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Photoelectron Emission Spectroscopy of

Inorganic Cations in Aqueous Solution

by

Paul Delahay, Kathrin von Burg and Andrew Dziedzic

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Threshold energies ($6.1 < E_t \leq 8.6$ eV) are determined for photoelectron emission by 16 inorganic cations in aqueous solution. E_t's are correlated with gas-phase ionization potentials, solvation and reorganization free energies, standard reduction potentials and ligand field stabilization energies (five transition metals). Dielectric saturation is shown to drastically lower threshold energies.

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PHOTOELECTRON EMISSION SPECTROSCOPY OF INORGANIC CATIONS IN AQUEOUS SOLUTION

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Threshold energies ($6.1 < E_t \leqslant 8.6$ eV) are determined for photoelectron emission by 16 inorganic cations in aqueous solution. E_t's are correlated with gas-phase ionization potentials, solvation and reorganization free energies, standard reduction potentials and ligand field stabilization energies (five transition metals). Dielectric saturation is shown to drastically lower threshold energies.

Threshold energies (ca. ± 0.1 eV) were recently obtained and interpreted [1,2] for photoelectron emission by various inorganic anions in aqueous solution. This work is extended to aqueous solutions of inorganic cations in the present paper.

1. Determination of threshold energies

Yields for photoelectron emission by solutions into water vapor were measured [1] as a function of photon energy (fig. 1). Anions with high threshold energies were selected to avoid emission by anions in the range of photon energies in which cations were investigated. Complexation [3] of the cation was avoided whenever possible. Hydrolysis [4] of the cation being photoionized and/or its photoionization product was minimized by addition of acid whenever necessary. Photoelectron emission by water [1] was totally negligible.

Threshold energies for emission were obtained [1,2] from emission spectra by extrapolation on the basis of the Brodsky-Tsarevsky theory [5]. A plot of the yield Y to the power 0.4 or 0.5 against photon energy is linear according to this theory, and extrapolation to zero yield gives the threshold energy

E_t. Selection of the proper exponent is discussed in [2] and [5] . The best plots were obtained by least square treatment of data, and the proper choice of exponent of Y was confirmed by calculation of correlation coefficients. The proportionality between the yield and the cation concentration was verified within ~ 10 percent for all cations (in general, 1, 0.5 and 0.2 M solutions) at constant anion concentration. This procedure insured that emission by the anion was negligible in the range of photon energies in which the yield was proportional to the cation concentration.
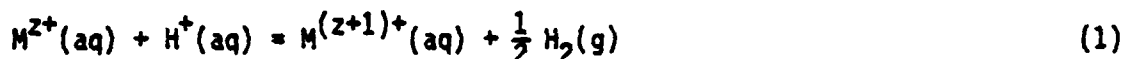
Threshold energies E_t are listed in Table 1. The slight variation of E_t from one anion to another for a given cation is essentially within experimental error. The standard deviation was 0.01 to 0.03 eV except for the 6 M acid solutions for which it was 0.06 to 0.08 eV because of a low signal (presumably because of scavenging of electrons by hydrogen ions). The effect of scavenging was also observed with reducible cations and is being investigated. E_t increased by 0.5 to 1 eV (vs. neutral solution) by addition of acid ($\geqslant$ 2 M) for Mn^{2+}, Co^{2+}, Ni^{2+}. Addition of an alkali metal salt of the acid ($\geqslant$ 2 M), instead of the acid, did not cause E_t to increase significantly. E_t for the acid was higher than for the alkali metal salt of the same anion as the acid, e.g., 9.08 eV for 6 M HCl vs. 8.77 eV for Cl^- in neutral solution (presumably because of removal of low-energy electrons by scavenging). This increase in E_t for Cl^- in 6 M HCl, for instance, allowed the determination of E_t = 8.60 eV for Co^{2+} without interference by the anion.

2. Threshold energy, ionization potential, reorganization and solvation free energies

The free energy for photoelectron emission by an aqueous solution of cation $M^{2+}(aq)$ will be correlated with the free energy for gas-phase ionization of $M^{2+}(g)$. The reference level of free energies must be changed

for that purpose. The problem is discussed in detail by Noyes [7,8], and only essential points will be mentioned. The surface potential at the solution-water vapor interface ($\sim$ 0.1 to 0.2 V for water [9]) is neglected here.

Free energies must be changed from their conventional values (i.e., in tables [10]) to the reference level of an electron in the gas phase at infinity at the same outer potential [11] as the solution. Consider the reactions,

$$M^{z+}(aq) + H^+(aq) = M^{(z+1)+}(aq) + \frac{1}{2} H_2(g) \qquad (1)$$

$$\frac{1}{2} H_2(g) = H^+(aq) + e^-(g) \qquad (2)$$

$$M^{z+}(aq) = M^{(z+1)+}(aq) + e^-(g). \qquad (3)$$

for which (g) and (aq) specify the gas phase and solution, respectively. The free energies are (fig. 2): the conventional value ΔG for (1); ΔG_H = 4.50 eV [8] for (2); $\Delta G + \Delta G_H$ for (3). Likewise, the conventional free energies of solvation of $M^{z+}(aq)$ and $M^{(z+1)+}(aq)$ ΔG_c^z and ΔG_c^{z+1} are converted to $\Delta G_s^z = \Delta G_c^z + z \Delta G_H$ and $\Delta G_s^{z+1} = \Delta G_c^{z+1} + (z+1) \Delta G_H$, respectively.

Photoionization of $M^{z+}(aq)$ yields initially the species $M^{(z+1)+}(aq*)$ under conditions governed by the Franck-Condon principle. The solvent about this ion undergoes reorganization to yield the ion $M^{(z+1)+}(aq)$ in a process characterized by the _reorganization_ free energy ΔG_r^{z+1}.

Two basic equations follow from fig. 2:

$$\Delta G_i^z(g) = \Delta G_s^z - \Delta G_s^{z+1} + \Delta G + \Delta G_H \qquad (4)$$

$$\Delta G_e^z(aq) = \Delta G_i^z(g) + \Delta G_s^{z+1} - \Delta G_s^z - \Delta G_r^{z+1} \qquad (5)$$

where $\Delta G_e^z(aq)$ and $\Delta G_i^z(g)$ are, respectively, the free energies for photoelectron emission by the solution and gas-phase ionization. A third equation follows from fig. 2 or can be obtained by combination of (4) and (5),

$$\Delta G_e^Z(aq) = \Delta G + \Delta G_H - \Delta G_r^{Z+1}. \tag{6}$$

One has $\Delta G_e^Z(aq) = E_t$ to a good approximation since measurements yield free energies for photoelectron emission. $\Delta G_i^Z(g)$ can be calculated from (4), e.g., 29.81 and 30.51 eV, respectively, for Cr^{2+} and Fe^{2+} (data from Table 1). The necessary ΔG_s^{Z+1}, however, is generally not available and therefore we shall use ionization potentials I^Z instead of $\Delta G_i^Z(g)$ in sec. 3. The I^Z's are enthalpies, but we set $\Delta G_i^Z(g) = I^Z$ and thus neglect a minor entropy correction.

Reorganization free energies ΔG_r^{Z+1} can be computed from (5), e.g., -2.05 and -2.11 eV, respectively, for Cr^{2+} and Fe^{2+} on the basis of the $\Delta G_i^Z(g)$ calculated above from (4) for these two cations. Since $\Delta G_s^3 = 45.04$ and 44.87 eV for Cr^{3+} and Fe^{3+}, respectively, one has $|G_r^3| \ll |\Delta G_s^3|$ for Cr^{2+} and Fe^{2+}. Hence, the ion $M^{(Z+1)+}(aq*)$ produced by vertical transition in the photoionization of $M^{Z+}(aq)$ is energetically not very different from $M^{(Z+1)+}(aq)$.

The orientation polarization of the solvent about $M^{(Z+1)+}(aq*)$ is the same as for $M^{Z+}(aq)$ (Franck-Condon principle). Conversely, the electronic polarization of the solvent about $M^{(Z+1)+}(aq*)$ is the same as for $M^{(Z+1)+}(aq)$ except for a secondary effect arising from the different orientation polarizations for these two ions. Since the free energies of $M^{(Z+1)+}(aq*)$ and $M^{(Z+1)+}(aq)$ are quite close, as shown above, the orientation polarization about $M^{Z+}(aq)$ and $M^{(Z+1)+}(aq)$ must not be very different. This can be the case only if there is <u>strong dielectric saturation</u> of the solvent about these ions.

The preceding conclusion is in complete agreement with Noyes [7,8] who calculated effective dielectric constants from solvation free energies and crystallographic radii by application of the Born equation. The resulting

effective dielectric constants (well below 10) indicated strong dielectric saturation. It should be noted that the foregoing analysis, based on eq. (5) does not involve the Born model of solvation.

3. Threshold energy and dielectric saturation

The calculation of ΔG_r^{z+1} in sec. 2 was restricted to Cr^{3+} and Fe^{3+} because of the lack of complete data for other cations. A method will be developed in this section by which the essential role of dielectric saturation will be established for all the cations of Table 1. The method involves the Born model of solvation, but the approximation inherent to the use of this model does not affect the conclusion to be drawn. Thus,

$$\Delta G_r^{z+1} = \Delta G_s^{z+1} + P_o^z + P_e^{z+1}(P_o^z) , \tag{7}$$

where P_o^z (> 0) is the orientation polarization about $M^{(z+1)+}(aq*)$ and $P_o^{z+1}(P_o^z)$ the corresponding electronic polarization (see above). A very small correction (< 0.05 eV) is neglected in (7) for the change of solvation free energy of an electrically neutral species [7] as the cavity radius changes from the value for P_o^z to that for P_o^{z+1}.

One has,

$$|\Delta G_s^z| < P_o^z + P_e^{z+1}(P_o^z) < |\Delta G_s^{z+1}| , \tag{8}$$

and consequently we set

$$P_o^z + P_e^{(z+1)}(P_o^z) = - \beta \Delta G_s^z \tag{9}$$

where the coefficient β (> 1) is to be determined from experimental data and calculated from a model. Equation (5), (7) and (9) yield,

$$\Delta G_e^z(aq) = \Delta G_i^z(g) + (\beta - 1) \Delta G_s^z. \tag{10}$$

The coefficient β can be calculated from the Born equation. Thus,

$$\beta = [(z+1)/z]^2(1 - \epsilon_o^{-1})/(1 - \epsilon^{-1})^{-1} + (\epsilon_o^{-1} - \epsilon^{-1})/(1 - \epsilon^{-1}), \tag{11}$$

where ϵ_o is the optical dielectric constant of water and ϵ its static dielectric constant in the absence of dielectric saturation. One computes

from (11), β = 2.33, 1.55, 1.34 for z = 1, 2, 3, respectively, in the absence of dielectric saturation (ϵ = 78.36). Conversely, one has $\beta = [(z+1)/z]^2$ for complete saturation ($\epsilon = \epsilon_0$), that is, β = 4, 2.25, 1.78, respectively, for z = 1, 2, 3.

Values of β calculated from (10) with $\Delta G_i^Z(g) = I^Z$ (see above) are listed in Table 1. The ionization potentials I^Z (from [4] except for Cr^{2+} and Fe^{3+} [6]) are more recent and in some cases slightly different from those used by Noyes (from [10]) in the calculation of conventional solvation free energies. This minor inconsistency is of no consequence in the interpretation of β. The β-values of Table 1 are close to those calculated from (11) for complete dielectric saturation. Equation (11) is approximate (Born model), and partial hydrolysis and/or complexation may cause β to be higher than expected for the ionic charge z+. Moreover, the value of β is not very sensitive to $\Delta G_e^Z(aq)$. Despite these reservations, one can safely conclude that there is strong dielectric saturation of water about the cations of Table 1. Reorganization free energies would be much higher in the absence of dielectric saturation, and the threshold energies of cations in aqueous solution are drastically lowered by strong dielectric saturation of the solvent about the cations.

4. Threshold energy, standard potential and reorganization free energy

The free energy ΔG in (6) pertains to reaction (1) which is written as an oxidation. Hence, $\Delta G = |e|E^0$ (in eV) where E^0 is the standard reduction potential for the $M^{z+1}(aq)/M^{z+}(aq)$ couple. Equation (6) becomes (in eV)

$$\Delta G_e^Z(aq) = |e|E^0 + 4.50 - \Delta G_r^{z+1}, \tag{12}$$

where ΔG_H = 4.50 eV was introduced (sec. 2). The free energy (ca. 0.1 to 0.2 eV [9]) for the surface potential should be added on the right hand side of (12).

Equation (12) clearly shows the relationship between the free energies $\Delta G_e^Z(aq)$ and ΔG for photoelectron emission and thermodynamics in the oxidation of $M^{Z+}(aq)$ to $M^{Z+1}(aq)$. ΔG_H arises from the change of reference levels for free energies, and $-\Delta G_r^{Z+1}$ is the additional free energy required by the Franck-Condon principle. The negative of the reorganization free energy can be regarded as the counterpart of the overvoltage in the electrochemical oxidation of $M^{Z+}(aq)$.

Reorganization free energies calculated from (12) are nearly the same (-2.05 to -2.25 eV) for the five cations of fig. 3 (E^0's from [4], [12]). Since ΔG_r^3 is nearly constant, a plot of E_t against $|e| E^0$ [2,13] is linear, to a first approximation, and has a unit slope (fig. 3). Standard reduction potentials can be deduced from such a plot and E_t (for chemically similar cations) for species that are unstable in aqueous solution, e.g., $E^0 \approx 1.75$ V for Ni^{3+}/Ni^{2+} in 6 M HCl (using $\Delta G_r = -2.1$ eV).

5. Threshold energy, ionization potential and ligand field stabilization energies (LFSE)

The $M^{2+}(aq)$ and $M^{3+}(aq)$ ions of V, Cr, Mn, Fe, Co exist as hexaquo ions [4]. $\Delta G_e^Z(aq)$ therefore include the difference ΔE_f between the LFSE's for $M^{3+}(aq)$ and $M^{2+}(aq)$ ions in an octahedral field. I^2 for these metals obviously does not include ΔE_f. Hence the correlation between I^2 and E_t is partially masked (for Cr, in particular, fig. 4) by the LFSE effect. The correlation, however, is evident when I^2 and $E_t + \Delta E_f$ are compared. Thus, $I^2 - (E_t + \Delta E_f)$ and $\Delta G_s^2 - \Delta G_s^3 + \Delta G_r^3 - \Delta E_f$ (cf. eq. (5)) are nearly constant from V to Co whereas $I^2 - E_t$ is not.

Acknowledgment

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Table 1

Threshold energies, coefficient β and relevant data

	E_t eV	I^Z [4,6] eV	$-\Delta G_S^Z$ eV	β
Ag^+	7.60 (ClO_4^-), 7.52 (ClO_4^-, 5 M $HCLO_4$)	21.48	4.95	3.80
Tl^+	7.40 (F^-), 7.46 (ClO_4^-, 5 M $HClO_4$)	20.42	3.55	4.67
V^{2+}	6.38 (SO_4^{2-}, 1 M H_2SO_4)	29.31	19.14	2.20
Cr^{2+}	6.14 (Cl^-, 1 M HCl)	30.95	19.32	2.28
Mn^{2+}	8.08 (Cl^-, 2 M HCl)	33.70	18.96	2.35
Fe^{2+}	7.35 (Cl^-, 1 M HCl), 7.38 (SO_4^{2-})	30.64	19.63	2.18
Co^{2+}	8.60 (Cl^-, 6 M HCl)	33.49	20.88	2.19
Ni^{2+}	8.35 (Cl^-, 6 M HCl)	36.16	21.40	2.30
Cu^{2+}	7.83 (ClO_4^-, 1 M $HClO_4$), 7.84 (SO_4^{2-})	36.74	21.55	2.34
Sn^{2+}	7.21 (Cl^-), 7.42 (ClO_4^-, 6 M $HClO_4$)	30.49	16.14	2.44
Pb^{2+}	7.23 (ClO_4^-), 7.10 (ClO_4^-, 6 M $HClO_4$)	31.92	15.49	2.59
In^{3+}	7.15 (ClO_4^-)	54.42	42.57	2.11
Ti^{3+}	6.90 (Cl^-)	43.25	42.01	1.87
V^{3+}	7.06 (Cl^-)	47.7	44.2	1.92
Cr^{3+}	7.33 (Cl^-), 7.27 (SO_4^{2-})	50.8	45.04	1.97
Fe^{3+}	7.03 (Cl^-), 7.05 (Cl^-, 6 M $HClO_4$)	56.8	44.87	2.11

See sec. 1 for standard deviation. ΔG_z^3 for V^{3+} calculated from (4) with $\Delta G = -0.25$ eV (sec. 4).

10

Captions to Figures

Fig. 1. Photoelectron emission spectra of Fe^{2+}. (A) 1 M $FeSO_4$; (B) 0.5 M $FeSO_4$ + 0.5 M Li_2SO_4; (C) 0.2 M $FeSO_4$ + 0.8 M Li_2SO_4. Photoelectron emission only by Fe^{2+} below the threshold energy (E_t = 8.65 eV) for SO_4^{2-}.

Fig. 2 Free energy diagram. Upward and downward arrows, respectively, for positive and negative free energies. The superscrips z and z+1 denote the positive ionic charge. The symbols (g) and (aq) refer to gas phase and solution, respectively. Free energies: $\Delta G^z(g)$, $\Delta G^{z+1}(g)$, $\Delta G^z(aq)$, $\Delta G^{z+1}(aq)$ of ions; ΔG for reaction (1); ΔG_H for (2); ΔG_C^z and ΔG_C^{z+1} conventional values for solvation; ΔG_S^z, ΔG_S^{z+1} values for solvation corrected for ΔG_H; ΔG_r^{z+1} reorganization; $\Delta G_i^z(g)$ gas-phase ionization; $\Delta G_e^z(aq)$ photoelectron emission by solution.

Fig. 3 Plot of threshold energy E_t and reorganization free energy ΔG_r [3] against $|e|E^0$. Standard reduction potentials E^0 from [4,12]. The E^0's for Mn^{3+}/Mn^{2+} and Co^{3+}/Co^{2+} are approximate (ca. ± 0.05 V). The symbol M_*^{3+} stands for $M^{3+}(aq*)$.

Fig. 4 Correlation between the ionization potential I^2 and the threshold energy E_t corrected for the difference ΔE_f between the LFSE's for M^{3+} and M^{2+} in an octahedral field. Values of ΔE_f from [14].
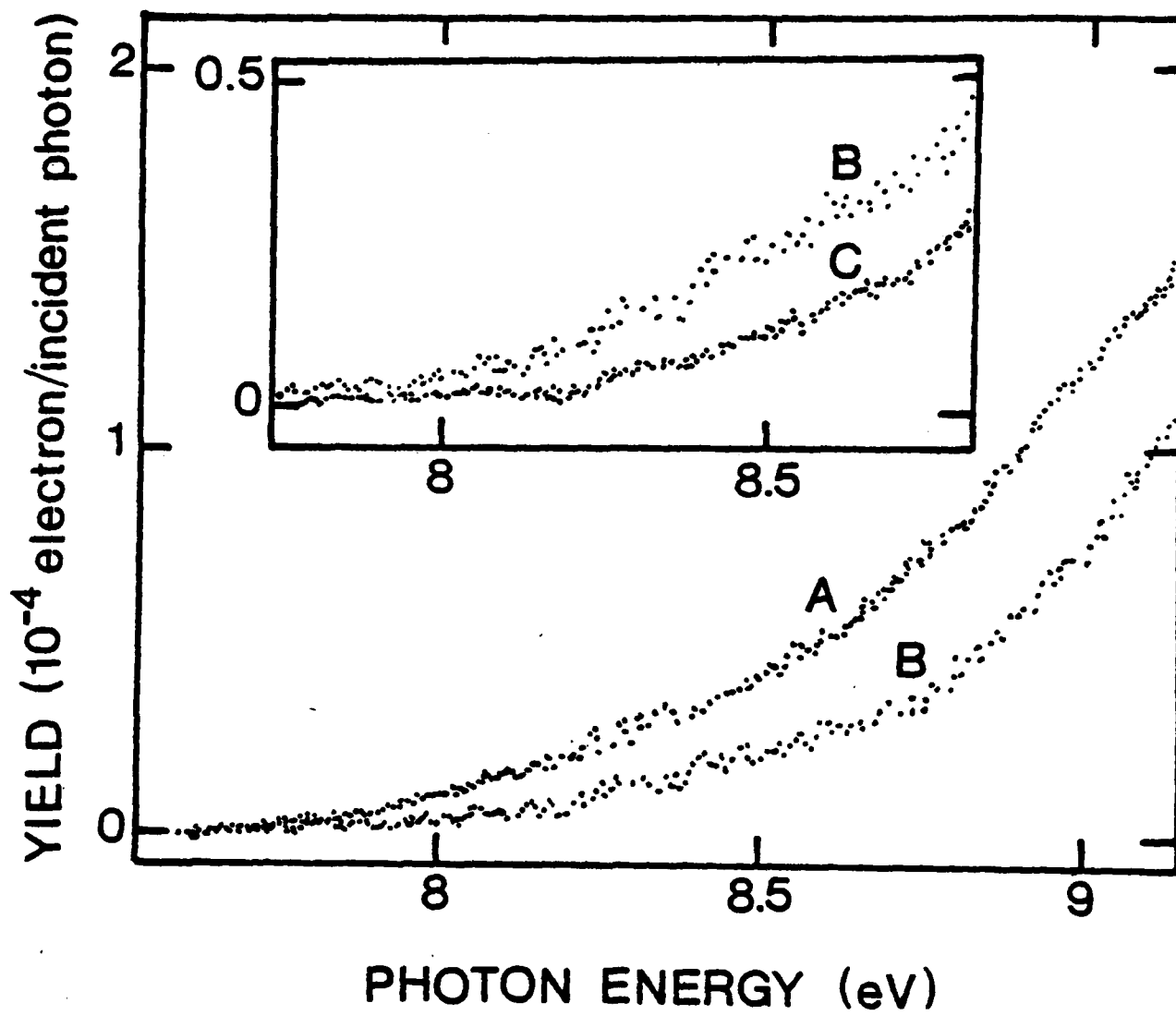
FIG. 1

FIG. 2

FIG. 3
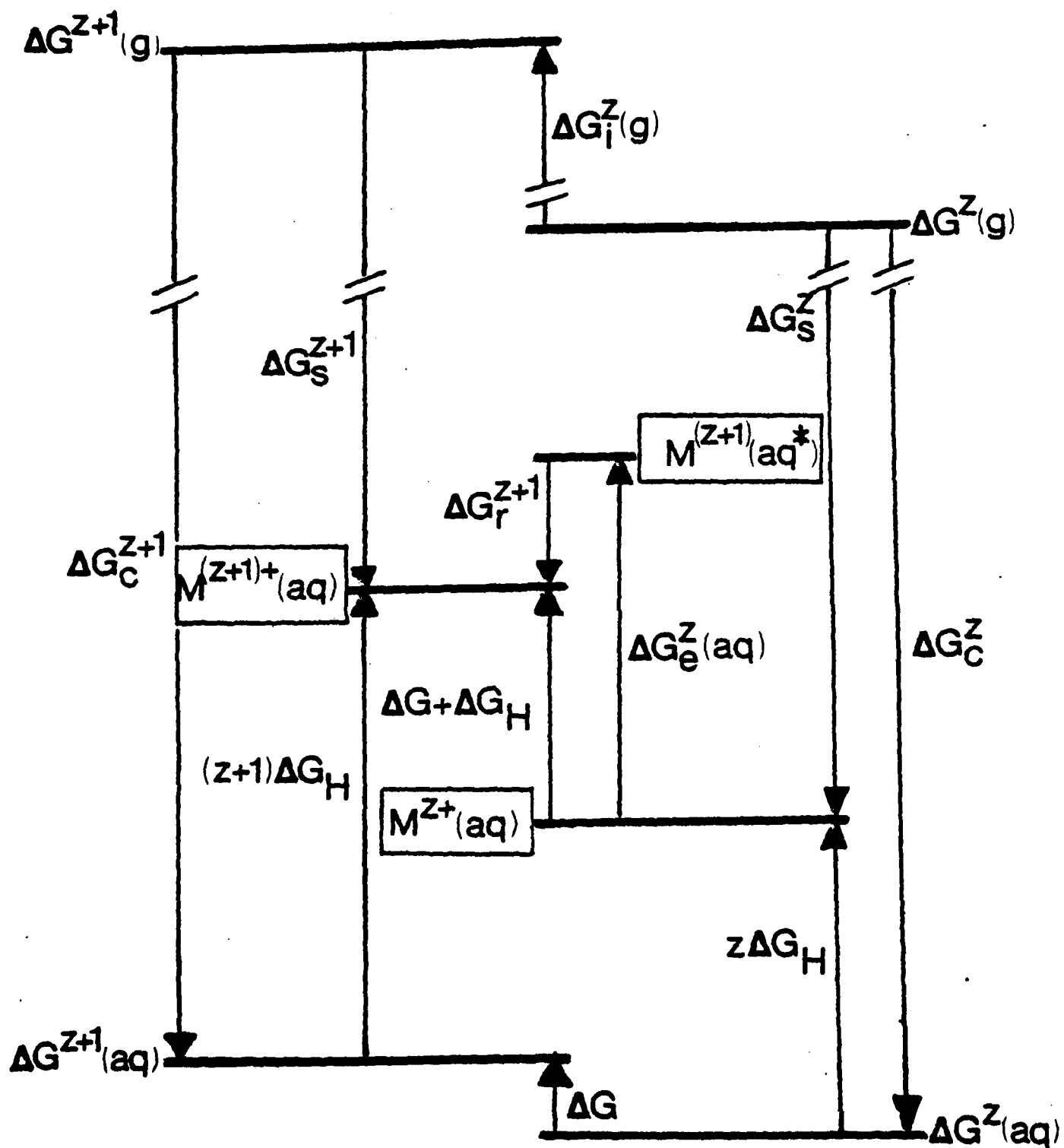
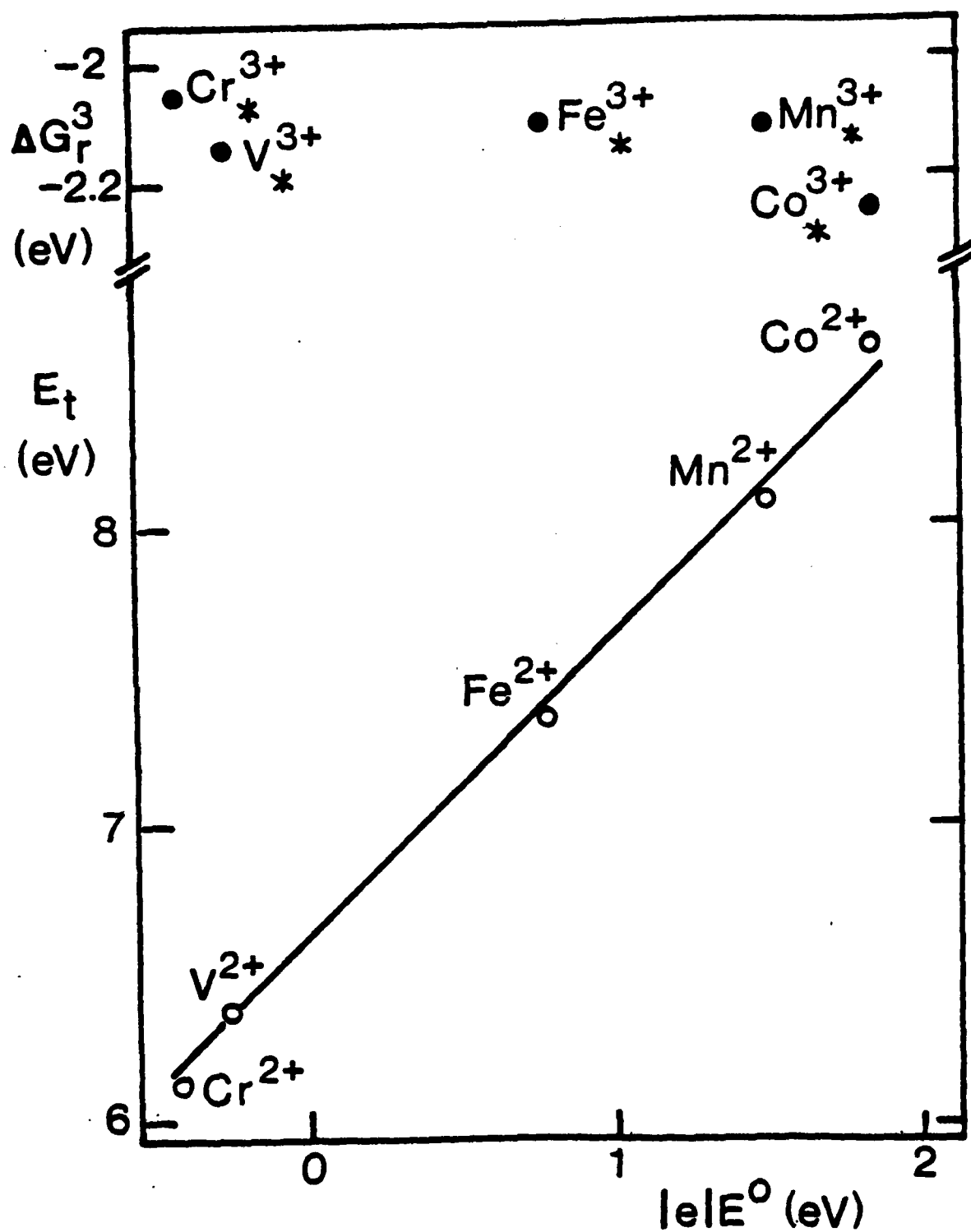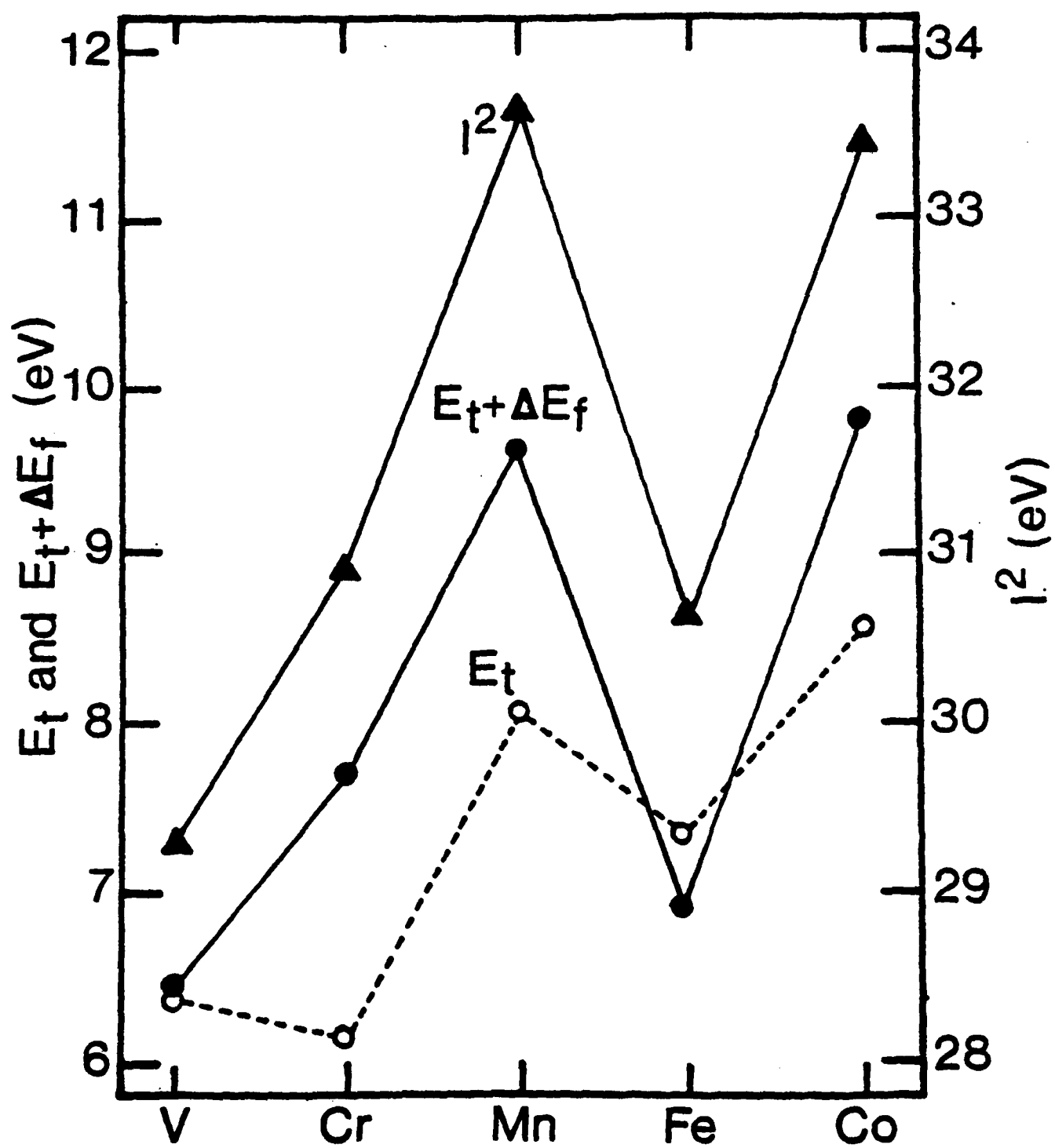FIG. 4

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