
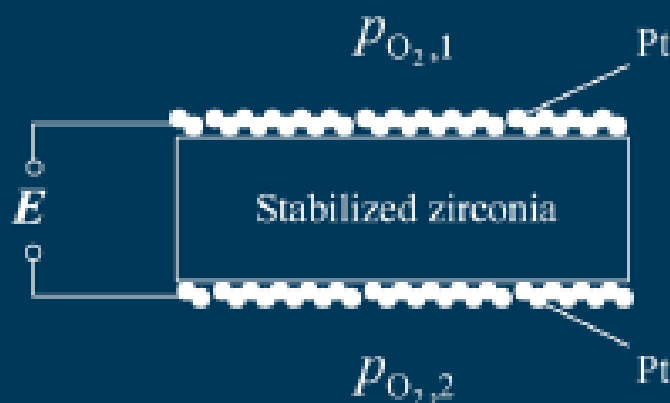


Allen J. Bard · György Inzelt · Fritz Scholz  
(Eds.)

# Electrochemical Dictionary

With 469 Figures and 18 Tables

 Springer



Allen J. Bard · György Inzelt · Fritz Scholz *Editors*

# Electrochemical Dictionary

with more than  
**2.770**  
entries

$$E = E^{\ominus} + \frac{RT}{nF} \ln \frac{a_{\text{ox}}}{a_{\text{red}}}$$

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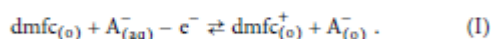
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# Three-phase electrode

## Rubin Gulaboski RG

→ *three-phase boundaries* [i], i.e., when droplets of a liquid are attached to a solid electrode that is immersed into another liquid that is immiscible with the droplet-phase. The three-phase arrangement allows performing a simultaneous electron and ion transfer reactions, giving access to the → *Gibbs energies of ion transfer*: A droplet of a solution of an electroactive and lipophilic compound is dissolved in a water-immiscible liquid and the droplet is attached to the surface of a working electrode, e.g., a → *paraffin-impregnated graphite electrode*. The Figure depicts the coupled electron and ion transfer reactions for the example of decamethylferrocene (dmfc). The overall process occurring at the three-phase electrode can be described by the following reaction scheme:



The thermodynamic treatment to the reaction scheme (I), leads to the following equation [ii, iii]:

$$E_c^{\text{Ox/Red}} = E_{\text{dmfc}_{(o)}^+/\text{dmfc}_{(o)}}^{\text{Ox/Red}} + \frac{\Delta_W^{\text{org}} G_{\text{A}^-}^{\text{Ox/Red}}}{F} - \frac{RT}{F} \ln(c_{\text{A}^-}_{(w)}) + \frac{RT}{F} \ln\left(\frac{c_{\text{dmfc}_{(o)}}}{2}\right) \quad (I)$$

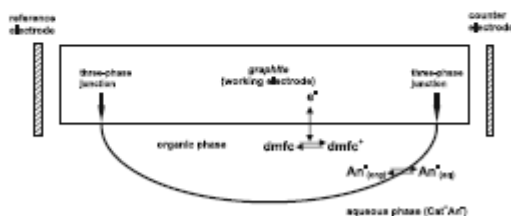
Similarly, the reduction of an *electroreducible* lipophilic compound will provoke the transfer of cations from the aqueous to the organic phase:



The thermodynamic treatment of the reaction given in Eq. (2) leads to the following form of the → *Nernst equation*, Eq. (2) [i]:

$$E_c^{\text{Ox/Red}} = E_{\text{Ox}_{(o)}^+/\text{Red}_{(o)}^-}^{\text{Ox/Red}} - \frac{\Delta_W^{\text{org}} G_{\text{Cat}^+}^{\text{Ox/Red}}}{F} + \frac{RT}{F} \ln(c_{\text{Cat}^+}_{(w)}) + \frac{RT}{F} \ln\left(\frac{2}{c_{\text{Ox}_{(o)}}}\right) \quad (2)$$

Various mechanistic studies concerning the electrochemistry of redox active liquids have been reported by Marken and Compton et al. [iv, v].



Droplets, electrochemistry of immobilized ~ — Figure. Scheme of the processes taking place at a three-phase electrode

Refs.: [i] Scholz F, Schröder U, Gulaboski R (2005) *Electrochemistry of immobilized Particles and droplets*. Springer, Berlin, pp 183–253; [ii] Scholz F, Komorsky-Lovrić Š, Lovrić M (2000) *Electrochem Commun* 2:112; [iii] Scholz F, Gulaboski R (2005) *ChemPhysChem* 6:16; [iv] Banks CE, Davies TJ, Evans RG, Hignett G, Wain AJ, Lawrence NS, Wadhawan JD, Marken F, Compton RG (2003) *Phys Chem Chem Phys* 5:4053; [v] Davies TJ, Banks CE, Compton RG (2005) *J Solid State Electrochem* 9:797

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**Dropping mercury electrode (DME)** — Polarizable electrode formed by sequence of mercury drops falling from a small aperture. After its first description in 1871 [i] it was explored by many authors in connection with development of the concept of → *electrode potential* [ii]. In that way it represented a scientific problem, and only after 30 years it was first used as a scientific tool [ii]. At that time the DME was mostly formed at the end of a narrow glass capillary connected by elastic tubing with a reservoir of mercury. By changing the height of mercury level in the reservoir above the orifice of the capillary the time of formation of the drop was usually adjusted to several seconds. With radius of the orifice of the capillary of less than 0.2 mm the shape of the drop is strictly spherical. Such DME is then characterized by two parameters: rate of flow of mercury  $m$  (in grams per second) and drop-time  $t_1$  (in seconds). Its weight  $w$  can then be expressed thus:  $w = mt_1g = 2\pi r\gamma$ , where  $g$  is gravitational constant (980.67 cm per  $s^2$ ),  $\pi$  is 3.14,  $r$  is radius of the orifice of the capillary (in cm), and  $\gamma$  is surface or interfacial tension (in dynes per cm). On the basis of this relation → *Kučera* introduced a method of determining interfacial tension between polarized mercury, and a solution by measuring drop-weight or drop-time [ii], later I. Oref [iii] used DME for studying the adsorption from the gas phase. — Mercury as electrode material (→ *mercury, and mercury electrodes*) has specific advantages: a) it has of all metals the highest → *hydrogen overpotential*, which enables polarization of the electrode to fairly negative potentials; b) the surface of mercury drops is homogeneous and isotropic, allowing sensitive and precise study of adsorption; in addition, DME has special features: c) periodic renewal of ideal liquid surface, unaffected by preceding events, which leads to reproducible results in electrolytic measurements irrespective of direction of polarizing scan; d) the small size of the mercury drop guarantees negligible consumption of electroactive species during electrolysis, which is the condition of quantitative reproducibility of electrolytic measurements. Aware of these advantages → *Heyrovský* used DME for electrolysis [iv], which developed into



resultant current–voltage (or current–time or current–voltage–time) display is commonly referred to as the “voltammogram” [i–ii]. The term “voltammetry” was coined by → *Laitinen* and → *Kolthoff* [iii–iv]. The protocol for potential control may vary, leading to a number of different techniques each with their common names. See, for example → *AC voltammetry*, → *pulse voltammetry*, → *cyclic voltammetry*, → *stair case voltammetry*, → *faradaic rectification voltammetry*, → *Fourier transform voltammetry*, → *hydrodynamic voltammetry*, → *electrochemical impedance spectroscopy*, → *linear scan voltammetry*, interdigitated array voltammetry, → *microelectrode voltammetry*, → *polarography*, → *rotating disk voltammetry*, → *scanning electrochemical microscopy (SECM)*, → *square-wave voltammetry*, → *stripping voltammetry*, → *ultramicroelectrode voltammetry*. The objectives of a voltammetric experiment also vary, ranging from analytical measurements designed to determine the concentration of an analyte to measurements designed to elucidate complex mechanisms and the values of the associated thermodynamic and kinetic (homogeneous and heterogeneous) parameters [i].

Refs.: [i] Bard AJ, Faulkner LR (2001) *Electrochemical methods*, 2<sup>nd</sup> edn. Wiley, New York; [ii] Oldham KB, Myland JC (1994) *Fundamentals of electrochemical science*. Academic Press, San Diego; [iii] Laitinen HA, Kolthoff IM (1941) *J Phys Chem* 45:1061; [iv] Laitinen HA, Kolthoff IM (1941) *J Phys Chem* 45:1079

**Voltammetry, cyclic** → *cyclic voltammetry*

**Voltammetry of Immobilized microparticles (VIM)** — Technique for studying the → *electrochemistry* of solid particles, esp. microparticles, that are insoluble (or very poorly soluble) in a certain → *electrolyte* solution. The particles are mechanically transferred to the surface of a suitable electrode. That electrode should have a rather soft surface as to allow mechanical embedding of the particles in the electrode surface. Very well suited are → *paraffin-impregnated graphite electrodes*, however, other → *graphite electrodes* also can be used. The transfer of particles to the electrode surface can be accomplished by placing the sample powder on a glass plate, on a glazed tile, or on filter paper and rubbing the electrode on the sample spot. Although the amount of transferred sample cannot be controlled exactly, it can be determined in many cases from the electrochemical measurements (integration of current versus time or potential curves), and the characteristic potentials of voltammetric measurements can be evaluated in a quantitative way. The technique was initially developed to analyze the

quantitative composition of alloys [i] where the transfer of alloy traces to the electrode was achieved by rubbing the electrode surface on a clean alloy surface (→ *abrasive stripping voltammetry*). Later the technique was employed in studies of various materials, ranging from alloys, minerals, inorganic powder mixtures, metal complexes, oxides etc. to metal organic compounds and organic dyes. The technique found application for analytical studies and basic electrochemical research of reactions of solid particles [ii, iii].

Refs.: [i] Scholz F, Nitschke L, Henrion G (1989) *Naturwissenschaften* 76:71; [ii] Scholz F, Meyer B (1998) *Voltammetry of solid microparticles immobilized on electrode surfaces*. In: Bard AJ, Rubinstein I (eds) *Electroanalytical chemistry*, vol. 20. Marcel Dekker, New York; [iii] Scholz F, Schröder U, Gulaboski R (2005) *Electrochemistry of immobilized particles and droplets*. Springer, Berlin

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**Voltammogram** — A plot of → *current* versus → *potential* as measured in → *voltammetry*. When voltammetry is performed using a → *dropping mercury electrode*, i.e., → *polarography*, the voltammogram is traditionally termed → *polarogram*.

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**Voltfluorometry** — may also be called volt-fluorimetry, is a technique to study the → *ion transfer at liquid–liquid interfaces* driven by the change in the phase-boundary potential (→ *potential*, subentry → *surface electric potential*) as a change in fluorescence intensity [i]. The excitation light is introduced from the organic solvent phase, e.g., 1,2-dichloroethane, so that the condition of the total internal reflection at the liquid–liquid interface is satisfied. The change in the fluorescence intensity in the organic solvent phase is proportional to the total quantity of fluorescent ions transferred from the aqueous phase to the organic phase, and, hence, a fluorescence intensity vs. potential curve is essentially isomorphic to a → *coulometry* response. The sensitivity of voltfluorometry is, at least, 1000 times higher than that of coulometry or → *voltammetry*; a voltfluorogram for the transfer of 2 nanomolar fluorescent ions can be detected [ii]. The method can also be applied to the transfer of nonfluorescent ions such as alkali and alkaline earth metal ions if a fluorescent → *ionophore* is present in the organic phase [iii]. The adsorption of transferring ions at the interface is sensitively detected by the voltfluorometry technique with ac modulation of the applied voltage [iv].

Refs.: [i] Kakiuchi T, Takasu Y, Senda M (1992) *Anal Chem* 64:3096; [ii] Kakiuchi T, Takasu Y (1994) *Anal Chem* 66:1853; [iii] Kakiuchi T,

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