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# Characterization of an Erbium-Doped Fiber Amplifier as a Light Source and Development of a Near-Infrared Spectrophotometer Based on the EDFA and an Acoustooptic Tunable Filter

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**A novel light source for the near-infrared region which has the highest intensity and widest spectral bandwidth of all near-IR light sources has been developed. The system is based on a single-mode fiber (about 18 m long) doped with $Er^{3+}$ ion. The doped ion produces amplified spontaneous emission (ASE) in the near-IR region (from 1500 to 1600 nm) when it is excited by a diode laser at 980 nm. Because the diode laser is fusion-spliced directly to the doped fiber, the system is compact, all-solid-state, reliable, and stable and requires little maintenance. Its ASE output intensity was found to be comparable with those of diode lasers currently available for this near-IR region and is much higher than those of conventional halogen−tungsten lamps and the so-called (high-intensity) superluminescent light emitting diodes (SLEDs). Its spectral bandwidth is, however, much wider than those of the diode lasers and the SLEDs. Even higher intensity can be obtained from the doped fiber when a low-intensity (1 mW) light from a 1550-nm laser diode is introduced into the doped fiber. The intensity is enhanced (up to 7 times compared to the ASE) because the input light is amplified by the doped fiber. Furthermore, the output intensity of this erbium-doped fiber amplifier (EDFA) can be appropriately adjusted to provide relatively higher output intensity at any range of wavelengths (within this 1500−1600-nm region) by simply changing the temperature and/or the driven current of the input diode laser. Subsequently, an acoustooptic tunable filter was used to provide a means to spectrally tune the EDFA rapidly and to develop an all-solid-state, compact near-IR spectrophotometer which not only is very sensitive, stable, and reliable but also has a very high throughput. This spectrophotometer can detect water in ethanol at a limit of detection of 10 ppm. More importantly, the high throughput makes it possible to use the instrument to measure spectra of highly absorbing samples (e.g., absorption spectrum of 1.0 M $Pr^{3+}$ aqueous solution through four sheets of paper); measurements which are currently not possible with halogen−tungsten lamp-based spectrophotometers.**

The use of the near-infrared spectrometry in chemical analysis has increased significantly in the last few years.[1−4] The popularity stems from the advantages of the technique: its wide applicability, noninvasiveness, and on-line characteristics. However, application of the technique is not as widely spread as one would expect on the basis of its potential. This may be due to a variety of reasons, but the most likely ones are the limitations on the speed, stability, and light throughput of the currently available instruments. To be effectively used as a detector for on-line measurements, the near-IR instrument needs to have high and stable light throughput, to suffer no drift in the baseline, and to be able to be rapidly scanned. These impose severe limitations on conventional near-IR spectrophotometers, because such instruments generally have relatively low and unstable light throughput, suffer some degree of baseline drift, and can only be scanned very slowly. It will be demonstrated for the first time in this paper that such limitations can be effectively alleviated by synergistic use of an erbium-doped fiber amplifier as a light source and an acoustooptic tunable filter as a dispersive element.

Acoustooptic tunable filter (AOTF) is an all-solid state, electronic dispersive device which is based on the diffraction of light by acoustic wave in an anisotropic crystal.[8−12] Compared to conventional gratings, the AOTFs have such advantages as rapid scanning ability (microseconds), high diffraction efficiency (>90%) and resolution (e.g., 0.8 Å at 253.4 nm[13]), and wide spectral tuning range (from the ultraviolet through the visible and near-IR to the IR).[8−12] The filters can also provide a unique means to maintain the intensity of the light source (by controlling either the frequency or the power of the applied radio frequency (rf) signal through a feed-back loop).[14] As a consequence of this development, a spectrophotometer based on the AOTF not only can be scanned very quickly but also is very stable and suffers no drift in the baseline. Such a spectrophotometer makes it possible, for the first time, to perform various types of measurements including

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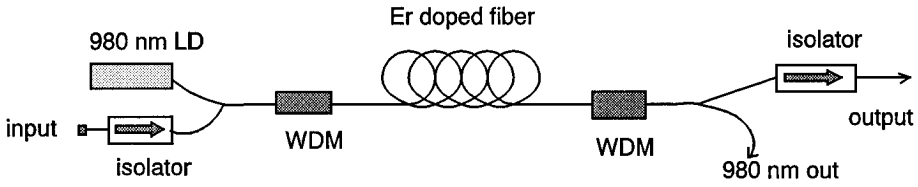


Figure 1. Schematic diagram of the erbium-doped fiber amplifier system: LD, laser diode; WDM, wavelength division multiplexer.

sensitive and fast scanning detection for HPLC[13] and flow injection analysis[15] (i.e., to record entire absorption spectra of compounds (and hence to identify them) when they are eluted from the column of HPLC[14] or the FIA[15]), determination of the inclusion complex formation between cyclodextrins and aromatic compounds,[16] and simultaneous determination of water and benzene in ethanol by flow injection analysis.[17] However, in spite of their advantages, these AOTF-based spectrophotometers still suffer from low light throughput. This is because these instruments are based on the use of halogen−tungsten lamps as light source, and such lamps have relatively low output in the region around and above 1500 nm. This is rather unfortunate, because this region covers the overtone and combination transitions of the O−H and C−H groups,[18] and hence is particularly important to spectrochemical analysis.

It has been shown recently that stimulated emission can be achieved in a fiber when the fiber is doped with rare earth ions such as $Er^{3+}$ and optically pumped by an ion laser or YAG laser.[19,20] Now, for the first time, a lasing medium can be confined in a material as flexible and as small (<5 $\mu$m) as a single-mode fiber.[19,20] Because of such features, the length of the doped fiber can be adjusted to as long as few miles to enable the fiber to have optical output in the range of kilowatts. Undoubtedly, this discovery is of scientific and technology importance. However, its practical applications have not been realized until quite recently. This is partly due to the use of either a large frame ion or a solid-state laser as the pump source. This limitation was alleviated recently when high-power diode lasers became available. Such an all-solid-state, compact, and high-power diode laser can be used as the pump source, and since this type of laser can be pigtailed to a single-mode fiber which, in turn, is fusion-spliced directly into the erbium-doped fiber, the whole fiber laser system is all-solid-state and very compact, required neither a water cooling system nor maintenance, and is much more reliable and stable than the system pumped by an ion laser. This type of diode laser-pumped erbium-doped fiber laser makes it possible, for the first time, to transmit optical signals over land and/or under sea for long distances. In fact, erbium-doped fiber lasers have been used extensively in telecommunications. Furthermore, because of their great potential in the field of telecommunications, scientific and technological advances of the fiber lasers have been moving at an unprecedented speed.

The size, stability, reliability, and high output make the erbium-doped fiber lasers particularly suited as a near-IR light source for spectroscopy. Unfortunately, such an application has not been realized. This may be due to a variety of reasons but the most likely one is the fact that the spectral tuning range of such a laser is very narrow (only about a few namometers[19,20]). Furthermore, to spectrally tune the laser over such a narrow wavelength range, an intracavity dispersive device (e.g., grating) is needed. The installation of such an intracavity device is rather difficult and, undesirably, eliminates some of the advantages of the lasers (e.g., all-solid-state and compactness). This limitation can be alleviated by operating the (diode laser-pumped) erbium-doped fiber not as a laser but as an amplified spontaneous emission (ASE) source.[19,20] This can be accomplished by installing an optical isolator to prevent the reflection (which, when compounding, will lead to oscillating and stimulated emission). Because the light in this case is not the stimulated emission but rather spontaneous emission, the spectral bandwidth of the light output from the fiber can be as wide as several hundred nanometers. Its total power over the entire spectral bandwidth is comparable with that of the (very narrow spectral bandwidth) stimulated emission light and is much higher than those of any other near-IR light sources, including the quartz−tungsten−halogen lamps and the so-called superluminescent light emitting diodes.

The information presented is, indeed, provocative and clearly indicates that it is possible to use the erbium-doped fiber amplifier (EDFA) and the AOTF to develop a novel, compact, all-solid-state, fast-scanning near-infrared spectrophotometer which has no moving part and high and very stable light throughput. Such considerations prompted us to initiate this study, which aims to construct a diode laser-pumped EDFA. The characteristics of this EDFA, namely its output intensity, ispectral distribution, and bandwidth, will be determined and reported in the first part of the paper. Comparison will then be made between this EDFA and both halogen−tungsten lamps and superluminescent light emitting diodes. The second part of the paper is focused on the instrumentation development of the first near-IR spectrophotometer which is based on the use of the EDFA as the light source and the AOTF as the dispersive element. The specifications of the spectrophotometer—specifically, its light throughput, stability, and noise—will be evaluated and compared with those of a spectrophotometer based on a halogen−tungsten lamp. The developed spectrophotometer will then be used for preliminary measurements which include near-IR spectra of highly absorbing samples (e.g., papers) and the purity of a variety of different solvents.

## EXPERIMENTAL SECTION

The schematic diagram of the erbium-doped fiber amplifier is shown in Figure 1. A diode laser was used as the pump laser. This laser, which was purchased from Lasertron (Burlington, MA, Model QLM9S450-007), was pigtailed to an 8-$\mu$m (core diameter) single-mode fiber. A modular laser diode controller (ILX Lightwave Model LDC 3900, equipped with an LCM 39420 current and temperature controller plug-in) was used to drive the laser diode

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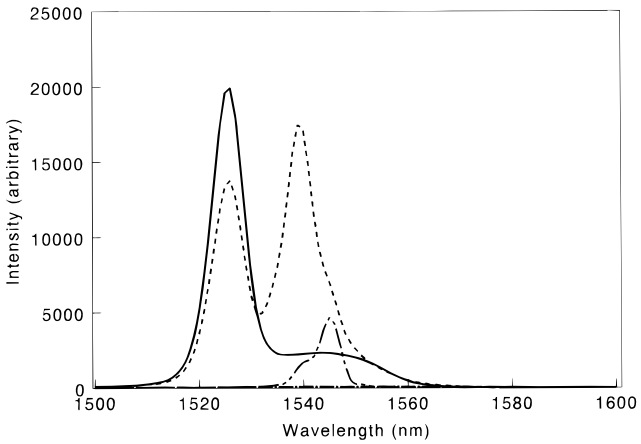


Figure 2. Output intensity as a function of wavelength of the superluminescent light emitting diode (— - —), laser diode operated at −6 °C and 10 mA (— - - —), ASE (—), and amplified light from the EDFA when the output of the LD was introduced into it (- - -).

and to control its temperature. The laser provides 70-mW output power (from the fiber) at 980 nm. The erbium-doped fiber was of the type FGM-P-027-01, obtained from Corning (Corning, NY). The total length of the fiber was 18.65 m. To reduce the size and facilitate handling, the fiber was rolled into a coil with a diameter of about 4 cm. A wavelength division multiplexer (WDM; E-Tek Corp., San Jose, CA, Model SWDMC100PSA10) was used to facilitate the coupling of the 980-nm light from the pump diode laser and the (optional) 1550-nm seed-light from a light emitting diode into the doped fiber. This WDM has two (fiber) inputs and one output. The fiber input, which has a bandpass transmission at 980 nm, was fusion-spliced directly to a fiber of the pigtailed diode laser. The other fiber input, which has a bandpass transmission at 1533−1557 nm, was terminated with a FC/PC connection. Light from a light emitting diode or a laser diode can be introduced to the EDFA system through this connector. The output fiber end of the WDM was fusion-spliced into the doped fiber. A similar WDM was fusion-spliced to the other end of the doped fiber. This WDM enables the isolation of the ASE output light at about 1550 nm and the removal of the 980-nm pump beam. An isolator (E-Tek, Model PIFI21BO44100) was fusion-spliced into the fiber of the WDM which transmits the 1550-nm output beam. This isolator prevents the 1550-nm output beam from reflecting back into the doped fiber, which will eventually lead to oscillation and stimulated emission.

## RESULTS AND DISCUSSION

**1. Characterization of the Erbium-Doped Fiber Amplifier System.** The doped erbium ions become excited when they are excited by the 980-nm light from the pump diode laser. The spontaneous emission is amplified by internal reflection throughout the length of the doped fiber. As expected, the intensity of the light emitted from the fiber is very high because the (erbium) spontaneous emission is amplified. However, because it is not the stimulated emission but rather the spontaneous emission, the spectral bandwidth is very broad. In fact, as illustrated as the solid line in Figure 2, the spectral bandwidth of the amplified spontaneous emission (ASE) is more than 50 nm (from about 1510 to 1564 nm).

To date, semiconductor devices which are widely used as light sources for this near-IR region (i.e., around 1550 nm) include diode lasers (LDs) and superluminescent light emitting diodes (super-LEDs). The former are known for their high output power (but have very narrow spectral bandwidths). While the intensities of the latter are much smaller than those of the LDs, they have much wider spectral bandwidth (and much higher output intensity than conventional LEDs). A study was made to compare the EDFA with these LDs and super-LEDs. Figure 2 shows the output ASE intensity of the EDFA together with those of the 1550-nm, 1-mW pigtailed LD and pigtailed super-LED. This super-LED is the commercially available LED which has the highest intensity in this 1550-nm region. As illustrated, the intensity of the super-LED is much lower than that of the LD. However, its spectral bandwidth is much wider. The ASE of the EDFA has the combined advantages of the LD and super-LED, namely its intensity is about 4 times higher than that of the LD, and its spectral bandwidth is about 25 nm wider than that of the super-LED.

It is possible to achieve amplification for light at wavelength region covered by the ASE when such light is introduced into the EDFA (through the input, as shown in Figure 1). To confirm this possibility, the output of the aforementioned pigtailed 1545-nm, 1-mW LD was connected to the input of the EDFA (through a FC/PC connector). To protect the LD from the reflected (strong) ASE light, an isolator was fusion-spliced between the LD and the EDFA. As illustrated (by the dotted line) in Figure 2, the spectral profile and the intensity of the EDFA changed drastically when light from the LD was introduced into the doped fiber. Specifically, the output intensity of the EDFA in the wavelength region corresponding to that of the LD (i.e., 1545 nm) increased by about 7 times, while there was only a marginal decrease (about 1.4 time) in the intensity of the peak corresponding to the ASE at about 1525 nm. This, in effect, makes the output of the EDFA not only wider but also higher and more evenly distributed over the entire bandwidth. Furthermore, the output intensity of the EDFA is not limited to any single spectral profile but rather is dependent on the intensity profile of the input LD. Because output intensity of the LD is dependent on the driven current and/or temperature, the output intensity of the EDFA should depend on these two factors as well. As illustrated in Figure 3, this indeed is the case. The spectral profile and intensity of the EDFA output can be appropriately adjusted by changing either the driven current (Figure 3A) or the temperature (Figure 3B) of the input LD.

Collectively, the results presented clearly demonstrate that the near-IR light from the EDFA has the highest intensity and widest spectral bandwidth of all other near-IR light sources currently available. Furthermore, its spectral output intensity can be appropriately adjusted to provide relatively higher output intensity at a certain range of wavelengths by simply changing the temperature and/or the driven current of the input diode laser. Its (short- and long-terms) intensity fluctuation was found to be comparable with those of halogen−tungsten lamps. However, its stability can be substantially improved by use of an acoustooptic tunable filter. Furthermore, the AOTF will also provide a unique and synergistic means to develop a compact, all-solid-state, sensitive, and very stable near-IR spectrophotometer which has a very high light throughput. Detailed information on the development of the spectrophotometer and applications of the developed instrument in the determinations of water impurity in organic solvents all described in the next section.

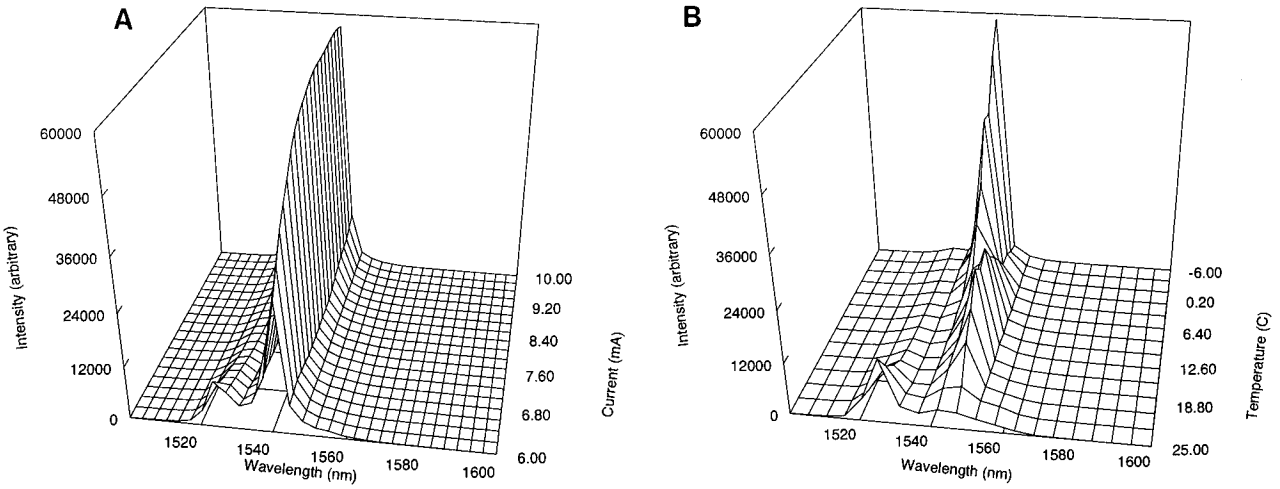


Figure 3. Three-dimensional plots of the output intensity of the EDFA as a function of wavelength and (A) driven current (at constant temperature of −6 °C), and (B) temperature (at constant driven current of 10 mA).

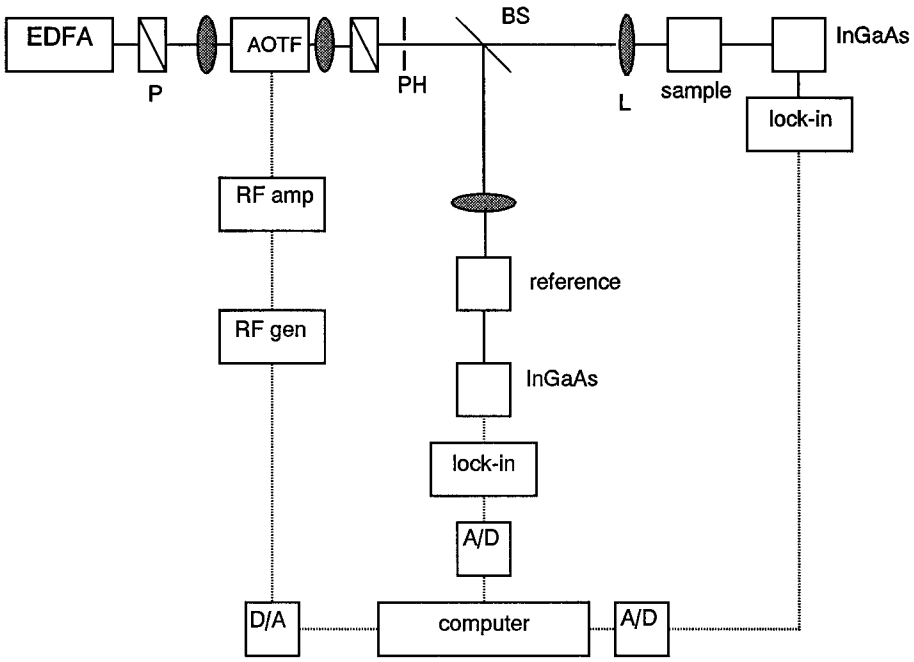


Figure 4. Schematic diagram of the near-infrared spectrophotometer based on the use of the EDFA as a light source and AOTF as a dispersive element: EDFA, erbium-doped fiber amplifier system; P, polarizer; AOTF, acoustooptic tunable filter; PH, pinhole; BS, beam splitter; L, lens; RF amp, radio frequency power amplifier; RF gen, ratdio frequency generator; InGaAs, detector.

**2. Development of a Near-Infrared Spectrophotometer Based on the EDFA and AOTF.** The schematic diagram of the near-IR spectrophotometer based on the use of the EDFA as the light source and an AOTF as a dispersive element is shown in Figure 4. As illustrated, the near-IR light from the output of the EDFA was dispersed to monochromatic light and spectrally scanned by means of a non-collinear AOTF (Crystal Technology Model 97-01457-01) which was fabricated from $TeO_2$. A driver constructed from a voltage control oscillator (VCO)[14−17] provided the rf signal to the AOTF. The rf signal from this driver was amplitude-modulated at 50 kHz by a home-built modulator and amplified by a rf power amplifier (ENI Model 411LA) prior to being applied to the AOTF. The light diffracted from the AOTF was split into two beams (i.e., sample and reference beams) by means of a beam splitter (BS). Intensity of the light in the sample and reference beams was detected by thermoelectrically cooled InGaAs detectors (Epitaxx Model ETX1000GR26TE). Each detector is equipped with a thermoelectric cooler, and a temperature controller (ILX Lightwave Model LDT-5412) was used to cool it down and to keep its temperature constant. The output signals from the (reference and sample) detectors, which were amplitude modulated at 50 kHz, were connected to lock-in amplifiers (Stanford Research Systems Model SR 810) for demodulation and amplification. The signal from the reference beam can be used either as a reference signal for a double-beam spectrophotometer or as a reference signal for the feed-back loop to stabilize the intensity of the light in the sample beam in a manner similar to those used previously.[12,13] The signals from the lock-in amplifiers were then connected to a microcomputer (Gateway P5-60 60 MHz) through a 16-bit A/D interface board (National Instruments Model AT-MIO 16X).

As indicated in the previous section, the output light from the EDFA in the 1511−1564-nm region is particularly useful for the determinations of organic compounds, because it covers the overtones and combination absorption of the O−H and C−H groups. As an example, the EDFA−AOTF spectrophotometer was

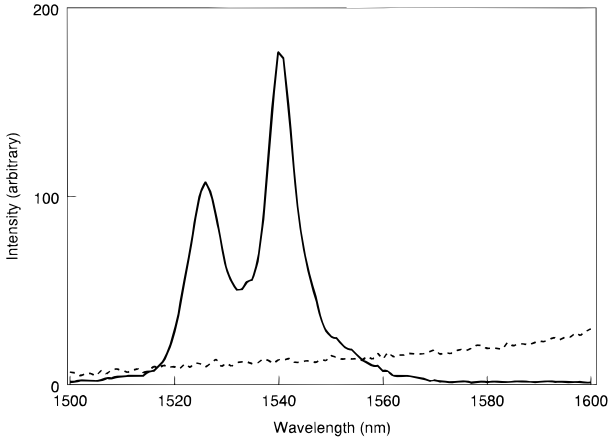


Figure 5. Relative intensity of the light transmitted through six sheets of paper, measured with a 250-W halogen−tungsten lamp-based spectrophotometer (- - -), and with the EDFA-based spectrophotometer (—).

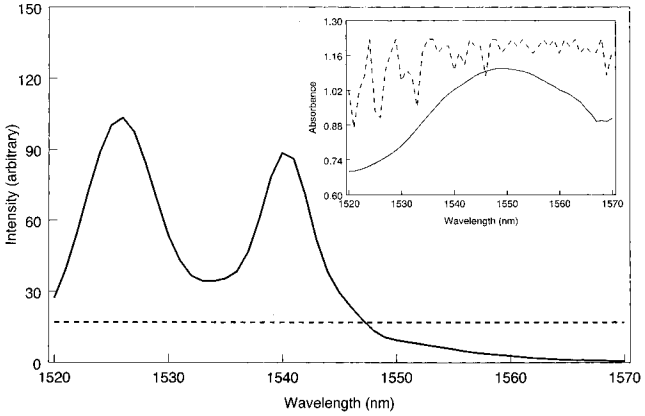


Figure 6. Relative intensity of the light transmitted through four sheets of paper and 1.0 M solution of $Pr^{3+}$ in $D_2O$ (in a 2-mm path length cell), measured with a 250-W halogen−tungsten lamp-based spectrophotometer (- - -) and with the EDFA-based spectrophotometer (—). Inset: spectra of the same sample plotted as absorption spectra.

used to determine the concentrations of water in ethanol. The determination was possible because the $\lambda_{max}$ of the first overtone for the O−H group in water is at 1450 nm, which is different from the value of 1550 nm for the O−H group in ethanol.[17,18,21] Accordingly, absorption spectra of ethanol samples containing different amounts of water were measured, and the spectra obtained were analyzed by the partial least-squares method. It was found that this EDFA−AOTF near-IR spectrophotometer is capable of detecting water in ethanol with a limit of detection of 11 ppm ($\mu$L/L) (at 1540 nm). This LOD value is comparable with the value of 10 ppm previously obtained using an AOTF-based near-IR spectrophotometer equipped with a halogen−tungsten lamp.[17]

The instrument can also be used to determine the concentration of ethanol in hexane. This is because the $\lambda_{max}$ of the overtones of the O−H group is at 1550 nm, which is different from the $\lambda_{max}$ = 1410 nm for the stretching and bending combination transition of the C−H group.[17,18,21] The LOD for this determination was found to be 50 ppm, which is relatively higher than the LOD of 11 ppm for water in ethanol. This is as expected, because in this case, the background signal due to the C−H groups (of ethanol and hexane) is much higher than the background signal for the water−ethanol mixture. In fact, this 50 ppm LOD value is comparable with the LOD value of 51 ppm obtained for similar measurements, i.e., benzene in ethanol.[17]

As expected, the results presented indicate that the sensitivity of this EDFA−AOTF-based spectrophotometer is comparable with those of the halogen−tungsten lamp−AOTF-based instruments. One of advantages of this EDFA−AOTF-based instrument is its high light throughput. The intensity of this EDFA light source is about 20 times higher than that of 250-W halogen−tungsten lamp. As a consequence, it can be used for measurements which are not possible with lamp-based instruments. Two measurements were performed to demonstrate this advantage. Shown in Figure 5 is the intensity of the light transmitted through six sheets of photocopy paper (Cascade X-9000 white paper). As illustrated, because of the high absorption (of the papers) and low intensity (of the halogen−tungsten lamp), no light was transmitted when the halogen−tungsten lamp based spectrophotometer was used. Because of its high intensity, a substantial amount of EDFA light was transmitted through the papers. In the second measurement, which is shown in Figure 6, it was not possible to use a lamp-based spectrometer to measure the absorption spectrum of a 1.0 M solution of $Pr^{3+}$ in $D_2O$ (in a 2-mm cell) placed after four sheets of photocopy paper (dashed line). Because of its high intensity, a substantial amount of EDFA light was transmitted through the papers and $Pr^{3+}$ solution, and as a consequence, the absorption spectrum of the latter can be recorded (Figure 6 inset). This high-throughput advantage is of particular importance in near-IR measurements, because near-IR techniques are often used for measurements in which the signal of interest is very small and riding on top of a very large background signal. As such, it is very difficult, inaccurate, and sometimes not possible to perform such measurements with low-light-throughput spectrophotometers.

In summary, we have successfully demonstrated that the light in the 1500−1600-nm region provided by the EDFA has the highest intensity and widest spectral bandwidth. It is important, however, to realize that (1) the intensity reported here is not the highest intensity which can possibly be obtained from the EDFA system and (2) the spectral region reported is not the only region which can be obtained from an erbium-doped fiber. Higher intensity can be obtained from the EDFA by increasing the power of the 980-nm pump diode laser or by pumping it with more than one diode laser.[19,20] Increasing the length of the doped fiber and the concentration of the doped ion may increase the output intensity. However, it may also produce self-absorption, self-quenching, and saturation, which will decrease the output intensity. The highest output power can be achieved when the length and concentration of the doped ion are appropriately adjusted. Since the present system is far from optimization, higher output power can be achieved when these two factors are determined and used.

With the present configuration (i.e, silica-based, erbium-doped fiber pumped with the 980-nm diode laser), the output wavelength is in the 1500−1600-nm region. Substituting the silica-based EDF with a fluoride-based EDF is known to provide output light in the

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region around 2.75 $\mu$m.[22] Replacing erbium ion with other rare earth ions makes it possible to obtain output light in other regions, including 1.31 $\mu$m (by praseodymium-doped fiber[20,23]), 1.06 $\mu$m (by neodymium-doped fiber[19,20]), 820 and 1480 nm (by thulium-doped fiber[24,25]), and 2 $\mu$m (by holmium-doped fiber[26]). Light in the visible and ultraviolet regions can also be obtained from these rare earth element-doped fiber amplifier systems. This is because it is relatively easy to mode-lock these all-solid-state doped fiber amplifier systems. The output lights from such mode-locked systems have very high peak power and, as a consequence, can be readily and efficiently converted into visible and ultraviolet light by a variety of nonlinear optical techniques (e.g., two- and four-wave mixing, stimulated Raman, and frequency doubling, tripling, and quadrupling[27]).

The unique features of the rare earth element-doped fiber amplifiers (e.g., compactness, all solid state, reliability, and high throughput) make it possible, for the first time, to bring this intense light source in the form of chemically inert and small size single-mode fiber (8 $\mu$m or less) directly to any medium for measurements. As a consequence, smaller medium and lower concentration of samples are needed for measurements which will have very high spatial resolution. It is expected, therefore, that this type of light source will provide a revolutionary and unique means to improve the sensitivity, selectivity, and spatial resolution for a variety of analytical techniques, including spectral chemical analysis (e.g., fiber-optic (chemical, biochemical) sensors), spectroelectrochemistry, chromatography, and electrophoresis.

The EDFA−AOTF-based near-IR spectrophotometer has sensitivity similar to those of halogen−tungsten lamp-based spectrophotometers. Advantages of the former, however, lie in its compactness, all solid state, reliability, low maintenance, and more importantly, its high light throughput. This feature makes this spectrophotometer uniquely suited for on-line, real-time, and in vivo measurements, which currently are not possible using existing spectrophotometers. This is the subject of our current intense investigation.


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