Chapter 2

INSTRUMENTATION

In this chapter mainly the instrument details and the specific utility is given. However the various aspects of the furnace design, specific chamber design for combustion synthesis is not included even though the work was undertaken. Various general arrangements for specific wet chemical methods and the apparatus required for the same are not listed. The reasons being that the things are standard and could be obtained from the fundamental books of chemistry. Only the major and specific Instrumentation used to study different aspects is described in the following sections:

1. Fluorescence spectrophotometer for studying photoluminescence.
2. Apparatus for PL quantum efficiency (Q) measurement.
3. Sources for irradiation. Co$^{60}$ gamma ray source.
4. The Harshaw TLD System 3500
5. Mettler Balance

2.1 FLUORESCENCE SPECTROPHOTOMETER

The Photoluminescence spectra (excitation and emission) were recorded on the Hitachi F-4000 Fluorescence Spectrophotometer. The functional block diagram of model F-4000 is shown in figure 2.1; and sketch of the optical system configuration is shown in figure 2.2. The spectrophotometer consists of two monochromators (one on the excitation side and the other on the emission side), a light source, two detectors (one for measurements and the other for monitoring), a sample holder, a data processor and a graphics plotter.

2.1.1 Specifications

(i) **Monochromators**: Large stigmatic concave gratings having 900 lines/mm are used on both excitation and emission sides with eagle mounting (F: 3) Blaze wavelengths are 300 nm on excitation side and 400 nm on emission side.

(ii) **Measuring wavelength range**: 220 to 730 nm and zero order light on both excitation and emission sides.

(iii) **Light source**: 150 W xenon lamp with ozone self-dissociation function.
(iv) **Detectors:** Photo multiplier R 372 F for measurements and photoelectric tube R 518 for monitoring.

### 2.1.2 Procedures for measurement of the Excitation and Emission Spectra

A powder sample cell was used to record the photoluminescence spectra. The sample cell consists of a round sample holder, quartz disk (window) and threaded cap. The quartz disk was fixed into the sample holder and the powder was spread on it. The threaded cap was fitted to hold the powder sample. The metal frame was put on the sample-cell containing sample so that the front protrusion of the cell could fit into the metal frame aperture. When analyzing the sample, optical axis will run along the centerline of powder surface (Quartz window). First the excitation (EX) spectra were recorded by setting the emission wavelength at the zero order and keeping other parameters as specified in the manual. The excitation bands (EX) were identified from these spectra and the emission (EM) spectra were scanned for identified excitation wavelengths.

It was necessary to know approximate nature of EX and EM spectra. While doing so, the direct scattered light may superimpose on the EX spectrum. So it is necessary to select a particular band in the emission for scanning the EX. Therefore, for proper excitation wavelengths, EM was set at the position as identified from the earlier emission spectrum. Again same procedure was followed for identifying correct EX positions and EM was recorded for each EX band separately.

In the ordinary measurements, a spectrum is affected by wavelength characteristics of the analysis system (monochromator, photo multiplier, etc.). In order to measure a spectrum of a sample free from the influence due to wavelength characteristics of the analysis system, excitation spectrum correction was performed during rhodamin B as a standard. Similarly emission spectrum was corrected using diffuser and attenuators mentioned in the instrument manual. Both the spectra were correctable in the range of 220-600 nm. The samples whose emission wavelengths are within 220-600 nm were scanned in the correct spectrum mode and the samples whose wavelengths were beyond 600 nm were scanned in ordinary mode.

Emission spectra were recorded with excitation band pass 5 nm and emission band pass 1.5 nm, while the excitation spectra were recorded with excitation band pass of 1.5 nm and emission band pass 5 nm.
Figure: 2.1 Functional block diagram of the fluorescence spectrophotometer

Figure: 2.2 Optical system configuration of fluorescence spectrophotometer
2.2 APPARATUS FOR MEASUREMENT OF PL QUANTUM EFFICIENCY

One of the important selection criterions for any specific application of a phosphor is its efficiency of energy conversion. Two important applications of inorganic powder phosphor are:

1. Conversion of UV to visible light in mercury discharge lamps; and
2. Conversion of electron beam (EB) energy to visible light in a cathode ray screen.

For UV to visible light conversion, the quantum efficiency \( Q \) is defined as the ratio of the number of photons emitted to that absorbed by the phosphor. The radiant or power conversion efficiency, better known as cathodoluminescent efficiency (CL), when the phosphor is used for energy conversion in a Cathode ray screen, is defined as the ratio of the power of the luminescent radiation emitted by the phosphor to that absorbed by it from the EB. An apparatus used for the measurement of \( Q \) is given in figure 2.3. It consists of a demountable stainless steel (SS) assembly of two chambers: one upper (1) and the lower (2). A HPMV lamp (3) (125 W), without its outer shell, is placed vertically in this chamber. The MV lamp provides the UV radiator of wavelengths 254, 312 & 360 nm. The light beam from the HPMV lamp is collimated with two aluminium rings (4) fitted inside the SS tube. A thin metal plate (5), introduced through a side cutting in the tube, acts as the shutter.

The lower chamber of SS tube holds the interference filter (6) and quartz lens (7) on suitable support, the aluminium sample holder (8) and the SPD (9). The base of an apparatus (10) is a graphite disk. A channel (11)
is cut in this disk to slide the sample holder from the side, inward to the centre. A sample holder is an aluminium plaque of ID 20 mm and depth 3 mm. The plaque is loaded with a 3 mm phosphor layer of uniform thickness. The SPD, mounted on cylindrical bakelite frame (12) placed at the viewing port (13), will view the light coming out from the phosphor surface at an angle 45°. The mercury line interference-filter selects the wavelength for UV excitation. A quartz lens (f = 5 cm) focuses the UV light onto the phosphor surface. The light falling on the SPD surface (area = 10x10 mm) produces a photocurrent, which is measured by an electrometer amplifier. A pyrex filter (14) introduced through the viewing port (13) cuts off UV when required. The beam of UV (15) strikes the phosphor surface and the fluorescent emission (16) falls on the SPD (type no. S1337-1010 BQ, Hamamatsu). The light collected by the SPD is proportional to the solid angle subtended by it on the phosphor surface.

To measure the currents, the aluminium plaque is loaded with MgO and placed in the apparatus, and UV radiation is focused on to a small area using the quartz lens. The reflected UV rays from MgO produces a photocurrent, \( C_1 \), in the SPD, which is measured by the electrometer amplifier. The plaque is now emptied and filled with the phosphor for measuring its \( Q \). As before, the current \( C_2 \) is now measured. The pyrex glass filter is then placed in front of the SPD, to cut off the reflected UV rays from the phosphor and the current due to the remaining fluorescent radiation \( C_3 \) alone is also measured. From these measurements, we get \( C_4 = C_2 + C_3 \) strictly this \( C_3 \) is not the exact current component in \( C_2 \) due to fluorescence alone. The measured current is equal to \( C_1 \) which is an attenuated value of \( C_3 \) due to the transmission coefficient \( T_r \) of the pyrex filter. The real current \( C_3 = C_1 / T_r \). The value of \( T_r \) was found to be 0.92±1% for the visible region of emission.

Expression for \( Q \), can be rewritten as [1]:

\[
Q = \{C_3 / C_1\} \{l_e / l_r\} \{p / (1-r)\}
\]

Where, \( r = p \{C_4 / C_1\} \).

The value of \( Q \) is determined from equation 1 by substituting all the known values in which \( C_1 \) & \( C_3 \) are actually measured and \( l_e \) & \( l_r \) can be deduced from characteristic curve of the SPD.

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2.3 SOURCES FOR IRRADIATION

$^{60}$Co source was used for $\gamma$-ray irradiation. Set-up for irradiation essentially consists of the following parts:

1. Radiation
2. Biological shield for the source
3. Central drawer incorporating the sample chamber
4. Driving system
5. Control panel
6. External cabinet

Gamma chamber 900 was received from the Isotope Division, Bhabha Atomic Research Centre (BARC), Bombay (India). This unit initially had 2370 curies Co$^{60}$ source on 11-12-1992 with its dose rate 0.56 MR/hr.

Gamma chamber 900 is a compact unit, offering an irradiation volume approximately 1000 cc. It has a sample chamber of 10 cms in diameter and 14 cms in height. The central drawer can be raised or lowered by a steel rope passing over a geared motor. This enables the sample chamber to be moved up and down as required and this movement is controlled from the front control panel through an electrical circuit. For low exposures, similar facilities involving weaker Co$^{60}$ source, in the RST cancer Hospital, Nagpur were also available.

2.4 HARIWAL TLD READER - MODEL 3500

The Harshaw TLD System 3500 Manual TL Reader is a PC-driven, manually operated, tabletop instrument for thermo luminescent dosimetry (TLD) measurement. It economically provides both high performance and high reliability and complies with the latest International Standards Organization (ISO) requirements. The 3500 read one dosimeter per loading and accommodate a variety of TL configurations, including powder.

The flexible design of the controlling software enables the user to configure the workstation for use in different applications. For example, for radiation therapy and planning, the software can configured to calibrate the instrument in Grays, track the dosimeters primarily by
Patient Identification, and retain some medical history. For personnel radiation protection monitoring, the primary data tracking may be by dosimeter and Employee ID, and the instrument calibrated in Sieverts. Other calibration unit's identification combinations are available.

The Reader's basic external components include a front control panel consisting of three LED status lights and a Read pushbutton, a sample drawer assembly that features an interchangeable planchet and a built-in test light for periodic monitoring of Reader performance, and a drawer for neutral density filters. The rear panel houses a voltage selectable power input module with fuse access, an instrument Reset button, a fitting for nitrogen gas tubing an RS-232-C serial communication port, and a recessed pressure sensor adjusting screw.

The technical architecture of the system includes both the Reader and a DOS-based IBM-compatible computer connected through a standard RS-232 serial communication port. The dosimetry functions are divided between the Reader and the specialized TLD Shell software that runs on the PC. All dosimetric data storage, instrument control, and operator inputs are performed on the PC; signal acquisition and conditioning are performed in the Reader. This simple architecture allows users the flexibility and economy of using an existing computer, and provides for the simple addition of optional functions and the updating of application software. The software provides real-time monitoring of the instrument's operating conditions and display of the glow curves and response values.

The Reader uses contact heating with a closed loop feedback system that produces linearly ramped temperatures accurate to within ±1°C to 400°C in the standard Reader, or 600°C with the High Temperature Profile (HTP) is user-defined in three segments: Preheat, Acquire, and Anneal, each with independent times and temperatures.

To improve the accuracy of low-exposure readings and to extend planchet life the 3500 provides for nitrogen to flow around the planchet. By eliminating oxygen in the planchet area, the nitrogen flow eliminates the unwanted oxygen-induced TL signals. Nitrogen is also routed through the Photo multiplier Tube (PMT) chamber to eliminate moisture caused by condensation.

A full complement of hardware and software accessories is available from Bicron to facilitate the effective use of the 3500. These include annealing ovens, external irradiators,
dosimeter handling tools and storage bins, neutral density filters, a nitrogen pressure regulator and valve, an uninterruptible power supply (UPS), and specialized applications software.

The 3500 TLD Reader – PC system operates interactively. Most of the controlling features are implemented on the TLD Shell software running on the PC. The Reader does have LED displays to indicate basic status for power, reading, and heating, but the principal information is displayed on the PC monitor and can print on a printer attached to the PC.

2.5 METTLER BALANCE

All the calculated weights of various chemicals and of the fuel are weighed on Mettler Toledo AG Balance. The analytical balance of the AG 204 combine numerous weighing and adjustment possibilities with an exceptional ease of operation. It is fully integrated doors of the draft shield. These balance are the most compact of their type and also equally convenient to operate for right and left-handers. The model of the AG 204 has the following common features.

i] Extremely compact construction.

ii] Ergonomic, one-handed operation of the draft shield, equally convenient for right and left handlers.

iii] Convenient keypad for one-handed operation and wide, easily readable display.

iv] FACT (Fully Automatic Calibration technology), fully automatic, motorized adjustment (calibration) with internal weight (naturally, the balance can also be calibrated with external weights)

v] Built-in functions for piece counting, percent weighing, formulation and dynamic weight determination.

vi] Built-in interface of the latest generation (Local CAN universal interface) allows the attachment of upto 5 peripheral devices. use of an adapter cable also allows attachment of devices with an RS 232C interface.

vii] Line independent operation (upto 10 hours) with optional PP B10 power pack.

AC balance conforms with the current standards and guidelines. It supports standard procedures, specifications, work practices and records following good laboratory
practice and standard operating procedure. The technical data of the AG 204 balance is as given below.

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Readability</td>
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</tr>
<tr>
<td>Maximum capacity</td>
<td>210 g</td>
</tr>
<tr>
<td>Taring range</td>
<td>0.210 g</td>
</tr>
<tr>
<td>Repeatability</td>
<td>0.1 mg</td>
</tr>
<tr>
<td>Linearity</td>
<td>± 0.2 mg</td>
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<tr>
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<tr>
<td>Calibration weight, external</td>
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