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SYNTHESIS AND CHARACTERIZATION OF THREE SULFUR-CONTAINED SCHIFF BASES

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ABSTRACT

Three sulfur-contained schiff-bases were synthesized in high yields. The structure of these compounds was characterized by IR, MS and NMR.

INTRODUCTION

Compared to other types of materials, schiff bases have many advantages, such as easy to prepare and purify, good coordination property (Das *et al*., 2013; Zhang *et al*., 2015; Zhang *et al*., 2012 and Li, 2015). Thus, this kind of compounds has a wide use in many fields. Sulfur-contained compunds showed good affinity to heavy metal ions, and attracted the interests of many scientists (Zhang *et al*., 2012; Zhang *et al*., 2011; Wu *et al*., 2012 and Yu, 2012). In this work, three sulfur-contained Schiff bases were synthesized and characterized (Figure 1).

Experimental Section

Reagents and Instruments

All of the materials were analytical reagent grade and used without further purification. IR spectra (KBr) were recorded on a Perkin–Elmer 1430 spectrometer. NMR spectra were measured with TMS as an internal standard. MS spectra were recorded on a Thermo TSQ Quantum Access Agillent 1100.

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Synthesis

General method: Compound 1a-c (1 mmol) and 2 (Zhang *et al*., 2012) (1 mmol) were mixed in ethanol (20 mL). The reaction mixture was stirred at 80 °C for 4 h, and then cooled to room temperature. The precipitate so obtained was filtered and dried in vacuum.

La: Yields 85.4%; IR: 3312 (N-H), 1053 (C=S); MS: M/z 302.31 $[M+H]^+$; ^{1}H NMR: ^{1}H NMR: 13.39 (s, 1H), 10.25 (s, 1H), 8.55 (s, 1H), 7.64 (t, 1H, J = 7.5), 7.38 (d, 2H, J = 8.5), 7.32 (t, 2H, J = 7.0), 7.27 (t, 2H, J = 7.75), 6.93 (d, 1H, J = 7.5), 6.89 (d, 1H, J = 7.6), 4.50 (s, 2H). ^{13}C NMR: 195.93, 157.70, 145.11, 137.23, 132.76, 129.71, 128.97, 127.73, 127.58, 120.10, 119.53, 116.84, 38.02.

Lb: Yields 86.7%; IR: 3312 (N-H), 1058 (C=S); MS: M/z 288.42 $[M+H]^+$; ^{1}H NMR: 13.51 (s, 1H), 8.62 (d, 1H, J = 5.0 Hz), 8.28 (s, 1H), 7.89 (d, 1H, J = 8.0 Hz), 7.85 (t, 1H, J = 8.5 Hz), 7.43 (d, 3H, J = 7.5 Hz), 7.34 (t, 2H, J = 7.5 Hz), 7.28 (t, 1H, J = 7.5 Hz), 4.50 (s, 2H). ^{13}C NMR: 197.99, 152.70, 150.27, 147.19, 137.51, 137.02, 129.75, 128.99, 127.77, 125.42, 120.64, 38.20.

Lc: Yields 84.8%; IR: 3316 (N-H), 1056 (C=S); MS: M/z

293.46 $[M+H]^+$; ^1H NMR: 13.33 (s, 1H), 8.43 (s, 1H), 7.70 (d, 1H, *J* = 5.0 Hz), 7.54 (s, 1H), 7.42 (d, 2H, J = 7.5), 7.33 (t, 2H, *J* = 7.5 Hz), 7.27 (t, 1H, *J* = 7.3 Hz), 7.14 (t, 1H, *J* = 9.0 Hz), 4.49 (s, 2H). ^{13}C NMR: 196.14, 142.13, 138.29, 137.35, 133.18, 130.60, 129.70, 128.95, 128.75, 127.69, 38.00.

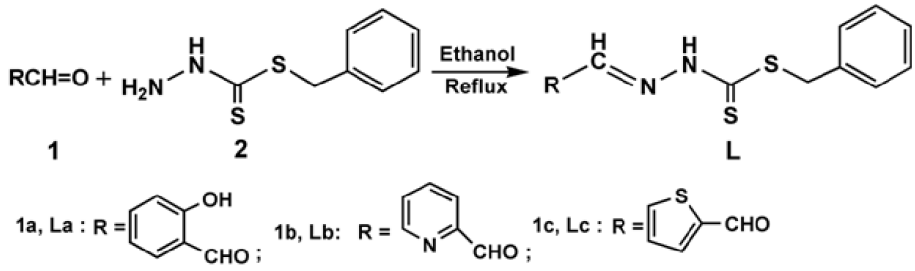

Figure 1. Synthesis route of the proposed Schiff bases

RESULTS AND DISCUSSION

The design and synthesis of sulfur-contained schiff bases has been developing rapidly because of its applications in the field of coordination chemistry. In this experiment, a series of schiff bases La-c were synthesized in a simple route with high yields.

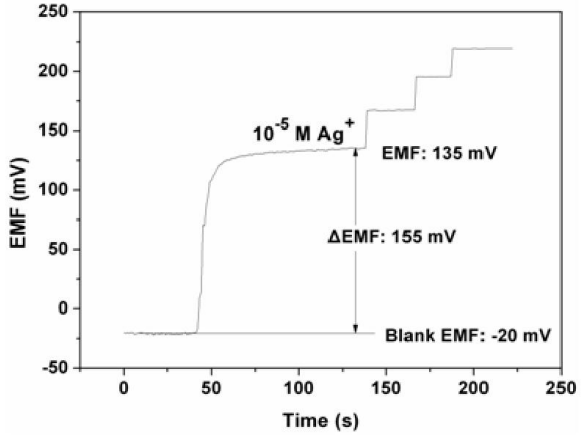

Figure 2. Selectivity of La

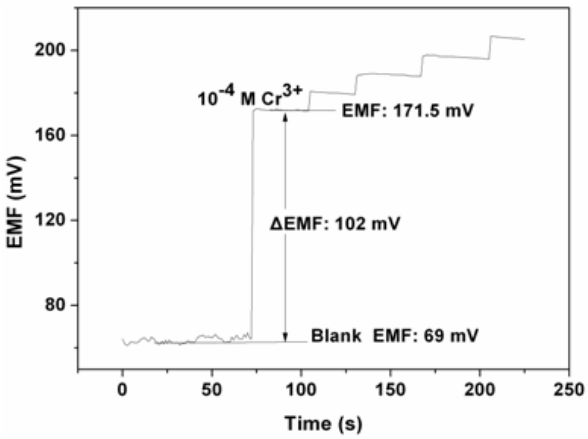

Figure 3. Selectivity of Lc

The Ms spectrum confirmed the formation of target moleculars, and the IR and NMR data gave a more detailed information of the proposed schiff bases, which also proved the obtaintion of the target schiff bases. The coordination property of these three compounds was also studied using ion-selective electrode method. The results showed that La has good affinity to Ag^+ (Figure 2), Lc has good selectivity to Cr^{3+} (Figure 3).

This provides some useful information for the synthesis of new ionophore for the construction of ion-selective electrodes.

Conclusions

In summary, a series of schiff bases were presented in this work. The conception may expand a promising approach to develop new ionophore for the construction of ion-selective electrodes.

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