X-ray Absorption Spectroscopy

The technology at a glance

X-ray absorption spectroscopy techniques provide information on the atomic organisation and chemical bonding around an absorbing atom in whatever medium it is embedded, i.e. solids and liquids. There are essentially two types of absorption spectroscopy: X-ray Absorption Fine Structure (EXAFS) and X-ray Absorption Near Edge Structure (XANES). Both techniques are element-selective, which means that scientists and researchers can study and characterise elements in their "working state" within compounds.

The added value of the ESRF absorption spectroscopy facilities

The ESRF offers a suite of three beamlines for absorption spectroscopy, each optimised for different applications, thus enabling research on a wide range of materials to detect very diverse factors under various conditions.

• ID24 is ideal for fast chemical processes and can be used for rapid micrometre-resolution *in situ* mapping of heterogeneous samples. Currently, the core activity of ID24 is to investigate catalysts in operation in subsecond sequences and to produce relatively high-resolution mapping. It is also well suited for studies at extreme pressure levels and within high magnetic fields in pulsed mode.

> "Absorption spectroscopy allows us to know not only what a molecule does, but above all why. It helps us find underlying principles and changes in molecular activity *in situ*."
>
> - Mark Newton and Pieter Glätzel, beamline scientists

• ID26 is a high-brilliance X-ray spectroscopy beamline for absorption studies on samples with low levels of concentration, such as highly-diluted trace elements or toxic chemicals. Detailed information on the electronic structure can be obtained by employing a high-energy resolution setup. It is equipped for different sample environments to perform *in situ* studies. It can also be adapted to a variety of user experimental stations. Fast scanning software allows efficient data collection.

• BM29 is the general purpose X-ray absorption spectroscopy beamline at the ESRF. It offers conventional X-ray absorption spectroscopy to perform experiments which do not require the specialist characteristics of other ESRF X-ray absorption instruments. It offers a large-energy range bending magnet which provides high-quality data based on the quick scanning of spectrums. It offers a good balance between quality of data and speed of utilisation.

Recently, BM29 undertook micro XAS measurements, producing quality of data that surpasses other similar beamlines worldwide.

Fields of application

Environmental science: trace elements, including identification of toxic concentrations of heavy metals, analysis of air pollution filters, effluent separation techniques, etc.
Earth and planetary science: geological studies, mapping, mineral characterisation.
Medicine and pharmacology: cancer cell research, observation of underlying activities and molecular activity of active ingredients and formulations *in situ*.
Automobile industry: study of key components, including fuel cells and catalytic converters.
Oil industry: analysis of trace elements in petroleum and petrochemical products.

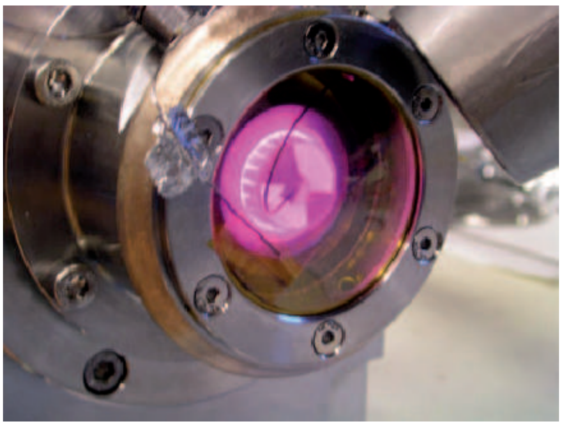
"We come to the ESRF since they offer unique techniques and are the only synchrotron facility able to build us a dedicated sample cell for *in situ* measurement."
- Toyota Motor Europe nv/sa (Belgium)

Corporate clients include Toyota, BASF, Total, Johnson-Matthey, IFP (Institut Français du Pétrole – French Petroleum Institute), Daihatsu

CASE STUDY

Toyota studied exhaust catalysts using X-ray techniques to examine catalyst surface structure and the chemical reactions taking place.

The challenge: To study the noble metal components of a working vehicle exhaust catalyst under *in situ* conditions and in real time.
Background: A supported metal catalyst is responsible for much of the oxidation of CO and unburned hydrocarbons in car exhaust gases. These catalysts lose efficiency as they age, due in particular to the sintering of the supported noble metals used in the catalysts. Toyota wanted to study the sintering process in detail to improve the catalysts.
Results: Data analysis led to the discovery of an unexpected phenomenon: efficient oxidative redispersion of Pt nanoparticles on some metal oxide supports during quick redox cycling. This redispersion process led to a tangible potential for incorporation into "on board" methodology for extending vehicle catalyst lifetime through curtailing or reversing the effects of metal sintering during operation.



Heating a catalyst sample in the *in situ* cell for time-resolved XAFS.

How did the synchrotron help? Using a purpose-built cell, energy-dispersive X-ray absorption scattering was used to study the local environment and electronic structure of the Pt metallic active site. The experiment was done at working exhaust conditions (high temperature and fluctuating oxidative and reductive gas compositions), while *in situ* TEM was used to study the catalyst surface. Infrared/EXAFS experiments were also used to study other major components of the catalytic system, Rh and Pd. Spectra were recorded on a millisecond timescale.