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APPLIED PHYSICS

A special design of the measuring cell was proposed, which makes it possible to determine the optical and electrochemical characteristics of thinfilm electrochromic electrodes simultaneously. Also, the proposed cell provides constant temperature control using a small-sized thermostating unit built on Peltier elements and digital boards of W1209 thermostats.

The cell was made using 3D printing with ABS plastic by the fused deposition method (FDM), followed by a sealing stage using a solution of polymethyl methacrylate dissolved in dichloroethane.

In the course of the research, the use of a green laser with a wavelength of 520 nm was substantiated. Separately, the linearity of optical readings, the dependence of the indicators of the optical characteristics measurement system on temperature, as well as the uniformity of electrolyte heating in the cell, were studied. In addition, the pattern of the electric field was determined, which was an indicator of the uniformity of the current density distribution on the measured electrode.

The obtained dependences made it possible to assert that the characteristics of the cell and the measuring system as a whole are suitable for the stated research purposes.

It was also shown that the cost of the cell, together with the optical measuring system and the constant temperature control system, is more than two times cheaper than simple electrochemical cells offered by manufacturers.

The proposed algorithm for the development of the cell design, the approach to the selection of components, as well as the given technical details, allow us to manufacture measuring equipment for the specific goals of the researcher. In this case, the given schematic, structural, and hardware solutions can be used separately from each other

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DEVELOPMENT OF A SPECIAL CELL FOR OPTICAL AND ELECTROCHEMICAL MEASUREMENTS USING 3D PRINTING AND MODERN ELECTRONIC BASE

Valerii Kotok PhD, Associate Professor Department of Processes, Apparatus and General Chemical Technology* Senior Researcher Competence center «Ecological technologies and systems»** E-mail: valeriykotok@gmail.com Vadym Kovalenko PhD, Associate Professor Department of Analytical Chemistry and Chemical Technology of Food Additives and Cosmetics* Senior Researcher Competence center «Ecological

technologies and systems»** E-mail: vadimchem@gmail.com *Ukrainian State University of Chemical Technology Gagarina ave., 8, Dnipro, Ukraine, 49005 **Vyatka State University Moskovskaya str., 36, Kirov, Russian Federation, 610000

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1. Introduction

The fields of chemistry, materials science, and the development of new materials are constantly expanding. The abundance of publications related to the development and research of new «smart», multifunctional materials often poses non-standard tasks for researchers to measure their characteristics. As an example, we can cite a class of materials related to chromogenic – electrochromic films [1, 2]. These films, when exposed to electricity, can change their optical characteristics, color, transparency, opacity, reflectance, and selectively block infrared radiation. Such materials

are used to create «smart» windows [3], mirrors [4], which adjust their optical characteristics. Commercial «smart» windows use materials in which electrochemical reactions take place [5, 6].

The development, study, and testing of electrochromic materials require specific conditions, including cells of a special design. These conditions, firstly, include the simultaneous measurement of changing optical and electrochemical characteristics. Secondly, it is necessary to ensure the uniformity of the current distribution over the electrode area and maintain a stable temperature (as in the case of studying classical electrochemical systems). Thirdly, the hardware implementation of all of the above should not affect the measurement of the characteristics of a transparent thin-film electrode.

At the same time, the proposed solutions may not be suitable in terms of characteristics, and may also be expensive. Nevertheless, in the last decade, there has been a rapid development of the elementary electronic base, the market for ready-made electronic modules, as well as a significant reduction in the cost of 3D printing. This makes it possible to create sufficiently accurate and at the same time cheap measurement systems, as well as to develop special cells for carrying out measurements based on the narrow needs of the researcher.

2. Literature review and problem statement

Composite electrochromic Ni(OH)2-polyvinyl alcohol film, which is planned to be investigated in the designed cell, is tested in a weakly alkaline solution [7]. In this case, the compositions of the tested films may differ [8]. The deposition takes place on a thin (0.9 mm) glass electrode covered with a layer of electrically conductive transparent oxide -SnO₂ doped with fluorine (FTO glass). In the colored state, the film becomes dark brown [9]. The color of the finished electrochromic device in the darkened state can remain brown, provided that transparent or partially transparent non-chromogenic counter electrodes are used [10]. Since the kinetics of chemical and electrochemical reactions depends on temperature, it is important to be able to stabilize this parameter over time. In this case, the minimum requirement for the cell is the possibility of parallel measurement of optical and electrochemical characteristics with the possibility of local temperature control. Another important requirement is the measurement of optical characteristics without additional bulky and expensive equipment. In addition to all of the above, the cell should be designed in such a way as to ensure a uniform distribution of the current density over the electrode area [11]. In this case, as shown in [12], the current density distribution is critically important in the electrochemical deposition of inorganic films.

Search, comparison of characteristics, as well as the availability of the proposed solutions showed a small number of proposals from manufacturers of such equipment. Besides, existing solutions have certain disadvantages.

According to [13], the cost of a three-electrode classical cell for carrying out electrochemical tests without a thermostating jacket, depending on the volume and type, is 240–650 USD. This manufacturer does not offer solutions suitable for optical testing.

Another manufacturer of electrochemical equipment [14] offers several designs of three-electrode classic jacketed cells for 660 USD. The presented cell allows only electrochemical tests at a controlled temperature. Additional equipment is required to maintain the temperature.

An analysis of the cell design and parameters [15] showed that this cell was developed for the study of electrochromic materials. However, it requires additional equipment to determine the optical characteristics. Also, the analysis of the cell design revealed the absence of acceptable options for maintaining a given temperature in the working space. In addition, due to the cell design, it is possible to use only an electrode with certain dimensional characteristics. At the same time, one of the big disadvantages of this cell is that the counter electrode has a spiral design. So, in the case of gas formation on the counter electrode, the rising bubbles will cross the transparent window for measuring the optical characteristics. The latter will violate the correctness of the optical indicators.

A cell of another manufacturer has a similar design to the previous one [16]. In accordance with the design, the disadvantages of such a cell are similar to the previous one.

According to the product description and photographs in [17], this cell has the same disadvantages as the previous ones. However, in comparison with the previous versions, the manufacturers provided the ability to maintain the temperature in a wide range of -25, +105 °C. In addition, the presented product can be used to determine optical characteristics in a wide range of wavelengths from 200 to 5,500 nm. However, it is clear that additional equipment is required for both temperature control and optical characterization.

A quartz cell in [18] can potentially be used to measure the parameters of electrochromic electrodes. However, as with the previous cells, additional equipment is required. The absence of a jacket allows the temperature to be maintained by immersion in the thermostat, but it will not be possible to measure the optical characteristics.

It should be noted that the cost of cells with the ability to measure optical characteristics is not indicated. It can be assumed that their cost will be many times more expensive, which makes their use problematic. Besides, additional expensive equipment is required to carry out measurements. Thus, the study of electrochromic materials may not be possible for developing scientific groups.

As a solution, a simple approach, design, and instrumentation are proposed for the implementation of the set goals.

3. The aim and objectives of the study

The study aims to develop a fully functional system for complex measurement of the characteristics of electrochromic materials based on 3D printing and a modern base of electronic components.

To achieve this aim, several objectives can be identified:

– to develop a cell design for simultaneous electrochemical and optical measurements with the ability to maintain temperature and justify the choice of the laser used in the installation;

- to check the chemical resistance of plastic for 3D printing;

to check the characteristics of the optical system;

- to check the uniformity of the temperature in the cell with the given design features and the distribution of the current density on the working electrode.

4. Materials and methods of research in the development of the cell for optical and electrochemical measurements

Reagents and materials.

For 3D printing, ABS plastic (copolymer of acrylonitrile, butadiene, and styrene) of the black color of the ABS+ grade (1.75 mm filament) was used. The black color was chosen for maximum absorption of light rays when reflected from solution boundaries, glass partitions, etc. A 6 M KOH solution was used to test the plastic for chemical stability. To improve the sealing of the cell, the inner surface of the cell was coated with a 10 % solution of polymethyl methacrylate (PMMA) dissolved in dichloroethane. The linearity of the indicators of the optical system was checked using aqueous solutions of the Carmoisin (Azorubin) dye with a concentration below 0.104 g/L. Carmoisine was chosen based on the maximum absorption, which corresponds to 516-519 nm [19], which in turn corresponds to the wavelength of the green laser used (520 nm).

The determination of the electric field pattern was carried out in a 3 % Na₂SO₄ solution. In the measurements, a current source powered from the mains was used, which made it possible to regulate the output voltage. The measurements were carried out on alternating current.

The temperature in the cell was measured using a thermocouple complete with a UNI-T UT70B multimeter (China).

To test the electrochromic material, measure the shape of the electric field in the cell, as well as the uniformity of heating from a thermostat, 0.1 M KOH was used. An electrochromic film on a transparent electrically conductive substrate (FTO glass R<10 Ohm/sq., Zhuhai Kaivo Optoelectronic Technology Co. Ltd. (China)) was prepared of nickel nitrate solution with the addition of polyvinyl alcohol by electrochemical method [20]. Before applying the film, the FTO glass was degreased by wiping with a paste based on water and soda. Then, the substrate was rinsed with running and distilled water and also treated with ultrasound in an ultrasonic bath in 96 % ethanol (10 minutes, 60 W, 41.5 kHz). After that, it was dried immediately before the next operation and wiped again with a lint-free cloth with ethyl alcohol. In this case, the dimensions of the working area of the FTO glass were 2×2 cm, the total dimensions were 3×2 cm.

To determine the absorption spectrum of electrochromic films, the studied electrodes were colored by sweeping the potential from +201 to +701 mV (NHE) at a sweep rate of 1 mV/s. The coloration-bleaching of the electrode was carried out by cyclic voltammetry, setting the potential from +201 to +701 mV (NHE) at a rate of 1 mV/s, 5 cycles. An Elins P-8 potentiostat (Russia) was used in all electrochemical tests.

Cell model making.

For the manufacture of the model, a 3D model of the cell was developed, which is shown in Fig. 1.



Fig. 1. Cell design:
a - left side view; b - top view; c - right side view.
1 - place for a laser module; 2, 3 - glasses; 4 - the main section of the cell; 5 - section for intermediate
electrolyte (0.1 M KOH); 6 - section for the thermostat heat exchanger; 7 - section for the reference electrode (silver chloride reference electrode in a saturated KCl solution);
8 - section for temperature sensors; 9 - tube for the light intensity sensor; -- - recesses for the counter electrode

The cell was printed on a MakerBot Replicator 2 3D printer using the FDM method (150 mm/min 0.15 mm, 20 % coverage). After printing, the supports were removed mechanically, then the inner surfaces of the cell were covered in several layers with a PMMA solution with intervals of one day for complete drying. The last stage was gluing the glasses into the sidewalls of the cell (positions 2, 3 in Fig. 1). Slides for optical microscopy with dimensions of 25×76 mm were used as glasses.

Optical system.

The cell design assumes that the investigated electrode with an electrochromic coating will be located in the main section 4 near glass 2 (Fig. 3). On one side, the laser module is located (position 1, Fig. 1). On the other side, there is a photodiode in the long tube 9. A long tube is needed to cut off light from above and from the sides. The tube diameter corresponds to the photodiode diameter. Thus, light enters the photodiode only from a narrow direction angle. A laser LED is used as a light source. This choice is associated with two circumstances. First, the laser has a nearly non-diverging beam of light. Secondly, the laser emits powerful radiation, which is several times brighter than the light in the room. Thus, the part of the light in the room that still reaches the photodiode has low power compared to laser radiation and does not introduce measurement errors. In this case, the main difference from the cells offered by manufacturers is that there is no need for additional equipment to measure optical characteristics. It is also important that when using the developed cell, it is possible to directly visually assess the quality and color characteristics of the coatings under study. For the proposed optical system, a laser module with a D18X65 lens system (China) with a radiation wavelength of 520 nm (green) and a power of 10 W was chosen. A BPX61 large-area silicon photodiode (China) was used to fix the light flux after passing through the measured electrode (Fig. 2).



Fig. 2. Appearance of the BPX61 photodiode used (TO-39 package)

The advantages of the product are a wide range of operating temperatures and detectable wavelengths (-40÷125 °C, 400–1,100 nm). It should be noted that the system works as follows. The focused beam from the laser module (position 1, Fig. 1) passes through the glass, then through the transparent electrode with the investigated film, through the electrolyte, and the second glass, passing through the tube, hits the photodiode. The photodiode is connected via an ADC (E-154, Russia) to the computer. When the color of the electrode changes, part of the light does not pass through the film, and the power of the beam on the photodiode decreases, which leads to a change in the photodiode voltage. In order for the evolution of gases not to interfere with the course of the beam and, thereby, not to affect the measurement, a counter-electrode of a special design was proposed (Fig. 3).

The counter electrode is inserted into the cell in the recesses indicated by the red dotted line (Fig. 1). The design is implemented so that there is a relatively uniform current distribution on the working electrode. In this case, the design assumes that gases are released at the edges and rise mainly inside the n-shaped cavity of the half-electrode.



Fig. 3. Design of the counter electrode for the experimental cell: nickel foil is marked in gray; pink – copper bus; green – laser beam; the arrow shows the direction of gas rise

Temperature control.

Temperature control is implemented using two Peltier elements (TEC1-12703, China), two digital thermostat boards (W1209, China), two aluminum radiators, and two small-sized fans (DC12S4010M, China) in accordance with Fig. 4, *a*, *b*. All heat transfer surfaces were coated with GD66 thermal paste (China) before assembly.

The power of the Peltier elements was selected based on the heating/cooling rate of the aqueous solution. Considering that the working volume of the solution is about 100 ml, heating by 1 °C takes time according to a simplified calculation (excluding heat loss, efficiency of the elements) according to formula (1):

$$\tau = m \cdot C/P,\tag{1}$$

where *m* is the mass of water, 0.1 kg; *C* is the heat capacity of water, in 4183 J/(kg·deg); *P* is the power of the Peltier element, 29.7 W.

Then, it takes 14 seconds to heat or cool the water for each degree. Taking into account the losses and thermal inertia, as well as the heat required to change the temperature of the elements, it is possible to take 200 % heating (cooling) time per 1 °C, i. e. no more than 28 s, which is quite acceptable.

The installation works as follows. On one digital thermostat (W1209, Fig. 4, c), the lower temperature limit is set at which the heating is turned on; on the second, the upper temperature limit is set at which the cooling is turned on. The limits are set based on the needs of the researcher, while the difference between them should not be less than 0.5 °C. Two temperature sensors connected to each of the W1209 thermostats (not shown in Fig. 4, b) are located in section 8 for temperature sensors (Fig. 1). If the temperature is below the set threshold value, the Peltier element TEC1 is activated, which heats the aluminum plate A1. The latter is in a solution, in which the required temperature is maintained (section 6, Fig. 1). If the temperature of the solution exceeds the upper threshold value set on another thermostat, the second part of the circuit with the TEC2 cooling element is switched on. For efficient cooling or heating, the second side of the elements is heated or cooled using a radiator and a small fan. It should be noted that aluminum is not resistant in an alkaline environment, so the working part of the plate was treated with KPT-8 thermal paste and wrapped in nickel foil. Nickel foil exhibits high corrosion resistance in alkaline environments.

The advantage of such a thermostat is the absence of the need for additional equipment, ease of manufacture, no branch pipes, and a high heating (cooling) rate.

5. Results of parameter measurements of materials and cells

5. 1. Determination of the chemical stability of the plastic used

In the literature, it was found that ABS plastic is resistant in concentrated alkaline solutions [21–23]. However, since ABS plastic of the latest generation was used for printing (ABS+ grade with a low coefficient of thermal expansion), it was necessary to accurately determine the resistance of the latter.

When the used plastic was kept in 6 M KOH for a month, no changes in weight, color, plastic characteristics, as well as delamination, before and after exposure were found (Fig. 5).

Proceeding from this, it can be argued that the selected plastic is resistant to the required concentration limits used in the studies [19, 23].



Fig. 4. Thermostat on Peltier elements:

a - simplified installation diagram (connecting elements are not shown); b - simplified circuit diagram; c - digital thermostat board (without case): M1, M2 - fans; TEC1 and TEC2 - Peltier elements (cooling and heating the aluminum plate A1, respectively), the elements related to the heating and cooling circuit are shown in red and blue, respectively; \blacksquare - fans; \square - aluminum radiator; \blacksquare - Peltier elements (shown without wires); \square - aluminum plate



Fig. 5. Plastic support wall kept in a 6 M KOH solution for a month

5. 2. Improvement of the optical system, verification of measurements, and evaluation of the temperature dependence of the sensor readings

Justification for the choice of the wavelength of the laser used. In order to select the laser wavelength, the absorption spectrum was recorded for the investigated electrochromic Ni(OH)₂-polyvinyl alcohol (PVA) film in the colored state (Fig. 6, a).

As seen in Fig. 6, a, the maximum absorption of the film is at a wavelength of 422 nm, which is also confirmed by other studies [25]. In turn, the sharp absorption of UV radiation in the short wavelength region is associated with the glass substrate. Obviously, it is desirable to measure as close to 422 nm as possible. Nevertheless, the character of the sensitivity curve of the photodiode used as a function of the wavelength of light [24] indicates that it is not desirable to carry out measurements in the region of 400 nm. This is due to the low sensitivity of the photodiode $S_r=10$ %. Considering that the market offers three visible lasers – violet (405 nm), green (520 nm), and red (650 nm, Fig. 6, *b*), it makes sense to use a green laser. Thus, the choice of the green laser is a compromise between acceptable photodiode sensitivity ($S_r=42$ %) and good absorption of the electrochromic coating under study. *Elimination of dark currents*.

During the evaluation experiments, it was determined that the voltage of the sensor in the fully illuminated state is about 0.5 V. However, in the fully colored state of the sensor, the photodiode produced variable-sign chaotic voltage values at the output (Fig. 7). Analysis of the information on this issue showed that semiconductor devices can generate so-called dark current noise, which is expressed through the appearance of a chaotic voltage at the output. Since these currents are much lower than the main ones, it was proposed to put a shunt resistance of 5 kOhm (0.25 W) in parallel with the photodiode.

Comparison of the curves in the region of the signal of the unlit photodiode showed that the proposed method completely eliminated noise.

Determining the linearity of the characteristics.

In order to evaluate the linearity of the signal characteristics, it was proposed to use dilute solutions of the Carmoisine dye, obtained by diluting the initial solution in Fig. 8 (the insert). The obtained characteristics of the photodiode signal in volts showed an almost linear relationship between the photodiode voltage and the Carmoisine concentration (Fig. 8). In this case, the reliability of approximation by the linear equation was almost equal to unity (R^2 =0.98). A slight bend cannot be attributed to deviations from the Bouguer-Lambert-Beer law, since it is observed in the region of dilute concentrations.



Fig. 6. Spectral characteristics:

a – electrochromic electrode Ni(OH)₂-PVA; b – relative sensitivity of the photodiode (S_r) depending on the length of the incident light [24]. Blue indicates UV absorption by glass



Fig. 7. Photodiode voltage under laser illumination: a - without shunt resistance; b - with shunt resistance of 5 kOhm

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Since the temperature of the sensor corresponds to the room temperature and may vary slightly, the temperature drift of the reading must be taken into account. For this, the curve of the dependence of the illuminated sensor on temperature was taken (Fig. 9). The dependence also turned out to be almost linear, and the calculated voltage temperature coefficient was -3.2 mV/K.



Fig. 8. Dependence of the photodiode voltage on the Carmoisine concentration. The insert shows that the initial solution of Carmoisine is 0.104 g/L



Fig. 9. Dependence of the photodiode voltage at maximum constant illumination on the photodiode temperature

This value is somewhat different from the reference value for this photodiode: -2.6 mV/K [24]. This coefficient should be taken into account when recalculating the voltage – optical transmission of the material.

5. 3. Evaluation of the temperature distribution in the cell using the presented thermostat design

The temperature distribution in the working chamber of the cell was estimated when the digital thermostat was set to maintain a temperature of 22 °C. It is worth noting that the chambers of sensors 1 and thermostat 2 are interconnected with the main chamber 3 through windows. This approach made it possible to achieve temperature uniformity even in the corners of the cell (Fig. 10).



Fig. 10. Results of temperature measurements during thermostating in the cell (a part of the cell is shown). The set temperature on the digital thermostat is 22 °C. Ambient temperature is 17 °C: 1 - section for temperature sensors;
2 - section for the thermostat plate; 3 - the main section of the cell (corresponds to section 4 in Fig. 1)

As you can see, out of four measurements in the cell, only in the far corner the temperature is 1 degree lower, which is insignificant, since there is an auxiliary electrode (counter electrode) there.

5. 4. Evaluation of the uniformity of the current density distribution in the main section of the cell

An important factor in the uniformity of the electrode operation is the uniformity of the current density distribution over the electrode area. For such a design of an electrochemical cell, the electric field is two-dimensional. The resulting field pattern indicates a uniform distribution of the current density, which can be determined from the parallelism of the equipotential lines near the working electrode (Fig. 11).



Fig. 11. Pattern of the electric field in the cell (a part of the cell is shown) Ambient temperature is 17 °C: 1 - section for temperature sensors; 2 - section for the thermostat plate; 3 - the main section of the cell (corresponds to section 4 in Fig. 1). Equipotential lines and voltage values for a particular line are marked in red, both lines and numbers. The counter electrode is marked in blue; the working electrode is in scarlet

At the same time, the auxiliary electrode has an uneven current density distribution, which is due to the selected shape of the electrode. In this case, the latter fact will not affect the quality of measurements.

6. Discussion of the cell characteristics, possible ways of improvement, and cost estimation

The proposed cell has small dimensions (Fig. 12), does not require additional expensive equipment, and can be adopted for studying other similar materials. It should be added that the cell can also be used to measure the concentration of non-ferrous solutions - dyes and some metal salts. A great advantage of the cell is the direct visual control of the processes on the electrochromic electrode. The disadvantages include the inability to measure spectral characteristics in this design. It should be said that the rest of the characteristics are acceptable and the cell can be used to evaluate electrochromic materials. Although the characteristic of the photodiode voltage – solution color (dye concentration) is slightly different from the linear one, the proposed system can be used in a series of experiments. That is, the comparison will be correct if the materials are examined in one cell. In this case, some curvature can be taken into account in the calculations. Most likely, the non-absolute linearity of this characteristic is due to the presence of shunt resistance in the circuit.

It is worth noting that the thermostat also copes well with the task. Moreover, due to the wide functionality of the W1209 digital board, the settings are stored in memory and the temperature sensor readings can be corrected. The thermostat itself can be used both in the cell and separately from it. Moreover, using special controller settings (hysteresis and power-on delay [26]) it is possible to achieve heating and cooling between two temperatures, which can be useful for solving specific problems.



Fig. 12. Photos of the finished cell: a - without a thermostat; b - with a thermostat

The raw signal from the sensor in the coloration-bleaching processes is shown in Fig. 13. Analysis of the form of the presented dependence illustrates that the cell fully copes with the task of recording changes in the optical characteristics of the electrode under study.



Fig. 13. Coloration-bleaching curve of the film electrochromic electrode. Raw photodiode signal data

After data recording, additional data processing is required, which takes into account the individual characteristics of the photodiode, which is not difficult in standard office programs. *Cell cost*

The analysis of the funds spent on the elements of the measuring system is presented in Table 1.

The cell costs 92.8 USD, which is more than half the price of simple electrochemical measurement cells. Moreover, in

this case, there is no need for additional costs for an external thermostat.

The only additional element for optical measurements is the need for an ADC with appropriate software. However, the market offers many solutions to solve this issue. These are digital oscilloscopes, multimeters, and ADCs for industrial automation. It can also be solved by attracting cheap automation solutions using Arduino, Raspberry Pi, modules based on ESP8266, STM32 (Blue pill), and extension boards to them [27].

Table 1

List of components and costs for the manufacture of a single cell

No.	Title/Description	Quantity	Cost*, USD
1	3D model printing	1	21
2	Consumables: glass, glue, PMMA, di- chloroethane, thermal paste (KPT-8), heat shrink tubes, nickel foil	_	5
3	Peltier element (TEC1-12703)	2	5.8
4	Thermostat power supply $(12 \text{ V}, 10 \text{ A})$	1	11.4
5	Aluminum plate	1	4.2
6	Controller (W1209)	2	4.6
7	Cooling/Heating kit (DC12S4010M fans, CG66 thermal paste, heatsinks, and mounts)	1	12
8	Laser module with power supply (D18X65)	1	27.5
9	Photodiode (BPX61)	1	1.3
Total			92.8

Note: * - taking into account the quantity.

It should be noted that for greater convenience, it is necessary to strive for an integrated temperature setting and control, signal pickup, and information processing system. The solution to this issue can be achieved through the development of special software.

In conclusion, we can say that using the proposed algorithm for designing cells and measuring systems, the latter can be easily changed for the specific goals of the researcher.

7. Conclusions

1. A cell with the possibility of thermostating and simultaneous measurement of optical and electrochemical characteristics of electrochromic electrodes in alkaline solutions of 0.1 - 6 M KOH has been developed and tested.

2. An approach to the development of cells and measuring systems for specific purposes of the researcher using a modern electronic base has been proposed. It is shown that in the case of measurements in the proposed cell of an electrochromic electrode based on Ni(OH)₂ films, a green laser λ =520 nm should be used.

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