<br>$2(C_4H_{14}N_2)$<br>$Cd_2Cl_8O$ | Monoclinic<br>$P2_1/c$ | $1.4x10^{-3}$ | 0.6413 | 165.64 | 13.03 | 465 | 280, 330, 375 | 3.6 |

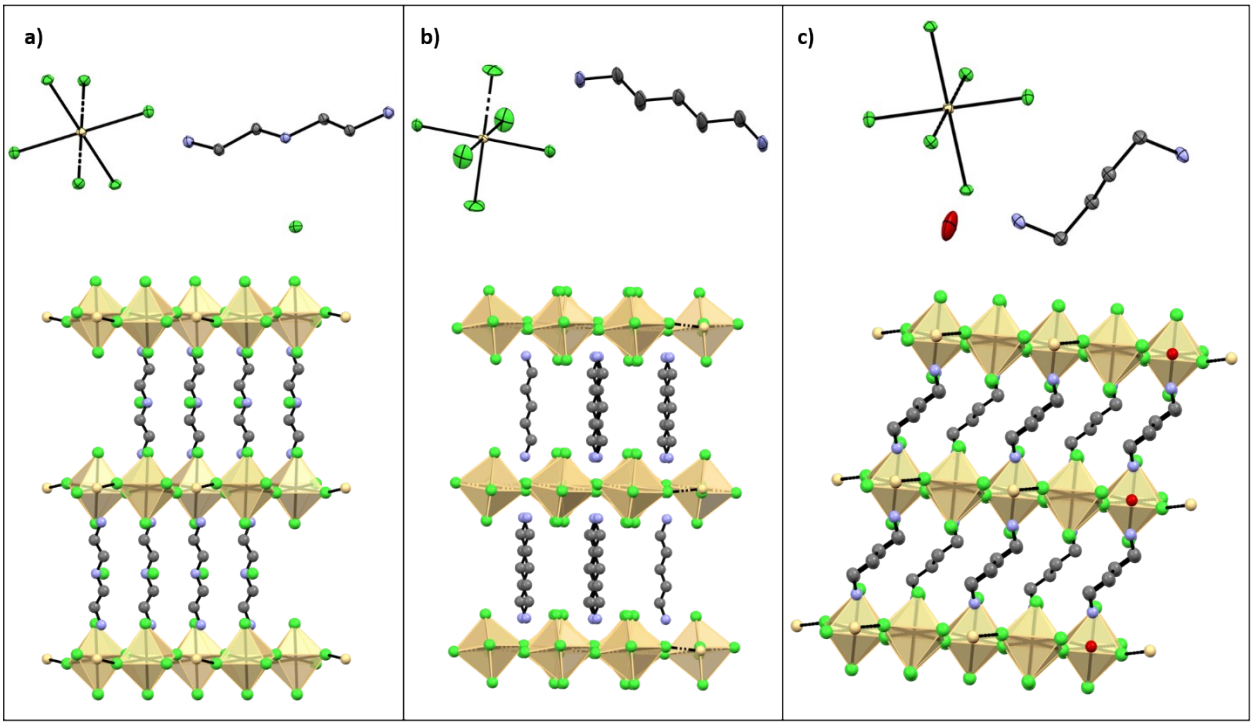


Figure S4: Asymmetric unit and packing diagram of host hybrids- a) DETACdCl, b) PDACdCl, c) DABCdCl.

Table S3: SCXRD parameters of doped hybrids

| Identification code | Sb:DETACdCl | Sb:PDACdCl | Sb:DABCdCl |
|---|---|---|---|
| **Empirical formula** | $C_4H_{16}CdCl_5N_3Sb$ | $C_5H_{16}CdCl_4N_2Sb$ | $C_8H_{28}Cd_2Cl_8N_4OSb$ |
| **Formula weight** | 395.85 | 358.40 | 704.74 |
| **Temperature/K** | 150.00 | 150.00 | 150.00 |
| **Crystal system** | orthorhombic | orthorhombic | monoclinic |
| **Space group** | Pnma | Imma | $P2_1/c$ |
| **a/Å** | 7.1960(4) | 7.2902(3) | 9.4098(4) |
| **b/Å** | 24.6826(14) | 24.0668(12) | 7.5645(3) |
| **c/Å** | 7.2487(4) | 7.4496(4) | 7.6395(4) |
| **α/°** | 90 | 90 | 90 |
| **β/°** | 90 | 90 | 102.01 |
| **γ/°** | 90 | 90 | 90 |
| **Volume/Å$^3$** | 1287.49(12) | 1307.05(11) | 531.89(4) |
| **Z** | 4 | 4 | 1 |
| **$\rho_{calc}$g/cm$^3$** | 2.042 | 1.821 | 2.200 |
| **μ/mm$^{-1}$** | 2.698 | 2.448 | 3.009 |
| **F(000)** | 776.0 | 704.0 | 344.0 |
| **Crystal size/mm$^3$** | 0.22 × 0.21 × 0.2 | 0.21 × 0.2 × 0.19 | 0.21 × 0.2 × 0.2 |
| **Radiation** | MoKα (λ = 0.71073) | MoKα (λ = 0.71073) | MoKα (λ = 0.71073) |
| **2Θ range for data collection/°** | 5.858 to 52.764 | 5.724 to 52.736 | 6.972 to 52.716 |
| **Index ranges** | -8 ≤ h ≤ 9, -30 ≤ k ≤ 30, -9 ≤ l ≤ 9 | -9 ≤ h ≤ 9, -30 ≤ k ≤ 30, -9 ≤ l ≤ 9 | -11 ≤ h ≤ 11, -9 ≤ k ≤ 9, -9 ≤ l ≤ 9 |
| **Reflections collected** | 27418 | 15463 | 5946 |
| **Independent reflections** | 1343 [$R_{int}$ = 0.0289, $R_{sigma}$ = 0.0122] | 742 [$R_{int}$ = 0.0506, $R_{sigma}$ = 0.0203] | 1092 [$R_{int}$ = 0.0366, $R_{sigma}$ = 0.0282] |
| **Data/restraints/parameters** | 1343/0/65 | 742/0/43 | 1092/3/70 |
| **Goodness-of-fit on F$^2$** | 1.107 | 1.093 | 1.163 |
| **Final R indexes [I>=2σ (I)]** | $R_1$ = 0.0132, $wR_2$ = 0.0379 | $R_1$ = 0.0318, $wR_2$ = 0.0939 | $R_1$ = 0.0251, $wR_2$ = 0.0651 |
| **Final R indexes [all data]** | $R_1$ = 0.0138, $wR_2$ = 0.0383 | $R_1$ = 0.0323, $wR_2$ = 0.0946 | $R_1$ = 0.0260, $wR_2$ = 0.0655 |
| **Largest diff. peak/hole / e Å$^{-3}$** | 0.37/-0.33 | 1.65/-0.90 | 0.57/-0.95 |

Table S4: SCXRD parameters of host hybrids

| Identification code | DETACdCl | PDACdCl | DABCdCl |
|---|---|---|---|
| **Empirical formula** | $C_4H_{16}CdCl_5N_3$ | $C_5H_{16}CdCl_4N_2$ | $C_8H_{28}Cd_2Cl_8N_4O$ |
| **Formula weight** | 395.85 | 358.40 | 704.781 |
| **Temperature/K** | 150.00 | 150.00 | 150.00 |
| **Crystal system** | orthorhombic | orthorhombic | monoclinic |
| **Space group** | Pnma | Imma | $P2_1/c$ |
| **a/Å** | 7.1977(3) | 7.2884(4) | 9.4203(6) |
| **b/Å** | 24.7093(9) | 24.0679(13) | 7.5615(4) |
| **c/Å** | 7.2486(3) | 7.4490(4) | 7.6317(5) |
| **α/°** | 90 | 90 | 90 |
| **β/°** | 90 | 90 | 101.956(2) |
| **γ/°** | 90 | 90 | 90 |
| **Volume/Å$^3$** | 1289.16(9) | 1306.68(12) | 531.83(6) |
| **Z** | 4 | 4 | 1 |
| **$\rho_{calc}$g/cm$^3$** | 2.040 | 1.822 | 2.201 |
| **μ/mm$^{-1}$** | 2.695 | 2.449 | 3.010 |
| **F(000)** | 776.0 | 704.0 | 343.7 |
| **Crystal size/mm$^3$** | 0.22 × 0.21 × 0.21 | 0.16 × 0.15 × 0.15 | 0.16 × 0.15 × 0.15 |
| **Radiation** | MoKα (λ = 0.71073) | MoKα (λ = 0.71073) | Mo Kα (λ = 0.71073) |
| **2Θ range for data collection/°** | 5.858 to 52.768 | 5.724 to 52.792 | 4.42 to 52.74 |
| **Index ranges** | -9 ≤ h ≤ 8, -30 ≤ k ≤ 30, -9 ≤ l ≤ 8 | -9 ≤ h ≤ 9, -30 ≤ k ≤ 30, -9 ≤ l ≤ 9 | -11 ≤ h ≤ 11, -9 ≤ k ≤ 9, -9 ≤ l ≤ 9 |
| **Reflections collected** | 15415 | 17999 | 8524 |
| **Independent reflections** | 1338 [$R_{int}$ = 0.0313, $R_{sigma}$ = 0.0141] | 737 [$R_{int}$ = 0.0262, $R_{sigma}$ = 0.0110] | 1082 [$R_{int}$ = 0.0429, $R_{sigma}$ = 0.0266] |
| **Data/restraints/parameters** | 1338/0/65 | 737/1/48 | 1082/3/70 |
| **Goodness-of-fit on F$^2$** | 1.137 | 1.145 | 1.007 |
| **Final R indexes [I>=2σ (I)]** | $R_1$ = 0.0140, $wR_2$ = 0.0366 | $R_1$ = 0.0205, $wR_2$ = 0.0646 | $R_1$ = 0.0290, $wR_2$ = 0.0880 |
| **Final R indexes [all data]** | $R_1$ = 0.0144, $wR_2$ = 0.0369 | $R_1$ = 0.0208, $wR_2$ = 0.0650 | $R_1$ = 0.0293, $wR_2$ = 0.0882 |
| **Largest diff. peak/hole / e Å$^{-3}$** | 0.37/-0.43 | 0.67/-0.66 | 0.69/-1.10 |

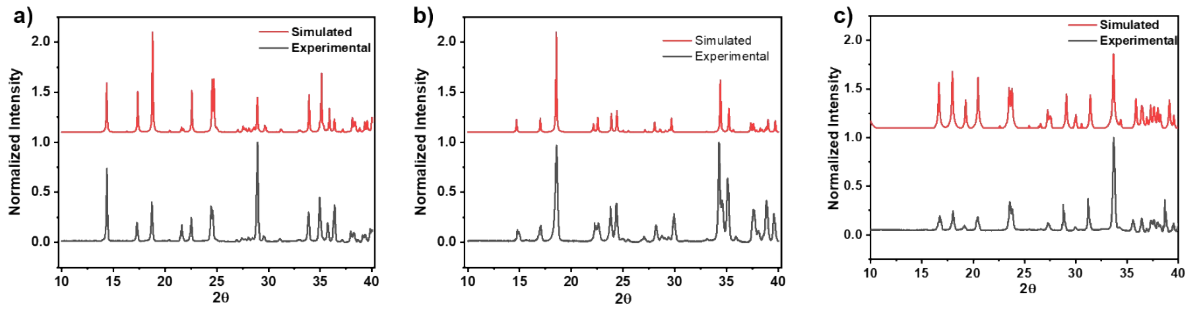


Figure S5: Simulated Vs. experimental PXRD patterns of doped hybrids- a) Sb:DETACdCl, b) Sb:PDACdCl, c) Sb:DABCdCl.

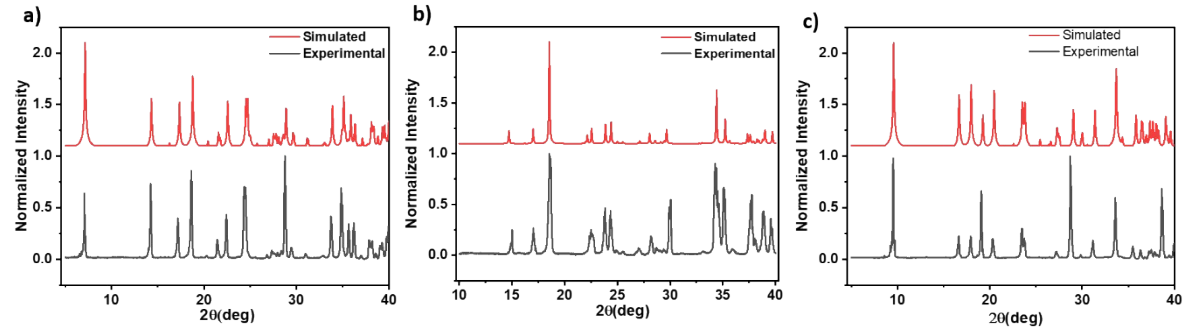


Figure S6: Simulated Vs. experimental PXRD patterns of host hybrids- a) DETACdCl, b) PDACdCl, c) DABCdCl.

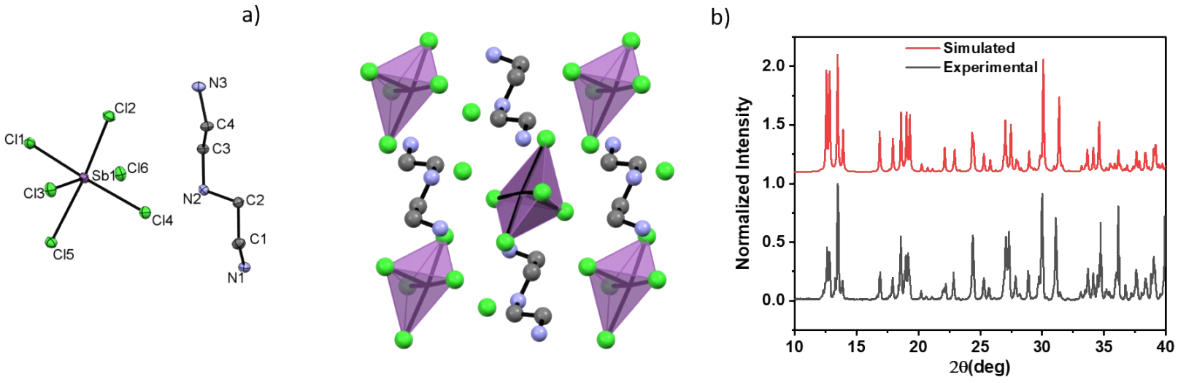


Figure S7: a) SCXRD structure of DETASbCl control hybrid; b) Comparison of simulated and experimental PXRD of DETASbCl control hybrid.

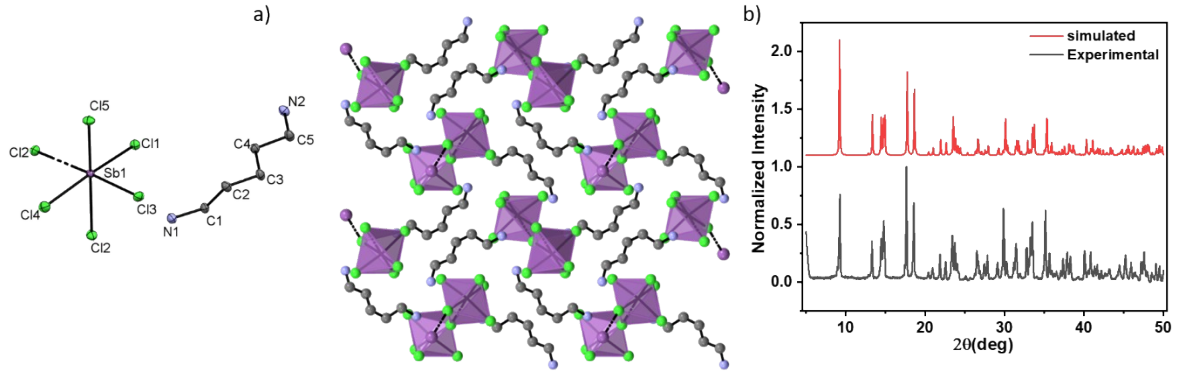


Figure S8: a) SCXRD structure of PDASbCl control hybrid; b) Comparison of simulated and experimental PXRD of PDASbCl control hybrid

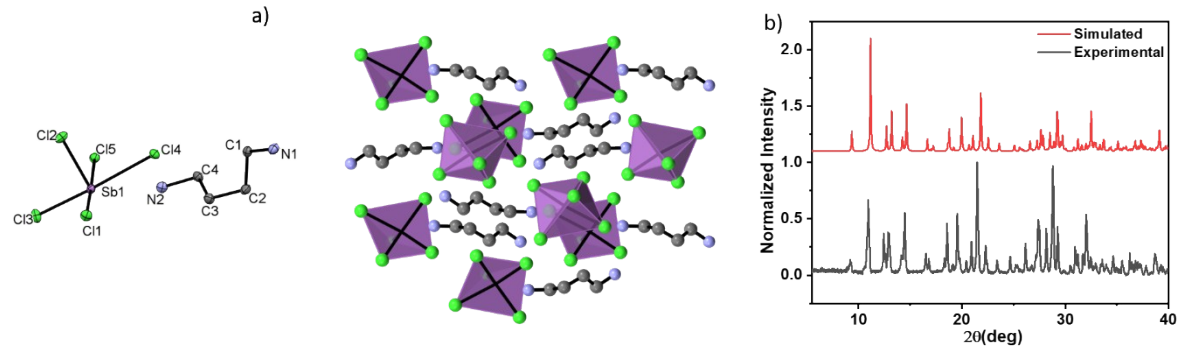


Figure S9: a) SCXRD structure of DABSbCl control hybrid; b) Comparison of simulated and experimental PXRD of DABSbCl control hybrid.

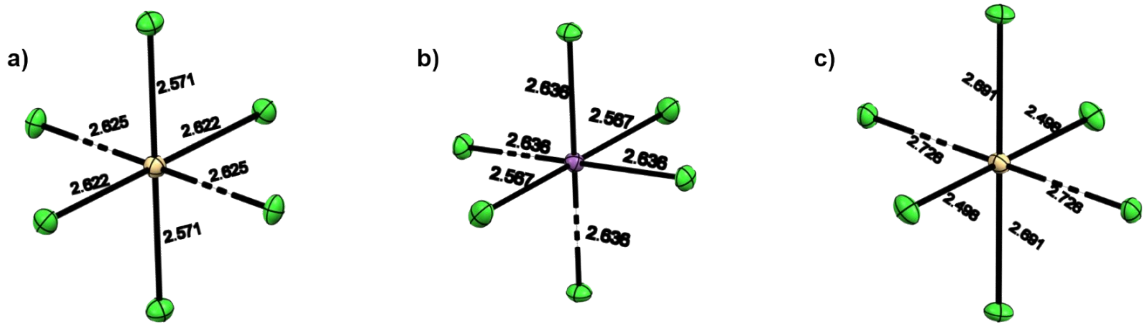


Figure S10. Single crystal XRD structure showing Cd-Cl bond lengths in a) Sb:DETACdCl hybrid,

b) Sb:PDACdCl hybrid, c) Sb:DABCdCl hybrid.

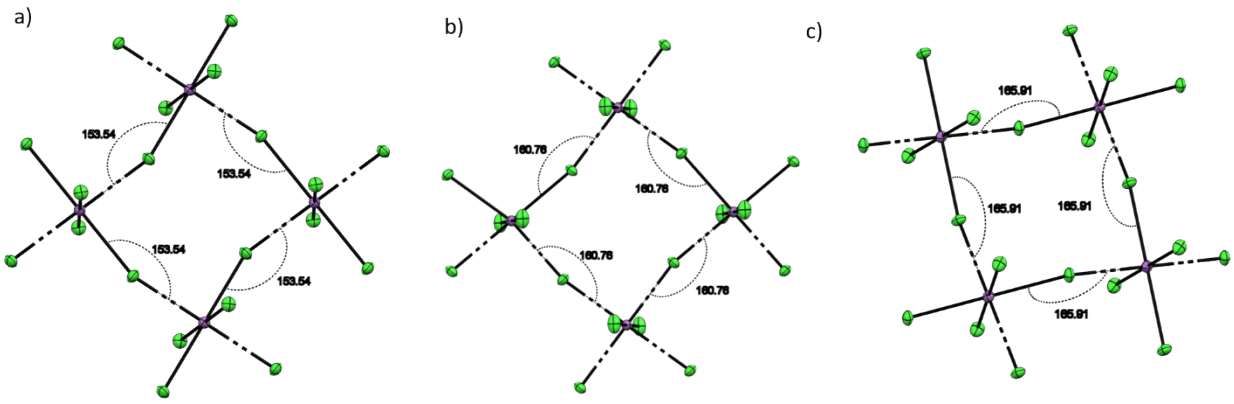


Figure S11: M-X-M bond angles in a) Sb:DETACdCl hybrid, b)Sb:PDACdCl hybrid, c) Sb:DABCdCl hybrid.

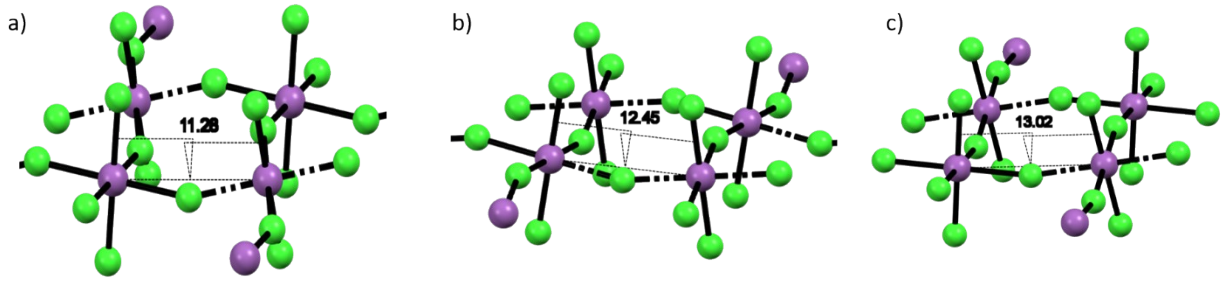


Figure S12: Dihedral angles in a) Sb: DETACdCl hybrid, b) Sb: PDACdCl hybrid, c) Sb:DAbCdCl hybrid.

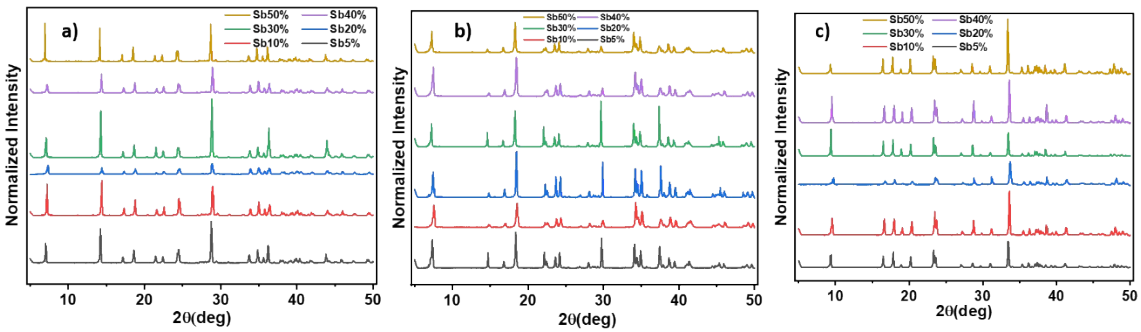


Figure S13: Comparison of PXRD patterns of different Sb feed percentages for a) Sb:DETACdCl hybrid, b) Sb:PDACdCl hybrid, c) Sb:DABCdCl hybrid.

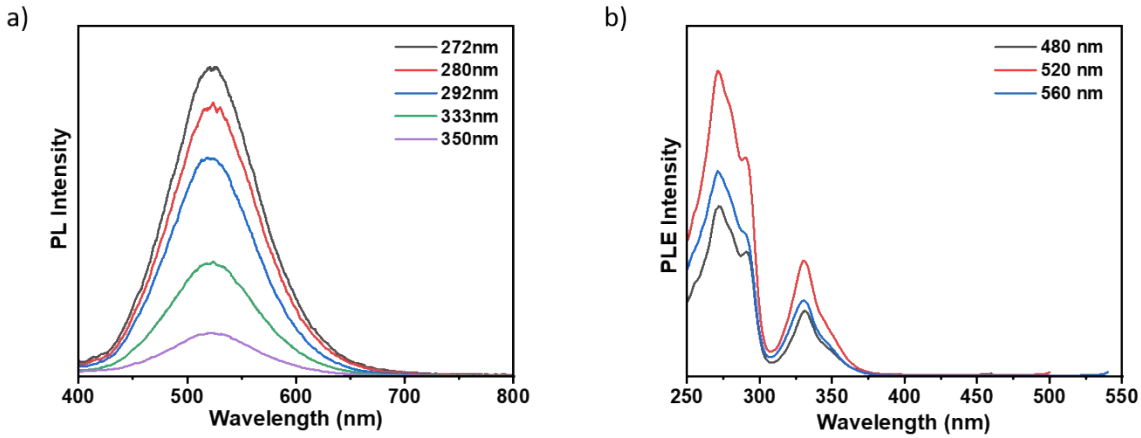


Figure S14: a) Excitation dependent PL, b) Emission dependent PLE of Sb:DETASbCl hybrid.

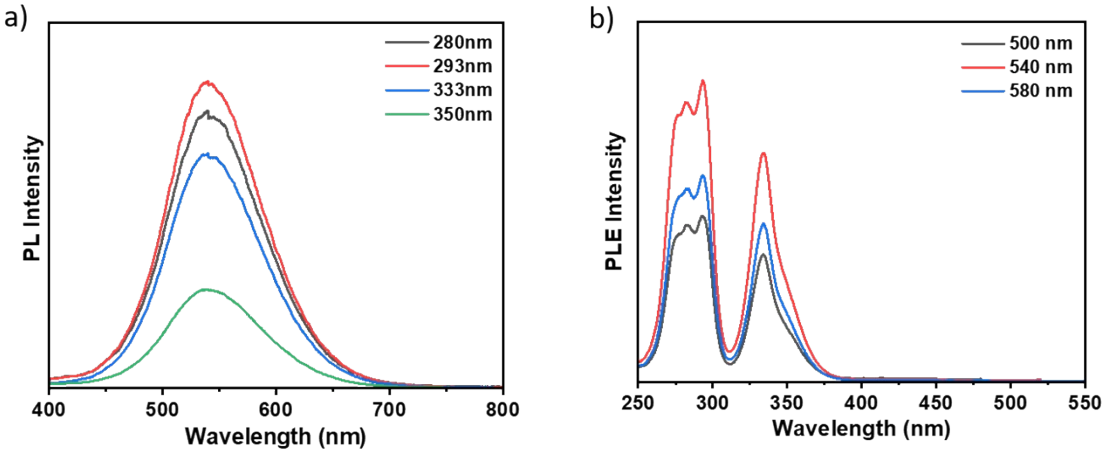


Figure S15: a) Excitation dependent PL, b) Emission dependent PLE of Sb:PDASbCl hybrid.

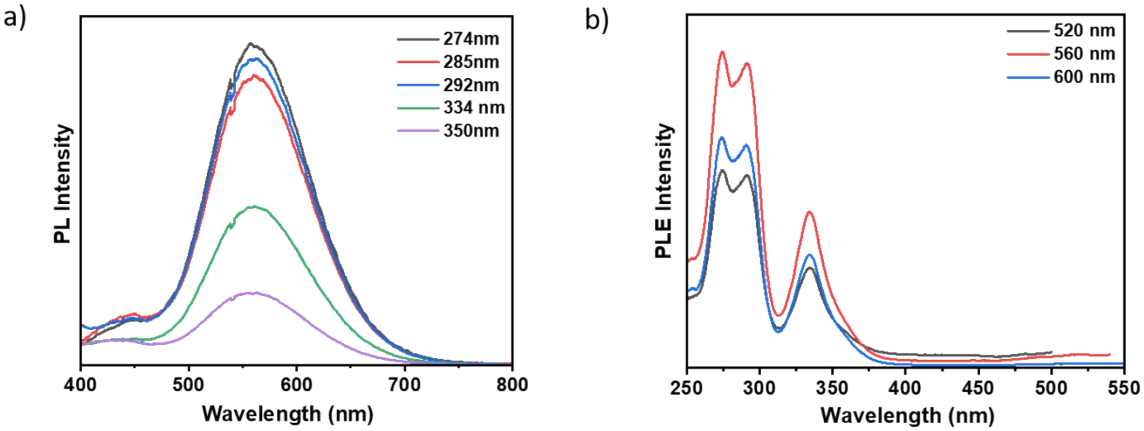


Figure S16: a) Excitation dependent PL, b) Emission dependent PLE of Sb:DABSbCl hybrid.

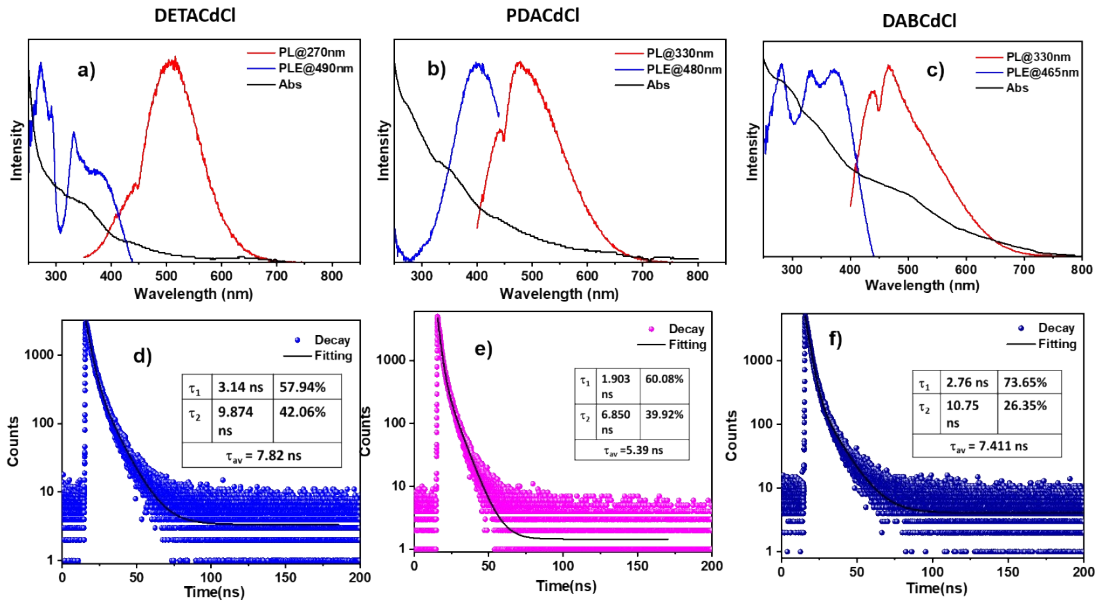


Figure S17: Optical characterization (PL, PLE and Absorption) of host hybrids- a) DETACdCl hybrid, b) PDACdCl hybrid, c) DABCdCl hybrid; Lifetime decay profiles of d) DETACdCl hybrid, e) PDACdCl hybrid, f) DABCdCl hybrid.

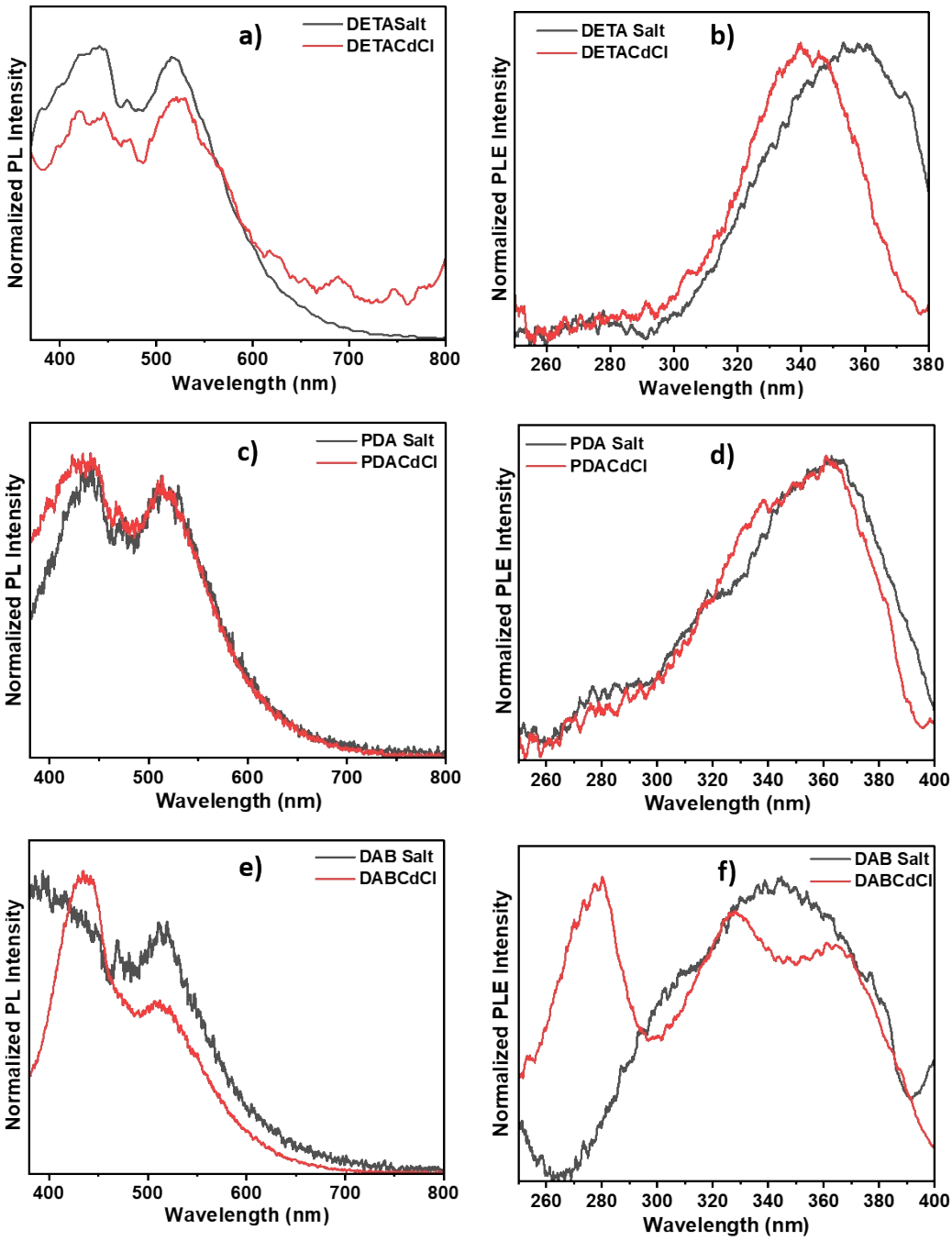


Figure S18: a) PL of DETA salt and DETACdCl excited at 275 nm b) PLE of DETA salt and DETACdCl (Emission: 420 nm) c) PL of PDA salt and PDACdCl excited at 275 nm d) PLE of PDA salt and PDACdCl (Emission: 435 nm) e) PL of DAB salt and DABCdCl excited at 275 nm f) PLE of DAB salt and DABCdCl (Emission: 425 nm)

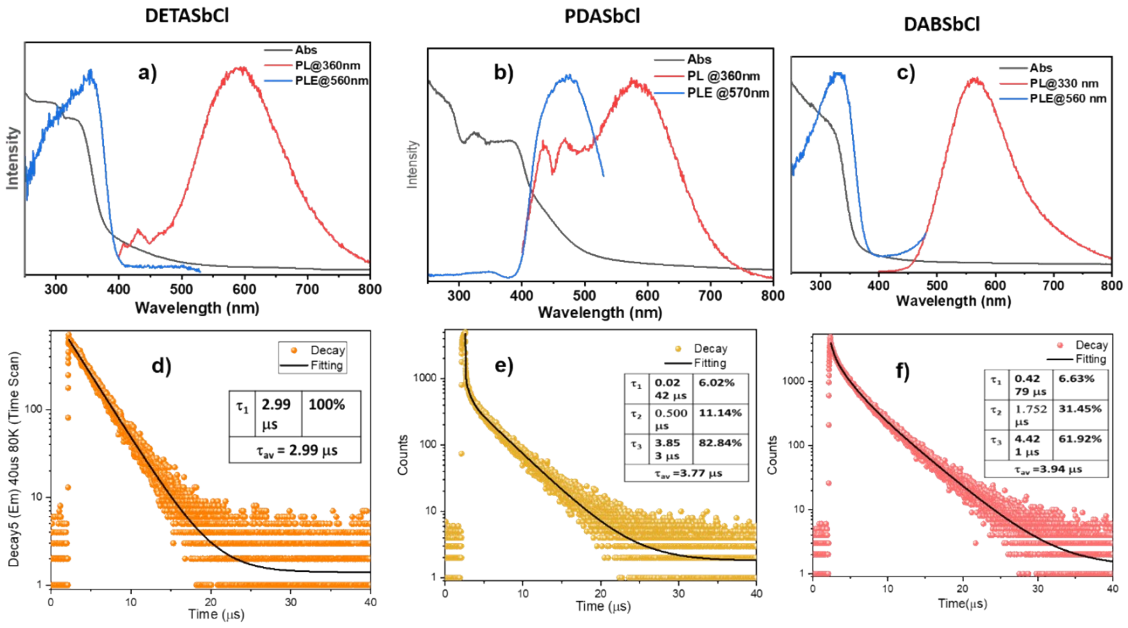


Figure S19: Optical characterization (PL, PLE and Absorption) of Control hybrids- a) DETASbCl hybrid, b) PDASbCl hybrid, c) DABSbCl hybrid; Lifetime decay profiles of d) DETASbCl hybrid, e) PDASbCl hybrid, f) DABSbCl hybrid.

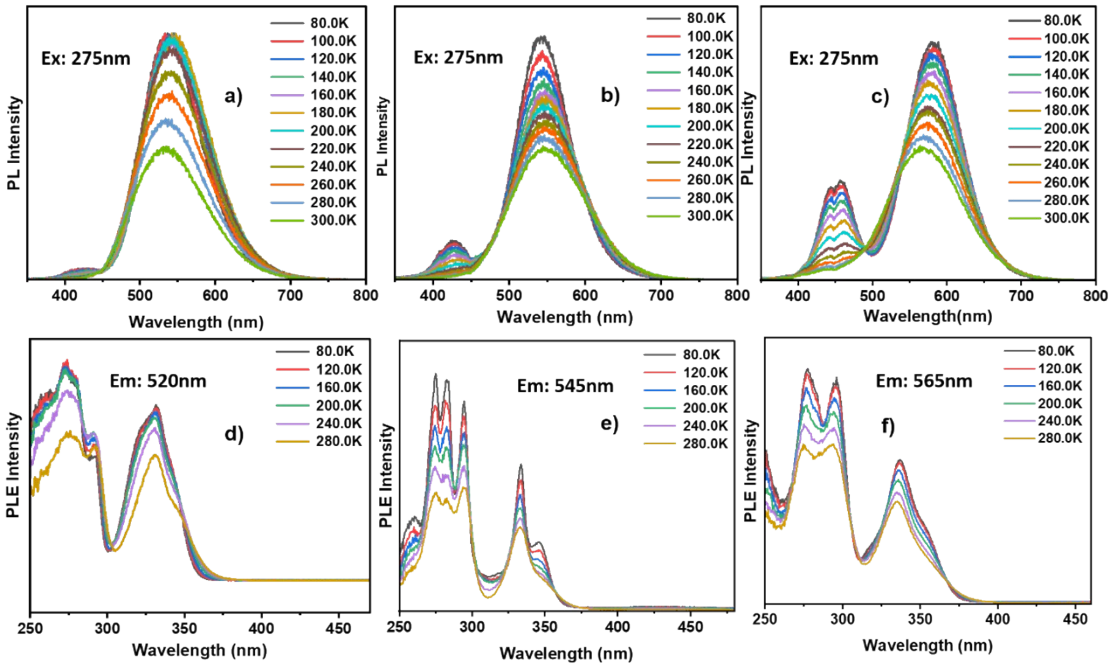


Figure S20: Temperature dependent PL of a) Sb: DETACdCl hybrid, b) Sb:PDACdCl hybrid, c) Sb:DABCdCl hybrid; Temperature dependent PLE of d) Sb:DETACdCl hybrid, e) Sb:PDACdCl hybrid, f) Sb:DAbCdCl hybrid.

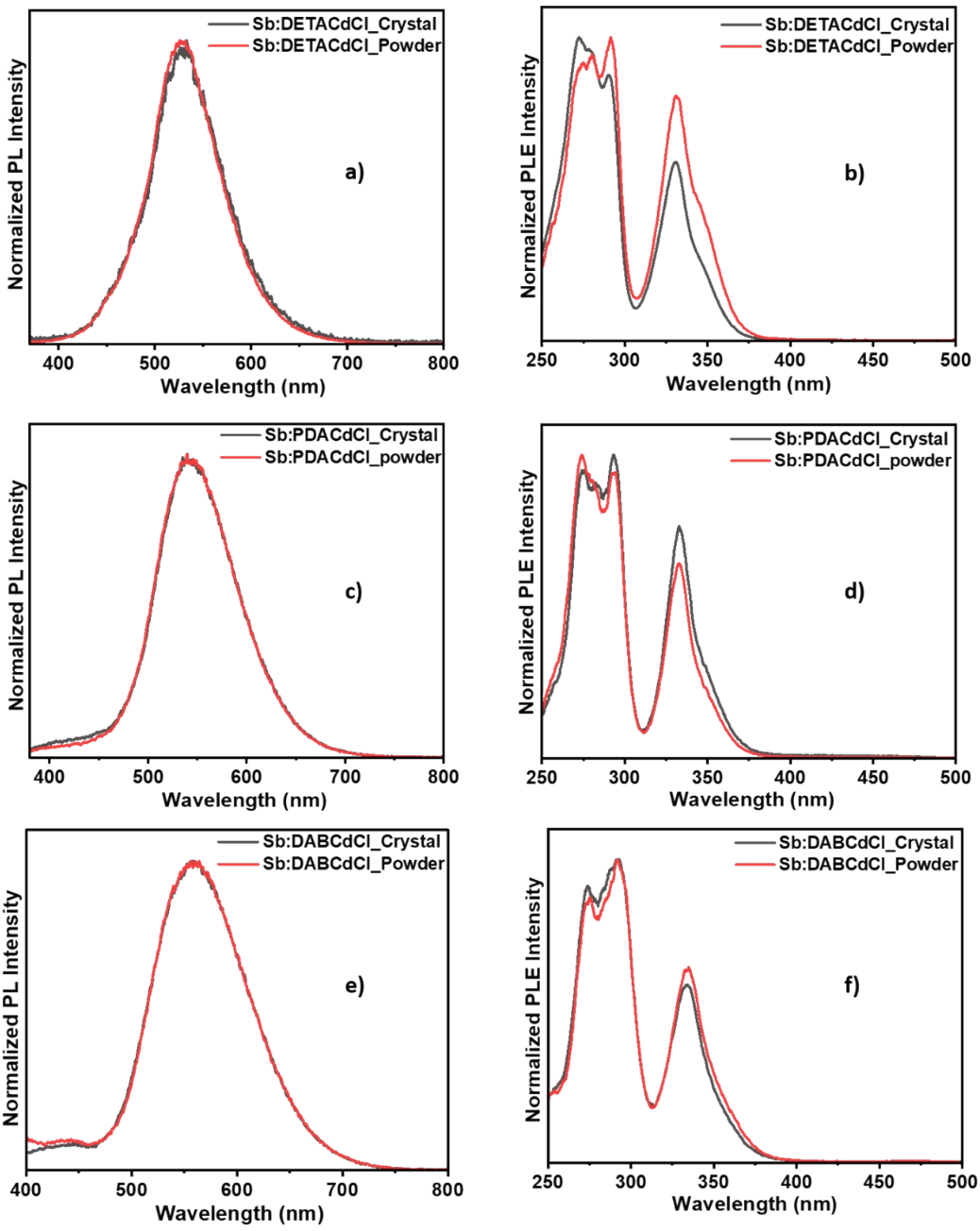


Figure S21: a) PL of Sb:DETACdCl crystal and powder excited at 330 nm b) PLE of Sb:DETACdCl crystal and powder (Emission: 520 nm) c) PL of Sb:PDACdCl crystal and powder excited at 330 nm d) PLE of Sb:PDACdCl crystal and powder (Emission: 540 nm) e) PL of Sb:DABCdCl crystal and powder excited at 330 nm f) PLE of Sb:DABCdCl crystal and powder (Emission: 560 nm).

Table S5: Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for DETACdCl

# DETACdCl

**Table S5-1 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for DETACdCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Cd1 | 10000 | 5000 | 5000 | 12.48(7) |
| Cl1 | 7006.4(5) | 4865.5(2) | 7011.0(5) | 17.38(9) |
| Cl2 | 10120.2(5) | 3969.3(2) | 4508.4(6) | 18.02(9) |
| Cl3 | 4350.2(7) | 2500 | 9655.0(7) | 19.18(11) |
| N1 | 5069(2) | 2500 | 5454(3) | 14.4(4) |
| N2 | 4562(2) | 4001.5(5) | 4828.8(17) | 17.5(3) |
| C1 | 4328(2) | 3007.7(5) | 4628(2) | 15.8(3) |
| C2 | 5249(2) | 3482.3(6) | 5601(2) | 17.2(3) |

**Table S5-2 Anisotropic Displacement Parameters (Å$^2$×10$^3$) for DETACdCl. The Anisotropic displacement factor exponent takes the form: -2π$^2$[h$^2$a*$^2$U$_{11}$+2hka*b*U$_{12}$+…].**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Cd1 | 11.87(10) | 13.82(9) | 11.74(10) | -0.29(5) | 0.29(5) | -0.09(5) |
| Cl1 | 14.86(16) | 22.47(17) | 14.81(17) | -1.67(13) | 4.52(13) | -2.36(13) |
| Cl2 | 20.55(19) | 14.43(17) | 19.09(18) | -0.40(14) | -0.17(13) | -0.93(12) |
| Cl3 | 16.9(3) | 23.5(2) | 17.1(2) | 0 | -0.02(19) | 0 |
| N1 | 15.5(9) | 13.7(9) | 14.0(8) | 0 | -1.0(6) | 0 |
| N2 | 19.8(6) | 14.2(6) | 18.5(6) | -0.3(5) | -1.1(5) | -0.3(5) |
| C1 | 16.7(7) | 13.6(7) | 17.0(6) | 1.0(5) | -2.3(6) | -0.2(5) |
| C2 | 20.5(7) | 14.4(7) | 16.8(7) | -0.1(6) | -3.4(6) | 0.5(5) |

**Table S5-3 Bond Lengths for DETACdCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Cd1 | Cl1 | 2.6226(3) | Cd1 | Cl2[1] | 2.5732(4) |
| Cd1 | Cl1[1] | 2.6226(3) | N1 | C1[4] | 1.4888(16) |
| Cd1 | Cl1[2] | 2.6249(3) | N1 | C1 | 1.4889(16) |
| Cd1 | Cl1[3] | 2.6249(3) | N2 | C2 | 1.485(2) |
| Cd1 | Cl2 | 2.5731(4) | C1 | C2 | 1.521(2) |

[1]2-X,1-Y,1-Z; [2]1/2+X,+Y,3/2-Z; [3]3/2-X,1-Y,-1/2+Z; [4]+X,1/2-Y,+Z

**Table S5-4 Bond Angles for DETACdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl1 | Cd1 | Cl1[1] | 180.000(15) | Cl2 | Cd1 | Cl1[2] | 88.310(12) |
| Cl1 | Cd1 | Cl1[2] | 88.693(5) | Cl2[1] | Cd1 | Cl1[1] | 88.807(11) |
| Cl1 | Cd1 | Cl1[3] | 91.307(5) | Cl2 | Cd1 | Cl1[3] | 91.690(12) |
| Cl1[3] | Cd1 | Cl1[2] | 180.0 | Cl2[1] | Cd1 | Cl1[3] | 88.310(12) |
| Cl1[1] | Cd1 | Cl1[2] | 91.307(5) | Cl2 | Cd1 | Cl2[1] | 179.999(15) |
| Cl1[1] | Cd1 | Cl1[3] | 88.693(5) | Cd1 | Cl1 | Cd1[4] | 153.475(14) |
| Cl2[1] | Cd1 | Cl1 | 91.193(11) | C1[5] | N1 | C1 | 114.83(16) |
| Cl2 | Cd1 | Cl1 | 88.807(11) | N1 | C1 | C2 | 107.88(12) |
| Cl2 | Cd1 | Cl1[1] | 91.194(11) | N2 | C2 | C1 | 110.26(12) |
| Cl2[1] | Cd1 | Cl1[2] | 91.690(12) | | | | |

[1]2-X,1-Y,1-Z; [2]1/2+X,+Y,3/2-Z; [3]3/2-X,1-Y,-1/2+Z; [4]3/2-X,1-Y,1/2+Z; [5]+X,1/2-Y,+Z

**Table S5-5 Torsion Angles for DETACdCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| N1 | C1 | C2 | N2 | 179.92(13) | C1[1] | N1 | C1 | C2 | 178.97(11) |

[1]+X,1/2-Y,+Z

**Table S5-6 Hydrogen Atom Coordinates (Å×10[4]) and Isotropic Displacement Parameters (Å[2]×10[3]) for DETACdCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H1A | 6327.9 | 2499.99 | 5334.81 | 17 |
| H1B | 4804.68 | 2500 | 6681.86 | 17 |
| H2A | 4836.48 | 4017.58 | 3604.43 | 21 |
| H2B | 5119.46 | 4282.3 | 5423.66 | 21 |
| H2C | 3310.02 | 4022.91 | 4984.66 | 21 |
| H1C | 2964 | 3025.05 | 4790.04 | 19 |
| H1D | 4607.97 | 3018.77 | 3291.42 | 19 |
| H2D | 4971.19 | 3466.6 | 6937.77 | 21 |
| H2E | 6613.57 | 3459.75 | 5443.93 | 21 |

Table S6: Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for Sb:DETACdCl

# Sb:DETACdCl

**Table S6-1 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:DETACdCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Cd1 | 10000 | 5000 | 5000 | 13.30(7) |
| Cl1 | 7009.6(4) | 4864.7(2) | 7013.7(4) | 18.06(9) |
| Cl2 | 9879.4(4) | 6031.1(2) | 5492.4(6) | 18.72(9) |
| Cl3 | 651.1(7) | 7500 | 4654.5(7) | 19.90(11) |
| N1 | 4930(2) | 7500 | 4543(3) | 15.2(4) |
| N2 | 5436(2) | 5997.6(5) | 5170.9(16) | 18.0(3) |
| C2 | 5674(2) | 6992.6(5) | 5373.0(19) | 16.6(3) |
| C1 | 4749.0(19) | 6516.5(6) | 4399(2) | 17.8(3) |
| Sb1 | 10000 | 5000 | 5000 | 13.30(7) |

**Table S6-2 Anisotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:DETACdCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Cd1 | 12.58(10) | 14.66(10) | 12.66(10) | -0.33(4) | 0.36(4) | -0.01(4) |
| Cl1 | 15.47(16) | 23.29(17) | 15.42(16) | -1.76(13) | 4.67(12) | -2.48(13) |
| Cl2 | 21.05(18) | 15.18(17) | 19.93(18) | -0.29(15) | -0.20(12) | -0.81(11) |
| Cl3 | 17.4(3) | 24.9(2) | 17.5(2) | 0 | -0.17(18) | 0 |
| N1 | 16.5(9) | 14.1(9) | 14.9(8) | 0 | -0.7(6) | 0 |
| N2 | 20.7(6) | 15.0(6) | 18.3(6) | -0.3(4) | -0.8(5) | -0.1(5) |
| C2 | 17.5(7) | 14.5(7) | 17.7(6) | 1.1(5) | -2.8(5) | 0.2(5) |
| C1 | 21.1(7) | 15.5(7) | 17.0(7) | -0.2(6) | -3.3(5) | -0.5(5) |
| Sb1 | 12.58(10) | 14.66(10) | 12.66(10) | -0.33(4) | 0.36(4) | -0.01(4) |

**Table S6-3 Bond Lengths for Sb:DETACdCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Cd1 | Cl1 | 2.6216(3) | Cl1 | Sb1 | 2.6216(3) |
| Cd1 | Cl1$^1$ | 2.6246(3) | Cl2 | Sb1 | 2.5713(4) |
| Cd1 | Cl1$^2$ | 2.6216(3) | N1 | C2$^4$ | 1.4890(16) |
| Cd1 | Cl1$^3$ | 2.6246(3) | N1 | C2 | 1.4890(16) |
| Cd1 | Cl2$^2$ | 2.5713(4) | N2 | C1 | 1.4826(19) |
| Cd1 | Cl2 | 2.5713(4) | C2 | C1 | 1.5241(19) |

[1]3/2-X,1-Y,-1/2+Z; [2]2-X,1-Y,1-Z; [3]1/2+X,+Y,3/2-Z; [4]+X,3/2-Y,+Z

**Table S6-4 Bond Angles for Sb:DETACdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl1 | Cd1 | Cl1[1] | 180.0 | N1 | C2 | C1 | 107.71(12) |
| Cl1 | Cd1 | Cl1[2] | 91.328(5) | N2 | C1 | C2 | 110.22(12) |
| Cl1[1] | Cd1 | Cl1[2] | 88.672(5) | Cl1 | Sb1 | Cl1[1] | 180.0 |
| Cl1[2] | Cd1 | Cl1[3] | 180.000(15) | Cl1[2] | Sb1 | Cl1[3] | 180.000(15) |
| Cl1 | Cd1 | Cl1[3] | 88.672(5) | Cl1[1] | Sb1 | Cl1[2] | 88.672(5) |
| Cl1[1] | Cd1 | Cl1[3] | 91.328(5) | Cl1 | Sb1 | Cl1[3] | 88.672(5) |
| Cl2 | Cd1 | Cl1[1] | 88.787(11) | Cl1 | Sb1 | Cl1[2] | 91.328(5) |
| Cl2 | Cd1 | Cl1[2] | 88.275(11) | Cl1[1] | Sb1 | Cl1[3] | 91.328(5) |
| Cl2[1] | Cd1 | Cl1 | 88.788(11) | Cl2[1] | Sb1 | Cl1 | 88.788(11) |
| Cl2 | Cd1 | Cl1 | 91.211(11) | Cl2 | Sb1 | Cl1[2] | 88.275(11) |
| Cl2 | Cd1 | Cl1[3] | 91.725(11) | Cl2 | Sb1 | Cl1 | 91.211(11) |
| Cl2[1] | Cd1 | Cl1[1] | 91.213(11) | Cl2[1] | Sb1 | Cl1[1] | 91.213(11) |
| Cl2[1] | Cd1 | Cl1[2] | 91.724(11) | Cl2 | Sb1 | Cl1[1] | 88.787(11) |
| Cl2[1] | Cd1 | Cl1[3] | 88.275(11) | Cl2[1] | Sb1 | Cl1[2] | 91.724(11) |
| Cl2 | Cd1 | Cl2[1] | 179.999(14) | Cl2[1] | Sb1 | Cl1[3] | 88.275(11) |
| Cd1 | Cl1 | Cd1[4] | 153.540(14) | Cl2 | Sb1 | Cl1[3] | 91.725(11) |
| C2[5] | N1 | C2 | 114.49(16) | Cl2 | Sb1 | Cl2[1] | 179.999(14) |

[1]2-X,1-Y,1-Z; [2]3/2-X,1-Y,-1/2+Z; [3]1/2+X,+Y,3/2-Z; [4]3/2-X,1-Y,1/2+Z; [5]+X,3/2-Y,+Z

**Table S6-5 Torsion Angles for Sb:DETACdCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| N1 | C2 | C1 | N2 | 179.93(12) | C2[1] | N1 | C2 | C1 | 178.91(10) |

[1]+X,3/2-Y,+Z

**Table S6-6 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:DETACdCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H1A | 5195.22 | 7500 | 3315.51 | 18 |
| H1B | 3671.01 | 7500 | 4662.67 | 18 |
| H2A | 4891.02 | 5716.38 | 4565.79 | 22 |
| H2B | 5148.16 | 5979.61 | 6391.76 | 22 |

**Table S6-6 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:DETACdCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H2C | 6690.85 | 5978.36 | 5028.92 | 22 |
| H2D | 5395.48 | 6982.11 | 6709.94 | 20 |
| H2E | 7038.86 | 6975.38 | 5209.82 | 20 |
| H1C | 3384.81 | 6539.23 | 4557.65 | 21 |
| H1D | 5026.08 | 6532.04 | 3062.12 | 21 |

**Table S6-7 Atomic Occupancy for Sb:DETACdCl.**

| Atom | *Occupancy* | Atom | *Occupancy* | Atom | *Occupancy* |
|---|---|---|---|---|---|
| Sb1 | 0.0003 | | | | |

Table S7: Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for DETASbCl

# DETASbCl

**Table S7-1 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for DETASbCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Sb01 | 7272.0(2) | 7512.4(2) | 7633.9(2) | 14.18(4) |
| Cl02 | 10691.5(5) | 6155.9(4) | 7926.9(4) | 19.06(8) |
| Cl03 | 8582.9(5) | 8919.8(4) | 4856.1(4) | 18.75(8) |
| Cl04 | 4073.1(5) | 8512.8(4) | 7603.5(4) | 21.87(9) |
| Cl05 | 7365.9(6) | 9545.6(4) | 8584.9(4) | 22.07(9) |
| Cl06 | 5873.1(5) | 6488.2(4) | 10154.2(4) | 19.60(8) |
| Cl07 | 7747.0(6) | 4666.8(4) | 6507.7(4) | 22.91(9) |
| N008 | 7969.3(18) | 6893.0(14) | 3161.7(14) | 15.0(3) |
| N009 | 2808.5(18) | 7794.2(14) | 4835.3(15) | 19.5(3) |
| N00A | 11716.3(19) | 6671.7(15) | 556.3(15) | 20.5(3) |
| C00B | 6062(2) | 6666.5(16) | 3806.4(17) | 16.2(3) |
| C00C | 4649(2) | 8053.3(17) | 3937.9(18) | 17.9(3) |
| C00D | 8374(2) | 7677.4(17) | 1692.5(17) | 18.3(3) |
| C00E | 10282(2) | 7954.8(17) | 1053.2(18) | 20.6(3) |

**Table S7-2 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for DETASbCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Sb01 | 14.89(6) | 13.75(6) | 15.41(6) | -0.82(4) | -6.50(4) | -4.13(4) |
| Cl02 | 17.91(19) | 18.96(18) | 21.05(19) | -4.32(15) | -7.83(16) | -2.20(15) |
| Cl03 | 20.43(19) | 17.30(18) | 19.50(18) | -3.97(15) | -6.88(15) | -4.42(15) |
| Cl04 | 16.74(19) | 24.5(2) | 24.8(2) | 1.81(16) | -9.96(16) | -4.05(15) |
| Cl05 | 29.7(2) | 17.41(19) | 23.6(2) | -3.14(15) | -12.26(17) | -7.17(16) |
| Cl06 | 19.43(19) | 23.77(19) | 18.31(18) | 2.10(15) | -8.08(15) | -9.42(15) |
| Cl07 | 29.6(2) | 21.3(2) | 22.1(2) | -0.72(16) | -13.20(17) | -7.13(16) |
| N008 | 14.7(6) | 15.2(6) | 16.2(6) | -1.3(5) | -6.9(5) | -2.7(5) |
| N009 | 15.5(7) | 16.7(7) | 24.7(7) | -3.0(6) | -5.5(6) | -2.4(5) |
| N00A | 14.3(7) | 27.5(8) | 18.5(7) | -1.7(6) | -5.2(6) | -3.9(6) |
| C00B | 13.5(7) | 14.9(7) | 18.7(8) | 0.1(6) | -4.7(6) | -3.4(6) |
| C00C | 12.4(7) | 16.2(8) | 22.1(8) | -0.6(6) | -3.6(6) | -2.5(6) |
| C00D | 15.5(8) | 19.7(8) | 16.7(8) | 2.0(6) | -5.1(6) | -2.5(6) |
| C00E | 20.1(8) | 19.3(8) | 22.4(8) | -2.4(7) | -4.5(7) | -8.3(7) |

**Table S7-3 Bond Lengths for DETASbCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Sb01 | Cl02 | 2.8306(4) | N008 | C00D | 1.494(2) |
| Sb01 | Cl03 | 2.8230(4) | N009 | C00C | 1.488(2) |
| Sb01 | Cl04 | 2.5201(4) | N00A | C00E | 1.482(2) |
| Sb01 | Cl05 | 2.4100(4) | C00B | C00C | 1.521(2) |
| Sb01 | Cl06 | 2.4904(4) | C00D | C00E | 1.518(2) |
| N008 | C00B | 1.4981(19) | | | |

**Table S7-4 Bond Angles for DETASbCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl03 | Sb01 | Cl02 | 98.139(12) | Cl06 | Sb01 | Cl03 | 174.276(13) |
| Cl04 | Sb01 | Cl02 | 173.306(12) | Cl06 | Sb01 | Cl04 | 87.594(13) |
| Cl04 | Sb01 | Cl03 | 88.203(12) | C00D | N008 | C00B | 113.49(12) |
| Cl05 | Sb01 | Cl02 | 88.714(13) | N008 | C00B | C00C | 111.24(13) |
| Cl05 | Sb01 | Cl03 | 86.090(13) | N009 | C00C | C00B | 108.99(13) |
| Cl05 | Sb01 | Cl04 | 93.878(15) | N008 | C00D | C00E | 113.84(13) |
| Cl05 | Sb01 | Cl06 | 90.320(14) | N00A | C00E | C00D | 112.38(13) |
| Cl06 | Sb01 | Cl02 | 86.220(13) | | | | |

**Table S7-5 Torsion Angles for DETASbCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| N008 | C00B | C00C | N009 | 169.15(13) | C00B | N008 | C00D | C00E | 178.04(13) |
| N008 | C00D | C00E | N00A | 73.19(18) | C00D | N008 | C00B | C00C | -58.02(17) |

**Table S7-6 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for DETASbCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H00A | 8143.48 | 7375.77 | 3774.07 | 18 |
| H00B | 8776.11 | 6044.38 | 3090.74 | 18 |
| H00C | 2584.56 | 7129.29 | 4448.14 | 23 |
| H00D | 1943.2 | 8603.95 | 4845.35 | 23 |
| H00E | 2772.09 | 7491 | 5761.13 | 23 |
| H00F | 11410.32 | 6244.74 | -23.46 | 25 |
| H00G | 11812.04 | 6074.83 | 1342.31 | 25 |
| H00H | 12806.96 | 6907.97 | 44.98 | 25 |
| H00I | 5877.42 | 6158.92 | 4782.27 | 19 |
| H00J | 5892.01 | 6078.87 | 3184.94 | 19 |
| H00K | 4947.49 | 8712.6 | 4401.17 | 21 |
| H00L | 4658.33 | 8477.83 | 2958.76 | 21 |
| H00M | 7467.25 | 8585.88 | 1764.09 | 22 |
| H00N | 8224.49 | 7135.24 | 1019.04 | 22 |
| H00O | 10344.63 | 8671.72 | 219.35 | 25 |
| H00P | 10523.12 | 8336.08 | 1797.09 | 25 |

**Table S8:** Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for PDACdCl

# PDACdCl

**Table S8-1 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for PDACdCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Cd1 | 5000 | 5000 | 5000 | 12.63(16) |
| Cl1 | 5000 | 6053.2(3) | 4467.5(12) | 25.3(2) |
| Cl2 | 7500 | 5110(20) | 7500 | 50(20) |
| N1 | 5000 | 5945.5(16) | 205(4) | 44.7(13) |
| C3 | 5000 | 7500 | -659(8) | 45.6(16) |

**Table S8-1 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for PDACdCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| C2 | 5000 | 6974.2(19) | 206(6) | 70(3) |
| C1 | 5000 | 6459.7(16) | -666(5) | 51.6(13) |
| Cl3 | 7783(7) | 5140.1(19) | 7211(6) | 15.5(10) |

**Table S8-2 Anisotropic Displacement Parameters (Å$^2$×10$^3$) for PDACdCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Cd1 | 11.4(2) | 15.2(2) | 11.3(2) | 0.49(9) | 0 | 0 |
| Cl1 | 42.6(5) | 15.8(4) | 17.7(4) | 0.6(3) | 0 | 0 |
| Cl2 | 50(30) | 60(30) | 50(40) | 0 | -40(30) | 0 |
| N1 | 100(4) | 13.5(17) | 20.5(15) | 0.3(11) | 0 | 0 |
| C3 | 93(5) | 19(3) | 25(3) | 0 | 0 | 0 |
| C2 | 168(9) | 15(2) | 26(2) | -1.2(15) | 0 | 0 |
| C1 | 117(4) | 17.0(17) | 20.5(18) | 1.6(15) | 0 | 0 |
| Cl3 | 14.7(17) | 19.3(12) | 12.5(16) | -0.1(6) | -3.1(12) | -0.5(6) |

**Table S8-3 Bond Lengths for PDACdCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Cd1 | Cl1[1] | 2.5657(8) | Cd1 | Cl3 | 2.635(5) |
| Cd1 | Cl1 | 2.5658(8) | Cd1 | Cl3[6] | 2.653(5) |
| Cd1 | Cl2[2] | 2.618(5) | Cd1 | Cl3[7] | 2.635(5) |
| Cd1 | Cl2[1] | 2.618(5) | N1 | C1 | 1.397(5) |
| Cd1 | Cl2 | 2.618(6) | C3 | C2 | 1.420(5) |
| Cd1 | Cl2[3] | 2.618(6) | C3 | C2[8] | 1.420(5) |
| Cd1 | Cl3[1] | 2.635(5) | C2 | C1 | 1.398(6) |
| Cd1 | Cl3[4] | 2.653(5) | Cl3 | Cl3[4] | 0.596(10) |
| Cd1 | Cl3[5] | 2.635(5) | | | |

[1]1-X,1-Y,1-Z; [2]-1/2+X,+Y,3/2-Z; [3]3/2-X,1-Y,-1/2+Z; [4]3/2-X,+Y,3/2-Z; [5]+X,1-Y,1-Z; [6]-1/2+X,1-Y,-1/2+Z; [7]1-X,+Y,+Z; [8]1-X,3/2-Y,+Z

**Table S8-4 Bond Angles for PDACdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl1[1] | Cd1 | Cl1 | 180.0 | Cl2[3] | Cd1 | Cl3[5] | 98.38(16) |

**Table S8-4 Bond Angles for PDACdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl1 | Cd1 | Cl2 | 90.7(12) | Cl2[2] | Cd1 | Cl3[5] | 81.62(16) |
| Cl1[1] | Cd1 | Cl2[1] | 90.7(12) | Cl2[3] | Cd1 | Cl3[6] | 6.7(4) |
| Cl1[1] | Cd1 | Cl2[2] | 89.3(12) | Cl2[3] | Cd1 | Cl3[4] | 81.62(16) |
| Cl1 | Cd1 | Cl2[2] | 90.7(12) | Cl2[2] | Cd1 | Cl3[7] | 6.7(4) |
| Cl1[1] | Cd1 | Cl2[3] | 90.7(12) | Cl2[1] | Cd1 | Cl3[5] | 173.3(3) |
| Cl1[1] | Cd1 | Cl2 | 89.3(12) | Cl2[2] | Cd1 | Cl3[6] | 173.3(4) |
| Cl1 | Cd1 | Cl2[1] | 89.3(12) | Cl2[1] | Cd1 | Cl3[4] | 6.7(3) |
| Cl1 | Cd1 | Cl2[3] | 89.3(12) | Cl2 | Cd1 | Cl3[6] | 85.50(19) |
| Cl1 | Cd1 | Cl3[4] | 90.26(10) | Cl2[2] | Cd1 | Cl3[4] | 98.38(16) |
| Cl1 | Cd1 | Cl3[5] | 89.74(10) | Cl2[2] | Cd1 | Cl3[1] | 85.50(19) |
| Cl1 | Cd1 | Cl3[6] | 91.70(10) | Cl2 | Cd1 | Cl3[5] | 6.7(3) |
| Cl1[1] | Cd1 | Cl3[5] | 90.26(10) | Cl2[1] | Cd1 | Cl3[6] | 94.50(19) |
| Cl1[1] | Cd1 | Cl3[6] | 88.30(10) | Cl2[1] | Cd1 | Cl3[7] | 85.50(19) |
| Cl1 | Cd1 | Cl3[7] | 88.30(10) | Cl2[3] | Cd1 | Cl3[7] | 173.3(4) |
| Cl1[1] | Cd1 | Cl3[7] | 91.70(10) | Cl2[1] | Cd1 | Cl3[1] | 6.7(4) |
| Cl1 | Cd1 | Cl3 | 88.29(10) | Cl2[3] | Cd1 | Cl3[1] | 94.50(19) |
| Cl1[1] | Cd1 | Cl3 | 91.71(10) | Cl2 | Cd1 | Cl3[4] | 173.3(3) |
| Cl1 | Cd1 | Cl3[1] | 91.70(10) | Cl3[6] | Cd1 | Cl3 | 79.3(2) |
| Cl1[1] | Cd1 | Cl3[1] | 88.30(10) | Cl3[7] | Cd1 | Cl3 | 100.7(2) |
| Cl1[1] | Cd1 | Cl3[4] | 89.74(10) | Cl3[1] | Cd1 | Cl3[7] | 79.3(2) |
| Cl2 | Cd1 | Cl2[2] | 88.2(2) | Cl3[1] | Cd1 | Cl3 | 180.0 |
| Cl2 | Cd1 | Cl2[1] | 180.0 | Cd1 | Cl2 | Cd1[8] | 169(2) |
| Cl2 | Cd1 | Cl2[3] | 91.8(2) | C2 | C3 | C2[9] | 126.0(5) |
| Cl2[1] | Cd1 | Cl2[3] | 88.2(2) | C1 | C2 | C3 | 125.3(4) |
| Cl2[2] | Cd1 | Cl2[3] | 180.0 | N1 | C1 | C2 | 124.7(3) |
| Cl2[1] | Cd1 | Cl2[2] | 91.8(2) | Cd1 | Cl3 | Cd1[8] | 160.4(2) |
| Cl2 | Cd1 | Cl3[7] | 94.50(19) | Cl3[5] | Cl3 | Cd1 | 85.3(10) |
| Cl2 | Cd1 | Cl3[1] | 173.3(4) | | | | |

[1]1-X,1-Y,1-Z; [2]-1/2+X,+Y,3/2-Z; [3]3/2-X,1-Y,-1/2+Z; [4]-1/2+X,1-Y,-1/2+Z; [5]3/2-X,+Y,3/2-Z; [6]+X,1-Y,1-Z; [7]1-X,+Y,+Z; [8]3/2-X,1-Y,1/2+Z; [9]1-X,3/2-Y,+Z

**Table S8-5 Torsion Angles for PDACdCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| C3 | C2 | C1 | N1 | 180.000(2) | C2[1] | C3 | C2 | C1 | 180.000(3) |

[1]1-X,3/2-Y,+Z

**Table S8-6 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for PDACdCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H1A | 3832.17 | 5813.14 | 265.73 | 67 |
| H1B | 5455.9 | 5986.49 | 1334.37 | 67 |
| H1C | 5711.94 | 5702.13 | -418.58 | 67 |
| H3B | 3913.77 | 7500 | -1457.2 | 55 |
| H3A | 6086.23 | 7500 | -1457.2 | 55 |
| H2B | 6086.78 | 6972.71 | 1003.62 | 83 |
| H2A | 3913.22 | 6972.71 | 1003.61 | 83 |
| H1D | 3912.68 | 6459.74 | -1462.42 | 62 |
| H1E | 6087.32 | 6459.74 | -1462.42 | 62 |

**Table S8-7 Atomic Occupancy for PDACdCl.**

| Atom | *Occupancy* | Atom | *Occupancy* | Atom | *Occupancy* |
|---|---|---|---|---|---|
| Cl2 | 0.1 | H1A | 0.5 | H1B | 0.5 |
| H1C | 0.5 | H3B | 0.5 | H3A | 0.5 |
| H2B | 0.5 | H2A | 0.5 | H1D | 0.5 |
| H1E | 0.5 | Cl3 | 0.45 | | |

Table S9: Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for Sb:PDACdCl

# Sb:PDACdCl

**Table S9-1 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:PDACdCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Cd01 | 5000 | 5000 | 0 | 13.0(2) |

**Table S9-1 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for Sb:PDACdCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Cl02 | 5000 | 6053.8(5) | 531.1(17) | 25.3(3) |
| N004 | 5000 | 5946(2) | 4803(6) | 46.3(19) |
| C005 | 5000 | 7500 | 5644(12) | 48(3) |
| C006 | 5000 | 6975(3) | 4787(9) | 70(4) |
| C007 | 5000 | 6460(2) | 5661(8) | 54(2) |
| Sb01 | 5000 | 5000 | 0 | 13.0(2) |
| Cl1 | 7781(3) | 4863.0(6) | 2217(3) | 18.2(5) |

**Table S9-2 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for Sb:PDACdCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Cd01 | 12.4(3) | 13.5(3) | 13.2(3) | -0.49(13) | 0 | 0 |
| Cl02 | 41.8(7) | 14.2(5) | 19.8(6) | -0.9(4) | 0 | 0 |
| N004 | 103(6) | 14(3) | 22(2) | 2.9(17) | 0 | 0 |
| C005 | 100(8) | 16(4) | 28(4) | 0 | 0 | 0 |
| C006 | 165(13) | 16(4) | 29(3) | -2(2) | 0 | 0 |
| C007 | 125(7) | 15(2) | 22(3) | 0(2) | 0 | 0 |
| Sb01 | 12.4(3) | 13.5(3) | 13.2(3) | -0.49(13) | 0 | 0 |
| Cl1 | 15.4(16) | 21.9(6) | 17.3(16) | 0.3(6) | -5.8(9) | 0.7(6) |

**Table S9-3 Bond Lengths for Sb:PDACdCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Cd01 | $Cl02^1$ | 2.5669(11) | C005 | C006 | 1.415(7) |
| Cd01 | Cl02 | 2.5669(11) | C005 | $C006^8$ | 1.415(7) |
| Cd01 | $Cl1^2$ | 2.650(3) | C006 | C007 | 1.400(9) |
| Cd01 | $Cl1^3$ | 2.650(3) | Sb01 | $Cl1^1$ | 2.636(3) |
| Cd01 | $Cl1^1$ | 2.636(3) | Sb01 | $Cl1^7$ | 2.636(3) |
| Cd01 | $Cl1^4$ | 2.650(3) | Sb01 | $Cl1^6$ | 2.650(3) |
| Cd01 | $Cl1^5$ | 2.636(3) | Sb01 | $Cl1^2$ | 2.650(3) |
| Cd01 | $Cl1^6$ | 2.650(3) | Sb01 | Cl1 | 2.636(3) |
| Cd01 | Cl1 | 2.636(3) | Sb01 | $Cl1^4$ | 2.650(3) |
| Cd01 | $Cl1^7$ | 2.636(3) | Sb01 | $Cl1^5$ | 2.636(3) |
| Cl02 | Sb01 | 2.5669(11) | Sb01 | $Cl1^3$ | 2.650(3) |
| N004 | C007 | 1.394(8) | Cl1 | $Cl1^2$ | 0.588(3) |

[1]1-X,1-Y,-Z; [2]3/2-X,+Y,1/2-Z; [3]-1/2+X,+Y,1/2-Z; [4]3/2-X,1-Y,-1/2+Z; [5]1-X,+Y,+Z; [6]-1/2+X,1-Y,-1/2+Z; [7]+X,1-Y,-Z; [8]1-X,3/2-Y,+Z

**Table S9-4 Bond Angles for Sb:PDACdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl02[1] | Cd01 | Cl02 | 180.0 | Cl02 | Sb01 | Cl1[6] | 91.55(4) |
| Cl02[1] | Cd01 | Cl1[2] | 90.14(4) | Cl02 | Sb01 | Cl1[7] | 88.45(4) |
| Cl02 | Cd01 | Cl1[3] | 90.14(4) | Cl02[1] | Sb01 | Cl1[5] | 90.14(4) |
| Cl02 | Cd01 | Cl1 | 91.55(4) | Cl02[1] | Sb01 | Cl1[7] | 91.55(4) |
| Cl02 | Cd01 | Cl1[4] | 90.14(4) | Cl02 | Sb01 | Cl1[2] | 89.86(4) |
| Cl02[1] | Cd01 | Cl1[3] | 89.86(4) | Cl02 | Sb01 | Cl1[5] | 89.86(4) |
| Cl02 | Cd01 | Cl1[5] | 89.86(4) | Cl02 | Sb01 | Cl1[3] | 90.14(4) |
| Cl02[1] | Cd01 | Cl1[1] | 91.55(4) | Cl02[1] | Sb01 | Cl1[4] | 89.86(4) |
| Cl02 | Cd01 | Cl1[6] | 91.55(4) | Cl02 | Sb01 | Cl1[1] | 88.45(4) |
| Cl02 | Cd01 | Cl1[2] | 89.86(4) | Cl02[1] | Sb01 | Cl1[3] | 89.86(4) |
| Cl02[1] | Cd01 | Cl1[7] | 91.55(4) | Cl02 | Sb01 | Cl1[4] | 90.14(4) |
| Cl02 | Cd01 | Cl1[1] | 88.45(4) | Cl02[1] | Sb01 | Cl1 | 88.45(4) |
| Cl02 | Cd01 | Cl1[7] | 88.45(4) | Cl02[1] | Sb01 | Cl1[6] | 88.45(4) |
| Cl02[1] | Cd01 | Cl1[5] | 90.14(4) | Cl02[1] | Sb01 | Cl1[2] | 90.14(4) |
| Cl02[1] | Cd01 | Cl1 | 88.45(4) | Cl02 | Sb01 | Cl1 | 91.55(4) |
| Cl02[1] | Cd01 | Cl1[6] | 88.45(4) | Cl02[1] | Sb01 | Cl1[1] | 91.55(4) |
| Cl02[1] | Cd01 | Cl1[4] | 89.86(4) | Cl1[1] | Sb01 | Cl1[2] | 12.77(6) |
| Cl1[6] | Cd01 | Cl1[7] | 180.00(7) | Cl1 | Sb01 | Cl1[4] | 12.77(6) |
| Cl1[7] | Cd01 | Cl1[4] | 92.079(16) | Cl1[1] | Sb01 | Cl1[3] | 92.079(16) |
| Cl1[1] | Cd01 | Cl1[6] | 79.44(6) | Cl1[2] | Sb01 | Cl1[3] | 104.77(6) |
| Cl1[6] | Cd01 | Cl1[5] | 167.23(6) | Cl1[1] | Sb01 | Cl1 | 180.0 |
| Cl1[7] | Cd01 | Cl1[5] | 12.77(6) | Cl1[2] | Sb01 | Cl1[5] | 75.23(6) |
| Cl1 | Cd01 | Cl1[5] | 92.078(16) | Cl1 | Sb01 | Cl1[3] | 87.922(16) |
| Cl1[7] | Cd01 | Cl1[3] | 167.23(6) | Cl1[7] | Sb01 | Cl1[2] | 87.921(16) |
| Cl1[4] | Cd01 | Cl1[5] | 104.77(6) | Cl1[2] | Sb01 | Cl1[4] | 180.00(7) |
| Cl1[3] | Cd01 | Cl1[5] | 180.00(7) | Cl1[1] | Sb01 | Cl1[5] | 87.921(16) |
| Cl1[1] | Cd01 | Cl1[3] | 92.079(16) | Cl1[6] | Sb01 | Cl1 | 100.57(6) |
| Cl1[1] | Cd01 | Cl1[7] | 100.56(6) | Cl1 | Sb01 | Cl1[2] | 167.23(6) |
| Cl1[7] | Cd01 | Cl1[2] | 87.921(16) | Cl1[6] | Sb01 | Cl1[2] | 92.079(16) |
| Cl1[1] | Cd01 | Cl1[4] | 167.23(6) | Cl1[7] | Sb01 | Cl1[3] | 167.23(6) |
| Cl1[6] | Cd01 | Cl1[3] | 12.77(6) | Cl1[4] | Sb01 | Cl1[3] | 75.23(6) |
| Cl1[2] | Cd01 | Cl1[3] | 104.77(6) | Cl1[1] | Sb01 | Cl1[6] | 79.44(6) |
| Cl1 | Cd01 | Cl1[2] | 167.23(6) | Cl1[3] | Sb01 | Cl1[5] | 180.00(7) |
| Cl1[7] | Cd01 | Cl1 | 79.43(6) | Cl1[7] | Sb01 | Cl1 | 79.43(6) |
| Cl1 | Cd01 | Cl1[4] | 12.77(6) | Cl1[6] | Sb01 | Cl1[5] | 167.23(6) |
| Cl1[1] | Cd01 | Cl1[2] | 12.77(6) | Cl1[1] | Sb01 | Cl1[7] | 100.56(6) |
| Cl1[2] | Cd01 | Cl1[4] | 180.00(7) | Cl1 | Sb01 | Cl1[5] | 92.078(16) |

**Table S9-4 Bond Angles for Sb:PDACdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl1 | Cd01 | Cl1[3] | 87.922(16) | Cl1[7] | Sb01 | Cl1[5] | 12.77(6) |
| Cl1[1] | Cd01 | Cl1 | 180.0 | Cl1[4] | Sb01 | Cl1[5] | 104.77(6) |
| Cl1[2] | Cd01 | Cl1[5] | 75.23(6) | Cl1[1] | Sb01 | Cl1[4] | 167.23(6) |
| Cl1[6] | Cd01 | Cl1 | 100.57(6) | Cl1[6] | Sb01 | Cl1[3] | 12.77(6) |
| Cl1[6] | Cd01 | Cl1[2] | 92.079(16) | Cl1[6] | Sb01 | Cl1[7] | 180.00(7) |
| Cl1[4] | Cd01 | Cl1[3] | 75.23(6) | Cl1[6] | Sb01 | Cl1[4] | 87.921(16) |
| Cl1[1] | Cd01 | Cl1[5] | 87.921(16) | Cl1[7] | Sb01 | Cl1[4] | 92.079(16) |
| Cl1[6] | Cd01 | Cl1[4] | 87.921(16) | Cd01 | Cl1 | Cd01[9] | 160.76(6) |
| C006 | C005 | C006[8] | 126.4(8) | Cl1[4] | Cl1 | Cd01 | 85.0(5) |
| C007 | C006 | C005 | 125.5(6) | Cl1[4] | Cl1 | Cd01[9] | 82.3(5) |
| N004 | C007 | C006 | 125.0(5) | Cl1[4] | Cl1 | Sb01 | 85.0(5) |
| Cl02[1] | Sb01 | Cl02 | 180.0 | | | | |

[1]1-X,1-Y,-Z; [2]-1/2+X,1-Y,-1/2+Z; [3]-1/2+X,+Y,1/2-Z; [4]3/2-X,+Y,1/2-Z; [5]3/2-X,1-Y,-1/2+Z; [6]1-X,+Y,+Z; [7]+X,1-Y,-Z; [8]1-X,3/2-Y,+Z; [9]3/2-X,1-Y,1/2+Z

**Table S9-5 Torsion Angles for Sb:PDACdCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| C005 | C006 | C007 | N004 | 180.000(4) | C006[1] | C005 | C006 | C007 | 180.000(5) |

[1]1-X,3/2-Y,+Z

**Table S9-6 Hydrogen Atom Coordinates (Å×10[4]) and Isotropic Displacement Parameters (Å[2]×10[3]) for Sb:PDACdCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H00A | 6150.23 | 5799.03 | 4833.32 | 69 |
| H00B | 4640.47 | 5989.23 | 3642.24 | 69 |
| H00C | 4209.3 | 5712.55 | 5374.9 | 69 |
| H00E | 6085.67 | 7500 | 6442.07 | 57 |
| H00D | 3914.33 | 7500 | 6442.06 | 57 |
| H00G | 3913.6 | 6973.51 | 3989.83 | 84 |
| H00F | 6086.4 | 6973.51 | 3989.83 | 84 |
| H00I | 6086.78 | 6461.36 | 6458.33 | 65 |
| H00H | 3913.22 | 6461.36 | 6458.33 | 65 |

**Table S9-7 Atomic Occupancy for Sb:PDACdCl.**

| Atom | *Occupancy* | Atom | *Occupancy* | Atom | *Occupancy* |
|---|---|---|---|---|---|
| H00A | 0.5 | H00B | 0.5 | H00C | 0.5 |
| H00E | 0.5 | H00D | 0.5 | H00G | 0.5 |
| H00F | 0.5 | H00I | 0.5 | H00H | 0.5 |
| Sb01 | 0.0001 | Cl1 | 0.5 | | |

Table S10: Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for PDASbCl

# PDASbCl

**Table S10-1 Crystal data and structure refinement for PDASbCl.**

| | |
|---|---|
| Identification code | PDASbCl |
| Empirical formula | $C_5H_{16}Cl_5N_2Sb$ |
| Formula weight | 403.20 |
| Temperature/K | 100.00 |
| Crystal system | orthorhombic |
| Space group | $P2_12_12_1$ |
| a/Å | 7.5550(5) |
| b/Å | 13.3233(10) |
| c/Å | 13.7812(10) |
| α/° | 90 |
| β/° | 90 |
| γ/° | 90 |
| Volume/Å$^3$ | 1387.18(17) |
| Z | 4 |
| $\rho_{calc}$g/cm$^3$ | 1.931 |
| μ/mm$^{-1}$ | 2.917 |
| F(000) | 784.0 |
| Crystal size/mm$^3$ | 0.21 × 0.2 × 0.19 |
| Radiation | MoKα (λ = 0.71073) |
| 2Θ range for data collection/° | 5.912 to 52.94 |
| Index ranges | $-9 \le h \le 9$, $-16 \le k \le 16$, $-17 \le l \le 17$ |
| Reflections collected | 31654 |
| Independent reflections | 2864 [$R_{int}$ = 0.0329, $R_{sigma}$ = 0.0165] |
| Data/restraints/parameters | 2864/0/120 |
| Goodness-of-fit on $F^2$ | 1.116 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0094, $wR_2$ = 0.0231 |
| Final R indexes [all data] | $R_1$ = 0.0096, $wR_2$ = 0.0232 |
| Largest diff. peak/hole / e Å$^{-3}$ | 0.23/-0.31 |

Flack parameter -0.010(5)

**Table S10-2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for PDASbCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Sb01 | 4828.1(2) | 8674.4(2) | 8970.2(2) | 11.13(4) |
| Cl02 | 5557.0(6) | 7300.4(3) | 7592.2(3) | 15.92(10) |
| Cl03 | 2352.5(6) | 9423.4(4) | 8002.9(4) | 18.79(10) |
| Cl04 | 7638.8(7) | 7919.5(4) | 10092.4(3) | 18.43(10) |
| Cl05 | 6937.4(7) | 9804.6(4) | 8225.2(4) | 19.47(11) |
| Cl06 | 4322.9(7) | 9809.8(4) | 10471.4(4) | 21.64(11) |
| N007 | 9874(2) | 4672.2(11) | 3935.2(12) | 18.1(3) |
| N008 | 9714(2) | 7416.3(13) | 8080.6(11) | 18.0(3) |
| C009 | 10000(3) | 6059.7(14) | 5129.8(13) | 15.3(4) |
| C00A | 10911(3) | 7043.6(15) | 5398.7(14) | 17.2(4) |
| C00B | 10139(3) | 7522.3(14) | 6312.0(13) | 16.9(4) |
| C00C | 10525(3) | 6917.3(15) | 7218.1(13) | 15.9(4) |
| C00D | 10719(3) | 5657.9(15) | 4182.8(14) | 18.7(4) |

**Table S10-3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for PDASbCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Sb01 | 11.19(6) | 11.08(6) | 11.10(6) | 0.24(4) | -0.36(5) | -0.46(4) |
| Cl02 | 17.4(2) | 14.4(2) | 15.9(2) | -0.63(17) | 0.87(18) | -0.64(17) |
| Cl03 | 16.4(2) | 19.1(2) | 20.9(2) | -0.3(2) | -4.92(19) | 3.22(18) |
| Cl04 | 17.3(2) | 21.4(2) | 16.6(2) | 0.93(18) | -1.25(19) | 4.74(19) |
| Cl05 | 19.6(2) | 19.6(2) | 19.2(2) | 4.0(2) | -2.01(19) | -8.68(19) |
| Cl06 | 19.1(3) | 22.2(2) | 23.6(3) | -9.2(2) | -0.1(2) | 2.54(19) |
| N007 | 22.1(8) | 15.5(7) | 16.7(7) | -2.7(6) | -0.5(9) | 0.6(7) |
| N008 | 17.2(8) | 22.8(8) | 14.1(7) | -2.3(7) | -0.1(7) | 1.4(7) |
| C009 | 16.7(9) | 15.5(8) | 13.8(8) | -1.3(7) | 0.9(8) | -1.3(8) |
| C00A | 22.8(11) | 16.0(10) | 12.7(9) | -0.1(8) | 2.9(8) | -4.0(8) |
| C00B | 21.1(10) | 15.0(9) | 14.6(8) | -1.4(7) | -0.4(8) | -0.8(8) |
| C00C | 16.0(10) | 19.1(9) | 12.6(9) | -2.0(7) | 1.3(7) | 0.8(8) |
| C00D | 24.0(11) | 16.2(9) | 15.8(10) | -2.8(8) | 2.9(8) | -4.6(8) |

**Table S10-4 Bond Lengths for PDASbCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Sb01 | Cl02 | 2.6947(5) | N008 | C00C | 1.494(2) |
| Sb01 | Cl03 | 2.5041(5) | C009 | C00A | 1.526(3) |
| Sb01 | Cl04 | 2.8129(5) | C009 | C00D | 1.512(3) |
| Sb01 | Cl05 | 2.4209(5) | C00A | C00B | 1.527(3) |
| Sb01 | Cl06 | 2.5911(5) | C00B | C00C | 1.515(3) |
| N007 | C00D | 1.500(2) | | | |

**Table S10-5 Bond Angles for PDASbCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl02 | Sb01 | Cl04 | 89.444(15) | Cl06 | Sb01 | Cl02 | 171.657(16) |
| Cl03 | Sb01 | Cl02 | 92.762(16) | Cl06 | Sb01 | Cl04 | 83.163(16) |
| Cl03 | Sb01 | Cl04 | 177.409(16) | C00D | C009 | C00A | 110.57(16) |
| Cl03 | Sb01 | Cl06 | 94.725(17) | C009 | C00A | C00B | 112.74(17) |
| Cl05 | Sb01 | Cl02 | 89.378(17) | C00C | C00B | C00A | 112.57(16) |
| Cl05 | Sb01 | Cl03 | 91.033(18) | N008 | C00C | C00B | 109.88(15) |
| Cl05 | Sb01 | Cl04 | 87.631(17) | N007 | C00D | C009 | 110.69(16) |
| Cl05 | Sb01 | Cl06 | 94.156(18) | | | | |

**Table S10-6 Torsion Angles for PDASbCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| C009 | C00A | C00B | C00C | 68.1(2) | C00A | C00B | C00C | N008 | −179.15(17) |
| C00A | C009 | C00D | N007 | 177.92(17) | C00D | C009 | C00A | C00B | 175.80(17) |

**Table S10-7 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for PDASbCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H00A | 10057.28 | 4533.36 | 3296.49 | 22 |
| H00B | 8690.65 | 4706.78 | 4053.67 | 22 |
| H00C | 10361.14 | 4178.49 | 4305.09 | 22 |
| H00D | 8515.82 | 7428.2 | 8011.95 | 22 |
| H00E | 10127.97 | 8055.7 | 8128.57 | 22 |
| H00F | 10002.53 | 7069.04 | 8626.67 | 22 |

**Table S10-7 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for PDASbCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H00G | 10194.73 | 5560.03 | 5650.63 | 18 |
| H00H | 8709.51 | 6172.67 | 5068.52 | 18 |
| H00I | 10795.63 | 7520.37 | 4851.37 | 21 |
| H00J | 12188.33 | 6915.38 | 5501.58 | 21 |
| H00K | 10637.61 | 8204.79 | 6389.25 | 20 |
| H00L | 8841.6 | 7588.6 | 6234.76 | 20 |
| H00M | 10036.62 | 6231.82 | 7146.51 | 19 |
| H00N | 11820.94 | 6861.28 | 7310.78 | 19 |
| H00O | 10485.24 | 6147.06 | 3657.45 | 22 |
| H00P | 12016.77 | 5569.08 | 4236.4 | 22 |

Table S11: Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for DABCdCl

# DABCdCl

**Table S11-1 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for DABCdCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Cd1 | 5000 | 5000 | 0 | 12.35(18) |
| Cl1 | 5349.3(10) | 2613.0(10) | 2669.5(10) | 16.5(2) |
| Cl2 | 2323.0(8) | 4475.2(12) | -591.4(11) | 18.8(2) |
| N1 | 7315(4) | 5311(4) | 5670(5) | 19.3(7) |
| C1 | 8606(4) | 4261(5) | 6419(5) | 22.5(7) |
| C2 | 9633(4) | 4133(4) | 5138(5) | 19.6(7) |
| O1 | 5000 | 5000 | 5000 | 56(4) |

**Table S11-2 Anisotropic Displacement Parameters (Å$^2$×10$^3$) for DABCdCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| Cd1 | 12.4(2) | 12.3(3) | 12.5(2) | -1.13(10) | 2.90(15) | 0.14(9) |
| Cl1 | 18.7(4) | 15.1(4) | 16.3(4) | 1.8(3) | 5.5(3) | 5.4(3) |
| Cl2 | 13.3(4) | 16.5(4) | 26.1(4) | -0.7(3) | 3.2(3) | 0.1(3) |
| N1 | 20.0(16) | 14.0(12) | 26.8(18) | 2.7(13) | 11.8(14) | -0.3(14) |
| C1 | 19.8(17) | 24.9(19) | 23.2(17) | 2.4(14) | 5.6(14) | 5.2(14) |
| C2 | 20.7(16) | 15.6(17) | 23.5(17) | 0.8(15) | 7.0(13) | -0.6(12) |
| O1 | 25(6) | 55(9) | 75(11) | -4(4) | -18(6) | -2(4) |

**Table S11-3 Bond Lengths for DABCdCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Cd1 | Cl1[1] | 2.6905(7) | Cd1 | Cl2[1] | 2.4999(8) |
| Cd1 | Cl1 | 2.6905(7) | N1 | C1 | 1.466(5) |
| Cd1 | Cl1[2] | 2.7236(7) | C1 | C2 | 1.514(5) |
| Cd1 | Cl1[3] | 2.7236(7) | C2 | C2[4] | 1.517(7) |
| Cd1 | Cl2 | 2.4999(8) | | | |

[1]1-X,1-Y,-Z; [2]+X,1/2-Y,-1/2+Z; [3]1-X,1/2+Y,1/2-Z; [4]2-X,1-Y,1-Z

**Table S11-4 Bond Angles for DABCdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl1 | Cd1 | Cl1[1] | 180.0 | Cl2[1] | Cd1 | Cl1 | 90.53(3) |
| Cl1 | Cd1 | Cl1[2] | 90.261(7) | Cl2 | Cd1 | Cl1 | 89.47(3) |
| Cl1[3] | Cd1 | Cl1[1] | 90.261(7) | Cl2[1] | Cd1 | Cl1[3] | 88.81(3) |
| Cl1[3] | Cd1 | Cl1[2] | 180.0 | Cl2 | Cd1 | Cl1[1] | 90.53(3) |
| Cl2[1] | Cd1 | Cl1[1] | 89.47(3) | Cl2 | Cd1 | Cl2[1] | 180.0 |
| Cl2 | Cd1 | Cl1[3] | 91.19(3) | C2 | C1 | N1 | 111.9(3) |
| Cl2 | Cd1 | Cl1[2] | 88.81(3) | C2[4] | C2 | C1 | 113.9(4) |
| Cl2[1] | Cd1 | Cl1[2] | 91.19(3) | | | | |

[1]1-X,1-Y,-Z; [2]1-X,1/2+Y,1/2-Z; [3]+X,1/2-Y,-1/2+Z; [4]2-X,1-Y,1-Z

**Table S11-5 Torsion Angles for DABCdCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| N1 | C1 | C2 | C2[1] | 68.7(4) | C1 | C2 | C2[1] | C1[1] | -180.0(4) |

[1]2-X,1-Y,1-Z

**Table S11-6 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for DABCdCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H1d | 9118(4) | 4808(5) | 7553(5) | 27.0(9) |
| H1e | 8303(4) | 3056(5) | 6695(5) | 27.0(9) |
| H2a | 9085(4) | 3709(4) | 3964(5) | 23.5(8) |
| H2b | 10389(4) | 3243(4) | 5600(5) | 23.5(8) |
| H1a | 7520(40) | 6300(20) | 5420(50) | 16(10) |
| H1b | 6940(50) | 4850(50) | 4740(30) | 17(13) |
| H1c | 6960(60) | 5410(70) | 6530(50) | 38(15) |

**Table S11-7 Atomic Occupancy for DABCdCl.**

| Atom | *Occupancy* | Atom | *Occupancy* | Atom | *Occupancy* |
|---|---|---|---|---|---|
| O1 | 0.500000 | | | | |

**Table S12:** Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for Sb:DABCdCl

# Sb:DABCdCl

**Table S12-1 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:DABCdCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Cd1 | 5000 | 0 | 5000 | 13.35(13) |
| Cl1 | 5343.9(9) | 2389.3(11) | 7664.5(12) | 17.2(2) |
| Cl2 | 2321.9(9) | 523.6(12) | 4409.4(13) | 20.4(2) |
| N1 | 2696(4) | 4681(4) | 4329(5) | 20.4(7) |
| C1 | 368(4) | 5864(5) | 4862(6) | 21.7(8) |
| C2 | 1385(4) | 5746(6) | 3581(6) | 24.7(9) |
| Sb1 | 5000 | 0 | 5000 | 13.35(13) |
| O2 | 5000 | 5000 | 5000 | 69(4) |

**Table S12-2 Anisotropic Displacement Parameters ($Å^2×10^3$) for Sb:DABCdCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Cd1 | 12.58(19) | 13.6(2) | 14.2(2) | -0.15(13) | 3.58(13) | 1.21(13) |
| Cl1 | 19.9(4) | 15.6(4) | 17.4(4) | -5.5(3) | 6.7(3) | -2.4(3) |
| Cl2 | 13.5(4) | 18.7(4) | 28.7(5) | -0.5(4) | 3.4(3) | 0.7(3) |
| N1 | 19.8(16) | 14.6(16) | 30.0(19) | -0.3(14) | 12.6(14) | 2.4(13) |
| C1 | 22.0(19) | 19(2) | 25(2) | -0.8(16) | 7.8(16) | 1.1(16) |
| C2 | 21.0(19) | 26(2) | 27(2) | 5.4(17) | 4.5(16) | 2.1(16) |
| Sb1 | 12.58(19) | 13.6(2) | 14.2(2) | -0.15(13) | 3.58(13) | 1.21(13) |
| O2 | 60(8) | 72(9) | 69(9) | 7(7) | 0(7) | -9(7) |

**Table S12-3 Bond Lengths for Sb:DABCdCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Cd1 | $Cl1^1$ | 2.6908(8) | Cl1 | Sb1 | 2.6908(8) |
| Cd1 | $Cl1^2$ | 2.7256(8) | Cl2 | Sb1 | 2.4976(9) |
| Cd1 | $Cl1^3$ | 2.7256(8) | N1 | C2 | 1.485(5) |
| Cd1 | Cl1 | 2.6908(8) | C1 | $C1^4$ | 1.515(8) |
| Cd1 | Cl2 | 2.4976(9) | C1 | C2 | 1.507(6) |
| Cd1 | $Cl2^1$ | 2.4975(9) | | | |

[1]1-X,-Y,1-Z; [2]1-X,-1/2+Y,3/2-Z; [3]+X,1/2-Y,-1/2+Z; [4]-X,1-Y,1-Z

**Table S12-4 Bond Angles for Sb:DABCdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| $Cl1^1$ | Cd1 | Cl1 | 180.0 | N1 | C2 | C1 | 111.8(3) |
| $Cl1^2$ | Cd1 | $Cl1^3$ | 180.0 | Cl1 | Sb1 | $Cl1^2$ | 90.197(6) |
| $Cl1^1$ | Cd1 | $Cl1^3$ | 90.197(6) | $Cl1^1$ | Sb1 | $Cl1^3$ | 90.197(6) |
| Cl1 | Cd1 | $Cl1^2$ | 90.197(6) | $Cl1^2$ | Sb1 | $Cl1^3$ | 180.0 |
| $Cl1^1$ | Cd1 | $Cl1^2$ | 89.803(6) | $Cl1^1$ | Sb1 | $Cl1^2$ | 89.803(6) |
| Cl1 | Cd1 | $Cl1^3$ | 89.803(6) | $Cl1^1$ | Sb1 | Cl1 | 180.0 |
| $Cl2^1$ | Cd1 | $Cl1^3$ | 88.88(3) | Cl1 | Sb1 | $Cl1^3$ | 89.803(6) |
| $Cl2^1$ | Cd1 | $Cl1^1$ | 89.32(3) | Cl2 | Sb1 | $Cl1^2$ | 88.88(3) |
| Cl2 | Cd1 | Cl1 | 89.32(3) | Cl2 | Sb1 | $Cl1^1$ | 90.68(3) |
| Cl2 | Cd1 | $Cl1^1$ | 90.68(3) | $Cl2^1$ | Sb1 | $Cl1^1$ | 89.32(3) |
| Cl2 | Cd1 | $Cl1^2$ | 88.88(3) | $Cl2^1$ | Sb1 | $Cl1^2$ | 91.12(3) |
| Cl2 | Cd1 | $Cl1^3$ | 91.12(3) | $Cl2^1$ | Sb1 | Cl1 | 90.68(3) |

**Table S12-4 Bond Angles for Sb:DABCdCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl2[1] | Cd1 | Cl1 | 90.68(3) | Cl2[1] | Sb1 | Cl1[3] | 88.88(3) |
| Cl2[1] | Cd1 | Cl1[2] | 91.12(3) | Cl2 | Sb1 | Cl1 | 89.32(3) |
| Cl2[1] | Cd1 | Cl2 | 180.00(4) | Cl2 | Sb1 | Cl1[3] | 91.12(3) |
| Cd1 | Cl1 | Cd1[4] | 165.91(4) | Cl2[1] | Sb1 | Cl2 | 180.00(4) |
| C2 | C1 | C1[5] | 114.2(4) | | | | |

[1]1-X,-Y,1-Z; [2]1-X,-1/2+Y,3/2-Z; [3]+X,1/2-Y,-1/2+Z; [4]1-X,1/2+Y,3/2-Z; [5]-X,1-Y,1-Z

**Table S12-5 Torsion Angles for Sb:DABCdCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| C1[1] | C1 | C2 | N1 | -68.8(5) | | | | | |

[1]-X,1-Y,1-Z

**Table S12-6 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:DABCdCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H1D | -388.43 | 6756.77 | 4409.02 | 26 |
| H1E | 921.87 | 6283.54 | 6034.76 | 26 |
| H2A | 867.7 | 5204.15 | 2447.18 | 30 |
| H2B | 1686.08 | 6951.99 | 3309.04 | 30 |
| H1A | 3220(30) | 4650(80) | 3450(30) | 53(18) |
| H1B | 3140(50) | 5210(50) | 5370(30) | 27(13) |
| H1C | 2530(50) | 3528(18) | 4560(60) | 32(13) |

**Table S12-7 Atomic Occupancy for Sb:DABCdCl.**

| Atom | *Occupancy* | Atom | *Occupancy* | Atom | *Occupancy* |
|---|---|---|---|---|---|
| Sb1 | 0.0001 | O2 | 0.5 | | |

Table S13: Summary of SCXRD structural analysis and tables of bond lengths, bond angles, dihedral angles for DABSbCl

# DABSbCl

**Table S13-1 Crystal data and structure refinement for DABSbCl.**

| | |
|---|---|
| Identification code | DABSbCl |
| Empirical formula | $C_4H_{14}Cl_5N_2Sb$ |
| Formula weight | 389.17 |
| Temperature/K | 100.00 |
| Crystal system | monoclinic |
| Space group | $P2_1/c$ |
| a/Å | 9.8543(4) |
| b/Å | 10.2894(4) |
| c/Å | 12.9617(5) |
| α/° | 90 |
| β/° | 106.6220(10) |
| γ/° | 90 |
| Volume/Å$^3$ | 1259.33(9) |
| Z | 4 |
| $\rho_{calc}$g/cm$^3$ | 2.053 |
| μ/mm$^{-1}$ | 3.209 |
| F(000) | 752.0 |
| Crystal size/mm$^3$ | ? × ? × ? |
| Radiation | MoKα (λ = 0.71073) |
| 2Θ range for data collection/° | 4.314 to 52.802 |
| Index ranges | $-12 \le h \le 12$, $-12 \le k \le 12$, $-16 \le l \le 16$ |
| Reflections collected | 28477 |
| Independent reflections | 2586 [$R_{int}$ = 0.0339, $R_{sigma}$ = 0.0157] |
| Data/restraints/parameters | 2586/0/111 |
| Goodness-of-fit on $F^2$ | 1.111 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0122, $wR_2$ = 0.0292 |
| Final R indexes [all data] | $R_1$ = 0.0132, $wR_2$ = 0.0297 |
| Largest diff. peak/hole / e Å$^{-3}$ | 0.37/-0.34 |

**Table S13-2 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for DABSbCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Sb01 | 3134.7(2) | 9166.2(2) | 4324.0(2) | 11.66(4) |
| Cl02 | 6043.2(4) | 9489.5(3) | 3539.0(3) | 14.96(7) |
| Cl03 | 11446.8(4) | 3712.1(3) | 8061.9(3) | 16.27(7) |
| Cl04 | 4512.2(4) | 7370.6(3) | 5337.1(3) | 17.78(8) |
| Cl05 | 1089.6(4) | 8539.5(4) | 4830.6(3) | 19.47(8) |
| Cl06 | 2420.7(4) | 7862.2(3) | 2703.3(3) | 19.91(8) |

**Table S13-2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for DABSbCl. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| N007 | 8706.1(14) | 5448.7(13) | 7587.3(10) | 17.7(3) |
| N008 | 6024.2(14) | 6455.0(12) | 3001.8(10) | 16.1(3) |
| C009 | 6700.7(17) | 5657.9(14) | 3977.8(12) | 15.6(3) |
| C00A | 7818.1(16) | 5474.6(14) | 6441.4(11) | 15.2(3) |
| C00B | 7999.4(16) | 6360.5(15) | 4663.5(12) | 16.3(3) |
| C00C | 8754.0(17) | 5631.0(15) | 5690.3(12) | 17.9(3) |

**Table S13-3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for DABSbCl. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Sb01 | 10.91(6) | 11.33(6) | 12.32(6) | 0.83(3) | 2.67(4) | -0.21(3) |
| Cl02 | 16.79(18) | 13.39(16) | 14.77(16) | 0.41(13) | 4.65(13) | 1.26(13) |
| Cl03 | 14.64(17) | 15.25(16) | 18.37(17) | 0.10(13) | 3.84(14) | 1.08(13) |
| Cl04 | 17.50(18) | 14.33(16) | 20.17(18) | 3.15(13) | 3.23(14) | 3.52(13) |
| Cl05 | 12.90(17) | 25.11(19) | 20.60(18) | 5.26(14) | 5.11(14) | -2.04(14) |
| Cl06 | 26.3(2) | 16.04(17) | 15.60(17) | -2.53(13) | 3.05(15) | -3.34(14) |
| N007 | 14.3(7) | 23.6(7) | 14.8(6) | -0.2(5) | 3.3(5) | 3.5(5) |
| N008 | 16.3(6) | 15.3(6) | 15.8(6) | -2.1(5) | 3.2(5) | -1.3(5) |
| C009 | 18.0(8) | 13.3(7) | 15.6(7) | -0.2(6) | 4.9(6) | -0.4(6) |
| C00A | 13.6(7) | 15.8(7) | 15.0(7) | 0.7(6) | 2.1(6) | 1.4(6) |
| C00B | 16.3(8) | 17.2(7) | 15.1(7) | 0.0(6) | 4.0(6) | -1.3(6) |
| C00C | 13.7(8) | 21.3(8) | 18.9(7) | 1.4(6) | 5.3(6) | 3.8(6) |

**Table S13-4 Bond Lengths for DABSbCl.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Sb01 | Cl04 | 2.4415(4) | N008 | C009 | 1.4950(19) |
| Sb01 | Cl05 | 2.3802(4) | C009 | C00B | 1.517(2) |
| Sb01 | Cl06 | 2.4213(4) | C00A | C00C | 1.529(2) |
| N007 | C00A | 1.4930(19) | C00B | C00C | 1.524(2) |

**Table S13-5 Bond Angles for DABSbCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl05 | Sb01 | Cl04 | 91.776(13) | N007 | C00A | C00C | 110.28(12) |

**Table S13-5 Bond Angles for DABSbCl.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl05 | Sb01 | Cl06 | 91.741(13) | C009 | C00B | C00C | 113.35(12) |
| Cl06 | Sb01 | Cl04 | 91.501(13) | C00B | C00C | C00A | 112.05(12) |
| N008 | C009 | C00B | 109.37(12) | | | | |

**Table S13-6 Torsion Angles for DABSbCl.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| N007 | C00A | C00C | C00B | 149.41(13) | C009 | C00B | C00C | C00A | -62.92(17) |
| N008 | C009 | C00B | C00C | 179.16(12) | | | | | |

**Table S13-7 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for DABSbCl.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H00A | 9359.52 | 4801.78 | 7678.04 | 21 |
| H00B | 8144.03 | 5303.18 | 8023.53 | 21 |
| H00C | 9156.12 | 6225.21 | 7758.81 | 21 |
| H00D | 5236.54 | 6040.77 | 2594.88 | 19 |
| H00E | 6646.76 | 6564.74 | 2608.35 | 19 |
| H00F | 5777.56 | 7244.85 | 3207.96 | 19 |
| H00G | 6976.79 | 4799.26 | 3757.92 | 19 |
| H00H | 6019.16 | 5516.34 | 4399 | 19 |
| H00I | 7138.91 | 6205.95 | 6332.42 | 18 |
| H00J | 7270.35 | 4656.84 | 6269.43 | 18 |
| H00K | 7712 | 7226.85 | 4858.97 | 20 |
| H00L | 8670.57 | 6495.16 | 4232.51 | 20 |
| H00M | 9625.25 | 6108.36 | 6071.94 | 21 |
| H00N | 9037.3 | 4761.1 | 5499.05 | 21 |

# Sb:DABCdCl_Dehydrated

**Table S14-1 Crystal data and structure refinement for Sb:DABCdCl_Dehydrated.**

| | |
|---|---|
| Identification code | Sb:DABCdCl_Dehydrated |
| Empirical formula | $C_4H_{14}CdCl_4N_2Sb$ |
| Formula weight | 344.37 |
| Temperature/K | 100.00 |

| | |
|---|---|
| Crystal system | monoclinic |
| Space group | $P2_1/c$ |
| a/Å | 9.3984(5) |
| b/Å | 7.5570(4) |
| c/Å | 7.6313(4) |
| α/° | 90 |
| β/° | 102.104(2) |
| γ/° | 90 |
| Volume/Å$^3$ | 529.95(5) |
| Z | 2 |
| $\rho_{calc}$g/cm$^3$ | 2.158 |
| μ/mm$^{-1}$ | 3.014 |
| F(000) | 336.0 |
| Crystal size/mm$^3$ | 0.18 × 0.17 × 0.17 |
| Radiation | MoKα (λ = 0.71073) |
| 2Θ range for data collection/° | 6.982 to 61.014 |
| Index ranges | $-13 \leq h \leq 13$, $-10 \leq k \leq 10$, $-9 \leq l \leq 10$ |
| Reflections collected | 10427 |
| Independent reflections | 1619 [$R_{int}$ = 0.0513, $R_{sigma}$ = 0.0331] |
| Data/restraints/parameters | 1619/0/53 |
| Goodness-of-fit on $F^2$ | 1.091 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0248, $wR_2$ = 0.0611 |
| Final R indexes [all data] | $R_1$ = 0.0276, $wR_2$ = 0.0619 |
| Largest diff. peak/hole / e Å$^{-3}$ | 0.91/-0.88 |

**Table S14-2 Fractional Atomic Coordinates (×10$^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:DABCdCl_Dehydrated. $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| Cd01 | 5000 | 5000 | 5000 | 12.20(8) |
| Cl2 | 4647.6(6) | 7379.5(6) | 2319.3(7) | 15.65(12) |
| Cl1 | 2313.5(6) | 4476.1(7) | 4414.6(8) | 16.97(12) |
| N1 | 2716(2) | 314(3) | 4314(3) | 19.1(4) |
| C2 | 368(2) | -870(3) | 4864(3) | 16.3(4) |
| C1 | 1390(2) | -754(3) | 3570(3) | 18.0(4) |
| Sb1 | 5000 | 5000 | 5000 | 12.20(8) |

**Table S14-3 Anisotropic Displacement Parameters ($Å^2×10^3$) for Sb:DABCdCl_Dehydrated. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Cd01 | 13.52(12) | 11.23(12) | 11.85(13) | 0.12(7) | 2.68(8) | -0.75(7) |
| Cl2 | 18.7(2) | 13.7(2) | 15.2(2) | 3.38(19) | 5.02(19) | 1.25(17) |
| Cl1 | 14.5(2) | 14.3(2) | 21.5(3) | 0.5(2) | 2.2(2) | -0.43(18) |
| N1 | 16.0(9) | 16.4(9) | 25.9(12) | 0.1(8) | 6.6(8) | -0.4(7) |
| C2 | 17.3(10) | 13.7(10) | 18.5(11) | 1.9(9) | 5.1(8) | -0.8(8) |
| C1 | 17.7(10) | 18.9(11) | 17.9(11) | -3.3(9) | 5.3(9) | -1.9(8) |
| Sb1 | 13.52(12) | 11.23(12) | 11.85(13) | 0.12(7) | 2.68(8) | -0.75(7) |

**Table S14-4 Bond Lengths for Sb:DABCdCl_Dehydrated.**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| Cd01 | Cl2[1] | 2.6915(5) | Cl2 | Sb1 | 2.6915(5) |
| Cd01 | Cl2[2] | 2.7221(5) | Cl1 | Sb1 | 2.5016(5) |
| Cd01 | Cl2 | 2.6915(5) | N1 | C1 | 1.493(3) |
| Cd01 | Cl2[3] | 2.7221(5) | C2 | C2[4] | 1.521(4) |
| Cd01 | Cl1[1] | 2.5016(5) | C2 | C1 | 1.517(3) |
| Cd01 | Cl1 | 2.5016(5) | | | |

[1]1-X,1-Y,1-Z; [2]1-X,-1/2+Y,1/2-Z; [3]+X,3/2-Y,1/2+Z; [4]-X,-Y,1-Z

**Table S14-5 Bond Angles for Sb:DABCdCl_Dehydrated.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl2[1] | Cd01 | Cl2 | 180.0 | N1 | C1 | C2 | 112.06(19) |
| Cl2[1] | Cd01 | Cl2[2] | 89.760(5) | Cl2[1] | Sb1 | Cl2[2] | 89.760(5) |
| Cl2 | Cd01 | Cl2[2] | 90.240(5) | Cl2 | Sb1 | Cl2[3] | 89.760(5) |
| Cl2[2] | Cd01 | Cl2[3] | 180.000(17) | Cl2[1] | Sb1 | Cl2 | 180.0 |
| Cl2[1] | Cd01 | Cl2[3] | 90.240(5) | Cl2 | Sb1 | Cl2[2] | 90.240(5) |
| Cl2 | Cd01 | Cl2[3] | 89.760(5) | Cl2[1] | Sb1 | Cl2[3] | 90.240(5) |
| Cl1[1] | Cd01 | Cl2[2] | 88.590(17) | Cl2[2] | Sb1 | Cl2[3] | 180.000(17) |
| Cl1 | Cd01 | Cl2[2] | 91.410(18) | Cl1 | Sb1 | Cl2[2] | 91.410(18) |
| Cl1 | Cd01 | Cl2[1] | 89.354(18) | Cl1[1] | Sb1 | Cl2[3] | 91.410(17) |
| Cl1[1] | Cd01 | Cl2[1] | 90.646(18) | Cl1 | Sb1 | Cl2[3] | 88.590(18) |
| Cl1[1] | Cd01 | Cl2 | 89.354(18) | Cl1 | Sb1 | Cl2[1] | 89.354(18) |
| Cl1 | Cd01 | Cl2 | 90.646(18) | Cl1 | Sb1 | Cl2 | 90.646(18) |
| Cl1[1] | Cd01 | Cl2[3] | 91.410(17) | Cl1[1] | Sb1 | Cl2[1] | 90.646(18) |
| Cl1 | Cd01 | Cl2[3] | 88.590(18) | Cl1[1] | Sb1 | Cl2[2] | 88.590(17) |

**Table S14-5 Bond Angles for Sb:DABCdCl_Dehydrated.**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| Cl1[1] | Cd01 | Cl1 | 180.0 | Cl1[1] | Sb1 | Cl2 | 89.354(18) |
| Cd01 | Cl2 | Cd01[4] | 165.44(2) | Cl1[1] | Sb1 | Cl1 | 180.0 |
| C1 | C2 | C2[5] | 114.0(2) | | | | |

[1]1-X,1-Y,1-Z; [2]1-X,-1/2+Y,1/2-Z; [3]+X,3/2-Y,1/2+Z; [4]1-X,1/2+Y,1/2-Z; [5]-X,-Y,1-Z

**Table S14-6 Torsion Angles for Sb:DABCdCl_Dehydrated.**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| C2[1] | C2 | C1 | N1 | 68.9(3) | | | | | |

[1]-X,-Y,1-Z

**Table S14-7 Hydrogen Atom Coordinates (Å×10$^4$) and Isotropic Displacement Parameters (Å$^2$×10$^3$) for Sb:DABCdCl_Dehydrated.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H1A | 3304.37 | 342.72 | 3507.97 | 23 |
| H1B | 3201.91 | -186.77 | 5352.8 | 23 |
| H1C | 2448.66 | 1435.65 | 4535.49 | 23 |
| H2A | 924.52 | -1288.16 | 6038.49 | 20 |
| H2B | -391.24 | -1762.82 | 4412.24 | 20 |
| H1D | 1686.82 | -1962.16 | 3296.62 | 22 |
| H1E | 868.91 | -210.68 | 2435.77 | 22 |

**Table S14-8 Atomic Occupancy for Sb:DABCdCl_Dehydrated.**

| Atom | *Occupancy* | Atom | *Occupancy* | Atom | *Occupancy* |
|---|---|---|---|---|---|
| Sb1 | 0.0003 | | | | |

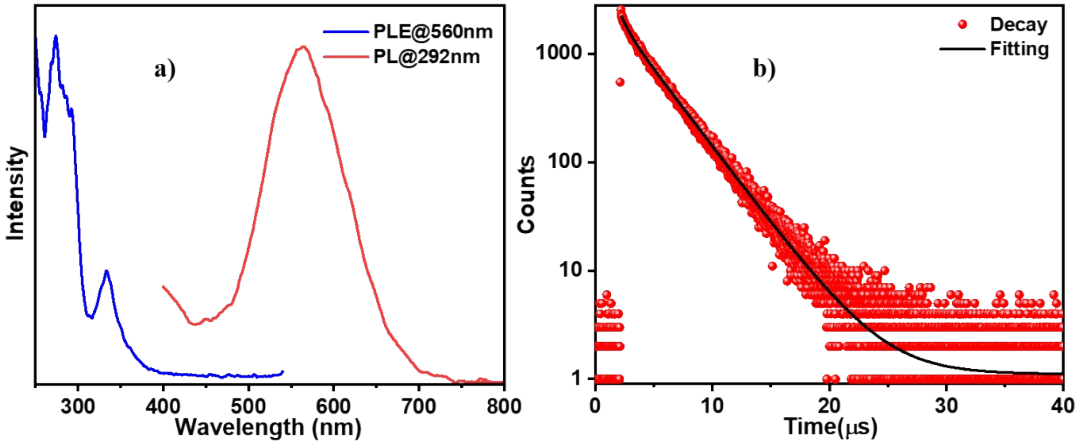


Figure S22: a) PL / PLE of dehydrated Sb:DABCdCl b) Lifetime Decay profile of dehydrated Sb:DABCdCl (Ex:310 nm Em:560 nm ).

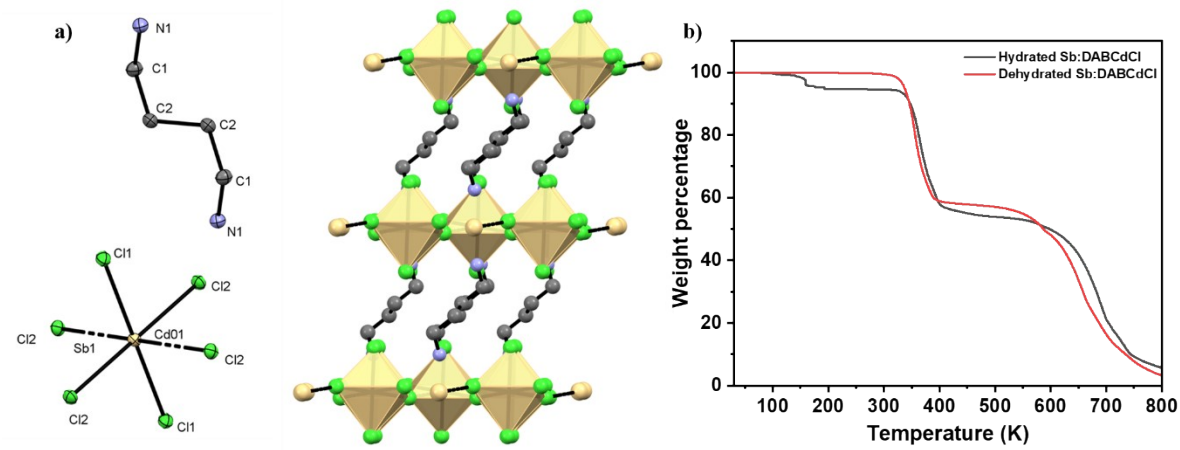


Figure S23: a) SCXRD structure of dehydrated Sb:DABCdCl hybrid b) TGA of Sb:DABCdCl [Black-Hydrated Sb:DABCdCl, Red- Dehydrated Sb:DABCdCl]

Table S14: Distortion parameters and optical properties of Hydrated and dehydrated Sb:DABCdCl

| Compound | Space group | Octahedral distortion (d) | Bond angle variance ($deg^2$) | M-Cl-M (deg) | $\phi_{tilt\ (deg)}$ | PL Max (nm) | PLE Peak positions (nm) | PLQY(%) | Lifetime (μs) |
|---|---|---|---|---|---|---|---|---|---|
| Sb:DABCdCl $2(C_4H_{14}N_2)Cd_2Cl_8O$ (Hydrated) | Monoclinic $P2_1/c$ | $1.4x10^{-3}$ | 0.6409 | 165.91 | 13.02 | 560 | 275, 291, 333 | 96 | 3.1 μs |
| Sb:DABCdCl $C_4H_{14}N_2CdCl_4$ (Dehydrated) | Monoclinic $P2_1/c$ | $1.5x10^{-3}$ | 0.8956 | 165.44 | 12.99 | 560 | 275,291, 333 | 39 | 3.02 μs |

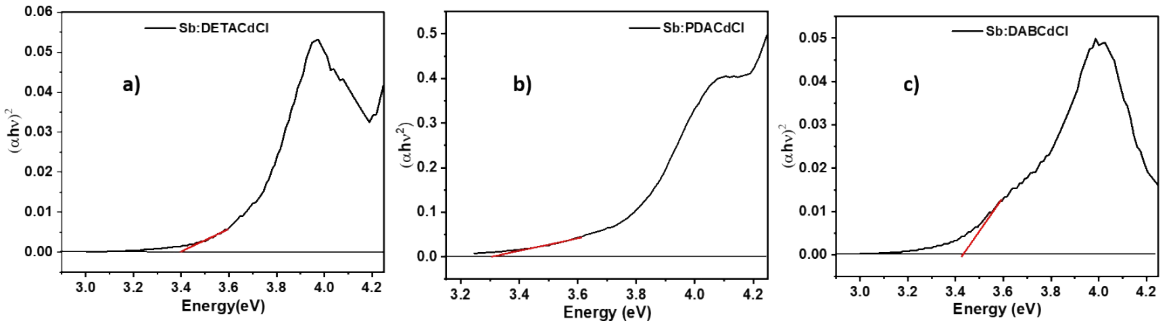


Figure S24: Tauc plot analysis of the doped hybrids- a) Sb:DETACdCl, b) Sb:PDACdCl, c) Sb:DABCdCl

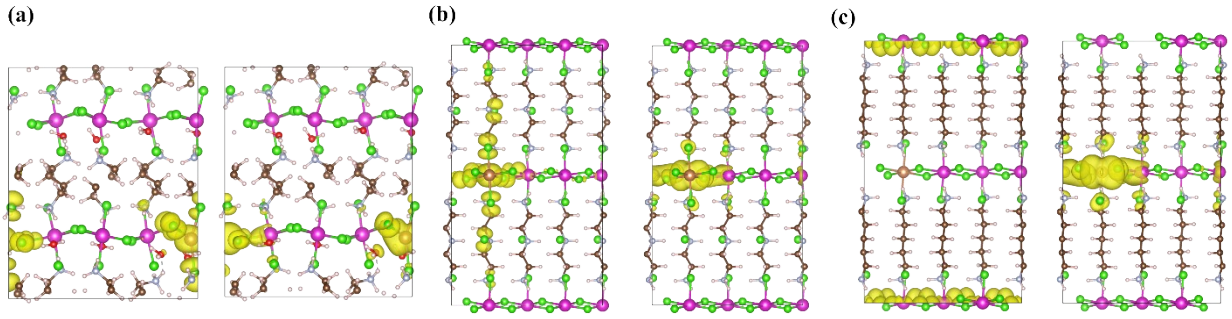


**Figure S25**: Band edge charge density localization of (a) Sb:DABCdCl (b) Sb:DETACdCl (c) Sb:PDACdCl.

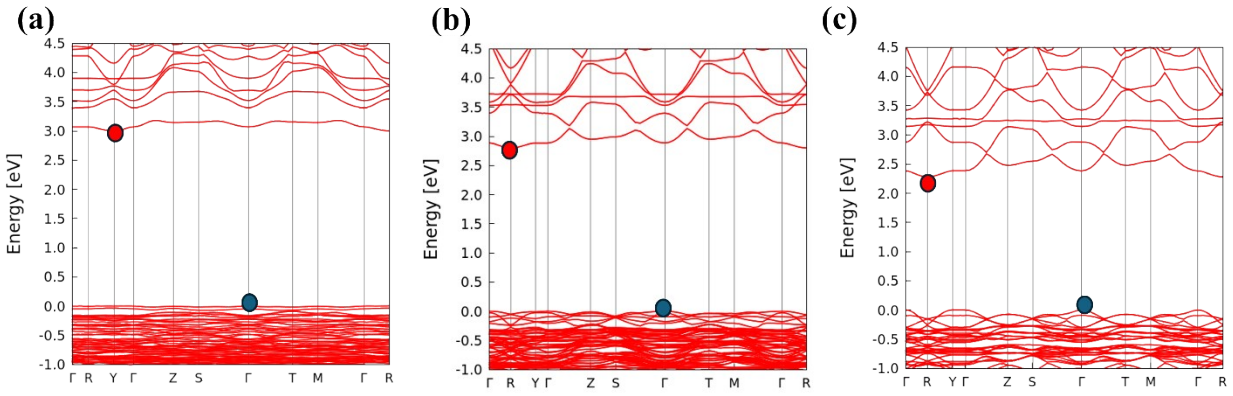


**Figure S26:** Band structure of (a) Sb:DABCdCl (b) Sb:DETACdCl (c) Sb:PDACdCl.

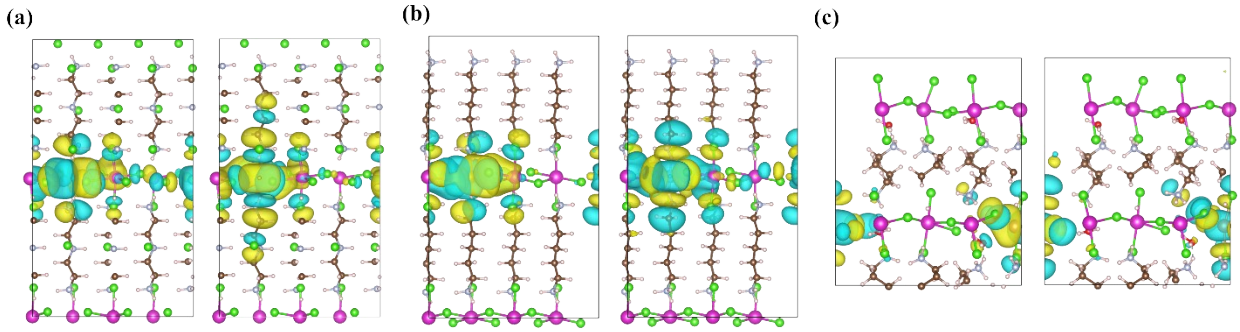


**Figure S27:** STE formation in the studied structure and their Kohn-Sham (KS) orbitals of the electron and hole in triplet state of (a) Sb:DETACdCl (b) Sb:PDACdCl and (c) Sb:DABCdCl.