	Experiment title: High-pressure XANES study of sulphur speciation in crude oil.	**Experiment number**: ES-181
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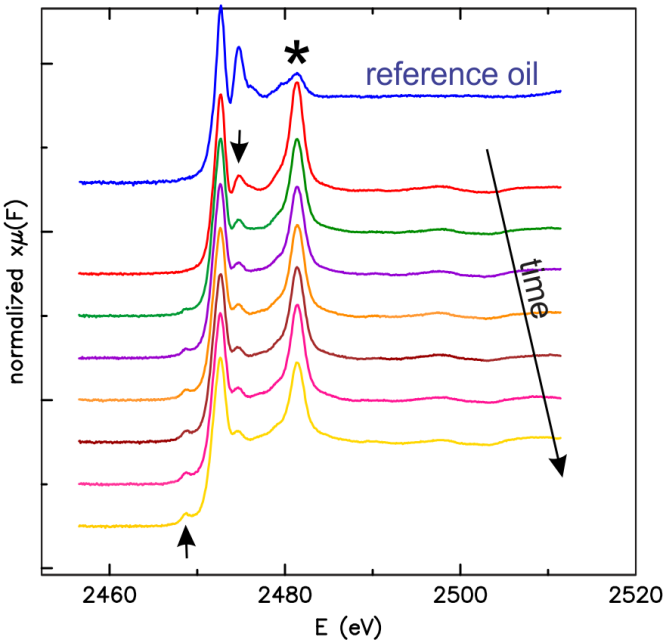
Report:

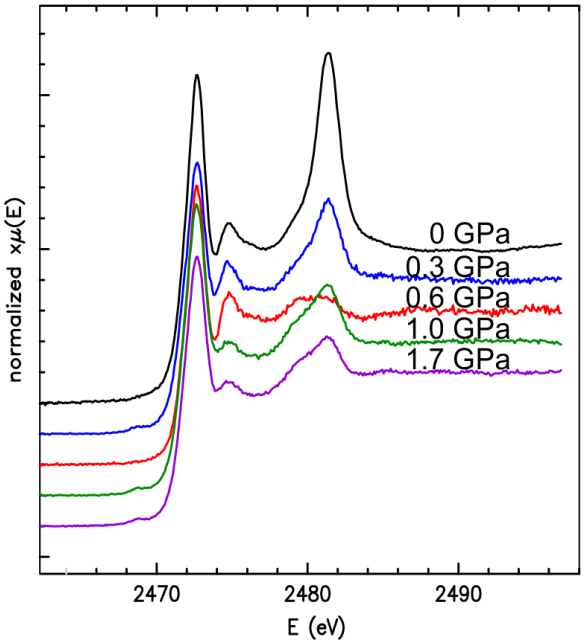
Experiments under high pressures at a low energy range (~2.5 keV) imposes a lot of technical challenges, and most of them were succesfully resolved. Ultra-thin (30 microns) diamond windows demonstrate sufficient strength to maintain pressure at least up to 7 GPa (in one test run), while still having enough X-ray transmittance.

However, two main technical problems are still not resolved completely: sulpher contamination of the sample environment and radiation damage of the soft matter samples.

As was found experimentally, the commercially available diamond windows contain a detectable amount of sulpher in a sulfate form (Fig. 1), mostly at the laser-cut edges. This contamination occurs due to the windows cleaning in a sulphuric acid (that penetrates inside the microcracks) during manufacturing, and it cannot be easily removed. To reduce the contamination effect due to the X-ray beam tales we used CRL focusing instead of a mirror, which helped to a certain extent, but at the same time also enhansed the radiation damage of the sample due to the harmonics (see Fig. 1).

Nevertheless, we demonstrated a possibility to obtain a high-quality XAS spectra in a tender X-ray domain under high pressure, also from natural samples with a low (~1.5%) concentration of sulphur (Fig. 2). The observed changes of S K-edge XANES spectra in crude oil as a function of pressure were very subtle, and these small changes could be probably attributed to the radiation damage of the sample.





Left (Fig. 1). The reference S K-edge XANES spectrum of the crude oil from West Siberia (top blue curve) and a sequence of spectra of the same sample insite the diamond anvil cell taken as a function of time (top to bottom). The star mark shows a feature at ~2482 eV, coming from the sulphur contamination of the diamond windows. Two arrows indicate spectrl features, specific to the sample radiation damage: a growing pre-edge peak at ~2468 eV and a reducing feature at ~2454 eV.

Right (Fig. 2). XANES spectra of the natural oil sample as a function of pressure at ambient temperatures. No changes in the sulphur oxidation state were observed, and the small spectra changes are probably attributed to the radiation damage.