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## **Design and development of a visible-to-near- infrared spectrograph for determination of moisture content of ground wheat**

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**Abstract.** *The paper briefly reviews available instrumentation techniques for near-infrared spectroscopy, and compares a diffraction grating spectrograph with other available instrumental setups. A spectrograph working in the spectral range of 450nm-1100nm was assembled with several lenses, a diffraction grating, and a linear charge coupled device (CCD) detector. Data acquisition and logging programs were developed using Virtual Instruments (VI). Ground wheat samples with different moisture contents were prepared and their near-infrared spectra were collected using the spectrograph. Linear regression models were calibrated using Partial Least Squares (PLS) method. Near-infrared spectra of test samples were collected and their moisture content was determined with the developed calibration model. Validation results gave a root mean square error of prediction (RMSEP) of 1.34%.*

**Keywords.** Near-infrared spectroscopy, spectrograph, moisture, ground wheat.

## Introduction

Near-infrared spectroscopy (NIRS) has been serving the agricultural and food industry for several decades since its initial applications by the United States Department of Agriculture (Norris 1962). In the years thereafter, significant upgrades on NIRS instrumentation were received constantly due to an increasing interest in utilizing NIRS in various agricultural and biological fields (Coates 1998). As an optical analysis method, NIRS is non-destructive, fast, reliable, and low in operation cost (Osborne et al. 1986). This allowed great flexibility of using the NIRS technique in routine quality analysis of agricultural products. Many commercial NIRS instrumentations are available in the market nowadays. Based on application needs, the instrumentation setups vary greatly. However, fast, inexpensive, and reliable instrumentations are always most preferred regardless of application purposes of end-users.

Moisture content in wheat is an important quality factor. This is mainly because moisture level is crucial in marketing, handling, and storage of wheat and its byproduct (Sauer 1992). Several reference and practical methods are available to determine the moisture content. Basic reference methods include vacuum oven methods, air oven methods, and distillation. Secondary methods include electronic methods and NIRS. Although very accurate, basic reference methods usually take a long time to get satisfactory results.

This study focused on development of a visible to near-infrared spectrograph and its application in quantitative determination of the moisture content in ground wheat. The assembled spectrograph was robust in structure, has good wavelength accuracy and long term stability. The system was applied to acquire near-infrared (NIR) spectra of ground hard red winter (HRW) wheat samples. A linear calibration model using partial least square regression (PLSR) (Martens and Næs, 1989) was set up using NIR spectra of ground wheat with different moisture contents and tested with an independent test set.

## Brief Review on NIRS Instrumentation

Most NIRS instruments utilize broadband halogen-tungsten light sources that are rich in NIR radiation. Based on different optical principles, there are two groups of instruments.

One group of these instruments works on principle of Michelson interferometer and Fourier transform (FT). The FT system possesses a moving mirror in the optical system to modulate polychromatic light into an interferogram and utilizes FT to decompose the interferogram into its spectral components. The FT system has very high wavelength accuracy, and good optical throughput. However, system performance is subject to mechanical vibration and environmental conditions. Most FT systems are restricted to laboratory use only.

The other group of instruments utilizes many different optical components to select light at different wavelengths. The wavelength selection components in commercial instruments are interference filters, electronically tunable filters, and diffraction gratings.

**Interference filter based instruments:** Multiple interference filters are often installed on a filter wheel controlled by microprocessor to change the scanning wavelengths. Filter-based instruments are very low in construction cost and usually dedicated to very specific analysis purposes.

**Electronically tunable filter based instruments:** Two most common types of electronically tunable filters are acousto-optic tunable filter (AOTF) and liquid crystal tunable filter (LCTF). Electronically tunable filter instruments are very fast in scanning, robust in structure with no moving parts, have random wavelength access and good wavelength accuracy. Despite the

aforementioned virtues, the construction cost of this kind of instrument is relatively, which has restricted their application.

**Diffraction grating based instruments:** This type of instruments are a good compromise between instrument flexibility and equipment cost. Such an instrument can be configured in a scanning mode by rotating the diffraction grating to change the detection wavelength. A scanning diffraction grating instrument is slow, and its performance degrades due to mechanical fatigue of rotating parts. The diffraction grating can also be mechanically fixed, and instead of using single element detector, a detector array on the focal plane of the diffraction grating can be employed to construct a spectrograph. A spectrograph is robust in structure, moderate in construction cost, and possess a good wavelength accuracy and measurement flexibility.

Mayes and Callis (1989) described a spectrograph system working in the 600 nm-1100 nm wavelength range and used it for absorption and diffuse reflectance measurement. The paper addressed the problems associated with scanning spectrometers and stated that a spectrometer based on multi-channel detector, like PhotoDiode Array (PDA) detector can provide more rugged, compact, and reliable solution.

Instead of using polychromatic light source, light emitting diodes (LED) based instruments employ light sources that produce narrow-band NIR radiation. The instrument is very simple, compact and only dedicated to very limited range of applications. Meanwhile, like filter-based instruments, its low construction cost makes it a good choice for specific analysis tasks.

## Materials and Methods

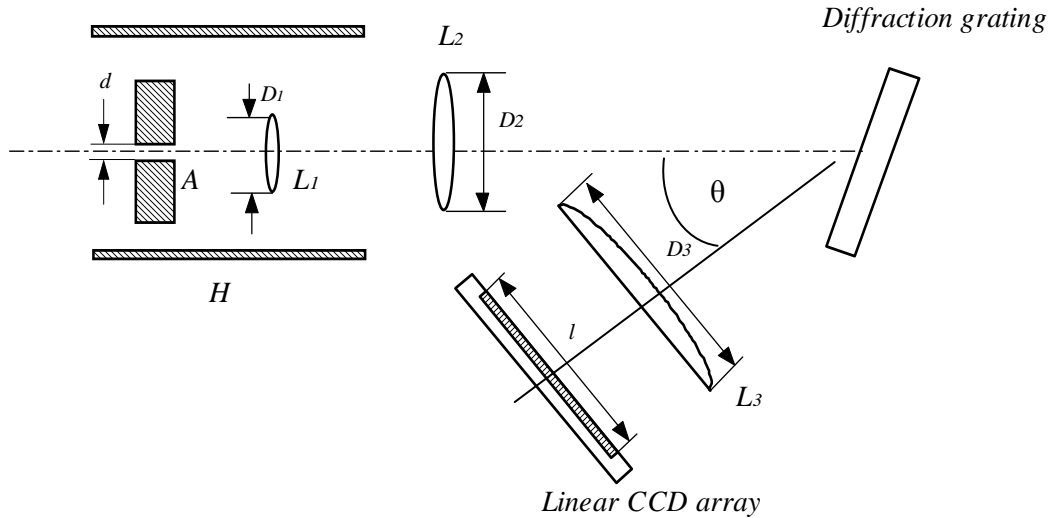
### *The Diffraction Grating Spectrograph*

A schematic of the developed spectrograph system is shown in Fig.1. An optical fiber bundle was installed in front of the spectrograph system. This enabled the system for reflectance measurement. An optical aperture  $A$  was placed right behind the optical fiber bundle output to limit the off-axis light propagation. The condenser  $L_1$  collected light emitting from the aperture and imaged the aperture with a magnification factor of 0.5. The collimating lens  $L_2$  was arranged in cascade with the condenser and had its focus on the image plane of the aperture. Thus, in a simplified optical system, incident light for the diffraction grating were parallel to the input optical axis. To reduce stray light, the lens  $L_2$  was slightly under-filled by incoming light from the condenser. The achromatic aberration of the collimating optics was minimized by employing a doublet lens instead of singlet.

A diffraction grating with groove density of 1200 per mm with optimized performance in the 800 nm-1100 nm region was used. The governing equation of a reflection diffraction grating is:

$$Gm\lambda = (\sin \alpha + \sin \beta_m) \quad (1)$$

where  $m$  is the diffraction order,  $\lambda$  the wavelength,  $G$  the groove density of the diffraction grating,  $\alpha$  the incident angle, and  $\beta_m$  is the diffraction angle.



**Figure 1. Optical system of the spectrograph. In the diagram, A is an aperture,  $L_1$  is a beam condenser;  $L_2$  is a collimating NIR doublet;  $L_3$  is an imaging plano-convex lens, and  $H$  is the housing for entrance optics.**

In the spectrograph, the first positive diffraction order was selected. When incident light went in parallel, the incident angle was the same. Therefore, light of different wavelengths were diffracted into different angles. The imaging lens  $L_3$  was placed on the same side of the incoming light path with respect to diffraction grating normal. The angle between the input optical axis and the optical axis of the imaging lens was set to  $45^\circ$ . A charge coupled device (CCD) image sensor (Alphas GmbH, Germany) with 2048 sensing elements was placed on the focus plane of imaging lens  $L_3$ . Thus, light of different wavelengths was focused onto different sensing elements along the linear CCD sensor. The corresponding wavelengths for different sensing elements were calculated using Eq.1.

### **Data Acquisition and Control**

A 16-bit multi-channel data acquisition board (NI-PCI-6013, National instruments, Austin, TX) was configured to work under differential input mode to acquire the output from the linear CCD image sensor. The linear CCD sensor array generated a trigger signal, which was used to control the timing of the data acquisition. To acquire one single spectrum, 50 output periods from CCD image sensor were acquired and averaged. The data sampling rate was set to 5K samples/s as a default value. Software was developed to change data sampling rates and average times. The software interface displayed the output spectra detected by linear CCD sensor, and the coefficient of variation (CV) curves, if multiple times were required to sample a single spectrum.

### **Wheat Samples**

Hard red spring wheat was used in the study. The initial moisture content (wet basis) of whole wheat was 9%. Moisture content was increased by adding calculated amount water according to:

$$W = \frac{K(E - I)}{1 - E} \quad (2)$$

where  $W$  (g) is the amount of water to be added,  $K$  (g) is the initial sample weight,  $E$  (%) is the expected moisture content level, and  $I$  (%) is the initial moisture content level.

Two high moisture levels (12% and 15%) were prepared. Moisture content level was decreased by heating wheat kernels in a Thermolyne OV35025 air oven (Dubuque, IA, USA) at  $130^{\circ}$  continuously for 1 hr. The approximate moisture content after heating was 5.0%. Thus, four moisture levels, which were 5%, 9%, 12%, and 15%, were used in this study. For each moisture level, 5 samples were prepared. To accurately determine the moisture content, the American Association of Cereal Chemists (AACC) standard procedures (AACC-44-15A) on moisture measurement of grain products using air oven method were followed (AACC, 1983). Wet sample based moisture content calculation formula was used. Wheat sample were ground using a Cyclotec 1093 sample mill (FOSS, Höganäs, Sweden). Of the twenty samples with different moisture content, 12 samples (three samples from each moisture level) were used to calibrate the linear regression model, and eight samples were used as an independent test set.

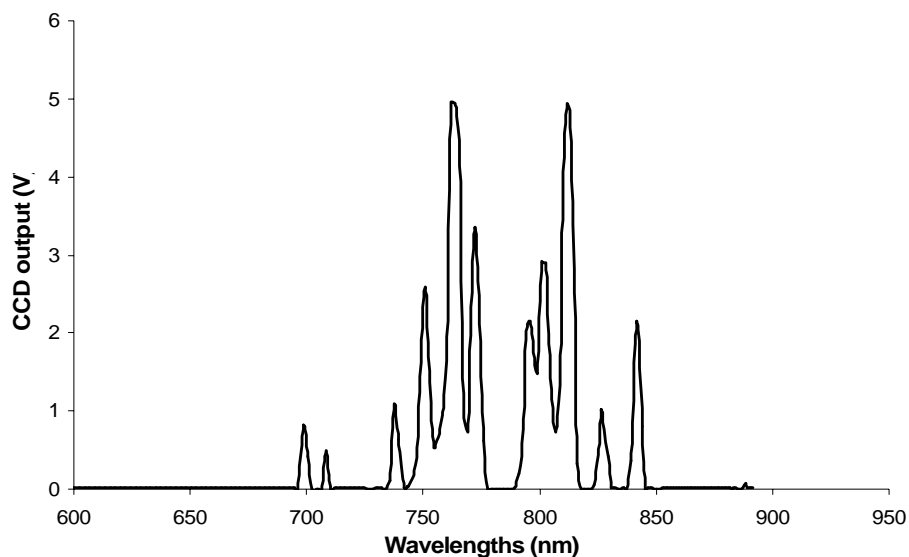
### ***Software and Computing facility***

GRAMS/AI from Thermo Electron Corp (Waltham, MA) was used to process spectra data and its PLS/IQ software package was used to calibrate and validate moisture content model. Data acquisition board and LabVIEW software were installed in a personal computer (CPU 1.7GHz, RAM 256M).

## **Results and Discussion**

### ***System Test***

Two of the most important parameters for the spectrograph are wavelength accuracy and optical band-pass. An argon lamp from Oriel instruments (Stratford, CT) was used to test the wavelength accuracy of the constructed spectrograph. The diffraction grating in the spectrograph was set to receive light at an incident angle  $7^{\circ}$  to test the wavelength accuracy. At an incident angle of  $7^{\circ}$ , the wavelength range was 624 nm - 888 nm. Within this wavelength range, there are many characteristic emission lines of the argon lamp (Appendix.1).



**Figure. 2. Emission spectra of an argon lamp measured by the spectrograph.**

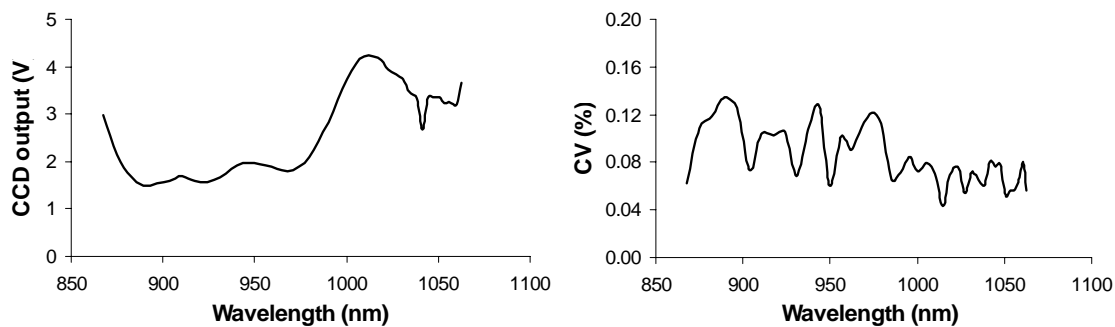
The spectra of the argon lamp was acquired using the spectrograph and shown in Fig. 2. The emission spectrum was sampled with a sampling rate of 20K samples/s, and resulted in a maximum wavelength interval of 2.2 nm on the lower wavelength end (624 nm), and a minimum wavelength interval of 0.88 nm on the higher wavelength end (888 nm). The wavelength accuracy was checked by comparing the wavelength positions of emission peaks in the acquired spectrum and that published by Oriel instruments. The test results indicated a maximum wavelength error of 1.8 nm that occurred at the 696.54 nm emission line.

A single-mode 650nm diode laser from World Star Tech Inc (Toronto, Ont.) was used to test the optical bandpass. The incident angle of diffraction grating was set to 0°. Assuming a Gaussian shape of the laser spectral emission and instrumental optical line profile, experimental data was fitted with a Gaussian curve with a standard deviation of 1.4, and full width at half maximum (FWHM) was calculated according to:

$$FWHM = 2\sqrt{-2\log\left(\frac{1}{2}\right)}\sigma = 2.35\sigma \quad (3)$$

Corresponding wavelength values were calculated and an optical bandpass of 6.0 nm was determined at an incident angle of 0°. When the incident angle was changed, optical bandpass was changed by multiplying the optical bandpass at an incident angle of 0° with cosine value of new incident angle. Considering the system was used in the near-infrared region, which corresponds to an attainable spectral region between 869 nm and 1066 nm at an incident angle of 17°, the optical bandpass was recalculated by multiplying 6.0 nm with  $\cos 17^\circ$ . Therefore, the actual bandpass was 5.5 nm for the near-infrared region.

An experiment to quantitatively determine the moisture content in ground wheat required that intensity of spectral data reflected the change of moisture content. Thus the stability of spectral intensity was important. In this experiment, the spectrograph system stability was tested by acquiring reflectance spectra of a Polytetrafluoroethylene (PTFE) disk every 3 s and repeated for 10 times. The average spectrum of PTFE disk and corresponding coefficient of variation (CV) curve over the 30 s period are shown in Fig.3. The CV values for the entire wavelength range (869 nm -1100 nm) were under 0.16% within the 30 s period, which indicated a high intensity measurement stability of the spectrograph.

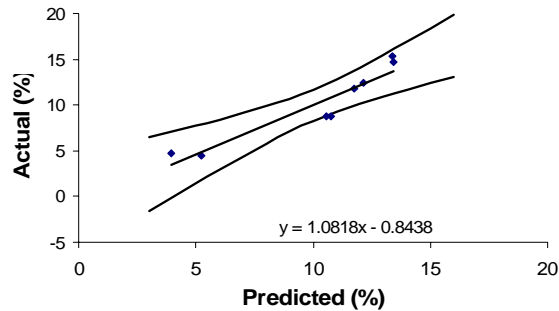


**Figure 3. Average spectra of the PTFE disk in 30 seconds and its corresponding CV curve. (a) reflectance spectra of PTFE disk; (b) corresponding CV curve.**

### ***Moisture Content Determination of Ground Wheat***

The ground wheat samples were put into a dish tray and pressed using a spatula to ensure consistent compactness and flatness of the measurement surface. The sample was then

illuminated and the reflectance spectra of the sample were taken using the spectrograph. The CV curves were monitored to ensure that spectra measurement was in control. Spectra of 20 samples were acquired and a calibration model was set up. Multivariate scatter correction (MSC) was applied on the reflectance spectra to reduce light scattering effect. Regression statistics showed that two factors were optimum for the PLSR model. Eight test samples were used to validate the calibration equation. Regression line and Working-Hotelling 95 percent confidence bands are shown in Fig.4.



**Fig. 4. Regression equations for validation. (a) regression equation for model calibrated with MSC corrected spectra;**

Validation statistics were listed in Table.1.

Table.1. Validation statistics

RMSEP (%)	Slope	Bias	R <sup>2</sup>
1.34	1.08	-0.84	0.88

RMSEP- Root mean square error of prediction.

It was observed that the prediction error was 1.34%. The regression equation had a coefficient of determination of 0.88, and a slope of 1.08.

## Conclusion

A diffraction grating spectrograph working in the visible to near-infrared region was constructed. When tested with an argon lamp in the 624 nm to 888 nm region, the spectrograph had a maximum wavelength error of 1.8 nm. An optical bandwidth expressed as full width at half maximum (FWHM) of 5.5 nm was obtained for the 869 nm to 1066 nm spectral region. An experiment was run to quantitatively determine the moisture content of ground wheat. A root mean square error of prediction (RMSEP) of 1.34% was obtained.

## References

- Approved methods of the American Association of Cereal Chemists.* 1983.44-15A: Moisture-Air-oven methods. St. Paul, MN: AACC.
- Coates, J. 1998. Vibrational spectroscopy: instrumentation for infrared and Raman spectroscopy. *Applied spectroscopy reviews* 33(4):267-425.
- Martens, H. and T. Næs 1989. *Multivariate calibration*, Chichester, U.K.: John Wiley & Sons, Inc.
- Mayes, D.M. and J.B. Callis. 1989. A photodiode-array-based near-infrared spectrophotometer for the 600-1100nm wavelength region. *Applied Spectroscopy* 43(1): 27-32.
- Norris, K. H. 1962. Instrumentation of infrared radiation. *Trans. ASAE* 5: 17-20.

Osborne, B.G., T. Fearn, and P.H. Hindle. 1993. *Practical NIR spectroscopy: with applications in food and beverage analysis*. 2nd edition. Essex, England: Longman Scientific & Technical; New York: J. Wiley.

Sauer, D.B. 1992. *Storage of cereal grain and their products*. 4th ed. St. Joseph, Mich.: ASAE.

## Appendix

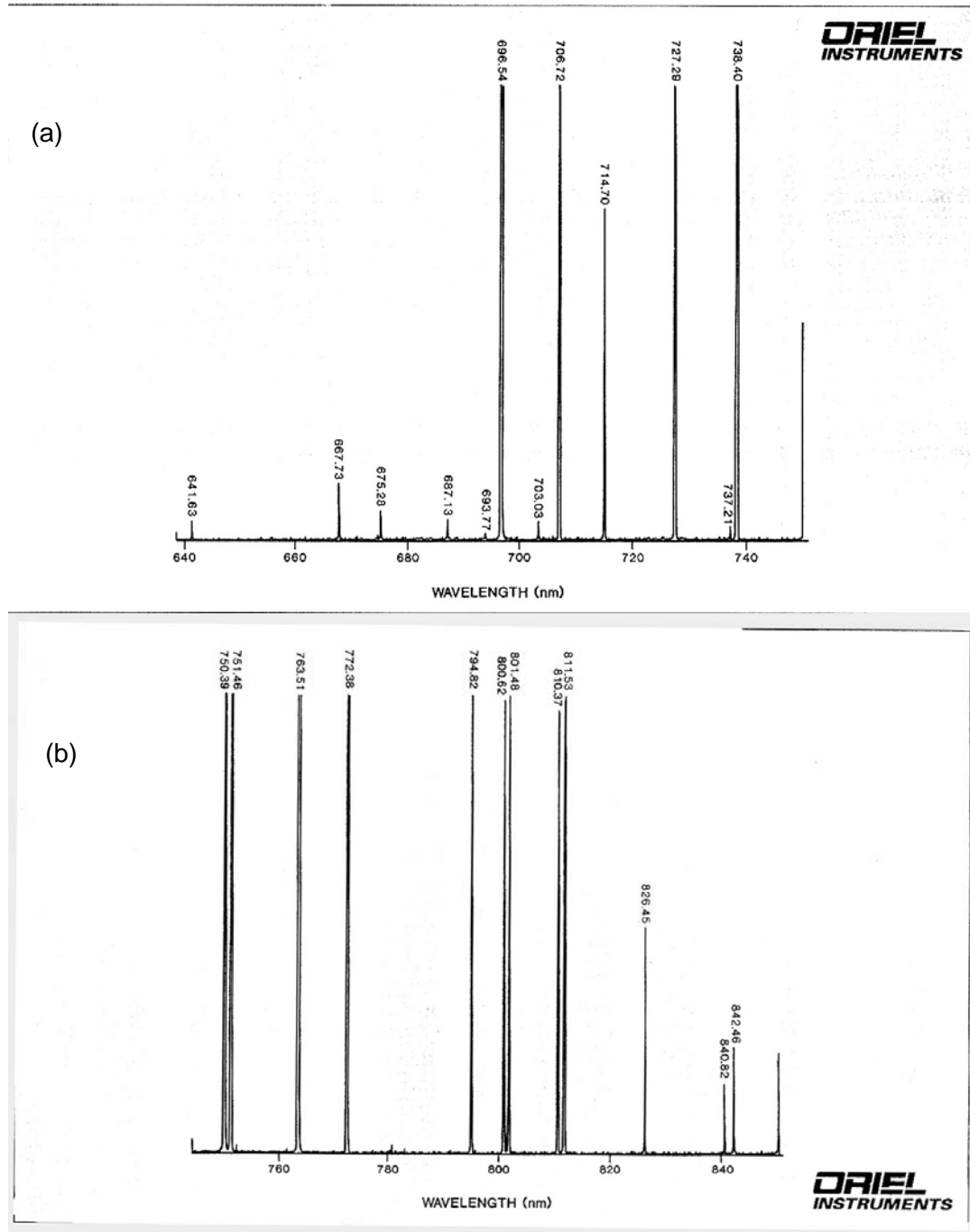


Figure. A1. Emission spectra provided by Oriel instruments. (a) emission spectra within the 640nm-740nm wavelength range; (b) emission spectra within the 740nm-850nm wavelength range. Source: Oriel instruments