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SYNTHESIS, GROWTH AND STRUCTURAL ANALYSIS OF 2-AMINO 4, 6 DIMETHOXYPYRIMIDINE P-TOLUENESULFONIC ACID MONOHYDRATE

RO.MU. Jauhar and P. Murugakoothan*
MRDL, PG & Research Department of Physics, Pachaiyappa's College, Chennai - 600 030, Tamil Nadu.

Abstract

A new organic nonlinear optical crystal (NLO) 2-amino 4,6dimethoxypyrimidinep-toluenesulfonic acid monohydrate (2ADPTS) was synthesized and good quality crystals were obtained from ethanolic solution containing stoichiometric quantities of the components. The species crystallizes in the triclinic crystal system with space group Pī. Single crystal and powder X-ray diffraction techniques were used to estimate the cell parameters and crystalline nature of the title compound. The presence of strain was calculated using Hall-Williamson equation. The presence of functional groups was identified by FTIR spectral analysis.

Key words: Optical crystal, Ethanolic solution, Triclinic crystal and FTIR spectral analysis.

1. Introduction

In recent years, organic compounds with large π-electron delocalization have attracted interests among materials scientists because of their large optical nonlinearity, photorefractivity and potential applications in optical signal processing, optical communication and they often possess interesting ferroelectric, ferromagnetic, superconducting properties, etc (Chemla and Zyss, 1987). For intelligent crystal engineering the charge transfer, electrostatic, hydrogen bond and π-π interactions have been focused to control the structure of solids (Prasad and Williams, 1990). As amino pyrimidines readily pair up with carboxylic acids to form a variety of adducts with mono and dicarboxylic acids, the 2 amino 4,6 dimethoxy pyrimidine has been taken to form molecular building blocks with p-toluene sulfonic acid. 2 amino 4,6 dimethoxy pyrimidine is used in various supramolecular recognition processes across the spectrum of organic, biological and medicinal chemistry with special interest motivated by their potential applications in nonlinear optics (Kushwaha *et al*., 2012). In this regard bulk crystals of 2ADPTS have been grown adopting slow evaporation solution growth technique. Herein we report the synthesis, growth and structural analysis of 2ADPTS.

2. Experimental Results

Synthesis and crystal growth

The initial reagents 2-amino 4, 6 dimethoxypyrimidine and p-toluenesulfonic acid monohydrate were used for synthesis of the title compound without additional purification in an equimolar stoichiometric ratio. The saturated solution of p-toluenesulfonic acid monohydrate was prepared using ethanol. Then, 2-amino 4, 6 dimethoxypyrimidine was added gradually in the above solution which results in white precipitation immediately. The precipitate was dissolved using the same solvent and the resulting solution was stirred for an hour to attain homogeneity. After a span of 20 days, hexagonal shaped crystal of 2ADPTS was harvested. The photograph of the as grown 2A46MP crystal is shown in Fig.1 (a).

*Corresponding author: **P. Murugakoothan**
E-mail: murugakoothan03@yahoo.co.in
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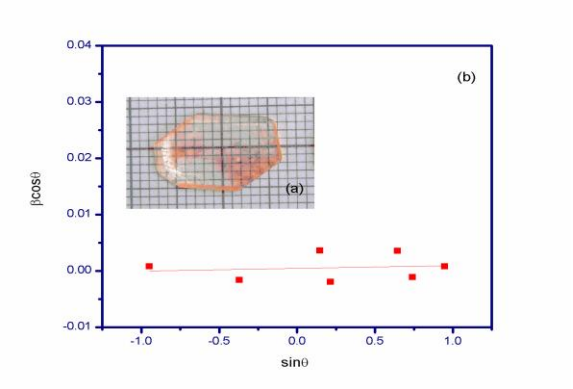

Fig - 1 (a) As grown 2ADPTS crystal and (b) Hall- Williamson plot of 2ADPTS

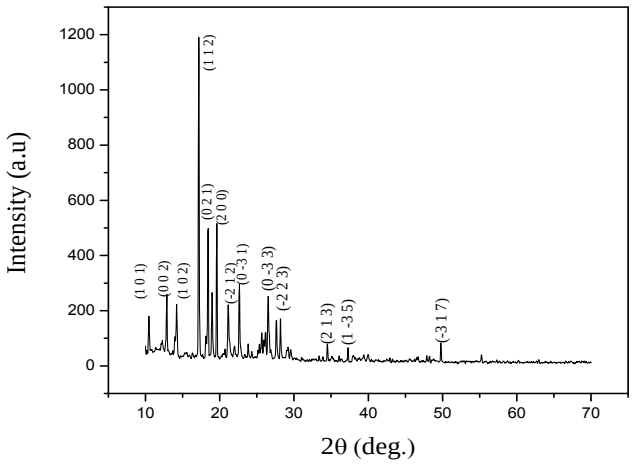

Fig – 2: Powder X-ray diffraction pattern of 2ADPTS

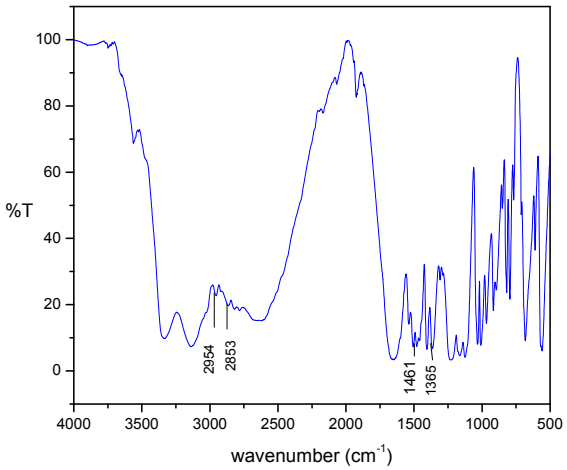

Fig – 3: FTIR spectrum of 2ADPTS crystal

Powder X-ray diffraction analysis was performed for the title material to determine the lattice parameters and space group of the grown crystal. A 2ADPTS crystal free of visual defect was crushed into fine powder and subjected to PXRD study using Reich Seifert diffractometer with CuKα (λ = 1.5405 Å) radiation over the range 10-70° at a scanning rate of 1°/minute. The 2ADPTS crystallizes in the triclinic crystal system with calculated unit cell parameters are, a = 9.63 (3) Å, b = 12.13 (2) Å, c = 14.41 (4) Å, α = 100.97°, β = 101.39°, γ = 103.61° and volume V = 1573.38(7) Å3 with space group Pī. The strain in the lattice of grown crystal is calculated by Hall Willamson equation $\beta Cos\theta = K\lambda/\tau + \eta sin\theta$, where β, θ, K, λ and τ are full width at half maxima (FWHM) of diffraction peak, Bragg diffraction angle of the peak, Scherrer constant, wavelength of X-rays and crystallite size respectively. The plot is drawn between βCosθ and Sinθ, such that slope of the curve gives the value of strain and is shown in Fig – 1 (b). The value of strain (η) is -9.59×10^{-3}. Generally, negative value of η depicts the presence of vacancy type of defects in 2ADPTS crystal which lead to expansion of lattice around the defect core and cause strain to the crystal (Kushwaha *et al*., 2012).

FTIR spectral studies

The functional groups of 2ADPTS crystal were identified by FTIR analysis using KBr pellet technique. The FTIR spectrum was recorded using Perkin Elmer FTIR spectrometer in the range 4000 – 450 cm^{-1} and was shown in Fig - 3. The stretching vibrations of secondary and primary amines occurred at 3333 and 3138 cm^{-1} respectively. The C-H stretching of methyl group of p-toluene sulfonate moiety yielded its signal at 2954 cm^{-1}. The sharp isolated bands at 2863 and 2819 cm^{-1} are due to methoxy C-H stretching vibrations. The peaks at 2168, 2067 and 1924 cm^{-1} are due to C-C stretching vibrations. The CH3 deformation bands are found at 1461 and 1366 cm^{-1} respectively. The C=C stretching vibration occurred at 1651 cm^{-1}. The peak at 1539 cm^{-1} was due to N-H bending vibration. The asymmetric and symmetric stretching vibrations of SO3 occurred at 1365 and 1163 cm^{-1} respectively. The aromatic C-H in-plane and out-of-plane bending vibrations occurred at 1231 and 815 cm^{-1} respectively. The N-H out-of-plane bending vibration occurred at 682 cm^{-1}.

3. Conclusion

2ADPTS single crystal was successfully grown by slow evaporation solution growth technique. Unit cell dimensions were found using powder XRD. The strain present in the specimen has been calculated using Hall–Williamson equation and found that vacancy type defects were present in the title compound. The functional groups present in the title material were confirmed using FTIR spectral study.

4. References

1) Chemla, D.S and J. Zyss. 1987. Nonlinear Optical Properties of Organic Molecules and Crystals. Academic Press, New York.

2) Prasad, P.N and D.J. Williams. 1990. Introduction to Nonlinear Optical Effects in Molecules and Polymers, Wiley, New York.

3) Zyss. J. 1994. Molecular Nonlinear Optics: Materials, Physics, and Devices.Academic Press, San Diego, California.

4) Kushwaha, S.K., K.K. Maurya, N. Vijayan, B. Kumar, R. Bhatt, S. Ganesamoorthy, G. Bhagavannarayana. 2012.Cryst. Eng. Comm. 14, 3297–3305.