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This dissertation is dedicated to God,  
the great "I AM"  
without whom I would not exist.  
To my wife, Erin, for her infinite  
reserve of grace that made this all  
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## **ABSTRACT**

The sol-gel process is a synthetic approach that can yield silicon dioxide-based inorganic polymer materials of varying densities, pore volumes, and crystallinity. Depending on the initial conditions of the reaction, materials such as high density amorphous glasses called xerogels, intermediate density crystalline powders called zeolites, or low density-high surface area glasses called aerogels can be fabricated. Both the xerogels and the zeolites have been doped with transition metal ions to produce hybrid metal-silica materials that exhibit catalytic properties. The process for synthesizing these hybrid metal-silica materials has been extended to create a novel type of metal doped silica, low density-high surface area aerogel.

The results of this investigation elucidate the electronic properties of vanadium and chromium metal centers that reside in amorphous xerogel matrices and in crystalline zeolites. Differences in the metal coordination and electronic structure imposed by the amorphous and crystalline matrices have been quantified. Spectroscopic studies of this nature should help in the development of new and more efficient catalysts for industrial organic synthesis.