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Report

In order to investigate the rhodium and palladium K-edges by energy dispersive EXAFS, it is necessary to use a Laue geometry monochromator crystal since at these relatively high energies (> 23 kev) the penetration of the radiation into the crystal degrades the resolution. Considerable difficulty was encountered with the monochromator, with one crystal being mis-cut and a second providing an unstable focus. Several samples proved to be inappropriate for the catalysis environmental cell. For some, durable and homogeneous self-supporting discs could not be made. Thus either the samples could not sustain a heat treatment process or the EDE spectra (which are very sensitive to sample inhomogeneities) did not give reliable and interpretable data.

For some systems, prepared from the deposition of $[RhCl(CO)_2]_2$ onto alumina from acetone solution, strong, homogeneous discs could be made which also provided a substantial edge jump. The rhodium is introduced to the surface as *cis*-$Rh(CO)_2$ centres in this way to yield a yellow solid. The formation of small particles of rhodium was monitored during *in vucuo* thermolysis on a temperature ramp to 500 K, and reduction under hydrogen to 423 K. An example of the data obtained is shown in Figure 1, which shows 100 spectra during the activation process. It appear that the rhodium has formed metal particles below 373 K under hydrogen.

Exposure of the supported rhodium to CO caused some changes in the XAFS pattern; further analysis is required to establish the extent of any regeneration of the $Rh(CO)_2$ centres.

Figure 1.