

# New quantum cascade laser – FT-IR combined spectrometer for microsecond time-resolved *in situ/operando* spectroscopy

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To study the kinetics of dynamic phenomena that are not reversible (typically because the material can get modified under working conditions), the interferometer of FT-IR spectrometers needs to be handled in the so-called rapid-scan mode. So, its mobile mirror needs to be moved in a continuous way with a scanning rate that dictates the time resolution, namely tens of milliseconds to the best [1-2]. To boost the time resolution of our rapid-scan FT-IR spectrometer without compromising its standard functionalities, we have coupled it with quantum cascade lasers (QCLs) tunable within the 1876 – 905 cm<sup>-1</sup> spectral range, within one single setup. The spectrometer now enables a fast and easy switch between the standard FT-IR mode, used for classical broadband scans from 6000 to 650 cm<sup>-1</sup> (30 ms maximum time resolution), and the new QCL-irradiation mode, used for ultrafast recording at specific wavenumbers (5 μs time resolution). So, for the first time, one can study a sample during a physical or chemical transformation first as a whole, and then immediately switch to the QCL mode to follow a selected vibration (associated to an intermediate, a structural change, a diffusing substance, ...) *versus* time [3].

In the present work, we demonstrate how useful this new methodology is when monitoring adsorption/diffusion phenomena within porous materials, by taking the example of gaseous ammonia diffusing into a commercial H-ZSM-5 zeolite. We *in situ/operando* monitor a zeolite sample 1) during a pre-treatment procedure with a standard 4 cm<sup>-1</sup> spectral resolution (standard FT-IR spectroscopy), and 2) under working conditions with microsecond time resolution (QCL diagnostic), in a single experiment. The QCL diagnostic reveals changes in the rate-limiting step with increasing ammonia pressure which were impossible to observe via standard FT-IR spectroscopy. Indeed, in contrast to FT-IR, the QCL mode catches the whole uptake curves instead of only their slowest period, i.e. their very end [3].

Applications of the new spectrometer to unravel catalytic reaction mechanisms are currently in progress.

## References

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