

## Supplementary Information

### *Identical XAFS Spectra in all Four Channels*

To demonstrate that identical XAFS data are collected from all four sample positions the following experiment was conducted. The same catalyst (2.7 wt% Ni/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, calcined at 400°C for 8 hr in air) was loaded into all four positions of the sample holder and the samples reduced in a flow of hydrogen by ramping to 650°C at 5 deg/min. The resulting Ni K-edge TPR-XANES data are shown in Figure S1. Visually it appears that the sequence of XANES spectra as a function of temperature is similar for each of the four samples. The resulting Ni reduction profiles obtained from the linear combination fit to these XANES spectra are shown in Figure S2. All four profiles are essentially indistinguishable, with a measured maximum deviation of 1% between the four values at a given temperature. To add true meaning to this error value these data would have to be compared to an experiment where the catalyst is divided into four aliquots and the experiment repeated four times, and the LCF conducted on the four separate data sets. This experiment has not been conducted, but it seems reasonable to hypothesize that the inherent difficulties of repeating a measurement four times would produce greater variability than a single simultaneous experiment. The data of Figs S1 and S2 are interpreted to indicate that the same spectra are obtained in all four sample positions, and that there are no measurable differences in the individual channels of the four-channel ion chamber.

Figure S1

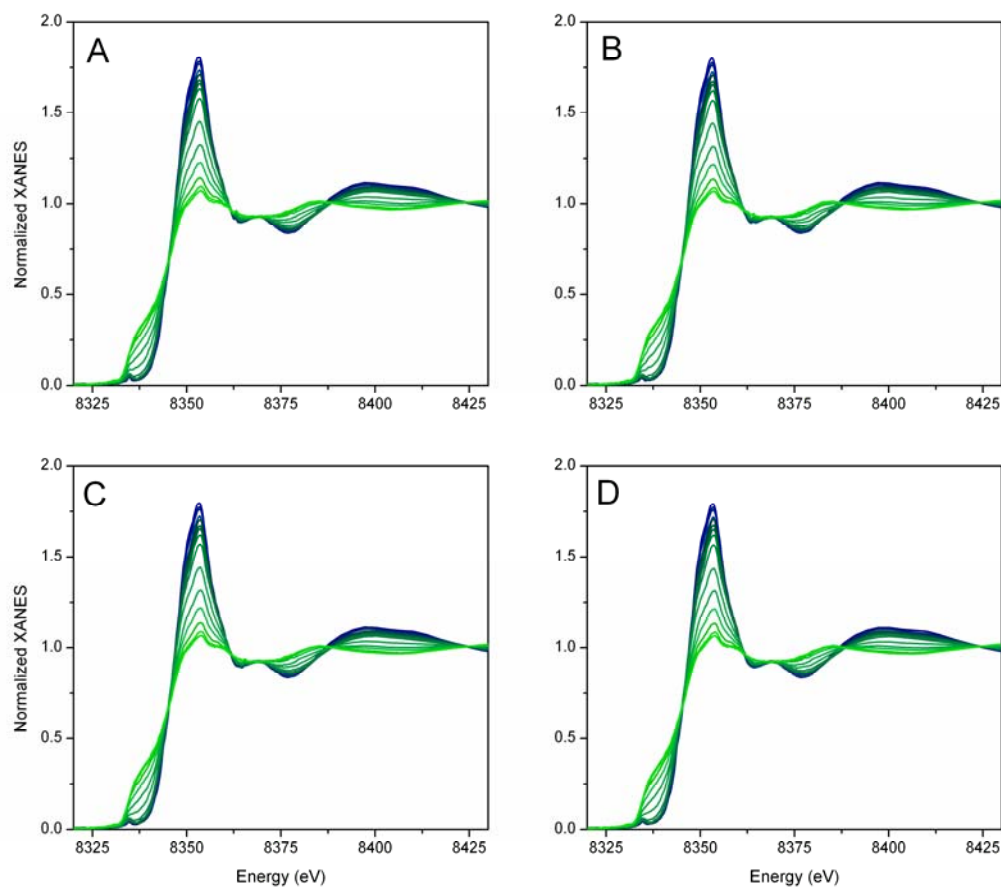


Figure S1. Simultaneous Ni K-edge TPR-XANES of the 400°C oxidized Ni/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> catalyst. Each panel shows the sequence of spectra from one of the four sample positions measured as the temperature was ramped to 650°C at 5deg/min. (A) = sample position 1, (B) = sample position 2, (C) = sample position 3, (D) = sample position 4.

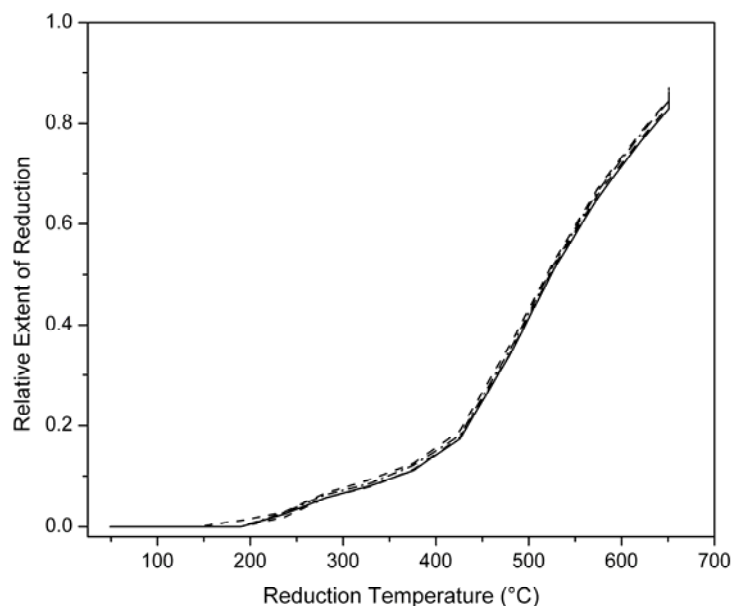


Figure S2. Simultaneously determined Ni reduction profiles determined by linear combination fitting of the data shown in Fig. 3. Sample position 1 (solid), sample position 2 (dashed), sample position 3 (dotted), and sample position 4 (dot-dash).

### ***Simultaneous Measurement of XAFS Reference Spectra***

Measuring XAFS data from reference compounds is a somewhat tedious and time-consuming chore in an XAFS research program. This task uses valuable beamtime when the time would be better used to conduct the planned research. The community has not developed an acknowledged set of quality reference spectra. Moreover it is usually the case that the individual researcher prefers to collect their own reference spectra to add to their own library. Using the 4-channel detector it is now possible to drastically reduce the amount of time it takes to collect these spectra. Additionally, by collecting the spectra simultaneously any uncertainties due to beamline artifacts are removed<sup>9</sup>. Figure S3 shows the simultaneously collected Ni XAFS data from nickel (II) nitrate hexahydrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ), Ni(II) sulfate hexahydrate ( $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ ), Ni(II) oxide (NiO), and nickel aluminate ( $\text{NiAl}_2\text{O}_4$ ). Each of these reference samples was mounted so that its

XAS spectrum was measured by one of the channels in the four channel ionization chamber. The XAFS spectra of all four samples (Fig. S3) were collected simultaneously, with a single energy scan of the monochromator. The signal/noise of both the XANES and EXAFS data is excellent and comparable to that if these spectra were collected singly<sup>9</sup>, and the time required to obtain this data is a factor of four less then if the spectra were acquired singly.

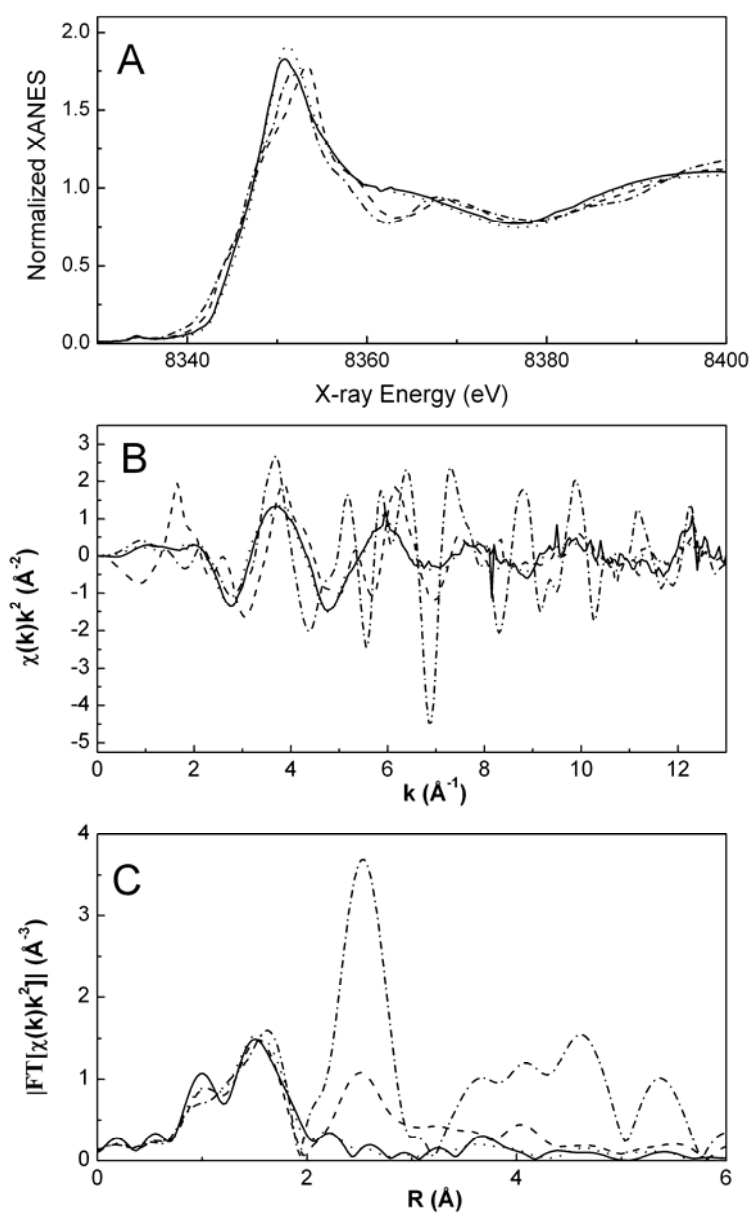


Figure S3. The simultaneously measured Ni K-edge XAFS of the four Ni reference compounds, Ni sulfate hexahydrate (dashed), Ni nitrate hexahydrate (solid); Ni aluminate (dots); Ni oxide (dash-dot). The XANES data (A),  $k^2\chi(k)$  (B), and magnitude of the FT (C) are shown.