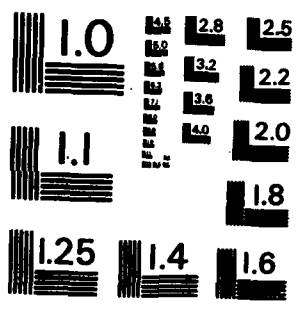


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DIELECTRIC RELAXATION IN POLY(ETHYLENE OXIDE) COMPLEXED
WITH ALKALI METAL PERCHLORATES

by

John J. Fontanella and Mary C. Wintersgill

Prepared for Publication

in

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Annapolis, MD 21402

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20. ABSTRACT (continued)

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DIELECTRIC RELAXATION IN POLY(ETHYLENE OXIDE) COMPLEXED
WITH ALKALI METAL PERCHLORATES

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ABSTRACT

Audio frequency complex admittance measurements over the temperature range 5.5-350K have been performed on poly(ethylene oxide) (PEO) complexed with lithium and sodium perchlorates. No evidence of an α relaxation was seen in any sample. In the Li-complexed material, the γ relaxation occurs at about 174K and 1000 Hz which is the same as for pure PEO. In the Na-complexed materials, there appear to be more than one peak in this region, and the relative peak heights are concentration dependent. The result is that at the 8:1 doping level, there is a broad, skewed relaxation whose peak occurs at about 203K and 1000 Hz, while at 4.5:1 NaClO₄ the relaxation appears much closer to a normal peak with a maximum at 185K for the 1000 Hz data. Combined with previous results for thiocyanate complexed PEO, these results show that distortions in the polymer chain depend on both the cation and anion. Next, two new relaxations are observed at low temperatures. The first, occurring at 60-100 K is very weak and is common to all PEO samples. The second occurring at very low temperatures, is only found for perchlorate complexed PEO and is attributed to the localized motion of perchlorate-induced defects on the exterior of the chains. The high temperature electrical conductivity for PEO₈:NaClO₄ is found to be larger than PEO_{4.5}:NaClO₄ in agreement with the trend observed by other workers for PEO complexed with different alkali metal salts.

1. INTRODUCTION

There is currently a great deal of interest in "fast ion conductors," both in terms of the conduction mechanisms which lead to the anomalously high conductivities in these solids, and in their applications in devices such as fuel cells, electrolytic cells and high energy density batteries. Interest in ion conducting poly(ethylene oxide) (PEO) (1-25) has been stimulated by the possibility of their use as electrolytes in solid state batteries, since their ability to carry lithium ion conduction and the physical characteristic of allowing thin, stable sheets to be constructed relatively easily, hold the promise of high power density, high voltage (3V) secondary cells. PEO complexed with various Li, Na and K salts have been the focus of much attention and the present electrical relaxation studies supplement the existing results.

2. EXPERIMENT

The complex admittance measurements were performed at five audio frequencies, 10^2 , $10^{2.5}$, 10^3 , $10^{3.5}$ and 10^4 Hz using a fully automated, microprocessor-controlled impedance bridge constructed by one of the authors (CGA). The sensitivity and accuracy of the bridge are equal to that of the best commercially available manual bridge.

Films of PEO (Polysciences MW 5×10^6) complexed with LiClO₄ and NaClO₄ were prepared as described previously (21). The concentrations of PEO:LiClO₄ were in the range 19:1 to 8:1

while the PEO:NaClO₄ concentrations ranged from 12:1 to 4.5:1. Attempts were made to prepare PEO:KClO₄ and higher concentrations of PEO:LiClO₄ but the results proved unsatisfactory. Samples were typically 0.05 cm thick. For all these samples, aluminum electrodes were evaporated onto the surfaces, with a central electrode diameter of about 8mm on one side and about 10mm on the other. This electrode configuration is primarily suited to comparative admittance studies, since the usual equations

$$C = \frac{\epsilon_0 \epsilon' A}{d} \quad (1)$$

and

$$G = \frac{\sigma A}{d} \quad (2)$$

where A is the area and d is the separation between the plates, strictly only apply to a standard parallel plate capacitor. Therefore the average of the electrode diameters was used in transforming the capacitance and conductance to the complex dielectric constant;

$$\epsilon^* = \epsilon' - j\epsilon'' \quad (3)$$

and hence to the conductivity σ , since

$$\sigma = \epsilon_0 \epsilon'' \omega \quad (4)$$

The procedures used place an absolute uncertainty of about 10% on these values, however the relative uncertainty is better, being on the order of 0.1%. Due to an apparent lack of thermal expansion values for PEO in the literature, no correction was included for this effect.

3. RESULTS AND DISCUSSION

3.1. Relaxation Spectrum

As shown in a previous paper (21), the electrical relaxation spectrum at 1000 Hz in the low temperature region, for pure PEO, consists of peaks at about 174 K (γ) and 224 K (α_a). It is apparent from Figs. 1-7 that α_a does not exist in any of the samples complexed with alkali metal perchlorates. This is the same result as for PEO complexed with alkali metal thiocyanates (21). As discussed before, this implies that there is no amorphous pure PEO in the present samples. The remaining possibilities are crystalline pure PEO and various complexed phases. Crude DSC measurements, performed on a Perkin Elmer DSC-1B Differential Scanning Calorimeter, tended to confirm the absence of amorphous, pure PEO in the complexed materials. Further, they indicate that there may be some pure crystalline PEO in the PEO19:LiClO₄, PEO8:LiClO₄ and the PEO8:NaClO₄, since there were indications of a thermal anomaly in the 350 K region, where a major endothermic event is seen for the pure PEO. The DSC traces for the PEO4.5:NaClO₄ samples showed no signs of any pure material.

Considering the γ relaxation, comparison of Fig (1c) of the previous paper (21) with Fig (1a) of the present paper shows that the γ relaxation in PEO8:LiClO₄ is essentially the same as for pure PEO. This is similar to previous results for PEO4.5:LiSCN and PEO4.5:NaSCN. These results are consistent with

attributing the γ relaxation to a $tg+t \rightleftharpoons tg-t$ transition within the helical chain. Our interpretation of these results is that the cations reside within the helical channels at low temperatures and, because of their small size, do not interfere with the $tg+t \rightleftharpoons tg-t$ transition.

However, as can be seen from Figs (2a) and (3a) two effects are noticeable, the first being that the maximum of the relaxation peak now occurs at about 190K and 1000 Hz and the peak is markedly skewed for the PEO8:NaClO₄ samples. The higher concentration NaClO₄-complexed materials appear to have a normal, approximately symmetric relaxation peak, with the maximum at 185K for 1000 Hz. Thus there seems to be a clear shift in peak temperature for all the Na-complexed materials as compared with the Li-complexed (or pure) samples, while at the lower concentration at least, an additional peak at about 195K is probably the cause of the asymmetry.

These results are to be compared with similar measurements of alkali-metal thiocyanate complexed PEO, published in a previous paper (21). There, the Na-complexed materials showed a single broad peak associated with the γ relaxation very similar to that for pure PEO, while the KSCN complexed PEO showed three peaks in this region. These were attributed to the distortion of the helix by the large potassium ion. However, for the perchlorate complexed material, large distortions in the chain are occurring for a smaller cation, Na. Thus, assuming that the same type of phenomenon is being seen in the sodium perchlorate

complexed material as in the KSCN complexed material, it follows that anions interact strongly with the polymer chain. This implies that the roles of the cation and anion are more equivalent than is usually thought. However, these results may still be understood in terms of a model where the cations reside inside the helices at low temperatures, while the anions occupy the spaces between the polymer chains. Our interpretation is that the thiocyanate ions, being approximately linear fit more easily between the chains than the tetrahedral perchlorate ions. This being the case, the perchlorate ions may, for example, tend to reduce the diameter of the helix, making it more sensitive to the size of the cation contained within.

This model is also consistent with the second feature found in perchlorate complexed PEO, electrical relaxation at very low temperatures. For both PEO₁₂:NaClO₄ and PEO₈:NaClO₄, and all concentrations of PEO:LiClO₄, the peak occurs at about 15 K. Fig. 4 shows a typical low temperature region for one of the PEO₃-LiClO₄ samples. In the case of the PEO_{4.5}:NaClO₄ samples, only the high temperature side of the peak is observed, implying a peak maximum position below 5 K. The observation of very low temperature electrical relaxations is not surprising as cryogenic relaxations in polymers are well known and are, in fact, usually found to be extremely sensitive to impurities (26). However, it is to be emphasized that no evidence of such a peak was seen in any of the thiocyanate-complexed or the pure PEO samples (21).

In the present model, the relaxation can be attributed to localized motion of perchlorate-induced defects on the exterior of the chains. The evidence for a strong interaction between the perchlorate ion and the chain has already been pointed out in the discussion of the γ relaxation region. The reason for considering defects at the surface of the chains is that the shift to lower temperatures for higher perchlorate concentrations can be easily understood. Specifically, if the perchlorate ions increase the interchain spacing proportional to their concentration, the barriers associated with the localized motion at the surfaces of the chains will be lowered and the peaks will shift to lower temperatures.

Finally, there are traces of a relaxation in the 60-100K temperature range in all the samples. Closer analysis of previous results (21) showed traces of a peak in this region in those samples also. However, the peaks were too small to allow reliable determination of the characteristics of the peak.

3.2. CONDUCTIVITY

The conductivity results shown in figs. 1b, 2b and 3b display varying amounts of dispersion at the highest temperatures studied. However, the 10^4 Hz data represent the conductivity of the sample fairly well, as shown in fig. 1b, by the very good agreement between the 10^4 Hz data of the present work and the results of Weston and Steele (20) obtained using complex impedance analysis.

Comparison of figs. 2b and 2c shows that the conductivity of PEO8:NaClO4 is about an order of magnitude larger than that for PEO4.5:NaClO4. This appears to be a general result since the conductivity for PEO8-NaSCN and PEO8-KSCN are much larger than for PEO4.5-NaSCN and PEO4.5-KSCN (27), respectively, and a similar dependence of the conductivity on concentration has been observed previously by Weston and Steele (18), Papke et al. (11), and Sorensen and Jacobsen (23). In fact, Sorensen and Jacobsen (24) have developed a model for this behavior based on a two-phase system.

4. SUMMARY

In summary then, several results have been obtained by a complex admittance study of PEO complexed with lithium- and sodium-perchlorate:

(1) The γ relaxation in PEO8:LiClO₄ is found to be the same as for pure PEO but differences are found in the γ relaxation region for PEO complexed with NaClO₄. These results are interpreted as indicating that the perchlorate ion has an appreciable interaction with the polymer chain.

(2) Two new relaxations are reported. The first occurs in the region 60-100 K and is extremely weak. The second occurs at very low temperatures and is attributed to perchlorate induced defects on the exterior of the polymer chain.

(3) The conductivity of the PEO8:NaClO₄ samples is larger than for PEO4.5:NaClO₄ in agreement with the trend observed by other workers for PEO complexed with different alkali metal salts.

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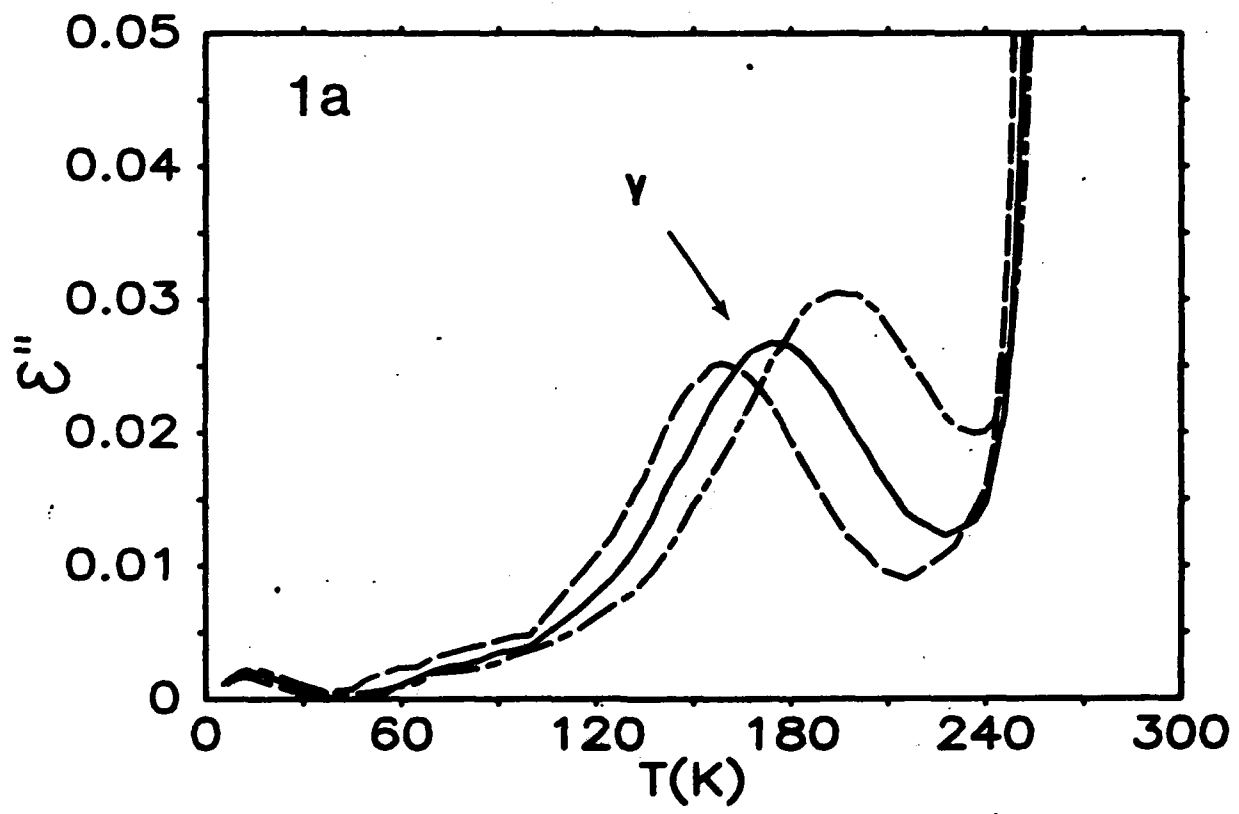
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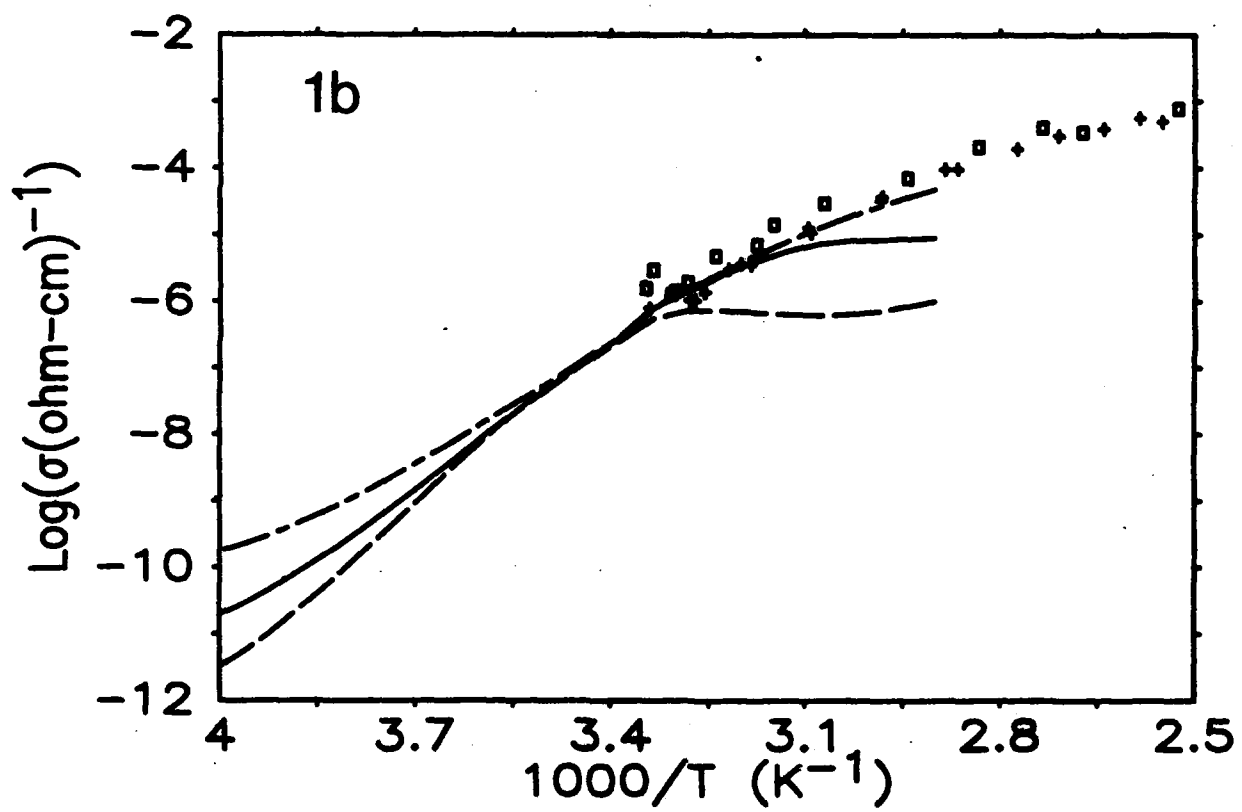
Fig. 1. $\text{PEO}_8\text{-LiClO}_4$. All data are: Dash curve 100 Hz; Solid curve 1000 Hz; Chain link 10,000 Hz. The data are for decreasing temperatures only and straight lines connect the datum points. (a) ϵ'' vs. $T(\text{K})$. (b) $\sigma(\text{ohm-cm})^{-1}$ vs. $1000/T(\text{K})^{-1}$. Also included are the datum points of Weston and Steele (20): \square Polysciences PEO and 99.7% acetonitrile plasticizer; $+$ BDH PEO and distilled acetonitrile plasticizer.

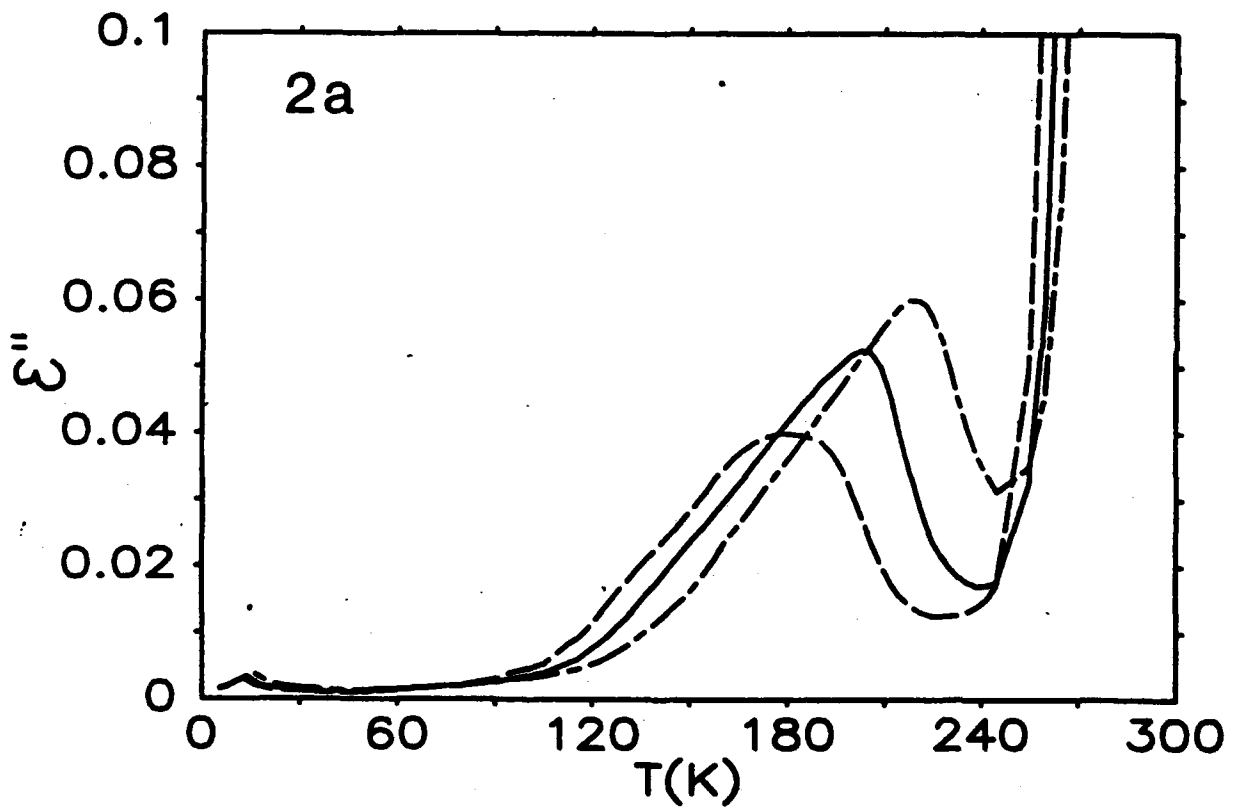
Fig. 2. $\text{PEO}_8\text{-NaClO}_4$. All data are: Dash curve 100 Hz; Solid curve 1000 Hz; Chain link 10,000 Hz. The data are for decreasing temperatures only and straight lines connect the datum points. (a) ϵ'' vs. $T(\text{K})$. (b) $\sigma(\text{ohm-cm})^{-1}$ vs. $1000/T(\text{K})^{-1}$.

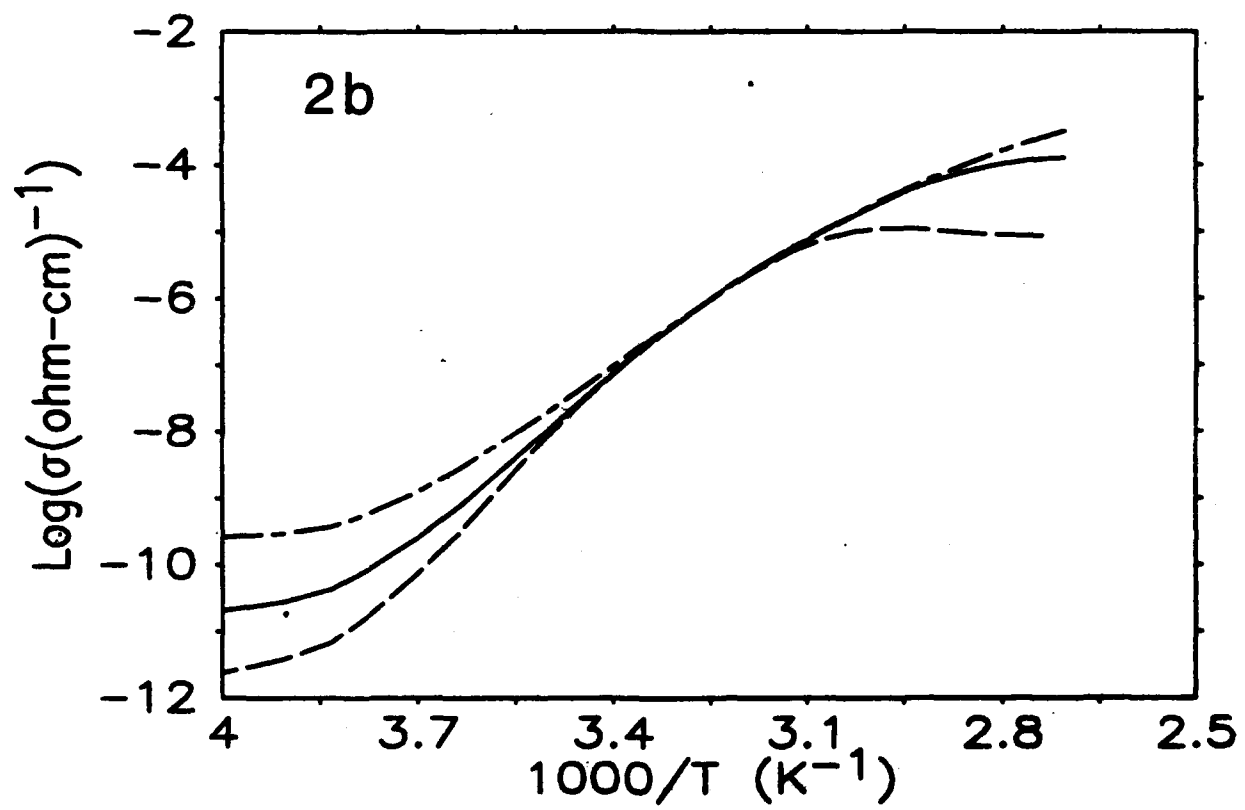
Fig. 3. $\text{PEO}_{4.5}\text{-NaClO}_4$. All data are: Dash curve 100 Hz; Solid curve 1000 Hz; Chain link 10,000 Hz. The data are for decreasing temperatures only and straight lines connect the datum points. (a) ϵ'' vs. $T(\text{K})$. (b) $\sigma(\text{ohm-cm})^{-1}$ vs. $1000/T(\text{K})^{-1}$.

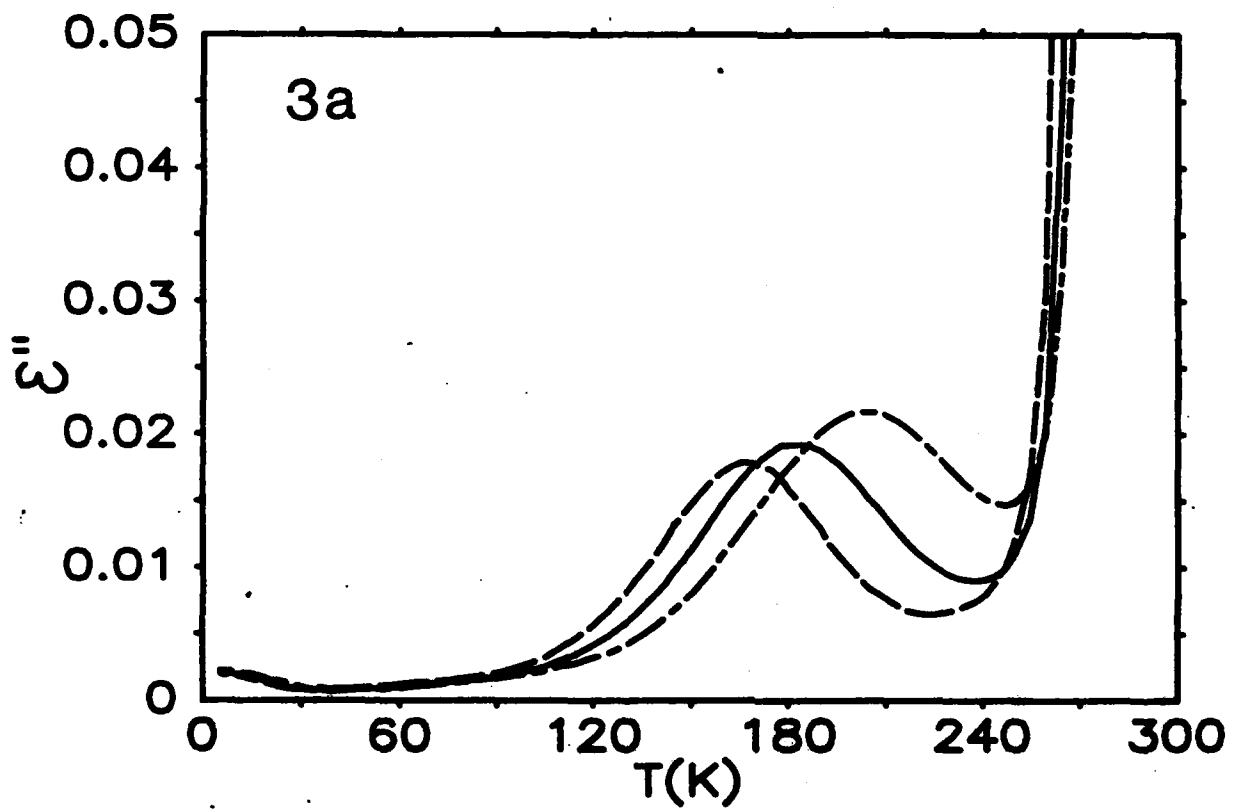
Fig. 4. Low temperature ϵ'' vs. $T(\text{K})$ data for $\text{PEO}_8\text{-LiClO}_4$. The datum points are: Smallest peak height (x) 100 Hz; Middle peak height (Δ) 1000 Hz; Largest peak height (\square) 10,000 Hz. Straight lines connect the datum points.

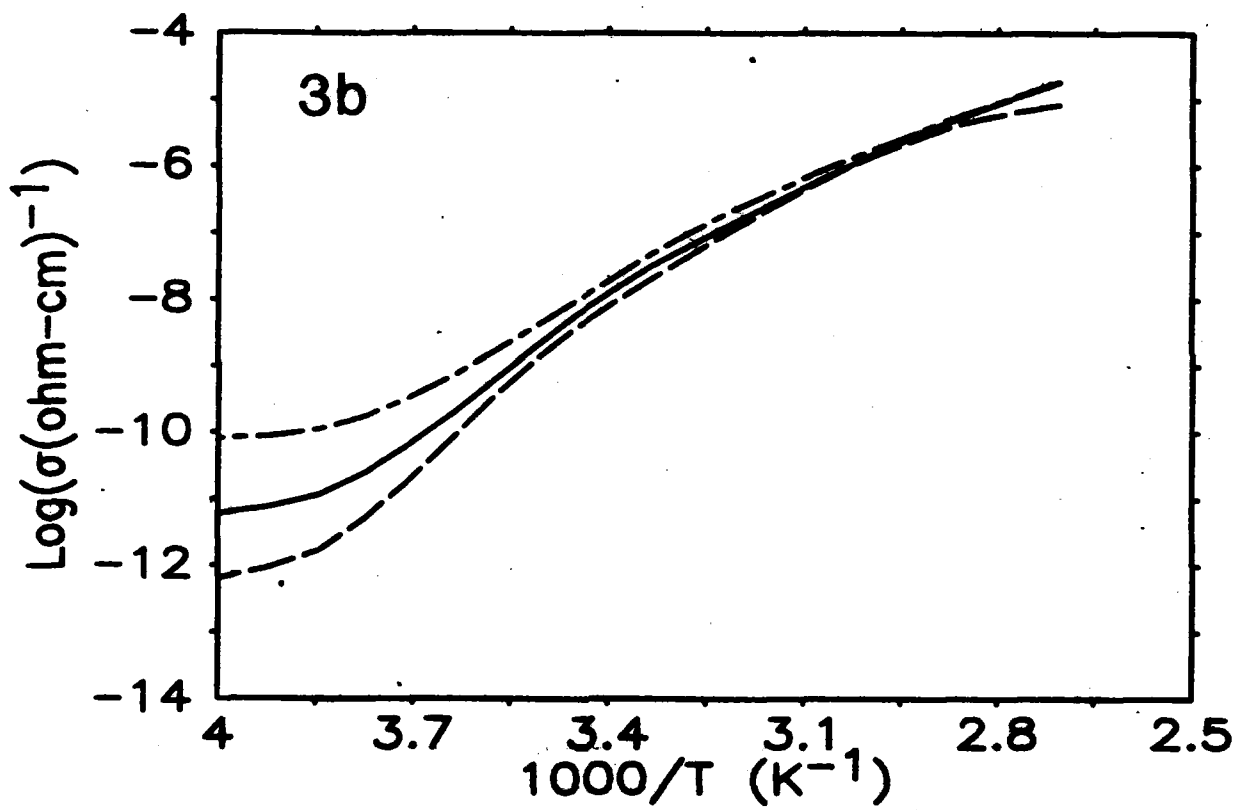


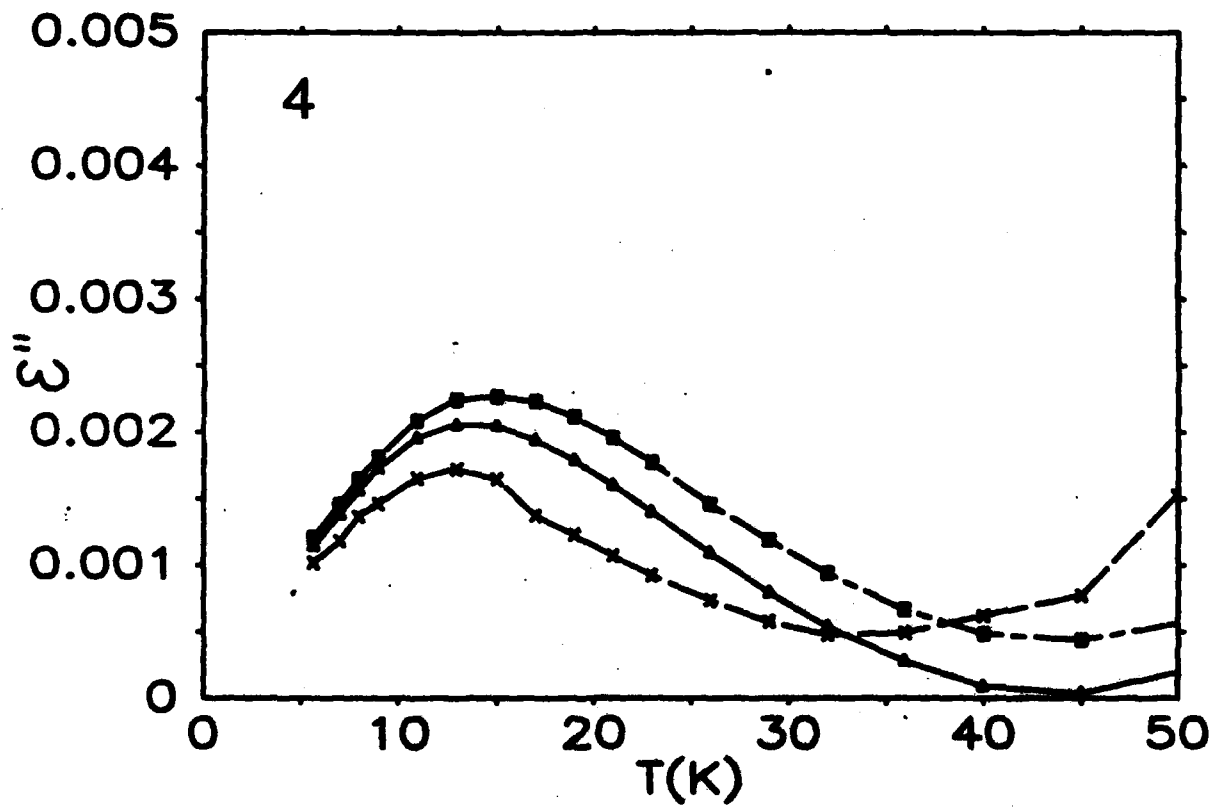












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