

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

TABLE OF CONTENTS

ABSTRACT	ii
ACKNOWLEDGMENTS	iii
TABLE OF CONTENTS.....	iv
TABLE OF FIGURES.....	vi
CHAPTER I. INTRODUCTION.....	1
I.1- THE PRINCIPLES OF TIME-OF-FLIGHT MASS SPECTROMETRY	1
I.2- ADVANTAGES/DISADVANTAGES OF TOF-MS	1
I.3- HISTORY OF TOF-MS	2
I.4- THE TIME-OF-FLIGHT MASS SPECTROMETER AND RELATED INSTRUMENTATION.....	4
I.5- NEUTRAL SOURCES AND IONIZATION METHODS	7
I.6- THEORY OF MASS SEPARATION BY TOF-MS.....	13
I.7- MASS CALIBRATION	15
I.8- RESOLUTION.....	16
CHAPTER II. EXPERIMENTAL SETUP.....	31
II.1- INSTRUMENTAL DESIGN OVERVIEW	31
II.2- VACUUM SYSTEM	31
II.3- IONIZATION CHAMBER.....	34
II.4- FLIGHT TUBES.....	35
II.5- REFLECTRON.....	35
II.6- LASER IONIZATION.....	37
II.7- DETECTORS	38
II.8- ELECTRONICS AND TRIGGERING	39
II.9- DATA HANDLING.....	41
CHAPTER III. INSTRUMENT CHARACTERIZATION	43
III.1- INITIAL INSTRUMENT OPERATION	43
III.2- REPRODUCIBILITY OF EXPERIMENTAL TIMES-OF-FLIGHT ..	46
III.3- SPACE FOCUS.....	46
III.4- RESOLUTION	59
III.5- MASS CALIBRATION.....	63
III.6- ION YIELDS FOLLOWING LASER ABLATION	66
III.7- ION VELOCITIES FOLLOWING LASER ABLATION	72
CHAPTER IV. DOPED METAL OXIDE SYSTEMS.....	84
IV.1- INTRODUCTION	84
IV.2- 5% Eu:Y ₂ O ₃ results	85
IV.3- 0.033% Eu:CaO results.....	88

TABLE OF CONTENTS (cont.)

IV.4- ISOTOPE ANALYSIS	93
CHAPTER V. FULLERENES ANALYSIS.....	99
V.1- BACKGROUND OF THE ALL-CARBON FULLERENE MOLECULES	99
V.2- GOALS OF OUR FULLERENES ANALYSIS.....	102
V.3- SAMPLES ANALYZED AND SAMPLE PREPARATION.....	103
V.4- C ₆₀ RESULTS.....	103
V.5- C ₇₀ RESULTS.....	107
V.6- DISCUSSION.....	110
V.7- ACTUAL SAMPLE CONSTITUENT VERSUS GAS-PHASE FORMATION ISSUE.....	112
V.8- QUANTITATION OF SPECIES IN THE C ₇₀ SOOT	113
CHAPTER VI. ANALYSIS OF HIGH MOLECULAR MASS INORGANIC COMPLEXES USING MATRIX-ASSISTED-LASER DESORPTION/IONIZATION (MALDI)	115
VI.1- HIGH MOLECULAR WEIGHT INORGANIC COMPLEXES.....	115
VI.2- MALDI.....	116
VI.3- GOALS.....	118
VI.4- SAMPLES ANALYZED.....	119
VI.5- INSTRUMENTAL PARAMETERS	124
VI.6- LASER BEAM DEFOCUSING AND SIGNAL AVERAGING.....	124
VI.7- MATRIX SELECTION FOR MALDI AND SAMPLE PREPARATION.....	125
VI.8- [Ir(dpp) ₂ Cl ₂]PF ₆ (“Dbl-Ir”) RESULTS	126
VI.9- {[(bpy) ₂ Ru(dpp)] ₂ RuCl ₂ } (PF ₆) ₄ (“Tri-Ru”) RESULTS	136
VI.10- {[(bpy) ₂ Ru(dpp)] ₂ iRCl ₂ } (PF ₆) ₅ (“JSB”) RESULTS	144
VI.11- OVERALL CONCLUSIONS.....	156
CHAPTER VII. CONCLUSIONS.....	158
VITA	160