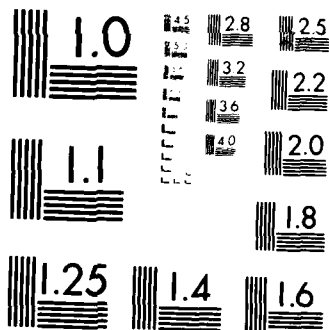


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IONIZATION ENERGIES OF LIQUIDS FROM ENERGY DISTRIBUTION,
QUANTUM YIELD AND SECOND DERIVATIVE CURVES

by

K. P. Cheung, I. Watanabe, A. Dziedzic, K. von Burg and P. Delahay

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New York, NY

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IONIZATION ENERGIES OF LIQUIDS FROM ENERGY DISTRIBUTION, QUANTUM YIELD AND SECOND DERIVATIVE CURVES

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(Received)

ABSTRACT

The energies of the lowest ionization band of eight liquids of low vapor pressure are determined from energy distribution curves (EDC), quantum yield spectra (collected electrons per incident photon as a function of photon energy), and second derivative curves (SDC) of retarding potential curves. Threshold energies from EDC's and quantum yield spectra agree if one takes into account a 0.15 eV shift caused by the spectrometer's rather low resolution and a small difference (0.15 eV or less) resulting from the use of approximate extrapolation methods. Threshold energies from EDC's and SDC's agree to within 0.1 eV after correction for the half-width of the high-energy branch of SDC's. Multiple ionization bands are exhibited by the SDC's of some of the liquids, and the observed splittings agree well with the results from gas-phase UPS spectra. A new spectrometer for the measurement of EDC's of liquids is described. The liquids studied are 6-chloro-1-hexanol, 2-ethyl-1-hexanol, ethylene cyanohydrin, ethylene glycol, 1,5-pentanediol, tetraglyme, triethylene glycol, tetraethylene glycol.

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INTRODUCTION

Three methods are available for the determination of the lowest ionization energy for photoelectron emission by liquids and solutions: (i) the determination of the quantum yield γ for emission as a function of photon energy E , that is, the measurement of the number of emitted electrons per incident photon as a function of E ; (ii) the determination of the kinetic energy distribution curve (EDC) of the electrons emitted into the gas phase upon irradiation at a fixed photon energy; (iii) the determination of second derivative curves (SDC), that is, the derivative of EDC's with respect to retarding potential. The quantum yield method is of general scope whereas EDC and SDC determinations are restricted to liquids having such low vapor pressure that electron-molecule inelastic scattering is negligible in the gas phase. The three methods have been applied [1], the quantum yield method to aqueous solutions of inorganic salts, the EDC method to various organic liquids, solutions of organic anion radicals and solvated electrons, and the SDC method to organic liquids. Determination of ionization energies by the three methods for the same liquid has not been reported to our knowledge. This comparison is made in the present paper for eight different liquids. The limitations of quantum yield and EDC methods are also examined, and the usefulness of second derivative curves (SDC) is demonstrated in the comparison with gas-phase UPS spectra.

EXPERIMENTAL

EDC's were measured with a rotating disk target [2-4] and a newly designed optical system (Fig. 1). The exit beam of monochromator A was focused through the lithium fluoride window B on the rim of the rotating disk target C. The monochromator was a McPherson instrument, model 218, with a

1200-groove/mm grating blazed at 150 nm. The source of radiation was a modified McPherson model 630 Hinteregger hydrogen lamp with a hot filament. The window B separated the monochromator and the mirrors D (coated with MgF_2 and optimized at 120 nm) from the target chamber filled with the organic vapor from the liquid sample. The target chamber was continuously evacuated with a 4-inch diffusion pump and a cryogenic pump J. The latter was a copper cylinder (9 cm radius, 20 cm long) maintained at -30°C by circulating coolant. The attenuation of the photon flux in the vapor phase was quite negligible because of the prevailing very low vapor pressure (10^{-3} torr). The photon flux was monitored by means of the converter E (platinum wire coated with sodium salicylate) and the photomultiplier F (Hamamatsu model R647). The stray light level was also taken into account.

The target C was an hexagonal disk (0.25 inch thick, 1.5 inch diameter). The focused light spot on the rim of the disk was 2×8 mm. Measurements were synchronized with rotation (7.2 Hz) of the hexagonal disk in such a way that the liquid film being irradiated was horizontal when data were taken. The liquid was cooled by a coldfinger in contact with the external bottom of the sample container. The temperature of the liquid was monitored by a thermocouple and was held constant within 0.2 degree. Operation near the freezing point of the liquid was avoided to prevent accidental freezing of the liquid surface as a result of evaporation.

Retarding potential curves at given photon energies were obtained by varying the potential of the hemispherical collector grid G with respect to the liquid. The sample was made conductive by addition of a trace of lithium chloride or iodide (10^{-4} M). The contribution to photoelectron emission from the added salt was totally negligible. Contact with the liquid was made by a silver electrode coated with silver chloride or iodide to achieve a

stable electrode potential in the dilute halide solution in the target container. Escape of energetic electrons through the grid G was prevented by the grid H maintained at -5 V with respect to the collector grid G. Gas-phase photoionization in the target chamber was negligible because of the very low vapor pressure.

EDC's were obtained from retarding potential curves by analog differentiation through superposition of a low (0.2 V peak to peak) AC voltage on the DC retarding potential. The resulting AC component of the collected current was measured while the DC retarding potential was scanned, and plots of the AC signal as a function of the DC potential were taken as the EDC [4]. The entire experiment was automated and controlled by a Digital Equipment Corp., model PDP-11/34 computer. Nine scans were obtained for each EDC with the same sample under identical conditions, and each point on the curves of Fig. 2 represents the average of the nine measurements.

The resolution of the spectrometer expressed as the full width at half-maximum of a band was estimated [4] at ca. 0.5 eV. The resolution of the analyzer was ca. 0.3 eV, which is typical [5] for the retarding potential method. The other contribution arose from the monochromator whose slits were open at 2 mm (to achieve a sufficient photon flux).

Quantum yield spectra were determined with the equipment used in earlier work [6] after numerous improvements [7]. The instrument resolution was better than 0.1 eV at 10 eV and lower photon energies.

IONIZATION ENERGIES

Energy Distribution Curves

The EDC's (Fig. 2) of eight liquids selected for their very low vapor pressure were determined under the conditions of Table 1. The zero of the abscissa scale was taken at the inflection point of the EDC ascending branch

in agreement with standard practice in the study of photoelectron emission by condensed phases (see, e.g., ref. 8 for molecular crystals). The threshold energy E_t (Table 1) for the lowest ionization band of the liquid was determined from the relationship, $E_t = E - T_{\max}$, where E is the photon energy and $T_{\max}$ the kinetic energy obtained by extrapolation of the essentially linear segment in the high-energy tail of the EDC (Fig. 2). This procedure follows directly from the Brodsky-Tsarevsky theory of photoelectron emission by liquids [9,10]. The extrapolation strictly holds for measurements of the kinetic energy corresponding to the velocity component of electrons in the gas phase which is normal to the emitting liquid surface. Brodsky has argued [9,10] that experimental EDC's of liquids obtained by the retarding potential method essentially satisfy the foregoing conditions.

Comparison of the E_t -values from EDC's in Table 1 is possible only for ethylene glycol on the basis of the EDC's in [11,12] obtained at 21.2 eV. Application of the linear extrapolation of the EDC tail to Fig. 7 in [11] yielded $E_t = 8.3$ eV in excellent agreement with the value $E_t = 8.36$ eV in Table I. Conversely, Fig. 2 in [12] yielded the higher value of $E_t = 9.0$ eV. The difference between the energies in [11] and [12] was ascribed in [13] to an inadequate determination of the zero of kinetic energy in [12]. The abscissa of 21.2 eV in Fig. 2 of [12] corresponds to the peak of the EDC whereas one would expect the zero of kinetic energy to be at the midpoint of the ascending branch of the EDC. This is indeed the case for Fig. 7 in [11] and in later work (Fig. 1 in [14]) from the laboratory in which the work of [12] was done. The corresponding shift in Fig. 2 of [12] is ca. 0.8 eV, and the corrected value of E_t is then ca. $9.0 - 0.8 = 8.2$ eV in rather good agreement with the values of 8.3 eV obtained from [11] and 8.36 eV in Table I.

Quantum Yield Spectra

Quantum yield curves, Y against E (Fig. 3), can be approximated [5,9,10] by the functional dependence $(E - E_t)^n$ on photon energy, where E_t is the threshold energy and the exponent n is taken to be 3, 5/2 or 2 depending on the range of E above E_t . The value $n = 3$ holds very near the threshold energy E_t whereas $n = 5/2$ and $n = 2$ apply successively at higher photon energies. Such a functional dependence of the yield Y on photon energy is not rigorous. Moreover, the threshold energy E_t varies with the photon energy E at which Y is measured because of the dielectric dispersion of the liquid [7,15]. Variations of $Y^{1/n}$ with E therefore are modulated by the dispersion effect. Correction of E_t for dispersion is possible [15] from the experimental dependence of the real and imaginary parts of the dielectric constant of the liquid on photon energy, but such data are not available for the liquids studied here. If the fitting range is sufficiently large, the distortion of $Y^{1/n}$ vs. E plots caused by dispersion can be taken to average out, and the emission threshold can be found by a least-square treatment. This was done here at photon energies sufficiently remote from E_t for the value $n = 2$ to hold (Fig. 4). The choice of the exponent n is not critical, and plots of $Y^{2/5}$ against E yielded threshold energies which were only 0.16 eV lower on the average than the values of E_t deduced from the plots of Fig. 4.

Comparison of Threshold Energies from EDC's and Yield Spectra

Threshold energies from EDC's and yield spectra are comparable (Table 1), but the EDC values of E_t are on the average higher by 0.15 eV than the E_t 's from yield spectra. This systematic shift appears to be an artifact of instrumental origin. The resolution of the EDC spectrometer is definitely lower than that of the instrument for the determination of quantum yield

spectra (see Experimental). Hence, the descending tail of EDC's is distorted and poorer in high-energy electrons than it would be with higher instrumental resolution. The segment of the EDC therefore extrapolates to an energy $T_{\max}$ which is too low, and the threshold energy $E_t = E - T_{\max}$ (E , photon energy) is too high. Moreover, differences between the two sets of threshold energies in Table 1 are not surprising since the extrapolation methods by which the E_t 's were obtained are approximate on several counts: (i) The distribution of configuration in the liquid is not considered in the theory of emission in refs. 9 and 10. The EDC's therefore do not fall off until the maximum kinetic energy $T_{\max}$ is reached, as predicted by theory, but exhibit a drawn-out tail (Fig. 2). Selection of the segment of EDC tail taken to be linear is not totally unambiguous (e.g., for the EDC of ethylene cyanohydrin in Fig. 2). (ii) Threshold energies from yield spectra depend somewhat on the range in which $\gamma^{1/2}$ is presumed to vary linearly with E . (iii) These extrapolation methods apply to liquids exhibiting a single ionization band widely separated from higher bands. This condition is not fully satisfied for some of the liquids studied here, as will be shown in the next section, and extrapolation of the EDC and $\gamma^{1/2}$ plots to E_t is affected accordingly.

SECOND DERIVATIVE CURVES AND CORRELATION WITH GAS-PHASE UPS SPECTRA

Second derivative curves [16] (SDC) of retarding potential curves were determined for the liquids of Table 1. The SDC's (Fig. 5) were obtained by numerical differentiation of EDC's using 5-point Savitzky-Golay filters [17,18]. SDC's exhibit a maximum at the kinetic energy $T_{\max}(\text{SDC})$ such that the energy, $E_{\text{SDC}} = E - T_{\max}(\text{SDC})$, is independent of the photon energy E .

According to ref. 16, the energy E_{SDC} is higher than the ionization energy by $0.52w_{1/2}(\text{SDC})$, where $w_{1/2}(\text{SDC})$ is the half-width of the

high-energy descending branch of the SDC. The method of determining $w_{1/2}(\text{SDC})$ is illustrated for curve 6 in Fig. 5. The difference $E_t(\text{EDC}) - E_{\text{SDC}}$ therefore should be proportional to $w_{1/2}(\text{SDC})$ if one identifies the threshold energy $E_t(\text{EDC})$ obtained from EDC's with the ionization energy. This relationship is verified (Fig. 6) quite well for the liquids of Table 1 since a least-square fit of the eight points of Fig. 6 yields a slope of 1.010 ± 0.106 and intercept of 0.067 ± 0.045 . These results can be approximated by the relationship

$$E_t(\text{SDC}) = E_{\text{SDC}} - 1.1w_{1/2}(\text{SDC}). \quad (1)$$

The resulting threshold energies $E_t(\text{SDC})$ agree within 0.1 eV with the E_t 's obtained from EDC (Table 1). The difference between the coefficient 1.1 in eq. (1) and the theoretical value 0.52 in ref. 16 arises from SDC distortion caused by the low spectrometer resolution and the limitations of the classical model used in [16].

SDC's of liquids having well-separated ionization bands exhibit successive bands not unlike the corresponding gas-phase UPS spectra but without the detailed structure observed in the latter. This is not the case for EDC's which generally exhibit (Fig. 2) a single descending branch (except sometimes for EDC's obtained at 21.2 eV [11]). SDC's therefore complement the results obtained from EDC's and quantum yield spectra and they allow comparison with gas-phase UPS spectra.

Multiple bands were evident in the SDC's (Fig. 5) for most of the liquids of Table 1. The SDC of 6-chloro-1-hexanol may be compared with the gas-phase UPS spectrum of chloroethanol [19], and the separation of 0.44 eV between the two SDC maxima is comparable to the difference, $11.45 - 10.90 = 0.55$ eV, between the first two ionization energies of chloroethanol. The SDC of 2-ethyl-1-hexanol is comparable to the gas-phase UPS spectrum of pentanol

[20] inasmuch as the first peak is followed by a very broad band consisting of overlapping bands typical of alcohols. The SDC of ethylene glycol exhibits two maxima separated by 0.63 eV whereas the first two gas-phase ionization energies differ by 0.67 eV [12,21]. The SDC of tetraglyme may be compared with the gas-phase UPS spectrum of ethylene glycol dimethyl ether because of the similarity in structure, namely $\text{H}(\text{CH}_2\text{OCH}_2)_5\text{H}$ (tetraglyme) and $\text{H}(\text{CH}_2\text{OCH}_2)_2\text{H}$. The five oxygen atoms in the tetraglyme are the counterparts of the two oxygen atoms in the ether. The gas-phase UPS spectrum of the ether exhibits [21] a lone pair splitting of 0.28 eV, and therefore the very broad SDC band of tetraglyme is assigned to five closely spaced overlapping bands.

CONCLUSIONS

(i) Threshold energies obtained from EDC's and quantum yield spectra agree except for a small shift (0.15 eV) arising from the spectrometer's rather low resolution and a small difference (0.15 eV or less) introduced by the approximate extrapolation methods thus applied. (ii) Threshold energies from EDC's and from SDC's agree to within 0.1 eV after correction for the half-width of the high-energy branch of the SDC. (iii) Good correlation exists between the second derivative curves of liquids and the corresponding gas-phase UPS spectra.

ACKNOWLEDGEMENTS

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TABLE 1

THRESHOLD ENERGIES FROM EDC'S, $\gamma^{1/2}$ VS. E PLOTS AND SDC'S

symbol in Fig. 2	liquid	temp ^a (EDC) (°C)	photon energy (eV)	E_t from EDC (eV)	E_t from yield spectrum (eV)	E_{SDC} (eV)	E_t^b from SDC (eV)
1	6-chloro-1-hexanol	-14	10.6	9.14	8.86	9.30	9.15
2	2-ethyl-1-hexanol	-17	10.2	8.76	8.45	9.05	8.85
3	ethylene cyanohydrin	-14	10.4	9.31	9.04	9.85	9.41
4	ethylene glycol	-11	10.2	8.36	8.04	8.76	8.43
5	1,5-pentanediol	-9	10.6	8.67	8.65	9.05	8.63
6	tetraglyme ^c	-14	10.0	8.09	8.03	8.71	8.09
7	triethylene glycol	-4.5	9.6	8.05	8.22	8.63	8.06
8	tetraethylene glycol	-3	10.0	8.13	8.04	8.85	8.15

^aYield spectra obtained at 0°C. Corresponding vapor pressures (in torr) from data in [22-24]: $<10^{-1}$ (1), $<10^{-2}$ (2), ca. 1 (3), $<10^{-2}$ (4), $<10^{-1}$ (5), $<10^{-4}$ (6-8).

^bfrom eq. (1).

^ctetraethylene glycol dimethyl ether.

Figure Legends

Figure 1. Optical system for the determination of EDC's. A, vacuum UV monochromator; B, lithium fluoride window; C, hexagonal rotating disk target; D, folding and focusing mirrors; E, platinum wire coated with sodium salicylate; F, photomultiplier; G, collector grid; H, grid electrode at -5V with respect to G; J, cryogenic pump.

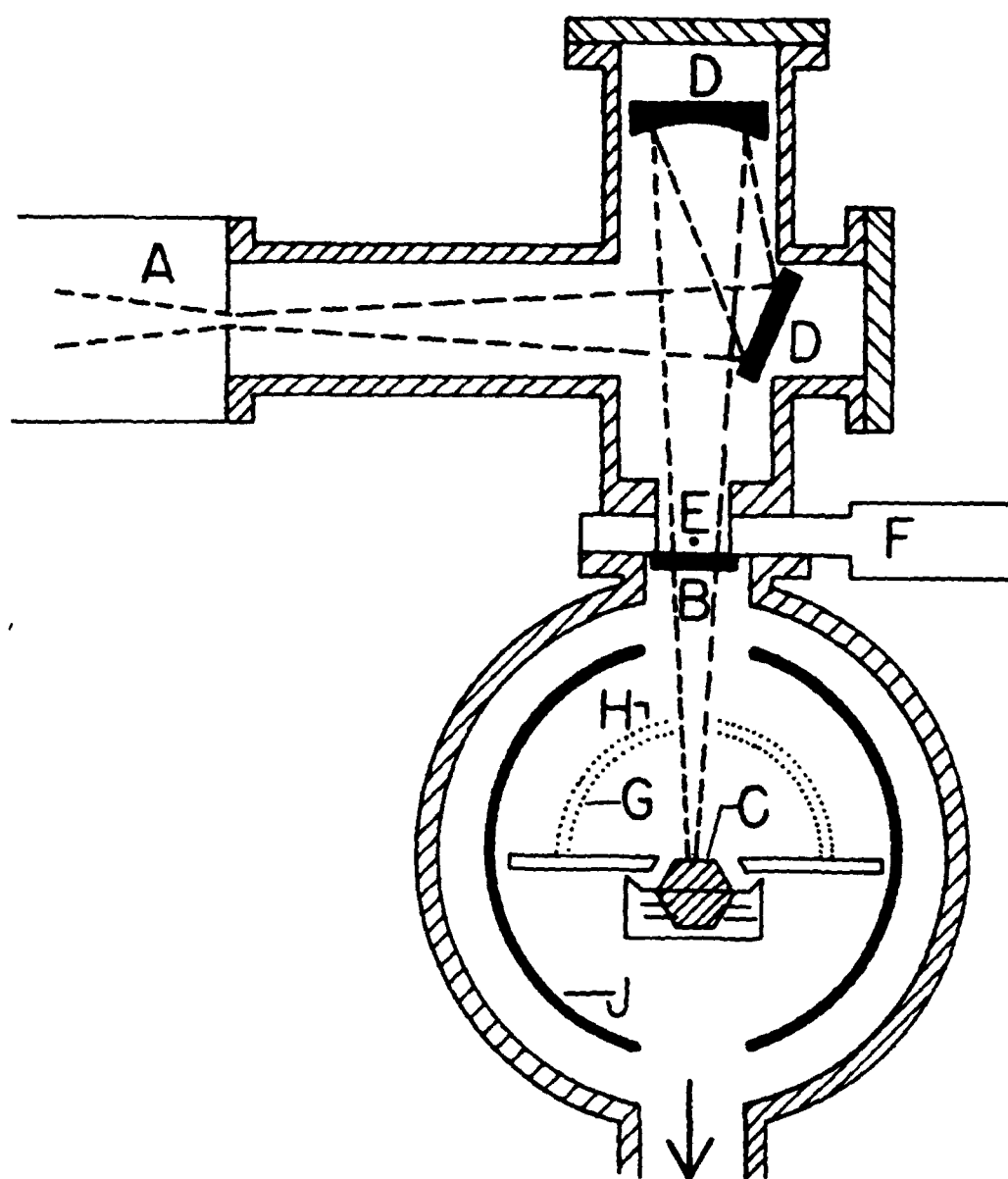
Figure 2. EDC's of the liquids of Table 1 and extrapolation to the kinetic energy $T_{\max}$. Zero of ordinate scale shown for each EDC.

Figure 3. Quantum yield Y (electrons collected per incident photon) against photon energy E for the liquids of Table 1. Zero of ordinate scale shown for each curve.

Figure 4. Plot of $Y^{1/2}$ against photon energy E for the data of Figure 3 and the liquids of Table 1. Zero of ordinate scale and extrapolation to the threshold energy E_t shown for each liquid.

Figure 5. SDC's of some of the liquids of Table 1. The kinetic energy $T_{\max}$ (SDC) displayed for each maximum. Zero of ordinate scale shown for each SDC. Determination of $w_{1/2}$ (SDC) of eq. (1) shown for curve 6.

Figure 6. Correction for the half-width $w_{1/2}$ (SDC) of SDC's. Straight line from a least-square fit.



TO 4-INCH DIFFUSION PUMP

FIG. 1

ELECTRONS PER INCIDENT PHOTON PER ELECTRONVOLT
(ARBITRARY UNITS)

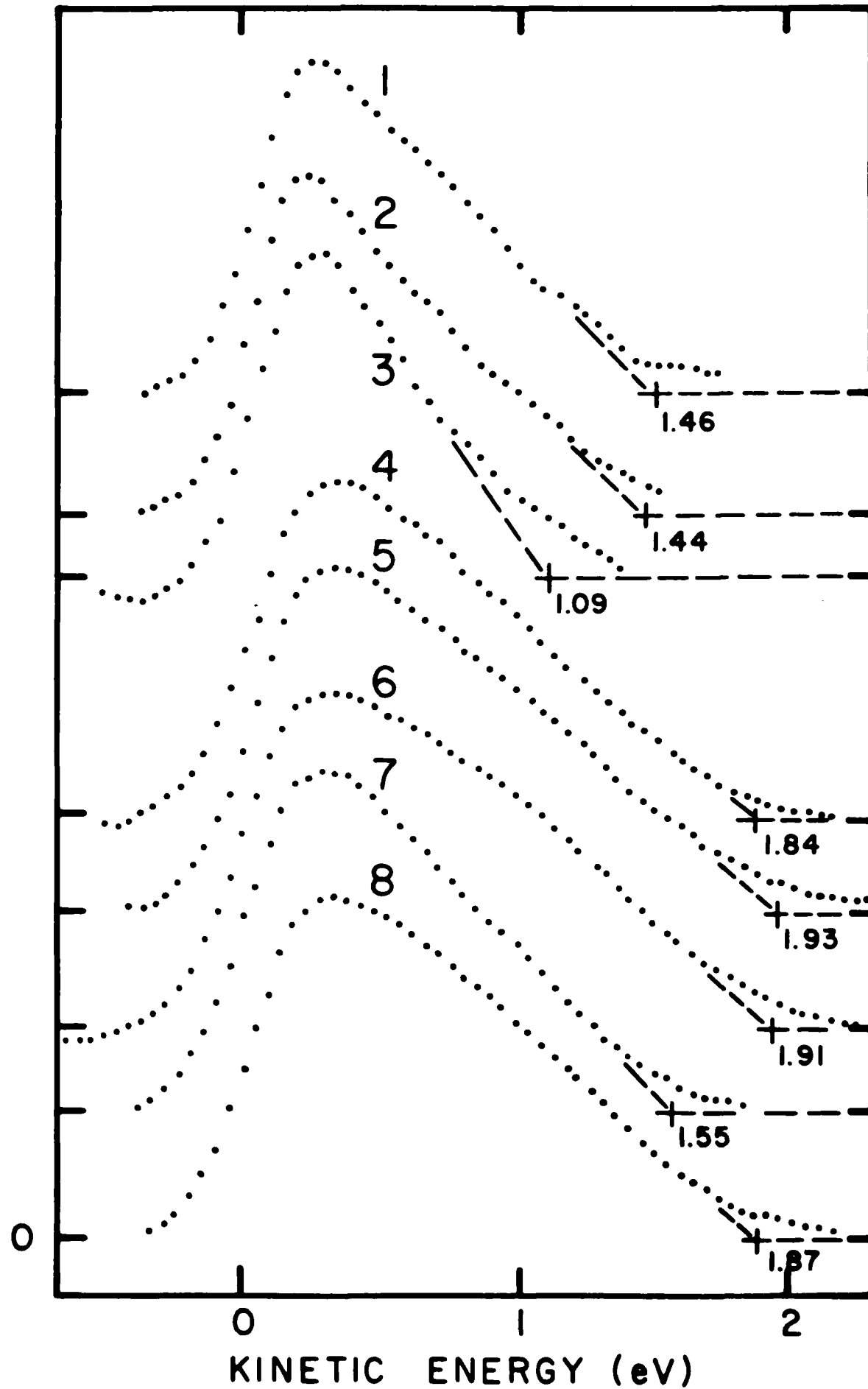


FIG. 2

YIELD (ELECTRONS PER INCIDENT PHOTON, ARBITRARY UNITS)

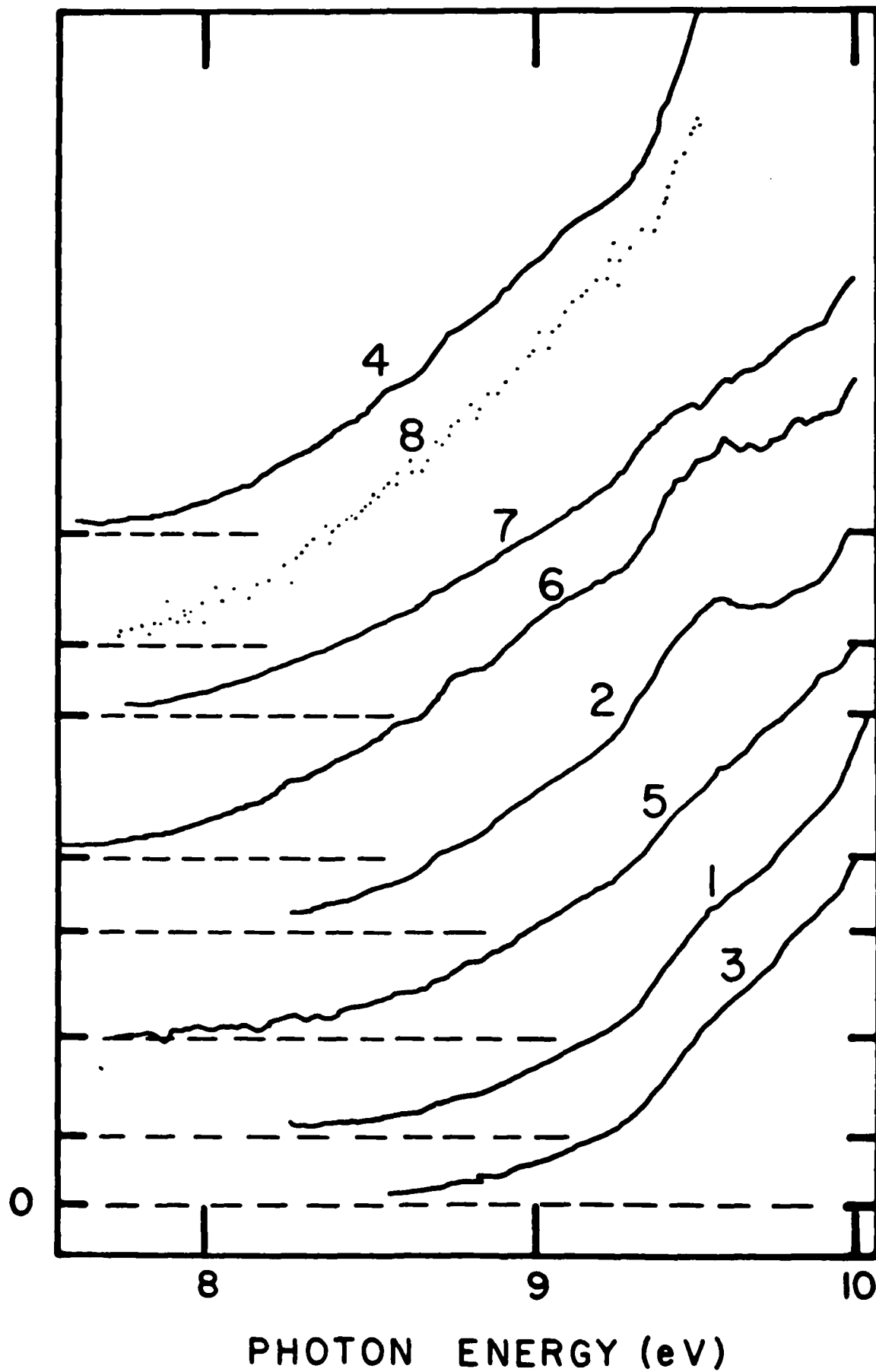


FIG. 3

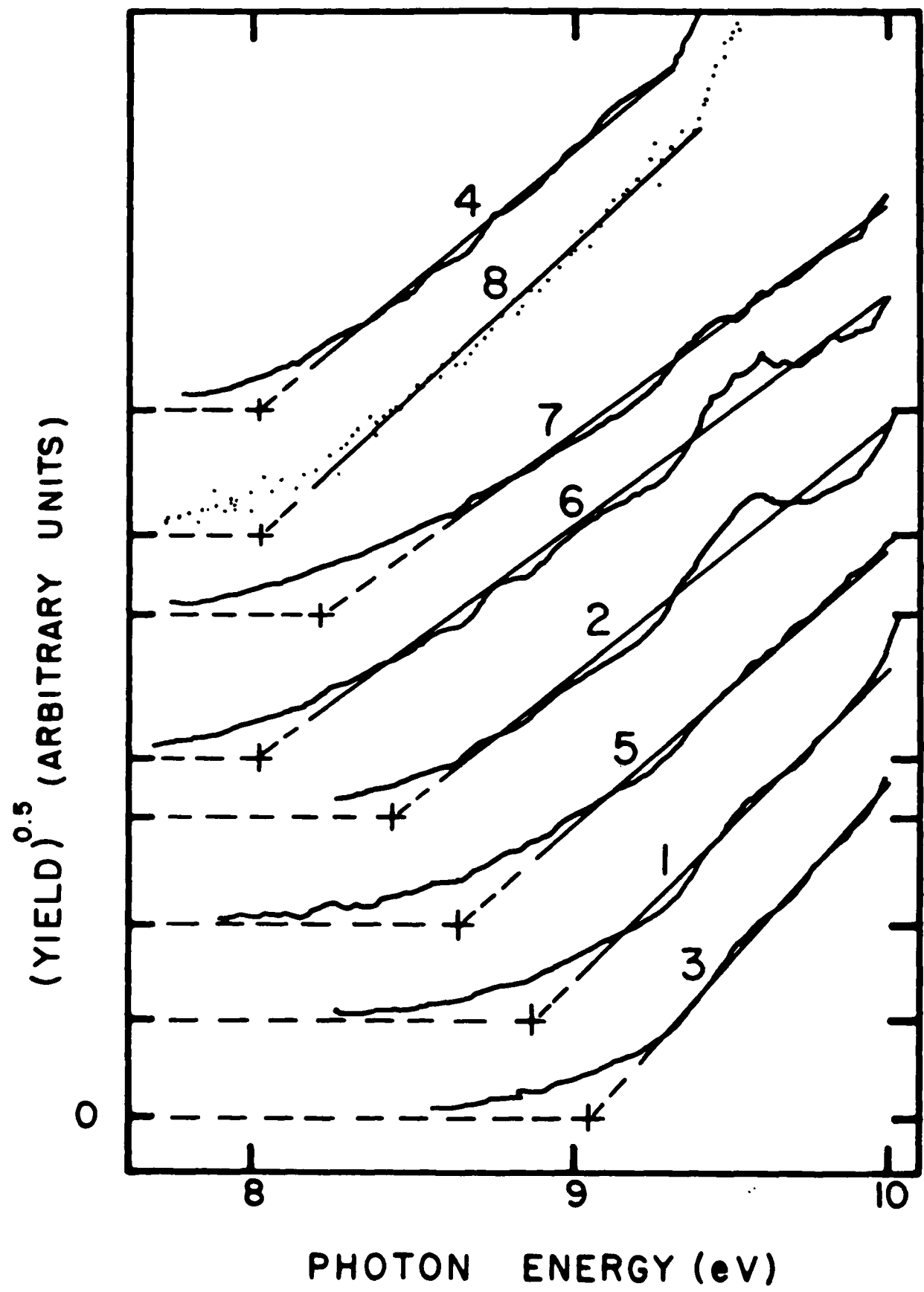


FIG. 4

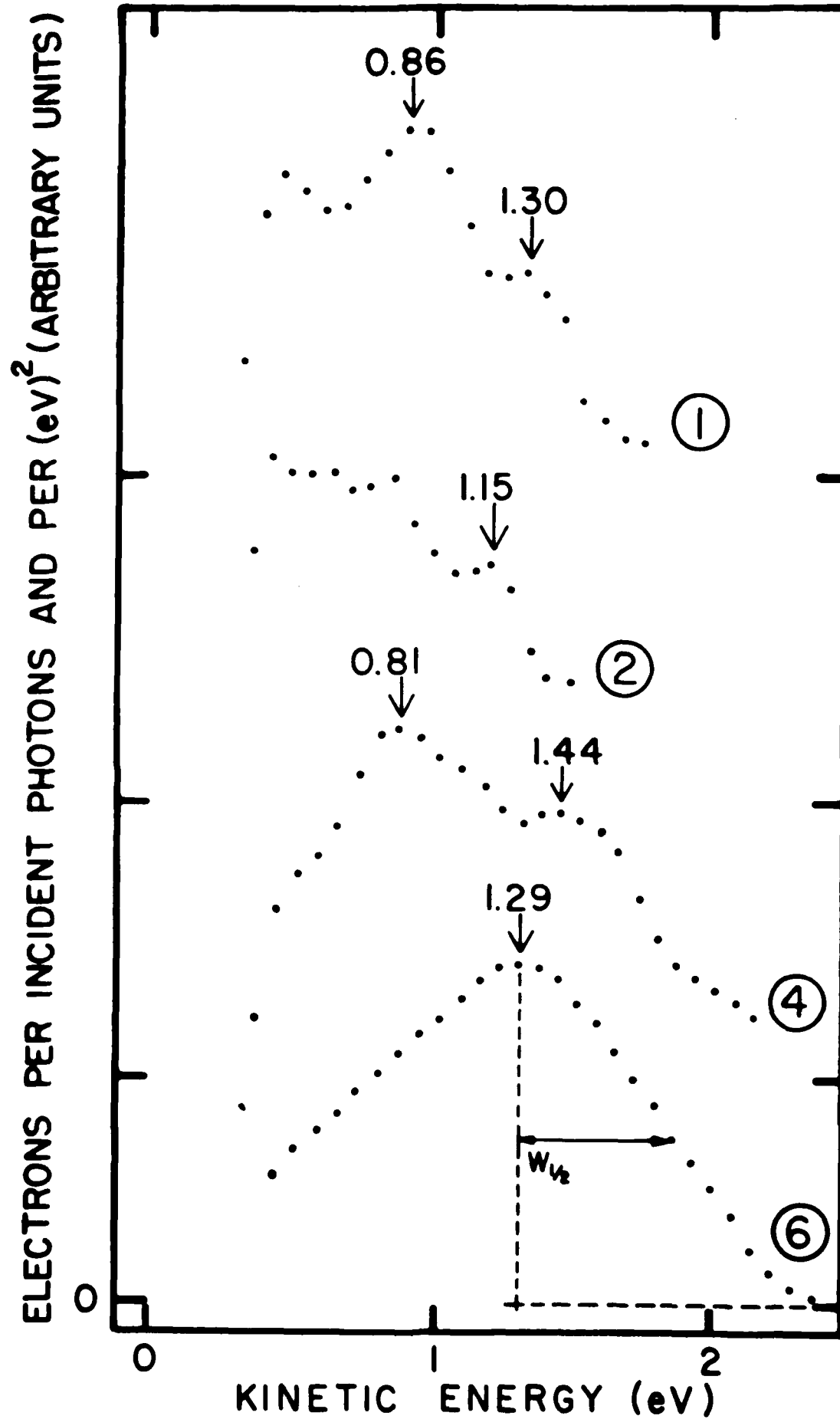


FIG. 5

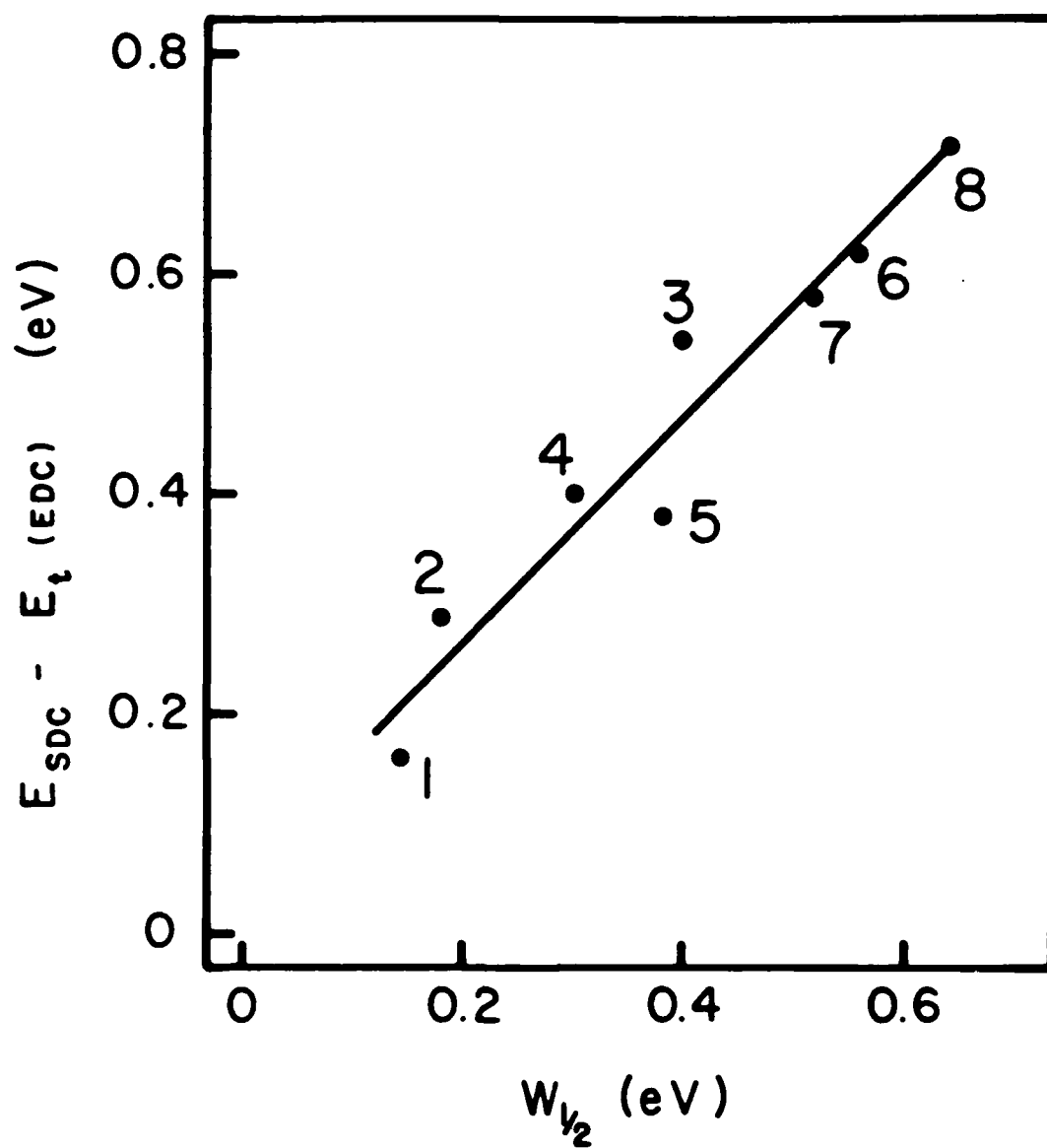


FIG. 6

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