

# DC Conductivity and Structural Studies in Potassium, Strontium Doped Boro-Phosphate Glasses

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**Abstract:** A series of five samples of boro-phosphate glasses doped with K<sub>2</sub>O and SrO within the composition of 20(B<sub>2</sub>O<sub>3</sub>) + 30(P<sub>2</sub>O<sub>5</sub>) + x (SrO) + (50-x) (K<sub>2</sub>O) where x = 10, 20, 30, 40 and 50 were prepared by the melt quench technique. The samples were annealed to remove if any thermal strains present. The Non-crystalline nature of the glass samples was confirmed by XRD studies. The room temperature density of the glass samples has been measured by adopting Archimedes principle and the molar volume was estimated. The density decreases and molar volume increases up to 30 mole fraction of SrO and further increase of SrO the density increases and molar volume decreases. The high temperature dc activation energy has been calculated by fitting the dc experimental data for Nernst Einstein equation. The high temperature activation energy and conductivity were varies non-linearly and behaving appositively with mole fraction of SrO. The diffusion coefficient has been calculated using Nernst-Einstein relation which varies nonlinearly with mole fraction of SrO. The resultant nonlinear variation of density, molar volume, high temperature conductivity, dc activation energy occurs in the present series. Key words: - Boro-Phosphate glasses, SrO, K<sub>2</sub>O, ionic conductivity, NBO's.

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## 1. INTRODUCTION:

Glassy electrolytes characterized as amorphous structures are now widely used for solid state ionic devices such as lithium ion batteries, sensors, fuel cells, electro chromic displays and in high energy density batteries etc. Warburg et al in 1984 explored ion transport in glasses by applying dc voltage [1-5]. The ionic conductivity in oxide glass systems doped with alkali and alkaline earth ions is due to migration of ions from one ionic site to another ionic site by hopping [6-7]. However the amorphous structure and non-equilibrium nature of oxide glass systems often hinder the fundamental understanding of their ionic transport mechanism [8]. Generally the fast ion conducting glasses are formed by three components; glass network former, metal oxides acting as a glass network modifier and dopant cations. The glass network formers are oxide materials of covalent nature, the assembly of oxygen tetrahedral or trigonals for example B<sub>2</sub>O<sub>3</sub> and P<sub>2</sub>O<sub>5</sub> etc [9]. In ion conducting glasses the electronic contribution to the total conductivity is usually very weak, which is the consequence of periodic potential fluctuations imposed by the disordered structure [10]. Researchers reported mixed cation effect in ionic diffusivity as well as in the glass transition temperature in a glass system xNa<sub>2</sub>O-(1-x) CaO-SiO<sub>2</sub> and xK<sub>2</sub>O-(1-x) BaO-SiO<sub>2</sub> [11]. In these glasses the mixed cation effect is due to movement of two dissimilar ions of unequal size are escorted by the formation of intrinsic defects in glass forming network when mobile cation enters foreign sites. It is somewhat similar and opposite to mixed alkali effect in mixed alkali glasses but size is very less pronounced due to smaller mobility of the alkaline earth cations. In the alkali and alkaline earth glass systems the energy land scape is contain monocharge alkali ion sites and double charged alkaline earth ion sites and both types mobile cations apply either of vacant sites without readjustment [11 12]. The presence of single charged alkali ions in a number of alkali and alkaline earth glass systems, binary charged alkaline earth cations proved greater diffusivities than in two alkaline earth glasses [13, 14]. In many oxide glass systems mixed cation effect has not been observed [15, 16]. Only few of the researchers reported the classical mixed cation effect in few of the oxide glass systems due to migration of single cation species [17-18]. It is known that the structure and properties of oxide glasses are strongly dependent on the nature and concentration of the constituent oxides and dopants. Although there exist numerous studies on binary and ternary tellurite and phosphate glasses containing B<sub>2</sub>O<sub>3</sub>, MgO, NiO and WO<sub>3</sub>, on thermal and structural behaviour studied in detail [19-20]. In many ion conducting glass systems the Nernst-Einstein relation have been used to relate the diffusion coefficient and electrical conductivity [28]. In the present paper, the experimental results on XRD studies, room temperature density, molar volume, temperature dependent dc conductivity, high temperature activation energy studies on boro-phosphate glass systems doped with K<sub>2</sub>O and SrO in the composition range 20(B<sub>2</sub>O<sub>3</sub>) + 30(P<sub>2</sub>O<sub>5</sub>) + x (SrO) + (50-x) (K<sub>2</sub>O) where x = 10, 20, 30, 40 and 50 are presented.

## 2. THEORY :

### 2.1 Density and molar volume

The density of glass samples generally is given by Archimedes principle equation as,

$$\rho = \frac{W_{\text{air}}}{W_{\text{air}} - W_L} \rho_L \text{ g/cm}^3 \quad (1)$$

molar volume of the glass samples is given by the following formula,

$$V_i = \frac{X_i M_i}{\rho_i} \quad (2)$$

Where ' $V_i$ ' molar volume, ' $x_i$ ' is the mole fraction, ' $M_i$ ' is the molecular weight and ' $\rho_i$ ' is the density of  $i^{\text{th}}$  glass sample where  $i = 1, 2, 3, 4$  and  $5^{\text{th}}$  glass sample. In oxide glasses mobile cationic concentration ( $N$ ) has been evaluated using density values and the following equation,

$$N = 2[(\rho m_{\text{Alkali}}/M_{\text{Alkali}})N_A] \quad (3)$$

### 2.2 Electrical conductivity

As per as many theoretical predictions and experimental results, various theoretical models were proposed to explain the conduction mechanism in ion conducting glass systems [29-30]. However no single theoretical model investigated till today which would explain completely the ion conduction mechanism over entire temperature range. In general the total conductivity of material  $\sigma_T$  is equal to the sum of the contributions of all charge carrying species  $\sigma_i$  and is given by

$$\sigma_i = \sum_i \sigma_i = \sum_i (t_i \sigma_i) \quad (4)$$

Where ' $t_i$ ' represents the transference number or the fractional contribution of a particular species of  $\sigma_T$ . For glass systems in which the conduction mechanism is dominated by a single type of ion species, for example  $\text{Ag}^+$ ,  $\text{Li}^+$ ,  $\text{K}^+$  etc. the equation (1) simplified as,

$$\sigma_i = \sigma_{\text{ion}} \quad (5)$$

In general the conductivity of glass systems in which one type of mobile ion species taking part in conduction mechanism is given as,

$$\sigma = nZe\mu \quad (6)$$

Where ' $n$ ' is the number of mobile ions per unit volume and ' $\mu$ ' is the mobility. Both are temperature dependent. Nernst-Einstein assumed the equivalence of the gradient in particle concentration which results from driving force for diffusion equal to external force. In case of ionic conduction the drift force is related to applied electric field, and Nernst-Einstein obtained the expression for diffusion coefficient and is given by the relation i.e.

$$D(\sigma) = \frac{\sigma kT}{n(Ze)^2} \quad (7)$$

Where ' $D$ ' the diffusion coefficient, ' $\sigma$ ' the electrical conductivity, ' $k$ ' the Boltzmann constant, ' $T$ ' the temperature of the glass system, ' $n$ ' the mobile cation concentration, ' $Ze$ ' the charge of the ion. From statistical random walk calculations on the basis of thermal activated process, the free energy for motion of cations for electrical conductivity in the oxide glass systems is given by [7],

$$\sigma = \frac{Ne^2 l^2 \nu_0}{6kT} \exp\left(-\frac{\Delta G_F}{2kT}\right) \exp\left(-\frac{\Delta G_m}{kT}\right) \quad (8)$$

$\Delta G_F = \Delta H_f - \Delta S_f$  the free enthalpy associated with the formation of an interstitial ionic pair,  $\Delta G_F = \Delta H_f - \Delta S_m$  the free enthalpy (entropy) associated with the migration of cations,  $\Delta H_m$  (enthalpy of motion) is the height of the potential barrier between two sites.

For glasses in which single type of ion transport dominating, for example alkali and alkaline earth doped oxide glass systems in which alkali ion is mobile and alkaline earth ion is immobile, the electrical conductivity from the above equation Eq. (7) can be simplified as [31-32].

$$\sigma T = \sigma_0 \exp\left(-\frac{E_a}{kT}\right) \quad (9)$$

Where ' $\sigma$ ' is the temperature dependent dc electrical conductivity, ' $T$ ' is the temperature of the sample and ' $\sigma_0$ ' is the pre exponential factor and is given by,

$$\sigma_0 = \frac{Ne^2 l^2 \nu_0}{6kT} \exp\left(\frac{\Delta S_f + \Delta S_m}{2}\right) \quad (10)$$

Where ' $N$ ' is the mobile cation number density, ' $e$ ' the charge of the proton, ' $l$ ' is the jump distance between two adjacent ionic sites, ' $\nu_0$ ' the vibrational frequency of ion in a potential well, ' $k$ ' is the Boltzmann constant, ' $T$ ' is the absolute temperature, and the dc activation energy is given by the expression,

$$E_a = \frac{\Delta H_f}{2} + \Delta H_m \quad (11)$$

In the present work, the conductivity of the glassy system has been found to obey the Nernst Einstein relation above the glass transition temperature as given by Eq. (7). Assuming a homogenous distribution of ions in the glass, the mean jump distance between adjacent alkali ion sites is related to alkali ion concentration as  $l = (1/N)^{1/3}$ .

### 3. EXPERIMENTAL TECHNIQUES:

The required amount of analytical grade chemicals such as Boric acid ( $H_3BO_3$ ), Ammonium Sulphate ( $NH_4H_2PO_4$ ), Potassium Sulphate ( $K_2SO_4$ ) and Strontium oxide (SrO) were thoroughly mixed by manually grinding in an agate mortar then transferred to porcelain crucible. The grinded chemicals mixture was heated to melting temperature at 1253K in a high temperature electrical muffle furnace. When homogeneous transparent melt was formed, then the melt was rapidly quenched using two stainless steel plates and the random glass pieces formed were collected. The samples were subjected to annealing by heating them for an hour up to 573K and left them for slow cooling for 4 hour. The samples were subjected to X-ray diffraction studies using Bruker D8 Advanced Diffractometer at IISER, Pune.

Room temperature density,  $\rho$  was determined by following Archimedes principle equation (1) using sensitive single pan balance (Citizen Cy204) with an accuracy of 0.1 mg and toluene  $C_7H_8$  as an immersion liquid.

Glasses were well shaped to the required sizes for the measurement of dc conductivity. The thickness of the samples was lying between 1.3mm to 2.5mm and the cross sectional area ranging between  $1.5 \text{ mm}^2$  to  $1.8 \text{ mm}^2$ . Silver paste was painted on two major surfaces of the samples. The DC conductivity measurements were carried out by following two-probe technique for the temperature range from 308K to 503K. The temperature of the sample was determined to the accuracy of  $\pm 1K$  using a chromel– alumel thermocouple and digital micro voltmeter. A constant dc voltage of about 5V was applied across the sample. The current passing through the sample and voltage was measured with the help of a digital Pico ammeter and digital multimeter respectively to an accuracy of  $\pm 0.1 \text{ pA}$  and  $\pm 10$  volts. The conductivity,  $\sigma$  of the samples were calculated using  $\sigma = (l/\rho)$  where the electrical resistivity,  $\rho = (RA/l)$ , where  $R$  is resistance, 'A' the cross sectional area and 'l' thickness of the sample.

## 4. RESULTS AND DISCUSSIONS:

### 4.1 XRD Diffraction

All the samples showed no sharp peaks in their XRD pattern [33]. The XRD pattern of one sample BPSK2 and BPSK3 is depicted in Fig 1. Similar patterns were obtained for the remaining samples which confirm the non-crystalline nature of all the samples. To save the space only the graph of two samples is depicted.

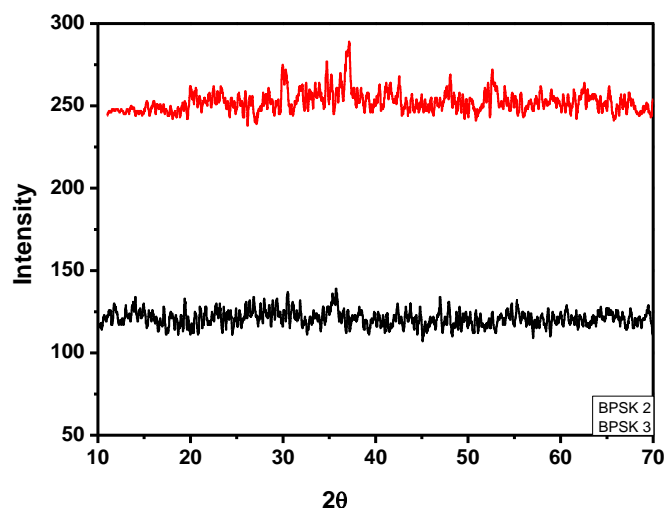


Fig.1. XRD Patterns of sample.

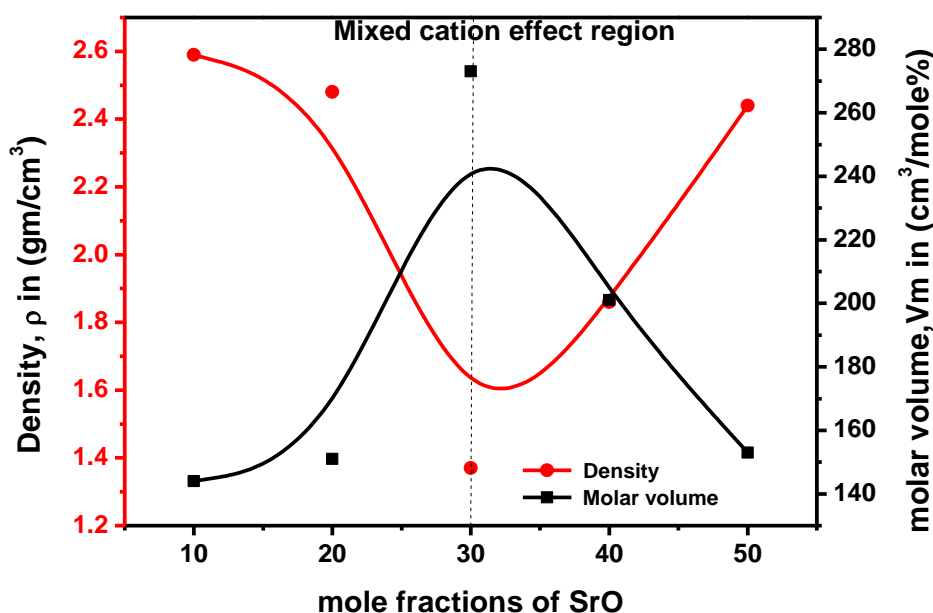
### 4.2 Density and molar volume discussion

The estimated room temperature density ' $\rho$ ' of annealed present series of glass samples lies between the values  $1.37 \text{ gm/cm}^3$  to  $2.59 \text{ gm/cm}^3$  and recorded in Table.1. The density of present series of glass samples decreases with increase of SrO at the cost of  $K_2O$  up to  $x = 30$  mole fraction, further increase in SrO, the density decreases (Fig.2). The total ion concentration sum of potassium and strontium ion in the present series of glass samples were kept constant.

The molar volume of these glass samples are ranging from 151 cm<sup>3</sup>/mole to 273 cm<sup>3</sup>/mole and varies in opposite fashion as that of density with K<sub>2</sub>O concentration. These values are comparable with that of many reported alkali and alkaline earth ion doped borate, phosphate and borophosphate glasses [34]. The density and molar volume verses mole fraction of SrO plot variation trend is nonlinear [35-36]. The topology of glass network is not same for all compositions in the present series. When the SrO ion concentration is go on replacing by K<sub>2</sub>O the molar volume decreases hence glass network continuously close packed up to x = 30 mole fractions of SrO and further increase the of SrO the glass network relaxes. In the studied composition range of present series of glass samples the nonlinear variation of density  $\rho$  and molar volume  $V_M$  with SrO ion concentration indicates that there is substantial change taking place in the topology of glass network after every particular mole fractions of Alkaline earth ion concentrations. The nonlinear variation of density  $\rho$ , and molar volume  $V_M$  with SrO ion concentration may be attributed as mixed cation effect taking placed in present glass systems within the studied range of composition [11, 12].

**Table: - 1** The variation of density,  $\rho$  and molar volume  $V_m$  with glass composition.

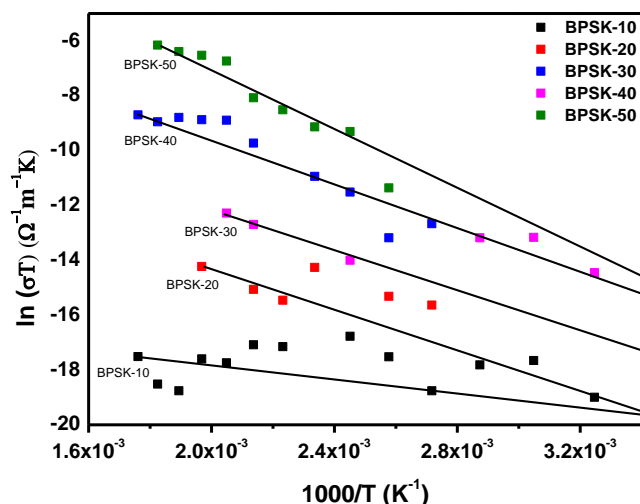
Glass samples	Glass composition				$\rho$ in gm/cm <sup>3</sup>	$V_m$ In cm <sup>3</sup>
	20P <sub>2</sub> O <sub>5</sub>	30B <sub>2</sub> O <sub>3</sub>	x SrO	(50-x) K <sub>2</sub> O		
BPSK 1	20	30	10	40	2.59	144
BPSK 2	20	30	20	30	2.48	151
BPSK 3	20	30	30	20	1.37	273
BPSK 4	20	30	40	10	1.86	201
BPSK 5	20	30	50	00	2.44	153



**Fig.2** Compositional Variation of Density,  $\rho$  and molar volume,  $V_m$  of BPSK glasses.

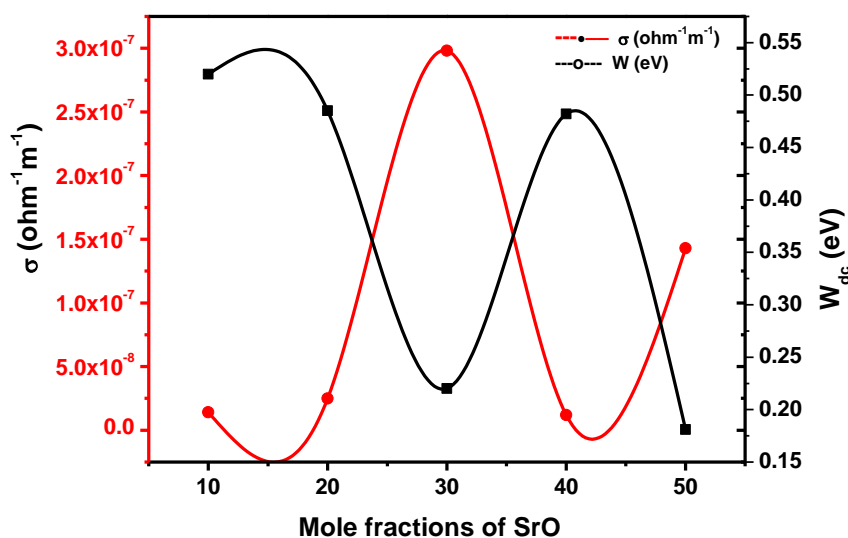
#### 4.3. DC Conductivity

The measured experimental data was fitted to the equation (9). The plots of  $\ln(\sigma T)$  versus  $(1/T)$  were plotted and depicted in Fig 3. During the measurement of dc conductivity of all the samples, because the second sample of the series 20B<sub>2</sub>O<sub>3</sub> + 30P<sub>2</sub>O<sub>5</sub> + 30SrO + 20K<sub>2</sub>O, the instruments could not measure any conductivity related readings. Hence in the figure 3 the graph was plotted for data of four samples only. From these figures it is observed that curves are linear in the high temperature region. The linear lines were fit to the data in the high temperature region using origin graphics - version 8.1. The degree of linear fits was noted by noting correlation coefficient, R, from the graphs and it was found to be in the range 0.9998 to 0.9995 for all the glass samples. The slopes of linear fits are determined from the figures. The temperature independent dc activation energies,  $W_{dc}$  values were determined and are in the range of 0.64 eV to 1.11 eV. The conductivity values at 500 K were in the range of  $3.13 \times 10^{-11}$  (ohm-m)<sup>-1</sup> to  $3.75 \times 10^{-08}$  (ohm-m)<sup>-1</sup>. The data are comparable with that of reported values for similar kind of glasses in literature [11, 12].



**Fig.3** plots of  $\ln(\delta T)$  vs  $1/T$  of BPSK glasses. Solid lines are fits to the high temperature data.

The high temperature activation energy decreases and conductivity increases up to  $x = 30$  mole fractions of SrO. For further increase in SrO ion concentration, the activation energy increases and conductivity decreases. The variation of activation energy and conductivity as a function of 'x' mole fractions of SrO is shown in fig 4. Because addition of two different cation species in to the glass matrix, among two, the species of one type ions such as  $K_2O$  ions dominating to contribute to the conductivity. That is due to mobility or diffusion of  $K_2O$  ion species in the glass matrix greater than Alkaline earth SrO ions, Because of less mobility of SrO ion it contribute less to the conductivity in the present series of glasses. Because in the oxide glasses doped with alkali and alkaline earth ions, the alkali ions contribute more and the alkaline earth ions less to the conductivity [11, 12]. Hence in the present studies initially the conductivity increases up to 30 mole fraction of SrO where within this composition range both SrO and  $K_2O$  ions contributing to the conductivity. Above the 30 mole fraction of the SrO ions where in the composition region the  $K_2O$  ion concentration decreases hence the conductivity decreases. This is because of less mobile SrO and more mobile  $K_2O$  ions and the ratio of mobile ion concentration go on reducing by the addition of SrO. In the present glasses systems the diffusion coefficient value can be estimated by using the equation (9). The diffusion coefficient varies nonlinearly and hits maximum value with up to 30 mole fractions of SrO further increase of SrO it decreases. This nonlinear variation of activation energy and dc conductivity in studied composition range of present series glasses is attributed as mixed cation effect is taking place [11, 12]. This classical mixed cation effect in the present series of glass samples is due to movement of two dissimilar ions of unequal size, are accompanied by the formation of intrinsic defects in glass forming network when mobile cations SrO and  $K_2O$  exchanges their sites [11, 12].



**Fig.4.** Variation of activation energy,  $w_{dc}$ , conductivity,  $\sigma$ , at 503 K with mole fraction SrO of present series of glasses.



**Table: - 2** The variation of dc activation energy and conductivity,  $\sigma$ , at 500K

glass samples	$N$ ( $\text{m}^{-3}$ )	$l$ (nm)	$D$ ( $\text{m}^2/\text{s}$ )	$\sigma$ ( $\text{ohm m}^{-1}$ )	$W_{dc}$ (eV)
BPSK 1	$31.97 \times 10^{28}$	$6.788 \times 10^{-10}$	$8.78 \times 10^{-19}$	$1.40 \times 10^{-8}$	0.52
BPSK 2	$22.95 \times 10^{28}$	$7.581 \times 10^{-10}$	$2.16 \times 10^{-18}$	$2.48 \times 10^{-8}$	0.48
BPSK 3	$8.45 \times 10^{28}$	$0.105 \times 10^{-10}$	$7.07 \times 10^{-17}$	$2.98 \times 10^{-7}$	0.22
BPSK 4	$5.70 \times 10^{28}$	$0.120 \times 10^{-10}$	$4.22 \times 10^{-18}$	$1.20 \times 10^{-8}$	0.48
BPSK 5	$0.749 \times 10^{28}$	$0.237 \times 10^{-10}$	$7.66 \times 10^{-16}$	$1.43 \times 10^{-7}$	0.18

In our present glasses ‘N’ ion concentration, ‘ $l$ ’ jump distance, and observed the nonlinear variation in dc conductivity ‘ $\sigma$ ’ activation energy ‘ $W_{dc}$ ’, and diffusion coefficient ‘ $D$ ’ can be attributed that the mixed cation effect taking place at  $x=30$  mole fractions of SrO ion concentration.

## 5. CONCLUSION:

A set of borophosphate glasses incorporated with SrO and  $\text{K}_2\text{O}$  have been prepared their non-crystalline nature was confirmed by XRD, The samples were measured for room temperature density and molar volume was estimated. The density and molar volume behaves in opposite fashion and nonlinear with increasing mole fractions of SrO and hits minimum and maximum peaks respectively at 30 mole fraction of SrO. The high temperature dc activation energy was determined by fitting the measured experimental data for Nernst Einstein relation. The Conductivity and activation energy found to behave in opposite fashion and hits maximum and minimum peaks respectively, at 30 mole fractions of SrO. The nonlinear variation of the observed physical parameters such as density, molar volume, dc conductivity, high temperature activation energy, diffusion coefficient with respect to mole fractions of SrO ion at the cost of potassium ion revealed that the classical mixed cation effect taking place in the present series of glasses.

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