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6 Photoelectron Emission Spectroscopy of Weak Acids and Bases and Their Ions in Aqueous Solution,

by

10 Kathrin von Burg [redacted] Paul Delahay

Prepared for Publication

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PHOTOELECTRON EMISSION SPECTROSCOPY OF WEAK ACIDS AND BASES AND THEIR IONS IN AQUEOUS SOLUTION

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Threshold energies E_t are determined for 14 weak acids and three bases and their ions. Values of E_t are interpreted in terms of dissociation or protonation reactions involving species produced by photoionization. Electron transfer to weak acid molecules in solution is shown to occur for emission by inorganic anions and cations and weak acids.

The threshold energies E_t for photoelectron emission by aqueous solutions of a few anions of weak acids were reported in earlier papers [1,2]. No correlation of the E_t values was attempted at that stage. This is done in the present paper on the basis of rather extensive data for weak acids and bases and their ions. Weak acids will also be shown to be excellent electron acceptors in solution.

1. Weak acids and their anions

The experimental methods developed in [1] and applied in [2-4] were used in the present work. Emission spectra obtained for aqueous solutions of acids and their salts displayed the yield Y (collected electrons per incident photon) as a function of the photon energy E. The threshold energies E_t^j (table 1) were obtained by extrapolation of the linear plots Y^n vs. E to Y = 0 for n = 0.5 or 0.4 [5]. The choice of exponent is discussed in [2-5]. The yield for formic acid (4 M) was very low and only about twice that for pure water (E_t = 10.06 eV [4]).

The threshold energies will be interpreted on the basis of the equation [2-4],

$$\Delta G_e^j = \Delta G_H + \Delta G^j - \Delta G_r^j, \qquad (1)$$

for the free energy of emission ΔG_e^j of the species $H_{n-j}A^{j-}(aq)$ (n = 1 to 3, j = 0 to 3). The quantity $\Delta G_H = 4.50$ eV [6] is the free energy of formation of the electron in vacuum on the assumption that the surface potential at the solution-water vapor interface is negligible (± 0.1 V for water [7]). ΔG^j is the change of free energy for the reaction

$$H_{n-j}A^{j-}(aq) + H^+(aq) = H_{n-j}A^{(j-1)-}(aq) + 1/2H_2(g), \qquad (2)$$

and ΔG_r^j (< 0) is the reorganization free energy for the process in which there is vibrational relaxation and the solvation nuclear configuration adjusts from j- to (j-1)- ionic charge.

Equation (1) will be applied first to the acid H_nA and its anion $H_{n-1}A^-$. One has (fig 1)

$$\Delta G_e(H_nA) - \Delta G_e(H_{n-1}A^-) = [\Delta G(H_{n-1}A^-) - \Delta G(H_nA)]$$
$$+ [\Delta G(H_nA^+) - \Delta G(H_{n-1}A)] + [\Delta G_r(H_{n-1}A) - \Delta G_r(H_nA^+)] \qquad (3)$$

where the symbol $\Delta G(X)$ represents the free energy of formation of species X in aqueous solution, and the ΔG_r's are the reorganization free energies. Equation (3) is similar to the corresponding relationship for water and hydroxide ion [4]. Each of the three terms on the right hand side of eq 3 will be discussed.

One has

$$\Delta G_K^1 = \Delta G(H_{n-1}A^-) - \Delta G(H_nA)$$
$$= - 0.0592 \log K_1 \quad , \qquad (4)$$

where ΔG_K^1 is expressed in electronvolts and K_1 is the first dissociation constant of H_nA. One has $0.1 < \Delta G_K^1 < 0.3$ eV for the acids of table 1. The third terms between brackets on the right hand side in eq (3) pertains to the change of ionic charge from -1 to 0 and 0 to 1 in the photoionization of $H_{n-1}A^-$ and H_nA, respectively. The ions $H_{n-1}A^-$ and H_nA^+ have comparable sizes although the latter is presumably slightly bulkier than the former. Contributions from vibrational relaxation should nearly cancel out in the difference of ΔG_r's. Since the ΔG_r's are negative, one concludes on the basis of ionic sizes and equality of charge in absolute value that the third term in eq (3) is negative and small (a few tenths of electronvolt).

Equation (3) can be rewritten in the form,

$$\Delta^1 = E_t^o - E_t^1 - \Delta G_K^1$$
$$= [\Delta G(H_nA^+) - \Delta G(H_{n-1}A)] + \Delta^1(\Delta G_r), \qquad (5)$$

where the free energies of emission are equated to the threshold energies [2-4] and the difference of reorganization free energies is denoted by $\Delta^1(\Delta G_r)$. The superscripts o and 1 correspond to H_nA and $H_{n-1}A^-$, respectively. The first term between brackets in eq (5) is the change of free energy for the protonation reaction,

$$H_{n-1}A + H^+ = H_nA^+, \qquad (6)$$

involving the species produced by photoionization of $H_{n-1}A^-$ and H_nA, respectively. The ion H_nA^+ is thermodynamically unstable and the radical $H_{n-1}A$ does not protonate to any significant extent if the condition, $\Delta^1 - \Delta^1(\Delta G_r) > 0$, is satisfied (eq (5)). Since $\Delta^1(\Delta G_r)$ is negative and $\Delta^1 > -0.2$ eV, the foregoing conclusion is fulfilled for all the acids of table 1, albeit marginally for maleic acid. Thus, photoionization of a weak acid produces an ion which dissociates quite completely into a H^+ ion and the radical $H_{n-1}A$. This result may be compared with the frequently observed enhancement of the strength of a weak acid upon excitation to its first electronic excited state [8].

Equation (5) is readily generalized (fig 1). The difference of reorganization free energies $\Delta^2(\Delta G_r)$ pertains to the photoionization of $H_{n-2}A^{2-}$ and $H_{n-1}A^-$, respectively. Since these two ions have comparable sizes, one expects from the Born equation that the ratio of ΔG_r's be approximately equal to the square of the ratio of ionic charges in the absence of dielectric saturation. The Born equation is approximate and there is saturation [3,6], especially for divalent ions, but one can safely conclude that $\Delta^2(\Delta G_r)$ is negative and not negligible. Hence, the condition $\Delta^2 - \Delta^2(\Delta G_r) > 0$ is satisfied for all the acids of table 1. The radical $H_{n-1}A$ produced by photoionization of $H_{n-1}A^-$ ion is unstable and the ion $H_{n-2}A^-$ resulting from photoionization of $H_{n-2}A^{2-}$ does not protonate to any extent. The same analysis and conclusion are applicable to photoelectron emission by the ions HA^{2-} and A^{3-} of table 1.

2. Weak bases and their cations

Weak bases in aqueous solution have lower threshold energies than their cations (table 1). One has (cf. eq (3)) for the base B and its cation BH^+

$$\Delta G_e(B) - \Delta G_e(BH^+) = [\Delta G(BH^+) - \Delta G(B)] + [\Delta G(B^+) - \Delta G(BH^{2+})] + [\Delta G_r(BH^{2+}) - \Delta G_r(B^+)], \qquad (7)$$

where $\Delta G(X)$ represents the free energy of formation of X in aqueous solution. The first term (< 0) on the right hand side of eq (7) is (in eV)

$$\Delta G_K^1 = \Delta G(BH^+) - \Delta G(B) = -0.0592 \log K_p \ , \qquad (8)$$

where K_p is the equilibrium constant for the protonation reaction $B + H^+ = BH^+$. The difference between the reorganization free energies $\Delta^1(\Delta G_r)$ is negative and not negligible because of the difference in ionic charge (and despite dielectric saturation).

One has

$$\Delta^1 = E_t^1 - E_t^o + \Delta G_K^1$$
$$= [\Delta G(BH^{2+}) - \Delta G(B^+)] - \Delta^1(\Delta G_r), \qquad (9)$$

where the superscripts o and 1 refer to B and BH^+, respectively, and the first term between brackets on the right hand side is the change of free energy for the protonation reaction, $B^+ + H^+ = BH^{2+}$. This change of free energy, equal to $\Delta^1 + \Delta^1(\Delta G_r)$, is negative for triethylamine and ethylenediamine since $\Delta^1 \leqslant 0.2$ eV for these bases and $\Delta^1(\Delta G_r) \leqslant -0.5$ eV (see above). Thus, the ion B^+ produced by photoionization of B protonates and the cation BH^{2+} is stable provided the pH is not too high. A similar condition is reached for the photoionization of the ion BH_2^+ of ethylenediamine (table 1). The quantity $\Delta^1 + \Delta^1(\Delta G_r)$ for aniline is probably positive or close to zero since $\Delta^1 = 0.8$ eV and protonation of B^+ must be minor even in strongly acidic solution. Thus, photoionization of a weak base B (aniline) produces an ion B^+ which is a weaker base than B. A similar effect is frequently observed as a result of the excitation of a weak base to its first electronic excited state [8].

3. Weak acids as electron acceptors in electron transfer reactions

The emission yield Y of a solution is proportional to the emitter concentration in the absence of complications [1-5]. This proportionality relationship also holds for the squared slope S^2 of the linear extrapolation plot Y vs. $E^{0.5}$ (sec 1, n = 0.5 here). The plots of the squared slope S^2 against acid concentration for acetic or propionic acid actually exhibit a maximum (fig 2). This result, as was recently shown [9], is typical of electron transfer between the emitting species and an electron acceptor upon absorption of a photon. The emitter and electron acceptor are different molecules of the same acid in the present case. Thus, some electrons which would be emitted into the vapor phase in the absence of electron transfer in solution are not emitted and the yield is lowered as a result of electron transfer.

The emission yield with electron transfer in solution was shown [9] to obey the following equation to a good approximation

$$Y = kC_m \exp(-C_a/C_c). \tag{10}$$

There k is a proportionality constant; C_m and C_a are the emitter and acceptor concentration, respectively; and C_c is a concentration which is characteristic of the kinetics of electron transfer. Dissociation of the acids of fig 2 can be neglected, and one has $C_m = C_a = C$ to a good approximation, where C is the analytical acid concentration. The plot of Y (or S^2) vs. C exhibits a maximum at $C = C_c$ according to eq (10), and the plot of log (S^2/C) vs. C is linear. These predictions are verified in fig 2. The concentration $C = C_c$ at the maximum of the S^2 vs. C plot defines a volume $1/C_c$ in which electron transfer is supposed to occur according to the model leading to eq (9) [9]. One has ca. v = 0.4 nm^3 for the data of fig 2, that is, a radius of ca. 0.5 nm if the volume v is supposed to be spherical. This length can be interpreted as the separation of two acid molecules undergoing electron transfer. Caution is in order since a simplified model was assumed in the derivation of eq (10), and the quantity v can be viewed as the product of an actual volume and a probability of electron transfer [9].

Emitter and acceptor need not be the same acid. Thus, the yield of acetic acid is lowered by addition of formic acid. The latter does not contribute to emission since it has a higher threshold energy (10.0 eV, table 1) than acetic acid (9.00 eV). Log S^2 at constant (2 M) acetic acid concentration indeed was found to decrease linearly with formic acid concentration from 0 to 7 M.

Aliphatic acids also lower the emission yield of inorganic anions (fig 3) and cations (fig 4). The slope S to the power 1/n is proportional to the emission yield, where n is the exponent in the extrapolation plot Y^n vs. E of the anions or cations. Thus, the plots of log $S^{1/n}$ vs. acceptor concentration at constant emitter concentration in fig 3 and 4 are linear as expected from eq (10). A strong anion effect on the rate of electron transfer is displayed in fig 3 just as in a similar plot in [9] for emission by halides in presence of Ba^{2+} ions (electron acceptor).

The results of fig 4 for Tl^+ ion are the first ones obtained for the lowering of the emission yield of a cation. The Tl^+ ion was selected because complexation with acetic or formic acid is negligible ($K_1 = 10^{-0.11}$ for acetic acid [10]). Lowering of the emission yield of anions by electron transfer to cations is common [9], e.g., the effect of Fe^{2+} on emission by Cl^-. The Fe^{2+} ions, however, do not affect emission by other Fe^{2+} ions because of electrostatic repulsion between Fe^{2+} ions (ionic atmosphere). Lowering of the yield of a cation (Tl^+) is observed with an electrically neutral electron acceptor (fig 4). Conversely, the yield of Tl^+ is not lowered by H^+ ion even in 6 M solution of a strong acid (trifluoroacetic), although H^+ ion is an excellent electron acceptor in the emission by anions.

Acknowledgment

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Table 1

Threshold energies and relevant quantities for weak acids and bases and their ions[a]

Acid	E_t^j (eV)	ΔG_K^j (eV)	Δ^j (eV)
formic	10.0 , 7.55	0.22	2.2
acetic	9.00, 7.82	0.28	0.9
monochloroacetic	9.24, 8.50	0.17	0.6
trichloroacetic	9.46, 8.55	0.04	0.9
propionic	9.08, 8.42	0.29	0.4
n-butyric	8.99, 8.23	0.29	0.5
carbonic	- , 9.07, 7.40	- , 0.61	- , 1.1
oxalic	8.26, 7.50, 7.32	0.08, 0.25	0.7, -0.1
tartaric	8.55, 7.72, 7.37	0.18, 0.26	0.6, 0.1
malonic	8.74, 8.33, 7.80	0.17, 0.34	0.2, 0.2
maleic	8.20, 8.31, 8.40	0.11, 0.36	-0.2, -0.4
phosphoric	9.45, 9.23, 8.79, 7.44	0.13, 0.43, 0.73	0.1, 0.0, 0.6
arsenic	9.44, 9.09, 8.50, 8.30	0.13, 0.40, 0.69	0.2, 0.2, -0.5
citric	8.66, 8.52, 8.39, 7.48	0.19, 0.28, 0.38	0.0, -0.1, 0.5
triethylamine	6.73, 7.57	-0.64	0.2
aniline	7.39, 8.44	-0.27	0.8
ethylenediamine	7.20, 7.47, 8.13	-0.59, -0.41	-0.3, 0.3

---

[a] 1 M solutions in all cases except for formic (4 M) and oxalic (0.4 M) acids, oxalate (0.4 M), bitartrate (0.05 M), mono- and dibasic arsenate (0.5 M), tribasic arsenate (0.25 M), aniline (0.05 M).

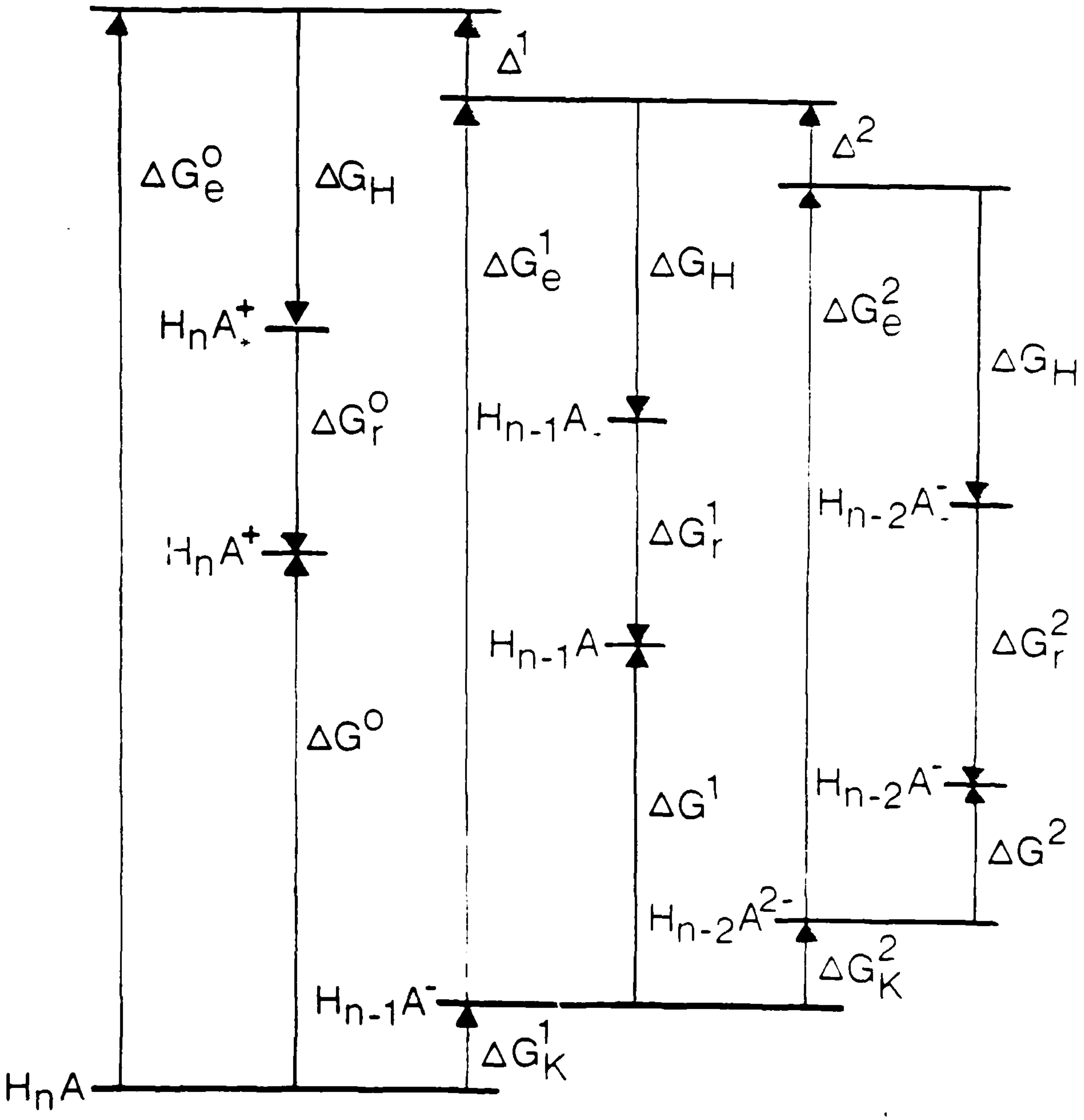
Captions to Figures

Fig. 1. Free energy diagram for photoelectron emission by H_nA, $H_{n-1}A^-$ and $H_{n-2}A^{2-}$. All species shown are in aqueous solution. See text for notations.

Fig. 2. Squared slope $S^2 = (dY^{0.5}/dE)^2$ for emission by acetic (A) and propionic (B) acid vs. acid concentration C. Points are experimental, and the curves were calculated from eq (10). Log (S^2/C) vs. acid concentration for acetic (C) and propionic (D) acids.

Fig. 3. Log $S^{1/n}$ for emission by anions vs. formic acid concentration for 2 M KCl (A), 2 M KBr (B), 2 M KI (C) and 1 M Li_2SO_4 (D). $S = dY^n/dE$ with $n = 0.5$ for Cl^-, SO_4^{2-} and $n = 0.4$ for Br^- and I^-.

Fig. 4. Log $S^{2.5}$ vs. acid concentration for emission by Tl^+ ions (0.25 M $TlClO_4$) in presence of trifluoroacetic (A), formic (B) or acetic (C) acid. $S = dY^{0.4}/dE$ for Tl^+ ion.


ΔG_e^0
ΔG_H
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ΔG_H
$H_{n-1}A_*$
ΔG_r^1
$H_{n-1}A$
ΔG^1
$H_{n-1}A^-$
ΔG_K^1
Δ^2
ΔG_e^2
ΔG_H
$H_{n-2}A_*^-$
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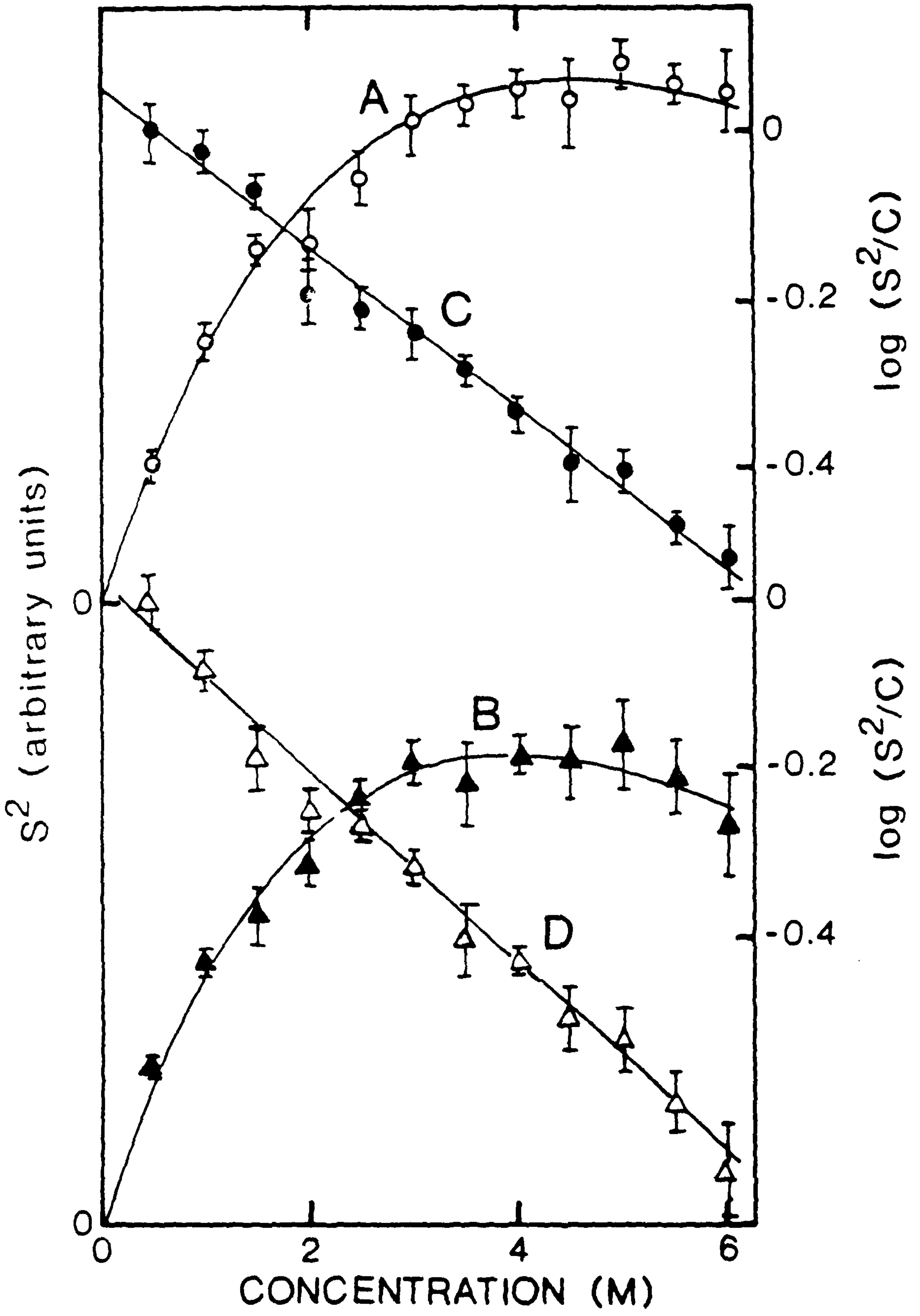
$H_{n-2}A^{2-}$
ΔG_K^2

FIG. 1


A
B
C
D
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CONCENTRATION (M)

FIG. 2

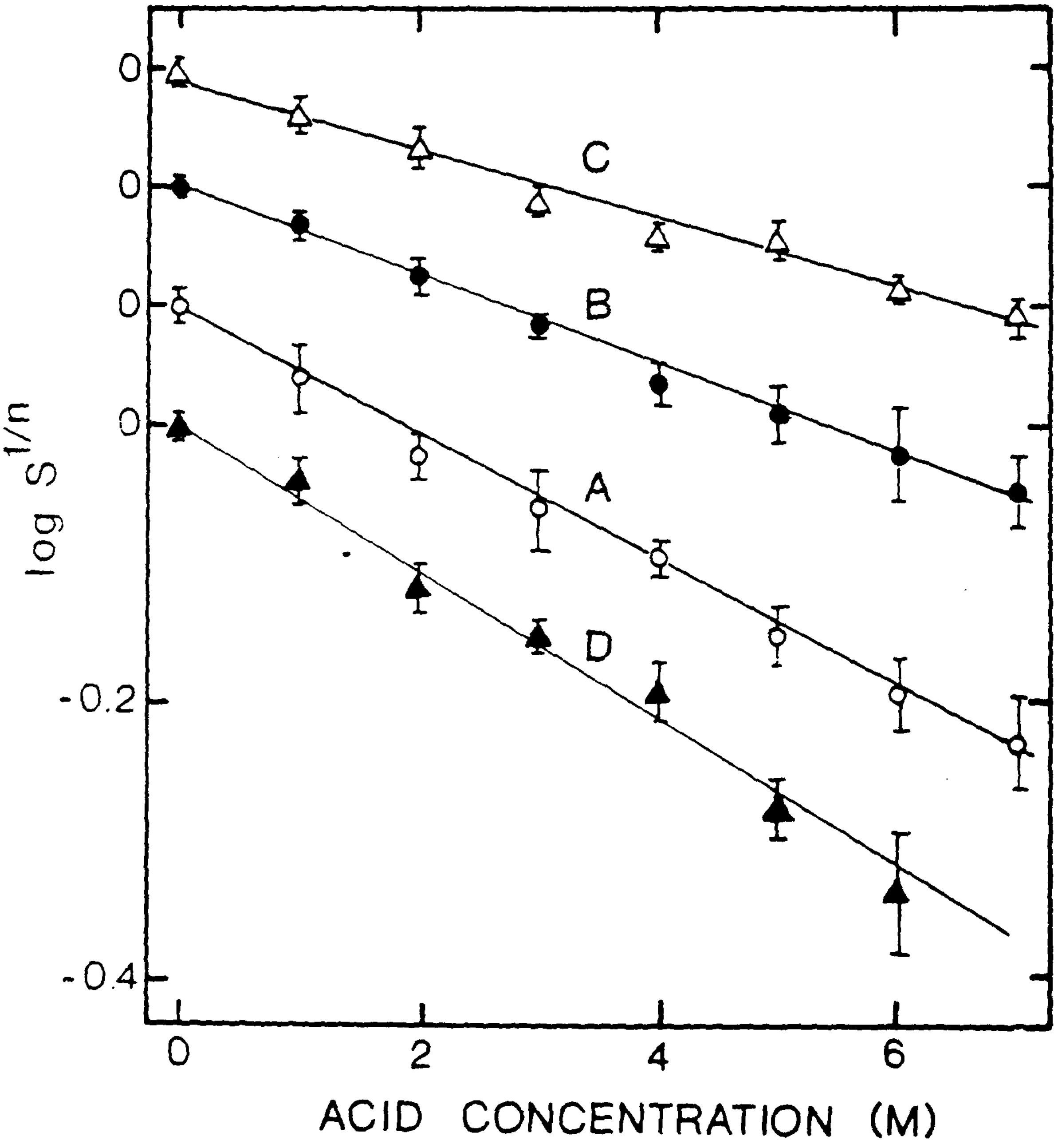

FIG. 3

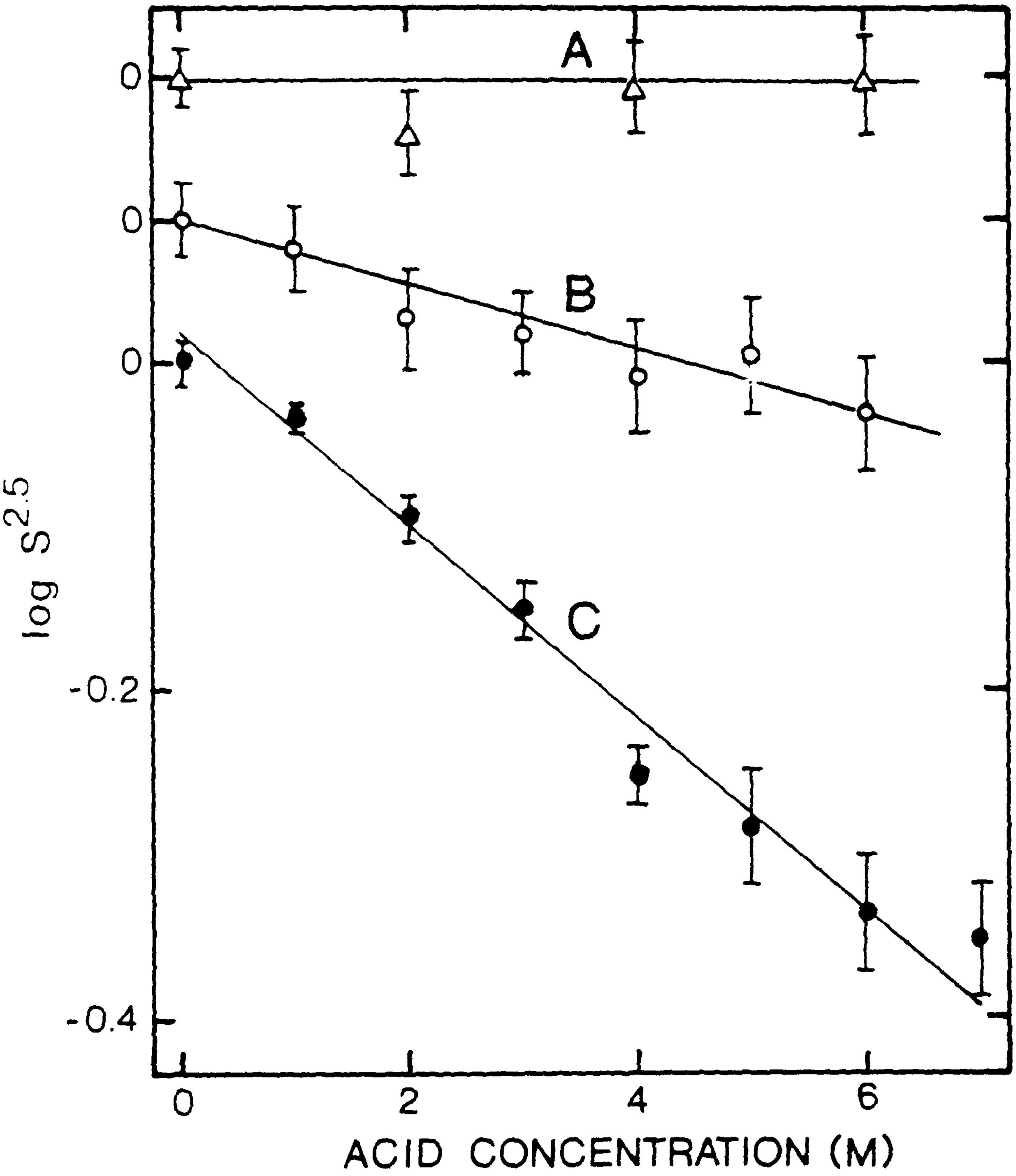

FIG. 4

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