Gasses R Us, LLC 1415 Engineering Drive
Madison, WI 53706

To: Professor Mallette
From: Nguyen Khoa Bui
CC: Lab Manager
Date: July 26, 2022
Subject: Re: Membrane Formal Experiment Memorandum Guidelines

A membrane gas separation procedure was setup to explore the TN 0800 separation unit under testing. Conditions for both oxygen and nitrogen enrichment processes were tested. Nitrogen enrichment only achieved 94% N_2 purity, which is below many industrial standard needs. The process also required retentate flowrate as low as 100 sccm/min and compressed air at 83 psig. Which are slow for industrial production and resource intensive. On the other hand, the module shown high selectivity for oxygen, allowing less use of compressed air to achieve at least 40% O_2 purity, an acceptable level in industrial standards for small-scale medical uses. While production at permeate stream can reach a high of up to 6500 sccm/min, this purity level only had O_2% recovery around 20%. Overall, we recommend the company to use the unit for oxygen enrichment only, but demand should be analyzed carefully to justify the intensive use of compressed air at the inlet with little recovery.

The membrane module was set up in accordance with the Membrane section of the CBE 424 Lab Manual (CBE 424 lab manual: membrane gas seperation protocol). The TN2 0800 separation unit under study was tested by alternating the inlet feed pressure, permeate rotameter flowrate, and retentate rotameter flowrate. The inlet feed consisted of dry air with its flowrate controlled by changing feed pressure and measured by a rotameter. The dry air passed through the membrane and split into a permeate and a retentate streams. The oxygen composition for both separated streams are measured with oxygen sensor. Their outlet pressures are measured by analog pressure sensors. These data were used to evaluate the module performance and

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efficiency by analyzing the outlets $O_2\%$ and $N_2\%$ purity and recovery and membrane behavior

with respect to R/F.

The module's integrity was ensured by performing mass balance closure on the overall

air, N_2 and O_2 flowrates. The mass balance has an average error of 8.2, 9.0, and 8.1% for air, O_2,

and N_2 respectively for all runs collected. These errors are within the error range computed total

equipment uncertainties of up to 9.4%. Despite the strong mass balance closure, the module

should be avoided operating with retentate flowrates below 1000 sscm. For this range, the mass

balance error was high at 17 to 18%. This was due to low retentate flowrate causing atmospheric

air to flow backward into the stream, contaminating the sampling line.

Unfortunately, the low retentate flowrate fell into the operating range for the nitrogen

enrichment trial, resulting in low $N_2\%$ purity at 94% and below at the retentate stream. This

purity level does not meet many industrial standards, such as tank inerting application at 95% or

food packaging at 99% purity or more according to the Membrane section of CBE 424 Lab

Manual (CBE 424 lab manual: membrane gas seperation protocol). This process condition also

operated at a low feed flowrate between 1700 and 2200 sccm, and high feed pressure of at least

75 psi. Furthermore, the recovery rate for 94% purity can range between 10-20% according to

figure 1. The company would be wasting highly compressed air for low and slow production.

While the module condition did not favor nitrogen enrichment process, the membrane

portrayed high selectivity for oxygen, ranging on average between 4.3 to 4.7 from data analysis.

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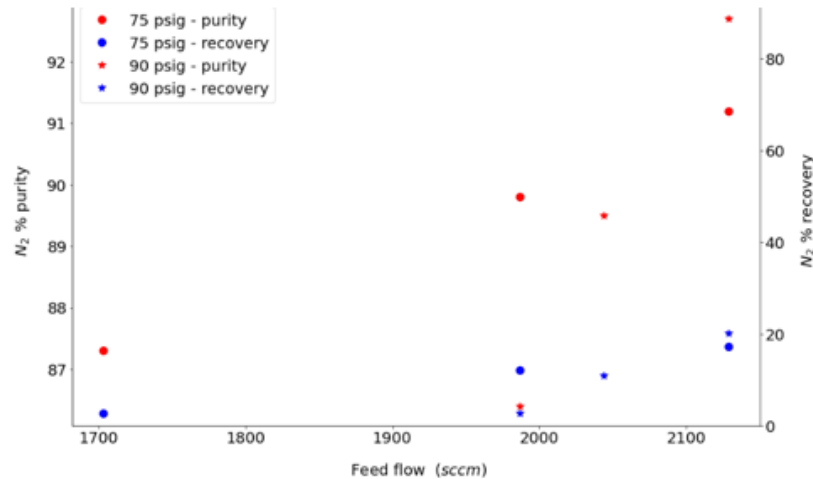Subject: Re: Membrane Formal Experiment Memorandum Guidelines

Figure 1. N_2% purity and recovery plotted against feed flowrate for two feed pressure 75 psig and 90 psig at room temperature T = 298K. Purity data (red points) are read with left y-axis and recovery data (blue points) are read with right y-axis.

The selectivity is within the range of the high-performance polymer as reported by other literatures. (L. M. RobesonW. F. BurgoyneM. LangsamA. C. Savoca, 1994) high selectivity, O_2% purity of up to 47% was achieved with feed flowrate above 10,000 sccm according to figure 2. This purity level is within the 40-60% satisfactory range for medical use in the industry. The resulting permeate flowrate also reached as high as 6500 sccm, a production rate up to 6 times higher than nitrogen enrichment process. Furthermore, feed pressure as low as 50 psi could achieved more than 40% purity level, a lower compressed air requirement compared to nitrogen enrichment process.

However, operating at such condition required high R/P ratio of up to at least 10. While data shown that higher R/F led to exponential growth of O_2% purity, the O_2% recovery rate

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would drop to 20% and below for product purity above 40%. As a result, large amount of

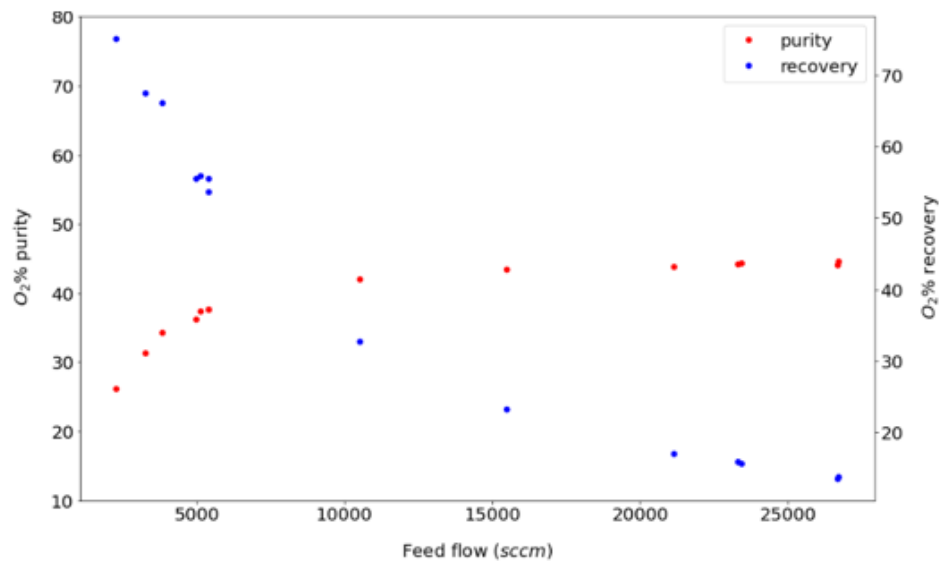processed air was wasted into the retentate stream.

Figure 2. O_2% purity and recovery plotted against feed flowrate for various feed and retentate pressures at room temperature T = 298K. Purity data (red points) are read with left y-axis and recovery data (blue points) are read with right y-axis.

In conclusion, the membrane module is a viable choice for oxygen enrichment process

due to high selectivity and satisfactory O_2% purity at permeate stream. The company should

ensure that the revenue in selling high purity oxygen would justify the use of the membrane

module. For nitrogen enrichment, we believe further testing would be needed to verify the

module's performance. However, even if we characterized N_2% purity to reach industrial

standard, production rate shown to be too slow, and the process required high inlet pressure

stream. The company should look for other membrane module that specialize in nitrogen

enrichment process.

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<u>References</u>

(n.d.). *CBE 424 lab manual: membrane gas seperation protocol.*

L. M. RobesonW. F. BurgoyneM. LangsamA. C. Savoca, C. F. (1994). *High performance polymers for membrane.* Retrieved from https://reader.elsevier.com/reader/sd/pii/0032386194906513?token=BB2A3198A22A46B50AE77D70592AFC322B52397A313307FF82FB2EECA7DF7BEAE18EE95B8DDAD9C24AE363E20ECE6568&originRegion=us-east-1&originCreation=20220724225420

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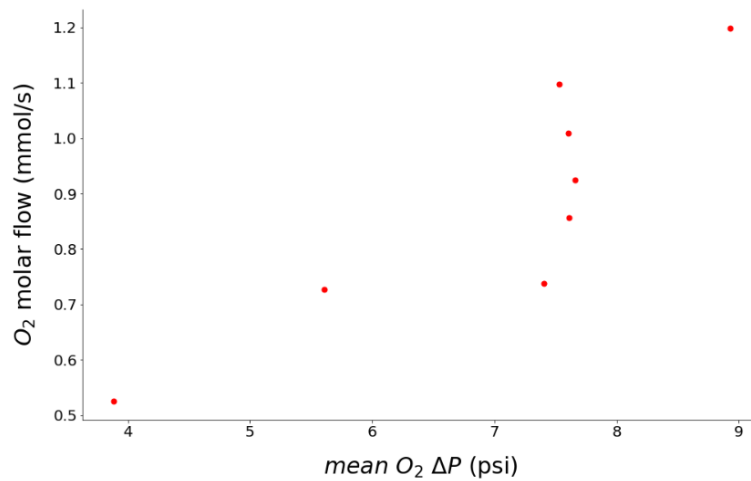Appendix:

Figures of data:

Figure 1. calculated O_2 molar flow rate versus the mean O_2 pressure difference at the permeate with room temperature T = 298 K. Data (red points) of varying feed pressure and retentate pressures are shown.
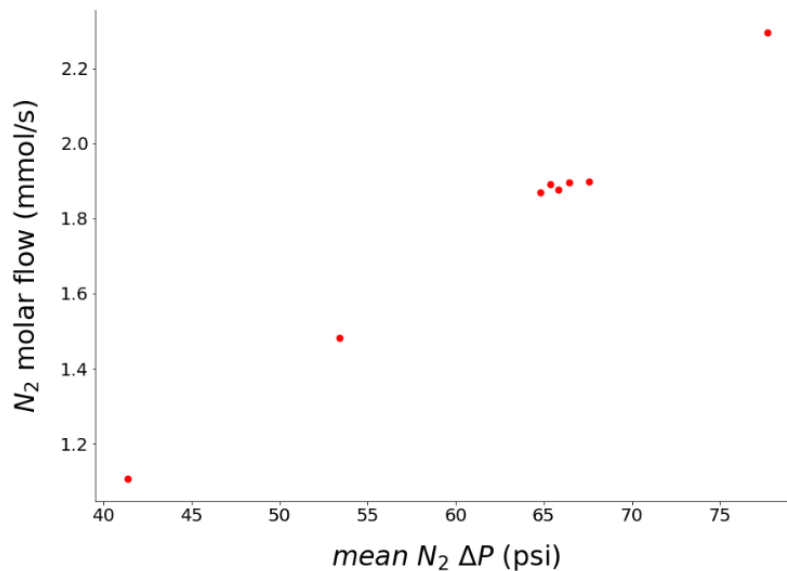

Figure 2. calculated N_2 molar flow rate versus the mean N_2 pressure difference at the permeate with room temperature T = 298 K. Data (red points) of varying feed pressure and retentate pressures are shown.

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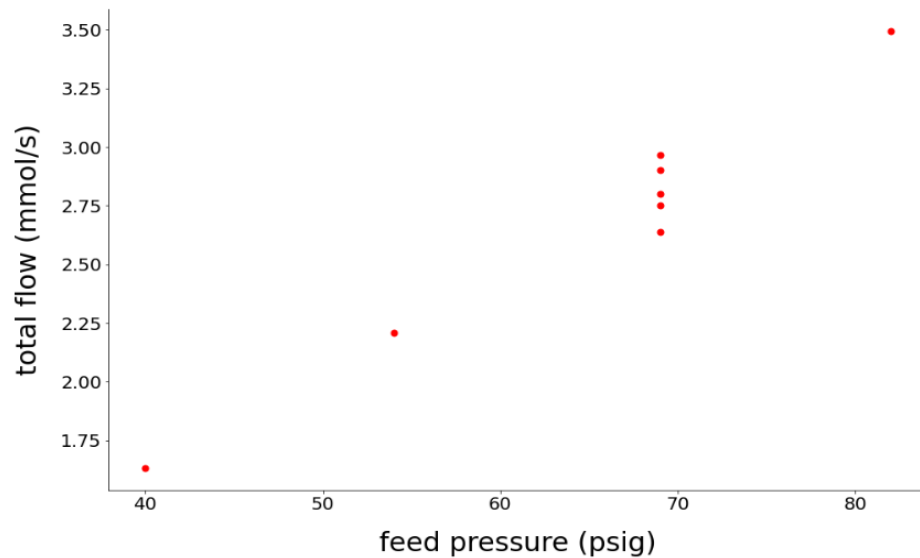

Figure 3. total molar flow rate versus the mean N_2 pressure difference at the permeate with room temperature T = 298 K. Data (red points) of varying feed pressure and retentate pressures are shown.
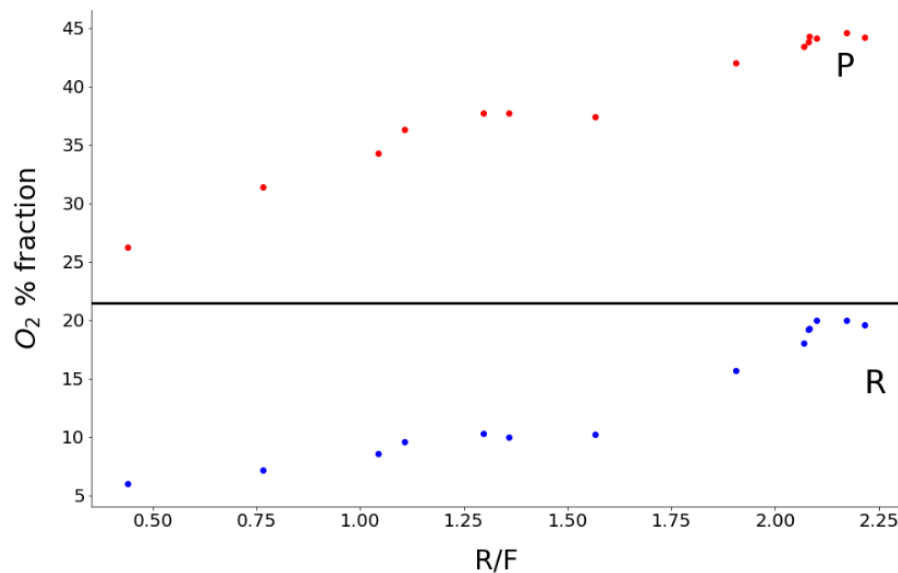

Figure 4. O_2% fraction at the permeate (red points) and the retentate (blue points) plotted against flowrate at the retentate over feed ratio R/F. The constant air O_2% fraction at the feed (black line) is also shown, separating the two stream's O_2% content.

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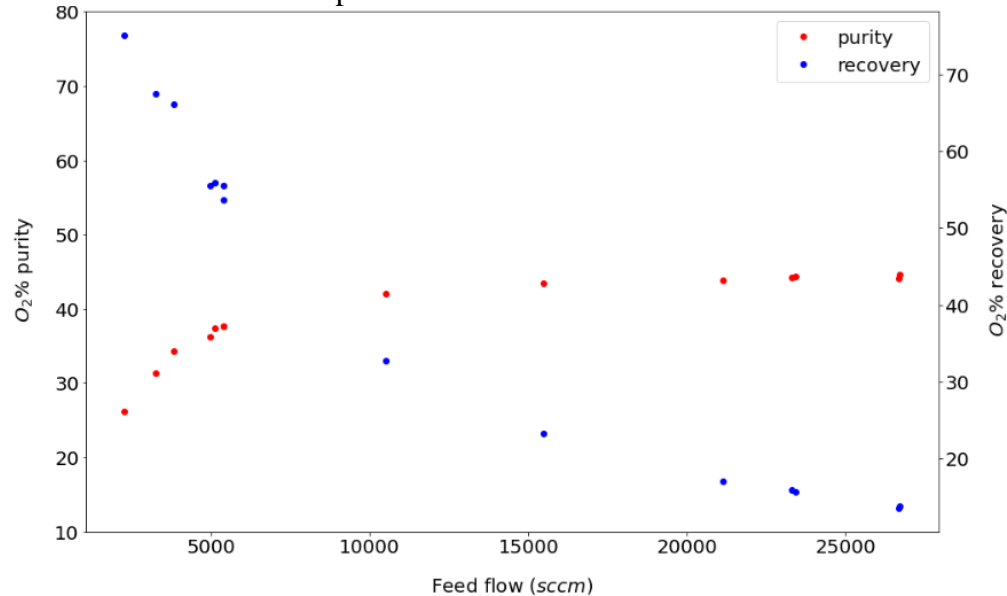
Figure 5. O$_2$% purity and recovery plotted against feed flowrate for various feed and retentate pressures at room temperature T = 298K. Purity data (red points) are read with left y-axis and recovery data (blue points) are read with right y-axis.

Figure 6. N$_2$% purity and recovery plotted against feed flowrate for two feed pressure 75 psig and 90 psig at room temperature T = 298K. Purity data (red points) are read with left y-axis and recovery data (blue points) are read with right y-axis.

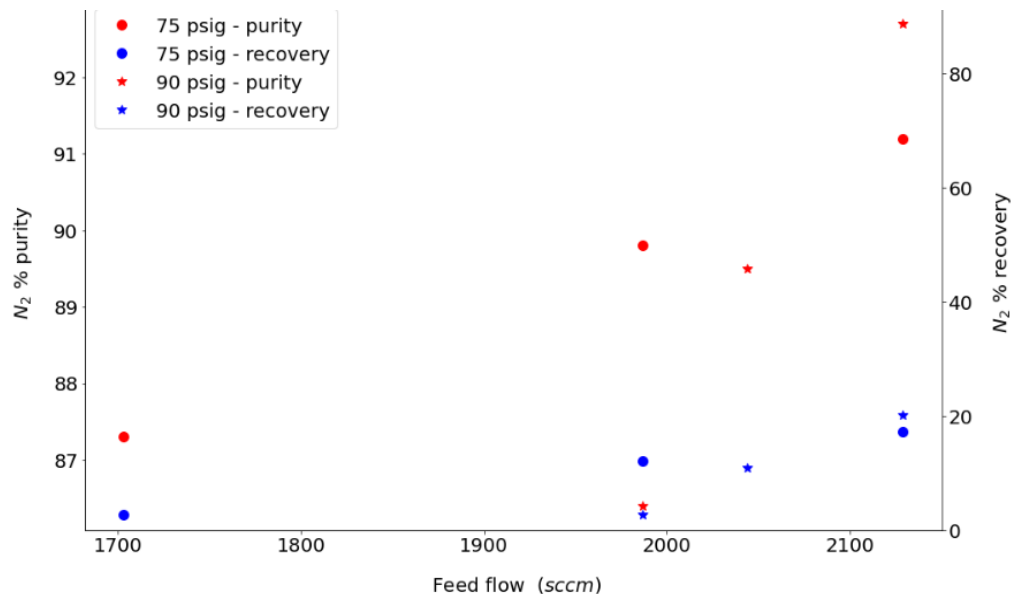Gasses R Us, LLC 1415 Engineering Drive
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Figure 7. O_2% composition in permeate stream plotted against ratio of permeate to retentate flowrate P/R. Data (red points) were collected at feed pressure of 75 psig and standard room temperature T = 298 K.

Figure 8. O_2% composition (red points) and O_2% recovery (blue point) in permeate stream plotted against feed pressure at room temperature T = 298 K.

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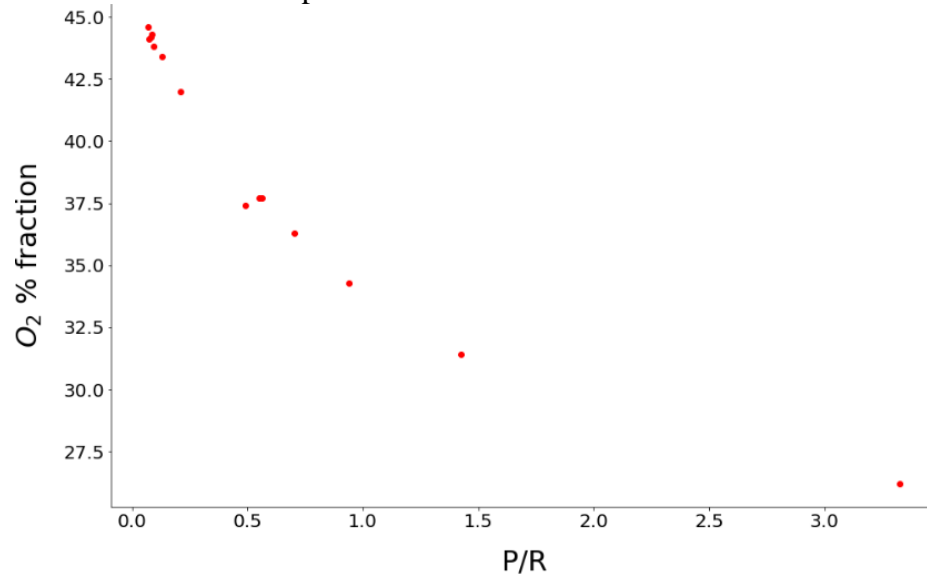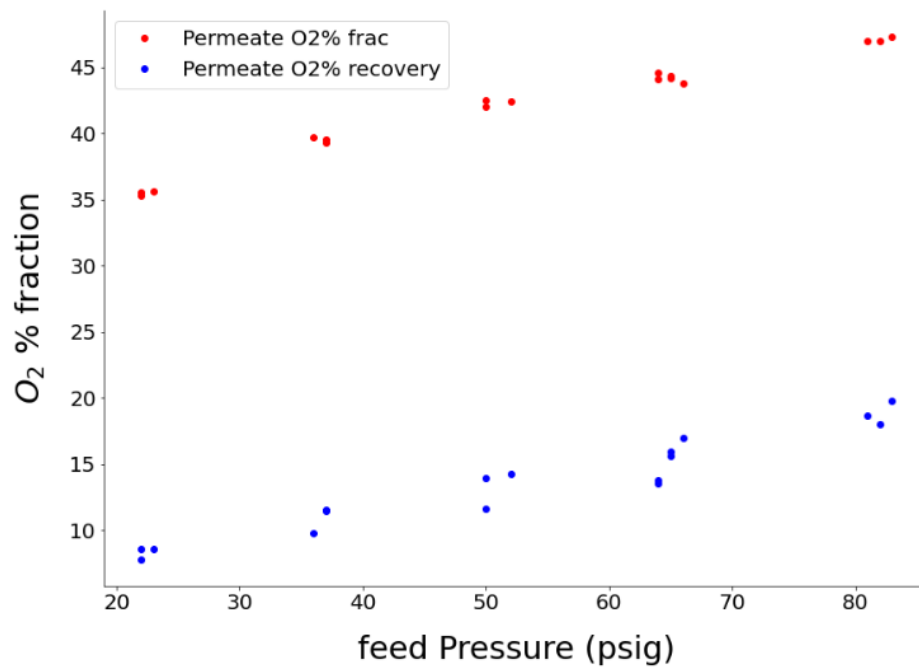Subject: Re: Membrane Formal Experiment Memorandum Guidelines

Figure 9. $O_2\%$ fraction (red and blue points) and the fitted permeability Q (red and blue lines) for permeate and retentate streams plotted against R/F at room temperature T = 298 K. The permeability model fitting is $y = a \ln x + b$, where a = 4.79, b = 23.29 for permeate stream and a = 4.189, b = 0 for retentate stream.

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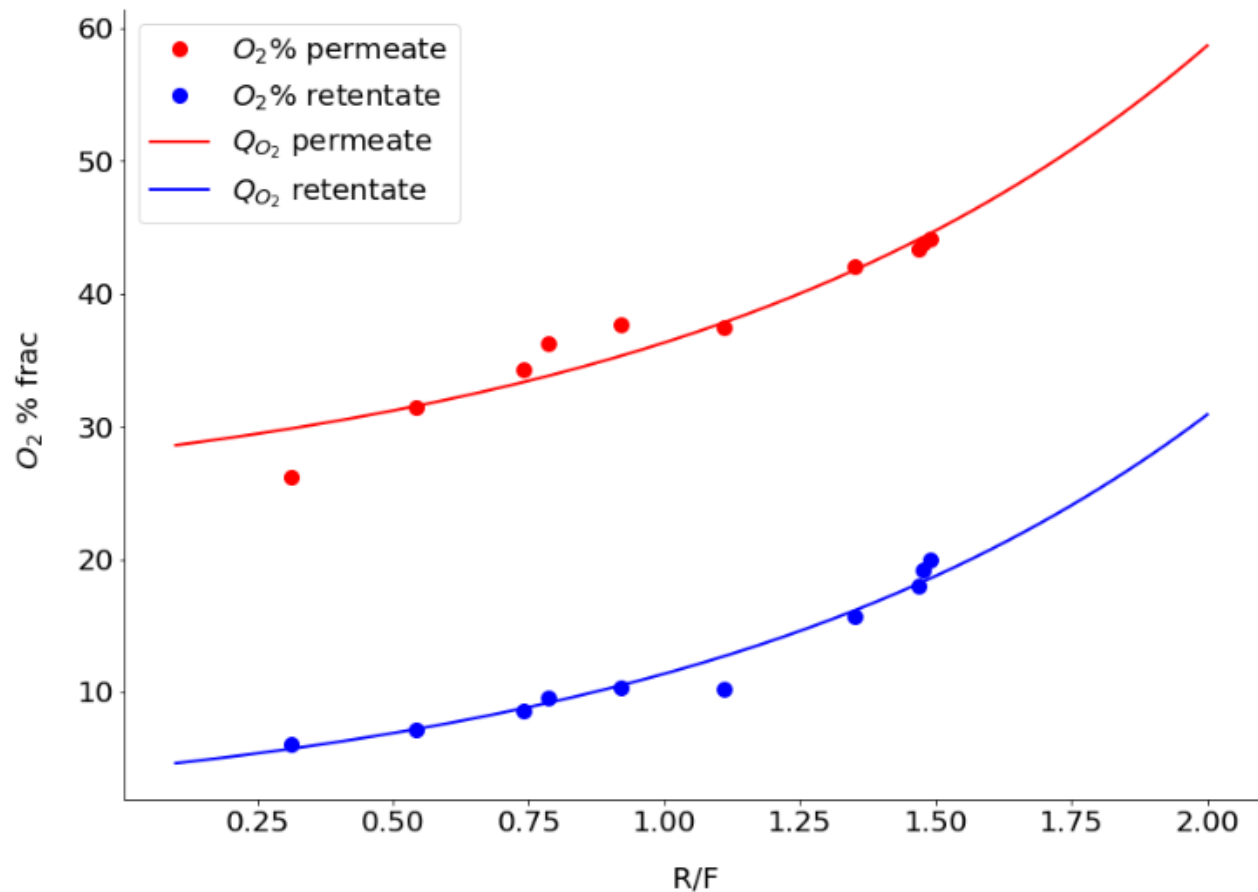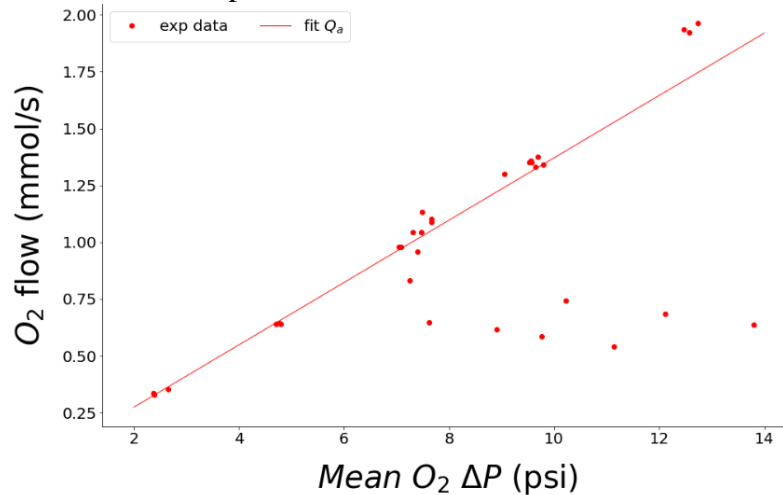
Figure 10. O_2 permeate flux flowrate (red points) and fitted permeability line (red line) plot against the mean pressure difference at the permeate stream. Data were fitted using linear equation $y = Q_b \Delta P$ with $Q_a = 0.1371$ mmol/psi s. Data were collected with various feed flowrate and at standard room temperature T = 298K.

Figure 11. O_2 permeate flux flowrate (red points) and fitted permeability line (red line) plot against the mean pressure difference at the permeate stream. Data were fitted using linear equation $y = Q_b \Delta P$ with $Q_b = 0.0313$ mmol/psi s. Data were collected with various feed flowrate and at standard room temperature T = 298K.

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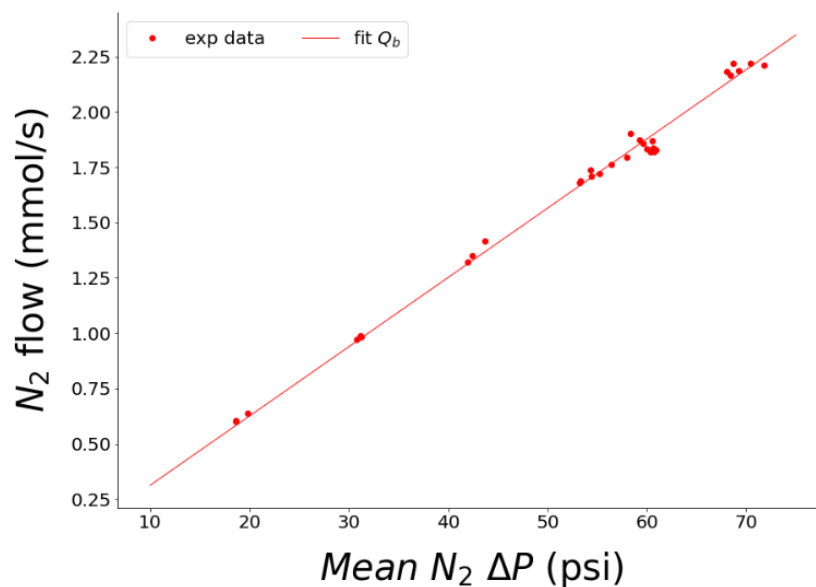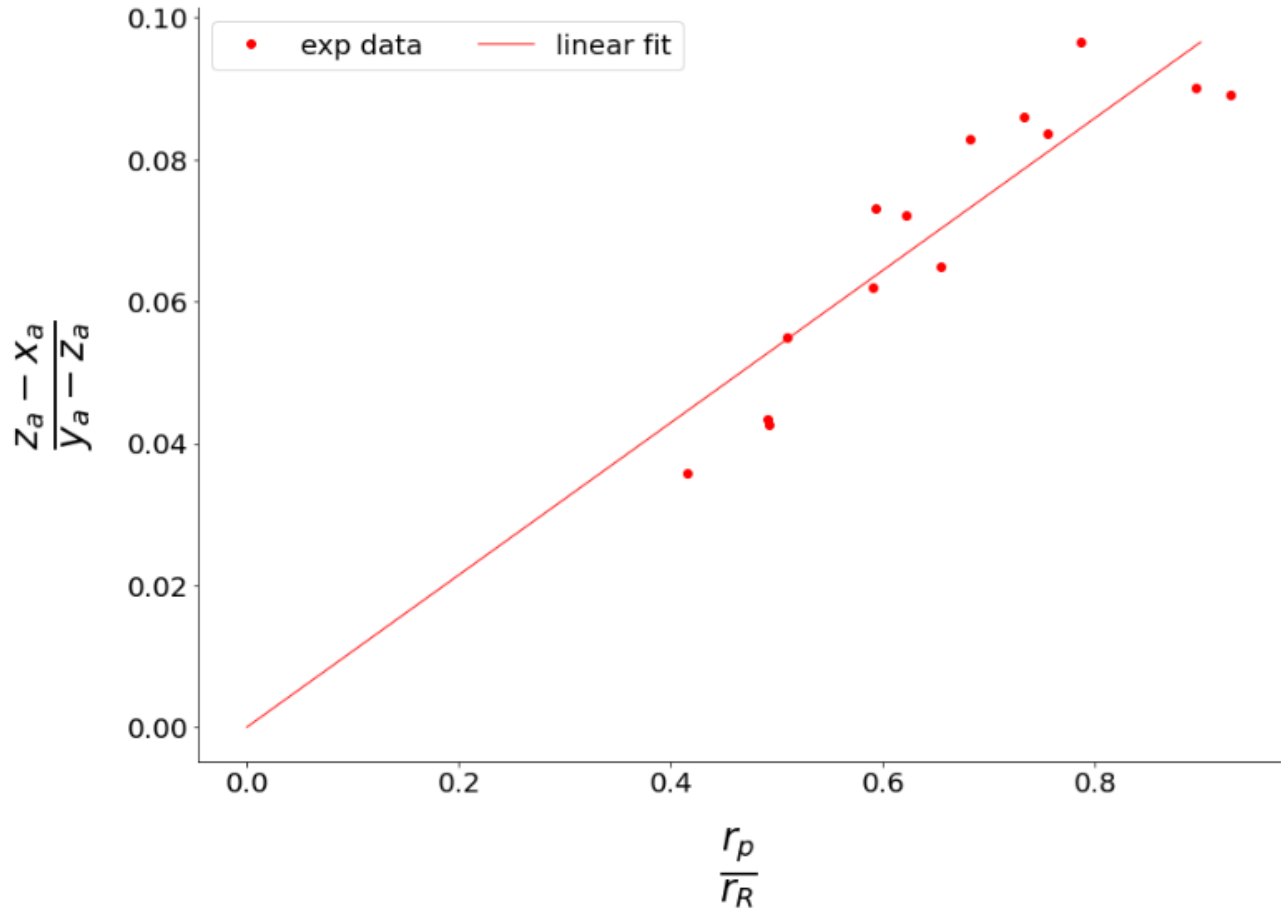
Figure 12. mass reconciliation for permeate rotameter reading by plotting $\frac{z_a - x_a}{y_a - z_a}$ against $\frac{r_p}{r_R}$ with 0 intercept. The slope of the line (red line), m, was determined at m = 0.89 when fitted with experimental data (red points). The slope is used for $C_p = m\ C_r$, with $C_r = 50$ sccm/mm to determine $C_p = 44.3$ sccm/mm.

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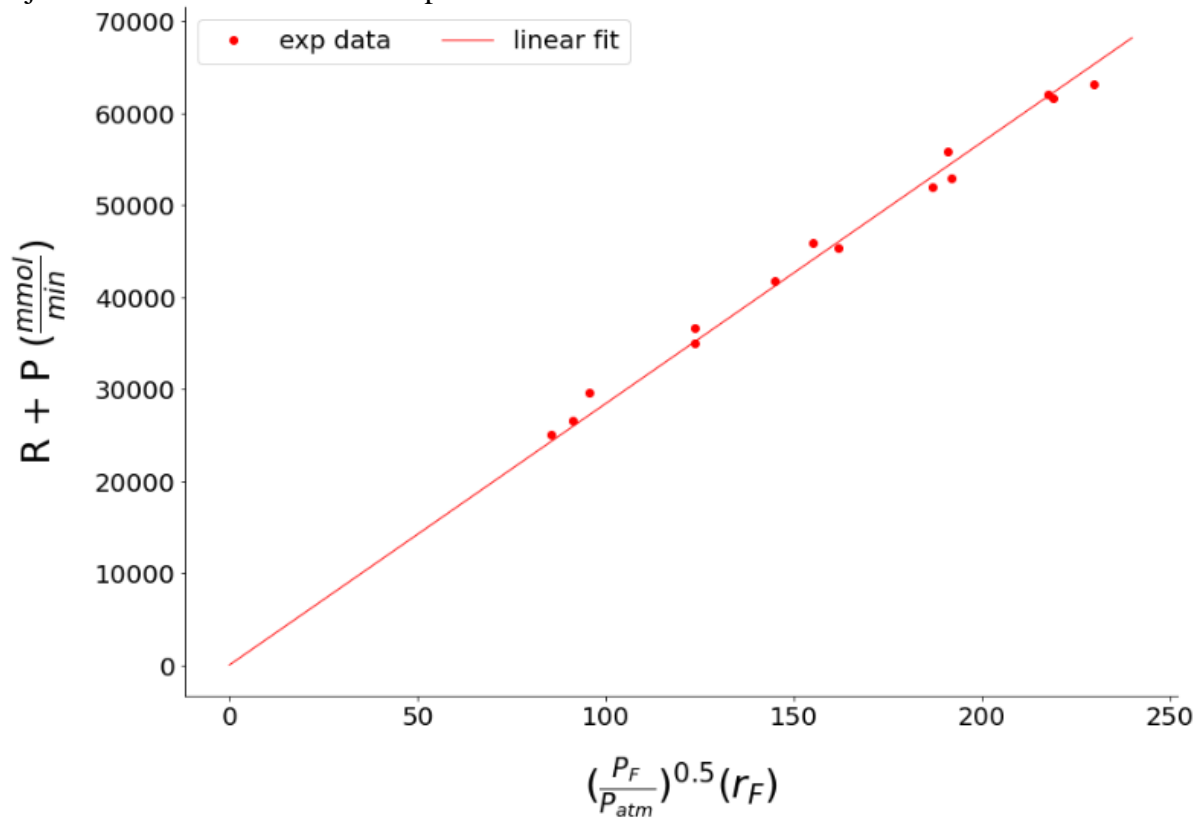
Figure 13. mass reconciliation for feed rotameter reading by fitting R + P against $\left(\frac{P_F}{P_{atm}}\right)^{0.5} (r_F)$ with 0 intercept. The slope of the line (red line), C_F, was determined at $C_F = 234$ sccm/mm when fitted with experimental data (red points).

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<u>Sample calculation:</u>

Calibrating C_F and C_p.

C_p : grab data from task 1 or others.

calculate : $\dfrac{z_a - x_a}{y_a - z_a} = \dfrac{R}{P} = \dfrac{C_p}{C_r}$. set $C_r = 50$ sccm $mmol/mm$

O_2 vol : $\dfrac{0.215 - \frac{8.5}{100}}{\frac{33}{100} - 0.215} = 0.9398 \, m_1 = \dfrac{C_p}{C_r}$ $\left[\text{for data 1 of task 1} \right]$

$\Rightarrow$ $0.93 = C_r = C_p$ $\dfrac{C_p}{C_r} = \dfrac{(\text{permeate reading})}{(\text{retentate reading})} = \dfrac{88 \text{ mm}}{89.5 \text{ mm}} = 0.983$

plot $\dfrac{z_a - x_a}{y_a - z_a} = 0.934$ against $\dfrac{C_p}{C_r} = 0.983$ with other points.

find m_1 with excel linest : $m_1 = 0.886$ with 0 intercept.

$\boxed{\text{get } C_p = 0.886 \cdot 50 = 44.3 \text{ sccm}/mm}$

Error :

assume both reading has $\pm \overset{2}{10}\%$ error accuracy from manufacture.

assume $\%$ O_2 reading has $\pm 0.2\%$ (0.002) error.

high error
$\dfrac{C_p + 0.1 C_r}{C_r - 0.1 C_r} = \dfrac{C_p (1 + 0.1)}{C_r (1 - 0.1)} = \dfrac{C_p}{C_r} \left(\dfrac{1.02}{0.98} \right)$

$\quad = 1.2014 \; 1.023$

low. error
$\dfrac{C_p}{C_r} \left(\dfrac{0.98}{1.02} \right) = \dfrac{0.944}{1.0047}$

high error
$\dfrac{z_a - (x_a - 0.002)}{(y_a + 0.002) - z_a} = 1.168$

low:
$\dfrac{z_a - (x_a + 0.002)}{(y_a + 0.002) - z_a} = 1.094$

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then,

m, high : $\dfrac{0.968}{0.829} \Rightarrow C_p = 56.883$ sccm/mbar.

low : $0.703 \Rightarrow C_p = 35.3$ sccm/mbar

$C_p = 44.3 + \dfrac{(56.9 - 44.3)}{(44.3 - 35.3)} = 44.3 \,{}^{+\,4.1}_{-\,2.3}$ sccm

avg error $\dfrac{12.6 + 9}{2} = 10.8$ $\dfrac{4.1 + 2.8}{2} = 3.55$

Calibrate C_F.

$\boxed{C_p = 44.3 \pm 3.55 \text{ sccm}}$

Sccm of retentate : $50 \cdot 89.5 = 4475 \,{}^{\pm\,89.5}$ sccm $= R$

permeate : $88 \cdot 44.3 = 3898$ sccm $\,{}^{+\,312.4}$ sccm $= P$

$R + P = 4475 + 3898 = 8373.8 \pm 401.9 \;[4.8\%]$ sccm

Feed P (psi) $= 69 + 14.7 = 83.7$ psi   and ± 1 psi on P_F:

error 20% on reading:

$\left(\dfrac{P_F}{P_{atm}}\right)^{\frac{1}{2}} r_F = \left(\dfrac{83.7}{14.7}\right)^{\frac{1}{2}} 14.5 = 34.6 \pm 0.692$

$\hookrightarrow \left(\dfrac{3.48 + 3.44}{2}\right) = 3.46$

plot $(R + P)$ vs $\left(\dfrac{P_F}{P_{atm}}\right)^{\frac{1}{2}} r_F$ with 0 intercept.

$C_F = 233.97 = 234$ sccm/mbar.

high $C_F = 254$ sccm/mbar

low $C_F = 213$ sccm/mbar   214.4

avg error $\left(\dfrac{(254 - 234) + (234 - 213)}{2}\right) \approx 20.5$

$\boxed{C_F = 234 \pm 20.5 \text{ sccm/mbar}}$

(8.77%)

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Calculating mol flux from sccm → mmol/s.

Feed flow actual: (data 2 on tash 1)

$$\dot{V}_F = C_F \cdot r_F \cdot \left(\frac{P_F}{P_{atm}}\right)^{\frac{1}{2}} = 12 \cdot 14.5 \cdot 234 \cdot \left(\frac{83.2}{14.7}\right)^{\frac{1}{2}}$$

$$\pm 762.5 \quad \boxed{9.4\%}$$

$$= 8095.5 \text{ sccm}$$

total:

$$J_{Feed} = \frac{\dot{V}(cm^3/s)}{22.4 \left[\frac{mol}{dm^3}\right]} \cdot \frac{1 \, dm^3}{1000 \, cm^3} \cdot \frac{1000 \, mmol}{mol} \cdot \left(\frac{1 \, min}{60 s}\right) = \left[\frac{mmol}{s}\right]$$

standard air den
air molar density
air room temp

$$= \boxed{\frac{8095.5}{22.4} \cdot \frac{1}{1000} \cdot 1000 \cdot \frac{1}{60} = 6.02 \text{ mmol/s}} \quad \pm \frac{0.566}{\cancel{0.704}} \quad 0.566$$

$$J_{per} = 6.02 \cdot$$

$$\dot{V}_p = C_p r_p = 88 \cdot 44.30 = 3.899 \text{ mmol/s} \quad \text{sccm}$$

$$\boxed{J_{per} = \frac{38.99}{22.4} \cdot \frac{1}{60} = 2.9 \text{ mmol/s}} \quad \pm \cancel{0.25} \quad 0.058 \, (2\%)$$

$$89.5 \, (2\%)$$

$$\dot{V}_r = C_v r_v = 86 \cdot 50 = \cancel{3500} \text{ mmol/s} \quad \pm \cancel{330} \quad \begin{array}{c} 0.566 \\ 0.78 \end{array}$$

$$89.5 \qquad 4475 \text{ sccm}$$

$$\pm 0.066$$

$$\boxed{J_{ret} = 3.33 \text{ mmol/s}}$$

$$\%\, err: \quad \frac{0.02 \cdot 100}{6.02} \cancel{} \quad 3.49\%$$

$$\frac{0.02}{6.02} \cdot 100$$

$$9.4 \qquad 0.21$$

$$\begin{array}{c} 3.33 \\ \end{array} \begin{array}{c} 0.21 \\ 0.78 \end{array}$$

Mass balance: $6.02 - (2.9 + 3.33) \approx -0.02 \text{ mmol/s}$

close to 0. and within error bound. $\left[\frac{0.21}{6.02} \cdot 100\right] = 3.48\%$

Mass conserved for total. and most other data.

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Calc J flux.

O_2 flux:

$$J_{O_2,F} = 0.215 \cdot 6.02 = 1.295 \text{ mmol/s}$$

$$J_{O_2,P} = 2.9 \text{ mol} \cdot (O_2 \% \text{ reading at perm}) = 2.9 \cdot (0.348) = 1.009 \text{ mmol/s}$$

$$J_{O_2,V} = \frac{2.9 \text{ m}}{3.33} \left(\underline{\quad\quad} \text{ ret} \right) = 3.33 \cdot (0.09) = 0.2996$$

$$\approx 0.30 \text{ mmol/s}$$

N_2 flux:

$$J_{N_2,F} = 0.715 \cdot 6.02 = 4.73 \text{ mmol/s}$$

$$J_{N_2,P} = 2.9(1 - 0.348) = 1.89 \text{ mmol/s}$$

$$J_{N_2,V} = 3.33(1 - 0.09) = 3.03 \text{ mmol/s} \qquad \frac{0.014}{1.295} \cdot 100 = 1.08\%$$

M.B of O_2: $1.295 - (1.009 + 0.3) = -0.014$. conserved account for err.

N_2 : $4.73 - (1.89 + 3.03) = -0.19$ conserved account for err.

$$\frac{0.19}{4.73} \cdot 100 = 4.017\%$$

All M.B are less than 5% error, well conserved, considering rotameter reading has 5% - 10% error.

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recover. of O_2 and N_2: | Perm | retentate |

% O_2%:
$$\frac{J_{per, O_2}}{J_{Feed, O_2}} = \frac{\frac{1.0095}{2\!\!\!/} \cdot 100}{\frac{6.02}{1.295}} \pm a = 78 \%$$

N_2%:
$$\frac{J_{ret, N_2}}{J_{Feed, N_2}} = \frac{\frac{3.0299}{4.728}}{} \cdot 100 = 64\%$$

done for other data and plot on graph:

mean O_2 ΔP (psi): (for ~~standard points~~):
ex data point.

Avg $\Delta P = \frac{\left(P_{feed} \cdot z_{O_2} - P_{per} \cdot x_{O_2}\right)}{2} + \frac{\left(P_{ret} \cdot x_{O_2} - P_{per} \cdot y_{O_2}\right)}{2}$

$$= \frac{\left(\frac{(22+14.7)}{2}{} \cdot 0.215 - 14.7 \cdot 0.353\right)}{2} + \frac{\left(34.7 \cdot 0.209 - 14.7 \cdot 0.353\right)}{2}$$

$$= \frac{2.37}{18.618} \text{ psi}$$

mean N_2 ΔP (psi): (same data):

Avg $\Delta P = \left[\frac{\left(36.7 \cdot 0.785 - 14.7 \cdot (1-0.353)\right)}{2}\right] + \left[\frac{\left(34.7 \cdot (1-0.209) - 14.7(1-0.353)\right)}{2}\right]$

$$= 18.63 \text{ psi}$$