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Electrochemical Instrumentation - From Potentiostat to Artificial Intelligence

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Abstract

Electrochemical instrumentation has advanced over the past several decades from the early potentiostats to the present evolution towards AI integration, profoundly impacting all aspects of electrochemical studies. Heyrovský's polarograph introduced controlled potential analysis with a dropping mercury electrode, enabling voltammetric quantification of metals and organics. Later, Hickling's potentiostat invention established precise potential control via three-electrode feedback, supporting techniques like Cyclic Voltammetry (CV) and chronoamperometry. The turning point was the advent of Operational amplifiers, which enabled stable, low-noise measurements, expanding to Electrochemical Impedance Spectroscopy (EIS) and Rotating Disk Electrode (RDE) methods for kinetic and mechanistic studies. Digital potentiostats incorporated fast AD/DA converters, facilitating pulse techniques (DPV, SWV) with superior sensitivity for trace analysis and fast-scan CVs. The PCB-based potentiostats further advanced this era by integrating all circuitry onto compact printed circuit boards, reducing size, cost, and power consumption while maintaining high precision for field-deployable electroanalysis. Modern electroanalysis leverages AI for real-time data processing, automating equivalent circuit fitting in EIS and anomaly detection in noisy datasets. Machine learning models predict redox behaviours, deconvolute overlapping signals in multi-analyte sensors, and optimise scan parameters adaptively. This progression from manual polarography to AI-augmented potentiostats has elevated the quality of research contributions by driving breakthroughs in biosensing, energy electrochemistry such as batteries, fuel cells and materials characterisation.

Keywords: Artificial Intelligence (AI), Electroanalysis, Electrochemical Impedance Spectroscopy (EIS), PCB-based Potentiostats, Potentiostat, Polarography

1. Introduction

The development and application of potentiostats have been a turning point in the progress of experimental electrochemistry and several electroanalytical techniques. The primary goal in these studies is to obtain meaningful quantitative and mechanistic information from electrochemical systems under controlled potential or current as may be desired for any particular study. Among the various classes of techniques, voltammetry is the earliest and was first introduced by Laitinen and Kolthoff in 1940. It broadly refers to the several methods involving the measurement of electrical current as a function of electrode potential[1].

Electrochemical reactions are dictated by well-defined potentials, and therefore a stringent control of electrode potential is indispensable for the systematic study of these processes. However, maintaining a desired potential at the test (or working) electrode is not an easy exercise due to dynamic changes in the concentrations of reactants, intermediates, and adsorbed species during the reaction. This makes it an appropriate electronic instrumentation to set the desired potential and current control indispensable for such studies.

1.1 Historical Development of the Potentiostat

Initially, the control of electrode potentials employed simple circuitry with battery-powered supplies, resistors and galvanometers. However, these arrangements were inadequate for producing the stability required for precise

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measurements. The decisive advance was due to Hickling[2] in 1942 through the introduction of the three-electrode potentiostat system using vacuum triodes and thyristors[3]. For the first time, an automatic real-time control of electrode potential was achieved by means of applying negative feedback within a 3-electrode system comprising a working electrode (potential-controlled), a reference electrode (potential-measured), and a counter electrode (current path).

Arguably, the modern potentiostat design owes very much to the work of Wenking in Germany, whose use of transistor amplifiers and analogue circuits greatly enhanced the stability and versatility of the potentiostat[4]. Unlike previous designs, which commonly grounded the counter electrode and left both working and reference electrodes "floating", Wenking grounded the working electrode, aligning the potentiostat's operation with that of a modern operational amplifier. His implementation featured a differential input stage, high-gain amplification, common-mode rejection, and dynamic internal feedback- features which are all present in contemporary operational amplifier-based potentiostats. Wenking's approach not only improved phase stability but also allowed precise tracking of fast kinetic processes, setting a new benchmark for electrochemical instrumentation that persists today. With his design, the potentiostat became an indispensable tool in electrochemical studies. Though Wenking never attempted to publish his work in any journal, he made the complete design and layout available in the instrument manuals. With his mastery of analogue electronics, he developed several other instruments for electrochemists, such as integrators and waveform generators, which produced true linear scans down to a few millivolts per hour[4].

1.2 Operational Amplifier-based Potentiostats

The advent of Integrated Circuit (IC) technology, especially the widespread adoption of operational amplifiers (op-amps), represented a revolutionary advance in potentiostat design. The Op-amp-based potentiostats offer high bandwidth, low noise, and simplified integration of signal generation, potential control, and signal processing. It also facilitated seamless integration of the device with a variety of Electrochemical techniques based on the potential and current range requirements. It is no exaggeration to say that the operational amplifier-based potentiostats have given rise to newer and more versatile adoption of electrochemistry as a premier analytical and research tool in the laboratories.

A typical configuration of a potentiostat design includes a control amplifier, a voltage follower, and a current-to-voltage converter, tightly coupled to maintain the working electrode potential with respect to the reference electrode by a feedback loop. These advances now enable the study of complex reactions with greater resolution and reliability.

2. Development of Handheld Potentiostats

Potentiostats developed in the late 1970s and beyond were primarily built around two-stage operational

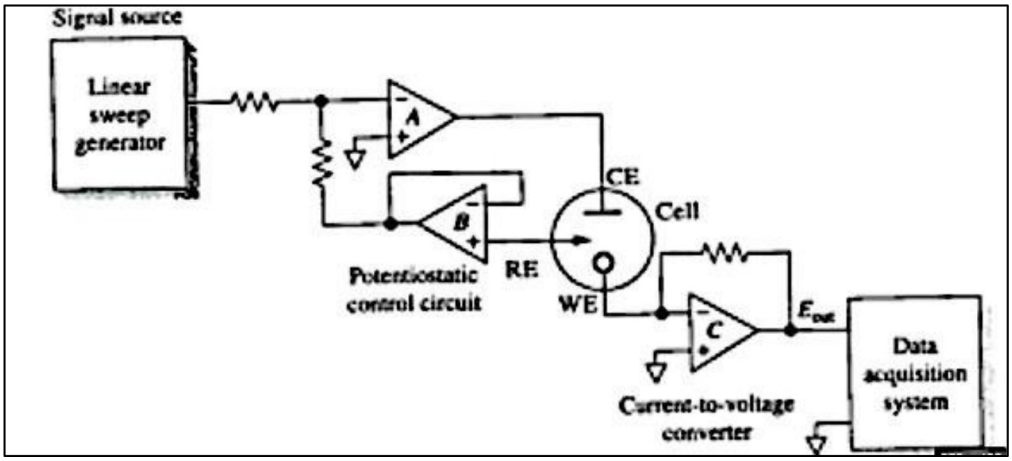


Figure 1. Block diagram of a typical Op amp based potentiostat. RE,CE and WE are Reference, Counter and Working Electrodes. A,B,C are operational amplifires

amplifier circuits, incorporating a control amplifier and a voltage follower within the classic three-electrode configuration. Later, the operational amplifiers handled the functions of input signal generation and current-to-voltage conversion in addition to post-signal processing. This gave rise to the development of devices with increasing functionality as newer electroanalytical techniques started emerging. Over time, enhancements such as programmable Analog-to-Digital (ADC) and Digital-to-Analog (DAC) converters became standard features, enabling more flexible input signals and automated current readouts.

The subsequent simplification and miniaturisation of potentiostat designs were necessitated by the need for handheld potentiostats, which were required for health care and environmental monitoring devices. They are also facilitated by the advent of integrated circuits containing multiple operational amplifiers (quad op-amps) and the widespread adoption of Surface-mount Device (SMD) technology. Today, potentiostat hardware PC cards are being manufactured by companies such as Texas Instruments and Analogue Devices, which can be readily programmed for different electrochemical techniques.

2.1 The AD5940/5941 Platform: Advanced Analogue Front End

The AD5940 analogue front end, alongside its variant the AD5941, represents another significant milestone in the miniaturisation and performance of electrochemical instrumentation[5]. Designed for high-precision, low-power operation, the AD5940 is particularly well-suited for bioimpedance and general electrochemical measurements. It is capable of accurate assessments of voltage, current, and impedance, making it ideal for applications ranging from skin or body impedance analysis to electrochemical gas and liquid sensing.

A defining feature of the AD5940 is its multi-parameter measurement capability, made possible by a high-speed ADC that rapidly switches between current, voltage, and impedance channels through a programmable multiplexer and switch matrix. This design allows for effectively simultaneous measurements on multiple sensors. The system includes two excitation loops, one with an ultra-low-power DAC and ADC for AC signals up to 200 kHz, enabling both time-domain and frequency-domain analysis. The AD5940's current consumption is remarkably low, as the potentiostat draws as little as 6.5 μA as a bias current, making it very well-suited for wearable and portable applications, especially for home-based health monitoring.

3. Applications and Future Directions

The versatility and integration seen in platforms like AD5940 have catalysed a wide range of applications. In the health sector, these devices find applications in continuous glucose monitors and bio impedance analysis[6]. In Industrial settings, they are employed for battery and fuel cell impedance analysis, as well as for monitoring air quality and other electrochemical sensor arrays. Their small size, low power consumption, and autonomous operation also make them ideal for wearable diagnostic devices and distributed sensor networks. These innovations herald a future in which real-time, on-site, and even personal electrochemical measurements become routine.

3.1 Polarography and the Evolution of Electrochemical Instrumentation

Prior to the development of polarography, electrochemical analyses were largely restricted to gravimetric and potentiometric techniques. The discovery of polarography by Heyrovsky[7], utilising a Dropping Mercury Electrode (DME), revolutionised the field by enabling the systematic study of electrode kinetics and adsorption processes. Polarography not only led to innovative techniques within electroanalysis but also spurred the development of associated instrumentation.

The term "polarography" was formally introduced in 1924, denoting methods that monitor the current–potential relationship using a DME. Over time, "voltammetry" emerged to describe a broader class of techniques utilising small electrodes and variable potential, now encompassing a range of specialised methods. Early polarographic measurements were laborious and relied on manual recording with galvanometers, whereas modern approaches leverage electronic instrumentation and digital interfaces for precise, rapid, and versatile analysis.

It is widely acknowledged that the widespread adoption of polarography in the latter half of the 20th century played a pivotal role in the development of modern electrochemical instrumentation. The evolution of polarographic strategies led directly to the genesis of numerous voltammetric techniques, including Cyclic

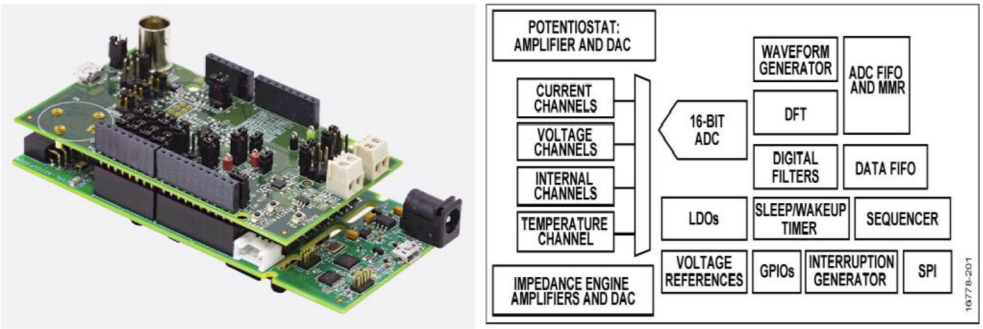


Figure 2. **(a)** AD5940 analogue front end, **(b)** Functional block diagram (from the analogue device data sheet).

Voltammetry (CV), Differential Pulse Voltammetry (DPV), Normal Pulse Voltammetry (NPV), and Square Wave Voltammetry (SWV). The integration of operational amplifiers into experimental setups revolutionised the study of electrode kinetics and mechanisms, directly stimulating the invention and refinement of new electroanalytical methods.

The versatility of polarography, in accommodating a diversity of modes—such as DC, AC, and pulse techniques—necessitated the design of increasingly sophisticated potential-controlling devices. These requirements spurred the creation of rapid-response potentiostats, high-precision operational amplifier current followers, as well as advanced devices for signal processing and conditioning. Of particular importance was the persistent need to effectively distinguish double-layer charging currents from faradaic currents arising from electrochemical processes. This necessitated the development of specialised instrumental techniques and circuit designs capable of such discrimination.

3.2 Faradaic and Capacitive (Double Layer Charging) Currents

A core consideration in polarography and related techniques is the distinction between faradaic and capacitive (charging) currents. The faradaic current refers to the current flow due to the electron transfer processes of interest, corresponding to the redox reactions at the electrode surface. In contrast, the charging (capacitive) current arises from the transient storage of charge at the interface between the electrode and the electrolyte in response to potential change, similar in behaviour to a charging capacitor. The latter component, particularly pronounced at the initial stages of a voltage sweep, adds to the faradaic signal. Since the faradaic current is, in fact is of actual interest for analytical purposes, it is essential to separate it from the charging current in the total measured current.

The charging (*i.e.*, capacitive) current decays more quickly than the faradaic component and approaches zero at the end of the mercury drop period. To accurately interpret faradaic processes and derive mechanistic or analytical conclusions, compensation or elimination of this charging current is necessary. The charging current can be modelled as:

$$I_{ch} = A(E - E_z)\left(\frac{dC_{dl}}{dE}\right)\left(\frac{dE}{dt}\right) + C_{dl}A\left(\frac{dE}{dt}\right) + (E - E_z)C_{dl}\left(\frac{dA}{dt}\right)$$

where, A is the electrode area, C_{dl} the double layer capacitance, the applied potential, and E_z the potential of zero charge.

9. Enhancing Analytical Sensitivity through Charging Current Compensation

The challenge of optimising the ratio between faradaic current and double-layer charging current has historically driven significant advancements in electroanalytical methodology and instrumentation[8]. Notably, Barker's pioneering contributions in this area established the conceptual and instrumental foundation for what can be regarded as the second generation of polarography[9]. Barker introduced the innovative technique of superimposing an Alternating Current (AC) signal onto a Direct Current (DC) input, thereby enabling more precise correction of capacitive or double-layer charging currents. Furthermore, the development of square wave and pulse polarography methods under his leadership provided enhanced discrimination between faradaic and non-faradaic

currents, significantly improving analytical sensitivity. These methodologies also underpinned further theoretical developments in transient electrochemical phenomena.

10. Instrumental Strategies for Improved Sensitivity in Polarography

The sensitivity achieved in classical DC polarography is fundamentally constrained by the unavoidable presence of double-layer charging current. Quantitatively, when the diffusion current approaches the magnitude of the charging current, for example, at a ratio of 1:1—analytical quantitation becomes unreliable.

To address this constraint, A. Poojary and S.R. Rajagopalan demonstrated that non-linear charging currents in DC polarography could be systematically separated from the total electrolytic current[10,11]. Their method involved the application of a small AC voltage perturbation to the cell, subsequent rectification of the cell's AC response, analogue integration of this signal, and the subtraction of a suitably scaled resultant from the total DC response. The researchers designed and developed an instrument implementing this automatic nonlinear charging current compensation, leveraging the capabilities of the then-emergent operational amplifiers and integrated circuit switches. Experimental results established that this approach successfully extended the quantifiable concentration range by two orders of magnitude, achieving detection limits as low as 100 nM.

Further methodological refinement was articulated through the development of three in situ techniques for Charging Current Compensation (CCC) in DC polarography. These techniques are predicated on the superposition of an AC ripple on the DC signal, coupled with analogue processing of the readout to recover a component proportional to the DC charging current. The systematic separation and compensation of charging currents in polarography represent significant progress in electrochemical analysis, enabling more accurate detection of trace analytes. Improvement in the analytical detection limit was not confined to DC polarography alone, but extended to the development of charging current elimination in AC polarography by a novel peak sampling technique where the charging current is effectively zero when the current is sampled at the peak of the applied AC potential.

The method of charging current compensation was not just confined to the polarography technique alone. The method of sampling the potential or current at the at specific life time of the transient response to the potential perturbation in order to separate the faradaic current from double-layer charging current was developed by Lakshminarayanan and Rajagopalan[12]. This strategy led to the development of a novel method of measurement of instantaneous corrosion rates using galvanostatic exponential relaxation techniques[13].

11. Impact of Polarography in other Electrochemical Techniques

The evolution and expanding popularity of polarography, particularly in the latter half of the twentieth century, have been instrumental in shaping the field of electrochemical instrumentation. Techniques derived from polarography such as cyclic voltammetry, differential pulse, normal pulse and square wave voltammetry have driven the need for sophisticated potential control devices, fast and precise potentiostats and advanced signal processing architectures. As a result, operational amplifier-based circuits and digital processing methods now form the basis of cutting-edge electrochemical instrumentation.

12. Recent Advances in Electrochemical Instrumentation: The Role of Scanning Probe Microscopy, Mass Spectrometry, and Artificial Intelligence

The landscape of electrochemical instrumentation has undergone profound transformation with the advent of scanning probe techniques, advanced mass spectrometry integration, and the incorporation of Artificial Intelligence (AI). These innovations have collectively enhanced the molecular-level understanding of electrochemical reactions and driven significant progress in materials science, catalysis, biosensing, and diagnostics.

13. Scanning Probe Microscopy in Electrochemistry

The introduction of Scanning Probe Microscopy (SPM) heralded a new era in the investigation of electrochemical phenomena at surfaces, providing molecular and, in some cases, atomic resolution. Scanning Tunnelling Microscopy (STM), invented by Gerd Binnig and Heinrich Rohrer at IBM in the 1980s, which won them the Nobel Prize for Physics in 1986, was pivotal in allowing direct atomic-scale imaging of conductive surfaces[14]. Immediately after this development, there was a flurry of activity in probing the surfaces; several laboratories around the world came out with a variety of homemade STMs. One of the early homemade STMs was developed in the author's laboratory, which uses a simple 'inertial sliding' mechanism for positioning the tip to the tunnelling distance of the Sample[15]. Rooted in the principle of quantum tunnelling, STM soon became invaluable for electrochemists for examining electrode reactions, as both surface science and electrochemistry share a focus on interfacial phenomena.

The evolution of SPM techniques proceeded with the development of Atomic Force Microscopy (AFM), enabling imaging of surfaces regardless of their electrical conductivity[16]. This extension was crucial for mapping organic molecules, biomolecules, and macromolecular processes at the molecular level, especially on non-conductive substrates. Consequently, EC- STM and AFM became indispensable for studying a range of systems, from Organic thin films to monolayers on single crystal surfaces, biological membranes[17].

14. Electrochemical Scanning Tunnelling Microscope (EC-STM)

EC-STM uniquely adapts STM for use in an electrochemical environment, where both the tip and substrate are immersed in an electrolyte and participate in redox processes[17]. Integration of a bipotentiostat is essential, as it independently controls the electrochemical potentials of both electrodes relative to a common reference, ensuring precise bias control for imaging. As a result, EC-STM facilitates real-time observation of adsorption, desorption, restructuring, and other surface processes at atomic resolution, under in situ electrochemical conditions.

EC-STM is based on the quantum mechanical tunnelling of electrons between a sharp metallic tip and a conductive sample surface. When the tip is brought extremely close (within a nanometer) to the surface, and a bias voltage is applied, electrons tunnel through the gap, generating a tunnelling current. This current is highly sensitive to the tip-sample distance, allowing for atomic-scale resolution. The EC-STM instrumentation integrates the core hardware of a scanning tunnelling microscope with specialised components for electrochemical control, enabling real-time, atomic-scale imaging of electrode surfaces during electrochemical reactions, adsorption, desorption and surface restructuring.

Unlike traditional STM, in EC-STM both the tip and the sample are immersed in an electrolyte solution. A bipotentiostat is used in EC-STM to independently control the electrochemical potentials of both the STM tip and the sample (substrate) with respect to a common reference electrode. Both electrodes are immersed in an electrolyte and can participate in redox reactions. A bipotentiostat ensures that the potential of each electrode can be set and maintained independently, allowing accurate control of the bias for STM imaging. The tunnelling current in STM depends on the potential difference (bias) between the tip and the sample. In an electrochemical environment, both electrodes are immersed in an electrolyte and can participate in redox reactions. A bipotentiostat ensures that the potential of each electrode can be set and maintained independently, allowing accurate control of the bias for imaging and spectroscopy.

15. Scanning Electrochemical Microscopy (SECM)

Coinciding with the early advances in EC-STM, the SECM was introduced by A.J.Bard's group[18]. Distinct from imaging-focused SPM techniques, SECM enables the mapping of local electrochemical activity by scanning a biased Ultramicroelectrode (UME) probe over a substrate in an electrolytic medium. The measured Faradaic current is shaped by hemispherical diffusion of a redox mediator around the probe, providing fine spatial resolution and mechanistic insights.

The mode of operation in SECM is decided on the basis of the particular information to be sought from the experiment. In the feedback mode, a conductive substrate regenerates the redox mediator, boosting probe current

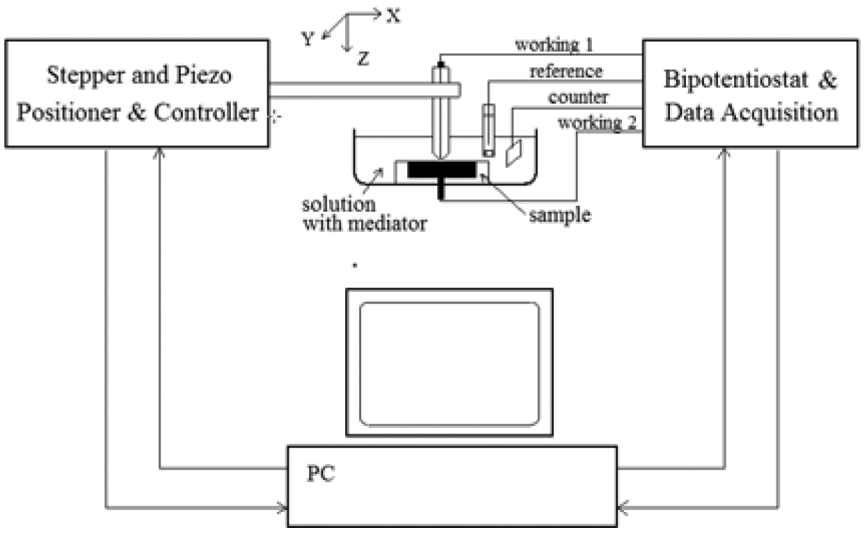


Figure 3. Block diagram of the Scanning Electrochemical Microscope with a bipotentiostat, which controls the potential of the tip and substrate with respect to the reference electrode[19,20].

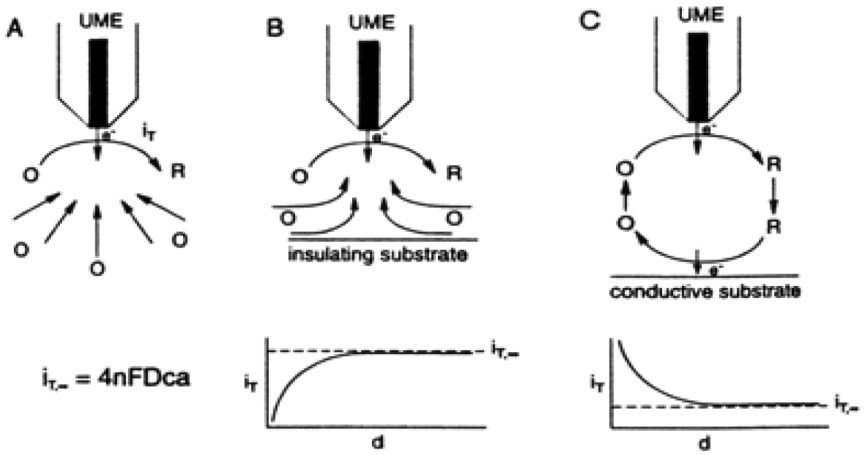


Figure 4. **(A)** The redox process at the UME, **(B)** Positive feedback mode on the conductive surface, **(C)** Negative feedback mode on an insulating surface[20].

and thus highlighting areas of catalytic activity. On the other hand, an insulating region suppresses the current due to diffusion hindrance. In the redox competition mode, which is more specialised, the positions of both probe and substrate are in direct competition for the same redox species. This competition enables sensitive mapping of catalytic activity. The tip current drops over catalytically active regions, allowing visualisation and comparison of catalyst spots or enzyme-modified surfaces. The high spatial and mechanistic resolution of SECM makes it a versatile tool in catalysis research, biosensing, material science, and energy applications, as it complements the primarily topographical information yielded by Electrochemical Scanning probe techniques.

16. Electrochemical Mass Spectrometry[21,22]

Electrochemical Mass Spectrometry (EC-MS), including its variant Differential Electrochemical Mass Spectrometry (DEMS), provides direct and real-time analysis of volatile and gaseous products from electrochemical reactions. In EC-MS, the electrochemical cell's reaction products permeate a hydrophobic membrane that interfaces with a mass spectrometer, which then detects and quantifies these species based on their mass-to-charge ratios. DEMS further integrates the control and data acquisition functions of the potentiostat with the mass spectrometer, achieving synchronised, real-time measurement of potential, current,

and product evolution. This integration is crucial for linking the formation of volatile products to specific electrochemical events, thereby offering insights into reaction mechanisms and kinetics. While these techniques excel at analysing volatile products, their selectivity and operational requirements are shaped by the choice of pervaporation membrane, with materials like PDMS and PVA being particularly common.

17. Artificial Intelligence in Electrochemical Instrumentation[23,24]

The incorporation of AI into electrochemical instrumentation is fostering a paradigm shift, especially in biosensing, materials discovery, and portable diagnostics. AI integration enhances the sensitivity and selectivity of electrochemical biosensors, which is a key advantage when analysing complex biological samples where traditional statistical or rule-based methods struggle with overlapping signals from similar analytes.

Biosensor data, particularly in personalised healthcare and telemedicine, often consists of large, complex datasets that cannot be efficiently analysed by manual or simple algorithmic means. AI algorithms are leveraged to categorise and interpret such data, aid in quick diagnosis, and propose effective treatment pathways. Furthermore, AI-driven wearable biosensors, capable of seamless integration with smartphones, are opening avenues in continuous health monitoring and real-time fitness tracking.

A notable technical challenge in point-of-care diagnostics using portable potentiostats has been the prevalence of noise and interference. Recent research demonstrates how De-noising Auto Encoders (DAEs), a subset of machine learning models, can be trained to transform noisy measurement data by simulating conditions of portable devices into clean data matching the fidelity of benchtop potentiostats. DAE models are trained on paired datasets of clean and artificially noised signals, enabling real-time correction of measurements in use.

Beyond noise reduction, AI-enabled systems are being developed for automatic calibration, interference detection, and correction for interfering analytes in biosensing. This is particularly consequential where signals from structurally similar molecules, such as dopamine and ascorbic acid, overlap, complicating clinical diagnostics[25]. AI-based pattern recognition aids in extracting meaningful information from these congested electrochemical signals, thus improving accuracy and lowering the turnaround time for diagnostics, which provide key benefits in resource-limited settings.

Machine learning is also rapidly accelerating the discovery and optimisation of functional materials[26]. Rather than relying solely on empirical or physics-based models, machine learning-based material design leverages data-driven pattern recognition to predict promising candidates and mechanisms, expediting innovation across energy storage, catalysis, and related fields.

18. Conclusion

The convergence of several electrochemical techniques along with scanning probe microscopy, advanced mass spectrometric techniques, and artificial intelligence has impacted modern electrochemical instrumentation profoundly. The ability to investigate dynamic surface phenomena at the molecular level, map catalytic and biological activity at micrometre and nanometer scales, and link chemical reactivity to specific electrode events is providing fresh opportunities to understand reaction mechanisms. Meanwhile, AI's transformative potential is just beginning to be realised, promising further advances in sensitivity, data interpretation, diagnostic speed, and the rational design of complex electrochemical systems.

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