

**CO<sub>2</sub>-selective, Hybrid Membranes by Silation of Alumina****David R. Luebke\* and Henry W. Pennline\*\*****United States Department of Energy  
National Energy Technology Laboratory  
P.O. Box 10940****Pittsburgh, PA 15236-0940****Fax: (412) 386-6004****\* Phone: (412) 386-4118****\*\* Phone: (412) 386-6013****ABSTRACT**

Hybrid membranes are feasible candidates for the separation of CO<sub>2</sub> from gas produced in coal-based power generation since they have the potential to combine the high selectivity of polymer membranes and the high permeability of inorganic membranes. An interesting method for producing hybrid membranes is the silation of an inorganic membrane. In this method, trichloro- or alkoxy-silanes interact with hydroxyl groups on the surface of  $\gamma$ -AlO<sub>3</sub> or TiO<sub>2</sub>, binding organic groups to that surface. By varying the length of these organic groups on the organosilane, it should be possible to tailor the effective pore size of the membrane. Similarly, the addition of “CO<sub>2</sub>-phillic” groups to the silating agent allows for the careful control of surface affinity and the enhancement of surface diffusion mechanisms. This method of producing hybrid membranes selective to CO<sub>2</sub> was first attempted by Hyun [1] who siled TiO<sub>2</sub> with phenyltriethoxysilane. Later, Way [2] siled  $\gamma$ -AlO<sub>3</sub> with octadecyltrichlorosilane. Both researchers were successful in producing membranes with improved selectivity toward CO<sub>2</sub>, but permeability was not maintained at a commercially applicable level. XPS data indicated that the silating agent did not penetrate into the membrane pores and separation actually occurred in a thin “polymer-like” surface layer. The present study attempts to overcome the mass transfer problems associated with this technique by producing the desired monolayer coverage of silane, and thus develop a highly-permeable CO<sub>2</sub>-selective hybrid membrane.

## INTRODUCTION

Many believe that the Integrated Gasification Combined Cycle (IGCC) process will one day account for the majority of the energy production in the United States [3-4]. The process, which includes gasification of coal to produce CO and H<sub>2</sub>, pollutant abatement, and combustion of the synthesis gas to produce electricity, offers great potential for the clean, efficient production of energy from coal. Substantial efforts are currently underway to develop the necessary technologies for removal of pollutants, such as sulfur, from the process gas [5]. The opportunity exists to concurrently develop a means of removing CO<sub>2</sub> that readily integrates within IGCC and in doing so, lay the groundwork for energy production from coal that is not only clean and efficient but addresses greenhouse issues as well.

Membranes, though well established in liquid filtration applications, are only beginning to come into use in more difficult gas separations. Simplicity, flexibility, and the potential to perform separations at lower energy penalties than other methods make them interesting for CO<sub>2</sub> removal from IGCC process gas streams [6-8].

In addition to the standard requirement of obtaining high permeability, two primary challenges exist in the development of membranes capable of selectively separating CO<sub>2</sub> from the process gas stream: reverse selectivity and hydrothermal stability. Most membranes pass smaller, lighter molecules and retain heavier ones. Membranes which selectively pass heavier molecules, such as CO<sub>2</sub>, at the expense of lighter ones, like H<sub>2</sub>, exhibit what is known as reverse selectivity. To achieve reverse selectivity, it is necessary to produce a membrane which relies on surface or bulk

diffusion as its primary transport mechanism. Polymer membranes rely on bulk diffusion, but producing a useful membrane in which surface diffusion is the primary transport mechanism has been more difficult [2,9].

The problem of hydrothermal stability is complicated by the unusual conditions under which the membrane is expected to operate. The likely minimum useful temperature for these membranes would be near 150°C with the preferred temperature near 300°C. Pressures in the process gas stream are expected to be approximately 3.6 MPa with transmembrane pressures possibly reaching 1.5 MPa [10]. In addition, reducing conditions of the gas stream and the presence of water and various minor contaminants necessitate the design of membranes with exceptional chemical and physical stability.

One possible means of achieving stability, permeability, and selectivity is the modification of an inorganic substrate, such as the innately stable and permeable alumina with organic groups to increase selectivity. The grafting of organosilanes to the inorganic surface is a well-studied method. The grafting is accomplished when halogen atoms on the silane molecule interact with hydroxyl groups on the inorganic surface eliminating HCl and forming a covalent attachment. As many as three such interactions can occur per silane molecule anchoring it to the surface. Since the silane molecule can also contain nearly any organic group, this method provides an extremely flexible tool for surface modification [11].

The procedures required to produce a uniform monolayer of silane on a porous alumina membrane surface and the effects of pretreatment and silation conditions, on structure and stability of the resulting monolayer are examined in this study.

## EXPERIMENTAL

### Fabrication

Membranes were prepared by modifying a 2 to 5  $\mu\text{m}$  layer of  $\gamma$ -alumina supported on the inner surface of 2.5 cm cylindrical segments of 1 cm diameter  $\alpha$ -alumina tubes purchased from Pall Corporation. The nominal pore diameter of the active  $\gamma$ -alumina layer was 5 nm. As a surrogate for these membranes for use in FTIR and XPS studies, Whatman Anodiscs with diameter 13 mm and nominal pore size of 20 nm were used.

The initial procedure followed the technology of McCarley and Way in their silation study of  $\gamma$ -alumina with octadecyltrichlorosilane [2]. The tubes or discs were first subjected to heating in air at a rate of 10 K/min to 500°C and were maintained there for 18h to remove organic impurities associated with handling. The cooled tubes were placed in a 500 ml round-bottomed flask with approximately 75 ml of 30 percent  $\text{H}_2\text{O}_2$  (Fisher, 31.1%) and refluxed for 30 min. Afterward, the tubes were rinsed in deionized water and returned to the heating apparatus to reflux in 75 ml of deionized water for 30 min. These steps were designed to fully hydroxylate the alumina surface before silation. After completion of this procedure, the tubes and discs were dried at 80°C for 1h in air.

Silation was initially conducted by placing 120 ml of anhydrous toluene (Aldrich, 99.8%) in a 250 ml Erlenmeyer flask. A mixture of 5 ml of n-butyltrichlorosilane (99%) and 10 ml of toluene was added. The graduated cylinder was then rinsed with an additional 10 ml of toluene and this too was placed in the flask. A magnetic stirrer was added, and mixing was begun at a slow speed. After a few seconds, the tube or disc was added and a stopper inserted to seal the flask. The reaction was allowed to continue for

6h. When it was complete, the tube or disc was removed, rinsed with 1 liter of toluene, and allowed to soak in 400 ml of toluene for 24 h. The silation procedure was conducted entirely in a glove box under dry nitrogen. The silated tubes and discs were then dried in a lab oven at 50°C in ambient air for 24h.

In order to optimize the pretreatment procedure, various steps were altered and measurements taken to determine their effect on silation. Membranes and models were produced which had been heated initially to 750°C rather than 500°C. Hydroxylation was eliminated entirely, conducted for 1h in deionized water rather than H<sub>2</sub>O<sub>2</sub>, and conducted for 2 h in H<sub>2</sub>O<sub>2</sub>. Post-hydroxylation and post-silation drying temperatures were each altered to 100 and 150°C. The silation was also altered by refluxing the reaction solution under vacuum or beginning the reaction in 0°C toluene.

## **FTIR**

A Thermo-Nicolet Nexus 670 Fourier Transform Infrared (FTIR) spectrometer was used to conduct transmission analysis of Anodisc samples after various pretreatments in order to confirm the presence of silanes in the samples. Samples were examined at room temperature under ambient pressures of N<sub>2</sub> using a liquid N<sub>2</sub> cooled MCT detector. The Anodiscs were affixed to index cards after pretreatment and held in the beam at an angle approximately 30 degrees from the perpendicular to limit fringence effects. Samples were placed in the chamber under dry N<sub>2</sub> and allowed to equilibrate for one hour. Two hundred scans were taken for each sample at a resolution of 4 cm<sup>-1</sup>.

## **XPS**

X-ray photoelectric spectroscopy (XPS) measurements were performed on a PHI 5600ci spectrometer with a monochromatic Al X-ray source. Anodisc samples were

examined with an analyzer pass energy of 58.7 eV. Sputter profiling to a depth of 1000 Å was performed using 3 kV Argon ions at a sputter rate of approximately 235 Å/min.

### **Performance Testing**

The cylindrical membrane was mounted in 0.5-in OD stainless steel tubing to allow mounting into the HMT-2 unit, Figure 1. Gas analysis of the retentate and permeate streams was conducted with a HP 5890 Series II Gas Chromatograph equipped with HaySep D columns for gas separation and thermal conductivity detectors (TCD) for gas detection at 15-minute intervals. The membrane was tested at constant retentate and permeate flow rates for each gas combination of interest while the pressure of the retentate was varied by computer controlled pressure valves. Thus the pressure of the retentate was varied in 2 psi increments from approximately 21psi to the maximum in an effort to develop a data set that would allow an understanding of the pressure-flux relationship.

## **RESULTS AND DISCUSSION**

### **Fabrication**

Since the goal of the study was to develop an optimal procedure for the fabrication, characterization, and testing of membranes prepared by modification of tubular alumina composite membranes, careful observation regarding procedures began from the outset. Studies involving similar tubular composite membranes have generally used low-fire ceramic glazes to aid in attachment of the ceramic tube to stainless steel performance testing systems [2, 12-14]. Most such glazes require firing at a minimum of 1000°C to achieve the desired glassy surface. In early testing, a similar procedure was used. It was noted that the active  $\gamma$ -alumina layer became discolored when the tubes were

fired at 1000°C for 4 hours. Further testing using the sapphire Anodisc surrogates revealed that these thin wafers curled and lost their shape at temperatures as low as 750°C. It is believed that both these behaviors represent major changes in the active membrane layer. Procedures were tailored to avoid temperatures which might cause such morphological changes, and the ceramic glaze was eliminated from the fabrication procedure.

## **FTIR**

A comparison of the transmission FTIR spectra of Anodiscs after various pretreatments helps to elucidate the degree of silation achieved by allowing the sample disc to be examined through its entire cross-section rather than simply on the surface. All spectra showed stretches between 2800 and 3000  $\text{cm}^{-1}$  and between 1300 and 1700  $\text{cm}^{-1}$  as well as a single peak at approximately 2325  $\text{cm}^{-1}$ . Other researchers have assigned the 2800-3000  $\text{cm}^{-1}$  stretches to the hindered motion of bound alkylsilanes coupled to the alumina surface [2,12,15].

Figure 2 shows 4 spectra representing a disc prepared by the standard technique alongside discs in which one of the three main pretreatment parameters was varied. Table 1 shows the full pretreatment conditions of each disc. Minor differences in absorbance were observed with the unfired sample giving the strongest signal followed by the standard, the water hydroxylated sample, and the sample dried at 150°C after silation. The lower absorbance of the fired samples suggest that firing removes a portion of the surface hydroxyl groups intended to act as silane attachment sites in such a way that they cannot be replaced during the hydroxylation step. From the lower absorbance of the water hydroxylated sample over those hydroxylated in  $\text{H}_2\text{O}_2$ , it can be concluded

as expected that water is a less efficient hydroxylation agent than  $\text{H}_2\text{O}_2$ . The low signal obtained from the sample dried at elevated temperature indicates that some silane is removed from the surface, probably via oxidation, during high-temperature drying. This result has both negative and positive implications for the stability of the silated membranes. While it is reasonable to view the removal of a portion of the silane as a setback, it is fortuitous that a large portion of the silane was stable at elevated temperatures in an oxidizing environment, conditions under which the membrane was never designed to operate. Work is currently underway to further examine the stability of the membranes in a variety of conditions.

Figure 3 compares spectra obtained for discs prepared with the standard pretreatment followed by three different silation techniques. The standard sample was silated at room temperature under atmospheric pressure while the vacuum silation sample was silated in refluxing toluene at a pressure of 400 Torr at room temperature. In the cold silation sample, the reaction began at  $0^\circ\text{C}$  under atmospheric pressure and the mixture was allowed to slowly rise to room temperature as the reaction progressed. Some increase in absorbance was noted when the sample was silated under vacuum. When the reaction was begun at the lower temperature, however, the intensity of the silane absorbances was increased by approximately an order of magnitude. For the explanation of this result, it is necessary to examine the samples using a technique like XPS which allows for examination of the surface and near surface region of the membrane.

## **XPS**

The XPS technique has the ability to elucidate the elemental composition of the membrane surface as well as within the first 1000 Å below it through sputter profiling.



Table 2 shows the relative amount of Si at the surface of discs after vacuum silation, silation at reduced temperature, and after vacuum silation followed by drying at 150°C. While the difference between the atomic concentration of Si observed in the vacuum silated sample and the 150°C dried sample is comparable to the difference in FTIR peak areas for similar samples, the minor difference in atomic concentration for the vacuum and cold silated samples is not large enough to account for the differences observed with the FTIR in that case.

In order to understand the difference in FTIR signal for the cold and vacuum silated discs, it is necessary to examine the amount of silane which penetrates the disc's pores. By sputtering away the surface of the disc and performing XPS analyses every 200 Å, a profile was developed for the silane distribution over the first 1000 Å of the disc, see Figure 4. The Si fraction drops almost immediately to zero in the vacuum silated sample while considerable penetration is achieved in the cold silated sample. One possible explanation of this observation is that at room temperature the silane undergoes a polymerization reaction which is catalyzed by the alumina surface resulting in a layer of silane on the surface that blocks the pores and prevents silation of the internal pore surface area. Reducing the temperature would slow the kinetics of the polymerization reaction, but would do little to limit the much more favorable reaction with surface hydroxyl groups [16]. It is then reasonable to suggest that an optimized cold silation procedure would be capable of producing a silane monolayer over the entire surface area of the membrane.

## **Performance**

The performance studies conducted with the cylindrical membranes were at an elementary level and designed primarily to test the apparatus and procedure as well as to confirm that pretreatment introduced no defects into the membrane layer. Figure 5 shows single gas permeance for H<sub>2</sub>, N<sub>2</sub>, and CO<sub>2</sub> as a function of transmembrane pressure. For Knudsen diffusion, expected since the mean free path of the gases ranges from 35 to 130 nm or 7 to 26 times the pore diameter [2], the plot should be linear with an insignificant slope [17]. The observed data show the expected linearity with scatter due to experimental deviation. Slopes are approximately two orders of magnitude below the observed permeances, indicating that error in the data is of that order as well. The lack of a more substantial slope indicates that bulk flow as a result of membrane defects is not occurring.

The observed permeances are two orders of magnitude less than those reported by McCarley and Way for a similar membrane [2]. The most likely explanation is the use of Ar as a sweep gas in these experiments. Atoms of Ar are large enough to interfere destructively with permeance of the test gases. Counter-diffusion of the Ar sweep was detected in the experiments. Future experiments will use a lighter sweep gas less able to interfere with the desired gas diffusion.

## **CONCLUSION**

The study was designed to lay the foundation for more ambitious work aimed at developing membranes capable of selective CO<sub>2</sub> removal in reducing environments, such as those in the process gas of the IGCC process. A pretreatment has been developed which includes no initial firing of the membrane, hydroxylation using H<sub>2</sub>O<sub>2</sub>, and post

silation drying at 50°C. It has been discovered that silation beginning at 0°C allows pore penetration and development of a silane monolayer capable of significantly affecting membrane performance. Procedures have been developed for testing membranes produced by silation. Future testing in the membrane test system will include various combinations of gases as well as tests with membranes with CO<sub>2</sub>-selective functional groups. These accomplishments place the researchers in an excellent position to move forward toward a significant contribution in the membrane community and a considerable advance in CO<sub>2</sub> separation technology.

### **ACKNOWLEDGEMENTS**

The authors wish to thank John Baltrus for instruction and collaboration in our XPS studies, Angela Goodman and Lisa Campus for instruction and collaboration in our FTIR studies, Bryan Morreale, Mike Ciocco, and the Parsens HMT crew for excellent work with the performance studies, and the Oak Ridge Institute for Science and Education (ORISE) for its support of the study.

### **DISCLAIMER**

Reference in this report to any specific commercial process, product or service is to facilitate understanding and does not necessarily imply its endorsement or favoring by the United States Department of Energy.

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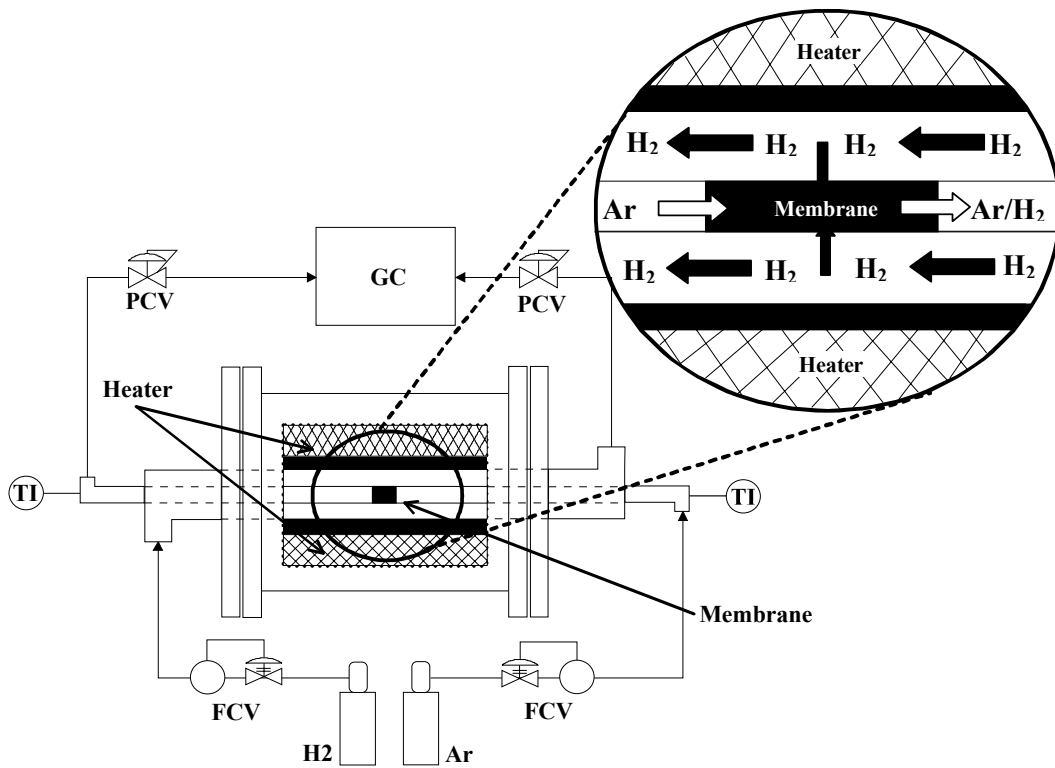


Figure 1. Flow schematic for performance testing system [18].

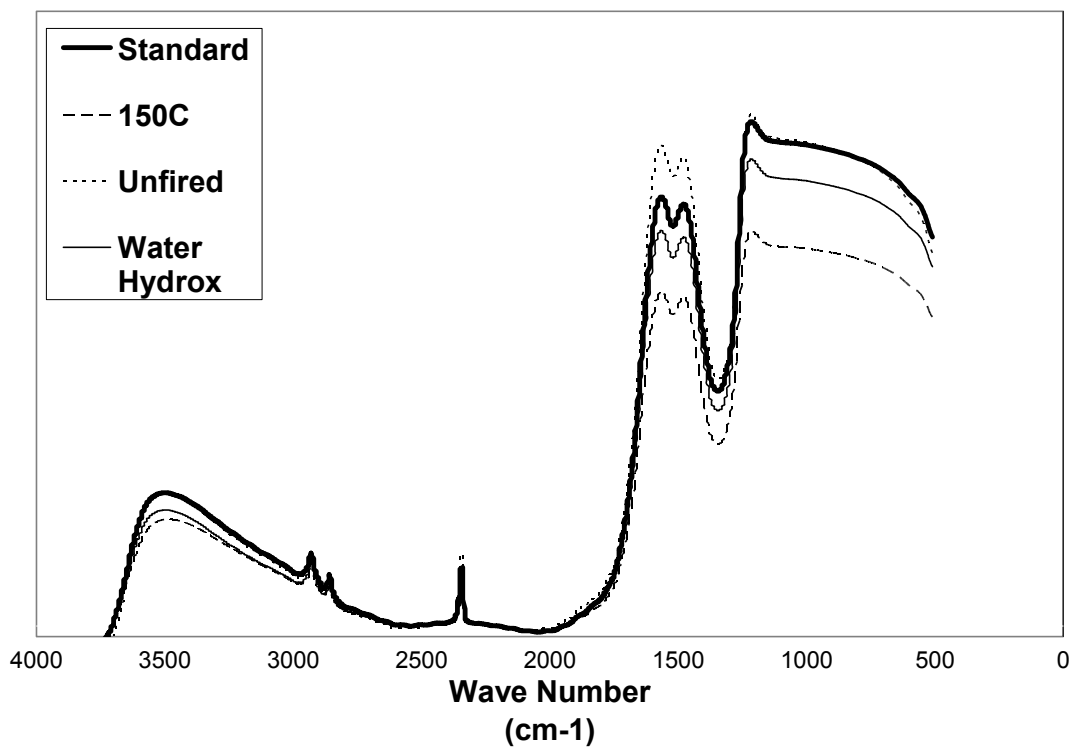


Figure 2. FTIR spectra for Anodiscs subjected to a variety of treatments prior to silation.

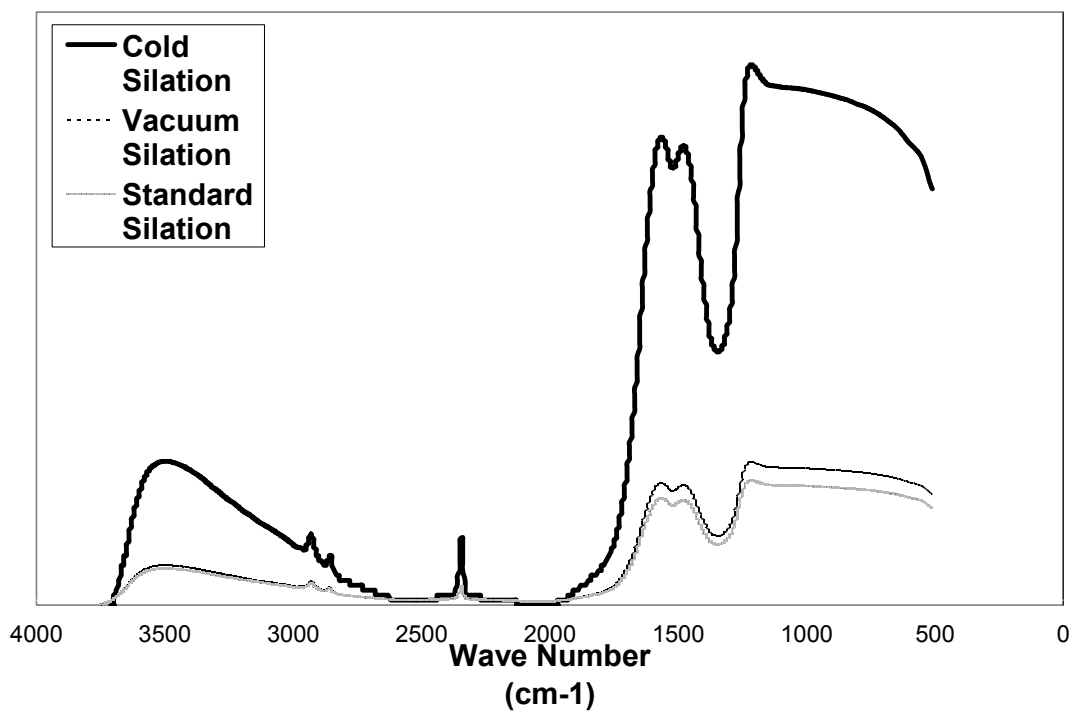


Figure 3. FTIR spectra for Anodiscs after a variety of silation procedures.

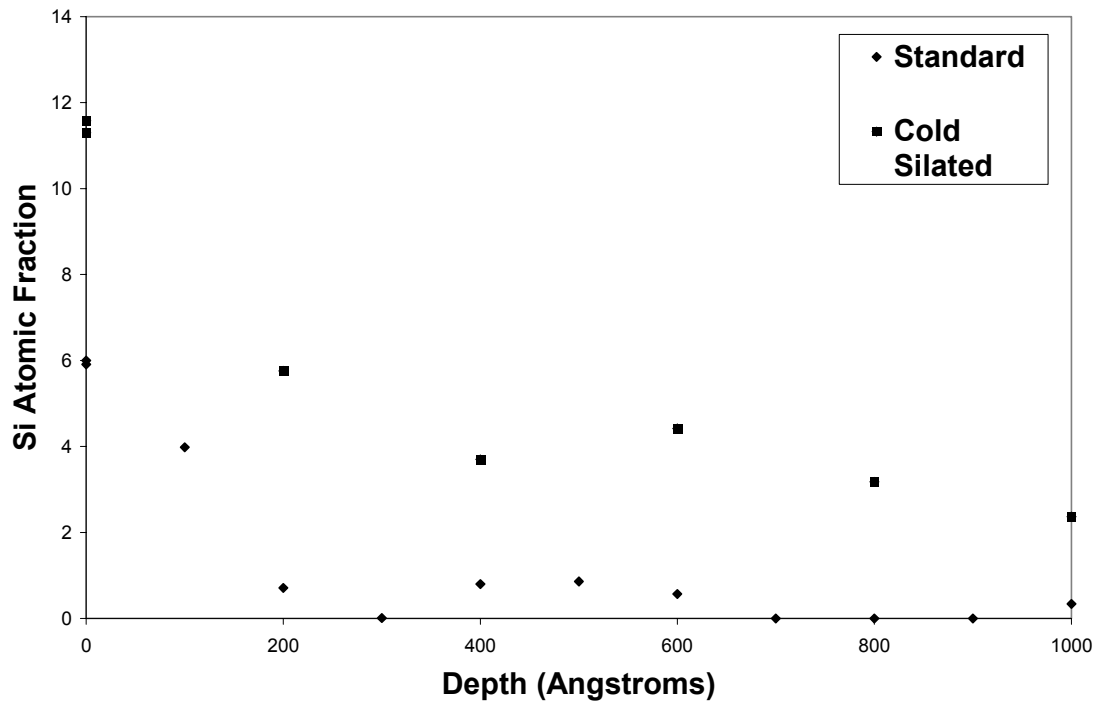


Figure 4. Silane penetration as determined by XPS sputter profiling.



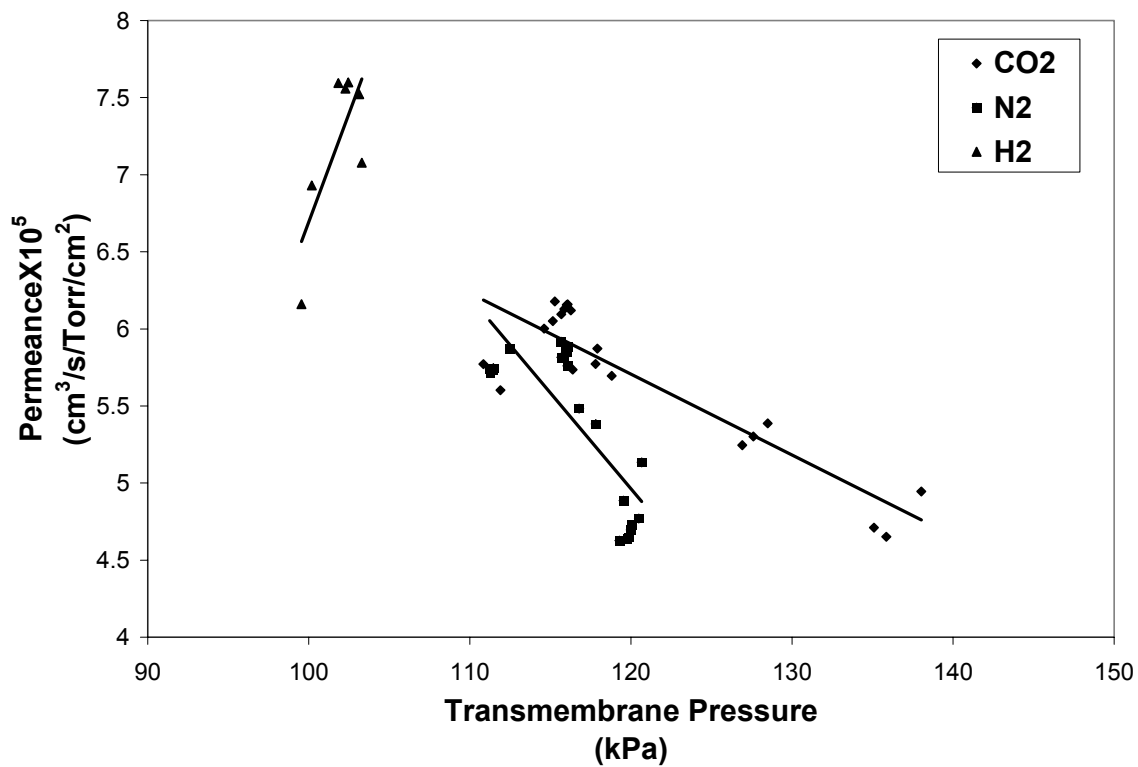


Figure 5. Room temperature permeance data for a blank alumina membrane tube calcined at 500°C.

**Table 1. Pretreatment summary for Anodisc samples.**

Sample	Firing	Hydroxylation	Post Silation Drying
Standard	500°C	H <sub>2</sub> O <sub>2</sub>	50°C
Unfired	None	H <sub>2</sub> O <sub>2</sub>	50°C
Water Hydrox	500°C	H <sub>2</sub> O	50°C
150°C	500°C	H <sub>2</sub> O <sub>2</sub>	150°C

**Table 2. Relative surface silane atomic concentrations for various silation procedures as determined by XPS.**

Sample	Relative Si Quantity
Hydroxylation Only	1.0
Vacuum Silation	7.2
Cold Silation	8.1
150°C Drying	4.1

# **CO<sub>2</sub>-Selective Hybrid Membranes by Silation of $\gamma$ -Alumina**

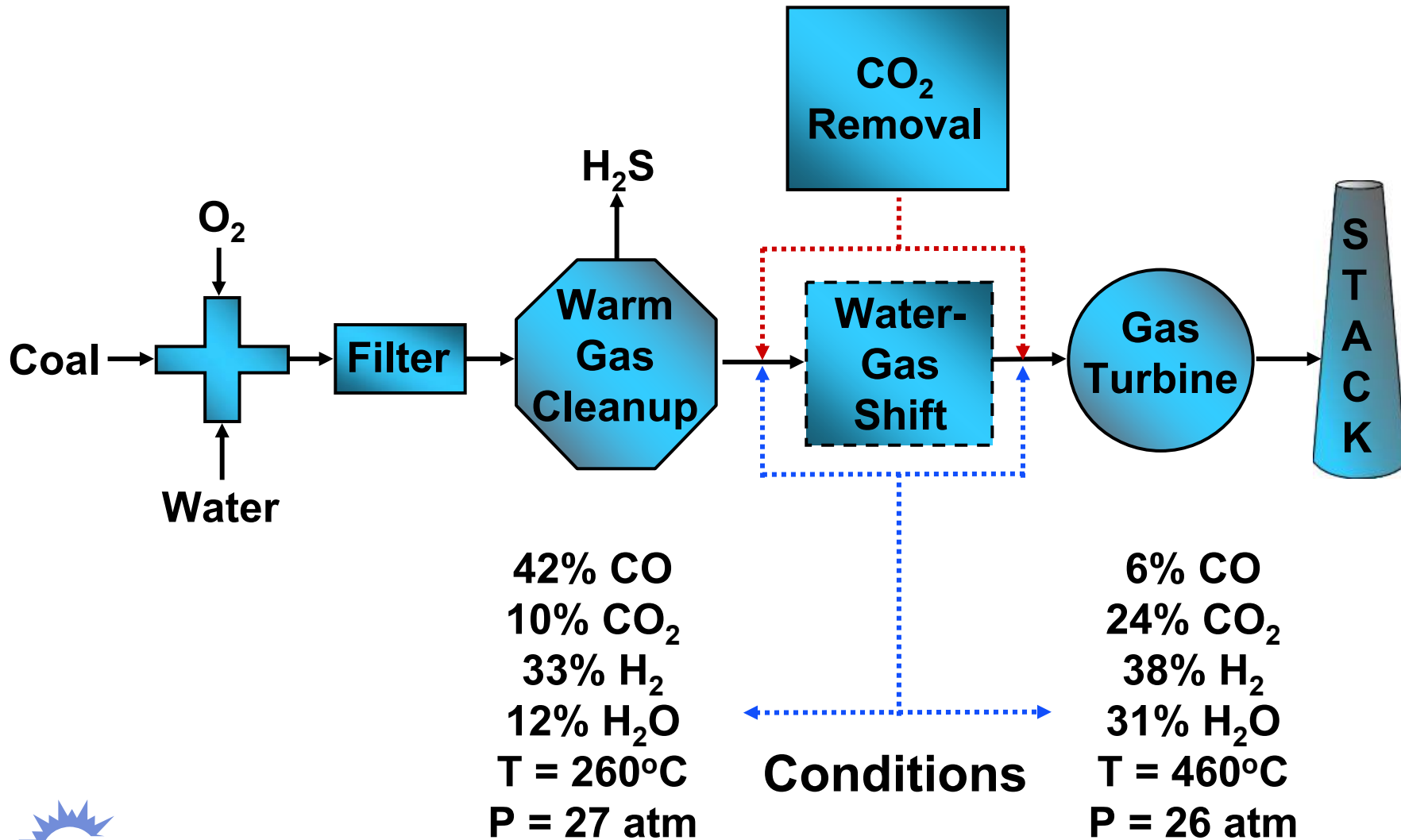
*David Luebke and Henry Pennline  
U.S. Department of Energy  
National Energy Technology Laboratory*

*September 17, 2003*

*Twentieth Annual International  
Pittsburgh Coal Conference*



# Two Possible Locations in IGCC



1. "Clean Coal Reference Plants: IGCC, Texaco", Final Report, February 2002.

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# Why a CO<sub>2</sub>-Selective Membrane?

- **Simplicity**
- **Relatively low operating cost**
- **Capable of producing a relatively pure CO<sub>2</sub> stream ready for sequestration with little further refinement**



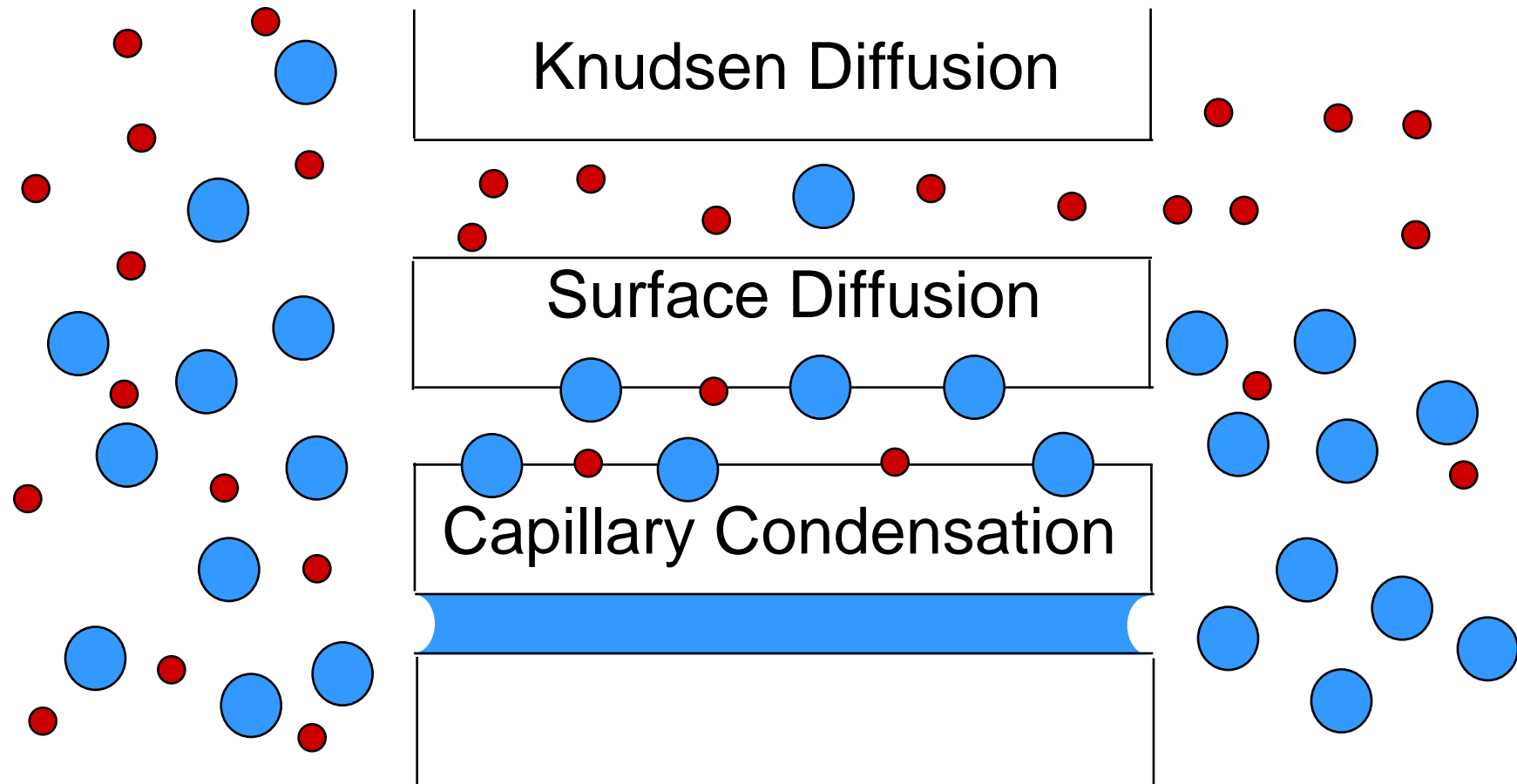
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# Challenges in Development

- **Operating conditions**
  - Temperature and Pressure
  - Moisture
  - Gaseous Impurities
- **CO<sub>2</sub>/H<sub>2</sub> size difference**
- **Must take advantage of the most difficult form of diffusion to optimize**



# Membrane Transport





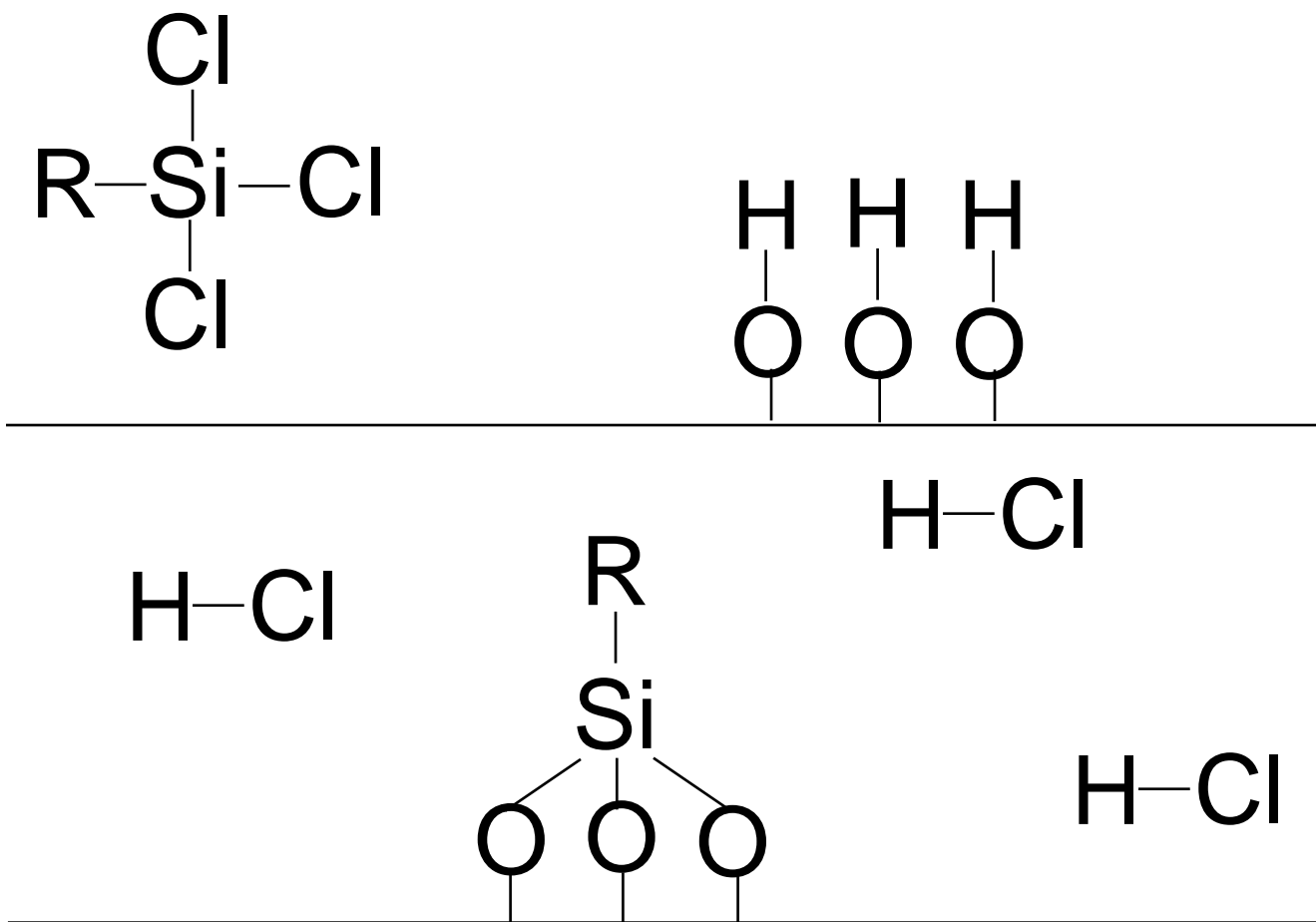
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# Organic/Inorganic Hybrids

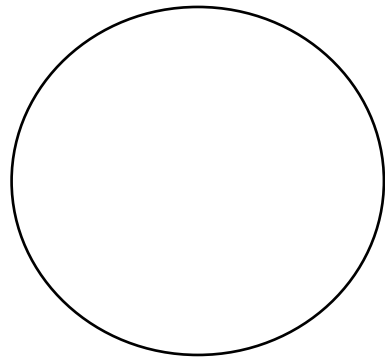
- **Have the potential to show the positive attributes of both membrane types .**
- **Should allow for a level of “fine-tuning” in performance not possible in other membrane types.**
- **Can be prepared by silation of alumina, a relatively well-understood and inexpensive process.**



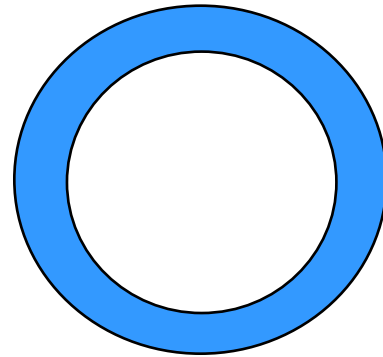
# Silation of Inorganic Membranes



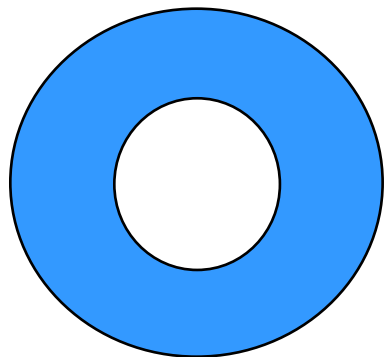
# Regulated Pore Size Shuts Down Knudsen Diffusion



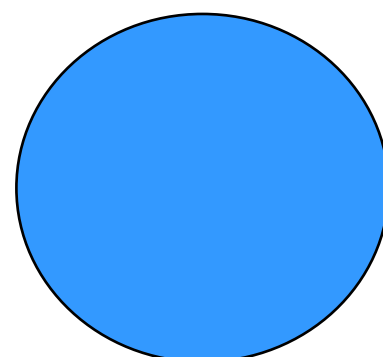
Unmodified  
 $\text{AlO}_3$



Short  
R-group

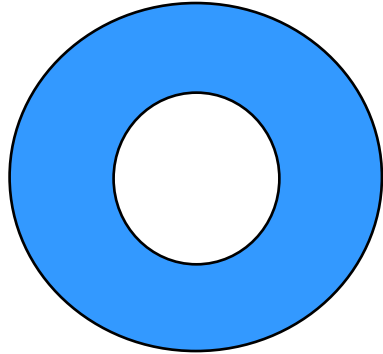


Moderate  
R-group



Long  
R-group

# CO<sub>2</sub>-philic Groups Enhance Surface Diffusion



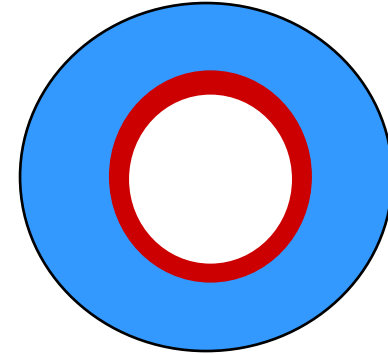
Ideal Pore Size

+

Carboxyl-  
Phenyl-  
Halo-

CO<sub>2</sub>-philic  
Groups

=



Ideal Membrane

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# Previous Work Produced Only Polymer-like Membranes

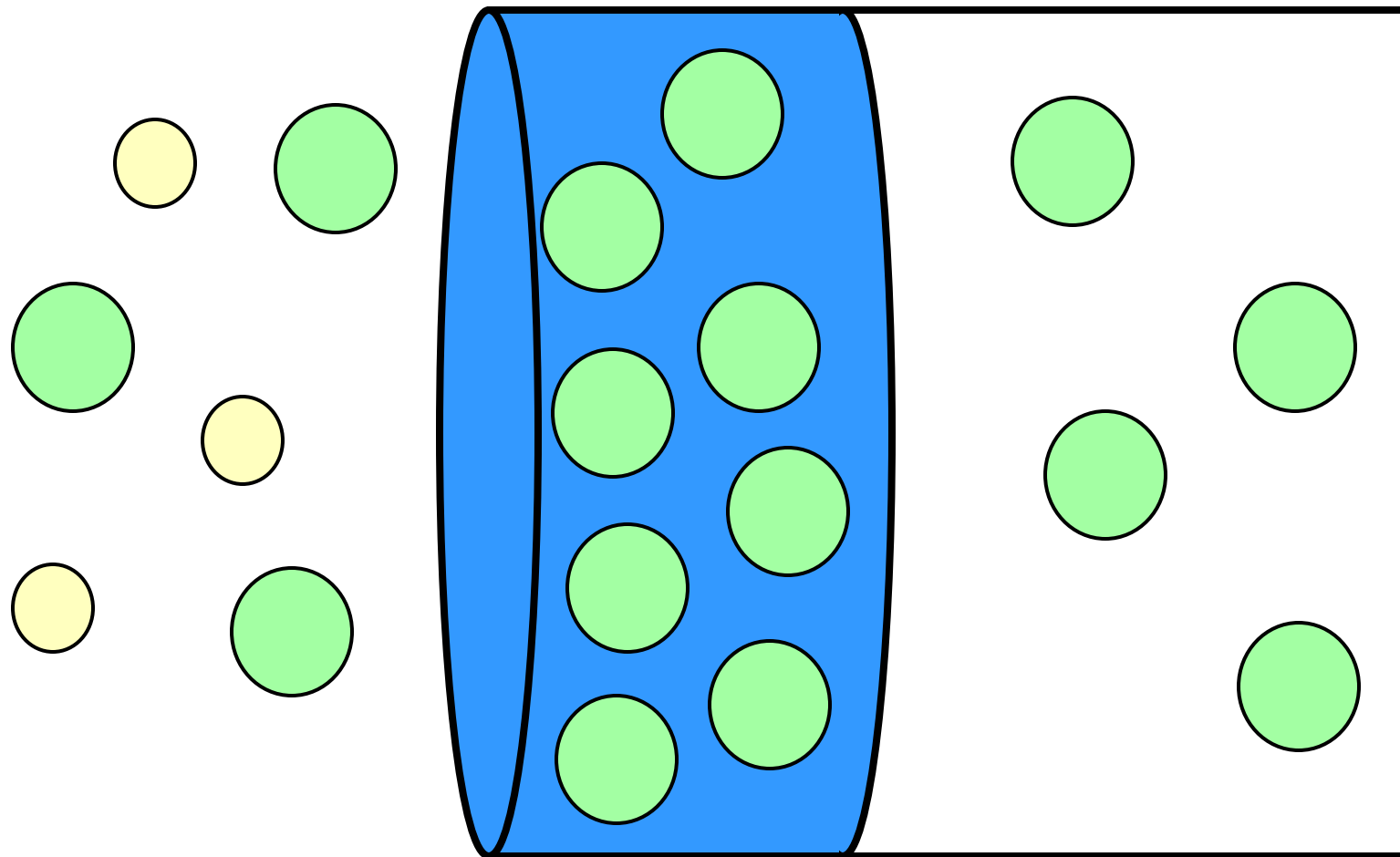
- Sang and Coworkers Silated  $\gamma$ -Alumina and  $\gamma$ -Alumina/Titania with Phenyltriethoxysilane<sup>2</sup>
- McCarley and Way Silated  $\gamma$ -Alumina with Octadecyltrichlorosilane<sup>3</sup>
- McCarley and Way Show Lack of Pore Penetration Using XPS Sputter Profiling

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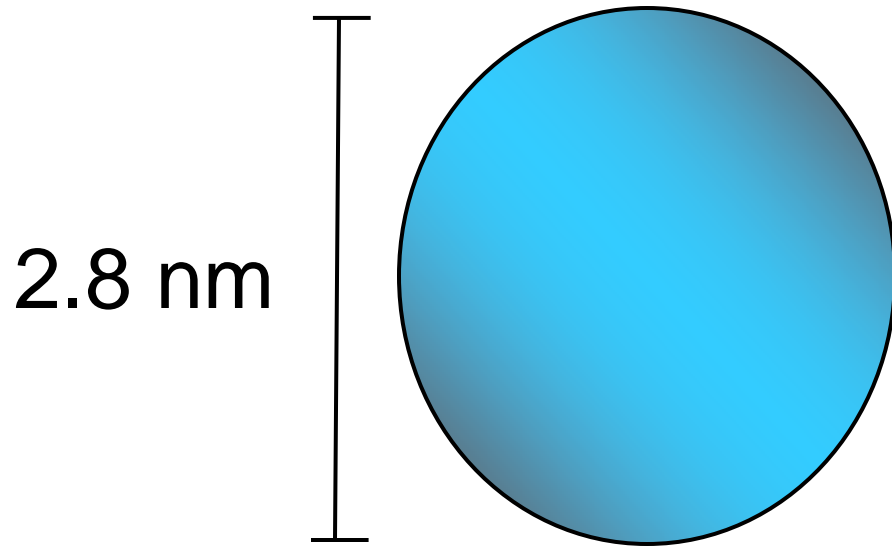


# Polymer-like Membrane

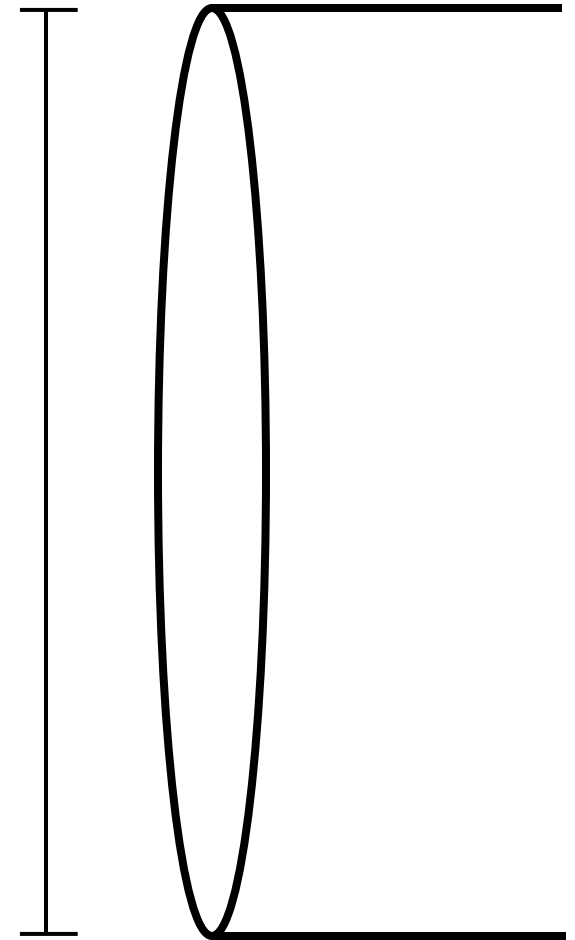


# Silane Diameter < Pore Diameter

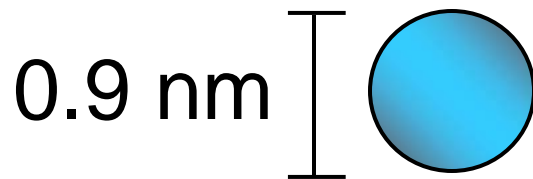
n-Octadecyltrichlorosilane



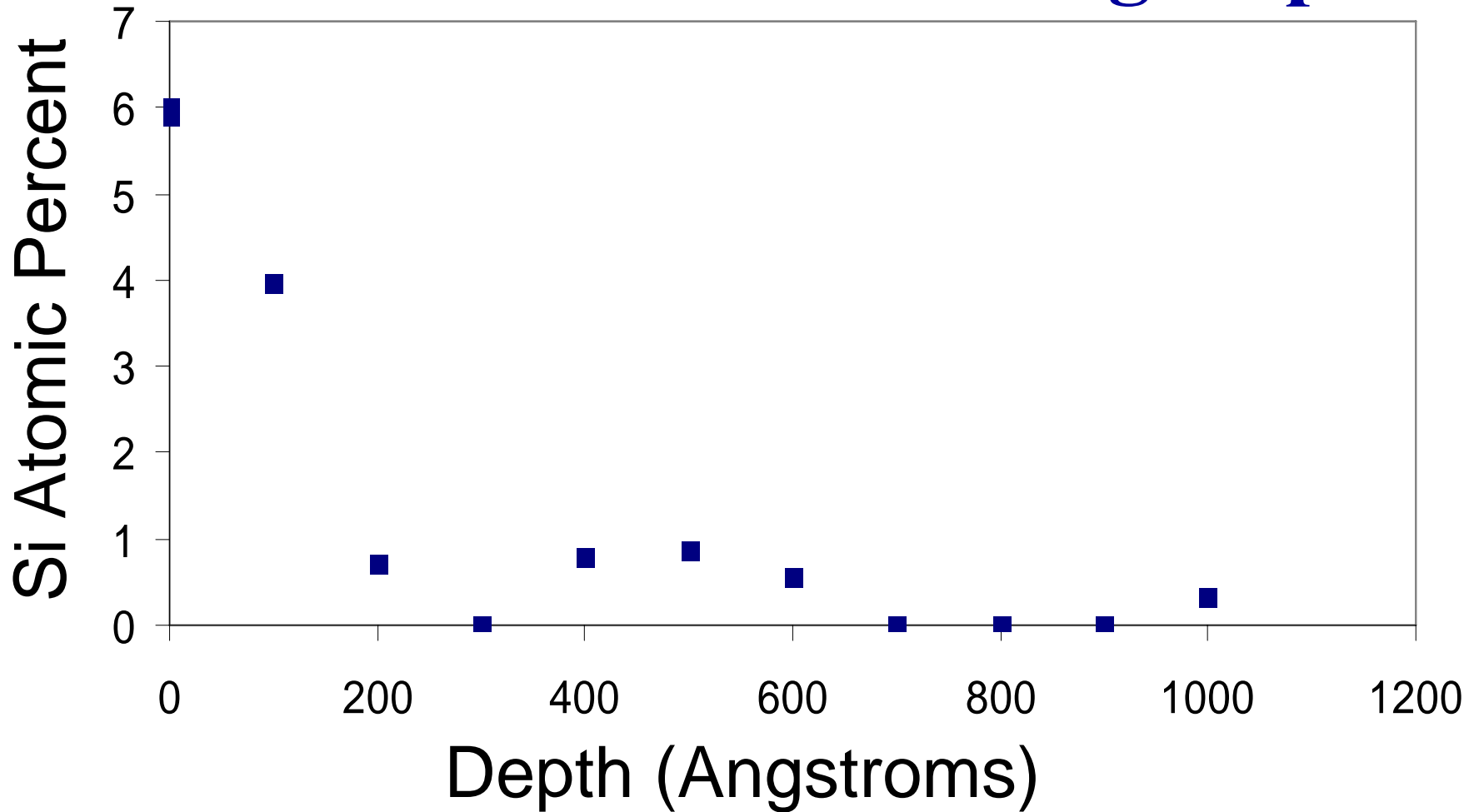
5.0 nm



n-Butyltrichlorosilane

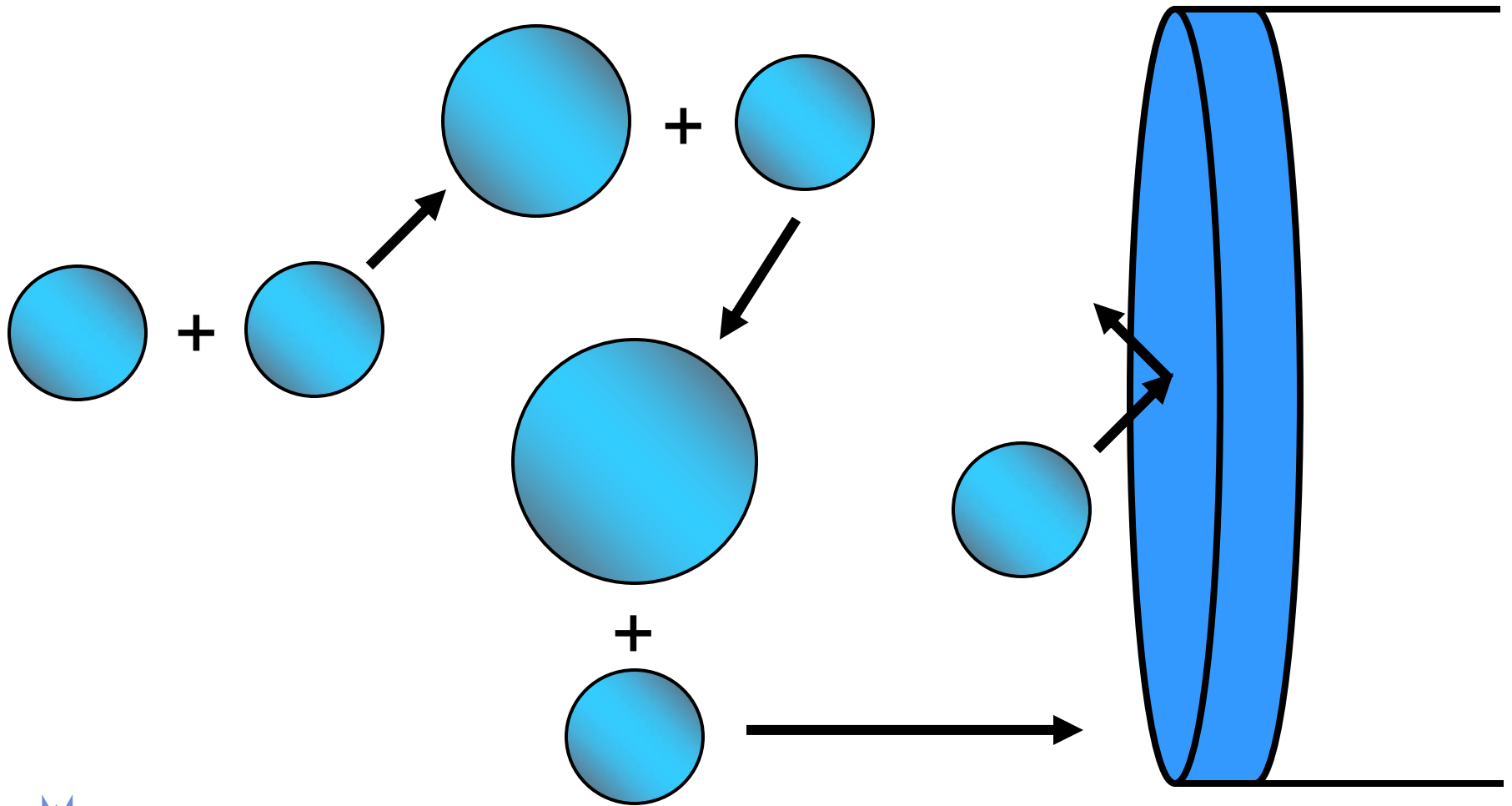


# Pore Penetration Unimproved by Substitution of Smaller R-group





# Agglomeration: Another Possible Cause?



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# Hypothesis

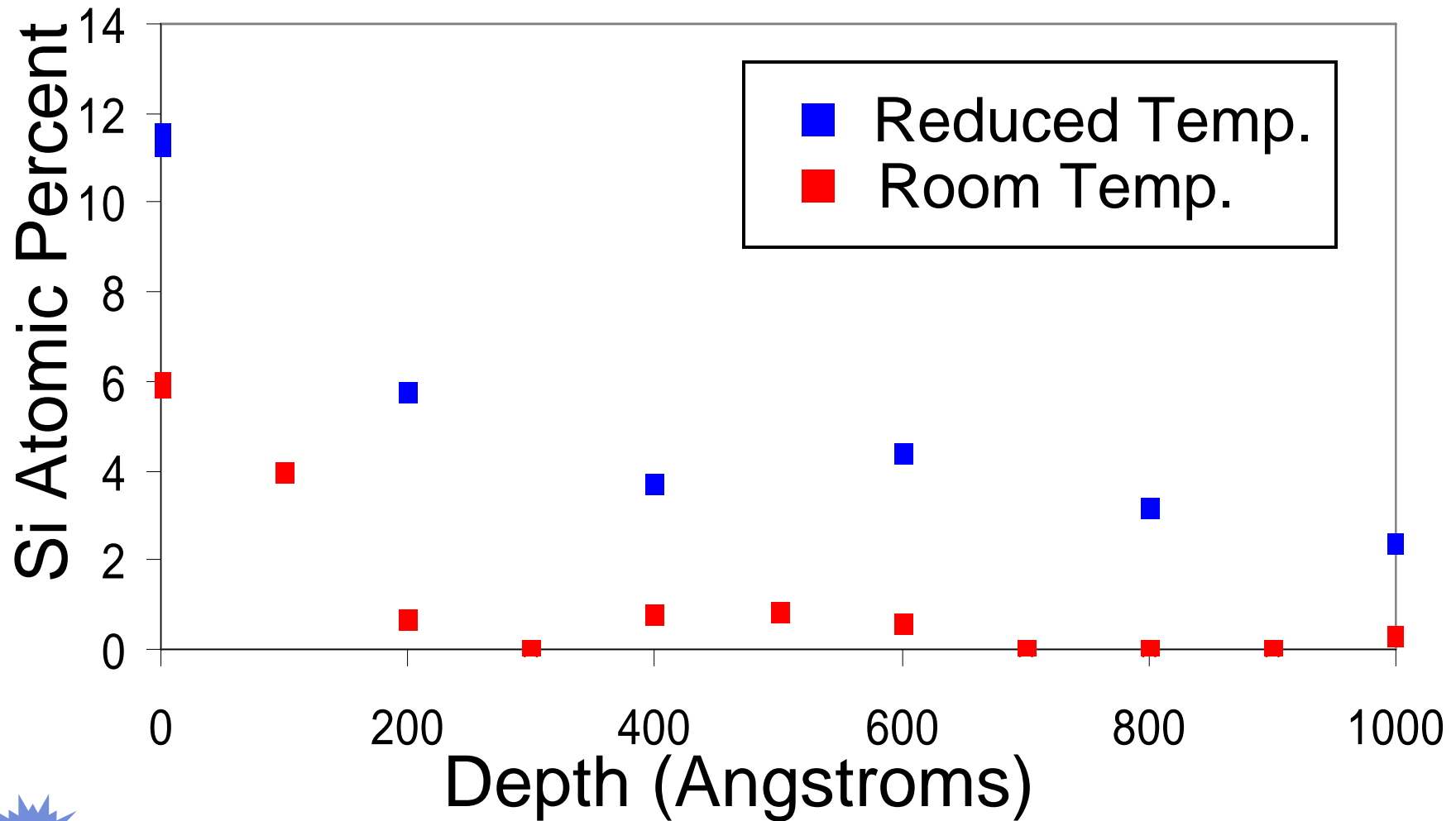
Reduction of reaction temperature could slow the rate of agglomeration to the extent where silitation becomes dominant resulting in silitation of the interior pore surface.

# Hypothesis Consistent with Work of Carraro<sup>4</sup> on Non-porous Surfaces

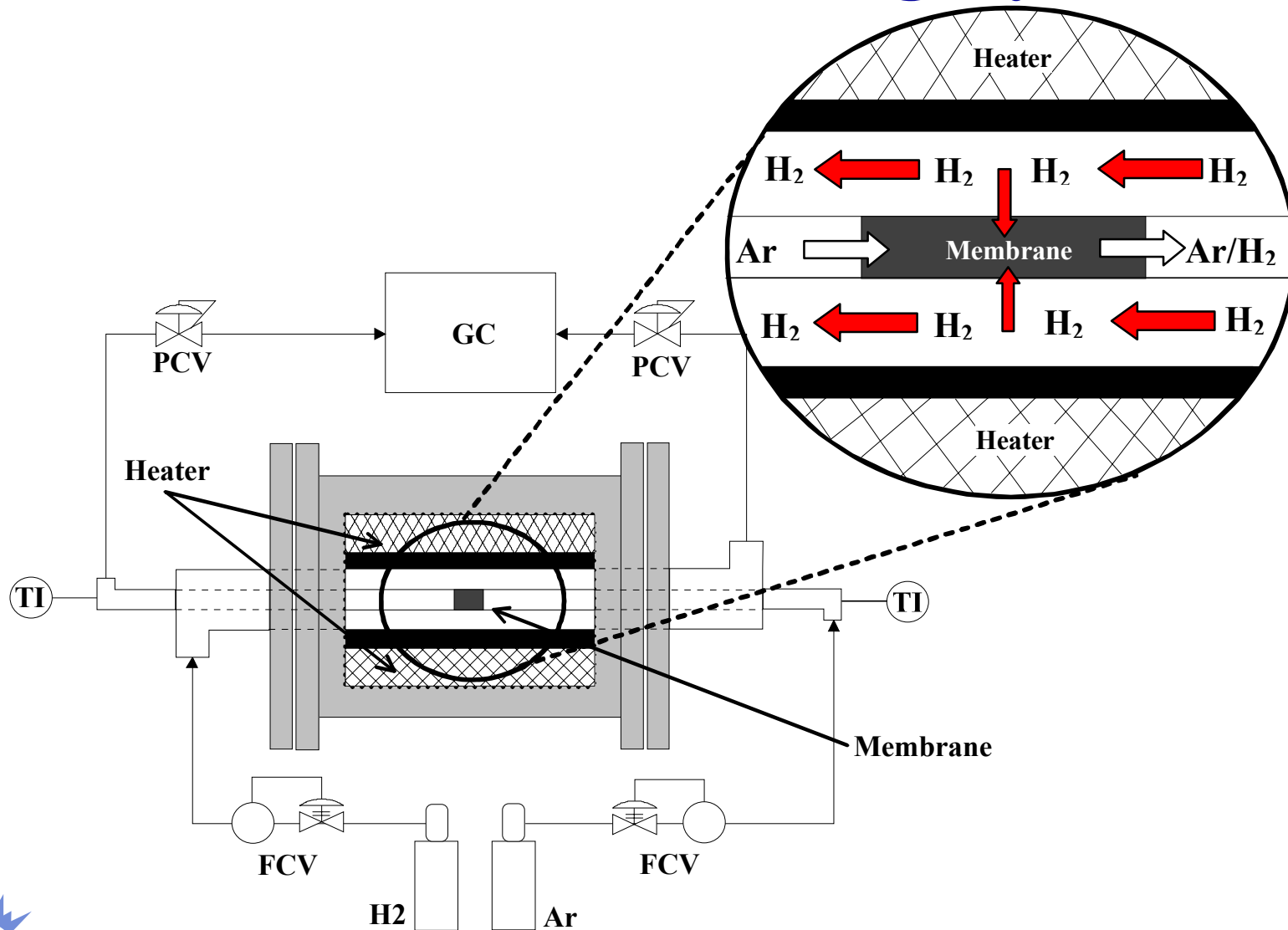
- Below 10°C octadecyltrichlorosilane formed islands on the surface which expanded to form a contiguous monolayer.
- Above 40°C no island formation occurred. Instead reaction in solution took place to form larger agglomerate species which eventually condensed on the surface.
- Between these temperatures a mixed regime was present in which islands formed but ceased their expansion before a contiguous monolayer was present.



# Silation at Reduced Temperature Improves Pore Penetration

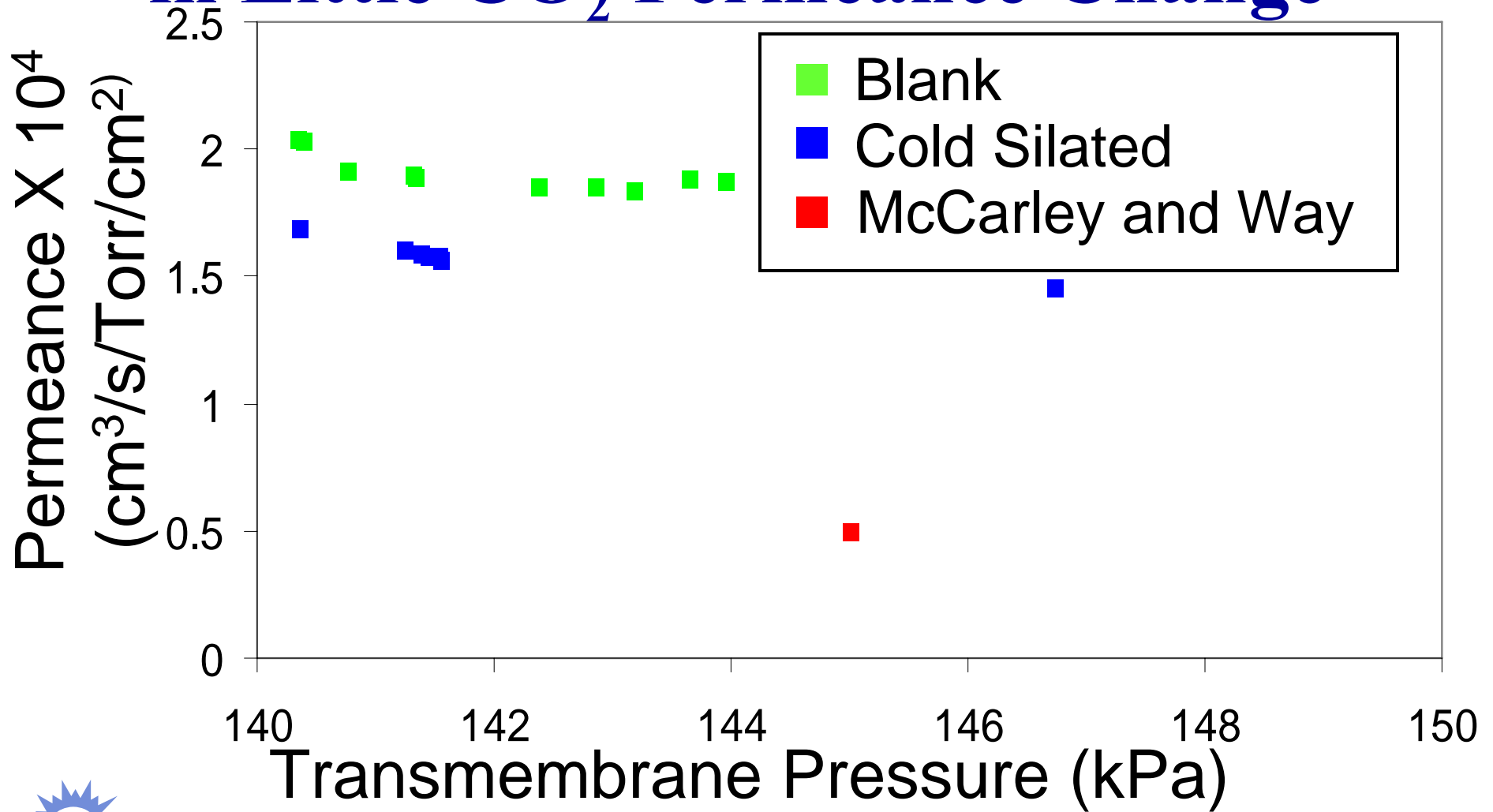


# Performance Testing System



5. Kurt S. Rothenberger, et al., Submitted to the J. of Mem. Sci., (2003).

# Addition of Butylsilane Groups Results in Little CO<sub>2</sub> Permeance Change



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# Conclusions

- **Silane pore penetration can be achieved by reducing fabrication temperature.**
- **Pore blockage does not occur with silation at reduced temperature.**
- **Minor reduction in permeance in cold silated membrane encourages optimism about the permeance of CO<sub>2</sub>-selective membranes.**



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# Future Work

- **By changing the length of the silane R-group, a high degree of control over effective pore diameter should be achievable.**
- **Addition of groups to the silane with a strong CO<sub>2</sub> affinity should make it possible to develop highly selective membranes.**





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