# The re-determination of the molecular structure of antimony (III) oxide using very-high-temperature gas electron diffraction (VHT-GED)

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## Electronic Supplementary Information

Electronic supplementary information (ESI) available:

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Figure S1: Experimental and final weighted difference (experimental minus theoretical) combined molecular-scattering intensities for $Sb_4O_6$.

**Table S1** Conditions of the synchronous GED/MS experiments.

| Nozzle-to-plate distance, mm | 338 | 598 |
|---|---|---|
| Fast electrons beam, μA | 1.4 | 1.0 |
| Accelerating voltage, kV | 72 | 72 |
| Temperature of effusion cell, K | 748(5) | 752(5) |
| Ionization voltage, V | 50 | 50 |
| Exposure time, s | 85 | 50 |
| Residual gas pressure, Torr | $2.5 \cdot 10^{-6}$ | $3.5 \cdot 10^{-6}$ |

**Table S2** Mass spectrum data for the saturated vapour of $Sb_2O_3$ at $U_{ioniz}$ = 50 V.

| | | Abundance | |
|---|---|---|---|
| Ion | m/e[a] | Long camera | Short camera |
| $Sb^+$ | 121 | 7.7 | 6.1 |
| $SbO^+$ | 137 | 70 | 72 |
| $Sb_2O_2^+$ | 276 | 21 | 22 |
| $Sb_3O_4^+$ | 429 | 68 | 71 |
| $Sb_4O_6^+$ | 584 | 115 | 112 |

[a] The isotopic species of higher abundance are shown.

**Table S3** Interatomic distances ($r_a$ / pm), experimental and theoretical {HF/[cc-pVDZ(O)/cc-pVDZ-PP(Sb)]} amplitudes of vibration ($u$ / pm), and $k$ values for the GED structure of $Sb_4O_6$.[a]

| | Atom pair | $r_a$ | Expt. $u$ | $k$ | Calc. $u$ |
|---|---|---|---|---|---|
| $u_1$ | Sb(1)-O(5) | 195.86(4) | 5.99(9) | 0.27 | 5.78 |
| $u_2$ | O(5)...O(6) | 296.02(12) | 12.52(27) | 0.37 | 12.90 |
| $u_3$ | Sb(1)...Sb(2) | 353.23(3) | 7.97(4) | 0.04 | 7.55 |
| $u_4$ | Sb(1)...O(7) | 377.71(7) | 14.02(20) | -0.02 | 13.59 |
| $u_5$ | O(5)...O(8) | 418.53(17) | 15.37(19) | 0.42 | 16.41 |

[a] For more on the $r_{a3,1}$ method see Reference 20.

**Table S4** Least-squares correlation matrix (× 100) for $Sb_4O_6$.[a]

| | $p_2$ | $k_1$ | $k_2$ |
|---|---|---|---|
| $p_1$ | -91 | | |
| $u_1$ | | 53 | |
| $u_3$ | | 52 | 61 |
| $k_1$ | | | 51 |

[a] $k_1$ and $k_2$ are scale factors.

**Table S5** Experimental coordinates (pm) determined from the GED analysis of $Sb_4O_6$.

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Sb(1) | -176.6 | 124.8 | 0.0 |
| Sb(2) | 176.6 | 124.8 | 0.0 |
| Sb(3) | 0.0 | -124.8 | -176.6 |
| Sb(4) | 0.0 | -124.8 | 176.6 |
| O(5) | -147.8 | 0.0 | -147.8 |
| O(6) | 0.0 | 209.0 | 0.0 |
| O(7) | 147.8 | 0.0 | -147.8 |
| O(8) | 147.8 | 0.0 | 147.8 |
| O(9) | 0.0 | -209.0 | 0.0 |
| O(10) | -147.8 | 0.0 | 147.8 |

**Table S6** Calculated coordinates (pm) for $Sb_4O_6$ at the MP2/d-aug-cc-pVQZ//aug-cc-pVQZ-PP level.

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Sb(1) | -124.4 | 124.4 | 124.4 |
| Sb(2) | 124.4 | -124.4 | 124.4 |
| Sb(3) | 124.4 | 124.4 | -124.4 |
| Sb(4) | -124.4 | -124.4 | -124.4 |
| O(5) | 0.0 | 204.8 | 0.0 |
| O(6) | 0.0 | 0.0 | 204.8 |
| O(7) | 204.8 | 0.0 | 0.0 |
| O(8) | 0.0 | -204.8 | 0.0 |
| O(9) | 0.0 | 0.0 | -204.8 |
| O(10) | -204.8 | 0.0 | 0.0 |

Total Energy in Hartrees: -1409.6325

**Table S7** Calculated coordinates (pm) for $Sb_4O_6$ at the B3LYP/d-aug-cc-pVQZ//aug-cc-pVQZ-PP level.

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Sb(1) | -126.5 | 126.5 | 126.5 |
| Sb(2) | 126.5 | -126.5 | 126.5 |
| Sb(3) | 126.5 | 126.5 | -126.5 |
| Sb(4) | -126.5 | -126.5 | -126.5 |
| O(5) | 0.0 | 209.0 | 0.0 |
| O(6) | 0.0 | 0.0 | 209.0 |
| O(7) | 209.0 | 0.0 | 0.0 |
| O(8) | 0.0 | -209.0 | 0.0 |
| O(9) | 0.0 | 0.0 | -209.0 |
| O(10) | -209.0 | 0.0 | 0.0 |

Total Energy in Hartrees: -1413.1833

**Figure S1** Experimental and final weighted difference (experimental minus theoretical) combined molecular-scattering intensities for $Sb_4O_6$.