EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:** Investigation of the local environment of cobalt in cobalt-containing mesoporous catalysts	**Experiment number**: 01-01-659
Beamline: BM 01B	**Date of experiment**: from: Jun 14th 2004 to: Jun 17th 2004	**Date of report**: Aug 24th 2004
Shifts: 6	**Local contact(s)**: Wouter Van Beek	*Received at ESRF:*

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Report:

Background

The experimental work done at the ESRF which this report is based on, is part of a larger project entitled *"Interfacial chemistry of cobalt (II) during sol-gel synthesis of cobalt-containing mesoporous materials"*. A manuscript including all the results from this project is now under preparation. Cobalt-containing mesoporous materials have potential applications within heterogeneous catalysis, and the objective of this project was to understand the chemistry during the synthesis of these materials. Cobalt was incorporated into two types of mesoporous materials; MCM-41 and MCM-48.

At the ESRF, several samples were investigated by EXAFS spectroscopy. The purpose of this work was to investigate the local environment of cobalt in the samples before and after calcination, i.e. to verify that the cobalt was actually incorporated into the silica mesoporous framework during synthesis, and to observe any changes during calcination.

Results

The results from the EXAFS curve fitting for some of the samples are listed in Table 1, and the EXAFS curves and Fourier Transforms for as-synthesised and calcined Co-MCM-41 are shown in Figures 1 and 2, respectively. These figures are representative for all the samples investigated.

Table 1: Results of EXAFS curve fitting for as-synthesised and calcined samples Co-MCM-41 and Co-MCM-48

Sample	**Shell**	**N**	**R (Å)**	**$2\sigma^2$ (Å^2)**
41 as-synth	Co – O	4.9	2.073 (3)	0.0096 (9)
	Co ··· Si	6.9	3.297 (3)	0.0091 (9)
41 calcined	Co – O	4.0	2.055 (4)	0.013 (1)
	Co ··· Si	6.3	3.275 (4)	0.014 (1)
48 as-synth	Co – O	5.5	2.056 (5)	0.016 (2)
	Co ··· Si	8.0	3.300 (4)	0.010 (4)
48 calcined	Co – O	4.3	2.026 (3)	0.015 (1)
	Co ··· Si	4.1	3.254 (4)	0.0108 (9)

Table 1 show that only two backscattering shells were detected; oxygen and silicon. No cobalt shells were detected for any of the samples, which indicate that no cobalt oxide or metallic cobalt is present in the samples. Some changes in local environment occured during calcination, but the cobalt remained incorporated into the silica framework.

Conclusion

The EXAFS investigations performed at the ESRF verified that the cobalt was successfully incorporated into the silica mesoporous framework in all the samples.

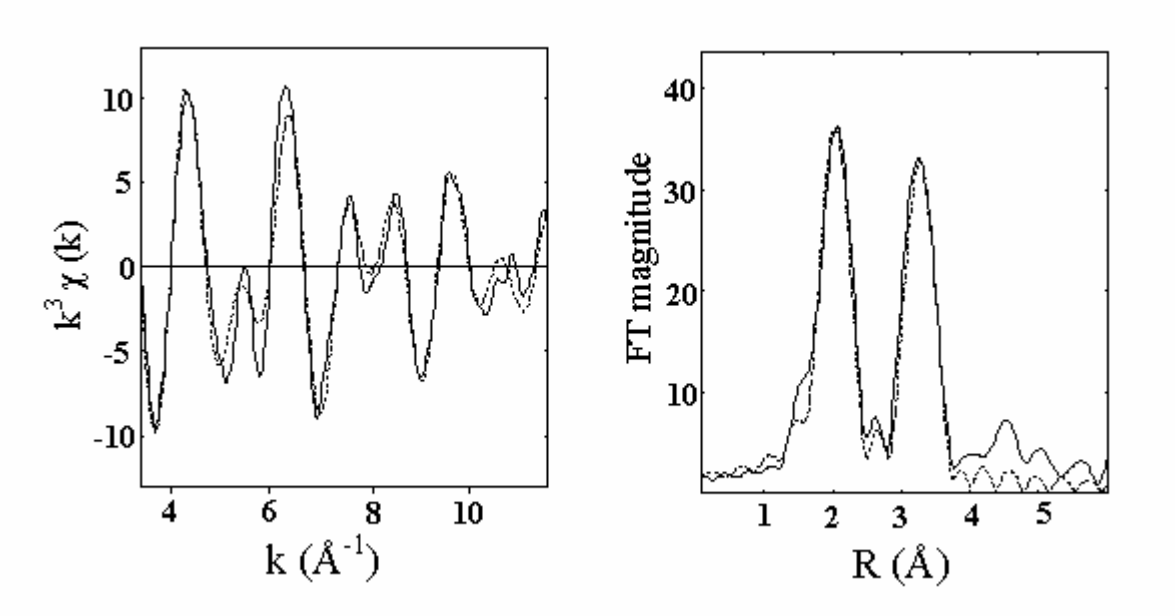

Figure 1: EXAFS-curve (left) and Fourier Transform (right) of as-synthesised sample Co-MCM-41

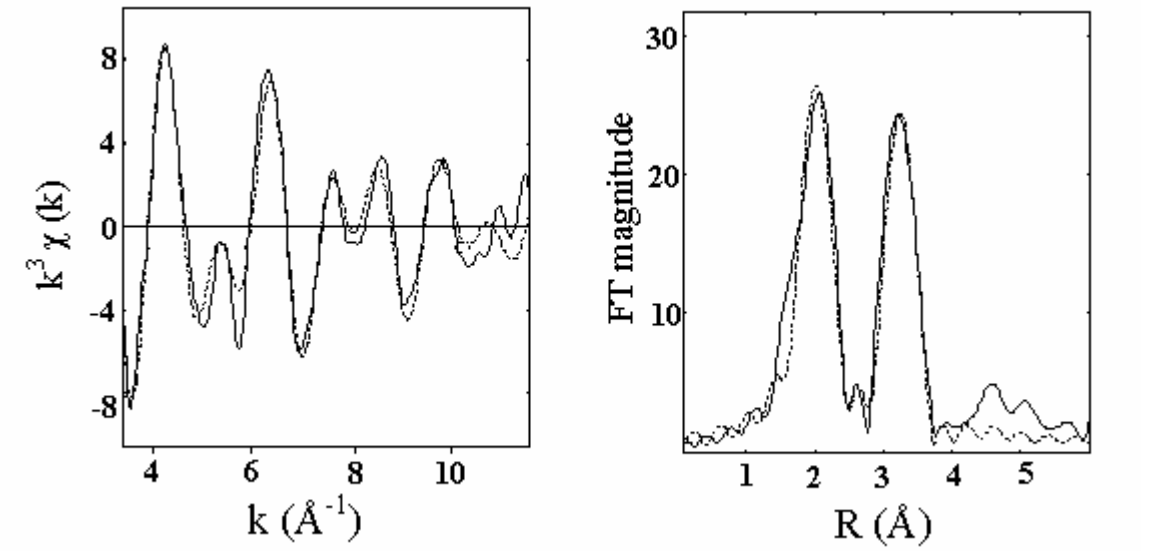

Figure 2: EXAFS-curve (left) and Fourier Transform (right) of calcined sample Co-MCM-41