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Contract N00014-75-C-0397

Task No. NR 051-258

TECHNICAL REPORT NO. 18 (New Series)

Correlation Between Thermal Electron Transfer in Solution and Photoelectron Emission

by

Paul Delahay

Accepted for Publication

in

Chemical Physics Letters

New York University
Department of Chemistry
New York, NY

February 1982

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 18 (New Series)	2. GOVT ACCESSION NO. AD-A111 177	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) CORRELATION BETWEEN THERMAL ELECTRON TRANSFER IN SOLUTION AND PHOTOELECTRON EMISSION		5. TYPE OF REPORT & PERIOD COVERED Technical Report 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Paul Delahay		8. CONTRACT OR GRANT NUMBER(s) N00014-75-C-0397
9. PERFORMING ORGANIZATION NAME AND ADDRESS New York University New York, NY 10003		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NR 051-258
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research Arlington, VA 22217		12. REPORT DATE February 1982 13. NUMBER OF PAGES 19
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

Accepted for publication in Chemical Physics Letters.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Activation free energy
Electrode reactions
Electron transfer, optical
Electron transfer, thermal
Photoelectron emission
Reorganization free energy

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

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(CN)6(4-)

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S/N 0102-LF-014-6601

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CORRELATION BETWEEN THERMAL ELECTRON TRANSFER IN SOLUTION AND PHOTOELECTRON EMISSION

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The activation free energy for electron transfer in solution or at electrodes is correlated to the corresponding Franck-Condon determined reorganization free energy R_m for photoelectron emission. Excellent to fair agreement is obtained between the activation free energies predicted from R_m and experimental values. Data are given for V^{2+}, Cr^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+} and $Fe(CN)_6^{4-}$ in aqueous solution.

1. Introduction

Electron transfer between species M^{z+} and $M^{(z+1)+}$ ($z \gtrless 0$) in solution can be studied either as a homogeneous process such as electron exchange between isotopes of M or as a heterogeneous process at a metal or semiconductor electrode. The kinetics of these processes is characterized by a free energy of activation $\Delta G_x^{\ddagger}$ for electron transfer between isotopes and $\Delta G_e^{\ddagger}$ for electrode processes. These thermal electron transfer processes have their optical counterparts, namely intervalence-transfer absorption and photoelectron emission by solutions.

Correlations can be established between the preceding four types of electron transfer. The theories of electron transfer [1,2] of Marcus [3] and Hush [4] provide the key to such correlations. (See [1,2] for other theories.) The free energies of activation $\Delta G_x^{\ddagger}$ and $\Delta G_e^{\ddagger}$ in these theories are expressed as functions of reorganization free energies R_x and R_e, respectively. The latter are related to each other. Marcus [5] correlated in this fashion electron transfer in solution and at electrodes. Hush [6]

established the correlation between thermal electron transfer and intervalence-transfer absorption spectra. We examine in the present paper the relationship between thermal electron transfer and the energetics of photoelectron emission by solutions. This type of optical electron transfer is characterized by a reorganization free energy R_m which can be determined experimentally as was recently shown [7-9]. The correlation between the experimental quantities $\Delta G_x^{\ddagger}$ or $\Delta G_e^{\ddagger}$ and R_m thus obtained provides a very direct test of theories of electron transfer in solution. Such a test is also provided, albeit in a different way, by the widely obeyed Marcus cross relation [2,10]. The present test, just as the cross relation, is less dependent on model calculations of the reorganization free energies R_x and R_e than previous comparisons of experimental and predicted values of $\Delta G_x^{\ddagger}$ [2] and $\Delta G_e^{\ddagger}$ [11].

2. Reorganization free energy for photoelectron emission by solutions

Photoelectron emission by a species M^{z+} ($z \gtrless 0$) into the vapor phase of the solution of M^{z+} is represented by the two equations

$$M^{z+}(z,z) = M^{(z+1)+}(z+1,z) + e^-(g), \tag{1}$$

$$M^{(z+1)+}(z+1,z) = M^{(z+1)+}(z+1,z+1). \tag{2}$$

The symbol (z,z) denotes that both electronic and nuclear configurations of the ligand (if any) and solvent correspond to the ionic charge z+. The same definition applies to (z+1,z+1) for the ionic charge (z+1)+. Conversely, (z+1,z) indicates that the electronic configuration pertains to the charge (z+1)+ whereas the nuclear configuration corresponds to the charge z+ (Franck-Condon principle). Thus, the species produced by (1) reorganizes according to process (2).

The free energy of emission ΔG_m for (1) in the case of aqueous solutions is given by [7-9]

$$\Delta G_m = \Delta G_H + \Delta G - R_m. \tag{3}$$

There, ΔG_H (= 4.50 eV) correlates the free energy level of the normal hydrogen electrode to the electron vacuum level on the assumption of a negligible surface potential; ΔG is the change of standard free energy for the oxidation of $M^{z+}(z,z)$ to $M^{(z+1)+}(z+1,z+1)$; R_m (< 0) is the reorganization free energy for (2). The free energy ΔG_m was shown [7-9] to be equal (within ca. $\pm$0.1 eV) to the threshold energy determined from experimental emission spectra. Thus, R_m can be determined experimentally by application of eq. (3) provided that ΔG is known.

The reorganization free energy R_m can be subdivided into inner- and outer-sphere contributions as is done in electron transfer theories for the quantities R_x (chemical) and R_e (electrochemical). Thus,

$$R_m = R_m^{in} + R_m^{out}. \tag{4}$$

The outer-sphere contribution is interpreted by treating the solvent macroscopically as a continuous medium, and the inner-sphere contribution is treated microscopically [1,2].

The outer-sphere contribution R_m^{out} is calculated by application of the Marcus theory [12] of non-equilibrium polarization. By starting with eqs. (6.59) and (6.62) in [2] or eq. (25) in [12], one obtains (rationalized units)

$$R_m^{out} = -(\varepsilon_o/2)(\varepsilon_{op}^{-1} - \varepsilon_s^{-1})\int_V (\vec{E}_{z+1} - \vec{E}_z)^2 dV, \tag{5}$$

where ε_o is the permittivity of free space; ε_{op} and ε_s are the optical and static dielectric constants of the solvent, respectively; $\vec{E}_{z+1}$ and $\vec{E}_z$ are the electric fields <u>in vacuo</u> of the charges $(z+1)|e|$ and $z|e|$, respectively; and V is the integration volume. It will be assumed to simplify matters that the ions involved have the same size. This is a minor approximation. The volume V extending to infinity is supposed to surround a sphere of radius a for both fields in (5), and the Coulombic potential is integrated from r = a to ∞. Thus,

$$R_m^{out} = -(1/4\pi\varepsilon_o)(\varepsilon_{op}^{-1} - \varepsilon_s^{-1})e^2/2a. \tag{6}$$

The contribution R_m^{out} given by (6) is independent of the ionic charge of the species emitting photoelectrons. Thus, R_m^{out} according to this model should be the same for emission by $M^{z+}(z,z)$ and $M^{(z+1)+}(z+1,z+1)$ ions provided the radius a is the same. Conversely, the inner-sphere contribution R_m^{in} is expected to depend on the ionic charge z of the emitter because the force constants determining its magnitude [2,11] vary with z. The dependence of R_m on z nevertheless should not be a strong one because R_m^{out} is markedly larger than R_m^{in} (sec. 3). The application of the Born equation with the factor $(\epsilon_{op}^{-1} - \epsilon_s^{-1})$ to the calculation of the reorganization free energy in [8] leads to a $(z+1)^2 - z^2$ dependence and is not justified.

3. Correlation between $\Delta G_x^{\ddagger}$ for electron exchange and R_m for photoelectron emission

Electron exchange between cations of ionic charge z+ and (z+1)+ (isotope labeling) will be considered in this section. The change of free energy is equal to zero in that case. Moreover, the work w_r required to bring from infinity the two reactants together in the activated complex is equal to the work w_p needed to perform the same operation for the products. Hence, $w_p - w_r = 0$, and one has [1-4]

$$\Delta G_x^{\ddagger} = w_r + R_x/4. \tag{7}$$

A relationship between R_x of (7) and R_m of (3) will be obtained. The method of calculating R_x is briefly recalled for that purpose. The outer-sphere contribution R_x^{out} is calculated [1-4] by assuming that the separation between the two ions of the activated complex is sufficient to preserve the spherical symmetry of the field of each ion but not sufficient to allow the neglect of Coulombic interaction. R_x^{out} is then given by an equation identical to (6) (with a plus sign) and the factor $(1/2a_1 + 1/2a_2 - 1/r_{1,2})$ instead of 1/2a. The change of sign arises because R_x is

defined in terms of the difference of free energies between the activated complex and the product of electron transfer (eq. (6.73) in [2]) whereas R_m is the change of free energy for process (2). The quantities a_1 and a_2 are the radii of the ions and $r_{1,2}$ is the distance between the centers of the reactants in the activated complex. In general, the radius a_i is set equal to the sum of the crystallographic radius of the ion and the diameter (2 x 1.38 Å) of the water molecule [13]. Moreover, one sets $r_{1,2} = a_1 + a_2$. If one assumes $a_1 = a_2 = a$, the final equation for R_x^{out} is the same as eq. (6) except for the sign. Thus, $R_x^{out} = |R_m^{out}|$ according to this model. Two ions are involved as reactants in the exchange reaction whereas photons are absorbed by single ions. Hence, $R_x^{in} = 2|R_m^{in}|$. In view of eq. (4) and a similar relationship for R_x, one has

$$R_x = |R_m| + |R_m^{in}|. \tag{8}$$

<u>This is the relationship needed to test eq. (7)</u>. The quantity R_m is experimental (cf. eq. (3)) and R_m^{in} is calculated for a bond-stretching model from eq. (6) in [10].

The computation of R_x by application of (8) does not require the use of the continuous medium model and the attending selection of the radius a and the usual assumption of dielectric unsaturation. The use of eq. (8) therefore removes the uncertainty resulting from the model computation of the outer-sphere contribution R_x^{out} in the verification of eq. (7). Equation (8), it should be noted, is not rigorous because it was obtained by assuming that the fields of the two reactants in the activated complex are spherically symmetrical. This is an approximation for which somewhat tentative corrections have been proposed [2]. Such corrections will not be attempted here to preserve the simplicity of the treatment.

Equations (7) and (8) were applied to the calculation of $\Delta G_x^{\ddagger}$ and comparison of the results with experiment. The work w_r in (7) was

calculated from [3]

$$w_r = z(z+1)(1/4\pi\epsilon_0\epsilon_s)(e^2/2a)F(\mu), \tag{9}$$

where the function $F(\mu)$ of the ionic strength μ corrects the Coulombic term in (9) for finite dilution. The linearized expression [14], $F(\mu) = (1 + 2a\kappa)^{-1}$, was used where κ is the reciprocal Debye length. This expression for $F(\mu)$ and the corresponding exponential form $\exp(-2a\kappa)$ are very crude at high ionic strength. Fortunately this does not matter much because w_r is small in comparison with $\Delta G_x^{\ddagger}$ for the systems studied here.

Values of $\Delta G_x^{\ddagger}$ calculated from (7) and (8) are compared with the experimental results in Table 1. The agreement is excellent for Mn^{2+}, Fe^{2+}, Co^{2+} but not as good for the other ions. The discrepancy may have several possible causes: e.g., kinetic complications in the experimental determination of $\Delta G_x^{\ddagger}$, transmission coefficient appreciably smaller than unity, departure from the medium-overlap case assumed in [3,4]. Errors in the determination of R_m from threshold energy measurements could hardly account for the discrepancies in Table 1.

The value of $|R_m^{out}| = |R_m| - |R_m^{in}|$ computed from the data in Table 1 will be compared with the values of $|R_m^{out}|$ calculated for the continuous-medium model. The latter is 1.14 ± 0.01 eV for the five cations [11] whereas the values of $|R_m^{out}|$ calculated from the data in Table 1 are 1.72, 1.47, 1.28, 1.65, 1.85 eV from V^{2+} to Co^{2+}. These values hold within ca. ± 0.3 eV because of the uncertainty on R_m (surface potential, extrapolation method) and the approximations and estimates in the computation of R_m^{in}. The minimum for Mn^{2+} matches perhaps not fortuitously the minimum in the corresponding sequence of the absolute values of the free energies of solvation [15], 19.12, 19.28, 18.92, 19.58, 20.85 eV. It seems that the continuous-medium model yields values of $|R_m^{out}|$ which are too low for the values of a in Table 1 for the five cations examined here.

The opposite holds for $Fe(CN)_6^{4-}$, but the difference between the value of 0.86 eV from the model [11] and 0.29 eV from Table 1 appears too great and probably reflects errors, at least in part (preliminary value of R_m in [9] from analysis of literature data). The foregoing conclusion about the relative inadequacy of the continuous-medium model is not surprising since this model is not very satisfactory either in the treatment of ionic solvation [16,17].

4. Correlation between $\Delta G_e^{\ddagger}$ for electron transfer at electrodes and R_m for photoelectron emission

The counterpart of (7) for electron transfer at electrodes at zero overvoltage is [5,10]

$$\Delta G_e^{\ddagger} = w_r + (R_e/4)[1 + (w_p - w_r)/R_e]^2. \tag{10}$$

There w_r is the work done to bring the reactant from the bulk of the solution to the site near the electrode at which electron transfer occurs. The same definition applies to w_p for the product of the reaction. The work terms in (10) are of the form $w_r = z|e|\phi$ and $w_p = (z+1)|e|\phi$ for the oxidation of a species with ionic charge $z|e|$ to one with charge $(z+1)|e|$. The potential ϕ can, in principle, be calculated from double layer theory. Thus, $w_p - w_r = w_r/z$, and eq. (10) becomes, after neglecting the term $(1/z^2)(w_r/R_e)^2$,

$$\Delta G_e^{\ddagger} = (1 + 1/2z)w_r + R_e/4. \tag{11}$$

The reorganization free energy R_e is given according to Marcus [5,10] by an equation similar to (6) except that the factor 1/2a is replaced by $(1/2)(a^{-1} - r_f^{-1})$, where a is now the distance from the center of the reactant to the electrode and $r_f = 2a$ pertains to the image forces. The value of a of sec. 3 is used in practice. Thus, the factor 1/2a of eq. (6) is now replaced by 1/4a, and therefore $R_e^{out} = |R_m^{out}/2|$. Taking $R_e^{in} = R_m^{in}$, one obtains

$$R_e = (1/2)[|R_m| + |R_m^{in}|]. \tag{12}$$

By comparing (8) and (12) one verifies that $R_e = R_x/2$ in agreement with [5,10]. This relationship is not rigorous because it corresponds to a simplified treatment of the image force problem.

Equations (11) and (12) are the counterparts of (7) and (8). The quantity $\Delta G_e^{\ddagger}$ can be calculated from R_m provided that R_m^{in} and w_r can be computed. The term R_m^{in} is treated as in sec. 3, but the calculation of w_r from double layer theory is tentative at best (except for the mercury electrode [11]). We therefore use R_m and experimental values of $\Delta G_e^{\ddagger}$ to compute w_r. The electrochemical work term w_r can also be computed as follows from the experimental values of $\Delta G_x^{\ddagger}$ and $\Delta G_e^{\ddagger}$ of Tables 1 and 2. Thus, R_x is computed from (7) using the experimental $\Delta G_x^{\ddagger}$'s and calculated chemical w_r's from Table 1. The quantity R_e follows from $R_e = R_x/2$ according to Marcus [5,10]. The electrochemical work w_r is then computed from (11) using the experimental $\Delta G_e^{\ddagger}$'s from Table 2. The agreement between the two sets of electrochemical w_r's in Table 2 is very good especially for a comparison involving three different experimental methods ($\Delta G_x^{\ddagger}$, $\Delta G_e^{\ddagger}$, R_m).

5. Conclusion

Excellent to fair agreement was obtained between experimental activation free energies for chemical and electrochemical electron transfer in solution and the values predicted from the corresponding Franck-Condon determined reorganization free energies for photoelectron emission. The results reported here support the basic theoretical model [3-5] for electron transfer in solution used in this work.

Acknowledgment

This work was supported by the Office of Naval Research and the National Science Foundation. The author thanks Professor R. A. Marcus for pointing out the need for a finite dilution correction in (9) and the model-free character of cross relation tests (sec. 1).

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Table 1

Correlation between $\Delta G_x^{\ddagger}$ for electron exchange and R_m for photoelectron emission

	$-R_m$ a) (eV)	$-R_m^{in}$ b) (eV)	a b) (A)	w_r c) (eV)	$\Delta G_x^{\ddagger}$ (calculated) (eV)	$\Delta G_x^{\ddagger}$ d) (experimental) (eV)
V^{2+}	2.13	0.41	3.48	0.04	0.68	0.87
Cr^{2+}	2.05	0.58	3.49	0.05	0.71	1.03
Mn^{2+}	2.02	0.74	3.56	0.03	0.72	0.75
Fe^{2+}	2.11	0.46	3.51	0.06	0.70	0.69
Co^{2+}	2.26	0.41	3.48	0.05	0.72	0.72
$Fe(CN)_6^{4-}$	0.67	0.38	4.65	0.04	0.30	0.47

a) From [8] except for $Fe(CN)_6^{4-}$ (preliminary result [9] from analysis of literature data)).

b) From [11].

c) From (9) with $F(\mu)$ calculated for the prevailing μ's in the experimental determination of the $\Delta G_x^{\ddagger}$'s.

d) From [2] and references cited therein.

Table 2

Correlation between $\Delta G_e^{\ddagger}$ for electron transfer at electrodes and R_m for photoelectron emission

	R_e a) (eV)	$\Delta G_e^{\ddagger}$ b) (experimental) (eV)	w_r c) (eV)	w_r d) (eV)
V^{2+}	1.27	0.38	0.05	-0.03
Cr^{2+}	1.32	0.54	0.17	0.04
Mn^{2+}	1.38	0.43	0.07	0.06
Fe^{2+}	1.29	0.37	0.04	0.04
Co^{2+}	1.34	0.59	0.20	0.20
$Fe(CN)_6^{4-}$	0.53	0.30	0.15	0.08

a) From values of R_m and R_m^{in} in Table 1 and eq. (12).

b) From [11] and references therein.

c) From eqs. (11) and (12).

d) From experimental values of $\Delta G_x^{\ddagger}$ and $\Delta G_e^{\ddagger}$ (see text).

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