Chapter 10

Investigation of Phase Transition in Potassium Sulphamate Single Crystal: AC Electrical Conductivity and Dielectric Studies
10.1 Introduction

Potassium Sulphamate, having the structural formula $\text{K}[\text{NH}_2\text{SO}_3]$, crystallizes in orthorhombic structure. The lattice parameters are $a = 8.32$, $b = 8.28$ and $c = 5.90$ Å. The space group of the crystal was found to be $\text{Pbma}$. The literature data of Potassium Sulphamate crystal is scanty. The X-ray diffraction studies of this crystal were carried out by C J Brown and E G Cox in (1940)[1]. The structure was later refined using neutron diffraction method by Cox et al. (1967) [2]. The infrared spectra of this crystal were studied by A M Vuagnat and E L Wagner [3]. The elastic properties of sulfamic acid and its sulfamates was reported by E Haussuhl and S. Haussuhl (1995) [4]. The literature review is detailed in the last chapter.

Chapter 9 describes the dc electrical conductivity studies and its variation with temperature. The measurements are carried out both in the low temperature and in the high temperature region. Low temperature studies show an anomaly at 265 K, and the studies carried out in the high temperature shows anomalies at around 340K and at around 420K, both anomalies may be attributed as a phase transition.

The aim of the present study is to study the variation of ac electrical conductivity with temperature along [100], [010] and [001] direction and to investigate any phase...
transition occurring in the crystal. The present study is used to confirm the phase
transition that observed in the dc electrical conductivity studies.

10.2 Experimental

Single crystals of potassium sulfamate were grown from aqueous solution of pure
sulfamic acid neutralized with pure potassium carbonate by slow evaporation method.
The crystal growth and sample preparation is detailed in the last chapter. Temperature
variation of ac electrical conductivity measurements are carried out along a, b and c-axes
for selected frequencies and also the frequency variation of the ac conductivity is studied
for certain temperatures.

10.3 Results and Discussion

10.3.1 Complex Impedance Analysis along a-axis

The variation of imaginary part of impedance with real part of impedance as a function
of frequency gives Cole-Cole plots. The complex impedance analysis of the potassium
sulphamate single crystal along a-axis is shown in fig 10.1-10.3 for different
temperatures. Typical semi circles are obtained for several temperatures. The incomplete
semicircle in the low frequency side in fig 10.2 and 10.3 for temperatures 50°C (323K),
100°C (373K) and 150°C (423K) is due to the unavailability of frequency in the low
frequency range. The appearance of only one semi circle at low temperature is an
indication of well-compressed sample confirming the absence of grain boundary effect.
The angle of inclination of the semicircle gives a measure of the breadth of the
distribution of relaxation times [5]. The spike observed in fig 10.1 for 30°C (303K) may
be attributed as a polarisation effect at the electrode /electrolyte interface. The presence
or absence of electrode features provides an indication whether ionic conductivity is
present. The presence of an electrode effect at 30°C (303K) in fig 10.1 suggests that the
sample has an ionic conduction [6]. Cole-Cole parameters such as bulk resistance ($R_b$)
and bulk capacitance ($C_b$) are extracted from these plots and are shown in table 10.1. The
dielectric relaxation time has also been calculated for several temperatures and the values
are shown in table 10.1.
Investigation of Phase Transition in Potassium Sulphamate single crystal

Fig 10.1  Complex impedance plots of Potassium Sulphamate crystal along a-axis for several temperatures (temp in °C)

Fig 10.2  Complex impedance plots of Potassium Sulphamate crystal along a-axis for 50°C (323K) & 100°C (373K)

Fig 10.3  Complex impedance plots of Potassium Sulphamate crystal along a-axis for 150°C (423K)
Temperature (K) | $R_b (K\Omega)$ | $C_b$ (pF) | $\tau (\mu s)$
---|---|---|---
243 | 264 | 6.02 | 1.59
263 | 269 | 5.91 | 1.59
273 | 838 | 6.33 | 5.30
283 | 888 | 5.97 | 5.30
303 | 891 | 8.93 | 7.95
323 | $30 \, M\Omega$ | 26 | 795
373 | $38 \, M\Omega$ | 20 | 795
423 | $48.5 \, M\Omega$ | 51.54 | 397

Table 10.1 Cole-Cole parameters of Potassium sulphamate crystal along $a$-axis

10.3.2 Complex Impedance Analysis along $b$-axis

The Cole -Cole plots of the potassium sulphamate single crystal along $b$-axis is shown in fig 10.4-9 for different temperatures. Typical semi circles are obtained for several temperatures. At lower temperatures the low frequency end of the curve does not intercept at the real axis indicating a decrease in the value of conductivity. As temperature increases they curve towards real axis and almost semicircular indicating the increase of conductivity [7]. At higher temperatures electrode polarisation was observed and may be attributed, as the sample is an ionic conductor. The Cole-Cole parameters are extracted from the curves and are depicted in table 10.2. The equivalent circuit can be represented by a parallel combination of capacitance and resistance only. All these curves start at the origin and hence there is no series resistance that can be ascribed to the equivalent circuit representation [7]. The radius of these semicircles gives the exact relaxation time of the lumped parameters that are associated with the sample. The semi circles have their centers located away from the real axis, indicating the presence of relaxation species with distribution of relaxation times in the sample.
Fig 10.4: Complex impedance plots of Potassium Sulphamate crystal along b-axis for 10°C (283K) & -10°C (263K)

Fig 10.5: Complex impedance plots of Potassium Sulphamate crystal along b-axis for 0°C (273K)

Fig 10.6: Complex impedance plots of Potassium Sulphamate crystal along b-axis -30°C (243K)
Fig 10.7: Complex impedance plots of Potassium Sulphamate crystal along b-axis for 30°C (303K)

Fig 10.8: Complex impedance plots of Potassium Sulphamate crystal along b-axis for 50°C (323K) & 150°C (423K).

Fig 10.9: Complex impedance plots of Potassium Sulphamate crystal along b-axis for 100°C (373K)
Temperature (K) | R_b (MΩ) | C_b (pF) | τ(s)  
--- | --- | --- | ---  
243 | 24.6 | 64 | 1.59 ms  
263 | 10.2 | 156 | 1.59 ms  
273 | 6.65 | 119 | 0.79 ms  
283 | 13.0 | 30.6 | 0.39 ms  
303 | 0.252 | 10.5 | 2.65 µs  
323 | 2.17 | 10.9 | 0.19 µs  
373 | 18.3 | 8.69 | 0.159 µs  
423 | 2.13 | 3.73 | 7.95 µs

Table 10.2: Cole-Cole parameters of Potassium sulphamate crystal along b-axis

10.3.3 Complex Impedance Analysis along c-axis

The shapes of the Cole-Cole plots obtained in the present study along c-axis depend on temperature and are shown in fig 10.10-10.13. At lower temperature, -30°C, (243K) the semi circle could not observe clearly, may be due to frequency limitation. The high frequency data for all other temperatures are represented by a nearly perfect circular arc, which passes through origin. The bulk resistance and bulk capacitance are calculated from the Cole-Cole plots and are shown in table 10.3. The distribution of relaxation time for various temperatures is also calculated.

Fig 10.10: Complex impedance plots of Potassium Sulphamate crystal along c-axis for 0°C (273K) & -10°C (263K)
Fig 10.11: Complex impedance plots of Potassium Sulphamate crystal along c-axis for -30°C (243K)

Fig 10.12: Complex impedance plots of Potassium Sulphamate crystal along c-axis 30°C (303K) & 150°C (423K)

Fig 10.13: Complex impedance plots of Potassium Sulphamate crystal along c-axis for 50°C (323K) & 150°C (423K)
Table 10.3: Cole-Cole parameters of Potassium sulphonate crystal along c-axis

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>R_6 (MΩ)</th>
<th>C_b (pF)</th>
<th>τ(µs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>243</td>
<td>0.418</td>
<td>95.1</td>
<td>0.39</td>
</tr>
<tr>
<td>263</td>
<td>10.6</td>
<td>2.5</td>
<td>0.26</td>
</tr>
<tr>
<td>273</td>
<td>3.27</td>
<td>1.62</td>
<td>5.3</td>
</tr>
<tr>
<td>303</td>
<td>0.517</td>
<td>3.07</td>
<td>1.59</td>
</tr>
<tr>
<td>323</td>
<td>170</td>
<td>2.34</td>
<td>397</td>
</tr>
<tr>
<td>373</td>
<td>76.7</td>
<td>2.59</td>
<td>198</td>
</tr>
<tr>
<td>423</td>
<td>0.455</td>
<td>3.49</td>
<td>1.59</td>
</tr>
</tbody>
</table>

10.4 Dielectric Analysis

10.4.1 Frequency Dependant Dielectric Spectra

The variation of real part of dielectric constant with frequency along a-, b- and c-axes are shown in figs 10.14-16. The dielectric constant is found to be independent of frequency in the frequency region 1KHz - 1 MHz. A slight decrease in the value of dielectric constant was observed in the high frequency region, typically after 1 MHz along a- and c-axes. The behaviour of dielectric constant with frequency is related to the application of the field which assists electron jumping between filled and empty sites. This lead to an increase of the electronic component in dielectric dispersion. On the other hand when the frequency is increased the dipoles will no longer be able to rotate sufficiently rapidly, so that their oscillation will begin to lay behind this field, which explains the observed decrease in dielectric constant with increasing frequency [8]. Very high value of dielectric constant is obtained in the low frequency region, below 1 KHz, may be due to the electrode effect. The room temperature dielectric constant along a, b and c-axes are 9.96, 10.15 and 9.14 respectively at 10 KHz. The plots of imaginary part of dielectric constant with frequency are shown in fig 10.17-10.19. In general dielectric loss decreases as the frequency increases. In the high temperature region all spectrum shows large frequency dispersion. The dielectric loss is found to be minimum along a-axis. Below room temperature the dielectric loss is independent of frequency along b and c-axes.
Fig 10.14: Variation of Real part of dielectric constant with frequency along a-axis for different temperatures

Fig 10.15: Variation of Real part of dielectric constant with frequency along b-axis for different temperature
Fig 10.16: Variation of Real part of dielectric constant with frequency along c-axis for different temperature.

Fig 10.17: Variation of Imaginary part of dielectric constant with frequency along a-axis for different temperature.
Fig 10.18: Variation of Imaginary part of dielectric constant with frequency along b-axis for different temperature.

Fig 10.19: Variation of Imaginary part of dielectric constant with frequency along c-axis for different temperature.
10.4.2 Temperature dependant Dielectric Spectra in the low temperature region

Fig 10.20  Variation of real part of dielectric constant with temperature along a-axis for different frequency in the temperature region 243 K-303 K

Fig 10.20 -10.22 shows the variation of dielectric constant with temperature along a, b and c-axes respectively in the temperature region 303K- 243K for selected frequencies of 1 KHz, 10 KHz and 100KHz. Fig 10.20, 10.21 and 10.22 shows dielectric peaks at 262K. The value of dielectric constant is high along c-axis. All figs show a strong dispersion in frequency. As frequency increases the dielectric constant found to decrease. The anomalies obtained at 262 K are very distinct and may be attributed as phase transition. The variation of dielectric constants around this temperature is very predominant and this variation is more observed for lower frequencies. The value of dielectric constant at room temperature along a, b and c-axes are 6.92, 6.49 and 6.43 at 10 KHz. Also the value of dielectric constant at 262K is 13.90, 13.01 and 928 along a, b and c-axes at 10 KHz respectively. A large variation of dielectric constant was observed along c-axis at around 262K. The anomalies observed at this temperature are reported for the first time.
Fig 10.21: Variation of real part of dielectric constant with temperature along b-axis for different frequency in the temperature region 243 K-303 K

Fig 10.22: Variation of real part of dielectric constant with temperature along c-axis for different frequency in the temperature region 243 K-303 K
10.4.3 Temperature dependant Dielectric Spectra in the High temperature region

![Graph](image)

Fig 10.23  Variation of real part of dielectric constant with temperature along a-axis for different frequency in the temperature region 303 K-450 K

The variation of dielectric constant with temperature are shown in fig 10.23 –10.25 along a, b and c-axes respectively in the temperature region 303K- 450K for selected frequencies of 1 KHz, 10 KHz and 100KHz. A change of slope was observed along the three principal axes at around 340K. The value of dielectric constant is independent of temperature in the temperature region 340 K-400K. This temperature independent region is dependant on frequency. A slight increase in the value of dielectric constant was observed along a- and b- axes after 400K while a large increase in the value of dielectric constant was observed along c-axes. A weak anomaly was also observed at around 420K along a and c-axes. Hence the increase in the value of dielectric constant after 400K may be due to the beginning of a phase transition occurring at around 420K. A change of slope at around 340 K may be attributed as phase transition occurring in the crystal around this temperature. The value of dielectric constant decreases as frequency increases. The variation of loss tangent with temperature along a, b, and c-axes are shown in figs 10.26-10.28. All figures show a change of slope at around 340 K. Along c-axes a loss peak was observed at 340 K for 1KHz. The loss tangent is very less (<10)
Fig 10.24: Variation of real part of dielectric constant with temperature along b-axis for different frequency in the temperature region 303 K-450 K.

Fig 10.25: Variation of real part of dielectric constant with temperature along c-axis for different frequency in the temperature region 303 K-450 K.
**Fig 10.26:** Variation of loss tangent with temperature along a-axis for different frequency in the high temperature region.

**Fig 10.27:** Variation of loss tangent with temperature along b-axis for different frequency in the high temperature region.
Fig 10.28: Variation of loss tangent with temperature along c-axis for different frequency in the high temperature region along a & b- axes and found to increase with temperature after 380 K along all measured axes. Loss tangent curve also shows some weak anomaly at around 425K along a, b and c-axes.

10.5 Conductivity Analysis

10.5.1 Frequency Dependant Conductivity Spectra along a, b and c-axes

Fig 10.29-31 illustrates the frequency dependence of total conductivity, $\sigma_{tot}$, for different temperatures along a, b and c-axes respectively. The dependence of the electrical conductivity with frequency can be expressed by an equation follow

$$\sigma_{tot} = \sigma(0) + A\omega^s$$

where $\sigma(0)$ is the conductivity at limiting zero frequency being termed as dc conductivity. $A$ is a constant and $s$ is the characteristic parameter. The value of $s$ lies in the range $0<s<1$, both values $A$ and $s$ are temperature dependent. $A$ and $s$ are parameters to be adjusted and can be extracted from the plots of frequency Vs conductivity curve.

Table 10.4, 10.5 and 10.6 shows the values of $A$, $s$ and $\sigma(0)$ for a, b and c-axes.
respectively for different temperatures. Theoretical fitting was done using the above equation and a good agreement between experimental and theoretical fitting is observed. Along all axes the parameters A and s are temperature dependent. In this work a-axis showed s values between 0.057 and 1.06, b-axes showed the values between 0.24 and 1.004 and c-axis showed a value between 0.05 and 1.00. The exponent s is the measure

Fig 10.29: Variation of ac conductivity with frequency along a-axis for different temperatures.

<table>
<thead>
<tr>
<th>Temp(K)</th>
<th>σ(0)</th>
<th>A</th>
<th>s</th>
</tr>
</thead>
<tbody>
<tr>
<td>243</td>
<td>$4.09 \times 10^{-3}$</td>
<td>$2.05 \times 10^{-4}$</td>
<td>0.057</td>
</tr>
<tr>
<td>263</td>
<td>$4.60 \times 10^{-3}$</td>
<td>$2.06 \times 10^{-4}$</td>
<td>0.06</td>
</tr>
<tr>
<td>273</td>
<td>$1.05 \times 10^{-5}$</td>
<td>$2.03 \times 10^{-4}$</td>
<td>0.013</td>
</tr>
<tr>
<td>283</td>
<td>$1.27 \times 10^{-5}$</td>
<td>$7.72 \times 10^{-5}$</td>
<td>0.335</td>
</tr>
<tr>
<td>303</td>
<td>$7.78 \times 10^{-5}$</td>
<td>$9.7 \times 10^{-6}$</td>
<td>1.06</td>
</tr>
<tr>
<td>323</td>
<td>$5.25 \times 10^{-9}$</td>
<td>$6.02 \times 10^{-11}$</td>
<td>0.99</td>
</tr>
<tr>
<td>373</td>
<td>$1.69 \times 10^{-8}$</td>
<td>$1.68 \times 10^{-10}$</td>
<td>0.996</td>
</tr>
<tr>
<td>423</td>
<td>$2.04 \times 10^{-7}$</td>
<td>$5.03 \times 10^{-9}$</td>
<td>0.77</td>
</tr>
</tbody>
</table>

Table 10.4: Power low parameters along a-axis for different temperatures
of the degree of interaction with the environment [9]. In the low frequency region the conductivity is independent of frequency irrespective of temperature while in the high frequency region it depends on frequency.

![Graph showing variation of ac conductivity with frequency along b-axis for different temperatures.](image)

Fig 10.30: Variation of ac conductivity with frequency along b-axis for different temperatures.

<table>
<thead>
<tr>
<th>Temp(K)</th>
<th>$\sigma(0)$</th>
<th>$A$</th>
<th>$S$</th>
</tr>
</thead>
<tbody>
<tr>
<td>243</td>
<td>$2.28 \times 10^{-8}$</td>
<td>$2.34 \times 10^{-9}$</td>
<td>1.004</td>
</tr>
<tr>
<td>263</td>
<td>$2.2 \times 10^{-8}$</td>
<td>$2.33 \times 10^{-9}$</td>
<td>1.00</td>
</tr>
<tr>
<td>273</td>
<td>$4.74 \times 10^{-8}$</td>
<td>$2.33 \times 10^{-9}$</td>
<td>1.00</td>
</tr>
<tr>
<td>283</td>
<td>$2.89 \times 10^{-8}$</td>
<td>$2.37 \times 10^{-9}$</td>
<td>1.003</td>
</tr>
<tr>
<td>303</td>
<td>$8.6 \times 10^{-9}$</td>
<td>$7.19 \times 10^{-9}$</td>
<td>0.24</td>
</tr>
<tr>
<td>323</td>
<td>$3.09 \times 10^{-6}$</td>
<td>$2.63 \times 10^{-6}$</td>
<td>0.28</td>
</tr>
<tr>
<td>373</td>
<td>$3.02 \times 10^{-7}$</td>
<td>$3.84 \times 10^{-7}$</td>
<td>0.41</td>
</tr>
<tr>
<td>423</td>
<td>$1.2 \times 10^{-6}$</td>
<td>$6.91 \times 10^{-7}$</td>
<td>0.45</td>
</tr>
</tbody>
</table>

Table 10.5: Power law parameters along b-axis for different temperatures
Fig 10.31: Variation of ac conductivity with frequency along c-axis for different temperatures.

<table>
<thead>
<tr>
<th>Temp(K)</th>
<th>$\sigma(0)$</th>
<th>A</th>
<th>s</th>
</tr>
</thead>
<tbody>
<tr>
<td>243</td>
<td>$1.54 \times 10^6$</td>
<td>$1.25 \times 10^8$</td>
<td>0.99</td>
</tr>
<tr>
<td>263</td>
<td>$1.05 \times 10^5$</td>
<td>$1.08 \times 10^8$</td>
<td>1.00</td>
</tr>
<tr>
<td>273</td>
<td>$1.06 \times 10^6$</td>
<td>$1.07 \times 10^8$</td>
<td>1.00</td>
</tr>
<tr>
<td>303</td>
<td>$7.05 \times 10^6$</td>
<td>$5.87 \times 10^5$</td>
<td>0.114</td>
</tr>
<tr>
<td>323</td>
<td>$3.69 \times 10^8$</td>
<td>$7.84 \times 10^{10}$</td>
<td>0.817</td>
</tr>
<tr>
<td>373</td>
<td>$3.63 \times 10^7$</td>
<td>$1.29 \times 10^7$</td>
<td>0.353</td>
</tr>
<tr>
<td>423</td>
<td>$7.05 \times 10^6$</td>
<td>$8.43 \times 10^5$</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Table 10.6: Power law parameters along c-axis for different temperatures
10.5.2 Temperature Dependant Conductivity Spectra along a, b and c-axes in the Temperature Region 243 K- 303 K

Fig 10.32-34 shows the variation of ac conductivity for different frequency in the temperature region 243-303 K along a, b and c-axes respectively.

Fig 10.32: Variation of ac conductivity with temperature along a-axis below room temperature.

Fig 10.33: Variation of ac conductivity with temperature along b-axis below room temperature.
Fig 10.34: Variation of ac conductivity with temperature along c-axis below room temperature.

The variation of ac conductivity with temperature is reporting for the first time. All figs shows conductivity anomalies at around 260 K. Anomalous peaks are observed at 260 K along a, b and c-axes. Large variation of conductivity values is observed along a, b and c-axes for all measured frequencies. The anomalies observed in the heating cycle could also been observed in the cooling cycle indicating a possibility of possible phase transition. Variation of dielectric constant with temperature in also showed anomalous peaks at this temperature. The temperature dependent conductivity is found to be frequency independent. The value of conductivity at room temperature is given by $3.03 \times 10^{-5}$, $1.70 \times 10^{-8}$ 3.20 x10^{-8}$

The conductivity data are then represented by Arrhenius relation, given as follows

$$\sigma = \sigma_0 \exp \left(-\frac{E}{K_B T}\right)$$

where $\sigma_0$ is the pre exponential factor and E, $K_B$ and T represents the apparent activation energy for conduction process, Boltzmann's constant and the absolute temperature, respectively. The activation energy for conduction process (E) was extracted by the slope of the straight line in the log $\sigma$ against reciprocal temperature $1/T$
plot. The activation energy along different axes for different frequencies are given in table 10.7.

<table>
<thead>
<tr>
<th>Axis</th>
<th>Activation energy (eV) for</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Freq.</td>
</tr>
<tr>
<td>a-axis</td>
<td></td>
</tr>
<tr>
<td>b-axis</td>
<td></td>
</tr>
<tr>
<td>c-axis</td>
<td></td>
</tr>
</tbody>
</table>

Table 10.7: Activation energies along different axes in the temperature region 243 K-303K

Very high activation energy was obtained along c-axes and the activation energy is found to decrease with increasing frequencies.

10.5.3 Temperature Dependant Conductivity Spectra along a, b and c-axes in the Temperature Region 303K-450 K

![Variation of ac conductivity with temperature along a-axis in the high temperature region.](image)

Fig 10.35: Variation of ac conductivity with temperature along a-axis in the high temperature region.
Fig 10.36: Variation of ac conductivity with temperature along b-axis in the high temperature region.

Fig 10.37: Variation of ac conductivity with temperature along c-axis in the high temperature region.
Figures 10.35-10.37 show the variation of ac conductivity with temperature along a, b and c-axes respectively in the temperature region 303 K - 450 K for different frequencies. Measurements are carried out for both in the heating and cooling cycles. Points with and without solid lines represent cooling and heating curves respectively. Conductivity first decreases with increasing temperature shows minimum at around 340K and then increases with further increase of temperature. All the figs show a slight change of slope at this temperature. The cooling cycle along b and c-axes showed a conductivity dip at around 340 K. This can be attributed as phase transition occurring at this temperature. Another anomaly that could observe on the conductivity measurement was at 425K. After 380K the conductivity values increases upto 425K. After 425K the value starts decreasing. In the heating run a weak anomaly was also observed at around 420K along a, b and c-axes. The conductivity values at 303 K is given by $1.28 \times 10^{-5} \Omega^{-1} \text{cm}^{-1}$ along a-axis, $2.77 \times 10^{-5} \Omega^{-1} \text{cm}^{-1}$ along b-axis and $4.75 \times 10^{-4} \Omega^{-1} \text{cm}^{-1}$ along c-axis.

The activation energy can be calculated for different frequencies and are given in table 10.8. The activation energy is high along c-axis also the activation energy found to decrease with increase with increasing frequency. Since these anomalies are observed for the first time an exact reason for these anomalies require further investigation.

<table>
<thead>
<tr>
<th>Axis</th>
<th>Activation energy (eV) for</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Freq. 1 KHz</td>
</tr>
<tr>
<td>a-axis</td>
<td>0.445</td>
</tr>
<tr>
<td>b-axis</td>
<td>0.633</td>
</tr>
<tr>
<td>c-axis</td>
<td>0.99</td>
</tr>
</tbody>
</table>

Table 10.8 Activation energies along different axes in the temperature region 303 K-450 K

10.6 Conclusion

The Cole-Cole plots of the potassium sulphamate crystal were drawn and the bulk parameters such as bulk resistance and bulk capacitance are extracted from the plots. The
relaxation time of the dielectric relaxation for several temperatures is also extracted. From the Cole–Cole plots one can infer that the crystal was an ionic conductor. The ac conductivity of potassium sulphamate crystal was measured along a- b and c axes. The dielectric studies are also conducted along a, b and c-axes. The variation of dielectric constant with temperature in the temperature region 243- 303K shows an anomalous behaviour at around 262 K. The variation of ac conductivity with temperature also shows an anomaly at this temperature. These anomalies are observed for both heating and cooling cycles, and may be attributed as a phase transition occurring in the crystal at this temperature. The present study is consistent with the dc conductivity studies detailed in the last chapter. These conductivity and dielectric anomalies observed at 262 K is reporting for the first time. The dielectric constant for various frequencies is measured for the first time. The variation of dielectric constant with temperature in the high temperature region shows no dielectric peaks. However a dielectric minimum was noticed at around 340 K and a weak anomaly was observed at around 425K. AC Conductivity studies also showed a conductivity minimum at 34K. Conductivity increases gradually after 340 K showed a change of slope at 340 K. This kind of anomaly was observed both for heating and cooling run and may be attributed as phase transition. Whether this can be connected with the phase transition is still a subject of further investigation. The present study is consistent with the earlier dc conductivity studies. AC conductivity measured on this crystal showed that conductivity is high long c-axis. The activation energies of the conduction is calculated in the low temperature region and in the high temperature region. The activation energy is found to decrease with increasing frequency and its value is high along c-axis. High activation energies are obtained along c-axes in the temperature region 243K- 303 K. The ac and dielectric studies in the high temperature region shows an exponential increase in conductivity values after 400K along a, b and c-axes. A weak anomaly was observed at 425K for heating and cooling cycles for a, b and c-axes. An exponential increase in the value of ac conductivity after 400K may be due to beginning of a phase transition that occurring at 425K. The anomalies observed at 265 K, 340 K and at 400K are consistent with the dc conductivity study and DSC studies and the reason for this anomaly needs further investigation.
References


---

73544110-087ca4e860b592e56c8747558ef55963