CHAPTER 2
Abstract

This chapter deals with the important aspects of the different experimental techniques presented in this thesis. The amplified fs laser system used throughout the thesis is introduced. The basic experimental set-up describing second-order intensity autocorrelation for measuring the fs pulse-width is explained in detail. Different experimental techniques namely, Supercontinuum generation, four-wave mixing and Z-scan that form the basis of the studies presented in this thesis are explained in detail.
Experimental details and techniques

2.1 Laser system used:

The laser system used in this dissertation is a commercially available diode-pumped mode-locked Ti:Sapphire Laser purchased from Spectra Physics Inc. The laser system consists of (i) oscillator unit producing high repetition rate, nJ, fs pulses, Mai-Tai and (ii) regenerative amplifier producing amplified fs pulse, Spitfire [1]. The pictorial overview of the fs laser facility present at the laser lab of University of Hyderabad is as shown in figure 2.1.

![Diagram of laser facility](image)

**Fig. 2.1:** Overview of the fs laser facility

2.1.1 The fs oscillator [Mai-Tai™ System]

The Mai Tai comprises of two lasers: a cw diode-pumped laser and a mode-locked Ti: Sapphire pulsed laser. As seen in figure 2.1, the laser head has two chambers, a cw pump chamber and a pulsed output chamber. The cw pump chamber contains a diode-pumped, intracavity, frequency doubled, solid-state Nd: YVO4 laser giving 532 nm laser output. The pulsed output chamber contains a mode-locked Ti: Sapphire cavity. Because of Ti: Sapphire’s broad absorption
band in the blue and green, the 532 nm output of the cw laser is an ideal pump source for the Ti: Sapphire laser. The fluorescence band of Ti: Sapphire medium extends from 600 nm to wavelength greater than 1000 nm making it possible for wide broadband tunable laser. The mode locking is achieved by an acousto-optic modulator (AOM) to ensure reliable mode-locked operation when the laser starts up and provides smooth wavelength tunings. It also allows the laser to operate for extended periods without dropouts or shut-downs associated with pure kerr-lens mode locking. The fs pulse laser can be wavelength tuned using a prism sequence and a slit. The prism sequence provides a region in the cavity where the wavelengths are spatially spread, and the slit is placed in the dispersed beam. By changing the position of the slit in the dispersed beam, the output wavelength is tuned.

The pulse-width tunings characteristics of a Ti: Sapphire laser are generally influenced by three factors: those inherent in the Ti: Sapphire material itself, those from cavity parameters, and to a degree, from the wavelength selection. While we cannot readily modify the Ti: Sapphire material to change pulse width, we can modify the net group velocity dispersion (GVD) of the cavity. The optical components in the laser cavity introduce positive GVD and cause temporal pulse spreading. Further pulse spreading is caused by self-phase modulation (SPM) in the Ti: Sapphire rod, which results from the interaction of the short optical pulse with the nonlinear refractive index. In order to obtain stable, short optical pulses, these effects must be compensated with negative GVD. Because positive GVD in the cavity changes with wavelength, the amount of compensating negative GVD must be varied with wavelength. In *Mai Tai*, prism pairs are used to produce a net variable negative GVD in the cavity. This compensation scheme is fully automated and results in an optimized pulse at any chosen wavelength.

*Mai Tai* delivers a ~ 80 fs, 82 MHz pulse train with pulse energy of 1 nJ. The output spectrum with its peak at 800 nm is as shown in figure 2.2.
2.1.2 The fs amplifier [Spitfire]

As the output energy of the oscillator is of the order of nJ, we make use of an amplifying unit, *Spitfire*, to amplify the energy without introducing much temporal broadening. *Spitfire* is a regenerative amplifier (RGA) system that employs the conventional chirped pulse amplification (CPA) technique. The general principle of RGA is as follows: (a) Trap seed pulse in optical resonator, (b) Multipass pulse through the active medium (in our case Ti: Sapphire rod) until amplified to desired energy level, and (c) Switch pulse out. But with such short high energy pulses there is always risk of laser induced damage of optics used for the amplification and other effects like self-focusing. To overcome this difficulty *Spitfire* employs Chirped Pulse Amplification (CPA) technique. The general principle of CPA is as follows: (a) pass the fs seed pulses from the output of the oscillator through a stretching mechanism that broadens the pulse to an order to $10^3$ by introducing chirp using a grating (in most cases); (b) amplify the temporally broadened pulse; and (c) recompress the amplified pulse to its original pulse-width by pass it through a pulse compressor mechanism. The amplification
is done by the RGA cavity described above. Figure 2.3 shows the figurative explanation of the CPA technique used in *Spitfire*

![Fig. 2.3: CPA technique basic principle](image)

As shown in figure 2.1, the regenerative amplifier consisting of a Ti: Sapphire rod is pumped by the output of *Evolution* which is a 125 ns, 1 kHz repetition rate Nd: YLF laser delivering high power laser pulses of 532 nm. The amplification of the seed pulse from *Mai Tai* is obtained as follows:

1. The seed pulse from *Mai Tai* having a pulse-width of ~82 fs is stretched using a combination of grating and mirror to about 250 ps as shown in the top half of the figure 2.1.
2. The temporally stretched pulse is then made to oscillate in the regenerative cavity pumped by the laser pulses of *Evolution* laser head to attain maximum desired energy amplification. The entry and the exit of the seed pulse is achieved by the use of two high-voltage Pockel cells whose delay is adjustable by a Signal Delay generator (SDG) as shown in figure 2.1.
3. The amplified seed pulse exited from the regenerative cavity is then recompressed following the reverse mechanism of stretching procedure to obtain amplified pulses of ~100 fs of a maximum of 1mJ average energy at
a repetition rate of 1kHz. The output of Spitfire is of 9 mm diameter with a spectral bandwidth of 9.4 nm at the peak wavelength operation at 800 nm as shown in figure 2.4.

![Spitfire output spectrum](image)

Fig. 2.4: Spitfire output spectrum

Table 2.1 summarizes the general and most important characteristics of the fs laser system used throughout the present studies in this thesis.

<table>
<thead>
<tr>
<th></th>
<th>Mai Tai</th>
<th>Spitfire</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pulse-width</td>
<td>~82 fs</td>
<td>~100 fs</td>
</tr>
<tr>
<td>Max output energy</td>
<td>1 nJ</td>
<td>1 mJ</td>
</tr>
<tr>
<td>Rep. rate</td>
<td>80 MHz</td>
<td>1 kHz</td>
</tr>
<tr>
<td>Beam diameter</td>
<td>3 mm</td>
<td>9 mm</td>
</tr>
<tr>
<td>Polarization</td>
<td>Horizontal</td>
<td>horizontal</td>
</tr>
</tbody>
</table>

2.2 Pulse-width measurement- The autocorrelation technique

An autocorrelator is the most common set-up used for measuring an femtosecond (fs) or picosecond (ps) optical pulse [2]. By using the speed of light to convert optical path lengths into temporal differences, we use the pulse to measure itself. The basic optical configuration is similar to that of a Michelson interferometer. An incoming pulse is split into two pulses of equal intensity and an adjustable optical delay is imparted to one. The two beams are then
recombined within a nonlinear crystal for second harmonic generation (SHG). The efficiency of the SHG resulting from the interaction of the two beams is proportional to the degree of pulse overlap within the crystal. Monitoring the intensity of SHG as a function of delay between the two pulses produces the autocorrelation function directly related to pulse width. Two types of autocorrelation configurations are possible. The first type, known as interferometric collinear autocorrelation as shown in figure 2.5(a), recombines the two beams in a collinear fashion. This configuration results in an autocorrelation signal on top of a constant dc background, since the SH generated by each beam independently is added to the autocorrelation signal. Alternatively, if the beams are displaced from a common optical axis and then recombined in a noncollinear fashion as shown in figure 2.5(b) the background is eliminated because the SHG from the individual beams is separated spatially from the autocorrelation signal. This configuration is called “background-free” and is employed in the pulse-diagnostics of the amplified fs pulses from Spitfire used for all the experiments presented in this dissertation. In the subsequent section we explain the details of the autocorrelation in the non-collinear geometry.

2.2.1 Experimental details:

While performing the retro-reflection in longer-pulse regime, retro-prisms are usually employed but in the case of fs pulse regime, the retro-prisms are replaced by a pair of mirrors as shown in figure 2.5(b). This is to come over dispersion-effects created within the fs pulses owing to the propagation in the prism-material medium that leads to temporal broadening of the pulses resulting in wrong results. As shown in the figure the incoming beam is first split into two arms and passed through the retro-reflecting arrangement such that the two separated beams follow a parallel path before focusing into a 1mm BBO crystal used for SHG. The retro-reflector on the delay stage is moved with a resolution of 6 μm that corresponds to a minimum temporal resolution of ~ 20 fs. From the experimental configuration the autocorrelation signal (SHG) which is generated as a resultant of temporal and spatial overlap of the two fundamental input beams
is collected into a fast photodiode (FND100) after passing through a SHG filter to cut-off the residual input pulses. Different neutral density filters are used for attenuation to ensure that the photodiode does not get saturated. The photodiode output is fed to a lock-in amplifier (SRS 830) and is finally recorded. The averaged signal is then sent to an interfaced ADC card and then to a computer. The autocorrelation trace is obtained by recording the SHG signal as a function of delay obtained by translation of the stage on which the retro-reflector arrangement is mounted.

![Interferometer autocorrelation set-up](image)

(a) collinear autocorrelation

(b) Non-collinear autocorrelation

Fig. 2.5: Interferometer autocorrelation set-up

### 2.2.2 Signal interpretation:

In order to determine the actual pulse width from the obtained experimental autocorrelation data, it is necessary to make an assumption about the pulse shape. Table 2.2 shows the relationship between the FWHM of the intensity envelope of the pulse ($\tau_p$) and the FWHM of autocorrelation function of the pulse ($\tau_\omega$), for several pulse shapes. It also shows the time-bandwidth, for transform-limited pulses as discussed in chapter 1.
Table 2.2: Second-order autocorrelation functions and time-bandwidth products for various pulse shape models

<table>
<thead>
<tr>
<th>Function</th>
<th>$I(\tau)$</th>
<th>$\frac{\tau_p}{\tau_{ac}}$</th>
<th>$\tau_p\Delta\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gaussian</td>
<td>$I(\tau) = \exp\left[-\frac{(4\ln 2)\tau^2}{\tau_p^2}\right]$</td>
<td>0.707</td>
<td>0.44</td>
</tr>
<tr>
<td>Hyperbolic Secant</td>
<td>$I(\tau) = \sec^2 \left(\frac{1.76\tau}{\Delta\tau_p}\right)$</td>
<td>0.648</td>
<td>0.315</td>
</tr>
<tr>
<td>Lorentzian</td>
<td>$I(\tau) = \frac{1}{1 + \left(4\tau^2/\Delta\tau_p^2\right)}$</td>
<td>0.5</td>
<td>0.221</td>
</tr>
</tbody>
</table>

The autocorrelation trace obtained for the amplified fs pulse generated from *Spitfire* is as shown in figure 2.6. The error bars shown corresponds to an experimental error of ~5% which is within the acceptable limits. Similarly we considered a maximum error of ~5% in the x-axis where the source of error is mainly in the movement of the translational stage.

![Autocorrelation trace](image)

**Fig. 2.6:** Autocorrelation trace obtained for the *Spitfire* amplified fs pulses fitted with Gaussian pulse shape
By performing Gaussian fit to the experimental autocorrelation trace we obtained the FWHM, \( \tau_{ac} \), to be \((148 \pm 7) \) fs. From Table-2.2 we have the relation for Gaussian pulse:

\[
\frac{\tau_p}{\tau_{ac}} = 0.707 \quad \Rightarrow \quad \tau_p = 0.707 \tau_{ac} \quad \Rightarrow \quad \tau_p = (104 \pm 7)
\]

Thus the pulse-width obtained from the second-order intensity correlation is \((104 \pm 7) \) fs. Since the FWHM of the spectral envelope of Spitfire as shown in the previous section is \( \Delta \lambda \sim 9.4 \) nm with its peak at \( \lambda_0 = 800 \) nm.

\[
\tau_p \Delta \nu = \tau_p \frac{c \Delta \lambda}{\lambda_0^2} = 0.458.
\]

Thus we have nearly-transformed limited pulses assuming Gaussian pulse shape.

### 2.3 Calculating pulse broadening due to GVD:

Because the pulses produced by the Spitfire are \(~ 100 \) fs, as it propagates through optical materials, the pulse get temporally broadened due to the group-velocity dispersion (GVD) discussed in the previous chapter. Thus while a making intensity calculation which depends on the pulse-width it is important to estimate the pulse broadening due to the GVD as it propagates though various optical components before actually reaching the sample. Below is some simple formulae for calculating the effects of GVD and compensation. B (broadening) is defined as the ratio defined as the ratio of the output pulse-width to the input pulse-width (in terms of fs) i.e.,

\[
B = \frac{\tau_{out}}{\tau_{in}} \Rightarrow \tau_{out} = B \times \tau_{in}
\]

For a transform-limited guassian pulse: \( B = \left\{ 1 + \left[ 7.68 \left( k_2 \times \frac{L}{\tau_{in}^2} \right)^2 \right] \right\}^{1/2} \), where \( k_2 \) (in terms of \( \text{fs}^2/\text{cm} \)) is the second order dispersion coefficient. Table 2.3 gives the values of \( k_2 \) for different standard materials at 800 nm.
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Table 2.3: Dispersion values of different materials at 800 nm

<table>
<thead>
<tr>
<th>Material</th>
<th>$k_2$ (fs$^2$/cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fused silica</td>
<td>300</td>
</tr>
<tr>
<td>BK-7 glass</td>
<td>450</td>
</tr>
<tr>
<td>Ti: Sapphire</td>
<td>580</td>
</tr>
<tr>
<td>KDP crystal</td>
<td>25 ($o$-ray); 38 ($e$-ray)</td>
</tr>
<tr>
<td>SF-10</td>
<td>1590</td>
</tr>
</tbody>
</table>

2.4 Details of the Experiments presented in the dissertation:

2.4.1 Supercontinuum generation:

In a typical SCG experiment [3], the beam is focused into the transparent media under consideration and the generated continuum is detected using a fiber coupled CCD spectrometer (Ocean Optics - SD2000). As shown in figure 2.7, the output of the amplified laser pulses is first passed through a variable aperture to control the input beam diameter. Various neutral density filters are made use to attenuate the input power. It should be noted here that the control over the input power of the incident laser beam is important as from the definition of the critical power of self-focusing ($P_{cr}$) depends only on the input peak power and not on the overall input peak intensity. Upon focusing the incident laser beam of suitable input power into the transparent media supercontinuum is generated as a conical emission. With increase in input power the emission becomes brighter until the media experiences laser induced damage. The laser beam depicted as red line focusing into the media and the supercontinuum output is depicted as yellowish-white conical emission to depict broadband output. The use of a large aperture lens is dual-purpose: (a) to collect all the conical emission and (b) either
collimate the output for further study of SCG properties or focus it onto a screen to record the spectrum with a spectrometer.

**Fig. 2.7.** Experimental schematic for supercontinuum generation in transparent bulk media

The spectra of continuum are recorded using a fiber coupled spectrometer (Ocean Optics USB2000) after collimation and suppressing fundamental by an IR filter thus limiting our study of SC to the visible region (400 – 780 nm). To avoid saturation of the detector used in the spectrometer the continuum was focused onto a white board and the scattered light was collected by placing the fiber tip very close to the board. The choice of the screen is crucial as the material of the screen used (for example, white paper) can absorb a part of broadband SCG emission and produce its own fluorescence emission that gets eventually added to the actual SCG spectrum while recording the spectrum with the spectrometer. The detector used in the spectrometer was a silicon charge coupled device that has a range from 350-800 nm with maximum sensitivity at 500 nm. Beyond 750 nm there is a monotonic decrease in the detector sensitivity. The fiber used for collection has excellent transmission for the range 300-900 nm. Neutral density filters with known absorbance spectra in the region was used to collect the continuum spectra obtained at high intensities. Taking into account the absorbance spectra of the filters used, the resultant spectra thus obtained was
corrected. The integrated intensity measurements of the SC are measured by focusing onto a photodetector (FND100). The laser power that was incident on the material under study was measured using a power meter (OPHIR) with a nearly flat and wide spectral response. The same power meter was also used to measure the power of the entire white light continuum after attenuation of the fundamental through an IR filter.

2.4.3 Degenerate Four Wave Mixing (DFWM)

A Four Wave Mixing experiment [4] can be considered as an interaction of three optical fields in a medium leading to the generation of fourth field, via third order polarization. The presence of a third-order optical nonlinear susceptibility $\chi^{(3)}$ leads to the creation of various components of material polarization, giving rise to new optical fields. If the phase-matching condition is fulfilled (i.e. the phase relation between the waves emitted by different parts of the nonlinear medium leads to constructive build up of the resulting wave), new beams of light are created. If the fields are of identical frequencies, the process is called Degenerate Four Wave Mixing and the output beam will have the same frequency. The time resolution of the FWM measurements depends on two parameters. The first is related to the time duration of the laser pulses and the second is related to the coherence time of the laser pulses. DFWM provides information about the magnitude and response of the third-order nonlinearity. In this process, three coherent beams incident on a nonlinear medium generate a fourth beam due to the third order nonlinearity. The strength of this fourth beam is dependent on a coupling constant that is proportional to effective $\chi^{(3)}$ and hence measurements on observed signal will yield information about the $\chi^{(3)}$ tensor components of the medium. DFWM can be employed in backward (or generally called the Phase Conjugate), forward or boxcar configurations, with the choice on the experimental conditions and the requirements. Using different polarizations of the three beams it is possible to measure all the independent $\chi^{(3)}$ tensor components of a material. Though the backward geometry is the most popular
for DFMW measurements, in the case of femtosecond pulses the box-car geometry is preferred.

**Box-car geometry:**

The basic principle of the method is as follows: Two synchronized pulses (called the $k_1$ and $k_2$ pulse) propagating in two slightly different directions ($k_1$, $k_2$) interfere in the sample to form a grating by spatially modulating its optical properties. Depending on the experimental conditions, the absorption (amplitude grating), or the refractive index (phase grating) can be modulated. After the laser pulse interaction, the amplitude of the modulation decreases: the dynamics of the modulation (i.e. the grating) will depend on the population lifetime (for amplitude grating) and/or the dynamics of the changes in the refractive index (for phase grating). A third beam, $k_3$, propagating in direction $k_3$, is sent into the sample and is diffracted by the grating in directions $k_4$, which satisfies the conservation of momentum: $k_4 = k_3 - k_2 + k_1$.

The experimental schematic of the DFWM set-up used in our studies is as shown in the figure 2.8. Since all the pumps are in same plane, the three beam form three corners of the square and the signal is seen at the fourth corner of the square. The delays of beams 2 and 3 could be varied using the stepper motor. Observing and optimizing the diffracted signals, in both horizontal and vertical directions, ensured the zero delay of different beams. The signal(s) obtained are spatially filtered before being fed to the fast PD to lock-in amplifier and to ADC which finally gets recorded in the computer. In the set-up a lens of focal length 400mm ($f=50$mm) is used to focus the three beams into the sample. For the relative measurements of $\chi^{(3)}$ of the samples under study we used pure CCl$_4$ solvent in case of samples in solution and an optically polished fused silica slide for solid samples. Other details related to the specific experiments carried out in the thesis work are explained in the subsequent chapters appropriately.
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Fig. 2.8. Experimental schematic for femtosecond Degenerate Four wave mixing set-up in box-car configuration. The inset shows the wave-vector representation of the geometry of the DFWM experiment. Beams 1-3 are coincident on the sample. The resultant fourth beam is the DFWM signal that occurs because of the interaction $k_4 = k_3 - k_2 + k_1$. The lower panel of the figure shows formation of the fourth beam at the vacant corner of the box formed by the interacting three waves.

The time-resolved DFWM profiles were obtained by delaying beam 3 with respect to the other two beams with a time resolution of ~33 fs. Since we used 100 fs input pulses we used CCl$_4$ solvent taken in a 1 mm cuvette as a standard sample for relative studies. CCl$_4$ is known to show only the electronic response (~few fs) and thus the time-resolved signal obtained can be treated as an autocorrelation trace of the input fundamental pulses. Autocorrelation trace for
CCl4 is shown in figure 2.9. Open circles are the experimental data and the solid line is the fit assuming the Gaussian profile of the pulse and has the FWHM ~ 145 fs similar to the FWHM of the autocorrelation trace obtained earlier with second order intensity correlation.

![Fig. 2.9: Autocorrelation trace of CCl4](image)

Non-collinear two beam geometry:

The schematic of a non-collinear two beam four-wave mixing (FWM) is as shown in figure 2.10. As shown in the figure the fundamental beam is split into two beams of almost equal intensity and they are brought back to form parallel beams before focusing into the sample. The FWM geometry is such that if the pump beams are represented by the wave vectors $k_1$ and $k_2$, the generated phase matched FWM signals are at $2k_1 + k_2$ and $k_1 + 2k_2$ respectively, and appear well separated from the fundamentals $k_1$ and $k_2$. As denoted in the figure the FWM signals do not have the same frequency as that of the fundamental and hence this geometry is called non-degenerate four-wave mixing. A more detailed analysis and the utility of this geometry is presented in the studies presented in chapter-6.
Fig. 2.10: Schematic of a femtosecond non-collinear two beam four wave mixing set-up

Fig. 2.11: Schematic of a femtosecond z-scan set. (a) closed aperture; (b) open aperture
2.4.4 Z-scan

The Z-scan technique is a single beam technique, which allows the determination of the real and imaginary parts of the third order susceptibility [5]. This technique is a simple, sensitive, single beam method that uses the principle of spatial beam distortion to measure both the sign and the magnitude of refractive nonlinearities of optical materials. The experiment uses a Gaussian beam from a laser in tight focus geometry to measure the transmittance of a nonlinear medium through a finite aperture in the far field as a function of the sample position Z, from the focal plane. In addition to this, the sample transmittance without an aperture is also measured to extract complementary information about the absorptive nonlinearities of the sample.

Closed-aperture Z-scan for sign and refractive nonlinearity

Consider, for instance, a material with a negative nonlinear refraction and of thickness smaller than the diffraction length ($\pi \omega_0^2 / \lambda$) of the focused beam being positioned at various positions along the Z-axis as shown in figure 2.11(a). This situation can be regarded as treating the sample as a thin lens of variable focal length due to the change in the refractive index at each position ($n = n_0 + n_2 I$). When the sample is far from the focus and closer to the lens, the irradiance is low and the transmittance characteristics are linear. Hence the transmittance through the aperture is fairly constant in this region. As the sample is moved closer to the focus, the irradiance increases inducing a negative lensing effect. A negative lens before the focus tends to collimate the beam. This causes the beam narrowing leading to an increase in the measured transmittance at the aperture. A negative lens after the focus tends to diverge the beam resulting in the decrease of transmittance. As the sample is moved far away from the focus, the transmittance becomes linear in Z as the irradiance becomes low again. Thus the curve for Z versus transmittance has a peak followed by a valley for a negative refractive nonlinearity. The curve for a positive refractive nonlinearity will give rise to the opposite effect, i.e. a valley followed by a peak.
Open-aperture Z-scan for absorptive nonlinearity

In the above discussion a purely refractive nonlinearity was considered assuming that absorptive nonlinearities are absent. The presence of multi-photon (two or more) absorption suppresses the peak and enhances the valley, while saturation of absorption produces the opposite effect. The sensitivity of the experiment to refractive nonlinearities is entirely due to the aperture. The removal of the aperture will make the Z-scan sensitive to absorptive nonlinearities alone. Thus by doing the Z-scan with and without aperture both the refractive and absorptive nonlinearities of the sample can be studied. The schematic of an open aperture Z-scan is as shown in figure 2.11(b). Spatially filtered input beam is focused using a lens. In the case of fs pulse excitation we used an achromatic doublet of focal length 120 mm. Usage of achromatic doublet for focusing the fs pulses is essential to overcome the chromatic aberration at the focus owing to the large bandwidth of the input spectrum when compared to ns or ps pulses. The sample is scanned across the focus using a stepper motor controlled by PC. The transmitted light is then collected using another lens (large area) of f ~ 100 mm and fast photodiode (FND100). Different neutral density filters are used for attenuation to ensure that the photodiode does not get saturated. The photodiode output is fed to a lock-in amplifier or a boxcar averager/gated integrator and is finally recorded. The averaged signal is then sent to an interfaced ADC card and then to a computer.

2.5 References

1. Spectra-Physics Inc. operation manuals for Mai-Tai™ and Spitfire™.