

Identification of Ruthenium Dendrimers by Direct Infusion ESI-TOFMS

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1. Introduction

Enormous interest has surrounded the synthesis of organometallic dendrimers, in particular those containing redox-active transition metals. These novel supramolecular species may yield materials having potential applications such as chiral molecular recognition, conversion of light into chemical energy and catalysis. To date, the most significant challenge to the synthetic chemist has been the characterization of these nanoscopic molecules. Spectroscopic characterizations are often inconclusive because polymers of similar composition would give similar data.

LECO's Unique® TOFMS (Time-of-Flight Mass Spectrometer) provides an elegant and unambiguous solution to the characterization of transition metal dendrimers. The resolution and sensitivity of this instrument allows for identification of parent compounds as well as side products. In this application we demonstrate the characterization by mass spectrometry of di- and tetra-metallic ruthenium (II) compounds¹ shown below in Figure 1.

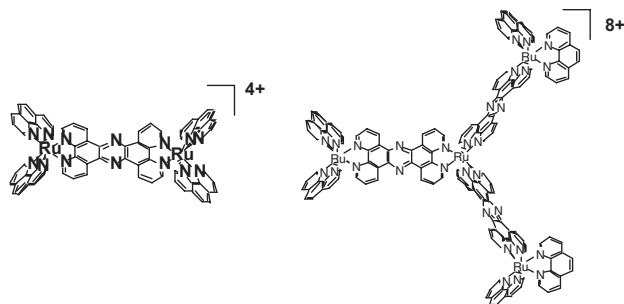


Figure 1. Structure of di- and tetra-metallic ruthenium (II) compounds.

2. Experimental Conditions

MS:	Unique® TOFMS
Source:	Low Flow ESI
Data Acquisition Rate:	2.5 spectra/second
Syringe Pump:	Harvard, model PHD 2000
Flow rate	1.5 μ L/min

3. Results

A standard solution (1.5×10^{-4} M in acetonitrile) of ruthenium dendrimers was prepared prior to analysis. Figure 2 represents a mass spectrum obtained for dimetallic ruthenium (II) dendrimer PF₆ salt (MW1308.19 Da). Three peaks with three different charge states dominate the mass spectrum. Peaks at mass-to-charge (m/z) 328, 485, and 800 can be assigned to the quadruply-charged $[M - 4PF_6]^{+4}$, the triply-charged $[M - 3PF_6]^{+3}$, and the doubly-charged $[M - 2PF_6]^{+2}$ ionic species, respectively. The inset of Figure 2 shows the detail of the $[M - 2PF_6]^{+2}$ ion at m/z 800. Each ruthenium contributes 8 isotopes to the observed isotopic spread of the peak. The resolution necessary to observe these individual isotopes was ca. 2,700.

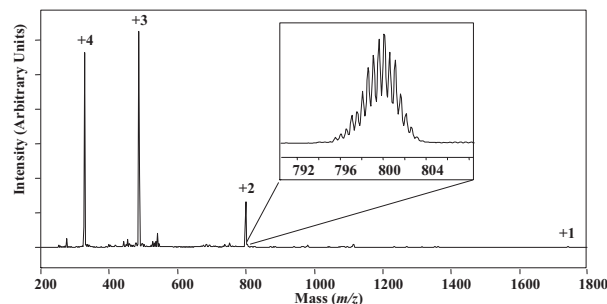


Figure 2. ESI-TOF mass spectrum of dimetallic ruthenium (II) complex.

Electrospray TOFMS yields mass spectra containing a series of cations that correspond to consecutive PF₆- counterion loss. These series of peaks provide unequivocal evidence for the tetrametallic ruthenium (II) dendrimer salt (MW 2640.36 Da) as shown in Figure 3.

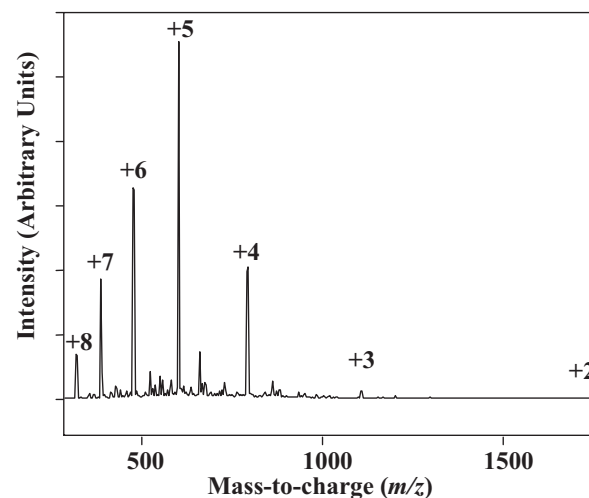


Figure 3. ESI-TOF mass spectrum of tetrametallic ruthenium (II) complex.

4. References

¹S. Bodige, A. S. Torres, D. J. Maloney, D. Tate, G. R. Kinsel, A. K. Walker, F. M. MacDonnell, *J. Am. Chem. Soc.* 119 (1997) 10364.

