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# INPUT CONTROL OF SENSOR PARAMETERS AND REPRODUCIBILITY OF SENSOR MEASUREMENT

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Abstract - Fast detection of pesticide toxicity in the field conditions is very important in many aspects (longtime influence of low concentrations on man's health, pesticide storage, price of one test etc.). The pesticide concentration measurements are performed on a biosensor with acetylcholinesterase (AChE). The AChE is a very sensitive compound. The measurement uses principle of an artificial inhibition synapse (AS)and of an enzyme Acetylcholinesterase (AChE). The AS is a very effective detector of the pesticide toxicity. Therefore properties of the sensor have to be measured without AChE at first. This process is called input control of the sensor. The reproducibility of electrochemical detector is studied first using hydrogen peroxide and second ferri-ferrokyanide redox couple. The parameters of electrochemical detector itself were evaluated statistically.

Keywords: hydrogen peroxide, acetylcholinesterase

# 1. INTRODUCTION

# 1.1. Input control of sensor

Input control is used to measure properties of sensor without AChE. There are two methods for determination of biosensor properties. One is using ferroferricyanide [1] and the next is using hydroxen peroxide  $(H_2O_2)$  detection.pointed out. The restrictions of the procedure, possibility and range of application of the results have to be stated.

# 1.2. Method for analysing properties of biosensor exploiting AChE

Next step is to create standard method for description properties of biosensor with AChE. This method uses principles, which were implemented in similar projects [2]. The compound ATCh is for analysis properties of biosensor exploiting AChE.

# 1.3. Output control of biosensor

This method is defined for measuring properties of bisoesensor with AChE after inhibition. The syntostigmin is used as inhibition compound.

#### 1.4. Evaluation method

Evaluation method is created for input control now. Evaluation is executed by using standard form. Form is created in EXCEL program [3]. Form is advised to standard operation principles, which they are one of the requirements for GOOD LABORATORY PRACTICE [4]. Evaluation method is done for evaluation results of output control and biosensor properties with AChE enzyme.

#### 2. EXPERIMENTAL RESULTS OF INPUT CONTROL

#### 2.1. Objective

Study the behaviour of hydrogen peroxide electrochemical detection from solutions of different  $H_2O_2$  concentration. Determination of method to detect  $H_2O_2$  by AC1.W2.RS sensor.

#### 2.2. Chemicals

- 1. 3 wt. % hydrogen peroxide,
- 2. Phosphate buffer (3,3 mM KH<sub>2</sub>PO<sub>4</sub>, 63 mM N<sub>2</sub>HPO<sub>4</sub>)

All chemicals were of high purity and used as received. Distilled water used in all experiments.

## 2.3. Sample preparation

As a testing solution were prepared 3 %, 0,3 %, 0,03 %and 0,003 % hydrogen peroxide solutions in phosphate buffer. Hydrogen peroxide solutions was prepared just before the experiment, due to hydrogen peroxide unstabilty.

#### 2.4. Electrochemical experiment

The sensor with platinum electrode was put into a Micro Flow System (MFS, BVT Technologies). There is a three electrodes cell. The electrodes were connected to a bioanalyzer (BVT Technologies). All measurements were performed at potential 650 mV. Working solution quantity (buffer) used in the actual experiment was 10 ml. Experiment was stabilized for 15 min before additions of hydrogen peroxide. 50  $\mu$ l H<sub>2</sub>O<sub>2</sub> (0,003 wt. %) was added three times after stabilization of the system. The interval between the additions is determined by stabilization of output current. Further additions were added stepwise from concentration of 0,03 wt. % to 3 wt. %, three times for each concentration.

#### 2.5. Equipment

Standard analyzer (Polarographic analyzer PA4, Laboratorní přístroje Praha) alongwith XY chart recorder was used in experiment. Micro Flow System (MFS, BVT Technologies). Bioanalyzer (BVT Technologies).

# 2.6. Mechanism of measurement

Electrochemical process can be only in few examples described by a simple chemical reaction.

Mechanism shown below is supposed to proceed on working electrode (anode):

The first step is discharging a molecule of water on the anode surface according to the reaction

$$Pt + H2O = Pt...OH + H^{+} + e^{-1}$$
(1)

The second step is hydrogen peroxide adsorption on the anode surface and combination with anteriorly developed Pt...OH to give adsorbed molecule of water and unstable  $Pt...O_2H$ 

$$Pt...H_2O_2 + Pt...OH = Pt...H_2O + Pt...O_2H$$
(2)



Developed molecule of water discharges according to (1) and the last step is

$$Pt...O_2H + Pt...OH = Pt...H_2O + O_2 + Pt$$
(3)



and the process repeats up to consumption of all hydrogen peroxide.

The whole reaction is

 $H_2O_2 = O_2 + 2H^+ + 2e^-$ (4) which means adsorption.

#### 2.7. Graphs and results

The real results of the experiment are depicted in Fig. 1 to 4.



Fig.1. Current vs time plot for two additions of 0,003 wt. %  $H_2O_2$  solution



Fig. 2. Current vs time plot for two additions of 0,03 wt.  $\%~H_2O_2$  solution



Fig. 3. Current vs time plot for two additions of 0,3 wt. % H<sub>2</sub>O<sub>2</sub> solution (current range changed in point 1 and 3)







Fig. 5. Calibration curve (H<sub>2</sub>O<sub>2</sub>)

## 3. CONCLUSION

Input control of the sensors is a method for determination properties of sensor without immobilized AChE enzyme. This method unsure reproducibility of measurement of organophosphorous pesticides toxicity. All measurements were performed on sensor AC1.



Fig. 4. AC1 sensor

This sensor is manufactured by BVT Technologies a.s. These sensors are produced with the use of the screen printing technology.

#### REFERENCES

- [1] Student Workbooks, SYCOPEL SCIENTIFIC LIMITED, Great Britain, 2000
- [2] Krejčí, J., Detector BioNA Functional Tests, Internal Research Report, 2001
- [3] Sajdl, O., Standard evaluation of measurement with peroxide hydroxide – form, Internal Research Report of ANTOPE Project, BUT Brno, 2002
- [4] Finger, P., Ecotoxic Biotests Workshop, Proceedings, p. 35, Seč u Chrudimi, 2002

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