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Cent. Eur. J. Chem. • 8(4) • 2010 • 946-952
DOI: 10.2478/s11532-010-0062-7

Central European Journal of **Chemistry**

Spectroscopic evaluation of chiral and achiral fluorescent ionic liquids

Research Article

Hadi M. Marwani*

Department of Chemistry, King Abdulaziz University, Jeddah 21589, Saudi Arabia

Received 08 January 2010; Accepted 13 April 2010

Abstract: In this study, spectroscopic investigation of chiral and achiral room temperature ionic liquids is achieved. New ionic liquids were prepared *via* metathesis, accomplished by the reaction of either L-phenylalanine ethyl ester hydrochloride, chlorpromazine hydrochloride or 1,10-Phenanthroline monohydrate hydrochloride with lithium bis(trifluoromethane) sulfonamide in water. The resulting ionic liquids were produced in high yield and purity. The results obtained by use of ^{1}H NMR and IR experiments were in very good agreement with the chemical structures of the synthesized ionic liquids. In addition, the results of thermal gravimetric analysis suggested that these ionic liquids have good thermal stability. UV-Vis and fluorescence spectroscopy measurements indicated that these ionic liquids are strongly optically absorbent and fluorescent. Lastly, time-based fluorescence steady-state measurements demonstrated the high photostability of these ionic liquids.

Keywords: *Ionic liquids • Metathesis reaction • NMR • UV-Vis • Fluorescence*

1. Introduction

One of the most pressing industrial problems is the dependence of nearly all chemical plants on toxic, hazardous, and flammable organic solvents [1]. However, recently discovered room temperature ionic liquids (RTILs) show promise as a replacement for volatile organic compounds. There is evidence that ionic liquids (ILs) are exceptional and versatile solvents in a host of analytical and chemosensing applications [2,3]. ILs have also generated interest in environmental areas because of their potential as greener solvents for many organic, inorganic, and polymeric substances as compared to environmentally damaging organic solvents [4]. The good thermal stability, high ionic conductivity, miscibility with other solvents, negligible vapor pressure, and the non-reactive and recyclable nature of ILs are a few other properties that make these compounds appropriate solvents for diverse applications [5-7]. RTILs belong to a class of potentially benign solvents that exist as molten salts under ambient conditions [8]. Both ILs and molten salts are composed of ions. The presence of bulky organic cations in ILs interrupts the crystal packing and lowers the melting temperature.

RTILs have potential for many different applications, including catalysis and synthesis. For instance, conventional organic solvents have been replaced by ILs in organic synthesis [9]. They have also been used in solvent extractions [10], liquid-liquid extractions [11], enzymatic reactions [12], pharmaceutical studies [13], electrochemical studies [14], dye-sensitized solar cells and batteries [15], and as buffer additives in capillary electrophoresis [16], stationary phases in gas-liquid chromatography [17], and ultralow volatility liquid matrixes for matrix-assisted laser desorption/ionization (MALDI) mass spectrometry [18]. In addition, their high thermal stability allows for their use in high temperature gas sensing [19].

Chiral ILs have been used as chiral selectors in different applications because of their chiral recognition abilities [13,20-22]. It has also been found that chiral ILs can be good alternatives for other chiral selectors, such as, cyclodextrins, molecular micelles, antibodies and crown ethers. The use of other chiral selectors is limited because of their low solubility, difficult organic syntheses, instability at high temperature, and high cost. However, chiral ILs are at a preliminary stage of development, and although there have been many

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* E-mail: hmarwani@kau.edu.sa

publications on ILs, only a few examples of chiral ILs have been reported. In addition, the synthesis of reported chiral ILs has always involved expensive reagents and elaborate synthetic schemes [23,24]. For example, the preparation of the chiral imidazolium cation through Diels-Alder reactions by Howarth *et al.* [25] required an expensive chiral alkylating agent. In addition, the most commonly synthesized RTILs are based on alkylammonium, alkylphosphonium, *N*-alkylpyridinium, and *N,N'*-dialkylimidazolium cations with various anions [4,26]. Finally, a great deal of evaluation and attention has been focused either on one liquid or one class of ionic liquids, particularly imidazolium salts. Therefore, there is an increasing interest for synthesis and investigation of new chiral and achiral ILs.

Recently, there has been a large focus on the effectiveness of ILs as environmentally friendly, “green” solvents and on the hostof practical applications to which they are amenable [17,27]. In addition, understanding the mechanism of solvation in ILs is a difficult and challenging task because of their complex nature [28,29]. Several studies have focused on this topic and a series of analytical techniques have been applied to explore the properties of these novel media [30-33]. Most analytical methods involve fast atom bombardment (FAB) mass spectrometry [34], X-ray crystallography [35], and X-ray absorption fine structure (XAFS) [36]. These methods have shown considerable promise in studying the physicochemical properties of RTILs. However, there are several problems associated with these techniques and their application to IL study (*e.g.* the ability of ILs to dissolve most media, the production of uniform vertical liquid films, and the high levels of absorption/self-absorption when using XAFS).

In addition, several studies have been focused on exploring the spectroscopic behavior of ILs in association to their structure [37-40]. For example, electronic absorption and fluorescence techniques have shown considerable promise in studying the photophysical properties of RTILs. Fluorescence spectroscopy is one of the most widely used, dominant, and powerful methods for obtaining information on the structure and function of many molecules [41]. Many approaches have examined the optical behavior of several substances in these media by use of fluorescence measurements [42-44]. These studies have provided information on the polarity of the RTILs, dynamics of ionic diffusion, and internal motion of the dissolved solutes. However, although the optical behavior of a considerable number of dissolved solutes in the presence of these RTILs has been studied, the optical properties of the RTILs themselves have not yet been carefully investigated.

The current study introduced the synthesis and spectroscopic evaluation of new chiral and achiral ILs via a simple and straightforward route. Characterization of these ILs was accomplished by use of ^{1}H NMR, IR, UV-Vis spectroscopy, differential scanning calorimetry (DSC), thermal gravimetric analysis, and elemental analysis. Furthermore, fluorescence measurements of these RTILs were obtained, allowing for a deeper mechanistic understanding of ILs.

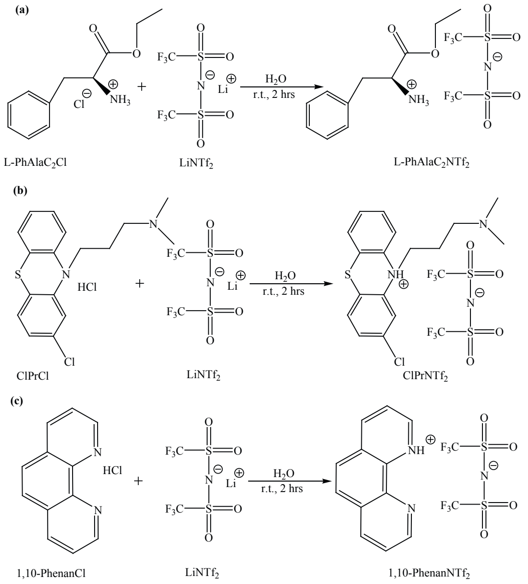
2. Experimental Procedure

2.1. Reagents and chemicals

L-Phenylalanine ethyl ester hydrochloride (L-PhAlaC$_2$Cl) was obtained from Sigma-Aldrich (Milwaukee, WI). Chlorpromazine hydrochloride (ClPrCl), 1,10-phenanthroline monohydrate hydrochloride (1,10-PhenanCl), and lithium bis (trifluoromethane) sulfonamide (LiNTf$_2$) were also purchased from Sigma-Aldrich.

2.2. Synthesis Procedures

All product ILs were prepared according to procedures previously reported by Bonhôte *et al.* [37]. Specifically, 1 g of the desired salt (L-PhAlaC$_2$Cl, ClPrCl, or 1,10-PhenanCl) was separately dissolved in 18.2 MΩ cm distilled deionized water and mixed with an equimolar amount of LiNTf$_2$. The resultant reaction mixture was stirred for 2 h at room temperature. The reaction resulted in two layers. The lower layer was separated and dried under vacuum overnight (Scheme 1). The purification by vacuum drying was performed at a drying temperature of 353 K and vacuum of 0.4 kPa.



Scheme 1. Synthesis of (a) L-PhAlaC$_2$NTf$_2$, (b) ClPrNTf$_2$, and (c) 1,10-PhenanNTf$_2$.

2.3.Sample Preparation

A stock solution of each ionic liquid was prepared individually in ethanol by dissolving an appropriate amount of the ionic liquid. All stock solutions were stored in the dark at 4°C. For both fluorescence and UV-Vis spectroscopic study, standard solutions of 10 μM L-PhAlaC$_2$NTf$_2$ and ClPrNTf$_2$ were prepared by adding appropriate amounts of IL stock solution. In addition, a standard solution of 1 μM 1,10-PhenanNTf$_2$ was prepared as above.

2.4. Apparatus

One-dimensional ^{1}H NMR spectra were acquired in the single-pulse mode on a Perkin Elmer 600 MHz spectrometer. All NMR measurements were recorded in CDCl$_3$ by use of tetramethyl silane (TMS) as an internal standard. ^{1}H NMR spectra were collected using a 7 μs pulse, 16 scans, and 1 s delay between scans. The spectra were processed by zero filling the FID from 16 to 32K and applying a line-broadening of 30 Hz. Thermal infrared (IR) spectra were recorded on a Shimadzu IR 470 spectrophotometer in the range 4000-600 cm^{-1}. The melting point (T$_m$) was determined by differential scanning calorimetry (DSC) using a thermal analysis instrument TA SDT2960 at a scanning rate of 5°C min^{-1}. Thermal gravimetric analyses (TGA) were performed using a thermal analysis instrument TGA-7 HR V6.1A (module TGA 1400°C). Determination of the thermal decomposition temperature (T$_{dec}$) for ILs was obtained at a heating rate of 5°C min^{-1} under nitrogen from 20 to 600°C. In addition, the UV/Visible absorption measurements were collected by use of a Perkin-Elmer UV-Vis scanning spectrophotometer. Absorption spectra were collected using a 10 mm quartz cuvet. Fluorescence measurements were performed using a Perkin-Elmer luminescence spectrofluorometer equipped with a 20-kW for 8-μs-duration xenon lamp, a gated photomultiplier tube (PMT), and a red-sensitive R928 PMT detector. All fluorescence measurements were collected at room temperature. The emission spectra of ILs were recorded in a 10 mm quartz fluorescence cuvette with slit widths set for entrance and exit bandwidths of 5 and 5 nm for the excitation and emission monochromators, respectively. Fluorescence emission spectra of ILs were recorded at 258 nm excitation wavelength for both L-PhAlaC$_2$NTf$_2$ and 1,10-PhenanNTf$_2$ and at 315 nm for ClPrNTf$_2$. All fluorescence spectra were blank subtracted before proceeding in data analyses. Time-based fluorescence steady-state experiments used to evaluate the photostability of ILs were acquired with excitation and emission bandpass set at 15 and 5 nm, respectively, in order to increase photobleaching. The excitation and emission wavelengths were set at 258 and 302 nm for L-PhAlaC$_2$NTf$_2$, 315 and 450 nm for ClPrNTf$_2$, and 258 and 370 nm for 1,10-PhenanNTf$_2$, respectively. The fluence level of the excitation source was open for a period of 30 min.

3. Results and Discussion

3.1. Characterization of ILs

ILs obtained via metathesis reaction were all liquids at room temperature, except for 1,10-PhenanNTf$_2$. The final products of L-PhAlaC$_2$NTf$_2$, ClPrNTf$_2$, and 1,10-PhenanNTf$_2$ were green, red, and pink in color, respectively. The ClPrNTf$_2$, however, was extremely more viscous in comparison to L-PhAlaC$_2$NTf$_2$. The physical properties of these ILs are tabulated in Table 1. The ^{1}H NMR and IR spectra (see Supplementary Figs. 1 and 2) of synthesized ILs were consistent with their chemical structure. In addition, synthesized ILs are found to be pure from data obtained by the elemental analysis.

L-PhAlaC$_2$NTf$_2$. This reaction shown in Scheme 1a produced 1.65 g (80% yield) of green room-temperature ionic liquid. ^{1}H NMR δ (ppm): 1.13 (t, 3H, CH$_3$), 3.15 (d, 2H, CH$_2$), 4.05 (q, 2H, CH$_2$O), 4.20 (t, 1H, CH), 5.15 (s, 3H, NH$_3$), 7.10 (d, 2H, ArH-2 and H-6), 7.23 (m, 3H, ArH-3, H-4 and H-5). Molecular species are simply identified by their characteristic absorbances *v* (cm^{-1}): 3572, 3166 (NH); 1738 (CO); 1613, 1506 (C=C); 1452, 1274 (C—S); 1340, 1188 (NSO$_2$); 856, 792, 744 (C—H). Elemental Anal. Calcd. for $C_{13}H_{16}N_2O_6S_2F_6$: C (32.90); H (3.38); N (5.91). Found: C (32.40); H (3.24); N (5.55).

The decomposition process of L-PhAlaC$_2$NTf$_2$ is complicated. This chiral ionic liquid shows two distinct decomposition stages, as illustrated in Fig. 1a. It initially starts to decompose at 250°C, and 70% weight loss is observed up to 400°C. The main gaseous products of this stage may correspond to CO$_2$, NH$_3$, H$_2$O, and benzeneethanamine. A subsequent decomposition of the residue occurs above 400°C.

However, a similar behavior was previously reported on phenylalanine by Jie *et al.* [45] using TG-FTIR. They concluded that the decarboxylation process takes place *via*

Table 1. Physical properties of L-PhAlaC$_2$NTf$_2$, ClPrNTf$_2$, and 1,10-PhenanNTf$_2$

ILs Product	Color	State at rt	Yield (%)	T$_{dec}$ (°C)
L-PhAlaC$_2$NTf$_2$	Green	Liquid	80	250 and 400
ClPrNTf$_2$	Red	Liquid	91	232
1,10-PhenanNTf$_2$	Pink	Solid	90	336

The symbols rt and T$_{dec}$ represent room and decomposition temperatures, respectively.

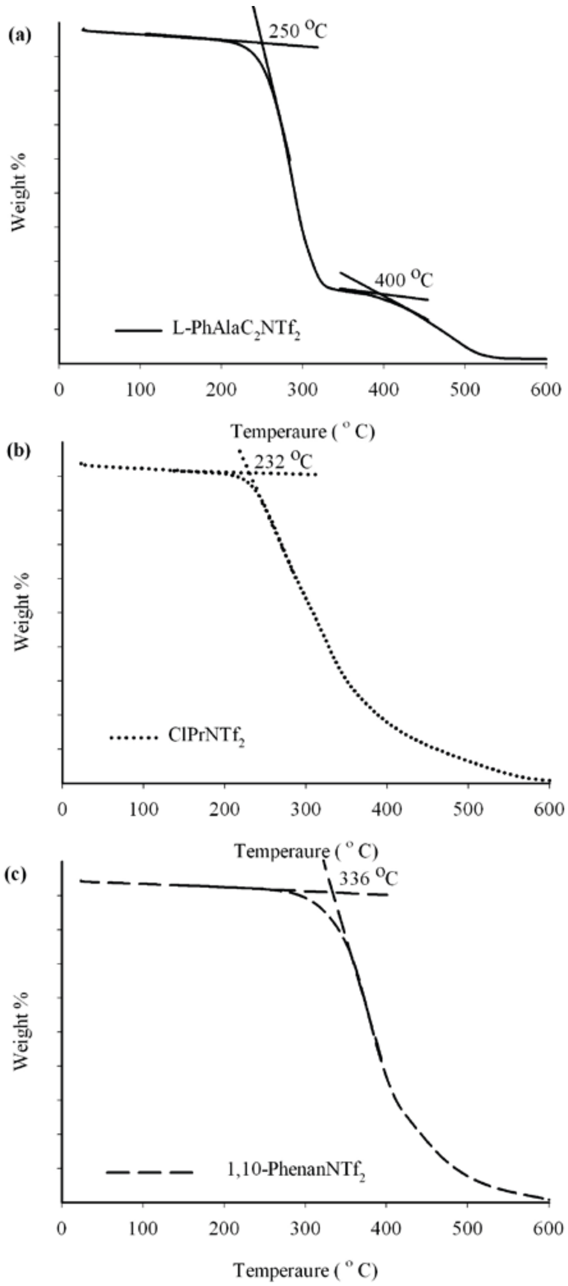


Figure 1. Thermal gravimetric analysis of (a) L-PhAlaC$_2$NTf$_2$, (b) ClPrNTf$_2$, and (c) 1,10-PhenanNTf$_2$, obtained at a heating rate of 5°C min^{-1} under nitrogen from 20 to 600°C.

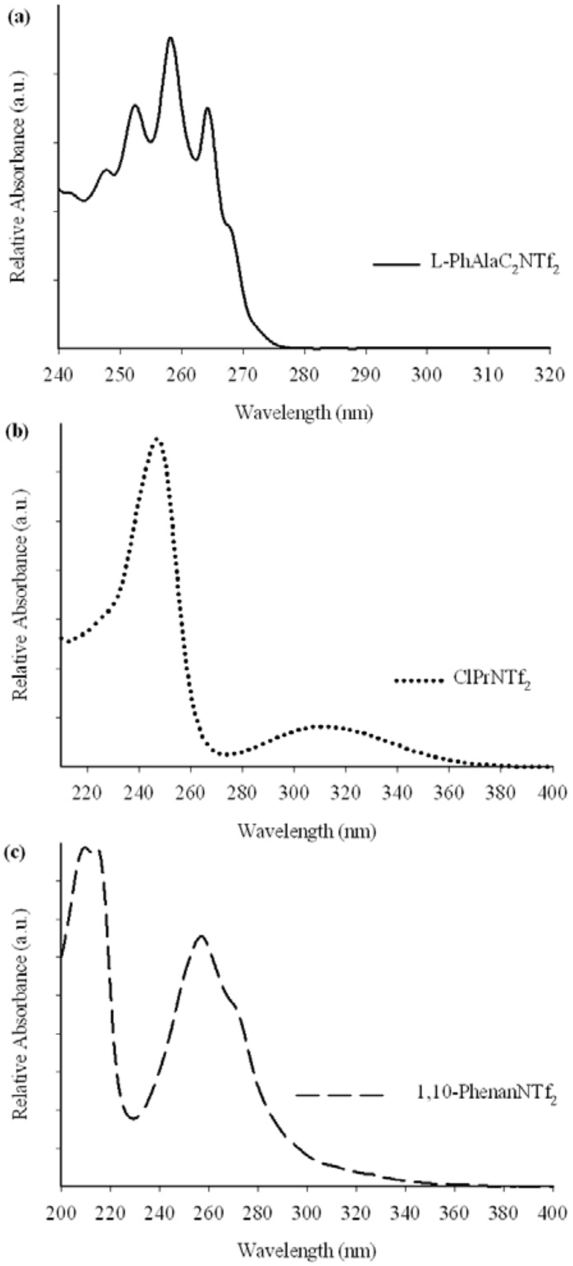


Figure 2. UV-Vis absorption spectra of 10 μM (a) L-PhAlaC$_2$NTf$_2$, (b) ClPrNTf$_2$, and 1 μM (c) 1,10- PhenanNTf$_2$ in ethanol.

two competing pathways. One is direct decarboxylation, resulting in benzeneethanamine, and the other is the concerted rupturing of the carbon chain to produce toluene.

In the first stage, they observed that the characteristic absorbance of CO_2 is the strongest as compared to those of NH_3 and H_2O. Thus, they suggested that the main primary decomposition step of phenylalanine is decarboxylation which produces CO_2 and benzeneethanamine, while dehydration and deamination are minor. For the second stage, they indicated toluene, accompanied by CO_2 formation, is the main organic product instead of benzeneethanamine.

ClPrNTf$_2$. This ionic liquid was red in color, and 1.54 g (91% yield) was produced. ClPrNTf$_2$ was, however, extremely more viscous at room temperature as compared to L-PhAlaC$_2$NTf$_2$. ^{1}H NMR δ (ppm): 2.00 (q, 2H, CH_2NH), 2.60 (m, 2H, CH_2), 3.05 (q, 2H, CH_2NH), 5.15 (s, 1H, NH), 6.70-7.10 (m, 7H, Ar-H), 7.50

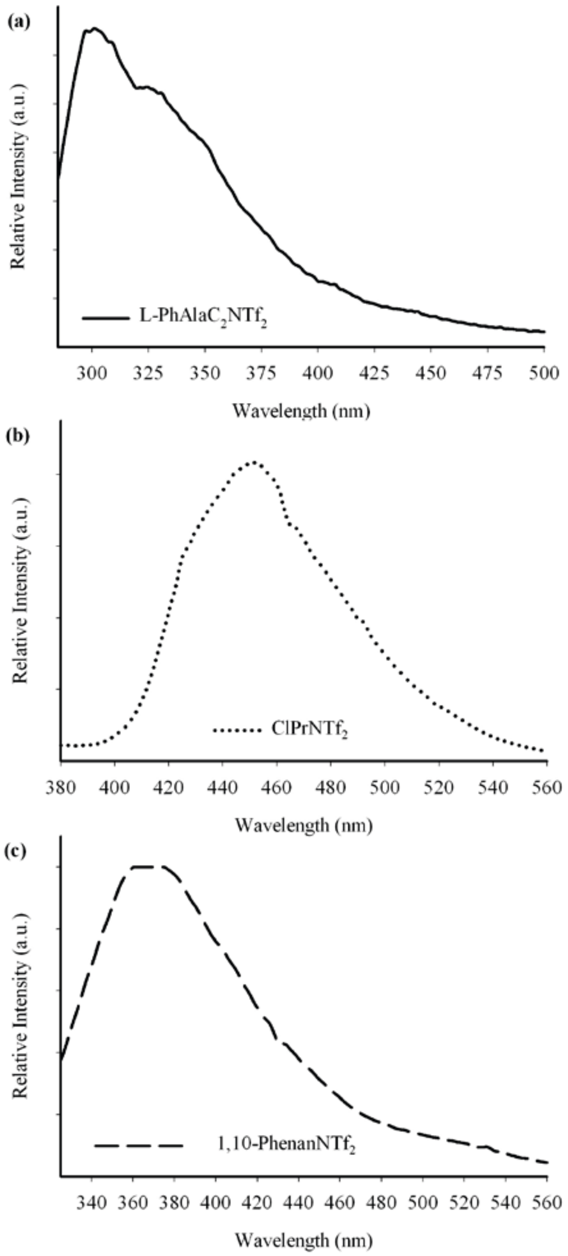


Figure 3. Fluorescence emission spectra of 10 μM (a) L-PhAlaC$_2$NTf$_2$, (b) ClPrNTf$_2$, and 1 μM (c) 1,10-PhenanNTf$_2$ in ethanol, excited at 258 nm for both L-PhAlaC$_2$NTf$_2$ and 1,10-PhenanNTf$_2$ and at 315 nm for ClPrNTf$_2$.

(s, 1H, NH). IR absorbances *v* (cm^{-1}): 3580, 3147 (NH); 1591, 1567 (C=C); 1457, 1276 (C—S); 1345, 1186 (NSO$_2$); 854, 794, 738 (C—H). Elemental Anal. Calcd. for $C_{19}H_{20}N_3ClO_4S_3F_6$: C (38.02); H (3.33); N (7.00). Found: C (37.32); H (3.18); N (6.39). This compound was thermally stable up to 232°C (Fig. 1b).

1,10-PhenanNTf$_2$. The reaction in Scheme 1c produced an ionic solid at room temperature that was pink in color. The final product obtained was 1.83 g (90% yield) with a melting point (T_m) of 134°C. ^{1}H NMR δ (ppm): 7.50 (d, 1H, H-6), 7.60 (d, 1H, H-5), 7.83 (s, 1H, NH), 7.88 (m, 2H, H-3 and H-8), 8.30 (d, 1H, H-7), 8.67 (m, 2H, H-4 and H-9), 9.20 (t, 1H, H-2). IR characteristic absorbances *v* (cm^{-1}): 3335 (NH); 1598, 1544 (C=C); 1424, 1284 (C—S); 1342, 1173 (NSO$_2$); 844, 770, 708 (C—H). Elemental Anal. Calcd. for $C_{14}H_{11}N_3O_5S_2F_6$: C (35.06); H (2.30); N (8.77). Found: C (34.26); H (2.18); N (8.05). The TGA measurement of this ionic liquid gave a decomposition temperature (T_{dec}) of 336°C, as shown in Fig. 1c. It is interesting to note that 1,10-PhenanNTf$_2$ initially starts to decompose at the highest decomposition temperature as compared to other ILs investigated in this study. This may be ascribed to the distinctive structure composition of 1,10-PhenanNTf$_2$ in comparison to other ILs. 1,10-PhenanNTf$_2$ is constituted of three fused benzene rings and contains no other functional groups.

3.2. Spectroscopic evaluation of ILs

3.2.1. UV-Vis and fluorescence spectroscopic study

As shown in Fig. 2a, three characteristic bands appeared in the UV-Vis absorption spectrum of L-PhAlaC$_2$NTf$_2$. These three intense bands are centered at 253, 258, and 264 nm. The absorption maximum was observed at 258 nm. In contrast, two broad bands were observed at 248 and 315 nm in the UV-Vis absorption spectrum of ClPrNTf$_2$ (Fig. 2b). The absorption maximum of ClPrNTf$_2$ was observed at 248 nm. The absorption spectrum of 1,10-PhenanNTf$_2$ also shows two bands at 214 and 258 nm, and the absorption maximum appeared at 214 nm, as displayed in Fig. 2c.

The optical behavior of all ILs included in this study not only indicates that these ILs do have significant absorption in the UV region, but it also brings to light very interesting fluorescence behavior. In general, high background fluorescence was observed in the emission spectra of these ILs (Fig. 3). These results further support the purity of synthesized ILs and are in good agreement with the data obtained from the elemental analysis. However, close examination of Figs. 2 and 3 indicates that these ILs absorb in the UV region and exhibit fluorescence that covers a large part of the visible region. This may lead to the limitations that these ILs may have in optical studies of dissolved solutes.

3.2.2. Photostability study

The photostability of all ILs included in this study was evaluated by use of time-based fluorescence measurements (Fig. 4). The excitation and emission bandpass was set at 15 and 5 nm, respectively, and all ILs were exposed to the maximum amount of irradiation for 30 min in order to induce photobleaching. However, there was no decrease in the fluorescence intensity of any of the ILs with an increase in the exposure time, as displayed in Fig. 4. This suggests that these ILs have a very high stability against photobleaching. The study of photostability is also in agreement with data obtained from thermal gravimetric analysis.

4. Conclusions

In this study, a successful synthesis of a series of novel ILs using a straightforward metathesis reaction is achieved. L-PhAlaC$_2$NTf$_2$ and ClPrNTf$_2$ are, desirably, liquids at room temperature and have high thermal stability. As a result, these ILs can be used in gas chromatography or in high temperature reactions. Additionally, L-PhAlaC$_2$NTf$_2$ is a chiral ionic liquid and can, therefore, serve both as solvent and chiral selector, alleviating the need to use environmentally toxic, hazardous, and flammable organic solvents to dissolve analytes. This ionic liquid could possibly be used as a chiral selector in the determination of enantiomeric composition of pharmaceutical products and in chiral separations. The resulted ILs are also found to be strongly optically absorbent, fluorescent, and very photostable. Finally, the preparation and spectroscopic evaluation of these ILs represent a step towards exploring their potential applications.

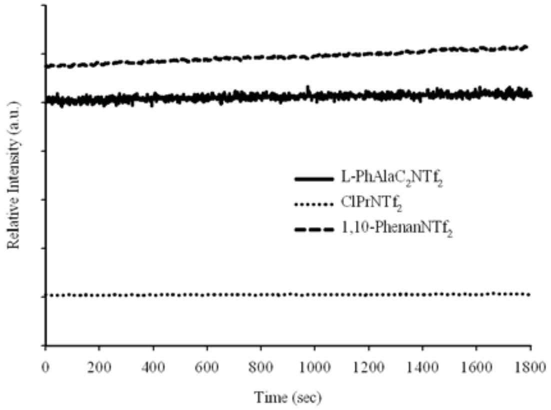


Figure 4. Time-based fluorescence steady-state measurements of 10 μM L-PhAlaC$_2$NTf$_2$, ClPrNTf$_2$, and 1 μM 1,10-PhenanNTf$_2$ in ethanol, acquired with excitation and emission bandpass set at 15 and 5 nm, respectively.

Acknowledgments

The author acknowledges King Abdulaziz University Research Center for its support. The author also thanks Prof. Hassan M. Faidallah for helpful discussions regarding this study.

Supporting Information

Additional ^{1}H NMR and IR data of L-PhAlaC$_2$NTf$_2$, ClPrNTf$_2$, and 1,10-PhenanNTf$_2$ are provided.

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