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**DEPARTMENT OF CHEMISTRY** 

# **SYNTHESIS AND CHARACTERIZATION OF HYDRAZIDE MACROCYCLIC AND ACYCLIC COMPOUNDS DERIVED** FROM DIPICOLINIC ACID

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#### **Materials and methods**

#### Introduction

Pyridine-2,6-dicarboxylic acid (pdc) and derivatives can be used as polydentate ligands for preparation of coordination compounds or detection of harmful chemical species. Hydrazide derivatives of pdc are special group of compounds that have specific N,N,N-donor site which serves for binding of a specific solvents, gases, or metal cations [1]. Hydrazide derivatives of **pdc** can also be used for the synthesis of macrocyclic Schiff bases. Depending on the design of the macrocyclic ligand, prepared compounds can selectively bind certain chemical species, especially metal cations [2]. The aim of this research is preparation, structural and electrochemical characterization of two hydrazide compounds (1 (acyclic compound) and 2 (macrocyclic compound)) using cyclic voltammetry, IR and NMR spectroscopy and X-ray structural analysis.



Compound 1 was synthesized according to the previously reported method [1]. Compound 2 was prepared in 3 steps starting from pdc: In the first step, pdc ethyl-ester was prepared. In the second step, pdc-dihydrazine derivate was prepared. In the last step macrocyclic hydrazide was prepared by the reaction of pdc-dihydrazine and 2-[5-(2-formylphenoxy)pentoxy]benzaldehyde (Scheme 1). Thus prepared compounds were characterized by IR and NMR spectroscopy, and single-crystal X-ray diffraction. Electrochemical measurements were performed in a tris-buffer pH = 9. A three-electrode electrochemical cell was used with a glassy carbon (geometrical area 0.02 cm<sup>2</sup>) as a working electrode, Ag/AgCl (inner electrolyte solution 3 mol dm<sup>-3</sup> NaCl) as a reference electrode, and a platinum wire as a counter electrode. Conditions used for differential pulse voltammetry in our study were: step potential 5 mV, pulse amplitude 25 mV, pulse width 70 ms, and scan rate 5 mV/s.



Scheme 1. Three-step synthetic pathway for the preparation of compound 2.



Fig 1. Molecular structure of compound 1.



Fig 3. Cyclic voltammograms of compound 1(A) and compound 2 (B) in tris-buffer (pH = 9).



Fig 4. Differential pulse voltammograms of compound 1 (A) and compound 2 (B) in tris-buffer (pH = 9). 1<sup>st</sup> scan (—), 2<sup>nd</sup> scan (—), 3<sup>rd</sup> scan (—).

### Conclusions

The results of characterizations of the compounds by mentioned methods are in accordance with the proposed molecular formulas (1 and 2). Cyclic voltammetry showed that compound 1 is not electrochemically active. In contrast, compound 2 exhibits a

#### References

- [1] I. Đilović, M. Molnar, M. Komar, R. Dimitarević, I. Balić, T. Balić, Polyhedron 249 (2024) 116795.
- [2] I. Yilmaz, M. Kandaz, A. R. Özkaya, A. Koca, Monatsh. Chem. 133 (2002)

#### single irreversible oxidation current peak at the potential, $E_{p,a} = 0.86$ V. Differential pulse voltammetry showed that compound **1** is slightly adsorbed on the glassy carbon electrode surface. In contrast, the adsorption of compound **2** is more pronounced.

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