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Absorbance measurements with light-emitting diodes as sources: Silicon photodiodes or light-emitting diodes as detectors?

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ABSTRACT

Light-emitting diodes may also serve as light detectors, and the combination of two of these devices, one serving as light source, the other for detection, has been reported repeatedly for use in analytical photometry. A comparative study of the performance of light-emitting diodes in this role and that of a standard photodiode is reported herein. The spectral sensitivities of the light-emitting diodes were found to be as narrow as their emission bands, but shifted to shorter wavelengths, so that the spectral overlaps between emission and sensitivity of the same devices are very limited. The photocurrents of the light-emitting diodes were found to be about ten times lower than those of the photodiode. In the discharge mode (the time for discharge of the p/n-junction by the photocurrent is measured) as well as the photovoltaic mode, both of which had previously been reported for light-emitting diodes used as detectors in photometric devices, the performance of a light-emitting diode was on a level that is adequate for many analytical purposes, but the photodiode generally gave better precision and the signals showed faster settling times.

1. Introduction

Light-emitting diodes (LEDs) from the near infrared to the UV-range are often employed as radiation sources for photometric detection in analytical chemistry. They are good alternatives to conventional incandescent sources or discharge lamps for optical measurement systems due to their advantages of small size, long lifetime, high stability, low heat production, low power consumption and low cost. As most LEDs emit only over a narrow wavelength range, monochromators are not needed, which allows the construction of very simple devices (see for example [1]). On the other hand, a change in wavelength requires an exchange of the light source, and it is not possible to acquire spectra of samples. A photometric measurement with an LED was first described by Flaschka in 1973 [2]. Since then a multitude of devices have been described in the literature. These include instruments for absorbance measurements in cuvettes (see for example [3–6]), detection in flow-injection analysis [7–9], on-line detection in process analysis [10,11], as well as detection in HPLC [12–14] and capillary electrophoresis [15–19]. Several reviews have appeared [1,20–23]. Commercial products are also available, in particular in the form of portable instruments for carrying out photometric tests in the field.

Photometric measurements are governed by the well known Lambert–Beer law, which relates absorbance, A, to concentration, c (ε is the molar absorptivity coefficient, and b the optical path-length). The absorbance is obtained from the light intensity before (I_0) and after passage (I) through the measuring cell. These parameters are usually determined with detectors which give current outputs that are proportional to light intensity, hence A can also be expressed as a function of the detector currents (i_0 and i):

$$A = \varepsilon bc = \log \frac{I_0}{I} = \log \frac{i_0}{i} \quad (1)$$

The mathematical transformation needed to obtain the value of A from the measured parameters is usually carried out by the instrument. As this is a complication, simple devices often give an output value which is proportional to transmittance, T, which is given by the simpler relationship

$$T = \frac{I}{I_0} = \frac{i}{i_0} \quad (2)$$

However, T is not proportional to concentration, and non-linear calibration curves are obtained for this parameter. For devices based on LEDs it is usually also not necessary to obtain the reference parameter (I_0 or i_0) as this stays fairly constant due to the inherent stability of these light sources. It is thus possible to work with a single detector for light intensity. The measured value may then be an arbitrary parameter (such as a voltage derived from the detector current by an electronic circuitry) and the exact relationship of this signal with the concentration is established by calibration.

Commonly LEDs are paired with silicon photodiodes (PD) as detectors. These devices are as easy to use as the LEDs, have good sensitivity and are frequently employed in modern commercial photometric instruments. Only when extremely low light levels need to be detected the more complex and expensive photomultiplier tubes

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are still required. Photodiodes are readily available in different forms from suppliers of electronic components.

It has also been known that LEDs can function as photodiodes as well [24]. In similarity to their emission performance, their spectral detection sensitivity is restricted to a narrow band. This feature has been made use of for applications in which the wavelength selectivity of detectors is a requirement, *i.e.* in a sun spectrometer reported in 1992 by Forrest M. Mims [24] and in remote sensing in agronomy (spectral reflectance to study vegetation coverage) [25]. In these instruments, the LEDs are inexpensive substitutes for photodiodes fitted with interference filters to restrict their wavelength sensitivity range. The latter are expensive due to the inclusion of the costly filters. Some fundamental studies concerning the spectral and dynamic behavior of a blue and of a red LED used as detectors were carried out by Miyazaki et al., (1998) [26].

The possibility of the pairing of two LEDs, one of which acts as detector, was mentioned in 1993 by Dasgupta et al., but was discouraged due to the low photocurrents found for the LEDs then available [1]. In the more recent years however, several research groups have reported the successful use of LED pairs for photometric devices for analytical chemistry [27–38]. The term PEDD (for Paired Emitter-Detector Diodes) has frequently been employed for this arrangement. Nevertheless, in these arrangements the wavelength selectivity of the LED used as detector was not required as this is already determined by the LED used as a source. A rationale for the substitution of silicon photodiodes in these devices has not often been given, but cost saving has been stated and it has been suggested that advantageous measuring modes require the use of LEDs.

This report examines the use of LEDs as an alternative for conventional photodiodes in photometric devices based on LEDs as emitters.

2. Experimental

2.1. Instrumentation

The LEDs (5 mm diameter plastic package, water clear) of different colors were obtained from Everlight (Shulin, New Taipei City, Taiwan) (R1 = Part No. 3832SURC/S530A3), (R2 = Part No. 3832SURC/S400A6), (R3 = Part No. 3832SURC/S530A3), (Y1 = Part No. 3832UYC/H2/S400), (G1 = Part No. 383SYGC/S530E2/H2), Kingbright (Chungho, New Taipei City, Taiwan) (RO1 = Part No. L7113SEC/H) and Avago (San Jose, CA, USA) (R4 = Part No. HLMP3750). The silicon photodiodes (SFH 203P) were products of Osram (Regensburg, Germany). The quartz glass cuvettes with an optical pathlength of 1 cm were obtained from Hellma (Model 1001040, Type 100QS, Müllheim, Germany). The spectrometer used to determine the emission spectra of the LEDs (Model S2000, with a spectral bandwidth of 3 nm) was obtained from Ocean Optics (Dunedin, FA, USA). The sensitivity data of the LEDs were acquired by placing them at the exit slit of the monochromator of a laboratory spectrophotometer fitted with a tungsten lamp (Model CE 303 from Cecil Instruments, Cambridge, England, spectral bandwidth = 10 nm). The beam splitter was sourced from Qioptiq Photonics (Part No. G344312000, Munich, Germany). Purpose made devices were employed for positioning and aligning of light sources, beam splitter, cuvettes and detectors according to different experiments and to exclude ambient light. The different measurement configurations employed are shown schematically in Fig. 1. The operational amplifier (Model OPA121) and the logarithmic ratio amplifier (Model LOG102) were products of Texas Instruments (Austin, TX, USA). The microcontroller used was an ATmega328 (Atmel, San Jose, CA, USA) on an Arduino Uno board (RS Components, Wädenswil, Switzerland). A standard multimeter obtained from Fluke (Model 75, Everett, WA, USA) was used to measure voltage signals. The e-corder data-acquisition system (Model ED401) and the Chart software package employed to measure the noise levels of the signals from the photodiodes and LED-detectors were obtained from EDAQ (Denistone East, New South Wales, Australia). The noise values were determined as the maximum deviations over a period of 30 s, no electronic filtering was applied. The light intensities of the LEDs used as light sources were controlled with a constant current supply built with a linear regulator from National Semiconductor (Model LM317, Santa Clara, CA, USA). The currents were set to 25 mA, except for the measurements concerning the effects of the light intensity. For these the currents were adjusted to different values between approximately 2.5 mA and 25 mA and the relative intensities were determined with the photodiode used in the photocurrent configuration. As can be seen in Fig. 2 for one of the LEDs the dependence of the intensities on current was found to be close to linear.

2.2. Reagents

Thymol Blue and sodium hydroxide of analytical grade were products of Siegfried (Zofingen, Switzerland) and Fluka (Buchs, Switzerland) respectively. Deionized water was used for all experiments and was obtained from a NANO-Pure purification system (Barnstead, IA, USA).

3. Results and discussion

3.1. Spectral considerations

It has been known that the spectral sensitivity of LEDs when used as photodiodes is restricted to a relatively narrow range but only limited quantitative information has been available. Miyazaki et al. measured the sensitivity spectra of a red and a blue LED and compared these with their emission spectra [26]. It was found that in both cases the detection sensitivity was shifted to shorter wavelengths. The emission and sensitivity spectra for 5 LEDs from green to red are shown in Fig. 3A and B respectively. The sensitivity measurements were carried out in the photocurrent (or photoconductive) mode, *i.e.* the currents produced by the photodiode were measured. The current follower circuit arrangement employed is shown in Fig. 1A. As can be clearly seen, the sensitivity spectra are shifted significantly to shorter wavelengths. For the selection of LEDs

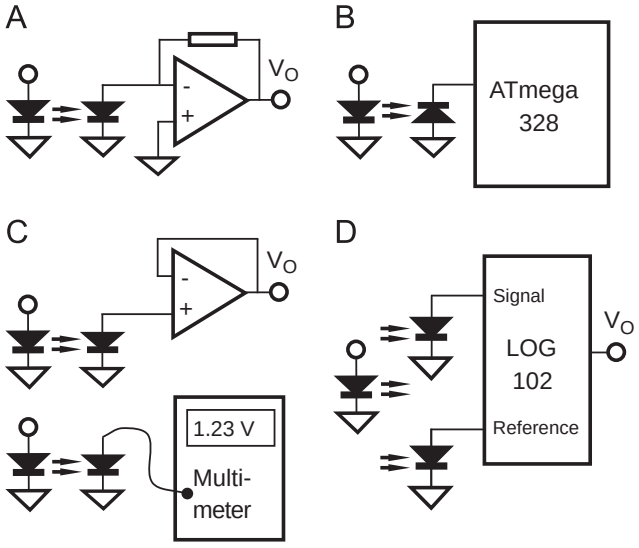

Fig. 1. Measuring configurations used. (A) Photocurrent mode with operational amplifier in the current follower configuration, (B) the capacitance discharge method with microcontroller, (C) photovoltaic mode with two options: high input impedance operational amplifier in the voltage follower configuration or direct connection to a multimeter, and (D) photocurrent mode with log ratio amplifier configuration.

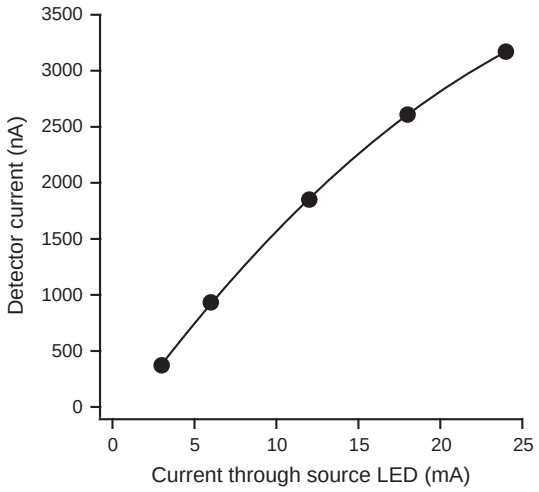

Fig. 2. Photocurrent of the photodiode (SFH203P) in dependence on the forward current of the yellow LED (Y1). Measured with the arrangement of Fig. 1A.

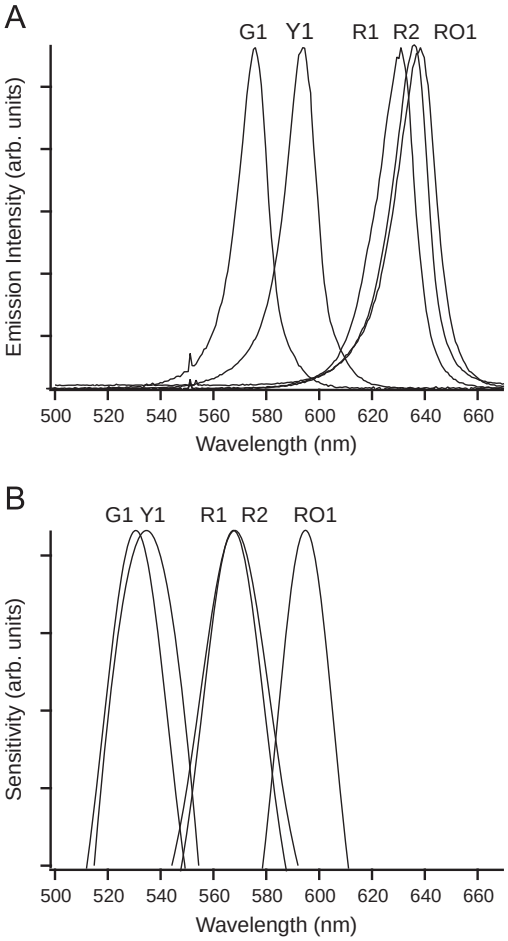

Fig. 3. Emission spectra of the LEDs (A), and sensitivity spectra of the same LEDs used as detector (B).

this shift was between about 40 and 60 nm. The extent of the shift thus varies, for instance the green (G1) and yellow (Y1) LEDs have almost identical sensitivity ranges despite their different emitted colors. Note that while the maxima of the peaks can be expected to be representative, the shapes of the bands of the sensitivity spectra of Fig. 3B should only be taken as a rough indication as they were affected by the relatively wide spectral bandwidth (10 nm) of the instrument available for these measurements. It was not possible to acquire any sensitivity spectra for blue LEDs, possibly because of the low intensity of this same system at shorter wavelengths, and these were therefore not further considered in this study. The results demonstrate that when the same type of LED is paired, the spectral overlap between emission and sensitivity is very limited.

3.2. Current yields for the photodiode and the LED-detectors

A comparison of the sensitivity of the LEDs when used as detectors with that of a standard photodiode was carried out by setting the monochromator of the light source to the wavelength of maximum sensitivity for each LED as determined in the previous section. The LEDs and the photodiode were individually mounted on a positioning stage in front of the exit slit and their geometric positions were optimized for the highest signal. The photocurrents generated by the detectors were again measured with the circuitry of Fig. 1A. As can be seen from Table 1, currents between about 0.5 and 4.5 nA were obtained. These levels are relatively low, but well within the capabilities of inexpensive modern electronic circuitry. The currents obtained for the photodiode, as expected, were higher than for the LEDs, but with a difference by a factor of about 5–40 times for the values between the two devices, the discrepancy was not as large as had been suspected. Note that the photodiode is a standard silicon type with a sensitive area of 1 mm^2, and is representative for similar models readily available from a range of suppliers. The comparison of the results for the 4 different red LEDs (R1–R4) indicates that there is no relationship between their sensitivity and brightness.

A further test was carried out by matching one of the LEDs as an emitter (Y1) with 3 different detector options: the photodiode, a second LED of the same type, and a different LED (RO1) which has a good match of its sensitivity spectrum to the emission spectrum of the emitter as can be seen from Fig. 3. This experiment was carried out by mounting the two devices on opposite sides of a holder for a standard cuvette of 1 cm pathlength. The positions of the emitter and detectors were again adjusted in each case to obtain the highest output signals which were measured in the photocurrent mode. The measurements were also carried out for different light intensities which were set by adjusting the currents through the LED serving as light source to 2.5 mA, 10 mA and 25 mA. The results are given in Table 2. The currents of the photodiode were again higher by about a factor of 10 compared to the LED with a good spectral match with the emitter. This is in agreement with the results reported in Table 1. The use of the twinned LEDs (same LED types as emitter and detector) led to currents which were about 3–5 times lower than those of the best match LED. The reason for this difference must be the mismatch between the responsitivity spectrum (λ_{max}=534 nm) and the emission spectrum (λ_{max}=595 nm) of the yellow LED (Y1) as

Table 1
Current yields of the LED-detectors and the photodiode placed at the exit slit of the monochromator set to the wavelengths of maximum sensitivity for each LED.

LED	Luminous intensity (mcd)	Peak emission wavelength, λ_{max} (nm)	Wavelength of maximum sensitivity (nm)	Current yield of LEDs (nA)	Current yield of photodiode SFH 203P (nA)
R1	2500	630	568	0.5	20.6
R2	6300	635	570	4.5	20.6
R3	800	639	596	1.8	23.6
R4	125	638	610	1.4	23.5
RO1	10,000	637	594	3.6	22.4
Y1	1000	595	534	2.3	13.4
G1	320	578	530	0.7	13.4

Table 2
Current yields and noise levels of two detector LEDs (twinned, Y1, and best spectral match, RO1) and the photodiode (SFH 203P) at different currents supplying the source LED (Y1).

Detector	25 mA			10 mA			2.5 mA		
	Current (nA)	Noise (nA)	S/N	Current (nA)	Noise (nA)	S/N	Current (nA)	Noise (nA)	S/N
Y1	93.6	3.8	24	57.2	2.6	22	11.5	2.0	5.8
RO1	474	2.2	215	223	1.5	149	38.9	1.3	30
SFH 203P	5564	0.8	6950	2516	0.35	7190	441	0.07	6300

illustrated in Fig. 3. The data of this figure leads to the expectation of an even lower current yield for the pairing of two of the yellow LEDs, but note again that the sensitivity spectra shown in Fig. 3 cannot be taken as fully quantitatively representative due to the limitations of the experimental system. Also given in Table 2 are the noise levels associated with these measurements. As can be seen, the absolute noise levels are lowest for the photodiode and highest for the yellow LED with the poor spectral match. Thus for the photodiode and the lowest light intensity (2.5 mA passed through the source LED) the signal-to-noise ratio is still excellent (>6000) while for the yellow LED as detector this drops to a low 5.8. Note that no electronic noise filtering was applied for these measurements.

3.3. Light intensity measurement by the discharge method

PEDD devices have been employed by measuring the discharge time for the incidental junction capacitance of an LED [36,38]. The capacitance is discharged by the photocurrent and thus the rate of discharge is faster for higher levels of irradiation. A plot of the logarithm of the discharge time was reported to be approximately linear with concentration in absorption measurements [36], presumably because the discharge of a capacitor follows a log-function. The method can be implemented with a simple microcontroller without requiring a separate analog-to-digital convertor or analog electronic circuitry. In this work, this mode was evaluated again for the favorable combination of the yellow LED (Y1) as emitter with the red–orange LED (RO1) as detector, as well as the photodiode as detector, again by mounting them on the cuvette holder at a distance of approximately 1 cm. The circuitry is illustrated in Fig. 1B. The reverse biased LED is first charged through the port of the microcontroller which is set to the output mode and turned to a logic HI (5 V). Then the port is switched to the input mode and the time taken for the voltage across the diode to decay to a logic LO level is determined with an internal counter. The discharge times in μs obtained for the two combinations in dependence of the intensity of the emitting LED are plotted in Fig. 4. Note the different scales for the two devices. The data indicates that both components, the LED as detector as well as the photodiode, may be employed in this mode, but the discharge times are shorter for the photodiode by a factor of about 10. This may be due to the higher current yields for the photodiode, as demonstrated above, but the junction capacitances are not known and can be expected to be different as well. The shapes of the response curves are non-linear with light intensity and different for the two devices. The readings differ by a factor of about 5 between the lowest and highest intensities for the LED while for the photodiode this spans a factor of about 10. A weak point was found to be the reproducibility of the measurements. The resolution of the system is 4 μs, which introduces digitization errors, especially for the shorter measured times, but the fluctuations are generally more pronounced. The relative standard deviations obtained from 5 readings were determined to be between about 2% and 6%. As the measurement is fast, an improvement can be obtained by averaging repeated readings to improve the precision. The standard deviations obtained for averaged readings (1000 individual measurements, which required about 10 s to acquire) for some of the measurements are given in Table 3. The values are generally better, very good for the low light intensities for the LED used as detector, but still around 3–4% for the high intensities. For the photodiode the pattern is more consistent with all values being around 1%. The data demonstrates that photodiodes are at least as suitable for this measurement approach as the LEDs.

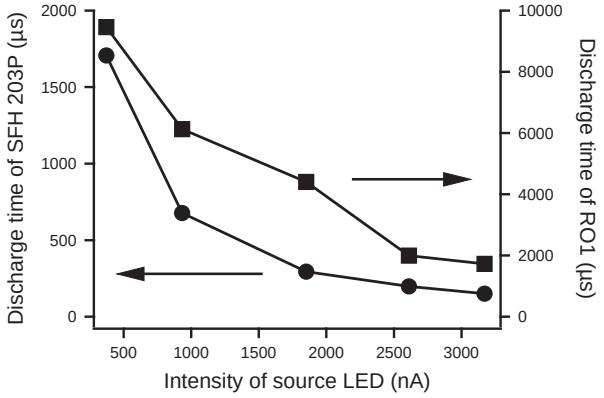

Fig. 4. Light intensity measurements using the junction discharge method (see Fig. 1B) for the red–orange LED (RO1) and the photodiode (SFH203 P) for different intensities of the source LED (Y1). These intensities refer to the photocurrents determined with the photodiode using the arrangement of Fig. 1A.

Table 3
Discharge times (mean of 1000 readings) and standard deviations for these averaged readings ($n=5$) obtained with the detector LED of best spectral match (RO1) and the photodiode (SFH 203 P) at different currents supplying the source LED (Y1).

Detector	24 mA		12 mA		3 mA	
	Mean discharge time (μs)	RSD (%)	Mean discharge time (μs)	RSD (%)	Mean discharge time (μs)	RSD (%)
RO1	1724.6	3.3	4401.4	2.9	9460.6	0.07
SFH 203P	149.8	0.99	294.4	0.57	1707.0	0.56

3.4. Light intensity measurement in the photovoltaic mode

Tymecki and coworkers suggested the use of LEDs as detectors in the photovoltaic mode [32,30]. In this approach the voltage developed across the diode on irradiation, without load, is measured. For a photodiode a logarithmic voltage (V) response to photocurrent (i), and hence light intensity, is expected [39]

$$V = \frac{kT}{e}\ln\left(\frac{i}{i_0}\right) \quad (3)$$

k is the Boltzman constant, T the absolute temperature, e the elementary charge, and i_0 the dark current of the photodiode. The

slope factor at room temperature is approximately 25 mV (or 58 mV for the decadic logarithm). The voltage is thus inversely proportional to absorbance, *A*, of Lambert–Beer's law which is also logarithmically dependent on light intensity (see Eq. (2) above). The approach is thus a simple method to obtain signals which are proportional to concentration. The measuring arrangement is illustrated in Fig. 1C. Two options were used in this work. The voltage was either determined via a high input impedance operational amplifier in the voltage follower mode or directly with a standard multimeter. The operational amplifier has a very high input impedance ($10^{13}\,\Omega$) and a negligible input bias current (<5 pA) and thus only draws an insignificant current, comparable to the input of a pH-meter (as used by Tymecki et al. [32,30]), but the multimeter has a standard input impedance of 10 MΩ so that a current in the high nano-ampere range is pulled from the source.

The photovoltaic responses of LED RO1 as detector and the photodiode (using again LED Y1 as emitter) are shown in Fig. 5A and B respectively. As can be seen, the LED as detector showed a much higher sensitivity than the photodiode. Note the different scales for the two plots. For the LED the change in voltage was several hundreds of mV for a change of light intensity of approximately one order of magnitude, but the response function was not linear with the logarithm of light intensity. For the LED as detector the signals were strongly dependent on whether the measurements were carried out by direct connection to the multimeter, or via the high impedance operational amplifier. The multimeter with the relatively low input impedance of 10 MΩ showed a strong loading effect. It was also found that for the readings with the LED as detector it always required about 1–2 min of time to achieve stable voltage signals in both measurements approaches. For the photodiode the response was almost linear with the logarithm of the intensity according to our measurements with a slope close to the theoretically expected (64 and 66 mV per decade for the measurements with the operational amplifier and multimeter respectively). In this case, the readings were almost identical for the two measuring systems. For the photodiode stable readings were established within seconds.

3.5. Measurements of dye solutions using the photovoltaic mode

As the photodiode was found to perform very well in the photovoltaic mode using the direct measurement with a multimeter, this promising new configuration was therefore tested for carrying out measurements of dye solutions. For comparison measurements with a conventional set-up using a beam splitter with a reference photodiode were also made. In this case the photocurrents were processed by a log-ratio amplifier in integrated circuit format which gives an output voltage, V_o, according to the following equation:

$$V_o = \log \frac{i_0}{i} \tag{6}$$

The output voltage therefore directly represents absorbance, *A*. The measuring arrangement is shown in Fig. 1D and corresponds to the standard set-up employed in molecular absorption photometry. Solutions of Thymol Blue prepared in 0.1 M sodium hydroxide solutions were tested, and the yellow LED-emitter (Y1) which has a peak at 595 nm was chosen as the light source because of its compatibility with the absorption spectrum of the dye ($\lambda_{max}=592$ nm). The current driving the LED-emitter was kept constant at 25 mA.

The results obtained for the photodiode in the photovoltaic and the log-ratio modes are shown in Fig. 6A and B respectively. The values for Fig. 6B correspond to mAU (milli-Absorbance units). The plot is almost linear (correlation coefficient, $r=0.9997$), the very slight curvature is a common feature for LED based absorbance measurements which is due to the not perfect monochromaticity of the light source, which is a violation of the prerequisites for strict adherence to Lambert–Beer's law [40]. The standard deviation for 5 measurements of the solution of 6 ppm ($n=5$) was determined as 0.12%. The photovoltaic response is also good ($r=0.9996$), but the slope is negative and the measured voltages do not directly represent absorbance values. The standard deviation for the measurement of the solution of 6 ppm (in terms of concentration) was 0.34%.

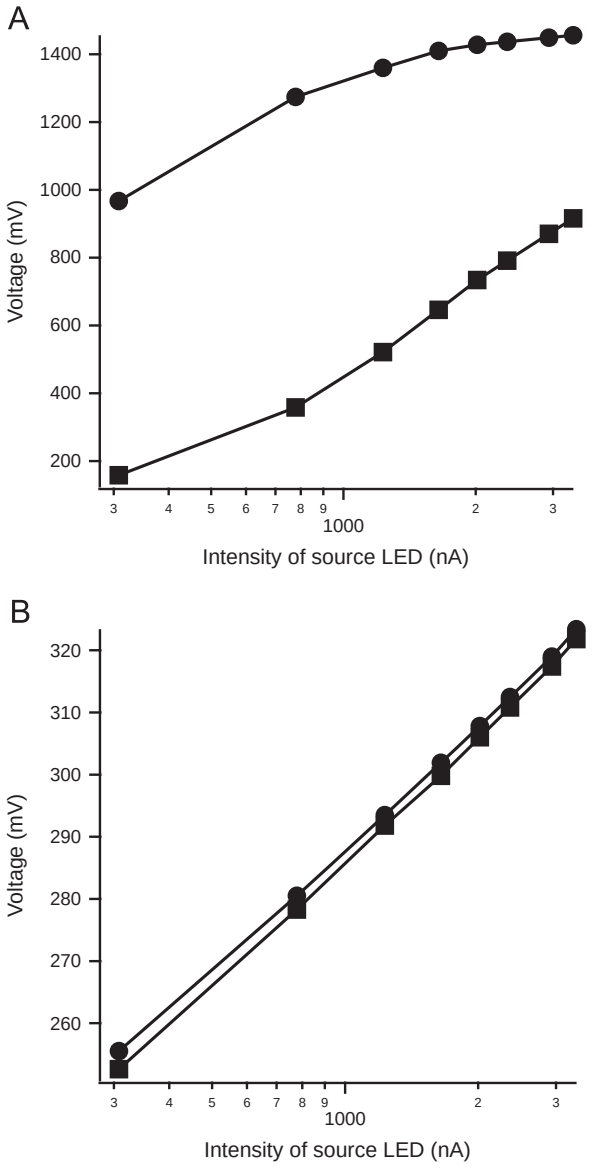

Fig. 5. Responses in the photovoltaic mode (see Fig. 1C) for the red–orange LED (RO1) (A) and the photodiode (SFH203P) (B) using either the high impedance version with the operational amplifier (circles) or the multimeter directly (squares) for different intensities of the source LED (Y1). The intensities refer to the photocurrents determined with the photodiode using the arrangement of Fig. 1A.

4. Conclusion

In the direct comparison between the use of LEDs as detectors and a photodiode it was found that, while the LEDs usually gave adequate results, the photodiode generally performed better. First of all, there is no difficulty regarding the spectral match between source and detector when using the latter. The photodiode also tended to give more predictable and reproducible results and stable readings were obtained instantly, while the LEDs settled slowly. This is not surprising,

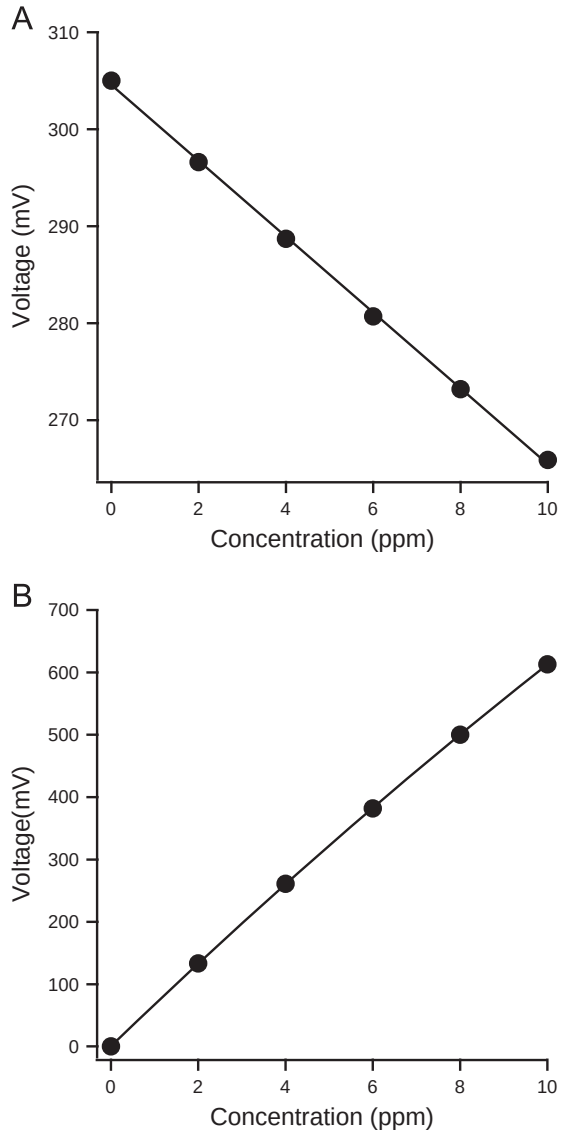

Fig. 6. Measurements of Thymol Blue solutions using the yellow LED (Y1) as emitter. (A) Photodiode (SFH203P) as detector in the photovoltaic mode using the direct measurement with the multimeter. (B) Photodiode (SFH203P) as detector in the log-ratio approach.

given that the latter have been optimized for emission, and hence the underlying physics are different for the two types of components (LEDs are direct bandgap devices, while photodiodes are indirect bandgap devices). For this reason, the reverse is also not possible, *i.e.* photodiodes do not emit light. The results also demonstrate that photodiodes tend to perform better in the special modes which had been suggested for the PEDD transducers (*i.e.* the junction discharge method and the photovoltaic mode). Note that the photodiode employed in these studies is a low cost version, widely available for approximately 1 US$ from distributors, which is comparable to the cost of LEDs. The use of a photodiode in combination with a log-ratio amplifier remains the best approach as it directly yields absorbance readings. Note that the reference is only necessary for the most demanding applications. The log-ratio amplifiers are now available from distributors for less than US$ 20 while only a few years ago the cost was significantly higher. If a truly low cost approach to photometry is sought, a photodiode used directly with an inexpensive multimeter in the photovoltaic mode should give acceptable results. We are not aware of prior reports on this set-up being employed in analytical chemistry. The only advantage of the use of LEDs as detectors is their spectral selectivity, but this is not generally relevant if paired with LEDs as sources.

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