

Synthesis and characterization of the ligands 2-(1H-pyrrol-2-yl)-1H-benzimidazole and 2-pyridine-benzimidazole and their Fe(II) and Ru(II) complexes.

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Abstract

The ligands 2-(1H-pyrrol-2-yl)-1H-benzimidazole (bimpr) and 2-pyridine-benzimidazole (2-bzimpy) were synthesized using the Trofimov's synthesis^[1] and characterized by ¹H-NMR, UV-Vis and FT-IR spectroscopies and mass spectrometry. The purification was optimized substituting the benzene as mobile phase. The complexes [Ru(2-bzimpy)₃]²⁺ and [Fe(2-bzimpy)₃]²⁺ were synthesized and characterized by ¹H-NMR, UV-Vis and FT-IR spectroscopies and mass spectrometry.

Key words: benzimidazole, optimized purification, Ru(II) and Fe(II) complexes.

Introduction

The interest in ligands derived from benzimidazole is focused on the functionalization of them by electrophilic substitution in the benzylic ring. Polar or ionic groups may be introduced in order to form hydrogen bond or electrostatic attraction between the complex and any interesting molecule.^[2] This characteristic is essential for the application in sensors and catalysis using electroactive ions such Fe(II) and Ru(II). These complexes will behave differently due to different electronic structure, e.g. the strengths of ligand field.^[3]

Results and Discussion

The ligands were synthesized by a two-step reaction. In the first step, o-phenylenediamine and carboxaldehyde of interest were mixed and stirred for 30 min at room temperature. In the second step, the crude product was oxidized with air flow maintaining the temperature between 60°-70°C.^[1] The purification was optimized using toluene: dichloromethane (7:3) and chloroform as mobile phase for bimpr and 2-bzimpy, respectively, instead of benzene. Posteriorly to the filtration column, recrystallization from ethyl acetate was performed. The mass spectra proved the formation of ligands (Figure 1).

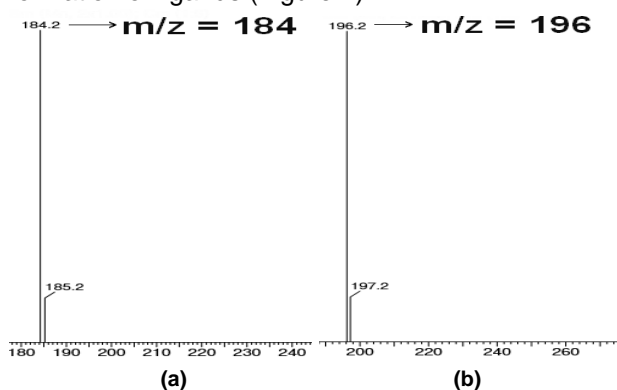


Figure 1. Mass spectra of (a) bimpr and (b) 2-bzimpy obtained in methanol and ESI+ mode.

The homoleptic complexes of Fe(II) and Ru(II) with the ligand 2-bzimpy were synthesized. The appearance of bands above 350 nm in the UV-Vis spectra confirmed the formation of them (Figure 2).

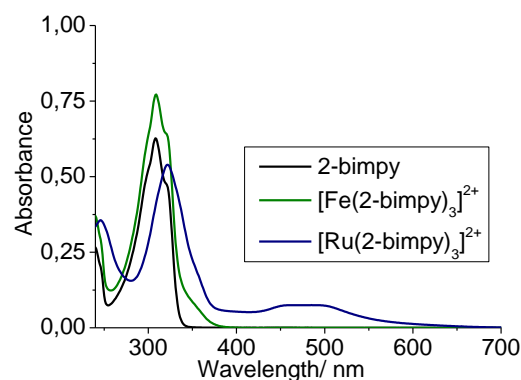


Figure 2. UV-Vis spectra of ligand 2-bzimpy and its complexes of Fe(II) and Ru(II) obtained in methanol.

Conclusions

The ligands bimpr, 2-bzimpy and the complexes [Fe(2-bzimpy)₃]²⁺ and [Ru(2-bzimpy)₃]²⁺ have their synthesis confirmed by ¹H-NMR, UV-Vis and FT-IR spectroscopies and mass spectrometry.

Acknowledgement

I would like to thank CNPq for the fellowship and support during this research, FAPESP and CAPES for financial support.

¹TROFIMOV, B.A. et al, *Synthesis*, 2009, 21, 3603-3610;

²McMURRY, J.E, *Organic Chemistry*, 8^a ed., CENGAGE Learning, 2008,972-977;

³HUHEEY, E.J.; KEITER, E.A.; KEITER, R.L., *Inorganic Chemistry*, 4^a ed., Harper Collings, 1993, 543-549.