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Note

Conversion constants for redox potentials measured versus different reference electrodes in acetonitrile solutions at 25°C

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Abstract

Caution should be exercised when one is comparing the redox potentials of complexes measured in CH₃CN solutions versus different reference electrodes. A critical review of conversion constants amongst various reference electrodes reported in the literature reveals that in most cases the comparisons of redox potential values are far from accurate. From measurements of relationships amongst various common reference electrodes in CH₃CN solutions, conversion constants for redox potentials measured versus different reference electrodes in acetonitrile solutions at 25°C are proposed, along with a convention for reporting redox potential values. © 2000 Elsevier Science S.A. All rights reserved.

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1. Introduction

The number of publications devoted to the redox chemistry of transition metal complexes in nonaqueous solutions has increased markedly in the last few years. This is especially true for those intended as models for the active sites of metalloproteins. These properties are consequently discussed in monographs [1,2] and reviews [3–5]. The redox potential of such species (normally denoted E_f or $E_{1/2}$ for $ML^{n+}/ML^{(n-1)+}$) is a key parameter that reflects not only the relative thermodynamic stabilities of the central metals' oxidation states, but also determines the chemical reactivity of such a complex in redox reactions.

However, use and comparison of these redox potential values is complicated by the fact that recalculation

is necessary when different reference electrodes have been used for measurements. The choice of the reference electrode is frequently determined by personal biases of the person carrying out the experiment, as well as the kinds of solvent, supporting electrolyte and complex. The saturated calomel electrode (SCE)¹ has been historically one of the most frequently used reference electrodes in laboratory practice, because of the inconvenience of the standard hydrogen electrode (SHE). However, its application in nonaqueous solutions is expected to be limited by: (a) water leakage into the nonaqueous phase and (b) the frequent incompatibility of KCl with these media, particularly when they contain perchlorate ion [70]. Thus, various nonaqueous half-cells based on silver salts have been introduced as useful reference electrodes [6], which also have the benefit of eliminating that part of the junction potential

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¹ Any references to a *standard* calomel electrode thus go undetected.

due to the phase boundary. Use of the ferrocenium/ferrocene redox couple as an internal standard has also become widespread during the last decade [7–9].

Nonetheless, the collation of potentials measured versus different reference electrodes continues to be a significant annoyance in reporting and discussing literature data. The $E_{1/2}$ for Fc^+/Fc in CH_3CN appears most commonly stated as being +400 mV versus the normal hydrogen electrode (NHE, vide infra) [7,8] and most authors adopt this in order to recalculate data standardised against Fc^+/Fc . This requires [7,9,10] the (actually false [11,12]) extrathermodynamic assumption that the redox potential of this couple is invariant with solvent². Furthermore, even recent publications give conflicting statements about the Fc^+/Fc potential in acetonitrile (either +450 or +400 mV versus SCE [4,13]). A logical inference from these statements could be that the redox potentials of couples measured against either of these two (SCE and NHE) electrodes could be reported as being the same, were it not for the fact that the potential of the SCE is actually +244 mV versus the SHE at 25°C [14]. The situation is worsened by the still-frequent usage of ‘NHE’ ($E_{\text{r}} = -6 \text{ mV}^3$) when ‘SHE’ is probably usually intended — an occurrence common despite its inappropriateness, as clearly pointed out by Ramette [15].

Redox potentials reported versus the Fc^+/Fc couple in some cases are also of poor reliability, because the cited $E_{1/2}$ for Fc^+/Fc has not actually been measured under the same experimental conditions [15–18], and/or has been substituted by a dogmatic reliance [19,20]⁴ on one of the values (usually +400 mV versus SCE) iterated in the literature. However, even a brief review (see Table 3) shows that this potential changes in an amazing manner as a function of geography and time, making it extremely difficult to correctly compare various $E_{1/2}$ values measured against an ‘ Fc^+/Fc standard’ without appropriate conversion.

Nonaqueous silver electrodes using silver nitrate or perchlorate are reliable reference electrodes for CH_3CN and other nonaqueous solutions [11]. However, the exact conversion scales for potentials measured against these electrodes are not always apparent, because the actual Ag^+ -concentration or salt anion in the Ag^+/Ag electrode is not reported in most papers. Also, some publications [5] report potentials against such a reference electrode without any indication of what would be involved in conversion of such potentials to a more

fundamental scale. Such logical absences and discrepancies in the recent literature lead to confusion when one needs to recalculate $E_{1/2}$ values measured versus different electrodes. In some accelerated communication journals, such as *Chem. Commun.* and *Angew. Chem.*, the rules for reporting redox potential values are lax and generally potentials reported in these journals are useless for external numerical comparison.

Therefore, we performed direct measurements of these reference electrodes versus each other in order to confirm the relationships amongst their $E_{1/2}$ values.

2. Experimental

Potentiometry measurements in acetonitrile solutions (thermostatted at $25.0 \pm 0.3^\circ\text{C}$) were performed using a PAR-173 potentiostat and a Radio Shack 22-163 digital multimeter. The SCEs were from Fisher Scientific and Broadley-James Inc., the saturated sodium chloride calomel electrode (SSCE) was itself prepared from a commercial SCE. Silver was 99.99% wire (Aldrich), CH_3CN was distilled off P_4O_{10} under N_2 , ferrocene (Sigma) was vacuum-sublimed, the supporting electrolytes tetraethylammonium perchlorate (TEAP) and tetraethylammonium hexafluorophosphate (TEAH) were prepared by metathesis between aqueous NET_4Br and NaClO_4 or HPF_6 , recrystallized from water (TEAP) or ethyl acetate (TEAH) and dried in vacuo over P_4O_{10} . TEAH is more suitable for direct potential measurements versus an SCE, because salt exchange at the junction in acetonitrile is less problematic [70]. However, in this study it was found that over the course of several minutes (a typical time for these measurements) the potential measured even versus an SCE immersed in $\text{CH}_3\text{CN}/\text{TEAP}$ was stable and was the same as measured in $\text{CH}_3\text{CN}/\text{TEAH}$ solutions. The electrodes used were: Ag^+ (0.1 M AgNO_3 , 0.1 M TEAP, CH_3CN)/Ag (‘ANE1’); Ag^+ (0.01 M AgNO_3 , 0.1 M TEAP, CH_3CN)/Ag (‘ANE2’); Ag^+ (0.001 M AgNO_3 , 0.1 M TEAP, CH_3CN)/Ag (‘ANE3’); Ag^+ (0.01 M AgClO_4 , 0.1 M TEAP, CH_3CN)/Ag (‘APE’) an SSCE and the SCE. These were measured pairwise. The Ag^+ -ion electrodes were separated from the bulk solution by a ‘thirsty’ Vycor™ frit. Experiments were also performed in which the SCE was separated from the bulk solution by a salt bridge containing 0.1 M tetraethylammonium perchlorate, chloride or iodide in acetonitrile in order to modulate the SCE junction transport consequences [8,57]. Our original choice [58] of AgClO_4 was based on the similar transfer numbers for its ions and the generally weaker complexing nature of ClO_4^- compared with NO_3^- [10,11,59]. Two different SCEs were used and were within 2 mV of each other. The redox potentials for Fc^+/Fc against these

² Although Strehlow and co-workers [9] demonstrated that $[E(\text{Fc}^+/\text{Fc}) - E(\text{CoCp}_2^+/\text{CoCp}_2)]$ is almost solvent-invariant at +1.32 V.

³ Arises because the cation activity coefficient < 1.00 . We did not investigate the alternative ‘ANE1’, viz. Ag^+ (0.1 M AgNO_3 , CH_3CN)/Ag.

⁴ The redox potential of Fc^+/Fc couple in MeCN was used in this paper as a reference for redox potentials in 1,2-dichloroethane.

Table 1
Conversion constants between different reference electrodes in CH₃CN at 25°C

↓From\to→	ANE1	ANE2	ANE3	APE	Fc ⁺ /Fc	NHE	SCE	SSCE	SHE
ANE1	0	+45	+97	+47	-37	+593	+343	+347	+587
ANE2	-45	0	+52	+2	-87	+548	+298	+302	+542
ANE3	-97	-52	0	-50	-133	+496	+246	+250	+490
APE	-47	-2	+50	0	-83	+546	+296	+300	+540
Fc ⁺ /Fc	+37	+87	+133	+83	0	+630	+380	+384	+624
NHE	-593	-548	-496	-546	-630	0	-250	-246	-6
SCE	-343	-298	-246	-296	-380	+250	0	+4	+244
SSCE	-347	-302	-250	-300	-384	+246	-4	0	+235
SHE	-587	-542	-490	-540	-624	+6	-244	-240	0

reference electrodes in 0.01 M TEAP/CH₃CN solutions were measured by cyclic voltammetry using a PAR-173/175/176/178 system or a Bioanalytical Systems BAS-100 electrochemical analyzer. The $E_{1/2}$ values were estimated as $1/2 (E_{pa} + E_{pc})$; (ΔE_p were 70 mV at $\nu = 100 \text{ mV s}^{-1}$). A three-electrode cell was used, the working electrode being a Pt-wire.

3. Results and discussion

The potentials obtained for the various electrodes referenced to the SCE were:

APE: $+296 \pm 2 \text{ mV}$
 ANE1: $+343 \pm 2 \text{ mV}$
 ANE2: $+298 \pm 2 \text{ mV}$
 ANE3: $+246 \pm 2 \text{ mV}$
 SSCE: $-4 \pm 2 \text{ mV}$

while the APE was at $+4 \pm 2 \text{ mV}$ versus the corresponding nitrate electrode ANE2, so that the variation in activity coefficient for Ag⁺ in CH₃CN as a function of anion and concentration is apparent.

Adding the well-known relationship between the SCE and the SHE [14], the various electrode potential difference values permit us to propose the correct conversions amongst $E_{1/2}$ values measured against them (Table 1):

This means that, e.g. in order to convert from $E_{1/2}$ values reported versus APE to the SCE scale, one should

Table 2
Experimental ($E_{1/2\text{-obsd}}$)^a and 'predicted' ($E_{1/2\text{-pred}}$) Fc⁺/Fc $E_{1/2}$ values measured vs. different reference electrodes in TEAP/CH₃CN at 25°C

Reference electrode	$E_{1/2\text{-obsd}}$ (mV)	$E_{1/2\text{-pred}}$ (mV)
APE	+82	$+84 \pm 2$
ANE(2)	+87	$+85 \pm 2$
SCE	+380	$+378 \pm 2$

^a By cyclic voltammetry at $\nu = 100 \text{ mV s}^{-1}$.

add 296 mV. It should be noted that these apply only to TEAP or TEAH used as an electrolyte, with or without a bridge. We found that the iodide salt bridge increased the apparent potentials for the APE and ANE2 by 10 mV, whereas the use of NEt₄Cl in the bridge led the potential to drift constantly in time in the positive direction. The same results were observed in the ferrocene cyclic voltammetry study. Previous recommendations [69] regarding the use of salt bridges are reinforced.

For instance, there appears to be a significant difference ($\sim 60 \text{ mV}$) between our values and the literature value (240 mV [4]) for conversion constants to SCE in systems with the usual APE and ANE2, although neither the Ag⁺ concentration nor the counterion were specified therein. These data also disagree with those in a recent report [60] in which ANE2 was stated to be at $+340 \text{ mV}$ versus SCE; again, however, the origin of that conversion was not stated.

As a check, we also used Table 1 to estimate the $E_{1/2}$ values expected for Fc⁺/Fc against each of these electrodes. The $E_{1/2}$ for Fc⁺/Fc values calculated in this manner were then directly measured by cyclic voltammetry of the Fc⁺/Fc couple redox potentials versus APE, ANE2 and SCE. The coincidence of the calculated and experimental values in Table 2 further evidences the applicability of the proposed conversions and suggests the typical experimental error involved in the $E_{1/2}$ values. No attempt was made here (or in other empirical work) to insert any additional adjustment for liquid junction potential; in any case, it would be difficult for a general agreement to be reached. These conversions are approximate to the extent that even at 25°C, the actual potential is influenced by other experimental conditions such as the supporting electrolyte concentration.

Special attention should be paid to the fact that the currently often-quoted values [7–9,18–20] for the E_f of Fc⁺/Fc of $+400$, $+410$ or $+450 \text{ mV}$ versus SHE are untenable, the actual value being around $+624 \text{ mV}$. On the other hand, results emanating from earlier, more detailed experiments [11] indeed yield values of $E_{1/2}$ for

Table 3
Literature data for $E_{1/2}$ of Fc^+/Fc couple^a in CH_3CN

$E_{1/2}$ (mV)	Reference electrode	Year	Ref.	$E_{1/2}$ (mV vs. SCE) ^a
348	NHE	1965	[43]	99
400	NHE	1994	[22]	151
400	NHE	1998	[21,39]	151
400	NHE	1970	[45]	151
400	NHE	1986	[20]	151
160	SCE	1986	[20]	160
410	NHE	1998	[32,33]	161
556	NHE	1979	[55]	307
315	SCE	1964	[56]	315
326	SCE	1965	[43]	326
342	SCE	1981	[38]	342
410	Ag/AgCl	1982	[52]	371
74	ANE2	1965	[43]	372
376	SCE	1986	[36]	376
380	SCE	1997	[29]	380
380	SCE	1990	[5]	380
421	Ag/AgCl	1998	[44]	382
90	APE	1972	[12]	386
430	Ag/AgCl	1994	[23]	391
50	ANE1	1994	[27]	393
400	SCE	1994	[4]	400
310	SCE	1996	[35]	310
400	SCE	1990	[51]	400
410	SCE	1991	[47]	410
450	Ag/AgCl	1994	[28]	411
665	NHE	1985	[30]	416
665	NHE	1998	[31]	416
665	NHE	1990	[50]	416
425	SCE	1985	[37]	425
425	SCE	1991	[48]	425
430	SCE	1998	[34]	430
430	SCE	1999	[41]	430
450	SCE	1998	[24]	450
480	SCE	1998	[26]	480
365	SSCE	1984	[54]	361
580	Ag/AgCl ^b	1987	[53]	
510	AgCl/Ag ^c	1991	[49]	
400	None given	1996	[40]	
400	Ag ^d	1998	[25]	
400	Ag ^d	1998	[42]	
409	Ag ^d	1991	[46]	

^a Adjusted to SCE in accordance with literature constants and those in this work. All $E_{1/2}$ values are positive in sign unless otherwise denoted.

^b In acetone.

^c EtOH, LiCl.

^d Silver wire pseudo-reference electrode, $[\text{Ag}^+]$ unspecified.

Fc^+/Fc which are close to those reported here, as do several other more recent reports [5,12,29,36,44] (Table 3). In some reviews [61], potentials have been summarised by reference to the Fc^+/Fc couple with little attention being paid to the fact that the Fc^+/Fc $E_{1/2}$ values used by the various research groups quoted in turn differed quite significantly (Table 3). The speculative nature of the assumption that

organometallic couples possess the same potential in different solvents [10] (leading to the direct comparison of $E_{1/2}$ values for various couples measured in different solvents) should not be forgotten either.

Care should be taken when one is comparing data from different papers, wherein potentials are referred to an 'Ag/Ag⁺ electrode'. Different research groups have used different Ag⁺ salts and concentrations, although mainly the nitrate and/or at 0.1 [32,62] or 0.01 M [11,13,63–65]⁵, the difference in potential between these two electrodes being 52 mV⁶. However, traditionally in reviews all nonaqueous silver electrodes seem to be treated as if they were of the same composition and thus only one conversion constant is used in order to recalculate potentials of diverse origins [4,66].

Considerable skepticism must be exercised when one is using the numerous literature data reported against SHE or especially the NHE. Our conversion constants for the APE and ANEs versus SHE [13,14,32,33] are indeed in the range of the +560 mV most often proposed as the recalculation constant [67] for various 'Ag⁺/Ag' electrodes. Recently, conversions of +300 and +520 mV from the ANE2 values to the SCE and NHE, respectively in $\text{CH}_3\text{CN}/0.1 \text{ M NBu}_4\text{ClO}_4$ were reported [68], which are also close to those estimated experimentally herein. Several recent papers in the field of organic electrochemistry also quote similar and self-consistent values for various ANEs [71,72].

In principle, one might expect $E_{1/2}$ values to be consistent within ca. ± 3 mV between different laboratories. However, we suspect that ± 10 mV is a more realistic margin at this stage. IUPAC has promulgated recommendations regarding reporting electrode potentials in nonaqueous solvents [69], and research in this area continues [73]. Nonetheless, ambiguities may be eradicated from publications in which authors (1) report the actual experimental $E_{1/2}$ of some standard redox couple, such as Fc^+/Fc versus their chosen reference electrode and (2) indicate exactly how conversions from that or any other reference electrode are being made.

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⁵ Actually, we previously reported in this journal +545 mV as a conversion constant from APE to SHE instead of +540 mV.

⁶ See footnote 3.

cluded in Table 3. V.V.P. personally appreciates the persistence of H.R.C.

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