USE OF CARBON MOLECULAR SIEVES FOR FOR CHROMATOGRAPHIC SEPARATIONS OF PERMANENT AND OTHER GASES

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Introduction

A sub-micron particle size carbon molecular sieve has been synthesized to possess monoporous, microporous characteristics. This CMS has been adhered to the inner surface of fused silica capillary tubing, using a proprietary, patented glue technology. The resultant porous layer open tubular (PLOT) column possesses gas-solid chromatographic performance characteristics which provide effective separation of hydrogen, oxygen, nitrogen, carbon monoxide, carbon dioxide, methane, acetylene, ethylene, ethane, propyne, propylene, propane, noble gases and nitrous oxide. This chromatographic performance is applicable to several fields of interest.

Experimental

The experimental approach consisted of three segments: carbon synthesis, column preparation and process reproducibility, and column applications. In segment one, preparation of the carbon was focused on preparing a series of spherical carbon molecular sieves possessing monoporous, microporous characteristics with increasing pore diameters. This series entailed CMSs with 3, 4, 5, 6, 7, 8, 9, or 10Å +/- 1.0Å pore diameters. Pore diameter was determined using a Micromeritics ASAP2010 porosimeter[1].

In segment two, 0.53 mm ID PLOT columns were prepared with increasing film thicknesses of the 5Å porous CMS until the resulting column effectively separated the oxygen and nitrogen masses. A 35 uM film thickness was determined to be optimum, and subsequent evaluations of the carbon pore diameters were performed with the columns possessing this layer thickness. Carbon dioxide was subsequently used as the probe molecule for determining absolute retention times.

A maximum pore diameter was determined by monitoring a decrease in the carbon dioxide retention time as a result in a decrease in the three dimensional adsorption on the analyte molecules in the pores. The molecular diameters of the analytes of interest for this segment study are listed in Table 1.

Table 1. Molecular diameters of permanent gases

Molecule	diameter(Å)
Oxygen	2.98
Nitrogen	3.15
Carbon monoxide	3.19
Carbon dioxide	3.34

Secondary evaluations were focused on the separation of hydrogen, oxygen and nitrogen, and in the third segment on the separations of the C2 and C3 hydrocarbons, noble gases and nitrous oxide. An HP 5890 gas chromatograph was used for all chromatographic evaluations.

Results and Discussion

The specific goal of the segment one experiments was to produce a CMS with a maximum number of pores possessing diameters effective in producing the desired maximum carbon dioxide retention time. The results obtained indicate that a pore diameter of 7.0 +/- 1.0Å is optimum to effectively adsorb/retain the carbon dioxide molecules and provide effective separation of oxygen and nitrogen.

In segment two, the porosity data presented above were related to the absolute retention time of carbon dioxide. The correlation data indicate that a CMS with a pore diameter of 7.0 +/- 1.0Å, a BET[1] surface area of 700 meters^2/gram and a pore volume of 0.35 cubic centimeters/gram provided optimum chromatographic performance.

In segment three, chromatographic application efforts were focused on separations of the permanent gases, light hydrocarbons, noble gases, and nitrous oxide.

Conclusion

A carbon molecular sieve has been synthesized to possess 7Å monoporous, characteristics. This submicron chromatographic sieve has been adhered to the inside walls of fused silica capillary tubing, and utilized for the chromatographic separation of permanent gases, light hydrocarbons, noble gases, and nitrous oxide. These columns are applicable to several fields of interest, specifically to the transformer oil industry and the general gas chromatography audience.

References

1. Webb, P.A. and Orr, C., *Analytical Methods in Fine Particle Technology*. Micromeritics, Norcross, GA, 1997.