EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Micro XAS determination of Iron speciation in anthropogenic and natural aerosol particles entering the Irish Shelf.	**Experiment number**: ES524
Beamline: ID21	**Date of experiment**: from: 9 Feb 2017 to: 13 Feb 2017	**Date of report**: 27/2/2017
Shifts: 12	**Local contact(s)**: Wout De Nolf, Bernhard Hesse	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

P.Croot (NUI Galway), **S. Nicholas** (NUI Galway), **M. Heller** (IFREMER), **H. Planquette** (LEMAR) and **G. Sarthou** (LEMAR).

Preliminary Report:

The primary objective of experiment ES-524 was to determine the speciation of iron in anthropogencic and natural aerosol particles that are deposited in the surface waters of the Irish shelf. The purpose of getting information on the particulate iron was to constrain estimates of the iron bioavailability and solubility of the aerosol particles based on the speciation data. Thanks to the help and guidance of the beamline scientists we were able to achieve far more than we had initially anticipated, particularly with regard to Sulfur maps and were able to obtain some seed data for related questions on marine particles. During our beam time at ID21 we were able to run a total of 21 sample experiments that were perfomed on 14 separate samples – 13 experiments for Sulfur K-edge and 8 experiments for Fe K-edge, 6 samples were run with both S-K and Fe-K edge experiments. 7 samples were from Mace Head, one was a Teflon filter blank for the filters used at Mace Head, three were volcanic ash from the 2011 Puyehue-Cordón Caulle eruption in Chile, one was from a culture of coccolithophorids (there is a possibility that these cells are blown by the wind into the atmosphere) and three samples were of marine particles collected during French research expeditions in the North Atlantic (Geovide) and the Arctic (TransArc). Xanes spectra for several sulfur and iron standards were also run, including a coquimibite sample as this mineral had been identified in other studies as a potential marker of anthropogenic iron. Blanks from the filters used to collect the aerosols was also assessed by running 4 different filter types Teflon, Cellulose Acetate (CA), Polyethersulfone (PES), Polycarbonate (PC) typically used to collect aerosols. We found that the Teflon PES, and PC filter worked fine but for the CA depth filters it was hard to focus the beam due to the different levels at which the samples were located on the filter. PES filters with low sample loadings showed a high S blank due to the S in the filter material itself.

The preliminary analysis of this data (obtained 2 weeks prior to the date of this report) has focused on the chemical mapping (examples shown below in Figure 1 and 2) and this revealed a number of interesting features with regard to the size of Fe and S particles in the Mace Head aerosols. Sulfur xanes spectre indicates that most of the S was present as coarse

mode particles of gypsum $CaSO_4$ or Na_2SO_4, consistent with a seawater source. Iron was found more in the fine mode particles and appeared to be colocated with S in the S maps but was not as obvious in the Fe maps. Further analysis of the collected data is currently underway, with checking and evaluation of the fits for the chemical mapping data and the resulting elemental distributions and for determining the S and Fe speciation of the indidvidal particles investigated with Xanes by fitting it to samples from the respective S and Fe reference libraries in order to deconvolute the possible source signals. It is anticipated that a final report can be completed on this within the next 2 months.

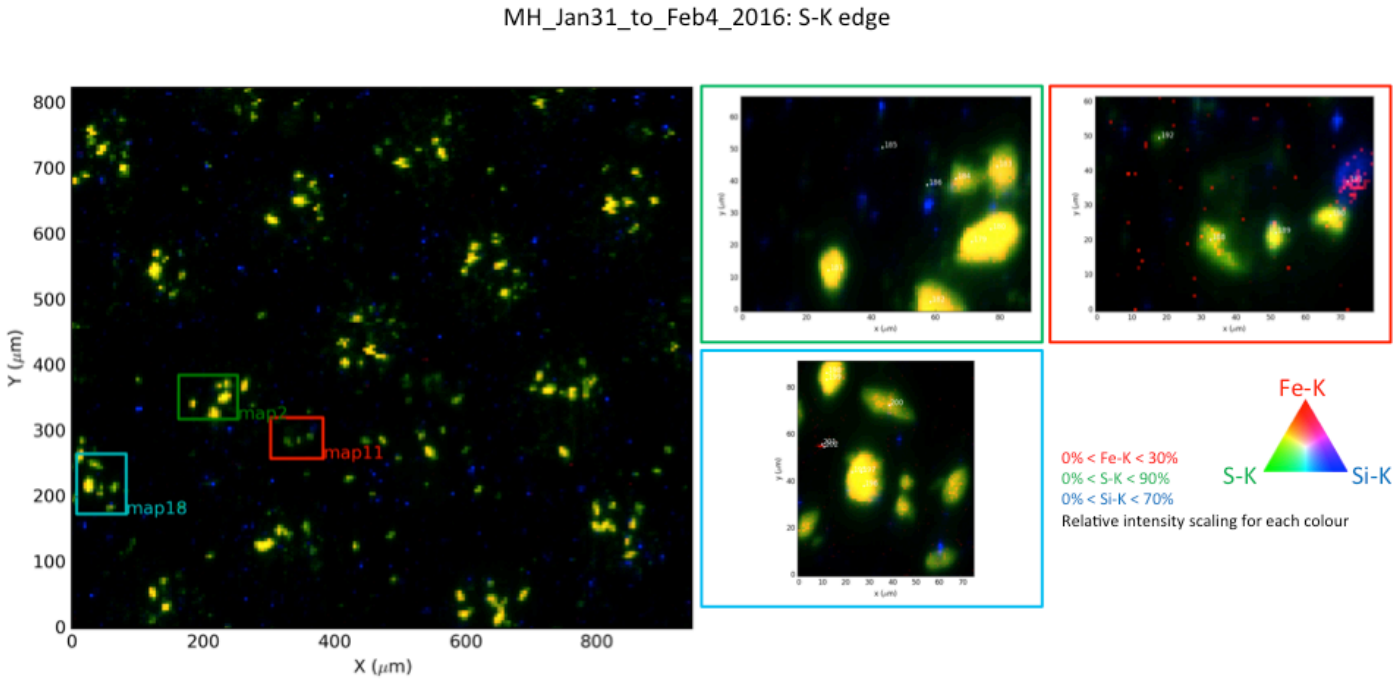

Figure 1: S-K edge chemical mapping of Mace Head sample collected Jan 31 to Feb 4, 2016.

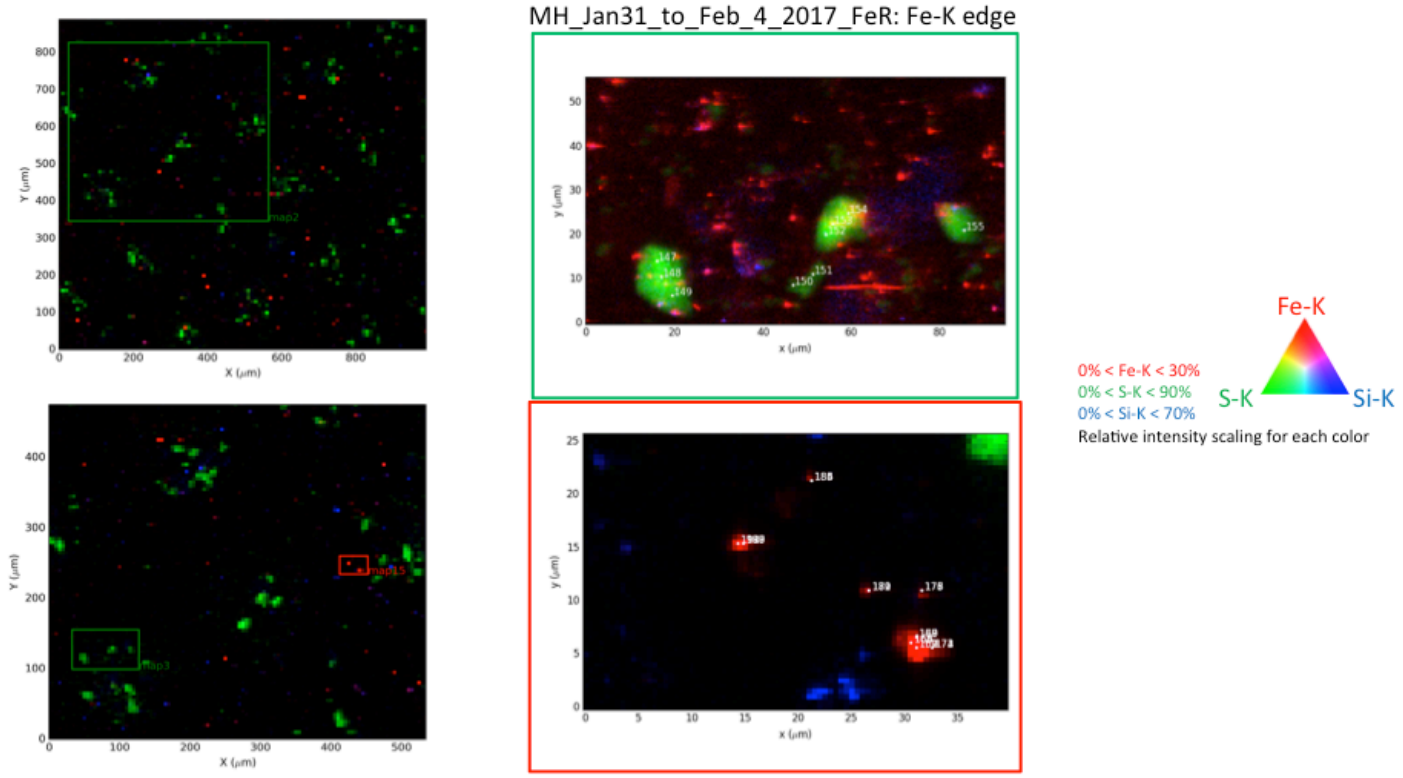

Figure 2: Fe-K edge chemical mapping of Mace Head sample collected Jan 31 to Feb 4, 2016.