Experimental techniques and theory

Abstract

Z-scan and degenerate four wave mixing (DFWM) are the two specific experimental techniques used for nonlinear optical measurements presented in this thesis. Details of these two techniques are described in this chapter along with relevant theory. Besides, the specifications and characteristics of the laser sources and those of other instruments utilized for the measurements are also included in this chapter.
1. Z-scan technique

Z-scan technique was originally introduced by Sheik Bahae et. al [1,2]. This is a simple and sensitive single beam technique to measure the sign and magnitude of both real and imaginary part of third order nonlinear susceptibility, $\chi^{(3)}$. In the original single beam configuration, the transmittance of the sample is measured, as the sample is moved, along the propagation direction of a focussed gaussian laser beam. A laser beam propagating through a nonlinear medium will experience both amplitude and phase variations. If transmitted light is measured through an aperture placed in the far field with respect to focal region, the technique is called closed aperture Z-scan experiment [1,2]. In this case, the transmitted light is sensitive to both nonlinear absorption and nonlinear refraction. In a closed aperture Z-scan experiment, phase distortion suffered by the beam while propagating through the nonlinear medium is converted into corresponding amplitude variations. On the other hand, if transmitted light is measured without an aperture (in this case the entire light is collected), the mode of measurement is referred to as open aperture Z-scan [2]. In this case, the throughput is sensitive only to the nonlinear absorption. Closed and open aperture Z-scan graphs are always normalized to linear transmittance i.e. transmittance at large values of $|z|$. Closed aperture Z-scan and open aperture Z-scan experiments respectively yield the real and imaginary parts $\chi^{(3)}$ [2]. Usually closed aperture Z-scan data is divided by open aperture data both measured simultaneously, to cancel the effect of nonlinear absorption contained in the closed aperture measurement [2]. The new graph, called divided Z-scan graph, contains information on nonlinear refraction alone. In a Z-scan measurement, it is assumed that sample is thin, i.e. the sample length is much less than Rayleigh’s range $z_0$, $z_0 = k\omega_0^2/2$ where $k$ is the wave vector and $\omega_0$ is the beam waist. This is essential to ensure that beam profile does not vary appreciably inside the sample. Experimental setup for single beam Z-scan technique is given in fig.1. Photodetector (PD1) monitors the input laser energy. PD2 and PD3 give open and closed aperture measurements respectively.
Different variants of this technique such as eclipsing Z-scan [3] and two colour Z-scan [4, 5] have also been introduced. In non degenerate (two colour) Z-scan, the effect of nonlinear refraction and absorption induced by a strong excitation beam at a frequency \( \omega_e \) on a weak probe beam at a different frequency \( \omega_p \) i.e. \( \Delta n(\omega_e, \omega_p) \) and \( \Delta \alpha(\omega_e, \omega_p) \), are measured. [\( \Delta n \) refers to the change in refractive index and \( \Delta \alpha \) refers to the change in absorption coefficient]. Non degenerate Z-scan technique has some advantages over conventional single beam technique [4]. The frequency difference \( (\omega_p - \omega_e) \) can be exploited to get information about the dynamics of the nonlinear response with time resolution much less than the laser pulse width. With two colour Z-scan technique, it is possible to make time resolved measurements by suitably delaying the probe pulse with respect to pump beam. Investigation of non-degenerate nonlinearity has technological importance in the area of dual wavelength all optical switching applications, where cross phase modulation is very important [4]. In eclipsing Z-scan technique, the far field aperture is replaced with an obscuration disk [3], which blocks most of the beam. This modification of the Z-scan technique enhances sensitivity to induced wave front distortion to an order of \( \lambda / 10^4 \). Two colour eclipsing Z-scan technique has also been suggested [6]. Measurements presented in this thesis have been made using single beam open aperture Z-scan technique.
Chapter 2. Experimental....

Z-scan technique is highly sensitive to the profile of beam and also to the thickness of the samples [1,2,7]. Any deviation from the gaussian profile of the beam and also from thin sample approximation will give rise to erroneous results. Therefore, some modifications have been suggested to overcome these disadvantages. One such modification is the use of non apertured multi channel Z-scan method, where the total far field beam profile distortion is recorded using a two dimensional CCD camera [8,9]. By image processing, the distorted beam for a given cell position is compared with undistorted beam (i.e. in the absence of nonlinearity). The module of the intensity variation over each pixel of CCD camera is then summed to define the total profile distortion signal (TPDS), which is proportional to nonlinear phase shift. This method has some advantages [9] like (a) the entire far field distribution of both signal and reference beams can be recorded simultaneously (b) after data acquisition the image recorded can be digitized and stored in a computer and the image is ready for further processing and analysis and (c) the dynamic range of a CCD detector is very high. Z-scan measurements can also be made using astigmatic gaussian beams [10] and top hat beams [11]. In the case of astigmatic gaussian beam, a slit is used instead of a (circular) aperture [10]. Z-scan measurement for non gaussian beams with arbitrary sample thickness [12] and arbitrary aperture [13] have also been suggested. In the case of the former, nonlinear parameters are measured in comparison with a standard sample, mostly CS$_2$[11].

1.1. Open aperture Z-scan

Nonlinear absorption of a sample is manifested in the open aperture Z-scan measurements. For example, if nonlinear absorption like two-photon absorption (TPA) is present, it is manifested in the measurements as a transmission minimum at the focal point [2]. On the other hand, if the sample is a saturable absorber, transmission increases with increase in incident intensity and results in a transmission maximum at the focal region [14-16]. It has been shown that the model originally developed by Bahae et. al for pure TPA can also be applied to excited state
absorption (ESA) [17]. ESA is a sequential TPA process, where two photons are successively absorbed [18]. However, in this case nonlinear absorption coefficient $\beta$ is renamed $\beta_{\text{eff}}$, the details of which are discussed in chapter 3.

1.2. Theory of open aperture Z-scan technique

In the absence of an aperture, transmitted light measured by the detector, in a Z-scan experiment, is not sensitive to phase variations of the beam and hence it can be neglected. The theory of Z-scan experiment given here is same as that in [2]. Only transmitted intensity need to be considered. The intensity dependent nonlinear absorption coefficient $\alpha(I)$ can be written in terms of linear absorption coefficient $\alpha$ and TPA coefficient $\beta$ as [2]

$$\alpha(I) = \alpha + \beta I$$  \hspace{1cm} (2.1)

The irradiance distribution at the exit surface of the sample can be written as

$$I_{r}(z, r, t) = \frac{I(z, r, t)e^{-\alpha l}}{1 + q(z, r, t)}$$ \hspace{1cm} (2.2)

where

$$q(z, r, t) = \beta I(z, r, t)L_{\text{eff}}$$ \hspace{1cm} (2.3)

$L_{\text{eff}}$ is the effective length and is given, in terms of sample length $l$ and $\alpha$ by the relation

$$L_{\text{eff}} = \frac{(1 - e^{-\alpha l})}{\alpha}$$ \hspace{1cm} (2.4)

The total transmitted power $P(z, t)$ is obtained by integrating eq. (2.2) over $z$ and $r$ and is given by
Chapter 2. Experimental...

\[
P(z, t) = P(t) e^{-\alpha t} \frac{\ln[1 + q_0(z, t)]}{q_0(z, t)}
\]  \hspace{1cm} (2.5)

\(P(t)\) and \(q_0(z, t)\) are given by the equations (2.6) and (2.7) respectively.

\[
P(t) = \frac{\pi \omega_0^2 I_0(t)}{2}
\]  \hspace{1cm} (2.6)

\[
q_0(z, t) = \frac{\beta I_0(t) L_{\text{eff}} z_0^2}{z^2 + z_0^2}
\]  \hspace{1cm} (2.7)

For a pulse of gaussian temporal profile, eq.(2.5) can be integrated to give the transmission as

\[
T(z) = \frac{C}{q_0 \sqrt{\pi}} \int_{-\infty}^{\infty} \ln[1 + q_0 e^{-t}] dt
\]  \hspace{1cm} (2.8)

Nonlinear absorption coefficient is obtained from fitting the experimental results to the eq. (2.8).

If \(|q_0| < 1\), the eq. (2.8) can be simplified as

\[
T(z, S = 1) = \sum_{m=0}^{\infty} \frac{[-q_0(z, 0)]^m}{(m+1)^2}
\]  \hspace{1cm} (2.9)

Saturable absorption (SA) occurs when a sample is excited at its resonant wavelengths. In this case, as indicated earlier, absorption in the sample decreases as input intensity is increased. Therefore, nonlinear absorption coefficient may be considered as negative. SA takes place because of the depletion of the ground state population. This type of phenomenon is characterized by a parameter called
Z-scan and DFWM studies in certain photonic materials

saturation intensity $I_s$ [15]. In such cases, we cannot directly apply the eq. (2.8), which was derived for TPA. The simplest model to explain SA is a two-level model [16]. Assuming that SA occurs due to depletion of ground state population, steady state can be expressed by the equation

\[ \frac{dN}{dt} = \frac{\sigma I}{h\nu} \left( N_g - N \right) - \frac{N}{\tau} = 0 \]  

(2.10)

Here $N$ is the concentration of the excited state molecules, $N_g$ is the undepleted ground state concentration, $\sigma$ is the absorption cross section, $h\nu$ is the photon energy and $\tau$ is the life time of the excited state. Absorption coefficient $\alpha$ is proportional to ground state population we can be written as

\[ \alpha = \sigma \left( N_g - N \right) \]  

(2.11)

In presence of SA, intensity dependent absorption coefficient $\alpha(I)$ can be written as

\[ \alpha(I) = \alpha_0 \left( \frac{1}{1 + \frac{\sigma I}{h\nu}} \right) = \alpha_0 \left( \frac{1}{1 + \frac{1}{I_s \frac{h\nu}{\sigma I}}} \right) \]  

(2.12)

where $h\nu/\sigma \tau = I_s$, the saturation intensity.

1.3. Closed aperture Z-scan

Closed aperture Z-scan is an example of self-refraction phenomenon or self phase modulation in space. In the absence of nonlinear absorption, a well-defined peak and valley are observed [1,2]. If the nonlinear refractive index $n_2$ of the sample is negative, the beam gets converged in the pre-focal region to get focused closer to the aperture. Consequently, the beam diameter decreases near the aperture, resulting in
large amount of throughput at the detector. This results in a peak in the pre focal region. In the post focal region, the same phenomenon results in the divergence of the beam, which results in the decreased transmission through the aperture. Hence, a valley appears in the post-focal region. If the sample has positive nonlinear refraction, we have just the opposite result (pre focal valley and post focal peak.). The former is called self-defocusing and the later is called self-focussing. One of the mechanisms of self-focussing is optical Kerr effect [19], which has instantaneous response. In this case the electric field of a light beam exerts a torque on anisotropic molecules by coupling to oscillating dipole induced in the molecule by the field itself. Resulting light induced molecular reorientation is the main mechanism for optical nonlinearity in transparent liquids. Nonlinear refractive index depends linearly on light intensity. The other mechanism of optical Kerr effect includes off resonant excitation of narrow band absorbers and consequent distortion of electronic distribution among energy levels in the materials. The resultant intensity dependant refractive index is responsible for self-focussing or self-defocusing. In self-focusing beam collapses upon itself spatially. Kerr like nonlinearity has very fast response time, of the order of picoseconds.

All the measurements mentioned in this thesis have been made in samples taken in solution form under nanosecond excitation. Therefore closed aperture measurements, in the present experimental conditions, can give only thermal response [20,21], which is not of interest in the scope of the present work. Therefore closed aperture Z-scan measurements were not attempted in detail. It may also be noted that the theory of closed aperture Z-scan experiments developed for Kerr type nonlinearity is not applicable for thermal nonlinear refraction.

2. Degenerate four-wave mixing (DFWM)

Four wave mixing refers to interaction of three input waves in a nonlinear medium through the nonlinear polarization corresponding to $\chi^{(3)}(\omega; \omega_1, \omega_2, \omega_3)$, to produce a
Z-scan and DFWM studies in certain photonic materials

fourth wave, at the resultant frequency and wave vector of the input fields [22]. If the frequencies of all the waves are the same (their wave vectors can be different) it is called DFWM. This is a sensitive interferometric method to determine magnitude of \( \chi^3(-\omega; \omega, \omega, -\omega) \). In the present case, standard back scattering geometry of DFWM, which gives rise to optical phase conjugation, is used to measure nonlinear coefficients \( \chi^{(3)}(-\omega; \omega, \omega, -\omega) \). Schematic diagram of DFWM is shown in fig.2.

![Schematic diagram of DFWM](image1)

**Fig. 2. Schematic diagram of DFWM**

P\(_f\), forward pump beam; P\(_b\), backward pump beam; P, probe beam

S, OPC signal; \( \theta \): angle between pump and probe beams

![Experimental setup of DFWM](image2)

**Fig. 3. Experimental setup of DFWM**

BS: Beam splitters; PD: Photo-detectors; M: Mirrors; \( \theta \): Pump-probe beam angle
In the experimental setup, two beams of equal intensity generally called forward and backward pump beams, are made exactly counter propagating. Therefore, sum of their wave vector is zero \((k_r + k_b = 0)\). The actual experimental setup for DFWM is given in fig.3. A third beam (called probe beam), which has an intensity less than those of the pump beams, make a small angle with respect to one of the pump beams (in the present case \(\approx 8^\circ\)) as shown in the fig. 3. Pump-probe intensity ratio was about five. The generated fourth wave is called phase conjugated beam, because its amplitude remains everywhere the complex conjugate of the probe beam. From phase matching condition \((\Delta k = 0)\) it can be seen that wave vector of the phase conjugate beam \(k_c = -k_p\), i.e. phase conjugate light retraces the path of the probe beam.

![Fig. 4. a](image)

**a. Reflection (ordinary mirror)**

![Fig. 4. b](image)

**b. Reflection (phase conjugate mirror)**

It is to be noted that in a phase conjugate reflection, the wave vector of the incident beam is totally reversed, whereas in an ordinary reflection, only normal component of the wave vector component is reversed. Thus phase conjugate reflection does not obey the Snell’s law. Reflection from a phase conjugate mirror and that from an ordinary mirror are illustrated in the fig.4 [22, 23].
Generation of phase conjugate light can be understood as diffraction of one of the input beams from a grating formed by interference of the other two input beams [24-27]. Grating essentially means spatial (or temporal) modulation of certain properties of the medium. Therefore, DFWM is also called real time holography (simultaneous writing and reading of the hologram). In an isotropic medium the vector form of nonlinear polarization is by [24]

$$\vec{P}_{nl} = \frac{1}{2} \left[ a(\vec{E}_f \cdot \vec{E}_p^*) \vec{E}_b + b(\vec{E}_b \cdot \vec{E}_p^*) \vec{E}_f + c(\vec{E}_f \cdot \vec{E}_b) \vec{E}_p^* \right] + \text{cc...} \quad (2.13)$$

The first term corresponds to the static grating formed by interference of forward pump beam (P₁) and probe beam (P). Backward pump beam (P₂) is Bragg diffracted from this grating. This is called transmission grating (fig.5.a). The second term corresponds to the static grating formed by the interference of backward pump beam (P₂) and probe beam (P).

![Fig. 5](image)

a. Transmission grating  
b. Reflection grating

The forward pump beam (P₁) appears to be reflected from this grating and hence this grating is called reflection grating (fig.5.b) The grating vector for transmission
Chapter 2. Experimental....

grating and reflection grating are given by \( (k_f - k_p) \) and \( (k_b - k_p) \) respectively. These two vectors are orthogonal. Hence, only one of them will dominate. The last term corresponds to a temporally modulated grating (at frequency \( 2\omega \)), which is stationary in space. The probe beam is diffracted from this grating. This grating is free from wash out effects. It is also to be noted that temporal grating does not have a holographic analogue. The eq. (2.14) gives the grating spacing [24] in terms of the wavelength \( \lambda \), refractive index of the medium \( n \) and the angle between the beams \( \theta \).

\[
\Lambda = \frac{2\pi}{k_G} = \frac{\lambda}{2n \sin(\theta/2)}
\]  

(2.14)

Periodic modulation of any material property can be considered as a grating. Nature of the grating formed depends on the characteristics of medium as well as those of interacting fields. The gratings formed as a result of the interference of the beams may be classified into a number of categories like population grating [28], orientational grating [29], thermal grating, polarization grating [26,27] etc. Population grating is formed in an absorptive medium due to electronic excitation of the molecules. Orientational gratings are formed by induced reorientation of anisotropic molecules in presence of electromagnetic fields. Absorption of light and subsequent nonradiative de-excitation results in temperature rise, which leads to the formation of thermal grating [26, 27].

An advantage of DFWM is that we can select a particular susceptibility component of \( \chi^{(3)} \) by suitably choosing the polarizations of input fields [24]. In the context of this thesis, we are interested in measuring the pure electronic contribution to nonlinearity. Therefore, polarizations of the interacting beams were adjusted such that the two pump beams are s-polarized (vertical polarization) and the probe beam is p-polarized (horizontal polarization). In this configuration a polarization grating is formed in the sample and \( \chi^{(3)} \) component measured is \( \chi^{(3)}_{xyyx} \). It is also to be noted that, while
formation of polarization grating, orientational grating etc. solely depends on local field (local response), thermal grating and the gratings formed in the photorefractive effect have nonlocal response [30].

Apart from back scattering geometry of DFWM, there are many other configurations for DFWM. Folded boxcar geometry and forward scattering geometry are a few of them [25].

2.1. Theory of DFWM

The theory of degenerate four wave mixing is based on coupled mode equations, which govern the spatial and temporal evolution of the signal under slowly varying amplitude approximation (SVAP) [23,25,31]. Using SVAP, second order Maxwell’s equations can be reduced to first order differential equation. Practically, this approximations implies that growth of the signal with a particular frequency and wave vector, is determined only by the nonlinear polarization having the same modulation frequency and wave vector. This follows from the fact that power transferred to electric field is given by the volume integral of E & P. If E & P do not have the same frequency and wave vector, their volume integral will vanish because they are orthogonal functions. Mathematically SVAP is given by the inequality

\[ \left| k^2 E \right| >> \left| k \frac{\partial E}{\partial z} \right| >> \left| \frac{\partial^2 E}{\partial z^2} \right| \]

(2.15)

It is assumed that two pump beams are of equal intensity and there is no pump depletion: i.e. \( |E_1| = |E_2| = \) a constant. Therefore probe beam and phase conjugate beam are only considered. Nondestructive buildup of the signal occurs when nonlinear polarization (the source term in Maxwell’s equation) is given by the relation
Chapter 2. Experimental....

\[ P_i(\omega = \omega + \omega - \omega) = 6\chi_{ijkl}(-\omega, -\omega, \omega, -\omega)E_{ij}(\omega)E_{2k}(\omega)E_{p}^{*}(\omega)\exp[-i(K_1 + K_2 - K_p)\cdot R] \]

(2.16)

The interaction between probe beam and phase conjugate beam are given by

\[ \frac{dE_p}{dz} = -i\kappa E_c^{*} \quad \frac{dE_c}{dz} = i\kappa E_p^{*} \]

(2.17)

where \( \kappa \) is the coupling constant, given by eq. (2.18)

\[ \kappa^{*} = (2\pi\omega/cn)\chi^{(3)}E_1E_2 \]

(2.18)

The solution to above equations are given by

\[ E_c(0) = -i \left[ \frac{\kappa}{|\kappa|} \tan(|\kappa|) \right] E_p^{*} \]

(2.19)

Eq. (1.18) shows that the phase conjugate signal is the complex conjugate of \( E_p \).

[Here we have used the boundary condition that \( E_p(0) \) is finite and \( E_c(l) = 0 \).] If the ratio of the intensities of the pump and probe beams is always constant (i.e. if all the three beams are taken from a parent source) intensity of the phase conjugate signal is given by the relation

\[ I_2(\omega) \propto \frac{\omega}{2\varepsilon_0cn^2} |\chi^{(3)}|^{2} l_2^2(\omega) \]

(2.20)

Third order susceptibility \( \chi^{(3)} \) is usually measured with respect to a standard sample \( \text{CS}_2 \). In absence of linear absorption, third order susceptibility is given by
Z-scan and DFWM studies in certain photonic materials

\[ \chi^{(3)} = \chi^{(3)}_{\text{ref}} \left[ \frac{\left( \frac{\gamma_{\text{ref}}}{\gamma_0} \right)}{\left( \frac{\gamma_{\text{ref}}}{\gamma_0} \right)} \right] \left[ \frac{n}{n_{\text{ref}}} \right] \frac{l_{\text{ref}}}{l} \]  \hspace{1cm} (2.21)

If sample has linear absorption, eq. (2.21) has to be modified by considering the effect of linear absorption. It is assumed that optical density of the sample (i.e. \( \alpha l \), where \( \alpha \) is the linear absorption coefficient and \( l \) is the sample length) is much less than one. In presence of linear absorption, susceptibility is given by the following equation

\[ \chi^{(3)} = \chi^{(3)}_{\text{ref}} \left[ \frac{\left( \frac{\gamma_{\text{ref}}}{\gamma_0} \right)}{\left( \frac{\gamma_{\text{ref}}}{\gamma_0} \right)} \right] \left[ \frac{n}{n_{\text{ref}}} \right] \frac{l_{\text{ref}}}{l} \frac{\alpha l}{1 - e^{-\alpha l}} \]  \hspace{1cm} (2.22)

The reflectivity of the phase conjugate mirror \( R \), is defined as the ratio of phase conjugate intensity to probe beam intensity.

\[ R = \frac{I_c}{I_p} \]  \hspace{1cm} (2.23)

Eq. (2.23) can be written in terms of the coupling coefficient \( \kappa \) as in eq. (2.24).

\[ R = \tan^2 \left( |\kappa| \right) \]  \hspace{1cm} (2.24)

2.2. Effects of absorption and pump depletion

In presence of pump beam depletion, phase conjugate reflectivity exhibit deviation in its behaviour from the eq. (2.24). In such cases phase conjugate reflectivity is given by

\[ R = \left| \frac{\kappa \tan(\beta I)}{B + (\alpha / 2) \tan(\beta I)} \right|^2 \]  \hspace{1cm} (2.25)
where \[ B = \left[ |\kappa|^2 - (\alpha/2)^2 \right]^{1/2} \] (2.26)

In the presence of pump depletion, the reflectivity becomes a multi-valued function of \( \kappa \). This arises from the fact that energy is transferred from the pump beam to conjugate beam such that the total energy is invariant. In the case of one photon resonance, it is the imaginary part of the susceptibility, which contributes to the formation of grating, unlike in the non resonant case (where only real part contributes to the grating formation). If \( \chi^{(3)} \) has an imaginary part of considerable magnitude, resonant TPA can take place at high intensities, giving rise to formation of thermal grating, concentration grating, free carrier gratings etc, depending on the sample. Net effect of TPA is to produce effective fifth order nonlinearity through cascading \( \chi^{(3)} \): \( \chi^{(1)} \) processes. Cascading is a situation where a few successive lower order linear or nonlinear effects are combined to produce an effective higher order nonlinear process. In such occasions, the phase conjugate signal may be fitted to an equation of the form [32]

\[ I_c = b_3 I_{\text{pump}}^3 + b_5 I_{\text{pump}}^5 \] (2.27)

\( \chi^{(3)} \) can also be calculated from the fitted value of \( b_3 \) of eq. (2.27).

In the case of instantaneous response like optical Kerr effect, the pulse width of the phase conjugate signal will be 57.77% of the input pulse width for a gaussian pulse. This is approximately true in the case of hyperbolic secant squared pulse also. Therefore, presence of phase conjugate signal temporally broader than 57.71% of incident laser pulse width is an indication of the presence of slower process getting mixed up with instantaneous response. In some cases cascaded effects will imitate a third order process. Such effects have been reported in non-centrosymmetric materials. A few examples are concurrent processes of optical rectification and linear
**Z-scan and DFWM studies in certain photonic materials**

electro optic effects. It has also been reported that piezoelectric effects can also give rise to signal in DFWM [33].

3. **Instruments specifications:**
Specifications of the laser sources, photodiodes and laser energy meters used for making measurements are given below.

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<th>3.1. Pulsed Nd: YAG laser [34]</th>
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<td>Wavelength</td>
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<td></td>
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<td>Pulse width</td>
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<tr>
<td>Beam shape</td>
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<td>Model and make</td>
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<td>Active area</td>
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46
### 3.4. Oscilloscope [38]

- **Model and make**: Tektronix TDS 360
- **Rise time**: 1.75 ns
- **Input resistance**: 1 MΩ
- **Sensitivity**: 2 mV/division to 10 V/division

### 3.5. Energy meter

- **Detector head (Pyroelectric)**: Rjp - 735
- **Model and make**: Rjp - 7620 Energy Ratiometer

### 3.6. Spectrophotometer [39]

- **Model and Make**: Jasco 570
- **Wavelength range**: 190 nm to 2500
- **Resolution**: 0.1 nm (UV/Visible region)
  - 0.5 nm (NIR region)
Z-scan and DFWM studies in certain photonic materials

References:

Chapter 2. Experimental....

[36] 818-UV Detector Calibration Report, Newport Corporation, CA, USA
[37] Melles Griot Catalogue 1997-98, Melles Griot Inc, USA