An Automated Setup for Vapor-Liquid Equilibrium Measurements with Raman Spectroscopy

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An Automated Setup for Vapor-Liquid Equilibrium Measurements with Raman Spectroscopy

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1 MOTIVATION

We need reliable data for process design decisions.

VLE data from databanks?

- Combinatorial complexity for mixture data.
- Classic VLE experiments are resource-intensive.
- Uneven distribution of available data.
- → Experimental VLE data is scarce.[1]

We need data fast!

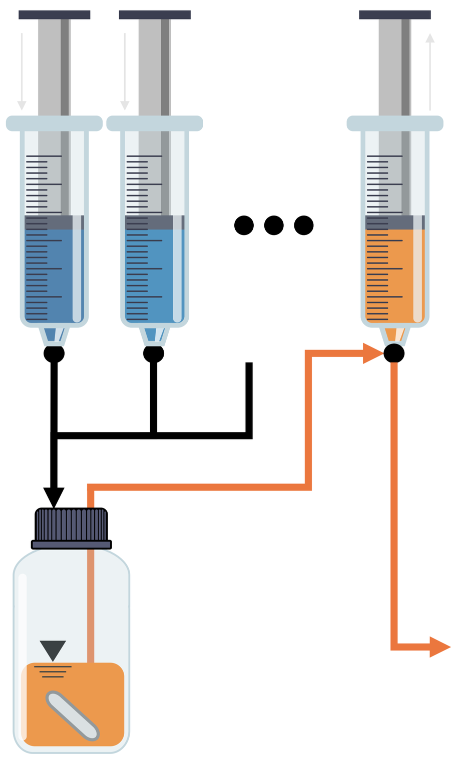
We must accelerate our experiments and ensure reliability!

Miniaturization

- $V_{sample} = 2$ ml
- Enhanced heat and mass transfer.
- Fast equilibration: 6-8 min
- L-shape shields liquid signal from vapor measurement.

Automation

- Automated sample preparation, handling and experiments.
- Avoids error-prone manual steps.
- Around-the-clock operation.
- LabVIEW controlled.
- Increased safety.



sample preparation

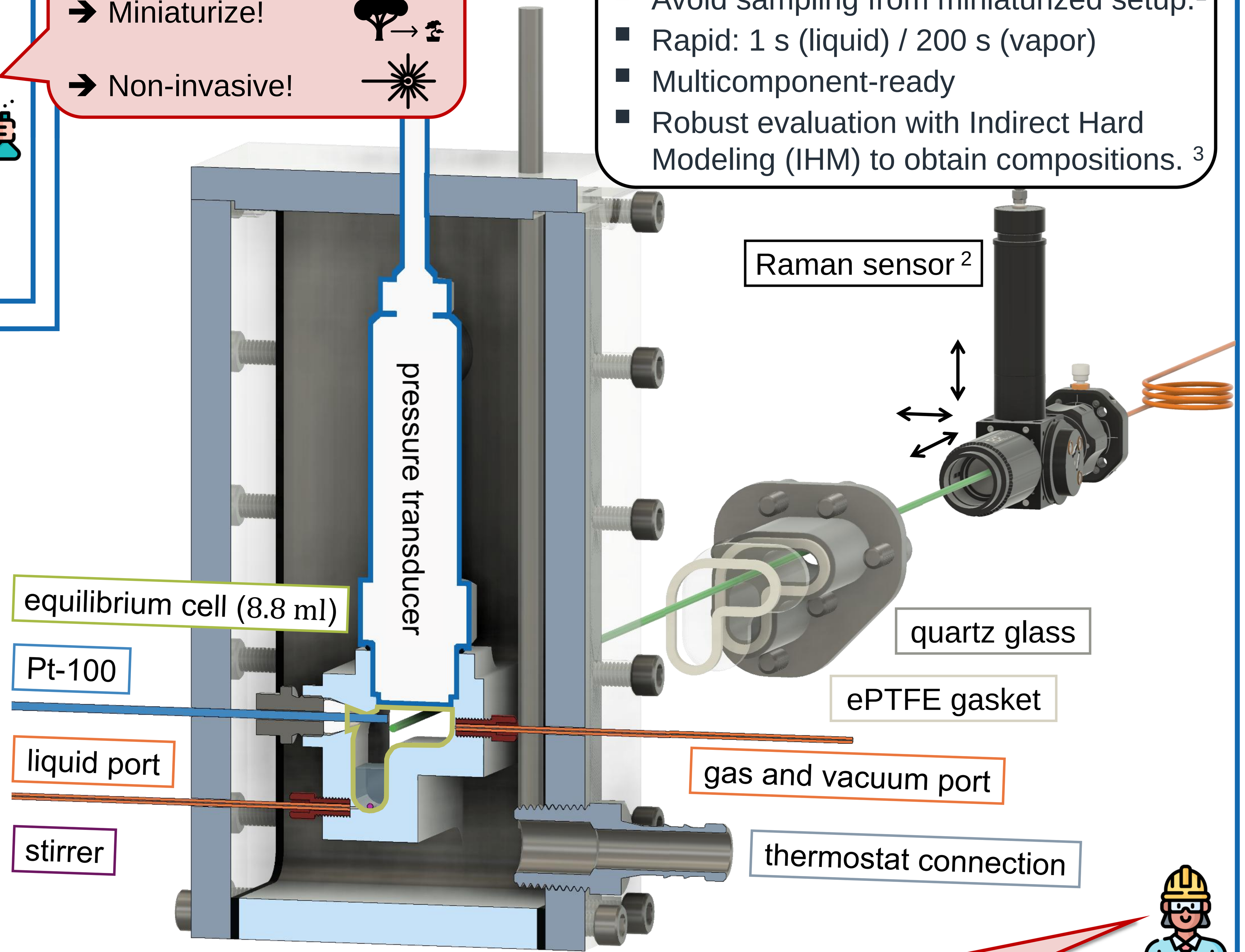
Millifluidics combined with Raman spectroscopy is a proven concept.[4,5]

2 SETUP

- → Automate!
- → Miniaturize!
- → Non-invasive!

Non-invasive measurements

- Avoid sampling from miniaturized setup.[2]
- Rapid: 1 s (liquid) / 200 s (vapor)
- Multicomponent-ready
- Robust evaluation with Indirect Hard Modeling (IHM) to obtain compositions.[3]



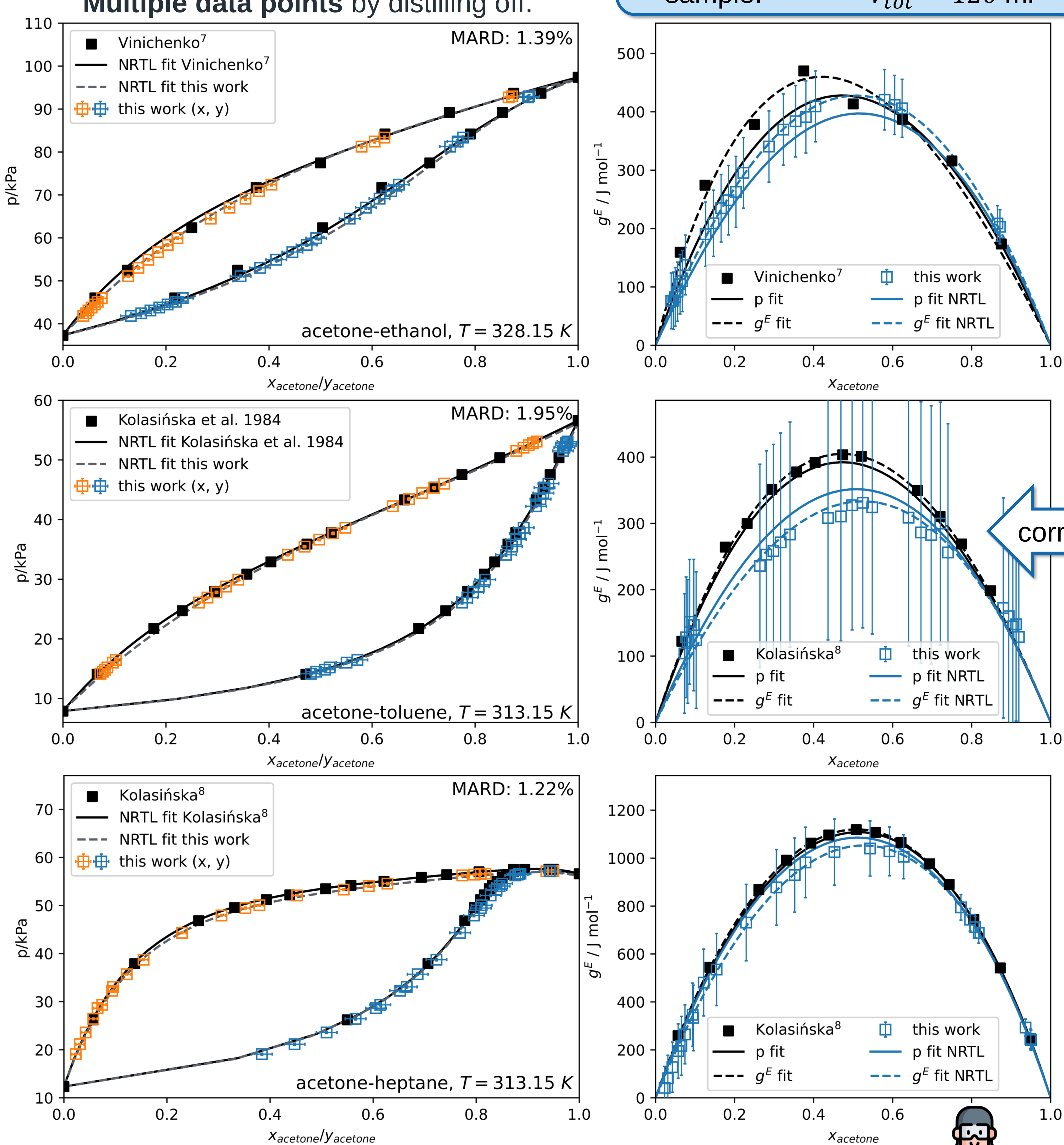
Fast data! But Reliable?

4 RESULTS – BINARY VLE AND CONSISTENCY TESTS

For each mixture:

- total duration t < 24 h
- sample: $V_{tot} = 120$ ml

Multiple data points by distilling off.



Consistent within measurement uncertainty!

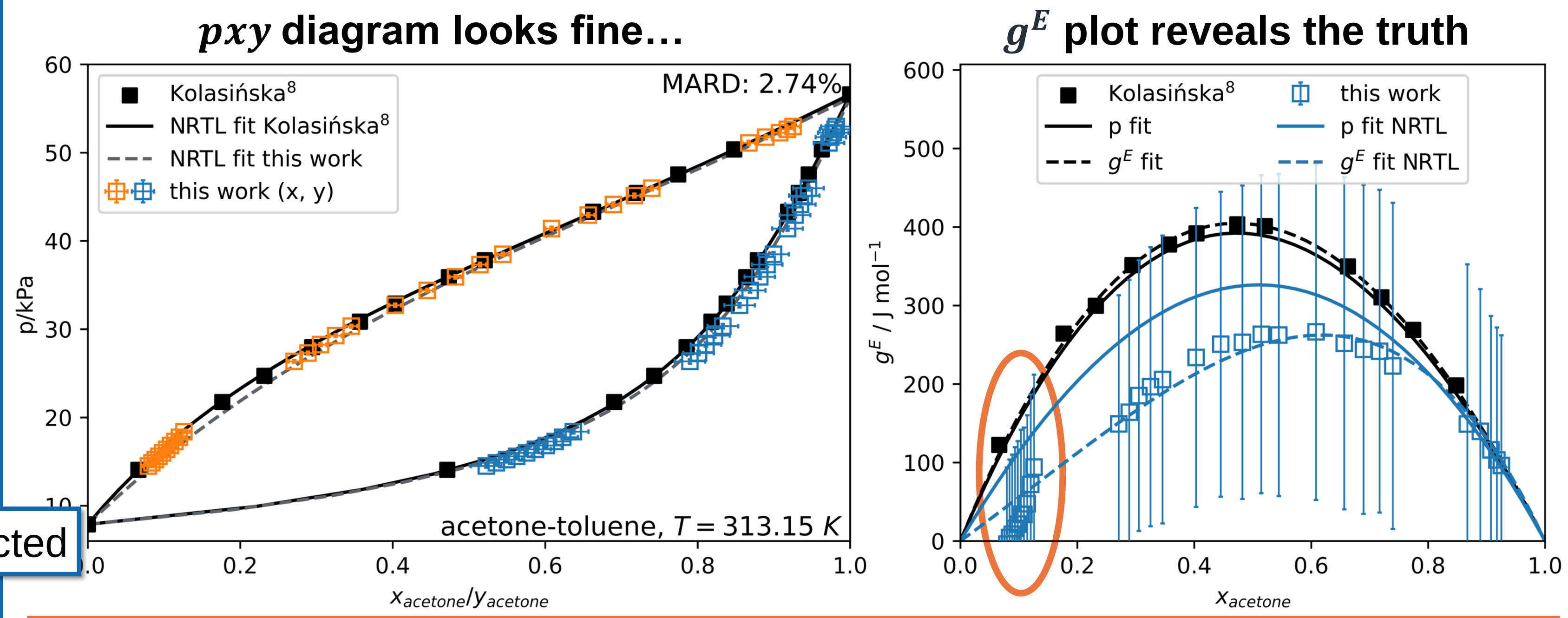
3 PRELIMINARY RESULTS AND CONSISTENCY TESTS

We can check consistency, but literature thresholds seem arbitrary.

Consider measurement uncertainty to derive consistency thresholds with error propagation!

- Smooth behaviour in g^E plot expected for consistent data.[6]
- Fit for different objectives (p or g^E) ideally identical.

pxy **diagram looks fine…** g^E **plot reveals the truth**



Discovery of erroneous calibration from consistency test!

5 CONCLUSIONS

- Full binary VLE in less than 24 h.
- Automation eliminates common sources of error and increases throughput.
- Consistency tests ensure reliability.
- Next step: multicomponent mixtures.

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