APPLICATION NOTE PA201

Unknown gas identification with photoacoustic spectroscopy and broadly tunable external cavity quantum cascade laser – a case of a clean room air quality issue in a production plant

This application note covers a real life gas analysis case of a technology company. Gasera PA201 research photoacoustic gas cell was successfully used in the analysis of air samples and identification of impurities. The problem and the outcome of the analysis is gone through step by step to illustrate the possibilities of photoacoustic spectroscopy (PAS) with broadly tunable external cavity quantum cascade lasers (EC-QCL).

Fig 1. PA201 photoacoustic cell for research use with its gas exchange controller and DSP modules.

Application

The customer had a problem of low yield in their production, and analysis of the clean room air in their production plant was needed. The objective of the analysis was to find and identify possible impurities. PA201 cantilever-enhanced research photoacoustic gas cell (Fig 1) with a broadly tunable EC-QCL (Block Engineering LLC.) as an infrared source was selected for the analysis because of the versatility of this combination. Broadly tunable EC-QCLs can record an infrared spectrum, similarly to FTIR spectrometer, for an analysis of various components. The tuning range of a modern EC-QCL can be over 1000 cm^{-1} in the mid-infrared area, which covers the full fingerprint region generally used for materials identification. Further, different EC-QCLs can be used to cover different spectral ranges.

Although the operating principles of EC-QCL-PAS and FTIR are completely different, they can be used similarly in this case. Therefore, the analysis is also done with an FTIR instrument in parallel for a comparison. EC-QCL-PAS has some advantages against the conventional FTIR spectrometers like higher spectral power density, low sample volume and no need for background measurement.

Measurement results

First, clean room air sample from the production plant was collected into a gas sample bag, and sent for analysis. Measurements were done with both EC-QCL-PAS and FTIR setups. PA201 has a 10 cm path length with a total volume of 30 ml and the EC-QCL range (981–1279 cm^{-1}) was chosen to cover the absorption lines of typical volatile organic compounds (VOCs). A Bruker Tensor 37 spectrometer with a short 10 cm gas cuvette was used in the FTIR measurements because of the limited sample amount. Nitrogen (6.0 pure) was used as a background gas in the FTIR measurement.

The second step was to determine, if there actually were impurities in the sample. The measured spectrum of the clean room air sample was compared to a clean reference air sample. The measured EC-QCL-PAS spectra are presented in Fig 2. The EC-QCL-PAS measurement shows a clear impurity in 1000–1100 cm^{-1} region when compared to the reference air sample. The sharp peaks are similar in both spectra and represent the water vapor. The FTIR measurement results are presented in Fig 3. No identification can be done based on FTIR measurements. The noise level is too high for reliable analysis. Actually, the FTIR measurement shows a higher signal in reference air sample between 1000 and 1050 cm^{-1}, which should indicate a presence of impurities in the reference air sample.

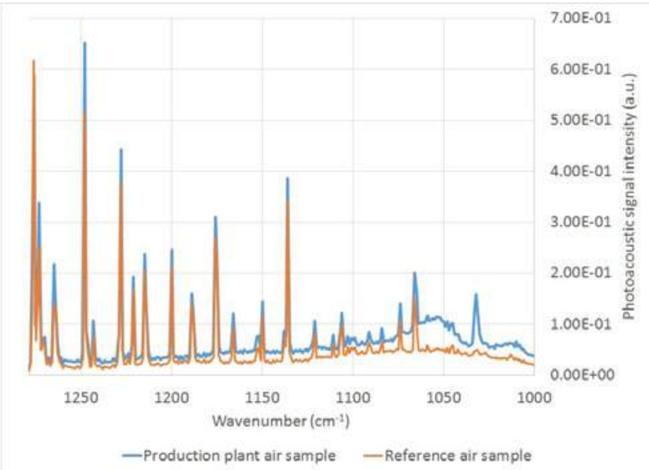

Fig 2. EC-QCL-PAS measurement of the production plant air sample and a reference air sample.

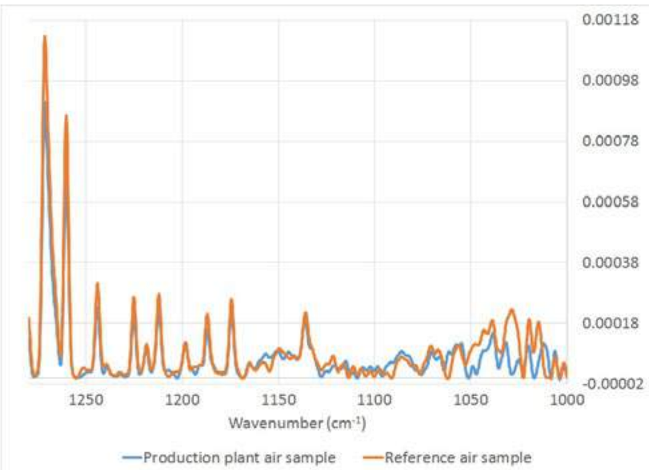

Fig 3. FTIR measurement of the production plant air sample and a reference air sample.

Third, the impurity has to be identified. PNNL spectral library was used to identify the impurity as methanol. The library spectrum of methanol was fitted into measured spectrum with a good match. The fit is demonstrated in Fig 4. The fourth and last step was to quantify the result. A known concentration of methanol was measured to calibrate the EC-QCL-PAS setup to analyze methanol from air. Based on the calibration, 3 ppm of methanol was found in the clean room air sample from the production plant. The detection limit of the EC-QCL-PAS system for methanol is 0.9 ppb for 60 s measurement time.

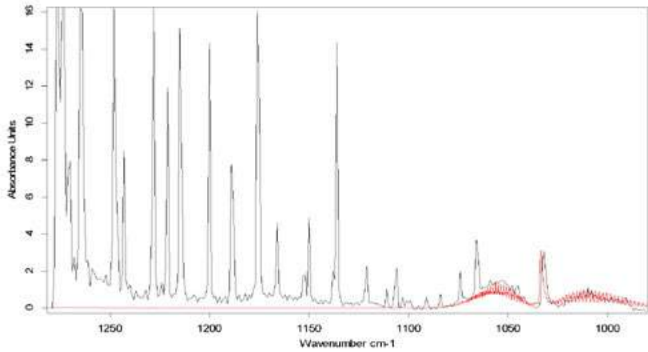

Fig 4. Library spectrum of methanol fitted to the measured EC-QCL-PAS spectrum. The variation in the optical power of the EC-QCL was corrected before the fit.

Conclusion

EC-QCL-PAS is a powerful alternative for FTIR with a long path cell. Low gas volume required for the photoacoustic detection and high sensitivity of the cantilever microphone can be combined with the high optical power and high resolution of the EC-QCLs. Broad tuning range enables multi-component analysis of various components in multiple research applications.