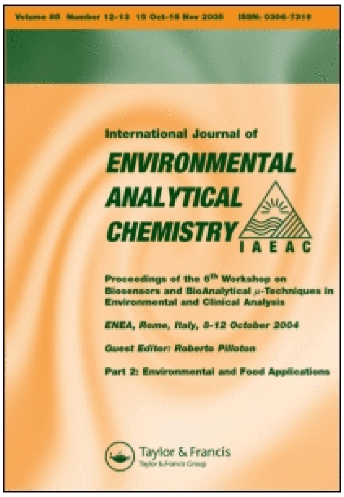

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PORTABLE SIMPLIFIED PHOTOMETER FOR IN SITU ENVIRONMENTAL WATER ANALYSIS

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The portable simplified photometer, in which high-luminous light emitting diode (LED) was used as a lamp, was manufactured as an useful tool for *in situ* environmental water analysis. This photometer is applicable to various spectrophotometric methods for the determination of chemical species exhibiting an absorption λ_{max} of the colored complex spectrum around 660 nm and/or 560 nm.

KEY WORDS: *In situ* water analysis, portable simplified photometer, light-emitting diode, photodiode. spectrophotometric method.

INTRODUCTION

The authors started to develop seven years ago simplified visual methods for the determination of chemical species in natural waters, in order to enlarge the population surveying the environmental quality of natural waters. Consequently, visual methods for the determination of F^-, PO_4^{3-}, NO_3^-, NO_2^-, NH_4^+, Fe^{2+} and $H_2S(HS^-)$ have been developed[1–3] and have already been introduced into practical use.[4] These visual methods include standard series methods using test tubes, so that they do not require complicate sample handling nor further instrumental analysis. They were all developed after establishing the spectrophotometric method which is to be used as the reference method, based on the red or blue coloration (500–650 nm) so as to visually judge the difference easily. However, there still remained room for improvement in precision.

Although a commercially available portable spectrophotometer was thought to be useful to increase precision and was used on the spot, it was found to be not a satisfactory tool for *in situ* environmental water analysis due to the following limitations: (1) The response was slow, (2) it was not so inexpensive, and (3) the kinds of such portable spectrophotometers are in short supply nowadays.

Therefore, the authors have assembled a simplified photometer equipped with high luminous light-emitting diode (LED) as a lamp and high-speed response positive-intrinsic-negative (PIN) type photodiode as a photodetector. Photometric modules of this kind have already been developed.[6–8] However, they are mostly used as a detector in flow injection analysis[9] and application to a portable simplified photometer has not, to the best of our knowledge, been reported. The relative sensitivity of this photometer with respect to that of commercially available spectrophotometers, which varied with the degree of overlapping between the emission spectrum of LED and absorption spectrum of the colored complex, was examined for the various spectrophotometric methods.

EXPERIMENTAL

Materials

The essential components of the assembled photometer are shown in Figure 1, and the electric circuit diagram of the amplifier and the constant voltage power supply system is shown in Figure 2. The digital voltage meter is not always necessary since a circuit tester can be used as a display device. Two kinds of LED, TLRA 180AP (Toshiba, λ_{max} 660 nm, 3000 mcd) and SLH 56MC (Rohm, λ_{max} 563 nm, 250 mcd) have been used as a lamp. A PIN photodiode, of which its spectral sensitivity is good in the infrared region, has been employed as photodetector in this study. Because the dark current of PIN photodiode is low and stable, it is not necessary to take into account the contamination of stray light so much.

Apparatus

The emission spectrum of LED was taken on a Hitachi 508A atomic absorption spectrophotometer. The spectrophotometric determination was carried out in a Shimazu UV-140.

RESULTS AND DISCUSSION

Selection of light source

It is difficult to manufacture a simplified portable photometer based on the commercially available ones, since they have miniaturized and complicated optical systems for laboratory use, that is, they obtain the sharp monochromatic light from a white light lamp by employing a monochromator, lens, and a collecting mirror. It is also necessary to take into account the radiation of heat and consumption of electric power in using the white light lamp as a photometer light source. The same stands for the filter photometer.

On the other hand, LED has excellent characteristics as a lamp. In fact: (1) Monochromator or monochromatic filter is not necessary since the peak width of emission spectrum is narrow, (2) the directional pattern of light is sharp in LED so

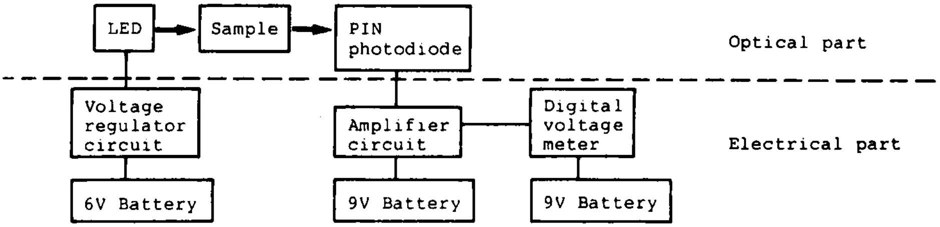

Figure 1 Block diagram showing the components of the simplified photometer.

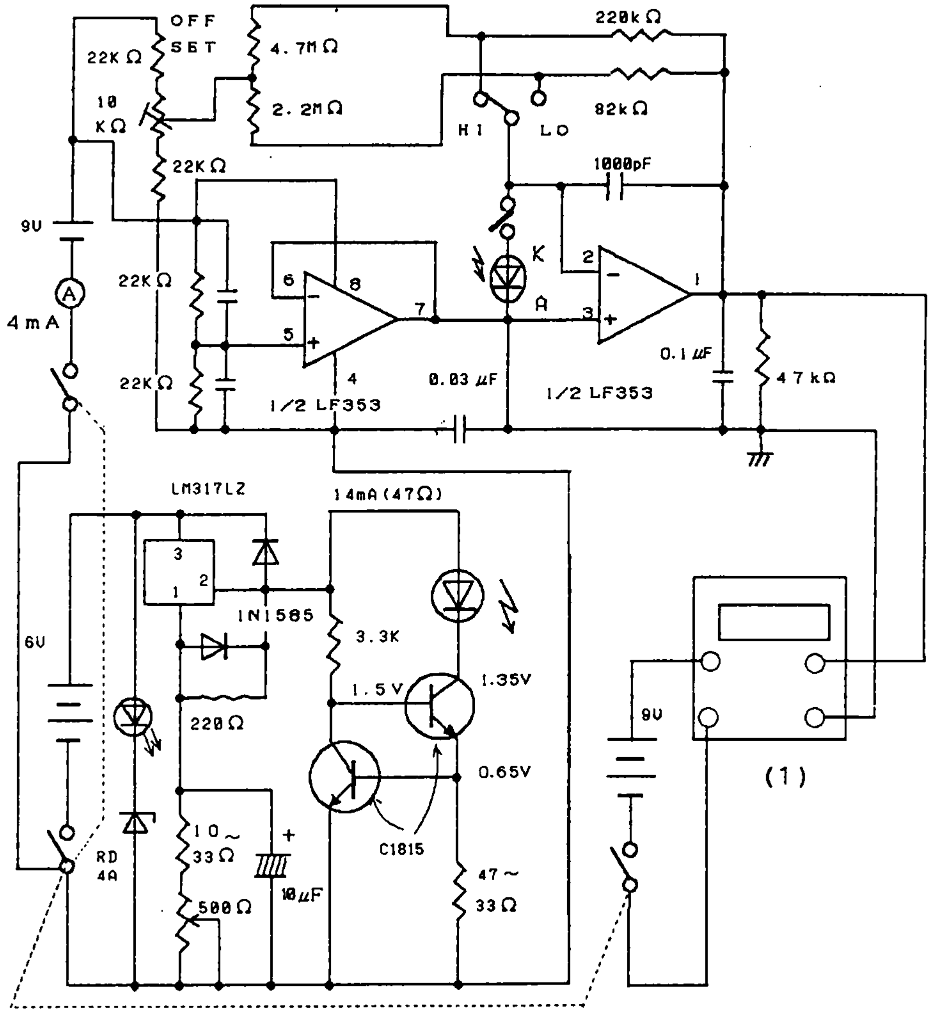

Figure 2 Electric circuit diagram of the amplifier and constant voltage power supply system. (1) Digital voltmeter.

that collecting mirror and lens are not necessary, and (3) the electric power consumption is lower compared with the white light lamp. However, a portable photometer equipped with LED has not been reported. This could be attributed to the fact that the variety of LED is not enough to cover all visible region. Moreover, only two kinds of LED are enough high-luminous among the commercially available LEDs. Although this is a weak point of LED for a general use in the laboratory, it is not so a big problem in the environmental water analysis. As can be seen in Table 1, most of spectrophotometric methods for the determination of chemical species of interest in water quality, use the limited wavelengths around 650 nm and 550 nm.

Analytical performance

In the present photometer, the absorbance (A) is calculated from the following equation, assuming that the Lambert–Beer's law holds,

$$A = -\log(mV/mV_0) = abc$$

where mV_0 and mV refer to the response of the loading water and sample in a cell, respectively.

The sensitivities obtained for NH_4^+—N, NO_2^-—N, PO_4^{3-}—P, Fe^{2+}—Fe, and F^- were compared with those obtained by a commercial spectrophotometer (Table 1). In the thymol method for NH_4—N (Figure 3-a) and the malachitegreen-molybdate

Table 1 Comparison of the relative sensitivity of the simplified photometer with that of a commercial spectrophotometer

			Absorbance per 1 μg[a,b]		
Chemical *species*	*Spectrophotometric method*	λ_{max}	*Spectrophotometer*	*Simplified photometer*	*Relative sensitivity*[c]
NH_4^+—N	Thymol photometry Conventional method	660 nm	0.0166 (0.999_9)	0.0152 (0.999_9)	0.91
	Improved method[f]	690 nm	0.0214 (0.999_9)	0.0163 (0.999_9)	0.76
NO_2^-—P	Photometry using GR reagent	530 nm	0.137 (0.999_9)	0.077 (0.998)	0.56
PO_4^{3-}—P	Malachitegreen-molybdate photometry	615 nm[d]	0.141 (0.999_8)	0.130 (0.999_6)	0.92
Fe^{2+}—Fe	1,10-Phenanthroline photometry	508 nm	0.0201 (0.999_7)	0.0040 (0.995)	0.20
F^-	Alfusone photometry	555 nm[e]	0.0147 (0.999_7)	0.0047 (0.998)	0.32

[a] n = 5.

[b] Values in parentheses correspond to the correlation coefficient of the calibration curves.

[c] Ratio of sensitivities.

[d] Absorbance is determined at 650 nm since the reagent blank exhibits relative absorption at λ_{max}.

[e] Absorbance is determined at 620 nm since the reagent blank exhibits relative absorption at λ_{max}.

[f] The procedure is reported in the previous paper[6].

method for PO_4^{3-}—P, in which the absorption λ_{max} of the spectrum of the colored complex is in fair agreement with that of the emission spectrum of LED, the relative sensitivity was close to 1. On the other hand, in the case of the GR reagent method for NO_2^-—N (Figure 3-b) and the 1,10-phenanthroline method for Fe^{2+}—Fe, in which the corresponding spectra do not overlap so much, the sensitivity obtained by the present photometer was smaller. It can be seen from Table 1 that the linearity of the calibration curve slightly decreased as long as the discrepancy in λ_{max} of the two spectra increased. However, the correlation coefficients were all better than 0.995 and it can be said that this photometer fairly improves the precision in the visual determination method.

Finally, it is difficult to apply the spectrophotometric method based on the bathochromic shift such as alfusone method for F^-—F (Figure 3-c) to the visual method. However, Table 1 clearly demonstrates that this photometer can be used for such spectrophometric methods as long as the two spectra overlap each other to a certain extent.

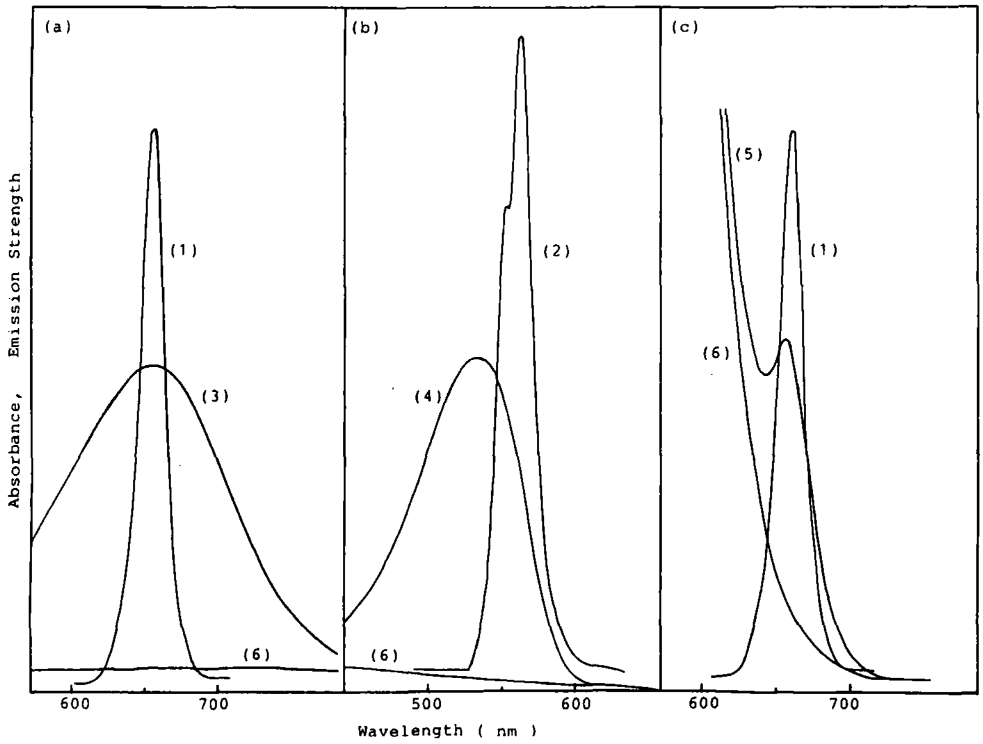

Figure 3 Comparison of the emission spectrum of LED with the adsorption spectrum of colored complex. (a), Thymol method for the determination of NH_4^+—N; (b), GR reagent method for the determination of NO_2^-—N; (c) alfusone method for the determination of F^-; (1) emission spectrum of TLRA 180 AP; (2) emission spectrum of SLH 56MC; (3), indothymolblue; (4) NO_2^-—GR reagent complex; (5) La-ALC-F complex; and (6) reagent blank.

CONCLUSION

In conclusion, whether this photometer is applicable to a spectrophotometric method or not depends on the extent of overlapping between the emission spectrum of LED and absorption spectrum of the colored complex. In this respect, the discrepancy in $\lambda_{\max}$ of the two spectra should be less than 50 nm. The dynamic range of the calibration curve and reproducibility of the present photometer were almost the same to that of a commercial spectrophotometer in all examined methods.

The manufacturing of this photometer was easy as the electronic parts used are all common stuff and can be accomplished within a few days. The total cost was less than 75 US dollars.

This photometer can be used not only for *in situ* environmental water analysis but also for educational and demonstration purposes, e.g., in high schools, where expensive spectrophotometers are not available.

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