Dmytro V. SNIZHKO[1], Olga A. SUSHKO[1], Elena A. RESHETNYAK[2], Dmitro H. SHTOFEL[3], Tomasz ZYSKA[4], Andrzej SMOLARZ[4], Nazarbek MUSSABEKOV[5], Aliya KALIZHANOVA[6]

Kharkiv National University of Radio Electronics (1), Kharkiv V.N. Karazin National University (2), Vinnytsia National Technical University (3), Lublin University of Technology (4), Kazakh National Research Technical University after K. I. Satpaev (5), Al-Farabi Kazakh National University (6)

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Colorimeter based on color sensor

***Abstract**. In this paper colorimeter based on color sensor is considered. The developed colorimeter experimental utilization is shown on example of detection Cu2+, Co2+ ions and Neutral Red indicator. Realized measurement optical system in the colorimeter supports operation with different types of sorbents, as possibility is shown an operation with silica, polyurethane foam and the indicator paper. Dynamic range of device at 12 bit per channel and well stability of characteristic are enough to work with fine reliability that is no more 0.7% for analyzed test systems.*

***Streszczenie.** W artykule przedstawiono kolorymetr zbudowany z wykorzystaniem czujnika barwy. Przedstawiono wykorzystanie przedstawionego kolorymetry na przykładzie detekcji jonów Cu^{2+}, Co^{2+} i neutralnego wskaźnika barwy czerwonej. Zrealizowany system optyczny kolorymetru umożliwia pracę z różnymi rodzajami sorbentów. Możliwości te pokazano z wykorzystaniem krzemionki, pianki poliuretanowej i papierka wskaźnikowego. (**Kolorymetr zbudowany z wykorzystaniem czujnika koloru**).*

Keywords: diffusion, reflection, color, analysis, indicator.
Słowa kluczowe: dyfuzja, odbicie, kolor, analiza, wskaźnik.

Introduction

Optical measurements stay a main analytic technics. They are used to control chemical composition, physical properties definition, supervision of technology processes in many areas as laboratory analyses, ecological monitoring, pharmacopoeias, field-test analyses and other [1-4].

In different fields historically the color is used as an optical characteristic to control object properties. The light interaction with matter is used for analysis of last one in different technics. Reflection technics is very attractive for non-destructive analysis. Generally reflection has mirror and diffusion components. Frequently color of non-transparent (and non-luminescent) objects is determined by a diffusion part of reflected rays. So to obtain information about object interactions with light we should analyze this component at different wavelength. A colorimeter is device that is used for reflected color measurements it contains two main elements: illuminant and photo detector. Their combinations represent different techniques of color measurements or colorimetry. As example, for realization of measurement different illuminants can use one-by-one to analyze response at different wavelength. In this method for sensing is use one detector with spectral sensitivity covering all illuminants spectral ranges. If need obtain color coordinate in with case three or more illuminants is applied. This method has limitation, because including of a big number of illuminants is difficult in one device. Alternative is using one type illuminant and analyze a diffusion component of a reflected part of a light in different spectral ranges. It's realized by using a system of several light filters and one photo detector. For color measurements a spectral characteristic of filter should be close to a human eye characteristic. To simplify color measurement filter matrix is combined with photo detector matrix. Last methods have place in color CMOS and CCD cameras and color sensors. Utilization of multipixels matrix in analytics is limited because analytical light from object is divided between pixels so for sensitive assay light collection at small numbers of pixels with possibilities to integration is preferable. It is realized in color sensors. Many firms represent different sensor constructions and packaging that can include some subsidiary electronics on a chip. A great interest is a high-end device that has all enough components that needed for measurements as digital interface, ADC, amplifier, matrix control logic and sensor. S9067 from Hamamatsu, ADJD-S311-CR999 from Avago as examples of this type devices are that include a detector matrix, pre-amplifier, analogue-digital converters, control logic and digital interface. Utilization color sensor in feedback loop for adaptive control of displays determines increasing number and quantity of color sensors.

In the article theoretical and technical problems for realization of simple, effective, and cheap colorimeter (fig. 1) as alternative to expansive spectrometers were analyzed. Device is based on Hamamatsu color sensor S9037 Proposed technician solutions are discussed. The device testing is shown by using various color reactions which are placed at different substrates.

Colorimetry is well alternative to spectroscopy in more situations. As well as the spectrum variation the color change of analytical system is connected with substances concentration in a sample. In many cases sensitivity of assay is determined by specificity of chemical reaction between a reagent and an analyte, so application of cheaper color detector is justified. In case of visual detection assays with color scale for analyte the determination of concentration is not precise. The application of the developed colorimeter helps to obtain quantitative determination and precision is enough to resolve many technical problems without complex and expensive instrumentation.

Fig.1. The colorimeter C1001 (working side)

Colorimeter versus spectrometer

Implementation of colorimeter in analysis accomplish by next benefits under spectroscopy. Colorimeter is instrument for psychophysical analysis and provides measurements that are correlated with a human eye-brain perception. A colorimeter data directly read from measurements versa spectroscopy. Spectrometry provides wavelength-by-wavelength spectra analysis, reflects optical properties objects without interpolation by a human. Colorimeter and spectrometer based on optical sensor but sensor for colorimeter detector is simplest so it is cheaper. Spectrometers construction is expensive because is include optical bench, prisms, gratings, lenses etc. thus are complex devices. Consequently work with spectrometer is more difficult, that neglected more complex automation. Comprehensive spectroscopy analysis is indispensable methods in research and development works. Colorimeters are usable for measurements with constant conditions when color changes are determined, i.e. investigator has a priori data about a sample composition, an analytical reaction and a relation between analyte and response. Simplicity of colorimeter allow constructing smart-device for realization different modern conceptions as pocket-test for object, screening test in technological processes, when object is unstable or non-transportable, express assays in urgent medical care, quality control on the spot, real time hazards monitoring.

The problem of tool complexity for spectroscopic method which is the most adequate for carrying out of color researches is reflected in the high price of this equipment. An example of high miniaturization spectrometer systems can be fiber optic systems, for example, such as Hamamatsu mini-spectrometer C10988MA.

There are today many field of colorimeter application as biologics and pharmaceutics, chemical industry, food industry and quality inspection in building, paint and coating, paper industry, plastic fabrications, textile and other. Colorimetry is utilized as a good approved modern technique so this method is in progress [5].

Proposition of construction

In proposed colorimeter we realized a color coordinates measurement method for a diffusion reflective light. Main idea of its design is a minimal cost with saving a most metrological characteristics and functionality. A block diagram of the colorimeter is represented on fig. 2. We use several technical conceptions to build it.

At first, color sensor is used as an optical detector. Several companies represent them on the market, Hamamatsu and Avago are leaders in construction of these optical transducers. The developed colorimeter is based on S9037 from Hamamatsu that has matrix of optical converters which have optical filters on ranges for red, green and blue spectral field with spectral area of sensitivity 400÷720 nm (conventional range of visible light). Besides the color sensor includes pre-amplifiers for three channels, 12-bit ADC, buffers and sensor control module which supervises a measurement process and a digital serial interface so it is a complete list for a signal processing. Important feature of the sensor is big sensitive area 0.32 mm2 for each channel Last one does this sensor especially convenient to analytical instrumentation and measuring systems. Low power consumption, 12 bit per each color channel signal resolution, and probability to adjust sensor sensitivity does the sensor a dominant device for color measurement.

At second, for lighting is used a light source with long life time, good stability, and shot start-up time. This source is white super luminescent light emitting diode (LED). LED is a high efficient illuminant that is significant for portable devices, perfect life time and negligible small start-up time is sufficient also. Spectral characteristics of sources are important because composition of reflected light (color) from object depend on a spectral characteristic of illuminant (blue object in red light would be black). So for color measurements is need ideal white illuminant. A real illuminant is not really linear so colorimeter optical transducers should have different sensitivity for color channels. Undoubtedly scaling of sensitivity of transducers is not precisely correct reproduce standard light. To solve this problem addition mathematical operation is used. However in many applications is not important absolute value calibration procedure and reference measurement is used according to obtained calibration curve or calibration by ideal white surface. Frequently as ideal white object it is use magnesium oxide or barium sulphate pressed powders, tetrafluorethylene (teflone) or spectrolone, spatial "milk" glasses and etc. In some situations as etalon use white paper but it must do not have luminescent bleach powdering that is in many office "white" paper.

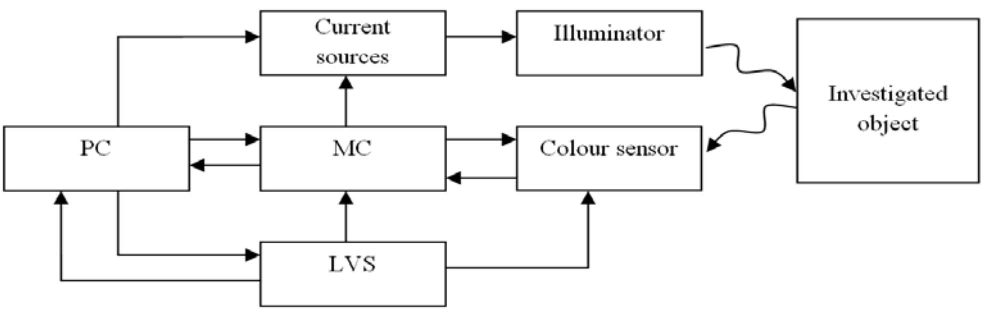

Fig.2. Block diagram of the colorimeter: PC – personal computer, MC – microcontroller, LVS – line voltage stabilizer

In our colorimeter color sensor with spectral sensitivity rough to human eye is that is etalon spectral sensitivity in color measurements. Four pure white LED from OptoSupply OSW5DA5201A is used as illuminant, small irradiation angle (for 50% power level it is 8 spherical deg.), high illumination (50000 mcd at 20 mA forward current), chromaticity coordinates (x = 0.27; y = 0.28 in XY-coordinates system) near to illuminant type C and low cost defined selection. Sensitivity of each channel is determined by colorimeter calibration with ideal white object in the colorimeter. To white balance procedure we use tetrafluorethylene 10 mm height cylinder stamped in black plastic holder.

The specific feature of the colorimeter is using of four LEDs for illumination that is placed around sensor and directed at 45° to an investigated object surface as seen on fig.3. This is needs to realize diffusion lighting of object and to remove shadows on rough surface as powders. This specificity in lightning is differ that use in paper scanner or mobile phone cameras so it significantly improve measurement metrology. Color sensor is placed between LEDs under focus place of LED illumination so as to place at 45° to each LEDs and is armored by a black cylindrical shield. This prevents a penetration of direct and mirror reflected beams to sensor, only diffusion component is detected. The illuminant construction is important in investigation of objects with rough surface. Positioning sensor under investigated object at distance 10mm and presence relatively large input window of color sensor 1.2mm ×1.2mm do integration measurements from a spot Ø5mm. This integrates a signal from surface and increases reproducibility and representation in real object measurements. Since real object frequently have microstructure of surface that need averaging between elements of structure on some representative square. Objects with 5 mm diameter as a minimal, easy-to-use, defensible size of investigated object.

At fig. 3 the schema of the lightning is shown. Sample (1) is illuminated by four light-emitting diodes (2) with the narrow irradiation diagram. The light irradiation is directed from four sides and turn to analyzed surface at 45°. The color sensor (3) is placed between light-emitting diodes on distance in 12 mm from analyzed surface of a sample object. Additionally the sensor is protected by cylindrical shield to prevent penetration of lateral radiation of light-emitting diodes. The measurement system satisfies to requirements of International Commission on Illumination (also known as Commission Internationale de l´Eclairage) [6, 7] and to color sensors application notes recommendations [8, 9].

Interface

Microcontroller ATMega164P is a smart bridge between the color sensor with the light source and PC. A concept of universal measurement tool for different assays demands a flexible control interface. A colorimeter connection with a personal computer (PC) is realized by USB. The colorimeter communication interface uses Human Interface Devices (HID) protocol that exclude a needing in a custom driver because standard HID USB driver is included in many OS as Microsoft® Windows XP, Vista, 7, 8, 9, 10.

Colorimeter supported USB 2.0 specification and work at low speed mode for HID protocol it is 8 bytes of data every 10 ms that equal 800 bytes per second. To transfer data and commands data packet at 8 bytes is used. It's a maximum packet size for low speed HID devises. The measurement process is controlled by the next sequence commands. Turn on/off command activates/deactivates illuminant (LEDs) and resets the sensor. Set sensitivity mode command chooses size of photo element matrix of S9706 sensor as small size 3×3 or standard size 9×9. Start measurement command initiates impulse generation with determined duration that is integration time and controls the sensor sensitivity. Get data command returns measurement data about color. The sensor response data 3 channels × 12bit transfer is packed into 3 words that is equal 6 bytes packet. Additional two functions control access to EEPROM that built-in MC and are used to story measurement conditions after calibration in the device.

The colorimeter is based on productive controller ATMega164P. Factors which have defined its use in colorimeter, is:

- manufacturing on 100 nanometers technology;
- low power consumption;
- high efficiency at low energy consumption, more 12MIPS for 3,3V and mA for core is important to program realization of USB support that has done without use additional interface circuit or signal level converter;
- presence of the advanced periphery allows to system scaling include probability us same MC (ATMega324P and ATMega644P) core with great volume of flash-memory (accordingly 32 kB and 64 kB instead of 16 kB for ATMega164P) that can be used for following improvement of colorimeter functionality.

Used MC as well as others MC of Mega series has built-in circuit for self-programming, support serial low-voltage in-system programming, built-in debug system that simplifies scaling and testing of developed device.

Power supply

The colorimeter consumes no more than 200 mA at voltage 4.4 that allow powering over USB (Universal Serial Bus). The device works with 3.3 V signal level electronics and compatible with USB signals so the low-dropout voltage regulator with low quiescent current LP2950-3.3 was used. It has allowed to conditions of a current consumption from USB. Possibility of color sensor and MC to work with 3,3V powering permits single voltage regulator in the device.

A current source as the power supply for light-emitting diodes connected seriously is used. Therefore the colorimeter scheme includes DC-DC converter with a current feedback [10]. Because power consumption directly is from USB the basic requirements for LED power supply is high efficiency, low noise and good stability. Serial LEDs connection decreases number of LED power current sources. The PWM up-converter is based on buster schema with a system of input and output filters which decreases pulsation. High efficiency of power source is obtained by use MC possibility to generate pulse signal.

Power save mode is realized in colorimeter and it is activated at no measurement time. Power JFET (P-channel) switch turns off power of DC-DC convertor, and MC turns off reference sources and pulse generator of MC as part of DC-DC convertor. A small energy consumption of MC does colorimeter utilizable in field-measurement with lap-top or pocket PC.

Control program and measurement process

To minimize construction of colorimeter all functions as data collection, storage, displaying and control interface are transferred to a host computer. This allows make a flexible and adaptive system, provides miniaturization and reduce cost of a colorimeter.

Colorimeter is connected to a personal computer (PC) by USB. A presence of standard protocol that is supported by many OS removes a necessity to a construction of a system driver for colorimeter. Colorimeter supports USB 2.0 specification and works in low speed mode for HID protocol is 8 bytes of data every 10 ms that equal 800 bytes per second. For transfer data and commands use 8 bytes long data packet as maximum packet length for low speed HID devises.

The calibration of the colorimeter is done in automated mode. The aim of a calibration is obtaining maximum signal (but not more 4095 counts) in each channel. As a calibration standard is main used a white surface, we used a cylindrical tetrafluorethylene solid block fixed in a plastic holder. Sequential step-by-step approach is used by adjusting from small to higher sensitivity of the color sensor to get maximum response available in the dynamic range of the sensor that is 12 bits so it is 4095. After calibration with white sample the colorimeter is ready to operation with test samples. Saving calibration data to EEPROM in the device exclude eliminate a needing at the next time. An operator can recall calibrated parameters of measurement from the device memory.

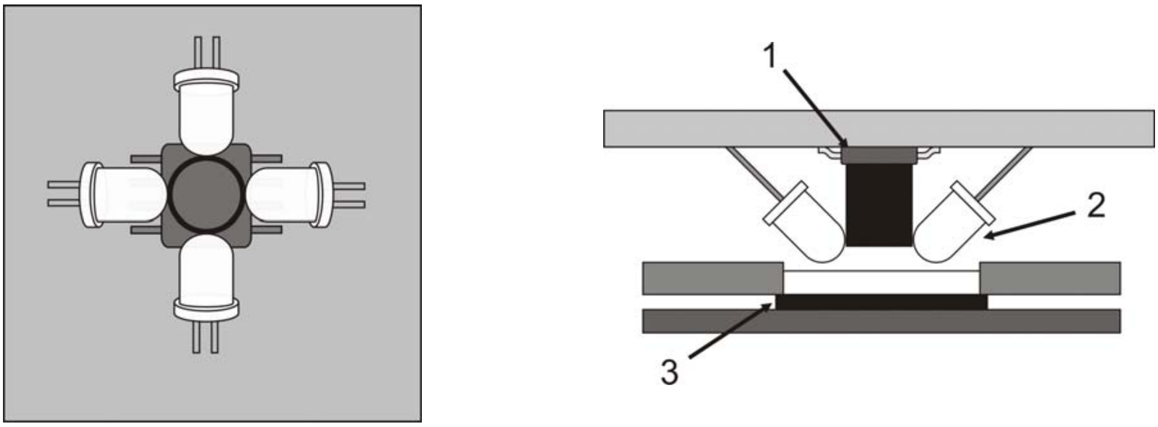

Fig.3. Optical schema of colorimeter: a – buttom view, b – side view. 1 – colour sensor; 2 – LEDs; 3 – sample

The measurement procedure of colorimeter contains sequential procedures (fig. 3). At the first step variables needed for operation are initialized. After that ports of MC are initiated it is included a turn on pulse generator, a reset of color sensor. Because sensitivity of sensor is changed together in all channels so settings of the measurement period is repeated three times to adjust sensitivity in each channel. The measurement procedure includes a command to transfer an integration time value, command to initiate a measurement, command to read obtained data. According to settings the colorimeter one-by-one measures brightness in red, green, blue channels. Alternative situation present in Avago sensor ADJD-S311-CR999 in which present probability for independent signal time integration control in channels, but sensors have small sensitive sensor matrix (0.1764 mm2 for four channels – red, green, blue, clean) and resolution only at 10 bits for channel contra 12-bit for Hamamatsu sensor S9706 with effective matrix area ~1 mm^2.

In the colorimeter probability presents possibility to average data for several measurements that increase reproducibility. In algorithm cycle to count measurement number is added and a test that an averaging process is complete. Then data exam on reproducibility is done a difference between current and previous measurements should be smaller than a clarified level. If color data difference is bigger new measurement is initiated. Measurements are repeated till obtaining reproducible data or iteration limit is finished. Condition for unstable measurement data is indicated by a mark "*".

After a measurement procedure command for turn off modules is called devices switch to power saving mode.

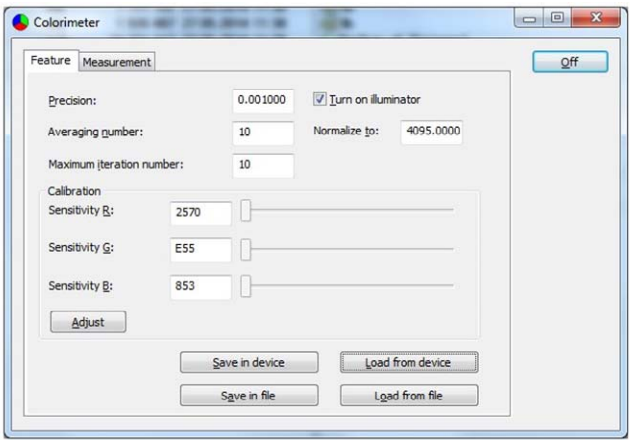

Fig.4. "Features" software window

The colorimeter software "Reflectometer 1.0" has graphical interface. On main window controls are separated to two pages. Control page «Features» (Fig. 4) contains controls for measurement features as for a calibration procedure as for adjusting of channels sensitivities, operation page "Measurements" (Fig. 5) contains controls for a measurement and a data management. Last one supports a measurement protocol saving to ASCII file.

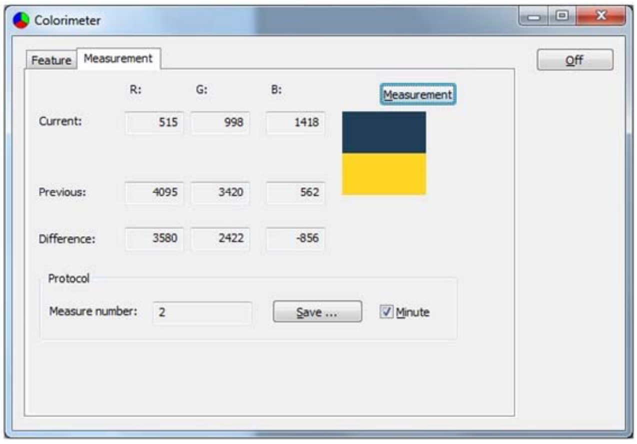

Fig.5. "Measurements" software window

Experimental tests

Repeatability of colorimeter measurements were done for different analytical system. In our testing we used the porous polyurethane (PU) foam, the dens reagent indicator paper and the silica gel. Test reactions with different colors were chosen (Table 1). [12, 13] The dependences of the measurement precision to sorbent material and to sorbate colors were done.

The diffuse reflectance spectra of the sorbates contained almost wavelengths in all visible range. The thiocyanate complex of Co^{2+} ion adsorbed on PU foam has absorption maxima 622 nm. The immobilized complex of Cu^{2+} ions with formazan is characterized by 558 nm. Neutral Red adsorbed on silica gel has absorbance at 540 nm.

After reaction with different analyte concentrations the test system colors were measured by the colorimeter (fig.6). The channel with maximum response was chosen for each system. The complexes of Co(II) and Cu(II) and Malachite Green have main changes in red channel, complexes of Fe(III) and indicator Neutral Red have biggest changes in green channel (table 2).

Discussion

The color measurement is determination of color coordinates in some color system. The RGB system is used for representation of a color as a sum of responses to a light that obtained from three types of receptors in a human eye, set as standard ICE 1931:

$$R = \int_{380}^{760} E(\lambda)\rho(\lambda)r(\lambda)d\lambda,$$

$$G = \int_{380}^{760} E(\lambda)\rho(\lambda)g(\lambda)d\lambda,$$

$$B = \int_{380}^{760} E(\lambda)\rho(\lambda)b(\lambda)d\lambda,$$

where: $r(\lambda)$, $g(\lambda)$, $b(\lambda)$ – color adding function, $\rho(\lambda)$ – spectrum reflection function, $E(\lambda)$ – spectrum energy distribution of light source function, R, G, B – brightness of red (r), green (g) and blue (b) lights.

Therefor color coordinates are depended to illuminator properties $r(\lambda)$, $g(\lambda)$, $b(\lambda)$ it is important to use standardized illuminator. But all of them is not compact and don't have long operation time. Alternative is frequently utilized to use mathematical correction of the sensor response as correction functions $K_r(G,B)$, $K_g(R,B)$, $K_b(R,G)$ for red green and blue components of color are determined. Final corrected data is $R_f = RK_r(G,B)$, $G_f = GK_g(R,B)$, $B_f = BK_b(R,G)$ consequently [11]. Because the applied color sensor has human like spectral sensitivity according datasheet information, for recalculation was used only data about spectrum of light emitted diodes. If a calibration curve is measured for a determined test system and determined colorimeter, this recalculation is note need, measurement will be in arbitrary units with depended connection with concentration.

Other colorimetric problem that same response can be obtained from different objects with different spectrum is not sufficient if to work with a verified analytical color reaction, when color changes have is bonded with an analyte concentration in a depended way.

Table 1. Used test systems in analyses

Analyte	Reagent	Indicator system	Sorbent	Concentration range, mol/l	Time, min	Color changing of a sorbent
Co^{2+}	SCN–	KSCN (1.0 mol/l), NaF (0.01 mol/l), H_2SO_4 (pH 2)	PU	$(0.5–40)\cdot10^{-6}$	30	white – blue
Neutral Red	–	HCl, pH 3	Silica	$(0.1–1)\cdot10^{-5}$	120	white – crimson
Cu^{2+}	Formazan	paper-Cu-Test, HCl (pH 3)	indicator paper	$(0.8–12)\cdot10^{-3}$	15	yellow – violet

Table 2. The meanings of the parameters of graduation curve (eq. 1)

Test system	Color channel	Concentration range, mol/l	Parameters of fitting curve $y_0+a\cdot e^{-x/t}$			R^2
			y_0	a	T	
Co^{2+}-SCN-PU	red	$(0.5–15)\cdot10^{-6}$	277.8	1119	32.71	0.989
Neutral Red-Silica	green	$(1–20)\cdot10^{-5}$	156.7	659.2	61.69	0.996
Cu-Paper test	red	$(0.5–24)\cdot10^{-3}$	142.7	85.09	2.796	0.957

Table 3. The precision characteristics of the R, G, B color components measurement results of different nature sorbents

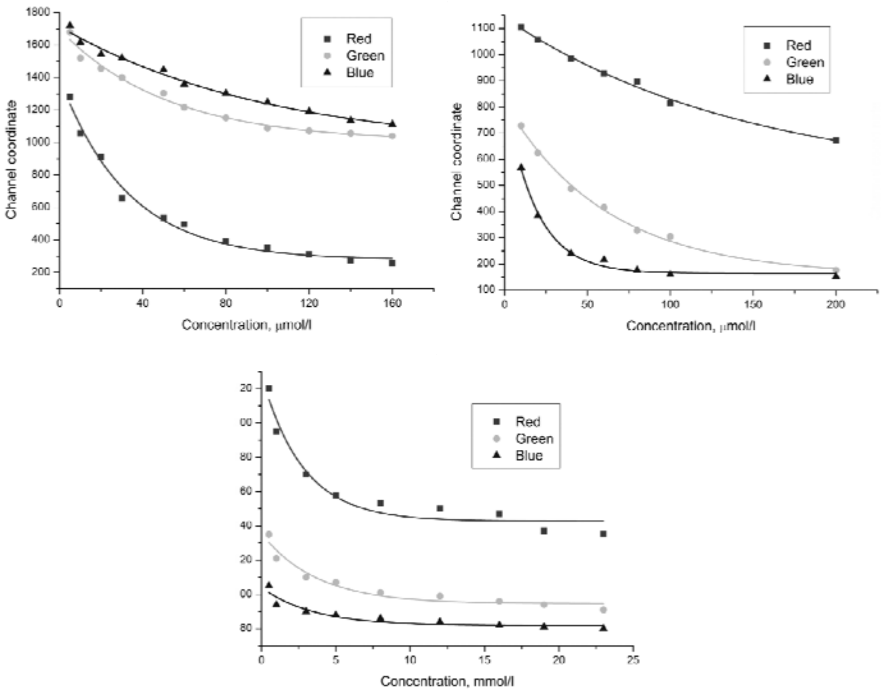

Fig.6. R-, G-, B- channels response in 4095 range (12 bit) for: (top – left) The thiocyanate complexes of Co2+ ions adsorbed on PU; (top – right) – indicator Neutral Red absorbed on silica; (bottom – center) – complex of Cu2+ ions with formazan on the paper

Conclusions

Application of colorimeter as an analytical system places an intermediate state between naked eye measurement with color verification by printed sample and spectroscopic measurements. Precision of colorimeter is comparable to spectrometer, so cheaper application of colorimeter is attractive for many practical tasks, though spectrometer stays universal instrument with more functional possibilities.

The testing of the developed colorimeter was done with indicator test system with different sorbents as polyurethane foam, silica and indicator paper. For all sorbents the colorimeter showed reproducible response. For analysis in selected range as 0.5 – 15μmol/l of Co^{2+} ions, 10 – 200 of Neutral Red indicator and 0.5 – 24mmol/l of Cu^{2+} reproducibility was not exceed 0,7%.

Colorimeter applications can be wider than operation with indicator test system. Flexibility of proposed device, small dimensions 70 mm × 60 mm × 35 mm, full USB compliance, low power consumption, peak power consumption does not exceed 0,5 W (in a measurement mode) carry out analytical assays in different task include medical analyses or monitoring of ecological situation as laboratory as field test conditions. The colorimeter can resolve analytical task in other different fields as well as industrial application, control of technological processes where color information is an adequate characteristic.

***Authors**: Dmytro V. Snizhko PhD, Olga A. Sushko, Department of Biomedical Engineering, Kharkiv National University of Radio Electronics, Nauki ave., 14, Kharkiv 61066, Ukraine, Elena A. Reshetnyak PhD, Department of Chemical Metrology, Kharkiv V.N. Karazin National University, Svoboda sq., 4, Kharkiv 61022, Ukraine, Dmitro H.Shtofel PhD, Vinnytsia National Technical University, Khmel'nyts'ke Hwy, 95, Vinnytsia 21000, Ukraine, dr inż. Tomasz Zyska, dr hab. inż. Andrzej Smolarz, Lublin University of Technology, Faculty of Electrical Technology and Computer Science, 38a Nadbystrzycka Str., 20618 Lublin, Poland, E-mail: t.zyska@pollub.pl, Nazarbek Mussabekov, Kazakh National Research Technical University after K. I. Satpaev, 22 Satpayev Str., 050013 Almaty, Kazakhstan, Aliya Kalizhanova, Al-Farabi Kazakh National University, 71, Al Farabi Street, 050013 Almaty, Kazakhstan.*

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