APPLICATION NOTE

MEASURING THE NIR ABSORPTION SPECTRA OF HOLMIUM AND DIDYMIUM GLASS FILTERS WITH THE PRONIR SPECTROMETER

INTRODUCTION

Rare-earth elements are a group of 17 chemical elements in the periodic table consisting of 15 lanthanide, from lanthanum to lutetium, plus scandium and yttrium, as defined by the International Union of Pure and Applied Chemistry (IUPAC) [1]. Certain rare-earth elements, such as neodymium, praseodymium, erbium, and holmium, when present as trivalent ions, display sharp absorption peaks in the ultra-violet (UV), visible (Vis), and near-infrared (NIR) regions [1,2].

Keywords:

NIR Spectroscopy;

ProNIR Spectrometer;

Rare-Earth Elements;

Didymium Glass Filter;

Holmium Glass Filter;

Holmium, didymium (mixture of neodymium and praseodymium), and cerium oxide are rare-earth elements with a series of sharp and well-characterized absorption peaks. They are used for validation of the wavelength scale of a spectrophotometer in the UV, Vis, and NIR regions. These elements are widely used for wavelength qualification and are accepted by all major pharmacopeias and regulatory authorities [3].

Measuring an absorption spectrum across a wide spectral range (i.e., from 190 to 2500 nm) requires two light sources (deuterium for UV and tungsten-halogen for Vis-NIR) and two detectors with different operational ranges [4]. The first detector is usually a silicon-based charge-coupled device (CCD) or a photomultiplier (PMT) and has the best operational range from 200 to 850 nm, while the second detector is typically an indium gallium arsenide (InGaAs) and has the best operational range from 850 to 2500 nm [4].

Due to the different detection ranges and instrumental conditions (e.g., slit width and gratings), wide range absorption spectra require users to perform two distinct measurements; the first spectrum is measured from 200 to 850 nm while the second is measured from 850 to 2500 nm, with different experimental conditions from the first one.

In this Application Note, the NIR absorption spectra (from 850 to 2500 nm) of holmium and didymium oxide glass filters are measured using the ProNIR spectrometer, in a setup using the W20 light source, standard cuvette holder, and solid sample holder. Furthermore, is also discussed the advantage of combining the data acquired from two spectrometers to obtain a wide range absorption spectra. The data herein presented was obtained with the new software package, the LightScan 2.0.

MATERIALS & METHODS

Reagents

- Holmium Glass Filter;
- Didymium Glass Filter;

Instruments and Accessories (Figure 1):

- W20 Light Source;
- Standard Cuvette Holder;
- Vis-NIR Optical Fibers with 600 µm of diameter;
- Solid Sample Holder;
- ProNIR Spectrometer (Slit: 50 µm; Grating: 150 grooves/1200 nm; Thermoelectrical Cooled Detector: Hamamatsu with 512 pixels);

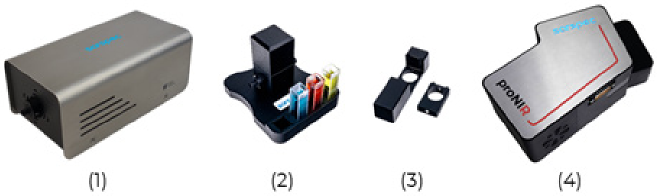

Figure 1 – W20 Light source (**1**), Standard Cuvette Holder (**2**), Solid Sample Holder (**3**), ProNIR Spectrometer (**4**) used to measure the NIR absorption spectra of holmium and didymium glass filters.

EXPERIMENTAL PROCEDURE

1. Each rare-earth glass filter was inserted in the solid sample holder;

2.The ProNIR detector was turned on and cooled down to -20ºC, to significantly increase the signal-to-noise ratio and acquisition stability;

3.The instrument settings selected on the LightScan software are specified in **Table 1**;

Table 1 – Instrument settings used for these experimental measurements.

Parameter	Value
Integration time (ms)	4
Average	150
Smoothing	0

RESULTS

The UV-Vis-NIR absorbance spectra (up to 900 nm) of holmium and didymium glass filters were previously obtained with the new FAbS spectrophotometer and are depicted in **Figure 2**. A detailed information about the absorption peaks of the holmium oxide glass filter can be found elsewhere [5].

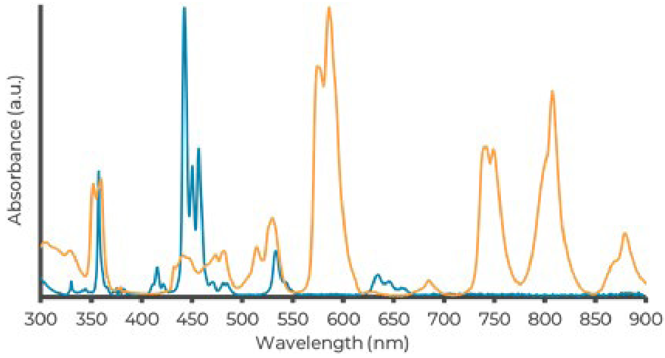

Figure 2 – Normalized absorption spectra of holmium (blue line) and didymium (yellow line) glass filters, previously obtained with the new FAbS spectrophotometer.

Both holmium and didymium glass filters provide sharp and well-defined absorption peaks within the range of 300 to 900 nm. In contrast with holmium, the didymium glass filter has a wider spectral range, which makes it more suitable to be used as a wavelength reference material to verify the wavelength scale of spectrophotometers. However, despite its wider spectral range, the didymium glass filter is not approved for wavelength accuracy tests by all major pharmacopeias. Instead, the holmium glass filter is the only solid reference approved for that purpose.

Data about holmium and didymium absorption spectra in the NIR region (from 900 to 2500 nm) is scarce. Few transmission spectra reports can be found for the holmium glass filter [6,7]. For the didymium glass filter, only one transmission spectrum was found [7]. In **Figures 3** and **4** are presented the absorption spectra of holmium and didymium glass filters from 300 to 2500 nm, respectively. In addition to the absorption peaks in the UV-Vis region, holmium glass filter is also characterized by two absorption peaks in the NIR region. The first band is observed around 1150 nm, while the second band is observed around 1900 nm and has a major peak with a maximum around 1960 nm.

The structure and wavelength of the NIR absorption bands presented in **Figure 3** are in agreement with the transmission spectra found in the literature for the holmium glass filter [6,7].

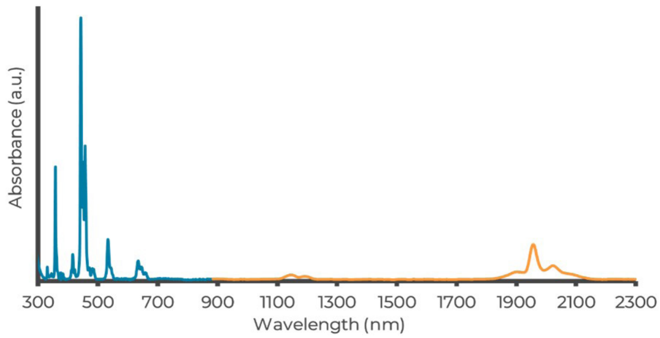

Figure 3 – Absorption spectrum of holmium glass filter obtained with FAbS from 300 to 900 nm (blue line), and ProNIR from 900 to 2300 nm (yellow line).

In contrast with holmium, didymium glass filter has three major broad absorption bands in the NIR region. The first band observed starts at 1300 nm and has a maximum around 1520 nm. The second band starts at 1750 nm has a maximum around 1920 nm. The third and last band starts at 2300 nm and has a maximum around 2430 nm. The structure and wavelength of the NIR absorption bands given in **Figure 4** are in agreement with the transmission spectrum found in the literature [7].

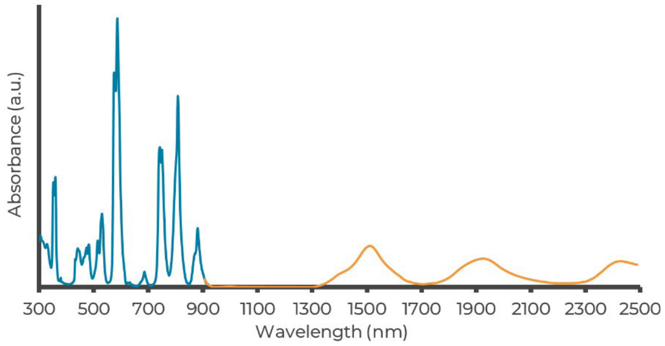

Figure 4 – Absorption spectrum of didymium glass filter obtained with FAbS from 300 to 900 nm (blue line), and ProNIR from 900 to 2300 nm (yellow line).

In this Application Note is demonstrated how simple and easy is to measure the wide absorption spectra of holmium and didymium oxide glass filters from 180 to 2600 nm (**Figures 3 and 4**, respectively) with the new FAbS and ProNIR spectrometers, powered by the new LightScan 2.0. This new software is a simple but powerful spectroscopic platform that allows the user to interconnect all the devices and get the full potential of the spectroscopic setup. The acquisition is controlled by the LightScan 2.0 and can be performed simultaneously, by using two spectrometers with different operational ranges.

CONCLUSIONS

The absorption spectra of holmium and didymium oxide glass filters were measured from 850 and 2500 nm by using the new LightScan 2.0 software and the new ProNIR Spectrometer.

The thermoelectrical cooled detector on the ProNIR provides an outstanding acquisition of absorption, transmittance, and reflectance spectra with very low noise, for acquisition times ranging the milliseconds to seconds. In addition, it also provides a great sensitivity for emission measurements in the NIR range.

The absorption spectra obtained with ProNIR were combined with those obtained using FAbS Spectrophotometer, from 300 to 900 nm, showing a perfect overlap between 850 and 900 nm. It is demonstrated that the absorption spectra of both holmium and didymium glass filters, two calibrating reference materials, measured using FAbS shows a perfect overlap with that measured using ProNIR. This suggests that by combining the two spectrometers in a simultaneous acquisition method a wide range absorption spectra can be obtained. The simultaneous acquisition method provides users with a wider spectral range that can go from 185 to 2500 nm, in a single measurement within a few seconds. This feature is already included in the new software, the LightScan 2.0.

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