

DOI: 10.19884/j.1672-5220.202412006

Development of a Micro-Spectrophotometer and Its Application in Water Quality Testing

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Abstract: Traditional spectrophotometers have a large volume and slow scanning speed, which limits their applicability for rapid on-site detection. Herein, a micro-spectrophotometer (named ATOM) is fabricated, and its performance is verified in water quality testing. An M-type Czerny-Turner light path structure, a broadband light emitting diode (LED) light source, and a linear charge-coupled device (CCD) photodetector were adopted in ATOM. The performance of ATOM was validated through iron content determination by using *o*-phenanthroline spectrophotometry. The experiment results showed that the linear correlation coefficient of determination R^2 was 0.9997 for mass concentrations ranging from 0 to 2.0 $\mu\text{g/mL}$. The relative standard deviation was 0.37%, and the relative error compared to a commercial large-scale spectrophotometer was below 1.4%. The dimensions of ATOM are 75 mm \times 60 mm \times 25 mm, with hardware costs of approximately 1 000 CNY. ATOM features compact size, low cost, rapid measurement, high integration and high precision, making it suitable for portable on-site rapid detection.

Keywords: micro-spectrophotometer; water quality testing; portability; low cost; linear CCD

CLC number: TH 744

Document code: A

Article ID: 1672-5220(2025)03-0283-09

Open Science Identity
(OSID)



0 Introduction

Spectrophotometry is well known for its high sensitivity and precision in the quantification of trace elements, as well as organic and inorganic compounds. This analytical technique is widely applied in scientific research, industrial manufacturing, and environmental assessment^[1]. Take water quality testing as an example, spectrophotometric methods can quickly identify the concentration of ammonia nitrogen, residual chlorine, heavy metals, volatile phenols and total phosphorus in water. The detection sensitivity is at the order of

milligrams per liter (mg/L)^[2]. Compared with traditional chemical analysis techniques, spectrophotometry offers distinct advantages in terms of sensitivity, precision, and operational simplicity^[3]. However, traditional spectrophotometers are typically characterized by their bulkiness and high cost, making it difficult to meet the practical needs of on-site testing^[4]. Therefore, the demand for lightweight, compact, low-cost and portable micro-spectrophotometers is becoming increasingly urgent. Scholars have conducted research in this direction.

Poh et al.^[5] utilized Arduino Nano microcontroller and three-dimensional (3D) printing technology to fabricate the micro-spectrophotometer, with a total volume of approximately 105 mm \times 90 mm \times 140 mm. The design had the merits of portability, low cost, and the ability to transmit data to smartphones via bluetooth for facilitating on-site testing and recording. Compared with commercial devices, the linear correlation coefficient of determination R^2 for protein concentration was 0.929 7, with a standard error (SE) of 0.042, which indicated that limited reproducibility still needed to be improved. Stephenson et al.^[6] employed a diode array and a microprocessor to develop a compact spectrophotometer. The size was approximately 120 mm \times 65 mm \times 60 mm and it weighed 200 g. This device had the advantages of portability and battery power supply. The measurement results of potassium permanganate concentration were similar to those of commercial equipment. However, the transmission grating used in the system had the problem of high cost and excessive stray light.

There were some breakthroughs achieved in China. Yu et al.^[7] from South China Normal University developed a wireless, cost-effective, open-source handheld spectrophotometer by utilizing 3D printing technology, with an Arduino Nano microcontroller, an AS7341 visible light sensor, and a light emitting diode (LED). The size of the device was 50 mm \times 50 mm \times 80 mm and it weighed 130 g. The device offered the advantages of portability and

Received date: 2024-12-06

Foundation items: National Natural Science Foundation of China (No. 62375048); Natural Science Foundation of Shanghai, China (No. 22ZR1402600)

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Citation: XU M Y, YU Y A, JIANG M, et al. Development of a micro-spectrophotometer and its application in water quality testing [J]. *Journal of Donghua University (English Edition)*, 2025, 42(3): 283-291.

affordability. However, the relative standard deviation (RSD) of the divalent copper concentration was 18%. Meanwhile, JIAN et al.^[8] from Sichuan Normal University developed a system employing an LED light source and an HR4000 miniature spectrometer. This system was simple to operate, easy to build, and had high accuracy, though at a relatively high cost.

In terms of commercial micro-spectrophotometers, both domestic and foreign enterprises have launched some representative products. The USB2000 spectrophotometer of Ocean Optics (USA) possessed the advantages of high spectral resolution, compact structure, and universal serial bus (USB) and recommended standard 232 (RS232) dual communication support, but the need for an external light source limited its application^[9]. The DR1900 spectrophotometer of HACH (USA) was provided with various built-in preset test methods along with powerful data storage and transmission functions. However, its hardware cost (approximately 60 000 CNY) and specialized consumables reduced the versatility of the instrument^[10]. The Unicosh 1600 spectrophotometer (China) possessed some advantages in terms of cost-effectiveness (approximately 5 000 CNY). However, its large size and heavy weight (6 kg) limited its portability^[11]. In terms of the light source system, traditional instruments commonly used deuterium tungsten lamps or xenon lamps, which were able to cover the continuous spectrum from the ultraviolet (200–400 nm) to the visible region. However, their service life was limited (about 1 000–2 000 h), and they were large in size, costly to replace, and inconvenient to maintain. According to the national environmental protection standards, the measurement wavelength of water pollutants was concentrated in the range of 400 to 665 nm, and the LED spectral range of 400 to 730 nm was able to meet the majority of water pollutants' measurement needs. It also possessed the advantages of long service life (20 000 h), small size, low cost and low-voltage direct current drive, making it an ideal alternative light source.

To address the problems above, this article proposes and describes the fabrication of a micro-spectrophotometer named ATOM. By utilizing a broadband LED and a linear charge-coupled device (CCD) within an M-type Czerny-Turner configuration, the instrument aims to achieve portability and cost-effectiveness. The subsequent sections detail the optical and hardware design, software implementation, and experimental validation of the performance of ATOM in water quality testing.

1 Methods

1.1 Principle

Spectrophotometry is an analytical technique based on the selective light absorption properties of substances,

utilized for qualitative or quantitative analysis. The absorbance is proportional to the concentration of the solution^[12]. Array spectrophotometers employ the post-dispersion spectral principle, where the light beam passes through the sample before being converted into monochromatic light by a splitting system^[13]. The typical structure of an array spectrophotometer is illustrated in Fig. 1. In this configuration, different wavelengths of light are absorbed by the sample in the cuvette simultaneously, and the transmitted light is diffracted into monochromatic beams by a grating. These monochromatic beams are then irradiated onto a linear CCD. The linear CCD is composed of a series of photosensitive pixel arrays by which spectral data are simultaneously collected across a specific wavelength range, without requiring any scanning mechanism. The linear CCD has an integration function, which can accumulate the measurement values of a single channel to enhance the signal and improve the signal-to-noise ratio.

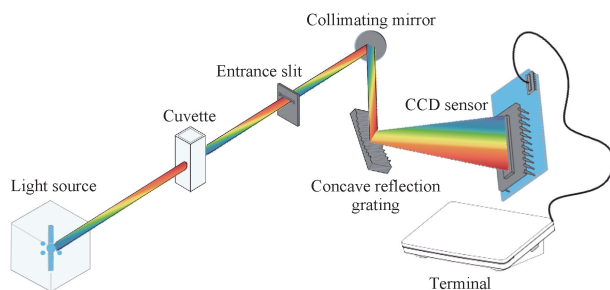


Fig. 1 Array spectrophotometer structure

1.2 Design of ATOM

The ATOM presented in this article employed post-dispersion spectrum technology and ATOM consisted of five core components: an LED light source, a sample cell, a spectroscopic system, a linear CCD and a photoelectric signal process system. The overall structural schematic is shown in Fig. 2. The spectroscopic system, linear CCD, and its driver module were integrated within a sealed aluminum alloy box with main structure dimensions. To minimize stray light interference in the monochromator's light path, the inside surface of the sealed box has undergone blackening treatment. The photograph of ATOM is shown in Fig. 3. The following sections introduce the optical system, spectrum acquisition hardware system and software in detail.

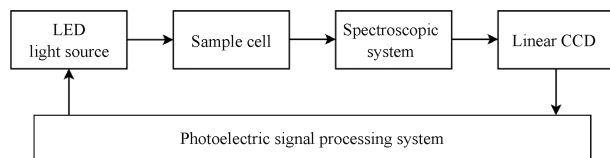


Fig. 2 Framework for the overall design of ATOM

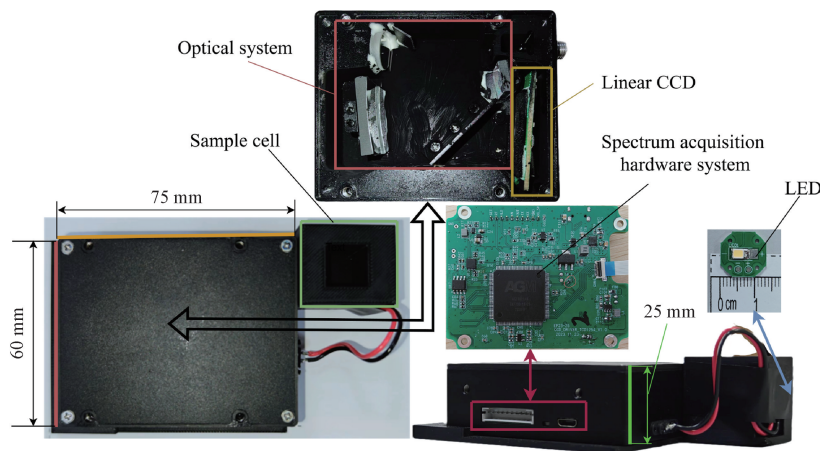


Fig. 3 Photograph of ATOM

1.2.1 Optical system

The ATOM designed in this article was based on the Czerny-Turner light path design principle. The light path structure was classified into two main types: cross-type and M-type^[14]. Although the cross-shaped structure is more compact, its resolution stability is relatively low, and the stray light is high. The M-shaped structure is simple and relatively less compact, but its optical path symmetry resolution is high and has a lower level of stray light^[15-16]. Therefore, the M-type structure was selected for this design. The concave mirrors were utilized for collimating and focusing, along with the plane diffraction grating as the dispersion element. The light path structure and its ZEMAX simulation are illustrated in Fig. 4. After

passing through the slit, the light was collimated by the collimating mirrors and directed onto the planar diffraction grating, which dispersed the light further and directed it to the focusing mirrors. The dispersed light was focused by the focusing mirrors, and directed onto the different pixels of the linear CCD with different wavelengths. In the linear CCD the optical signal was converted into an electrical signal output. Additionally, a filter was positioned in front of the linear CCD detector. It was used to filter the visible light and secondary diffracted light in the near-infrared region. Based on the design principles and performance parameter requirements for ATOM, the technical specifications of the selected optical system are determined and summarized in Table 1.

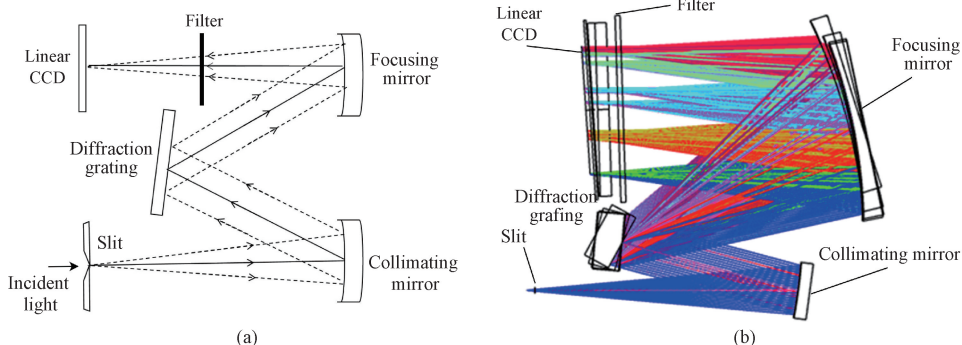


Fig. 4 ATOM light path structure: (a) schematic diagram of M-type Czerny-Turner light path structure; (b) ZEMAX light path simulation

Table 1 Optical system specifications

Component name	Parameter and value
Slit	Width: 50 μm
Collimating lens	Focal length: 50 mm
Grating	Spatial frequency: 500 lp/mm; blaze wavelength: 300 nm
Concave mirror	Focal length: 50 mm
Linear CCD	Resolution: 2 500 pixels; spectral response: 300–1 100 nm

1.2.2 Spectrum acquisition hardware system

The overall design scheme of the spectrum acquisition hardware system circuit is illustrated in Fig. 5. To achieve the design goal of miniaturization, the hardware circuit adopts separate main control and driver circuit boards connected via ribbon cables. The main control circuit board featured a double-sided layout and a four-layer printed circuit board (PCB) design, integrating a linear voltage regulator circuit, an LED driver circuit, a GD32F103RET6 (GD32) microcontroller unit (MCU), and an AG10KL144 H (AG10K) field-programmable gate array (FPGA)

chip. The MCU and FPGA communicate through the serial peripheral interface (SPI). The MCU and personal computer (PC) communicate through USB. The linear CCD sensor, signal conditioning circuit, and analog-to-digital converter(ADC) are integrated into the driver board. The dimensions of the main control circuit board are 55 mm × 70 mm, while those of the driver circuit board are 11 mm × 38 mm. The system’s analog power supply employs a direct current to direct

current (DC-DC) boost converter cascaded with a linear regulator circuit. This architecture contributes to minimizing the effect of external power fluctuations on the system’s internal power supply, as well as enhancing its resistance to noise and interference. Besides, the ADC is separately powered to minimize interference with other analog circuits. The photographs of the main control circuit board and the driver circuit board are presented in Fig. 6.

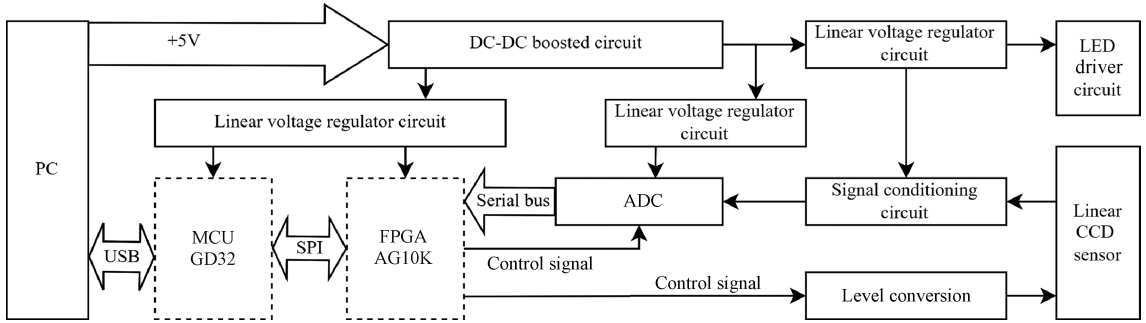


Fig. 5 Spectrum acquisition system hardware circuit design

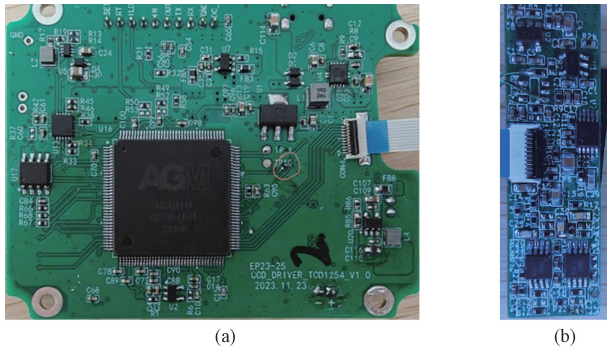


Fig. 6 Photographs of circuit boards: (a) main control circuit board; (b) driver circuit board

1. 2. 2. 1 Driver circuit of linear CCD sensor

Linear CCD sensors are widely utilized for spectral acquisition due to their high sensitivity, small pixel size and strong immunity to interference^[17]. In this article, a TCD1254GFG linear CCD sensor was employed, which integrated 2 500 effective light-sensitive pixels. Three drive pulse signals were essential for the operation of this linear CCD sensor; the shift pulse (SH) facilitated the line-by-line transfer of pixel charge; the integral control gate pulse (ICG) regulated the electronic shutter for accumulating photogenerated charge; the master clock signal governed the reading of the analog signals.

The master clock pulse was set to 2 MHz, and data was output from the output signal (OS) port of the linear CCD. The effective photoelectric OS contained both spectral signals and various noise interferences. To enhance the signal-to-noise ratio and obtain high-quality spectral signals, a signal

conditioning circuit was designed in this article. This circuit reduced noise, improved signal quality, and ensured that the CCD signal varied linearly with the measured light intensity within the dynamic range. The block diagram of the signal conditioning circuit is illustrated in Fig. 7. The output signal from the linear CCD was input into an amplifier circuit to increase the signal load level and achieve impedance matching. It was then passed through an addition and subtraction (add-subtract) arithmetic circuit and a voltage amplifier circuit to amplify the analog signal into the ADC measurable range. Finally, it was routed through a second-order low-pass filter circuit to reduce high-frequency noise, and the filtered analog signal was input into the ADC for analog-to-digital conversion.

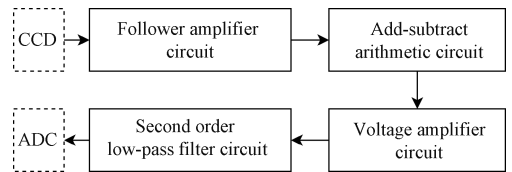


Fig. 7 Block diagram of signal conditioning circuit

1. 2. 2. 2 Driver circuit of LED

A broad-spectrum white LED with a wavelength range of 400 to 730 nm was selected as the light source for ATOM. This LED eliminated the sharp blue spikes to achieve a uniform spectral power distribution. To enable adaptive adjustment of LED brightness, ATOM was equipped with a constant current source that allowed for current adjustment through universal asynchronous

receiver/transmitter (UART) commands sent to the MCU, ensuring the stability of LED intensity. The schematic of the constant-current driver circuit is illustrated in Fig. 8. In this circuit configuration, DAVCC represents the digital-to-analog converter (DAC) output voltage; R1, R2, R3, R4 and R5 denote resistors; Q1 is a transistor; GND represents the ground, which serves as the reference point for zero electrical potential in the circuit.

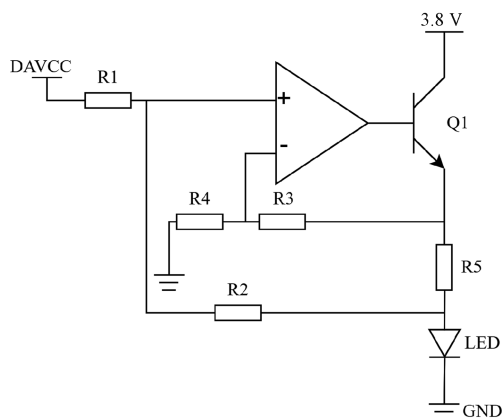


Fig. 8 LED constant-current driver circuit

The LED constant current source circuit comprised an operational amplifier, a current sampling resistor, a transistor, and its driver circuit, with the output current value being controlled by DAVCC. When the voltage across R5 fluctuated, the change was instantly transmitted to the inverting input of the operational amplifier. The difference between this signal and the voltage at the non-inverting terminal was amplified by the operational amplifier, and the output was used to regulate the base current of the transistor. The internal impedance of the transistor was altered by this process, which subsequently affected the voltage drop between its emitter and collector. Through this feedback mechanism, the stability of the voltage across R5 was maintained by the system, thereby achieving a constant output of load current.

1.2.3 Software design of spectrum acquisition

The primary task of the spectrum acquisition system was to control the linear CCD sensor based on the commands from the PC host, and measurement data was transmitted in real-time to the host PC. The linear CCD drive timing and ADC drive timing were generated internally within the FPGA. Bidirectional data transmission was required between the MCU, PC and FPGA. The GD32 MCU received and parsed control terminal instructions, and then transmitted them to the FPGA module. This process regulated the pulse output signal, thereby ensuring synchronized operation of the CCD and ADC. The software function modules of the system are shown in Fig. 9.

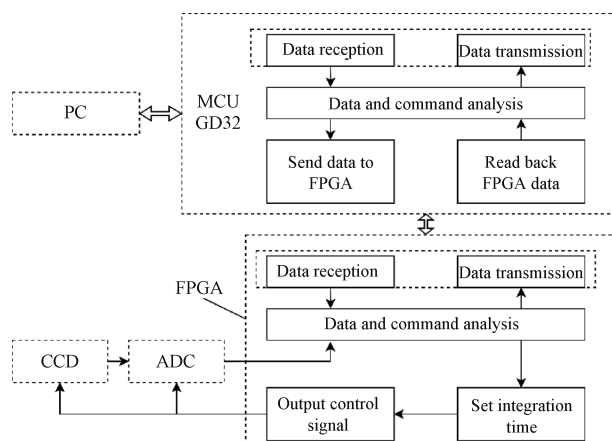


Fig. 9 Schematic diagram of software function modules

2 Experimental Section

Iron is categorized as an essential trace element and belongs to the heavy metal category. Excessive concentrations of iron ions in water can affect its color, odor and taste. Hexavalent chromium is identified as a toxic heavy metal pollutant that poses significant risks to human health. According to GB 3838—2002, environmental quality standards for surface water, the required accuracy for spectrophotometric measurement of iron is 0.3 mg/L, while that for hexavalent chromium is 0.01 mg/L. To verify the performance of ATOM, spectrophotometric analyses were conducted by using the 1, 10-phenanthroline method for divalent iron and the diphenylcarbazide method for hexavalent chromium^[18]. The results were compared with those of a traditional spectrophotometer.

2.1 Detailed performance

The detailed performance parameters of ATOM are shown in Table 2.

Table 2 Performance parameters of ATOM

Parameter	Value
Wavelength accuracy/nm	±0.7
Photometric accuracy	±0.004 (absorbance)
Wavelength resolution/nm	1.9
Stray light	< 0.2%
Signal-to-noise ratio	200:1
Wavelength range/nm	400–730
Photometric range	0–2.1 (absorbance)

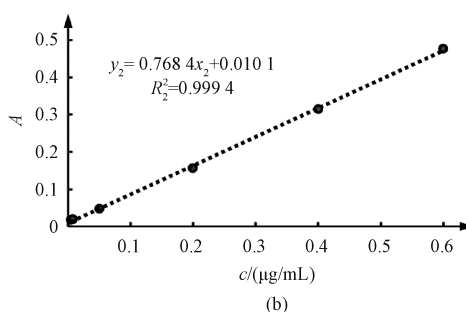
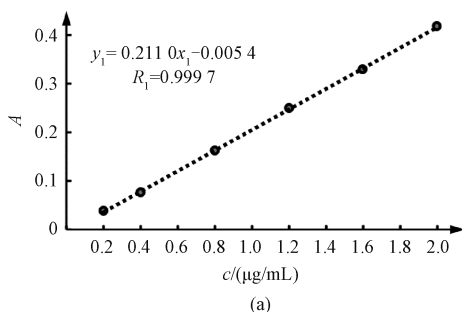
The wavelength range was measured by using a tungsten deuterium lamp, by which the lower and upper wavelength parameters of the spectral response curve were obtained. The wavelength accuracy was determined by using the characteristic spectral line of the mercury lamp, by which the peak positions of different wavelength bands were measured, and the maximum

deviation of the measured wavelengths of each peak from the standard value was taken as the wavelength accuracy of the instrument. The photometric accuracy in the visible wavelength range was verified by using the standard solution of copper sulfate. The wavelength resolution was determined from the half-height width extremes of the characteristic spectral lines of the mercury lamp. The signal-to-noise ratio was calculated by using the ratio of the mean saturated spectral value of the tungsten deuterium lamp to the standard deviation of the measurement noise, and the stray light was determined by using a cut-off filter at 360 nm. The absorbance range was calculated by using the absorbance formula based on the ADC full-scale signal and the smallest resolvable signal. The performance of ATOM was compared with that of other commercial instruments, which was presented in Section 3.

2.2 Linear relationship test

To verify the linear range of ATOM, a clean sample container was used, and deionized water was used for background correction. The experimental work utilized the following analytical-grade reagents: iron standard

solution (100 $\mu\text{g}/\text{mL}$) and *o*-phenanthroline solution (mass fraction: 0.1%) obtained from Tianjin Zhonglian Chemical Reagent Co., Ltd., China; hexavalent chromium standard solution (100 $\mu\text{g}/\text{L}$) procured from the General Research Institute for Nonferrous Metals, Beijing Co., Ltd., China; hexavalent chromium detection reagent (LH-CrA10) supplied by Zhejiang Luheng Environmental Technology Co., Ltd., China. Divalent iron complexes were analyzed with a 100 $\mu\text{g}/\text{mL}$ iron standard solution at mass concentrations of 0.2, 0.4, 0.8, 1.2, 1.6 and 2.0 $\mu\text{g}/\text{mL}$, and the absorbance of the iron solution at 510 nm was measured with the deionized water as the background, establishing the relationship between mass concentration c and absorbance A for divalent iron, as shown in Fig. 10 (a). For hexavalent chromium, standard solutions with mass concentrations of 0.005, 0.008, 0.050, 0.200, 0.400, and 0.600 $\mu\text{g}/\text{mL}$ were prepared, and the absorbance at 540 nm was measured by using the deionized water as the background to determine the relationship between mass concentration c and absorbance A for hexavalent chromium, as illustrated in Fig. 10(b).



x_1 —iron standard solution mass concentration; y_1 —iron standard solution absorbance; R_1^2 —coefficient of determination for iron standard solution; x_2 —hexavalent chromium standard solution mass concentration; y_2 —hexavalent chromium standard solution absorbance; R_2^2 —coefficient of determination for hexavalent chromium standard solution.

Fig. 10 Relationship between mass concentration and absorbance: (a) divalent iron; (b) hexavalent chromium

Figure 10 shows that R_1^2 is 0.999 7 for divalent iron solutions and R_2^2 is 0.999 4 for hexavalent chromium solutions across different concentrations. These results indicate that ATOM demonstrates good linearity and satisfies the measurement requirements.

2.3 Precision test

To verify the consistency of the results obtained from ATOM, the standard iron solutions at mass concentrations of 2.000 and 1.000 $\mu\text{g}/\text{mL}$ were selected as sample 1 and sample 2. The measurement was repeated 12 times to assess the precision and stability of the instrument. Based on the measured absorbance and combined with the absorbance-concentration working curve (Fig. 11 (a)), the concentration was inversely deduced. The mean value and RSD were calculated. The measurement results are illustrated in Table 3.

Table 3 Precision test results

Sample	Sample mass concentration/ ($\mu\text{g}/\text{mL}$)	Measured mass concentration/ ($\mu\text{g}/\text{mL}$)	RSD/%
1	2.000	2.003	0.26
2	1.000	0.997	0.37

According to Table 3, the RSDs are 0.26% for sample 1 (2.000 $\mu\text{g}/\text{mL}$) and 0.37% for sample 2 (1.000 $\mu\text{g}/\text{mL}$), indicating that ATOM exhibits high precision and good repeatability.

3 Discussion

To discuss the performance of ATOM developed in

this article, the main performance parameters, such as stray light level, photometric accuracy, wavelength

accuracy, wavelength resolution and wavelength range, were compared, as shown in Table 4.

Table 4 Comparison of parameters between ATOM and commercial spectrophotometers

Spectrophotometer	Stray light level	Photometric accuracy	Wavelength accuracy/nm	Wavelength resolution/nm	Wavelength range/nm
Ocean Optics USB2000	<0.10%	—	±0.2	0.3–10.0 (depends on the slit)	Depends on the external light source
HACH DR1900	<0.5%	±0.003 (absorbance)	±2.0	1.0	340–800
Unicosh 1600	<0.3%	±0.5% (transmittance)	±2.0	1.0	325–1 000
ATOM	<0.2%	±0.004 (absorbance)	±0.7	1.9	400–730
Shimadzu UV3600	<0.000 05%	±0.002 (absorbance)	±0.2	0.1	185–3 300

In Table 4, the first three rows present the data from micro-spectrophotometers, and the last row presents the data from Shimadzu UV3600, a commercial laboratory spectrophotometer widely used for high-precision spectral analysis. As shown in Table 4, Shimadzu UV3600 possesses significant advantages over micro-spectrophotometers in terms of stray light level, photometric accuracy, wavelength accuracy, wavelength resolution and wavelength range. ATOM was compared with Shimadzu UV3600 through a performance experiment to verify the accuracy of its measurement results.

The experimental method for performance comparison involved configuring iron standard solutions at mass concentrations of 0.3, 1.0 and 1.4 $\mu\text{g}/\text{mL}$ as Sample 1, Sample 2 and Sample 3, followed by the analysis of treated iron complex solutions from each group. ATOM and Shimadzu UV3600 were used to measure these samples separately, to directly compare the performance of the two instruments. The comparative measurement results are shown in Table 5.

According to Table 5, the relative error in measured values between ATOM and the commercial spectrophoto-

meter is below 1.4%, indicating that the measurement results of the two instruments are relatively consistent. However, Shimadzu UV3600 is 1 020 mm \times 660 mm \times 275 mm in size, 96 kg in weight, and needs wavelength scanning^[19], which limits its field application and portability. In contrast, ATOM is compact in size, highly integrated, and does not require time-consuming wavelength scanning, completing spectral data acquisition in only 7 ms. The cost, volume, weight and communication interface of ATOM were compared with those of typical commercial micro-spectrophotometers. The comparison results are shown in Table 6.

Table 5 ATOM and Shimadzu UV3600 spectrophotometer test results

Sample	ATOM measured value of $c/(\mu\text{g}/\text{mL})$	Shimadzu UV3600 measured value of $c/(\mu\text{g}/\text{mL})$	Relative error /%
1	0.313	0.315	0.63
2	1.013	1.000	1.31
3	1.373	1.372	0.07

Table 6 Comparison of parameters between ATOM and typical commercial micro-spectrophotometers

Model	Cost/CNY	Volume/ mm^3	Weight/g	Communication interface
Ocean Optics USB2000	10 000–30 000	89.1 \times 63.3 \times 34.4	190	USB/RS232
HACH DR1900	58 527	178 \times 267 \times 98	1 500	USB
Unicosh 1600	5 000	360 \times 280 \times 180	6 000	RS232
ATOM	1 000	75 \times 60 \times 25	140	USB/RS232/UART

The comparison results demonstrate that ATOM possesses advantages in portability, cost efficiency and interface expandability, compared with commercial micro-spectrophotometers.

4 Conclusions

A micro-spectrophotometer based on the Czerny-Turner design was designed in this article. A broad-spectrum LED was used as the light source, and a linear CCD was used as the photodetector. The linear CCD

photoelectric signal processing system was realized by using FPGA, GD32 MCU and SPI bus. To verify the system's performance, the 1, 10-phenanthroline spectrophotometric method was used to determine the iron mass concentration, and the key performance parameters of ATOM were experimentally validated. The experimental results indicated that a linear correlation coefficient of determination of 0.9997 was achieved for the working curve by using ATOM within the divalent iron mass concentration range of 0–2.0 $\mu\text{g}/\text{mL}$, while the RSD was 0.37%.

ATOM employs LEDs as its light source and adopts an array spectrophotometer optical path structure. It does not require wavelength scanning, with dimensions of 75 mm × 60 mm × 25 mm, and the hardware cost is approximately 1 000 CNY. ATOM possesses the advantages of small size, low cost, fast measurement, high integration and high precision. These characteristics align with current portable field detection requirements for water environment applications and enable its application in field rapid detection. The developed ATOM system is currently optimized for the visible spectrum. The wavelength range is expected to expand to ultraviolet and infrared regions in the near future.

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微型分光光度计的研制及其在水质检测中的应用

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摘要:传统的分光光度计体积大、扫描速度慢,不适合现场快速检测。该文研制了一台微型分光光度计(命名为 ATOM),并在水质检测中验证了其性能。ATOM 采用 M 型 Czerny-Turner 光路结构,宽光谱发光二极管作为光源,线阵电荷耦合器件(charge-coupled device, CCD)作为光电探测器。采用邻二氮菲分光光度法测定铁含量,对 ATOM 的性能进行了验证。试验结果表明,ATOM 在 0~2.0 $\mu\text{g}/\text{mL}$ 质量浓度范围内线性相关判定系数 R^2 达 0.999 7,相对标准偏差为 0.37%,与商用大型分光光度计测量结果的相对误差小于 1.4%。ATOM 的主体部分尺寸为 75 mm \times 60 mm \times 25 mm,硬件成本约为 1 000 元人民币,具有体积小、成本低、测量快速、集成度高、测量精度高等优点,可作为便携式分光光度计应用于现场快速检测。

关键词:微型分光光度计;水质检测;便携式;低成本;线阵 CCD