Monitoring Industrial Processes Using a Sensor Based on an External Cavity-Quantum Cascade Laser

Andreas Genner[1], Markus Brandstetter[1], Michael Harasek[2], Bernhard Lendl[1]

[1] Institute of Chemical Technologies and Analytics, Vienna University of Technology, Getreidemarkt 9, A-1060 Vienna, Austria
[2] Institute of Chemical Engineering, Vienna University of Technology, Getreidemarkt 9, A-1060 Vienna, Austria

Introduction

The increased performance and commercial availabilty of Quantum Cascade Lasers (QCL) made it possible to move from experimental setups in well protected labs to real world applications in industry. Especially lasers with an external cavity (EC-QCL), supporting a tuning range of up to 600 cm^{-1} in the mid-IR, are well suited for monitoring chemical processes in the liquid phase as they can cover broad absorption bands. Although conventional FTIR-spectrometers cover an even broader spectral range, they are outplayed by sensors based on EC-QCLs because they enable, due to their orders of magnitude increased optical power density, a higher flow cell thickness.

Here, we present a sensor based on an EC-QCL (Daylight Solutions, USA) for monitoring the cleaning behavoir of an industrially used vessel. The contamination of the vessel with residuals of a chemical or biological process taking place in the vessel were simulated by coating the vessel with different substances. During the cleaning process, the rinsing water is monitored with the EC-QCL sensor. The sensor is part of the PATOV-Project (Process Analytical Technology Unit for Online Verification), aiming to optimize the Clean-In-Place processes in pharmaceutical industry.

Experimental Setup

The scheme of the experimental setup used for monitoring the cleaning process of the PATOV testing facility is shown in Fig. 1. One can see that the EC-QCL sensor consists of two parts: the liquid handling system (left) and the optical system (right).

The concentration was determined by IR-absorption measurements whereas the EC-QCL acted as an intensive IR-source (>350 mW, 1024-1230 cm^{-1}). The beam passed a flow cell (160 µm optical pathlength) containing the liquid from the process and was focused towards a TEC-MCT-detector. Using a Boxcar Integrator, the high-frequent pulses were converted into a DC-signal, digitized (ADC7760) and finally recorded on a PC.

The liquid handling system was necessary as adsorption of the process agents on the flow cell windows (CaF_2) can occur. The flow cell could be cleaned by flushing it with ethanol or acetone. Another benefit was the possibility of injecting offline samples into the measurement system for establishing calibration curves.

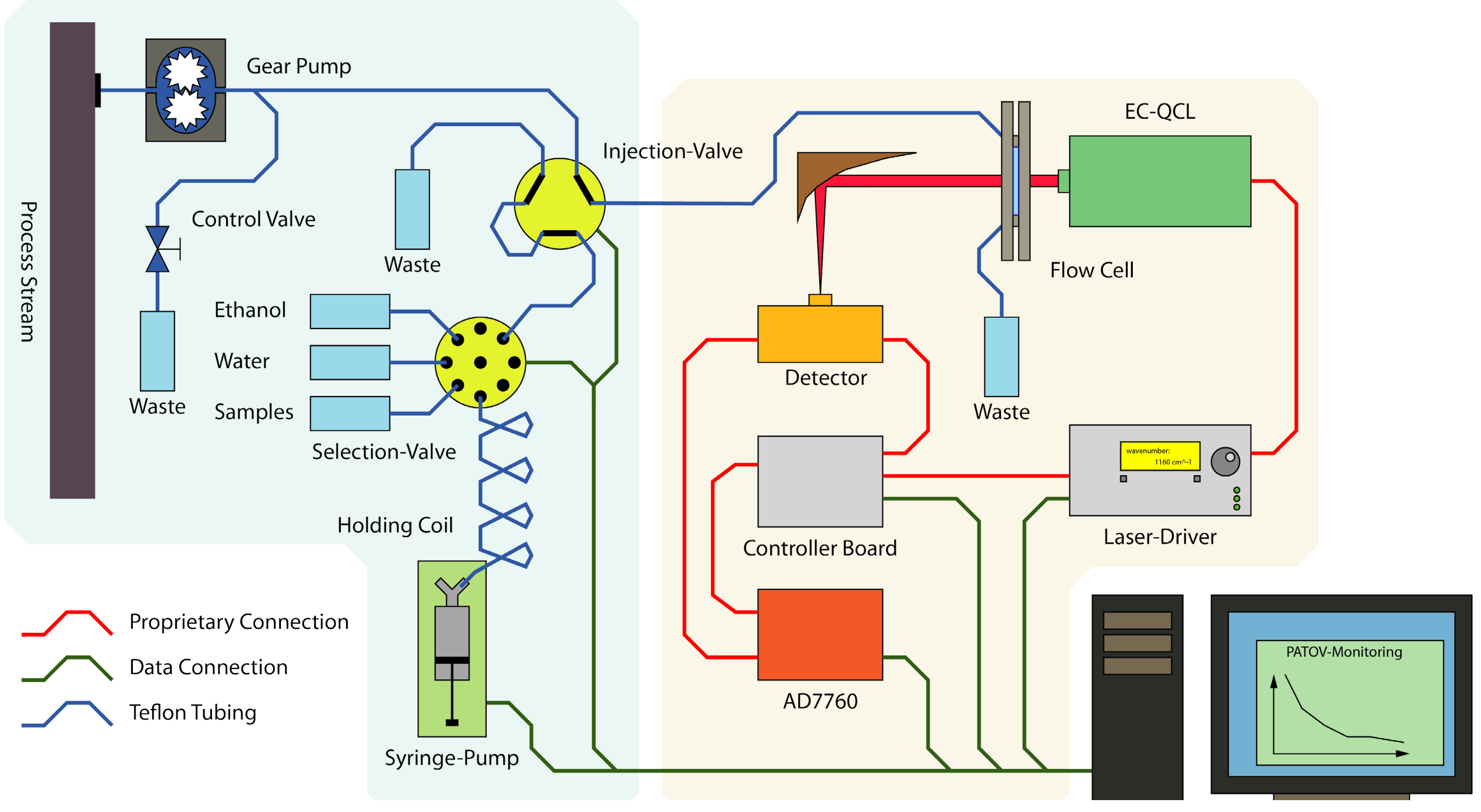

Fig. 1: Scheme of the EC-QCL sensor with the optical system (right) and the sample injection system (left)

Calibration

The linearity of the EC-QCL-based sensor was verified by analyzing calibration samples. The solid matter was weighed in, diluted with deionized water and finally measured with the sensor using the sample injection system (Fig. 1). As substance A showed a satisfying solubility, a wide concentration range was covered and calibrated. In contrast, the solubility of substance B water was limited, resulting in less calibration points and a narrower calibration range. The corresponding curves are shown in Fig. 2.

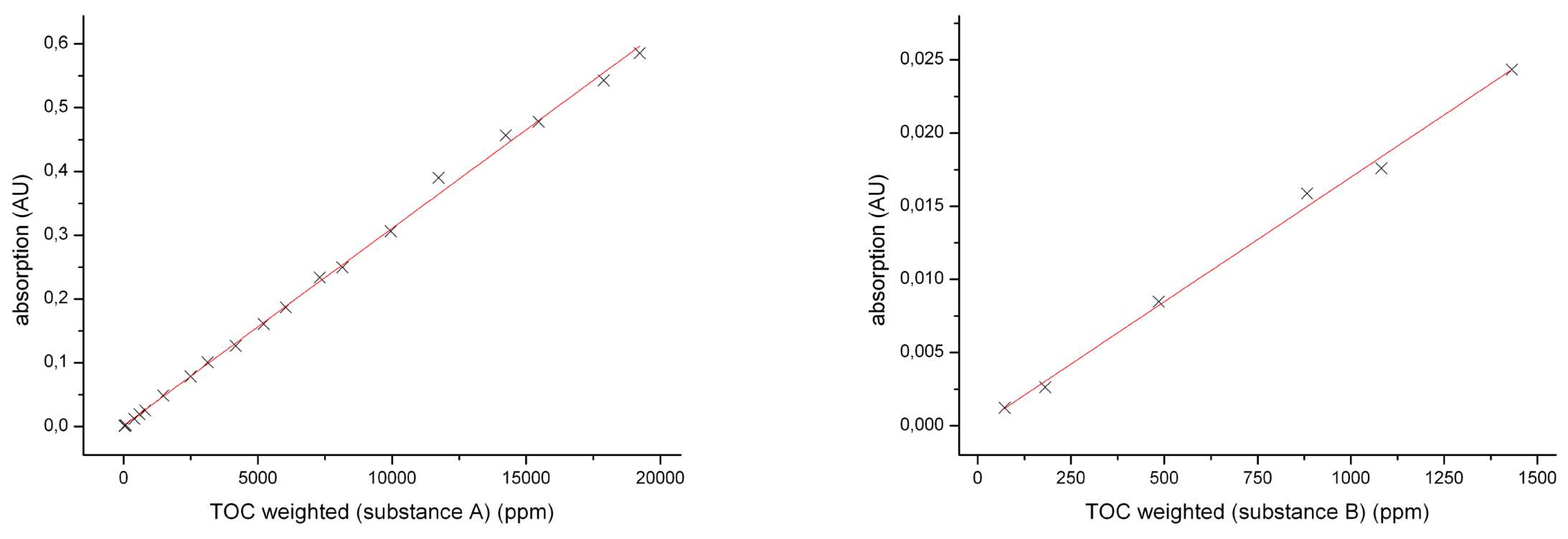

Fig. 2: Calibration curves of different organic substances

PATOV Testing Facility

The applicability of the EC-QCL-based sensor was tested in combination with a test plant shown in Fig. 4 and 5. It consisted of a water reservoir, a heating unit, a vessel simulating a chemical reactor and two pumps. A known amount of a viscouse organic substance was spread onto the vessel's surface and, immediately after that preparation step, the cleaning process was started. Shortly after the vessel, a small fraction of the stream was separated and injected into the sensor. The delay caused by pumping the liquid to the flow cell and analyzing the concentration was determined with ~15 s.

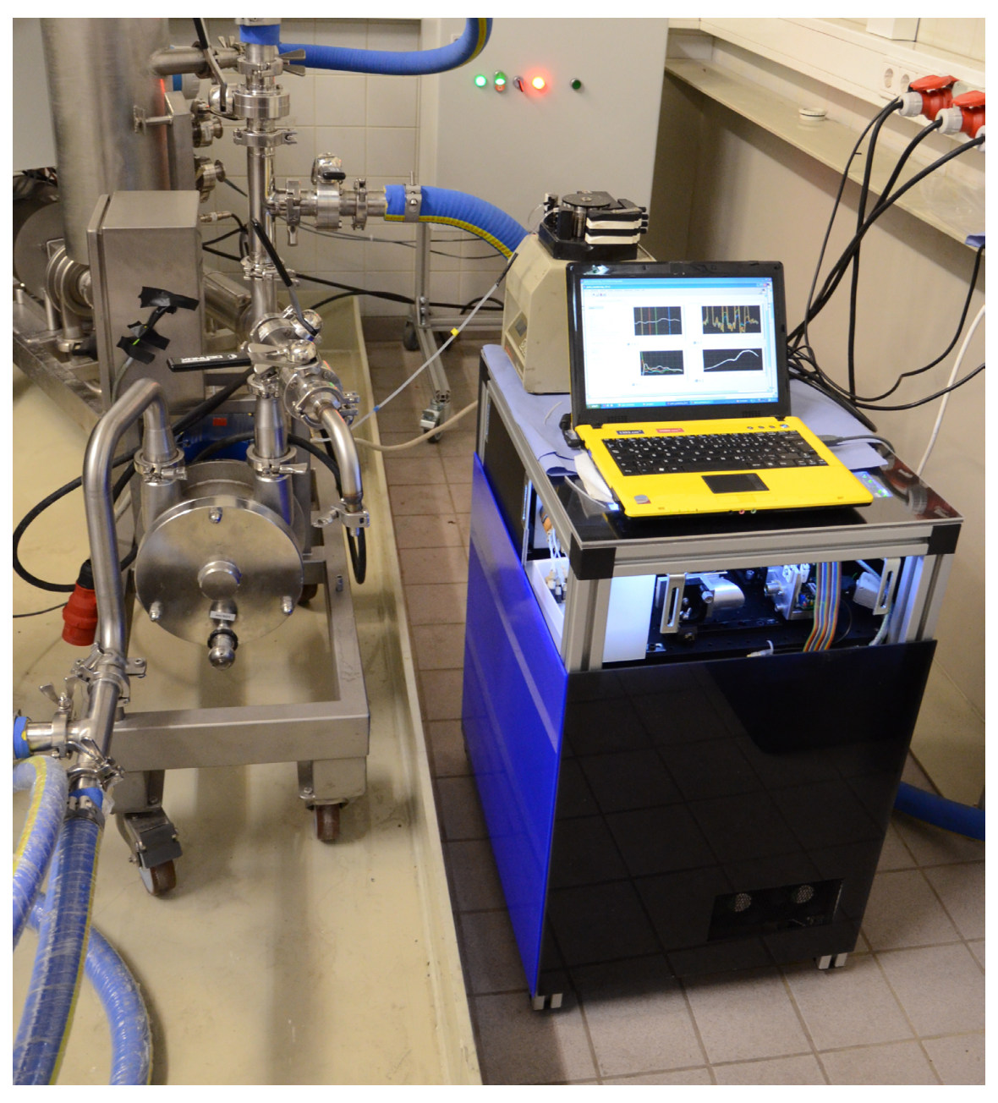

Fig. 3: Portable EC-QCL sensor setup next to the testing facility

Fig. 4: Assembled PATOV testing facility

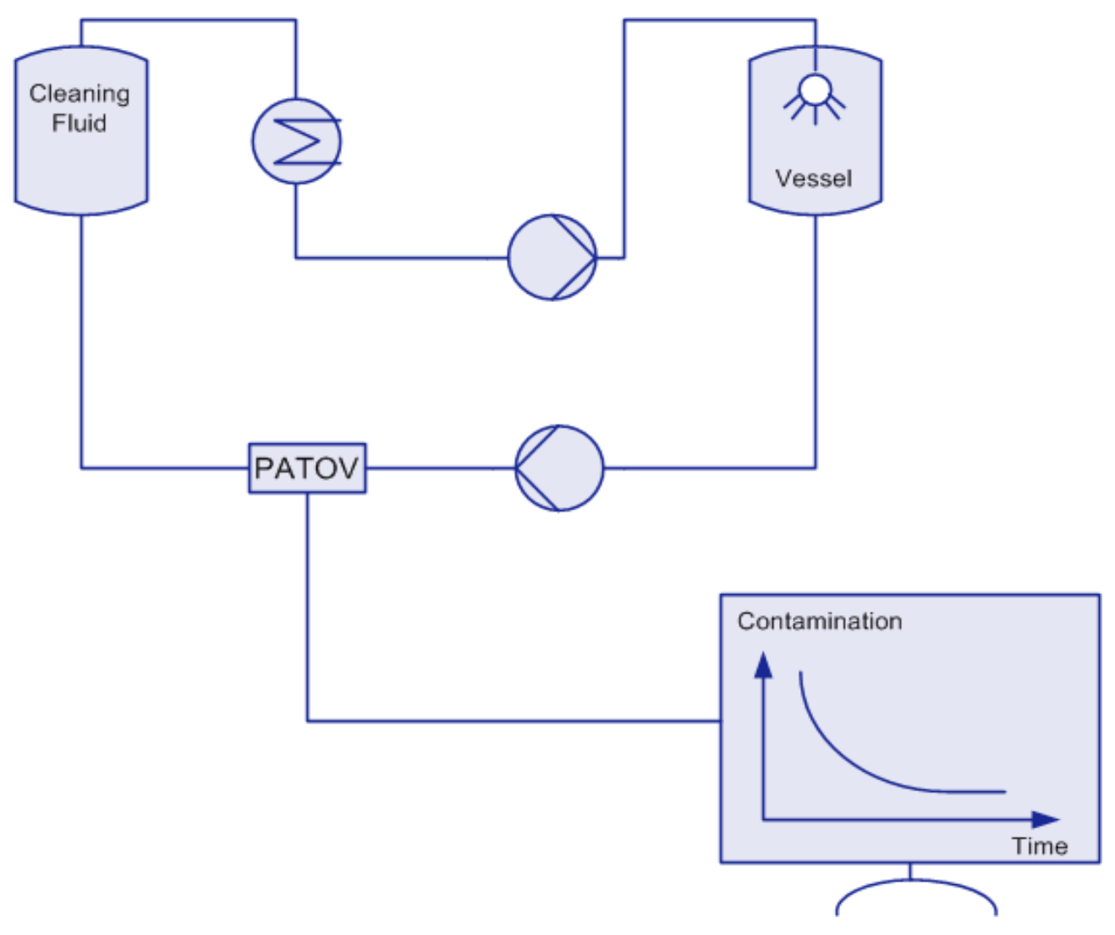

Fig. 5: PATOV testing facility scheme

Process Monitoring

A typical cleaning cycle of the PATOV testing facility is shown in Fig. 6. Deionized water was used as a reference, resulting in a flat line until the cleaning process was started. The slight increase after 25 seconds was caused by impurities in tap water. Several seconds later, two significant absorption peaks indicated the washout of the simulated residuals in the vessel. After another ~2 min, the absorption stayed constant, as the contamination was homogenuously distributed in the PATOV testing facility.

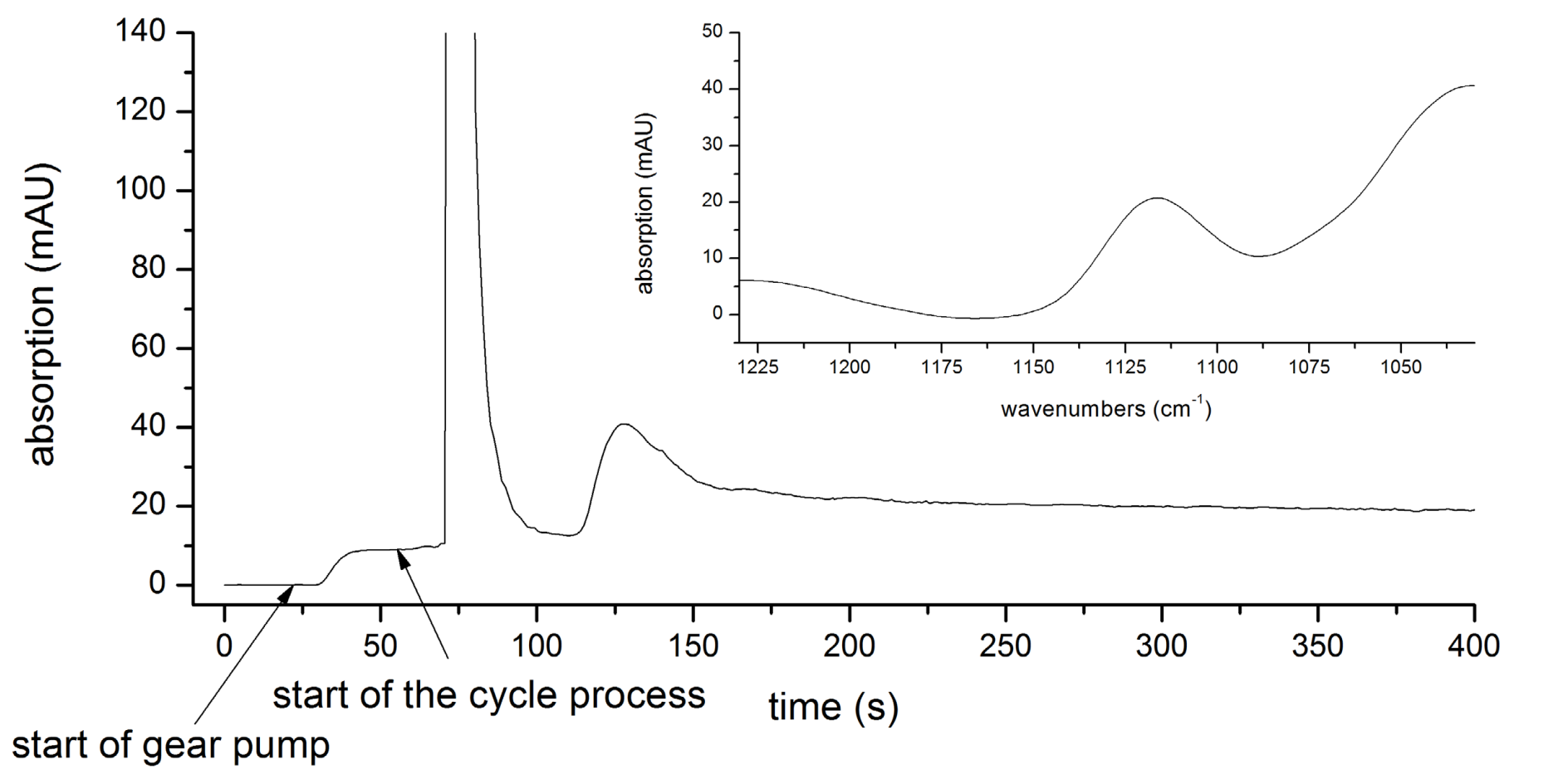

Fig. 6: Cleaning process of the PATOV testing facility. Inset: Mid-IR absorptionspectrum of a typical contamination

Outlook

The acceptance of the new EC-QCL-based sensor in the industry will be increased by the following improvements:

- interface for process control (4-20 mA current loop)
- data monitoring via TCP/IP
- expansion of the testing system towards protein contaminations
- evaluation on a differently sized/equipped testing facility

Project Partners: