Measuring optical densities over 10 Abs on the Agilent Cary 7000 Universal Measurement Spectrophotometer (UMS)

Technical Overview

Accurately measure highly attenuating optical filters

High blocking optical filters deliver critical optical control in a wide variety of consumer and industrial products. Highly attenuating (i.e., high optical density) filters are used in personal protective equipment, such as laser safety eyewear, through to precise, low light level control in optical systems for enhanced sensitivity at preferred wavelengths. This technical overview demonstrates the performance of the Cary 7000 UMS for the measurement of such materials. The procedure adopted uses filters of chosen nominal, but not precisely known, optical density to validate the photometric performance of the spectrophotometer. Using the addition of filters technique, photometric range, accuracy and linearity were demonstrated over 10 absorbance units (Abs) in the visible and over 8 Abs in the near-infrared (NIR) wavelength region.

Introduction

The measurement of high optical density (or absorbance) is of significant importance to scientists, engineers and manufacturers in applications ranging from the rapidly expanding area of biophotonics through 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 permanganate [1]) and the measurement of turbid biological samples (such as those containing cytochrome P450 [2]). Central to the ability to accurately measure these high optical density samples is 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 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.

Photometric accuracy and linearity are 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 other [3]. 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 that result from either dilutions, in case of liquids, or additional — sometimes destructive — modification of solid samples in order to measure them within the performance capabilities of the instrument.

In this instance, optical densities of materials used in the manufacture of optical filters have been measured in the visible and NIR spectral range. Using the addition of filters technique, photometric range, accuracy and linearity were demonstrated over 10 Abs at ca. 640 nm in the visible region and over 8 Abs at ca. 1500 nm in the NIR region.

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 elsewhere [4]. In this case, the principle has been applied in the visible and NIR to confirm photometric performance prior to sample analysis. Rear beam attenuation was used when appropriate.

When the incident light in the sample beam is highly attenuated, rear beam attenuation (RBA) is required to increase the dynamic range of the instrument, in order to balance the sample and reference signals (or light intensities) on the detector. Typical situations where RBA is of use include measurement of dense optical filters, beam balance when using sample holders or accessories that attenuate 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 Attenuator [5].

Measurements

For investigation of photometric range in the visible region, the addition of a BG25 filter with attenuating mesh filters was measured.

The collection procedure was as follows: 4.5 Abs attenuation was installed in the rear beam. A baseline was measured with a short spectral averaging time of 1 s. The BG25 and attenuating mesh filters are then measured separately. The spectral averaging time was then lengthened to 50 s to perform a blocked beam measurement followed by the measurement of the BG25 filter and attenuating mesh filters together. All measurements were made in %T. Particular care was taken with respect to filter positioning and movement between measurements.

The blocked beam measurement was then subtracted from all filter measurements and the results were converted to absorbance units. The individual spectra of the BG25 filter and the attenuating mesh filter were added together to calculate the predicted spectrum for the measurement of all the filters together. A summary of the collection parameters is given in Table 1.

Table 1. Summary of parameters used for addition of filter measurement in the visible

Parameter	Values
Data interval	1 nm
Spectral bandwidth	5 nm
Spectral averaging time	1 s or 50 s
Rear beam attenuation	4.5 Abs
Measurement mode	Double beam
Slit height	Full

Cary 7000 Universal Measurement Spectrophotometer

A similar protocol was used with measurements in the NIR region for the addition of 2 UG 11 filters. A summary of the parameters is shown in Table 2.

Table 2. Summary of parameters used for addition of filter measurement in the NIR

Parameter	Values
Data interval	4 nm
Spectral bandwidth	Variable
Energy	1.0
Spectral averaging time	1 s or 10 s
Rear beam attenuation	2.2 Abs
Measurement mode	Double beam
Slit height	Full
Detector	PbSmart Lead Sulfide (PbS)

Results and discussion

The results of the addition of filters measurements in the visible region can be seen in Figure 1. The actual (red spectrum) and predicted (green spectrum) measurements show excellent correlation with the maximum absorbance at approximately 640 nm reaching over 10 Abs. The good signal:noise evident in the combined filter measurement is indicative of the superior performance of the Cary 7000 UMS and its ability to detect extremely low light levels around the absorbance maximum.

The results of the addition of filters measurements in the NIR region can be seen in Figure 2. The actual (red spectrum) and predicted (green spectrum) measurements show exceptional correlation with the maximum absorbance at approximately 1500 nm reaching over 8 Abs.

The Cary 7000 UMS and the Cary 5000 UV-Vis-NIR are the only instruments on the market that use PbSmart Technology in their PbS NIR detector. PbSmart is a proprietary technique which controls Agilent PbS detectors. When coupled with the superior stray light and optical design of the instrument, industry leading NIR performance is achieved. While other commercial instruments adopt a wide band InGaAs detector to achieve a wider linear dynamic range in the NIR, the Cary 7000 UMS and Cary 5000 system design achieves this on the broader wavelength range PbSmart PbS detector. If your application demands exceed 8 Abs in the NIR the Cary 6000i spectrophotometer, which uses a narrow band InGaAs detector and a tailored diffraction grating for this detector, is designed to meet your needs.

While 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 over 10 Abs in the visible region and over 8 Abs in the NIR region. The results clearly demonstrate the ability of the spectrophotometer to make accurate high optical density measurements.

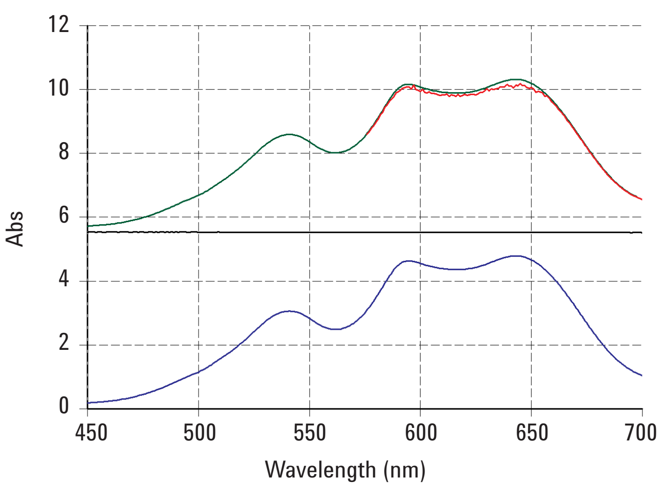

Figure 1. Spectra of BG25 filter (blue), attenuating mesh filter (black) and the spectrum of the BG25 filter and attenuating mesh filter together (red). The green spectrum is the predicted result based on the addition of blue and black spectrum.

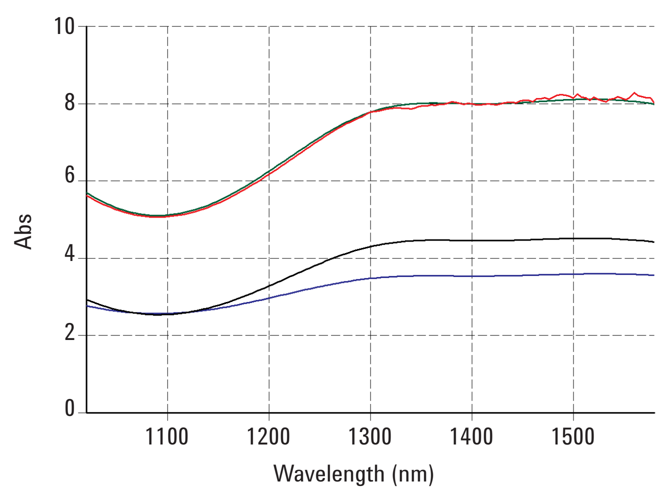

Figure 2. Spectra of UG11 filter 1 (blue), UG11 filter 2 (black) and the spectrum of the UG11 filter 1 and UG 11 filter 2 together (red). The green spectrum is the predicted result based on the addition of blue and black spectrum.

Conclusion

The addition of filters technique has been successfully used to demonstrate the photometric range, accuracy and linearity of the Cary 7000 Universal Measurement Spectrophotomter in the UV-Vis-NIR. Using the same instrument, spectra of samples having absorbance maxima above 8 Abs in the NIR region and over 10 Abs in the visible region were readily acquired.

References

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