

SUPPORTING INFORMATION

Effect of thermal treatment on the stability of Na-Mn-W/SiO₂ Catalyst for the Oxidative Coupling of Methane

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1. Experimental details

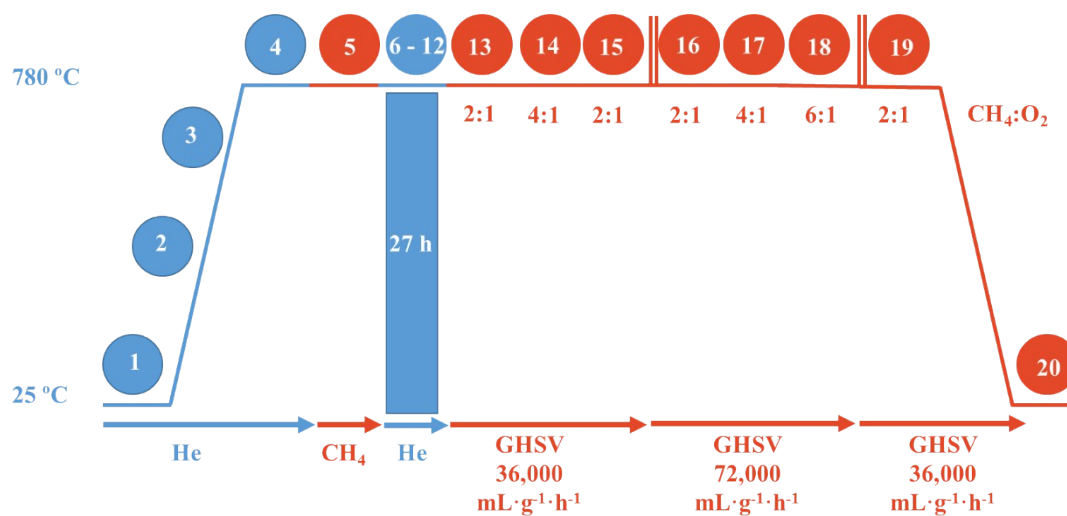


Figure S1. Schematic representation of the experimental protocol used for the operando XRD-CT measurements with Na-Mn-W/SiO₂ catalyst. Numbers correspond to the order of the XRD-CT measurements.

2. Operando XRD-CT measurements with Na-Mn-W/SiO₂ catalyst

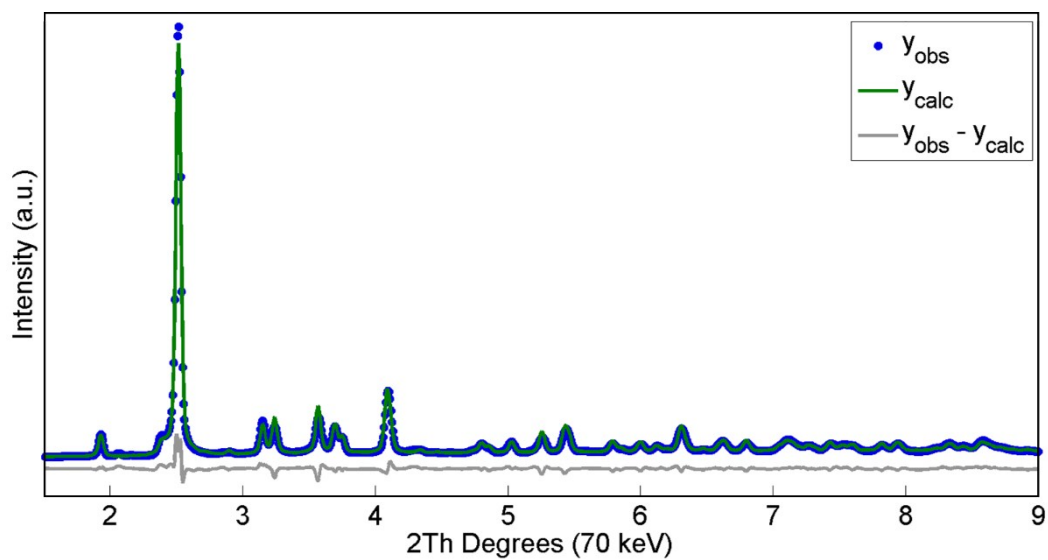


Figure S2. Results of the Rietveld refinement analysis of diffraction pattern collected at room temperature (an average diffraction pattern for XRD-CT dataset).

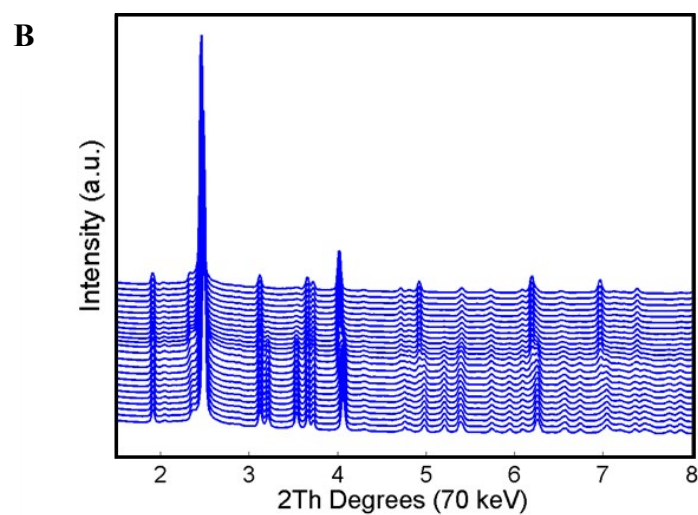
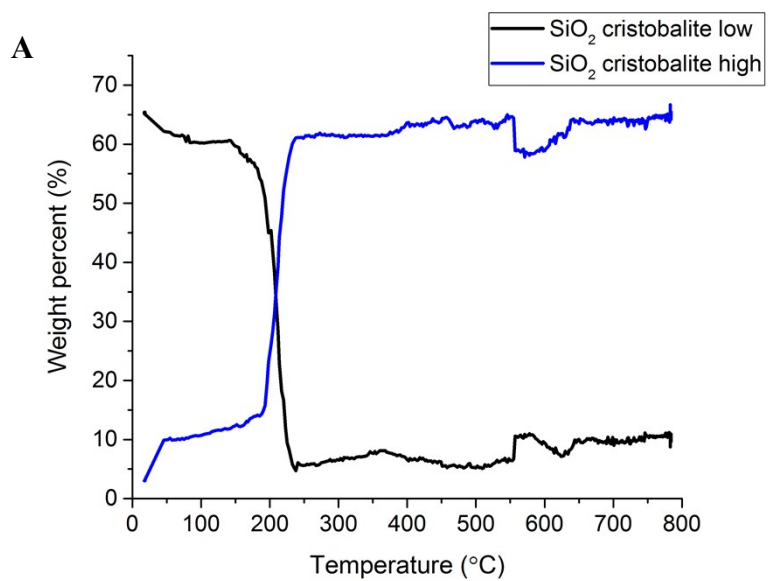


Figure S3. Panel A: Total weight percentage composition of two cristobalite polymorphs (low and high symmetry) in Na-Mn-W/SiO₂ catalyst as a function of temperature during the temperature ramp under He. Panel B: Line scans collected during the temperature ramp under He, showing the changes in diffraction pattern related to cristobalite phase transition from lower to higher symmetry.

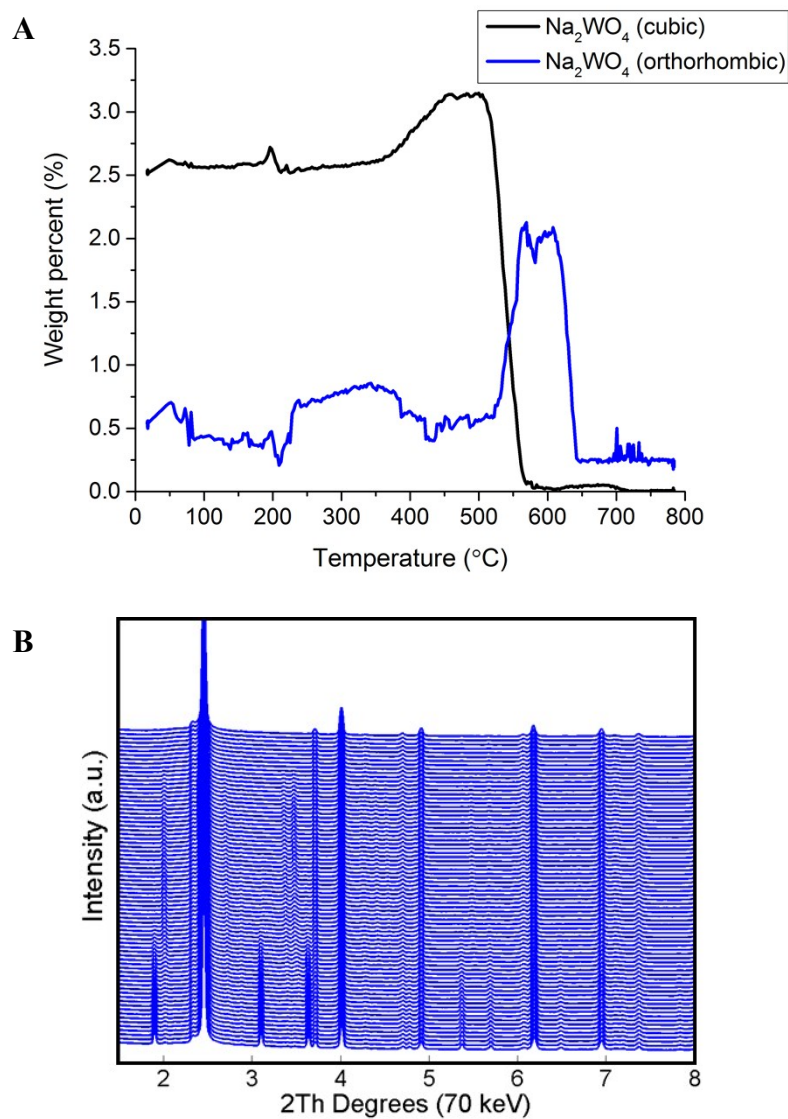


Figure S4. Panel A: Total weight percentage composition of two Na_2WO_4 polymorphs (low and high temperature) in Na-Mn-W/ SiO_2 catalyst as a function of temperature during the temperature ramp under He. Panel B: Line scans collected during the temperature ramp under He, showing the changes in diffraction pattern related to Na_2WO_4 phase transition from cubic to orthorhombic crystal structure.

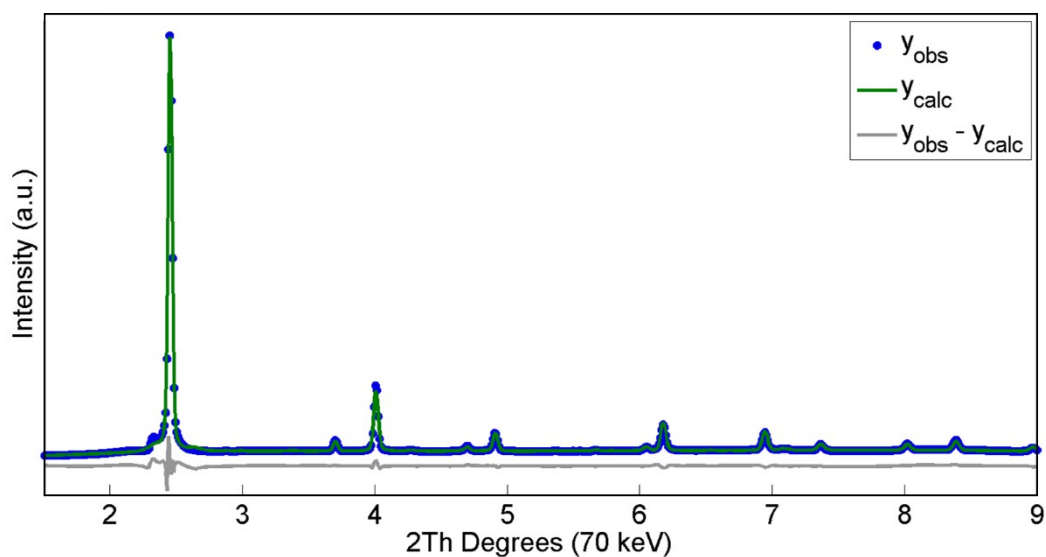


Figure S5. Results of the Rietveld refinement analysis of diffraction pattern collected at 780 °C under He (an average diffraction pattern for XRD-CT dataset).

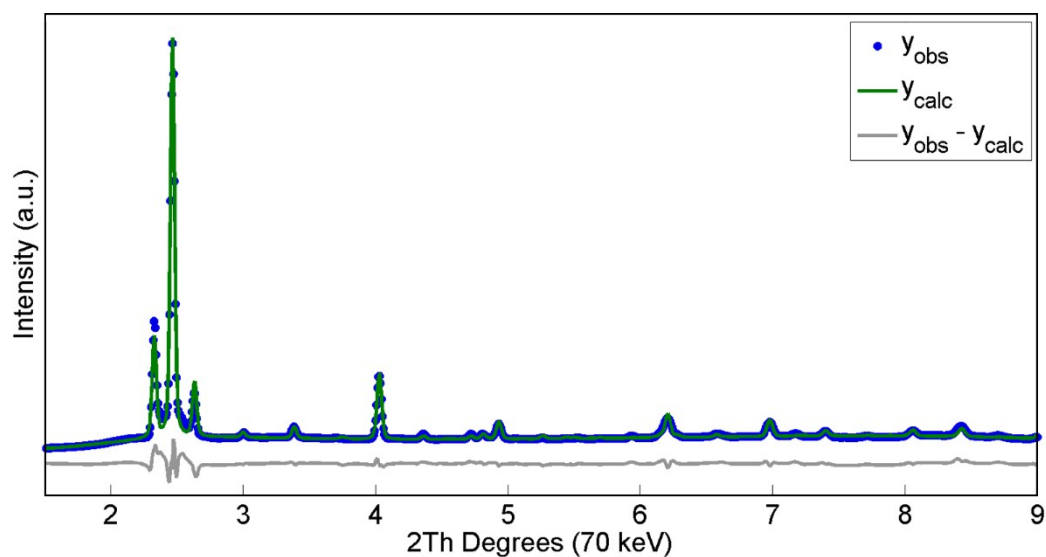


Figure S6. Results of the Rietveld refinement analysis of diffraction pattern collected at 780 °C under He after the prolonged calcination for 27 h (an average diffraction pattern for XRD-CT dataset).

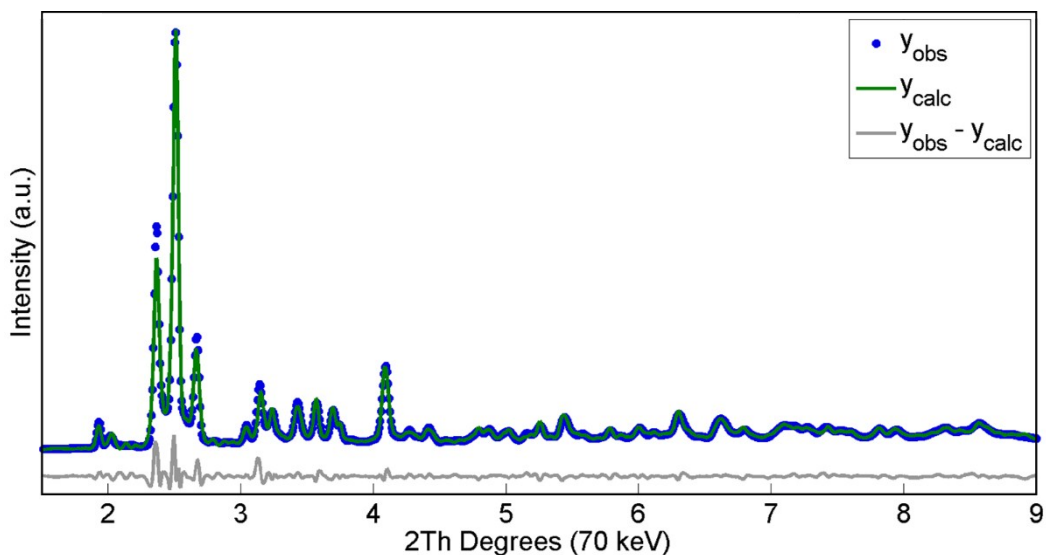


Figure S7. Results of the Rietveld refinement analysis of diffraction pattern collected at room temperature after the OCM reaction (an average diffraction pattern for XRD-CT dataset).

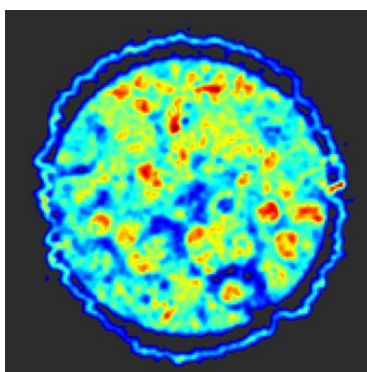


Figure S8. Spatial distribution of high symmetry cristobalite, collected at 780 °C under He after the prolonged calcination for 27 h. Significant interaction between Na⁺ species and reactor vessel, leading to crystallisation of amorphous silica can be observed in the XRD-CT image.



Figure S9. *Photograph of reactor tubes after the laboratory catalyst performance testing with Na-Mn-W/SiO₂ catalysts prepared with incipient wetness impregnation and calcined for 2 h and 8 h.*

Note: copies of the raw data presented in this manuscript are available on request by contacting one of the corresponding authors.