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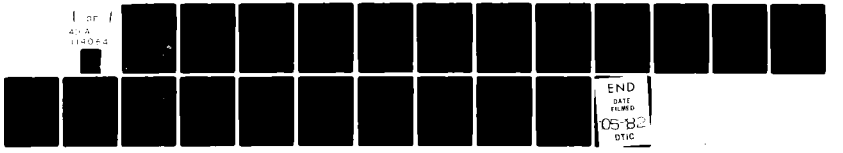

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Charge transfer absorption spectra are correlated to the energetics of photoelectron emission by donor and/or acceptor solutions. Donor-acceptor pairs include: ~10 inorganic anions and solvent (water); five cations (V^{2+} to Co^{2+}) and solvent; solvent and atoms (H, Cl, Br, I), OH radical, Fe^{3+}, Ce^{4+}; anion-cation pairs exhibiting charge transfer in the 7 to 10 eV range.

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CHARGE TRANSFER SPECTRA AND PHOTOELECTRON EMISSION BY SOLUTIONS

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Charge transfer absorption spectra are correlated to the energetics of photoelectron emission by donor and/or acceptor solutions. Donor-acceptor pairs include: 10 inorganic anions and solvent (water); five cations (V^{2+} to Co^{2+}) and solvent; solvent and atoms (H, Cl, Br, I), OH radical, Fe^{3+}, Ce^{4+}; anion-cation pairs exhibiting charge transfer in the 7 to 10 eV range.

Optical donor-acceptor charge transfer (CT) will be correlated to photoelectron emission by solutions of the donor. The interpretation involves the calculation of the free energies for the CT and emission processes and the consideration of the reorganization free energies [1,2] on the basis of the theories of electron transfer [2] of Marcus [3] and Hush [4].

1. Basic relationships

The free energy ΔG^{CT} for donor-acceptor CT will be obtained first. The donor and acceptor are represented by D^- and A, respectively, without consideration of the actual ionic charges (to simplify notations). Consider the following sequence: The separated reactants D^- and A in solution are brought within a distance at which CT occurs; the CT process is governed by the Franck-Condon principle, and CT therefore is followed by reorganization of the nuclear configurations of the products of CT and the solvent; finally, the products D and A^- are separated.

The preceding sequence is equivalent to the net reaction $D^- + A = D + A^-$ involving the change of free energy $\Delta G(DA^-)$. Hence, one has [1,2]

$$\Delta G^{CT} = \Delta G(DA^-) - R^{CT}, \qquad (1)$$

where R^{CT} (< 0) is the reorganization free energy for the change of nuclear

configuration following CT. The work terms for bringing together reactants and separating products (weak bonding) are omitted in (1) as they are generally negligible (< 0.1 eV) in the experiments considered here.

The contributions of the donor and acceptor in (1) will be separated. One sets,

$$\Delta G(DA^-) = \Delta G(D^-) + \Delta G(A), \tag{2}$$

where $\Delta G(D^-)$ and $\Delta G(A)$ are the changes of free energies for the reactions $D^- + H^+ = D + 1/2H(g)$ (oxidation of D^-) and $A + 1/2H_2(g) = A^- + H^+$ (reduction of A) in solution, respectively.

The reorganization free energy R^{CT} in (1) will be interpreted on the basis of the theories of electron transfer in [3,4]. The quantity R^{CT} is equated to the sum of inner- and outer-sphere contributions (denoted by "in" and "out"). The outer-sphere contribution is derived from the macroscopic model of non-equilibrium polarization of a continuous medium [3]. Thus,

$$R^{CT}_{out} = -(\epsilon_{op}^{-1} - \epsilon_s^{-1})e^2(1/2a_D + 1/2a_A - 1/r_{DA}), \tag{3}$$

where ϵ_{op} and ϵ_s are the optical and static dielectric constants of water, respectively; a_D and a_A are the radii of the assumed spherical boundaries between inner and outer regions of the donor and acceptor, respectively; r_{DA} is the distance between the centers of the donor and acceptor prevailing in CT. The fields of the donor and acceptor in (3) are assumed to have spherical symmetry without mutual influence [2]. This approximation will suffice here. Equation (3) is rewritten in the form,

$$R^{CT}_{out} = R_{out}(D_*) + R_{out}(A^-_*) + R(DA^-), \tag{4}$$

where the asterisks denote the non-equilibrium nuclear configurations prior to reorganization.

One obtains from (1), (2) and (4) by noting that reorganization free energies are the sum of inner- and outer-sphere contributions,

$$\Delta G^{CT} = \Delta G(D^-) - R(D_*) + \Delta G(A) - R(A_*^-) - R(DA^-). \quad (5)$$

This equation is combined with the relationship [1,2,5] for the free energy $\Delta G^m(D^-)$ for photoelectron emission by aqueous solutions of D^-,

$$\Delta G^m(D^-) = \Delta G_H + \Delta G(D^-) - R(D_*), \quad (6)$$

where $\Delta G_H = 4.50$ eV on the assumption of a negligible surface potential ($\pm$ 0.1 V) at the water vapor-solution interface. Thus,

$$\Delta G^m(D^-) - \Delta G_H = \Delta G^{CT} - \Delta G(A) + R \quad (7)$$

with

$$R = R(A_*^-) + R(DA^-) \quad (8)$$
$$= R_{in}(A_*^-) + R_{out}(A_*^-) + R(DA^-).$$

Equation (7) correlates the energetics of CT and emission by D^- and is the key to the interpretation of results in the present paper.

An expression for the computation of $R(DA^-)$ is obtained by addition of (7) to the equation,

$$\Delta G^m(A^-) = \Delta G_H + \Delta G(A^-) - R(A_*), \quad (9)$$

for photoelectron emission by aqueous solutions of the reduced form A^- of the acceptor A. The change of free energy $\Delta G(A^-)$ pertains to the oxidation of A^- whereas $\Delta G(A)$ is defined for reduction of A. Hence, $\Delta G(A^-) = -\Delta G(A)$. It will also be <u>assumed</u> that the quantities $R(A_*^-)$ and $R(A_*)$ pertaining to CT and emission, respectively, are equal. Conditions under which this assumption is justified are discussed in sec. 3. Thus,

$$\Delta G^m(A^-) - \Delta G^{CT} = 2\Delta G_H - \Delta G^m(D^-) + 2\Delta G(A^-) + R(DA^-). \quad (10)$$

The quantity $R(DA^-)$ can be computed from (10), and the distance r_{DA} between the centers of the donor and acceptor is obtained from (3). This is significant because r_{DA} is obtained from <u>experimental free energies without the introduction of the radii a_D and a_A</u>. The assumption of outer-sphere contact in electron transfer in solution may be verified in this way.

2. Charge transfer from donor to solvent

Equation (7) will be tested for anions and cations as donors. The species A^- produced by CT is the hydrated electron, and $\Delta G(A) = 2.77$ eV. One has [6] $\Delta G^m(D^-) \approx E^t$, where E^t is the experimental threshold energy for emission by an aqueous solution of the donor. Values of E^t were taken from [6-8] except for $Fe(CN)_6^{4-}$ ($E^t = 6.6$ eV [9]). One also sets ΔG^{CT} equal to the photon energy E^{max} (from [10]) at the maximum of the CT absorption band of the donor. E^{max} for $H_2PO_4^-$ and HPO_4^- were obtained by extrapolation and were not included in the least-square fitting discussed below. The photon energy for SO_3^{2-} was not used either since it corresponds to an extinction coefficient of 10^3 and not to the band maximum.

Least-square fitting yielded $E^t - 4.50 = -0.03 + 1.016(E^{max} - 2.77)$, with E^t and E^{max} in electronvolts. Hence, $E^t - 4.50 \approx E^{max} - 2.77$. One expects from (7) that these two quantities differ by R provided that one sets $\Delta G^m(D^-) = E^t$ and $\Delta G^{CT} = E^{max}$, and the reorganization terms in (8) therefore nearly cancel out. Since one has $R_{in}(A_*^-) < 0$, it follows that $R(DA^-) > -R_{out}(A_*^-)$ or, according to (3), $r_{DA} < 2a_A$. Taking $r_{DA} = a_A + a_D$, one must have $a_D < a_A$ to account for the value $R \approx 0$. This is not surprising since model calculations [11] yield an equilibrium cavity radius of ca. 0.37 nm for the hydrated electron. This radius may be compared with the crystallographic radii ($< a_D$) of ca. 0.2 nm for the donors of Fig. 1. The model leading to eq. (3) is approximate and so are radius values, but the preceding argument undoubtedly accounts for the near cancellation of terms in (8).

The quantities ΔG_H and $\Delta G(A)$ in (7) pertain to the final states of the electron, namely the vacuum level in emission and the level of the redox couple A/A^- in CT. Hence, emission and CT for the anions of Fig. 1 involve nearly the same free energies after correction for the difference in the final states of the

electron. This conclusion rests on the experimental values of E^t and E^{max} without recourse to model calculations. Such model calculations for the CT process, although somewhat crude, in fact yield results in agreement with the present work, namely $E^t \approx E^{max} + 1.7$ (eV) [6].

The testing of eq. (7) for CT from cations to the solvent is more tentative than for anions because CT absorption bands of cations are hidden by other transitions. Only Cr^{2+} among the cations M^{2+} whose spectra are given in [12] exhibits a well-defined shoulder at ca. 4.6 eV [1]. Thus, one sets tentatively $\Delta G_{CT} \approx 4.6$ eV for Cr^{2+}. The CT spectra of other cations can be characterized by the photon energy E^{low} [12] at which the extinction coefficient is 0.1. The plot (Fig. 2) with E^{low} instead of $E^{max} - 2.77$ (eV) appears quite linear. Least square fitting yielded $E^t - 4.50 = -1.63 + 0.996\ E^{low}$, where the energies are in electronvolts. One has $E^{low} = 3.35$ eV and $\Delta G^{CT} \approx 4.6$ eV (see above) for Cr^{2+}. The intercept of the plot of $E^t - 4.50$ (eV) against ΔG_{CT} therefore is $-1.63 - 0.996(4.6 - 3.35) = -2.88$ eV if one assumes that $\Delta G^{CT} - E^{low}$ is the same for the cations of Fig. 2. If this is indeed the case, it may be concluded that the quantities $E^t - 4.50$ and $\Delta G^{CT} - 2.77$ (eV) are equal within a few tenths of electronvolt for each of the cations of Fig. 2. This conclusion is obviously tentative in view of the limited spectroscopic evidence.

3. Charge transfer from solvent to acceptor

Absorption spectra are available for CT from the solvent (water) to a few oxidizing species [13,14]. The photon energies E^{max} of the CT absorption bands are listed in Table 1 together with $\Delta G(A)$ [15,16]. Values of R in Table 1 were computed from (7) for $\Delta G^m(D^-) = E^t = 10.06$ eV [7] for water.

The results for Fe^{3+} and Ce^{4+} will be interpreted first. The value $R(DA^-) = 1.58$ eV was computed from (10) for $\Delta G^m(A^-) = E^t = 7.35$ eV [5]

and $\Delta G(A) = 0.77$ eV [16] for emission by Fe^{2+}. One deduces $r_{DA} = 0.50$ nm from (3) for $R(DA^-) = 1.58$ eV. This length is nearly equal to the distance for contact between a water molecule and a hexaquo ion Fe^{3+}, namely $0.138 + (0.064 + 2 \times 0.138) = 0.48$ nm (0.064 and 0.138 nm, crystallographic radii of Fe^{3+} and H_2O, respectively). The agreement is remarkable, but experimental errors and the approximate character of eq. (3) and the relationship $R(A_*) = R(A_*^-)$ should not be overlooked.

The value $R = -0.50$ eV in Table 1 for Fe^{3+} is close to the inner-sphere reorganization free energy of -0.46 eV for Fe^{3+}/Fe^{2+} electron transfer computed in [17] from the bond-stretching model. This agreement suggests (cf. eq. (8)) that $R_{out}(A_*^-) \approx -R(DA^-)$ within experimental and systematic errors of a few tenths of electronvolt. Likewise, one has $R = -0.15$ eV for Ce^{4+} (Table 1) versus an inner-sphere term of -0.10 eV in [17] for Ce^{4+}/Ce^{3+}. It is concluded (eq. (7)) that the energetics are nearly the same for emission by water and CT to Fe^{3+} or Ce^{4+} once E^t for emission and E^{max} for CT are corrected for reorganization and the difference of the final states of the electron.

The other acceptors in Table 1 will now be examined. Values of $R(DA^-)$ between 0.6 and 0.9 eV are obtained for the halogens by application of (10), and the corresponding distance $r_{DA} \approx 1$ nm computed (from (3)) is approximately twice too long (see above). It is concluded that the equality, $R(A_*^-) = R(A_*)$, introduced in the derivation of (10) does not hold for the halogens. Thus, the reorganization free energies for the processes $A^- \rightarrow A_*$ and $A \rightarrow A_*^-$ are <u>not</u> the same. This is to be expected from consideration of the curves R and P representing the total (electron involved in CT, ions, solvent) energy U against generalized reaction coordinate for the reactants and products of CT [2]. The energy $U(R \rightarrow P)$ for the vertical transition from the

ground state of R to P is generally different from the energy U(P→R) for the vertical transition from the ground state of P to R. This comparison must be made after correction for the change of free energy for CT. The difference between the two energies for vertical transition increases (in absolute value) with the difference between the coordinates of the ground states of R and P.

The radii $a(Fe^{3+})$ and $a(Fe^{2+})$ are nearly the same, and the near equality, $R(Fe^{2+}_{\star}) \approx R(Fe^{3+}_{\star})$, assumed in the calculation of $R(DA^{-})$ from (10) is consistent with the foregoing discussion of vertical transitions. Conversely, the atomic radii of the halogen atoms are definitely smaller than the corresponding crystallographic radii of the halide ions, and it is not surprising that $|R(A^{-}_{\star})|$ for $A \rightarrow A^{-}_{\star}$ (CT) is different from $|R(A_{\star})|$ for $A^{-} \rightarrow A_{\star}$ (emission). The high values of $|R|$ for the halogens in Table 1 thus are accounted for by the higher (in absolute value) inner-sphere reorganization free energy for CT than for emission. The same conclusion holds for H and OH in Table 1. The increase of $|R|$ from the inner-sphere term is particularly large for OH because vibrational excitation of both solvent and radical is involved.

4. Donor-acceptor charge transfer in the 7 to 10 eV range

Equation (7) will be applied to the recently studied [8,18] CT processes between a wide variety of donors and acceptors in aqueous solution upon photon absorption in the 7 to 10 eV range. These results will be interpreted in terms of optical CT, but the analysis also holds if one assumes a scavenging mechanism. The confined volume model in [18], however, supports interpretation based on optical CT. The occurrence of CT was inferred from the lowering of the yield for photoelectron emission by the donor in presence of the acceptor. This method of detection of CT can be applied provided that the condition $\Delta G^{CT} \lesssim \Delta G^{m}(D^{-})$ holds, that is ((7) and (8)),

$$\Delta G_H - \Delta G(A) + R \gtrsim 0. \qquad (11)$$

If R = 0, condition (11) becomes $\Delta G(A) \lesssim \Delta G_H$, that is, $\Delta G(A) \lesssim 4.5$ eV. Strongly reducing species therefore may be produced by CT from the donor. CT was observed [18], for instance, with the following acceptors ($\Delta G(A)$ in eV [16,19] between parentheses): H^+ (2.1), Fe^{2+} (3.1), Ba^{2+} (4.85±0.25). CT to the alkali metal cations was not detected in [18] although the free energy change, $\Delta G(A) \approx 3.5$ to 3.6 eV [19], is much more favorable to CT than for the CT-exhibiting Ba^{2+} ion (4.85±0.25 eV). This seemingly paradoxical result can be understood by consideration of the term R in (11). Assume to simplify matters that $R \approx R_{in}(A^-_*)$ since the other two terms contributing to R in (8) should nearly cancel out. Thus, one expects $R_{in}(Na_*)$ to be significantly different from $R_{in}(Ba^+_*)$ because CT to Na^+ as acceptor would involve a much more pronounced change in size upon reduction to Na than CT to Ba^{2+} as acceptor. This difference in R more than compensates the difference in $\Delta G(A)$, and CT is only observed with Ba^{2+}. *In contrast to the case of* Na^+, CT to Tl^+ is observed despite the marked increase in radius from ion to atom. CT is possible in that case because $\Delta G(A)$ for the reduction of Tl^+ is sufficiently low (1.9 eV) to offset the large inner-sphere term in R.

Acknowledgment

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Table 1

Charge transfer from water to acceptor

Acceptor	E^{max} a) (eV)	$-\Delta G(A)$ b) (eV)	$-R$ (eV)	$-R_{in}(A_*)$ c) (eV)
H	>6.2	-0.1	>0.5	-
OH	5.40	1.9	1.74	-
Cl	3.95	2.55	0.94	-
Br	4.50	2.0	0.94	-
I	4.85	1.4	0.69	-
Fe^{3+}	5.29	0.77	0.50	0.46
Ce^{4+}	4.00	1.70	0.14	0.10

a) From [13,14]. E^{max} for H higher than upper limit (6.2 eV) of explored range of photon energies ($E^{low} \approx 5.0$ eV).

b) From [15,16].

c) From [17].

Captions to Figures

Fig. 1. Plot (eq. (7)) for CT from anion to solvent (water). E^t, threshold energy for photoelectron emission by anions; E^{max}, photon energy at maximum of CT-absorption band of the anions.

Fig. 2. Plot for CT from cation to solvent (water). E^{low}, photon energy at which the extinction coefficient is 0.1 on the cation absorption spectra from [12].

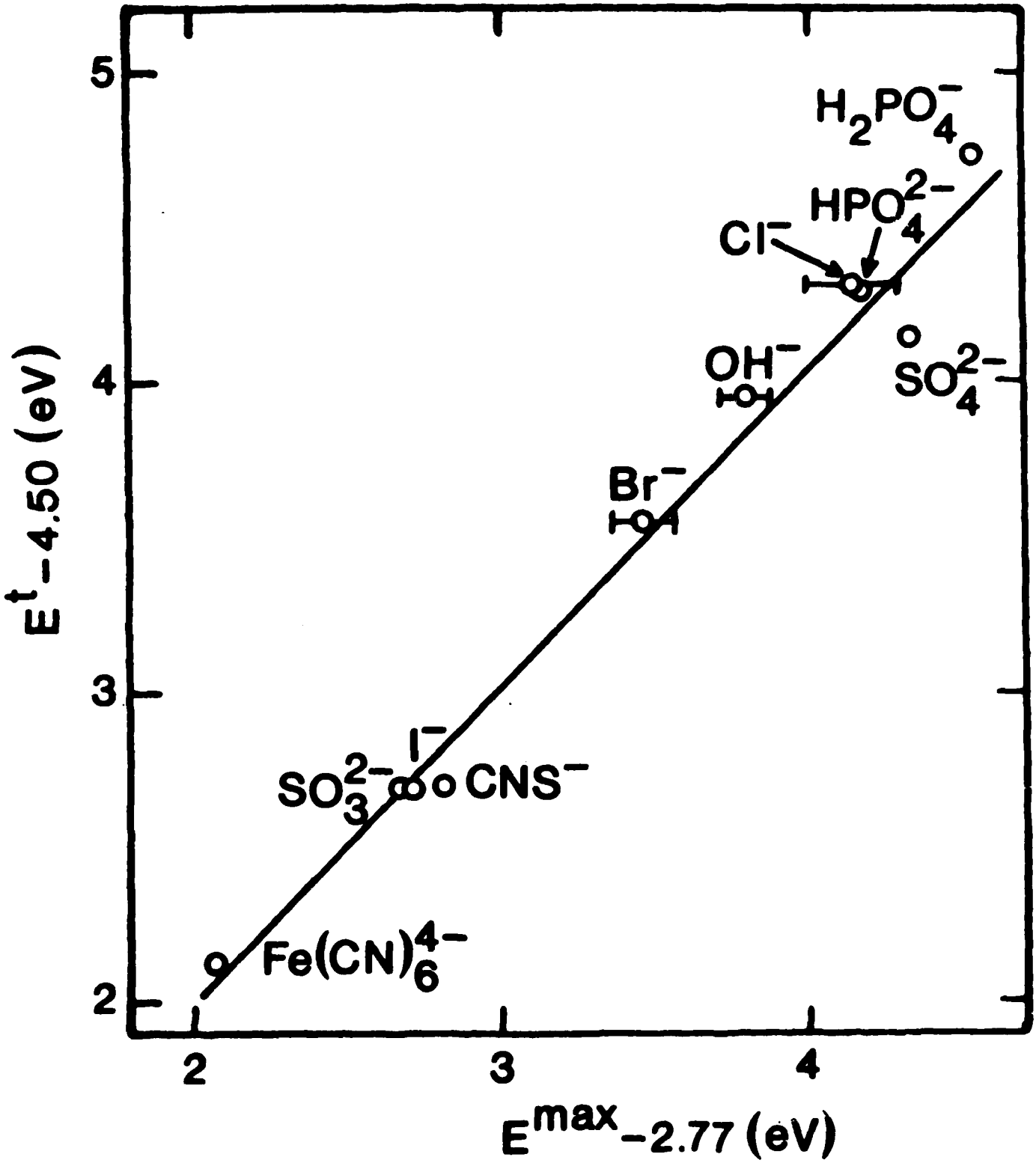
5
4
3
2
2
3
4
$E^t - 4.50$ (eV)
$E^{max} - 2.77$ (eV)
$H_2PO_4^-$
HPO_4^{2-}
Cl^-
OH^-
SO_4^{2-}
Br^-
I^-
SO_3^{2-}
CNS^-
$Fe(CN)_6^{4-}$

FIG. 1

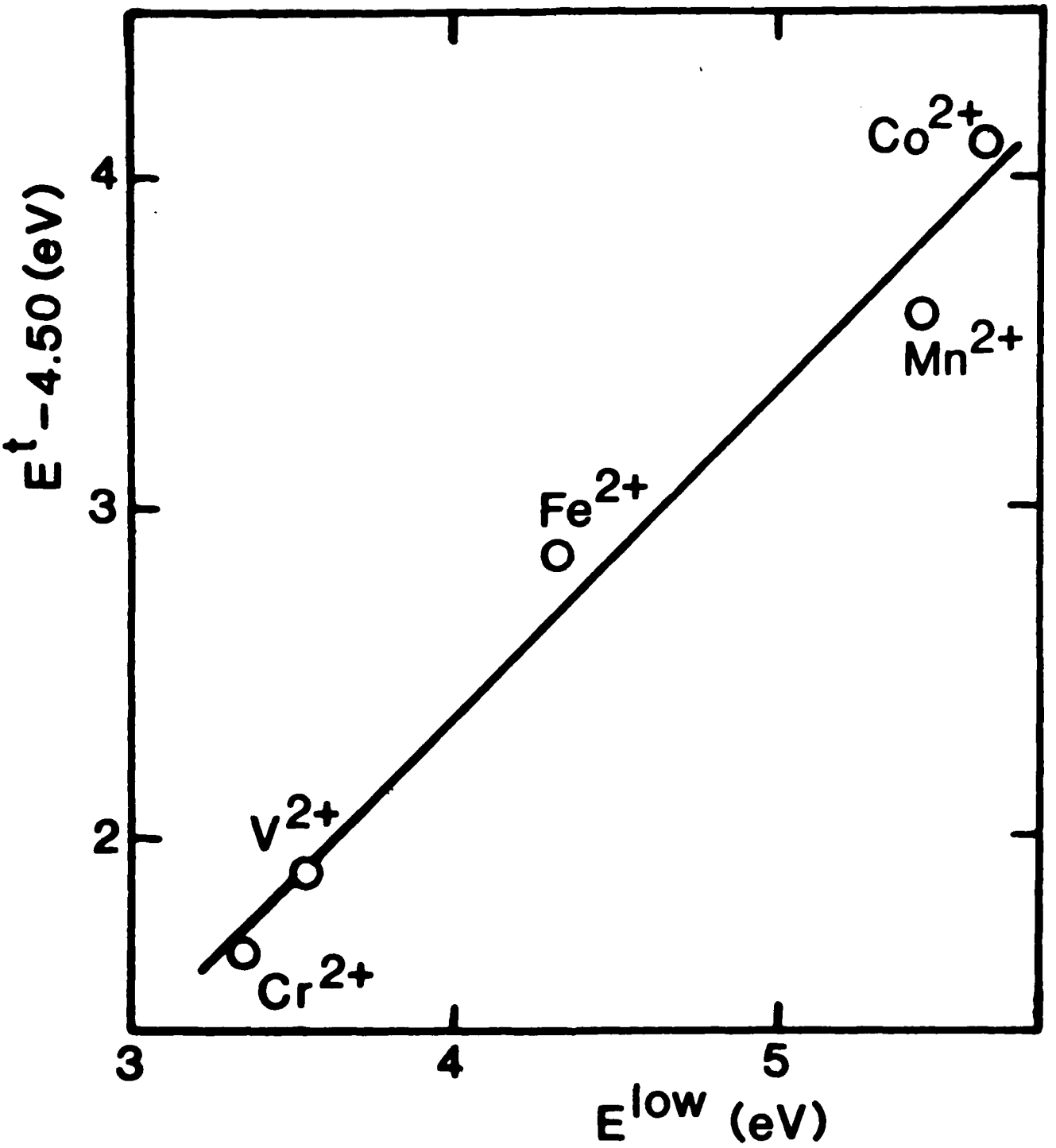

FIG. 2

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