

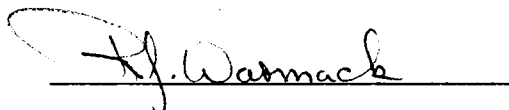
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PHOTOEMISSION OF n-ETHYLENE GLYCOLS
IN THE VACUUM ULTRAVIOLET

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

Lili Jiang
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Especially I would like to thank Dr. R. J. Warmack for his great help on research and computer programs.

ABSTRACT

Measurements of the absolute photoemission of liquids in the vacuum ultraviolet (UV) have been taken. Studies have focused on photoelectric yields of diethylene glycol, triethylene glycol, and tetraethylene glycol undoped and doped with lithium hydroxide solution (LiOH). The electron mean free paths calculated from experimental photoyields are submitted. Photon fluxes are determined by filling the ion chambers with argon gas. The absolute photoyield curves generally show monotonic behavior with the values in the neighborhood of 15% to 20%. These absolute photoyields are higher than expected. The data of our experiments indicate that the effects of doping with alkali salts on these n-ethylene glycols are small.

Two papers (Nemec, 1975; Casanovas, 1981) on photoemission into vacuum give the photoelectric threshold energy to be about 8.0 eV for 11 organic liquids. Our experimental data give other values. The relation between the photoyields and the electron mean free paths is broken down in the photon energy region 16 eV to 30 eV when the threshold energy is 8 eV. It is suggested therefore that the photoelectric threshold energy be less than 8 eV and larger than 7 eV.

Our electron mean free paths were compared with the empirical equation predicted from Seah and Dench (1978) for organics. The electron mean free paths calculated from our experimental data exceeded those predicted by Seah and Dench. Therefore the empirical

equation is not suitable for these n-ethylene glycols in the photon energy region 16 eV to 30 eV at low pressure.

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CHAPTER I

INTRODUCTION

Research concerning the phenomenon of photoemission from solids, liquids, and gases has been pursued since the explanation of photoemission in 1905. Most studies have focused on solid surfaces. Detailed theories have been constructed using quantum mechanics, band theories, plasma theory, and scattering theories. Many thousands of papers, both experimental and theoretical, have been devoted to photoemission, and their results have been extremely useful in revealing details of the electronic structure for many metals, semiconductors, and insulators. Although solids have been well characterized through numerous photoemission studies, the study of photoemission from liquid surfaces has much room for improvement. Research on photoemission from liquid surfaces has intrigued many scientists particularly because of the biological significance of liquids. These researches reveal details of the process of photoemission and consequently of the structure of liquids.

There are several reasons for studying the phenomenon of photoemission from liquid surfaces. The studies of optical and photoemission properties of liquids are important in areas such as photochemistry and photobiology. The chemical industry also benefits from such studies. In photochemistry and photobiology, many studies relating to solar UV radiation show effects arising from photochemical

changes in materials. In photobiology, studies of the interactions of radiation with molecular liquids have direct biological applications in understanding radiation damage in living material, radiation therapy, radiation dosimetry, and related problems in cell biology. Also, the studies give information on adhesive interactions between biological cells and between cells and artificial materials. In general, the interaction of electromagnetic radiation with matter can be described by the optical properties of the medium, and photo-emission data aids in determining optical properties.

The theory of the optical and dielectric properties of liquids is difficult because of the nature of the liquid state of matter. Useful theories have been formulated in the fields of solid state and molecular physics to describe the electronic structure of solids, which in many cases have long range order, and in gases, which are completely disordered. Because liquids have only short range order and no rigid structure, it is difficult to specify the interaction of the molecules in order to describe the liquid state. In the next section, we introduce the theory of photoemission on solid surfaces so that we can explain and use a similar method to study the photo-emission from a liquid surface. Application to the band structure and liquids has been made by Siegbahn (1974), Nemec (1975), and Nason (1976).

Studies of the photoelectric yield and the total electron mean free paths in liquids are the topics of this thesis. Experiments on optical and photoionization phenomenon were carried out. Studies have

focused on optical measurements of the photoemission from diethylene glycol, triethylene glycol, and tetraethylene glycol. The polyethylene glycols are of considerable use in biology for the incubation of cells. Details of the optical properties and calculation of photoelectric yields in the UV range are given in this thesis. Studies were completed on absolute photoyields of diethylene glycol, triethylene glycol, and tetraethylene glycol which were doped with an alkali solution. The absolute photoyield curves generally show monotonic behavior with values in the neighborhood of 15% to 20%. The associated total mean free paths exceed those predicted from Seah and Dench (1978). Comparison of electron mean free paths are extended with the empirical equation for dielectrics provided by Seah and Dench (1978). These investigations show that there are inference problems in using the empirical equation in the low energy region from 16 eV to 30 eV and the resulting electron mean free paths often differ by more than an order of magnitude.

Photoemission and electron mean free paths in liquid formamide on the vacuum UV were measured by Birkhoff, Painter, and Ashley (1982). The yields of photoelectrons for liquid formamide (HCONH) were obtained. These yield curves are similar to each other in the energy range 16 eV to 25 eV. They are nearly constant between 16 eV and 18 eV at 13.5% and decline toward higher energies.

Surface charging and ionic doping of liquids provide sufficiently high conductivity for the positive charge to be moved. When the photon beam strikes the surface, producing the photocurrent,

a net positive charge can remain on the liquid surface if the charge is not carried rapidly enough to the walls of the container. This thesis presents new measurements of dc conductivity and reports that addition of a 0.1 to 0.001 molar alkali solution increases the conductivity by at least an order of magnitude. Thus, a primary purpose of this thesis is to report the effects of impurities and surface charging on the apparent photoyields in liquids.

CHAPTER II

THEORY

Photoemission from solids can be modeled as a two-step process (Berglund, 1964). Electrons are first optically excited into states of higher energy; then they move to the surface of the solid, with or without scattering, and escape into vacuum. Studies of the spectral distribution of the photoelectric yield and studies of the energy distribution of photoemitted electrons can be used to explain both the optical excitation process and the electron-scattering process in solids. All points are simply considered in order to systematically describe all the problems. Thus the main purpose of these expressions is to explain the effects of electrons which escape after one or more inelastic-scattering events or escape without significant inelastic-scattering in photoemission. Subsequent to this model, complicating factors are introduced. These include the effects of electron-electron scattering, of electron scattering by plasmon creation, and of the Auger process. Photoemission at photon energies well above the threshold is emphasized.

I. Probability of Electron Escape without Inelastic Scattering

Consider an electron excited to some energy E and momentum p at a distance x from the surface of a semi-infinite solid as shown in

Figure 1. This electron has just been excited to this state by absorption of a photon. In order for the electron to escape from the solid without any loss of total energy, it must (1) reach the surface of the solid without suffering an inelastic collision, and (2) have a crystal momentum component perpendicular to the photoemitting surface greater than some critical value.

In order to simplify the theory, several assumptions are used below. These assumptions cannot be expected to be exact; however, they simplify the problems and can make it easier to understand. Such treatments as given here must be considered as only a first approximation. The assumptions to be made are:

1. the distribution in direction of the excited electrons is isotropic (actually the direction is determined by the direction of the incident electric field);
2. only inelastic-scattering events need be considered;
3. the probability of inelastic-scattering can be described in terms of a mean free path l , which is a function only of the electron energy;
4. the inelastic-scattering is isotropic; and
5. in order to escape over the surface barrier, the electron must have a component of its total crystal momentum p perpendicular to the surface which is greater than some critical value p_c .

The escape probability is assumed to be unity if condition (5) is satisfied. As will be seen in the equation derived, p and p_c

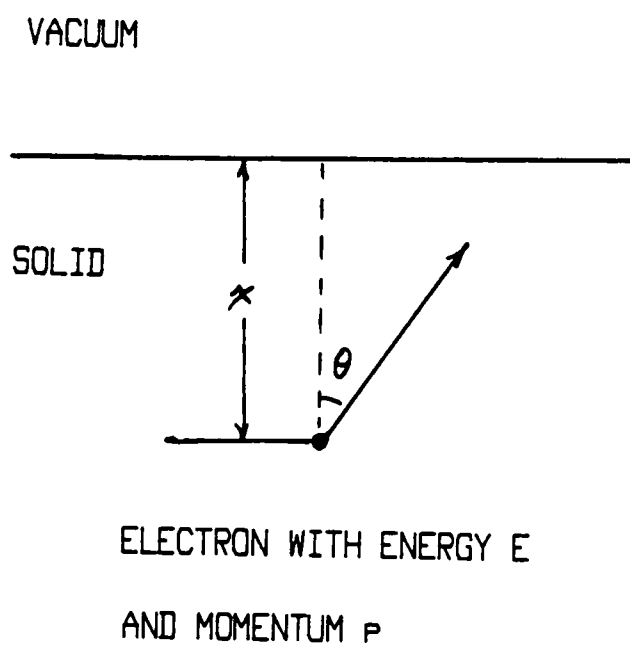


Figure 1. Diagram for symbols relating to an escaping photoelectron.

appear only in terms of the form $(1 - p_c/p)$. This can be calculated from the assumed band structure.

If θ is the angle between the direction of electron momentum upon excitation and the normal to the photoemitting surface as shown in Figure 1, the electron must move a distance $(x/\cos\theta)$ to reach the surface. The probability of the electron escaping without losing energy following excitation to energy E is given by Berglund (1964):

$$P_e(E, x) = \frac{1}{2} \int_0^{\theta_0} \sin\theta d\theta e^{-x/\lambda \cos\theta} \quad \text{if } p \geq p_c \quad (1a)$$

where

$$\theta_0 = \cos^{-1}(p_c/p) \text{ and}$$

$$P_e(E, x) = 0 \quad \text{if } p < p_c \quad (1b)$$

where λ is the mean free path for inelastic-scattering.

In optical absorption, the rate per unit area at which electrons are excited to energies between E and $E + dE$, in a slab of width dx located at distance x from the surface on which the light is incident, is $G(E, x)$. One may write

$$G(E, x)dE dx = G_0(E)dE e^{-\alpha(\nu)x} dx \quad (2)$$

where $\alpha(\nu)$ is the absorption coefficient at frequency ν defined as:

$$\alpha(\nu) = \int_0^\infty dE \alpha'_\nu(E)$$

and $G_0(E)$ is the amplitude of the excitation rate $G(E, x)$.

If all photons corresponding to frequency ν are absorbed in exciting electrons in the solid to higher energy states, then $\alpha'_\nu(E)dE$ is the part of $\alpha(\nu)$ due to electronic transition to energy states between E and $E + dE$. If n_p is the flux of photons at frequency ν per unit area absorbed by the photoemitting materials, the excitation rate in Eq. (2) due to optically excited electronic transitions satisfies

$$G(E,x)dE dx = n_p \alpha'_\nu(E) e^{-\alpha(\nu)x} dx .$$

$G_0(E)$ is thus defined to be $n_p \alpha'_\nu(E)$. Then the rate $r'(E)dE$ of escape of electrons with energy between E and $E + dE$ is:

$$r'(E)dE = G_0(E)dE \int_0^\infty dx P_e(E,x) e^{-\alpha(\nu)x} . \quad (3)$$

In Eq. (3), the photoemitting solid has been assumed to be semi-infinite to allow the upper limit in the integration to be infinite. Carrying out the integration in Eq. (3):

$$r'(E)dE = \frac{G_0(E)dE}{2\alpha} \left[1 - \frac{p_c}{p} - \frac{1}{\alpha\ell} \ln \left(\frac{1 + \alpha\ell}{1 + (p_c/p)\alpha\ell} \right) \right] \quad p \geq p_c \quad (4a)$$

and

$$r'(E)dE = 0 \quad p < p_c . \quad (4b)$$

The logarithm term is expanded as

$$\begin{aligned}
-\frac{1}{\alpha\ell} \ln \left(\frac{1 + \alpha\ell}{1 + (p_c/p)\alpha\ell} \right) &= \frac{1}{\alpha\ell} \ln \left(1 - \frac{[1 - (p_c/p)]\alpha\ell}{1 + \alpha\ell} \right) \\
&= -\frac{1}{\alpha\ell} \left[\frac{[1 - (p_c/p)]\alpha\ell}{(1 + \alpha\ell)} + \frac{[1 - (p_c/p)]^2 \alpha^2 \ell^2}{2(1 + \alpha\ell)^2} \right. \\
&\quad \left. + \frac{[1 - (p_c/p)]^3 \alpha^3 \ell^3}{3(1 + \alpha\ell)^3} + \dots \right] \quad (5)
\end{aligned}$$

Substituting Eq. (5) in Eq. (4) yields

$$\begin{aligned}
r'(E)dE &= \frac{\frac{1}{2} [1 - (p_c/p)] G_0(E) dE}{\alpha + (1/\ell)} \left\{ 1 - \frac{[1 - p_c/p]}{2(1 + \alpha\ell)} \right. \\
&\quad \left. - \frac{[1 - (p_c/p)]\alpha\ell}{3(1 + \alpha\ell)^2} + \dots \right\} \quad p \geq p_c \quad (6a)
\end{aligned}$$

and

$$r'(E)dE = 0 \quad p < p_c \quad (6b)$$

Equation (6) can be further simplified if a threshold function $C(E)$ is defined by

$$C(E) = \frac{1}{2} [1 - (p_c/p)] \quad p \geq p_c \quad (7a)$$

$$C(E) = 0 \quad p < p_c \quad (7b)$$

and a correction factor K is defined as

$$K = \left[1 - \frac{C(E)}{1 + \alpha \ell} - \frac{2}{3} \frac{C(E)^2}{(1 + \alpha \ell)^2} + \dots \right] .$$

K can be evaluated and plotted as a function of C(E) and as in Figure 2. Thus,

$$r'(E)dE = \frac{KC(E)G_0(E)dE}{\alpha + 1/\ell} \quad (8)$$

II. Probability of Electron Escape after One Inelastic Scattering Event

The probability of an electron escaping after being scattered once can be derived in a way similar to that for an electron escaping without scattering. Hence.

$$r''(E)dE = \frac{KC(E)dE}{\alpha + 1/\ell'} \int_E^{\infty} dE' P_2 G_0(E') \quad (9)$$

where ℓ' is the mean free path for inelastic scattering for an electron with energy E' and P_2 is the probability of the electron being scattered to an energy between E and $E + dE$. The probability P_2 will depend on the scattering mechanism. Combining Eq. (6) with Eq. (9), the rate of escape of electrons with energy between E and $E + dE$ can be found. Considering only electrons which do not scatter and those which scatter once, one finds

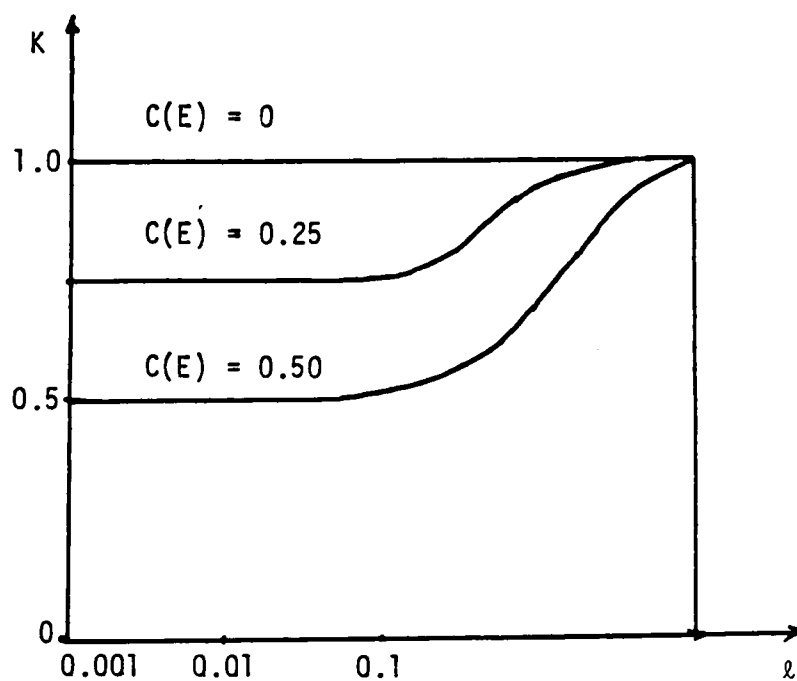


Figure 2. A graph of the correction factor K versus the mean free path l .

$$r(E)dE = \frac{KC(E)dE}{\alpha + (1/\ell)} \left[G_0(E) + \int_E^\infty dE' \frac{1}{2} \left(\frac{1}{\alpha\ell'} \ln(1 + \alpha\ell') \right. \right. \\ \left. \left. + \frac{\ell}{\ell'} \ln(1 + \frac{\ell'}{\ell}) \right) P_2 G_0(E') \right] \quad (10)$$

III. Effect of Elastic Scattering

The probability of escape of an electron with energy E a distance x from the surface of a photoemitter can be approximately written as:

$$P_e(E, x) = B(E)e^{-x/L}$$

where $B(E)$ is a function which takes into account the threshold energy and L is an attenuation length which depends on the mean free paths for inelastic and elastic collisions. Its calculation is similar to those resulting in Eq. (6). One has

$$r(E)dE = \frac{B(E)G_0(E)dE}{\alpha + (1/L)} \quad (11)$$

The number of electrons emitted with energy between E and $E + dE$ per absorbed photon of frequency ν is

$$N(E)dE = \frac{r(E)dE}{n_p} \\ = \frac{KC(E)dE}{\alpha + (1/\ell)} \left[\alpha\nu'(E) + \int_E^\infty dE' \frac{1}{2} \left(\frac{1}{\alpha\ell'} \ln(1 + \alpha\ell') \right. \right. \\ \left. \left. + \frac{\ell}{\ell'} \ln(1 + \frac{\ell'}{\ell}) \right) P_2 \alpha\nu'(E') \right] \quad (12)$$

The quantum yield is defined as the total number of electrons which escape from a photoemitter per absorbed photon at a given photon energy. Hence,

$$Y(\nu) = \int_{E_W}^{\infty} dE N(E) \quad (13)$$

where E_W is the energy corresponding to the Fermi energy plus the work function, i.e., the energy at the vacuum level. In general, the quantum yield per incident photon $Y'(\nu)$ is the quantity experimentally measured for a material. This is related to the yield per absorbed photon through the reflectivity $Re(\nu)$:

$$Y(\nu) = \frac{Y'(\nu)}{1 - Re(\nu)} . \quad (14)$$

IV. Photoemission from Liquids

Most papers have been devoted to studying the phenomenon of photoemission from solid surfaces rather than liquids. An independent-particle model, which includes many-body aspects in a phenomenological way by using an inelastic mean free path, is used to describe this process (Berglund, 1964). Recently similar methods have been used to study photoemission processes in insulating liquids (Pepper, 1970; Birkhoff, 1982). Our application of the theory is devoted to studying the relative yield of photoelectrons as a function of photon energy. Several quantum mechanical treatments (Birkhoff, 1982; Levinson, 1979 and 1981) of the photoemission process and reflectance are applied to

insulating liquids. The yield is understood to be the yield per incident photon. The photon energy is $\hbar\omega$.

In our experiment, the photon beam is incident at 15° from the normal to the liquid surface. Part of the photon beam is reflected, and part may produce photoelectrons. We are interested in the physical information derivable from absolute yield measurements. In order to simplify the problem, we assume a simple three-step process. First we assume a normally incident photon beam. Then surface plasmons are excited and decay by releasing electrons. This gives a maximum photoemission yield near the surface plasmon energy. Finally, some of these photoelectrons are scattered through inelastic processes such as excitation of volume plasmons which may act to lower the photoyield. If the electrons which arrive at the surface have enough energy to surmount the surface potential barrier, they will escape. This method was developed by Birkhoff (1982).

The photoemitted flux depends on the photon flux at x , the probability of generating a photoelectron in dx , and the probability that the photoelectron will find its way back to the surface and escape. The fraction, N , of photons which penetrate the surface are exponentially attenuated with a photon mean free path L_p :

$$N = (1 - R)e^{-x/L_p} \quad (15)$$

where x is the distance below the surface and R is the reflection coefficient.

We will assume that the probability of producing a photoelectron is equal to (dx/L_p) by neglecting photon attenuation processes which do not give rise to ionization. The probability that the electrons emitted isotropically will be headed toward the surface must take into account a relative differential solid angle:

$$\frac{d\Omega}{4\pi} = \frac{2\pi \sin\theta d\theta}{4\pi} . \quad (16)$$

The probability that they will survive an exponential attenuation on their way to the surface is equal to

$$e^{-x/L_e \cos\theta} \quad (17)$$

where L_e is the mean free path of an electron for inelastic interaction.

To penetrate through the surface and be detected externally, an electron must have a component of momentum along the normal. This component corresponds to an energy greater than that required to surmount the potential barrier into the vacuum. According to the classical barrier model, electrons will escape if they move within a cone of a solid angle:

$$\theta_c = \cos^{-1}(\phi/\hbar\omega) \quad (18)$$

where ϕ is the photoelectric threshold energy. The photoyield per incident photon is therefore

$$Y = \frac{1}{2} (1 - R) \int_0^{\infty} \frac{dx}{L_p} e^{-x/L_p} \int_0^{\theta_c} \sin\theta d\theta e^{-x/L_e \cos\theta} . \quad (19)$$

After integrating, the yield per incident photon is

$$Y = \frac{1}{2} (1 - R) \left[1 - \cos\theta_c - \frac{L_p}{L_e} \ln \left(\frac{1 + L_e/L_p}{1 + (L_e/L_p) \cos\theta_c} \right) \right] . \quad (20)$$

To use Eq. (20) to predict photoyields, we need k , L_e , and L_p as additional input information.

The photon mean free path L_p is equal to $(\lambda/4\pi k)$ where λ is the wavelength and k is the imaginary part of the refractive index. The k is related to the damping of the electric and magnetic field intensities of the wave by the medium. For an absorbing media, k is greater than zero. The real part of the index of refraction is n . This is equal to c/v where c and v are the velocities of phase propagation in vacuum and in the medium, respectively. The complex dielectric function is ϵ . It has its real part ϵ_1 related to the polarizability of the medium in the presence of an electromagnetic wave and its imaginary part ϵ_2 is related to the absorption of electromagnetic energy by the medium. The real and imaginary parts of the dielectric function and the index of refraction are connected by

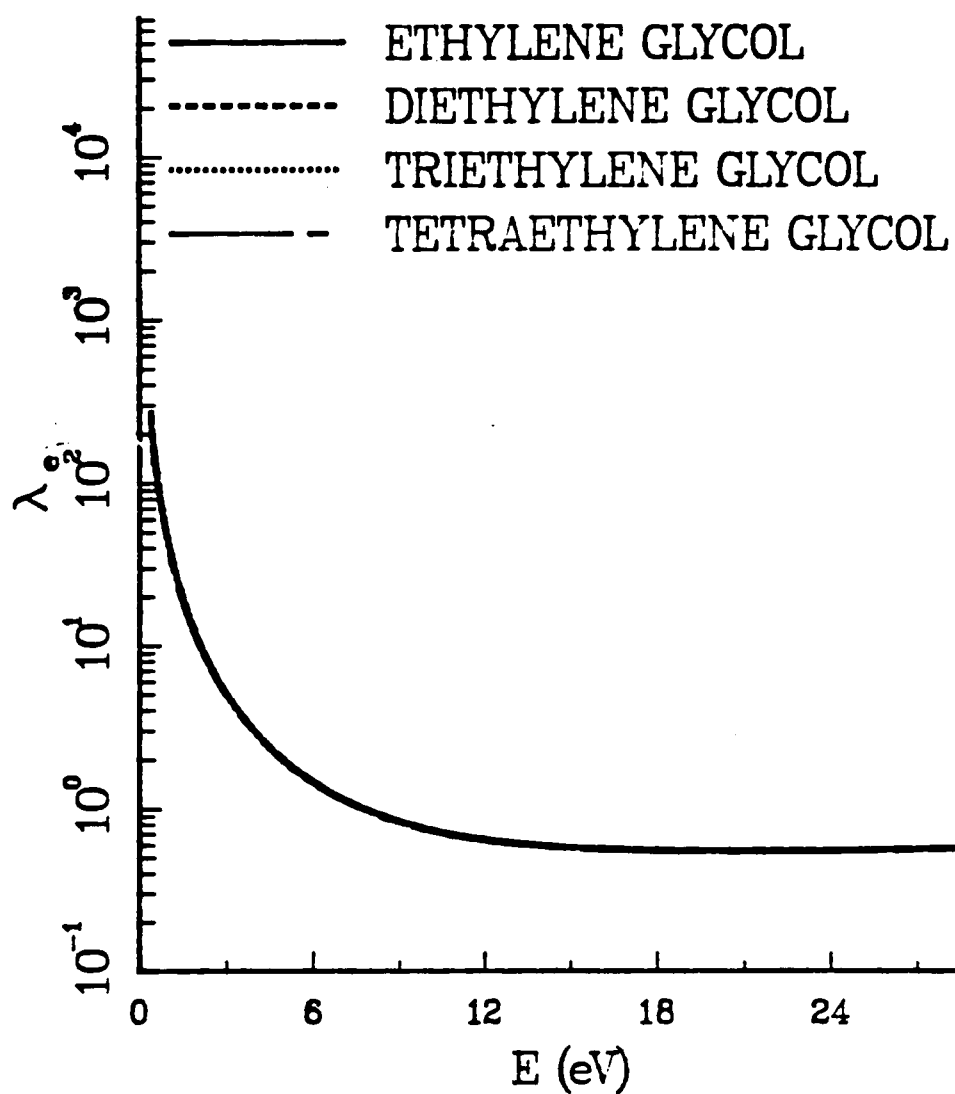
$$\epsilon_1 = n^2 - k^2 . \quad (21)$$

and

$$\epsilon_2 = 2nk \quad (22)$$

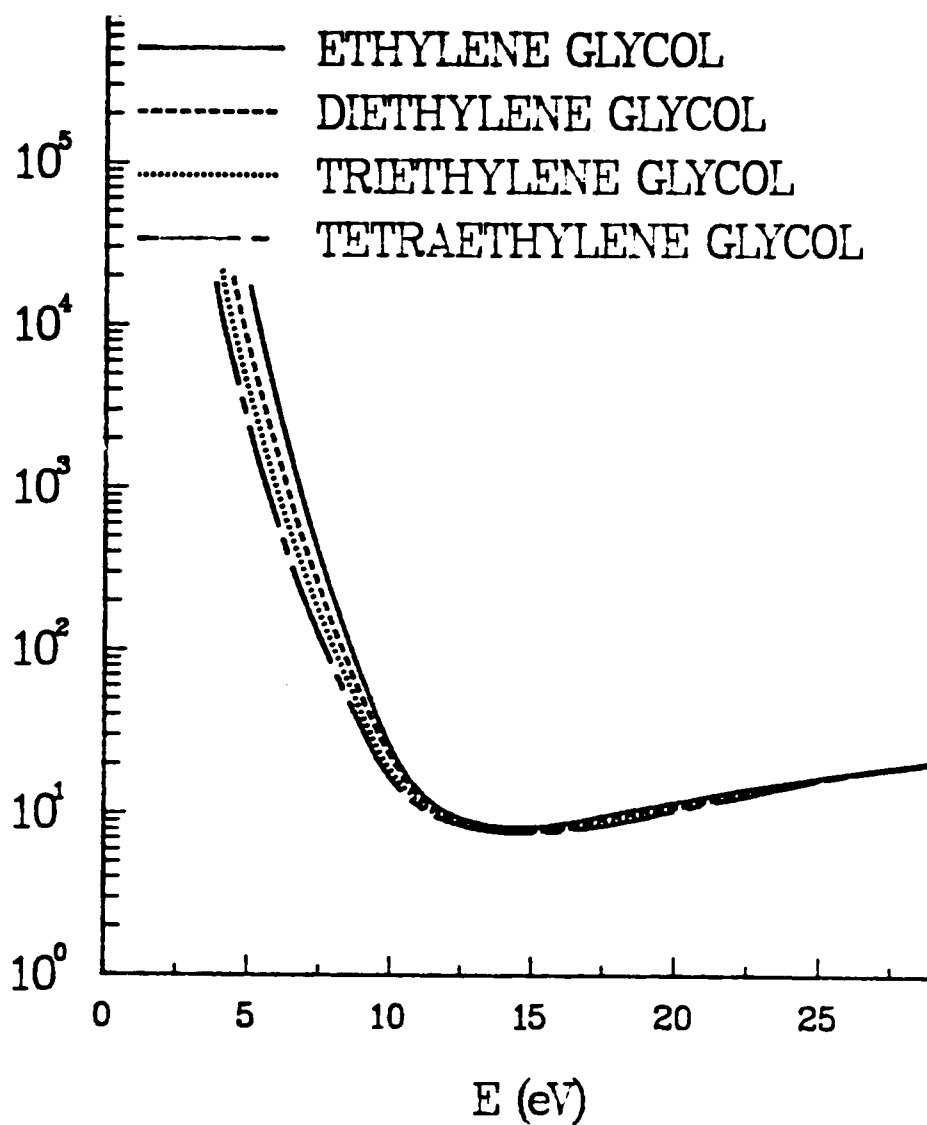
These are functions of frequency.

Figure 3 shows results of experimental measurements and calculations by Karickhoff, Warmack, and Painter (private communication). The curves of the photon mean free path, L_p , and the reflectance, R , are needed in the calculations of electron mean free path determined from the photoyield Y as shown in Section IV. These calculations will be compared to Figure 3a which gives the results of the empirical formula of Seah and Dench (1978).



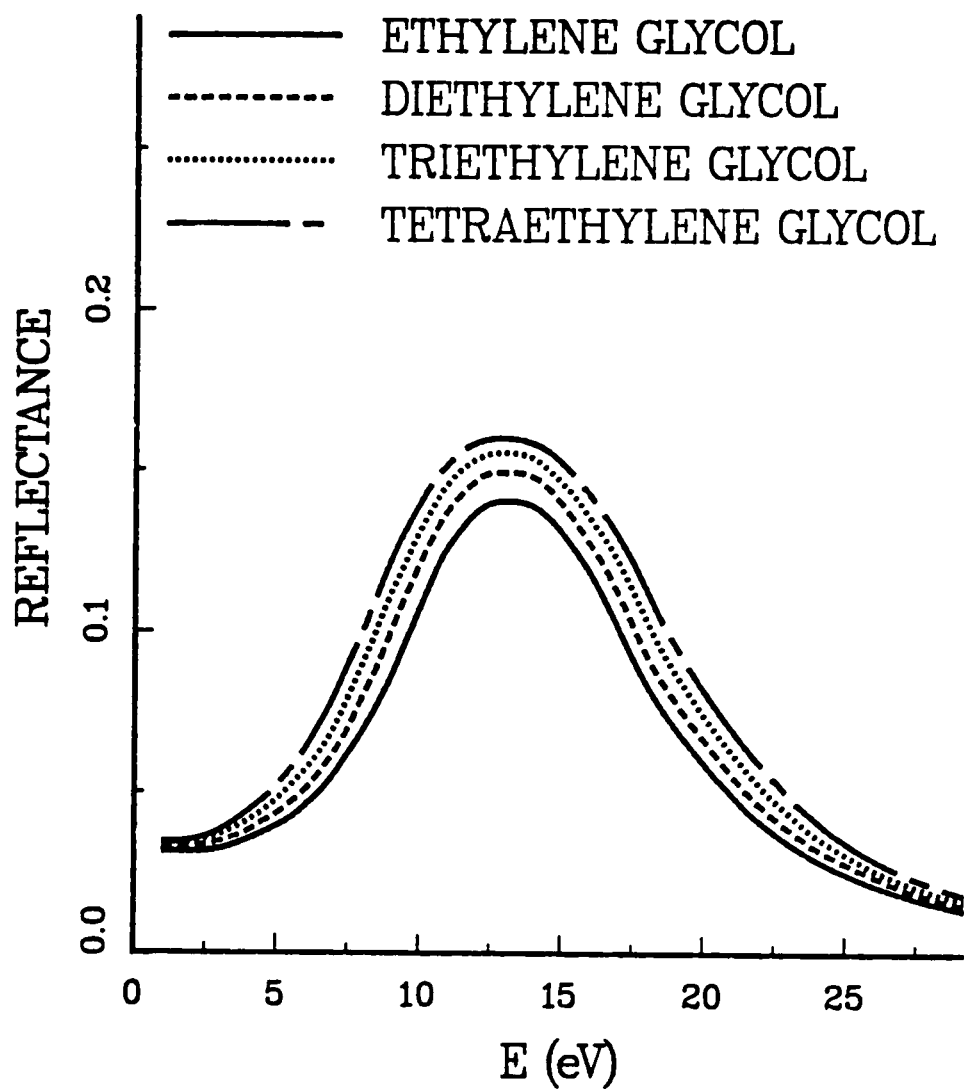
(a) Electron mean free path for n-ethylene glycols determined from the empirical equation of Seah and Dench.

Figure 3. Mean free path and reflectance data used for calculation and comparison.



(b) Photon mean free path experimentally determined from k .

Figure 3 (continued)



(c) Reflectance experimentally determined.

Figure 3 (continued)

CHAPTER III

EXPERIMENT

The photoemission chamber and monochromator are shown in Figure 4. The monochromator housing, ultraviolet source, hoist, differential pumping port, and photoemission chamber are located at (1), (2), (3), (4), and (5) as respectively designated. The photoemission chamber was made by two concentric cylinders: an inner stainless steel cylinder and an outer aluminum cylinder. The photon beam is transmitted from the monochromator to the photoemission chamber and the angle of the incident photon beam to the horizontal surface of a liquid sample is kept at 15° .

All experiments were conducted under low pressure in order to prevent unwanted absorption of the photon beam. Forepumps and oil-diffusion pumps were used to produce a vacuum base pressure of less than 10^{-5} torr. Figure 5 shows the schematic diagram of the vacuum system. The vacuum was obtained by systematically pumping the air out of volumes designated in Figure 5 as #1, #2, #3, and #4 by using oil-diffusion pump A, air-cooling oil-diffusion pump B, oil-diffusion pump C, and forepump D, respectively. The lamp is a high voltage low pressure spark discharge type. Argon was used as the filling gas in the ion chambers in order to assess the strength of the incident and reflected photon beams.

Figure 6 is a schematic diagram showing the measurement part of the experiment. The potentials on the walls of the ion chambers are

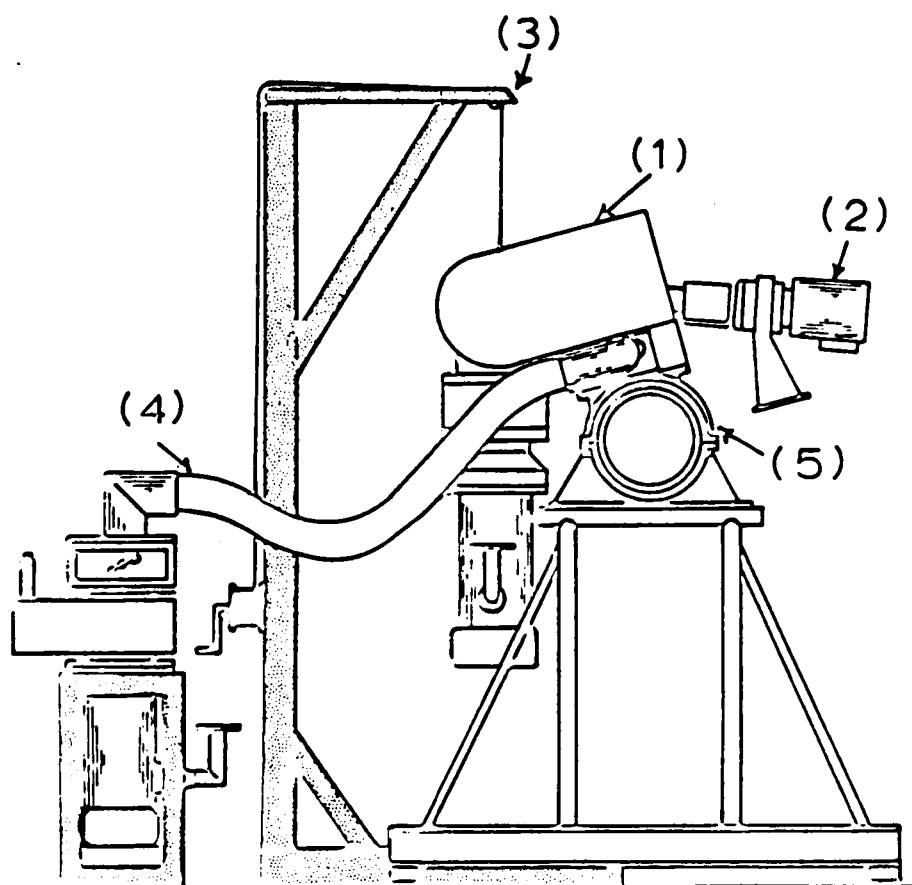


Figure 4. (1) Monochromator housing; (2) ultraviolet source; (3) hoist; (4) differential pumping port; (5) photo-emission chamber.

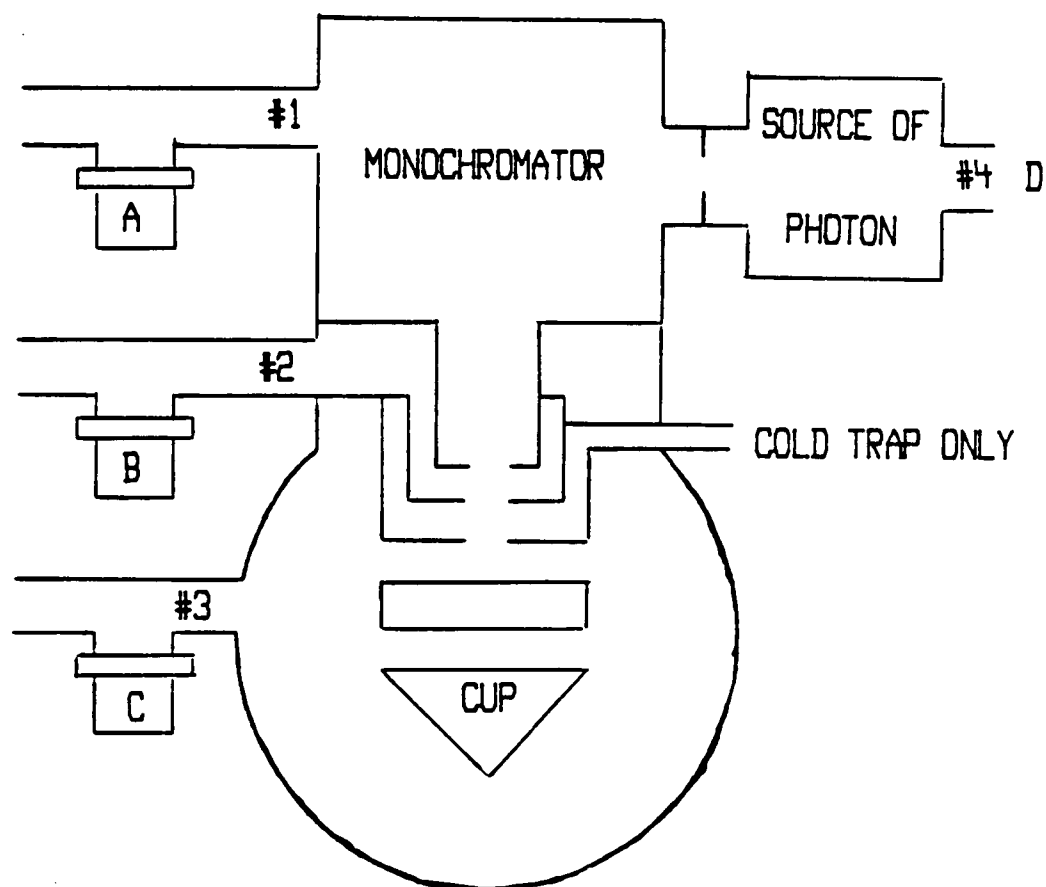


Figure 5. A schematic diagram of the vacuum system.

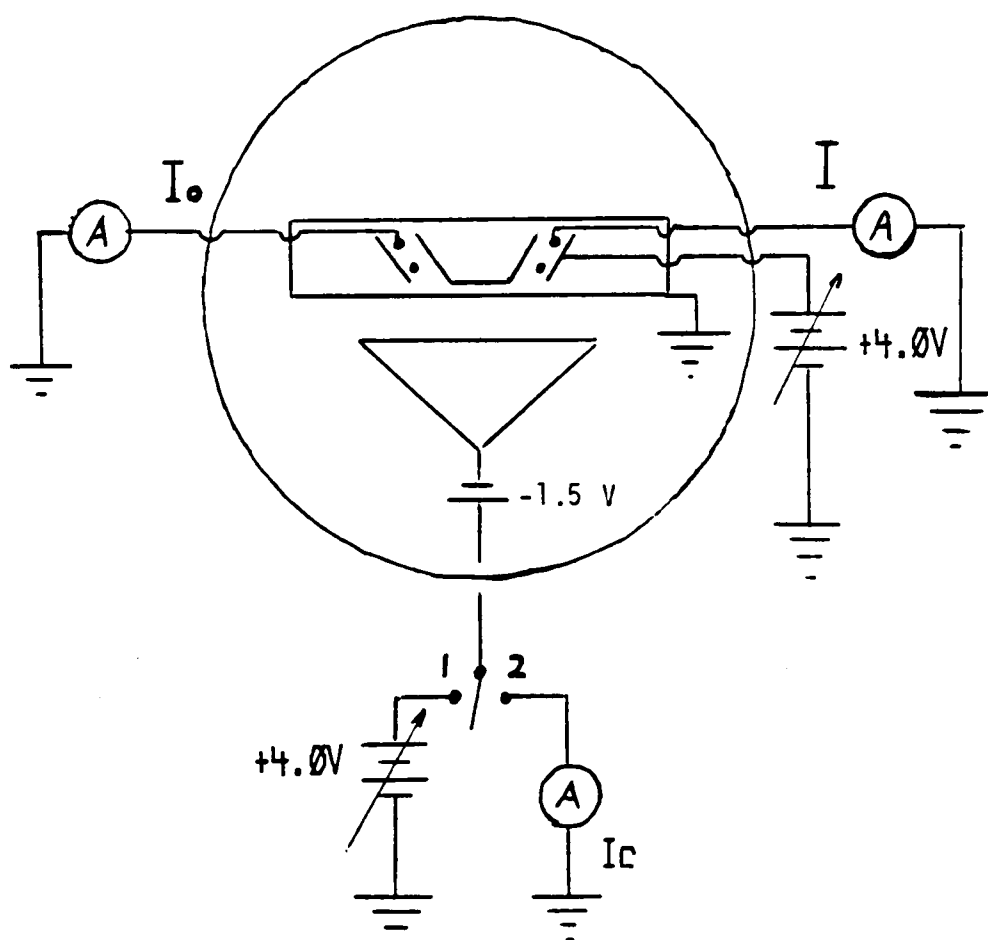


Figure 6. A schematic diagram of the measurement part for the experiment.

held constant at +4 V. Wires which collect the ions produced by the photon beam are connected with two electrometer inputs of a ratio meter. As shown in Figure 6, the sides of the ion chambers are 3 mm wide and the diameter of the wires is 0.05 mm. The chamber was filled with a pressure of argon in the range 80-260 mtorr for the ionization gas. The Ar^+ ions were pulled toward the collector wires and the electrons were pulled toward the positive sides in the chamber. The ion currents were measured by the electrometers.

Experiments used two modes of measurement: reflectance measurement and photoemission measurement. The reflectance measurement involves determination of the reflectance on the liquid surface. Determination of the absorption of the photon beam on the liquid surface was made by measuring the ion-chamber current ratio (I/I_0). The photoemission measurement is the determination of photoyield per incident photon. According to the degree of absorption of the photons, the photoyield could be determined by measuring the photoelectron current which was produced by each photon. The cup was first biased at +2.5 V in reflectance measurements. Then it was biased at -1.5 V in the photoemission measurement. The cup was connected to a battery in order that the cup be held steady biased at -1.5 V. Then the battery was connected to a switch which can be connected at point 1 with a bias voltage of +4 V and at point 2 with an electrometer as shown in Figure 6.

The cup in which the liquid samples were set was biased at +2.5 V in order to inhibit photoemission during the determination of

the currents I_0 , I , and the ratio (I/I_0) in the measurement of the reflectance. The cup was then biased at -1.5 V for the determination of the cup current I_c in order to repel all photoelectrons from the cup in the photoemission measurement. The above voltage biases on the chamber walls and on the cup were found to give the most satisfactory results (Birkhoff, 1982). In the early trials, bias potentials up to 50 V were used as a reference on the chamber walls and on the cup. This caused problems which affected the final results. While the reflectances calculated from the currents of the ion chambers I_0 and I are reasonable, the absolute photoyields vary with the bias voltage. Apparently higher bias voltages gave high enough electric fields to cause ion multiplication and thereby proportionately increased ion-chamber currents I_0 and I , but not affecting the current of the liquid cup I_c very much. Hence, the ratio (I_c/I_0) , which was included in the calculation of the photoyield, was much too small; it varied with the bias of the ion chambers. Lower voltages (meaning the bias voltage of the ion chambers set at $+4$ V and the bias voltage of the cup at $+2.5$ V for reflectance and -1.5 V for photoemission) were used to eliminate these problems.

All data were taken at various pressures between 80 mtorr and 260 mtorr of argon gas. The first set of data was taken with the pressure about 250 mtorr or more, which was kept constant for the whole photoemission measurement. Then several sets of data were taken at several intermediate pressures. Before the photoemission measurements were taken, the cup was located so that the reflected to incident

current ratio was maximized. Thus all photoemission measurements were conducted at the same cup position. The cup position was set near the maximum of the ratio I/I_0 . This was done on an intense light line of the lamp near $760 \text{ \AA}$ or $675 \text{ \AA}$.

The absolute photoyield was calculated as follows. The current I_c consists of the true photoelectron emission current I_p and two positive ion currents I_b and I_r . The positive ion current I_b is formed by the incident photon beam between exit 1 of the first ion chamber and the surface of the liquid sample. This produces positive ions and these are attracted to the negatively charged surface. The positive ion current I_r is due to ionization from the reflected photon beam between the liquid surface and the entrance at point (3) of the second ion chamber. Hence,

$$I_c = I_p + I_b + I_r . \quad (23)$$

Y is the absolute photoyield defined as the ratio of electrons emitted/photons absorbed. If ϕ_s is the photon beam flux at the surface of the liquid sample, then

$$I_p = Y\phi_s . \quad (24)$$

The ion chambers were built to be symmetric as shown in Figure 7. Thus the distance $X1$ between the entrance point (0) and the exit point (1) of the first ion chamber along the photon beam path is equal to the distance $(X4-X3)$ between the entrance point (3) and the

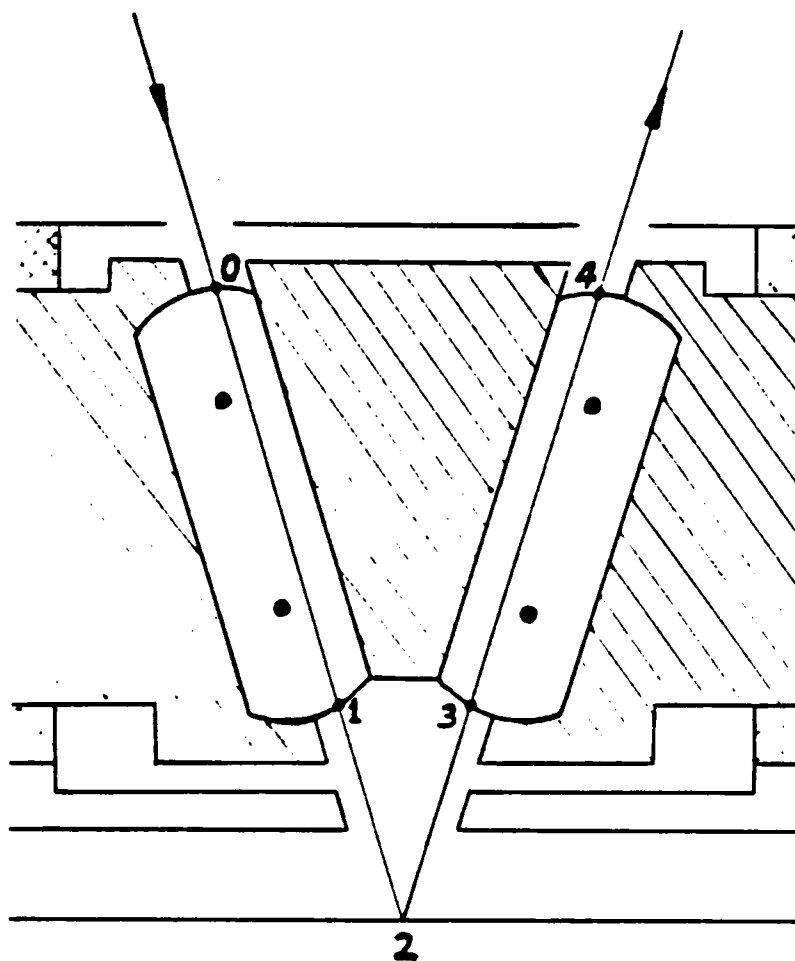


Figure 7. A cross section of the double ionization chamber for photoemission measurement on liquids.

exit point (4) of the second ion chamber. The distance $(X_2 - X_1)$ between point (1) and the liquid surface (2) is equal to the distance $(X_3 - X_2)$ between point (3) and the liquid surface (2). Hence,

$$X_1 = X_4 - X_3 \quad (25)$$

and

$$X_2 - X_1 = X_3 - X_2 = d \quad (26)$$

where X_1 is the distance between point (0) and point (1), X_2 is the distance between point (0) and point (2), X_3 is the distance between point (0) and point (3), and X_4 is the distance between point (0) and point (4).

Ionization currents for the incident and reflected photon beam are I_0 and I . For a single gas of argon with absorption coefficient K at pressure P , the change in photon flux over an incremental path dx is:

$$d\phi = -\phi K P dx \quad (27)$$

so that

$$\int \frac{d\phi}{\phi} = - \int K P dx \quad (28)$$

where the photon beam flux is ϕ . One has then

$$\phi = \phi(x_0) e^{-KP x} \quad (29)$$

where the photon beam flux at point (0) is $\phi(x_0)$.

Chamber currents I and I_0 are just the photon flux lost by absorption in the chambers since one photon absorbed yields argon-ion pair in the range 16-28 eV, one has

$$dI = KP\phi dx \quad (30)$$

so

$$I_0 = \int_0^{x_1} KP\phi(x_0)e^{-KPx} dx \quad (31)$$

so

$$I_0 = \phi(x_0)(1 - e^{-KPx_1}) . \quad (32)$$

The flux at x_3 and x_4 has been reduced by the reflection at x_2 ,

$$\phi(x_3) = \phi(x_0)R e^{-KPx_3} \quad (33)$$

where R is the reflectance of the surface. Thus,

$$\begin{aligned} I &= \int_{x_3}^{x_4} dx KP\phi(x_0)R e^{-KPx} \\ &= \phi(x_0)R(e^{-KPx_3} - e^{-KPx_4}) . \end{aligned} \quad (34)$$

Taking the ratio of I to I_0 :

$$\frac{I}{I_0} = \frac{R(e^{-KPx_3} - e^{-KPx_4})}{1 - e^{-KPx_1}} .$$

But

$$X_4 = X_3 + X_1$$

so

$$\frac{I}{I_0} = R e^{-KPX_3} . \quad (35)$$

The true photoelectrons emission from the liquid surface is

$$I_p = Y\phi . \quad (36)$$

The total cup current is

$$I_c = I_p + I_b + I_r$$

or

$$I_c = Y\phi(X_2) + I_b + I_r$$

also

$$\phi(X_2) = \phi(X_0) e^{-KPX_2} \quad (37)$$

and

$$dI = KP\phi dx$$

so

$$I_b = \int_{X_1}^{X_2} dI$$

or

$$I_b = \phi(x_0)(e^{-KPx_1} - e^{-KPx_2}) \quad (38)$$

giving

$$I_r = \int_{x_2}^{x_3} dI$$

or

$$I_r = \phi(x_0)R(e^{-KPx_2} - e^{-KPx_3}) . \quad (39)$$

Now,

$$\begin{aligned} I_c = & Y\phi(x_0)e^{-KPx_2} + \phi(x_0)(e^{-KPx_1} - e^{-KPx_2}) \\ & + \phi(x_0)R(e^{-KPx_2} - e^{-KPx_3}) \end{aligned}$$

where $x_2 = x_1 + d$ and $x_3 = x_2 + d$. Then from (32),

$$\phi(x_0) = \frac{I_0}{1 - e^{-KPx_1}}$$

so

$$I_c = \frac{I_0}{1 - e^{-KPx_1}} \left[Y e^{-KPx_2} + \left(e^{-KPx_1} - e^{-KPx_2} \right) + R \left(e^{-KPx_2} - e^{-KPx_3} \right) \right]$$

$$\frac{I_c}{I_0} (1 - e^{-KPx_1}) e^{+KPx_1} = Y e^{-Kpd} + (1 - e^{-Kpd}) + R e^{-Kpd} (1 - e^{-Kpd})$$

so

$$Y = \frac{I_c}{I_0} (1 - e^{-KP x_1}) e^{KP(x_1+d)} + (1 - e^{KPd}) + R(1 - e^{-KPd})$$

$$Y = \frac{I_c}{I_0} (e^{KP(x_1+d)} - e^{KPd}) + (1 - e^{KPd}) + R(1 - e^{-KPd})$$

where $x_2 = x_1 + d$, and the photoelectric yield is:

$$Y = \frac{I_c}{I_0} (e^{KP x_2} - e^{KPd}) + (1 - e^{KPd}) + R(1 - e^{-KPd}) \quad (41)$$

$$x_3 = x_2 + d .$$

From (35) the reflectance R is:

$$R = \frac{I}{I_0} e^{KP(x_2+d)} \quad (42)$$

where $[KP(x_2 + d)]$ is the slope of the line when $\ln(I/I_0)$ is plotted against the pressure.

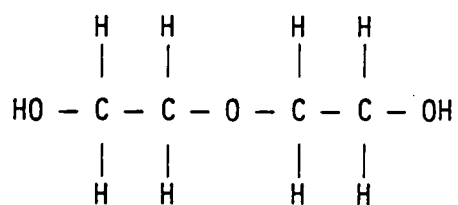
CHAPTER IV

RESULTS

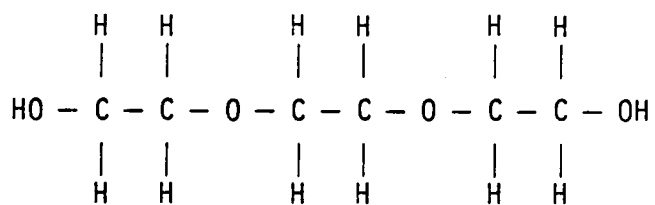
Three different liquid samples: diethylene glycol, triethylene glycol, and tetraethylene glycol, were used. These were then doped with liquids at 0.001, 0.01, and 0.1 molar concentrations of LiOH in successive experiments. The molecular structures of these ethylene glycols are shown in Figure 8.

These liquids are part of a series of n-ethylene glycols which are ethylene glycol, diethylene glycol triethylene glycol, tetraethylene glycol, and so on. They have a variety of industrial uses. Ethylene glycol is prepared by the hydration of ethylene oxide. It is used as an industrial coolant, heat transfer fluid, brake fluid, and so on. Diethylene glycol, made by heating ethylene glycol and glycol, is used in antifreeze solution, as a lubricating and finishing agent for a number of fabrics, including wool, cotton, and silk. Triethylene glycol, prepared from ethylene oxide and ethylene glycol in the presence of sulfuric acid, is used in plastics and in air disinfection. The polyethylene glycols are also used in biology in incubating mammalian cells.

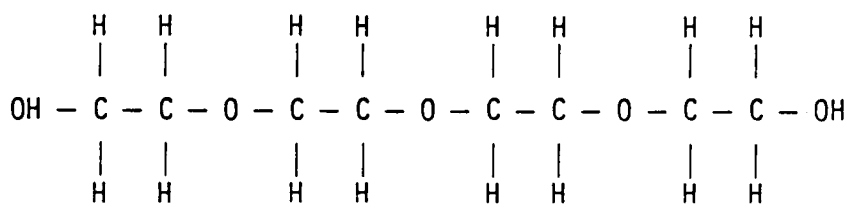
The chemical properties of triethylene glycol and tetraethylene glycol indicate that they are very stable. For example, they do not undergo chemical reaction with an alkali hydroxide solution. The vapor pressures of triethylene glycol and tetraethylene glycol are low enough



DIETHYLENE GLYCOL (mol. wt. 106)



TRIETHYLENE GLYCOL (mol. wt. 150)



TETRAETHYLENE GLYCOL (mol. wt. 194)

Figure 8. Structure of the liquids studied.

so that the photoemission and reflectance measurements can be taken under low pressure in nominal vacuum. However, the vapor pressure of the diethylene glycol is high enough to make the measurements under low pressure conditions more difficult. It also does not undergo chemical reaction with an alkali hydroxide solution.

The measurement of diethylene glycol was more difficult because the vapor pressure of diethylene glycol is higher than triethylene glycol and tetraethylene glycol. The cup was filled as full as possible with the liquid samples until a positive meniscus was seen. When the data of measurement of diethylene glycol were taken at low pressure between 80 mtorr and 260 mtorr, the liquid sample of diethylene glycol vaporized quickly enough so that the surface of diethylene glycol had to be adjusted during the measurement of the experiment. The cup was kept at the position which the reflected signal from the surface of diethylene glycol was maximized by raising the cup only one turn (0.032 mm/turn) per half hour.

I. Photoelectric Yields

All data were taken in the far ultraviolet between 400 Å and 800 Å corresponding to a photon energy between 15.5 eV and 31.0 eV. The pressures used were between 80 mtorr and 260 mtorr. Three kinds of liquid: diethylene glycol, triethylene glycol, and tetraethylene glycol, were used in the measurements and each kind of liquid sample was both doped and undoped with different concentrations of LiOH solution. Three sets of data were taken with dopings of 0.001 molar,

0.01 molar, 0.1 molar, and without doping of LiOH. Each liquid sample was measured at different pressures of about 90 mtorr, 110 mtorr, 130 mtorr, 160 mtorr, 200 mtorr, and 250 mtorr.

The photoyield per incident photon was taken by measuring the cup current I_c . We combined this with the data of reflectance. Then the photoyield per incident photon was calculated from Eq. (42) for each argon pressure at particular wavelengths in which the cup current I_c and ion chamber current I_0 have extrema. The maxima are at 425 Å, 444 Å, 465 Å, 475 Å, 502 Å, 519 Å, 542 Å, 573 Å, 593 Å, 608 Å, 637 Å, 665 Å, 680 Å, 698 Å, 712 Å, 744 Å, and 766 Å. The data on cup current was taken at these wavelengths. Data taking was separated into the two regions 400 Å-650 Å and 600 Å-800 Å. A strong photon beam was used in the region 400 Å-650 Å in order to get a strong signal of cup current I_c and ion chamber current I_0 . Then the data were calculated using computer programs for deconvolution and peak area integration. According to the theory of the yield calculation, the yield should be independent of pressure. The independence of pressure is demonstrated in a number of figures.

In Figures 9 through 13, the tetraethylene glycol doped at 0.1 M, 0.01 M, and 0.001 M concentrations of LiOH and undoped. The yield per incident photon is plotted as a function of the argon gas pressure. The yield per incident photon for the sample doped with 0.001 molar LiOH is shown in Figure 9. Each curve was measured at particular wavelengths. Statistically the curves of Figure 9 indicate that the yield per incident photon is independent of argon

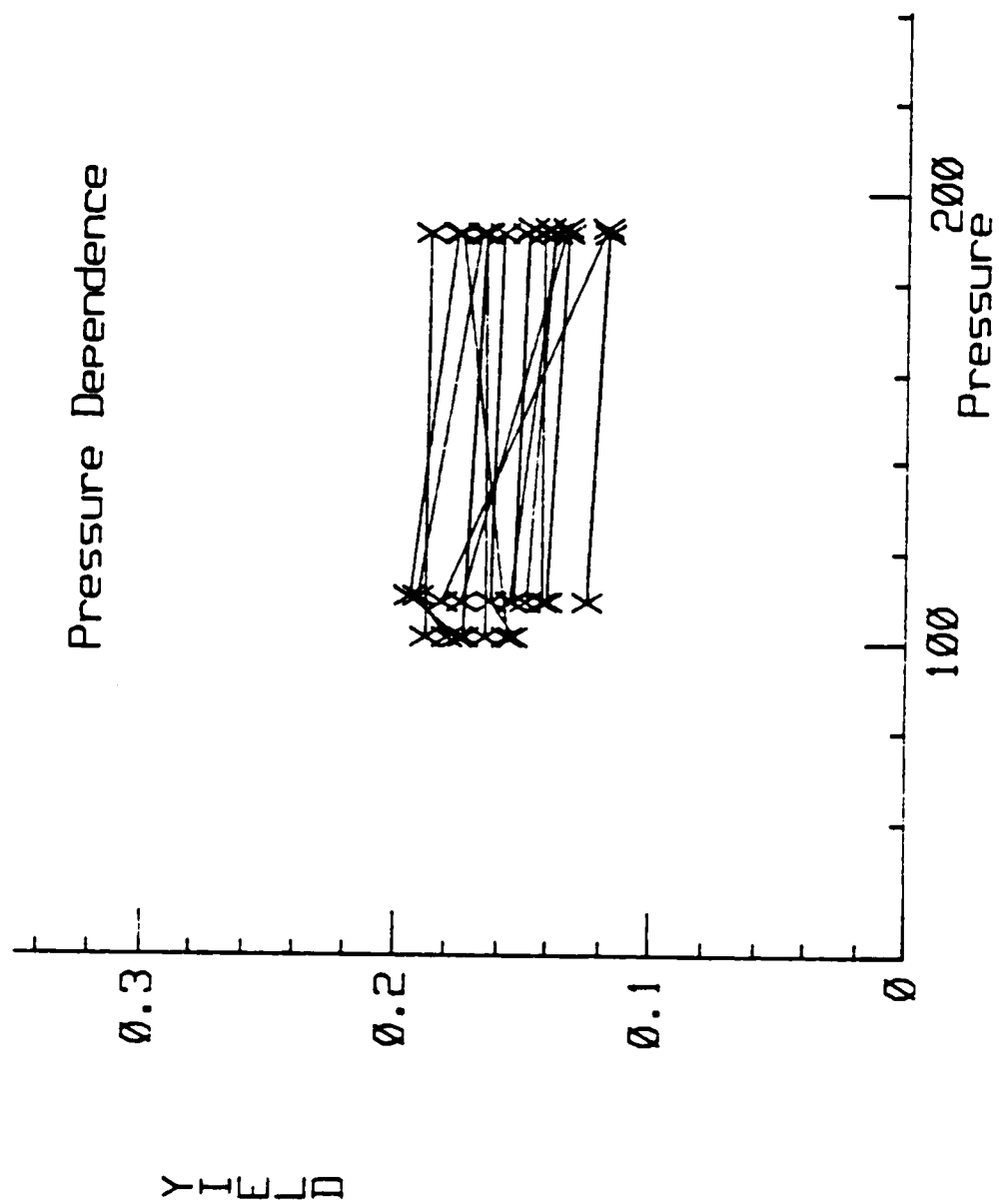


Figure 9. Tetraethylene glycol doped with 0.001 molar LiOH.

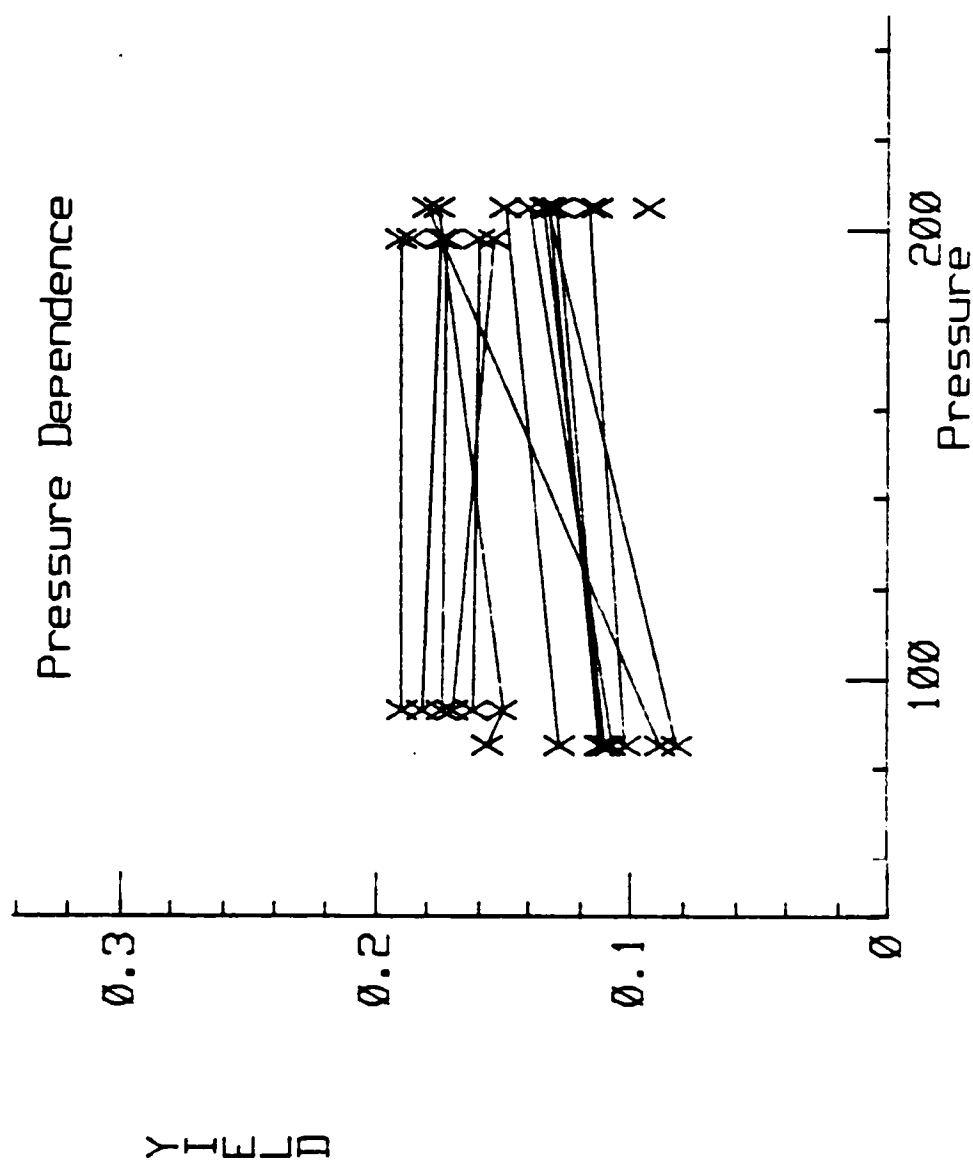


Figure 10. Tetraethylene glycol doped with 0.01 molar LiOH.

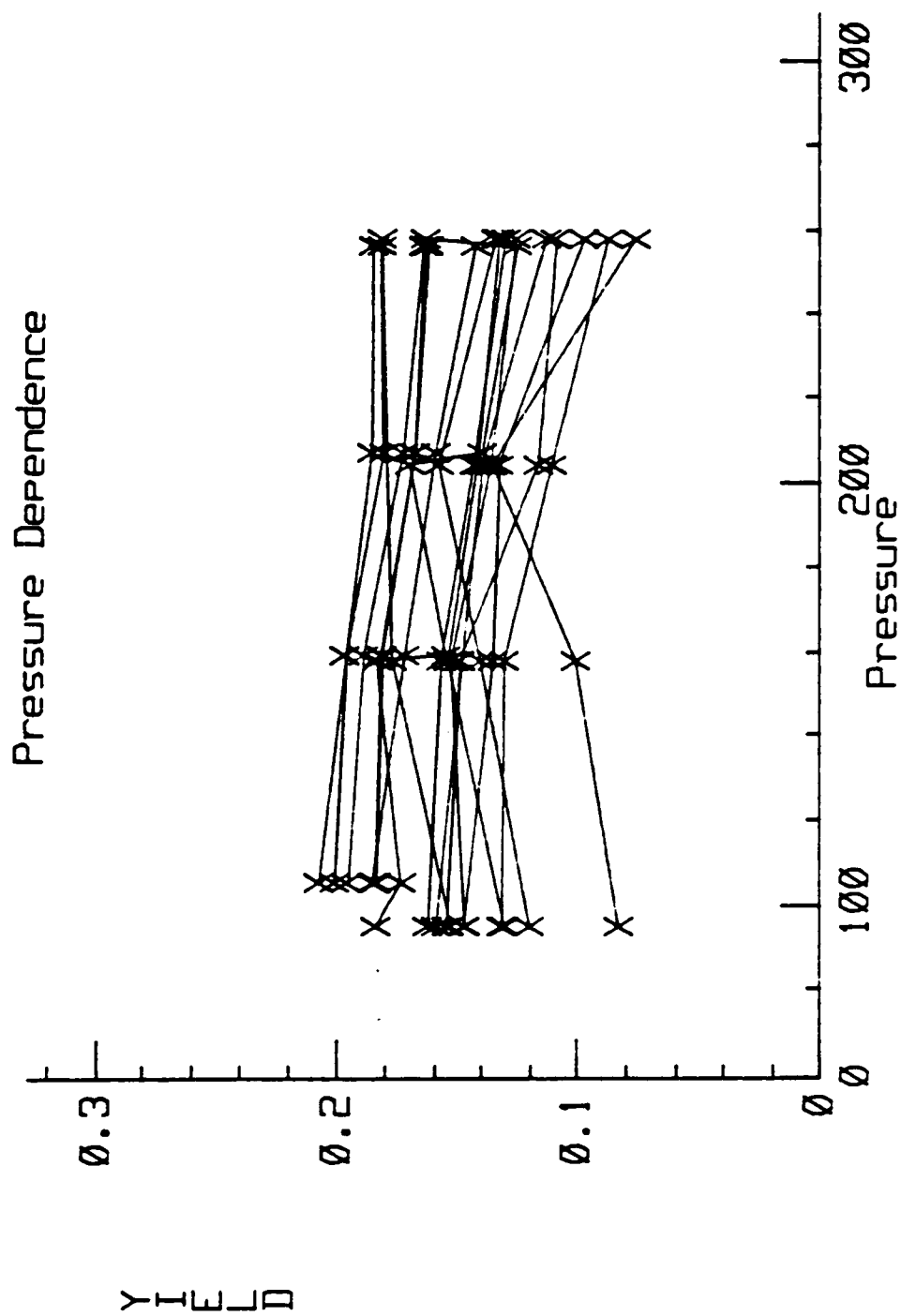


Figure 11. Tetraethylene glycol doped with 0.1 molar LiOH.

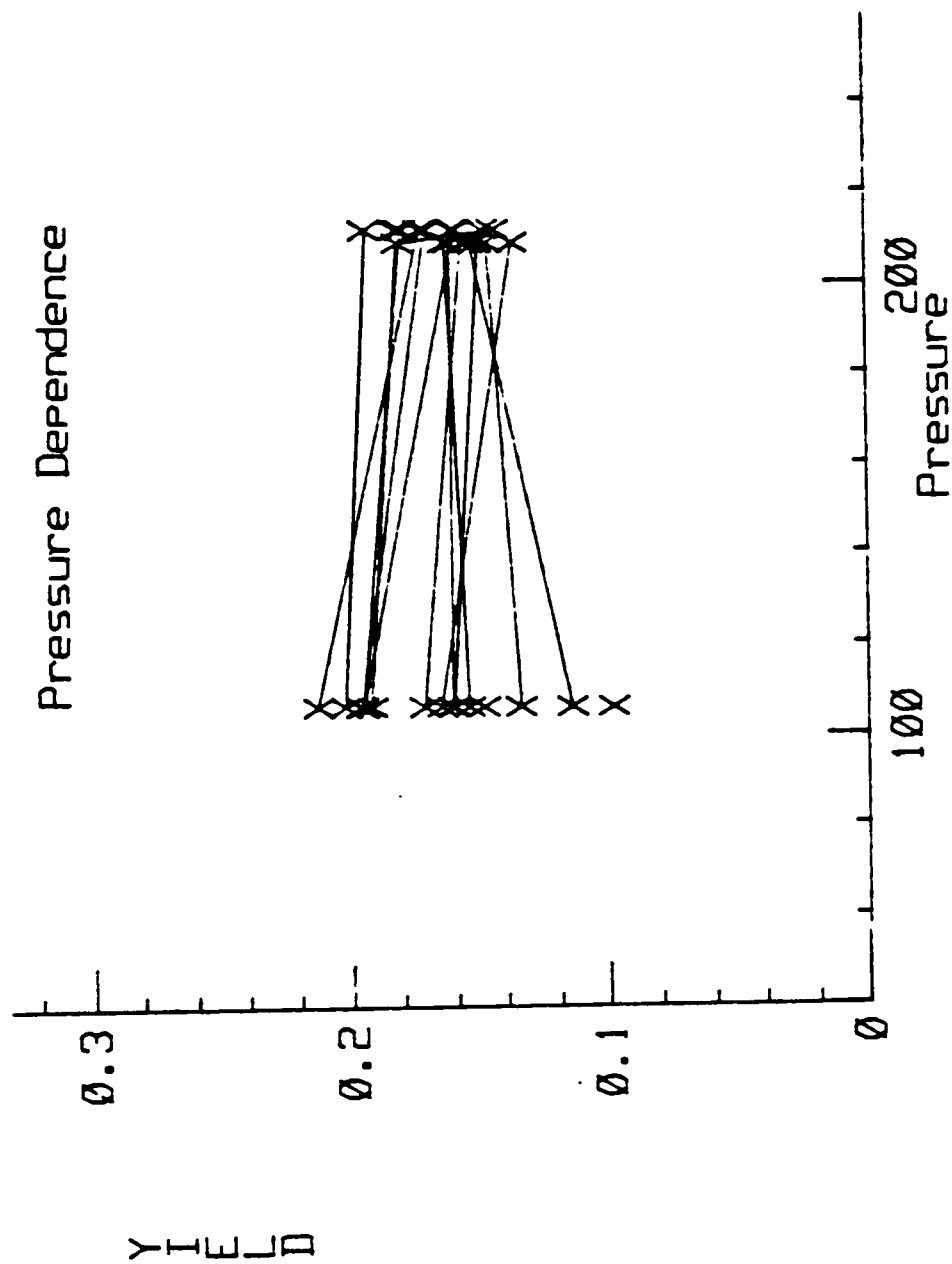


Figure 12. Undoped tetraethylene.

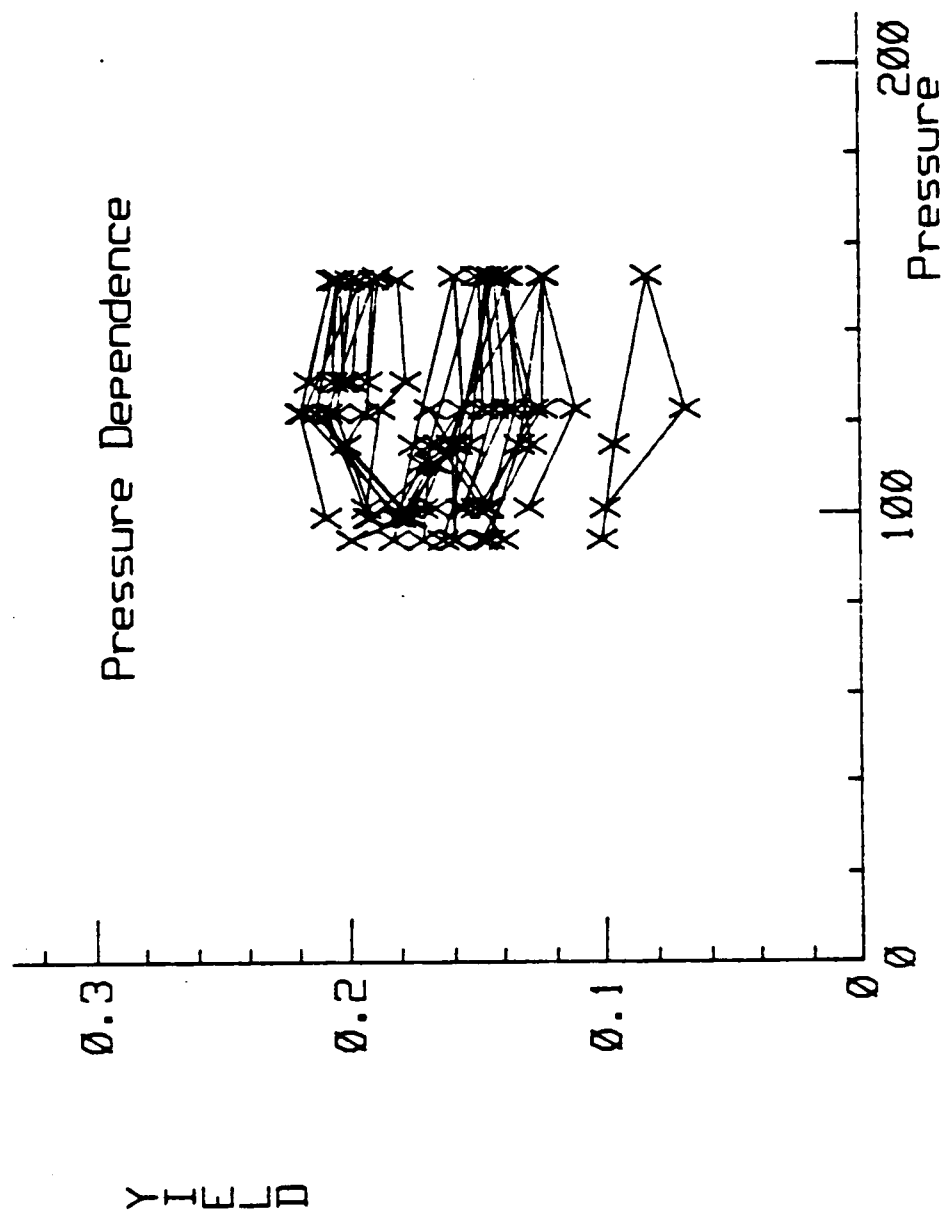


Figure 13. Triethylene glycol doped with 0.01 molar LiOH.

gas pressure. Tetraethylene glycol doped with 0.01 molar LiOH, 0.1 molar LiOH, and undoped are shown in Figure 10, Figure 11, and Figure 12. Triethylene glycol doped with 0.01 molar LiOH, 0.1 molar LiOH, and without LiOH solution are shown in Figures 13 through 15. Diethylene glycol doped with 0.01 molar LiOH and without LiOH solution are shown in Figures 16 and 17. The same results relating to independence from the argon gas pressure and independence from the doping with LiOH are shown in Figure 13, Figure 14, Figure 15, Figure 16, and Figure 17.

The yield per incident photon can be plotted as a function of photon wavelength (λ) and photon energy $h\nu$. The yield per incident photon of tetraethylene glycol, triethylene glycol, and diethylene glycol as a function of photon energy $h\nu$ are shown in Figures 18, 19, and 20. In Figure 18, the comparison of photoyields as a function of photon energy $h\nu$ from doped and undoped with LiOH for tetraethylene glycol are shown on curves marked "A," "B," "C," "D," and "E." The curve "A" is the tetraethylene glycol with doping of 0.001 molar LiOH. The curve "B" is the tetraethylene glycol with doping of 0.01 molar LiOH. The curve "C" is the tetraethylene glycol without doping. The curve "D" is the tetraethylene glycol with doping of 0.003 molar NaOH. The curve "E" is the tetraethylene glycol with doping of 0.1 mole LiOH. In Figure 19, the comparison of photoyields as a function of photon energy $h\nu$ from doped and undoped solutions for triethylene glycol are shown on the curves marked "A," "B," and "C." The curve "A" is the triethylene glycol with 0.01 molar LiOH, the curve "B" is

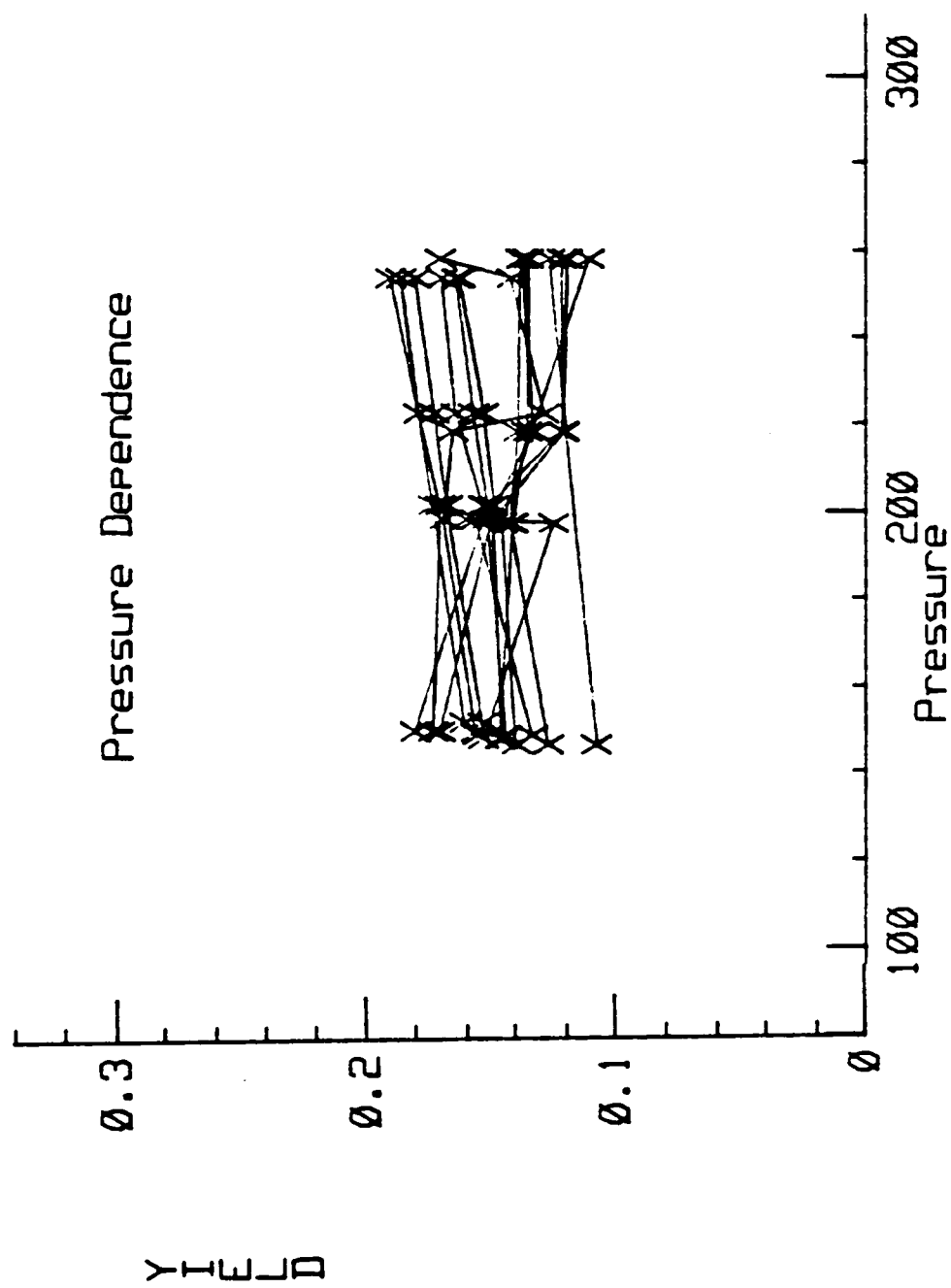


Figure 14. Triethylene glycol doped with 0.1 molar LiOH.

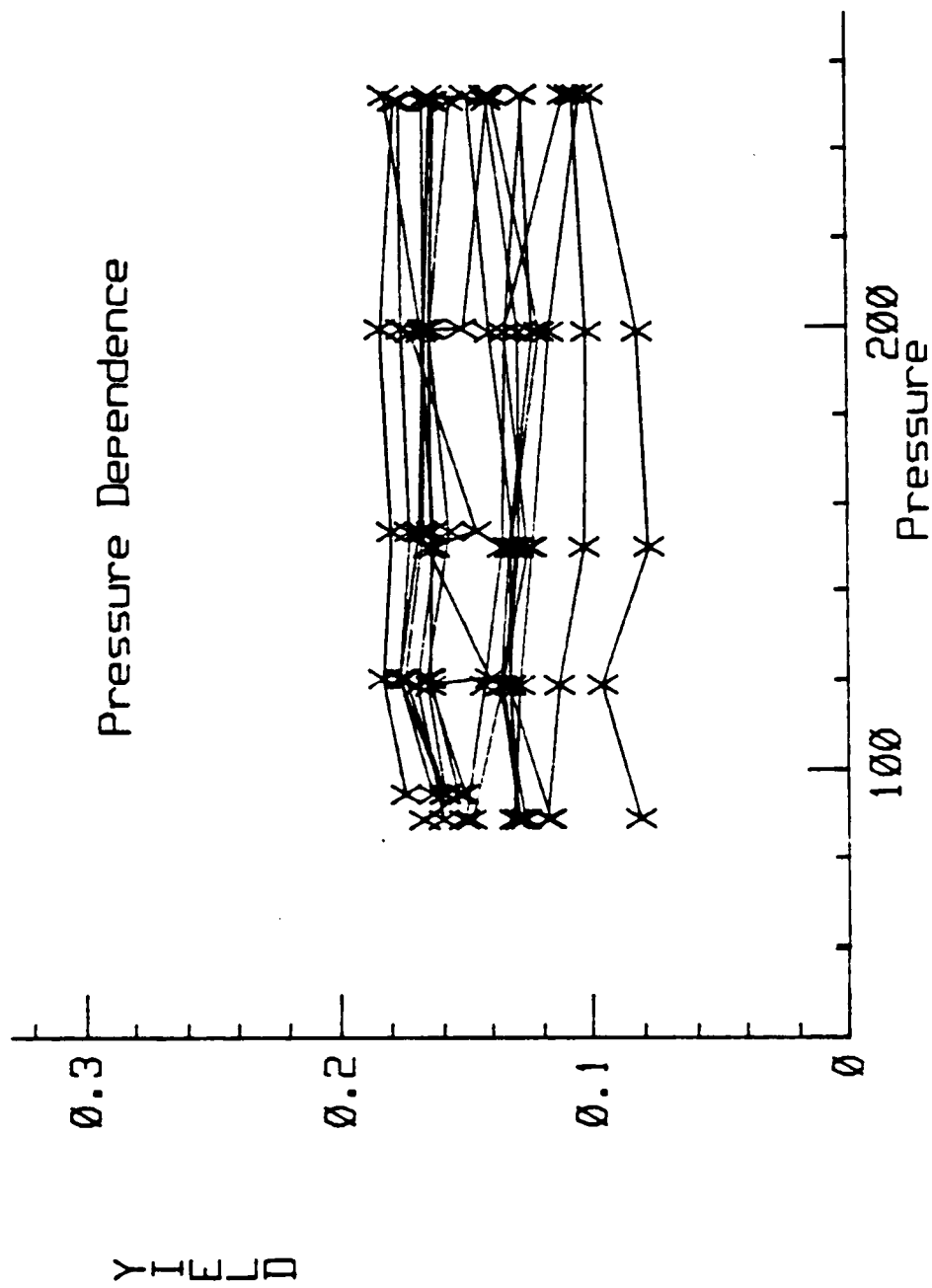


Figure 15. Undoped triethylene glycol.

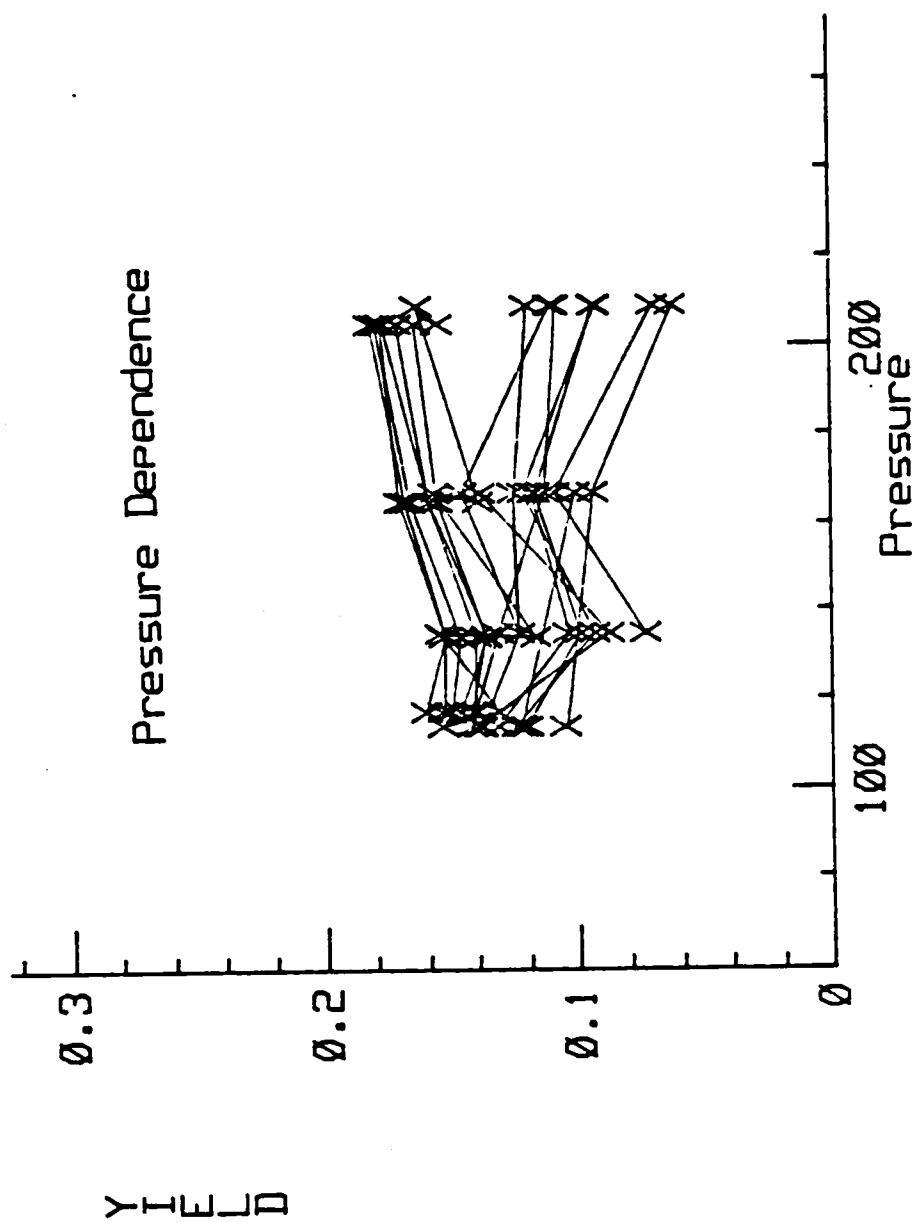


Figure 16. Undoped diethylene glycol.

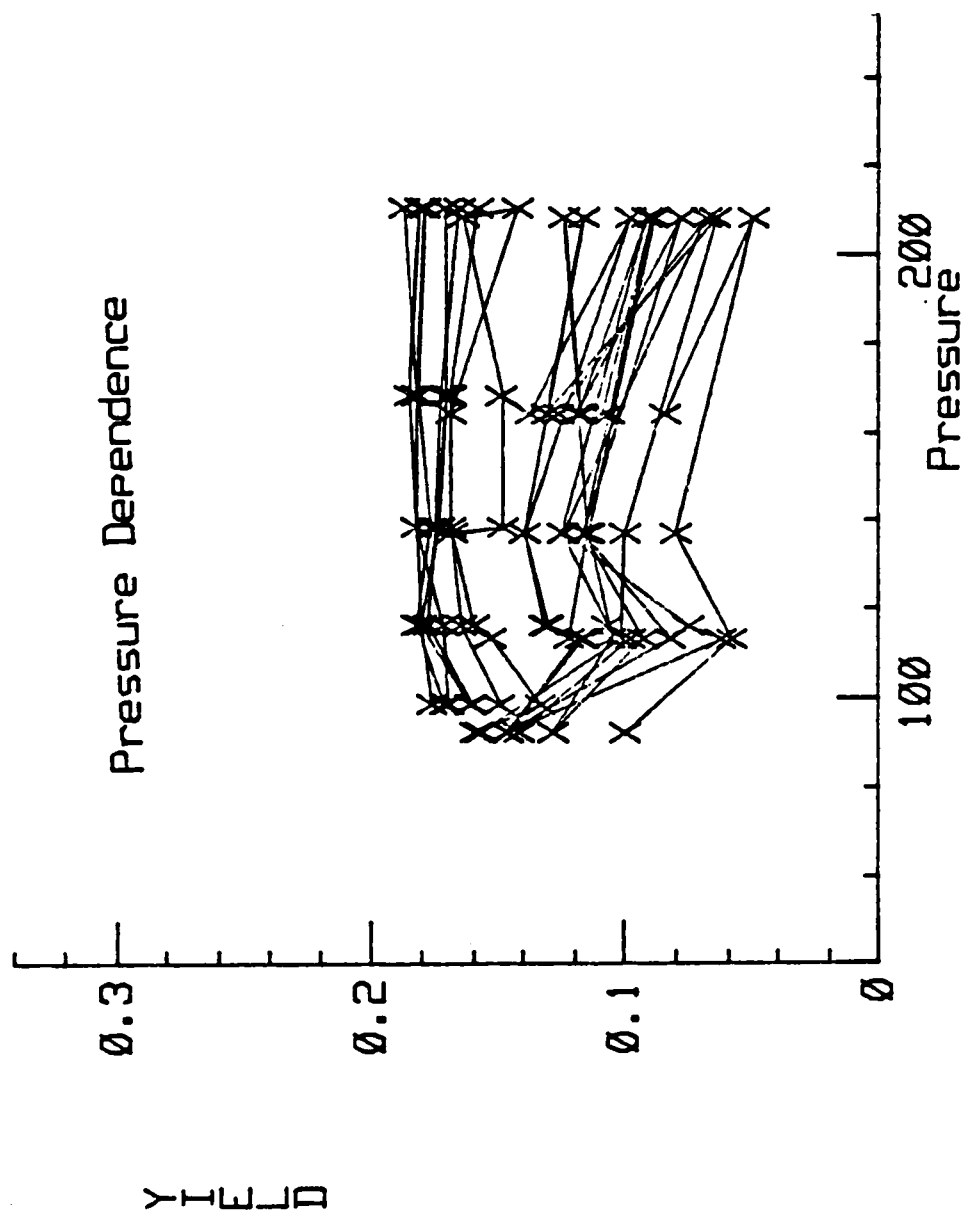


Figure 17. Diethylene glycol doped with 0.01 molar LiOH.

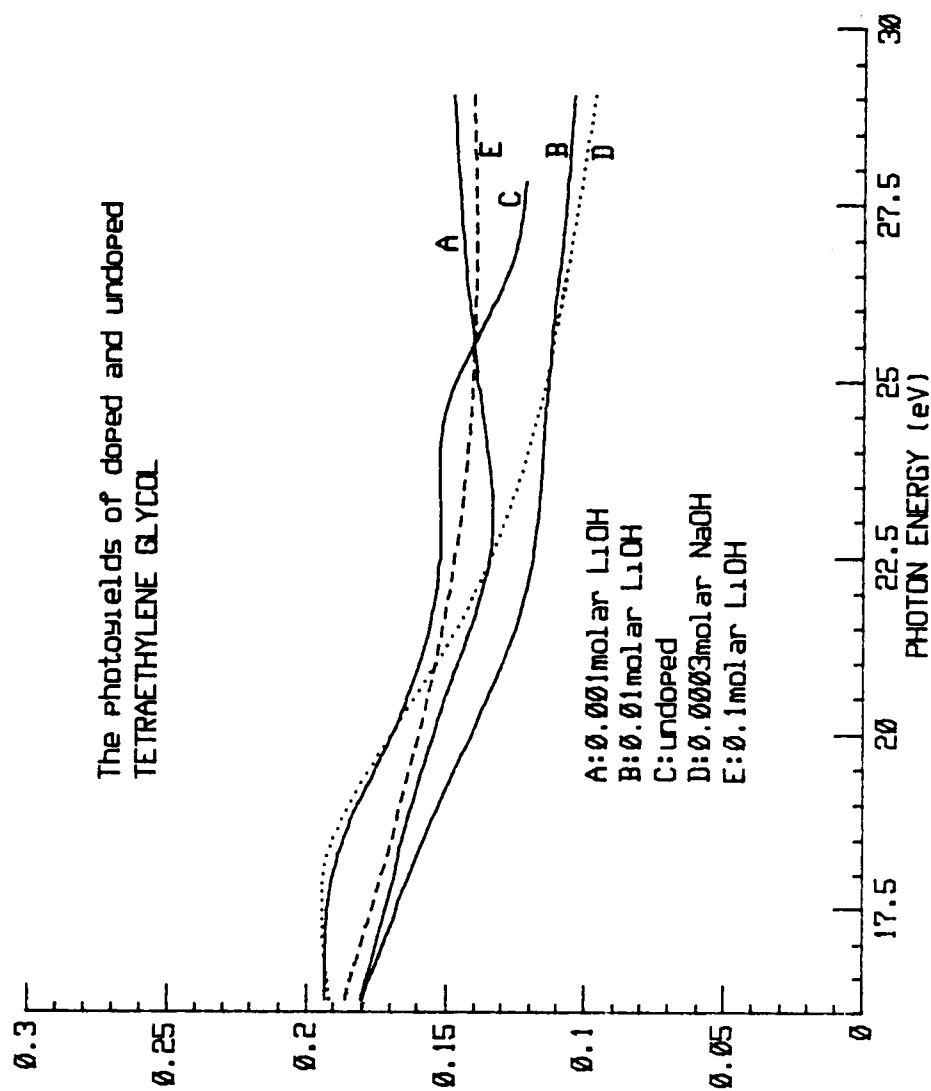


Figure 18. Comparison of photoyields from doped and undoped tetraethylene glycol.

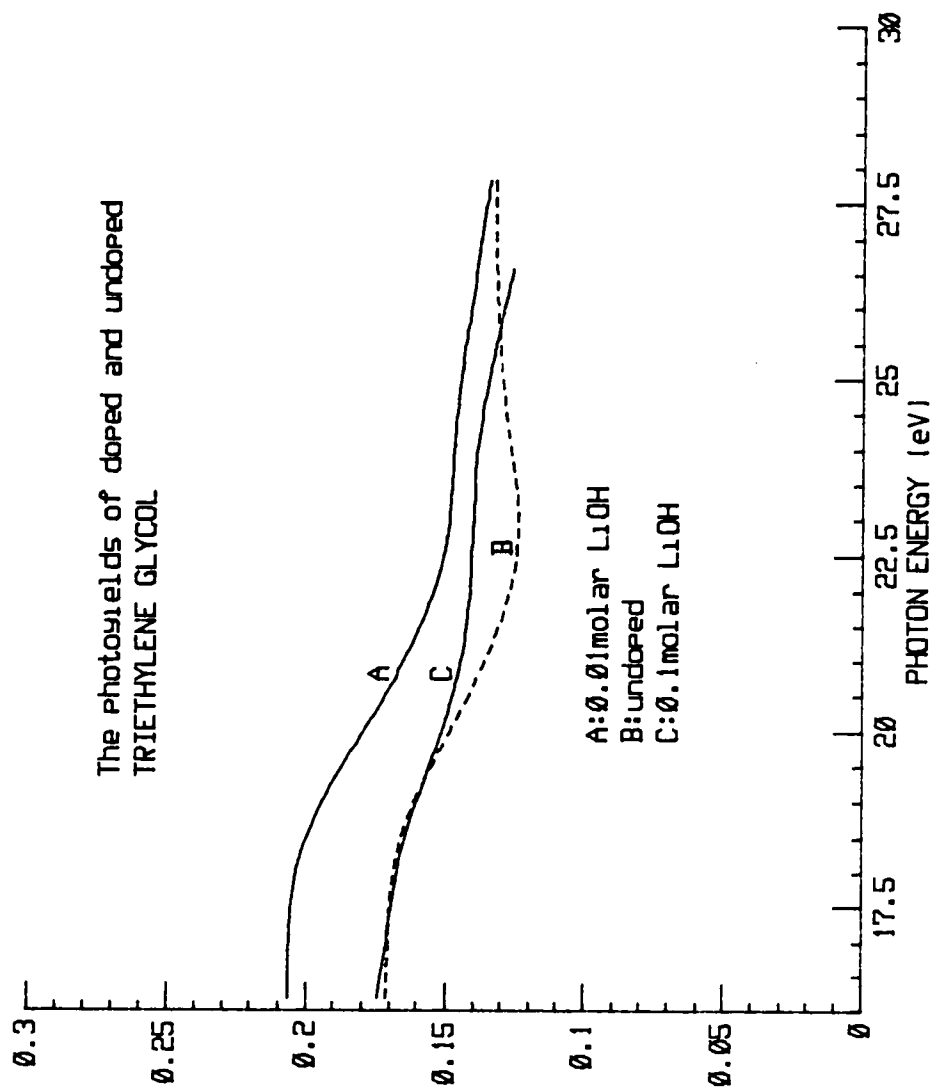


Figure 19. Comparison of photoyields from doped and undoped triethylene glycol.

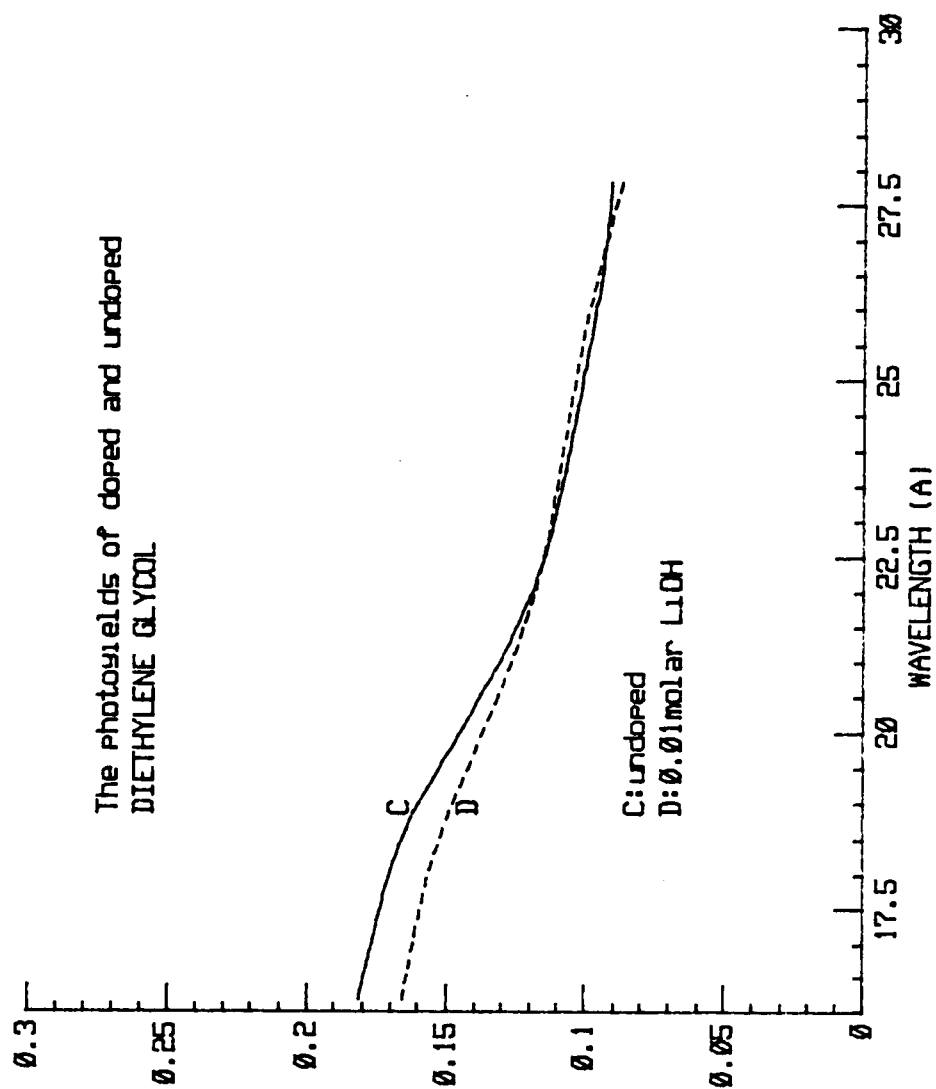


Figure 20. Comparison of photoyields from doped and undoped diethylene glycol.

the triethylene glycol without doping, and the curve "C" is the triethylene glycol with 0.1 molar LiOH. In Figure 20, the comparison of photoyields as a function of photon energy $\hbar\omega$ with doped and undoped for diethylene glycol are shown on the curves marked "C" and "D." The curve "C" is the diethylene glycol without doping. The curve "D" is the diethylene glycol with 0.01 molar LiOH. Figure 21, Figure 22, and Figure 23 show the comparison of photoyields as a function of wavelength for doped and undoped for tetraethylene glycol, triethylene glycol, and diethylene glycol.

From Figure 18, Figure 19, and Figure 20, one obvious indication is that the photoyields are independent of doping. The photoyields for tetraethylene glycol is seen to fall from between 0.18-0.20 at 16 eV to below 0.15 at 28 eV. The photoyield for triethylene glycol is seen to fall from a value of 0.17 at 16 eV to 0.14 at 28 eV and from 0.21 at 16 eV to 0.14 at 28 eV. The photoyield for diethylene glycol is seen to fall from about 0.18 at 16 eV to a value of 0.08 at 28 eV. The energy dependence from Eq. (20) is thus seen.

II. Electron Mean Free Paths

Using the preceding results, we sought the electron mean free paths and compared them to those predicted from Seah and Dench (1978). The electron mean free paths were derived from an empirical equation of Seah and Dench for organic materials and is given by $L_e = (49.0/E^2 + 0.11 E^{1/2})/\rho$, where ρ is the density of the liquid

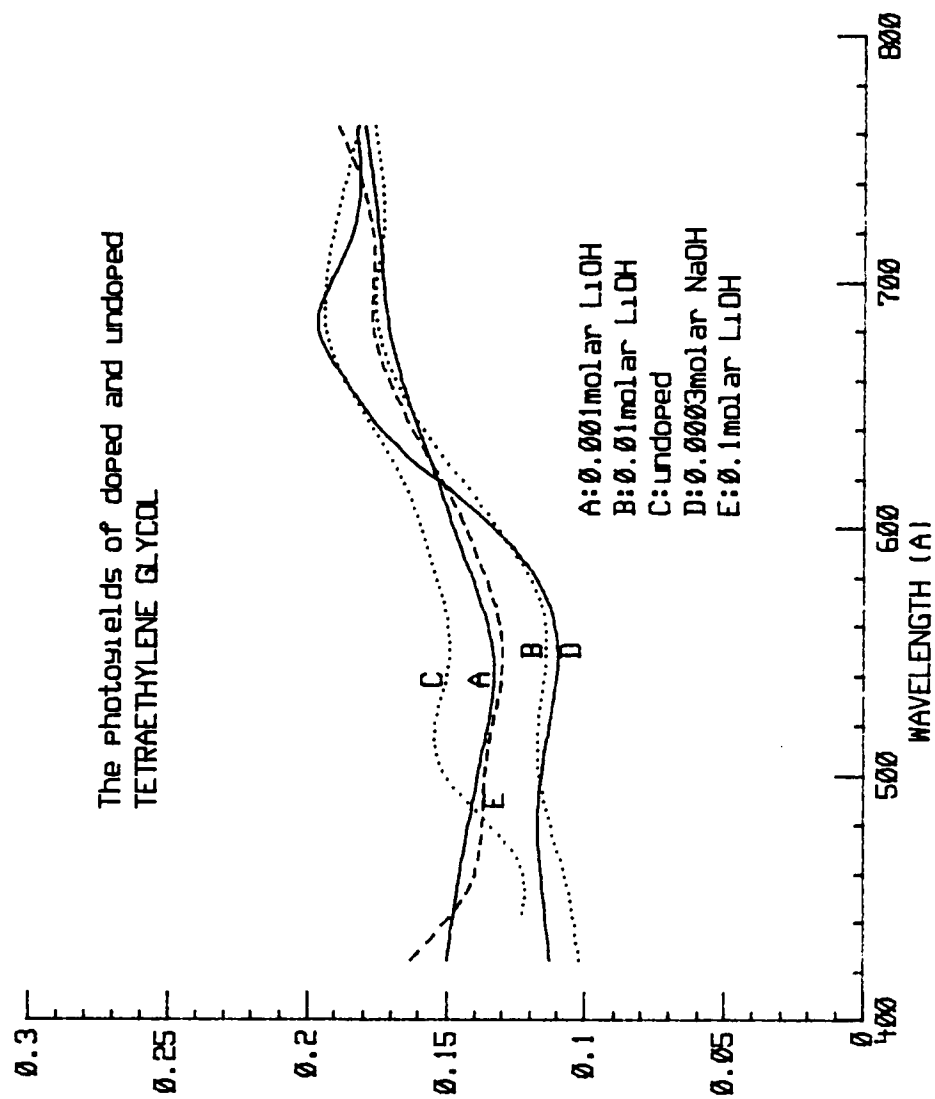


Figure 21. Comparison of photoyield from doped and undoped tetraethylene glycol.

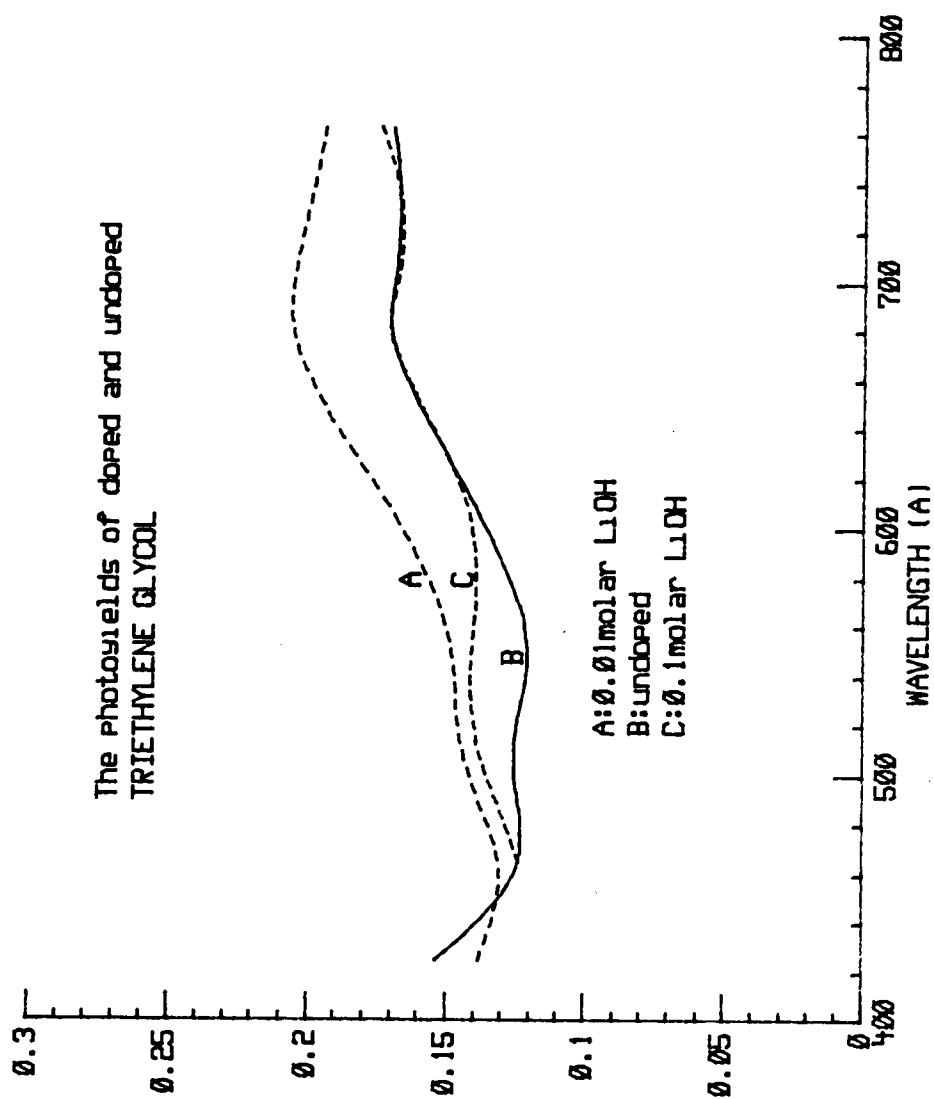


Figure 22. Comparison of photoyields from doped and undoped triethylene glycol.

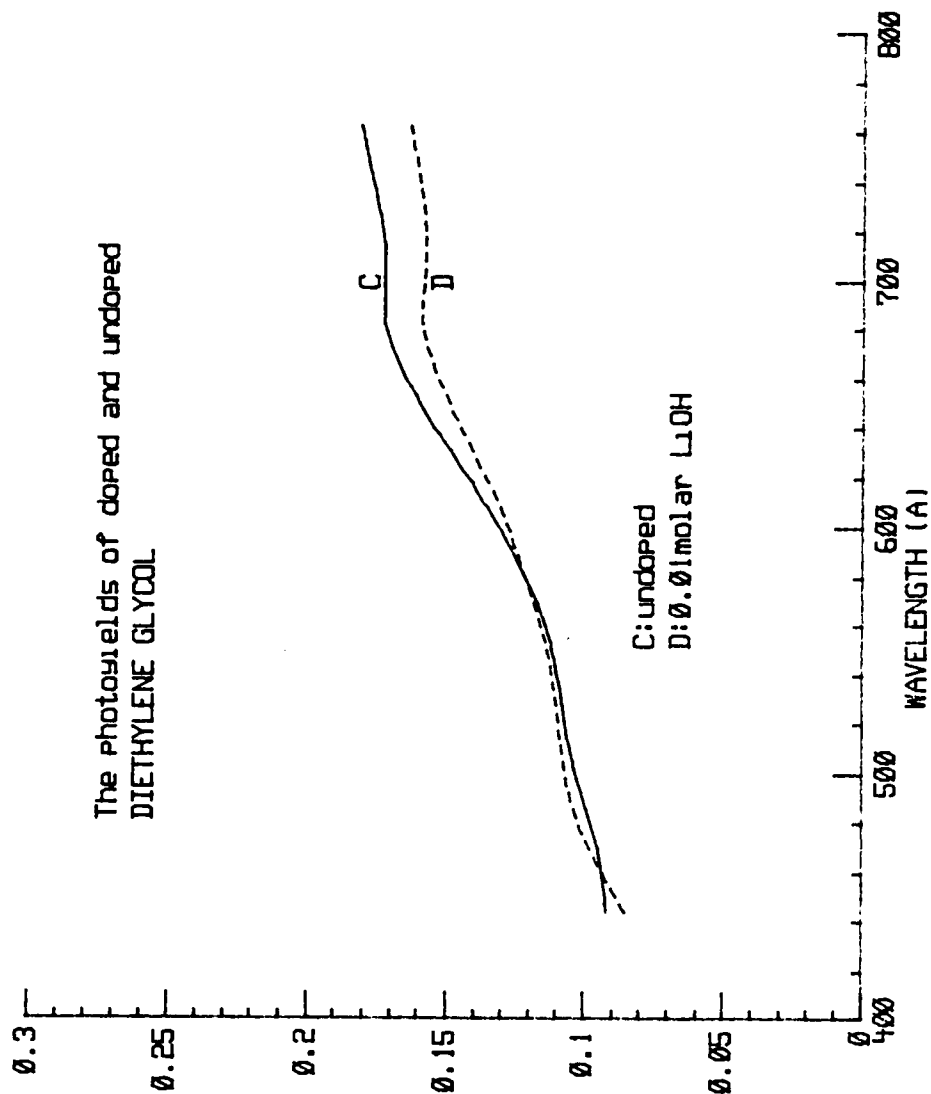


Figure 23. Comparison of photoyields from doped and undoped diethylene glycol.

(see Figure 3a, p. 19). As shown in the following, the electron mean free paths calculated from Eq. (20) with our experimental photoyields exceed those predicted from Seah and Dench's equation.

The photoyield can be calculated from the electron mean free path and photoelectric threshold energy by using Eq. (20). Conversely, the electron mean free path can be determined from the experimental photoyields and threshold energy by mathematically converting from Eq. (20). In Eq. (20),

$$\theta_c = \cos^{-1}(\phi/\hbar\omega)$$

where ϕ is the photoelectric threshold energy. Two papers (Nemec, 1975; Casanovas, 1981) on photoemission into the vacuum give about 8 eV for eleven organic liquids. Our data of the photoyield are higher so that the relation between the photoyields and the electron mean free paths cannot be satisfied when the threshold energy ϕ is 8 eV. In Eq. (20),

$$Y = \frac{1}{2} (1 - R) \left[1 - \cos\theta_c - \frac{L_p}{L_e} \ln \left(\frac{1 + L_e/L_p}{1 + (L_e/L_p)\cos\theta_c} \right) \right]$$

where $\theta_c = \cos^{-1}(\phi/\hbar\omega)$. If $\phi = 8$ eV, the data of the photoyields Y is larger than the term $\frac{1}{2} (1 - R[1 - \cos\theta_c])$ in the photon energy region between 16 eV and 30 eV.

$$Y - \frac{1}{2} (1 - R)(1 - \cos\theta_c) > 0$$

but

$$-\frac{L_p}{L_e} \ln \left(\frac{1 + L_e/L_p}{1 + (L_e/L_p) \cos \theta_c} \right) < 0 .$$

This cannot be true. Thus the formula (20) breaks down. We suggest that the threshold energy be less than 8 eV. Several groups of calculations in this thesis were done by arranging the threshold energy at 7.0 eV and 6.0 eV.

The electron mean free paths of tetraethylene glycol liquids doped with 0.1 molar, 0.01 molar, and 0.001 molar LiOH and undoped are shown in Figure 24. There the photoelectric threshold energy is 7.0 eV. The electron mean paths of these tetraethylene glycol liquids are above 10 (nm) from 20 eV to 28 eV and blow up near 17.5 eV. We compare them to the electron mean free paths derived from the empirical equation of Seah and Dench in Figure 3a (p. 19). The data of the electron mean free paths in our experiments are larger by about one order of magnitude than those in Figure 3a. The electron mean free path in Figure 24 blow up below 20 eV. Figure 25 shows the electron mean free paths in tetraethylene glycol, in which the photoelectric threshold energy is 6.0 eV. Figures 26 and 27 show the comparisons in the electron mean free paths of tetraethylene glycol doped with 0.1 molar LiOH and undoped, in which the photoelectric threshold energies are 7.0 eV and 6.0 eV. The dependence of the electron mean free paths of triethylene glycol and diethylene glycol are shown in Figures 28 and 29, in which the photoelectric threshold energy is 7.0 eV.

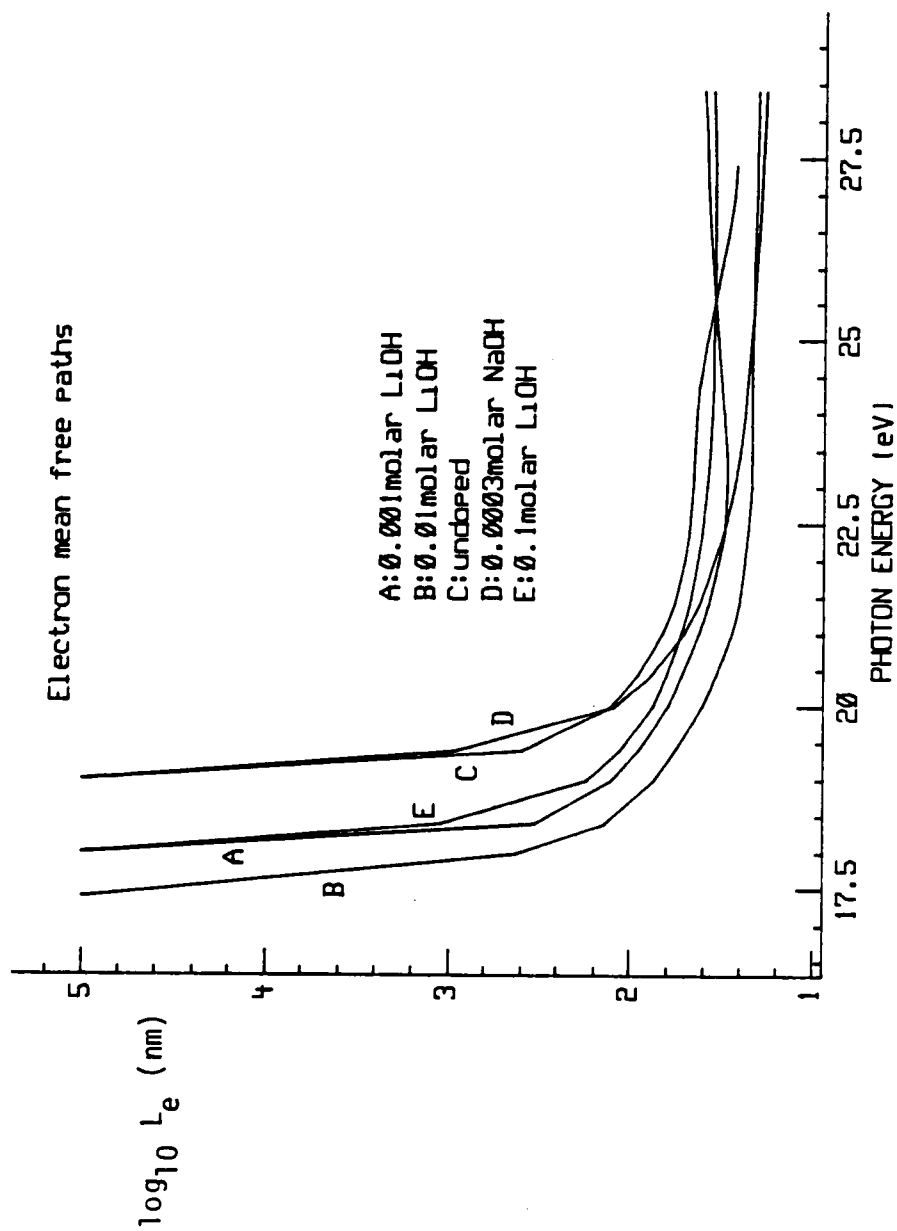


Figure 24. Electron mean free paths of doped and undoped tetraethylene glycol, $\phi = 7.0$ eV.

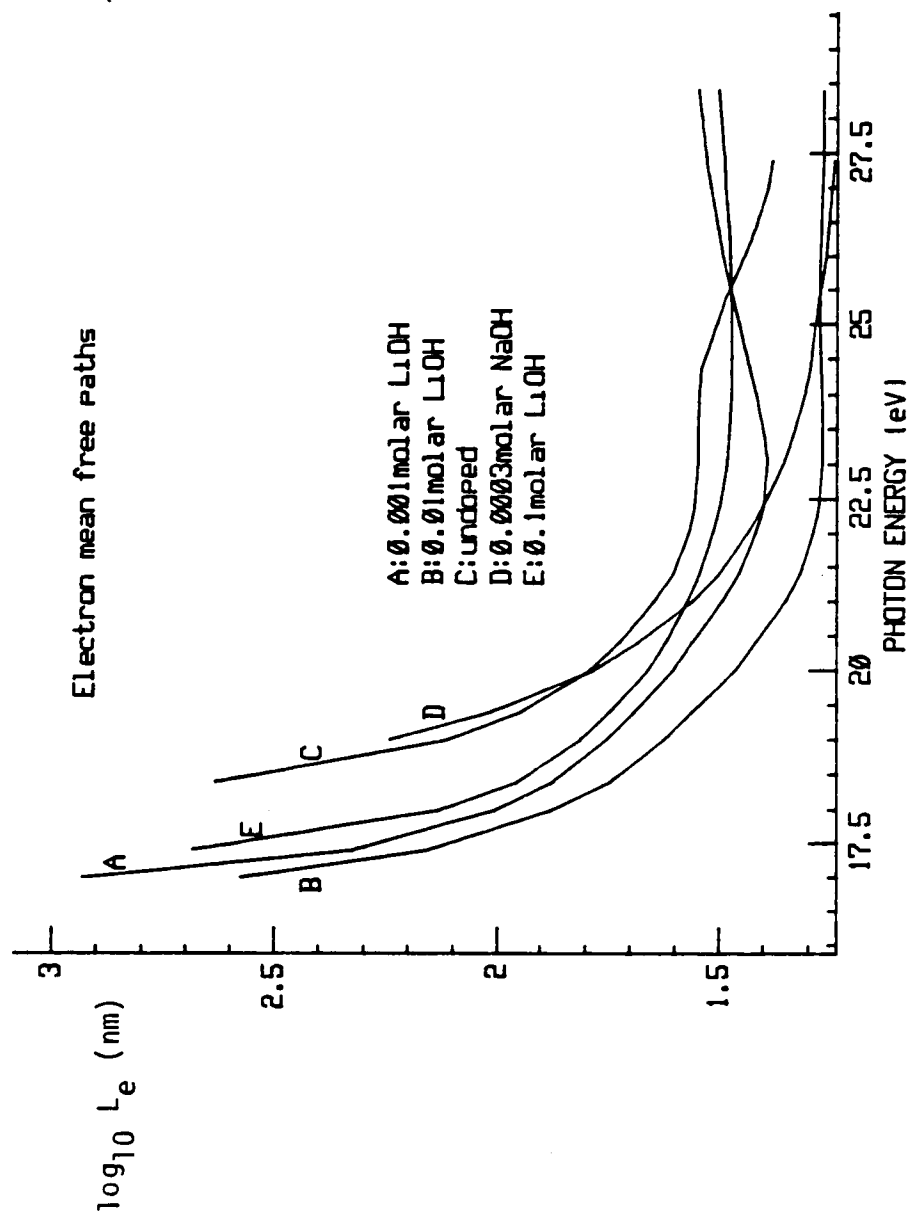


Figure 25. Electron mean free paths of doped and undoped tetraethylene glycol, $\phi = 5.0$ eV.

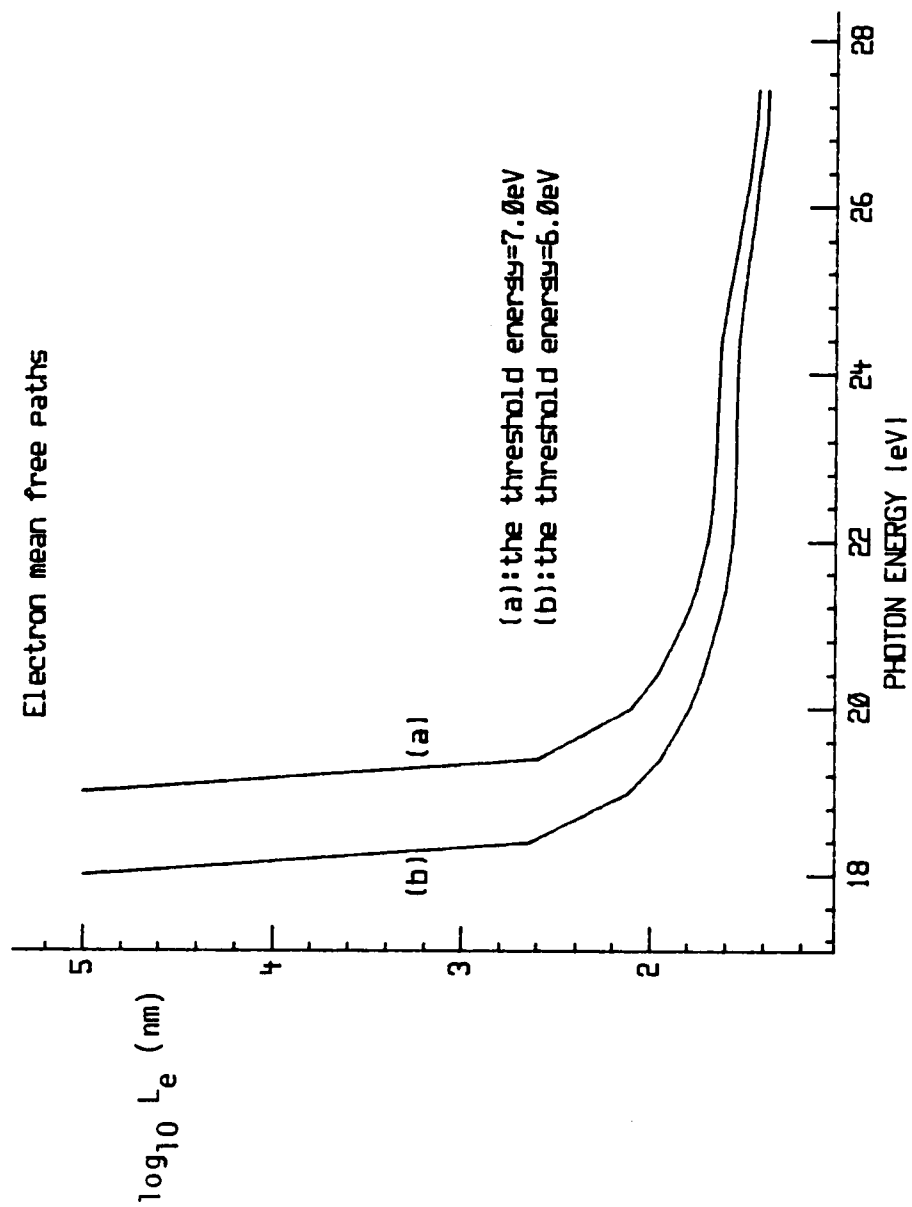


Figure 26. The comparison of electron mean free paths for undoped tetraethylene glycol in different threshold energy.

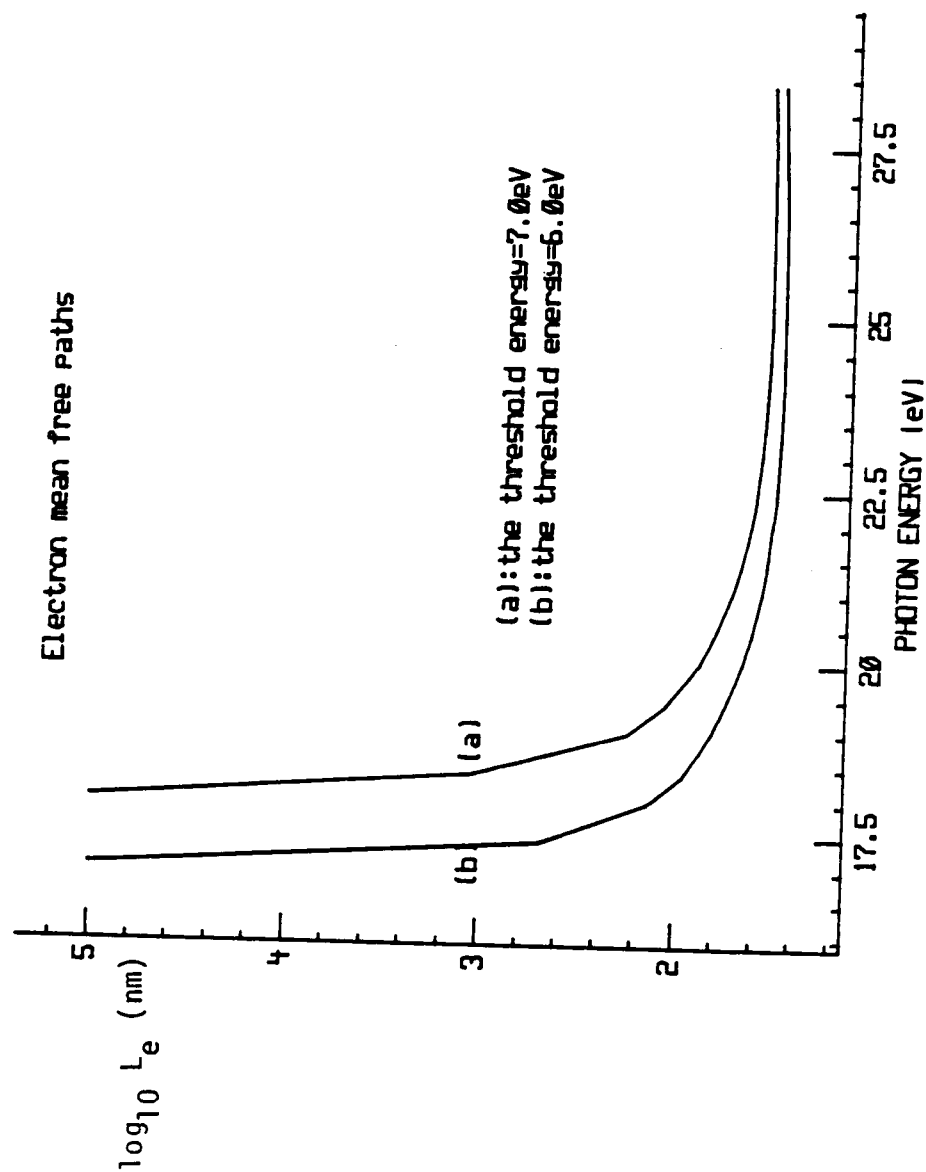


Figure 27. The comparison of electron mean free paths for doped tetraethylene glycol in different threshold energy.

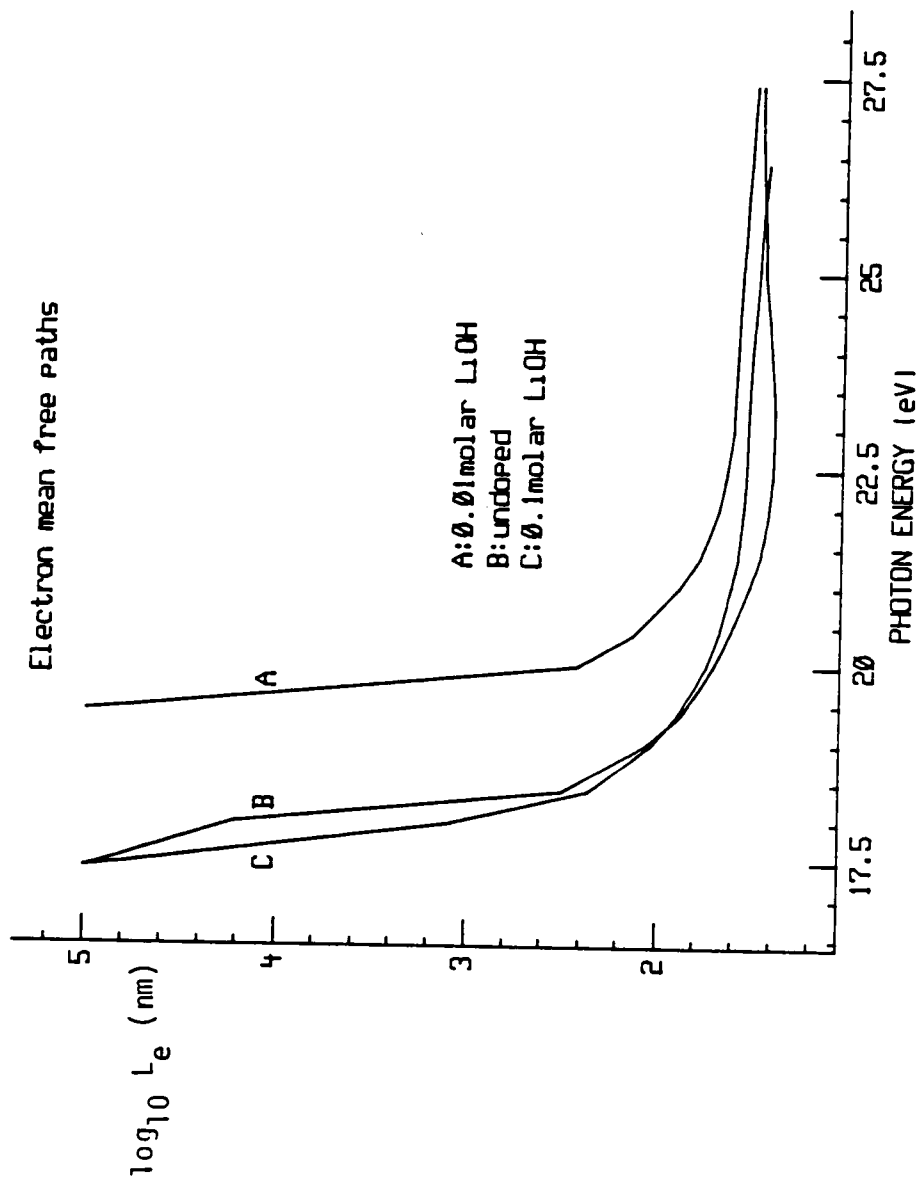


Figure 28. Electron mean free paths of doped and undoped triethylene glycol.

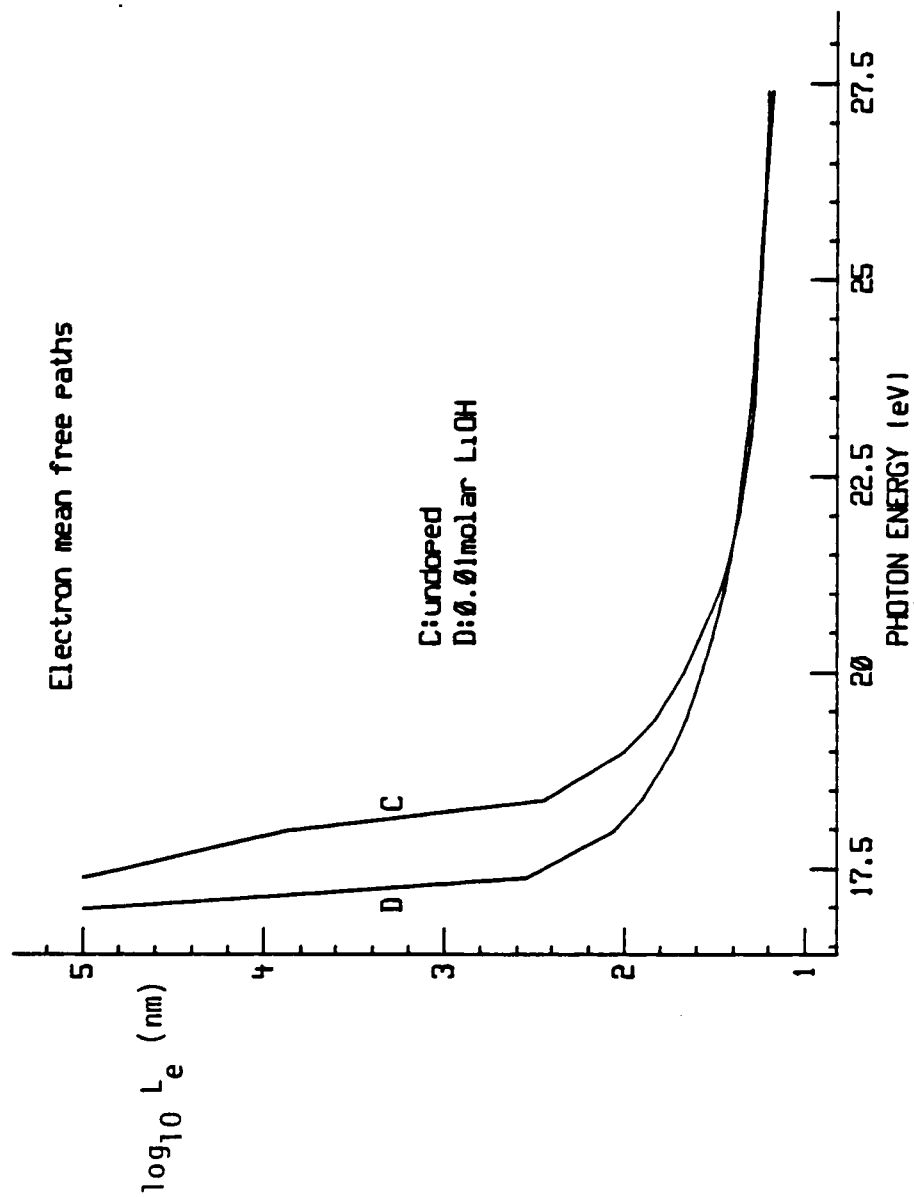


Figure 29. Electron mean free paths of doped and undoped diethylene glycol.

CHAPTER V

CONCLUSION

The doping in diethylene glycol, triethylene glycol, and tetraethylene glycol increases the conductivity to reduce the effects of surface charge. The addition of alkali salts increases the conductivity by orders of magnitude and increases speed of removing the charge away from liquid surfaces. Our measurements indicate that the effects of doping with alkali salts on diethylene glycol, triethylene glycol, and tetraethylene glycol are small. The photoyields of these n-ethylene glycols doped and undoped are shown from Figures 18 to 23. The monotonic behavior can be seen with values in the neighborhood of 15% to 20%. The increase in the photoyields for both doped and undoped is shown in the photon energy region 16 eV to 30 eV.

Electron mean free paths determined from the Seah and Dench empirical equation for organics and from the model insulator theory of Ashley and Williams is shown in Figure 3 (p. 19). The simple three-step model for photoemission was used earlier in calculation of photoyields from optical data for diethylene glycol, triethylene glycol, and tetraethylene glycol. The photoyields are higher than we expected so that the electron mean free paths calculated from experimental data by using Eq. (20) exceed those predicted from the Seah and Dench empirical equation. In the calculation of electron mean free paths from Eq. (20), the parameter ϕ (the photoelectric threshold

energy) causes the relation between photoyields and the electron mean free paths to be broken down. Two papers (Nemec, 1975; Casanovas, 1981) on photoemission into vacuum give 8.0 eV for 11 organics, but it is lower than 8 eV according to our experimental data. From the last chapter, we found that the better values for photoelectric threshold energy were less than 8 eV or larger than 7 eV. All experimental data support these results.

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