B.S. Junior Year – Spring Semester Lab

CEM 395 Analytical/Physical Laboratory

Description: Chemical kinetics, thermodynamics, and computer-based data analysis methods.

Credit: 2 Credits (1 hour lecture and 4 hours laboratory per week)

Prerequisite: CEM 483 and (CEM 262 or CEM 186H), credit or co-enrollment in CEM 484, and completion of Tier I writing requirement.

Recommended Background: One year of general chemistry, calculus, and general physics.

Initially the lecture focuses on technical report writing, propagation of error, and chemical kinetics. Formal laboratory reports, including propagation of error for all calculations, are required for each of the six experiments.

1. **Kinetics I: Method of Initial Rates**

   (Rate law determination for the iodine clock reaction; identification of mechanisms consistent with the rate law)

2. **Kinetics II: Activation Energy Determination and Kinetic Isotope Effect** (UV-Vis and Conductivity Measurements)

   (Mechanistic information elucidated for the spontaneous hydrolysis of acetic anhydride by studying the reaction (1) as a function of temperature (activation energy and Arrhenius pre-exponential factor) and (2) in D_2O and H_2O to determine kinetic isotope effect; non-linear curve fitting to determine rate constants)

3. **Determination of Substituent Effects on the Standard Reduction Potential of Benzonquinones** (Cyclic Voltammetry and Computational Chemistry)

   (Determination of standard reduction potentials using cyclic voltammetry; use of Hammett equation for evaluation of substituent electronic effects; comparison of reduction potential with computationally calculated LUMO energies (Spartan))

4. **Identification of a Crystalline Substance** (XRD and XRF)

   (Qualitative analysis of unknown powders with energy dispersive XRF; measurement of XRD powder patterns and determination of crystal structure and unit cell length; prediction of reflection intensities using structure factor calculations)

5. **Thermal Lens Calorimetry** (Green diode laser)

   (Detemination of heat capacity of various solvents using the time constant for the development of a thermal lens; parameter determination using non-linear least squares curve fitting; familiarity with lasers and assembly of optical components on a laser table)

6. **Enol-Keto Tautomerism** (NMR)

   (Effect of temperature, substituents, and solvent on the enol-keto equilibrium; influence of electron withdrawing/donating groups and ring currents on chemical shift; calculation of ΔH^0, ΔS^0, and ΔG^0 for the tautomerism reaction)

Alternative experiment:

Differential Scanning Calorimetry (DSC)

(Determination of transition temperatures, transition enthalpies, and % crystallization of polymers; discussion of polymers and polymerization as well as chain reactions, initiators, glass transition temperatures, heat capacities, and amorphous vs. crystalline polymers)