ESRF	**Experiment title:** Study of the metal binding properties of the C-terminal [56-61] peptidic fragment of metallothioneins and glutathione by EXAFS.	**Experiment number:** CH-641
Beamline:	**Date of experiment:** from: 17th February 1999 to: 23th February 1999	**Date of report:** 23th February 1999
Shifts:	**Local contact(s):** V. Armando Sole	*Received at ESRF:* 24 FEV. 1999

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Report:

This report describes the work performed at ID26 station, and it is mainly referred to the description of both the experiments performed and the problems suffered. Because of these problems and because the experiments have been just finished, only a very light reference to the results obtained and the data treatment is done.

The critical point of this investigation is that the measurements could not be performed at 77 K, according to the project presented to ESRF. After the setup of the line and some preliminary measurements of Zn and Cd solutions at low concentrations and room temperature with a photodiode, it was clear that the data quality was not good enough to study possible metal-metal distances around 4 Å. Great efforts were devoted to operate the energy resolved multielement detector to perform measurements with the sample at low temperatures, without success. As a consequence, measurements at 77 K could not be performed with more than three days of beamtime left.

After that insoluble problem, we tried to make the best use of the allocated beamtime by measuring model compounds and samples at room temperature. However, an additional problem arose. The system to control the temperature of the second crystal of the monochromator broke down and made the measurements hopeless.

The study of the model compounds $(EtN)_2Cd_4(SPh)_{10}$ and $(EtN)_2Zn_4(SPh)_{10}$, widely referred in the literature, at room temperature clearly indicated the lack of resolution of the performed measurements at the available conditions.

Sixteen metal ion - peptide solutions, at 2 mM metal concentration and different metal-to-peptide ratios, were studied. As example, Figure 1 shows an averaged EXAFS spectrum for the Zn-peptidic fragment system. Some glitches can be clearly observed, which may hinder the data treatment and interpretation. These irregularities were present in all the Zn K-edge spectra and could probably have been removed by using an energy discriminating detector. Figures 2 and 3 show an example of the results obtained after FT treatment for the same system. It can be clearly observed the presence of the Zn-S bond at *ca.* 2.32 A, which is in agreement with the previous literature data. However, the possible presence of a Zn-Zn bond, as a consequence of the formation of thiolate clusters, is not clear enough because the high noise level.

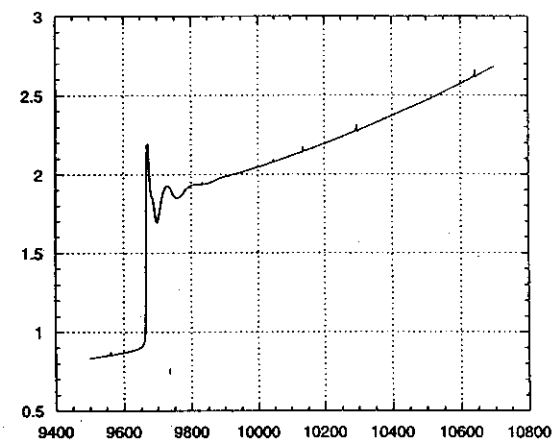

Fig. 1

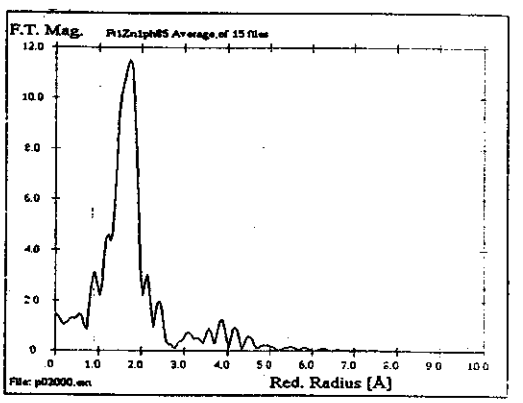

Fig. 2

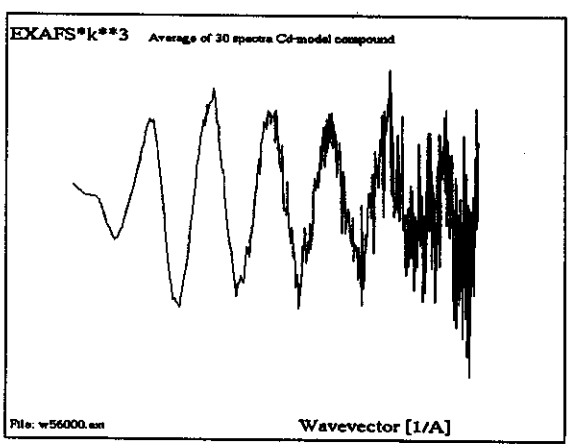

Fig. 3