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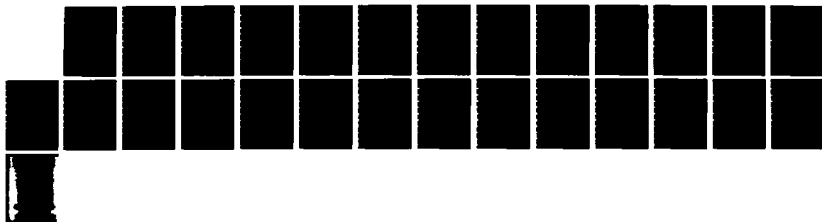
ELECTRICAL AND DIFFERENTIAL SCANNING CALORIMETRY
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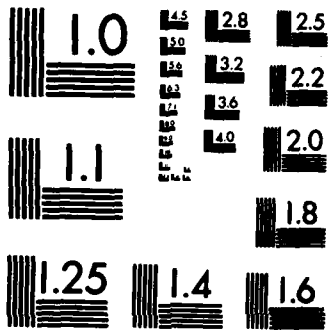
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TECHNICAL REPORT NO. 10

ELECTRICAL AND DIFFERENTIAL SCANNING CALORIMETRY STUDIES OF
POLY(ETHYLENE OXIDE) COMPLEXED WITH ALKALINE EARTH THIOCYANATES

by

John J. Fontanella & Mary C. Wintersgill

Prepared for Publication

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Department of Physics
Annapolis, MD 21402

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corresponding to the γ relaxation of pure PEO depends upon the size of the dopant cation. The activation enthalpy for the second peak is independent of the nature of the dopant cation and is very similar to that observed for the α_c relaxation observed in pure PEO. Next, the room temperature electrical conductivity for the complexed materials is much smaller than that for pure PEO and hence very much less than for PEO complexed with alkali metal salts. However, above T_g , the conductivity rises rapidly and is larger for the barium thiocyanate complexed PEO than for the calcium complexed material. Finally, the DSC studies show that an effect of the ions is to shift the glass transition temperature to high temperatures.



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Electrical and Differential Scanning Calorimetry Studies of
Poly(ethylene oxide) Complexed with Alkaline Earth Thiocyanates

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Synopsis

Audio frequency dielectric relaxation measurements and differential scanning calorimetry studies have been performed on poly(ethylene oxide) (PEO) complexed with calcium and barium thiocyanate. The measurements were performed over the temperature range 5.5–300K. The relaxation spectrum for the complexed material consists of two peaks. The activation enthalpy for the peak corresponding to the γ relaxation of pure PEO depends upon the size of the dopant cation. The activation enthalpy for the second peak is independent of the nature of the dopant cation and is very similar to that observed for the α_c relaxation observed in pure PEO. Next, the room temperature electrical conductivity for the complexed materials is much smaller than that for pure PEO and hence very much less than for PEO complexed with alkali metal salts. However, above T_g , the conductivity rises rapidly and is larger for the barium thiocyanate complexed PEO than for the calcium complexed material. Finally, the DSC studies show that one effect of the ions is to shift the glass transition temperature to higher temperatures.

INTRODUCTION

In recent papers the authors have presented electrical relaxation, electrical conductivity and differential scanning calorimetry (DSC) studies of pure poly(ethylene oxide) (PEO) and PEO complexed with alkali metal thiocyanates,¹ perchlorates,² iodides and trifluoromethane sulfonates.³ In addition, the effect of high pressure on the electrical conductivity has been determined for several of the complexed materials⁴ in order to gain information on the conductivity mechanism in these solid electrolytes. In the present paper, the work is extended to PEO complexed with alkaline earth thiocyanates.

EXPERIMENT

PEO (M.W. 5×10^6) was obtained from Polysciences, Inc. and $\text{Ca}(\text{SCN})_2 \cdot 4\text{H}_2\text{O}$ and $\text{Ba}(\text{SCN})_2 \cdot 3\text{H}_2\text{O}$ were obtained from Alfa Products. Both were dissolved in methanol at about 50°C in the ratio 6.5:1 polymer repeat unit to salt. The solution was cast on a teflon plate and allowed to dry in air. The materials were then vacuum dried at about 55°C . Thick, brittle films were the result.

No special precautions were taken to dry the polymer, salts or the methanol. The philosophy of this experiment was to treat all materials identically and identify differences which should then be due to the salts used to complex the polymer. However, it should be kept in mind that all measurements were performed in vacuum and, in fact, the effects described below were observed after the complexed samples had been annealed in vacuum at 100°C for several hours. Thus, none of the results are attributable to adsorbed water.

Aluminum electrodes were evaporated onto the samples in a two

terminal configuration. For the barium thiocyanate complexed material, one electrode was about 1.5 cm in diameter and the other was about 2.3 cm. For the calcium thiocyanate complexed material, the electrodes were about 0.9 and 1.3 cm in diameter. The geometry was chosen to maximize the efficiency of the sample holder. This configuration limits the accuracy of the strength of the peaks to about 20% because eq. 1 given below is only approximate. However, the relative change in loss is much more accurate.

The measurements were performed in a Cryogenics Associates temperature controlled dewar. Eight different specimens can be measured simultaneously thus allowing a useful comparison of materials. The data were taken with the temperature held constant with approximately 45 min equilibration time at the highest temperatures decreasing to about 20 min at the lowest temperatures. Above 45K the temperature is measured using a Rosemount Eng. Co. platinum resistance thermometer and below 45K it is measured using a CryoCal Inc. germanium resistance thermometer. The temperature is controlled using a 27 Hz feedback temperature controller and in general was controlled to within 0.001K. The absolute temperature is accurate only to about 0.1K. Other results obtained using this system are given elsewhere.¹⁻⁵

The loss measurements were made at the five audio frequencies, 10^2 , $10^{2.5}$, 10^3 , $10^{3.5}$ and 10^4 Hz using a fully automated, microprocessor controlled bridge constructed by one of the authors (C.G.A.) which is as sensitive and accurate as the best commercially available manual bridge. The complex dielectric constant, $\epsilon^* = \epsilon' - j\epsilon''$, was determined from the data as follows. First, ϵ' at 300K and 1000 Hz was determined from the

relation:

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

where C is the value of the capacitance and d and A are the approximate thickness and area and ϵ_0 is the permittivity of free space. The values of ϵ' at the other frequencies were then calculated assuming that the fractional change in ϵ' with frequency equals that for the capacitance. Due to a lack of thermal expansion coefficients, it was also assumed that the percentage change in ϵ' with temperature equals that for the capacitance. Next, values of ϵ'' at all temperatures were calculated using:

$$\epsilon'' = G\epsilon' / \omega C \quad (2)$$

where G/ω is the measured conductance divided by the angular frequency.

Finally, the conductivity was calculated using:

$$\sigma = \epsilon_0 \epsilon'' \omega \quad (3)$$

The DSC measurements were performed using a DuPont 990 differential scanning calorimeter.

DISCUSSION

DSC Studies

The results of the DSC studies are shown in Figures 1 and 2. In both samples a small endothermic event is observed at about 60°C. This is associated with the melting of pure crystalline PEO in the samples. The presence of pure PEO is not surprising as no attempt was made to synthesize fully complexed material. No traces of amorphous PEO are indicated as no low temperature (-60°C) glass transition was observed. However a strong

glass transition is observed in the calcium thiocyanate complexed material at about 35°C in both the as-prepared material and after quenching from 200°C. In the barium thiocyanate complexed material, the glass transition was observed at about 55°C after quench. The large increase in T_g for polyethers due to the incorporation of other inorganic salts is well known.⁶

Electrical Relaxation Studies

The results for ϵ'' at the three frequencies 10^2 , 10^3 and 10^4 Hz are shown in Figures 3 and 4. Two relaxations are apparent. An approximate activation enthalpy, E , and "reciprocal frequency factor," τ_0 , were determined for each sample from the relation:

$$\ln(\omega) = -\ln(\tau_0) - E/kT_{\max} \quad (4)$$

The results for each peak are listed in Table I.

The enthalpy for the higher temperature peak in both materials is found to be approximately 0.5 eV. The similarity of the peak positions for this peak in both materials is obvious from Figure 5 where the 100 Hz data for both complexed materials are plotted. Further, there is a similar peak in pure PEO which is apparent from the Arrhenius plot in Figure 1d of ref.

1. The peak position is close to that for the mechanical relaxation reported by Connor et al.⁷ and Kalfoglou,⁸ which was labeled α_c .

Since the DSC studies reported above show that there is some pure, crystalline PEO in the material, the higher temperature peak is attributed to the α_c relaxation in pure PEO, though the identification is tentative.

It is clear from Figure 5 that the lower temperature peaks in the complexed materials are different from the γ relaxation in pure PEO. In

fact, the peak for the calcium thiocyanate material occurs at a lower temperature than for pure PEO while that for the barium thiocyanate occurs at a higher temperature. This trend is reflected in the activation enthalpies listed in Table I.

The results correlate with ion size considerations and are consistent with attributing the γ relaxation to hydroxyl groups. The γ relaxation has been variously attributed to a "crankshaft mechanism,"^{9,10} "3-bond" or "4-bond" motions,^{11,12} "chain end hydroxyl groups"¹³ or the " $tg^+t \rightleftharpoons tg^-t$ " transition.¹⁴ In previous papers on dielectric relaxation in ion containing PEO¹⁻⁴ the data were interpreted in terms of the " $tg^+t \rightleftharpoons tg^-t$ " transition. However, by preparing deuterated samples of PEO, the authors have recently obtained evidence that the γ relaxation in PEO is more likely to be due to hydroxyl groups at least part of which exist at chain ends as suggested by Hedvig.¹³ That data will be presented elsewhere. The trends observed in the present work can then be explained as follows.

The effect of the ions is to modify the chain structure. Specifically, Ca^{2+} is smaller than the helical tunnel radius for pure PEO while Ba^{2+} is probably larger. Papke et al.¹⁵ concluded that the ttg^+ttg^- PEO conformation has an inner tunnel radius of 0.13-0.15 nm and the Ca^{2+} ion has a radius of 0.1 nm while Ba^{2+} has a radius of 0.135 nm (effective coordination number of 6).¹⁶ Consequently, if the cations reside within the helical tunnels, the effect of Ca^{2+} may be to "tighten" the helix thus making the motion of the contiguous hydroxyl groups easier (lower energy barrier). The effect of the Ba^{2+} , being larger than the tunnel radius, would then be

expected to be opposite.

Considering the current interest in ion conducting PEO^{1-4,9,17-24} the most important result of the present work is that at room temperature the electrical conductivity of the complexed material is less than for the "pure" PEO studied in the present work and thus many orders of magnitude lower than for PEO complexed with most alkali metal salts. This can be seen by comparing Figure 6, where the conductivities for the present samples are plotted, with the figures of reference 1. At 300 K and 100 Hz, for example, the effective electrical conductivity of the Ba(SCN)₂ complexed material is about $5 \times 10^{-12} \text{ (ohm-cm)}^{-1}$ and that for Ca(SCN)₂ complexed material is about twice as large. There is also a great deal of dispersion in this region with the conductivities approximately proportional to ω (ϵ'' approximately constant). These values are to be compared with a value of about $10^{-8} \text{ (ohm-cm)}^{-1}$ for "pure" PEO¹ and typical values of about $10^{-5} \text{ (ohm-cm)}^{-1}$ for PEO complexed with alkali metal salts. The most likely interpretation of the low conductivities is that the alkaline earth and thiocyanate ions form a non-conducting matrix at room temperature which has the additional effect of inhibiting the motion of the trace impurities which are presumably responsible for the conductivity in the "pure" PEO. A relevant observation is that samples of hot-pressed "pure" PEO show the same conductivity as the cast films. Consequently, the trace impurities are not introduced via the solvent used in casting the films and are probably present in the as-received polymer.

However, the conductivity in both samples rises rapidly with temperature above room temperature and particularly in the barium thiocyanate complexed material, reaches a relatively high value. The higher conductivity for the barium thiocyanate material is attributed to the large

polarizability of the barium ion. This suggests that PEO complexed with lead thiocyanate, for example, should exhibit higher conductivities still.

Finally, it should be pointed out that the low room temperature conductivity and onset of the electrical conductivity at higher temperatures for the alkaline earth thiocyanate complexed materials is probably related to the higher temperature glass transition for these materials.

Specifically, it has been suggested^{15,17,18} that large amplitude polymer segmental motion is important for ion transport. Consequently, large ionic conductivities are only expected above T_g where the chains are mobile and thus a consistent interpretation of the present results can be obtained assuming ionic conductivity. However, it should be kept in mind that the nature of the charge carriers in these materials has yet to be determined.

SUMMARY

In summary, then, DSC and audio frequency electrical relaxation measurements on PEO complexed with AE thiocyanates have yielded the following results:

- (1) T_g in the complexed materials is found to be shifted to higher temperatures than for pure PEO.
- (2) The DR spectrum consists of two peaks. One peak is tentatively attributed to α_c for pure PEO. The position of the second peak which occurs in the γ relaxation region, is lower (higher) in temperature for the calcium (barium) thiocyanate complexed material than for pure PEO. This correlates with the fact that calcium is smaller than the tunnel radius while barium is larger.
- (3) The room temperature σ for the complexed materials is smaller than for the "pure" PEO studied but rises rapidly above T_g and is larger for the

barium thiocyanate complexed material than for the calcium thiocyanate complexed material.

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TABLE I

Activation parameters for the relaxations observed in pure and calcium and barium thiocyanate complexed poly(ethylene oxide).

Relaxation	Material	τ_0 (10^{-14} s)	E (eV)
α_c	PEO _{6.5} Ba(SCN) ₂ :3H ₂ O	36	0.48
γ	PEO _{6.5} Ba(SCN) ₂ :3H ₂ O	4.3	0.37
γ	PEO	6.0	0.33
γ	PEO _{6.5} Ca(SCN) ₂ :4H ₂ O	14	0.30

Figure Captions

Fig. 1. DSC thermograms for $\text{PEO}_{6.5}:\text{Ca}(\text{SCN})_2:4\text{H}_2\text{O}$. The solid curve (Before) is for the as-prepared material. The dashed curve (After) is for the sample after being held at 200°C for 2 hr. then quenched to -100°C as rapidly as possible with the sample in the calorimeter using the cooling tower. The two curves are displaced for clarity.

Fig. 2. DSC thermograms for $\text{PEO}_{6.5}:\text{Ba}(\text{SCN})_2:3\text{H}_2\text{O}$. The solid curve (Before) is for the as-prepared material. The dashed curve (After) is for the sample after being held at 200°C for 2 hr. then quenched to -100°C as rapidly as possible with the sample in the calorimeter using the cooling tower. The two curves are displaced for clarity.

Fig. 3. ϵ'' vs. $T(\text{K})$ for $\text{PEO}_{6.5}:\text{Ca}(\text{SCN})_2:4\text{H}_2\text{O}$. Straight line segments connect the datum points and the frequencies are: dash, 100 Hz; solid, 1000 Hz; chain link 10,000 Hz.

Fig. 4. ϵ'' vs. $T(\text{K})$ for $\text{PEO}_{6.5}:\text{Ba}(\text{SCN})_2:3\text{H}_2\text{O}$. Straight line segments connect the datum points and the frequencies are: dash, 100 Hz; solid, 1000 Hz; chain link 10,000 Hz.

Fig. 5. ϵ'' vs. $T(\text{K})$ at 100 Hz for various materials: solid, "pure" PEO; chain link, $\text{PEO}_{6.5}:\text{Ca}(\text{SCN})_2:4\text{H}_2\text{O}$; dash, $\text{PEO}_{6.5}:\text{Ba}(\text{SCN})_2:3\text{H}_2\text{O}$.

Fig. 6. $\text{Log}_{10}(\sigma(\text{ohm-cm})^{-1})$ vs. $1000/T$ (K^{-1}) for
PEO_{6.5}:Ba(SCN)₂:3H₂O (upper set of curves) and
PEO_{6.5}:Ca(SCN)₂:4H₂O (lower set of curves). The frequencies
are: dash, 100 Hz; solid, 1000 Hz; chain link 10,000 Hz.

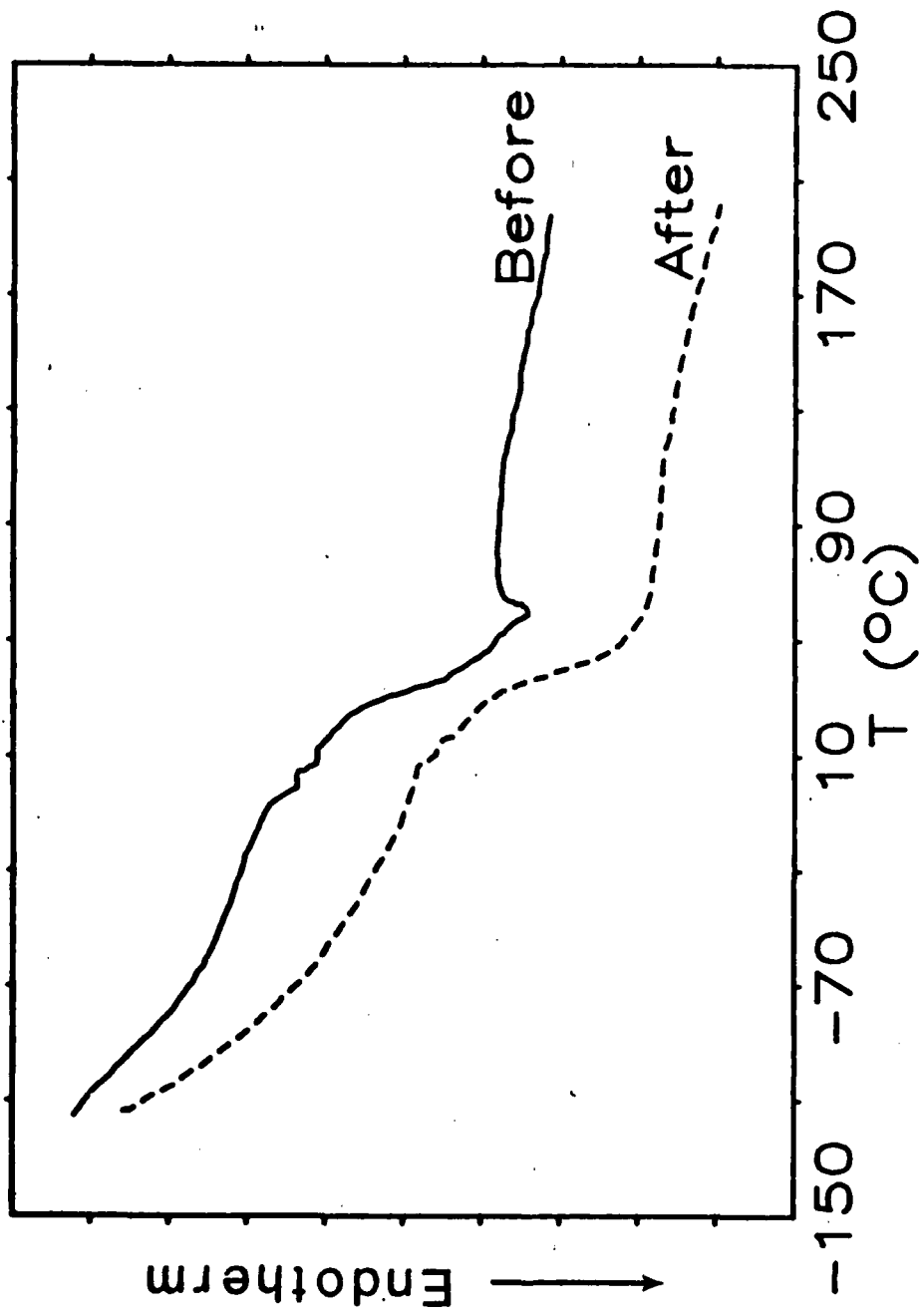


Figure 1

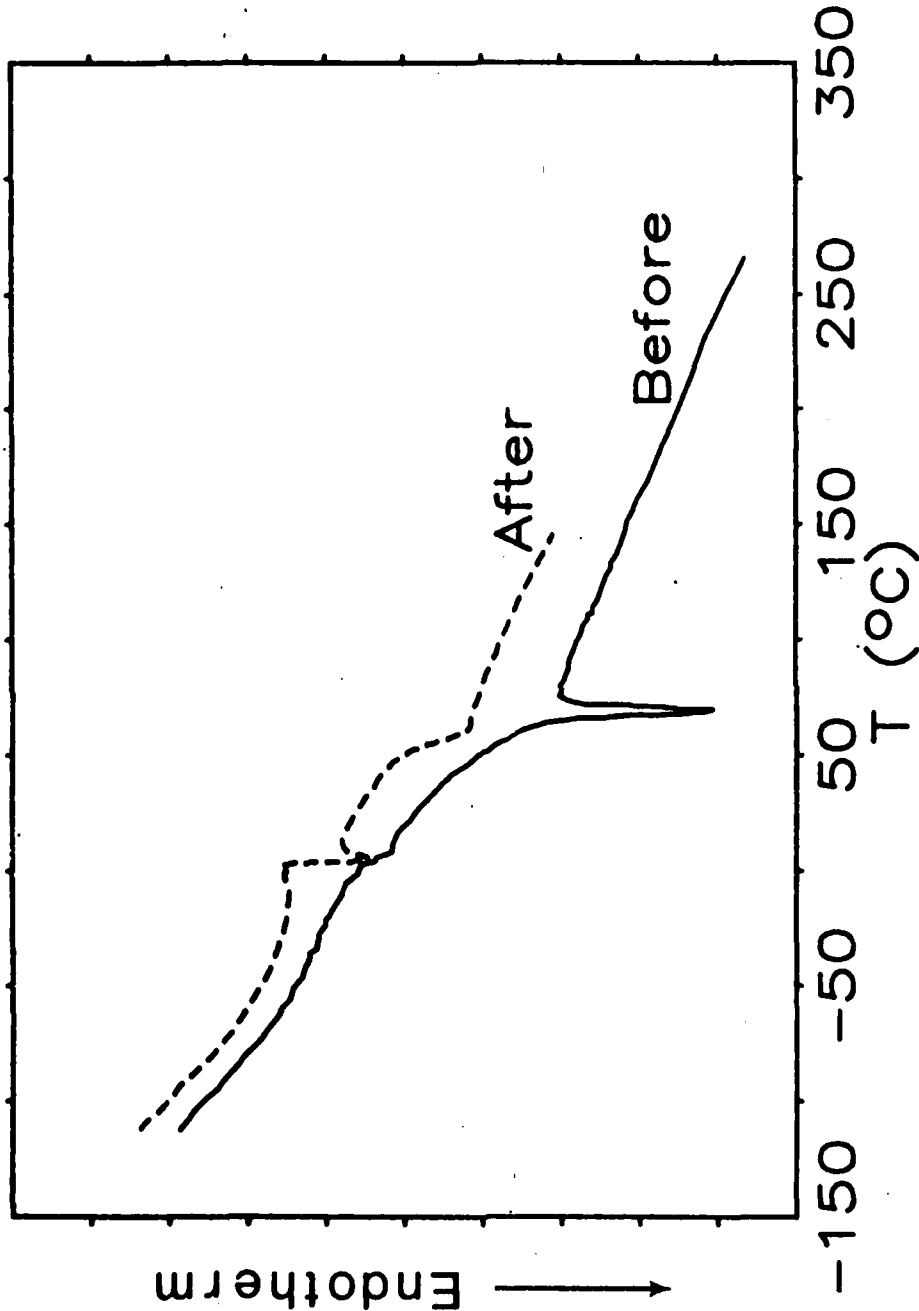


Figure 2

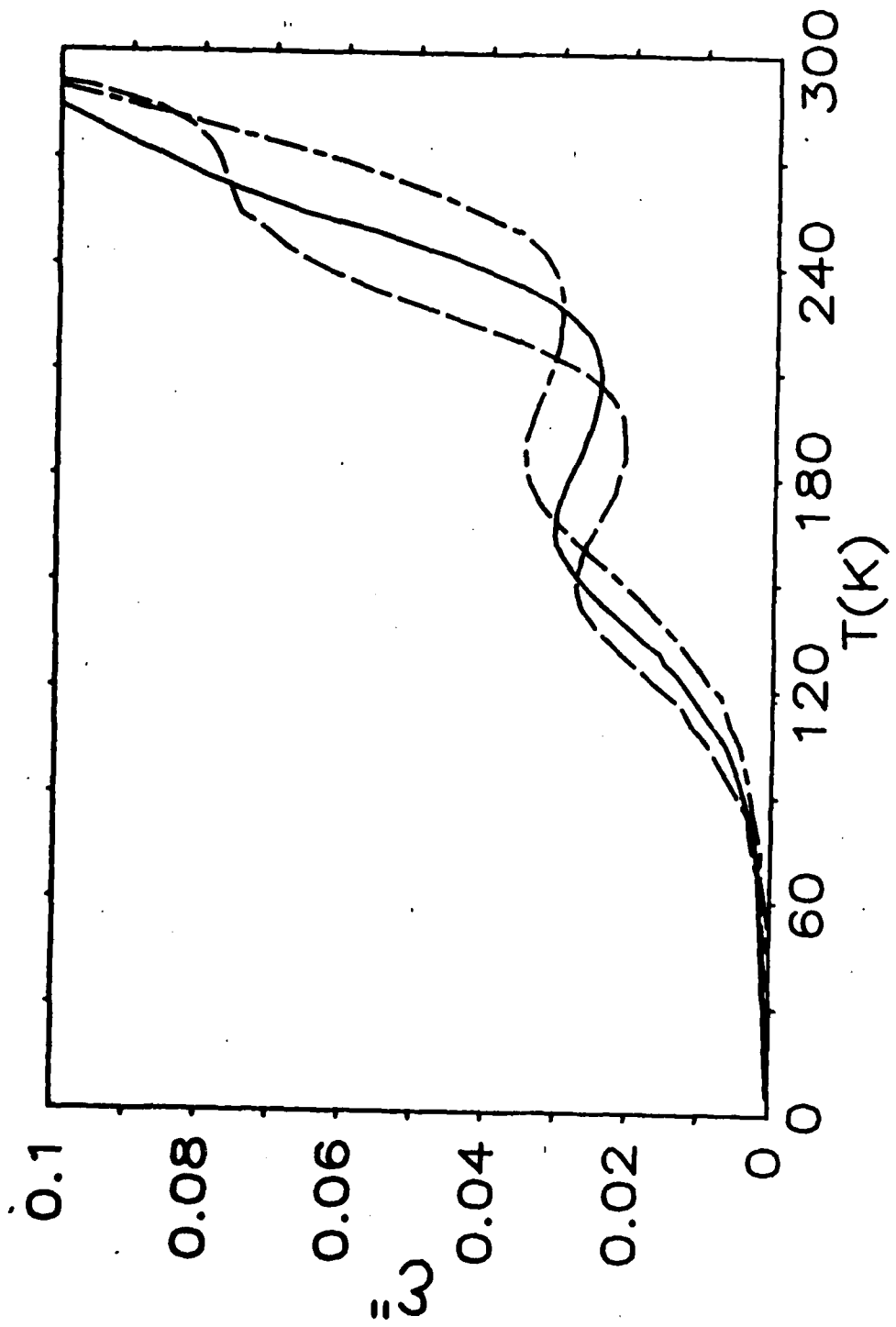


Figure 3

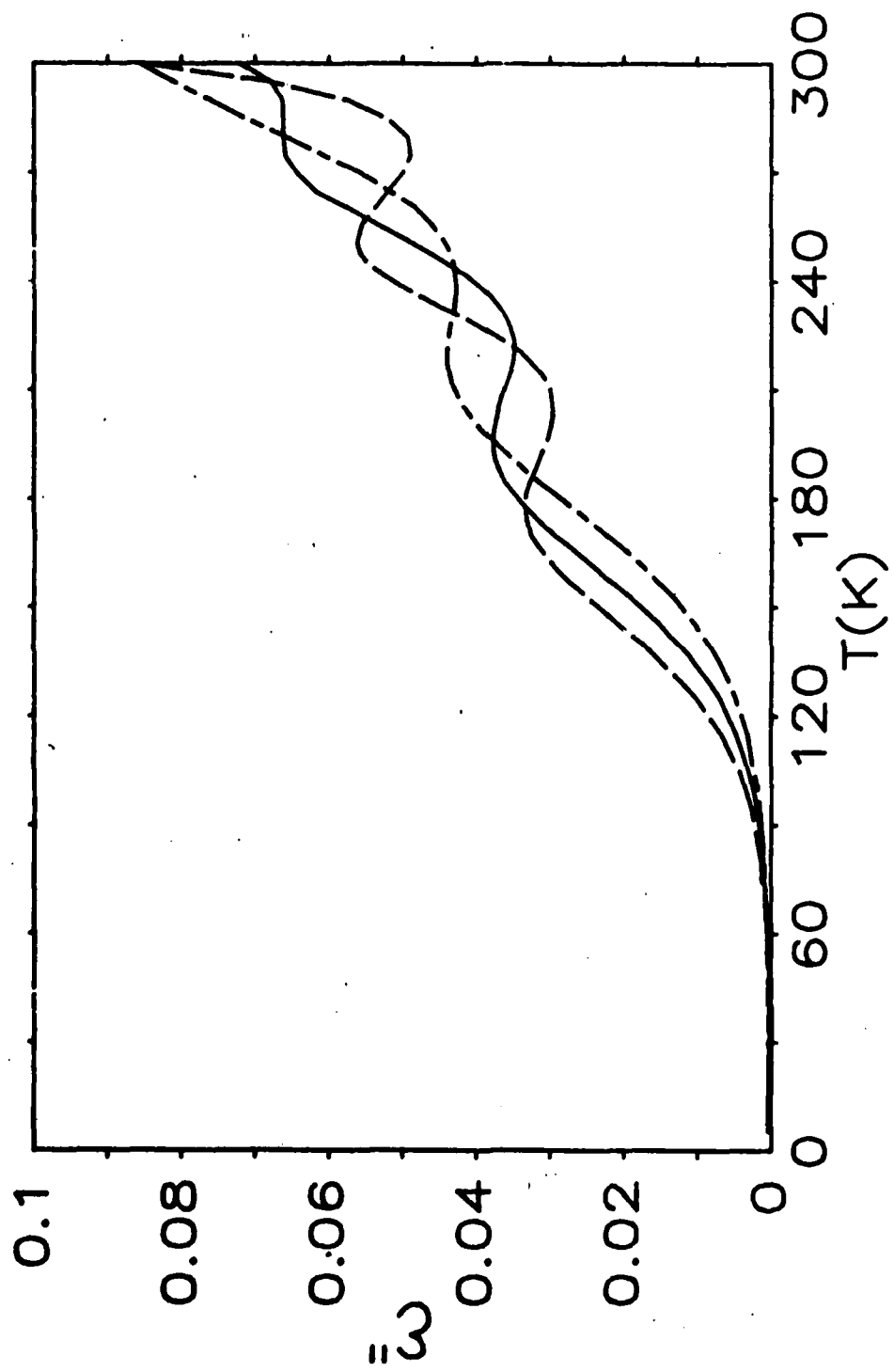


Figure 4

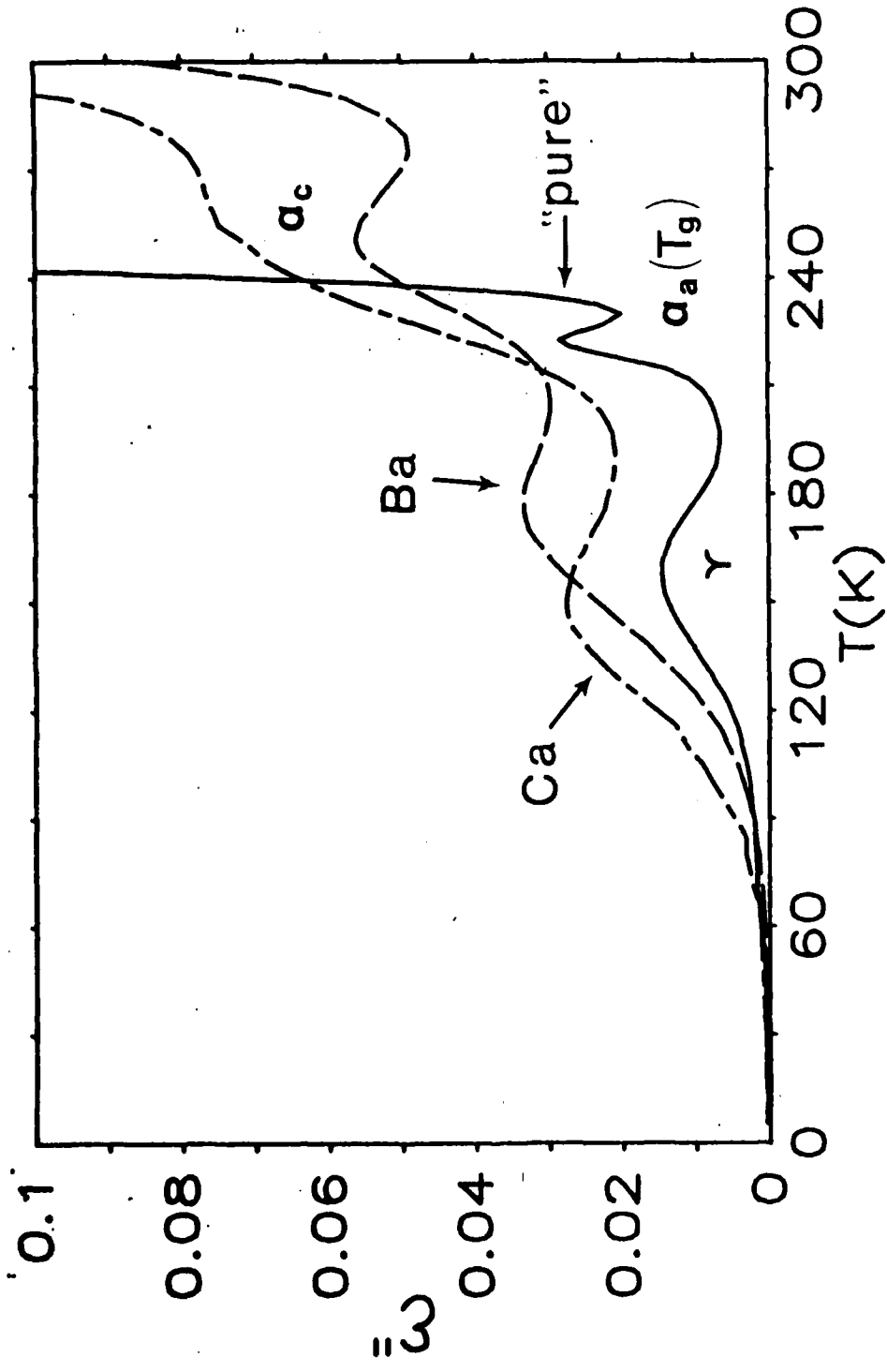


Figure 5

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