

**Experiment title:**

Synchronous, time-resolved XAS/DRIFTS study of Rh- and Ir-based systems active for H<sub>2</sub> production from Ammo

**Experiment number:**

EC-170

**Beamline:**
**Date of experiment:**

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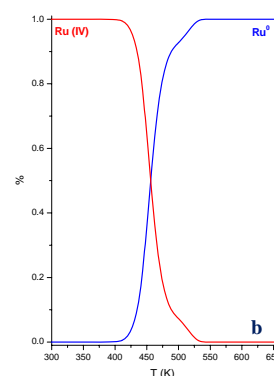
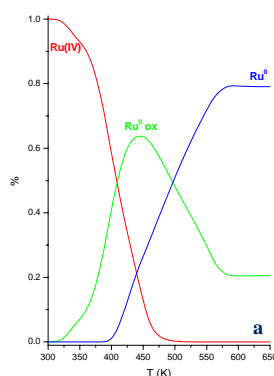
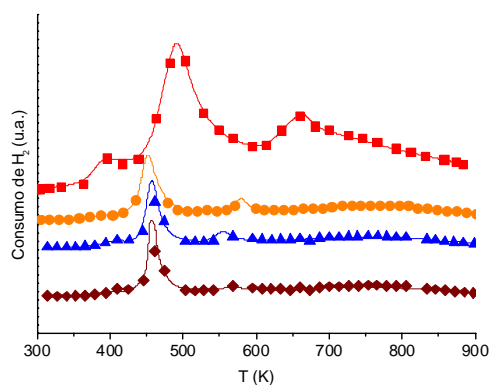
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**Report:**

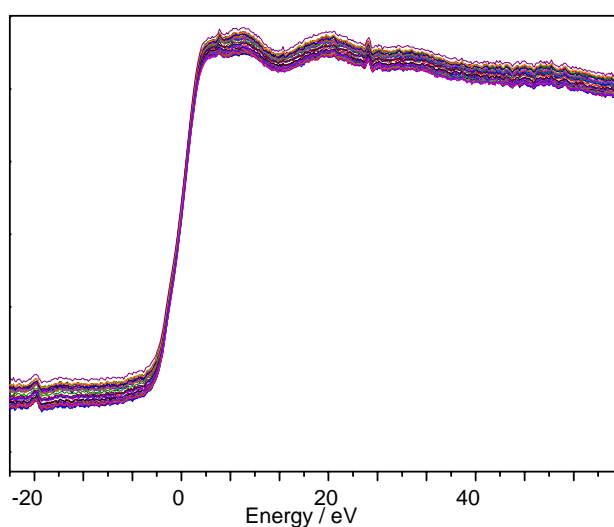
XAS (XANES and EXAFS) signals for 2 wt. % Ru samples supported in activated carbon calcined at different temperatures were analyzed by XANES-Factor Analysis and compared with laboratory TPR data (Fig. 1).



**Fig. 1** Left; TPR data of (■) RuCA-0, (●) RuCA-1, (▲) RuCA-2 y (◆) RuCA-3 samples. Right; Concentration profiles of a) RuCA-0 y b) RuCA-2 samples.

The XANES analysis of results showed the presence of 3 species in the low temperature calcined samples and 2 in the remaining cases. The interpretation of the reduction process provide evidence of two types of contributions coming from the metal reduction and the gassification of the carbon support. However, the joint analysis of both tecvhniques inidicate that main differences among samples concern the metal component and the stabilization (in some cases) of an intermediate species of the reduction process. The reduction temperature is thus used to control the state of Ru prior to reaction with  $\text{NH}_3$ .

Analysis of changes under  $\text{NH}_3$  interaction with the systems are very subtle (see Fig. 2) and are currently under study bu using Factor Analysis. It appears that contact with the gas produces a change in the first continuum resonance indicating a change of coordination distances and potential incorporation of N-adspecies.



**Fig. 2** XANES data obtained (50 ms) after  $\text{NH}_3$  contact with pre-reduced RuCa-0 sample.