



Experiment title: Zinc Speciation in the particulate matter from the Seine river : tracing zinc sources and modeling dissolved zinc concentrations	Experiment number: 30-02-942	
Beamline: BM30B	Date of experiment: from: June 24 to: June 30 2009	Date of report: September 28 2009 <i>Received at ESRF:</i>
Shifts: 18	Local contact(s): Dr Isabelle ALLIOT*	
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Report:

The objective of this proposal was to characterize zinc species in complex natural suspended matter from the Seine River collected from various sites and time period in relation to water chemistry and zinc bioavailability.

During the (18 shifts) beamtime EXAFS and XANES data were recorded at the Zn K-edge in fluorescence detection mode using 30 Ge-elements detectors, at a temperature of 5-15 K, using a liquid He cryostat. The monochromator was equipped with Si(220) crystals. XANES data were recorded within the same scans as EXAFS data by defining various regions in the scan.

12 samples were analyzed with Zn concentrations varying within the 100 – 600 ppm range. Special care was taken for preserving samples under anoxic conditions from the sampling to the analysis. Samples were dried and prepared as pellets under anoxic conditions at IMPMC laboratory. Samples were then brought to ESRF under controlled anoxic conditions and mounted in an anoxic glove box on the cryostat sample holder. It was brought to the beamline after sealing within an hermetic plastic bag and rapidly mounted on the cryostat rod. The sample rod was then put under low pressure He atmosphere for data analysis in the liquid He cryostat at a temperature or 10-15K.

Comparison of spectra taken on well preserved samples and on air-dried samples revealed dramatic changes in Zn speciation after air-drying, indicating the presence of redox sensitive Zn-bearing phases.

Data analysis is under progress and is based on linear least-squares fitting using a set of more than 50 model compounds, including Zn-bearing crystalline and amorphous phases as well as inorganic and organic sorption samples, already collected from previous synchrotron experiments from the group.

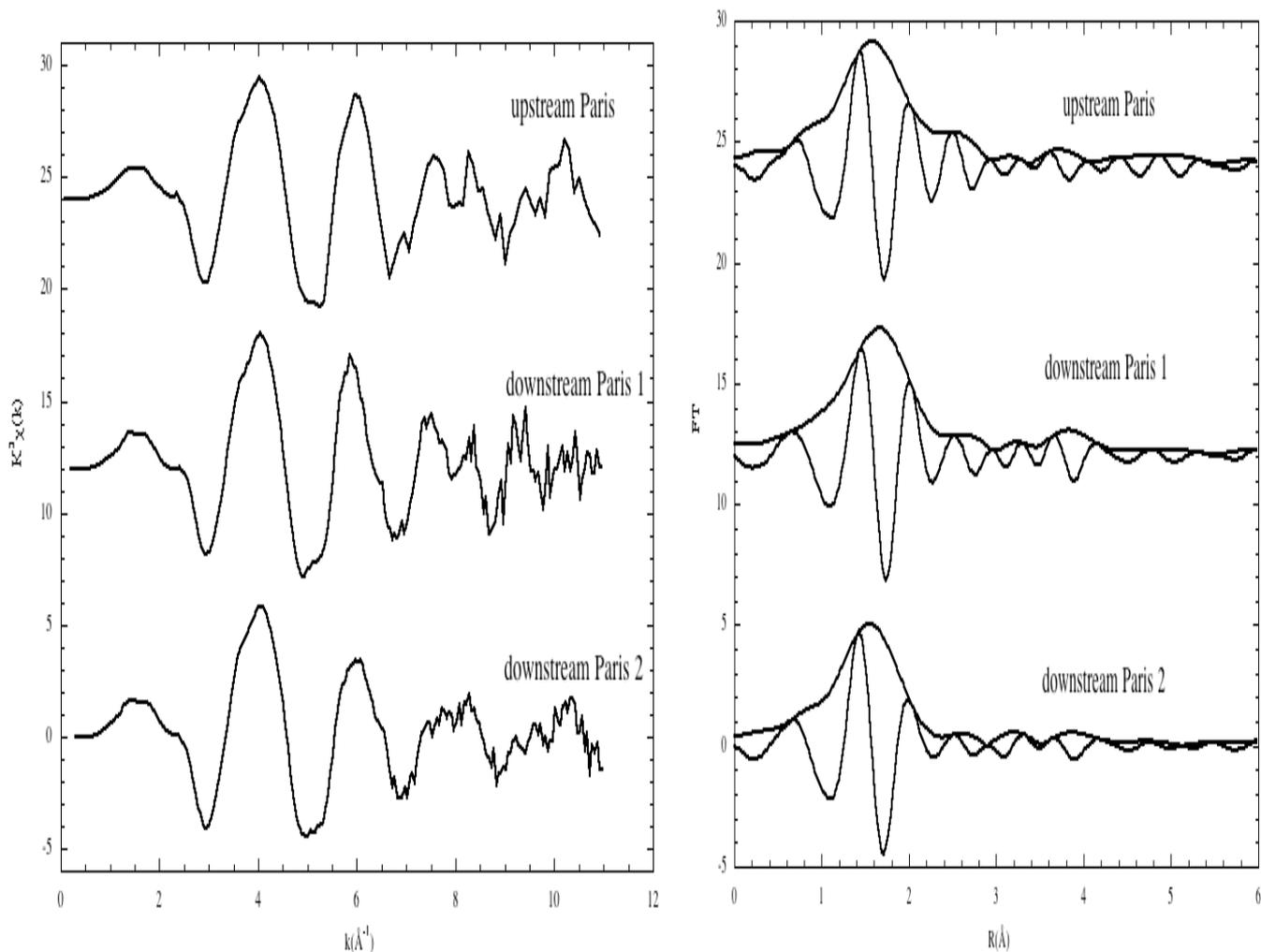


Fig.1 : Unfiltered EXAFS data at the Zn K-edge showing changes in Zn speciation in suspended matter from the Seine river from upstream to downstream Paris.

The data were of good quality, with a usable signal to noise ratio after 8 to 10 scans, depending on the Zn concentration (Fig. 1). The quality of the experiment also depended upon respecting the neighbouring Fe fluorescence (1- 5% wt Fe in samples).

Additional beamtime is required to complete the set of data in order to characterize the seasonal evolution of Zn speciation in relation with the increased Zn inputs for sewage water overflows .

In addition the complexity of Zn speciation in the samples studied requires to record an additional set of model compounds including redox sensitive mineral phases containing Zn at dilute levels.

Analyzing Zn speciation in particulate matter from the Seine river will give strong constraints on the biogeochemical cycling of this metal within the Seine river basin, and is required for modeling the spatial and seasonal evolution of dissolved Zn in the Seine water.

