Karlsruhe Institute of Technology

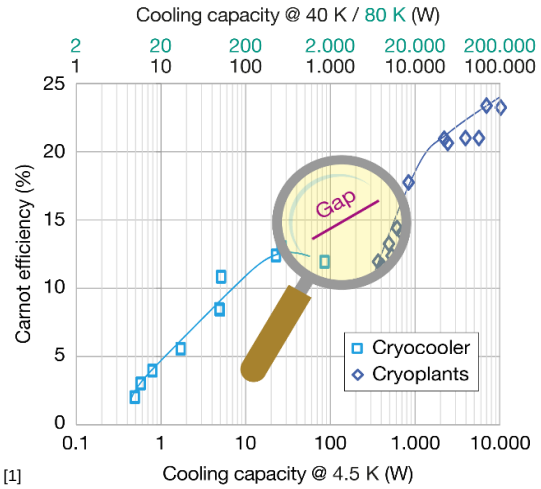
Referenzmessungen am kryogenen Phasengleichgewichtsprüfstand CryoPHAEQTS

Jens Tamson, Steffen Grohmann

DKV-Tagung 2022, Magdeburg

Motivation

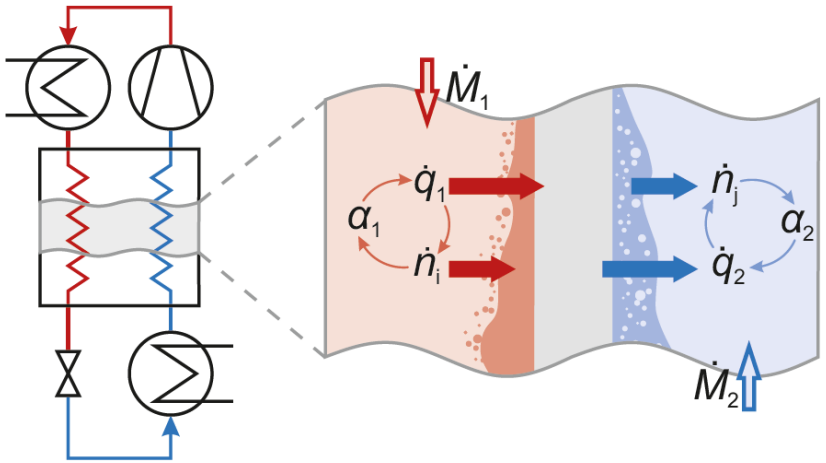
Est. low-temperature refrigerators



Source: [1]

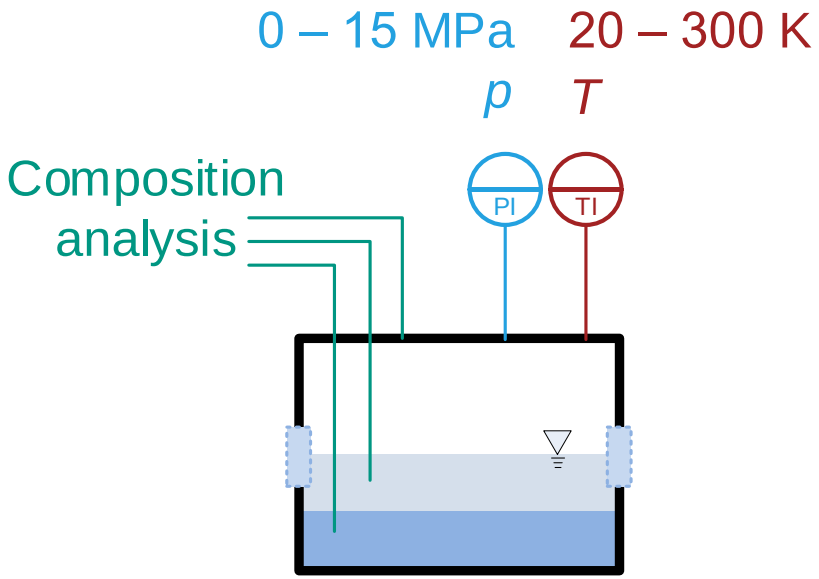
Technology gap to reach cryogenic temperatures at medium cooling capacities

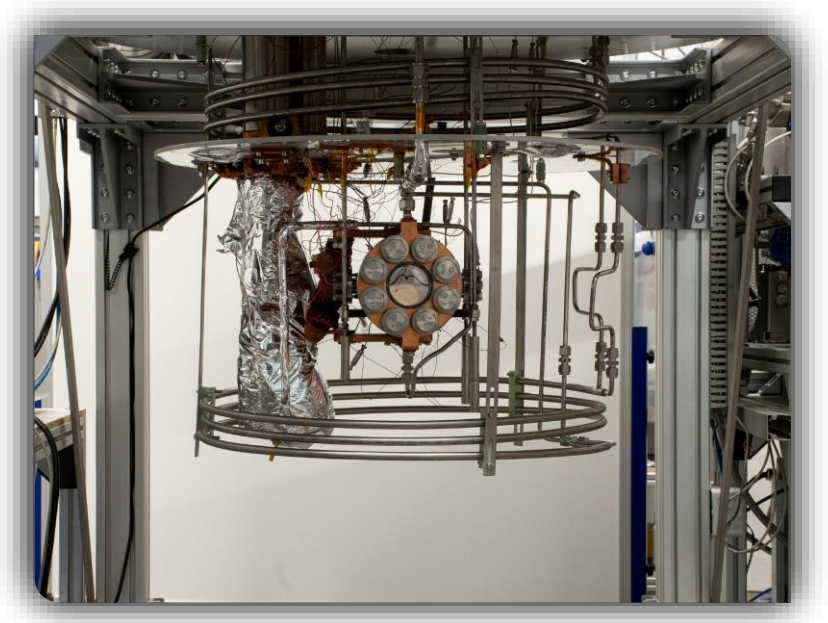
Cryogenic mixed refrigerant cycles



Physical property measurements are scarce at cryogenic conditions

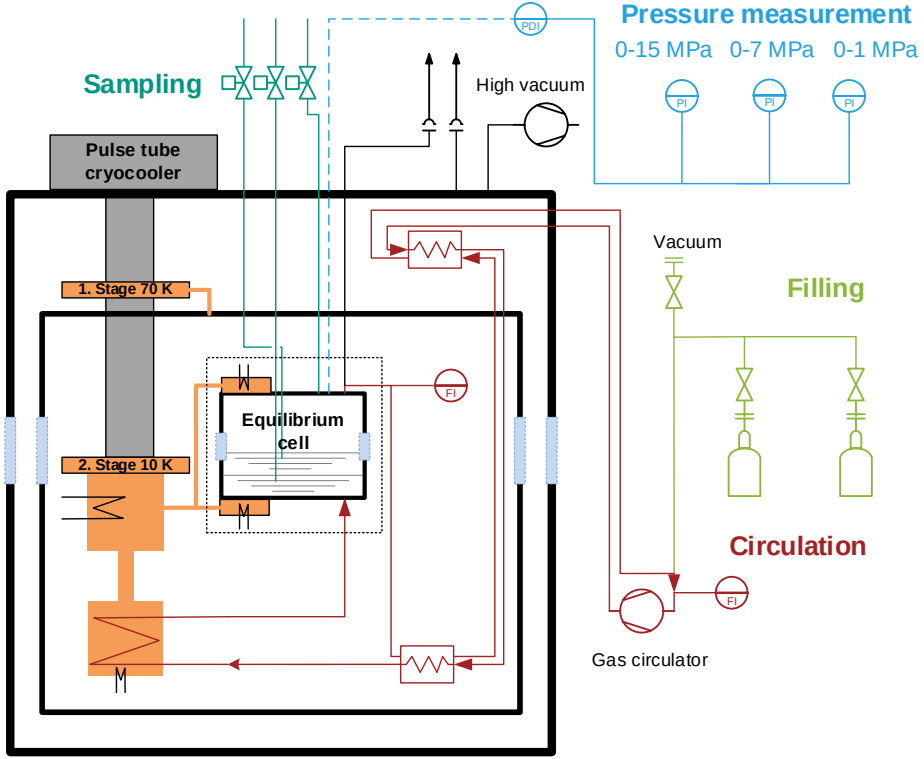
Cryogenic Phase Equilibria Test Stand CryoPHAEQTS

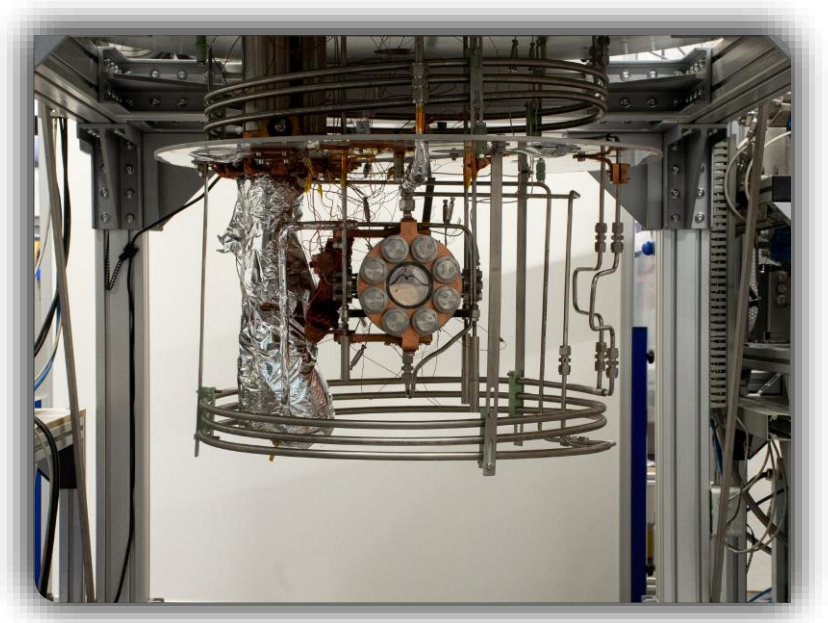




Institute for Technical Thermodynamics and Refrigeration
Refrigeration and Cryogenics

Cryogenic Phase Equilibria Test Stand CryoPHAEQTS





Institute for Technical Thermodynamics and Refrigeration
Refrigeration and Cryogenics

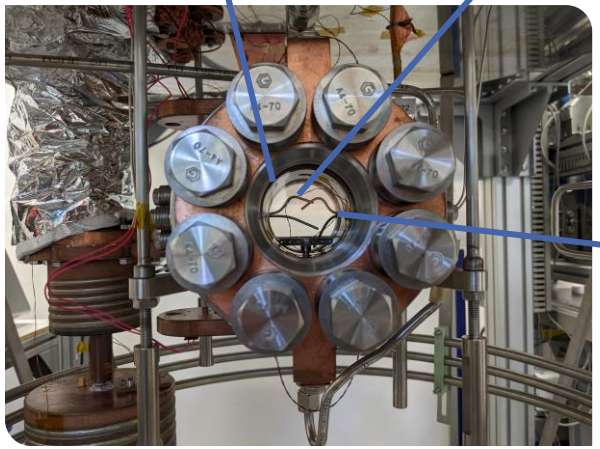
Measurement uncertainties w.r.t. GUM [2]

Temperature sensors

$u_{\mathrm{abs}}(T) \leq 20\ \mathrm{mK}$

Pressure sensor capillary

$u_{\mathrm{rel}}(p) \leq 0.1\%$ (above 1 bar)



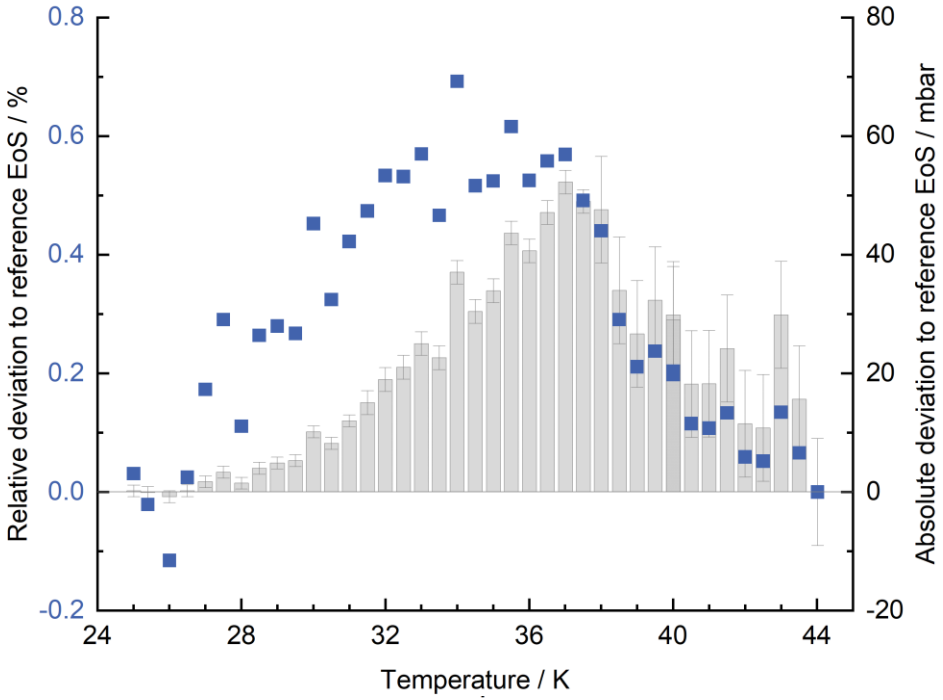
Sampling capillaries

$u_{\mathrm{rel}}(z_i) \leq 1\%$

Gas chromatography

- *T*-range: -30°C to 300°C
- Molecular sieve column & GS Gaspro column
- 18-point gravimetric calibration

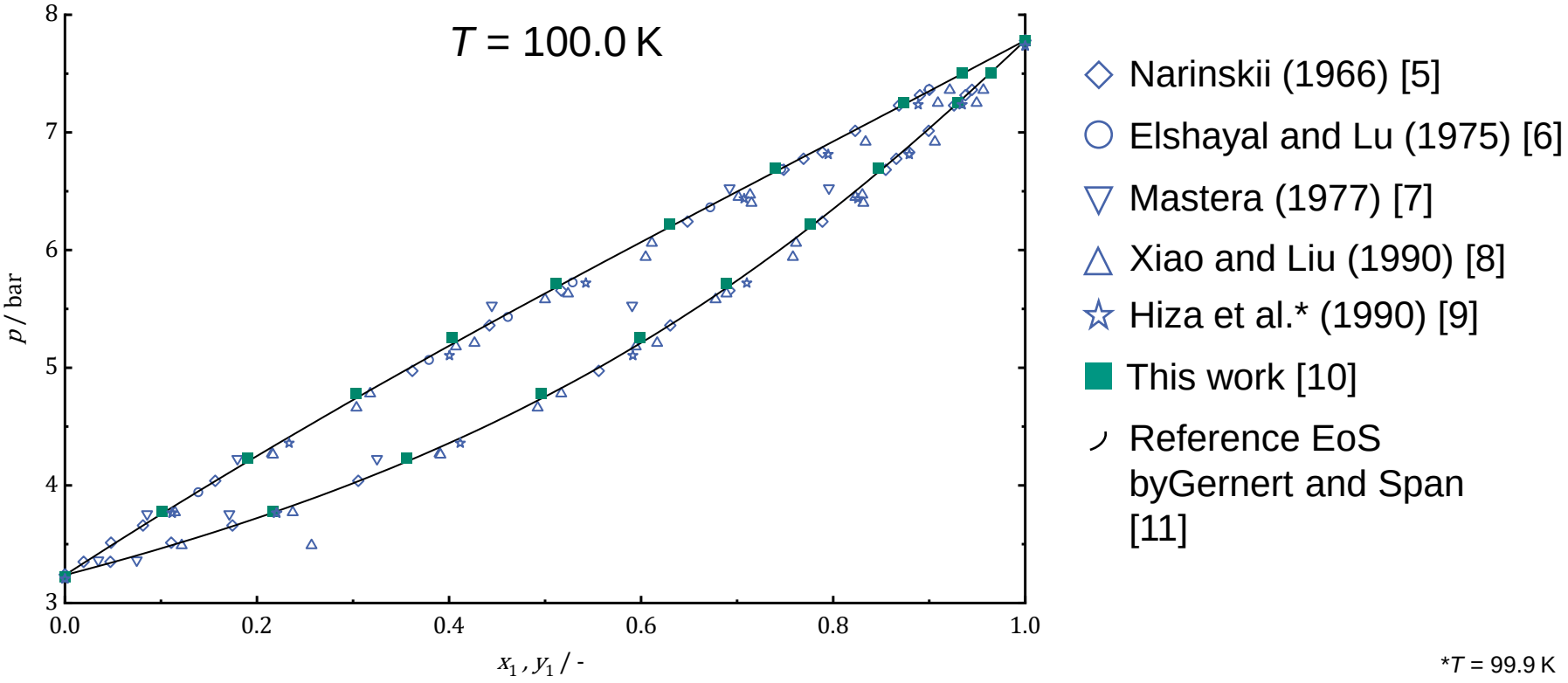
Results for vapor pressure of pure neon



- Measurements from 25 K to 44 K [15]
  - $u(T) = 13\ \mathrm{mK} \ldots 19\ \mathrm{mK}$
  - $u(p) = 1\ \mathrm{mbar} \ldots 8\ \mathrm{mbar}$
- Reference EoS: Thol et al. [3] accessed through REFPROP 10.0 [4]
- Good agreement close to triple point and critical point
- Lower curvature than reference EoS

Institute for Technical Thermodynamics and Refrigeration
Refrigeration and Cryogenics

Validation results of argon-nitrogen VLE

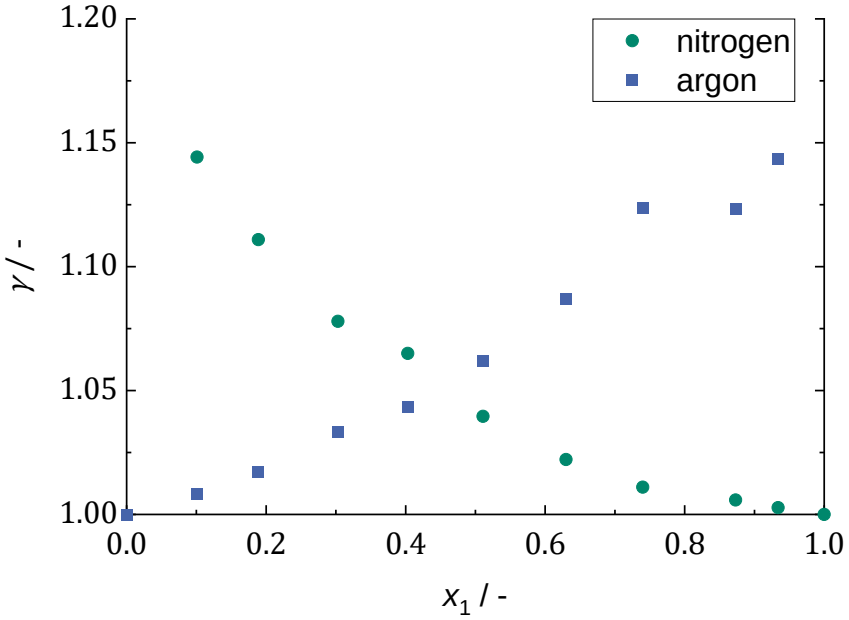


*T = 99.9 K

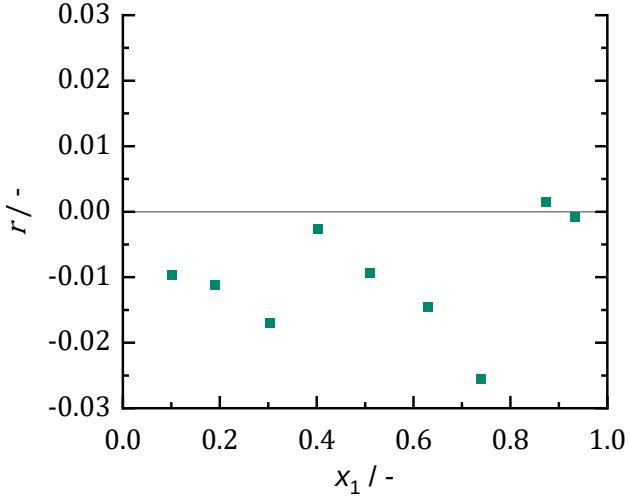
Institute for Technical Thermodynamics and Refrigeration
Refrigeration and Cryogenics

Thermodynamic consistency

- ‘Experimental‘ activity coefficents



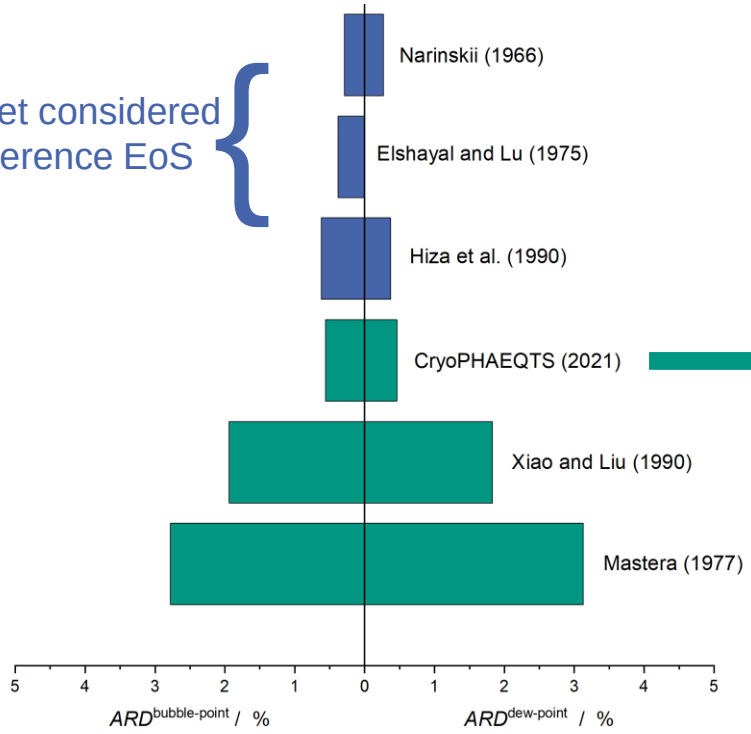
- Check for thermodynamic consistency
  - Redlich-Kister test [12]: fail
  - Fredenslund test [13]: pass
  - Van Ness test [14]: fail

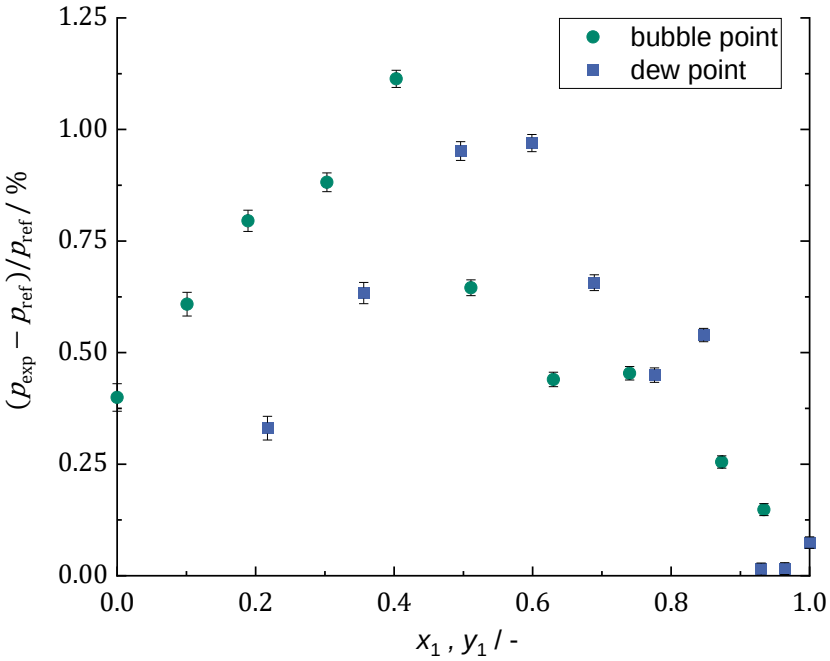


$$r = \ln\left(\frac{\gamma_{k,1,\mathrm{exp}}}{\gamma_{k,2,\mathrm{exp}}}\right) - \ln\left(\frac{\gamma_{k,1,\mathrm{calc}}}{\gamma_{k,2,\mathrm{calc}}}\right)$$

Comparison with literature and reference EoS

Data set considered in reference EoS



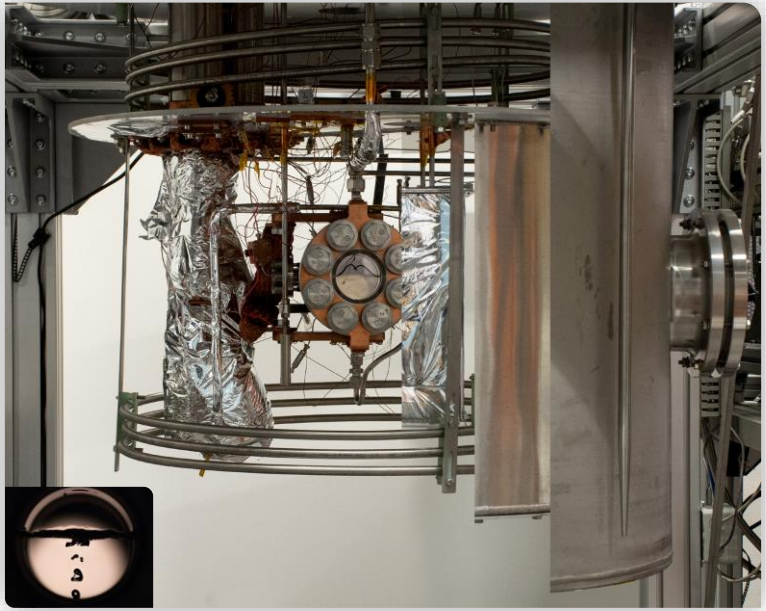


Institute for Technical Thermodynamics and Refrigeration
Refrigeration and Cryogenics

Conclusion & Outlook

- Successful validation of CryoPHAEQTS with the Ar-N_2-VLE
- Remaining inaccuracies due to GC-analysis requiring daily calibration

- Currently measuring He-Ne-VLE
- Test stand upgrade with a gas phase c_p-sensor

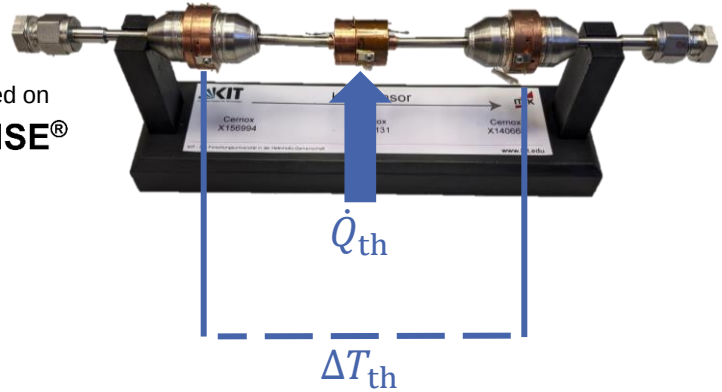


Institute for Technical Thermodynamics and Refrigeration
Refrigeration and Cryogenics

Outlook: Gas phase c_p sensor

Combination of cryogenic thermal mass flow sensor and Coriolis flow meter at ambient temperature

Design based on
WEKASENSE®



Source: Bronkhorst

$$c_p = \frac{\dot{Q}_{\mathrm{th}}}{\dot{M}_{\mathrm{Cor}} \Delta T_{\mathrm{th}}}$$

$$u_{\mathrm{aim}}(c_p) \leq 1\%$$

- First results unsatisfying due to axial heat conduction
- Re-design with additional piping length

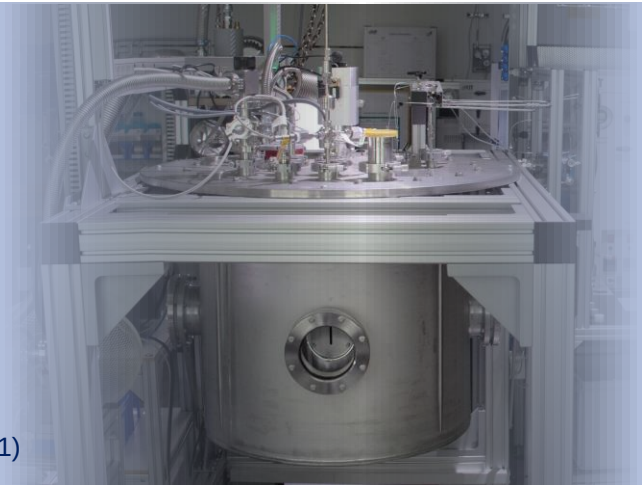
Please ask your questions!

Acknowledgements

Karlsruhe School of Elementary Particle and Astroparticle Physics: Science and Technology (KSETA)

Deutsche Forschungsgemeinschaft

German Research Foundation (DFG):
Simultaneous description of caloric and thermal properties of cryogenic fluid mixtures (Gr 4168/2-1)



References

[1] Decker, L.: Overview on cryogenic refrigeration cycles for large scale HTS applications. International Workshop on Cooling System for HTS Applications (IWC-HTS), October 14-16, 2015, Matsue, Japan
[2] Guide to the expression of uncertainty in measurement. International Organization for Standardization, 1995, ISBN 92-67-10188-9 .
[3] M. Thol, R. Beckmüller, R. Weiss, A. H. Harvey, E. W. Lemmon, R. T. Jacobsen, and R. Span, "Thermodynamic properties for neon for temperatures from the triple point to 700 K at pressures to 700 MPa," J. Phys. Chem. Ref. Data (unpublished)
[4] Lemmon E W, Bell I H, Huber M L and McLinden M O 2018 NIST Standard Reference Database 23: Reference Fluid Thermodynamic and Transport Properties - REFPROP Version 10.0 National Institute of Standards and Technology
[5] Narinskii G B 1966 Russian Journal of Physical Chemistry 40
[6] Elshayal I M and Lu B C Y 1975 The Canadian Journal of ChemicalEngineering 5383–87 ISSN 00084034
[7] Mastera S G J 1977 Dampf-Flüssig-Gleichgewichtsdaten der Systeme Ar-N2, Kr-Ar, Kr-N2 und Xe-Kr sowie Löslichkeitsgrenzen des festen Xenons und des festen Kryptons in flüssigen Luftkomponenten Ph.D. thesis RWTH Aachen Jülich
[8] Xiao J and Liu K 1990 J. Chem. Eng.(China) 88–12
[9] Hiza M J, Kidnay A J and Haynes W M 1990 Liquid-vapor equilibria in binary systems containing nitrogen, oxygen, and argon between 63 and 100K: Unpublished results (*private communication with E. W. Lemmon*)
[10] J Tamson et al 2022 IOP Conf. Ser.: Mater. Sci. Eng. 1240 012159
[11] J. Gernert and R. Span, J. Chem. Thermodyn., 93:274-293, 2016. DOI:10.1016/j.jct.2015.05.015
[12] Redlich O and Kister A T 1948Industrial & Engineering Chemistry40345–348
[13] Fredenslund A 1977Vapor-Liquid Equilibria Using Unifac: A Group-Contribution Method2nd ed (Elsevier Science) ISBN 0-444-41621-8
[14] van Ness H C 1995Pure and Applied Chemistry67859–87
[15] Tamson J, Grohmann S, Measurement of vapor pressure of neon from 25 K to 44 K, Fluid Phase Equilibria, 565, 2023, 113621