

## Supporting information

### Near-unity quantum yield hybrid antimony halide for solid-state lighting and fingerprint detection

*Siyu Zhang<sup>1</sup>, Sha Bai<sup>1,\*</sup>, Xiaoyu Liu<sup>1</sup>, Panrong Zhao<sup>1</sup>, Binbin Su<sup>2</sup>, Huidong Xie<sup>1,\*</sup>*

*1. School of Chemistry and Chemical Engineering, Xi'an University of Architecture and Technology, Xi'an, 710055, Shaanxi, China;*

*2. Department of Materials Science and Engineering, Xi'an University of Science and Technology, Xi'an, 710054, Shaanxi, PR China*

*Corresponding authors: [baisha@xauat.edu.cn](mailto:baisha@xauat.edu.cn), orcid: 0000-0002-8683-7834*

*[xiehuidong@tsinghua.org.cn](mailto:xiehuidong@tsinghua.org.cn) (Xie H.), orcid: 0000-0001-5722-0635, Tel: 00-86-13679297751*

**Table S1. Fractional Atomic Coordinates ( $\times 10^4$ ) and Equivalent Isotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for (ATPPCl)SbCl<sub>5</sub>.  $U_{\text{eq}}$  is defined as 1/3 of the trace of the orthogonalised  $U_{\text{IJ}}$  tensor.**

| Atom | <i>x</i>  | <i>y</i>  | <i>z</i>  | $U_{\text{eq}}$ |
|------|-----------|-----------|-----------|-----------------|
| Sb1  | 5977.1(2) | 6191.7(2) | 6421.6(2) | 45.38(6)        |
| Cl1  | 6393.4(6) | 5419.0(2) | 6785.6(6) | 78.2(2)         |
| Cl2  | 6235.6(7) | 6388.6(3) | 7716.0(5) | 81.5(3)         |
| Cl3  | 7529.3(6) | 6340.1(4) | 6040.3(8) | 105.9(4)        |

|     |            |            |            |           |
|-----|------------|------------|------------|-----------|
| C14 | 5501.3(6)  | 6990.6(2)  | 6213.7(5)  | 68.6(2)   |
| C15 | 4373.1(6)  | 6049.8(3)  | 6759.8(8)  | 94.8(3)   |
| P2  | 4344.9(4)  | 7696.6(2)  | 3833.6(4)  | 41.30(14) |
| O2  | 3700.1(14) | 7314.6(6)  | 2383.9(12) | 63.6(5)   |
| C22 | 4512.7(16) | 8086.6(7)  | 4575.5(13) | 42.9(5)   |
| C23 | 4381(2)    | 7983.8(9)  | 5336.9(16) | 66.8(8)   |
| C24 | 4528(3)    | 8282.6(11) | 5900.7(18) | 84.1(11)  |
| C25 | 4799(2)    | 8685.1(10) | 5708.2(18) | 72.2(9)   |
| C26 | 4928(2)    | 8788.1(9)  | 4957.5(18) | 62.9(8)   |
| C27 | 4788.3(18) | 8493.3(8)  | 4385.0(16) | 53.5(6)   |
| C28 | 5326.3(16) | 7416.7(8)  | 3677.3(14) | 43.8(5)   |
| C29 | 5799.3(17) | 7285.4(9)  | 4301.6(16) | 53.2(6)   |
| C30 | 6552.3(18) | 7065.7(9)  | 4200.7(18) | 59.6(7)   |
| C31 | 6846.2(19) | 6983.8(10) | 3476.3(19) | 62.5(8)   |
| C32 | 6388(2)    | 7120.1(12) | 2857.2(19) | 77.0(10)  |
| C33 | 5631(2)    | 7334.8(10) | 2944.4(17) | 65.3(8)   |
| C34 | 3514.1(15) | 7342.1(7)  | 4158.3(14) | 41.7(5)   |
| C35 | 3707.3(17) | 6977.3(8)  | 4570.4(16) | 50.1(6)   |
| C36 | 3061.4(19) | 6726.6(9)  | 4861.4(17) | 56.5(7)   |
| C37 | 2226.0(19) | 6834.6(9)  | 4728.0(17) | 56.7(7)   |
| C38 | 2027.5(18) | 7194.9(9)  | 4311.6(17) | 55.1(7)   |
| C39 | 2672.0(17) | 7451.6(8)  | 4035.5(15) | 51.8(6)   |
| C40 | 4012.9(17) | 7976.3(8)  | 2984.6(14) | 47.3(6)   |
| C41 | 3620.9(17) | 7698.6(9)  | 2367.2(14) | 47.8(6)   |

|     |            |            |            |           |
|-----|------------|------------|------------|-----------|
| C42 | 3164(2)    | 7927.9(11) | 1753.6(19) | 72.9(9)   |
| P1  | 3130.2(4)  | 4768.4(2)  | 6807.7(4)  | 42.20(14) |
| O1  | 4272.9(15) | 4612.5(7)  | 8169.2(13) | 71.3(6)   |
| C1  | 4010.8(16) | 4521.5(8)  | 6334.1(14) | 45.8(6)   |
| C2  | 4716.2(18) | 4769.6(9)  | 6163.8(17) | 56.8(7)   |
| C3  | 5352(2)    | 4609.0(11) | 5709(2)    | 70.7(9)   |
| C4  | 5288(2)    | 4201.2(12) | 5416.4(18) | 71.5(9)   |
| C5  | 4599(2)    | 3953.2(10) | 5583.4(18) | 65.2(8)   |
| C6  | 3955.3(19) | 4108.6(9)  | 6043.7(17) | 56.5(7)   |
| C7  | 2429.9(16) | 4378.9(7)  | 7217.6(14) | 44.3(5)   |
| C8  | 2750(2)    | 4029.5(9)  | 7621.2(17) | 60.2(7)   |
| C9  | 2198(2)    | 3738.5(10) | 7937.5(18) | 68.3(9)   |
| C10 | 1336(2)    | 3789.2(10) | 7864.2(18) | 64.5(8)   |
| C11 | 1013.0(19) | 4132.6(10) | 7482.7(19) | 61.7(7)   |
| C12 | 1554.4(17) | 4427.2(8)  | 7154.5(16) | 52.2(6)   |
| C13 | 2591.4(16) | 5072.0(8)  | 6076.4(14) | 45.8(5)   |
| C14 | 2249(2)    | 5468.6(9)  | 6229.6(16) | 59.1(7)   |
| C15 | 1897(2)    | 5705.3(11) | 5644.8(19) | 73.3(9)   |
| C16 | 1899(2)    | 5555.6(12) | 4914.1(19) | 79.9(10)  |
| C17 | 2240(2)    | 5168.8(14) | 4755.6(19) | 91.1(13)  |
| C18 | 2581(2)    | 4921.1(11) | 5334.9(18) | 74.1(9)   |
| C19 | 3489.8(17) | 5147.3(8)  | 7512.2(16) | 50.1(6)   |
| C20 | 4130.2(18) | 4986.8(11) | 8086.0(16) | 57.1(7)   |

|     |         |            |         |           |
|-----|---------|------------|---------|-----------|
| C21 | 4561(3) | 5325.2(14) | 8542(2) | 102.0(14) |
|-----|---------|------------|---------|-----------|

**Table S2. Anisotropic Displacement Parameters ( $\text{\AA}^2 \times 10^3$ ) for (ATPPCl)SbCl<sub>5</sub>. The Anisotropic displacement factor exponent takes the form:  $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\dots]$ .**

| Atom | $U_{11}$  | $U_{22}$  | $U_{33}$  | $U_{23}$  | $U_{13}$ | $U_{12}$  |
|------|-----------|-----------|-----------|-----------|----------|-----------|
| Sb1  | 47.62(10) | 44.67(10) | 43.84(10) | -2.93(7)  | 1.21(7)  | -3.63(7)  |
| Cl1  | 83.3(6)   | 49.9(4)   | 101.5(7)  | -6.6(4)   | -20.1(5) | 11.0(4)   |
| Cl2  | 116.8(7)  | 79.9(6)   | 47.9(4)   | -9.0(4)   | -12.3(4) | 19.1(5)   |
| Cl3  | 61.4(5)   | 109.8(8)  | 146.5(10) | -26.9(7)  | 30.3(6)  | -25.0(5)  |
| Cl4  | 88.8(5)   | 51.9(4)   | 64.9(5)   | 16.6(3)   | 11.3(4)  | -0.6(4)   |
| Cl5  | 61.6(5)   | 55.6(4)   | 167.2(10) | 13.6(6)   | 31.9(6)  | -5.8(4)   |
| P2   | 52.2(3)   | 37.3(3)   | 34.4(3)   | -1.1(2)   | 3.2(3)   | -1.0(3)   |
| O2   | 81.9(14)  | 49.4(11)  | 59.7(12)  | -10.0(9)  | -4.9(10) | -0.9(10)  |
| C22  | 51.5(14)  | 39.1(12)  | 38.0(12)  | -4.1(10)  | 2.6(10)  | -1.1(10)  |
| C23  | 110(3)    | 48.3(16)  | 41.7(15)  | -0.7(12)  | 10.8(16) | -17.9(16) |
| C24  | 142(3)    | 73(2)     | 37.0(15)  | -7.3(15)  | 12.7(18) | -22(2)    |
| C25  | 101(3)    | 62.3(19)  | 53.0(17)  | -20.0(15) | 5.3(17)  | -16.1(18) |
| C26  | 82(2)     | 45.7(15)  | 61.2(18)  | -5.8(13)  | -0.1(16) | -13.0(14) |
| C27  | 66.8(17)  | 49.3(15)  | 44.5(14)  | 0.2(12)   | 3.8(12)  | -10.9(13) |
| C28  | 49.3(13)  | 40.5(12)  | 41.5(13)  | -2.6(10)  | 7.1(10)  | -3.9(10)  |

|     |          |          |          |          |           |           |
|-----|----------|----------|----------|----------|-----------|-----------|
| C29 | 56.6(16) | 57.6(16) | 45.4(14) | -4.5(12) | 0.5(12)   | 0.7(13)   |
| C30 | 55.3(16) | 58.3(17) | 65.1(18) | -1.2(14) | -6.4(14)  | -1.2(13)  |
| C31 | 51.8(16) | 58.1(17) | 78(2)    | -4.4(15) | 11.3(15)  | 2.7(13)   |
| C32 | 75(2)    | 100(3)   | 55.4(19) | -4.9(18) | 21.6(16)  | 20.2(19)  |
| C33 | 69.7(19) | 81(2)    | 45.0(15) | 1.8(15)  | 7.7(14)   | 14.1(16)  |
| C34 | 48.7(13) | 36.9(12) | 39.6(12) | -3.3(10) | 4.4(10)   | 2.1(10)   |
| C35 | 52.2(14) | 42.9(13) | 55.2(15) | 2.5(12)  | -1.1(12)  | 1.1(11)   |
| C36 | 65.1(18) | 41.8(14) | 62.5(17) | 9.8(13)  | 2.9(14)   | -1.3(12)  |
| C37 | 60.9(17) | 48.6(15) | 60.7(17) | -1.0(13) | 15.0(14)  | -7.5(13)  |
| C38 | 47.7(14) | 56.1(16) | 61.3(17) | -4.1(13) | 9.2(13)   | 5.1(12)   |
| C39 | 58.0(15) | 44.6(14) | 52.8(15) | 3.5(12)  | 7.2(12)   | 7.6(12)   |
| C40 | 61.0(15) | 42.9(13) | 38.1(13) | 0.5(10)  | -0.5(11)  | 0.7(12)   |
| C41 | 51.7(14) | 53.1(15) | 38.7(13) | -2.9(11) | 3.7(11)   | -2.3(12)  |
| C42 | 84(2)    | 72(2)    | 62.2(19) | 7.8(16)  | -22.9(17) | -11.1(17) |
| P1  | 47.0(3)  | 36.5(3)  | 43.0(3)  | 2.7(3)   | -0.1(3)   | 1.8(3)    |
| O1  | 80.5(15) | 70.2(14) | 63.2(13) | 0.5(11)  | -13.4(11) | 15.7(12)  |
| C1  | 49.2(13) | 43.4(13) | 44.9(14) | 4.3(11)  | 0.0(11)   | 5.8(11)   |
| C2  | 54.5(15) | 53.9(16) | 62.1(17) | -1.6(13) | 5.4(13)   | -0.4(13)  |
| C3  | 57.8(18) | 80(2)    | 74(2)    | 5.4(18)  | 14.2(16)  | 2.8(16)   |
| C4  | 70(2)    | 85(2)    | 59.3(19) | 1.5(17)  | 12.0(16)  | 27.6(18)  |
| C5  | 78(2)    | 54.1(16) | 63.6(19) | -6.5(14) | 3.0(16)   | 17.0(16)  |
| C6  | 63.2(17) | 43.6(14) | 62.8(17) | 1.4(13)  | 2.6(14)   | 4.3(13)   |
| C7  | 51.3(14) | 38.6(12) | 43.0(13) | -0.4(10) | 2.1(11)   | -1.5(11)  |

|     |          |          |          |           |           |           |
|-----|----------|----------|----------|-----------|-----------|-----------|
| C8  | 60.1(17) | 58.7(17) | 61.7(18) | 14.9(14)  | 5.8(14)   | 6.2(13)   |
| C9  | 89(2)    | 54.7(17) | 61.5(19) | 19.1(14)  | 17.2(17)  | 10.4(16)  |
| C10 | 76(2)    | 54.9(17) | 63.0(19) | -1.4(15)  | 22.3(16)  | -12.5(15) |
| C11 | 55.3(16) | 60.9(17) | 68.9(19) | -4.2(15)  | 7.4(14)   | -8.5(14)  |
| C12 | 52.3(15) | 44.1(14) | 60.2(16) | -0.7(12)  | -2.4(13)  | 0.5(11)   |
| C13 | 47.1(13) | 46.0(13) | 44.2(13) | 4.9(11)   | -0.1(11)  | 3.8(11)   |
| C14 | 81(2)    | 49.6(15) | 46.9(15) | 7.3(12)   | 0.3(14)   | 12.1(14)  |
| C15 | 95(2)    | 63.1(19) | 61.6(19) | 14.1(16)  | -3.6(18)  | 24.5(17)  |
| C16 | 87(2)    | 98(3)    | 54.8(19) | 22.3(18)  | -0.2(17)  | 33(2)     |
| C17 | 98(3)    | 127(3)   | 48.3(18) | -13(2)    | -14.6(18) | 48(2)     |
| C18 | 83(2)    | 82(2)    | 56.8(18) | -14.3(16) | -12.1(16) | 34.1(18)  |
| C19 | 56.6(15) | 41.4(13) | 52.4(15) | -2.5(12)  | 2.0(12)   | -2.9(11)  |
| C20 | 55.7(16) | 68.6(19) | 47.1(15) | -5.9(14)  | -0.9(12)  | -1.9(14)  |
| C21 | 112(3)   | 100(3)   | 94(3)    | -20(2)    | -41(3)    | -16(3)    |

**Table S3. Bond Lengths for (ATPPCl)SbCl<sub>5</sub>.**

| Atom | Atom | Length (Å) | Atom | Atom | Length (Å) |
|------|------|------------|------|------|------------|
| Sb1  | Cl1  | 2.5891(8)  | C40  | C41  | 1.515(3)   |
| Sb1  | Cl2  | 2.3745(8)  | C41  | C42  | 1.475(4)   |
| Sb1  | Cl3  | 2.5689(9)  | P1   | C1   | 1.787(3)   |
| Sb1  | Cl4  | 2.6398(8)  | P1   | C7   | 1.792(3)   |
| Sb1  | Cl5  | 2.6248(8)  | P1   | C13  | 1.802(2)   |
| P2   | C22  | 1.800(2)   | P1   | C19  | 1.800(3)   |

|     |     |          |     |     |          |
|-----|-----|----------|-----|-----|----------|
| P2  | C28 | 1.794(3) | O1  | C20 | 1.204(4) |
| P2  | C34 | 1.805(2) | C1  | C2  | 1.386(4) |
| P2  | C40 | 1.798(2) | C1  | C6  | 1.393(4) |
| O2  | C41 | 1.211(3) | C2  | C3  | 1.371(4) |
| C22 | C23 | 1.382(4) | C3  | C4  | 1.381(5) |
| C22 | C27 | 1.387(3) | C4  | C5  | 1.365(5) |
| C23 | C24 | 1.377(4) | C5  | C6  | 1.379(4) |
| C24 | C25 | 1.374(5) | C7  | C8  | 1.396(4) |
| C25 | C26 | 1.363(4) | C7  | C12 | 1.387(4) |
| C26 | C27 | 1.378(4) | C8  | C9  | 1.375(4) |
| C28 | C29 | 1.381(4) | C9  | C10 | 1.369(5) |
| C28 | C33 | 1.389(4) | C10 | C11 | 1.364(4) |
| C29 | C30 | 1.380(4) | C11 | C12 | 1.380(4) |
| C30 | C31 | 1.369(4) | C13 | C14 | 1.382(4) |
| C31 | C32 | 1.366(5) | C13 | C18 | 1.377(4) |
| C32 | C33 | 1.374(4) | C14 | C15 | 1.377(4) |
| C34 | C35 | 1.385(3) | C15 | C16 | 1.358(5) |
| C34 | C39 | 1.383(4) | C16 | C17 | 1.355(5) |
| C35 | C36 | 1.380(4) | C17 | C18 | 1.382(4) |
| C36 | C37 | 1.375(4) | C19 | C20 | 1.505(4) |
| C37 | C38 | 1.379(4) | C20 | C21 | 1.489(5) |
| C38 | C39 | 1.380(4) |     |     |          |

**Table 4 Bond Angles for (ATPPCl)SbCl<sub>5</sub>.**

| Atom | Atom | Atom | Atom | Angle (°) | Atom | Atom | Atom | Atom | Angle (°) |
|------|------|------|------|-----------|------|------|------|------|-----------|
| P2   | C22  | C23  | C24  | 178.7(3)  | P1   | C1   | C2   | C3   | -170.9(2) |

|     |     |     |     |             |    |     |     |     |           |
|-----|-----|-----|-----|-------------|----|-----|-----|-----|-----------|
| P2  | C22 | C27 | C26 | -179.0(2)   | P1 | C1  | C6  | C5  | 170.3(2)  |
| P2  | C28 | C29 | C30 | 179.3(2)    | P1 | C7  | C8  | C9  | 179.2(2)  |
| P2  | C28 | C33 | C32 | 179.8(3)    | P1 | C7  | C12 | C11 | -178.7(2) |
| P2  | C34 | C35 | C36 | 175.3(2)    | P1 | C13 | C14 | C15 | 175.2(3)  |
| P2  | C34 | C39 | C38 | -176.9(2)   | P1 | C13 | C18 | C17 | -174.0(3) |
| P2  | C40 | C41 | O2  | -15.1(3)    | P1 | C19 | C20 | O1  | -13.9(4)  |
| P2  | C40 | C41 | C42 | 166.1(2)    | P1 | C19 | C20 | C21 | 166.7(3)  |
| C22 | P2  | C28 | C29 | 43.9(2)     | C1 | P1  | C7  | C8  | 44.1(3)   |
| C22 | P2  | C28 | C33 | -135.0(2)   | C1 | P1  | C7  | C12 | -137.6(2) |
| C22 | P2  | C34 | C35 | -90.2(2)    | C1 | P1  | C13 | C14 | -141.4(2) |
| C22 | P2  | C34 | C39 | 85.6(2)     | C1 | P1  | C13 | C18 | 33.3(3)   |
| C22 | P2  | C40 | C41 | -164.60(19) | C1 | P1  | C19 | C20 | -52.2(2)  |
| C22 | C23 | C24 | C25 | 0.6(6)      | C1 | C2  | C3  | C4  | 0.4(5)    |
| C23 | C22 | C27 | C26 | 0.1(4)      | C2 | C1  | C6  | C5  | -0.7(4)   |
| C23 | C24 | C25 | C26 | -0.4(6)     | C2 | C3  | C4  | C5  | -0.8(5)   |
| C24 | C25 | C26 | C27 | 0.0(6)      | C3 | C4  | C5  | C6  | 0.4(5)    |
| C25 | C26 | C27 | C22 | 0.1(5)      | C4 | C5  | C6  | C1  | 0.4(5)    |
| C27 | C22 | C23 | C24 | -0.4(5)     | C6 | C1  | C2  | C3  | 0.3(4)    |
| C28 | P2  | C22 | C23 | -90.0(3)    | C7 | P1  | C1  | C2  | -163.4(2) |
| C28 | P2  | C22 | C27 | 89.2(2)     | C7 | P1  | C1  | C6  | 25.5(3)   |
| C28 | P2  | C34 | C35 | 28.4(2)     | C7 | P1  | C13 | C14 | 98.1(2)   |
| C28 | P2  | C34 | C39 | -155.7(2)   | C7 | P1  | C13 | C18 | -87.2(3)  |
| C28 | P2  | C40 | C41 | 76.8(2)     | C7 | P1  | C19 | C20 | 72.8(2)   |
| C28 | C29 | C30 | C31 | 1.4(4)      | C7 | C8  | C9  | C10 | -0.4(5)   |
| C29 | C28 | C33 | C32 | 1.0(5)      | C8 | C7  | C12 | C11 | -0.4(4)   |
| C29 | C30 | C31 | C32 | -0.2(5)     | C8 | C9  | C10 | C11 | -0.7(5)   |
| C30 | C31 | C32 | C33 | -0.6(5)     | C9 | C10 | C11 | C12 | 1.3(5)    |

|     |     |     |     |           |     |     |     |     |           |
|-----|-----|-----|-----|-----------|-----|-----|-----|-----|-----------|
| C31 | C32 | C33 | C28 | 0.2(5)    | C10 | C11 | C12 | C7  | -0.7(5)   |
| C33 | C28 | C29 | C30 | -1.8(4)   | C12 | C7  | C8  | C9  | 0.9(4)    |
| C34 | P2  | C22 | C23 | 30.7(3)   | C13 | P1  | C1  | C2  | 76.5(2)   |
| C34 | P2  | C22 | C27 | -150.2(2) | C13 | P1  | C1  | C6  | -94.6(2)  |
| C34 | P2  | C28 | C29 | -74.2(2)  | C13 | P1  | C7  | C8  | 161.0(2)  |
| C34 | P2  | C28 | C33 | 107.0(2)  | C13 | P1  | C7  | C12 | -20.7(3)  |
| C34 | P2  | C40 | C41 | -47.7(2)  | C13 | P1  | C19 | C20 | -166.3(2) |
| C34 | C35 | C36 | C37 | 1.4(4)    | C13 | C14 | C15 | C16 | -1.3(5)   |
| C35 | C34 | C39 | C38 | -1.0(4)   | C14 | C13 | C18 | C17 | 0.8(5)    |
| C35 | C36 | C37 | C38 | -0.7(4)   | C14 | C15 | C16 | C17 | 0.7(6)    |
| C36 | C37 | C38 | C39 | -0.8(4)   | C15 | C16 | C17 | C18 | 0.7(6)    |
| C37 | C38 | C39 | C34 | 1.7(4)    | C16 | C17 | C18 | C13 | -1.4(6)   |
| C39 | C34 | C35 | C36 | -0.5(4)   | C18 | C13 | C14 | C15 | 0.5(5)    |
| C40 | P2  | C22 | C23 | 149.6(3)  | C19 | P1  | C1  | C2  | -38.3(3)  |
| C40 | P2  | C22 | C27 | -31.3(3)  | C19 | P1  | C1  | C6  | 150.7(2)  |
| C40 | P2  | C28 | C29 | 162.0(2)  | C19 | P1  | C7  | C8  | -80.6(2)  |
| C40 | P2  | C28 | C33 | -16.8(3)  | C19 | P1  | C7  | C12 | 97.6(2)   |
| C40 | P2  | C34 | C35 | 152.7(2)  | C19 | P1  | C13 | C14 | -23.4(3)  |
| C40 | P2  | C34 | C39 | -31.5(2)  | C19 | P1  | C13 | C18 | 151.2(3)  |

**Table S5. Hydrogen Atom Coordinates ( $\text{\AA}\times 10^4$ ) and Isotropic Displacement Parameters ( $\text{\AA}^2\times 10^3$ ) for (ATPPCl)SbCl<sub>5</sub>.**

| Atom | <i>x</i> | <i>y</i> | <i>z</i> | <i>U</i> <sub>eq</sub> |
|------|----------|----------|----------|------------------------|
| H23  | 4192.28  | 7712.49  | 5468.96  | 80                     |

|      |         |         |         |     |
|------|---------|---------|---------|-----|
| H24  | 4442.93 | 8211.66 | 6412.98 | 101 |
| H25  | 4895.04 | 8886.87 | 6089.39 | 87  |
| H26  | 5111.75 | 9060.88 | 4830.01 | 75  |
| H27  | 4878.43 | 8566.42 | 3874.45 | 64  |
| H29  | 5608.66 | 7345.54 | 4794.87 | 64  |
| H30  | 6860.18 | 6973.17 | 4624.89 | 72  |
| H31  | 7354.22 | 6836.4  | 3406.22 | 75  |
| H32  | 6592.74 | 7066.4  | 2365.95 | 92  |
| H33  | 5326.53 | 7424.44 | 2516.43 | 78  |
| H35  | 4272.7  | 6901.15 | 4651.06 | 60  |
| H36  | 3191.05 | 6484.88 | 5147.46 | 68  |
| H37  | 1791.78 | 6663.49 | 4919.84 | 68  |
| H38  | 1461.43 | 7264.61 | 4217.23 | 66  |
| H39  | 2539.72 | 7698.79 | 3766.19 | 62  |
| H40A | 3601.7  | 8192.34 | 3130.66 | 57  |
| H40B | 4502.93 | 8121.69 | 2769.37 | 57  |
| H42A | 2655.86 | 8053.45 | 1959.34 | 109 |
| H42B | 3016.13 | 7731.66 | 1352.64 | 109 |
| H42C | 3524.21 | 8147.75 | 1549.36 | 109 |
| H2   | 4758.03 | 5045.01 | 6357.86 | 68  |
| H3   | 5827.08 | 4774.89 | 5598.12 | 85  |
| H4   | 5718.14 | 4094.82 | 5103.26 | 86  |

|      |         |         |         |     |
|------|---------|---------|---------|-----|
| H5   | 4562.33 | 3678.36 | 5386.05 | 78  |
| H6   | 3487.1  | 3938.83 | 6159.46 | 68  |
| H8   | 3335.19 | 3993.84 | 7675.96 | 72  |
| H9   | 2411.49 | 3504.91 | 8203.78 | 82  |
| H10  | 968.49  | 3588.49 | 8075.63 | 77  |
| H11  | 426.64  | 4168.35 | 7443.71 | 74  |
| H12  | 1331.5  | 4659.2  | 6889.78 | 63  |
| H14  | 2255.54 | 5575.56 | 6727.07 | 71  |
| H15  | 1656.81 | 5969.79 | 5751.08 | 88  |
| H16  | 1665.15 | 5718.83 | 4521.62 | 96  |
| H17  | 2244.56 | 5069.2  | 4253.14 | 109 |
| H18  | 2802.9  | 4653.22 | 5224.04 | 89  |
| H19A | 3737.27 | 5388.54 | 7244.82 | 60  |
| H19B | 2996.86 | 5251.09 | 7790.73 | 60  |
| H21A | 4902.87 | 5195.68 | 8935.27 | 153 |
| H21B | 4140.75 | 5505.93 | 8774.73 | 153 |

**Table S5. SCXRD data of (ATPPO)<sub>2</sub>SbCl<sub>5</sub> from 100 K**

| Formula           | (ATPPO) <sub>2</sub> SbCl <sub>5</sub>                                          |
|-------------------|---------------------------------------------------------------------------------|
| CCDC number       | /                                                                               |
| Empirical formula | C <sub>42</sub> H <sub>40</sub> Cl <sub>5</sub> SbP <sub>2</sub> O <sub>2</sub> |
| Formula weight    | 937.68                                                                          |

|                                           |                                        |
|-------------------------------------------|----------------------------------------|
| Temperature (K)                           | 100                                    |
| Crystal system                            | orthorhombic                           |
| Space group                               | Pbca                                   |
| $a$ (Å)                                   | 15.3858(11)                            |
| $b$ (Å)                                   | 31.184(3)                              |
| $c$ (Å)                                   | 17.3400(13)                            |
| Volume (Å <sup>3</sup> )                  | 8319.4(11)                             |
| $Z$                                       | 8                                      |
| $\rho_{\text{calc}}$ (g/cm <sup>3</sup> ) | 1.497                                  |
| Radiation                                 | Mo K $\alpha$ ( $\lambda$ = 0.71073 Å) |
| $2\theta$ range for data collection (°)   | 4.398 to 55.076                        |
| Reflections collected                     | 141152                                 |
| Goodness-of-fit on $F^2$                  | 1.058                                  |
| $R_{\text{obs}}, wR_{\text{obs}}$         | 0.0251, 0.0583                         |
| $R_{\text{all}}, wR_{\text{all}}$         | 0.0293, 0.0604                         |