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WATER ABSORPTION AND CONDUCTIVITY IN POLY(ETHYLENE
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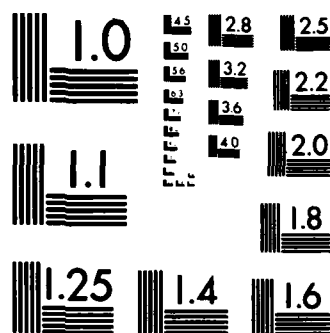
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Contract N00014-77-C-0636

Task No. NR 359-667

TECHNICAL REPORT NO. 6

Water Absorption and Conductivity in
Poly(Ethylene Oxide)-Lithium Tetrafluoroborate Films

by

M. M. Nicholson and T. P. Weismuller

Prepared for Publication

in the

Journal of the Electrochemical Society

Science Center
Rockwell International Corporation
Anaheim, California

October, 1984

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4. PERFORMING ORGANIZATION REPORT NUMBER(S) SC5384.1TR			5. MONITORING ORGANIZATION REPORT NUMBER(S)	
6a. NAME OF PERFORMING ORGANIZATION Rockwell International Science Center		6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION Office of Naval Research	
6c. ADDRESS (City, State and ZIP Code) P.O. Box 3105 Anaheim, CA 92803			7b. ADDRESS (City, State and ZIP Code) 800 North Quincy Arlington, VA 22217	
8a. NAME OF FUNDING/SPONSORING ORGANIZATION		8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER Contract N00014-77-C-0636	
8c. ADDRESS (City, State and ZIP Code)			10. SOURCE OF FUNDING NOS.	
			PROGRAM ELEMENT NO.	TASK NO. NR 359-667
11. TITLE (Include Security Classification): Water Absorption and Conductivity in Poly(ethylene oxide)-LiBF ₄ Films			PROJECT NO.	WORK UNIT NO.
12. PERSONAL AUTHOR(S) M. M. Nicholson and T. P. Weismuller				
13a. TYPE OF REPORT Technical		13b. TIME COVERED FROM 1-1-83 TO 12-31-83	14. DATE OF REPORT (Yr., Mo., Day) October 1984	15. PAGE COUNT 32
16. SUPPLEMENTARY NOTATION Prepared for publication in the Journal of the Electrochemical Society				
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB. GR.	Poly(ethylene oxide), lithium tetrafluoroborate, water, conductivity, syneresis	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) 0003 Water-vapor absorption and conductivity in spin-cast poly(ethylene oxide)-lithium tetrafluoroborate films were investigated near 21°C by in situ near-infrared spectroscopy and ac conductance measurements. The film conductivity was 2×10^{-5} ohms/cm until the water content exceeded that corresponding to LiBF ₄ ·3H ₂ O. It increased with additional water, reaching 3×10^{-3} ohms/cm at the composition equivalent to LiBF ₄ ·20H ₂ O. Most of the water above the trihydrate level was unstably absorbed, however, and began to escape from the film within a few minutes. This syneresis was attributed to spontaneous restructuring or crystallization of the polymer. The conductivity data were interpreted on the basis of a two-phase model in which one portion of the salt remained in a nonconductive solid region, while the other portion dissolved in a more fluid conductive polymer-water region.				
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS ☐			21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL Jerry J. Smith			22b. TELEPHONE NUMBER (Include Area Code) (202) 696-4409	22c. OFFICE SYMBOL ONR - Code 472

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WATER ABSORPTION AND CONDUCTIVITY IN
POLY(ETHYLENE OXIDE)-LITHIUM TETRAFLUOROBORATE FILMS

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ABSTRACT

Water-vapor absorption and conductivity in spin-cast poly(ethylene oxide)-lithium tetrafluoroborate films were investigated near 21°C by in situ near-infrared spectroscopy and ac conductance measurements. The film conductivity was $\leq 2 \times 10^{-5} \text{ ohms}^{-1} \text{ cm}^{-1}$ until the water content

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Key words: Poly(ethylene oxide), lithium tetrafluoroborate, water, conductivity, syneresis.



exceeded that corresponding to $\text{LiBF}_4 \cdot 3\text{H}_2\text{O}$. It increased with additional water, reaching $3 \times 10^{-3} \text{ ohms}^{-1} \text{ cm}^{-1}$ at the composition equivalent to $\text{LiBF}_4 \cdot 20\text{H}_2\text{O}$. Most of the water above the trihydrate level was unstably absorbed, however, and began to escape from the film within a few minutes. This syneresis was attributed to spontaneous restructuring or crystallization of the polymer. The conductivity data were interpreted on the basis of a two-phase model in which one portion of the salt remained in a nonconductive solid region, while the other portion dissolved in a more fluid conductive polymer-water region.



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Complexes of salts with aliphatic poly-ethers are a relatively new class of solid electrolytes (1,2). Some of these materials can attain conductivities of the order of $10^{-5} \text{ ohm}^{-1} \text{ cm}^{-1}$ at room temperature, and the conduction is known to increase greatly on absorption of water. Because of their potential importance for lithium battery electrolytes, most research on such complexes has been done with carefully dried preparations. For other types of batteries and electrochemical devices, however, some water is acceptable, or even required, and the higher conductivity is advantageous. The influence of water on these solids is, in any case, a point of scientific interest which has received only cursory treatment in previous studies.

This paper describes results of conductance measurements near 21°C on thin solid films of poly(ethylene oxide) (PEO) complexed with lithium tetrafluoroborate, and containing various amounts of water. The water content of the films was monitored in situ by near-infrared absorption spectroscopy while the ac conductance at 1,000 Hz was determined on an impedance bridge. This thin-film technique permitted much faster acquisition of data than conventional bulk methods for investigation of polymers. Both the water content and the conductance showed a peculiar but repeatable time dependence which probably was caused by spontaneous restructuring of the polymer.



EXPERIMENTAL

The PEO preparation, with an average molecular weight of 4,000,000, was from Polysciences, Inc. The LiBF_4 was from Ozark-Mahoning. Electrolyte films of the order of 1 μm thick were prepared on single-crystal sapphire plates by spin casting from a viscous aqueous solution containing 4.5 polymer repeating units per molecule of LiBF_4 . These plates had, near one edge, a pair of narrow-line electrodes formed from conductive silver paste, with an interelectrode spacing of 0.4 mm. Margins around the film were defined by masking tape, which was removed after the casting process.

The films were dried under vacuum at room temperature for 24 hr and transferred, without air exposure, to a helium-atmosphere glove box, where they were mounted in the double cell shown in Fig. 1. For the measurements of conductance and water absorption, the cell was placed in a Beckman DK-2A spectrophotometer, with the electrical leads connected to a Fluke Model 710B impedance bridge. Nitrogen containing known partial pressures of water was passed into the optical reference compartment of the cell and out through the sample compartment. The water pressure was controlled at known levels by passing the nitrogen through water in a cooled, thermostatted gas-washing bottle. The moist nitrogen then travelled through a long copper tube to reach room temperature, near 21°C, before entering the cell.



Resistances and near-infrared (IR) spectra of the films were determined at various times up to 2 hr, and, in some instances, the transmittance at the characteristic wavelength of the OH stretching band near $2.9\text{ }\mu\text{m}$ was recorded continuously as a function of time. The amount of water present per unit film area during an experiment was found by comparing the IR absorbance of the film with that of a known thickness of liquid water retained between two glass slides. Following a systematic run, the cell could be placed, open, in the vacuum chamber to re-dry the film in preparation for the next set of measurements.

Two methods were used to determine the original thickness of the PEO-LiBF₄ film after completion of the conductance measurements. The polymer electrolyte was too soft for measurement with a profilometer. For a rough thickness estimate (+40%) the used film was re-dried, then dissolved in water, and the resulting weight loss noted. For a more accurate evaluation, the lithium content of the dissolved film was determined by atomic absorption. The density of the anhydrous PEO-LiBF₄ preparation was found to be 1.23 g/cm^3 by weighing a known volume of the bulk solid packed into a glass tube.



RESULTS AND DISCUSSION

The System PEO-LiBF₄-H₂O.--The PEO-LiBF₄ system at the 4.5:1 mole ratio was selected for investigation primarily because the anhydrous complex of this composition was known to have the comparatively high conductivity of $10^{-5} \text{ ohm}^{-1} \text{ cm}^{-1}$ at 20°C (1). This suggests a tendency toward amorphous behavior which could be conducive to the incorporation of water in the organic phase.

The PEO-LiBF₄-H₂O films were colorless and highly transparent. As expected, water was readily absorbed from the vapor phase with an immediate increase in conductivity. Figure 2 shows a typical set of near-infrared spectra recorded after thirty-minute exposures to each of the indicated partial pressures of water. The maximum absorption due to OH stretching occurred at 2.90 μm in pure liquid water. This band acquired a doublet character in the PEO-LiBF₄-H₂O films, with increased absorption near 2.85 μm , which was more evident at the lower water levels. The shift to the shorter wavelength apparently was due to binding of water with the lithium ion, since it did not occur with PEO in the absence of the salt.

Conductances ($1/R$) of two PEO-LiBF₄ films are plotted as functions of the water absorbance in Fig. 3 and 4. The symbol R represents the measured resistance of the film specimen. The anhydrous film thickness was roughly 1.8 μm for Film 1 and, more accurately, 0.88 μm for Film 2. The circles and other symbols represent different runs which covered a wide range of water pressures



and standing times, as well as different pre-drying conditions. It is noteworthy that the data for each film can be adequately represented by a single line, even though many nonequilibrium points are included. The significance of the slopes of these lines is discussed later. The absorbance intercept for Film 2 in Fig. 4 corresponds to the composition $\text{LiBF}_4 \cdot 2.9\text{H}_2\text{O}$. The approximate composition $\text{LiBF}_4 \cdot 2.5\text{H}_2\text{O}$ was found in a similar way for Film 1 in Fig. 3. Two hydrates of LiBF_4 have been characterized in the literature (3,4). $\text{LiBF}_4 \cdot \text{H}_2\text{O}$ is stable above 23°C in the presence of an aqueous solution of the salt, while $\text{LiBF}_4 \cdot 3\text{H}_2\text{O}$ crystallizes below 23°C . In the present study, near 21°C , the measured film conductances dropped abruptly to $<10^{-7} \text{ ohm}^{-1}$ as the water content fell below the trihydrate composition. It is thus apparent that the initial portion of the absorbed water was utilized primarily in hydration of the salt. The "free" water above the trihydrate level was responsible for the higher magnitude of conductivity discussed in this paper.

The absorption of water by the PEO-LiBF_4 films displayed an unusual time dependence. Figure 5 illustrates this behavior in the IR absorbance recorded under a constant water vapor pressure of 18.7 torr. After 24 hr of pre-drying under vacuum, the film added water rapidly at first. The absorbance then reached a maximum in 7 to 10 min, after which it gradually decayed toward the level corresponding to $\text{LiBF}_4 \cdot 3\text{H}_2\text{O}$. This behavior was repeatable with thorough drying between runs. Less extensive drying produced a related, but distinctly different, result shown by the lower curve in Fig. 5. With a vacuum drying time of



0.5 hr, the initial absorbance again was essentially zero. The water uptake also was quite rapid at first, but the loss set in after only 1 min, and the IR absorbance approached the asymptotic value much earlier than it did with 24 hr of drying.

The change of conductance with time closely paralleled that of the water content, as one can infer from Fig. 3 and 4. This is shown explicitly by Fig. 6 for the same runs that are represented in Fig. 5.

The System PEO-H₂O.--It was of major interest to know whether the spontaneous water loss was a property of the salt or the polymer. A spin-cast film of PEO without the salt was prepared for this purpose and dried under vacuum 24 hr. Its infrared absorption under 18.7 torr of water vapor was then examined.

Infrared spectra recorded at various exposure times under constant water pressures are shown in Fig. 7. This group of spectra differs in several ways from the PEO-LiBF₄-H₂O spectra of Fig. 2. The salt-free film was visually cloudy, and infrared light scattering is evident in the lower transmission range of the spectra. Although the absorption maximum shifted slightly with water content, any doublet character of the OH band is not obvious. Finally, the salt-free spectra showed isosbestic points at 2.76 and 3.21 μ m. Such points are characteristic of a fixed stoichiometry between initial and final species in a chemical process. They were not observed with the nonscattering films



containing LiBF_4 . Fig. 8 indicates a time-dependent gain and subsequent loss of water by the PEO which is very similar to that for PEO- LiBF_4 in Fig. 5. The final water absorbance was relatively closer to zero, however, in the salt-free film.

Although the scattering effect prevented an absolute determination of water in the PEO, it is clear that the major time dependence was associated with the polymer. A comparable syneresis, or loss of solvent component, was reported by Weiss and Lenz for water in phosphonate esters derived from PEO oligomers (5). Those authors determined the absorbed water gravimetrically on bulk samples in experiments lasting several days. With the thin-film technique used here, the time dependence could be detected in a few minutes and well characterized in about an hour. An apparently related heat-induced syneresis in cross-linked PEO- H_2O gels was attributed by Graham *et al.* to increased ordering of the polymer, with some water still attached (6).

Further Correlations.--Conductivity relationships in the PEO- LiBF_4 - H_2O system can be examined in greater detail by means of Fig. 9, in which the total thickness of the swollen film is considered, and Fig. 10, which treats the free water, with its incremental thickness, as a separate region supporting of all the observed conduction.



For calculating the bulk conductivity σ plotted in Fig. 9, the total film thickness was taken, as a first approximation, to be the sum of the dry PEO-LiBF₄ thickness, based on bulk density, and the liquid water thickness equivalent to the IR absorbance of the film. This estimated total thickness ranged from 0.88 μm in the dry state to 2.26 μm for the swollen film at a ratio of 21 moles of H₂O per mole of LiBF₄. All of the smoothed data points for Film 2 fell close to a straight line intersecting the mole-ratio axis at 3.1. The corresponding plot for Film 1 was a slightly curved line with a similar intercept; it is not shown because of uncertainty in the dry-film thickness.

The cause of the linear relationship in Fig. 9. is not obvious. In a very simple model for the system, it could be envisioned that all of the salt dissolves in a homogeneous conductive phase, with water acting only as a diluent. The conductivity would then decrease with increasing water content. Factors opposing this effect could be the dissociation of ion pairs and the lowering of viscosity on addition of water. However, those trends, superimposed on the dilution, would not account for the straight line in Fig. 9. The actual behavior was more consistent with a system of two microphases, one a conductive fluid or amorphous region containing dissolved salt, and the other a nonconductive solid including the remainder of the LiBF₄·3H₂O. The linear relationship could then occur if the dissolved-salt concentration in the film as a whole were proportional to the free-water content and the mobilities of Li⁺ and BF₄⁻ remained constant. This model treats the



conductive microphase as an aqueous region saturated with both PEO and LiBF_4 and disregards effects of porosity or tortuosity created by the solid PEO-salt matrix.

A lower limit for the apparent equivalent conductance of LiBF_4 can be estimated as follows from the plot in Fig. 9. At the mole ratio of 20, the concentration c of LiBF_4 dissolved in the entire film had to be ≤ 1.65 M. With the corresponding experimental conductivity σ of $3.1 \times 10^{-3} \text{ ohms}^{-1} \text{ cm}^{-1}$, the equivalent conductance Λ_{LiBF_4} , expressed as $1000 \sigma/c$, would have been $\geq 1.9 \text{ ohms}^{-1} \text{ cm}^2 \text{ mole}^{-1}$. It may be noted from the work of Radchenko and Ryss that LiBF_4 completely dissolves in pure water at a ratio as low as 5.35 moles of water per mole of salt (7). In the presence of PEO, however, the salt probably is much less soluble. The lower limit of $1.9 \text{ ohms}^{-1} \text{ cm}^2 \text{ mole}^{-1}$ for Λ_{LiBF_4} is two orders of magnitude less than typical equivalent conductances of salts in water, but it is not inconsistent with their behavior in viscous polymer-water mixtures. For example, a paper by Isono *et al.* indicates an equivalent conductance of $7.9 \text{ ohms}^{-1} \text{ cm}^2 \text{ mole}^{-1}$ for NaCl in a concentrated poly(ethylene glycol)-water mixture at 25°C (8).

Figure 10 accentuates the contrast between the two types of film behavior encountered in this study. An incremental conductivity σ_{aq} was calculated by taking the effective film thickness as that equivalent to the free water. This incremental thickness was found from the IR absorbance



term $A - A'$, where A is the measured absorbance and A' is the intercept from Fig. 3 or Fig. 4. These are nonequilibrium data. The pronounced difference between the plots in Fig. 10 may reflect a difference in the rates of syneresis for the two films: The polymer apparently rearranged readily in Film 1 to an ordered, or crystalline, phase, forming an aqueous region with a constant conductivity σ_{aq} of $6.2 \times 10^{-3} \text{ ohms}^{-1} \text{ cm}^{-1}$. This water was subsequently lost by evaporation. With slower crystallization in Film 2, the data included some intermediate stages, where σ_{aq} was lower due to more polymer in solution. The same limiting level was eventually reached by Film 2, however, and water loss occurred as represented in Fig. 5.

Further research is planned to characterize related polymer-salt-solvent electrolyte systems.

ACKNOWLEDGMENT

This work was performed in part by the Office of Naval Research.



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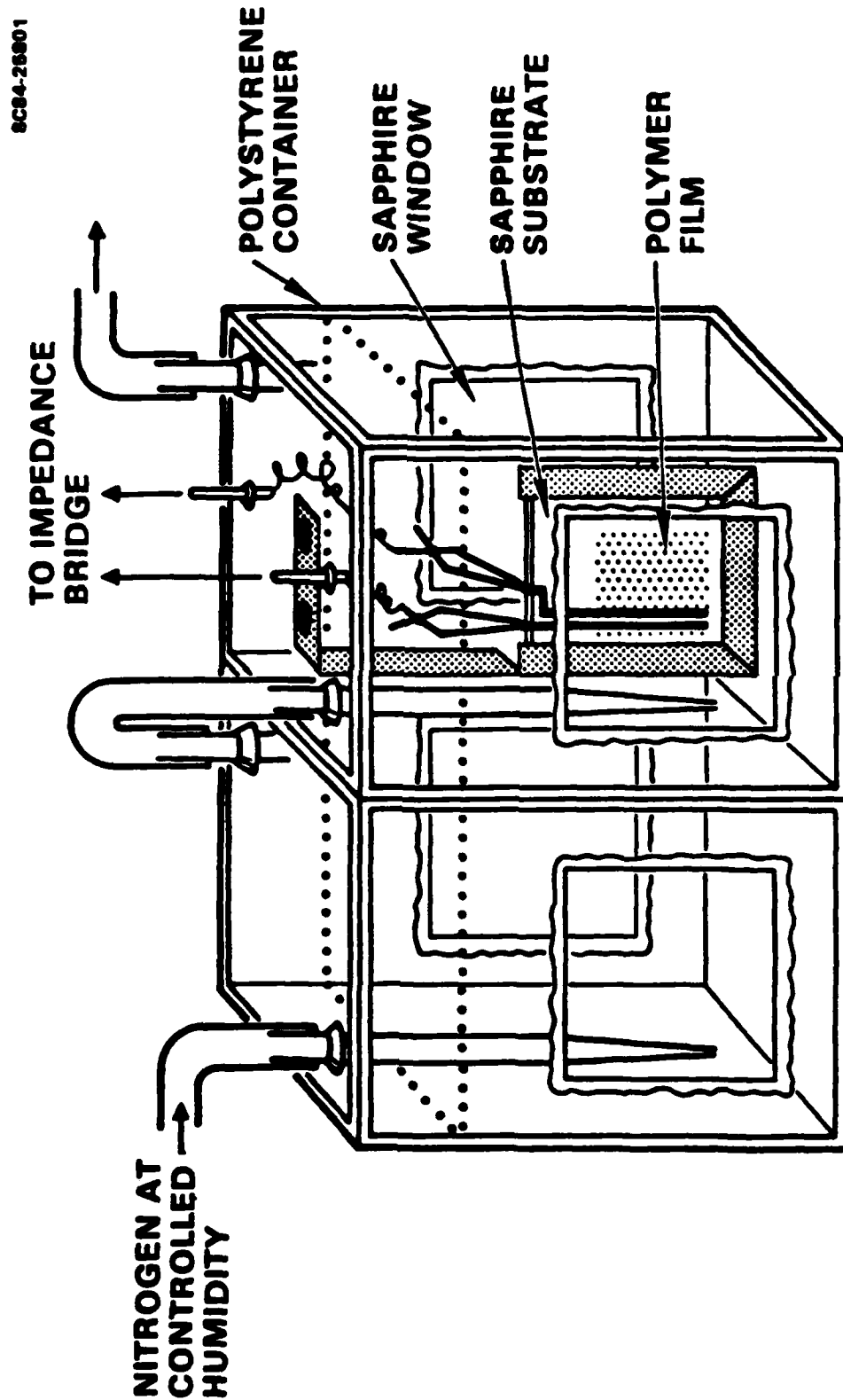


ILLUSTRATIONS

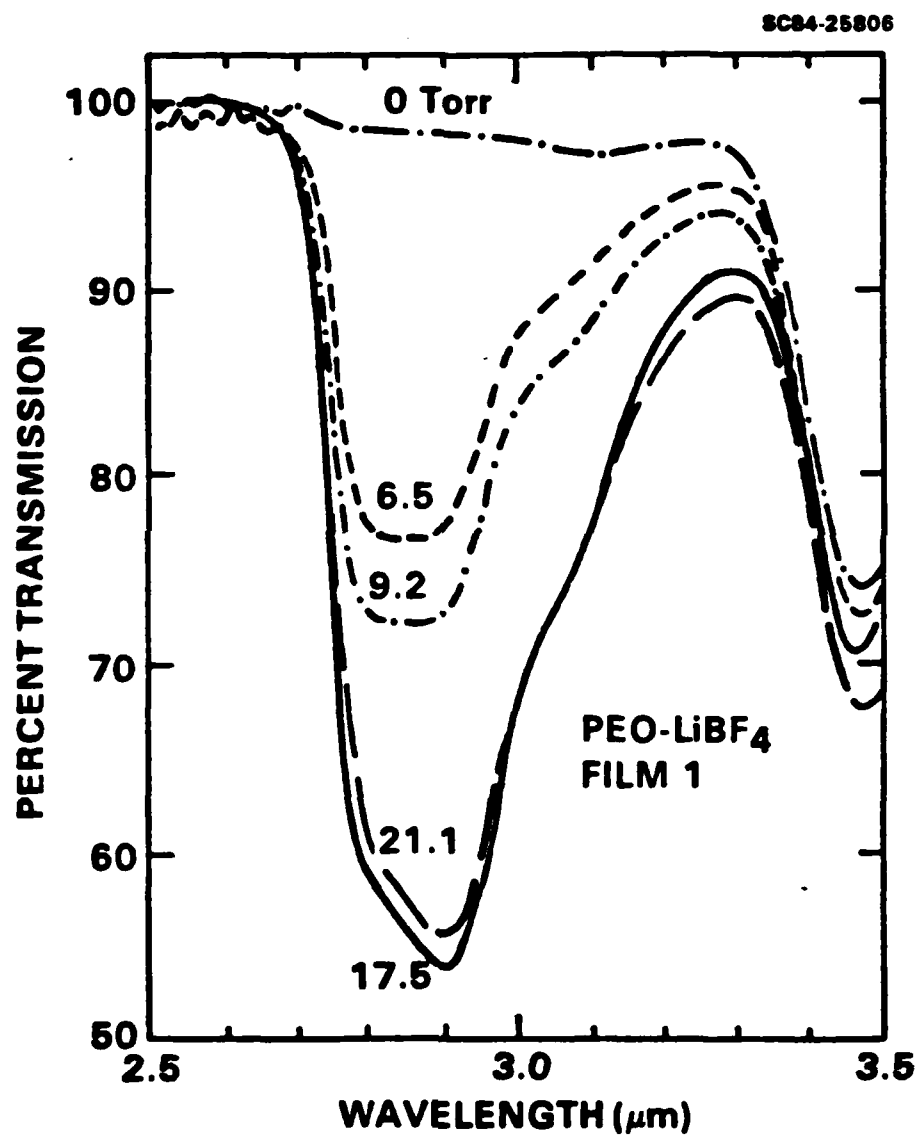
1. Experimental cell
2. Infrared spectra of $\text{PEO-LiBF}_4\text{-H}_2\text{O}$ film recorded 30 min after changes of ambient water vapor pressure
3. Dependence of measured conductance on IR absorbance due to water in Film 1
4. Dependence of measured conductance on IR absorbance due to water in Film 2. Inset shows the extrapolation in detail.
5. Time dependence of water absorbance in $\text{PEO-LiBF}_4\text{-H}_2\text{O}$ film recorded continuously under water pressure of 18.7 torr after different pre-drying times. All curves start at zero absorbance.
6. Time dependence of conductance of $\text{PEO-LiBF}_4\text{-H}_2\text{O}$ film under conditions of Fig. 5. All curves start at zero conductance on this scale.
7. Infrared spectra of $\text{PEO-H}_2\text{O}$ film recorded at different times under water pressure of 18.7 torr



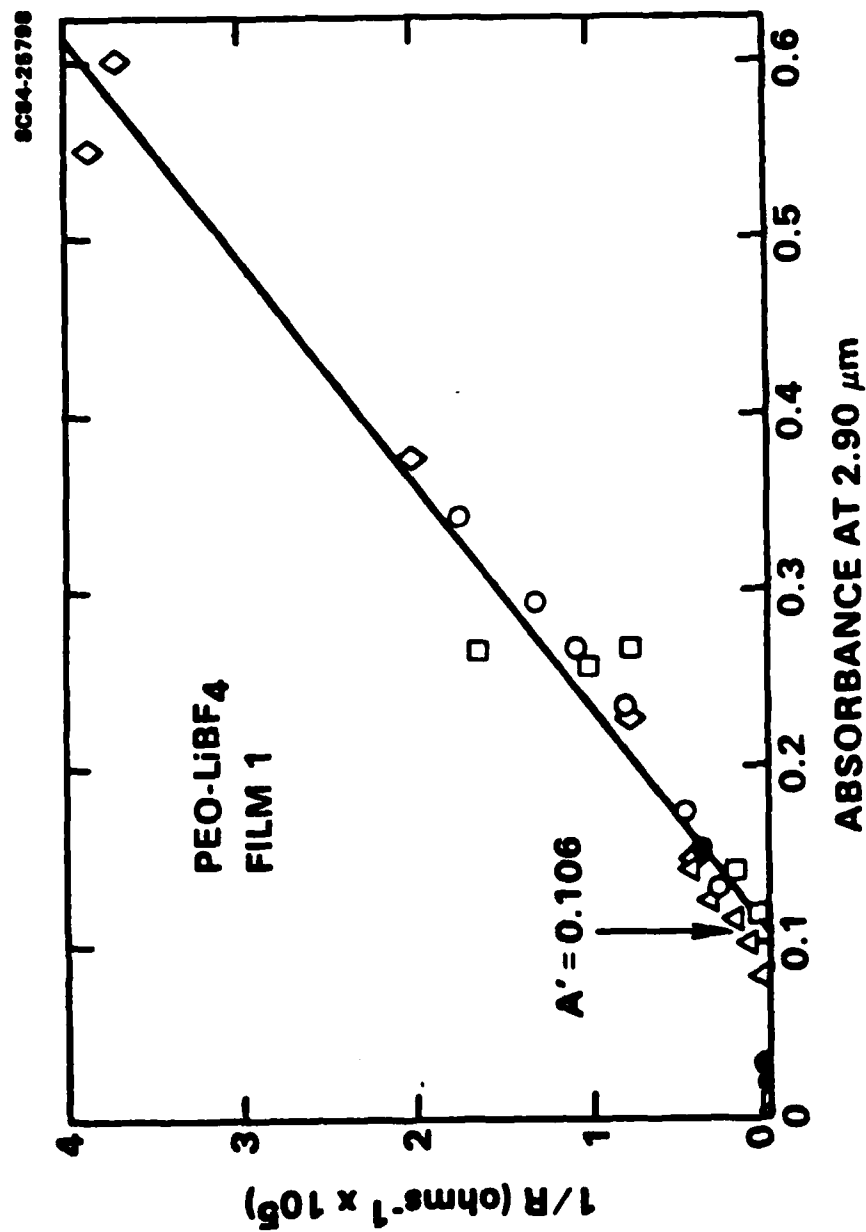
8. Time dependence of water absorbance in PEO-H₂O film recorded continuously under water pressure of 18.7 torr after pre-drying 24 hr
9. Conductivity of swollen PEO-LiBF₄-H₂O film as a function of the mole ratio of H₂O to LiBF₄. Points are smoothed data from Fig. 4.
10. Conductivity based on incremental thickness equivalent to free water in PEO-LiBF₄-H₂O films as function of free-water absorbance. Points are smoothed data from Fig. 3 and 4.



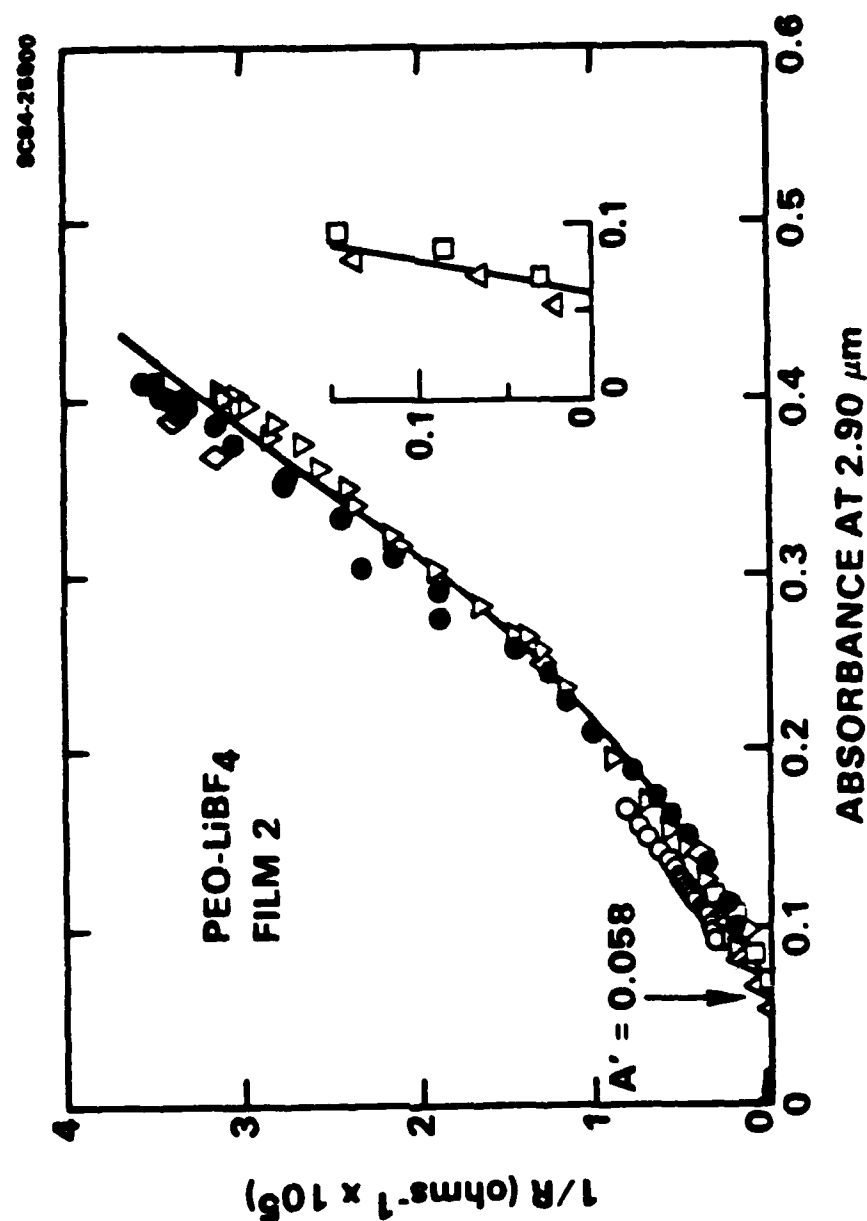
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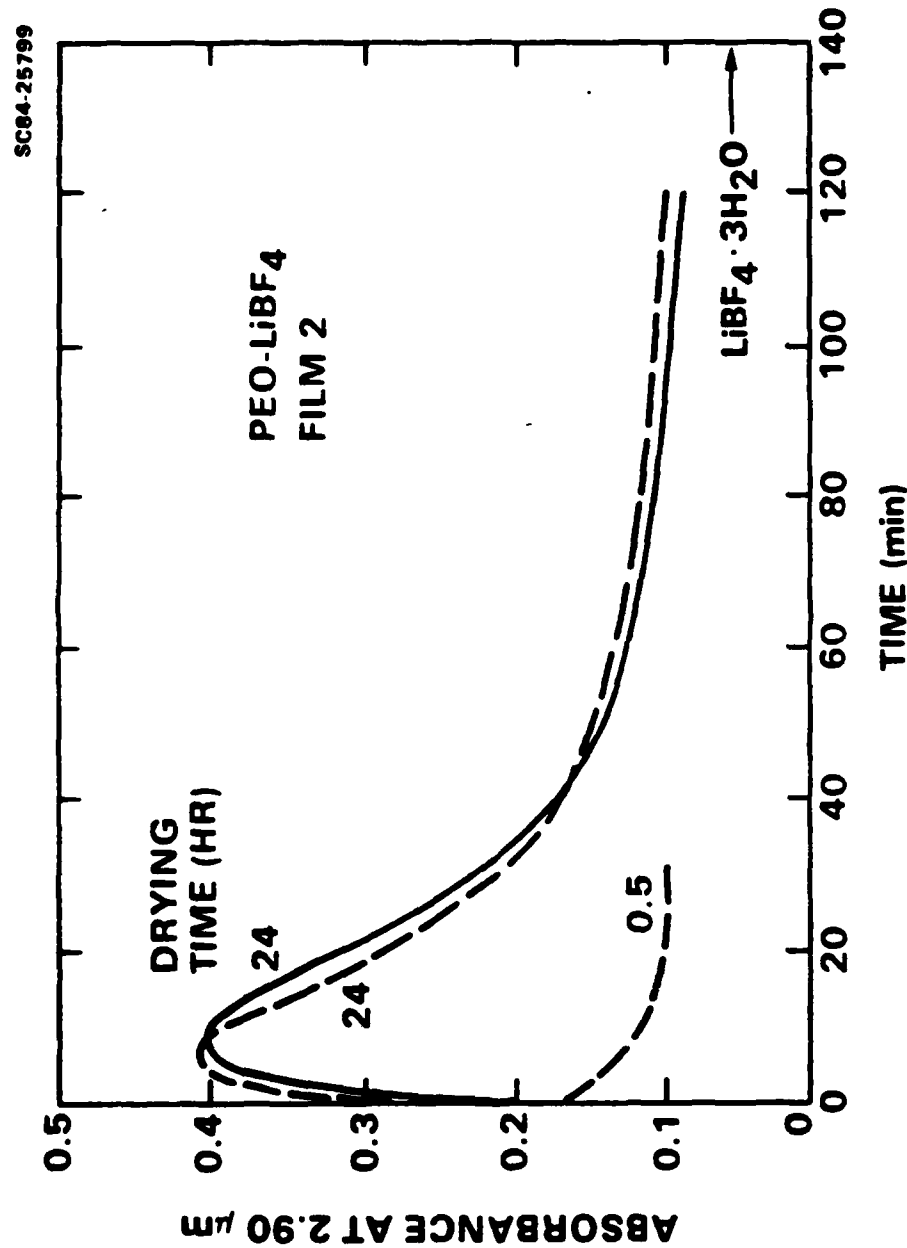
2. Infrared spectra of PEO-LiBF₄-H₂O film recorded 30 min after changes of ambient water vapor pressure



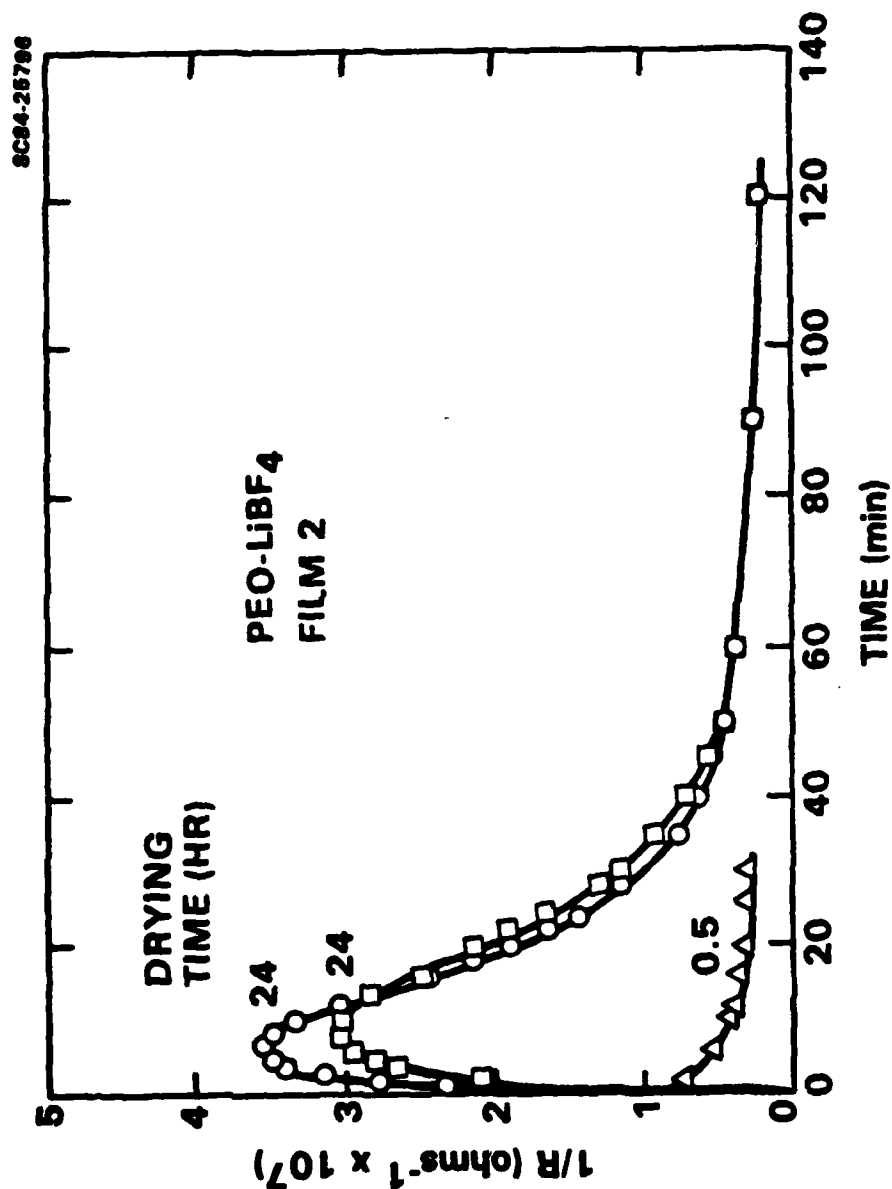
3. Dependence of measured conductance on IR absorbance due to water
in Film 1



4. Dependence of measured conductance on IR absorbance due to water in Film 2. Inset shows the extrapolation in detail.



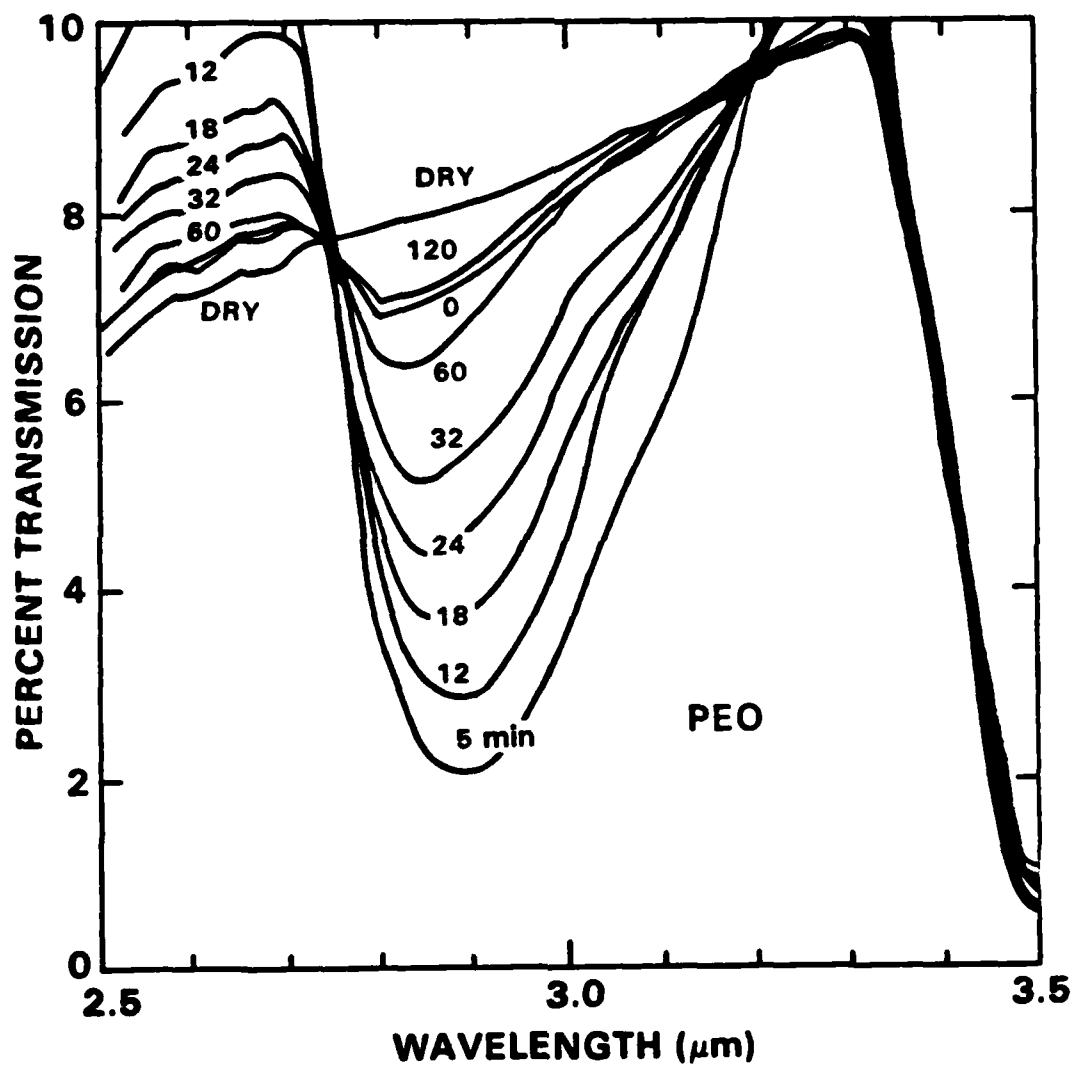
5. Time dependence of water absorbance in PEO-LiBF₄-H₂O film recorded continuously under water pressure of 18.7 torr after different pre-drying times. All curves start at zero absorbance.



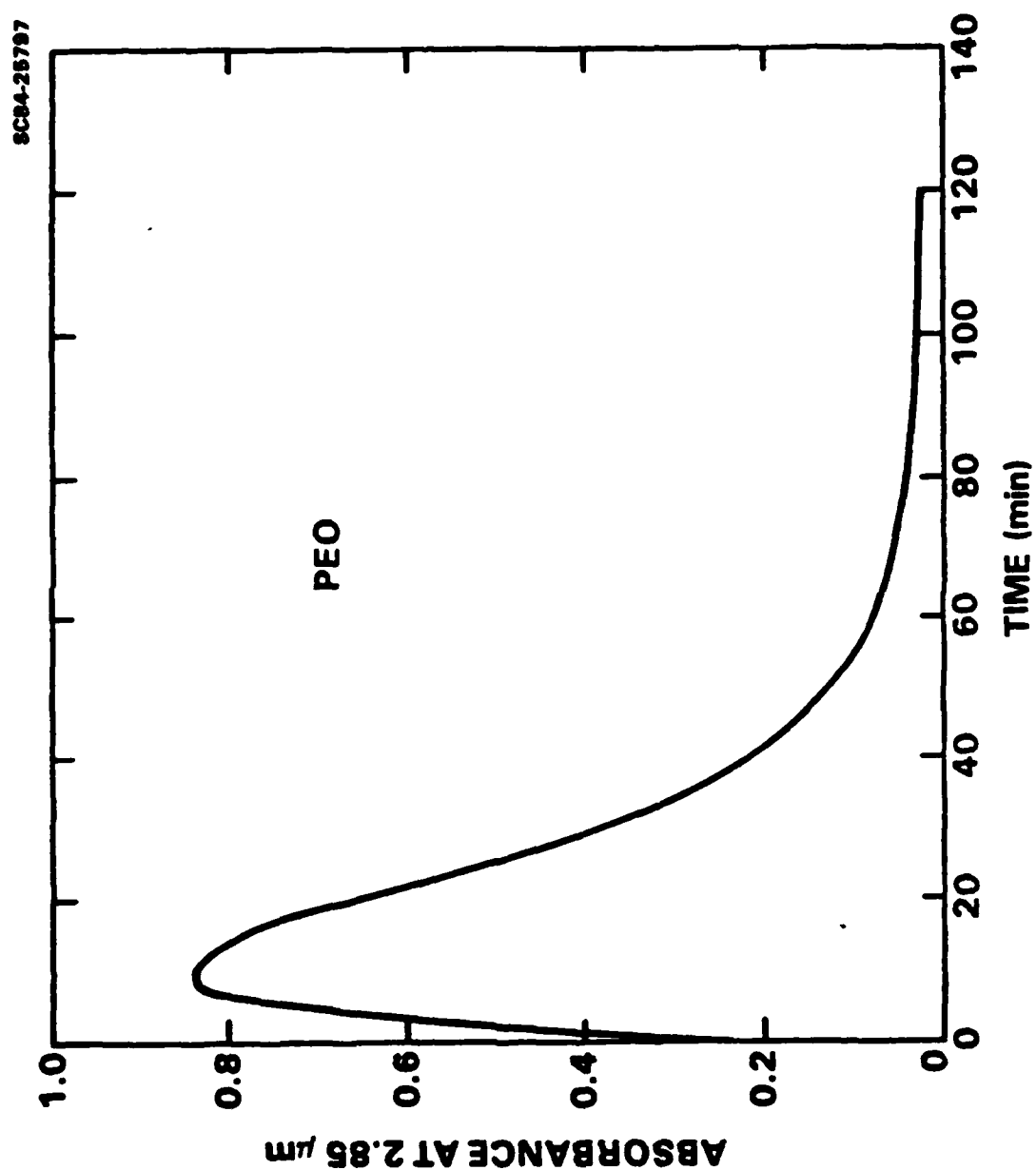
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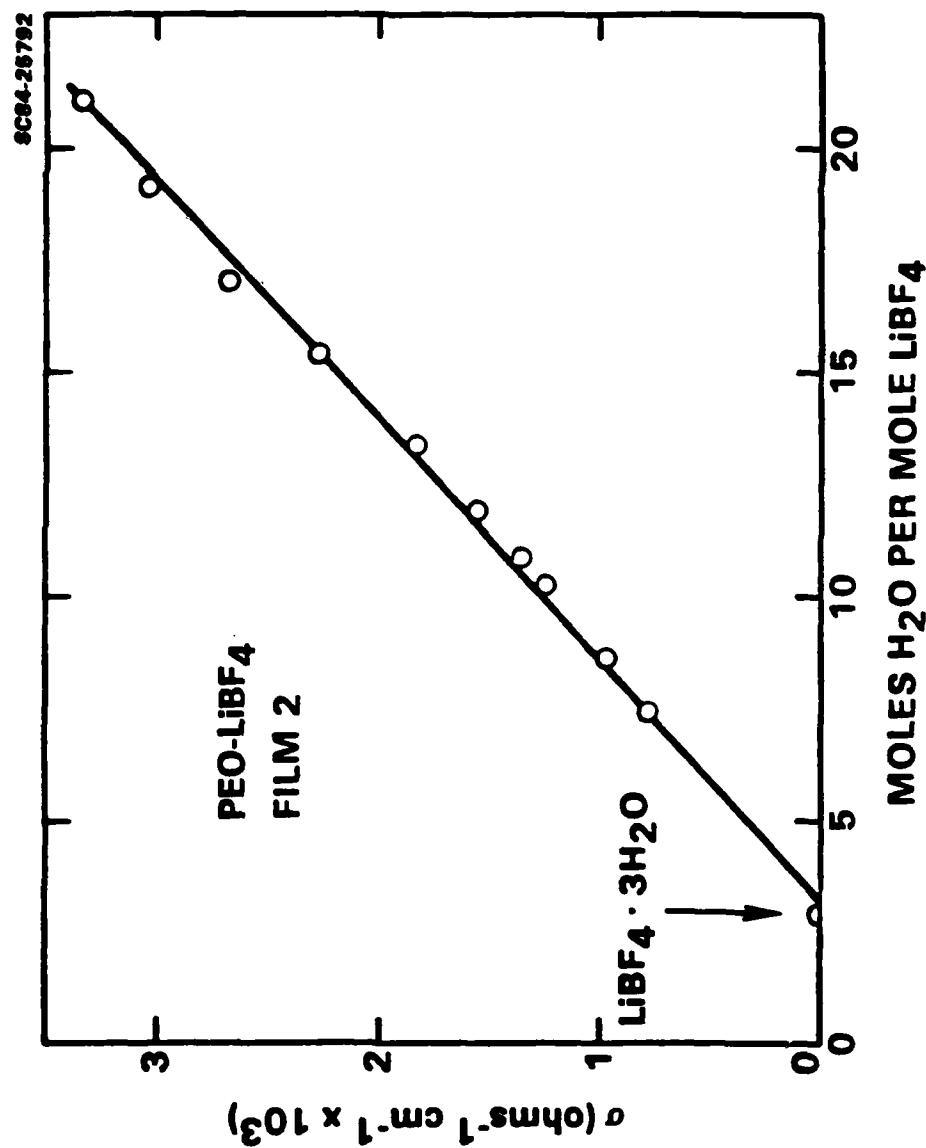
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7. Infrared spectra of PEO-H₂O film recorded at different times
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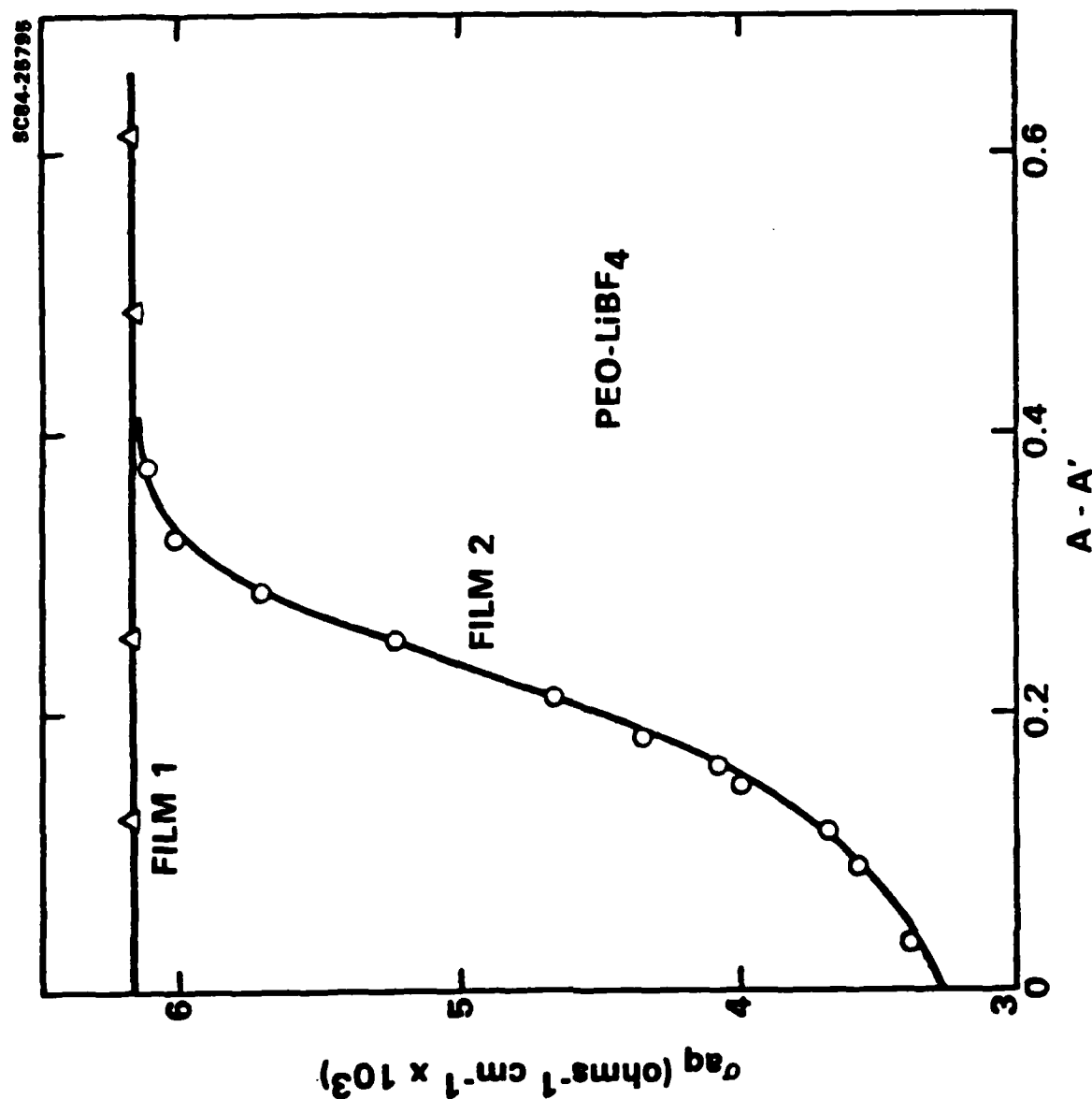


8. Time dependence of water absorbance in PEO-H₂O film recorded continuously under water pressure of 18.7 torr after pre-drying 24 hr



9. Conductivity of swollen PEO-LiBF₄-H₂O film as a function of the mole ratio of H₂O to LiBF₄. Points are smoothed data from

Fig. 4.



10. Conductivity based on incremental thickness equivalent to free water in PEO-LiBF₄-H₂O films as function of free-water absorbance. Points are smoothed data from Fig. 3 and 4.

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