



Influence of molecular structure on the photoluminescence of 2-methyl benzimidazolium picrate: A new fluorescent material



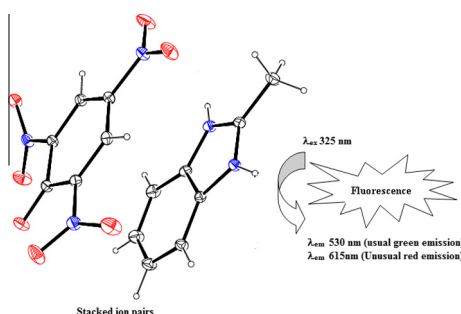
M. Obulichetty, D. Saravanabharathi*

Department of Chemistry and Applied Chemistry, PSG College of Technology, Coimbatore 641 004, India

HIGHLIGHTS

- 2-Methyl benzimidazolium picrate crystals were grown and characterized.
- Rare heteromolecular stacking of ion pairs and $n \rightarrow \pi$ interaction were identified.
- Spectral characterizations support the structural features.
- Stacking of ions causes red emission of the material upon UV excitation.

GRAPHICAL ABSTRACT



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ABSTRACT

This work reports the structural and photo luminescent characterization of the 2-methyl benzimidazolium picrate salt for the first time. This new material exhibits the π - π stacking of aromatic rings involving the benzimidazolium cation and picrate anion, which is additionally supported with a rare $n \rightarrow \pi$ interaction as well as with the extended networks of intermolecular hydrogen bonds in the solid state. The FT-IR and UV characterizations further support the solid state structural features of the salt. However, the spectroscopic results reveal that the stacking of ion pairs, which is observed in the solid state, is not maintained in solution. The DSC result indicates that the material is stable under ambient temperature conditions. Interestingly, upon photo excitation at 325 nm in the solid state, the material shows an unusual red emission around 615 nm, which is probably attributed to the supramolecular stacked nature of the ion pairs, along with a usual picrate centered green emission at 530 nm. However, the fluorescence measurement in solution, wherein the stacking of ion pairs are not maintained, shows only a single peak at lower wavelength. These observations highlight the influencing role of the supramolecular stacking interactions on the photoluminescence of the material, due to which, a chemical sensing action may be envisaged.

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Introduction

Crystalline picrates of organic compounds [1–6] have attracted a great deal of interest in the recent past owing to their applications in various fields such as molecular recognition, selective guest absorption [1–3], bio – mimicking models [1–3], magnetism

[3], second harmonic generation [3], fluorescence emission [1,3] solar energy harvesting [4] etc. It is well known that the picric acid, apart from being a Lewis acidic electron acceptor, can also function as a Brønsted acidic proton donor and hence its acidity could be induced either way depending upon the nature of the partners involved in the reactions [1,6]. For example, with π donating aromatic hydrocarbons, like naphthalene, or with weakly basic aromatic amines, particularly when the ΔpK_a of the reactants are less than 3.8, only the Lewis acidity of the picric acid is exemplified

* Corresponding author. Tel.: +91 9443220623.

E-mail address: dsbharathi@yahoo.com (D. Saravanabharathi).

with the formations of π - π stacked charge transfer (CT) molecular complexes [1,5]. On contrary, with the strong organic heterocyclic bases, the *Brønsted* acidity of the picric acid is readily shown up with the proton transfer from the -OH of the acid to the basic nitrogen centers, resulting in the formations of ionic salts with distinct anion-cation pairs [1,6–8]. It is to be noted that the organic picrates, although invariably exhibit extensive network of hydrogen bonds in the solid state, do not show the overall planar stacking interactions of the anion-cation pairs very regularly, except in few cases of ionic heterocyclic picrates [7–11]. Such materials have become attractive, not only due to their fascinating structural topologies [12], but also to their interesting optical functions, which result from the delocalized nature of orbitals and the non centrosymmetric manner of the crystal packing in the solid state [13–15]. In this context, the picrates of the benzimidazole family of compounds are also interesting and need to be scrutinized.

As the crystalline benzimidazolium picrates are scanty in the literature [7,16], this paper reports the synthesis, growth and characterizations of 2-methyl benzimidazolium picrate crystal. In fact, this is the first report, which highlights the influencing role of the supramolecular structure on the photoluminescence of the crystalline benzimidazolium picrates.

Experimental

Synthesis and characterizations

2-Methylbenzimidazole was prepared and purified according to the known procedure [17] using commercially available 1,2 phenylenediamine (Thomas Backer) and acetic acid (Ranbaxy). Picric acid (Ranbaxy) was purchased and used without further purification. The salt was obtained as a yellow precipitate by mixing the methanolic solutions of the precursors in 1:1 stoichiometric ratio (0.003 mol in 20 ml). The precipitated compound was filtered off, air-dried and dissolved in a minimum amount of acetone, which on slow evaporation yield bulkier crystals of centimeter dimension. The ^1H NMR of the material in acetone showing signals at δ = 8.78 (s, 2H, picrate), 7.91 (m, 2H, benzimidazolium – aromatic), 7.57 (m, 2H, benzimidazolium – aromatic), 3.06 (s, 3H, benzimidazolium – methyl) confirms the occurrence of a neat reaction. Recrystallization of the powdered sample in methanol has offered the X-ray quality single crystals. The purity of the single crystals was ascertained with the elemental analysis. [Calculated for $\text{C}_{14}\text{H}_{11}\text{N}_5\text{O}_7$ is C = 46.53%, H = 3.04%, N = 19.39%; Found: C = 46.50%, H = 3.09%, N = 19.54%].

The FT-IR spectra was recorded on a Shimadzu 8400S FT-IR spectrophotometer as a KBr pellet. The UV-Visible spectra was recorded using Shimadzu UV -1700 spectrophotometer. The ^1H NMR was examined using Bruker AV III – 400 MHz instrument. The elemental analysis was performed on Thermo finnigan, Flash EA 1112 CHN analyzer. Fluorescence measurement was carried out on the powdered sample using the Jobin Yvon Fluorimeter instrument. The DSC study was performed in a Pyris 6 DSC instrument with a heating rate of 10 °C/min.

Crystallographic studies

X-ray diffraction and data collection was performed on a Bruker Axs Kappa Apex 2 CCD Diffractometer. The structure was solved by direct method with SHELXS-97 and refined using SHELXL-97 [18]. The non-hydrogen atoms were refined with anisotropic displacement parameters. The C bound hydrogen atoms were placed at calculated positions and were treated as riding on their parent atoms. A summary of crystallographic data and refinement parameters are given in Table 1.

Table 1

Crystallographic data for 2-methyl benzimidazolium picrate.

Empirical formula	$\text{C}_{14}\text{H}_{11}\text{N}_5\text{O}_7$
Formula weight (g mol^{-1})	361.28
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, $\text{P2}_1/\text{n}$
Unit cell dimensions	$a = 9.147(5)$ Å, $\alpha(^{\circ}) = 90.000(5)$ $b = 13.916(5)$ Å, $\beta(^{\circ}) = 100.321(5)$ $c = 11.961(5)$ Å, $\gamma(^{\circ}) = 90.000(5)$
Volume	$1497.9(12)$ Å ³
Z, Calculated density	4, 1.602 Mg/m ³
Absorption coefficient	0.132 mm^{-1}
F(000)	744
Crystal size	$0.20 \times 0.20 \times 0.20 \text{ mm}$
Theta range for data collection	2.27 to 25.00 deg.
Limiting indices	$-10 \leq h \leq 10$, $-16 \leq k \leq 16$, $-12 \leq l \leq 14$
Reflections collected/unique	13865/2634 [R(int) = 0.0306]
Completeness to theta = 25.00	100.0%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9979 and 0.9310
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2634/0/245
Goodness-of-fit on F^2	1.019
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0424$, $wR2 = 0.1071$
R indices (all data)	$R1 = 0.0541$, $wR2 = 0.1172$
Extinction coefficient	0.0089(12)

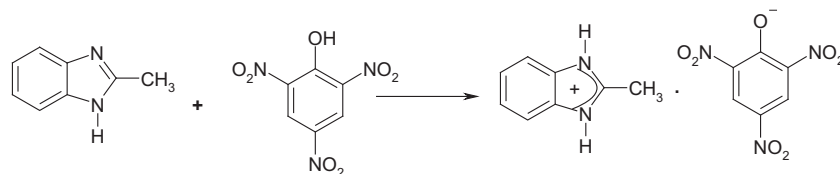
Results and discussion

Synthesis and X-ray structural investigations

The salt was readily prepared as a yellow powder in a near quantitative yield by simply mixing the methanolic solutions of the precursors in 1:1 stoichiometry (Scheme 1). It has also been found that the powdered sample can be easily grown either as bulkier crystals of centimeter dimension (Fig. S1 A) or as fine single crystals, by simply changing the medium from acetone to methanol respectively. The integrity of re crystallized sample was ascertained through elemental analysis and by its ^1H NMR spectrum in acetone- d_6 .

The molecular structure of the salt is shown in Fig. 1. The constituents of the asymmetric unit possess all the relevant structural features of a picrate anion and a protonated benzimidazolium cation, indicating an acid-base reaction between the precursors. As seen in the other structural reports [19], the delocalized nature of the constituents is discernible through the bond lengths, angles of the near planar aromatic rings of the system (Table S1). Interestingly, the pair of ions present in the asymmetric unit shows the π - π stacking interactions involving the six membered rings of picrate anion and the benzimidazolium moiety (Fig. 1 and (Fig. S2)) such that the inter planar centroid-centroid distance between the stacking is in the range of 3.6 Å, which is well within the characteristic magnitude of the CT complexes [20]. The rings of the stacks are twisted against each other with an angle of 22° and resemble the usual values of the stacked aromatic rings [5,20].

In addition, the overlap of the imidazolium five membered ring of the cation with the para nitro group of the picrate anion deserves attention (Fig. S3). It is interesting to note that the carbon atom (C8) of the five membered segment of the cation overlaps the nitrogen (N1) of the para nitro group of the anion, with a non bonded C8--N1 distance at 3.38 Å, which is very similar in pattern to those found in the structures of serotonin picrate [21] and in the supramolecular tapes of bis-benzimidazoles [22], wherein such interactions play crucial roles in the assembly of solid state structures. The observed non bonded distance, although is not within the sum of the van der Waals radii of the atoms involved, is



Scheme 1. Synthesis of the 2-methyl benzimidazolium picrate.

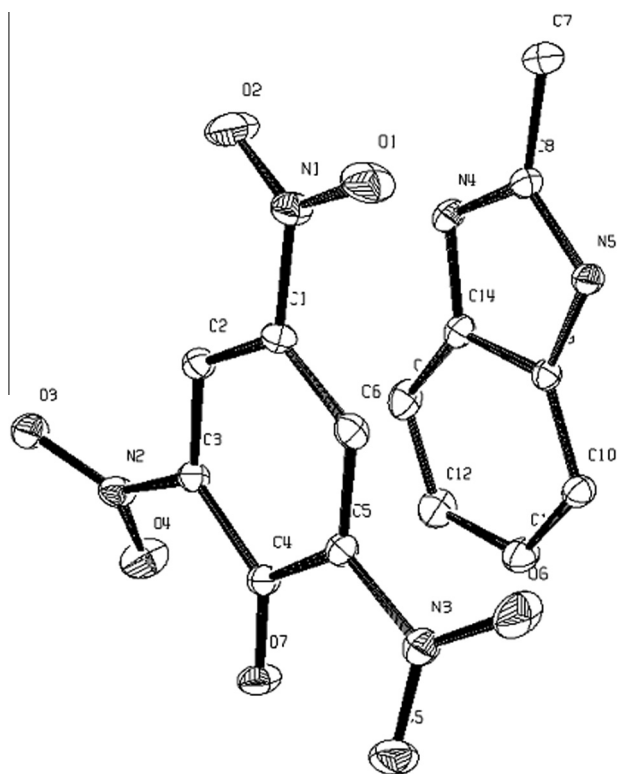


Fig. 1. The molecular structure of 2-methyl benzimidazolium picrate with atom labeling. (Hydrogen atoms were omitted).

comparable to the ranges found in those structures, suggesting that the nitro group does indeed interact with the five membered segment of the imidazolium cation. In fact, by combining these X-ray structural outcomes, with the FT-IR results (discussed in the next section), it is possible to infer an $n \rightarrow \pi$ interactions [12,21–25] involving a donor site of the imidazolium ring with the vacant π orbital of the nitro group. Probably, to maximize such interactions, the nitrogen of the para nitro group (N1) is situated approximately in between the imidazolium nitrogen atoms of the rings (the non bonded distances of N1---N4, and N1---N5 respectively are at 3.58 Å and 3.77 Å (Fig. S4).

The stacked sets of ion pairs generate an extended network of intermolecular hydrogen bonds (Table 2) through the $-N^+-H$

Table 2
Intermolecular hydrogen bonding details (Å, °) for the 2-methyl benzimidazolium picrate.

D–H...A	d(D–H)	d(H...A)	d(D...A)	<(DHA)
N(5)–H(5A)...O(7)#1	0.89(2)	1.89(2)	2.696(2)	150(2)
N(5)–H(5A)...O(4)#1	0.89(2)	2.38(2)	3.049(3)	131.7(18)
N(4)–H(4A)...O(5)#2	0.85(2)	2.10(3)	2.937(3)	169(2)

Symmetry transformations used to generate equivalent atoms: #1 $-x + 3/2, y + 1/2, -z + 5/2$ #2 $x + 1, y, z$.

moiety of the imidazolium group and the symmetry related O atoms of the picrate group (Figs. S5 and S6) in the solid state.

As reviewed in the introduction, the ionic picrate crystals with the π – π stacking interactions of the aromatic rings are not very recurring in nature and therefore the present structure represents one such example of the ionic picrates, in which the stacking is further facilitated with a rare $n \rightarrow \pi$ interaction as well as by the intermolecular hydrogen bonds in the solid state. The spectral studies also substantiate the observed solid state structural features and are discussed in the following sections.

Spectroscopic analysis

The FT-IR spectral appearance of the product (Fig. 2 and (Fig. S7), differing from those of its precursors, points out a clean ionic reaction. Particularly, the sensitivity associated with the ν_{as} N–O vibration is worth discussing. While it occurs as a single band at 1525 cm^{-1} for the free picric acid, it shows splitting with one higher wave number band at 1560 cm^{-1} and another as a broadened line in the region between 1548 cm^{-1} and 1528 cm^{-1} for the product. In addition, the aromatic C–H out of plane bending vibrations of the product, occurring at 790 cm^{-1} is also slightly higher in wave numbers when compared to the value of 783 cm^{-1} shown by the picric acid. These observations, fitting well with the IR spectral classification of organic picrates by the Kross et al. [23] and further resembling the IR spectral outcome of the earlier reports on the molecular picrates containing both the $n \rightarrow \pi$ and π – π interactions [24], provide clear evidences for the existence of a similar $n \rightarrow \pi$ interaction involving a donor site of the imidazolium ring to the vacant orbital of the nitro group, which are oriented due to the π – π stacking of the aromatic rings in the solid state, thus supporting the X-ray structural features.

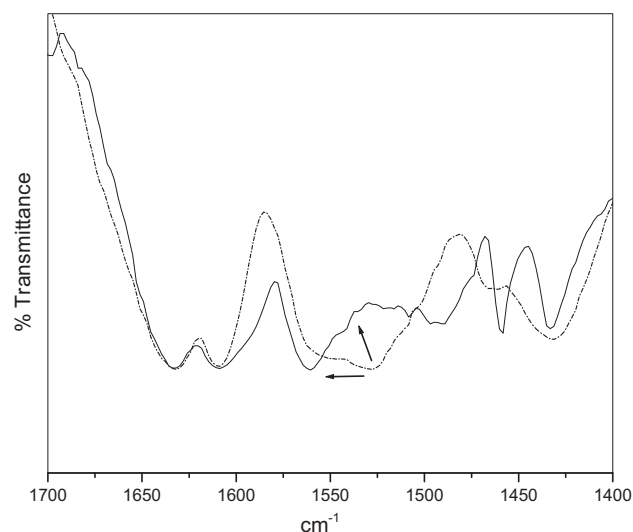


Fig. 2. The FT-IR showing the higher wave number shift of the ν as N–O vibration of the salt (solid line) when compared to the free picric acid (dashed line).

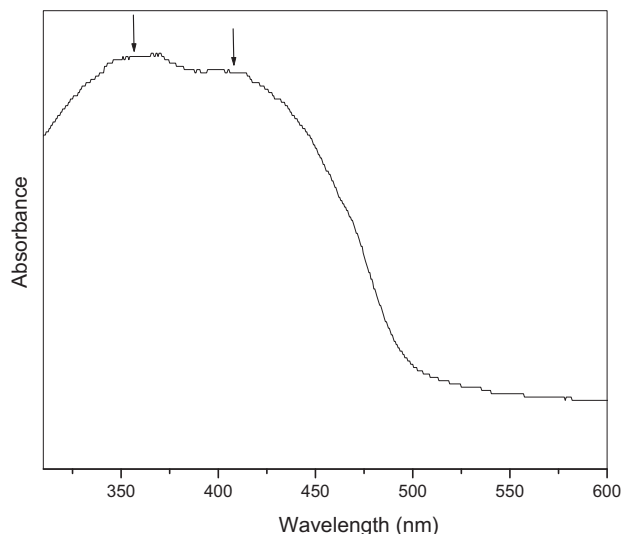


Fig. 3. Electronic absorbance spectrum of the material as a transparent film.

The electronic spectrum of the material also gives direct evidence to the solid state structural features. Although as an acetone solution it shows only a single absorption with a λ_{max} at 377 nm due to the picrate group (Fig. S8), as a transparent solid film (which was obtained after the evaporation of the solvent), it shows two absorption maxima (Fig. 3), one at 369 nm and the other at 412 nm. Since the absorption at 369 nm is due to the picrate moiety, the additional lower energy absorption of the film could be ascribed to the formation of the π - π stacking of the aromatic rings [26–28], which indicate that the supramolecular stacking interactions are inevitable in the solid state.

Since the variable temperature NMR (VT NMR) is a powerful technique in exploring the solution dynamics of the π - π stacked systems [29,30], it has been utilized in the present study as well to examine the nature of the stacking in solution, by carrying out the experiments in the range from 25 °C to –50 °C in acetone- d_6 . It is quite clear from the results (Table 3 and Fig. S9) that upon lowering the temperature, the singlet of the methyl protons show a slight but gradual upfield shift, whereas, the singlet of the picrate protons and the multiplet from the set of benzimidazolium protons show definite downfield shifts. These results, indeed confirm the existence of a dynamic motion of the ion pairs in the solution. Since the charges are located almost entirely on the rings of the ionic species, the self association phenomenon among the identical ions is highly improbable [31] and therefore the observed temperature dependence of the chemical shifts, could only be attributed to some loose stacking of the ion pairs, which could make the protons at different parts of the molecule to feel the magnetic anisotropy effect at different levels [30], particularly when there is a reduction in the dynamic motion at lower temperatures.

While the aromatic protons may be located away [32] from the molecular center of the ion pairs, possibly due to the influence of

the strong hydrogen bonding interactions, thus showing downfield shifts, the methyl protons on the other hand, may get placed in the core of the shielding zone, due to which they could exhibit upfield shift upon lowering the temperature. Although this VT NMR study, does not pinpoint the through-space interactions, it provides convincing evidences to realize that the stacking of the ion pairs can occur readily in the absences of any destabilizing forces and thus go in line with the inference derived from the UV–Visible experiments.

Thermal studies

In order to check the influence of temperature on this material, a DSC investigation has been carried out. The result (Fig. 4) indicates that the material is stable up to 150 °C. A sharp melting of the crystal occurs around 212 °C, coinciding with the observation made through the melting point apparatus. The endothermic event around 150 °C may be possibly attributed to a transition of the system from the state of ionic salt to the molecular CT complex [1] as there is a characteristic color change from yellow to red, which is a well known phenomenon associated with this type of transitions [1]. In general, the DSC results point out the stability of the material for ambient temperature applications.

Fluorescence studies

As the supramolecular structural features of the material in the solid state, such as stacking of the delocalized planar aromatic units and coplanar arrangement of the picrate anion with respect to the cation are well desired for a fluorescence emission character, which is indeed witnessed upon exposing the material even under an ordinary household UV lamp (Figs. S1B and S1C), an attempt has been made to explore the photoluminescent character of the material. When the powdered sample of the material was excited in the absorption range of the picrate group at 325 nm in the solid state, it shows an efficient luminescence in the longer wavelength ranges (Fig. 5). A green emission around 520–550 nm, and a longer wavelength red emission around 615 nm are clearly seen in the photoluminescence profile. While the green emission may be attributed to originate from the picrate anion moiety of the system [33], the occurrence of a red emission around 615 nm is an interesting phenomenon and deserves attention. This additional red emission may be attributed to the stacked nature of the ion pairs of the system in similar line to that of the picrate–peptide complex [27], which was characterized for its π stacking related emissions.

It is to be noted that, no such red emission has been reported in any of the benzimidazoles in their neutral or in their protonated state [34–36] and in fact they are known to possess emission maxima only within 450 nm. Similarly, the plain imidazolium picrate, which cannot exhibit any stacking interaction as that of the present compound, showed only in the 500–530 nm range and did not exhibit the red emission [37]. Therefore, from these correlations, it may be understood that the observed red emission is a consequence of the supramolecular stacking interactions of the ion pairs in the solid state. A shoulder around 575 nm may be attributed for the shallow traps, which are known in the literature [37].

As the stacking interactions are not sustained in solution, one would expect that the longer wavelength emission to disappear in the solution state photoluminescence profile. In fact, the fluorescence measurement in the acetone has shown only a single and shorter wavelength peak (Fig. S10A) around 470 nm. Further it has been observed in the solid state that the nature of the emission profile does not change significantly with respect to excitation wavelength (Figs. S10B–10D). These results highlight the influencing role of supramolecular structural motifs on the luminescence

Table 3
Temperature dependent chemical shift values of the different types of protons.

Temperature (°C)	Chemical shift values			
	Picrate	Benzimidazolium [A]	Benzimidazolium [B]	Methyl
25	8.78	7.91	7.57	3.06
0	8.81	7.92	7.57	3.06
–25	8.82	7.93	7.58	3.05
–50	8.83	7.94	7.58	3.04

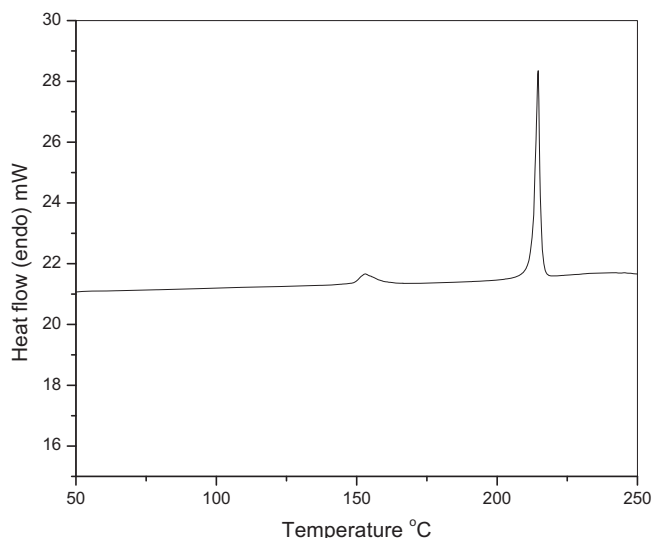


Fig. 4. The DSC result of the material.

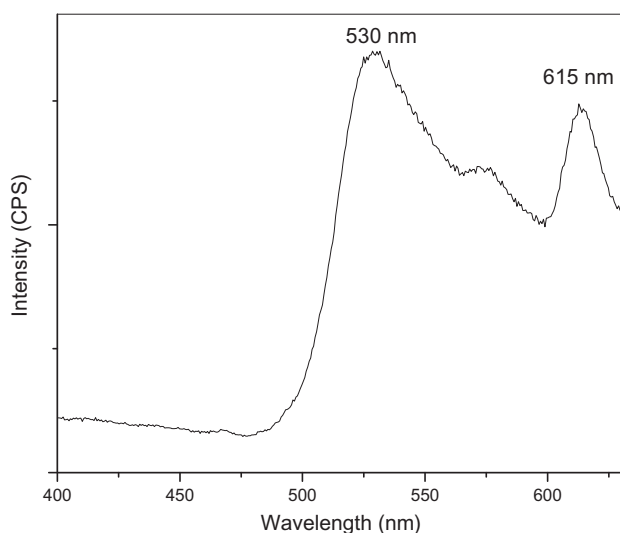


Fig. 5. The emission spectrum of the material in the solid state upon excitation at 325 nm.

character of the material as those of other reports [38–40] and stimulate the need to explore various structural motifs in the crystalline benzimidazolium family of picrates as well.

Other salient features of the material such as high crystalline nature, its ability to form a film/layer, its insoluble nature in less polar solvents etc. bring out additional advantage for this material in order for sensor application. Further study in this direction is in progress in our laboratory.

Conclusion

The synthesis, structural and photo luminescent characterizations of the 2-methyl benzimidazolium picrate have been performed in this study. The solid-state structural characterization has revealed the proton transfer reaction from picric acid to 2-methyl benzimidazole resulting in the formation of ion pairs. The ion pairs are found to possess a rather unusual heteromolecular π – π stacking along with $n \rightarrow \pi$ interactions involving the five membered imidazolium ring and the para nitro group of the

picrate moiety. The stacks are interlinked with extensive hydrogen bonds. The FT-IR and UV–Visible characterizations of the material also support the structural characterizations. This is the first report, showing the photoluminescence of a crystalline benzimidazolium family of picrates. The supramolecular stacking interaction in the solid state influences the fluorescence character of the material with an unusual longer wavelength red emission in the solid state. The report highlights the need to explore the structure–luminescence relationship in several other compounds of this family.

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Appendix A. Supplementary material

CCDC 912688 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html> (or from the Cambridge Crystallographic Data Center, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033). Figs. S1–S10 and Table S1 are given in the electronic supplementary information.

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.saa.2013.09.096>.

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