

Thermal properties of $Ba_xSr_{1-x}(NO_3)_2$ mixed crystals

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ABSTRACT: $Ba(NO_3)_2$ and $Sr(NO_3)_2$ are isomorphous materials and have cubic structure. The use of these crystals as stimulated Raman shifters, optical filters and intracavity Raman lasers made these crystals the subject of numerous investigations in to various physical properties. $Ba_xSr_{1-x}(NO_3)_2$ mixed crystals (x is composition of Barium) have been grown from solutions by slow evaporation and their X-ray patterns have been recorded using a powder diffractometer. From measurements of Bragg reflections, lattice constants, mean Debye-Waller factors, Debye temperatures have been determined. The miscibility and the Vegard's law have been tested. Single phase mixed crystals were formed. The mean Debye-Waller factor and Debye temperature shows a non-linear dependence of composition, with positive and negative deviations from linearity respectively. Phase transition temperatures have been identified from specific heat and resistance measurements. Debye temperatures estimated from specific heats were compared with those from X-rays. Composition dependence of specific heat and resistance both show negative deviation from linearity. The anomalies observed from specific heat and resistance measurements were compared with those obtained from other methods and discussed in light of metastable behavior of the mixed crystals. Debye typed transitions were observed which were attributed to structural disorder and is due to reorientation of NO_3^- group. The composition dependence of melting points measured from DTA experiments found to be non-linear with negative deviations from linearity.

I. INTRODUCTION

Barium nitrate, strontium nitrate, lead nitrate and calcium nitrate are molecular cubic crystals and belong to a family of isomorphous materials. These crystals are used as Stimulated Raman Shifters (SRS) and as optical filters [1,2]. $Sr(NO_3)_2$ is a promising crystal for coupled intra-cavity Raman lasers [3]. The space group of these crystals is $Pa\bar{3}$ instead of $P2_13$ [4]. Crystallization behavior of $Pb_xSr_{1-x}(NO_3)_2$ and $Ba_xSr_{1-x}(NO_3)_2$ solid solutions from aqueous solutions was studied by Wu et al [5,6]. For both the systems, mixed crystals were grown over all compositions and reported Vegard's law behavior for lattice constants. The results on the crystal growth, habit surface characterization, dielectric studies of these mixed crystals were studied in this laboratory [7]. In this paper the results on some of the properties of $Ba(NO_3)_2$ - $Sr(NO_3)_2$ mixed crystal system are reported. Accurate lattice parameter measurements have been carried out. The mean Debye-Waller factors have been determined from the integrated intensities and the Debye characteristic temperatures were derived. Their composition dependence is also discussed in terms of the degree of disorder of the arrangement of metal ions in the lattice. Critical examination of the temperature variation of the specific heat and resistance is also made with respect to the onset offset of the transitions. Thermal behavior is also tested and melting points were identified for these mixed crystals.

II. EXPERIMENTAL METHODS

The details of the crystal growth and composition determinations are described elsewhere [8]. The powder X-ray diffraction (PXRD) patterns were recorded using JEOL-JDX-8P X-ray diffractometer fitted with a scintillation counter and nickel filtered CuK_α radiation ($\lambda=1.54056 \text{ \AA}$). The Bragg reflections were recorded in θ - 2θ scan with a goniometer speed of $0.5^\circ/\text{min}$ and a chart speed of 40 mm/min , over an angular range of 10 to 120° . The integrated intensities have been corrected for thermal diffuse scattering (TDS) [9]. Specific heats are measured using semi-adiabatic heat pulse

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 11, November 2015

method. The change in material temperature is measured before and after the supply of heat pulse. The instrument is calibrated with copper and the values are accurate upto 0.5%. The low temperature resistance measurement is done in a liquid nitrogen bath using Keithley model 6517A programmable electrometer. A Lakeshore model 340 temperature controller is used for controlling and measuring the temperature. The measured resistance is accurate within 5%. Melting points were determined by recording thermograms using Mettler Toledo STAR system. The accuracy in temperature is $\pm 1^\circ\text{C}$. The resistance is standardized for KBr sample. The accuracy in melting point is $\pm 5^\circ\text{C}$.

III. RESULTS AND DISCUSSIONS

Fig. 1. shows the fast scan X-ray powder diffractograms of pure $\text{Ba}(\text{NO}_3)_2$ and three of the $\text{Ba}_x\text{Sr}_{1-x}(\text{NO}_3)_2$ mixed crystals. For all the crystals the diffraction peaks are well defined and sharp indicating the formation of single phase mixed crystals. Each diffraction line split in to two in the composition range $0.3 \leq x \leq 0.6$ indicating the presence of two phases.

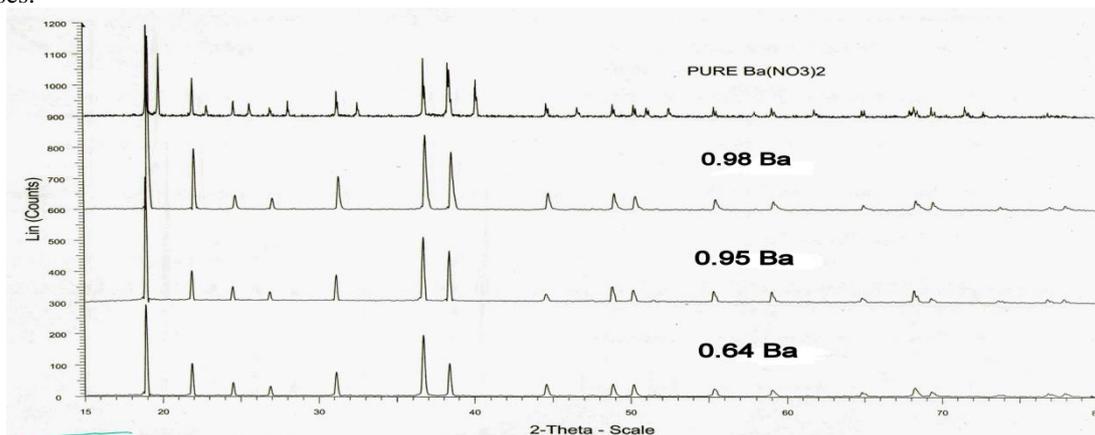


Fig. 1. Typical X-ray diffractograms for pure $\text{Ba}(\text{NO}_3)_2$ and some of the mixed crystals of $\text{Ba}_x\text{Sr}_{1-x}(\text{NO}_3)_2$ system.

A.LATTICE CONSTANT

Accurate lattice constant (a_0) was determined by least squares treatment of the values of lattice constants obtained for several reflections and the error function $\cos^2 \theta$. The values of lattice constants obtained in this study for $\text{Ba}_x\text{Sr}_{1-x}(\text{NO}_3)_2$ mixed crystals follow a linear equation below indicating the Vegard's law behavior.

$$y = (7.767 \pm 0.009) + (0.364 \pm 0.014)x \tag{1}$$

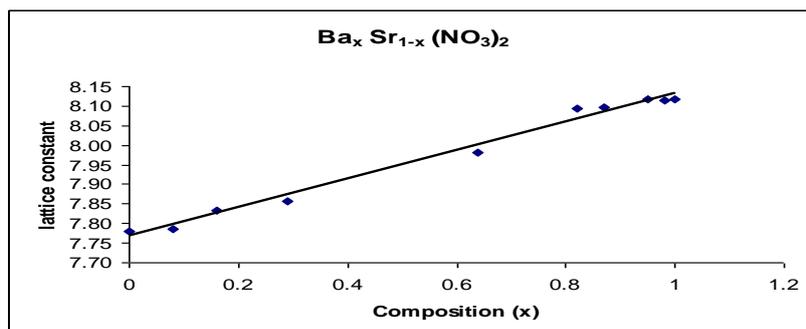


Fig.2. Plot of lattice constant (a_0) versus composition (x).

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 11, November 2015

The lattice constants for pure $\text{Ba}(\text{NO}_3)_2$, $\text{Sr}(\text{NO}_3)_2$ are in good agreement with those from National Bureau of Standards (NBS) circular values [4]. There is scattering in the distribution of experimental values of the lattice constants for mixed crystals. But the deviations are on either side of the line connecting end members, indicating Vegard's law behavior for the lattice constants. The deviations are very small and are of the order of accuracy in the measurement of lattice constants. Wu et al [5, 6] also reported Vegard's law behavior for lattice constants of this system.

In the present work the samples with intermediate compositions were found to consist of two phases indicating the miscibility gap. In a series of growth experiments conducted we obtained single phase mixed crystals only in the range $x < 0.3$ and $x > 0.6$. The particle size is estimated using the Scherer formula. The crystallite size of the samples varied between 70-90 nm.

B. DEBYE-WALLER FACTOR AND DEBYE TEMPERATURE

The Debye-Waller factor is determined from intensities. The experimental conditions and procedure of sample preparation are such that errors in intensity due to surface roughness, porosity and extinction are minimized. The integrated intensity I_o of a Bragg reflection of a powder sample may be written as

$$I_o = C I_c \exp(-2B \sin^2 \theta / \lambda^2) \quad (2)$$

where C is the scale factor and B, the mean Debye-Waller factor and I_c , the calculated intensity which is given by

$$I_c = (LP) J F^2 \quad (3)$$

where LP is the Lorentz Polarization factor, J the multiplicity factor and F, the structure factor. For alkaline earth metal nitrates the structure factor can be written using the data of coordinates of equivalent positions [10] as

$$F(hkl) = f_m^o \exp(-M_m) + 8 f_N^o \{ \cos(2\pi)hu \cos(2\pi)ku \cos(2\pi)lu \} \exp(-M_N) + 8 f_O^o \left\{ \begin{array}{l} \cos(2\pi)hx \cos(2\pi)ky \cos(2\pi)lz + \cos(2\pi)hy \cos(2\pi)kz \\ \cos(2\pi)lx + \cos(2\pi)hz \cos(2\pi)kx \cos(2\pi)ly \end{array} \right\} \exp(-M_o) \quad (4)$$

where m, N and O stand for metallic ions (Ba and Sr), nitrogen atoms and oxygen atoms respectively; f_m^o , f_N^o and f_O^o are atomic scattering factors [11] and u, x, y, and z are positional parameters [12]. Since the masses of nitrogen and oxygen differ by only two atomic mass units, one can approximately take $M_N \approx M_O$. The atomic scattering factors are corrected for anomalous dispersion [13]. The mean Debye-Waller factor is determined from a least squares treatment of data on the log (I_o/I_c) versus $(\sin \theta / \lambda)^2$.

For the mixed crystals the Debye-Waller factor thus determined (B_{obs}) is the sum of the true thermal Debye-Waller factor ($B_{thermal}$) and a static component (B_{static}) due to the presence of two types of metallic ions in the mixed crystal. $B_{thermal}$ is obtained from B_{obs} by applying a correction for B_{static} [14].

$$B_{obs} = B_{thermal} + B_{static} \quad (5)$$

B_{obs} is determined using Debye-Waller theory for a cubic crystal extensively discussed [15-19].

Values of the mean Debye-Waller factor (B) and Debye temperature (θ_M) for $\text{Ba}_x\text{Sr}_{1-x}(\text{NO}_3)_2$ crystals are plotted in Fig. 3. From Fig. 3(a) it can be seen that the mean Debye-Waller Factor increases with mixing from either end assuming the highest value in the equimolar region. In the absence of any earlier work on this property and the results in the present case are compared with those of alkali halide mixed crystal systems. The treasure of data on alkali halide mixed crystals has been reviewed extensively [19]. The review reveals that in all the alkali halide mixed crystal systems the Debye-Waller factor vary non-linearly with composition with positive deviations from linearity. The composition dependence of the Debye temperature is non linear with negative deviations from linearity. The deviations reported are of the order of the experimental error except in the case of RbBr-RbI, where it is larger than the experimental error. In the present case also the deviations from linearity are much larger than the experimental error. This is the clear indication that the nature of lattice vibrations in $\text{Ba}_x\text{Sr}_{1-x}(\text{NO}_3)_2$ lattices is not the same as that of the alkali halide mixed crystal systems. The larger difference between experimental and calculated values of B and θ_M also

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 11, November 2015

reflects the degree of the disorder of the arrangement of the metal ions in the lattice. Therefore it is concluded that the $Ba_xSr_{1-x}(NO_3)_2$ lattices is more disordered than that in alkali halide mixed crystals.

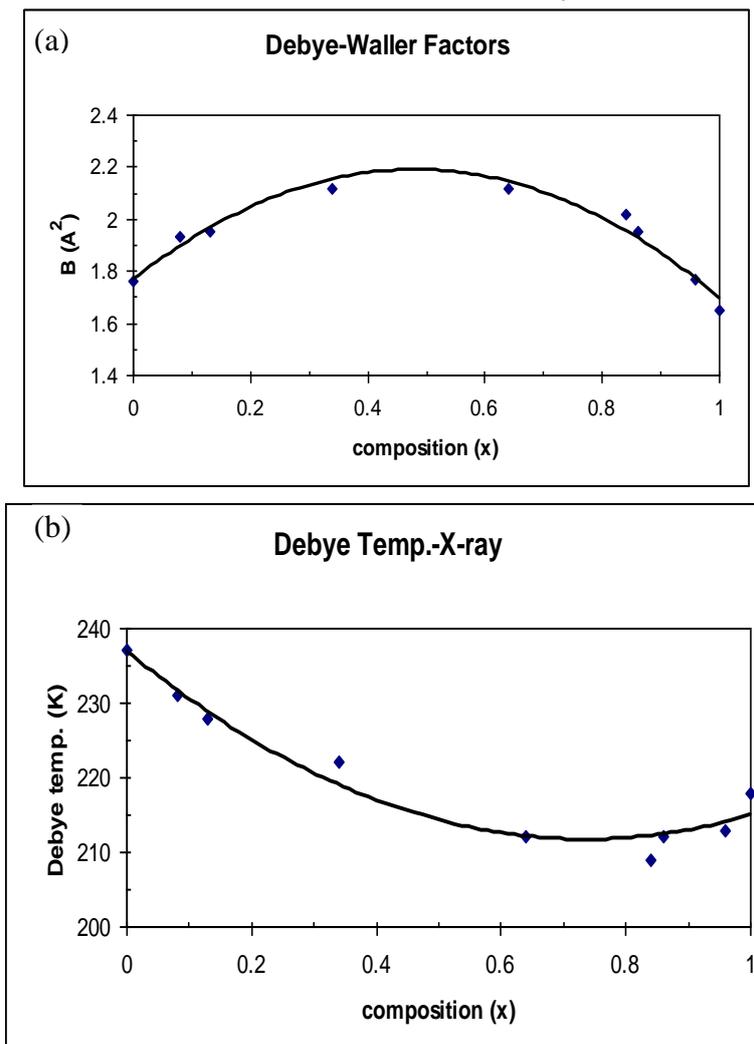


Fig. 3. (a) Plot of Debye Waller factors (B) for $Ba_xSr_{1-x}(NO_3)_2$ crystals, (b) Plot of Debye temperature (θ_M) for $Ba_xSr_{1-x}(NO_3)_2$ crystals.

C. SPECIFIC HEAT AND DEBYE TEMPERATURE

Fig. 4(a) & (b) shows the typical variation of C_p in temperature range from liquid helium to 320 °K, in the units of J/mol-K, for pure $Sr(NO_3)_2$ and $Ba_{0.66}Sr_{0.34}(NO_3)_2$ crystals. The most prominent feature of the C_p Vs.T plot is the appearance of two peaks for $Sr(NO_3)_2$. The maximum of these peaks correspond to 256 °K and 302 °K for $Sr(NO_3)_2$. These peaks are sharper compared to that reported for the polycrystalline sample [20]. Similar observations were made in the case of the $Ba(NO_3)_2$ and mixed crystals with four compositions.

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 11, November 2015

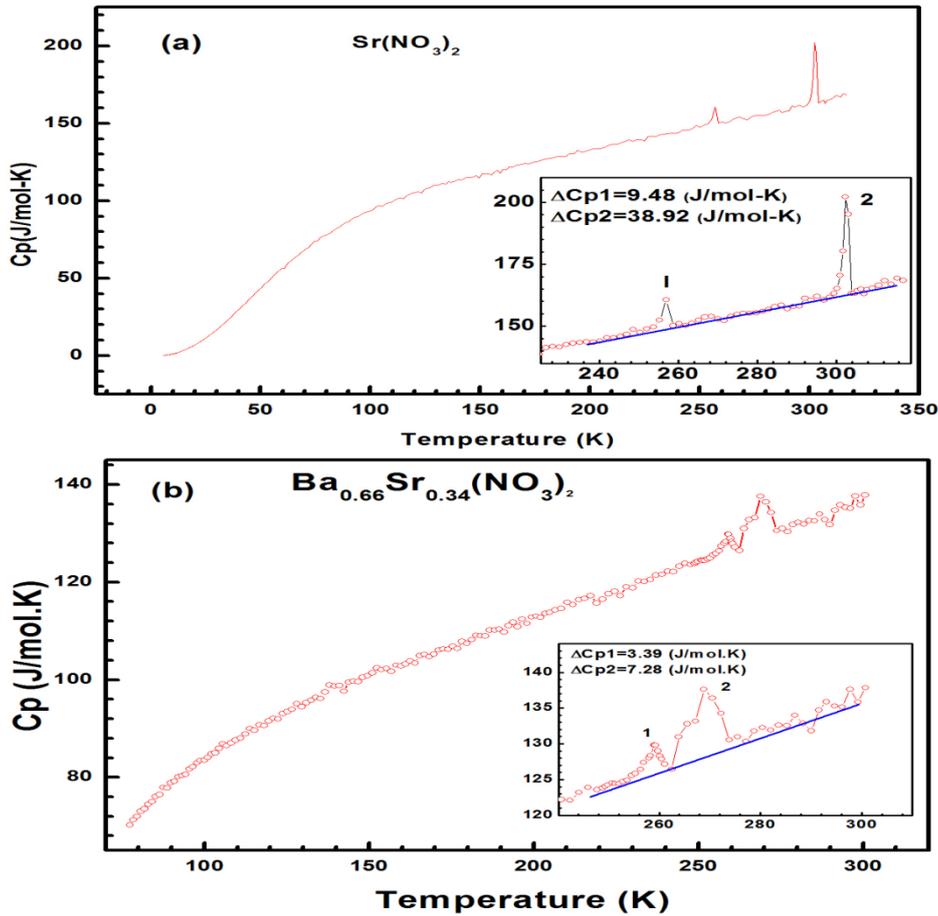


Fig. 4 (a) & (b). Temperature dependence of specific heat for (a) $\text{Sr}(\text{NO}_3)_2$ (b) $\text{Ba}_x\text{Sr}_{1-x}(\text{NO}_3)_2$ with $x=0.66$.

The composition dependence of specific heat for the system under study is no linear with respective deviations from linearity [Fig.5]. The transition enthalpies ΔH was deduced from the excess heat capacities in the higher and the lower temperature regions [21]. The values of ΔH derived from ΔC_p are 5.63 J/mol, 27.36 J/mol for $\text{Ba}(\text{NO}_3)_2$ and 53.45 J/mol, 316.85 J/mol for $\text{Sr}(\text{NO}_3)_2$.

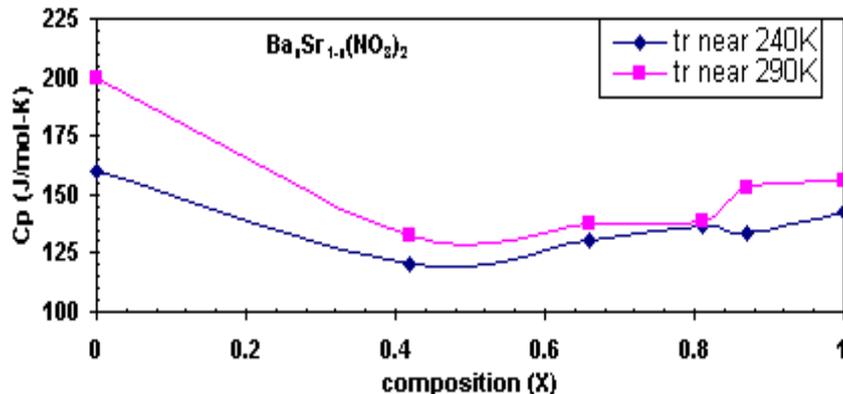


Fig. 5. Composition dependence of C_p for $\text{Ba}_x\text{Sr}_{1-x}(\text{NO}_3)_2$ mixed crystals.

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 11, November 2015

As seen from Fig. 4 (a) & (b) thermal variation of the heat capacity exhibits departure from the classical Debye typed one. The present experimental results evidence a continuous increase of the heat capacity till 320 K except in the surroundings of the transition phenomena. Such a large departure from the Debye typed behavior of the heat capacity is attributed to structural disorder. For a Debye typed behavior, a Dulong-Petit plateau is expected. In general, nitrates are expected to exhibit various disorders as a function of temperature due to re-orientation of NO₃⁻ group [22]. The study of the three nitrates namely, lead, barium and strontium nitrate in the temperature range 0-300 °K by resonance and pulse echo ultrasonic methods does not reveal any abnormal change of the elastic components C₁₁, C₁₂ and C₄₄ [23]. They reported a continuous decrease associated to an harmonic properties of the crystal.

The Debye characteristic temperature (θ_D) values obtained from the slopes of the C_p/T Vs. T² plots are $\theta_D = 196$ °K for Ba(NO₃)₂ and $\theta_D = 249$ °K for Sr(NO₃)₂. The Debye temperatures have also been obtained for these crystals from X-ray diffraction. The values are $\theta_M = 218$ °K for Ba(NO₃)₂ and $\theta_M = 237$ °K for Sr(NO₃)₂.

The specific heat for four mixed crystals of Ba_xSr_{1-x}(NO₃)₂ system are measured. The Debye temperatures have been obtained for two mixed crystals with compositions x=0.81 and 0.87 and are 205 K and 208 K respectively. For other two crystals since the measurements were made only from liquid nitrogen temperatures, the Debye temperatures were not obtained. The temperatures at which the occurrence of transition phenomenon observed along with the increase in specific heat are tabulated in the Table 1.

Table.1. Anomalies in C_p for Ba_xSr_{1-x}(NO₃)₂ mixed crystals.

composition (x)	at transition		
	temp.(°K)	C _p (J/mol K)	Δ C _p (J/mol K)
0.00	256	160	9.48
	302	200	38.92
0.42	235	120	3.25
	298	133	2.00
0.66	258	130	3.39
	269	138	7.28
0.81	259	137	4.62
	271	139	2.68
0.87	228	134	2.30
	272	153	7.72
1.00	227	143	2.31
	274	156	4.20

D.RESISTANCE

The resistance of Ba(NO₃)₂ and Sr(NO₃)₂ and their mixed crystals with various barium concentrations has been measured between 77°K-310°K under identical conditions. The typical results are shown in Fig. 6. Above 220°K, the resistance suddenly falls down to a value much smaller than that in the low temperature range. And on further heating, the resistance appears to be independent of temperature. In the case of barium nitrate the resistance increases at a faster rate below 220°K and exhibit two sharp peaks one at 236.6°K and the other at 260.2°K. The magnitude of resistance in this temperature range is of the order of 5 x 10¹² ohms. After transition the resistance decreased to a value of about 2x 10¹¹ ohms. On the whole the variation of resistance with temperature for barium nitrate is between 10¹¹ to 10¹² ohms.

Similarly in the case of Sr(NO₃)₂ the resistance varied from 2x10¹³ ohms to 5x 10¹² ohms when the temperature is increased from 100°K–300°K. Similar to Ba(NO₃)₂ and Sr(NO₃)₂ also exhibited anomalies in resistance at temperatures 213°K, 240°K and 256°K. In both the cases the order of the resistance is very much within the resistance range of insulators. In general the resistance of insulators varies between 10⁹ ohms to 10¹⁸ ohms. The resistance of these nitrates

International Journal of Innovative Research in Science, Engineering and Technology

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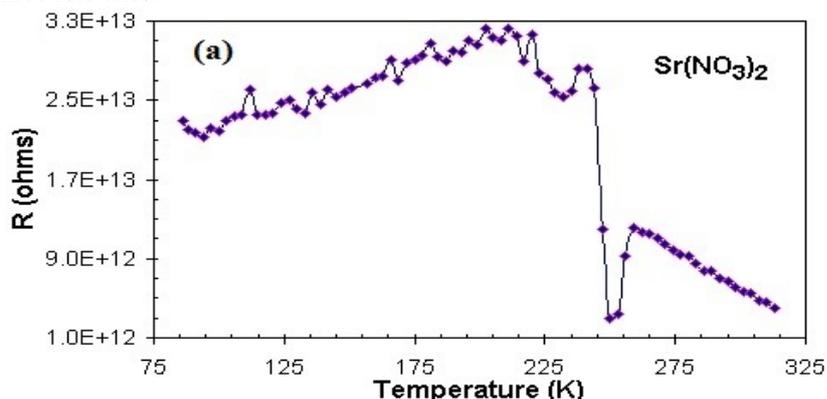
Vol. 4, Issue 11, November 2015

occupies the bottom of the resistance spectrum of insulators. Diamond, mica, marble and Bakelite are the insulators whose resistance is comparable to that of the alkaline earth nitrates. The anomalies observed in the present work are sharp, with the relaxation width varied between two degrees to about twenty one degrees. The display of more than one anomaly by these crystals is the clear indication that they are metastable in their electrical properties with temperature. The values of temperatures at the onset and off set of the transitions along with the resistance are shown in Table 2.

Table 2. On set and off set of transitions in Resistance measurements for the $Ba_xSr_{1-x}(NO_3)_2$ mixed crystals.

Compo- sition (x)	On set of transition		Off set of transition	
	Temp.(K)	Resistance (Ohms)	Temp.(K)	Resistance (Ohms)
0.00	238	2.81×10^{13}	259	1.21×10^{13}
0.13	244	6.66×10^{11}	313	3.10×10^{11}
0.23	238	3.10×10^{11}	---	---
0.38	232	6.75×10^{10}	244	5.36×10^{10}
0.57	240	4.26×10^{11}	256	6.98×10^8
0.73	255	5.83×10^{11}	258	1.36×10^9
0.86	244	4.82×10^{11}	247	4.58×10^{11}
0.93	237	7.07×10^{11}	264	3.53×10^{11}
0.98	260	9.69×10^{11}	267	6.59×10^{10}
1.00	261	5.8×10^{12}	270	2.16×10^{11}

From table 2, it can be observed that the anomalies in resistance occurred around 240 °K and 260 °K. Similar anomalies have also been observed by Badr and Kamel (1979) [24] from resistivity measurements. Their results have shown anomaly in resistivities at temperatures about 220 °K for $Ba(NO_3)_2$ and above 200 °K for $Sr(NO_3)_2$. Bjorseth (1971) [25] also studied the resistivity and obtained a minimum in the resistivity curve at about 200 °K for $Ba(NO_3)_2$ and the slope of the resistivity curve shown a change at about 240 °K. In the present measurement also small anomalies are observed at around 200-220 °K. But they are not strong. They indicate the metastable behavior of the crystals in the system. Badr and Kamel (1979) [24] attributed these low temperature transitions in the nitrate crystals to the re-orientational motion of nitrate group about its three fold axes.



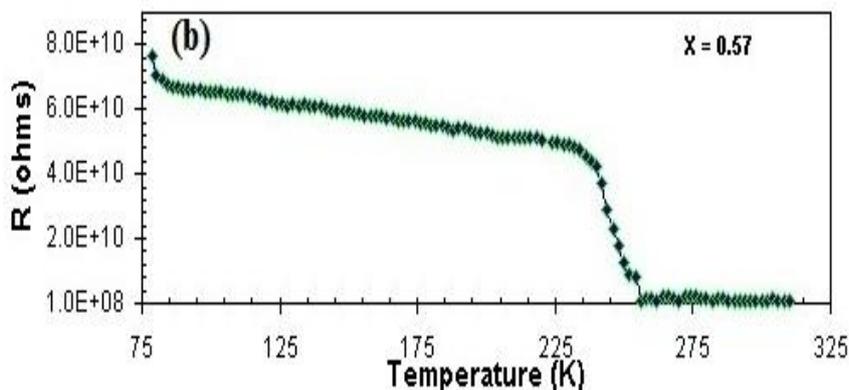


Fig. 6. Temperature dependence of resistance for (a) $\text{Sr}(\text{NO}_3)_2$ (b) $\text{Ba}_x\text{Sr}_{1-x}(\text{NO}_3)_2$.

Badr and Kamel (1979) [24] reported anomaly at 193 °K for $\text{Sr}(\text{NO}_3)_2$ and 220 °K for $\text{Ba}(\text{NO}_3)_2$ from resistivity measurements. Whereas Bjorseth (1971) [25] reported anomaly for $\text{Ba}(\text{NO}_3)_2$ at 200 °K from resistivity and 220 °K from dielectric constant measurements. In the present work the anomaly at 200 °K or 220 °K has not been detected by any method. May be the anomalies at 193 °K, 200 °K, 220 °K, indicate a single transition, which is common to both $\text{Ba}(\text{NO}_3)_2$ and $\text{Sr}(\text{NO}_3)_2$. This may also be due to the difference in sensitivities of the methods of measuring technique. The exact value of transition temperature varied from method to method and uncertainty in the transition temperatures from different methods may be taken as ± 5 °K to ± 10 °K. This large uncertainty is partly due to the different sensitivities of measurements by different methods and partly due to the large metastable behavior of these nitrates, evident from all the studies including the present one.

E.MELTING POINTS

A typical thermogram is shown in Fig. 7. As all the crystals are stable thermally upto about 500 °C indicates no water of hydration in the crystals. The melting point of $\text{Ba}(\text{NO}_3)_2$ obtained in the present work is 592°C which is in good agreement with the early reported value of 590°C [26]. Fig. 8 shows the composition dependence of melting points for the mixed crystal system under study. The composition dependence of melting temperature is slightly nonlinear with negative deviation from linearity. This is similar to the observation of Sirdeshmukh et al (1990) [27] for KCl-KBr and Subhadra and Hussain (1987) [28] for NaClO_3 - NaBrO_3 mixed crystals. The maximum deviation occurs at about the equimolar composition. This deviation is larger than the accuracy in the measurement.

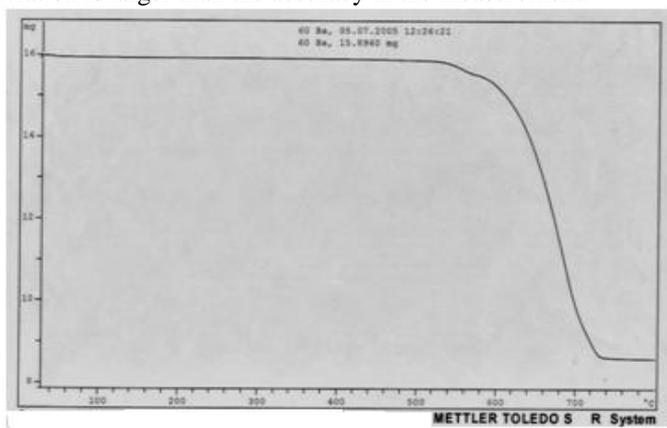


Fig. 7. Thermogram of $\text{Ba}_{0.58}\text{Sr}_{0.42}(\text{NO}_3)_2$.

International Journal of Innovative Research in Science, Engineering and Technology

(An ISO 3297: 2007 Certified Organization)

Vol. 4, Issue 11, November 2015

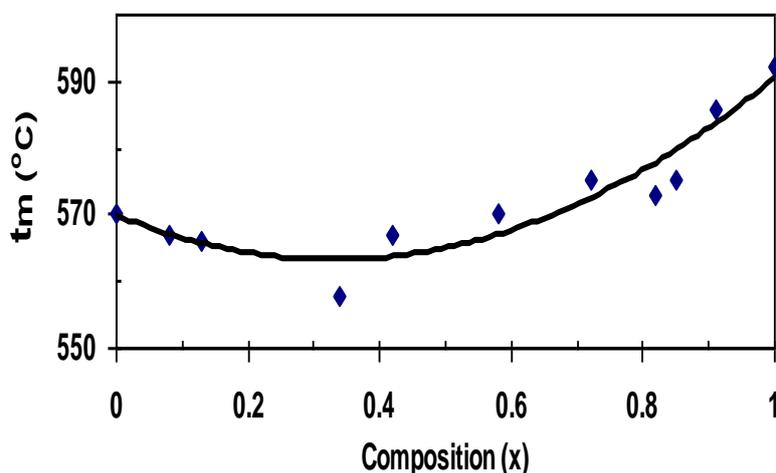


Fig. 8. Variation of melting points with composition (x) for $Ba_xSr_{1-x}(NO_3)_2$ mixed crystal system.

IV. CONCLUSIONS

A detailed X-ray study of $Ba_xSr_{1-x}(NO_3)_2$ mixed crystal system has been carried out. Lattice constants and thermal properties like Debye-Waller factors, Debye temperatures, specific heats and melting points have been determined. The composition dependence of

- i) the lattice constant follows Vegard's law,
- ii) Debye-waller factors show a non-linear behavior with positive deviations from linearity,
- iii) Debye temperatures show a non-linear behavior with negative deviations from linearity.
- iv) specific heats show a non linear behavior with negative deviations from linearity.
- v) resistance of these crystals is very much in the range of insulator before and after transition.
- vi) melting points show a non linear behavior with respective deviations from linearity.
- vii) The thermal behavior of $Ba_xSr_{1-x}(NO_3)_2$ crystals is highly disordered.

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