CHAPTER 2
SYNTHESIS METHODS AND CHARACTERIZATION TECHNIQUES

Synthesis of phase pure materials and their characterization is a very important aspect for materials research. For the current research work on Eu$^{3+}$ activated novel red phosphors two different methods of synthesis are adopted namely, solid state method and citrate gel method. Various techniques are used for structural, microstructural and optical characterization of these phosphors. This chapter outlines various methods of synthesis and characterization techniques employed for the development of Eu$^{3+}$ activated molybdate and tungstate based novel red phosphors.
2.1 Introduction

Synthesis of high quality, phase pure inorganic materials is one of the most important aspects of material science research. The proper choice of the chemical precursors and the preparation technique are essential to obtain a material with the desired chemical and physical properties and it is well known that material’s performances are closely related to the ways that they are processed (Kong L.B et al 2008). The solid samples can be synthesized in variety of shapes and sizes depending upon their requirements such as single crystals, amorphous solids, thin films, thick films, polycrystalline powder etc. Several methods available to synthesize high quality samples in bulk and thin film forms are, Solid State Reaction (SSR), Vapor Phase Transport (VPT), Co-precipitation, Citrate gel, Sol gel, Physical Vapor Deposition (PVD), Chemical Vapor Deposition (CVD), Pulsed Laser Deposition (PLD), Chemical Solution Deposition (CSD), Metal Organic Chemical Vapor Deposition (MOCVD), Sputtering, Flux Growth Technique, Electrochemical Methods etc.

Various techniques are there for structural and microstructural characterization of as prepared materials with desired properties. The structural characterization techniques include X ray Diffraction (XRD), Neutron Diffraction (ND) and Electron Diffraction (ED) while microstructural characterization methods are Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), Lateral Force Microscopy (LFM), Scanning Tunneling Microscopy (STM), Magnetic Force Microscopy (MFM), Transmission Electron Microscopy (TEM) etc. The optical properties such as UV Visible absorption and photoluminescence can be recorded by Spectrophotometer and Spectrofluorimeter respectively.

For the present research work of studies on luminescent properties of Eu$^{3+}$ activated molybdate and tungstate based novel red phosphors, Solid State Reaction (SSR) and Citrate Gel (CG) routes are selected as the synthesis methods and the crystalline structure, morphology, absorbance and luminescent properties of these phosphors were characterized by powder X ray diffraction (XRD), Scanning Electron Microscopy (SEM), UV Visible absorption spectroscopy and Photoluminescent spectroscopy. This chapter overviews various methods of synthesis and characterization techniques employed for the development of Eu$^{3+}$ activated novel red phosphors.

2.2 Synthesis Methods

2.2.1 Solid State Reaction Route

Most of the inorganic phosphors were synthesized conventionally via a solid state reaction process because of its facile operation. In solid state reaction route, powders are used as raw materials and solid - solid reaction occurs between these powders. Up to now, this
method has been the most widely and intensively used approach for the preparation of phosphors including oxides, (oxy) nitrides and fluorides because it is comparatively simple and very suitable for mass production. Because of the refractory nature of alumina and RE oxides, conventional solid state reaction synthesis of oxide based phosphors like YAG requires temperature higher than 1000°C. The solid state reaction is controlled by atomic diffusion between different raw materials. Hence, repeated grinding and heating are required. Furthermore, a controlled atmosphere is necessary to master the valence of the activator and the stoichiometry of the host lattice. The particle size of the phosphor prepared by this method is relatively large and less controllable. In solid state reaction method, the solid reactants react chemically without the presence of any solvent at high temperatures yielding a product which is stable. There are two factors, namely thermodynamic and kinetic, which are important in solid state reaction; the former determines the possibilities of any chemical reaction to occur by the free energy considerations which are involved while the later determines the rate at which the reaction occurs (Engler E.M 1987; West A. R 1984). The atoms diffuse through the material to form a stable compound of minimum free energy. In order to prepare a single phase sample, the conditions during any reaction are very important. During synthesis the parameters such as temperature, pressure, gas flow and time for the reaction are to be varied according to the phase requirements in the sample. The major advantage of solid state reaction method is that the final product in solid form is structurally pure with the desired properties depending on the final sintering temperatures. This method is environment friendly and non toxic or unwanted waste is produced after the SSR is complete.

The steps involved in solid state reaction for synthesis of phosphors are schematically illustrated in Fig. 2.1. They are;

**1) Selection and weighing of raw materials**

Take appropriate high purity (99.99%) fine grain powders as starting materials (reagents) in stoichiometric proportions depending upon the composition and weigh all of them as per the calculations performed.

**2) Mixing and drying**

The stoichiometrically weighed reactants are mixed together in an agate mortar with acetone as the mixing medium. After mixing, the powder is kept in a hot air oven at 100°C for 30 min. This process is repeated three times so as to get a homogeneous, finely powdered mixture. For solid state diffusion mechanism, the growth of the reaction product in powder system occurs at the contact points. The homogeneity of mixing is one of the most important factors which decide the ability of a process to produce homogeneous, single phase powder.
The size and distribution of the reactant particles are important factors for deciding the degree of homogeneity.

A homogeneous mixture has great influence on the diffusion distances and the number of contacts between the reactant particles. The presence of agglomerates results in porosity of the final product. Therefore it is necessary to mix the reactants to a high level and to powder them repeatedly. During mixing process, these agglomerates get broken and defects are introduced into the grains which enhance the diffusion mechanism.

![Schematic illustration of phosphor preparation by solid state reaction route.](image)

**Fig. 2.1** Schematic illustration of phosphor preparation by solid state reaction route.

(3) **Pelletization**

The homogenously mixed powders are pelletized to enhance intimate contact of reactants and minimize contact with the crucible. Organic binder may be used to help keep pellet together.

(4) **Calcination**

Calcination is an intermediate heating at a lower temperature through which chemical reactions between solid starting materials usually in the form of mixed powders take place. The reactants normally consist of oxides, carbonates, nitrates, sulphates etc. It involves heat treating a powder or mixture of powders at a temperature well below its melting point for
effective decomposition, to liberate unwanted gases and/or chemically bound waste, solid state reactions and structural transformations to produce the desired composition and phase product. The kinetics of solid state reactions occurring during calcinations may be controlled by any one of the three processes: (i) the reaction at the interface between the reactant and solid product (ii) heat transfer to the reaction surface (iii) gas diffusion or permeation from the reaction surface through the porous product layer.

(5) Grinding

The grinding of calcined pellets has an effective role in deciding the degree of homogeneity. Generally grinding to somewhere around 1 to 10 µm is advisable. Grinding the solid reactants to very fine powder (<1 µm) can lead to the formation of agglomerates that result in non uniform packing. Agate mortar with pestle is used for grinding the powder reactants. Finally the grinded, reacted powders were analyzed by X ray diffraction technique and analyze the structural formation. If the required phase purity of the phosphor materials is not achieved, repeat the calcination process twice / thrice with intermittent grinding till the completion of reaction.

2.2.2 Citrate Gel Route

In conventional synthesis routes, the reactants are mixed together manually by grinding the mixture of starting materials or mechanically by ball milling process and the subsequent reaction rate depends on a large degree on the particle size of the reactants, the degree of homogenization achieved on mixing and the intimacy of contact between the grains, as well as the obvious effect of temperature.

By chemical methods of preparation, it is possible to achieve a high degree of homogenization together with a small particle size and faster reaction rates. Various chemical methods include sol gel, citrate gel, co precipitation and combustion methods etc that ensure easy mixing of precursor solutions at the molecular level, provides a high degree of homogeneity at low processing temperatures, and makes doping of activators, coactivators or sensitizers straightforward and effective (Dhage S. R et al 2003, 2004 (a), (b); Gaikwad A. H et al 2004).

The citrate gel route includes the following steps and is schematically illustrated in Fig. 2.2.

- Select highly pure (99.99%) raw materials of corresponding metallic cations for the necessary phosphor material. (Nitrates, Chlorides).
- Stoichiometrically weigh and make the cationic solution in distilled water.
- Prepare the citrate solution by dissolving appropriate molar ratio of citric acid in distilled water (1:2).
- After completing homogenization of citrate solution, dissolve all the cationic solution in citrate solution (1:5).
- Keep for constant stirring for 1 hour for homogenous mixing.
- Concentrate the solution by keeping it in the water bath (maintained at 100°C) for 3 hours leading to the formation of gel.
- Dry the viscous gel to form a brown product and then powder it by grinding in an agate mortar, which is the precursor.
- Heat treat the precursor at various temperatures to get the required phase pure phosphors.

Fig. 2.2 Schematic illustration of phosphor preparation by citrate gel route.

2.3 Characterization Techniques

2.3.1 Structural Characterization – X ray Diffraction (XRD)

X rays are electromagnetic radiation with typical photon energies in the range of 100 eV - 100 keV. For diffraction applications, only short wavelength X rays (hard X rays) in the range of a few angstroms to 0.1 angstrom (1 keV - 120 keV) are used. Because the wavelength of X rays is comparable to the size of atoms, they are ideally suited for probing
the structural arrangement of atoms and molecules in a wide range of materials. The energetic X rays can penetrate deep into the materials and provide information about the bulk structure. X rays primarily interact with electrons in atoms. When X ray photons collide with electrons, some photons from the incident beam will be deflected away from the direction where they originally travel. If the wavelength of these scattered X rays did not change (meaning that X ray photons did not lose any energy), the process is called elastic scattering (Thompson Scattering) in that only momentum has been transferred in the scattering process. These are the X rays that we measure in diffraction experiments, as the scattered X rays carry information about the electron distribution in materials. On the other hand, in the inelastic scattering process (Compton Scattering), X rays transfer some of their energy to the electrons and the scattered X rays will have different wavelength than the incident X rays. Diffracted waves from different atoms can interfere with each other and the resultant intensity distribution is strongly modulated by this interaction. If the atoms are arranged in a periodic fashion, as in crystals, the diffracted waves will consist of sharp interference maxima (peaks) with the same symmetry as in the distribution of atoms. Measuring the diffraction pattern therefore allows us to deduce the distribution of atoms in a material (Cullity B. D 1978).

![Fig. 2.3 Illustrations of lattice planes and Bragg's Law.](image)

The peaks in an X ray diffraction pattern are directly related to the atomic distances. The interaction of X rays with the periodic arrangement of atoms arranged in two dimensions can be illustrated as follows. The atoms, represented as spheres in Fig. 2.3, can be viewed as forming different sets of planes in the crystal. For a given set of lattice planes with an interplanar distance \(d\), the condition for a diffraction (peak) to occur can be written as;

\[
2d \sin \theta = n \lambda
\]

which is known as the Bragg's law, after W.L. Bragg, who first proposed it. In the equation, \(\lambda\) is the wavelength of the X ray, \(\theta\) the scattering angle, and \(n\) an integer representing the order of the diffraction peak. The Bragg's Law is one of most important laws used for interpreting
X ray diffraction data (Warren B. E 1969). Although we have used atoms as scattering points in this example, Bragg's Law applies to scattering centers consisting of any periodic distribution of electron density. In other words, the law holds true if the atoms are replaced by molecules or collections of molecules such as colloids, polymers, proteins and virus particles.

![Reflection and transmission geometry in X ray diffraction and Goniometer in PANalytical X’pert PRO X ray diffractometer.](image)

**Fig. 2.4** Reflection and transmission geometry in X ray diffraction and Goniometer in PANalytical X’pert PRO X ray diffractometer.

Powder XRD (X ray Diffraction) is perhaps the most widely used X ray diffraction technique for characterizing materials. As the name suggests, the sample is usually in a powdery form consisting of fine grains of single crystalline material to be studied. The term 'powder' really means that the crystalline domains are randomly oriented in the sample. Therefore when the 2D diffraction pattern is recorded, it shows concentric rings of scattering peaks corresponding to the various d spacings in the crystal lattice. The positions and the intensities of the peaks are used for identifying the underlying structure (phase) of the material. Powder diffraction data can be collected using either transmission or reflection geometry, as shown in Fig.2.4.
Powder diffraction data are usually presented as a diffractogram in which the diffracted intensity is shown as function either of the scattering angle, 2θ or interplanar spacing, d. The diffractogram data can be analysed for phase identification and for calculation of lattice parameters, extent of crystallinity, crystallite size and strain present in the lattice. For the structural characterization of phosphor materials, here PANalytical X’pert PRO X-ray diffractometer is used. The instrumental parameters are summarized in Table 2.1.

Table 2.1 Instrumental parameters of PANalytical X’pert PRO X-ray diffractometer

<table>
<thead>
<tr>
<th>Diffractometer system</th>
<th>PANalytical X’pert PRO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Configuration</td>
<td>Flat sample stage</td>
</tr>
<tr>
<td>Goniometer</td>
<td>Theta – theta mode</td>
</tr>
<tr>
<td>Goniometer radius</td>
<td>240 mm</td>
</tr>
<tr>
<td>Scan Type</td>
<td>Continuous</td>
</tr>
<tr>
<td>Divergence slit type</td>
<td>Fixed</td>
</tr>
<tr>
<td>Divergence slit width</td>
<td>0.5°</td>
</tr>
<tr>
<td>Measurement temperature</td>
<td>25°C</td>
</tr>
<tr>
<td>Specimen length</td>
<td>10 mm</td>
</tr>
<tr>
<td>Anode material</td>
<td>Cu</td>
</tr>
<tr>
<td>K Alpha1</td>
<td>1.5406 Å</td>
</tr>
<tr>
<td>Generator settings</td>
<td>30 mA, 40 kV</td>
</tr>
<tr>
<td>2θ offset</td>
<td>Zero</td>
</tr>
</tbody>
</table>

2.3.2 Microstructural Characterization – Scanning Electron Microscopy (SEM)

Electron Microscopy can be defined as a specialized field of science that employs the electron microscope as a tool and uses a beam of electrons to form an image of a specimen (Bozzola J. J et al 1992; Heath J. P 2005). In contrast to light microscopy which uses visible light as a source of illumination and optical lenses (glass) to magnify specimens in the range between approximately 10 to 1,000 times their original size, electron microscopy is operated in the vacuum and focuses the electron beam and magnifies images with the help of electromagnetic lenses. The electron microscope takes advantage of the much shorter
The wavelength of the electron compared to the wavelengths of visible light (Flegler S. L. et al. 1993). When the accelerating voltage is increased in electron microscope, the wavelength decreases and resolution increases. In other words, increasing the velocity of electrons results in a shorter wavelength and increased resolving power (Flegler S. L. et al. 1993). The scanning electron microscope (SEM) is primarily used for the study of surface morphology of solid specimens, which gives image at very high magnification and high resolution. Due to the manner in which the image is created, SEM images have a characteristic three dimensional appearance and are useful for judging the surface structure of the sample.

Electrons in scanning electron microscopes are accelerated at voltages in the range of 2 to 40 kV. An electron beam < 0.01mm in diameter is focused on the specimen. These fast primary electrons (PE) interact in various ways with the surface layers of the specimen. The zone, in which such interaction occurs, and in which different signals are produced, is called "interaction volume" or "electron diffusion cloud". The size of the interaction volume is proportional to the energy of primary electrons and its shape is determined depending upon scattering processes by the mean atomic number. Secondary electrons (SE), back scattered electrons (BSE) are produced flowing off as specimen current. In addition X rays, Auger electrons, and cathodoluminescence are produced. Although secondary electrons are produced in the entire interaction volume, they can only escape from surface layers. Secondary electrons are very slow, their escape energy is ≤ 50 eV. Approximately half of all secondary electrons are produced very near to the point of impact of primary electrons. The SE signal, comprising all essential information on topography, produces electron-micrographs of high resolution.

The basic parts of a scanning electron microscope includes electron optical column, vacuum system, signal detectors and display system. Schematic illustration of a SEM is shown in Fig. 2.5. The primary function of the lenses is to demagnify the electron beam. The vacuum system consists of two pump systems. Scan generator coils control the magnifications. Electrons in the electron gun are emitted from the cathode and accelerated by the anode to the energy 1 – 50 keV. The electron beam is condensed by the condenser lenses (one or two). Magnetic field produced by the scan coils deflects the electron beam back and forth. The electron beam focused by the objective lens to very fine spot (1-5 nm) scans the sample surface in a raster pattern. Primary electrons interact with the atoms of the sample surface causing emissions of the secondary electrons which are detected and producing the image. The backscattered electrons of the electron beam may also be detected. The
backscattered electron image is used for contrasting the sample regions, having different chemical compositions.

Fig. 2.5 Schematic presentation of a Scanning Electron Microscope.

For microstructural analysis using SEM, non conductive solid specimens should be coated with a layer of conductive material except when observed with Variable Vacuum or Environmental SEM. Such coatings are made on the surface of non conductive samples using gold or palladium (Goldstein J. I et al 1981).

The microstructural analysis of the as prepared phosphors was carried out by JEOL JSM- 5600 LV scanning electron microscope. The details of the instrumental settings are summarized in Table 2.2.
2.3.3 Optical Characterization – UV Visible Absorption Spectroscopy

UV Visible absorption spectroscopy is a powerful analytical tool to understand the optical properties of materials and to identify inorganic and organic materials. UV Visible absorption spectroscopy involves absorption of UV/Visible light by a molecule causing the excitation of an electron from ground electronic state to excited electronic state. UV Visible absorption spectrum is a plot of degree of absorption of sample against the wavelength of the incident radiation. It may include both broad and sharp lines of absorption. In practice it is found that the ultraviolet and visible spectrum of most molecules consists of a few humps rather than sharp lines. The main reason for the band absorption is that an electronic level transition is usually accompanied by a simultaneous change between the more numerous vibrational levels. Thus a photon with too little energy to be accepted by the molecule for a 'pure' electronic transition can be utilized for a transition between one of the vibrational levels associated with the lower electronic state to one of the vibrational levels of a higher electronic state. A typical spectrophotometer set up for UV Visible absorption is shown in Fig. 2.6. In absorption spectrophotometer, the photon energy of the radiation incident on the sample is selected by a scanning monochromator (Ronda C 2007; Henderson B et al 1989; Yen W. M et al 2004)
The radiation selected by the monochromator is passed through a chopper that alternately directs the radiation along a reference beam path (going through compartment R) and through a path containing the sample (compartment S). One detector alternately records the signals from both beam paths. The transmittance $T(\lambda)$ is defined as the ratio of the intensity of the light passed through the sample $I(\lambda)$ to the intensity of the reference signal $I_0(\lambda)$. In a transmission spectrum, $T$ is plotted as a function of wavelength (an equivalent quantity). Instead of transmittance the absorbance or optical density, $A$ is often the quantity plotted on the y axis.

It is related to the transmittance by the equation,

$$A = -\log_{10}(T) \quad (2.2)$$

Absorption spectroscopy is used to measure the transmitted signal for transparent (nonscattering) samples such as single crystals or solutions. An alternative for strongly scattering materials such as polycrystalline powders is diffuse reflection spectroscopy. The difference in set ups for absorption and reflection spectroscopy is in the detection compartment. When diffuse reflection spectroscopy is used, the backscattered signal is detected and compared to the backscattered signal from a reference material. Polycrystalline powders of MgO or BaSO$_4$ can be used as a reference. These materials scatter all the light in the wavelength range 200–3000 nm. The reflectance, $R$, defined as $I/I_0(\lambda)$, is plotted on the y axis.
The absorbance of the phosphors was recorded by Shimadzu UV 2401 spectrophotometer. The instrumental parameters of Shimadzu UV 2401 are enlisted in Table. 2.3.

**Table. 2.3 Instrumental parameters of Shimadzu UV 2401 spectrophotometer**

<table>
<thead>
<tr>
<th>Spectrophotometer Model</th>
<th>UV 2401</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lamps</td>
<td>Tungsten, Deuterium</td>
</tr>
<tr>
<td>Detector</td>
<td>PMT</td>
</tr>
<tr>
<td>Wavelength Range</td>
<td>190-900 nm</td>
</tr>
<tr>
<td>Wavelength Reproducibility</td>
<td>0.1 nm</td>
</tr>
<tr>
<td>Wavelength Precision</td>
<td>0.3 nm</td>
</tr>
<tr>
<td>Monochromator</td>
<td>High performance holographic grating (single)</td>
</tr>
<tr>
<td>Power Needed</td>
<td>240 V</td>
</tr>
</tbody>
</table>

2.3.4 Optical Characterization – Luminescence Spectroscopy

Luminescence spectroscopy is a collective name given to three related spectroscopic techniques. They are molecular fluorescence spectroscopy, molecular phosphorescence spectroscopy and chemiluminescence spectroscopy. The basic process involved in phosphors is phosphorescence (photoluminescence), here phosphorescence spectroscopy can be referred as luminescence spectroscopy (Pelant I et al 2012). Photoluminescence spectra are measured using compact commercial equipment called spectrofluorimeters. Their main elements are also shown in Fig. 2.7.

The sample is excited with a lamp which is followed by a monochromator (the excitation monochromator) or a laser beam. The emitted light is collected by a focusing lens and analyzed by means of a second monochromator (the emission monochromator) followed by a suitable detector connected to a computer. Two kinds of spectra, (i) emission spectra and (ii) excitation spectra can be recorded. In emission spectra, the excitation wavelength is fixed and the emitted light intensity is measured at different wavelengths by scanning the emission monochromator. An emission spectrum yields information on the energetic positions of the optical transitions that are involved in the emission of light. From emission spectra, no information about the absolute value of the transition strengths can be obtained but
comparison of relative intensities (after correction for instrumental response) is straightforward for transitions originating from the same level. In this case, the observed intensities $I_{em}(\lambda)$ for transitions to various levels are proportional to the corresponding radiative transition rates.

![Diagram](image)

**Fig. 2.7** Schematic illustration of a photoluminescence spectrofluorimeter.

In excitation spectra, the emission monochromator is fixed at any emission wavelength while the excitation wavelength is scanned in a certain spectral range. In contrast to an absorption spectrum, an excitation spectrum yields information on the energetic position of absorption bands that lead to emission of the chosen wavelength.

The different experimental set ups for absorption/ reflection and emission/ excitation spectroscopy lead to a different performance. Excitation and emission spectroscopy have very low background levels and a much higher sensitivity. They are used in combination with samples containing very small quantities of luminescent species. Absorption and diffuse reflection spectroscopy have much larger background and noise levels and are used for more concentrated samples. Both types of techniques also show different requirements for the equipment used. For absorption and diffuse reflection spectroscopy, the requirements for the output powers of lamps and the sensitivity of the detector are less demanding than for emission and excitation spectroscopy. Collimated radiation from the excitation source is measured in the former two techniques while in the latter, diffuse and spectrally resolved emission radiation of much lower intensity has to be detected.
The photoluminescence properties of developed red phosphors are recorded by Horiba Yvon Fluorolog® 3 spectrofluorimeter. The instrumental parameters of Horiba Yvon Fluorolog® 3 spectrofluorimeter are enlisted in Table. 2.4.

<table>
<thead>
<tr>
<th>Table.2.4 Instrumental parameters of Fluorolog® 3 spectrofluorimeter</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Spectrofluorimeter</strong></td>
</tr>
<tr>
<td>Source</td>
</tr>
<tr>
<td>Excitation Monochromator</td>
</tr>
<tr>
<td>Emission Monochromator</td>
</tr>
<tr>
<td>Sample Compartment Module</td>
</tr>
<tr>
<td>Detector</td>
</tr>
</tbody>
</table>

2.3.5 Lifetime Measurement – Phosphorimeter

Luminescence lifetime is the time required for the luminescence intensity to decay from the initial value to 1/e of that value (e = 2.718...). Lifetimes can be measured by pulsed luminescence experiment. The Phosphorimeter is used to record the lifetime of luminescence. The lifetime of Eu$^{3+}$ activated red phosphors were recorded by the Phosphorimeter attached to Fluorolog® 3 spectrofluorimeter. The Phosphorimeter includes a pulsed Xe lamp, gating electronics to control the size and temporal displacement of the detection window. It is a part of dual lamp house that contain both pulsed and continuous Xe lamps, electronics and cables. By software control it is possible to switch between pulsed and continuous lamps. For the lifetime measurement the sample is excited with pulsed light. The emitted phosphorescence is measured by a photon counting detector (R928) with variable delay and open window between pulse and detection.

A typical sequence of data acquisition starts with a flash from the pulsed lamp, sensed by the control module at time $t = 0$. The light enters excitation spectrometer, where it is dispersed. Monochromatic light from the spectrometer excites the sample.
Fig. 2.8. The sequence of excitation, delay and sampling in lifetime measurement.

Luminescence emission from the sample then passes through emission spectrometer to photomultiplier tube detector. The control module includes a gate and delay generator, allowing the signal at detector to be integrated only during a specific period after flash (Delay after flash), for a predetermined length of time (Sample window). Any signal arriving before or after the gating is ignored. This sequence of excitation, delay and collection is repeated for each lamp pulse (Fig. 2.8). The total signal is accumulated for a predetermined number of pulses and the data is saved to the disk. The data can be manipulated and luminescence lifetime of the corresponding sample can be calculated.