College of Science and Mathematics

Department of Chemistry

Inorganic Chemistry Laboratory (CHEM 371)

Spring 2013 Syllabus

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Experiment 1: Semi-microscale preparation of copper(I) chloride

Students gain experience in the preparation and handling of air sensitive compounds. Knowledge is gained in the application of redox principles to synthesize Cu(I). Students determine the oxidation state of the final product by qualitative analysis.

Experiment 2: Microscale preparation of tin(IV) iodide

Both SnI_4 and SnI_2 are prepared as a mixture from elemental tin and iodine. SnI_4 is isolated based upon a difference in polarity explained by the *inert-pair effect*. The chemistry of SnI_4 is investigated using wet chemical methods.

Experiment 3: Preparation of tris(2,4-pentanedionato)manganese(III)

Students employ their knowledge of redox chemistry in the preparation of $Mn(acac)_3$. The latter complex is characterized by UV-vis absorption spectroscopy and an in depth knowledge of symmetry and crystal field theory are used to explain the concept of *Jahn-Teller distortion* and its consequences on the electronic transition observed. Students are introduced to the concept of *chelating* ligands.

Experiment 4: Microscale determination of magnetic susceptibility

Experience is gained in the determination of magnetic susceptibility using the Evan's method technique (Johnson-Matthey magnetic susceptibility balance) for transition metal complexes prepared in previous experiments, $[Cr(NH_3)_6](NO_3)_3$ and $Mn(acac)_3$. The magnetic moment is derived by application of the Curie-Weiss law and used to predict the *spin-state* (high-spin vs. low-spin) of the transition metal *d* subshell.

Experiment 5: Microscale preparation of ferrocene

Students conduct the preparation of ferrocene under inert conditions (N_2 atmosphere). The product is purified by sublimation and characterized by a combination of attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) and qualitative analytical methods. Knowledge is gained in organometallic chemistry and the concept of *hapticity* introduced.

Experiment 6: A synthetic and photophysical analysis of Ru(II) polypyridyl complexes

Students gain further experience with transition metal coordination chemistry and electronic transitions. The synthetic goal is to prepare a chromophore to be used in a dye-sensitized solar cells (re. Expt. 10). This complex is characterized by UV-vis absorption spectroscopy and the nature of *charge-transfer electronic transitions* is explained. Knowledge is gained in applications of inorganic photochemistry and photoelectrochemistry (electron transfer, dye-sensitized solar cells, light emitting diodes etc.).

Experiment 7: An introduction to cyclic voltammetry

Students learn how to perform cyclic voltammetry (CV) and how to analyze and process the data acquired. The concept of using ferrocene/ferrocenium redox couple as a *pseudo reference* is introduced (previously prepared in Expt. 7). Formal potentials are recorded for the previously prepared ruthenium complex (Expt. 8) and are correlated with UV-vis and emission spectra for this complex.

Experiment 8: Fabrication and power conversion efficiency of a dye-sensitized solar cell

Students learn how to fabricate an inorganic based solar cell device using the previously prepared ruthenium complex (Expt. 8). Knowledge of nanoparticle semiconductor electrodes is gained. The concepts of the solar cell are introduced and determination of solar cell efficiency is carried out. Further knowledge is gained in the understanding of photoinduced electron transfer and its applications.

Experiment 9: Electrocatalytic H_2 production using cobaloxime complexes

Thus, the so called ‘hydrogen economy’ has the potential to provide tremendous environmental and economic benefits for todays society. Current hydrogen production methods include the gasification of coal and the steam reforming of natural gas to produce a variable mixture of CO and H_2 known as “syngas”. This laboratory experiment is aimed at developing your understanding of the thermodynamic and molecular process involved in the green synthesis of H_2 gas using an inorganic catalyst inspired by the vitamin B-12 structure. Knowledge of organometallic catalysis is gained and students are introduced to electrochemical catalysis.

Experiment 10: Preparation and photophysical characterization of CdS quantum dots.

A one-pot synthetic procedure for the preparation of core-shell $CdSe/Zn_xCd_{1-x}S$ quantum dots is conducted. Photophysical properties of the QDs are investigated and basic theory of inorganic semiconductors is explored.