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Measurement Practice / Electro-Analytical

Overview Electro-Chemical Measurements

The benefits of Electro-Analytical Methods are:

- Very cost-effective
- Simple and easy to apply
- Suitable for continuous monitoring
- Suitable for laboratory use and for on-site use
- Sensors and instruments can be miniaturized

Electro-analytical methods usually use Sensors, which are separate from the Instrument, but are connected to the instrument by a cable. In some cases the sensor is integrated into the instrument.

The sensor is the part of the measuring equipment, which is in direct contact with medium (e.g. river water) or sample (e.g. toothpaste). Usually the sensor is immersed in the liquid to be analyzed, but in some cases it is only brought in close contact with the sample (e.g. the measurement of pH of meat).

Depending on the application the sensors come in various shapes and sizes (e.g. microelectrodes). Sometimes sensors are combined (e.g. pH electrode with integrated temperature sensor).

The Instrument contains the necessary electronic circuits to power the sensor and to convert the sensor signal into one of the standardized electrical signals for display, data recording or signal transmission.

Instruments come in various forms:

- Desktop Meters are used in laboratories. They display the measuring value. Sometimes they have built-in intelligence or can be connected to a computer for storing and processing the measuring data.
- Portable Meters are designed for on-site use. They display the measuring value. Sometimes they store the measuring data, which later can be transferred to a computer for processing and storage.
- Rugged Portable Meters for harsh environmental conditions (water-resistant, impact proof, etc).
- Sensor-Computer Interfaces which convert the sensor signal into digital data and communicate with the computer via the serial port (RS 232), printer port (LPT) or Universal Serial Bus (USB).
- Industrial Signal Transducers or Signal Converters which convert the sensor signal into a standardized industrial signal which can be transferred over a long distance (4-20 mA, RS485).
- Laboratory Signal Conditioners which convert the sensor signal into a signal suitable for data acquisition systems (0 to 10 V, -5V to +5 V, etc.).
- Educational Instruments with large displays, designed to demonstrate the results of chemical and biological experiments.

There are 3 types of measurements:

- Sample measurements on site
- Sample measurements in the laboratory
- Continuous Monitoring of parameters in Environment, Chemistry, Medicine, Biology, Science, Technology and Every-day life.

Sample Measurements are more and more replaced by:

- Continuous Monitoring and Recording
- Data Acquisition and Data Processing
- Computerized Measurements and Automated supervision.

The value of the electric signal of an electro-analytical instrument (Millivolts or Milliamperes) must be related to the property of the **Sample**, which is to be measured.

This is achieved by a procedure called **Calibration**. By measuring the response of the instrument to a so-called **Standard** with a known property, the relation between the property and the signal is established.

If it is known that the relation between the property and the signal is linear, a two-point calibration is sufficient. For control purposes more than two calibration point is recommended.

If the relation between the property and the signal is non-linear, the calibration procedure must be repeated for multiple values. The number of calibration points necessary, depends on the grade of non-linearity and the required accuracy of the measurement.

The graphic display of the result of a calibration procedure is called **Calibration Curve** or **Calibration Diagram**. In case of a linear function it is a straight line. A straight line can be defined by two points (start point and end point), or by one point (start point) and the **Slope** (gradient, gain). If the slope is known and stable, a one-point calibration or re-calibration can be sufficient.

The property of the sample is determined by comparing the signal (response) from the sample measurement with the calibration curve. In former times this was done manually by numeric or graphic interpolation, today electronic circuits or computers fulfil this task and the value of the property to be measured is directly displayed.

Electro-analytical measurements are influenced by various factors. There is always the influence of temperature, which makes it necessary to measure the temperature when an electro-chemical measurement is performed. The influence of temperature can be calculated and the result of the electro-analytical measurement adjusted. Some instruments have an **Automatic Temperature Compensation (ATC)** built in.

Depending on the type of measurements there can be other influences like the barometric air pressure and the salinity (salt content of the water) on the measurement of dissolved oxygen in water.

When measurements are performed the question of **Accuracy** and **Precision** comes up. Accuracy is the degree of agreement between the measured value and the true value. Precision is the degree of agreement between replicate measurements of the same quantity.

Another aspect of electro-analytical measurements is the stability of the signal (reading, display). Fast changes of the signal are called **Noise**, slow changes of the signal are called **Drift**.

Noise is caused by electromagnetic interference on sensors and cables. Noise can be reduced by improving the electromagnetic shielding, good ground connections and avoiding of so-called ground loops. The effect of noise can be reduced by smoothing the signal by electronic measures (noise filter) or by averaging multiple measurements.

Drift can be caused by chemical instabilities in the sample, and electro-chemical instabilities within the sensor. Measures against drift include stirring of the sample, waiting till chemical equilibrium is achieved and avoiding of temperature changes.

In the following chapters, the most important electro-analytical methods are discussed in more detail:

- **pH measurement** is the determination of the acidity or alkalinity of a sample. The pH value is of great importance for all biological, chemical and technical processes.
- **Conductivity measurement** is the determination of the ability to carry electric current. Conductivity is directly related to the salt content of a solution (Salinity).
- **Dissolved Oxygen measurement** is the determination of the amount of oxygen gas, which is dissolved in water and seawater. It is of the greatest importance for life and other biological processes in natural water and seawater.
- **Redox potential (ORP) measurement** determines the ability to oxidise or reduce chemical compounds. It is a decisive factor in chemical processes.
- **Ion-selective measurements** determine the concentration of specific ions in water, food, pharmaceutical products, etc. These measurements have a great importance for environmental, health and quality control.
- **Temperature measurements** are part of every analytical measurement, since the result of an electro-analytical measurement can only be interpreted correctly when the temperature during the measurement is known.
- Water quality measurements are a combination of the measurements above with some additional measurements.