

EXPERIMENTAL REPORT

RAPPORT D'EXPERIENCE

Programme Committee Proposal Number
N° Projet Comité de Programme
32-1-138

PROJECT TITLE : *TITRE DU PROJET* :

Characterization of sulfide catalysts doped by low contents of noble metals.

LIGNE :	D2AM	IF
INSTRUMENT :	PETITS ANGLES <input type="checkbox"/>	EXAFS <input checked="" type="checkbox"/>
	7 CERCLES <input type="checkbox"/>	GM <input type="checkbox"/>
	FIP <input type="checkbox"/>	SUV <input type="checkbox"/>

NUMBER OF RUNS USED

NOMBRE DE SESSIONS EFFECTUEES : 9

STARTING DATE

DATE DE DEMARRAGE : November 19, 1999

AUTHORS : *AUTEURS* : Geantet C., Pessayre S., Soldo Y., Hazemann J.L.

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An in-situ QEXAFS study of the reduction and sulfur poisoning of catalysts containing noble metal at a low loading (0.3 wt%) has been performed in fluorescence mode at Pt L_{III}-edge (11450-12000 eV).

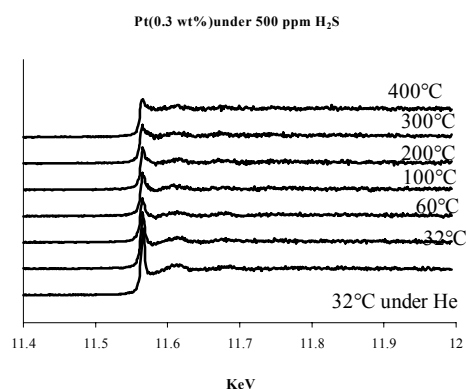


Fig1. :QEXAFS at Pt L_{III} edge Under *in-situ* sulfidation conditions.

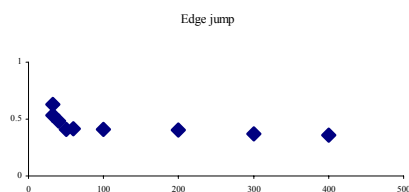


Fig2. : Edge jump intensity versus T

An in-situ cell using a T-quartz tube was designed for the fluorescence measurements upon thermal treatment (up to 673 K, ramp 4K/min).

The use of the Canberra detector allowed a time resolved study of the reduction and/or sulfidation of the Pt based catalysts. Each 10 K, a spectrum was acquired in 30 sec. Analysis of the XANES showed that on alumina reduction around Pt atoms proceeds rapidly and is almost completed at 100°C. When the sample is treated with H₂/H₂S, it rapidly reacts at room temperature. QEXAFS experiments were completed with conventional XAFS measurements. XAFS evidenced that, under 500 ppm of H₂S, a metallic contribution remains for 1 wt-% loading suggesting a surface sulfidation and a metallic core where for 0.3 wt% sample Pt is fully sulfided. When Pt is used as a dopant of sulfide hydrotreating catalysts, preparation is of primary importance. XAFS evidenced that after impregnation of platinum on the sulfided catalysts, sulfidation of Pt occurs at room temperature and is responsible for a better dispersion of the catalyst*.

* S Pessayre, thèse Lyon1 (Oct 2000)