AUTOMATIC STAND FOR IS-FET SENSORS PARAMETERS IDENTIFICATION

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ABSTRACT: Evaluation of chemical sensor parameters requires many measurements with variation of chemical sample composition, variation of electrical sensor conditions (i.e. polarisation, etc.) and with changes of sensors and bath temperature. The number of necessary measurement easily reaches thousands of tests. When manually controlled they are extremely costly and time consuming. The fully automated measurement stand for evaluation of matrix of IS-FET sensors is described with details in the paper.

KEYWORDS: chemical sensor, IS-FET, Chem-FET

INTRODUCTION

Reliable and efficient monitoring of natural environment parameters usually needs permanent field tests and measurement data evaluation. In particular this is very necessary for surface water quality monitoring (at rivers, lakes, etc). Nowadays technology gives possibility for both construction of smart chemical sensor matrix as well as building of distributed sensor measurement system for on line operation.
Such a system is realised under European Community Commission, FP5 in the project "System for European Water Monitoring - SEWING", IST - 2000 - 28084. According to general assumptions the system must be open for number of smart sensors, every smart sensor will contain the matrix of four IS - FET structures for 4 ion concentration measurement and will be facilitated with radio transmission unit based on mobile phone to transmit measurement data to local data acquisition system. Many careful investigations must be done to work out of the matrix of IS-FET sensors. To speed up researches on optimal IS-FET construction and on dedicated for different ions chemical membranes, the automatic stand for IS-FET sensors parameters identification has been worked out.
The automatic measurement stand has three main tasks to fulfil:

— automatic sample preparation for tests (with temperature control of bath)
— automatic IS-FET signal measurements
— measurement data processing and sensor characteristic visualisation

The automatic stand will be extremely useful for long term sensor parameter stability measurements and for final sensor matrix calibrations as well.

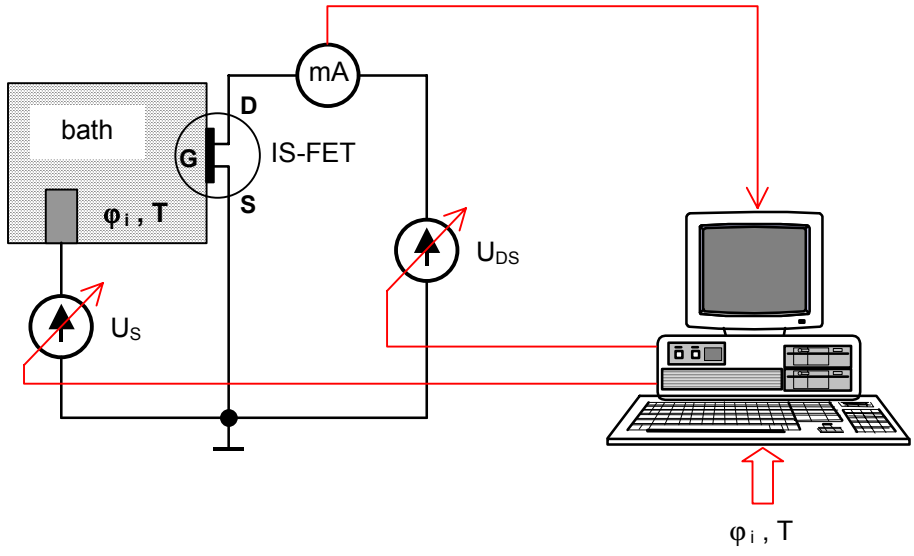

Fig. 1. The simplified measurement circuit for IS-FET sensor operation with computer controlled sensor polarisation (sensor electrical working point); φ_i – ion concentration, T – bath temperature.

IS-FET SENSORS UNDER TESTS

The Ion Selective - Field Effect Transistor must be optimised from three different point of view:

— to gain both highest sensitivity on ions concentration (on electrical charge) and highest stability - this is transistor construction optimisation
— to gain highest selectivity - this is chemical membrane optimisation
— to gain both highest sensitivity and measurement accuracy - optimisation of the electrical working point of the sensor transistor.

The last from listed above problems can be relatively easily resolved with the measurement circuit controlled by computer as it is presented on Fig.1. The reference electrode immersed in the sample is under potential Vs which can be varied from -5 to +5V with 10 mV potential step. The computer system controls also drain to source potential U_{DS} in the range of +2 ÷ +15V with Keithley 236 Source Measurement Unit which can also measure drain current I_D.

To speed up the sensors evaluation process and to save volume of liquid components for given ion concentration bath, the measurement head for simultaneously 10 sensor tests has been designed and constructed - Fig.2 [1]. The measurement head gives equal liquid bath flow conditions for every sensor under test (designed with radial symmetry) and is suited for silicon sensor structures with two back-side contacts on every sensors. This construction gives great advantage that sensors structures cut out from silicon wafer can be directly placed into measurement head (without sensor structures bonding and maintenance into a package).

ORGANISATION OF MEASUREMENT STAND

One of the main tasks of the measurement stand is automatic liquid sample (bath) preparation and continuous supply of it into the measurement head with up to 10 sensors structures. This part of the system is well seen on Fig.3. The system is equipped with 3 burettes - Metrohom 765 Dosimat, Reglo Digitall (Isematec) Peristatic Pump all with digital standard RS 232 interfaces, number of electrically driven valves, and especially designed fast operating thermostat with cooling/heating system. As an additional equipment the system is also endowed with electromagnetic stirrer (for obtaining uniform ions concentration in bath) and a few other containers for single ion baths, neutral liquid and wasted solutions. Organisation of computer controlled system is well shown on Fig. 4. The system is run under Pentium III PC embedded with IEEE 488 Interface Card and Serial 8 x RS 232 Card (both card provided by National Instruments). The sensors polarisation (voltages U_{DS} and U_s) and measurement of output signal (I_D) is performed by two 236 Source Measure Units (KETHLEY) type. Ten switching canals are performed by multiplexer of 34970A Data Acquisition/Switch Unit (Agilent) type. Some additional canals of that multiplexer are used to switch electromagnetic pinch valves for circulation control of bath liquid sample.

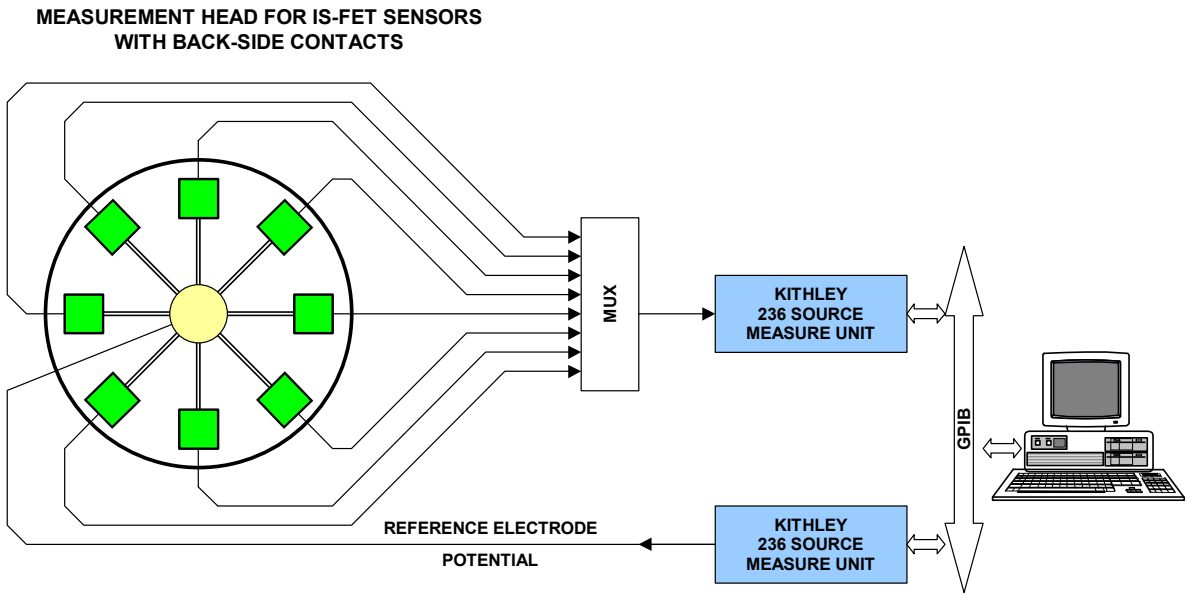

Fig. 2. The measurement head for 8 simultaneous IS-FET sensors evaluation with computer controlled measurement and data acquisition system.

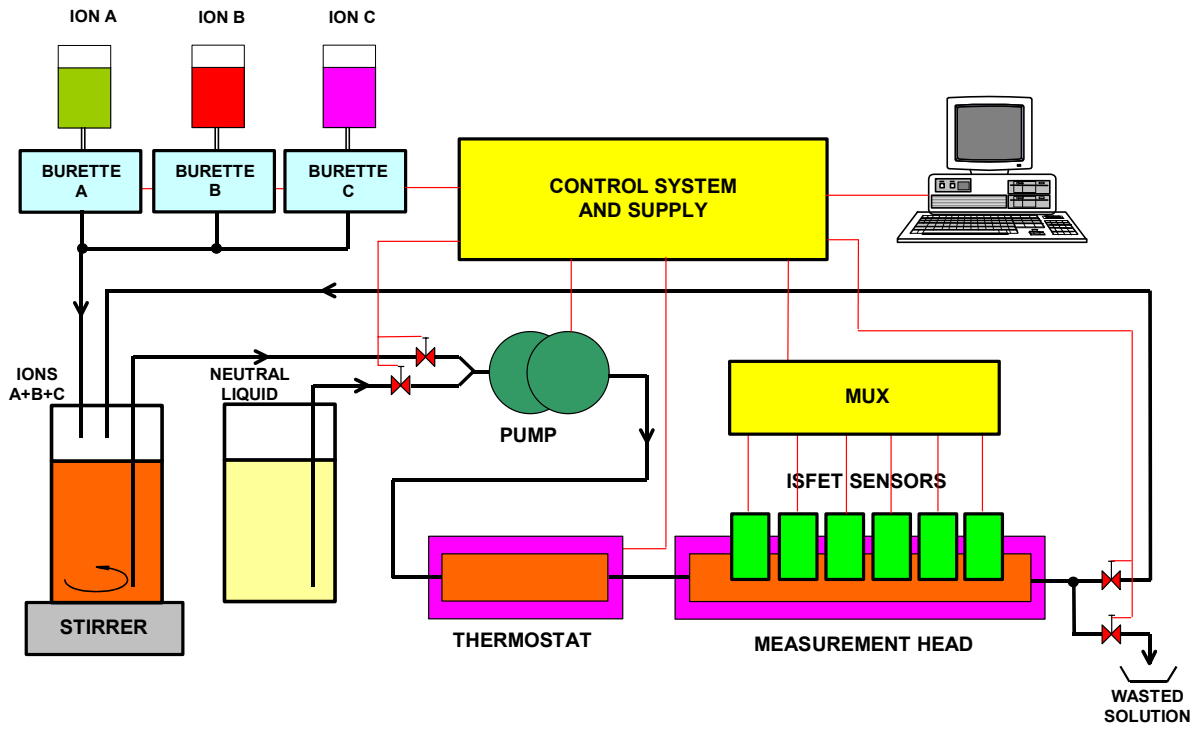

Fig. 3. General scheme of whole measurement and bath preparation automated stand.

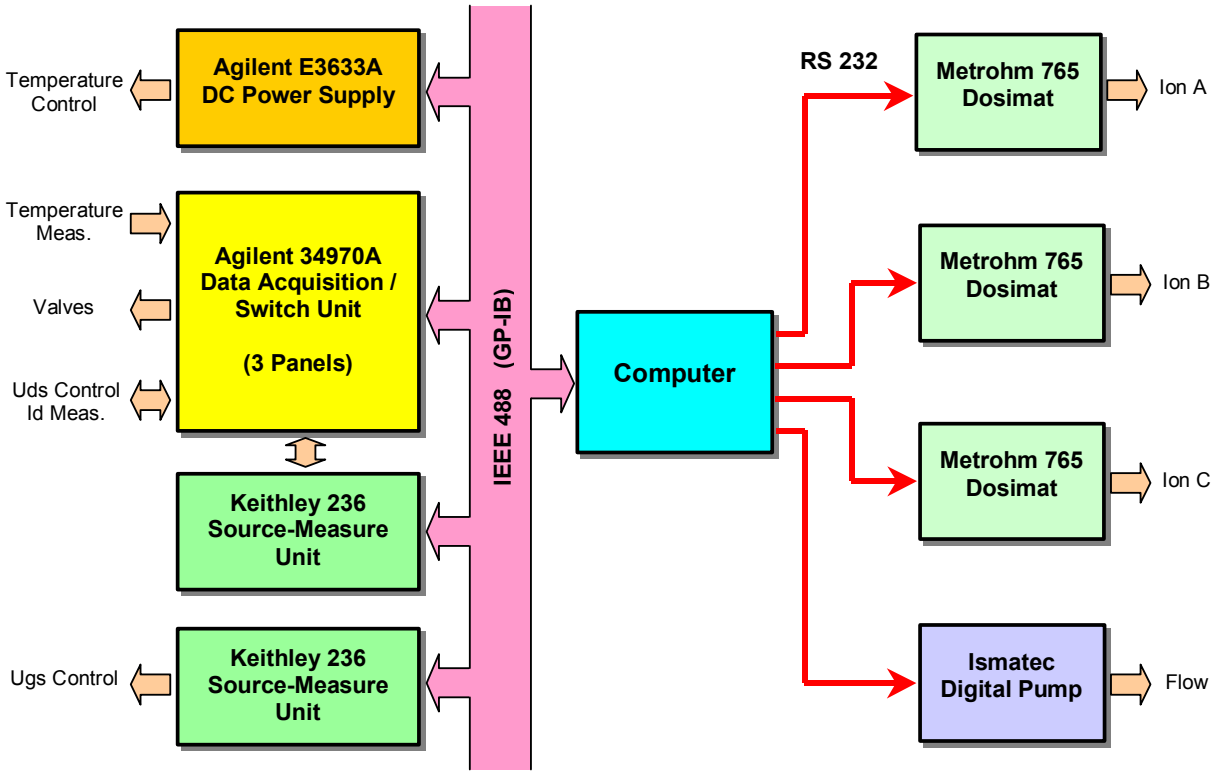

Fig. 4. The computer controlled system organisation for automatic sample preparation, temperature control and automatic data acquisition.

OPERATION ALGORITHMS AND COMPUTER PROGRAMS

The operational algorithms have been designed with special attention on great flexibility of operation of measurement stand. For that dedicated programs have been written to set up polarisation of IS-FET in given range of potential and with given potential variation step. The control and data acquisition program has been created in Lab Window C VI 5.5 software environment.

An other part of the computer program gives possibility to design whole measurement process, to design temperature change of ions samples and to determinate time interval between sensor immerse in particular ions concentration bath and measurement of output signal. Typically for each ion the system changes its concentration in the range of 5 decades with 2 concentration on decade which gives 11 measurement points for every ion, uniformly distributed on logarithmic scale. The temperature of both liquid samples and sensor structures can be automatically changed in the range +5ºC to +40ºC with step 1K.

Obtained measurement data, enriched with information about actual date, sensor type, number and measurement history and sensors identification number, are collected on hard disc in format allowing their searching, sorting, further analysis and sensor full characteristic visualisation.

TECHNICAL DATA OF THE MEASUREMENT AUTOMATIC STAND

The measurement stand can be described by following main parameters:

- up to 10 IS-FET chemical sensors can be tested simultaneously;
- up to 3 different ions solutions (extendable up to 7) can be added independently into measurement solution (bath) to obtain required ions concentration;
- the volume of measurement solution circulating in stand is some of 200 ml;
- the ion solution volume, separately injected into measurement solution, can be set in range from 10 nl to 20 ml (in one cycle) with resolution of 10 nl and accuracy of 30 nl;
- the temperature of measurement solution can be set in range from +5°C to +40°C with minimum step and accuracy of ±1°K;
- the minimum sample temperature (+5ºC) can be achieved in 12 min. (by chilling) and the highest temperature (+40ºC) within 10 min.
- U_{DS} and U_S voltage measurement error is less than 0,1%.
- I_D current measurement error is less than 0,25%.

CONCLUSIONS

The constructed measurement stand has been preliminary tested. Obtained technical parameters are satisfying.

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