



<b>Experiment title:</b> Fluorescence microscopy studies on the micro distribution of metals in estuarine sediments from the port of NY/NJ	<b>Experiment number:</b> ME-592	
<b>Beamline:</b>	<b>Date of experiment:</b> from: 17.7.2003 to: 22.7.2003	<b>Date of report:</b> 16.10.2003
<b>Shifts:</b>	<b>Local contact(s):</b> Dr. Ulrich Neuhäusler	<i>Received at ESRF:</i>

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Prior spectromicroscopy work on contaminated marine sediments done by the proposers included

- Mapping and chemical speciation by XANES of carbon in hydrated samples using the X1-A scanning transmission X-ray microscope operating near the carbon-K-absorption edge at 284 eV at the National Synchrotron Light Source, Brookhaven National Laboratory (see XRM99 proceedings).
- Mapping and chemical speciation by XANES of sulfur using the ID21 scanning X-ray microscope in fluorescence mode at 2.5 keV photon energy at the ESRF (see report ME-398)

In this experiment (ME-592), scientific scope was to link the elemental distribution information on sulfur and carbon already obtained before to the distribution of medium-Z heavy metals. In particular, we were interested to learn about the metal sulfides by colocalization of sulfur and heavy metals. Of further interest is to compare marine sediments heavily exposed to industrial activity to unexposed sediments, and, thanks to the full fluorescence spectrum available for each individual image point, to generally look for colocalization of elements. Furthermore, Fe-XANES has been performed on selected, iron-rich regions of interest within the sample.

Using the ID21 scanning X-ray microscope operating just above the iron-K-edge at 7.2 keV photon energy, metals were mapped with high spatial resolution (sub-half micron) in a variety of marine sediment samples, both heavily exposed and unexposed to human industrial activity:

- National Institute of Standards and Technology (NIST), Standard Reference Material 1944 coarse fraction ([http://patapco.nist.gov/srmcatalog/common/view\\_detail.cfm?srm=1944](http://patapco.nist.gov/srmcatalog/common/view_detail.cfm?srm=1944))
- National Institute of Standards and Technology (NIST), Standard Reference Material 1944 fine fraction ([http://patapco.nist.gov/srmcatalog/common/view\\_detail.cfm?srm=1944](http://patapco.nist.gov/srmcatalog/common/view_detail.cfm?srm=1944))
- Contaminated sediment from Newtown Creek, NY, treated by a cleaning procedure developed by Biogenesis enterprises
- Marine sediment from Blake Ridge, NC, taken 667 m below sediment surface, no human influence

- Marine sediment from Gowanus Canal, NY
- Marine sediment from Kearny Point, Newark Bay, NJ
- Marine sediment from Passaic River, NJ, a river that has seen heavy industrial activity over the last 200 years

Thanks to the excellent performance of the ID21 scanning X-ray microscope and to samples well suited to be studied with this technique, a large amount of high quality data has been acquired and analysis and publication are underway.

### NIST SRM 1944 coarse fraction, dataset # 0009

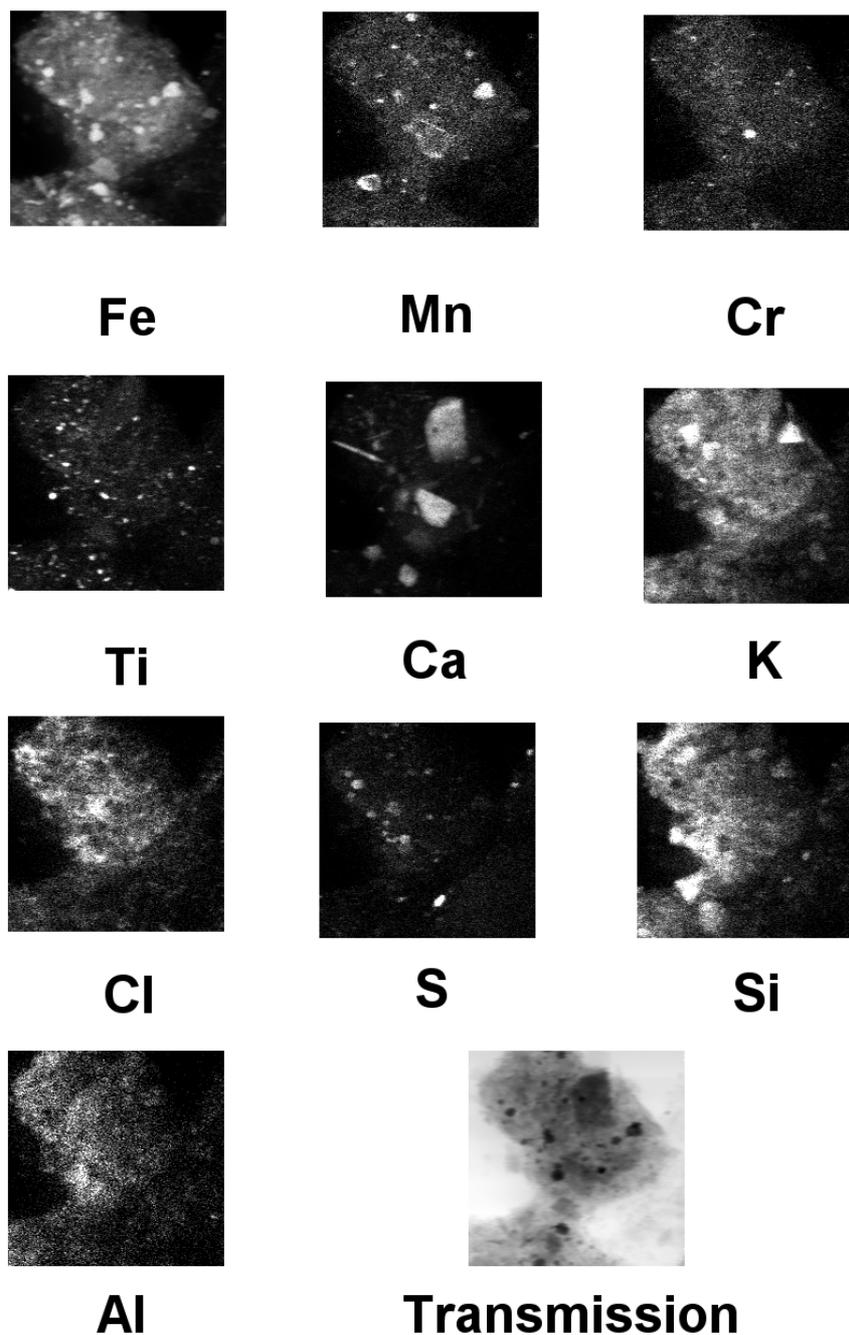


Figure 1: 100 x 100 μm elemental maps obtained with the ID21 scanning X-ray microscope at 7.2 keV photon energy with 0.5 μm pixel size. The sample is the coarse fraction of a NIST standard reference material 1944, a well characterized marine sediment sample from 6 locations in NJ/NY.