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# Synthesis, growth and characterization of Bis thiourea sodium nitrate with Kcl single crystals

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**Abstract :** Single crystals of Bis thiourea sodium nitrate(BTSN) doped with Kcl have been grown in a period of three weeks from solution by slow evaporation method at room temperature. The solubility study was carried out at various temperatures ranging from 35-55$^0$C in aqueous solution. Grown crystals were characterized by single crystal X-ray diffraction,Powder X-ray Diffraction and FT-IR technique. The range and percentage of optical transmission were ascertained by recording UV-Vis-NIR spectrum. Its mechanical behaviour was estimated by Vickers Microhardness test.

**Keywords :** Characterization, X-ray diffraction, single crystal, growth from solution.

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## 1. Introduction

Thiourea and its family of crystals have been reported to be very great interest for the non-linear optical (NLO) applications [1-4]. The thiourea molecule is an interesting inorganic matrix modifier due to its large dipole moment[5] and its ability to form an extensive network of hydrogen bonds[6]. The centrosymmetric thiourea molecule, when combined with inorganic salt yields noncentrosymmetric complexes, which have the NLO properties[7]. In the present study, thiourea is combined with potassium chloride to form a new semiorganic optical material.

In this paper, we are presenting a preliminary report on the growth and characterization of new semi-organic optical material bis-thiourea sodium nitrate (BTSN)with Kcl single crystals.

## 2. Experimental Procedure

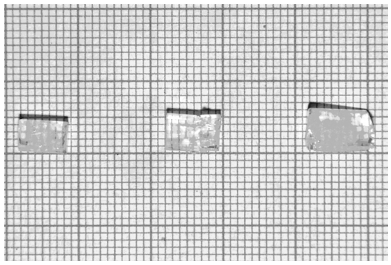
### 2.1. Crystal growth

The BTSN single crystals were synthesized using analar grade thiourea and sodium nitrate in a stoichiometric ratio of 2:1,1wt% 0f Potassium chlorideis added according to the following chemical reaction,

$2[CS(NH_2)_2]+NaNO_3 \rightarrow Na[CS(NH_2)_2]_2NO_3$

The mother solution was thoroughly stirred using magnetic stirrer and the mixture was heated to avoid decomposition of the solute molecules. Since thiourea has the coordinating capacity to form different phases of metal-thiourea complexes, the mixture of the reactants had to be stirred well to avoid coprecipitation of multiple phases[8].

The seed crystals were obtained by spontaneous nucleation technique. Bulk size of BTSN-Kcl crystals were crystallized with well defined faces in a period of 30-40 days. The crystals of BTSN-KL are shown in Figure 1.



2.2 Figure 1. Photograph of BTSN-Kcl crystals

### 2.3 Solubility

The synthesized salts of BTSN-Kcl has been determined for five different temperatures from 35-55°C. The solubility was determined by dissolving the solute by stirring in water in an airtight container maintained at the relevant temperature. After attaining saturation, the equilibrium concentration of the solute was estimated gravimetrically. The same process was repeated to estimate the equilibrium concentration of the solute at various temperatures. Figure 2 shows the solubility curve for BTSN-Kcl. Here solubility increases with temperatures.

# 3. Results and discussion

## 3.1. Single crystal XRD analysis

The title compound (BTSN-Kcl) was analyzed by single crystal X-ray diffraction method to determine the lattice parameters. The crystallographic parameters are given in Table 1.

| Crystal parameters | BTSN-Kcl |
|---|---|
| a (Å) | 5.456(2) |
| b (Å) | 7.624(3) |
| c (Å) | 8.5442(2) |
| α=β=γ | 90° |
| v ($Å^3$) | 355.4(2) |
| symmetry | orthorhombic |

Table 1. Single crystal XRD data for BTSN-Kcl crystal

## 3.3 UV-Visible spectrum

The UV-vis-NIR transmittance spectrum wasrecorded using UV-vis-NIR spectrophotometer. Figure 4. shows the UV-vis-NIR spectrum recorded with highly transparent single crystal of BTSN-Kcl of thickness 2 mm. It is observed that the lower cut-off-wavelength of BTSN-Kcl crystal is at 300 nm and the crystal is found to be transparent in the region of 300-1100 nm. Using the formula $E_g = hc/\lambda$, the band gap energy was found to be 4.141 eV.

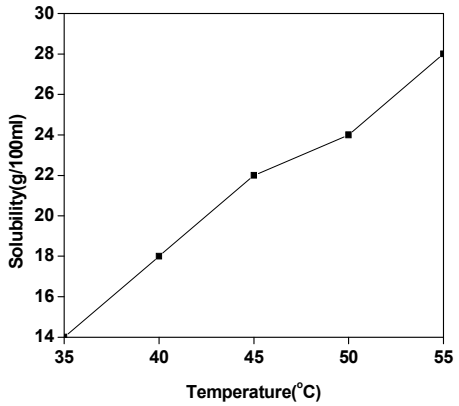



Figure 2. Solubility curve of BTSN-Kcl crystals

## 3.2 Characterization

## Powder X-ray diffraction analysis

The grown BTSN-Kcl crystals were finely powdered and have been subjected to powder XRD analysis. All the observed reflections were indexed. The well-defined Bragg's peaks at specific 2θ angles show high crystallinity of BTSN-Kcl.

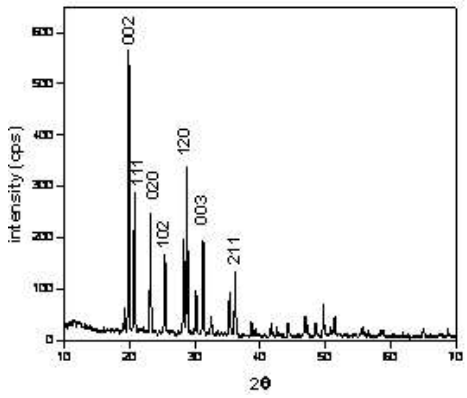



Figure 3. Powder X-ray diffraction

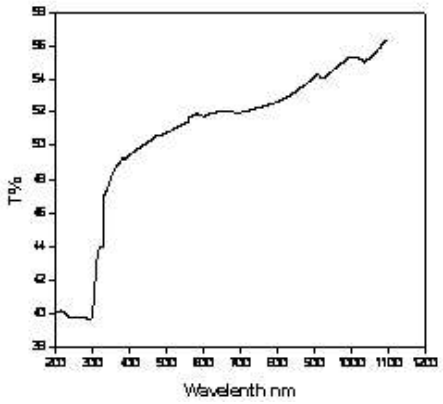



Figure 4. UV-Visible Spectrum of BTSN-Kcl

## 3.4 FTIR Studies

In this study the FTIR spectrum was recorded in the range 400-4000 $cm^{-1}$ for BTSN-Kcl single crystal. The FTIR spectrum of BTSN-Kcl is shown in Figure 3. In the FTIR spectrum of BTSN-Kcl, the broad and intense peak due to $NH_3^+$ stretching vibrations appeared as strong absorption band in the range 3153-3378$cm^{l}$ and hence hydrogen bond interactions involving $NH_3$ stretching vibrations could be strong. The absorption band at 1429 $cm^{-1}$ corresponds to $CH_2$ stretching vibration. The C=S asymmetric bending vibration at 1089 cm

$^{1}$ is shifted to lower frequency of 1080 cm$^{-1}$. The weak band observed at 480 cm$^{-1}$ in the Raman spectrum is assigned to K-Cl stretching vibrations. The peak observed at 2681 cm$^{-1}$ is due to $NH_2^-$ stretching vibrations. The absorption band at 623 cm$^{1}$ is assigned to out of plane bending vibration. The band at 1592 cm$^{-1}$ is assigned to N-C-N stretching vibration. Thus the FTIR spectrum confirms the formation of BTSN-Kcl and its characteristic frequencies are observed as mentioned above.

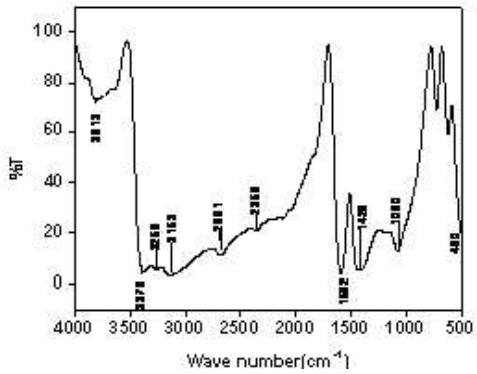



Figure 5. FTIR Spectrum of BTSN-Kcl

## 3.5 Vickers Microhardness Studies

Vickers microhardness measurements were carried out on BTSN-Kcl crystal using Vickers microhardness tester fitted with a diamond indentor. The indentations were made for various loads from 25 to 100 g. Vickers microhardness number ($H_v$) for BTSN-Kcl crystal is calculated using the following relation $H_v=1.8544\ P/d^2$ kg/mm$^2$ where P is the applied load in kg and d is the diagonal length of indentation impression in millimeter and 1.8544 is a constant of a geometrical factor for the diamond pyramid. A plot between the hardness number and the load is depicted in Figure 5. From the result, it is observed that hardness number increases with load.

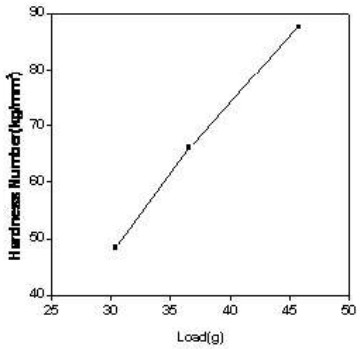



Figure 6. Variation of hardness with load for BTSN-Kcl crystal

## 4. Conclusion

BTSN-KcL single crystals was synthesized and its solubility was analysed in the temperature range 35-55°C.

Good quality of BTSN-Kcl crystals were grown by slow evaporation technique. Single crystal XRD analysis confirmed that the BTSN-Kcl crystallizes in orthorhombic system. The functional groups present in the grown crystal have been confirmed by FTIR spectral analysis. From UV-visible spectral studies, the cut-off wavelength was found. Powder X-ray diffraction pattern were obtained.

From Vickers microhardness studies, it is observed that the hardness number increases with the applied load.

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