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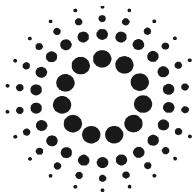

Experiment Report Form

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:** LTL and MCM-41 supports for Pt and Rh metal catalysts	**Experiment number**: 1-01-238
Beamline: BM1B	**Date of experiment**: from: 22/11/2000 to: 26/11/2000	**Date of report**: 28/3/2001
Shifts: 8	**Local contact(s)**: Hermann Emerich	*Received at UNIL:*

Names and affiliations of applicants (* indicates experimentalists):

L. Drozdová*, R. Cattaneo* and R. Prins

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

Report:

Introduction

Pt, Rh and Pd carbonyl complexes were successfully synthesized inside the cavities of zeolites by the "ship-in-bottle" synthesis. Their decomposition leads to uniform metallic particles homogeneously dispersed over the zeolite support. The carbonyl complexes and bare metallic particles are active in a variety of reactions, mainly in CO+NO conversion, water gas shift reaction and naphtha reforming. The aim of this study is to determine the structure of bimetallic PtRh carbonyl complexes prepared by the ship-in-bottle technique. The presence of Pt-Rh bonds in bimetallic particles can be proved by the coexistence of Pt-Pt and Pt-Rh shells in Pt L_{III} edge, as well as Rh-Pt and Rh-Rh shells in Rh K edge EXAFS spectra.

Experimental

Rh K and Pt L_{III} 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.

The complexes were prepared by carbonylation of $[Rh(NH_3)_5Cl]^{2+}$ and $[Pt(NH_3)_4]^{2+}$ ions

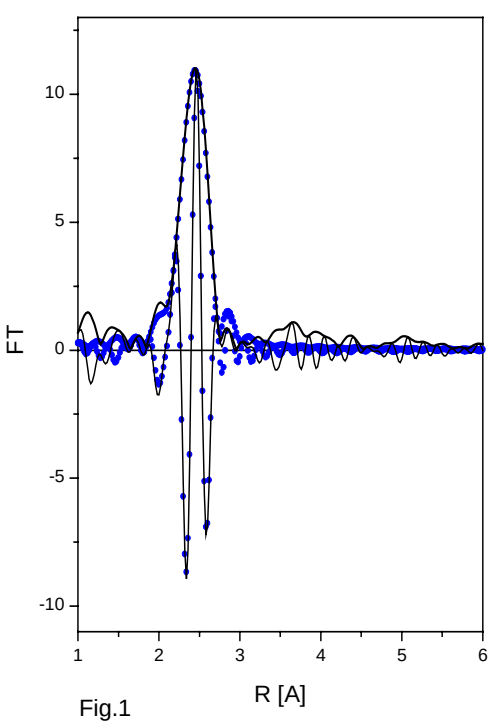

Fig.1

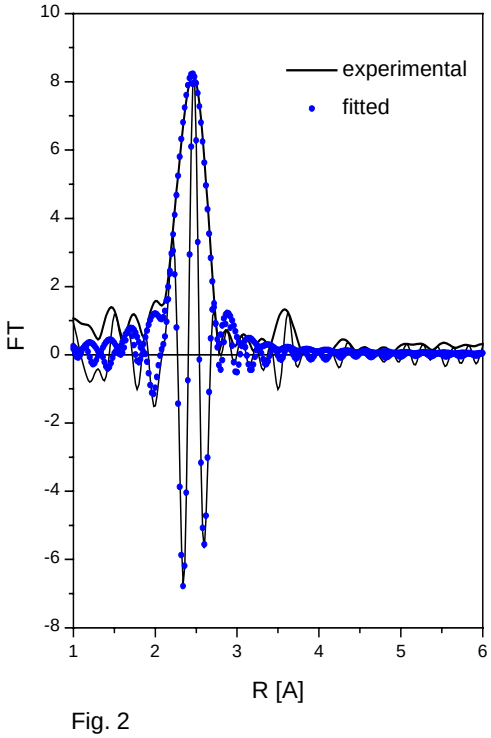

Fig. 2

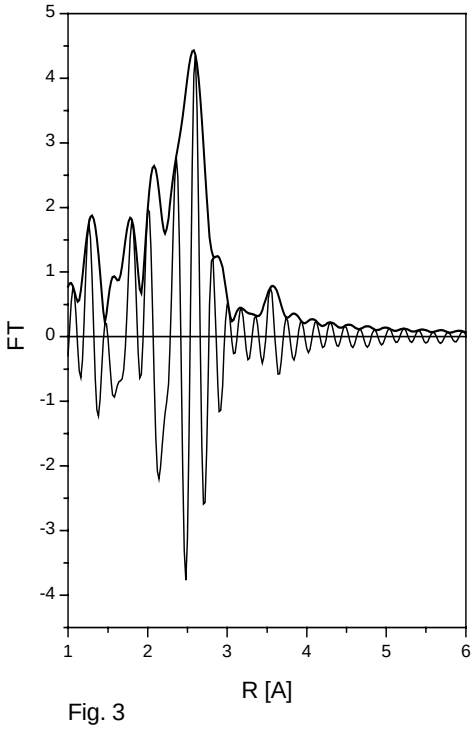

Fig. 3

exchanged into zeolite NaX.

Results

Rh edge EXAFS spectra

The first sample was carbonylated for 14 hours and the Fourier transformed spectrum is shown in Fig.1. The spectrum was fitted by one Rh-Rh shell and the interatomic distance of 2.79 Å is significantly longer than the one observed for pure metal (2.68 Å). The very low coordination number 2.6 points to the formation of very small Rh clusters. It can be concluded that no bimetallic particles are formed because it was impossible to fit the spectrum with Rh-Rh and Rh-Pt shells together.

The second sample was carbonylated for 80 hours and the Fourier transformed spectrum is presented in Fig.2. The best model to fit the spectrum consists of one Rh-Rh and one Rh-Pt shell. The Rh–Rh interatomic distance was again 2.79 Å and the Rh–Pt distance was found at 2.65 Å. The very low coordination numbers show that very small bimetallic clusters are formed and that a longer time of carbonylation is required to form bimetallic particles.

Pt edge EXAFS spectrum

The Fourier transformed spectrum of the sample carbonylated during 80 hours is shown in Fig. 3. The peak between 2 and 3 Å can correspond to Pt–Pt as well as Pt-Rh shells. Pt-C and possibly also Pt–O shells were detected in the range of 1 to 2 Å. Further data analysis of Pt edge spectra is in progress.