Technical Overview

Evaluation of Photometric Accuracy in the Near Infrared Region Using Metal-on-Quartz Filters

Comparing Agilent Cary 7000 UV-Vis-NIR spectrophotometer measurements against traceable reference material values

Authors

Wesam Alwan and Travis Burt
Agilent Technologies, Inc.

Abstract

UV-Vis-NIR spectroscopy technique provides quick and accurate characterization of solid and liquid analytes in a wide range of applications. To achieve high-quality measurements, photometric accuracy of the spectrophotometers can be verified using certified reference materials. These measurements are usually performed in the UV-Vis region, as required by global pharmacopeias. However, in this technical overview, photometric accuracy (absorbance) in the near infrared (NIR) region of the Agilent Cary 7000 universal measurement spectrophotometer (UMS) is evaluated using three metal-on-quartz filter reference materials.

Introduction

The photometric accuracy of a spectrophotometer can be determined by measuring certified reference materials of known absorbance values and concentration and comparing the measured absorbance with the reference material absorbance values at each specific wavelength. Poor photometric accuracy will produce incorrect results.

In transmission measurements, the photometric response of the spectrophotometer follows the Beer-Lambert law. According to the law (Equation 1), a linear relationship exists between absorbance and sample concentration (as represented in Figure 1). The simple linear relationship between absorbance and concentration, and the relative ease of measurement of ultraviolet-visible-near infrared (UV-Vis-NIR) light have made UV-Vis-NIR spectroscopy a fundamental tool for many routine quantitative analytical methods.

Equation 1.

$$A = \varepsilon bc$$

Where:

A = absorbance

ε = molar absorptivity ($M^{-1}cm^{-1}$)

b = pathlength (cm)

c = concentration (M)

The United States Pharmacopeia (USP) and the European Pharmacopeia (Ph. Eur.) guidelines describe how to verify that the analytical performance of UV-Vis spectrophotometers is suitable for the intended operational range of the analysis. The guidelines relate mainly to photometric accuracy in the UV-Vis region. For measurements below 1 Abs and above 1 Abs, the absorbance accuracy must be ±0.01% and ±1% of the absorbance measured, respectively.[1,2]

This technical overview evaluates the photometric (absorbance) accuracy in the NIR region of the Cary 7000 universal measurement spectrophotometer (UMS) using metal-on-quartz filter reference materials.

Experimental

Instrumentation

The Cary 7000 UMS UV-Vis-NIR instrument was used to evaluate photometric accuracy in the NIR region using the operating parameters given in Table 1.

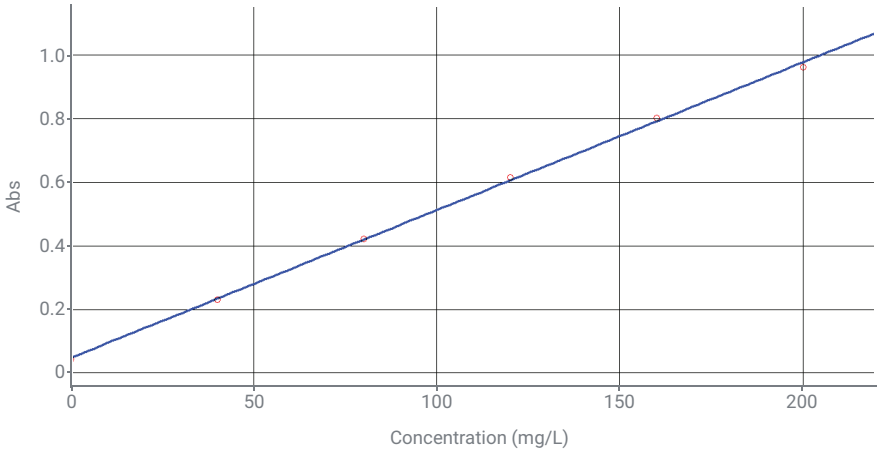

Figure 1. The Beer-Lambert law states that a linear relationship exists between the absorbance of a solution and the concentration of that solution.

Table 1. Agilent Cary 7000 UMS operating parameters used to evaluate NIR region photometric accuracy.

Parameter	Setting
Wavelength Range	1,000 to 3,000 nm
Signal Averaging Time	0.1 s
Data Interval	1 nm
Spectral Bandwidth	Auto (nm)
Slit Height	Full
Number of Measurements (n)	Reported results are the average of three successive measurements of the filter reference materials
Samples	1%T (2 Abs), 3%T (1.5 Abs), and 50%T (0.3 Abs) metal on fused silica filter reference materials

Solid filter reference materials (RMs) with different transmission properties were used to evaluate the photometric accuracy of the Cary 7000 in the NIR region. A metal-on-quartz NIR reference set, 1%T (~2 Abs), 3%T (~1.5 Abs), and 50%T (~0.3 Abs), was bought from Starna Scientific Ltd (product number RM-NQ1NQ35NQ, Figure 2). The filter RMs are traceable to NIST standard reference material (SRM) 2031a (for the UV-Vis wavelengths) and National Research Council of Canada (NRC) primary certified reference materials (CRMs) for the NIR wavelengths. The filters are orientated at 0 degrees to the incident beam in the spectrophotometer and are constructed with an optical sandwich. The design minimizes the fringing that is often caused by internal reflection effects from metal-on-quartz filters.[3]

Results and discussion

The NIR metal-on-quartz RMs were measured by the Cary 7000 using the operating parameters described in Table 1. To assess photometric accuracy in the NIR region, the absorbance at certified wavelengths 1,100, 1,700, 2,210, 2,500, and 2,800 nm of each filter was determined. The measured values were compared to the certified values, and the percentage error was calculated accordingly. As shown in Table 2, the error between measured absorbance and certified absorbance values was <1% for all filters and wavelength's measured, verifying the photometric accuracy of the Cary 7000 for NIR measurements. The data are also shown in Figure 3 (transmission) and Figure 4 (absorbance). Good repeatability of the three measurements of each filter RM was also achieved by the Cary 7000. Representative repeatability data for the 2 Abs filter reference material is shown in Figure 5 (transmission) and Figure 6 (absorbance).

Figure 2. Metal-on-quartz filters used for the NIR photometric accuracy test of the Agilent Cary 7000 UMS. Photo reproduced with permission from Starna Scientific Ltd.

Table 2. Photometric accuracy using three NIR metal-on-quartz filter reference materials. Air was used as a blank and the reference beam was unobstructed during measurements using the Agilent Cary 7000 UMS.

Wavelength (nm)	Certified Value (Abs)	Expanded Uncertainty of the Filter (Abs)	Cary 7000 Value (Abs, n = 3)	Difference Between Measured and Certified Values (Abs)	Percentage Error (%)
Metal-on-Quartz Filter 0.3 Abs (50%T)					
1,100	0.3688	±0.0025	0.3694	0.0006	0.1894
1,700	0.3709	±0.0025	0.3716	0.0007	0.2153
2,210	0.3676	±0.0025	0.3680	0.0004	0.1265
2,500	0.3665	±0.0025	0.3665	0.0000	0.0045
2,800	0.3786	±0.0025	0.3811	0.0025	0.6704
Metal-on-Quartz Filter 1.5 Abs (3%T)					
1,100	1.5371	±0.0058	1.5390	0.0019	0.1295
1,700	1.5068	±0.0058	1.5086	0.0018	0.1216
2,210	1.5064	±0.0058	1.5074	0.0010	0.0699
2,500	1.5124	±0.0058	1.5127	0.0003	0.0237
2,800	1.5291	±0.0058	1.5370	0.0079	0.5210
Metal-on-Quartz Filter 2 Abs (1%T)					
1,100	1.6564	±0.0059	1.6591	0.0027	0.1638
1,700	1.6254	±0.0059	1.6279	0.0025	0.1577
2,210	1.6274	±0.0059	1.6284	0.0010	0.0617
2,500	1.6347	±0.0059	1.6349	0.0002	0.0179
2,800	1.6521	±0.0059	1.6576	0.0055	0.3347

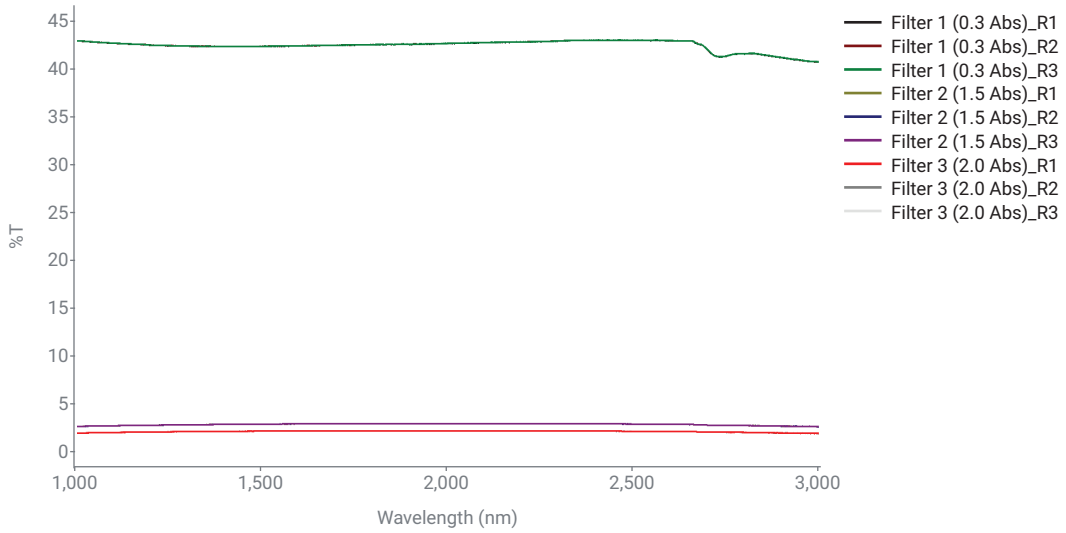

Figure 3. Spectra (n = 3) of the three metal-on-quartz filters measured using the Agilent Cary 7000 UV-Vis-NIR system in transmission. Absorbance values at 1,100, 1,700, 2,210, 2,500, and 2,800 nm of each filter were recorded and compared to the certified reference values.

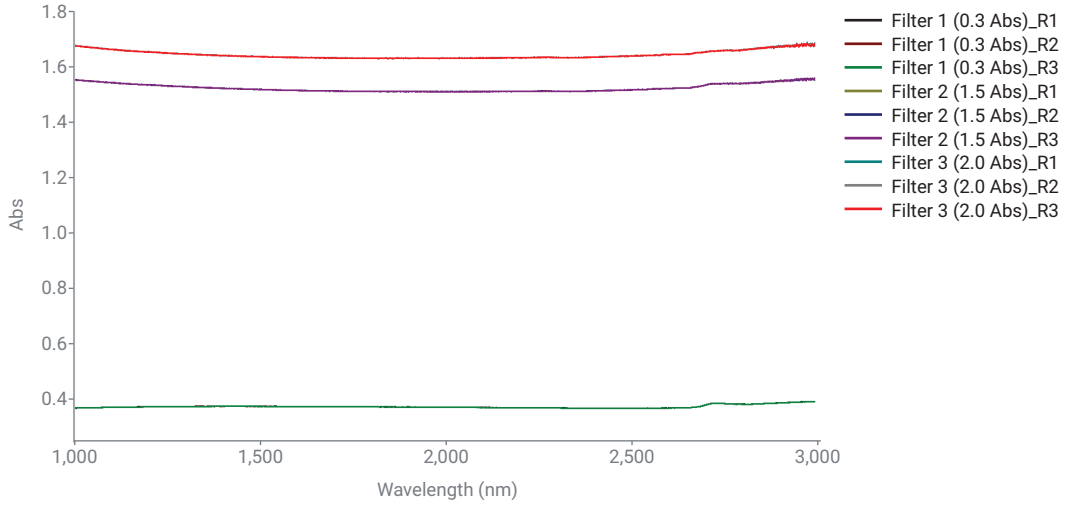

Figure 4. Spectra (n = 3) of the three metal-on-quartz filters measured using the Agilent Cary 7000 UV-Vis-NIR system in absorbance. Absorbance values at 1,100, 1,700, 2,210, 2,500, and 2,800 nm of each filter were recorded and compared to the certified reference values.

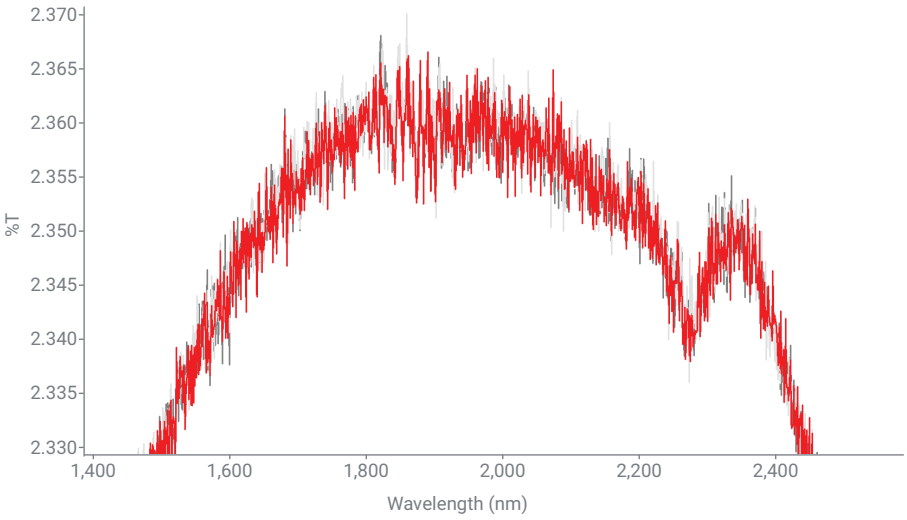

Figure 5. Zoomed in section of the 2 Abs filter measured on the Agilent Cary 7000 UV-Vis-NIR system in transmission, demonstrating repeatability (n = 3).

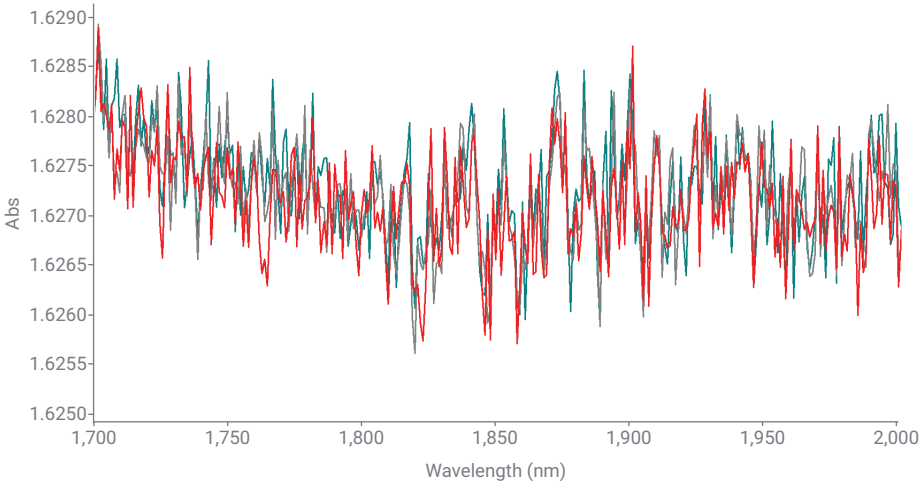

Figure 6. Zoomed in section of the 2 Abs filter measured on the Agilent Cary 7000 UV-Vis-NIR system in absorbance, demonstrating repeatability (n = 3).

Conclusion

The photometric accuracy of the Agilent Cary 7000 UV-Vis-NIR spectrophotometer was tested in the NIR region by measuring three metal-on-quartz filter reference materials ranging from 0.3 to 2 Abs. Excellent accuracy and repeatability results were obtained for all wavelengths with an error between measured absorbance and certified absorbance values of <1% for all filters.

Testing the photometric accuracy and repeatability of a spectrophotometer across its operational absorbance range using traceable reference materials provides confidence in the quality of the final data.

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