ESRF	**Experiment title:** Speciation of metal in aerosol emitted by metallurgic plants and incinerators : Pb and Cd case	**Experiment number:** 30-02-612
Beamline: BM30B FAME	**Date of experiment:** from: march 12th to 19th and Jully 16th to 22th, 2003	**Date of report:** 06-10-03
Shifts: 9	**Local contact(s):** Jean-Louis Hazemann	*Received at ESRF:*

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Report:

Currently, it is increasingly asked to manufacturers to make an evaluation of the sanitary impact of their atmospheric emissions. Knowing that it has been clearly demonstrated, especially for metals, that the chemical forms of an element governs its toxicological properties, as well as its mode of transfer in the environment, it becomes necessary to characterize metallic species emitted by the main transmitter manufacturers. But only few studies have been devoted to determine the speciation of heavy metal using XAS. Two industrial atmospheric particles transmitters have been studied. The first is representative of the steel industry and is located in the south of France and the second transmitter corresponds to a municipal waste incinerator plant in the area of Toulon (south of France). We have also used atmospheric filters to collect aerosols at various distances from the two transmitters.

XAS experiments have been conducted on the XAS CRG BM-30b French beamline (FAME). In the case of the Pb LIII edge, the Si(111) monochromator was used. The beam was focused vertically on the sample using the 2nd mirror and horizontally with the second crystal of the monochromator. The spot size was 200*200μm. At the Cd K edge, the second mirror was removed, and we used the Si(220) monochromator. XAS spectra were recorded in the fluorescence mode with a high purity Ge multichannel fluorescence detector. The quantity of atmospheric particles collected at the source was enough to prepare and press fine powders to be scanned. Unfortunately the mass of particles collected by the filters around the industrial plants were low and generally bellow 10 mg/cm^2. Therefore we have not removed the particles from the filters and decided to scan the filters. To increase the signal, the angle between the incident beam and the filters was decreased at maximum. Depending on the sample the angle was in the 4-8° range. Filter before use was scanned to determine the fluorescence background.

Figure 1 compares Pb-LIII edge EXAFS spectra of the two atmospheric samples with different reference compounds. The features of the EXAFS spectrum of the Pb+FeOOH sample (Pb coprecipitated with FeCl3) are similar to the features of the Steel plant EXAFS spectrum (figure 2). The modelling of the EXAFS signal indicates that the second coordination sphere of Pb is composed by Fe atoms. This result is not surprising. For the Municipal waste incinerator sample, the EXAFS curve presents some similarities with the EXAFS features of the PbSiO3 sample in the 2.5-5 Å range. But both spectra can not be superimposed.

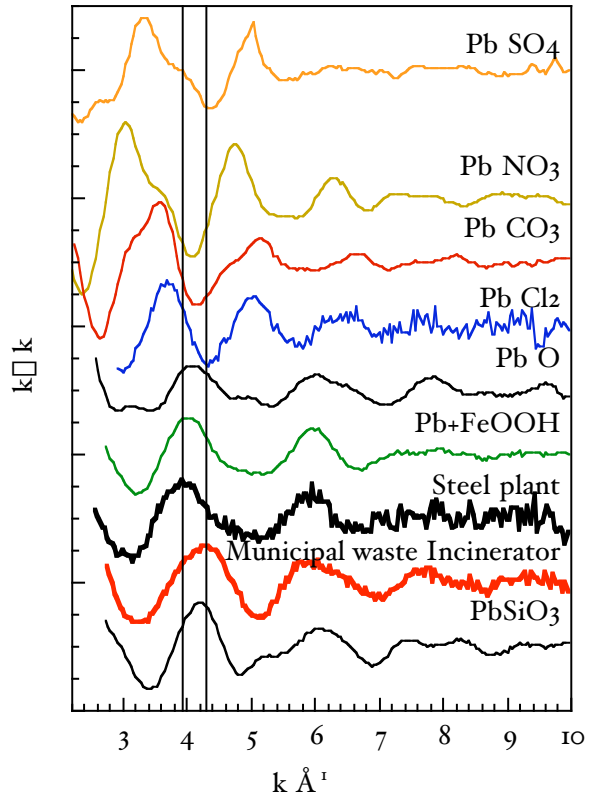

Figure 1: Pb-LIII EXAFS spectra of atmospheric particles from industrial transmitters compared with spectra of reference compounds

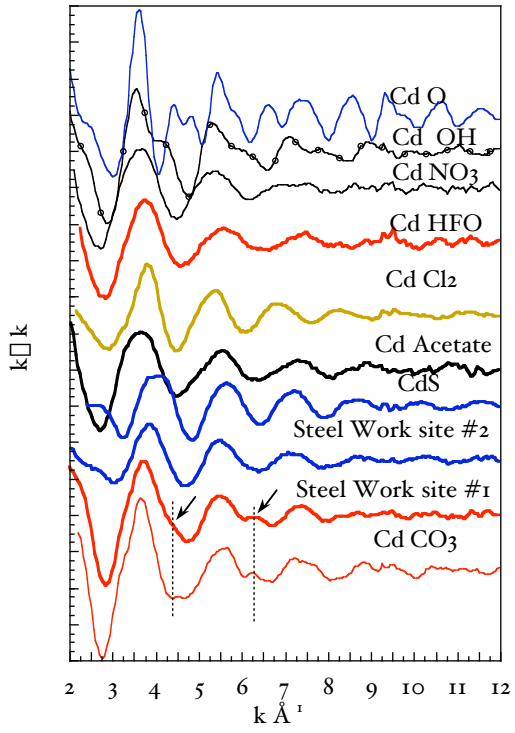

Figure 3: Cd K-edge EXAFS spectra of atmospheric particles from industrial transmitters compared with spectra of reference compounds

A very interesting point is that the XAS signal from filters positioned around the Municipal Waste incinerator is strongly different from XAS signal from the source (figure 2). This result suggests whether that the speciation of Pb in the atmospheric particles changes rapidly or that the particles collected by the filters are not originating from the plants.

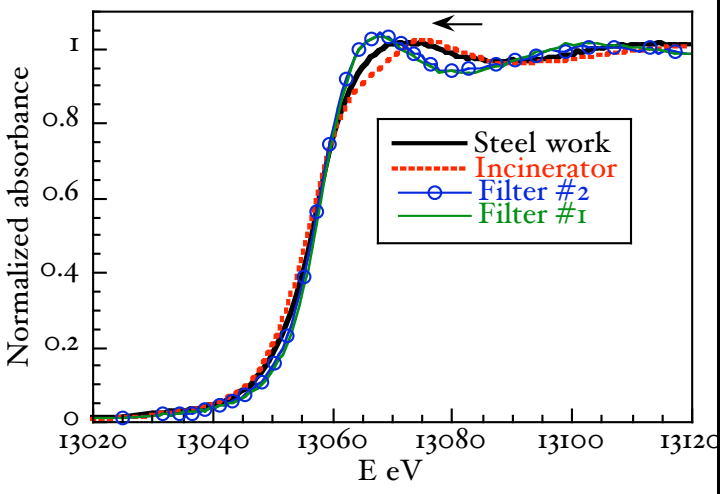

Figure 2 : XANES of the source and the filters

EXAFS modelling are still under progress.

In the case of the cadmium, the concentration was lower than for the Pb. Therefore the scanning time for each sample was high and ***we were not able to analyse a high number of samples***.

Figure 3 presents EXAFS spectra of the atmospheric particles from two sites of the steel work plant with reference compounds. The spectrum of the Steel work sample from site #1(furnace) presents some similarities with the spectrum of CdCO3. In the case of the sample from site #2 (agglomeration), the amplitude and phase of the EXAFS oscillations can be compared with that of CdS.

Further data treatments are under progress and correspond to the Ph'D of Magali Collet which started in October 2003.