ELECTROCHEMICAL SENSORS: MECHANISMS , APPLICATIONS IN ANALYTICAL CHEMISTRY, BIOTECHNOLOGY.

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ABSTRACT

Due to the fact that there is a strong need for sensors and that there are several technical advancements being made, the field of sensors is quite diversified and is expanding at a quick rate. Electrochemical sensors offer a solution that is both cost-effective and convenient for the detection of changeable analytes. These sensors find widespread application in a variety of industries, including agriculture, food, and oil, within the context of environmental and biological applications. The low theoretical detection limits that originate from the differences in the Faradaic and nonFaradaic currents are the primary reasons for the popularity of electrochemical sensing. The variability of the reporting signals, such as the voltage, current, overall power output, or electrochemical impedance, is another advantage that contributes to the popularity of electrochemical sensing. The purpose of this review article is to attempt to cover the most recent developments and applications of electrochemical sensors in a variety of areas of business. An investigation is also conducted into the role that nanomaterials play in the study and development of electrochemical sensors. With the knowledge that has been offered here, we expect that additional efforts will be encouraged to be made toward the study and advancement of electrochemical sensors.

Keywords:*Electrochemical sensors, applications in analytical chemistry.*

INTRODUCTION

The International Union for the Pure and Applied Chemistry (IUPAC) defines a chemical sensor as "a device that converts chemical data, ranging from the concentration of a single sample component to complete composition analysis, into a signal that can be used for analytical purposes." A receptor and a physicochemical transducer are the two elements that make up the majority of a chemical sensor. These are the two essential functional components that make up a chemical sensor. The receptors are diverse and can take the form of activated or doped surfaces, as well as complex (macro)molecules that generate highly specific interactions with the analyte.

In the event where the receptor is derived from biological sources, such as DNA, antibodies, or enzymes, the product in question is referred to as a biosensor. As a result of the interaction between the receptor and the analyte, the recognition event is transformed into a signal that is preset. Keeping a high degree of specificity for the analyte that is being measured in the presence of chemical species that could potentially interfere with the measurement is one of the most important requirements that sensors must fulfill in order to prevent false-positive results. The transducer is another essential component of sensors. Its primary function is to transform the signal that is produced by the interaction between the receptor and the analyte into a value that can be seen

and understood. In light of this, chemical and biosensors can be categorized as either affinity-based or catalytic devices. Affinity-based devices rely on highly specific interactions between the receptor and analyte, such as using the specific affinity of nucleic acids (i.e., ssDNA and aptamers), antibodies–antigens, or host–guest interactions. Catalytic sensors, on the other hand, generate the signal through the use of catalytic activity. This is the case in the case of enzymatic, DNAzyme, or functionalized surfaces that are capable of performing redox reactions under specific conditions. Depending on the type of transducer that is being utilized, the monitoring of the recognition events can be carried out utilizing a variety of approaches, such as gravimetric, electrochemical, or optical methods.

Because of their advantages, electrochemical sensors are by far the most often used type of sensor. These advantages include low detection limits, which can be as low as picomoles, rapidity, and the low-cost equipment that is used for sensing. Electrochemical sensors are the market leaders. Electrochemical sensors are available in a wide range of form factors, ranging from fully integrated wearable devices to top-bench sensors. The purpose of a chemical sensor is to provide precise information in real time about the chemical composition of the environment in which it is located. If all went according to plan, a device like this would be able to reply in a continuous and reversible manner without affecting the sample in any way. A biological or chemical identifying layer is coated on a transduction element during the manufacturing process of such devices. When it comes to electrochemical sensors, the information that is used for analysis is derived from the electrical signal that is generated as a result of the interaction between the recognition layer and the drug of interest. Depending on the nature of the analyte, the characteristics of the sample matrix, and the requirements for sensitivity or selectivity, a number of different electrochemical devices can be utilized for the purpose of environmental monitoring. A significant number of these devices can be classified into a number of different categories, including amperometric and potentiometric electrochemical sensors (depending on the nature of the device). The use of amperometric sensors allows for the detection of electroactive species that are engaged in the process of chemical or biological identification.

OBJECTIVES

1. To study electrochemical sensors.

2. To study applications in analytical.

Types of Electrochemical Sensors

It is possible to divide electrochemical sensors into a number of distinct categories, such as amperometric, potentiometric, impedimetric, photoelectrochemical, and electrogenerated chemiluminescence. For potentiometric sensors, a local Nernstian equilibrium is produced at the sensor interface as a result of certain sensor–analyte interactions. This equilibrium occurs when the system is not permitted to flow any current, which provides information about the concentration of the analyte. According to the Cottrell equation, amperometric sensors use a voltage that is applied between a reference electrode and a working electrode in order to commence electrochemical oxidation or reduction. The measurements of the current that is produced as a result of this process serve as a quantitative indicator of the concentration of the analyte on the sensor.

$$i = \frac{nFAc_j^0 \sqrt{D_j}}{\sqrt{\pi t}}$$

Where,

i = Current (in ampere);

n = Number of electrons;

F = Faraday constant (96,485 C/mol);

A = Area of the (planar) electrode in cm2

C_j^0 = Initial concentration of the reducible analyte {\displaystyle j}in mol/cm3

D_j= Diffusion coefficient for species {\displaystyle j}in cm2/s;

t = time in seconds.

On the other hand, conductometric sensors, which are also frequently referred to as impedimetric sensors, monitor changes in the surface impedance in order to detect and quantify analyte-specific recognition events that occur on the electrode. The extraordinary success of electrochemical sensor research, as well as its growing influence on analytical chemistry, makes it difficult to address all of the achievements that fall within the scope of this review. As a result, our goal was to demonstrate the variability in the field, rather than to completely immerse ourselves in a particular type of electrochemical sensor.

Electrochemical Sensor Applications

Electrochemical sensors have been highly sought after for the research of biological, environmental, industrial, and pharmaceutical species for a considerable amount of time. This is not only due to their long-term dependability, high sensitivity, and accuracy, but also because to their low cost, speed, and ease of downsizing. In order to improve analytical performance, several nanomaterials with remarkable properties have been used into electrochemical assays for more than twenty years. These nanomaterials include metals, conductive polymers, metal oxides, and frameworks based on metal–organic and carbon-based nanomaterials. This modification makes it possible to increase the loading capacity by utilizing recognition molecules, such as enzymes, antibodies, and aptamers, in addition to bioinspired receptors. These receptors have the ability to capture targets in a specific and efficient manner, which in turn increases the specificity of the electrochemical sensors. There is a close connection between this and the objective of delivering powerful electrocatalytic activity for specific electromagnetic chemical processes. In addition, it is feasible to increase both the electrical conductivity and the surface area of the surface by modifying the shape and structure of the surface. This should result in an increase in the sensitivity of these tests. Recently, electrochemical sensors have become increasingly popular as a result of the introduction of novel applications. These applications include single-molecule sensing, in vivo analysis, wearables, and point-of-care diagnostic imaging.

There are a number of benefits associated with electrochemical sensors. These benefits include their high sensitivity, which enables low limit of detection (LOD) and limit of quantification (LOQ) values; their rapid analytical response, which makes them ideal for flow analysis and alert systems; their simplicity, which enables a virtually limitless variety of geometries, electrode materials, and configurations; and their ease of use, which includes the ability to be integrated as a detection module in a variety of analytical systems.

Research methodology

The reagents. Poly(vinyl chloride) (PVC) high molecular weight; plasticizer bis(2-ethylhexyl) sebacate (DOS), heptyl-4-trifluoroacetylbenzoate (Carbonate Ionophore I), 4-butyl-α,α,α-trifluroacetophenone (Carbonate Ionophore IV), tetradodecyl ammonium chloride (TDACL), potassium tetra-p-chlorophenylborate (TpClPBK), tetrabutylammonium perchlorate (TBAClO4), 2-amino-2-(hydroxymethyl)- 1,3-propanediol (TRIS), tetrahydrofuran (THF), acetonitrile (ACN), dimethylformamide (DMF), pyridine, and aniline were purchased from Sigma-Aldrich. The 5,10,15-tris(4- aminophenyl)-20-phenyl porphyrinates of copper(II) and cobalt(II) were produced in accordance with the procedures described in the literature and were completed characterized using nuclear magnetic resonance and ultraviolet-visible spectroscopy. The remaining compounds were of an analytical grade, and hence, they were utilized without any additional purification. In the preparation of each and every solution, distilled water was utilized.

Evaluation and preparation of the sensors being used. The preparation of PVC membranes was carried out in accordance with a standard pattern. A membrane with a weight of 100 mg comprised between 1.5 and 3.5 weight percent of ionophore and/or 1 to 6 weight percent of lipophilic additive, which was distributed in a polymeric matrix composed of PVC and DOS 1:2. In order to enhance the solubility of an ionophore, the components of the membrane were dissolved in 1 milliliter of trifluoroethanol (THF), and 7 weight percent of pyridine was added to membranes III and IV. After that, membrane cocktails were cast out on a flat GC electrode surface that had been previously buffed with alumina slurries, cleaned in an ultrasonic bath, rinsed with methanol, and dried on air. The diameter of the electrode was three millimeters. Evaporation of THF was permitted to take place overnight. By using cyclic voltammetry, porphyrin electropolymers (EP) were formed on platinum wires with a surface diameter of three millimeters. These electropolymers were derived from solutions of 1.0 mM/L Co(II)TATPP or Cu(II)TATPP and 0.1 M/L TBAClO4 in the following solvents: (a) acetonitrile, (b) DMF, (c) pyridine, and (d) DMF: 0.5 M aniline in 1 M/L H2SO4. A deoxygenation process was performed on the solutions by bubbling N2 for ten minutes before to the experiment. By using an AMEL 7050 potentiostat (made in Italy), the potential of WE was scanned at a rate of 50 mV per second. The range of potentials that were cycled was from -0.2 to 0.8–1.4 V versus SCE. As a counterelectrode, a wire with a thickness of 0.5 millimeters was utilized.

Table 1: Membrane compositions and deposition details

	Ionophore, wt%	Additive, wt%	Film/solvent
I.a-d	Co(II)TATPP	—	EP/(a-d)[a]
II	Cu(II)TATPP	—	EP/(d)

III	Co(II)TATPP, 1.5%	TDACl, 1%	PVC/DOS
IV	Co(II)TATPP, 1.5%	—	PVC/DOS
V	Carb.Ion I, 2.7%	TDACl, 2%	PVC/DOS
VI	Carb.Ion IV, 3.5%	TDACl, 2%	PVC/DOS
VII	—	TDACl, 6%	PVC/DOS
VII	PANI	—	EP^{b}

At least twenty-four hours before to the initial measurement, freshly manufactured EP and solvent polymeric membrane sensors were immersed in 0.1 M/L NaHCO3 for a period of duration. In order to investigate the potentiometric responses of sensors, a range of 10^{-7}–10^{-1} M/L solutions containing various salts were utilized for the study. Both pure water and 0.1 M Tris-H2SO4 buffer with a pH of 8.6 were used as the medium for the addition of the known volumes of standard salt solutions, which were controlled simultaneously. Each membrane formulation was tested on three replica electrodes for the purpose of this study. The sensor potentials were measured in comparison to a doublejunction SCE reference electrode (AMEL, Italy), and the high-impedance 8-channel potentiometer LiquiLab (Ecosens, Italy) was used to record the data. Through the use of the separate solution method (SSM), selectivity coefficients were computed by utilizing EMF values that were measured in salt solutions with a concentration of 0.01 M and theoretical slope values.

Observations and Measurements. The Cary-50 Scan spectrophotometer was utilized in order to collect data for the UV-visible spectroscopic analysis. In this experiment, we utilized quartz and methacrylate cells that measured 45 × 10 × 10 mm and had a route length of 10 mm. For the potentiometric experiments, 15 microliters of the identical membrane concoctions were deposited on glass slides measuring 20 millimeters in length, 6 millimeters in width, and 1 millimeter in height. Following the evaporation of the THF, a thin polymer film was found to be connected to the glass slide. The absorption spectra of dry polymer films as well as those exposed for a duration of ten minutes in aqueous solutions of a number of different salts with varying concentrations were recorded. In order to assess the absorption spectra of Co(II)- and Cu(II)TATPP electropolymers, they were electrochemically deposited on transparent glass slides that were treated with indium tin oxide (ITO, Aldrich) measuring 15 × 7 × 1 mm. The nominal resistance of these glass slides was between 30 and 60 Ω/cm2.

Measurements of the Plasma! In this study, arterial blood samples were collected from five male individuals, three of whom were healthy and two of whom had respiratory acidosis (samples A and C). Blood plasma was extracted by centrifuging fresh samples and then removing suspended blood cells. This process was repeated until the plasma was obtained. A couple of hours after the samples were collected, they were analyzed. At the very least, tree replicas were carried out for every single plasma sample on the same day. In the event that

samples were not promptly tested, they were stored at a temperature of -20 degrees Celsius. Detection of the bicarbonate ion content in samples was accomplished by the use of the standard addition method. For this particular 50 μL of plasma, the sample was dissolved in 50 mL of 1 mM/L sodium chloride (E1), and then two sequential injections of 0.01 M sodium hydroxide (E2, E3) were carried out after the sample was dissolved. After that, the concentrations of CO_3^{2-} and HCO_3^{-} ions were assessed based on the ratio of $R = \Delta E3/\Delta E2$, the pH of the solution, and the dissociation constants of carbonic acid ($pK1 = 6.4$, $pK2 = 10.3$). A GEM Premier 3000 blood analyzer from Instrumentation Laboratory in the United States was used to determine the quantity of bicarbonate present in plasma samples for the purpose of comparison.

The sensor array was made up of five carbonate-selective electrodes in addition to a pH glass electrode. Before being used for measurements in actual plasma samples, the array was calibrated in 25 model solutions that were designed to simulate the composition of human plasma. There were four different salts present in each solution; the concentration of the salts was comparable to that of plasma and varied within the following range: 70–100 mM/L of sodium chloride, 20–60 mM/L of sodium hydroxide, 1–8 mM/L of sodium phosphate, and 1 mM/L of sodium salinity; the pH of the solution was adjusted to a range of 7.2–7.4 by adding 0.1 M/L of hydrochloric acid.

Analyzing the data. A method known as partial least square regression (PLS) was utilized in order to train a multisensory array in artificial solutions that were designed to simulate human blood plasma samples. Additionally, a correlation was established between the multisensory array response and the bicarbonate content estimated by a commercial blood analyzer. In order to process the data, the autoscaling approach was utilized. Due to the fact that the number of measurements that comprised the dataset was insufficient to divide the dataset into a training set and a test set, a validation method known as leave-one-out was utilized. With regard to the treatment of data, the Unscrambler version 9.1 (2004, CAMO PROSESS AS, Norway) was utilized.

Results and Discussion

Compounds of Co(II)- and Cu(II)TATPP that have been electropolymerized. Research has been done extensively on porphyrin electropolymers that are based on polyaniline (PANI). There have been prior reports from a number of writers that describe the electropolymerization of mono-, bis-, trisand tetra-2- or 4-aminophenyl substituted porphyrinates of different metals on platinum or GC WE. According to the findings of Bettelheim and colleagues, the electropolymerization of aminophenyl-substituted porphyrins takes place oxidatively through the meso-aniline rings in a head-to-tail form, just like aniline itself. The substance that is produced is a polyaniline chain that contains bridged porphyrin units in its practical application. It has been reported that anion-selective electrodes, which are based on such films, possess selectivity that is distinct from the Hofmeister selectivity series [25, 26]. Additionally, one of the intrinsic benefits of these electrodes is that they are stable and have a longer lifetime than other electrodes since the ionophore, which is contained within the polymer film, is retained.

In the current examination, we have concentrated on the development and investigation of the potentiometric behavior of sensors based on Co(II)- and Cu(II)-tris-4-aminophenyl porphyrinates. This is due to the fact that these porphyrinates are known to be sensitive to hydrophilic anions. To begin, an optimization of electropolymerization conditions for the deposition of poly-Co(II)TATPP films from four different solvents has been carried out (for more information, refer to Section 2 of this document). It has been shown that

acetonitrile does not result in the creation of any film on the surface of the Pt WE. On the other hand, insulating polyCo(II)TATPP films of yellow hue have been created from DMF and pyridine. On the other hand, a conductive film growth has been observed from the DMF/aniline solution (membrane I.d), as shown in Figure 1. A dominating PANI film generation process was observed in the latter instance, and it was accompanied by a partial Co(II)TATPP embedding in the PANI film during the first five cycles of the process. Figure 2 displays the cyclic voltammograms that were obtained after the first, fifth, and tenth potential scans that were performed during the electrodeposition of membrane I.d. in the voltage range of -0.2 to one and a half volts. It is possible that the creation of the PANI emeraldine form is responsible for the oxidative wave that occurs at around 0.2 V. As the scan number increases, this wave changes to a more positive potential and covers the reversible peak that occurs at approximately 0.4 V, which corresponds to the reversible Co(II)/Co(III) one-electron redox process. Particularly characteristic of the oxidation of arylsubstituted porphyrins and aniline are the sharp anodic peaks that can be seen during the first five scans. These peaks can be found at 0.65, 0.85, and 1.35 V. The presence of a high capacitive background anodic current, which is seen within the range of 0.3 to 1.1 V, is most likely a result of the incorporation of either free SO_4^{2-} ions or negatively charged Co(II)TATPP/SO_4^{2-} complexes into the PANI film.

By observing the existence of Soret's band ($\lambda = 454$ nm), which is a characteristic signature of the porphyrin aromatic ring, on the ultraviolet-visible absorption spectra of the membrane I.d., Figure 3, it was confirmed that the incorporation of Co-TATPP in the PANI backbone that was generated on the ITO glass electrodes was confirmed. On the other hand, the bathochromic shift of the peak maximum in the polymeric film in comparison to the fresh monomer solution in CH_2Cl_2 may be attributed to the axial coordination of porphyrin aminophenyl fragments (which in part remain nonoxidized during the electropolymerization) on the central Co ions of the neighboring porphyrin units. This is indicated by the broadening of Soret's band, which indicates the formation of a multilayer.

In this study, the potentiometric responses of poly-Co(II)TATPP electropolymerized membranes I.b–I.d that were deposited from DMF, pyridine, and DMF/aniline towards a number of anions were investigated. On the other hand, membranes I.b and I.c did not exhibit any significant reaction to any of the anions that were examined, most likely because of the abundance of insulating EP formation. When it comes to membrane I.d., selectivity patterns that were significantly different from the Hofmeister series were found. On the other hand, membrane II, which was based on Cu(II)TATPP-doped PANI film, was shown in Figure 4. For both membranes, the reaction towards CO_3^{2-} ions was found to be the strongest, with a slope that was near to the theoretical Nernstian value (27 and 28 mV/dec, respectively). In addition, it was observed that the I^- and SCN^- ions had a significant interference influence, with slopes of 56 and 59 mV/dec, respectively. Membrane VII (PANI) did not exhibit any particular reaction to any of the anions that were investigated, and the pH of the solution had a significant impact on its behavior. In point of fact, the pH sensitivity of PANI films is widely known, and a number of sensors for pH measurement that are based on polyaniline have been described in the past.
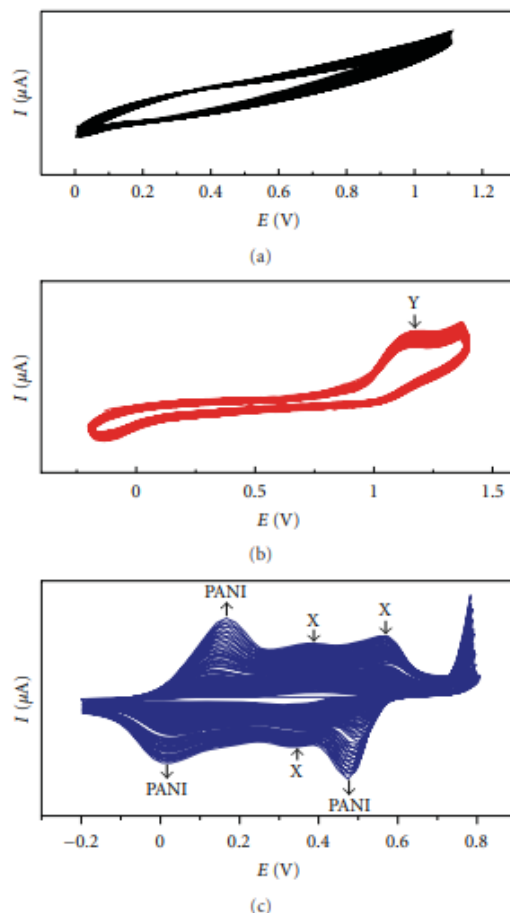
Figure 1: Cyclic voltammograms of PANI-Co(II)TATPP film electrodeposition on Pt WE from (a) acetonitrile; (b) DMF; (c) DMF: 0.5/L M aniline in 1 M/L H2SO4 = 1 : 1. The working and Platinum was used for the counterelectrodes, and the scan rate was 0.1 volts per second. The incorporation of Co(II)TATPP into the film is represented by the letter X in figure X, while the letter Y represents the reduction in current and the development of an insulating film.

Either the PH influence on Co(II)TATPP- and Cu(II)TATPP-doped PANI films or the selective complexation of bicarbonate/carbonate ions by metalloporphyrins could be responsible for the observation of a high sensitivity of membranes I.d and II to changes in the concentration of NaHCO3. As a matter of fact, the increase in the concentration of NaHCO3 causes the solution pH to rise. As a result, the accurate measurement of the different types of carbon dioxide (CO2) that are present in the sample under investigation necessitates either the simultaneous control of the pH or the utilization of an adequate buffer background.
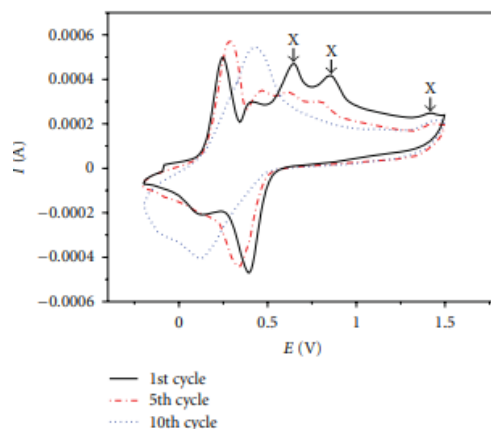
Figure 2: The details of PANI-Co(II)TATPP membrane I.delectropolymerisation from DMF/aniline solution. Peaks indicated as X show an incorporation of Co(II)TATPP in PANI film during first 5 cycles.
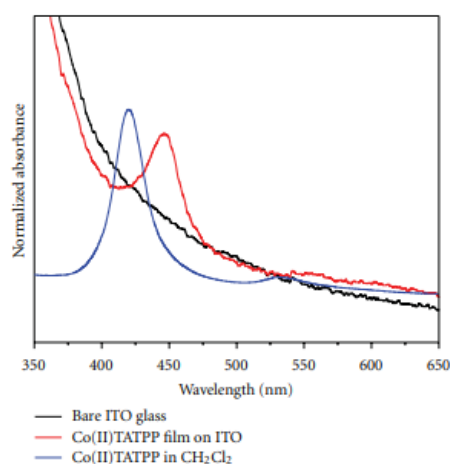
Figure 3: UV-visible absorption spectra of PANI-Co(II)TATPP electropolymer (membrane I.d) deposited on ITO glass slide. The spectra of Co(II)TATPP in CH2Cl2 and bare ITO glass are given for comparison.

Conclusions

The purpose of this review is to provide a summary of the most recent developments in electrochemical sensors. These sensors are designed to differentiate between minute biomolecules such as DNA, enzymes, and hormones, as well as to monitor a wide range of inorganic and organic contaminants that are updated electrochemically. Ongoing efforts are being made to develop new sensors that are constructed from a wide variety of chemical or biological sensing components. Furthermore, the development of exceedingly compact, reproducible, and economical (disposable) sensor devices is made possible by mass manufacturing technology, which is excellent for the microelectronics industry. Instrumentation that is based on microprocessors and is lightweight and user-friendly is integrated with these devices. There is little question that more advancements in selective and stable identification components, such as "smart" sensors and remote electrodes, molecular devices, multiparameter sensor arrays or micromachining, and nanotechnology, will absolutely have a substantial impact on the management of pollution. A further advancement in the field of biological research is the development of electrochemical sensors. Additionally, there are benefits in the future

industry in terms of sensitivity, selectivity, and processing speed. These advantages provide advantages. When it comes to studying a wide variety of targeted targets, electrochemical technologies are tools that are not only quick and accurate but also nondestructive. Increasing sensitivity has been accomplished through the utilization of functional peptides, aptamers, and nanomaterials (such as carbon nanotubes, graphene, graphene derivatives, metal nanoparticles, and gold nanoparticles, among others). During the electrochemical measurement, a measurable read signal is produced as a result of the interaction between the target and a specific probe or composite.

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