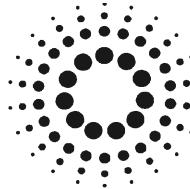


EUROPEAN SYNCHROTRON RADIATION FACILITY

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. 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.

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- 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.



	Experiment title: EXAFS study of supported phosphide catalysts	Experiment number: 01-01-256
Beamline: BM01B	Date of experiment: from: 04 June 2001 to: 08 June 2001	Date of report: 27/09/2001
Shifts: 9	Local contact(s): Mr Hermann EMERICH	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Dr. Viviane Schwartz Dr. Virginie Zuzaniuk Christoph Stinner		
Report: <i>Introduction</i> There has been an increasing interest in developing novel catalysts for hydrotreating reactions in order to comply with the more stringent environmental regulations. Recently, transition metal phosphides have been reported to show better hydrodenitrogenation activities than the traditional molybdenum sulfide catalyst [1]. The knowledge of the structure of the active component is mandatory for understanding the catalytic activity of these materials. However, the catalysts are usually prepared on a support, like Al ₂ O ₃ or SiO ₂ on which the active particles are highly dispersed with particle sizes below 5 nm. Since no other laboratory technique is suitable for the analyzes of the structure of the active components, we aim to utilize EXAFS as a characterization tool for the supported CoMoP catalysts. <i>Experimental</i> The catalysts were pressed in self-supported wafers, and mounted in a sealed EXAFS cell. In this work, we investigate the SiO ₂ -supported CoMoP and Co ₂ P catalysts and we compare them with the bulk materials. Mo K-edge and Co K-edge EXAFS spectra were recorded in transmission mode at liquid nitrogen temperature. The program XDAP – version 2.3.3 was used to analyze and fit the data as described in the literature [2].		

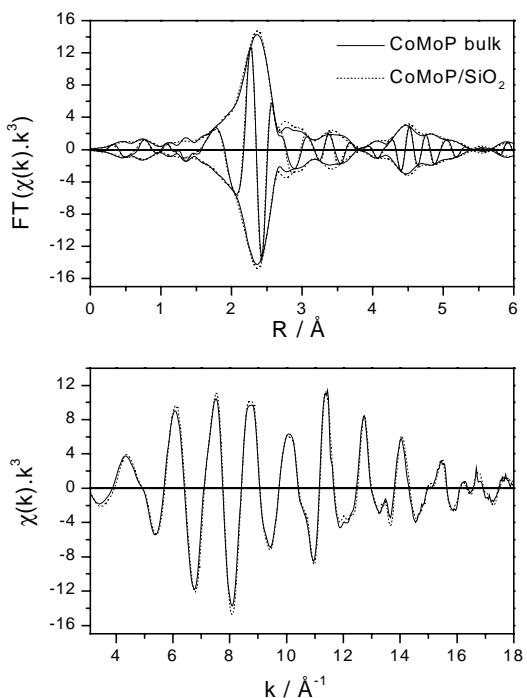


Figure 1: Fourier transformed and k^3 - weighted Mo K-edge EXAFS function ($k^3 \cdot \chi(k)$) of bulk and supported CoMoP.

Results

Fourier transformed and the $\chi(k) \cdot k^3$ Mo K-edge EXAFS functions of the bulk and supported CoMoP catalysts in the range of $3.1 \text{ \AA} < k < 18 \text{ \AA}$ are shown in Figure 1. The spectra are nearly identical revealing that the same structure is formed when the phosphide is prepared as a bulk material or supported on SiO_2 . The data analysis shows an overlapping of two P and one Co shells in the region from 2.4 and 2.7 \AA (phase-corrected). A second Mo-Mo shell was found at a distance of 3.05 \AA and a third P shell was found at 3.42 \AA (phase-corrected). Similar coordination numbers were obtained for the bulk and supported CoMoP materials. However, data analysis showed that less Co atoms and consequently more Mo atoms were found to be coordinated to a Mo atom. Investigation of the Co K-edge for both materials confirmed that Co are coordinated to more Co atoms than Mo atoms when comparing the data to the theoretical values of a CoMoP compound [3]. We believe that Co is not only present in a CoMoP structure but also present in a segregated phosphide phase.

References:

- [1] C. Stinner, R. Prins, and T. Weber, *J. Catal.* 191, 438 (2000).
- [2] M. Varkaamp, J.C. Linders, D.C. Koningsberger, *Phys. Rev. B* 209 (1-4), 159 (1995).
- [3] R. Guerin, M. Sergent *Acta Cryst B* 34, 3312 (1978).

