Laser-Ionization Time-of-Flight Mass Spectrometry of High Molecular Mass Inorganic Complexes

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Laser-Ionization Time-of-Flight Mass Spectrometry (LI-TOF-MS) is a sophisticated tool for the molecular-weight determination and structural characterization of a variety of molecules. Advances in instrumentation and ionization methods have recently expanded its role in the analysis of high-mass analytes. Large multimetallic complexes, which are efficient solar-energy converters, rely heavily on their chemical structure for optimum operation. Molecular mass determinations of these multimetallic complexes have been problematic due to their lability and high molecular weights.

This thesis describes the characterization of a LI-TOF-MS instrument and confirmation of theoretical time-of-flight mass-separation principles. Several test cases demonstrate the instrument's proper operation and calibration for a wide mass range of analytes. Mass spectral results of three organometallic compounds: i. $[Ir(dpp)_2Cl_2](PF_6)$, ii. $\{[(bpy)_2Ru(dpp)]_2IrCl_2\}(PF_6)_5$, and iii. $\{[(bpy)_2Ru(dpp)]_2RuCl_2\}(PF_6)_5$ under a variety of laser ionization and sample preparation conditions are compared. A complete structural characterization of the monometallic complex, $[Ir(dpp)_2Cl_2](PF_6)$, is presented. The two trimetallic analytes fragmented easily, but significant components of the molecules are successfully identified. After optimizing the ionization and analytical procedure, LI-TOF-MS proved useful in the analysis of high molecular mass metal complexes.

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