Purity of Fluids – LAr Division
Modeling Electron Lifetimes in LAr TPCs

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In collaboration with
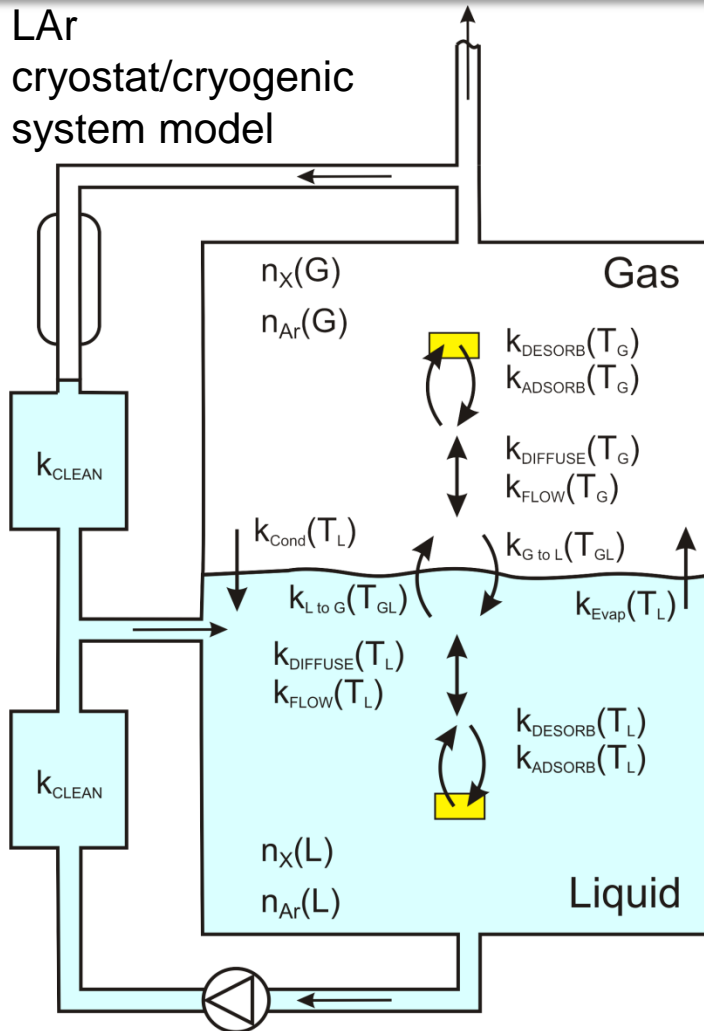Flavio Cavanna and
Brooke Russell

OUTLINE:

- o The model and the inputs

- o Dissolution/Devolution equilibrium and rates

- o Desorption and Diffusion rates

- o Electron Attachment and impact on LAr TPCs

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LAr cryostat/cryogenic system model

Note that transport by diffusion requires solving PDEs; other processes can be modeled by ODEs.

The rate constant, k, for each process and impurity implies a differential equation.

The solution of all twelve coupled differential equations determines the dynamic and steady- state impurity concentrations in LAr.

$$K_H(T_{GL}) = \frac{k_{L\,to\,G}(T_{GL})}{k_{G\,to\,L}(T_{GL})}$$ (Henry's Law)

This constrains the ratio of the unknown dissolution & devolution rate constants. At equilibrium, Henry's law determines impurity concentrations:

$x(liq) = K_{H,xx}\, x(gas)$, x is mole fraction of impurity

For LAr cryostats, dominant rate processes are k_{CLEAN} , $k_{DESORP}(T_G)$, and k_{Evap} ; $k_{G-L/L-G}$ may be important.

We need a complete and experimentally verified model, including diffusion and temperature distributions, to enable quantitative design decisions.

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Dissolution/Devolution equilibrium and rates

Scatchard-Hildebrand Theory of Liquid Solutions

$$\Delta E_{vap} = \Delta H_{vap} - RT$$

$$C_{ii} = \Delta E_{vap,i} / V_{M,i} \quad \longleftarrow \quad \text{Cohesive energy density}$$

$$C_{12} = \sqrt{C_{11}C_{22}}(1 - l_{12}) \quad \longleftarrow \quad \text{Exchange energy density}$$

$$\delta_{ii} = \sqrt{C_{ii}} \quad \longleftarrow \quad \text{Solubility parameters}$$

$$\Phi_i = x_i V_{M,i} / \sum_j x_j V_{M,j}$$

$$G_{E,mix} = (C_{11} + C_{22} - 2C_{12})(x_1 V_{M,1} + x_2 V_{M,2})\,\Phi_1\,\Phi_2$$

$$G_{E,mix} = \frac{x_1 V_{M,1} x_2 V_{M,2}\left(\delta_1^2 + \delta_2^2 - 2\delta_1\delta_2(1 - l_{12})\right)}{x_1 V_{M,1} + x_2 V_{M,2}}$$

$$\ln \gamma_i = \frac{\partial G_{E,mix}}{\partial x_i} \quad [Gibbs - Duhem\ relation]$$

$$\ln \gamma_1 = \frac{V_{M1} V_{M2}^2 (x_1 - 1)^2 \left(\delta_1^2 + \delta_2^2 - 2\delta_1\delta_2(1 - l_{12})\right)}{RT((x_1 - 1)V_{M2} - x_1 V_{M1})^2} \quad [liquid]$$

$$\ln \gamma_1 = \frac{P\,y_1}{P_{vap,1} x_1} \quad [impurity\ in\ gas]$$

$$\ln \gamma_2 = \frac{P(1 - y_1)}{P_{vap,2}(1 - x_1)} \quad [Ar\ in\ gas]$$

$$y1 = \frac{P_{vap,1} x_1 \gamma_1}{P_{vap,1} x_1 \gamma_1 + P_{vap,2}(1 - x_1)\gamma_2},\ P = P_{vap,1} x_1 \gamma_1 + P_{vap,2}(1 - x_1)\gamma_2$$

Vapour–liquid equilibria for cryogenic mixtures

E. R. Bazúa and J. M. Prausnitz

Although much attention has been directed towards developing a theoretically-satisfying theory of liquid solutions, most of the theoretical work so far reported is not as yet in a form useful for engineering calculations. There is good reason to believe that progress is being made and that new theoretical results will eventually be reduced to practice but for the immediate future it is likely that process design engineers will want to utilize results obtained from older and simpler theoretical descriptions of liquid mixtures.

A particularly simple, semi-empirical treatment of liquid solutions, based on solubility parameters, was given forty years ago by Scatchard and by Hildebrand.[1,2,3] Their description, known as the theory of regular solutions, provides a fair approximation of those properties of liquid solutions needed for vapour–liquid equilibrium calculations; the derivation of the Scatchard–Hildebrand theory shows that it is limited to mixtures of non-polar (or weakly polar) components whose molecules are approximately spherical and not very large. In other words, the theory should be most useful for those liquid mixtures which are of interest in cryogenics. In this work, we examine the applicability of Scatchard–Hildebrand theory to mixtures of simple liquids at low temperatures. In particular, we show that for reliable quantitative work, the original theory must be modified through the introduction of a binary parameter which relaxes the restrictive geometric-mean assumption. When this binary parameter is introduced into the Scatchard–Hildebrand solubility parameter equation, it is possible to make good vapour–liquid equilibrium calculations as needed for low temperature chemical process design.

Activity coefficients from solubility parameters

Three important assumptions are made in the derivation of the well-known solubility parameter equation for activity coefficients. They are

1. At constant temperature, the saturated liquid components mix with no volume change.

2. At constant temperature and volume the excess entropy of mixing is zero.

3. The cohesive energy density C_{12} corresponding to the interaction between dissimilar molecules 1 and 2, is given by the geometric mean of the pure-component cohesive energy densities, C_{11} and C_{22}.

At conditions remote from critical, the first assumption is usually correct to within less than 1 or 2%. The second assumption is not good but, because of a fortunate cancellation of errors, it has little effect on the calculation of partial molar Gibbs energies which, in turn, determine the activity coefficients. The third assumption is usually good to within a few percent but unfortunately the activity coefficients are very sensitive to small departures from the geometric mean. Therefore, if the solubilty parameter equation is to be useful for quantitative, rather than semi-quantitative purposes, it is necessary to relax the third assumption.

Using only assumptions 1 and 2, the molar excess Gibbs energy g^E for a binary mixture is written
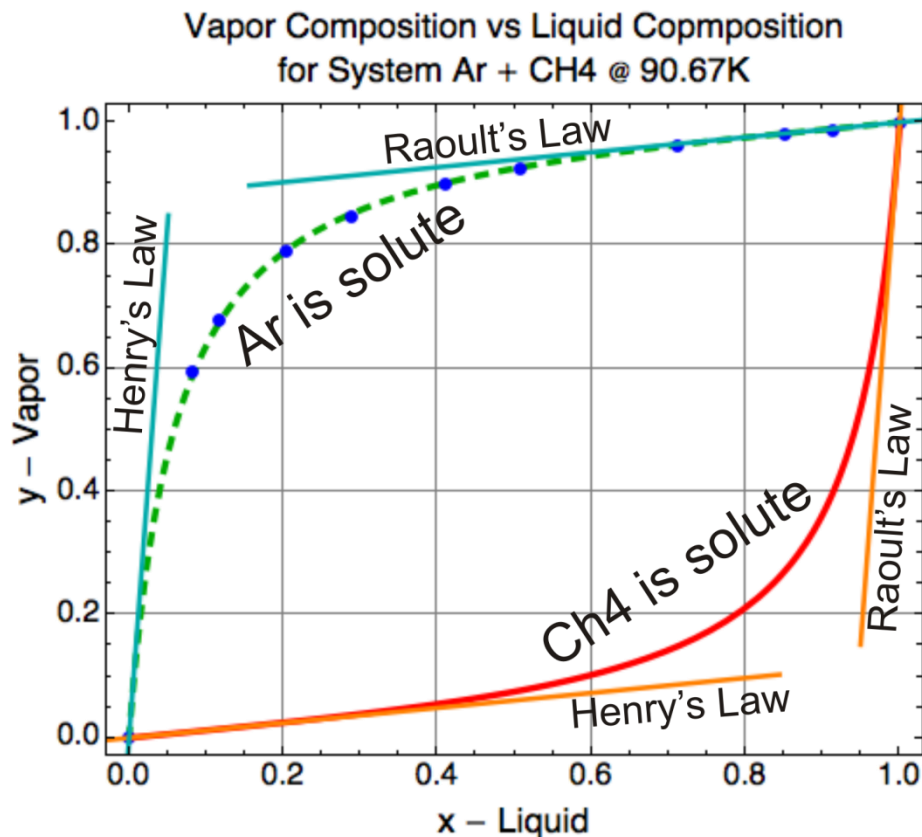
$$g^E = A_{12}(x_1 v_1 + x_2 v_2)\,\Phi_1\Phi_2 \qquad \ldots(1)$$

where, for component i, x_i is the liquid-phase mole fraction, v_i is the pure-component liquid molar volume, and Φ_i is the volume fraction

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$$K_H(T) = \lim_{x \to 0} \frac{\partial y_1}{\partial x_1}$$

Vapor Composition vs Liquid Copmposition
for System Ar + CH4 @ 90.67K

Henry's constant for CH_4 dissolved in Ar
Is
Raoult's constant for Ar dissolved in CH_4
and

Raoult's constant for CH4 dissolved in Ar
Is
Henry's constant for Ar dissolved in CH_4
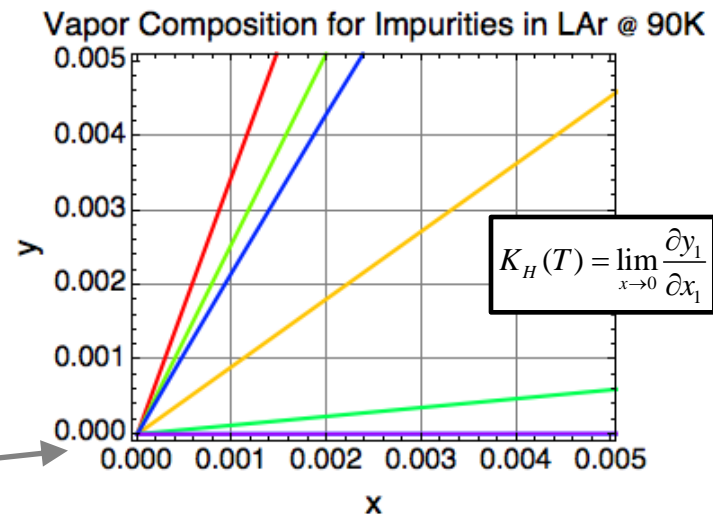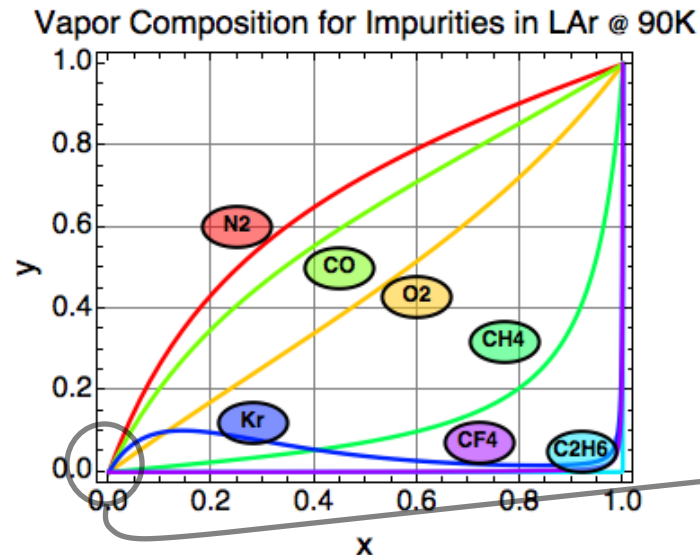
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Total Pressure for Impurities in LAr @ 90K
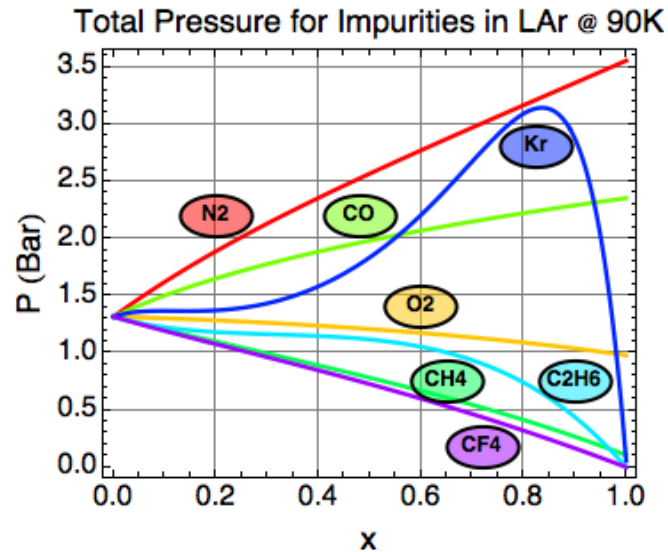
$$\Delta(T) = \frac{\delta_1^2 + \delta_2^2 - 2\delta_1\delta_2(1-\ell_{12})}{RT\left(V_{M,2}(1-x_1) + V_{M,1}x_1\right)^2}$$

$$y_1 = \frac{e^{-(V_{M,1}V_{M,2}^2(1-x_1)^2 - V_{M,1}^2V_{M,2}x_1^2)\Delta(T)}}{P_{Vap,2}(1-x_1)/(P_{Vap,1}x_1)+1}$$

$$P(x_1,T) = e^{V_{M,1}^2V_{M,2}x_1^2\Delta(T)}P_{Vap,2}(1-x_1) + e^{V_{M,1}V_{M,2}^2(1-x_1)^2\Delta(T)}P_{Vap,1}x_1$$

$$K_H(T) = \frac{P_{Vap,1}}{P_{Vap,2}}e^{V_{M,1}\Delta(T)}$$

Vapor Composition for Impurities in LAr @ 90K

Vapor Composition for Impurities in LAr @ 90K

$$K_H(T) = \lim_{x\to 0}\frac{\partial y_1}{\partial x_1}$$

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Where Do Impurities Reside?
Henry Coefficients describe impurity partition between liquid and gas phases

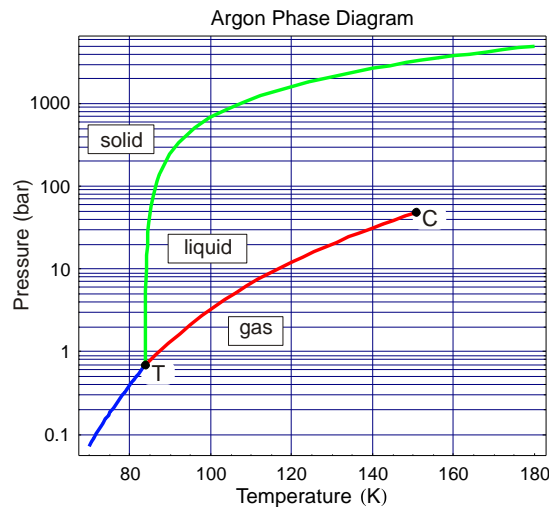$$k_{H,xx} = \frac{y_{Impurity}}{x_{Impurity}}$$

y = mole fraction in gas

x = mole fraction in liquid

solvent is 1

solute is 2

$$K_H(T) = \frac{P_{Vap,1}}{P_{Vap,2}} e^{V_{M,1}\Delta(T)}$$

$$\Delta(T) = \frac{\delta_1^2 + \delta_2^2 - 2\delta_1\delta_2(1-\ell_{12})}{RT\left(V_{M,2}(1-x_1) + V_{M,1}x_1\right)^2}$$

Argon Phase Diagram

Henry's Coefficients for LAr at 89K

Solute	T_T K	kH,xx	x2 Max	Ref
N_2	63.1	3.5	1	{ID1, ID2, ID13, ID18}
O_2	54.3	0.91	1	{ID1, ID13, ID18}
He	–	4150	?	{ID15}
Ne	24.6	955	0.9	{ID15, ID16, ID19, ID20, ID21}
Kr	116.	2.2	?	{ID1}
Xe	161.	$<10^{-5}$	0.05	{ID22}
H_2	14.	780	0.25	{ID7, ID14}
D_2	18.7	610	?	{ID7}
CO	68.1	2.8	1	{ID1, ID18}
CO_2	217.	?	$2.\times10^{-6}$	{ID6, ID9}
CH_4	90.7	0.12	?	{ID1, ID2, ID11}
CF_4	89.4	0.0012	?	{ID1}
Ethane	91.	0.000077	?	{ID1}
Propane	85.	$7.\times10^{-8}$	?	{ID1}
1,3–Butadiene	164.	?	$8.\times10^{-7}$	{ID5}
n–Pentane	143.	?	2.3×10^{-6}	{ID5}
2–Methylbutane	113.	?	0.000014	{ID23}
Neopentane	257.	?	0.00079	{ID5}
1–Pentene	108.	?	$7.\times10^{-7}$	{ID23}
Isoprene	127.	?	2.4×10^{-6}	{ID30}
Cyclopentane	180.	?	9.9×10^{-8}	{ID5, ID29}
Cyclopentene	138.	?	7.1×10^{-6}	{ID28}
Hexane	178.	?	1.6×10^{-7}	{ID26}
2–Methylpentane	120.	?	2.3×10^{-7}	{ID27}
1–Hexene	133.	?	6.7×10^{-7}	{ID27}
1–Hexyne	141.	?	1.5×10^{-7}	{ID28}
Cycloexane	280.	?	1.6×10^{-7}	{ID26}
2,3–Dimethylbutane	144	?	0.000012	{ID5, ID29}
H_2O	273.	?	$<10^{-8}$	{ID6}
N_2O	182.	?	0.000074	{ID6}
Dimethyl ether	135.	?	0.000013	{ID6}
Diethyl ether	157.	?	4.5×10^{-6}	{ID6}
Di–n–propyl ether	158.	?	3.7×10^{-6}	{ID6}
Di–isopropyl ether	188.	?	$2.\times10^{-7}$	{ID6}

Selected Henry's Coefficients
For liquids and solids dissolved in LAr

Solute	T_T K	kH,xx	x2 Max	Ref
N_2	63.1	3.5	1	{ID1, ID2, ID13, ID18}
O_2	54.3	0.91	1	{ID1, ID13, ID18}
He	–	4150	?	{ID15}
Ne	24.6	955	0.9	{ID15, ID16, ID19, ID20, ID21}
Kr	116.	2.2	?	{ID1}
Xe	161.	$<10^{-5}$	0.05	{ID22}
H_2	14.	780	0.25	{ID7, ID14}
D_2	18.7	610	?	{ID7}
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Cycloexane	280.	?	1.6×10^{-7}	{ID26}
2,3–Dimethylbutane	144	?	0.000012	{ID5, ID29}
H_2O	273.	?	$<10^{-8}$	{ID6}
N_2O	182.	?	0.000074	{ID6}
Dimethyl ether	135.	?	0.000013	{ID6}
Diethyl ether	157.	?	4.5×10^{-6}	{ID6}
Di–n–propyl ether	158.	?	3.7×10^{-6}	{ID6}
Di–isopropyl ether	188.	?	$2. \times 10^{-7}$	{ID6}

Where Do Impurities Reside?
Henry Coefficients describe impurity partition between liquid and gas phases

Extremely high purity LAr is required to avoid charge attenuation along drift.

(Not a problem for light collection, since for common impurities the optical attenuation length is ~1000 times greater than electron mean drift length)

H_2O is a problem:

- H_2O appears to have a large (but *unknown*) attachment rate.

- Henry coefficient for H_2O is *unknown*, but is presumably very small (<<0.1).

- Saturation solubility of H_2O in LAr is extremely low (<10^{-8}): therefore, the system may have three-phases (by the phase rule, all concentrations are then constant at a fixed temperature, independent of the amount of H_2O in the system).

- **We need to understand the location (partition) of H_2O in LAr TPCs, in order to determine how best to minimize it's concentration in the liquid, to maximize the electron mean drift length.**
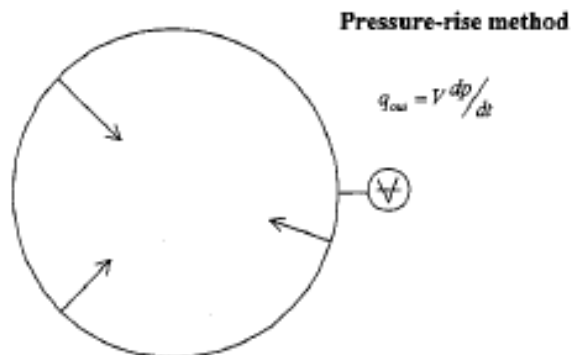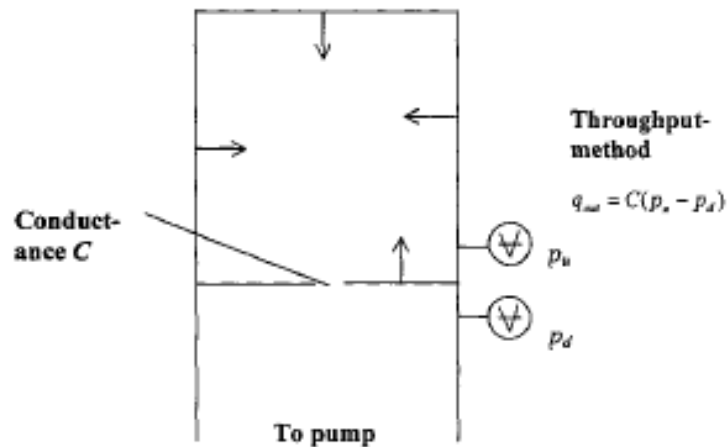
$$k_{H,xx} = \frac{y_{Impurity}}{x_{Impurity}} = \frac{k_{Liquid \to Gas}}{k_{Gas \to Liquid}}$$

$$k_{Mean} = \sqrt{k_{Gas \to Liquid} \times k_{Liquid \to Gas}}$$

y = mole fraction in gas

x = mole fraction in liquid

Nothing is known about rate constants; therefore, we cannot calculate over what time scales impurities distribute themselves in the system.

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Desorption and Diffusion rates

Outgassing Rate Measurement

Throughput-method

$$q_{out} = C(p_u - p_d)$$

Conduct-ance C

p_u

p_d

To pump

Pressure-rise method

$$q_{out} = V \frac{dp}{dt}$$

Total Outgassing Rate
for 304 SS

$E_A \sim 0.5 eV$

- ○ fully degassed
- △ oxidized

Outgassing rate (10^{-9} Pa m s^{-1})

$1/T$ (10^{-3} K^{-1})

$U = 0.43$ eV

diffusion coefficient, m^2/s

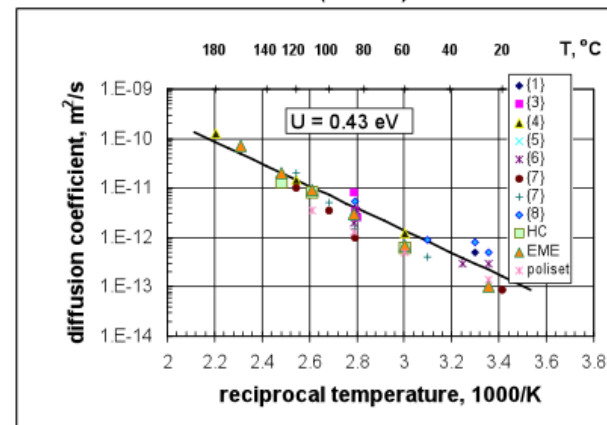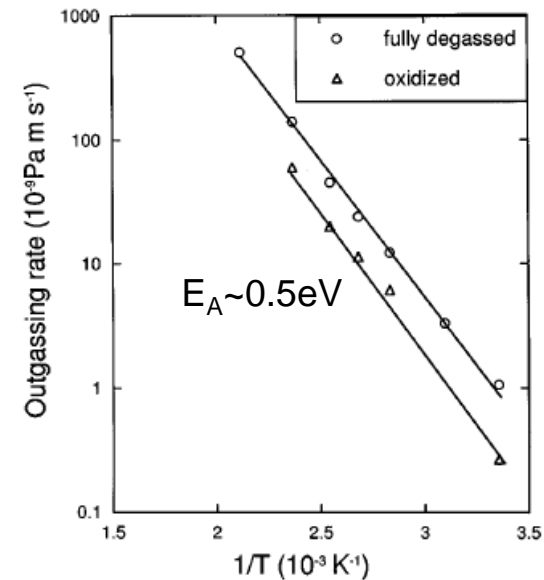reciprocal temperature, 1000/K

Figure 4. Experimental data on temperature dependence of moisture diffusion coefficient for different epoxy encapsulating materials. Figures in brackets are references.

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Water Desorption by FR4

$$k_D(T) = Exp[-E_A/k(T-T_0)]$$

Fig. 3. Relative weight loss for 2 epoxy based PCBs at different temperatures.

Normalized Reaction Rate for Water Desorption by FR4 & SS

FR4 E_A = 0.32 eV

SS E_A = 1.1eV

Data

Extrapolation

FR4: K. Weide-Zaage et al., Microelectronics Reliability 45 (2005) 1662
SS: J.H. Hendricks, Temperature Programmed Desorption Measurements of the Binding Energy of Water to Stainless Steel Surfaces, AVS 54th International Symposium (2007)

Each surface adsorbed species has a characteristic binding energy; different binding/diffusion for bulk absorbed species.

We re developing a cryostat for measurement of outgassing rates vs. temperature for common materials & impurities.

# Effects of Diffusion, Solution, & Adsorption Rates
Water desorption & oxygen adsorption by stainless steel

A. Bettini, *et al.*, *A study of the factors affecting the electron lifetime in ultra-pure liquid argon*, NIM A305 (1991) 177

Measurements in an isothermal cell 250 cm^3 volume

A. Bettini et al. / A study of the factors affecting the electron lifetime

Purified LAr doped with 3 ppb O$_2$
E = 400 V/cm for T ≤ 4000 min
E = 50 V/cm for T ≥ 4000 min
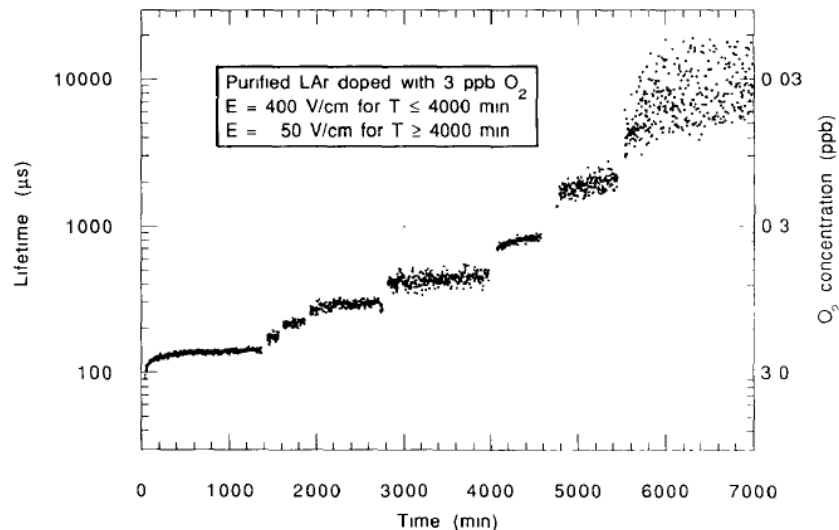
Lifetime and inferred [O$_2$] after doping with 3 ppb O$_2$. The jumps are due to stirring.

A. Bettini et al. / A study of the factors affecting the electron lifetime   185

Purified LAr
40 ppb O$_2$ introduced at T=250 min

E = 50 V/cm
E = 200 V/cm
E = 400 V/cm
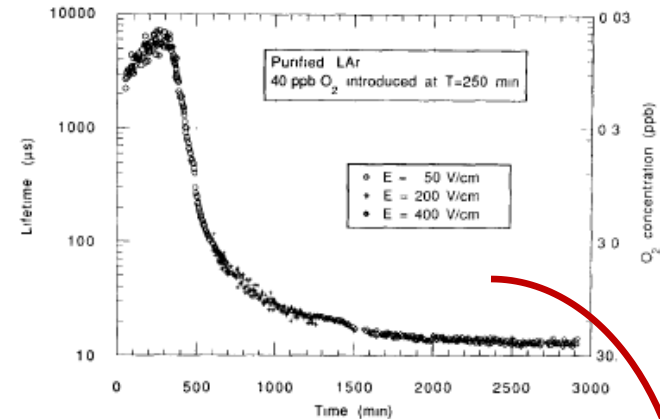
11. Electron lifetime τ measured in LAr doped with 40 ppb of O$_2$. The O$_2$ was introduced into the cell (in the gas phase) 250 min after it had been filled with purified LAr.

40 ppb O$_2$ introduced @ T=250 min
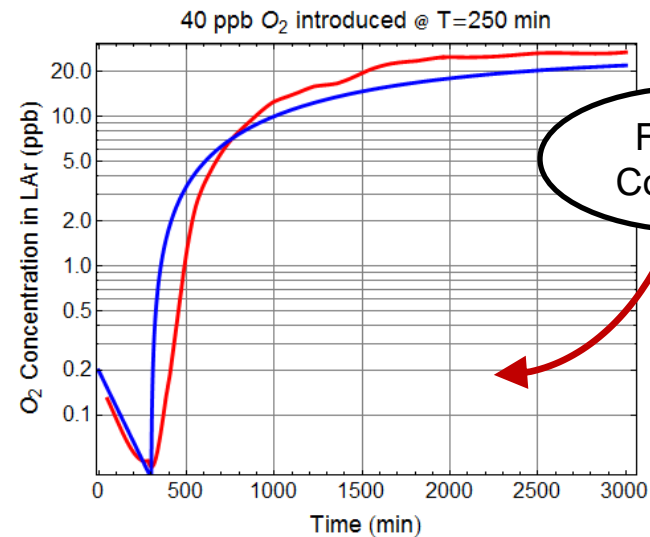
Fit Rate Constants

Need to quantify diffusion and adsorption rate constants (and Henry constants) vs. temperature for common materials & impurities!

Electron Attachment and impact on LAr TPCs

Properties of LAr – Response to ionizing radiation

X={N_2, O_2, H_2O, ...}

Ionization — **Excitation** — **Heat & Radiation**

Ar^+ ⟷ e^- — Penning Ionization — Ar^* — Decay — Ar

X^+ X

87-107nm

Recombination

Ar^*

Resonance Absorption

Attachment

Ar_2^* — Dissociation
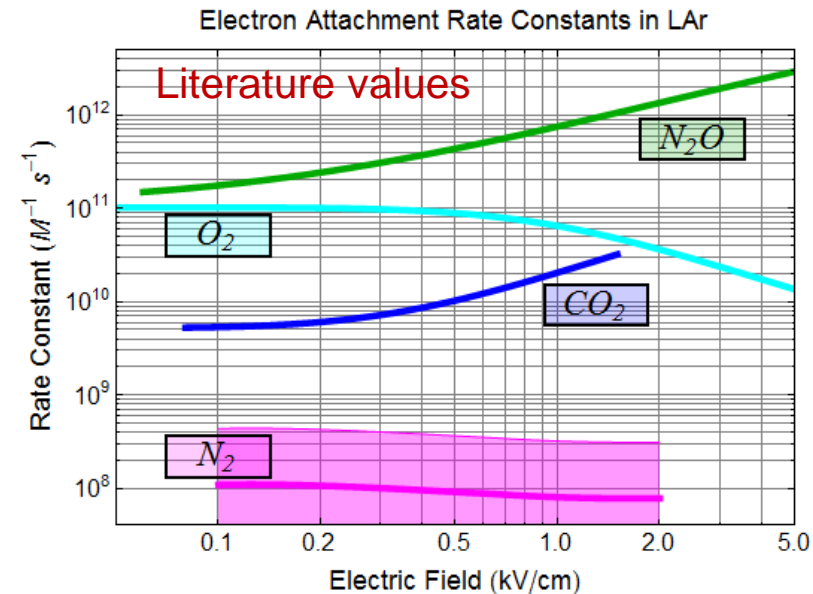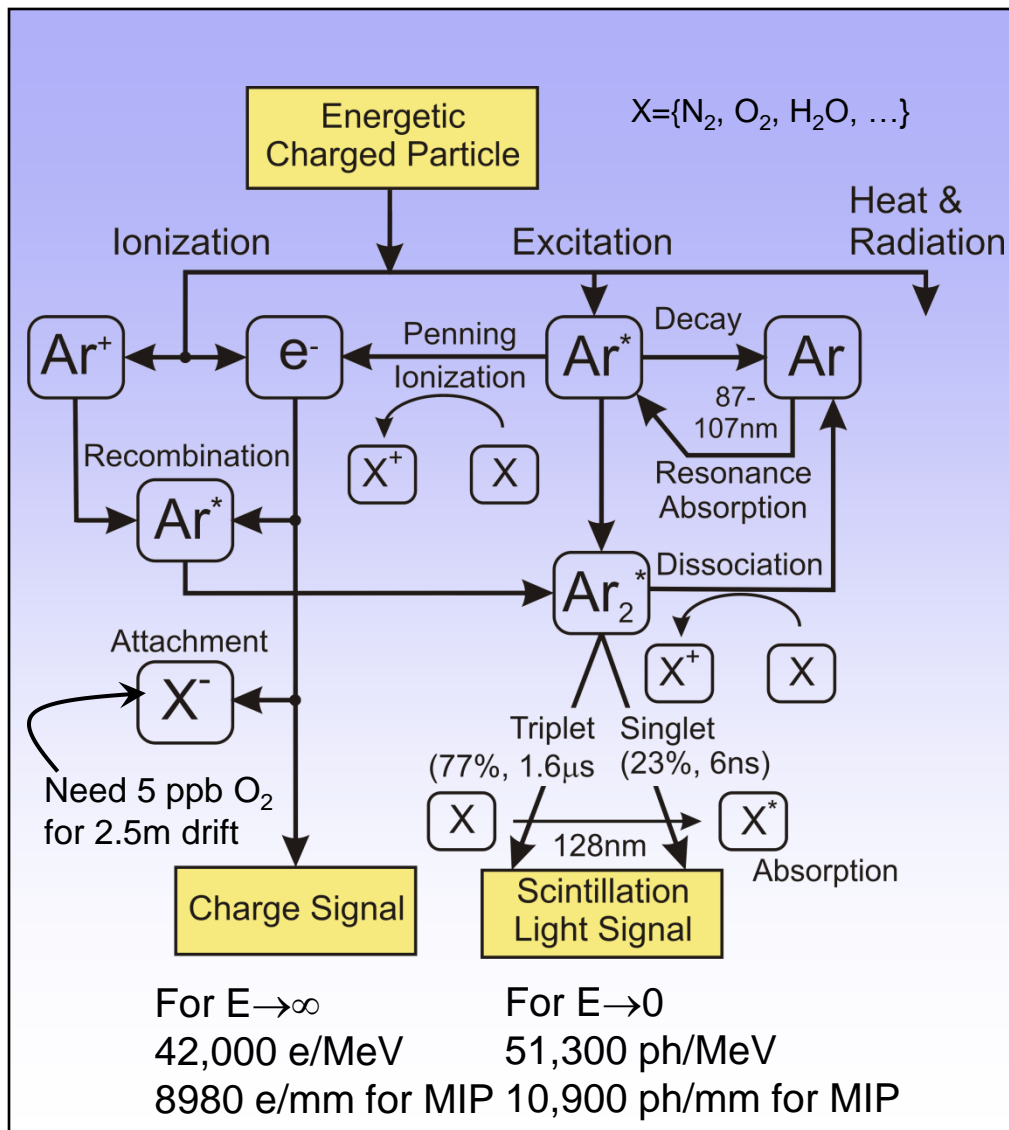
X^-

X^+ X

Triplet (77%, 1.6μs) Singlet (23%, 6ns)

X

X X^*

128nm — Absorption

Need 5 ppb O_2 for 2.5m drift

Charge Signal

Scintillation Light Signal

For E→∞
42,000 e/MeV
8980 e/mm for MIP

For E→0
51,300 ph/MeV
10,900 ph/mm for MIP

Electron Attachment Rate Constants in LAr

Literature values

N_2O

O_2

CO_2

N_2

Rate Constant ($M^{-1} s^{-1}$)

Electric Field (kV/cm)

To do:

1. Electron attachment to H_2O appears to be important process, but rate constant is not known.

2. Present value of attachment constant for N_2 appears to be too large to be compatible with known drift times and N_2 impurity levels.

Simple Kinetic Model for LAr Purity

Steady state solution (*as* $t \to \infty$):

$$n \to \frac{(A_{Cable,Eff} \, k_{Cable,Ef}(T_{Ref}) + A_{SS,Eff} \, k_{SS,Ef}(T_{Ref})) \, V_L}{dV_L / dt} \qquad with \; A_{X,Eff} = \frac{A_X}{S \, k_{Outgass,X}(T_{Ref})} \int_s k_{Outgass,X}[T(s)] \, ds$$

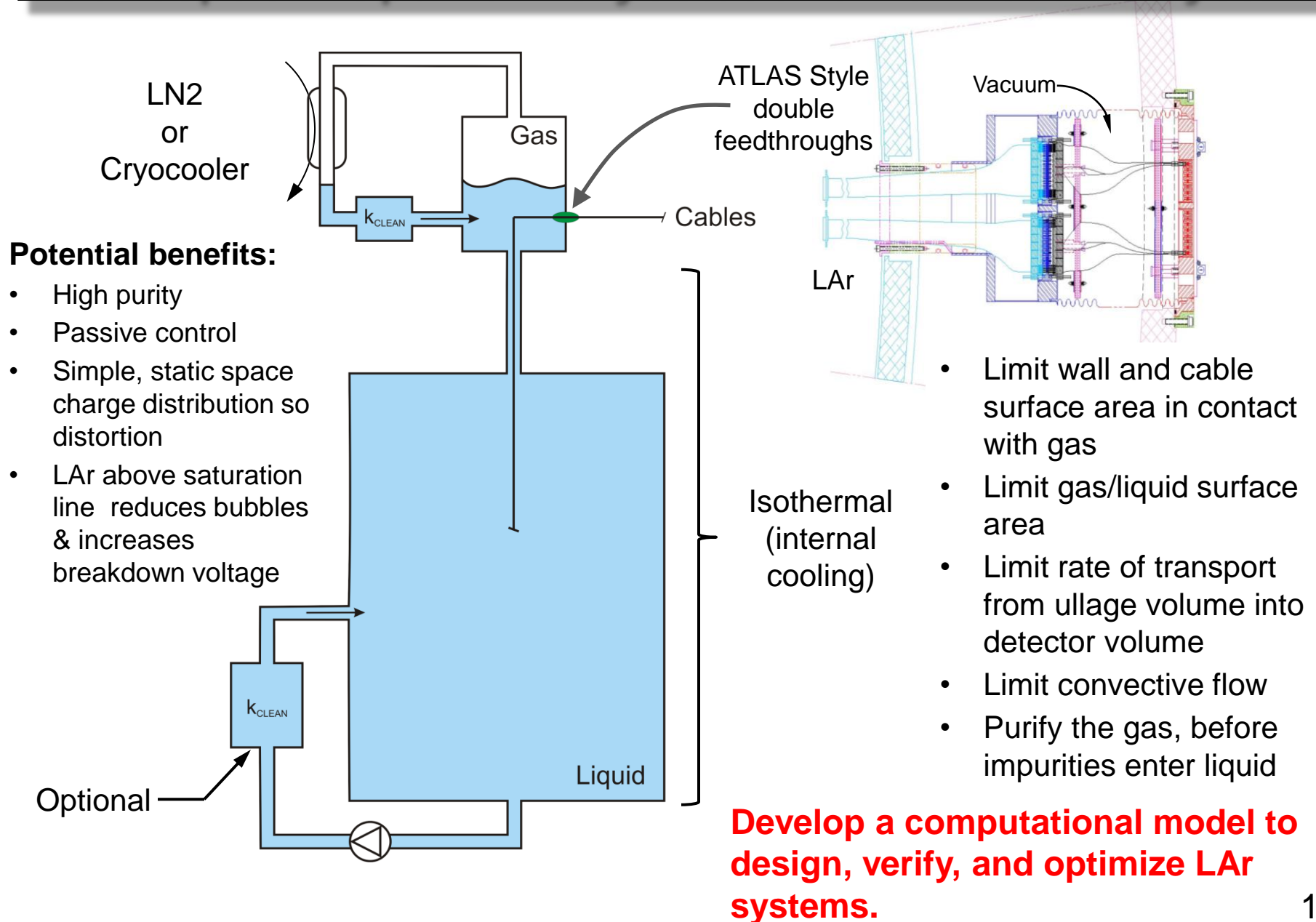use $\tau = \dfrac{1}{k_{Attach}(n/V)}$ and determine $k_{Cable,Eff}$ and $k_{SS,Eff}$ from LAPD & ICARUS data

find $k_{Outgass,SS} = 0.2 \, k_{Outgass,Cable} = 5 \times 10^{-12} \; mole \; m^{-2} \; s^{-1} \; (cf. \sim 2 \times 10^{-12} \; for \; SS \; scaled \; to \; 250 \, K)$

Values in black are input to, and red are output from, this model

Detector	LAr Volume (m³)	Effective Ullage Surface (m²)	Effective Cables	Volume Exchange (hr)	Recirculation Rate (liter/s) as liquid	Lifetime (ms) **Given** or **Deduced**
ICARUS	523	0.32	53000	144	1.0	7
LAPD	7.78	26.3	10	6.0	0.36	3
"MTS"	0.25	0.69	6	2.1	0.033	10 (cf. 7-10)
ArgoNeuT	0.55	0.20	960	170	0.0009	0.6 (cf. 0.7)
BNL "TS"	0.021	0.41	6	9	0.0007	1 (cf. >0.1)
MicroBooNE	136	31	8300	24	1.6	10

Concept of "Optimal" Cryostat and Purification System

LN2
or
Cryocooler

Gas

k_{CLEAN}

ATLAS Style double feedthroughs

Cables

LAr

Vacuum

Potential benefits:

- High purity
- Passive control
- Simple, static space charge distribution so distortion
- LAr above saturation line reduces bubbles & increases breakdown voltage

Isothermal (internal cooling)

k_{CLEAN}

Optional

Liquid

- Limit wall and cable surface area in contact with gas
- Limit gas/liquid surface area
- Limit rate of transport from ullage volume into detector volume
- Limit convective flow
- Purify the gas, before impurities enter liquid

Develop a computational model to design, verify, and optimize LAr systems.

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Summary

o Much is known about impurities in LAr. But we need to:

- Quantify partition of H_2O in LAr Henry's coefficient and solubility.
- Measure attachment coefficient vs E for H_2O.
- Investigate importance of more exotic contaminants (CO_2).
- Measure adsorption/desorption Henry's coefficients for SS, FR-4,…
- Measure solution/devolution and adsorption/desorption rates.
- Develop kinetic model of impurities in LAr TPCs

o Goal is to design cryostat and cryogenic systems for very high LAr purity to increase electron lifetimes to permit long drift distances with the highest signal quality, while reducing the cost of the cryogenics, electronics, and TPC structures.

ID1   E.R. Bazua and J.M. Prausnitz, Vapour-liquid equilibria for cryogenic mixtures, Cryogenics 11 (1971) 114.

ID2   F.B. Sprow and J.M. Prausnitz, Vapor-Liquid Equilibria for Five Cryogenic Mixtures, A.I.Ch.E. Journal 12 (1966) 780.

ID3   G.T. Preston and J.M. Prausnitz, A Generalized Correlation for Henry's Constants in Nonpolar Systems, Ind. Eng. Chem. Fundam. 10 (1971) 389.

ID4   G.T. Preston and J.M. Prausnitz, Thermodynamics of Solid Solubility in Cryogenic Solvents, Ind. Eng. Chem. Process Des. Develop. 9 (1970) 264.

ID5   G.T. Preston, E. W. Funk, and J.M. Prausnitz, Solubilities of Hydrocarbons and Carbon Dioxide in Liquid Methane and in Liquid Argon, Journal of Physical Chemistry, 76 (1971) 2345.

ID6   A.J. Rest, R.G. Scurlock and M. Fai Wu, The Solubilities of Nitrous Oxide, Carbon Dioxide, Aliphatic Ethers and Alcohols, and Water in Cryogenic Liquids, Chemical Engineering Journal, 43 (1990) 25.

ID7   H. Volk and G.D. Halsey, Jr., Solubility of Hydrogen and Deuterium in Liquid Argon, Journal Of Chemical Physics 33 (1960) 1132.

ID8   R.C. Miller and J.M. Prausnitz, Statistical Thermodynamics of Simple Liquid Mixtures: Henry's Constants, I & E C Fundamentals 8 (1969) 449.

ID9   A.L. Myers and J.M. Prausnitz, Thermodynamics of Solid Carbon Dioxide Solubility in Liquid Solvents at Low Temperatures, I & E C Fundamentals 4 (1965) 209.

ID10 A. Baba-Ahmed, P. Guilbot, D. Richon, New equipment using a static analytic method for the study of vapour-liquid equilibria at temperatures down to 77 K, Fluid Phase Equilibria 166 (1999) 225.

ID11 S.P. Barreiros, M. N. da Ponte and J.C.G. Calado, Excess Thermodynamic Properties of Argon + Methane: A Standard Simple Liquid System, Fluid Phase Equilibria, 49 (1989) 9.

ID12 M.G.Zellner,L.C.Claitor,and John M.Prausnitz,Prediction of Vapor-Liquid Equilibria and Enthalpies of Mixtures at Low Temperatures,Ind.Eng.Chem.Fundam.9 (1970) 549.

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