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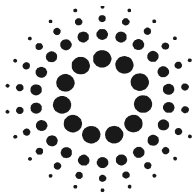

# Experiment Report Form

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. This double-page report will be reduced by ESRF to a one page, A4 format, and will be published in the Annex to the ESRF Annual Report.

Should you wish to make more general comments on the experiment, enclose these on a separate sheet, and send both the Report and comments to the User Office.

When preparing your report, please follow the instructions below:

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- make sure the report does not exceed the space available; tables and figures may be included if you wish.
- for work which is published or which is in press, you may simply include a copy of the abstract together with full reference details. If the abstract is in a language other than English, ensure that you include an English translation.
- bear in mind that the report will be reduced to 71% of its original size. A type-face such as “Times”, 14 points, with a 1.5 line spacing between lines for the text produces a report which can be read easily.

Note that requests for further beam time must always be accompanied by a report on previous measurements.

Postal address: User Office, ESRF, B.P. 220, F-38043 GRENOBLE Cedex, France.
Telephone: (33) 76 88 25 52; Fax: (33) 76 88 20 20; e-mail: useroff@esrf.fr.

| SNBL | **Experiment title:**<br>Fe ions and cation-ligand complexes in zeolites as catalysts for deNO$_x$ processes and benzene oxidation. | **Experiment number**:<br>01-01-239 |
|---|---|---|
| **Beamline**:<br>BM1B | **Date of experiment**:<br>from: 26/11/2000 to: 1/12/2000 | **Date of report**:<br>March 26, 2001 |
| **Shifts:**<br>9 | **Local contact(s)**:<br>Hermann Emerich | *Received at UNIL:* |

**Names and affiliations of applicants** (* indicates experimentalists):

L. Drozdová*, P. Marturano* and R. Prins

Laboratory for Technical Chemistry, ETH Zürich, 8092 Zürich, Switzerland

**Report**:

***Introduction***

Iron – exchanged ZSM-5 zeolite catalyst is one of the most promising systems for the selective catalytic reduction of nitrogen oxides. Our last EXAFS experiments confirmed that reproducible results are obtained for the catalyst prepared by solid state ion exchange via sublimation of $FeCl_3$, followed by washing and calcination in flowing oxygen. Binuclear Fe (hydr)oxo-species were revealed as active sites for the first time [1]. The present study focused on the individual preparation steps in order to explain the formation of the active binuclear species.

***Experimental***

Fe - K edge EXAFS spectra were recorded in transmission mode at liquid nitrogen temperature. The data were analyzed by standard procedures using the XAFS Data-Analysis

Program XDAP – Version 2.2.3.

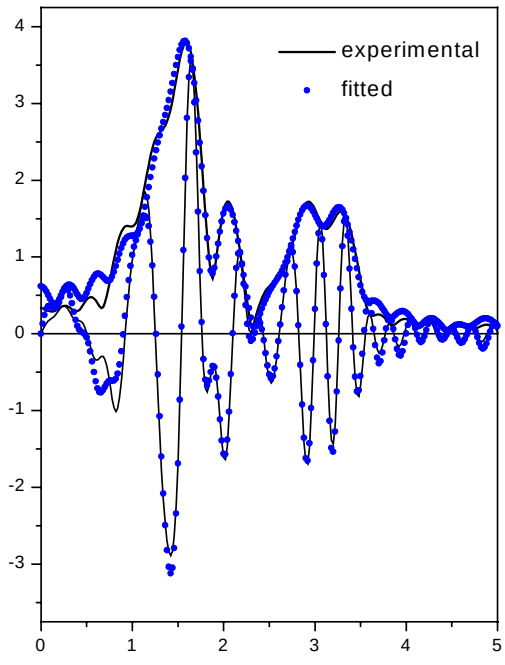


Fig.1

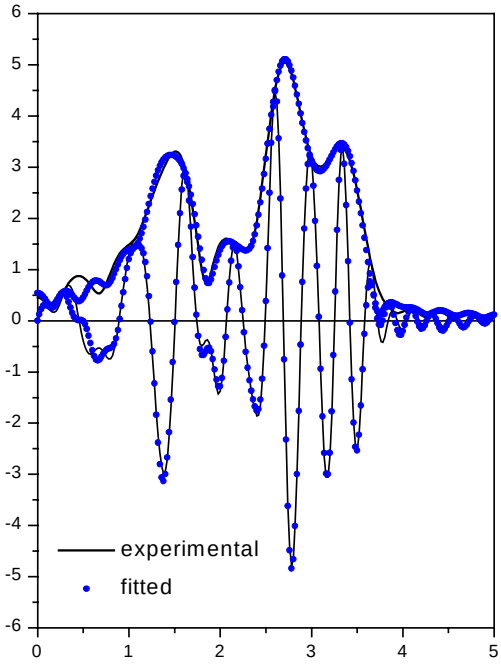


Fig.2

***Results***

*Sublimation*

EXAFS spectra of ZSM-5 sample were measured after sublimation of $FeCl_3$ without exposure to air. The absence of a Fe –Fe shell in the region from 2 to 3 Å indicates that isolated Fe ions are exchanged into the zeolite. This result was confirmed by data analysis which showed that the first shell consists of Cl and O ligands.

*Washing*

Washing seems to be a crucial step in the preparation of the active catalyst. The Fourier transformed spectrum of the sample after sublimation and washing is presented in Fig. 1. The data analysis showed that binuclear species are formed already during this washing step.

A sample after sublimation, directly calcined and not exposed to air, was measured for a comparison. Analysis of the spectrum presented in Fig. 2 confirmed that $Fe_2O_3$ particles are formed and that the washing is a necessary step to produce active binuclear species.

***References***

1. P. Marturano, L. Drozdová, A. Kogelbauer and R. Prins, *J. Catal*. **192**, 236 (2000).