

The measurement of high optical densities (up to 8 Abs) in the near-infrared

Application Note

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Abstract

The optical densities of various materials used in the manufacture of laser safety eyewear have been determined in the NIR. The lens materials were measured over wavelength ranges corresponding to the laser wavelengths for which the eyewear was designed (InGaAs, 980 nm and Nd:YAG, 1064 nm). Prior to measurement, a variety of filters of known optical density were used to validate the photometric performance of the spectrophotometer. Using the addition of filters technique, photometric range, accuracy and linearity were demonstrated up to 8 Absorbance units at 1200 nm in the near-infrared.

Introduction

The measurement of high optical density (or absorbance) in the NIR is of significant importance to scientists and engineers in applications ranging from the rapidly expanding area of biophotonics to the manufacture of designer sunglasses. Other areas where measurement of high optical density is important include the design, production and validation of bandpass, blocking and cut-off filters, quantitative analysis of strongly absorbing liquid media (such as potassium permanganate¹) and the measurement of turbid biological samples (such as those containing cytochrome P450²). Central to the ability to make these high optical density measurements are the photometric accuracy, linearity and dynamic range of the spectrophotometer in question.

Photometric linearity determines how accurately a spectrophotometer measures absorbance with increasing optical density or concentration. If an instrument has poor linearity, calibration curves (for instance) may deviate from linearity at high absorbance levels, reducing the photometric range of the instrument and accuracy of high optical density measurements. Together with linearity, photometric accuracy defines the ability of a spectrophotometer to accurately measure a given optical density or absorbance.



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Photometric accuracy and linearity are obviously vital wherever accurate and precise measurements are required. Of similar importance is the range over which the spectrophotometer response remains linear. This is known as the linear dynamic range and is traditionally defined as the range over which absorbance and concentration remain directly proportional to each other³. A wide linear dynamic range permits the measurement of a wide range of sample concentrations (optical densities) and can significantly reduce sample analysis and preparation (dilution) times. In this instance, optical densities of materials used in the manufacture of laser safety eyewear have been determined in the NIR. Prior to sample measurement, filters of known optical density were used to confirm the photometric performance of the spectrophotometer. Using this addition of filters technique, photometric range, accuracy and linearity were demonstrated up to 8 Absorbance units at 1200 nm in the near-infrared. Having demonstrated photometric accuracy, linearity and range, the lens materials in question were then measured at the appropriate wavelengths in the near-infrared.

Theory

The 'addition of filters' technique provides a straightforward and inexpensive means of determining the photometric linearity and range of a spectrophotometer without the need for expensive, calibrated standards. This method, applied to the visible portion of the electromagnetic spectrum, has been described elsewhere⁴. In this case, the same principle has been applied in the NIR to confirm photometric performance prior to sample analysis.

Rear beam attenuation was used when appropriate. Rear beam attenuation (RBA) is useful when the apparatus (or sample) in the sample beam attenuates the light beam considerably. In such situations, attenuation of the rear beam considerably increases the dynamic range of the instrument, as the detector does not 'see' two dramatically different signals (or light intensities). Typical situations where RBA may be of use include measuring the transmittance of dense

optical filters, compensating for sample holders/accessories that restrict the light beam, or (in general) measuring samples with high absorbance. RBA may be achieved using mesh filters of the type described below, or by using the fully automated Cary Rear Beam Attenuator⁵.

Materials and method

(For part numbers please see Reference 6)

Equipment

- Agilent Cary 6000i UV-Vis-NIR Spectrophotometer
- Lockdown Solid Sample Holder and Lockdown Cuvette Holder
- Mesh filter kit for attenuating reference beam

Protocol

Using the Lockdown Solid Sample Holder and Cuvette Holder, a Lockdown plate was prepared with optical rails and sample slide in the sample beam and cuvette base in the reference beam. The optical rails and sample slide were used to mount filters and samples, whilst the cuvette base was used to provide a platform for rear beam attenuation (RBA). This was achieved using V-holder and mesh filters (mesh filter kit for attenuating reference beam⁶). RBA of ~3.5 A was used for all filter and sample measurements. Measurements were made using independent UV-Vis and NIR control (fixed spectral bandwidth in the UV-Vis; variable spectral bandwidth in the NIR). Indicative parameters were as follows (longer signal averaging times were used in some instances):

- UV-Vis: 5 nm SBW, 1 nm data interval, 0.1 s signal averaging time
- NIR: 3 nm data interval, 0.3 s signal averaging time, Energy 3.00

All measurements were made in 'double beam' mode using a full slit height. Baseline correction (Zero/Baseline correction) was used in all cases.

Filters having nominal absorbance values ranging from 1 to 3 over the desired wavelength range were used for the addition of filter experiments (all filters were manufactured by Schott; www.schott.com). Particular care was taken with respect to filter positioning and movement between measurements. In the case of the two-filter addition, the first filter was measured, followed by the two together and then the second (by careful removal of the first).⁴ A similar approach was used for the three-filter addition.

Results and discussion

The results of the addition of filters measurements can be seen in Figure 1. For the two-filter addition, the actual and predicted measurements show excellent correlation across the entire NIR wavelength range measured, up to an absorbance maximum of 7.19 Abs at 1248 nm. In the case of the three-filter addition, where an additional absorbance of ~1 Abs is measured, good correlation between actual and predicted is again observed up to a maximum of 8.10 Abs at 1208 nm.

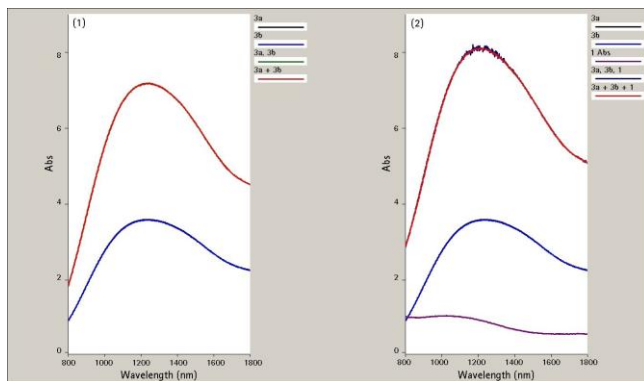


Figure 1. Addition of filters: (1) addition of two filters for an absorbance maximum of 7.19 (1248 nm) and (2) addition of three filters for an absorbance maximum of 8.10 (1208 nm). In each case, the red trace is the result of the mathematical addition of the individual filter spectra, and is overlaid on the spectrum of the combined filter measurement

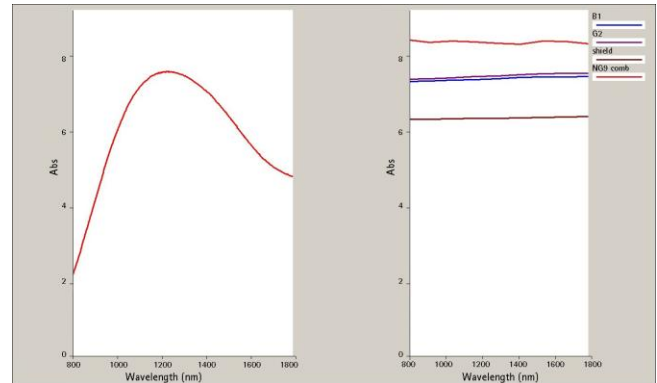


Figure 2. Sample scans of (left) high optical density lens material (absorbance maximum of 7.45 at 1230 nm, and (right) two plastic and two glass lens materials for use at 1064 nm (Nd:YAG)

The noise evident in the combined three-filter measurement is indicative of the very low light throughput around the absorbance maximum. However, the profile of the measured spectrum is essentially identical to that of the mathematical addition of the three separate filters. The noise is distributed symmetrically about the expected profile, and could be minimized using a longer signal averaging (acquisition) time.

Whilst not exhaustive, the addition of filter experiments described confirm the ability of the spectrophotometer to make photometrically accurate and precise absorbance measurements at optical densities up to 8 Abs. High optical density samples were subsequently analyzed using parameters similar to those used for the filter measurements. The results can be seen in Figures 2 and 3.

The spectra obtained show excellent signal-to-noise across the wavelength and absorbance ranges investigated. Furthermore, they clearly demonstrate the ability of the spectrophotometer to make accurate high optical density measurements.

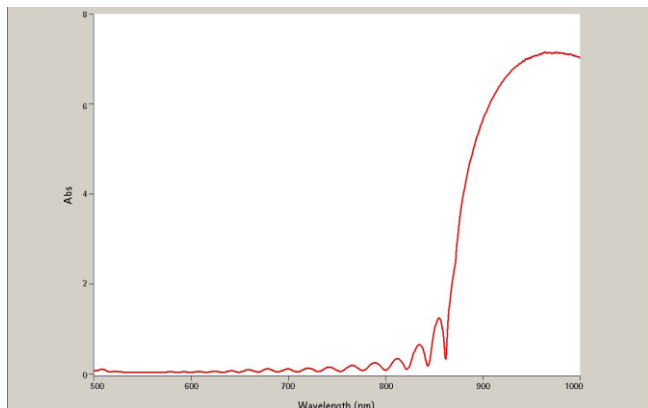


Figure 3. Visible-NIR spectrum of blocking filter for use over the 900 – 1100 nm wavelength range (e.g. InGaAs lasers at 980 nm). Maximum absorbance of 7.16 Abs observed at 964 nm

Conclusion

The addition of filters technique has been successfully used to demonstrate the photometric range, accuracy and linearity of the Cary 6000i UV-Vis-NIR spectrophotometer. Using the same instrument, spectra of a number of samples having absorbance maxima ranging from 7 to 8 were readily acquired.

Acknowledgements

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3. Hind, A.R., 'To improvements in spectrophotometry', American Laboratory, 34(24) 2002 32.
4. 'Photometric Linearity Range of the New Generation Cary 4000/5000/6000i spectrophotometers', Data Sheet, www.agilent.com.
5. Cary Rear Beam Attenuator accessory; part number 00 100441 00
6. Part numbers:

Product	Part Number
Agilent Cary 6000i UV-Vis-NIR Spectrophotometer*	00 100794 00
Mesh filter kit for attenuating reference beam	99 100477 00
Cary WinUV Analysis Pack Software	85 101950 00

*includes Lockdown Solid Sample Holder and Cuvette Holder as standard

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