



Real-Time In-Situ Reaction Monitoring

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INSITU-ATR Technology with the Bruker ALPHA II FT-IR Spectrometer

Real-time monitoring of chemical reactions provides valuable insight into reaction kinetics, conversion, and endpoint determination. Conventional offline sampling methods are time-consuming and may disturb sensitive reaction systems. This application note describes an in-situ FT-IR reaction monitoring approach using the Bruker ALPHA II FT-IR spectrometer in combination with an ATR light pipe sampling interface. The setup enables continuous, non-invasive monitoring of chemical reactions directly in the reaction vessel, delivering time-resolved infrared spectra without manual sampling.

Experimental Setup

Instrumentation

The reaction monitoring system consists of the ALPHA II FT-IR spectrometer, INSITU-ATR for in-situ sampling, and OPUS / Reaction Monitoring software for automated data acquisition and evaluation.

Reaction Monitoring

Ethyl acetate, NaOH, H₂O, and isopropyl alcohol were mixed to form a homogeneous solution. The INSITU-ATR was immersed directly into the reaction mixture, and a background spectrum was recorded at the start of the reaction.

The mixture was stirred at room temperature using a magnetic stirrer. Spectra were collected automatically over approximately 3 hours using the OPUS Reaction Monitoring function. Data analysis was carried out by integrating the band at 1686 cm⁻¹, assigned to acetate, and the band at 1240 cm⁻¹, assigned to the ester.

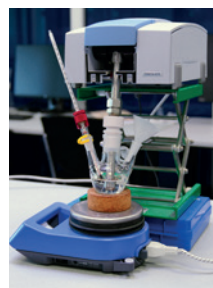
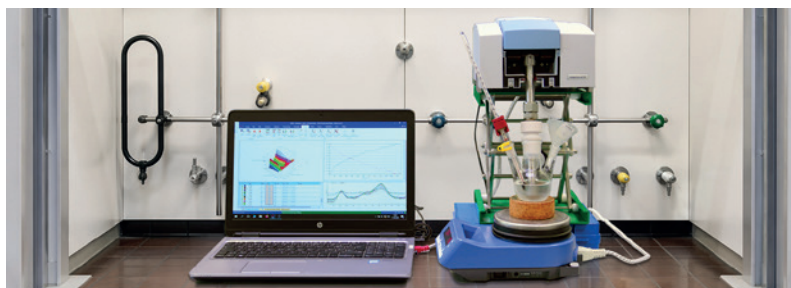


Fig. 1
ALPHA II spectrometer
with INSITU-ATR

Results and Discussion

The spectral changes are consistent with the conversion of ethyl acetate to sodium acetate. While the reaction follows second-order kinetics overall, the selected experimental conditions result in apparent pseudo-first-order behavior (Fig. 2C). This is reflected in the time-dependent evolution of the integrated band intensities. A linear relationship is obtained when plotting the natural logarithm of the ester signal versus time, whereas a plot of the reciprocal signal ($1/I$) shows a non-linear increase, confirming pseudo-first-order kinetics.

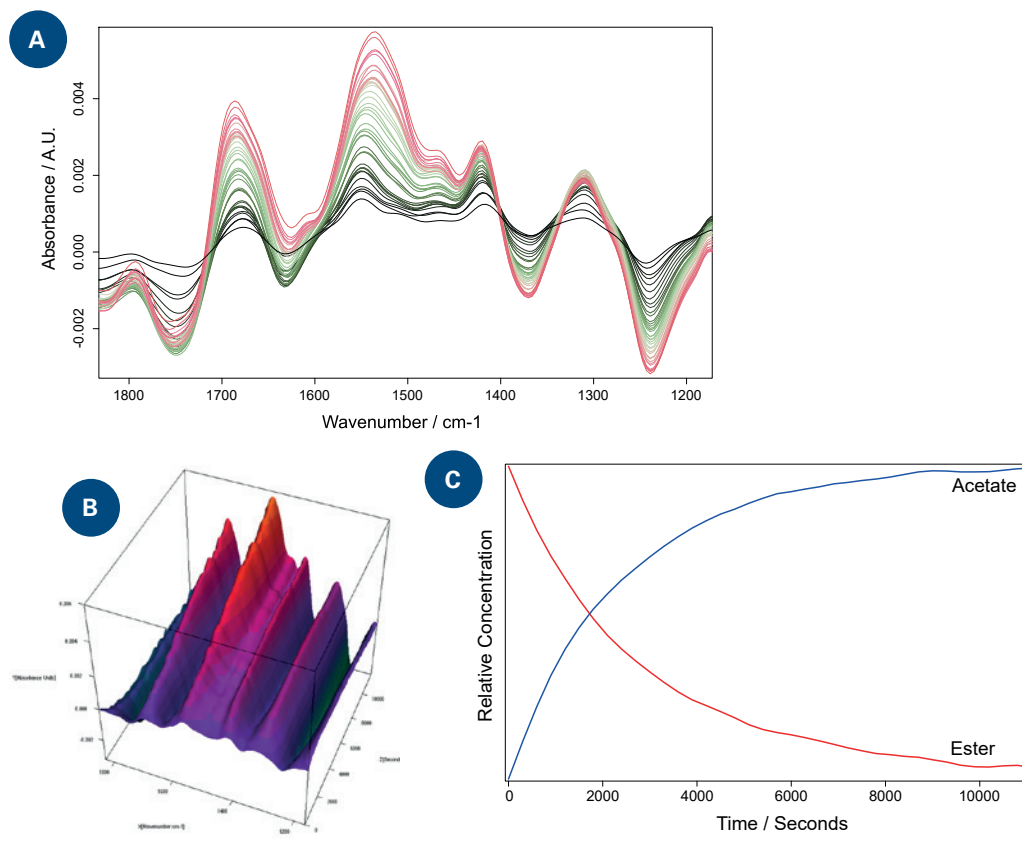


Fig. 2

A) Time-resolved spectra during saponification reaction B) 3D spectral evolution over reaction time C) Temporal evolution of ester and acetate concentrations.

Conclusion

The measurements demonstrate that Bruker's INSITU-ATR reaction monitoring module enables direct and continuous monitoring of liquid-phase reactions without sampling. Both reactant depletion and product formation can be tracked simultaneously using characteristic spectral bands. The approach provides reliable insight into reaction progress and kinetic behavior under real reaction conditions.

