REPORT ON THE EXAFS DATA COLLECTION ON BM 29 OF THE ESRF

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Experiment number: CH95; Beam line : BM29
Period: August, 19-26, 1996

The beamtime allocated to proposal CH95 was partly used to compare the performance of BM29 of the ESRF with XAFS station 9.2 of the SRS, Daresbury using samples relevant for experiment number CH95.
We checked the performance of BM29 using our standard $Pt/\gamma-Al_2O_3$ catalyst. The same sample batch, the same pre-treatment procedure, the same in-situ XAFS cell and the same measuring conditions were used for the experiments carried out at beamline BM 29.

Figure 1 shows the EXAFS curves obtained after pre-edge subtraction, normalisation, and background removal. The most obvious difference is the high frequency noise on the data collected at beamline BM 29. Above k=12 the noise reaches the same amplitude as the signal (fig. 2). The counting time per data point for the BM29 experiments amounted 2 sec, for the 9.2 this value was l-3 sec. The detectors used at station 9.2 were ionisation chambers. The small differences in amplitudes (see also Figure 3, showing a comparison of Fourier transforms) are most probably caused by the better energy resolution of station BM29 (see also Figure 4, displaying a comparison of absorption edges). From these results it can be concluded that the detection methods applied at station BM 29 has to be further optimised in order to get a comparable or better performance than station 9.2 of the SRS (Daresbury, UK).

A promising set of data on samples with a very low concentration of Nickel (Ni content varied between 0.3 wt% and 0.02 wt% measured in the fluorescence mode using the photodiodes with the samples mounted in Kapton rods. Since sample and detector were at a very small distance a very large solid angle could be realised. A typical example of an EXAES spectrum measured on a 0.02 wt% sample is given in figure 5. It is our experience that typical concentrations of 0.5 wt% Ni can be measured at station 9.2 using a 13 element Ge fluorescence detector. The higher solid angle and the higher number of fluorescence photons of XAFS station BM29 resulted in this much lower detection limit. It can be foreseen that a further optimised fluorescence detection system will lead to an even lower detection limit for this beamline.

Fig 1

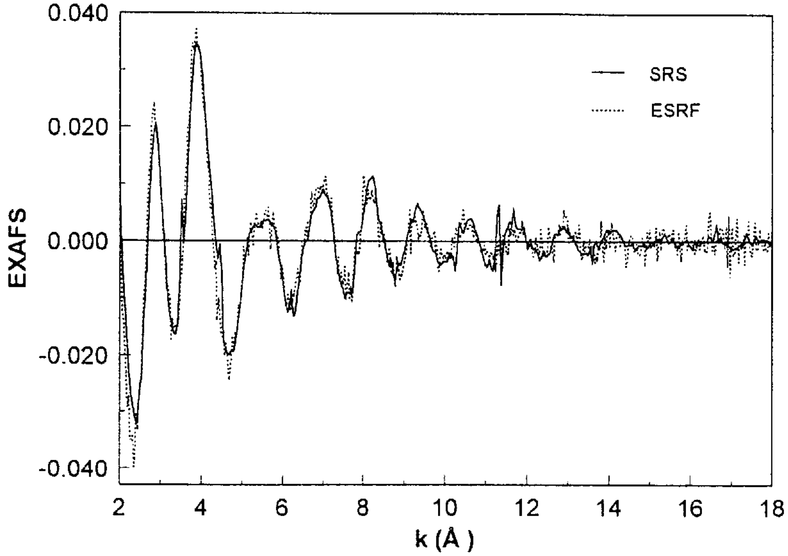

0.040
0.020
0.000
-0.020
-0.040
EXAFS
SRS
ESRF
2
4
6
8
10
12
14
16
18
k (Å)

Fig 2

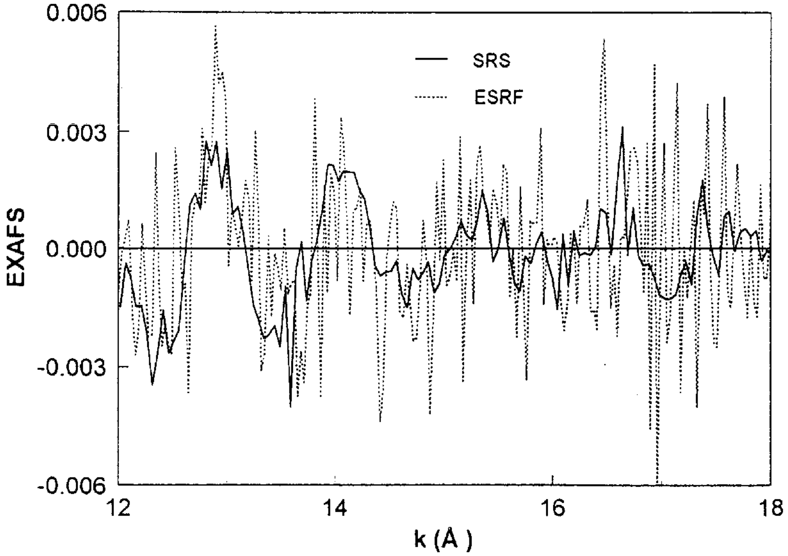

0.006
0.003
0.000
-0.003
-0.006
EXAFS
SRS
ESRF
12
14
16
18
k (Å)

Fig 3

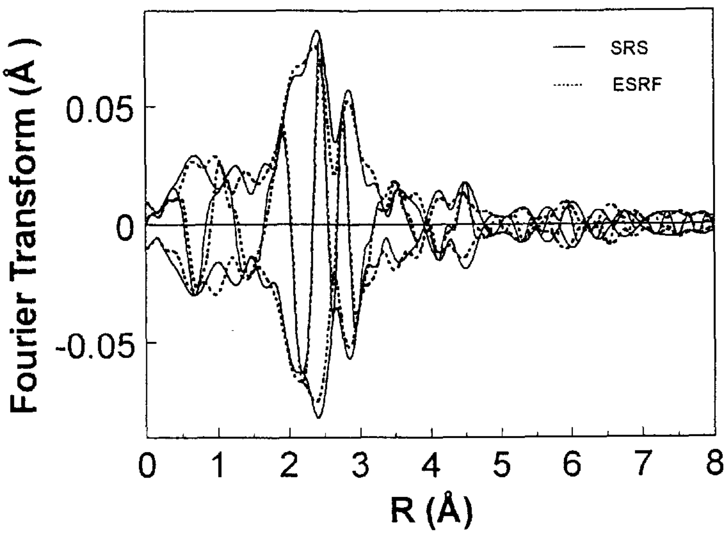

SRS
ESRF
0.05
0
-0.05
Fourier Transform (Å)
0
1
2
3
4
5
6
7
8
R (Å)

Fig 4

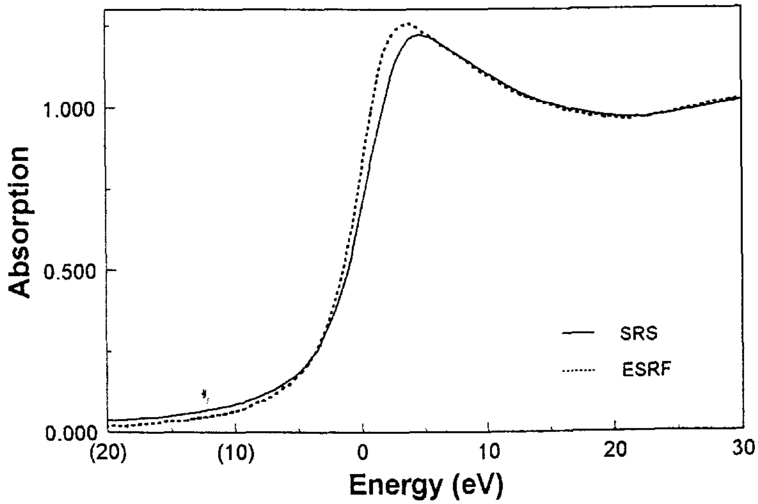
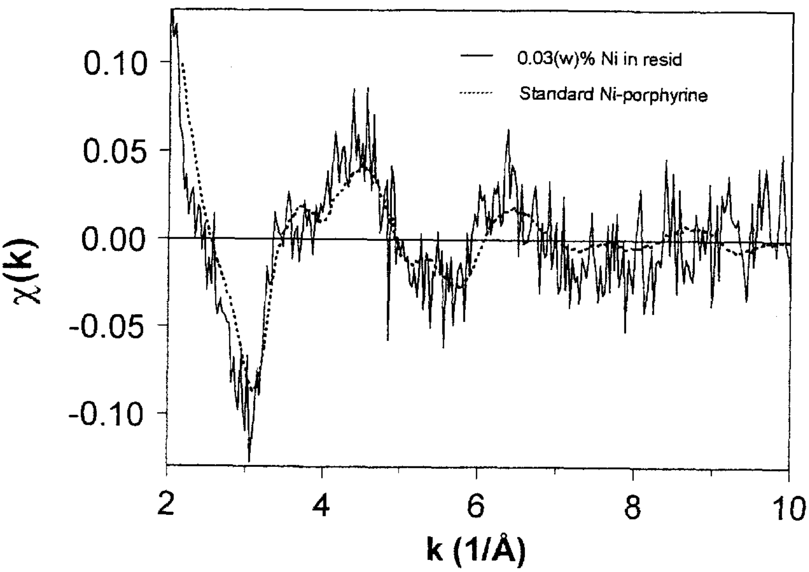

1.000
0.500
0.000
Absorption
SRS
ESRF
(20)
(10)
0
10
20
30
Energy (eV)

Fig 5



0.03(w)% Ni in resid
Standard Ni-porphyrine
0.10
0.05
0.00
-0.05
-0.10
χ(k)
2
4
6
8
10
k (1/Å)