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# Crystal Growth and Characterization of $Li^+$ Ion and Picric Acid Doped Triphenyl Phosphine Oxide

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## Research Article

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**ABSTRACT**

A semi organic nonlinear optical family single crystal of the title compound was grown by the solvent evaporation method at ambient temperature. The single crystal X-ray diffraction analysis shows that the grown crystals have a tetragonal structure. Fourier Transform Infrared spectral analysis and UV-vis spectral studies was also carried out. Micro hardness mechanical studies show that the hardness number of the title compound decreases with the load as measured by the Vicker's micro hardness method. The dielectric properties of the grown crystal when analyzed by varying the frequency. The nonlinear optical properties were studied using the Kurtz and Perry powder method and the second harmonic generation efficiency was found to be 0.15 times that of potassium dihydrogen phosphate crystal. The third order nonlinear studies were done using z scan technique. Magnetic studies using Vibration-sample magnetometer analyses have been studied.

## INTRODUCTION

Nonlinear materials of exceptional properties can be engineered properly for serving in real time applications. This has gained importance due to its application in the field of optical computing, color display, optical communication and many more. Crystal engineering interestingly helps in finding the mechanical properties based on crystal structures. Depending on the stress applied, the material can undergo deformation which may involve elastic or plastic or brittle or hard or viscoelastic or a combination of these properties also. The reasons may be due to either permanent change in the molecular structure, or stretching of intermolecular bonds, or when stress and strain are not proportional. When changes are made intentionally in the crystal by some additives, mechanical properties can be changed. Thus, an understanding at the molecular level of the crystal can improve mechanical properties. Organic additives can improve the properties like mechanical strength, particularly tensile strength and electrical stability in the crystal when compared to inorganic ones. As the presence of hyper polar molecules in organic NLO materials may get decomposed at higher temperatures, solution growth technique is employed [1-4]. Also, third order nonlinear optical properties lead to applications like optical signal processing, optical activity, etc.

Triphenylphosphine can react readily with almost all metals. The symmetric electron distribution helps in enhancing the optical nonlinearity of the crystal which may be either on both ground and excited states. This in turn is due to the overlap of π orbital delocalization of electronic charge distribution, as the organic NLO material structure is based on the π bond system [5-8]. The nonlinearity of the crystal is affected both by the anion group and the cation group especially which has larger radius and changeable electron cloud. The presence of the alkali cations helps in showing parametrical and acousto electronic properties, which may be observed in particularly Li ions, possessing greater polarizing power of all the metal ions. An attempt of addition of picric acid in the solution was due to the fact that, already picric acid is non centerosymmetric and, its addition results in quick binding with rest of the elements [9]. This addition is aimed towards increase in hyperpolarizabilty. Thus, in this study, the title compound lithium ion and picric acid doped triphenylphosphine oxide crystals was grown by solvent evaporation technique, which is one of the promising and easy approach for the preparation of single crystals.

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# EXPERIMENTAL

### Materials Preparation

The title compound was obtained by taking Lithium Chloride (LiCl) (obtained from Mody Chem-Pharma Limited), and Triphenylphosphineoxide (TPPO) (from Ningbo Huajia Chemical Co., Ltd) in a stoichiometric ratio of 1:2. Ethanol (obtained from Generic manufacturer), was taken as a solvent and after vigorous stirring, a turbid solution was obtained. The solubility of the compounds was determined for various temperatures, by dissolving the solute in ethanol at different constant temperatures with stirring and the solubility graph is shown in **Figure 1.** After refluxing for one hour, a clear transparent solution was obtained which was taken in a glass beaker covered with a polythene cover with 3 to 4 holes made. After 7 days, white precipitate was formed and dried.

Saturated lithium doped triphenylphosphine oxide solution was prepared from the precipitate obtained along with ethanol and Dimethyl Sulfoxide (DMSO) (obtained from Blessed organics manufacturer), in the ratio of 1:1 solvent. After 1 hour, 5 ml of picric acid (obtained from Sisco Research Laboratories) was added to the stirred transparent solution. The obtained slightly turbid solution has pH of 5. The solution was then further taken in a glass beaker with perforated cover and was kept in a dust and vibration free atmosphere for solvent evaporation. Crystals of size (2 × 3 × 7) $mm^3$ were harvested after about 35 days. The as grown crystal is shown in the **Figure 2**.

$$LiCl_2 + 2(C_6H_5)_3P = O \rightarrow LiCl_2[(C_6H_5)_3P = O]_2$$

$$LiCl_2[(C_6H_5)_3P = O]_2 + C_6H_5(NO_2)_3OH \rightarrow C_6H_5(NO_2)_3OH - LiCl_2[(C_6H_5)_3P = O]_2$$

# RESULTS AND DISCUSSION

### Powder XRD Analysis

The crystallinity of Lithium ion and picric acid doped triphenyl phosphine oxide crystals was confirmed by the powder X-ray diffraction study as shown in **Figure 3.** The X-Ray Diffraction (XRD) patterns of the sample was measured by XPERT-PRO diffractometer with monochromatic CuKα-radiation (λ=1.5406Å). The peaks in the powder XRD pattern infer crystalline property

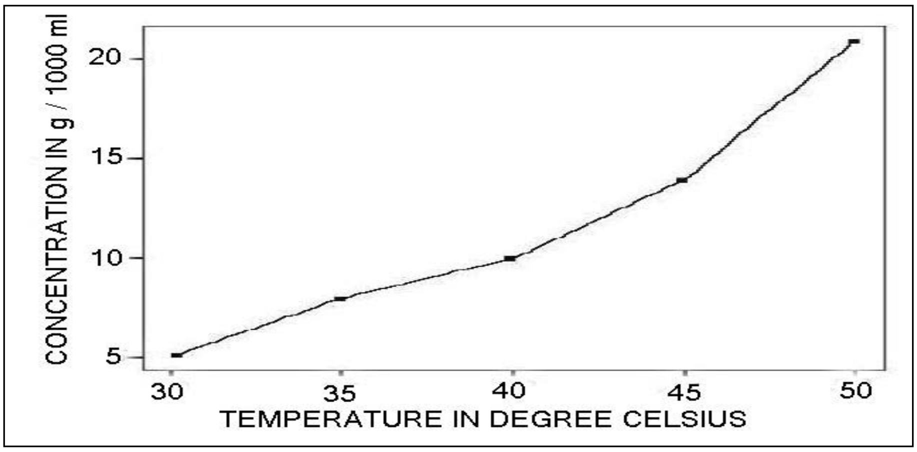


**Figure 1.** Solubility curve of lithium ion doped triphenyl phosphine oxide.

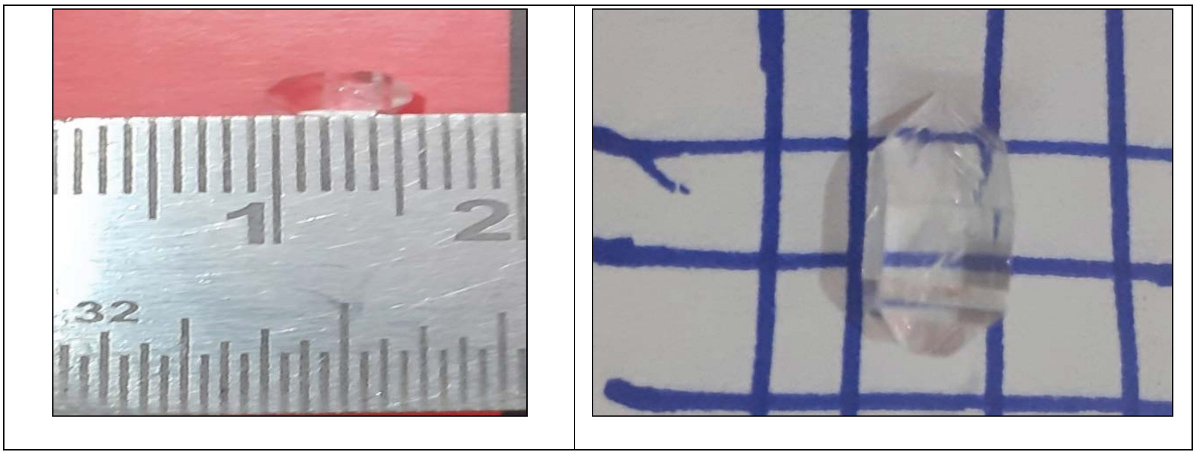

**Figure 2.** The as grown crystal of lithium ion and picric acid doped triphenyl phosphine oxide.

and single phase nature of the pure Lithium ion and picric acid doped triphenyl phosphine oxide crystals. There is change in intensity with slight shift in the reflection peaks of the crystal when compared to those of the original compounds, resulting in small variations in the cell parameters, with slight distortion in structure. This may be attributed to the strains due to the substitution of the semi-organic impurity.

**Single Crystal XRD Analysis**

Bruker Smart Apex CCD area detector using a scan mode SAINT program was used to carry out cell refinement and data reduction of the reflections obtained from the single crystal using MoKα radiation in the range of 2.2º-28º. It is observed that the C-C, C-P and P-O bond lengths agree with the reported literature values. They are 1.800 (13) Å, 1.803 (11) Å and 1.803 (10) Å respectively and the angles α=β=γ. It is found that the crystal structure is of tetragonal form with the space group P4/nmm **(Figure 4)**.

**FTIR Analysis**

Fourier Transform Infrared Spectroscopy or FTIR spectroscopy denotes the transitions between quantized vibrational energy states. The organic and inorganic compounds which are IR active, gives an absorption peak of IR radiation due to the absorption of photon by a molecule and after its excitation to a higher energy state [10,11]. FTIR analysis of the title compound was recorded in Perkin Elmer FTIR spectrophotometer using KBr pellet technique in the range of 400 $cm^{-1}$-4000 $cm^{-1}$. The influence of Li can be confirmed by the shifting of p=O (1187 $cm^{-1}$) to lower frequency (1144 $cm^{-1}$). Also, the influence of metal–oxygen vibrational modes is indicated by the frequency 540 $cm^{-1}$ in the spectra. 3370 $cm^{-1}$ peak denotes the stretching vibration of water molecules absorbed by KBr. The $NO_2$ vibrations observed at 663 $cm^{-1}$ in picric acid shifted to 635 $cm^{-1}$ in the title compound. The peaks 1211 $cm^{-1}$ and 635 $cm^{-1}$ confirm the presence of picric acid [12,13]. The vibrational frequencies of Lithium ion and picric acid doped triphenyl phosphine oxide crystals are shown in **Table 1**.

**UV-Vis-NIR Analysis**

The UV-vis-NIR analysis of the title compound can be studied by the transmission spectrum recorded using Lambda 35 (Instrument model) UV-vis-NIR spectrometer in the wavelength range of 190 $cm^{-1}$-1100 $cm^{-1}$. A sample crystal of 5 mm thickness has been used for this study. The lower cut off wavelength was found to be low before picric acid was added. This study was repeated after the addition of picric acid in the synthesizing part and the absorption cut off wavelength was found to be 260 nm.

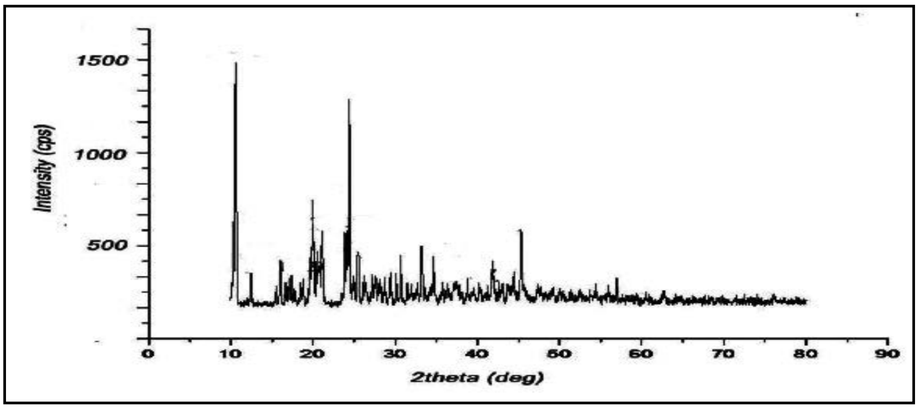


**Figure 3.** Powder X-ray diffractogram study of lithium ion and picric acid doped triphenyl phosphine oxide.

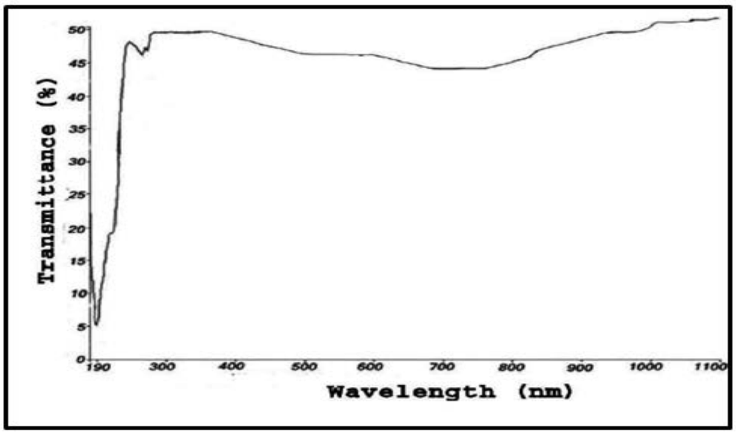


**Figure 4.** UV-vis-NIR studies of lithium ion and picric acid doped triphenyl phosphine oxide crystal.

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The addition of picric acid has been found to improve the transparency of the crystal [14]. Also, the crystal is highly transparent to the wavelengths 190 nm up to 1100 nm. This is a desirable property for an NLO crystal.

**Energy Band Gap Determination of Lithium Ion and Picric Acid Doped Triphenyl Phosphine Oxide**

The optical band gap energy of the title compound has been studied with the values of absorption coefficient (α) which obeys, α=2.3026log (1/T)t, where $E_g$ is the optical band gap energy of the crystal and A is constant. α is calculated from the transmittance. A graph is drawn for the variation of $(\alpha \cdot h\nu)^2$ and hv and the extrapolation of the linear part gives the optical band gap energy. $E_g$ is found to be 3.8 eV for the title compound. It can be inferred that the wider band gap energy leads to large transmittance in the visible region [15-18].

**NLO Activity**

**Laser damage threshold study**

Laser Damage Threshold (LDT) study is essential for crystal application, as it is an important property that a NLO crystal has to inherit is its optical surface and bulk damage tolerance. It has to withstand a high value of power intensity and hence, a high value of LDT is necessary for measuring second and third order non-linear optical property. A pyrometer is normally employed to record the energy of the input laser pulse which is taken as the reference and the energy at which the damage of the crystal occurs. The energy density can be calculated using the formula Energy Density=E/A; where, E is the input energy and A is the area of the circular spot. LDT was performed for the title compound using Q-switched Nd-YAG laser of 1064 nm as a pulse with duration of 10 nanoseconds. The LDT of Lithium ion and picric acid doped triphenyl phosphine oxide is 1.52 GW/$cm^2$ when compared to 0.20 GW/$cm^2$ of KDP [19].

**Second Harmonic Generation Study**

Kurtz and Perry setup with a Q-swtiched Nd-YAG laser beam of wavelength 1064 nm with an input power of 4.9 ns, pulse width of 8 ns and a repetition rate of 10 Hz were used for measuring the SHG conversion efficiency of Lithium ion and picric acid doped triphenyl phosphine oxide. After packing the powdered sample, of size sieved to 100 µm in a microcapillary tube of uniform bore, it was exposed to laser radiations by placing it between two transparent glass slides. A photomultiplier tube was used as a detector. The emission of 532 nm which is in green colour indicates the SHG conversion with a second harmonic signal of value 1.34 mJ, for an input energy of 0.68 mJ **(Figure 5)**. This can be compared with KDP as 8.8 mJ for the same input energy [20,21]. The SHG efficiency has been compared with KDP and other reported materials in the literature as given in **Table 2**.

**Z-scan Study**

Z-scan is important because it gives the details of coherence, transparency of propagating light rays, focusing ability etc. or the intrinsic photo physical property of the optical materials. He-Ne laser of wavelength 632.8 nm with beam diameter 1 mm has

**Table 1.** Vibrational frequencies.

| Assignments | Lithium ion and picric acid doped triphenyl phosphine oxide in $cm^{-1}$ |
|---|---|
| ν ( $NO_2$) | 1550 |
| ν ( P=O) | 1144 |
| ν ( C-N) | 1211 |
| ν (P-C) | 1308 |
| ν (C=C) | 1499 |
| δ (N-H) | 1621 |
| ν (C-H) | 3259 |
| ν (O-H) | 3370 |

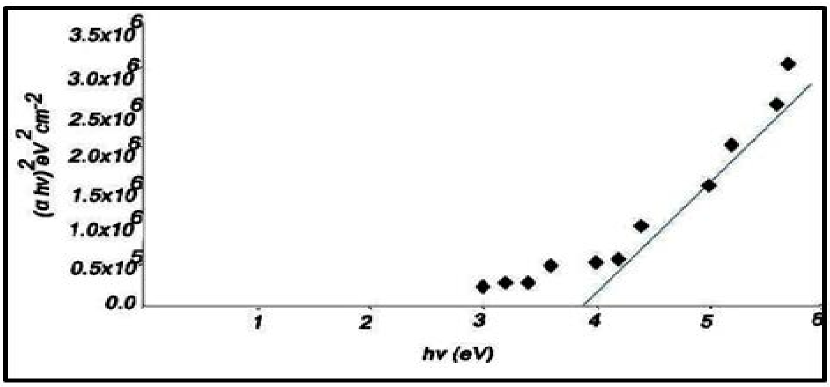


**Figure 5.** Tauc's plot of lithium ion and picric acid doped triphenyl phosphine oxide.

been used for the scan. Gaussian filters change the input laser beam to Gaussian form. This is used to scan the sample along the z axis. The diameter of the incident Gaussian beam is dependent on the focal length of the convex lens. In a closed aperture set up, the aperture restricts the outside scattered light. A lens focuses a laser beam to a point '$w_0$' called waist point and after this, the beam is defocussed. The sample is moved through a distance '$z_0$' along the z axis. The nonlinear refractive index $n_2$ is measured by the Rayleigh length. The nonlinear absorption coefficient can be measured by the open aperture scan [22-25]. The calculated Rayleigh length is 2 mm, with laser waist at the focus as 17 nm. The on-axis peak intensity is 4.21 GW/cm$^2$. This is due to second order hyperpolarizability which was confirmed to be -40.2 x 10-40 esu from B3LYP level by employing 6-31G (d,p) basis set, by Hatree-Fock method. Since the nonlinear refraction and absorption affect closed aperture transmittance, nonlinear refraction has to be separated from nonlinear absorption. To cure this, the ratio of closed aperture and open aperture z scan can be plotted. **Figure 6** shows the z-scan studies of Lithium ion and picric acid doped triphenyl phosphine oxide crystal. A valley obtained after the peak in the normalized transmittance curve indicates that the nonlinear refraction is negative. This is due to defocussing effect or the nonlinear refractive index is adaptable to the change in laser intensity (sample will defocus light). This property can be useful in the protection of optical sensors [26].

### Vickers Micro Hardness Test of Lithium Ion and Picric Acid Doped Triphenyl Phosphine Oxide Crystals

Micro hardness studies were performed using SHIMADZU HMV micro hardness tester fitted with a diamond pyramidal indenter. The measurements were made for different applied load (P) varying from 25 g to 150 g for an indentation time of 10 seconds. Hardness number Hv is calculated using the formula $H_v$=1.8544 P/d$^2$ (kg/mm$^2$), 'd' denotes the diagonal length measured **(Figure 7)**. It is seen that Hv is directly proportional to the applied load which is known as Reverse Indentation Size Effect (RISE). At the load of 100g, a crack was developed on the crystal due to the internal stress [27,28].

### Dielectric Studies of Lithium Ion and Picric Acid Doped Triphenyl Phosphine Oxide Crystal

The dielectric constant or relative permittivity (εr) or K is the macroscopic electrical property of a dielectric medium [29]. The dielectric study on Lithium ion and picric acid doped triphenyl phosphine oxide crystal was carried out using Jognic's (Model: 2816B) Multirange for CRL frequency: 50 Hertz to 200 KHz instrument. A sample of dimension (2 × 2.5 × 5.5) mm$^3$ was placed between electrodes (copper). The capacitance was measured at room temperature for the sample crystal for various frequencies. The dielectric constant (εr) and dielectric loss (tanδ) was calculated for the variation in frequency. Graphs are plotted for the variation of dielectric constant and dielectric loss as a function of frequency. Dielectric constant varies dependent on the dipole polarization and charge displacement in the sample.

The graphs reveal that, dielectric constant is higher at lower frequency and decreases as the frequency is increased and beyond a frequency, becomes constant and is calculated to be 32. Also, dielectric loss decreases at higher frequencies. At the lower frequency, the contribution of all the four polarizations is significant and at higher frequency, the decrease in dielectric constant is justified by the reduction in space charge polarization at the grain boundaries. This behavior of the crystal is very

**Table 2.** Comparison of conversion efficiency of some TPPO complexes.

| Material | SHG intensity(mV) |
|---|---|
| KDP (Reference material) [22] | 23 |
| $CdBr_2(TPPO)_2$ [23] (sieved-100 µm) | 27.6 |
| L Alanine Lithium chloride [24] | 9.93 |
| Lithium ion doped triphenyl phosphine oxide crystal (sieved-100 µm) | 3.5* |
| *:Present Work | |

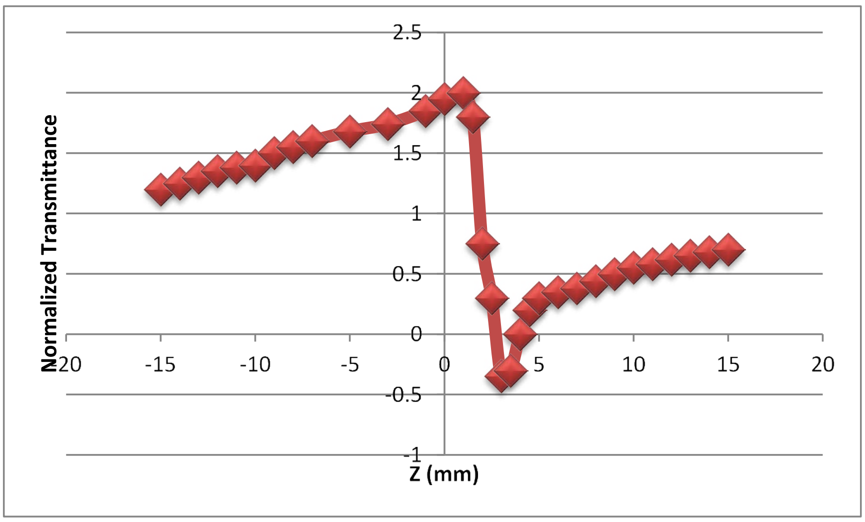


**Figure 6.** Z-scan studies of lithium ion and picric acid doped triphenyl phosphine oxide.

similar to normal dielectric and can find applications in electro-optics. **Figure 8** shows the variation of dielectric constant with frequency for Lithium ion and picric acid doped triphenyl phosphine oxide crystals. Also, variation of dielectric loss with frequency is shown in the **Figure 9**. The variation of dielectric loss versus frequency at room temperature infers that, they are inversely proportional [30-37].

**VSM Analysis**

The Vibrating Sample Magnetometer (VSM) analysis is important and accurate for measuring magnetic properties of materials. The principle is based on analysis of the dipole field from an oscillating sample placed in a magnetic field [38]. The room temperature magnetic measurement was carried out for Lithium ion and picric acid doped triphenyl phosphine oxide crystals by using VSM (Model: 7404: Lake Shore). The magnetization property of Lithium ion and picric acid doped triphenyl phosphine oxide crystals is shown in **Figure 10**.

About 15 mg of the sample was taken for measurement for 5 seconds. The study of plot reveals that, when the magnetic

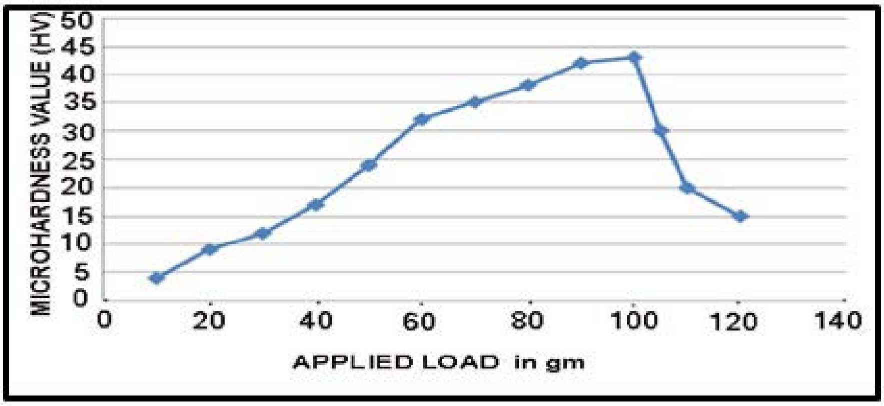


**Figure 7.** Micro hardness study of lithium ion and picric acid doped triphenyl phosphine oxide crystals.

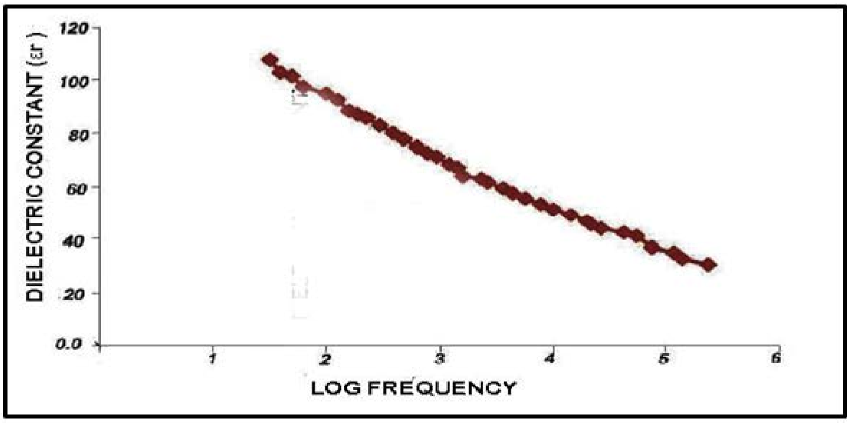


**Figure 8.** Variation of dielectric constant with respect to log frequency of lithium ion and picric acid doped triphenyl phosphine oxide crystals.

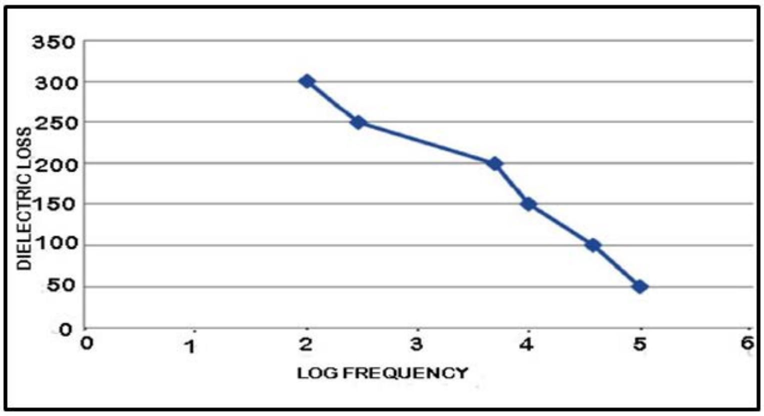


**Figure 9.** Variation of dielectric loss with respect to log frequency of lithium ion and picric acid doped triphenyl phosphine oxide crystals.

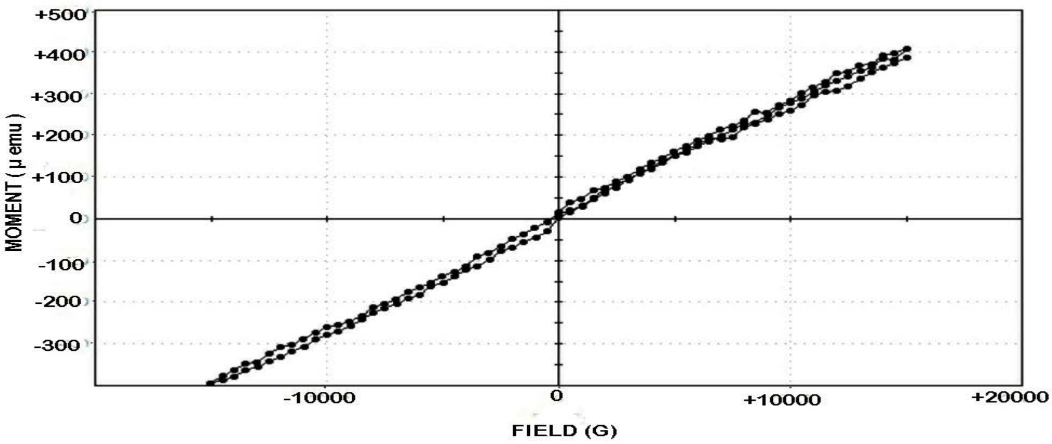


**Figure 10.** Magnetic studies of lithium ion and picric acid doped triphenyl phosphine oxide crystals.

field is applied, the dipole aligned with the applied field. This linear graph shows that the title compound is paramagnetic in nature with a positive susceptibility [39,40]. The saturation magnetization is 401.97E-6 emu. The above results can be justified, due to the charge transfer from metal dopant to molecule which can induce local spins in metal doped organic hydrocarbon.

## CONCLUSION

Lithium ion and picric acid doped triphenyl phosphine oxide crystals were grown by slow evaporation technique. The FTIR and NMR spectra confirm the formation of the Lithium ion and picric acid doped triphenyl phosphine oxide crystals. The transmission spectrum of Lithium ion and picric acid doped triphenyl phosphine oxide crystals laid importance on the fact that, the light of wavelength around 240 nm is absorbed. From the microhardness study, it may be suggested that, Lithium ion and picric acid doped triphenyl phosphine oxide crystals can withstand the external mechanical stress below 100 g load. The higher value of band gap (3.8 eV) suggests that Lithium ion and picric acid doped triphenyl phosphine oxide crystals can be polarized at higher frequencies [41]. From the Kurtz-Perry technique, it is found that, the output intensity of 532 nm was found to be ~0.15 times that of the intensity of KDP and the SHG efficiency is found to be 1.34 mJ when compared to 8.8 mJ for KDP. At room temperature, the interdependence of frequency, dielectric constant, dielectric loss has been studied taking polarization into account. The Lithium ion and picric acid doped triphenyl phosphine oxide crystals are found to behave like a normal dielectric and can find applications in electro-optics.

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## CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

## AUTHOR CONTRIBUTIONS

This manuscript is written with the contribution of all the authors.

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