Growth and Fourier Transform Infrared (FTIR) Spectroscopic Investigation of Potassium Pentaborate, $K(H_4B_5O_{10}).2H_2O$ [KB5] Crystal

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Abstract

Crystals of Potassium Pentaborate, $K(H_4B_5O_{10}).2H_2O$ [KB5] were grown by using solution growth technique at constant temperature 32°C. Spectroscopic grade of Potassium Carbonate, K_2CO_3, Boric Acid, H_3BO_3 and distilled-water were used to grow the crystals. The as-grown crystal was characterized by Fourier Transform Infrared (FTIR) spectroscopy. FTIR spectrum of the crystal was collected by PC-controlled SHIMADZU FTIR-8400 spectrophotometer in the wavenumber range of 400 cm^{-1} - 4000 cm^{-1} region using Potassium Bromide, KBr pellet method.

Key words: Potassium Pentaborate, FTIR, KBr pellet method

Introduction

In the recent past years, efforts have been made by many researchers to develop ultraviolet (UV) lasers for industrial and medical applications. The experiments conducted by Becker (1998) proved that inorganic borate crystals are superior to other commonly used Non-linear optics (NLO) materials for UV applications. Potassium pentaborate, $K(H_4B_5O_{10}).2H_2O$ (abbreviated as KB5) has been identified as an important material for non linear optical applications especially in visible and ultraviolet region. KB5 crystallizes in an orthorhombic crystal system with four molecules in the unit cell (Z=4) [1, 2, 4, 6].

Infrared spectroscopy is a versatile analytical technique. It is relatively easy to obtain spectra from solids, liquids and gases. The most popular way of obtaining infrared spectra is to pass the infrared beam directly through the sample, known as the transmission technique. Transmission method is the oldest and most basic infrared method. The advantage of this technique is the transmission spectra have signal-to-noise ratio. The method is based upon the absorption of infrared radiation at specific wavelengths as it passes through a sample. It is possible to analyze samples in liquid, solid or gas form by using this approach. Potassium Bromide (KBr) pellets are used to obtain the infrared spectra of solids, and are particularly well suited to powder samples. KBr is an inert, infrared transparent material, and acts as a support and a diluent for the sample. The most commonly used alkali halide is potassium bromide (KBr), which is completely transparent in the middle of IR region [3, 7, 8].

In the present work, Potassium pentaborate single crystals were grown in distilled-water using slow evaporation technique. The as-grown crystals were characterized by Fourier Transform Infrared (FTIR) spectroscopy.

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Experiment

Growth of Potassium Pentaborate, $K(H_4B_5O_{10}).2H_2O$ [KB5] Crystal

Crystals of Potassium Pentaborate [KB5] were grown by solution growth technique from the aqueous solutions containing the mixture of Potassium Carbonate, K_2CO_3 and Boric Acid, H_3BO_3 with equimolar ratio [4, 5, 9]. The solution was prepared at the temperature higher than room temperature (typically 40°C to 45°C). The saturated solution was filtered into the beaker and warmed about 5°C higher than the previous temperature. The beaker is then covered and set inside the water bath. The temperature of water bath was controlled by temperature controller to maintain the temperature at 305 K (32°C). After 15 days, seed crystals of KB5 were drawn in the saturated solution and collected with tweezer placed on filter paper to dry. Transparent and homogeneous crystal was selected for FTIR measurement. At room temperature, KB5 crystal is colourless. Photograph of the crystal growth condition is shown in Fig 1.

Fig 1 Photograph of the crystal growth condition of KB5

FTIR Spectroscopic Measurement

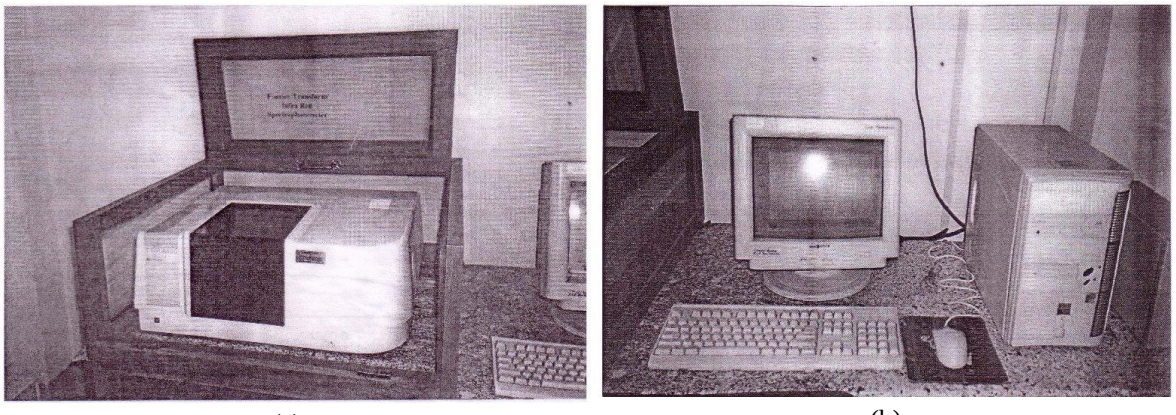
In the present work, FTIR transmission spectrum of the KB5 crystal was recorded on SHIMADZU FTIR-8400 Spectrophotometer at room temperature. This experiment was performed at the Medical Research Laboratory, Department of Medical Research (Lower Myanmar). Photographs of the SHIMADZU FTIR-8400 spectrophotometer and its PC-control system are shown in Fig 2(a) and (b). Experimental conditions were as follows:

Measurement mode : %T

Wavenumber range : 400 cm^{-1} – 4000 cm^{-1}

Number of scan : 60 s

Method : KBr pellet



(a) (b)

Results and Discussion

FTIR transmission spectrum of Potassium Pentaborate [KB5] crystal is shown in Fig 3. The observed wavenumbers (absorption lines) and corresponding vibrational mode assignments of molecules are tabulated in Table 1.

The bands observed in the IR spectrum at 448 cm^{-1} and 532 cm^{-1} are assigned to ring OBO symmetric bendings. The ring asymmetric bending vibration is found at 602 cm^{-1} with the couple of symmetric bendings.

Of course, the B – O vibrations of borate crystals have their absorption bands in the frequency region 784 cm^{-1} –1438 cm^{-1} [5, 7, 9]. The strong bands observed in the IR spectrum of KB5 at 833 cm^{-1} and 957 cm^{-1} are assigned to ring B – O symmetric stretching vibrations. The ring B – O asymmetric stretching vibrations were appeared at 1065 cm^{-1} and 1142 cm^{-1} with very strong intensity.

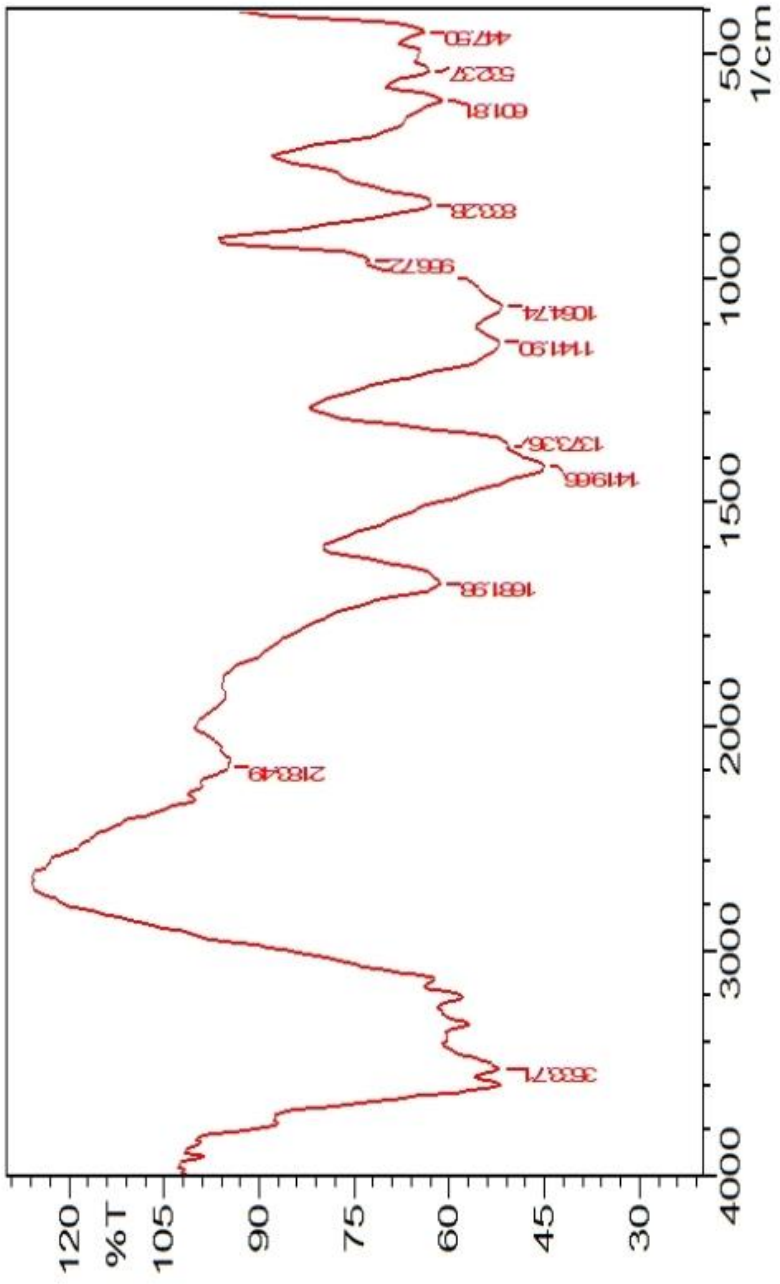


Fig 3 FTIR transmission spectrum of Potassium Pentaborate [KB5] crystal

Table 1 Wavenumbers (absorption lines) and corresponding vibrational characterizations and mode assignments of Potassium Pentaborate [KB5] crystal

Line No	Wavenumber (cm^{-1})	Vibrational Characteristics
1	448 / 532	ring OBO symmetric bendings
2	602	ring OBO asymmetric bendings
3	833 / 957	ring B – O symmetric stretching vibrations
4	1065 / 1142	ring B – O asymmetric stretching vibrations
5	1373	B – O terminal symmetric stretching
6	1420	B – O terminal asymmetric stretching
7	1682	bending vibration of H_2O
8	3534	asymmetric stretching vibration of H_2O
9	2183	bending vibration of CO_2

The very strong peak at 1373 cm^{-1} in the infrared spectrum was attributed to B – O terminal symmetric stretching vibration. The B – O terminal asymmetric stretching was observed in the IR spectrum at 1420 cm^{-1} with strong intensity.

The band observed at 1682 cm^{-1} was represented by the bending vibration of H_2O (water) and the very strong band observed at 3534 cm^{-1} were assigned to asymmetric stretching vibration of H_2O or O – H stretching vibration band. In this band, symmetric stretching vibration of H_2O may be overlapped under the shoulder between the wavenumbers range of 3100 cm^{-1} and 3700 cm^{-1}. The band observed at 2183 cm^{-1} was indicated by the bending vibration of CO_2.

Conclusion

Potassium Pentaborate [KB5] crystals were grown by solution growth technique at constant temperature 32°C. The as-grown KB5 crystal was characterized by FTIR spectroscopy. From the FTIR spectrum, twelve absorption lines were observed in the wavenumber range of 400 cm^{-1} – 4000 cm^{-1} region. These lines are characteristics of borate and water molecules that constituents in KB5 crystal. These lines are also represented by the four types of molecular group vibrations; namely, ring B – O, B – O terminal, H_2O and CO_2 respectively. The collected wavenumbers were precisely assigned.

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