**RESEARCH ARTICLE**

# CHARACTERIAZTION OF SPECTRAL, STRUCTURAL OF 2-METHYLIMIDAZOLINIUM PICRATE (MIMP) COMPOUND

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**Abstract**

There are many nitrogen containing heterocyclic-picrate complexes to be synthesized and characterized. Among the organic picrate complexes synthesized so far, there are many other physical properties yet to be studied. The complete structural studies for all the picrate complexes synthesized so far are not available in the literature. Thermal analyses (TG-DTA and DSC) and spectral studies (UV-visible, Emission, Polarized Raman, FTIR and NMR) for many picrate complexes are not reported. Properties like dielectric, nonlinear optical, mechanical of a number of picrate complexes are not studied)benzamide, PBB, have been reported under microwave radiation.  The product is obtained in good yield.

**Key Words:** Microwave, PBA, PBN, PBB, Antibacterial activity.

## Introduction

Picric acid has a tendency to form stable picrate compounds with various organic molecules due to the presence of active electron cloud and ionic bonds (Yamaguchi et al., 1988). In the past two decades, organic nonlinear optical (NLO) picrate crystals have attracted the scientists and technologists due to their potential applications in the area of photonics including optical information processing (Prasad, Polymer **32,** 1746 (1991; Marder et al., 1991; . Saleh, M.C. Teich, 1991; . Penn  et al., 1991), high-energy lasers for inertial confinement fusion research, colour display, electro-optic switches, frequency conversion, etc.. The organic picrate compounds exhibit larger NLO response than inorganic materials due to the presence of active    -bonds. However, these materials have poor mechanical strength, thermal stability, laser damage threshold, etc., than the inorganic materials.

As the organic materials possess prominent NLO properties, new organic NLO materials are synthesized and characterized. A detailed survey of literature on picrate crystals are presented in this chapter.

Paula Zaderenko *et al.* (1997) studied the crystal structure of the diethyl 2-benzimidazol-1-ylsuccinate-picrate. The structure of the crystal was determined by single crystal X-ray diffraction analysis. The X-ray diffraction analysis indicated that the compound belonged to orthorhombic crystal system with $Pna2_1$ space group. Cross Polarization and Magic Angle Spinning (CP/MAS) and solid NMR studies showed that the picric acid linkage depends on the nature of the azole group.

The crystal structure, IR absorption spectra, emission spectrum of the europium

compound, TG and DSC analyses of lanthanide 4-methylmorpholine N-oxide picrate complexes were reported by Fernandes *at el*. (2000). The grown crystals crystallized in triclinic crystal systems with $P\overline{1}$ space groups as determined by single crystal X-ray analysis. Thermal analysis showed that the Eu(III) crystal was thermally stable up to $300^{\circ}C$.

A good nonlinear optical materials, l-Asparaginium picrate and $Co^{2+}$ doped l-Asparaginium picrate were studied by Russel Raj and Murugakoothan [134]. The doped crystal has high SHG efficiency than undoped crystal when compared KDP. The TG-DTA studies showed that the doped crystal exhibit better thermal stability than pure l-Asparaginium picrate. The single crystal X-ray analysis shows that the doped and undoped crystals were crystallized in monoclinic crystal systems.

### Experimental study

### Powder X-ray diffraction pattern method

Obtain a few tenths of a gram (or more) of the material, as pure as possible Grind the sample to a fine powder, typically in a fluid to minimize inducing extra strain (surface energy) that can offset peak positions, and to randomize orientation. Powder less than ~10 µm(or 200-mesh) in size is preferred Place into a sample holder or onto the sample surface: smear uniformly onto a glass slide, assuring a flat upper surface pack into a sample container sprinkle on double sticky tape Typically the substrate is amorphous to avoid interference Care must be taken to create a flat upper surface and to achieve a random distribution of lattice orientations unless creating an oriented smear.

### UV-visible absorption spectral analysis

Experiments were carried out using an Evolution Array UV-Vis spectrophotometer running VISIONcollect™ software (Wine color intensity and Wine hue) in Equation Calculation mode, or Color Analysis mode (CIE L*a*b* color). Samples were measured in 1 mm pathlength cuvettes against a deionized water blank.

### FTIR spectral analysis

Exchange the ion pairing agent: dissolve the peptide/protein in 5mM HCl, freeze in liquid nitrogen ($LN_2$), lyophilize, and repeat this process 4–5 times. Alternatively, the peptide/protein can be repurified by RP-HPLC using HCl instead of TFA in the buffers. Exchange the residual $H_2O$ to $D_2O$: dissolve the peptide in a small amount of $D_2O$, incubate at room temperature for 30 min, freeze ($LN_2$), and lyophilize. Amyloid fibrils should be formed in $D_2O$ with the pD adjusted with DCl/NaOD. As a general rule, it is difficult to get quality FTIR spectra from fibrils formed in $H_2O$ and then exchanged to $D_2O$. Data acquisition in $D_2O$: purge sample chamber for 20 min, take a scan of the empty chamber (as a background), purge for 20 min, take a scan of the same $D_2O$ solution used for sample preparation, purge for 20 min, take a scan of the sample, and subtract the $D_2O$ spectrum from the sample spectrum*.  Data Acquisition in the solid state: purge sample chamber for 20 min, take a scan of the empty chamber (as a background), purge for 20 min, take a scan of the empty sample cell, purge for 20 min, dry the peptide or fibril sample onto the sample cell window using a gentle stream of nitrogen and take a scan of the sample, and subtract the $D_2O$ spectrum from the sample spectrum.  Examine the Amide I region (1600–1700 $cm^{-1}$) of the sample spectrum. -sheet secondary structure is detected by the presence of a band near 1620 $cm^{-1}$.

### Results

### Powder X-ray diffraction pattern method

The powder X-ray diffraction pattern of MIMP complex is shown in Figure 5.131 The observed 'd' values for different 2 with

**Figure.1**.UV-Vis absorption spectra
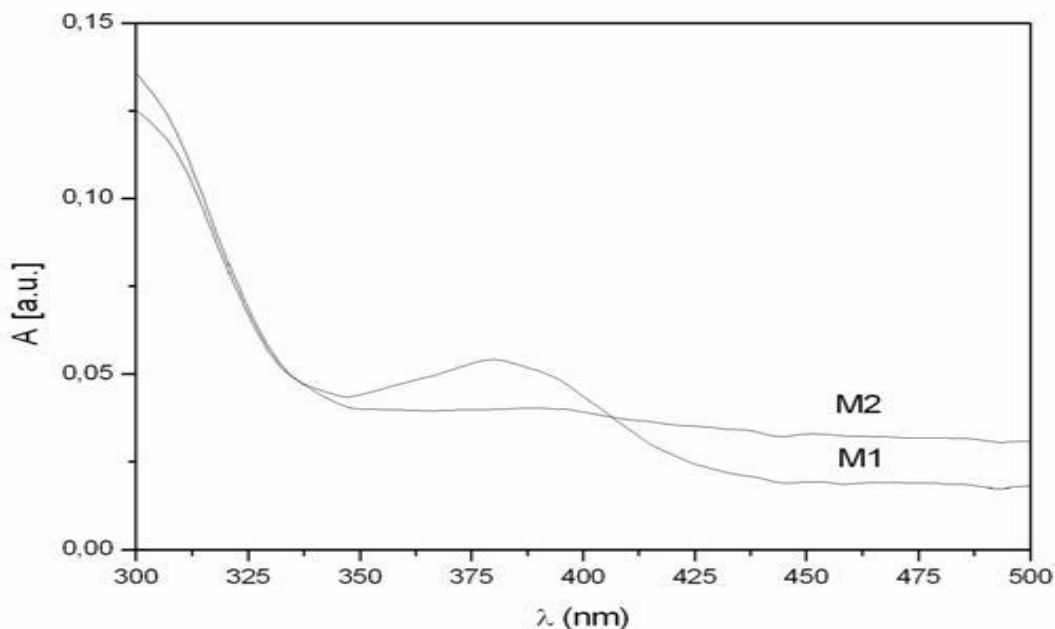


hkl indices of corresponding reflecting planes of the complex are given in Table 5.67. The sharp and well defined Bragg peaks observed in the powder X-ray diffraction pattern confirm its crystalline property. The prominent Bragg peaks in the powder X-ray diffraction pattern were indexed. The 2 values observed in the powder X-ray diffraction pattern were used to calculate the lattice parameters and unit cell volume by using the CRYSFIRE software package The unit cell parameters obtained are  a = 17.343Å, b = 12.8143Å, c = 14.7260Å and   =  = 90°,   90° and the unit cell volume is 2243.2Å$^3$. From the unit cell parameters, it is observed that the complex crystallizes in monoclinic system. The peak corresponding to -405 plane has a maximum counts of 2398. This is the strongest diffraction peak in the powder X-ray diffraction pattern. The unit cell parameters obtained from powder X-ray diffraction pattern almost fits well with the unit cell parameters obtained from single crystal X-ray diffraction method.

**UV-visible absorption spectral analysis**

The complex shows absorption at 381 nm which is due to the  -  * transition of the complex. The presence of  -  * transition in the complex is owing to the charge transfer in the picrate moiety. The absence of absorption observed in the visible region from 400 to 900 nm is required key factor for the material to exhibits the NLO property. This shows that the title complex can be used as a NLO material in the visible region. The higher intensity of absorption band presented in the UV region is due to the conjugated systems present in the grown material (Guru Prasad et al., 2010).

**FTIR spectral analysis**

The formation of the charge transfer complex by the reaction between picric acid and 2-methylimidazole is evident by the presence of the main characteristic infrared bands of the donor and acceptor in the spectrum of the product. The absorption peak at 3485 cm$^{-1}$ is due to N-H asymmetric stretching vibration of NH$^+$ and the corresponding symmetric stretching vibration is observed at 3144 cm$^{-1}$.. The frequencies at 557 and 575 cm$^{-1}$ are due to the rocking vibration of nitro group  (Uma Devi et al., 2008).

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