Supporting Information

**A crystalline and 3D periodically ordered mesoporous quaternary semiconductor for photocatalytic hydrogen generation**

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**Results and Discussion**

![Figure S1. Two-dimensional SAXS datasets before (left) and after (right) calcination showing several spots indicative of orientation or large grain sizes.](image)

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Figure S2. Low magnification SEM image of ISO-derived mesoporous CsTaWO$_6$.

Figure S3. XRPD pattern of the product of Cs$_2$CO$_3$ plus TaCl$_5$ dissolved in EtOH and dried at 80 °C. The bottom shows a reference card for CsCl (JCPDS card no. 02-2173).
Figure S4. In-situ XRPD patterns of the dried ISO-Cs$_2$WOCl$_5$ composite with reference lines for CsCl (orange, JCPDS card no. 02-1445) and CsTaWO$_6$ (purple, JCPDS card no. 25-0233), calcined at different temperatures (* assigned to reflections from the sample carrier).
Figure S5. TG and DTG data for ISO-derived mesoporous CsTaWO$_6$

Figure S6. MS traces of ISO-derived CsTaWO$_6$, a) water- and chlorine-derived signals; b) large bigger organic fragments from polystyrene (PS); c) smaller organic fragments from polyethylene oxide (PEO) and polyisoprene (PI); and d) other fragments, including CO$_2$, which is released up to 500 °C.