

# Supplementary information

## Innovative insights in plug flow microreactor for *operando* X-ray studies

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### Measure of the catalytic performance for the X-ray microreactor

The catalyst 12 wt.%CeO<sub>2</sub> in 10 wt.%Cu/Al<sub>2</sub>O<sub>3</sub> was prepared using standard impregnation methods. We have used meshed catalyst particles with mesh sizes in the range of 80 up to 120 mesh (corresponding to particle sizes of 177 to 125  $\mu$ m, respectively). The catalyst loading in the laboratory reactor was 30 mg, giving a bed of 4 mm in a quartz tube of 4 mm diameter. For the X-ray capillary microreactor a quartz capillary of 1mm diameter was used and the loading was 7.5 times less (4mg) then the gas flows was proportional reduce for this factor in order to have the same residence time per mass in each reactor. In both cases the catalyst was first reduced in situ in hydrogen prior to the water gas shift reaction (WGS). For simplicity we detail only the X-ray microreactor procedure, the other can easily derived: the reduction has proceed in 5% H<sub>2</sub>/He at 15ml/min flow rate (Brooks) as the temperature was ramped at 10 K/min up to 573 K and maintained at this temperature for 1h. Then the temperature was raised down in 15 ml/min of He flow to 473 K in order to start the measures of the CO conversion. Following 6ml/min of He flow was pass through a

saturator containing water at 338K this temperature was maintained downstream by a special heating line up to the microreactor entrance. There the flow was mixed with 13ml/min of 5%CO/He. Measures are taken before 40 min at each measured temperature: 473, 523, 573 and 623 K. The CO conversion was calculated using a mass spectrometer (OMNISTAR QMS 200) in the outlet of the microreactor and following CO<sub>2</sub> and CO lines, the CO conversion is defined as  $100 \times \text{CO}_2 / (\text{CO} + \text{CO}_2)$ . For error estimation four measures are taken in each reactor.

### **Pictures of the X-ray Microreactor**

