# Techniques for the Analysis of Trace Tin Concentrations

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Tin determination allows for the monitoring of pollution and assessment of the impact of human activities on the environment. The determination of tin in the environment is crucial for the protection of human health and ecosystems, and for maintaining sustainability. Tin can be released into the environment from various sources, such as industry, transportation, and electronic waste. The concentration of tin in the environment can be determined by different analytical methods, depending on the form of tin present and the purpose of the analysis. The choice of an appropriate method depends on the type of sample, concentration levels, and the available instrumentation.

tin stripping voltammetry polarography

## 1. Introduction

Tin is a shiny silvery metal in group 14 of the periodic table. It is well known for its historical importance, versatility, and many applications. Its addition into copper alloys to make bronze marked a major technological achievement in human history. In ancient times, tin was also used to make mirrors and decorative objects. Its ductility, low melting point, and corrosion resistance make it a valuable material for a wide range of industrial and technological applications. Tin compounds are readily used as catalysts and stabilizers in the plastics industry. Nowadays, tin is used to coat other metals because it forms a thin anti-corrosion layer that protects objects from rust. This type of protection is mainly used in the food industry for cans or steel trays. It is also used in the electronics industry for touch screens and solar cells <sup>[1][2][3][4][5]</sup>.

Tin occurs naturally in small amounts in soils and sediments. The concentration of tin in soils can vary depending on the geological characteristics of the region. Soil concentrations typically range between 1–4 ppm, sometimes falling below 0.1 ppm, while peat can be as high as 300 ppm. Although tin occurs naturally in the environment, human activities can significantly affect its distribution and concentration, potentially leading to environmental problems and the need for appropriate management and remediation in areas where tin mining and processing take place. The toxicity of most tin compounds is relatively low and they are not considered to be very hazardous. However, as with many chemicals, exposure to high concentrations or chronic exposure to certain tin compounds can cause adverse health effects. Organic tin compounds are more toxic than inorganic ones. This is due to their poor absorption and relative insolubility in inorganic tin compounds. Organic tin compounds, on the other hand, are better absorbed and easily penetrate between human organs. The exposure limit value for tin and its inorganic compounds has been set at 2 mg m<sup>-3</sup>. The WHO has set the maximum tolerable level of tin in food at 250  $\mu$ g g<sup>-1</sup>. The levels of tin in environmental and biological samples cover a wide range, from  $\mu$ g g<sup>-1</sup> in canned foods to ng g<sup>-1</sup> and even pg g<sup>-1</sup> in water. Consumption of food containing inorganic tin at a concentration of 200 mg kg<sup>-1</sup> may lead to side effects. Too much tin in the body can affect the functioning of the nervous system, cause thymus problems, and weaken the immune system. A deficiency, on the other hand, manifests itself in stunted hair and nail growth, vision problems, and delayed reaction time to stimuli. It is, therefore, important to balance the concentration of tin in the human body [1][2][4][5][6][7][8].

The interest in the determination of tin is related to its wide application in various fields. Many methods for the determination of tin have been described in the literature. Frequently used methods for the determination of tin include atomic absorption spectroscopy <sup>[9]</sup>, spectrophotometry <sup>[10][11][12]</sup>, atomic emission spectroscopy <sup>[13]</sup>, inductively coupled plasma-mass spectrometry (ICP–MS) <sup>[14]</sup>, inductively coupled plasma-optical emission spectroscopy (ICP–OES) <sup>[15]</sup>, X-Ray fluorescence spectrometry <sup>[16]</sup>, liquid–solid chromatography <sup>[17]</sup>, and chemiluminescence <sup>[18]</sup>. Although many analytical techniques, such as atomic spectroscopy with high trace element detection capabilities, have been used for many years, their high cost and, in some cases, low sensitivity and selectivity mean that there is a need for more sensitive and cheaper methods. Of these, ICP–MS and ICP–OES only allow the determination of organic and total tin concentrations. These methods are expensive and do not allow the selective determination of Sn(II) and Sn(IV) ions <sup>[9][10][11][12][13][14][15][16][17][18]</sup>.

Electrochemical methods can be an interesting alternative to other methods for the determination of traces of tin. Electrochemical methods rely on the measurement of electrical quantities such as potential, current, or charge. The main advantages of electrochemical methods over other detection systems are that high sensitivities, wide ranges of linearity, and low instrument costs are achieved <sup>[19]</sup>. Electroanalytical methods for the detection of tin include stripping voltammetry [20][21][22][23][24][25][26][27][28][29][30][31][32][33][34][35][36][37][38][39][40][41][42][43][44][45][46], polarography [47][48][49][50][51][52][53][54][55][56][57], potentiometry [58][59][60], and coulometry [61]. In the literature, most of the works on the electrochemical determination of tin are devoted to voltammetric methods. In stripping voltammetry methods, measurement is carried out in two stages. In the first stage, called concentration, a labeled component accumulates on the electrode; in the second stage, called stripping, the accumulated component undergoes an electrode reaction as a result of the change in the potential of the working electrode. In the stripping stage, the voltammetric curve is recorded as a peak. There are three known methods of stripping voltammetry, which differ in the way the analyte is accumulated on the working electrode. If the accumulation is by adsorption, the method is called adsorptive stripping voltammetry (AdSV). In this method, during the stripping stage, the determined substance is oxidized or reduced depending on the potential applied to the working electrode; in a few cases, the signal is obtained based on a desorption process. In anodic stripping voltammetry (ASV), the first step involves an electrolytic process carried out by electrochemical reduction of the labeled substance at a constant potential, which, depending on the nature of the separated metal and the electrode material, dissolves or forms a film on the electrode surface. After electrolytic concentration, the electrode is polarized in the anodic direction, and at an appropriate potential, the labelled metal is oxidized. Anodic stripping voltammetry is the oldest stripping method in use. In contrast, cathodic stripping voltammetry (CSV) involves an electrode reaction in the first step, resulting in the formation of a poorly soluble compound on the surface of the working electrode. In the stripping step in this method, decreasing potential is applied to the working electrode. At this potential, the labelled metal is reduced [22] <sup>[63][64]</sup>. As you can see, the most frequently used method in the voltammetric analysis of tin is the ASV method, which uses different working electrodes and supporting electrolytes <sup>[20][21][22][23][24][25][26][27][28]</sup>. The lowest limit of detection for tin was obtained using the AdSV method. Due to the accumulation of tin with the complexing agent on the working electrode, higher sensitivity is achieved compared to the ASV method, allowing the detection of lower analyte concentrations. Only one paper on the determination of tin by cathodic stripping voltammetry has been published in the literature <sup>[33]</sup>.

Polarography is an older technique than voltammetry. Polarography was introduced by Jaroslav Heyrovský in the 1920s. This electrochemical technique focuses on the study of redox reactions and is widely used in analytical chemistry. Polarographic methods have also been used for the determination of tin. The main difference between classical electrolysis and the polarographic method is that in the former, the total mass of the substances separated at the electrodes is determined, whereas in polarography, changes in the current flowing through the solution under test during electrolysis are studied. Polarographic methods should use working electrodes that are liquid electrodes with a continuously or periodically renewed surface, whereas voltammetric methods use working electrodes where the electrode area is constant <sup>[63][65][66]</sup>.

Tin(II) analysis is also carried out using potentiometric membrane sensors, known as ion-selective electrodes (ISEs) <sup>[58][59][60]</sup>. The main advantage of the ISE is that it provides precise analytical information. In this method, the electrode reaction takes place without an applied external voltage. The basis of potentiometry is the measurement of the electromotive force. The measurement involves examining the difference in electrochemical potential between the electrodes immersed in the diluent to be analyzed. ISE electrodes are provided with an ion-selective membrane, which separates the corresponding half–cell from the test solution. Such a membrane is sensitive to the activity of a particular type of ion. The main advantage of the ISE is that it provides a rapid yet accurate determination of the analyte <sup>[67][68][69]</sup>. Only a few ion-selective electrodes have been proposed for the determination of tin(II).

### 2. Types of Techniques Used

#### 2.1. Polarography

The most frequently used polarographic method is differential pulse polarography (DPV). In DPV methods, the current is sampled at two different points in the waveform, resulting in a differential output signal. This makes the developed methods highly sensitive. Weber proposed a DPV method for the determination of tin using the DME as the working electrode. The optimized procedure allowed for the determination of tin at the lowest concentration of  $8.4 \times 10^{-10}$  mol L<sup>-1</sup>, the lowest detection limit reported in the literature, using the polarography technique. Another advantage of the developed method is the achievement of a wide range of linearity in the determination of tin, from  $8.4 \times 10^{-9}$  to  $4.2 \times 10^{-5}$  mol L<sup>-1</sup> [50]. Of course, there are also works in which the detection limit obtained is less satisfactory, being 5 orders of magnitude higher. This results in a significant reduction in the sensitivity of tin determination.

#### 2.2. Voltammetry

Among the electrochemical methods for the determination of tin, stripping voltammetry offers many advantages, such as high sensitivity, accuracy, simplicity of the proposed analytical procedures, low cost of instrumentation and reagents, but also a wide range of applications of the developed procedures <sup>[19]</sup>.

The use of a buildup and stripping step in the procedures significantly increased the sensitivity of the determination of tin, making it possible to determine very low concentrations. The most commonly used electrode in voltammetric methods for the determination of tin has been the hanging mercury electrode, due to its high sensitivity, repeatability, and accuracy. However, due to the toxicity of mercury, other electrodes were introduced to eliminate or reduce the use of this metal in electrode manufacture. A bismuth film electrode was introduced for the determination of tin by anodic stripping voltammetry. Unfortunately, the sensitivity of the method decreased, and the detection limits obtained were not as low as when using a mercury-based electrode. Another electrode that replaced the mercury-based electrode was the carbon paste electrode. By using this electrode and a complexing agent, which was BPR, a sensitive and selective method for the determination of traces of tin was achieved <sup>[24]</sup>. In this case, the detection limit was of the same order of magnitude as the detection limit obtained with a hanging drop mercury electrode. The detection limit can also be lowered by increasing the accumulation time of the element being determined on the surface of the working electrode. Very low concentrations of tin required long accumulation times to build up sufficient material on the electrode for accurate measurement. In the paper [35], the accumulation time was 600 s and thus, the lowest detection limit was obtained. The authors in [39] calculated detection limits at two different accumulation times. Increasing the accumulation time of the tin-oxine complex on the HMDE electrode by up to 240 s resulted in a detection limit that was one order of magnitude lower than that obtained, using a 60 s accumulation time <sup>[39]</sup>. In conclusion, the introduction of an accumulation and stripping step results in a significant reduction in the detection limit and a high increase in sensitivity, accuracy, and selectivity of the developed tin determination methods.

#### 2.3. Potentiometry

A unique feature of potentiometry is that it measures the concentration of free ions and not the total ion concentration. Therefore, the potentiometric methods described determine the concentration of Sn(II) ions <sup>[58][59]</sup> <sup>[60]</sup>. Three ion-selective electrodes have been proposed for the determination of stannous. The lowest detection limit obtained in the determination of tin with the ion-selective electrode was  $8.0 \times 10^{-7}$  mol L<sup>-1</sup> <sup>[58]</sup>. Potentiometric techniques used for the determination of Sn(II) are characterized by a wide range of linearity. The range of linearity obtained in the work <sup>[58]</sup> was in the range from  $5.0 \times 10^{-7}$  mol L<sup>-1</sup> to  $1.0 \times 10^{-1}$  mol L<sup>-1</sup>. This was the widest range of linearity among the methods discussed in that article <sup>[58]</sup>. For ion-selective electrodes, dynamic response time is an important factor. The ion-selective electrodes developed for the determination of stannous are characterized by a short response time, with the electrodes reaching equilibrium in the range of 15 to 20 s. The lifetimes of the electrodes, during which the results obtained for the determination of tin were reproducible, were also investigated.

The longest lifetime was up to 90 days, and it was obtained for an electrode in which the ionophore was DB18C6

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