



A simple homemade light emitting diode based photometer for chromium speciation

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ABSTRACT

A simple spectrophotometric method utilizing a homemade light emitting diode (LED) based photometer is developed for chromium (VI) and total chromium determination in water samples. The photometer is composed of an LED (white color) as light source and a programmable light-to-frequency converter (three arrays of photodiodes each with a red, green or blue filter) as a detector. The chromium speciation is based on the complex formation reaction of Cr(VI) with 1,5-diphenylcarbazide. Considering the maximum absorption wavelength of the complex, the determination was performed using green filter detector. Prior to the total chromium determination, Cr(III) was oxidized to Cr(VI) with Ce(IV). Cr(III) concentration was calculated from the difference between total Cr and Cr(VI) concentrations. Under the optimum conditions, the calibration graph was linear in 10–200 $\mu\text{g L}^{-1}$ chromium concentration range. The relative standard deviation ($n=5$) and the limit of detection was calculated to be 2.45 and 2.99 $\mu\text{g L}^{-1}$ respectively. The proposed method was successfully applied to chromium speciation in spiked water samples.

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1. Introduction

Chromium is an element naturally found in rocks, animals, plants, soil, volcanic dust and gases. The properties of chromium strongly depend on its chemical state. While chromium (III) is essential but vital in the metabolism of glucose, fats, and proteins, the chromium (VI) is a toxic substance. Hence, highly sensitive methods with the ability to differentiate between Cr(III) and Cr(VI) is desirable [1]. Flame atomic absorption spectrometry [2,3], dispersive X-ray fluorescence spectrometry [4], inductively coupled plasma atomic emission spectrophotometry [5], inductively coupled plasma-mass spectrometry [6], electro-thermal atomic absorption spectrophotometry [7], electrochemical methods [8], fluorimetry [9,10], chemiluminescence methods [11], and high-performance liquid chromatography [12] are among the methods developed for separately determining the different chromium species. However, most of these methods are relatively expensive, time consuming, and or require complex instrumentation.

Due to the simplicity, cheapness, and ease of the availability, the spectrophotometric methods are widely used for chromium determination. Many chromogens have been utilized in the spectrophotometric determination of chromium [13–15]. Among these chromogenes, 1,5-diphenylcarbazide is a unique one in having high

selectivity and sensitivity [16,17]. Considering the low chromium concentration levels in the environmental and biological samples, pre-concentration is often a necessary step, prior to the spectrophotometric determination procedure. Columns containing Amberlite XAD resin [18,19], optode membranes optimized with anionic exchange resin [20], solid phase extraction [21,22], dispersive liquid–liquid microextraction [23] immobilized nanometer size titanium dioxide [24], and baker's yeast cells immobilized on controlled pore glass [25] are amongst the pre-concentration procedures utilized in spectrophotometric techniques for chromium speciation.

Due to the outstanding in situ determination ability and some other advantages, the portable devices using LED as light source are gaining a growing interest in developing simple light absorption techniques [26–31]. Monochromatic characteristics, low energy consumption, brightness, cheapness, long life, and excellent stability are among the characteristics with high performances. Morales-Rubio and co-workers [32] have developed LED based photometer employing solenoid micro-pumps to handle the reagent solutions. To improve the analytical sensitivity and attain the low concentration levels, a home-made flow cell with an optical path-length of 100 mm was used.

In this work, design and the development of a simple photometer for determination of the low chromium concentrations without a need for pre-concentration step or long optical path lengths is reported. The photometer designed employs a white color LED as light source, and a programmable color light-to-frequency converter (consisting of three separate sets of 16 arrays of photodiodes

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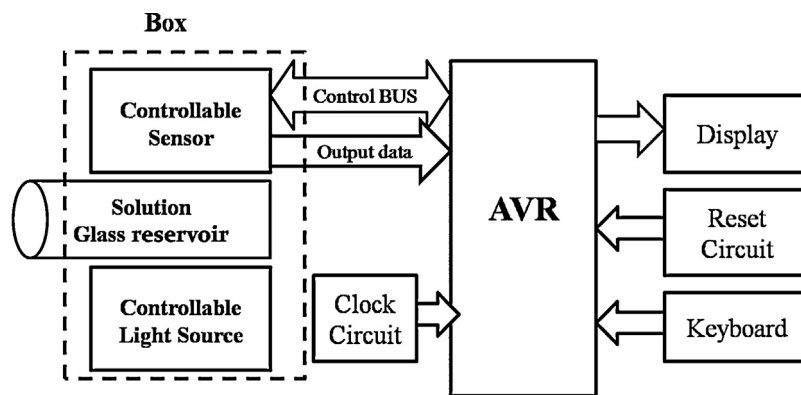


Fig. 1. Block diagram of circuit major.

each with a blue, green or red filter). Cr(VI) produce a purple colored complex with 1,5-diphenylcarbazide with a maximum absorption at 540 nm. Hence, a photodiode array with green filter was taken as determination mode of the detector. Cr(III) was oxidized to Cr(VI) by Ce(IV) before determination [33].

2. Experimental

2.1. Reagents and apparatus

Standard Cr(VI) and Cr(III) solutions (100 mg L^{-1}) were prepared by dissolving 0.3753 g of potassium dichromate or 0.7696 g of chromium nitrate (Riedel) in 1000 mL of water respectively. Working standard solutions ($10\text{--}200 \text{ } \mu\text{g L}^{-1}$) were made weekly by appropriate dilution with hydrochloric acid solution (0.5 mol L^{-1}). 1,5-diphenylcarbazide solution (0.052%, w/v) was prepared by dissolving 0.052 g in 2 mL of 99.5% (v/v) ethanol, making the volume up to 100 mL with sulphuric acid (0.5 mol L^{-1}), and storing in a refrigerator. This reagent was found to be stable at least for one week.

All the chemicals used, except those indicated, were of the analytical reagent grade purchased from Merck (Darmstadt, Germany).

A Shimadzu UV-1650 PC (Japan) double beam spectrophotometer was used for all spectral measurements and a Shimadzu 2554 (Japan) spectrofluorimeter to obtain LEDs light emission spectra.

2.2. The electronics and optics of the constructed photometer

The main circuit block diagram of the constructed photometer is shown in Fig. 1. The system is composed of two parts: concentration measurement part and data acquisition and processing part. The modular design method is used in the proposed system, including the programmable sensor, controllable light source, data-acquisition, central processing and power units.

The sensor used in this system is a TCS230 (the Texas Company Advanced Optoelectronics Solutions, TAOS) with four arrays of photodetectors with a red, green, or blue filter and one of them without any filter (clear). The filters of each color are distributed evenly throughout the array to eliminate location bias among the colors.

To maintain a constant light intensity, a common white-color LED (emitting a 450–620 nm continuous spectrum), was employed as a controllable light source. A small hole (3 mm d) was drilled at the center of the front side, and another hole (4 mm) at the center of the back side of the cell container (a metallic cube with 10 mm path length). Using some silicone glue, LED and the sensor, were glued on the front and the side holes respectively, in a way that the tips of the detector and the LED were positioned on the center of the holes. To reduce the interfering effects of the environmental

light on the analysis procedure, the solution reservoir, light source and the sensor were placed in a dark box.

For online output data (produced by the sensor), an ATMEGA32 (operating and processing) system was employed as the central processing unit.

2.3. The Analysis procedure

The colorimeter was turned on and the green filter mode (operation mode of the detector) was set. 1,5-diphenylcarbazide solution (0.052% w/v, 1 mL) transferred into a 10 mL volumetric flask containing a known volume of standard chromium(VI) solution ($10\text{--}200 \text{ } \mu\text{g L}^{-1}$), made up to the mark with distilled water, and after standing for 4 min, contents of the flask transferred into the cell, which was then placed in the colorimeter cell container. The frequency displayed on the LCD was read against a blank solution. The chromium content of sample solution was obtained using a calibration graph.

For total chromium concentration determination, 0.5 mL Ce(IV) (120 mg L^{-1}) was added into a volumetric flask containing a known volume of standard Cr(III) sample solution ($10\text{--}200 \text{ } \mu\text{g L}^{-1}$). After standing for 2 min, 1,5-diphenylcarbazide solution (0.052% w/v 1 mL) added and the determination procedure was performed as for chromium(VI).

3. Results and discussion:

3.1. The sensor function

The TCS230 programmable color light-to-frequency converter combines configurable silicon photodiodes and a current-to-frequency converter on single monolithic CMOS integrated circuit. TCS230 is an oscillator which produces a square-wave output whose frequency is proportional to the intensity of the chosen color.

The light-to-frequency converter reads an 8×8 array of photodiodes. 3 sets of sixteen photodiodes, having blue, green, or red filter, and one set being clear (with no filter) were employed (Fig. 2). These four types of photodiodes are interdigitated to minimize the effect of non-uniformity of incident irradiance. All 16 photodiodes of the same color in each set are connected in parallel. The type of photodiodes to be mounted in the designed photometer is pin-selectable. They were $120 \times 120 \text{ } \mu\text{m}$ in size and on $144\text{-}\mu\text{m}$ centers and their output signal is displayed as frequency on LCD.

3.2. Spectral characteristics and LED selection

The absorption spectrum of Cr(VI) - 1,5-diphenylcarbazide complex shows a peak at 540 nm. With due attention to the maximum absorption wavelength of the complex, a photodiode array with

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