

## Preliminary Experiment Report

Experiment Title: XANES and EXAFS Solution Studies of Insulin Mimetic Vanadium Complexes

Experiment number: CH448

Beamline: ID 26

Date of Experiment: from 18<sup>th</sup> June to 23<sup>rd</sup> June

Shifts: 15 shifts

Local contacts: Drs. Christophe Gauthier and Armando Sole

Names and affiliations of applicants:

Dr. Maria Rangel\*

Instituto de Ciências Biomédicas Abel Salazar and Departamento de Química  
Lg. Prof. Abel Salazar, 2 Rua do Campo Alegre, 687  
4050 PORTO 4 150 PORTO  
PORTUGAL PORTUGAL

Dr. Walkiria Schindwein\*

Department of Chemistry  
De Montfort University  
Leicester, UK

Dr. Ana Margarida Damas\*

Instituto de Ciências Biomédicas Abel Salazar  
Lg. Prof. Abel Salazar, 2  
4050 PORTO  
PORTUGAL

Report:

We will describe in this report the work that has been done along the days since as the beamline is new and many equipment was used by us for the first time.

a) 18<sup>th</sup> June

Cryostat and table set-up  
Tests with vanadium foil  
Beam Alignment  
Cryostat purge

b) 19<sup>th</sup> June

Experiments at RT with samples of 10mM aqueous solutions of vanadium complexes. The observed shifts in the pre-edge peak of the spectrum indicate that there is an effect of radiation damage (reduction of samples).

Interferences with chromium were also observed mainly because we were not using an energy dispersive multidetector element but a multidetector element with no energy resolution (photodiode).

The set-up of the cryostat and the liquid He transfer line were initiated in order to run experiments at low temperature. It was not possible to have the transfer line operational because it had been replaced by the manufacturer in a different size.

c) 20<sup>th</sup> June

Set-up of an liquid Nitrogen transfer line. The system was working fine with the N<sub>2</sub> line.

Problems arose with the cooling of the sample:

Although several procedures for the cooling of the samples had been tried ( liquid nitrogen and a salt/ice bath) diffraction peaks were always present in the spectrum and could not be discriminated by the detector.

In the future the samples will have to be cooled with dry ice and ethanol.

d) 21<sup>st</sup> June

It was not possible to run experiments because we could not change the gap on the undulator due to general ESRF failure.

e) 22<sup>nd</sup> June

Experiments at RT were performed in several conditions in order to get information about radiation damage for the next experiments. The quick scan acquisition mode was used and some experiments were performed with a high degree of resolution in energy in order to make possible the observation of 1eV shifts in the pre-edge peak.

As it may be concluded we were not able to perform the expected experiments and we think that the proposal with this project has to be resubmitted.

However we also think that the experiments with our compounds were useful for us in what concerns the next experiments and also for the set up of this new beamline.

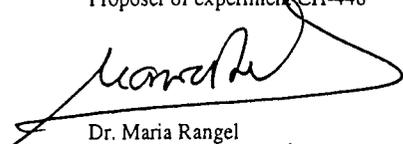
During our stay we had the opportunity:

- To contact with the scientists of the beam line and exchange ideas about the experiments and instrumentation.
- To get acquainted with the possibilities of the beamline in loco in particular the possibility of using the quickscan acquisition mode that we think will be very important to analyze our samples.
- To test our samples in what concerns radiation damage at room temperature.

Finally we would like to add that apart the assistance we got from the beamline scientists and technicians we also received very good support in the Chemistry Laboratory.

25<sup>th</sup> August 1998

Proposer of experiment CH-448



Dr. Maria Rangel