

Towards an Automated Setup for Vapor-Liquid Equilibrium Measurements with Raman Spectroscopy

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Experimental vapor-liquid equilibrium (VLE) data remain indispensable for reliable process design, yet their availability is often limited [1]. The traditional approach of conducting VLE experiments is resource-intensive and experimental efforts increase exponentially for mixtures due to the combinatorial complexity [2]. Manual sample preparation and transfer are common sources of errors [3]. Recent work on liquid-liquid equilibria has demonstrated the benefits of combining automation with spectroscopic in situ measurements [4]. Spectroscopic in situ measurements have already been employed for vapor-liquid equilibria. Still, the setups were either limited to high pressure to increase signal intensity [5] or required elaborate manual procedures [6]. While our collaborators at RWTH Aachen University focus on improving the ease of use, we aim to increase the throughput by automation.

In this work, we introduce a concept for a new automated setup designed for high-throughput VLE experiments at low pressures (2-400 kPa). Miniaturizing the system aims at improving heat and mass transfer, faster equilibration, and a reduced sample volume of 2 mL. Non-invasive, in situ Raman spectroscopy is employed in a backscattering configuration developed at RWTH Aachen University [4]. This configuration enables rapid composition measurements in both phases. The setup features two connections for introducing and removing gases and liquids, paving the way for automation with syringe pumps, valves, and flowmeters. The sample preparation also allows for full automation. Multiple measurements can be conducted with one sample by alternating the composition between measurements by applying a vacuum to the cell. Broad chemical resistance ensures general applicability.

We present the first measurements for mixtures of organic molecules, showcasing the potential of composition measurements via Raman spectroscopy. The proposed concept would enable the rapid and reliable measurement of vapor-liquid equilibria in multicomponent mixtures.

References

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