SOLID-STATE ION-SELECTIVE MICROELECTRODES FOR FLOW ANALYTICAL SYSTEMS

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Sensor design

Back-side contact transducers

Two structures of back-side contact transducers - solid-state Au and Ag/AgCl miniaturized electrodes (squares 5 x 5 mm, thickness 0.5 mm) - developed using silicon technology (Institute of Electron Technology, Warsaw) or on epoxy-glass laminate support have been used.

Au transducers fabricated on silicon (left) and on epoxy-glass laminate support (right)

Microelectrodes preparation

Solid-state microelectrodes sensitive towards inorganic charged pollutants (i.e. NO_3^-, NH_4^+, K^+, Na^+) were obtained by the deposition of polymeric membranes on the surface of Au microelectrodes.
The ion-sensitive membranes contained 1-2% of ionophore (4% of ion-exchanger TDAB in the case of nitrate-sensitive membranes), 10-50 mol% of KTFPB, 31-33% of PVC, 64-66% of DOS. Membrane components (~100 mg in total) were dissolved in 0.5 ml of THF.

The resulting membrane solutions (7µl) were deposited on the surface of back-side contact Au transducers. Ag/AgCl microelectrodes, developed in silicon technology, have been used as chloride-sensitive sensors.

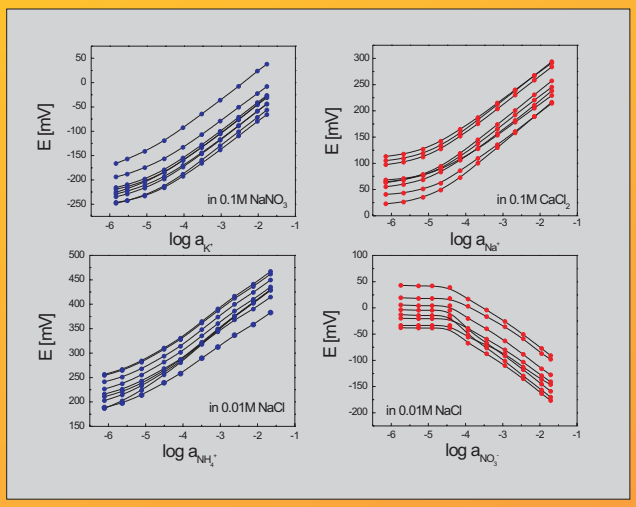
Laboratory set-up

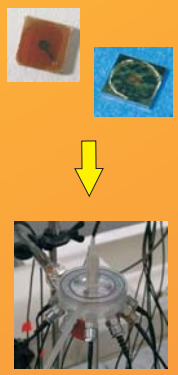
After the membrane solvent evaporation, the sensors were mounted in a flow-through cell and conditioned for 3 days.
The performances and the long-term stability of the ion-selective microelectrodes were determined in an automated set-up with data acquisition PC-based system (16-channel amplifier, LawsonLab). A customized double junction Ag/AgCl electrode was used as a reference electrode.

Laboratory tests

Response curves of the microelectrodes

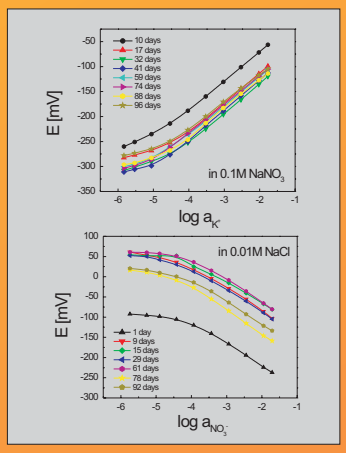
- the performances and the values of the selectivity coefficients towards primary ions against interferent ions of the microelectrodes were comparable with classical ion-selective electrodes based on PVC membranes,
- the microsensors exhibited good reproducibility in terms of: their values of the selectivity coefficients, linear response range (from -4 to -1.5 pX) and slopes (54-58 mV/pX) of calibration curves; a limited reproducibility of the standard potential was observed, which can be related to the simplified structure of the sensors.





Long-term stability of the microelectrodes

- good long-term stability of the microelectrodes i.e. only small variations of their values of selectivity coefficients and response slope was observed during 2-3 months of measurements,
- significant changes of the standard potential E° in time were recorded; however, the calibration of the electrodes in the monitoring probe before each measurement, can eliminate the influence of this effect on analytical results.



Industrialisation

- new modular flow-cells allowing easy mounting of the solid-state microsensors in the measurement loop-flow have been fabricated,
- special facility for the semi-industrial sensor fabrication, providing deposition of the polymeric membranes (with an automatic dispenser) and sensor testing has been developed in MEDBRYT,
- final solid-state microelectrodes for the determination of: NO_3^-, NH_4^+, K^+, Na^+ will be used in a portable analytical device (SYSTEA) dedicated to water quality monitoring.

automatic dispenser for the deposition of polymeric membranes

obtained solid-state microelectrodes

commercially available MEDBRYT's modular flow-cells for solid-state microelectrodes