A new system for electroanalytical mass screening

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Abstract

The work deals with n-channel system enabling many sample analysis at sort time from sensor array using electrochemical methods. The 8x12 sensor array is formed from 3-electrodes system created on PCB (Printed Circuit Board) with combination of galvanic deposition and screen-printing special paste on the electrodes which can be used for heavy metal analysis and also biomolecular complexes and organic toxic substances determination. The control unit switches each 3-electrode sensor to 8-channel precise potentiostat which was designed for electrochemical analysis with current sensitivity below 1 pA. The potentiostat can synchronize with control unit to depress time for analysis to minimum. Measured data are sent to user-friendly application in computer and analyzed or saved consequently.

Keywords: electrochemical methods, cyclic voltametry, sensor array

Introduction

Nowadays the ecology aspect of human activity is very important because of many toxic substances being produced by industry (Pollard et al., 2009; Yantasee et al., 2007). Rapid monitoring and mapping of toxic contamination of environment is required. The biggest objectives in today's world are to collect a lot of samples, simple and faster measurements by intelligent automatic devices, data archiving and studying saved records. Very promising

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*Tel: +420 545 133 350, Fax: +420 545 212 044 E-mail: kizek@sci.muni.cz application can be mass screening in cases of epidemic and pandemic using electrochemical methods (Adam et al., 2009). Time is significant and crucial issue in these cases.

The system design

The whole system consists of a sensor array, a sensor multiplexer, the main device – potentiostat and service program in computer (see Fig 1). Sensor array converts chemical values to electrical signals. There are 96×3 electrodes and their electrical interconnections on the sensor array. These connections are designed for parallel processing. Each electrode has its own connection pin in the connector on the one side of the sensor array, only working electrodes are connected together in each line of sensors. There is shielding layer near the electrodes (ring around and plate under the electrodes) for minimization of external interferences.



Figure 1: Block diagram of the whole system.

We need a multiplexer system to switch and to group signals to several multiple ones. The multiplexer system is able to select one of the sensors for serial processing or the whole sensor line (group of eight sensors) for parallel processing. The multiplexed signals are distributed to 8-channel potentiostat for real-time multichannel measuring.



Figure 2: Block schematic of the 8-channel potentiostat (main part of Fig 1).

Simplified schematic of the potentiostat is shown in the Fig 2. The potentiostat is controlled by service application in PC, because standalone measurement would be hard executable (too many measured data would be stored in internal RAM). The service application allows measuring by eight standard electrochemical methods (for example cyclic voltammetry, differential pulse voltammetry, etc.), one user defined method and other methods as EVLS (Elimination Voltammetry with Linear Scan) are being implemented to the application.

Results and Discussion



Figure 3: Sensor array (8 \times 12 3–electrode systems) on the left and multiplexer system on the right.

Electrodes were placed to the matrix of sensor array and this array was made on standard organic printed circuit board (PCB, 4-layers FR4) consequently (see Fig 3). Connector pins and sensor electrodes are golden plated (NiAu + galvanic gold). After making basic structure of the sensor array, the auxiliary electrode was deposited from Pt using galvanic method. Working electrode was deposited grow Pt using galvanic method. Working electrode was formed by printing using Ag/AgCl paste DuPont, USA). There will be isolating layer around the sensors used for making 3D structure to create holes for specimens. Many different shapes of sensors were made for testing response size and measured specimen stability and two of them were selected and used on sensor arrays (see Fig 4).



Figure 4: Two different shapes of electrodes used on sensor array (red - auxiliary, green – working and yellow – reference electrode)

Multiplexer system can select one column (eight sensors) or each sensor separately. The selection of auxiliary and reference electrodes is realized by analogue CMOS multiplexers (74HC4067 and 74HC4051). Working electrodes are selected by mechanical SMD relays with very low contact resistance. This solution connects maximal accuracy and minimization of the sensor multiplexer size. Sensor multiplexer provides three ways of controlling - manual sensor selection by user, time delayed sensor switching with user defined switching time and external controlling by the superior system, potentiostat for example. Internal multiplexers and relays are switched by AVR RISC microcontroller, actual state is signalized by simple LED display and multiplexer is powered by external stabilized power source. Synchronization link is designed as simple two wires with open collectors and pull-up resistors for maximum versatility (1st wire changes index actual selected sensor and 2^{nd} wire gets sensor to start position).



Figure 5: Input module of potentiostat - input analog part schematic

8-channel potentiostat is able to provide more than one measurement at the time which saves the measurement time. The device was build up with SW for controlling the measurement by PC. The potentiostat was designed for parallel high precise/low currents measurement (separated power sources, precise AD/DA converters and analogue circuits, battery powering when the measuring is running with automatic charging, etc.; see Fig 2 and Fig 5). Fig 2 shows modular concept of the potentiostat with one digital controlling block and up to eight input analog modules. To maximize the noise resistance the input modules were designed with separated and shielded blocks (see Fig 6) and their interconnection to the digital part of the potentiostat is realized only by digital bus. Each analog input modul can operate as independent universal measuring system because it contains all necessary parts (power source, programmable voltage output with DAC, voltage input with ADC and I/U converter with five ranges (100 pA, 10 nA, 1 µA, 100 µA and 10 mA).



Figure 6: Potentiostat - input analog part HW realisation

The potentiostat measures one row by one row at the time therefore it can synchronize the end of each row measurement with multiplexer to save the time. The instrument is presented in Fig 7. Advanced routines and circuits solutions for increasing resolution and decreasing noise are being implemented.



Figure 7: The 8-channel potentiostat

Different concentrations of PbCl₂ dissolved in redistilled and deionized water mixed with 4 ml 1 M KCl were used as test analyte. The analyte was applied as drops of 50 μ L on electrodes. The measurement method was standard cyclic voltammetry in the range of potentials from 0 V to -1 V with the scan rate of 25 mV/s. There were seven different concentrations measured at the same time. Testing results of Pb ions detection are shown in the Fig 8.



Figure 8: Testing measurement by cyclic voltammetry

Conclusion

This automatic measurement system for mass screening has been developed by accelerating of the precise and rapid electrochemical measurements and increasing their efficiency. Measured data are analyzed by the computer application, specially developed for this device. With the system the mass screening of several substances can be provided during a few minutes. If one measurement takes 2 minutes the 96 samples can be analyzed during 24 minutes.

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