TRACEABLE LNG DENSITY MEASUREMENT AND CALCULATION TRAINING
R. Span, M. Richter, N. von Preetzmann, P. Eckmann, M. Thol
„Metrology for LNG“, May 2020
Thermodynamics at Ruhr University Bochum

accurate measurement of thermophysical properties

accurate modeling of thermophysical properties

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Span et al. | LNG Density Measurement and Calculation | 05/2020
Thermodynamics at Ruhr University Bochum

\[
\alpha(\delta, \tau) = \alpha^0(\delta, \tau) + \alpha^r(\delta, \tau) = \frac{f^0(T, v)}{RT} + \frac{f^r(T, v)}{RT}
\]

\[
\frac{h}{RT} = 1 + \tau \left( \alpha^0_\tau + \alpha^r_\tau \right) + \delta \alpha^r_\delta
\]

with \( \delta = \frac{\nu_{\text{red.}}}{\nu} = \frac{\rho}{\rho_{\text{red.}}} \) and \( \tau = \frac{T_{\text{red.}}}{T} \)

\[
c_p = \frac{-\tau^2 \left( \alpha^0_\tau + \alpha^r_\tau \right) + (1 + \delta \alpha^r_\delta - \delta \tau \alpha^r_\delta)^2}{1 + 2 \delta \alpha^r_\delta + \delta^2 \alpha^r_\delta}
\]

\[
z = \frac{pv}{RT} = 1 + \delta \alpha^r_\delta
\]

Accurate modeling

\[
\alpha^o(\tau, \delta) = c_1 + c_2 \tau + n_0 \ln(\tau) - \sum_{i=1}^{l_{\text{POL}}} \frac{n_i}{t_i(t_i - 1)} \tau^{t_i} - \sum_{k=1}^{K_{\text{PE}}} m_k \ln(1 - e^{-\delta_k}) + \ln \delta
\]

\[
\alpha^r(\delta, \tau) = \sum_{i=1}^{N_{\text{POL}}} n_i \delta_i \tau^i + \sum_{i=N_{\text{POL}}+1}^{N_{\text{POL}}+N_{\text{EXP}}} n_i \delta^i \tau^i \exp(-\delta^k) + \text{special terms}
\]

\[
\alpha(\delta, \tau, x) = \sum_{i=1}^{N} x_i \left[ \alpha^0_{oi}(\rho, T) + \ln x_i \right] + \sum_{i=1}^{N} x_i \alpha^r_{oi}(\delta_m, \tau_m) + \sum_{i=1}^{N-1} \sum_{j=i+1}^{N} x_i x_j F_{ij} \alpha^r_{ij}(\delta_m, \tau_m)
\]
LNG Custody Transfer

General Formula for Calculating the LNG Energy Transferred
(According to GIIGNL - LNG Custody Transfer Handbook)

\[
E = V_{\text{LNG}} \cdot \rho_{\text{LNG}}(T, p, x) \cdot GCV_{\text{S,LNG}} - E_{\text{g, displaced}} \ [\text{MMBTU}]
\]

- \( E \): the total net energy transferred
- \( V_{\text{LNG}} \): the volume of LNG loaded or unloaded in m\(^3\).
- \( \rho_{\text{LNG}} \): the density of LNG loaded or unloaded in kg/m\(^3\).
- \( GCV_{\text{S,LNG}} \): the gross calorific value of the LNG in MMBTU/kg.
- \( E_{\text{g, displaced}} \): the net energy of the displaced gas in MMBTU
The Starting Point – Options for LNG-Density Calculations

Revised Klosek-McKinley
- 90 K - 115 K, $p \approx 0.1$ MPa
- $\Delta \rho / \rho = 0.1\%$
- fitted to sat. liquid densities

GERG-2008
- 90 K - 450 K, $p \leq 70$ MPa
- $\Delta \rho / \rho = 0.1 - 0.5\%$
- fitted to all kinds of data

COSTALD
- 90 K - 180 K, $p \leq 15$ MPa (CH$_4$)
- $\Delta \rho / \rho = ???$  AAD $\Delta \rho / \rho = 0.4\%$
- fitted to density data

Density is an important property!
The Starting Point – An Extremely Weak Experimental Basis

-0.4 0 0.4

100 \( \frac{\rho' - \rho'_{\text{EOS}}}{\rho'_{\text{EOS}}} \)

Temperature / K

100 105 110 115 120 125 130 135

-0.4 0 0.4

Revised Klosek-McKinley

very weak data basis

limit of validity

Ger 


Hiza & Hatnes (1980)

The Starting Point – An Extremely Weak Experimental Basis

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### The Starting Point – An Extremely Weak Experimental Basis

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<tr>
<th>Authors</th>
<th>Technique</th>
<th>$t$ - range</th>
<th>$p$ - range</th>
<th>Dr/r [%]</th>
<th>Cryogenic liquid mixtures</th>
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<tbody>
<tr>
<td>Klosek and McKinley (1968)</td>
<td>Pycnometer</td>
<td>-150°C to -180°C</td>
<td>$p = p_a$</td>
<td>0.5%</td>
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<td>Air Products, USA</td>
<td>Magnetically suspended sinker</td>
<td>-200°C to 50°C</td>
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<td>Kleinrahm and Wagner (1984)</td>
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<td>-40°C to 250°C</td>
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**Note:** Until today, mixtures were measured only in the **gas phase**.

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- Klosek and McKinley (1968), Air Products, USA
- Haynes and Frederick (1983), NIST
- Kleinrahm and Wagner (1984), RUB
- Brachthäuser, Kleinrahm, Lösch and Wagner (1993), RUB

**Technique:**
- Pycnometer
- Magnetically suspended sinker
- Two sinker hydrostatic balance with MSC
- Single sinker hydrostatic balance with MSC

**Temperature Range:**
- $t$ - range: -150°C to -180°C, -200°C to 50°C, -210°C to 70°C, -40°C to 250°C

**Pressure Range:**
- $p$ - range: $p = p_a$, $p \leq 350$ bar, $p \leq 120$ bar, $p \leq 300$ bar

**Dr/r (%)**:
- 0.5%
- 0.1% (plus uncert. of composition)
- 0.02% to 0.01%
- 0.02%

**Cryogenic Liquid Mixtures**:
- VLE-Measurement ⇒ demixing of the liquid ⇒ only **saturated** liquid densities
- Until today: mixtures measured only in **gas phase**
The Starting Point – An Extremely Weak Experimental Basis

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<td>VLE-Measurement</td>
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<td>Until today: mixtures measured only in gas phase</td>
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No state of the art measurements for LNG!

LNG Pipeline

The Starting Point – An Extremely Weak Experimental Basis

Pressure measurement (ambient temperature)

Measuring cell (cryogenic temperature)

Cryogenic liquid mixtures

VLE-Measurement

⇒ demixing of the liquid
⇒ only saturated liquid densities

VLE-Measurement

⇒ demixing of the liquid
⇒ only saturated liquid densities

Until today: mixtures measured only in gas phase

Until today: mixtures measured only in gas phase

Span et al. | LNG Density Measurement and Calculation | 05/2020
The Primary Densimeter for “Metrology for LNG”

Technical specifications:
- $T$-range: (90 to 300) K
- $p$-range: (0.05 to 12) MPa
- Hom. densities (liquid and gas) & saturated liquid density
- $U(\rho(T,p,x)) = \sim 0.02 \% \ (k = 2)$

Concept for densimeter from 10 years ago!
The Primary Densimeter for “Metrology for LNG”

Technical specifications:

- $T$-range: (90 to 300) K
- $p$-range: (0.05 to 12) MPa
- Hom. densities (liquid and gas) & saturated liquid density
- $U(\rho(T,p,x)) = \sim 0.02 \% (k = 2)$

Metrological traceability:

- $T$: ITS90 (SPRT calibr. at PTB)
- $p$: piston gauge (DAkkS calibr.)
- $V_S$: hydrostatic weighing at PTB
- $m_S$: mass comparison at PTB
- $x$: gravimetrical preparation
The Primary Densimeter for “Metrology for LNG”

Supercritical liquefaction:
- Filling at ambient $T$
- Isobaric cooling
- Waiting for equilibrium
- First density measurement

Pressure control with “VLE-cell”:
- Allows VLE in a defined place
- Reducing $T \rightarrow$ Reducing $p$
- No venting necessary
Evolution of the Primary Densimeter for “Metrology for LNG”

Reduction of „force transmission error“ to reduce uncertainty
Evolution of the Primary Densimeter for “Metrology for LNG”

Exchange of connection tube between measuring cell and VLE-CEL

Before:

After:

\[ p_{\text{cell}} = 6.164 \text{ MPa} \]

During the exchange:
- Fr 10:25 a.m.
- Th 4:30 p.m.
- Th 12:20 p.m.
- Tu 9:50 a.m.
- Fr 12:30 p.m.
- We 9:55 a.m.
- Fr 6:40 a.m.
- Th 8:30 p.m.
- Tu 6:00 p.m.
Evolution of the Primary Densimeter for “Metrology for LNG”

Density measurements:

- 6 synthetic LNGs (e.g. from Norway and Oman)
- 6 binary Mixtures (CH$_4$ + C$_2$H$_6$/C$_3$H$_8$/C$_4$H$_{10}$/C$_5$H$_{12}$)
- 3 binary Mixtures (CH$_4$ + N$_2$)
- 2 synthetic LBGs (CH$_4$ + N$_2$ + O$_2$ + H$_2$ + CO$_2$)
- Synthetic air
- 8 synthetic LNG-like mixtures (ongoing project)

A typical measurement schedule:

- 4 Isotherms: (100 to 160) K with pressures ≤ 10 MPa
- 7 measurement points along each isotherm
- 2 isotherms per week (since last development stage)
First density measurements on LNGs

- 6 synthetic LNG mixtures representing natural gas coming from different origins
  - e.g. Norway, Oman, Libya, …
  - $x_{\text{CH}_4}$ from (81.6 to 98.0) mole-%
  - 5 to 8 components
  - Hydrocarbons from methane to pentanes

- Densities compared to:
  - Revised Klosek and McKinley method
  - And GERG-2008 fundamental EOS
First density measurements on LNGs

Normalized composition of the 6 synthetic LNG mixtures in mole-%

<table>
<thead>
<tr>
<th>Component</th>
<th>LNG Norway</th>
<th>LNG Libya</th>
<th>LNG Oman</th>
<th>LNG 2</th>
<th>LNG 5</th>
<th>LNG 7</th>
</tr>
</thead>
<tbody>
<tr>
<td>CH₄</td>
<td>91.798</td>
<td>81.563</td>
<td>87.885</td>
<td>84.636</td>
<td>87.972</td>
<td>97.890</td>
</tr>
<tr>
<td>C₂H₆</td>
<td>5.698</td>
<td>13.375</td>
<td>7.274</td>
<td>12.800</td>
<td>7.240</td>
<td>0.999</td>
</tr>
<tr>
<td>C₃H₈</td>
<td>1.303</td>
<td>3.679</td>
<td>2.926</td>
<td>1.499</td>
<td>2.900</td>
<td>0.487</td>
</tr>
<tr>
<td>n-C₄H₁₀</td>
<td>0.396</td>
<td>0.688</td>
<td>1.565</td>
<td>0.210</td>
<td>0.692</td>
<td>0.209</td>
</tr>
<tr>
<td>i-C₄H₁₀</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>n-C₅H₁₂</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>0.030</td>
<td>0.100</td>
<td>0.016</td>
</tr>
<tr>
<td>i-C₅H₁₂</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>0.020</td>
<td>0.110</td>
<td>0.018</td>
</tr>
<tr>
<td>N₂</td>
<td>0.805</td>
<td>0.695</td>
<td>0.350</td>
<td>0.585</td>
<td>0.343</td>
<td>0.194</td>
</tr>
</tbody>
</table>
Results for “LNG Norway”

- Measurements from (105 to 135) K in the homogenous liquid region
- Saturated liquid densities determined via extrapolation to saturation pressure
- **Revised Klosek and McKinley method**
  - Predicts the saturated liquid densities within the exp. uncertainty of $U(\rho) = 0.044 \% \ (k = 2)$
  - "Accuracy" of the model of 0.1 % for < 115 K
- **GERG-2008 fundamental EOS**
  - Uncertainty $\Delta \rho'/\rho' = (0.1 \text{ to } 0.5) \%$
  - Prediction not within exp. uncertainty

---

Relative Deviations for LNG Norway

![Graph showing relative deviations between GERG and experimental results.](Image)
Results for “LNG Norway”

- **RKM** has no pressure dependent term
  - Calculating saturated liquid densities only
  - Deviations to exp. densities increase at higher pressures in the liquid region
  - Offset to measurements of up to 2.2 %
- **GERG-2008** predicting saturated and homogenous liquid densities
  - Consistent over the entire pressure range
  - No significant temperature dependency
  - Overall within the uncertainty of the model
Results for other LNGs

- Same observations for the RKM when comparing to density measurements on other LNGs
- e.g. “LNG Libya” with only 81.6 mole-% methane

Lessons learned:

- GERG-2008 predicting densities within the uncertainty of the model of (0.1 to 0.5) %, but Not within the experimental uncertainty
- RKM only for saturated liquid densities Especially relevant for small-scale applications at elevated pressures
Revised Klosek and McKinley method RKM

- ISO Standard (ISO 6578)
- Well-established for LNG custody transfer
- Range of validity: \( T < 115 \text{ K} \)
- Uncertainty: \( \Delta \rho '/\rho ' = 0.10 \% \)

\[
\frac{M_{\text{mix}}}{\rho_{\text{mix}}} = \nu_{\text{mix}}(T) = \sum_i x_i \cdot v_i - \left[ k_1 + (k_2 - k_1) \cdot \left( \frac{x_{N_2}}{0.0425} \right) \right] \cdot x_{\text{CH}_4}
\]

- Pressure dependency in the liquid region not taken into account

- Development of a new engineering model based on the RKM
- To be fitted to LNG density measurements performed on the primary Densimeter for “Metrology for LNG”
Enhanced Revised Klosek and McKinley (ERKM) method

- Expanded range of validity: $100 \text{ K} \leq T \leq 135 \text{ K}$
  $p \leq 10 \text{ MPa}$

- Uncertainties:
  - for $100 \leq T / \text{K} \leq 115$, $\Delta \rho / \rho = 0.10 \%$
  - for $115 < T / \text{K} \leq 135$, $\Delta \rho / \rho = 0.15 \%$

\[ \rho_{\text{LNG}} = \rho_{\text{RKM}} \cdot (1 + k_p) \]

\[ k_p = (p - p_{s, \text{mix}}) \cdot 4.06 \cdot \left( \frac{T_{\text{PC}}}{T_{\text{PC}} - T} \right)^{1.79} \]

The original RKM

Pressure-dependent term

Approximation for the mixture's vapor pressure

Pseudo-critical temperature to avoid deviations at higher temperatures

Enhanced Revised Klosek and McKinley (ERKM) method

- Performance of the ERKM
  - Representing all LNG densities within 0.15 %
  - Across the entire temperature and pressure range
- Adopted to the GIIGNL Custody Transfer handbook
  - Density calculation for small-scale applications

Results for “LNG Norway” versus “LNG Oman” and “LNG 5”

**GERG-2008** predicts densities for mixtures without or with small amounts of butane and pentane better!

- Consistent over the **entire pressure range**
- No significant temperature dependency
Planning the next measurements together with EOS developers

Available database:

Multicomponent mixtures:
- 3 mixtures including methane, ethane, propane, butane, nitrogen
- 3 mixtures including methane, ethane, propane, butane, isobutane, pentane, isopentane, nitrogen

Binary mixtures:
- ✓ Methane + ethane \((x_{C1} = 0.75)\)
- ☐ Methane + propane \((x_{C1} = 0.88)\)
- ☐ Methane + isobutane \((x_{C1} = 0.97)\)
- ☐ Methane + pentane \((x_{C1} = 0.99)\)
- ☐ Methane + nitrogen \((x_{C1} = 0.70, x_{C1} = 0.97, x_{C1} = 0.99)\)

Identify binary mixtures, which have to be measured to improve a fundamental equation of state.
Density measurements of methane-rich binary mixtures

Methane + Propane
\((x_C1 = 0.88)\)

Methane + i-Butane
\((x_C1 = 0.97)\)
Density measurements of methane-rich binary mixtures

Methane + n-Pentane
\((x_{C1} = 0.99)\)

Methane + N2
\((x_{C1} = 0.70)\)
Development of EOS-LNG – Binary Mixtures

No adjustment needed

Deviations calculated with:

- GERG-2008
- eRKM
Development of EOS-LNG - Binary Mixtures

Deviations calculated with:
- GERG-2008
- eRKM
- EOS-LNG

Adjusted binary-specific functions
Development of EOS-LNG – Binary Mixtures

If available, other properties such as
- Phase equilibria (e.g., VLE)
- Heat capacity
- Speed of sound
were also considered.

Example:
VLE for the system
methane + \textit{n}-pentane \((C_1C_5)\)
Development of EOS-LNG – Multicomponent Mixtures

Adjustment of binary-specific functions for:

- $C_1C_4$
- $C_1C_4i$
- $C_1C_5$
- $C_1C_5i$

Deviations calculated with:

- GERG-2008
- eRKM
- EOS-LNG
Fundamental Equation of State (EOS-LNG) – Helmholtz Energy

\[
\frac{\alpha(T, \rho, \bar{x})}{RT} = \alpha(\tau, \delta, \bar{x}) = \alpha^o(T, \rho, \bar{x}) + \alpha^r(\tau, \delta, \bar{x})
\]

\[
\alpha^o(T, \rho, \bar{x}) = \sum_{i=1}^{N} x_i [\alpha^o_{o,i}(\tau_{o,i}, \delta_{o,i}) + \ln x_i]
\]

\[
T_r(\bar{x}) = \sum_{i=1}^{N} x_i^2 T_{c,i} + \sum_{i=1}^{N-1} \sum_{j=i+1}^{N} 2x_i x_j \beta T_{ij} Y_{T,ij} \frac{x_i + x_j}{\beta^2_{T,ij} x_i + x_j} \sqrt{T_{c,i} T_{c,j}}
\]

\[
\frac{1}{v_r(\bar{x})} = \frac{1}{\rho_r(\bar{x})} = \sum_{i=1}^{N} x_i^2 \frac{1}{\rho_{c,i}} + \sum_{i=1}^{N-1} \sum_{j=i+1}^{N} 2x_i x_j \beta v_{ij} Y_{v,ij} \frac{x_i + x_j}{\beta^2_{v,ij} x_i + x_j} \frac{1}{8} \left( \frac{1}{\rho_{c,i}^{1/3}} + \frac{1}{\rho_{c,j}^{1/3}} \right)^3
\]

\[
\alpha^r(\tau, \delta, \bar{x}) = \sum_{i=1}^{N} x_i \alpha^r_{o,i}(\tau, \delta) + \Delta \alpha^r(\tau, \delta, \bar{x})
\]

Pure fluid contribution at corresponding states

Departure term

Arbitrary number of terms and term types
Fundamental Equation of State (EOS-LNG)

Exemplary calculation of thermodynamic properties:

Pressure: \[ p(T, \rho, \bar{x}) = 1 + \delta \alpha^r_{\delta} \]

Enthalpy: \[ h(T, \rho, \bar{x}) = 1 + \tau (\alpha^o_t + \alpha^r_t) + \delta \alpha^r_{\delta} \]

Speed of sound: \[ \frac{w^2(T, \rho, \bar{x})M}{RT} = 1 + 2\delta \alpha^r_{\delta} + \delta^2 \alpha^r_{\delta\delta} - \frac{(1 + \delta \alpha^r_{\delta} - \delta \tau \alpha^r_{\delta\tau})^2}{\tau^2(\alpha^o_{tt} + \alpha^r_{tt})} \]

Derivatives:

\[ \alpha_{\delta} = \left( \frac{\partial \alpha(T, \rho, \bar{x})}{\partial \delta} \right)_\tau \]
\[ \alpha_{\tau} = \left( \frac{\partial \alpha(T, \rho, \bar{x})}{\partial \tau} \right)_\delta \]
\[ \alpha_{\delta\tau} = \frac{\partial^2 \alpha(T, \rho, \bar{x})}{\partial \delta \partial \tau} \]

Natural variables for most properties and applications are not \( T, \rho \) but, e.g., \( T, p \) or \( p, h \) → iterative flash calculations required

The Software tool TREND is developed to enable easy and fast calculations of thermo-physical properties with the most accurate equations of state in research and industry.
Outlook

- Additional properties have to be measured:
  - thermodynamic properties: speed of sound, vapor-liquid equilibrium, and heat capacities
  - transport properties: viscosity

- EOS-LNG needs to be refined:
  - new measurements of the binary mixtures $C_1C_4$ and $C_1C_{5i}$ (LNG III) available
Outlook - Binary Mixtures (LNGIII)

Deviations calculated with:

- GERG-2008
- eRKM
- EOS-LNG

Binary-specific functions must be refined.
Outlook

- Additional properties have to be measured:
  - thermodynamic properties: speed of sound, vapor-liquid equilibrium, and heat capacities
  - transport properties: viscosity

- EOS-LNG needs to be refined:
  - new measurements of the binary mixtures $C_1C_4$ and $C_1C_{5i}$ (LNG III) available
  - further measurements on a ternary mixture $C_1C_4C_{5i}$ launching soon
  - include additional components such as CO$_2$ or H$_2$

- LLE and SLE need to be considered:
  - new measurements of liquid-liquid equilibria (LLE) of $C_1$ with higher alkanes required
  - LLE predicted by EOS-LNG need to be validated, if necessary refit of EOS-LNG
  - Models for solid hydrocarbons ($C_{4+}$) need to be developed and implemented into the EOS-LNG framework to describe solid-liquid equilibria (SLE), freezout of higher components

- Fitting techniques have to be modified to allow for:
  - the application of measurements of multicomponent systems to the fit
  - the development of a generalized functional form including higher hydrocarbons
Outlook

- The (liquefied) natural-gas community needs to prepare itself for the rise of hydrogen

HYSTRA – CO$_2$-free Hydrogen Energy Supply-Chain