TO: SEPARATION DESIGN GROUP

FROM: Dr. McBrain, Director of R and D

G-E Chemical Co.

SUBJECT: Use of PRISM Separator to Produce Nitrogen and Oxygen Rich Streams

Presently, our company is using a three stage membrane separation process to produce an oxygen-rich stream (>40% O2) and a nitrogen-rich stream (<5% O2) from air using three membrane separators as shown below. The existing system, shown in Fig 1 below, has often had difficulties producing a sufficiently pure nitrogen-rich stream. In addition, due to increased demand within the company, we would like to increase the production of both product streams. A fourth membrane module, of 2000 m2 area, has been made available for this effort. Note that the compressor for this process was conservatively designed, and is capable of producing a maximum of 17,500 slpm at 140 psia.

I would like your group to:

1. Determine the permeances for oxygen and nitrogen and the O2/N2 permeability ratio for the laboratory membrane module.
2. Determine experimentally the effects of the stage cut and retentate pressure on the permeate and retentate concentrations and the permeate flow rate. Over what conditions is the perfect mixing model valid?
3. Model the effect of fiber diameter on the permeate pressure along the length of the fiber. Check the assumption that the bore-side (permeate) pressure drop is negligible to confirm that perfect mixing is a valid assumption.
4. Design a new separation train using the four available modules to meet the oxygen and nitrogen requirements using the perfect mixing model. Optimize the process to yield the maximum production of the oxygen-rich and nitrogen-rich streams given the current compressor’s capabilities. How should the four modules be arranged, and at what pressure and stage cuts should they be operated?

1st stage

4000 m2

2nd stage

4000 m2

3rd stage

4000 m2

Air

14285slpm @ 125 psia

3140 slpm at 42-44% O2

6000 slpm @ 5-6 % O2

Vent to air

Vent to air

Figure 1 Existing Gas Membrane Process. Permeate streams are shown coming off the top, and retentate streams off the bottom of each unit. The process as shown often does not meet the <5% O2 specification for the final retentate stream. For the O2-rich permeate product, there is no benefit to producing greater than 40% O2.

**Operation of the Gas Separation System**

The pilot plant has a PRISM separator consisting of four columns, two co-current and two counter-current. The columns can be operated individually, or in groups of two or four. Using all four columns together is suggested for the bulk of your experiments. If time permits, investigate co- vs counter- current flow patterns. Gas cylinders of oxygen, nitrogen, and air will be used as feed streams. The oxygen concentration of the permeate and retentate streams are measured using oxygen analyzers. The permeate and retentate flow rates are measured using rotameters. Two pressure gauges are located on the apparatus (feed and retentate pressure) and a needle valve is included to control the retentate flow rate. Details of the operation of the unit should be obtained from the TA.

**Calibration**

Calibration curves will be provided for the rotameters. Make sure you make a copy and return the originals to the lab. The oxygen analyzers should be calibrated according to the following procedure:

1. Set the appropriate valves to bypass the columns and send gas directly to the oxygen sensors.
2. The meters should read 20.9% within 5 minutes after flushing the system with air.
3. Flush with oxygen. If readings within 5 minutes do not stabilize at 100± 2%, adjust the sensor to 100% and repeat step 1.
4. ***Do not calibrate using pure N2 – it’s not necessary and it puts a lot of pure N2 into the air.***

**Background Data**

Minimum Product Requirements:

Oxygen rich stream: 40% Oxygen at 3150 slpm

Nitrogen rich stream: 95% Nitrogen/5% Oxygen at 6000 slpm

Note: there is no benefit to producing streams of greater purity than required.

Column Dimensions:

Active Length (*lf*): 1.45 m

Pipe (Column) Diameter (*Dp*): 2.43 cm

Fiber Dimensions:

OD (*Do*): 495 μm

ID (*Di*): 245 μm

Packing Efficiency (*η*) 0.50

Experimental Operating Limits:

Feed Pressure (*P*): 100 – 140 psig (for operation with air)

Stage Cut (*θ*): 0.10 – 0.5

**Surface Area of Fibers and Permeability Factors**

The number of fibers in a PRISM bundle is a function of fiber outer diameter, fiber

packing efficiency, and the column inner diameter as shown below.

 (1)



Figure 2. Fiber packing arrangement in the PRISM Separator

Equation 1 is based on the fibers being in a hexagonal closed packed arrangement as shown in Figure 2. The fibers generally do not touch in which case the unit cell is larger and the packing less efficient, i.e., *η*<1. The total number of fibers in the system is given by:



(2)

Where *N*mod is the number of modules used in the system (usually four). The total membrane area is calculated using the following equation:



(3)

For a single component gas, then the gas transport equation can be written as:

(4)

where *A* is the area (calculated using Equations 1-3), *P* is the retentate pressure, *p* is the permeate pressure, *Vp* is the permeate flow rate, Q is the permeability coefficient and δ is the effective thickness of the membrane. This is one expression of Darcy’s law for flow through porous media. Note that δ is not the membrane thickness, (Do-Di), but rather the distance over which the separation actually occurs. In most membrane systems, this is a thin skin whose thickness can be difficult to determine. As a result, the permeability coefficient, Q, is generally not determined, and instead, the permeance, (Q/δ), is calculated. Q/δ can be determined from Eq 4 using a pure gas if the bore-side pressure drop along the fiber is negligible (i.e., if *ΔP* across the membrane, for the entire fiber length, is constant).

**The Relationship between Permeate Flow and Pressure**

The downstream pressure on the bore-side of the fiber is dependent on the flow rate of gas in the bore(the permeate). As stated in the previous section, the permeance can be calculated easily if the bore-side pressure drop is negligible. This property must be demonstrated to prove that the permeance values obtained are valid.

The bore-side pressure can be determined by numerical integration. The differential form of the flux expression (Equation 4) gives the change in permeate molar flow rate, *dV*, with respect to an incremental fiber length*:*

** (5)

where *dl* is the incremental fiber length. In this calculation, the feed pressure (*P*) is assumed constant while the permeate pressure (*p*) will vary over the length of the fiber. If the viscosity, *μ*, is independent of pressure, then the Hagen-Poiseuille equation governs the pressure drop per unit length in the bore of the fiber. Therefore, to a good approximation, one can write

 (6)

where the area-average gas velocity, *u*, is calculated using:



(7)

*. NOTE: The velocity is calculated by dividing the volumetric flow rate by the total cross sectional area the gas flows through. You have V as a flow rate in liters/minute – SO WATCH YOUR UNITS.*

You need to numerically integrate equations (5) and (6) to find *p*. Then you can calculate the permeate pressure of a module along the length of a fiber as Di is changed. Vary the inner diameter of the fiber between the limits of 40 and 400 *μm*. The fiber outer diameter can be calculated using:

 (8)

Where the units of *Di* and *Do* in the equation are *μm*. The permeance is independent of the fiber diameter. Keep in mind that the purpose of the calculation is to determine the conditions under which *P>>p* and the variation in permeate pressure (*p*) is negligible.

**Safety Considerations**

While the substances used in this experiment do not normally strike one as dangerous, several precautions must be taken. First of all, you are dealing with high pressure cylinders of gas which can do considerable damage if the stem or regulators are damaged. Therefore all cylinders must always be secured so they cannot fall and be damaged. All around you in the laboratory are experiments that may contain hazardous chemicals. Therefore, you must wear your lab coat and safety glasses. Your foreman’s report should also consider the hazards associated with pure oxygen and nitrogen and the physical lay-out of the apparatus.

**Foreman’s Report**

In the Foreman’s report, you must outline all the experimental runs required in order to calculate the pure gas permeances, and for air separation, to determine the effects of the retentate pressure and stage cut. For a given retentate pressure, the permeate flow rate is essentially constant. This means that you must change the feed gas flow rate to control the stage cut. Supply an Excel spreadsheet for the different studies in your Foreman’s report. These data tables must include all parameters you expect to measure in that section of the experiment and any formulas for values you may need to calculate.

**Final Comments**

In designing your process, flow rates and the composition of the exit streams can be specified using the perfect mixing model if the following parameters are supplied:

* 1. Membrane Area
  2. Mole Fraction of *O*2 in Feed: *z*
  3. Permeances: (*QO2 / δ*) and (*QN2 / δ*)
  4. Feed-Retentate Pressure: *P*
  5. Permeate Pressure (Bore-side Pressure): *p*
  6. Feed Flow Rate, F

In ECHE 361, the general procedure for handling membrane separation problems was that the stage cut was given, and a graphical technique was used to solve the RT and mass balance equations for xr and yp, the composition of the permeate and retentate. The stage cut and feed flow rate were then used to find Lr and Vp, the retentate and permeate flow rates. Finally, the membrane area was found using the flux equation and the known values of yp and Vp.

However, in your design calculations, the membrane area is fixed. For a given feed flow rate, there is only one possible set of xr, yp, Lr and Vp values that will satisfy the RT equation, the mass balance equations, and the flux equation. In other words, the stage cut is not a variable when the membrane area and feed flow rate are specified in the design. Write out the four equations and look at what variables appear in each. There is a relatively simply iterative procedure that will allow you to solve for the four unknowns.

**Units**

Please use the following units for this laboratory:

Gas flow rates: slpm (standard liters / minute)

Pressure: Psia

O2, N2 concentration mol fraction or mol%

O2, N2 permeance slpm/psia m2

*Note: the rotameters in the lab are calibrated for liters/min at P = 1 atm and T = 70F. Standard conditions in SI systems are generally taken as P = 1 atm and T = 273K, equivalent to 22.4 liters at STP = 1 mole. In historical engineering practice, the ‘standard’ condition has been defined in many different ways, (T = 32F, 60F, 68F, or 70F, P = 101,325 Pa or 100,000 Pa, etc). For the purposes of this lab, we will ignore the difference and assume that the rotameter readings in liters/min are equivalent to standard liters/min.*