Supplementary material (ESI) for Journal of Materials Chemistry


# Supporting Information

## Effects of the order-disorder phase transition on the physical properties of $A_8Sn_{44}\square_2$ (A = Rb, Cs)

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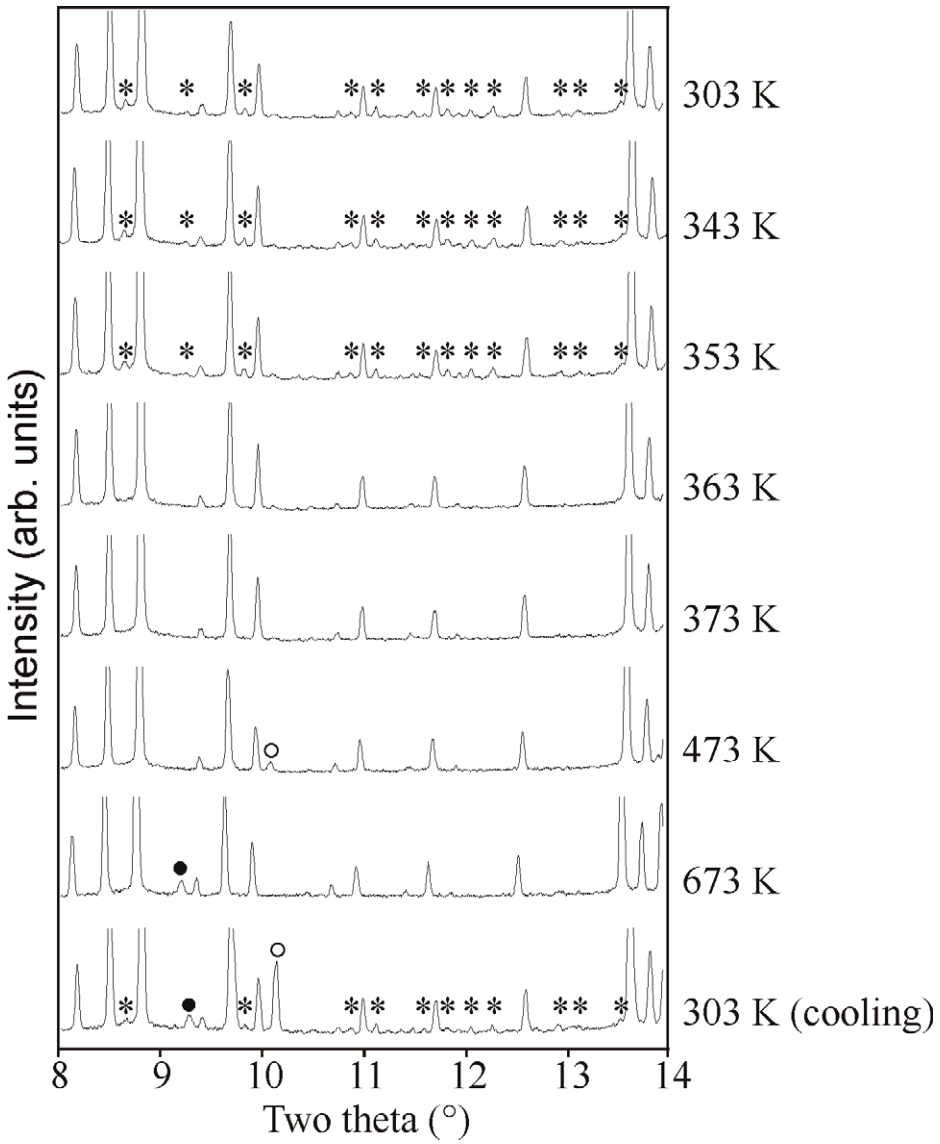


**Fig. S1** Temperature-dependent powder-diffraction diagrams of $Rb_8Sn_{44}$ ($\lambda$ = 0.499658 Å). Superstructure reflections, $\beta$-Sn and unindexed peaks are denoted as *, ○, and ●, respectively.

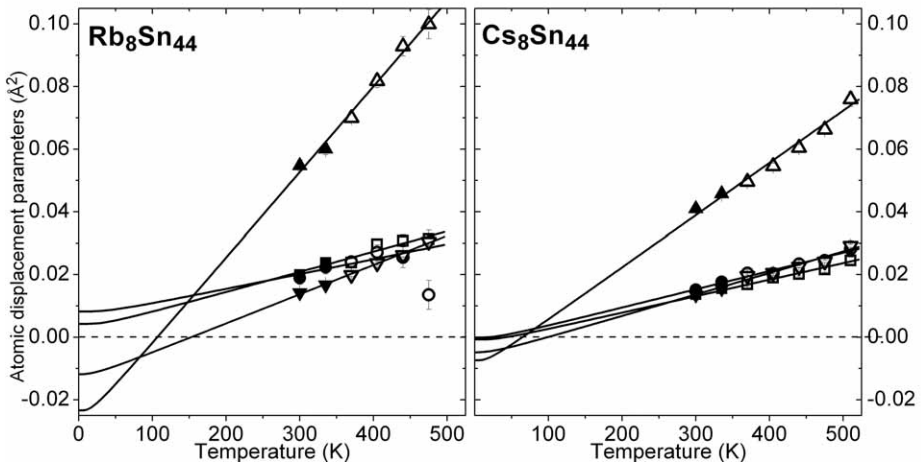


**Fig. S2** Atomic displacement parameters obtained from the data measured above room temperature. The ADPs were fitted using an Einstein model for the guest atom and a Debye model for the average host structure ADPs. (squares) $U_{iso}(2a)$, (circles) $U_{11}(6d)$, (triangles pointing up) $U_{22}(6d)$, and (triangles pointing down) host structure $U_{iso}$ average.

**Table S1**. Einstein and Debye temperature extracted from fits to the ADPs from the multi-temperature powder diffraction results.

| | T < 300 K | T > 300 K | | T < 300 K | T > 300 K | |
|---|---|---|---|---|---|---|
| | $Rb_8Sn_{44}$ | $Rb_8Sn_{44}$ | $Cs_8Sn_{44}$ | $Rb_8Sn_{44}$ | $Rb_8Sn_{44}$ | $Cs_8Sn_{44}$ |
| | $\theta_X$ (K) | $\theta_X$ (K) | $\theta_X$ (K) | $d^2$ (Å$^2$) | $d^2$ (Å$^2$) | $d^2$ (Å$^2$) |
| 2*a*(*E iso*) | 95(1) | 94(7) | 85(2) | –0.000(1) | 0.001(4) | –0.002(1) |
| 6*d*(*E*,11) | 84(1) | 109(16) | 79(3) | –0.007(1) | 0.006(5) | –0.002(2) |
| 6*d*(*E*,22) | 56(1) | 46(1) | 47(1) | 0.004(1) | –0.030(5) | –0.011(5) |
| *D*,host | 168(2) | 114(2) | 132(5) | –0.001(1) | –0.015(1) | –0.008(2) |

**Table S2.** $Rb_8Sn_{44}$: The superstructure refinement with unit cell 2a × 2a × 2a and space group $Ia\bar{3}d$: Rb1 = (0, 0, 0, $U_{iso}$), Rb2 = (1/8, 1/4, 0, $U_{11}$, $U_{22}$), Sn1 = (3/8, 0, 1/4), Sn1´ = (1/8, 0, 1/4, sof), Sn2 = (x, x, x), Sn2´ = (x, y, z), Sn3a = (x, y, z, sof), Sn3b = (x, y, z, 1-sof), Sn3´ = (x, y, z). The conventional type-I clathrate with unit cell a × a × a and space group $Pm\bar{3}n$: Rb1 = (0, 0, 0, $U_{iso}$), Rb2 = (1/4, 1/2 , 0, $U_{11}$, $U_{22}$), Sn1´ = (1/4, 0, 1/2, sof), Sn2 = (x, x, x), Sn3a = (0, y, z, sof), Sn3b = (0, y, z, 1-sof).

| Temperature | 300 K | 335 K | 370 K | 405 K | 440 K | 475 K |
|---|---|---|---|---|---|---|
| *a* (Å) | 24.1031 (4) | 24.1156 (4) | 12.0645 (1) | 12.0715 (2) | 12.0793 (1) | 12.0856 (1) |
| $U_{iso}$(Rb1) (·$10^{-4}$ Å$^2$) | 200 (14) | 238 (14) | 239 (12) | 297 (15) | 308 (19) | 313 (29) |
| $U_{11}$(Rb2) (·$10^{-4}$ Å$^2$) | 190 (21) | 223 (21) | 241 (19) | 271 (24) | 255 (33) | 135 (46) |
| $U_{22}$(Rb2) (·$10^{-4}$ Å$^2$) | 547 (18) | 601 (18) | 700 (16) | 818 (21) | 929 (31) | 999 (47) |
| $U_{iso}$(host) (·$10^{-4}$ Å$^2$) | 142 (6) | 167 (6) | 199 (5) | 236 (5) | 262 (6) | 305 (11) |
| sof(Sn1´) (%) | 34.75 (4) | 34.96 (4) | 68.47 (2) | 68.49 (3) | 68.62 (3) | 68.83 (5) |
| *x*(Sn2) | 0.0907 (1) | 0.0906 (1) | 0.1827 (1) | 0.1826 (1) | 0.1827 (1) | 0.1831 (1) |
| *x*(Sn2´) | 0.0896 (1) | 0.0897 (1) | | | | |
| *y*(Sn2´) | 0.4053 (1) | 0.4053 (1) | | | | |
| *z*(Sn2´) | 0.0904 (1) | 0.0904 (1) | | | | |
| *x*(Sn3a) | 0.0027 (6) | 0.0023 (6) | 0 | 0 | 0 | 0 |
| *y*(Sn3a) | 0.1516 (4) | 0.1517 (4) | 0.3071 (1) | 0.3071 (1) | 0.3068 (2) | 0.3062 (3) |
| *z*(Sn3a) | 0.0582 (4) | 0.0579 (4) | 0.1133 (1) | 0.1135 (2) | 0.1138 (2) | 0.1145 (3) |
| sof(Sn3a) (%) | 34.75 (4) | 34.96 (4) | 68.47 (2) | 68.49 (3) | 68.62 (3) | 68.83 (5) |
| *x*(Sn3b) | 0.0016 (4) | 0.0020 (4) | 0 | 0 | 0 | 0 |
| *y*(Sn3b) | 0.1739 (1) | 0.1739 (1) | 0.3475 (2) | 0.3477 (3) | 0.3477 (5) | 0.3486 (7) |
| *z*(Sn3b) | 0.0668 (1) | 0.0668 (1) | 0.1350 (3) | 0.1340 (3) | 0.1330 (5) | 0.1303 (7) |
| sof(Sn3b) (%) | 65.25 (4) | 65.04 (4) | 31.53 (2) | 31.51 (3) | 31.38 (3) | 31.17 (5) |
| *x*(Sn3´) | 0.4996 (2) | 0.4999 (2) | | | | |
| *y*(Sn3´) | 0.1539 (1) | 0.1540 (1) | | | | |
| *z*(Sn3´) | 0.0562 (1) | 0.0562 (1) | | | | |
| $M_{pearson-IIIV}$ | 0.79 (5) | 0.85 (5) | 0.78 (2) | 1.03 (3) | 1.23 (4) | 1.02 (9) |
| *U* | 0.049 (5) | 0.057 (5) | 0.053 (3) | 0.028 (3) | 0.012 (4) | 0.009 (5) |
| *V* | –0.0023 (9) | –0.0037 (9) | –0.0044 (7) | –0.0007 (7) | 0.0018 (8) | –0.0003 (9) |
| *W* | 0.00145 (4) | 0.00154 (4) | 0.00162 (3) | 0.00128 (3) | 0.00112 (4) | 0.00116 (4) |
| *X* | 0.055 (7) | 0.042 (8) | 0.048 (2) | 0.036 (3) | 0.019 (4) | 0.053 (8) |
| *a*(Sn) (Å) | 5.8299 (5) | 5.8336 (5) | 5.8379 (4) | 5.8430 (1) | 5.8469 (1) | 5.8508 (1) |
| *b*(Sn) (Å) | 3.1810 (3) | 3.1853 (3) | 3.1899 (2) | 3.1945 (1) | 3.1991 (1) | 3.2038 (1) |
| $U_{iso}$(Sn) (·$10^{-4}$ Å$^2$) | 93 (34) | 93 (34) | 159 (28) | 289 (12) | 343 (7) | 390 (5) |
| $R_p/R_{wp}$ | 6.5 6.2 | 6.6 6.2 | 5.9 5.2 | 7.5 6.6 | 8.9 7.8 | 9.6 7.9 |
| $R_{exp}$/Chi$^2$ | 3.7 2.8 | 3.8 2.7 | 4.0 1.7 | 4.1 2.6 | 4.2 3.5 | 4.2 3.6 |
| $R_F/R_I$ ($Rb_8Sn_{44}$) | 1.8 1.8 | 2.0 1.9 | 1.7 1.5 | 2.5 2.2 | 3.3 2.8 | 6.0 5.2 |
| $R_F/R_I$ (Sn) | 1.5 2.7 | 1.8 3.2 | 1.9 1.9 | 2.4 2.6 | 2.4 1.9 | 2.0 1.9 |
| Weight_frac ($Rb_8Sn_{44}$) (%) | 97.5 (6) | 97.4 (6) | 97.9 (5) | 91.8 (5) | 75.5 (5) | 49.3 (5) |
| Weight _frac (Sn) (%) | 2.6 (1) | 2.6 (1) | 2.1 (1) | 8.2 (1) | 24.5 (2) | 50.7 (4) |

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Parameters, Background | 78 | 46 | 78 | 46 | 69 | 46 | 69 | 46 | 69 | 46 | 69 | 46 |
| Constrains | 2 | | 2 | | 2 | | 2 | | 2 | | 2 | |

**Table S3.** $Cs_8Sn_{44}$: The superstructure refinement with unit cell 2a × 2a × 2a and space group $Ia\bar{3}d$: Cs1 = (0, 0, 0, $U_{iso}$), Cs2 = (1/8, 1/4, 0, $U_{11}$, $U_{22}$), Sn1 = (3/8, 0, 1/4), Sn1′ = (1/8, 0, 1/4, sof), Sn2 = (x, x, x), Sn2′ = (x, y, z), Sn3a = (x, y, z, sof), Sn3b = (x, y, z, 1-sof), Sn3′ = (x, y, z). The conventional type-I clathrate with unit cell a × a × a and space group $Pm\bar{3}n$: Cs1 = (0, 0, 0, $U_{iso}$), Cs2 = (1/4, 1/2 , 0, $U_{11}$, $U_{22}$), Sn1′ = (1/4, 0, 1/2, sof), Sn2 = (x, x, x), Sn3a = (0, y, z, sof), Sn3b = (0, y, z, 1-sof).

| Temperature | 300 K | 335 K | 370 K | 405 K | 440 K | 475 K | 510 K |
|---|---|---|---|---|---|---|---|
| *a* (Å) | 24.2125 (2) | 24.2234 (2) | 12.1176 (1) | 12.1233 (1) | 12.1301 (1) | 12.1381 (1) | 12.1464 (1) |
| $U_{iso}$(Cs1) (·10$^{-4}$ Å$^2$) | 137 (6) | 156 (7) | 169 (6) | 190 (7) | 202 (7) | 217 (9) | 246 (14) |
| $U_{11}$(Cs2) (·10$^{-4}$ Å$^2$) | 152 (11) | 177 (11) | 205 (11) | 203 (11) | 234 (12) | 245 (16) | 282 (26) |
| $U_{22}$(Cs2) (·10$^{-4}$ Å$^2$) | 409 (9) | 458 (10) | 496 (9) | 546 (9) | 604 (10) | 662 (14) | 759 (23) |
| $U_{iso}$(host) (·10$^{-4}$ Å$^2$) | 135 (3) | 155 (3) | 195 (3) | 206 (3) | 225 (3) | 242 (4) | 292 (5) |
| sof(Sn1′) (%) | 33.61 (4) | 33.86 (4) | 69.41 (2) | 69.57 (2) | 70.20 (2) | 70.47 (3) | 71.88 (5) |
| *x*(Sn2) | 0.0907 (1) | 0.0907 (1) | 0.1830 (1) | 0.1831 (1) | 0.1831 (1) | 0.1831 (1) | 0.1827 (1) |
| *x*(Sn2′) | 0.0897 (1) | 0.0897 (1) | | | | | |
| *y*(Sn2′) | 0.4049 (1) | 0.4048 (1) | | | | | |
| *z*(Sn2′) | 0.0906 (1) | 0.0905 (1) | | | | | |
| *x*(Sn3a) | 0.0025 (5) | 0.0025 (5) | 0 | 0 | 0 | 0 | 0 |
| *y*(Sn3a) | 0.1525 (3) | 0.1524 (3) | 0.3074 (1) | 0.3073 (1) | 0.3074 (1) | 0.3073 (1) | 0.3071 (2) |
| *z*(Sn3a) | 0.0578 (4) | 0.0580 (4) | 0.1140 (1) | 0.1138 (1) | 0.1140 (1) | 0.1143 (2) | 0.1142 (3) |
| sof(Sn3a) (%) | 33.61 (4) | 33.86 (4) | 69.41 (2) | 69.57 (2) | 70.20 (2) | 70.47 (3) | 71.88 (5) |
| *x*(Sn3b) | 0.0020 (3) | 0.0018 (3) | 0 | 0 | 0 | 0 | 0 |
| *y*(Sn3b) | 0.1725 (1) | 0.1725 (1) | 0.3447 (2) | 0.3443 (2) | 0.3444 (3) | 0.3447 (4) | 0.3438 (6) |
| *z*(Sn3b) | 0.0671 (1) | 0.0669 (1) | 0.1353 (3) | 0.1357 (3) | 0.1355 (3) | 0.1348 (4) | 0.1363 (7) |
| sof(Sn3b) (%) | 66.39 (4) | 66.14 (4) | 30.59 (2) | 30.43 (2) | 29.80 (2) | 29.53 (3) | 28.12 (5) |
| *x*(Sn3′) | 0.4995 (1) | 0.4995 (2) | | | | | |
| *y*(Sn3′) | 0.1535 (1) | 0.1536 (1) | | | | | |
| *z*(Sn3′) | 0.0564 (1) | 0.0565 (1) | | | | | |
| $M_{\text{pearson-IIIV}}$ | 1.09 (3) | 1.11 (3) | 1.00 (2) | 0.97 (2) | 1.05 (3) | 1.15 (4) | 1.87 (5) |
| *U* | 0.021 (2) | 0.020 (2) | 0.016 (2) | 0.017 (2) | 0.017 (2) | 0.013 (2) | 0.009 (2) |
| *V* | 0.0000 (5) | 0.0004 (5) | –0.0009 (4) | –0.0012 (4) | –0.0011 (4) | –0.0010 (4) | –0.0003 (6) |
| *W* | 0.00110 (2) | 0.00108 (3) | 0.00114 (2) | 0.00116 (2) | 0.00112 (2) | 0.00107 (2) | 0.00084 (3) |
| *X* | 0.032 (2) | 0.031 (2) | 0.035 (2) | 0.041 (2) | 0.042 (2) | 0.042 (4) | –0.022 (3) |
| *a*(Sn) (Å) | 5.8311 (7) | 5.8353 (7) | 5.8402 (7) | 5.8446 (7) | 5.8480 (3) | 5.8515 (1) | |
| *b*(Sn) (Å) | 3.1805 (5) | 3.1846 (5) | 3.1891 (5) | 3.1937 (4) | 3.2002 (2) | 3.2048 (1) | |
| $U_{iso}$(Sn) (·10$^{-4}$ Å$^2$) | 257 (94) | 335 (110) | 650 (171) | 609 (157) | 362 (47) | 383 (12) | |
| $R_p/R_{wp}$ | 7.1 7.4 | 7.4 7.4 | 7.1 6.7 | 7.2 6.8 | 7.6 7.0 | 9.0 8.6 | 13.5 14.1 |
| $R_{exp}$/Chi$^2$ | 2.9 6.6 | 3.3 5.1 | 3.4 3.8 | 3.5 3.7 | 3.5 4.0 | 3.7 5.5 | 4.7 9.2 |
| $R_F/R_I$ ($Cs_8Sn_{44}$) | 2.8 2.5 | 3.1 2.6 | 3.3 2.1 | 3.6 2.2 | 4.0 2.4 | 4.8 3.0 | 9.0 5.7 |
| $R_F/R_I$ (Sn) | 2.7 4.3 | 2.8 4.1 | 3.6 4.3 | 4.3 3.6 | 5.5 6.0 | 5.0 4.5 | |
| Weight_frac ($Cs_8Sn_{44}$) (%) | 99.3 (4) | 99.3 (4) | 99.2 (4) | 99.2 (4) | 98.4 (4) | 91.2 (5) | |
| Weight _frac (Sn) (%) | 0.7 (1) | 0.7 (1) | 0.8 (1) | 0.8 (1) | 1.6 (1) | 8.9 (1) | |

| | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Parameters, Background | 78 | 46 | 78 | 46 | 69 | 46 | 69 | 46 | 69 | 46 | 69 | 46 | 67 | 46 | | |
| Constrains | 2 | | 2 | | 2 | | 2 | | 2 | | 2 | | 2 | | | |

**Table S4.** Low temperature data from 90 to 300 K for $Rb_8Sn_{44}$: The superstructure refinement with unit cell 2a × 2a × 2a and space group $Ia\bar{3}d$: Rb1 = (0, 0, 0, $U_{iso}$), Rb2 = (1/8, 1/4, 0, $U_{11}$, $U_{22}$), Sn1 = (3/8, 0, 1/4), Sn1´ = (1/8, 0, 1/4, sof), Sn2 = (x, x, x), Sn2´ = (x, y, z), Sn3a = (x, y, z, sof), Sn3b = (x, y, z, 1-sof), Sn3´ = (x, y, z).

| Temperature | 90 K | 105 K | 120 K | 135 K | 150 K | 165 K | 180 K | 195K |
|---|---|---|---|---|---|---|---|---|
| *a* (Å) | 24.0522 (2) | 24.0563 (2) | 24.0604 (2) | 24.0646 (2) | 24.0683 (2) | 24.0726 (2) | 24.0768 (2) | 24.0809(3) |
| $U_{iso}$(Rb1) ($\cdot 10^{-4}$ Å$^2$) | 61 (8) | 61 (9) | 86 (9) | 85 (9) | 96 (9) | 107 (10) | 114 (10) | 126(10) |
| $U_{11}$(Rb2) ($\cdot 10^{-4}$ Å$^2$) | 9 (12) | 15 (13) | 42 (14) | 41 (14) | 62 (14) | 76 (14) | 77 (15) | 85(15) |
| $U_{22}$(Rb2) ($\cdot 10^{-4}$ Å$^2$) | 203 (11) | 235 (12) | 263 (12) | 297 (12) | 327 (12) | 349 (13) | 382 (13) | 396(13) |
| $U_{iso}$(host) ($\cdot 10^{-4}$ Å$^2$) | 39 (3) | 42 (4) | 52 (4) | 55 (4) | 64 (4) | 67 (4) | 71 (4) | 77(4) |
| sof(Sn1´) (%) | 31.67 (7) | 31.64 (7) | 31.37 (7) | 31.24 (7) | 31.11 (7) | 30.82 (7) | 31.00 (7) | 31.34(7) |
| *x*(Sn2) | 0.0908 (1) | 0.0908 (1) | 0.0908 (1) | 0.0907 (1) | 0.0908 (1) | 0.0907 (1) | 0.0908 (1) | 0.0907(1) |
| *x*(Sn2´) | 0.0895 (1) | 0.0896 (1) | 0.0895 (1) | 0.0896 (1) | 0.0896 (1) | 0.0896 (1) | 0.0896 (1) | 0.0896(1) |
| *y*(Sn2´) | 0.4054 (1) | 0.4053 (1) | 0.4053 (1) | 0.4054 (1) | 0.4053 (1) | 0.4053 (1) | 0.4054 (1) | 0.4053(1) |
| *z*(Sn2´) | 0.0904 (1) | 0.0903 (1) | 0.0903 (1) | 0.0904 (1) | 0.0903 (1) | 0.0904 (1) | 0.0903 (1) | 0.0904(1) |
| *x*(Sn3a) | 0.00323 (3) | 0.00296 (1) | 0.00297 (1) | 0.00318 (1) | 0.00319 (1) | 0.00308 (1) | 0.00321 (3) | 0.00319(1) |
| *y*(Sn3a) | 0.15193 (1) | 0.15187 (1) | 0.15192 (1) | 0.15201 (1) | 0.15209 (3) | 0.15189 (3) | 0.15198 (1) | 0.15167(1) |
| *z*(Sn3a) | 0.05766 (1) | 0.05777 (1) | 0.05773 (1) | 0.05788 (1) | 0.05767 (1) | 0.05789 (1) | 0.05782 (1) | 0.05783(1) |
| sof(Sn3a) (%) | 31.67 (7) | 31.64 (7) | 31.37 (7) | 31.24 (7) | 31.11 (7) | 30.82 (7) | 31.00 (7) | 31.34(7) |
| *x*(Sn3b) | 0.0012 (2) | 0.0014 (2) | 0.0014 (2) | 0.0012 (2) | 0.0013 (2) | 0.0014 (2) | 0.0012 (2) | 0.0013(2) |
| *y*(Sn3b) | 0.1740 (1) | 0.1740 (1) | 0.1741 (1) | 0.1740 (1) | 0.1741 (1) | 0.1740 (1) | 0.1740 (1) | 0.1739(1) |
| *z*(Sn3b) | 0.0671 (1) | 0.0671 (1) | 0.0671 (1) | 0.0671 (1) | 0.0671 (1) | 0.0671 (1) | 0.0670 (1) | 0.0669(1) |
| sof(Sn3b) (%) | 68.33 (7) | 68.36 (7) | 68.63 (7) | 68.76 (7) | 68.89 (7) | 69.18 (7) | 69.00 (7) | 68.66(7) |
| *x*(Sn3´) | 0.4995 (1) | 0.4995 (1) | 0.4996 (1) | 0.4995 (1) | 0.4995 (1) | 0.4996 (1) | 0.4995 (1) | 0.4996(1) |
| *y*(Sn3´) | 0.1540 (1) | 0.1541 (1) | 0.1541 (1) | 0.1540 (1) | 0.1540 (1) | 0.1540 (1) | 0.1540 (1) | 0.1540(1) |
| *z*(Sn3´) | 0.0560 (1) | 0.0560 (1) | 0.0560 (1) | 0.0560 (1) | 0.0560 (1) | 0.0560 (1) | 0.0560 (1) | 0.0560(1) |
| $M_{\text{pearson-IIIV}}$ | 0.939 (15) | 0.856 (15) | 0.861 (15) | 0.844 (15) | 0.873 (15) | 0.841 (14) | 0.848 (14) | 0.832(14) |
| *U* | 0.0340 (12) | 0.0281 (13) | 0.0269 (13) | 0.0268 (13) | 0.0289 (13) | 0.0283 (13) | 0.0308 (14) | 0.0305(14) |
| *V* | 0.0001 (3) | 0.0012 (3) | 0.0015 (3) | 0.0015 (3) | 0.0009 (3) | 0.0012 (3) | 0.0008 (3) | 0.0008(3) |
| *W* | 0.00124 (2) | 0.00120 (2) | 0.00118 (2) | 0.00117 (2) | 0.00121 (2) | 0.00118 (2) | 0.00119 (2) | 0.00119(2) |
| *X* | 0.0226 (27) | 0.0378 (27) | 0.0391 (26) | 0.0404 (25) | 0.0343 (26) | 0.0413 (25) | 0.0397 (24) | 0.0420(24) |
| Y | 0.0005 (1) | –0.0001 (1) | –0.0003 (1) | –0.0003 (1) | 0.0000 (1) | –0.0004 (1) | –0.0003 (1) | –0.0004(1) |
| $R_p/R_{wp}$ | 6.06 6.42 | 6.31 6.84 | 6.31 6.80 | 6.24 6.75 | 6.09 6.65 | 6.17 6.63 | 6.13 6.62 | 6.21 6.66 |
| $R_{exp}/Chi^2$ | 2.37 7.32 | 2.42 7.99 | 2.45 7.69 | 2.44 7.64 | 2.46 7.32 | 2.47 7.21 | 2.48 7.11 | 2.49 7.13 |
| $R_F/R_I$ ($Rb_8Sn_{44}$) | 1.68 1.87 | 1.67 1.82 | 1.63 1.78 | 1.62 1.81 | 1.66 1.74 | 1.55 1.72 | 1.68 1.77 | 1.69 1.83 |
| Parameters, Background | 82 52 | 82 52 | 82 52 | 82 52 | 82 52 | 82 52 | 82 52 | 82 52 |
| Constrains | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |

**Table S4 (continued).**

| Temperature | 210 K | 225 K | 240 K | 255 K | 270 K | 285 K | 300 K |
|---|---|---|---|---|---|---|---|
| $a$ (Å) | 24.0853 (3) | 24.0895 (3) | 24.0938 (3) | 24.0980 (3) | 24.1017 (3) | 24.1046 (3) | 24.1082 (3) |
| $U_{iso}$(Rb1) ($\cdot 10^{-4}$ Å$^2$) | 133 (10) | 147 (10) | 159 (11) | 165 (11) | 168 (11) | 177 (11) | 188 (11) |
| $U_{11}$(Rb2) ($\cdot 10^{-4}$ Å$^2$) | 100 (15) | 114 (15) | 127 (16) | 141 (16) | 145 (16) | 162 (16) | 180 (17) |
| $U_{22}$(Rb2) ($\cdot 10^{-4}$ Å$^2$) | 433 (14) | 459 (14) | 482 (14) | 515 (14) | 542 (15) | 557 (15) | 584 (15) |
| $U_{iso}$(host) ($\cdot 10^{-4}$ Å$^2$) | 85 (4) | 95 (4) | 103 (4) | 110 (4) | 115 (4) | 119 (4) | 125 (4) |
| sof(Sn1´) (%) | 30.71 (7) | 30.57 (7) | 29.98 (7) | 31.25 (7) | 30.27 (7) | 30.56 (7) | 30.47 (7) |
| $x$(Sn2) | 0.0908 (1) | 0.0907 (1) | 0.0908 (1) | 0.0907 (1) | 0.0908 (1) | 0.0907 (1) | 0.0907 (1) |
| $x$(Sn2´) | 0.0896 (1) | 0.0896 (1) | 0.0896 (1) | 0.0897 (1) | 0.0896 (1) | 0.0896 (1) | 0.0896 (1) |
| $y$(Sn2´) | 0.4054 (1) | 0.4053 (1) | 0.4053 (1) | 0.4054 (1) | 0.4054 (1) | 0.4053 (1) | 0.4054 (1) |
| $z$(Sn2´) | 0.0904 (1) | 0.0903 (1) | 0.0904 (1) | 0.0904 (1) | 0.0904 (1) | 0.0904 (1) | 0.0905 (1) |
| $x$(Sn3a) | 0.00321 (1) | 0.00312 (1) | 0.00310 (1) | 0.00319 (1) | 0.00308 (1) | 0.00352 (1) | 0.00331 (1) |
| $y$(Sn3a) | 0.15191 (1) | 0.15206 (1) | 0.15194 (1) | 0.15177 (1) | 0.15206 (1) | 0.15185 (1) | 0.15177 (1) |
| $z$(Sn3a) | 0.05782 (1) | 0.05760 (1) | 0.05794 (1) | 0.05794 (1) | 0.05788 (1) | 0.05792 (1) | 0.05792 (1) |
| sof(Sn3a) (%) | 30.71 (7) | 30.57 (7) | 29.98 (7) | 31.25 (7) | 30.27 (7) | 30.56 (7) | 30.47 (7) |
| $x$(Sn3b) | 0.0012 (2) | 0.0013 (2) | 0.0013 (2) | 0.0013 (2) | 0.0013 (3) | 0.0011 (2) | 0.0012 (3) |
| $y$(Sn3b) | 0.1740 (1) | 0.1740 (1) | 0.1740 (1) | 0.1739 (1) | 0.1740 (1) | 0.1740 (1) | 0.1739 (1) |
| $z$(Sn3b) | 0.0670 (1) | 0.0670 (1) | 0.0670 (1) | 0.0670 (1) | 0.0670 (1) | 0.0670 (1) | 0.0670 (1) |
| sof(Sn3b) (%) | 69.29 (7) | 69.43 (7) | 70.02 (7) | 68.75 (7) | 69.73 (7) | 69.44 (7) | 69.53 (7) |
| $x$(Sn3´) | 0.4996 (1) | 0.4996 (1) | 0.4996 (1) | 0.4996 (1) | 0.4996 (1) | 0.4996 (1) | 0.4996 (1) |
| $y$(Sn3´) | 0.1540 (1) | 0.1539 (1) | 0.1540 (1) | 0.1540 (1) | 0.1539 (1) | 0.1539 (1) | 0.1540 (1) |
| $z$(Sn3´) | 0.0560 (1) | 0.0561 (1) | 0.0560 (1) | 0.0560 (1) | 0.0560 (1) | 0.0560 (1) | 0.0559 (1) |
| $M_{\text{pearson-IIIV}}$ | 0.845 (14) | 0.838 (14) | 0.857 (15) | 0.831 (14) | 0.824 (14) | 0.825 (14) | 0.823 (14) |
| $U$ | 0.0321 (14) | 0.0310 (14) | 0.0315 (14) | 0.0310 (15) | 0.0315 (15) | 0.0334 (15) | 0.0349 (15) |
| $V$ | 0.0005 (3) | 0.0006 (3) | 0.0005 (3) | 0.0007 (3) | 0.0008 (3) | 0.0001 (3) | –0.0001 (3) |
| $W$ | 0.00120 (2) | 0.00120 (2) | 0.00120 (2) | 0.00119 (2) | 0.00117 (2) | 0.00123 (2) | 0.00122 (2) |
| $X$ | 0.0382 (24) | 0.0390 (24) | 0.0359 (25) | 0.0411 (24) | 0.0420 (24) | 0.0404 (24) | 0.0405 (24) |
| Y | –0.0003 (1) | –0.0003 (1) | –0.0001 (1) | –0.0004 (1) | –0.0004 (1) | –0.0002 (1) | –0.0003 (1) |
| $R_p/R_{wp}$ | 6.16 6.57 | 6.12 6.57 | 6.25 6.61 | 6.25 6.55 | 6.17 6.48 | 6.12 6.40 | 6.11 6.38 |
| $R_{exp}/Chi^2$ | 2.49 6.96 | 2.50 6.88 | 2.51 6.95 | 2.53 6.70 | 2.56 6.41 | 2.58 6.16 | 2.59 6.08 |
| $R_F/R_I$ ($Rb_8Sn_{44}$) | 1.61 1.77 | 1.66 1.76 | 1.83 1.87 | 1.72 1.85 | 1.60 1.69 | 1.67 1.75 | 1.75 1.81 |
| Parameters, Background | 82 52 | 82 52 | 82 52 | 82 52 | 82 52 | 82 52 | 82 52 |
| Constrains | 2 | 2 | 2 | 2 | 2 | 2 | 2 |