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A New Static Analytic Apparatus for Complete Determination of Phase Equilibrium Data from Sub-atmospheric Pressures up to 10 bar

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Introduction

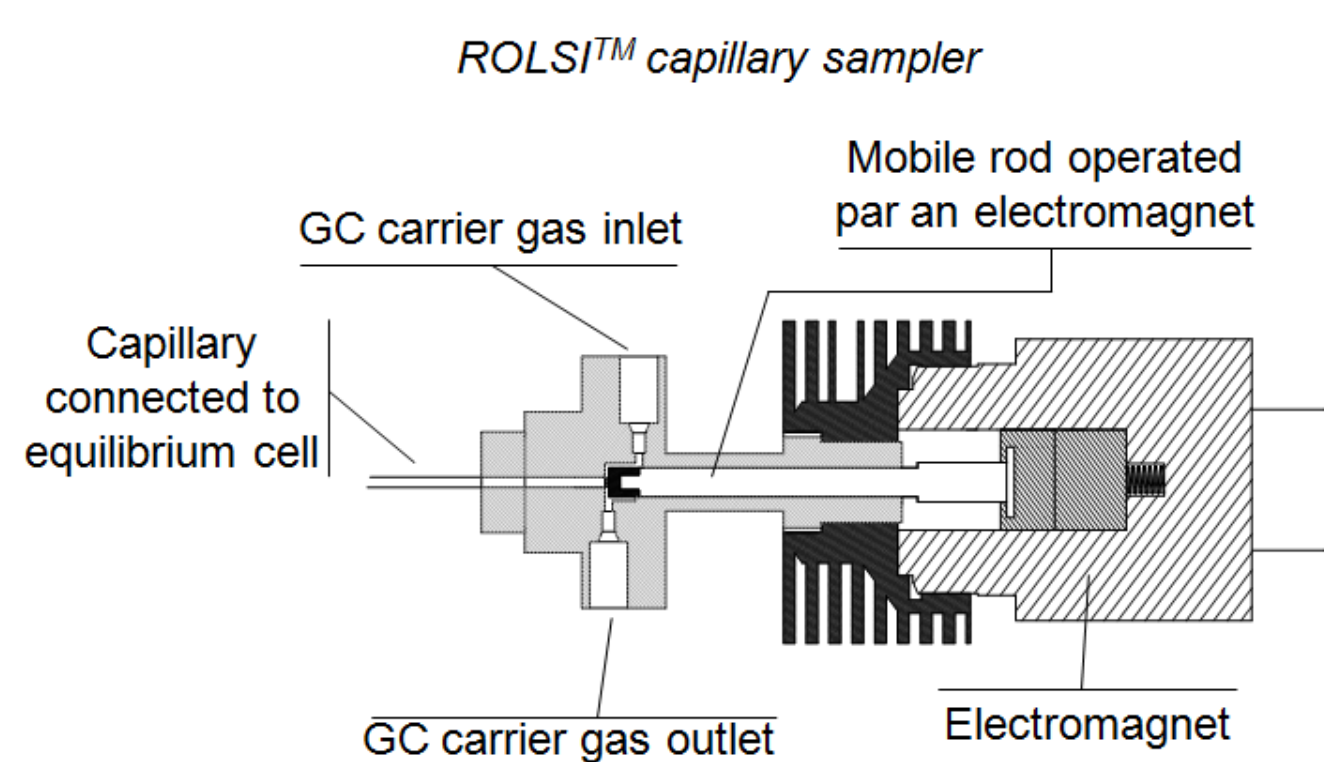
Reliable Vapor-Liquid-Equilibrium (VLE) data are of great importance in chemical engineering. Experimental data combined with appropriate thermodynamic models are essential for the design and optimization of separation processes. In most industrial applications, operating conditions vary from low (< 1 bar) to moderate (1 – 10 bar) pressures. For this pressure range, existing experimental methods show some drawbacks (see Table 1).

Here, we present a new apparatus based on static analytic method with improved sampling mechanisms.

Table 1. Common experimental methods in low and moderate pressure range

Method	Data type	Advantage	Drawback
Static-synthetic (Total pressure)	PT_x	•No sampling •Rapid measurement	•Thermodynamic models required for data reduction • PT_x (y calculated)
Circulation still	PT_{xy}	•Both phases sampled	•Large volume •Condensation required
Variable volume cell	PT_{xy}	•No sampling	•Discontinuous operation

Apparatus Description



ROLSI™ capillary sampler [1]:

- In-situ sampling
- Small sample volume (several μL per sample)
- On-line analysis by gas chromatograph (GC)

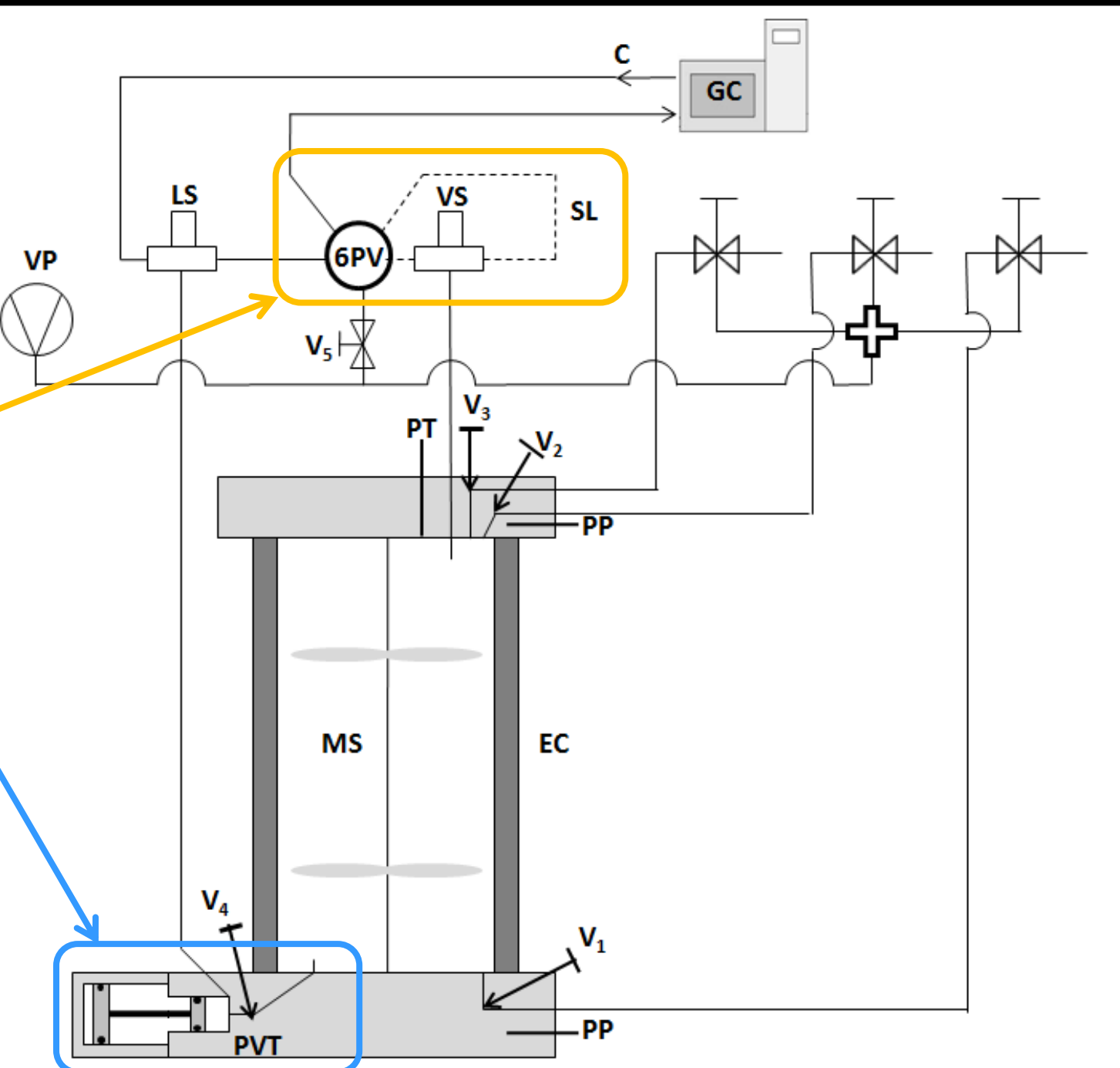
But...driving force needed: $P_{\text{cell}} > P_{\text{carrier gas}}$ (≈ 3 bar)

Improvements

- For vapor phase: a 6-port sample injection valve implanted in the transfer line
- For liquid phase: a small variable volume chamber (PVT) equipped with a piston for compression (Patent application is ongoing [2])

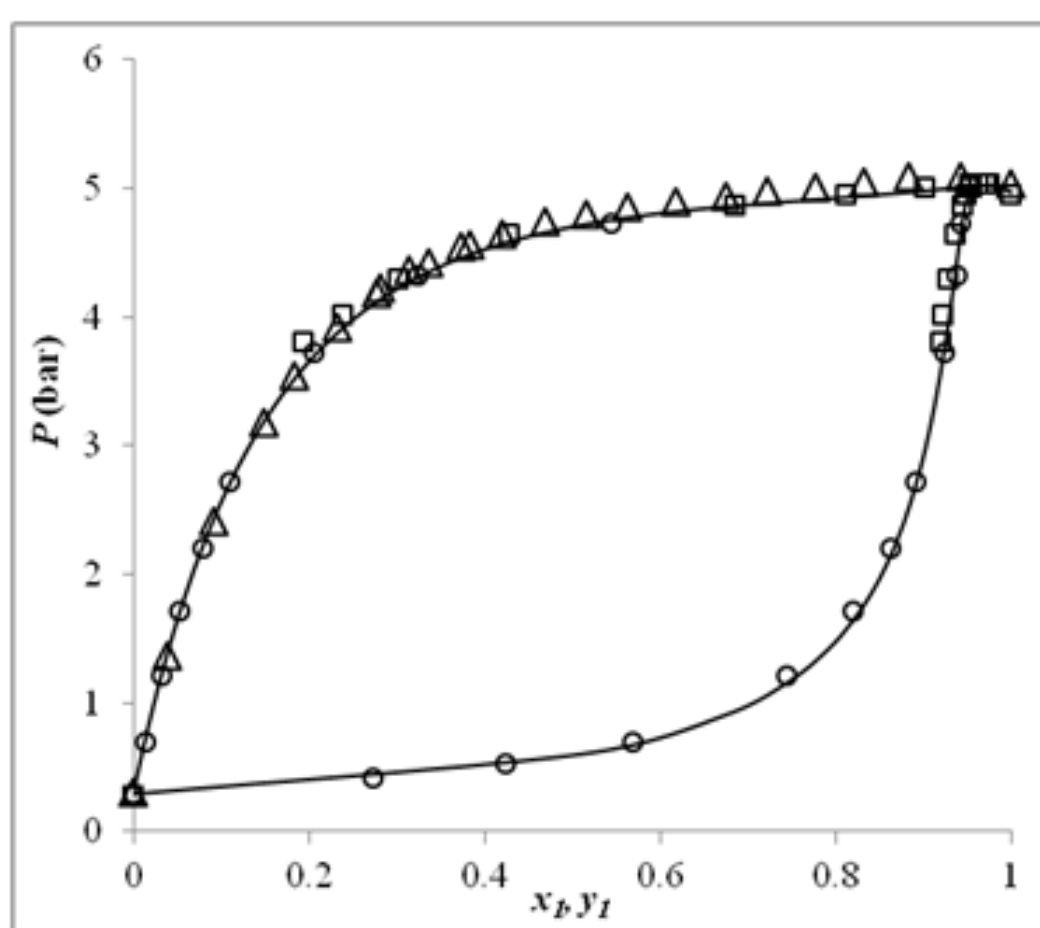
Main advantages:

- ✓ Sampling ability extended to $P < P_{\text{carrier gas}}$
- ✓ Complete VLE data measurement
- ✓ Continuous operation
- ✓ Less chemical consumption
- ✓ Ability of dealing with viscous fluids

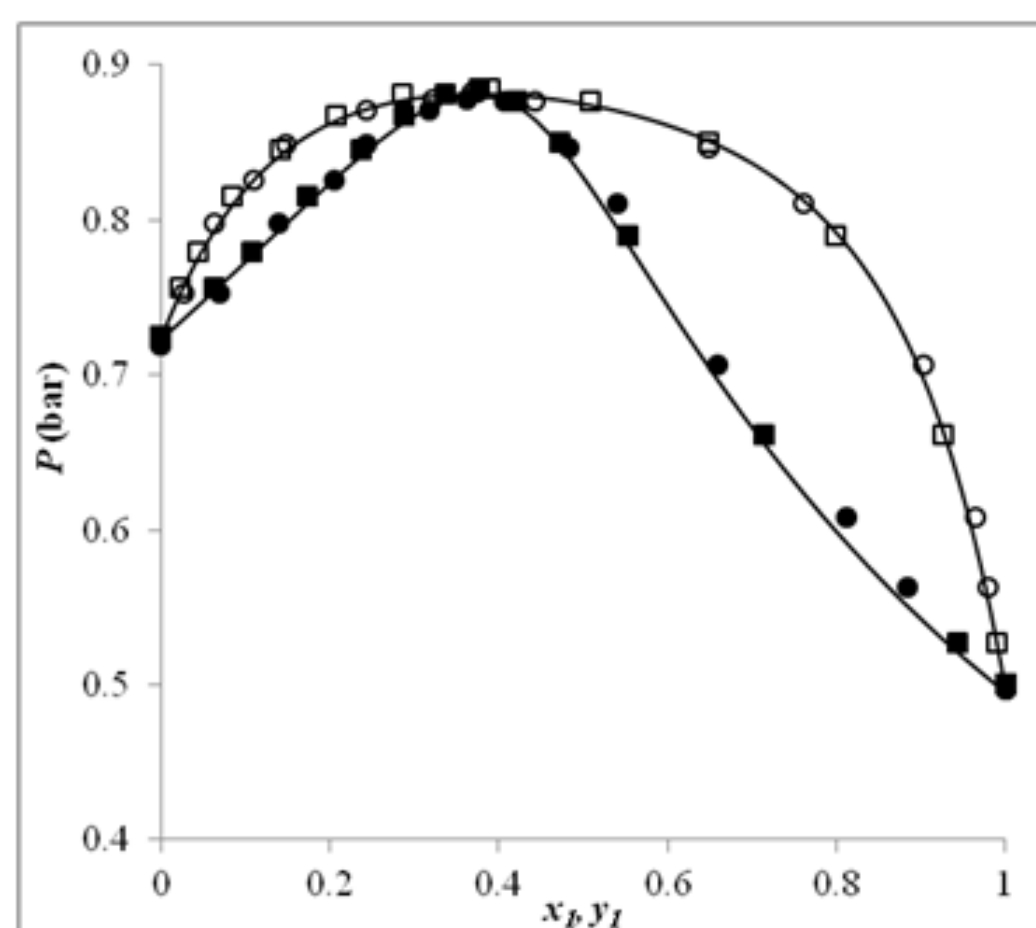


6PV: 6-port sample injection valve; C: carrier gas; EC: equilibrium cell; GC: gas chromatograph; LS: liquid ROLSI™ sampler; MS: magnetic stirrer; PP: platinum resistance thermometer probe; PT: pressure transducer; PVT: PVT chamber; SL: Sample Loop; V_i : valve; VP: vacuum pump; VS: vapor ROLSI™ sampler.

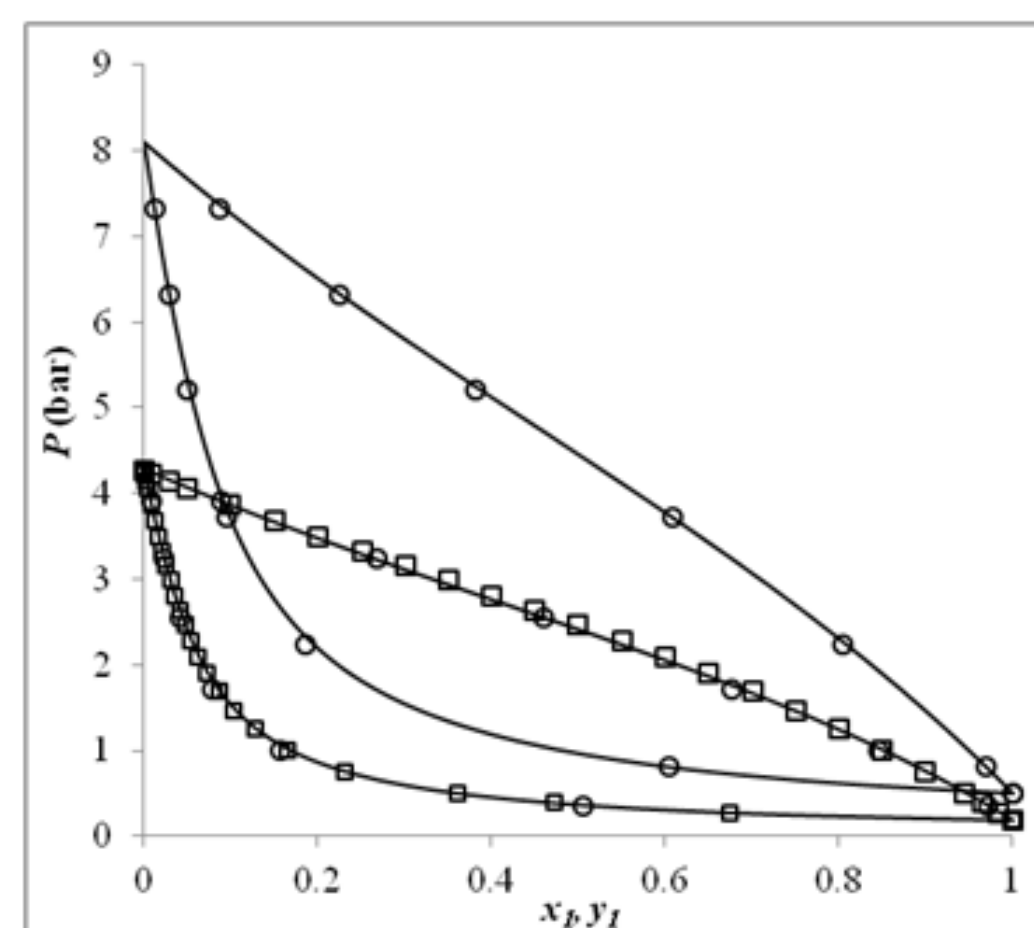
Experimental Results*



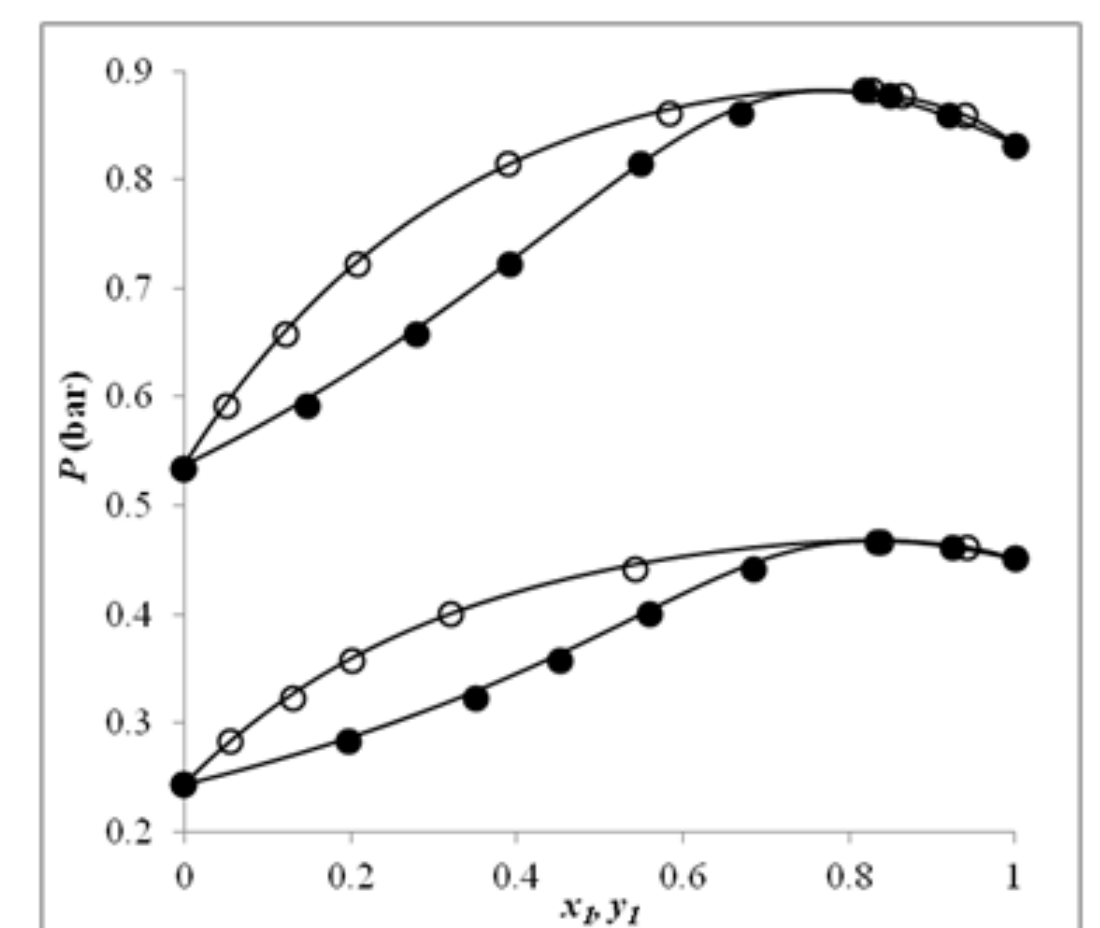
n-Butane (1) + Ethanol (2). (○) this work at 323.22 K; (△) static-synthetic method at 323.75 K [3]; (□) static-analytic method without the improvements at 323.25 K [4].



Diethyl sulfide (1) + Ethanol (2). (○, ●) this work at 343.13 K; (□) circulation still at 343.15 K [5].



Diethyl sulfide (1) + *n*-Butane (2). (○) this work at 317.62 K and 343.14 K; (□) static-synthetic method at 343.15 K [6] (y calculated by SRK EoS [7]).



1-Pentanethiol (1) + 1-Pentanol (2). (○, ●) this work at 372.75 and 392.72 K.

*Experimental were correlated through Wilson model [8] with SRK EoS [7]

Conclusion & Perspective

- A new static-analytic apparatus equipped two ROLSI™ capillary samplers was presented.
- The proposed improvements allow ROLSI™ to perform in-situ sampling even at $P < P_{\text{carrier gas}}$.
- The apparatus has shown comparable performances to existing methods, while showing some advantages.
- Two binary systems of interest were investigated.
- ❖ New systems are expected to be investigated for various applications in chemical engineering.
- ❖ The liquid sampling mechanism is expected to be used for liquid-liquid equilibrium measurement.

Reference

- [1]: Armines, French Patent No. 2853414, 2003
- [2]: Armines. French Patent Application No. 1460309, 2014
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- [4]: C.-B. Soo et al., Fluid Phase Equilib., 286 (2009) 79
- [5]: E. Sapei et al., Fluid Phase Equilib., 301 (2011) 200
- [6]: C. Dell'Era et al. Fluid Phase Equilib. 291 (2010) 180
- [7]: G. Soave, Chem. Eng. Sci. 27 (1972) 1197
- [8]: G.M. Wilson, J. Am. Chem. Soc. 86 (1964) 127