CHAPTER-IV

EXPERIMENTAL DETAILS
A. SAMPLE PREPARATION

In the present investigations the samples were prepared by using NaCl as host material in which Terbium was introduced in the form of its salt (TbCl₃). The base material used was "analar" grade sodium chloride supplied by BDH Laboratory Chemical Division, Bombay, which was certified to have a purity of 99.9 %.

The maximum probable impurities, according to the manufacturer, are insoluble matter 3 x 10⁻³ %, free acid (HCl) 1.8 x 10⁻³ %, free alkali 0.05 ml N¹⁻¹, bromide and iodide (Br) 5 x 10⁻³ %, ferrocyanide Fe(CN)₆⁻¹, 1 x 10⁻⁴ %, nitrate (NO₃⁻) 5 x 10⁻⁴ %, phosphate (PO₄⁻) 5 x 10⁻⁴ %, sulphate (SO₄²⁻) 2 x 10⁻³ %, ammonium (NH₄⁺) 5 x 10⁻⁴ %, arsenic (As) 4 x 10⁻⁵ %, barium (Ba) 1 x 10⁻³ %, calcium group and magnesium (Ca) 4 x 10⁻³ %, iron (Fe) 3 x 10⁻⁴ %, heavy metals (pb) 5 x 10⁻⁴ % and potassium (K) 1 x 10⁻² %.

Extreme care was taken with regard to the cleanliness and the purity while preparing and handling the samples. All the surfaces which were to come in contact with the phosphor had been cleaned with concentrated hydrochloric and nitric acids. After cleaning, the surfaces were kept in contact with boiling distilled water for nearly two hours and then dried thoroughly in an oven.

The impurity concerned in the present investigations is terbium used in the form of its salt viz. Terbium chloride. Terbium chloride was supplied by BDH Laboratory Chemical Division, Bombay, with 99.9 % purity. Introduction of the impurity into
sodium chloride was accomplished by the usual method of crystallization from aqueous solution. The weighed quantity of sodium chloride was dissolved in double distilled demineralized water and the weight of impurity as determined by the ppm calculation was added to the solution. Subsequently, the solution was heated slowly on a hot plate, until the excess water was completely driven out. Microcrystals were collected, dried at about 40°C, powdered and mixed homogeneously. The specimens of NaCl:Tb with four different Tb-impurity concentrations namely, 100, 200, 800, 1000 ppm prepared in this manner were designated as "as-received" samples.

B. THERMAL ANNEALING TREATMENTS

Thermal annealing of "as-received" samples was done in Muffle furnace supplied by Tempo Industrial Corporation (India). Temperature was adjusted by an energy regulator, which controlled the temperature by operating on a time cycle of ON and OFF, thus varying the average energy input as desired. Silica boats containing as-received samples were put in Muffle furnace maintained at about 500°C for two hours. On the completion of the annealing time, the samples were rapidly quenched to room temperature by withdrawing the boats on to a block of aluminium, maintained at room temperature by a blast of cold air. The rate of cooling for such samples was on an average of the order of 200°C per minute.

The second batch of the samples was annealed at 750°C for two hours and rapidly cooled to room temperature in the similar
manner. The 750°C annealed and quenched NaCl:Tb (1000 ppm) samples were designated as "NaCl:Tb (T)". The maximum variation observed in the value of the set temperature was ± 15°C.

C. RADIATION SOURCES

Samples were irradiated in $^{60}$Co gamma cell AECL-200 having uniform exposure rate of 800 Rad per minute at the irradiation position. The beta source used was a $^{90}$Sr 20 milli Curie having the dose rate of 700 Rad per minute. The dose rates were standardised and corrected periodically for $^{60}$Co and $^{90}$Sr decays respectively.

D. THERMOLUMINESCENCE GLOW CURVE READER

TL glow curve recorder system consists of a photomultiplier (PM) tube, a high voltage unit, a d.c. amplifier, a temperature programmer and a strip chart recorder. Block diagram of the unit is shown in Fig. IV-1. Sample is spread uniformly on a metallic Kanthal strip which is an alloy of Cr 23 %, Fe 72 %, Co 2 %, and Al 3 % acts as the heater. The size of the Kanthal strip is kept as 40 x 6 x 0.25 mm$^3$ with a 15 x 5 mm$^2$ of central area embossed to 0.5 mm depth and a chromel-alumel thermocouple is spot welded at the centre on the lower side of the Kanthal strip. Because of the small size of heater, high temperature can be attained with low power input and cooling is very fast after the power is switched off. Kanthal strip is secured tightly between two brass electrodes. The electrodes are fitted in a 233 x 100 x 24 mm$^3$
BLOCK DIAGRAM OF PROGRAMMED LINEAR HEATING TYPE TL GLOVE CURVE RECORDER.

FIG - IV - 1
bakelite plate which can slide in and out of a brass guide box, at the top of which an EMI 9514S photomultiplier tube was housed in a light tight brass cylinder. Negative voltage of 1050 volts is applied to the photomultiplier tube. When the bakelite plate containing the Kanthal strip and the sample is fully inside the guide box, sample is placed in front of the PM tube window.

Power to the strip is fed from Hewlett Packard F and M Model 240 temperature programmer which can heat the sample linearly from room temperature to 500°C and also a constant temperature anywhere from room temperature to 500°C can be maintained. A linear heating rate of 200°C per minute was used to record the TL glow curves in the present experiments. Light emitted by the sample uniformly spreaded over the kanthal strip on heating is detected by the PM tube and its output is fed to a Rakadenki (Japan) 10 mV strip chart recorder through a d.c. amplifier. Response time of the recorder was 0.3 second (full scale) and the chart speed used was 5 cm per minute. Different quantities of NaCl:Tb(T) material namely 2, 4, 6, 8, 10, 20, 30 and 40 mgs have been exposed to 800 Rad gamma dose. The TL glow curves exhibited by them were recorded by TL reader in identical experimental conditions. The plot of TL intensity at 200°C peak versus quantity is shown in Fig. IV-2. This brings out the fact that 10 mg of NaCl:Tb powder is sufficient to give substantial TL output. Therefore 10 mg of NaCl:Tb phosphor was selected as standard quantity for all TL glow curve measurement in the present work.
FIG-IV-2

TL-INTENSITY (AMP. X 10^5)

QUANTITY OF SAMPLE (mg.)
1. Recording of the Initial Part of the Glow Peak:

Initial portion of the 200°C glow peak was recorded for the purpose of determining the activation energy. Experimental arrangement was same as described in section-D except the difference that, the heating rate used was 100°C per minute. Requirements for determining the activation energy by Initial Rise method are (i) there should not be any contribution from neighbouring peaks, since only the initial portion of the glow peak is required for this purpose, therefore it must be assured that the lower temperature peaks are removed completely, (ii) population of charge carriers in the trap should remain unaffected.

After irradiation with gamma dose of 800 Rad the NaCl:Tb(T) sample was heated at 150°C for 30 minutes, so that the peaks on low temperature side were removed. Then it was warmed at the rate of 100°C/min. upto the temperature 167°C below the peak temperature so that even if a small part of any lower temperature peak is present, that is removed completely thus fulfilling the first condition. The second condition was satisfied by recording the glow peak upto the temperature when TL output is 1% or less than the peak intensity. This affects the trap population negligibly.

2. Glow Curve Recording With Different Heating Rates:

As an alternate method for the activation energy determination, TL glow curves were recorded with different heating
rates. The samples were given the post irradiation thermal annealing treatments described in section D-1, to remove the peak on low temperature side. Then the samples were heated linearly with the rates of 50, 100, 150 and 200 °C/min. and the glow curves were recorded. Fresh samples were taken for recording of the glow curve with each of the above mentioned heating rates.

3. Recording of Isothermal Decay:

In the study of isothermal intensity decay of TL glow peak, temperature of decay is chosen such that there is no TL emission of higher temperature peaks at that temperature. Secondly, the required temperature is attained by slowly warming the sample. If the required constant temperature is set on the temperature programmer in the beginning itself at the time of switching on, full electrical power to the Kanthal strip is fed and after the set temperature is attained power goes off. But because of thermal inertia, temperature of the Kanthal strip continues to increase by about 25 to 50°C. In order to avoid this 50°C lower temperature is set than the required temperature and the sample is warmed at the rate of 25°C/min. Temperature of the sample increases at a linear rate because of the constant increase in the power being fed to the Kanthal strip. When the required temperature is attained, further heating is stopped by setting the programmer in the isothermal position. In this setting a constant electrical power is fed to the Kanthal strip and the constant temperature is maintained. Practically no deviation from the set temperature is observed during a period of 30 minutes over which isothermal decay is recorded.
For studying the intensity decay of glow peak at 200°C, the post irradiation thermally treated NaCl:Tb(T) sample was spread on the Kanthal strip and warmed to 147°C at the rate of 25°C per minute. The temperature was maintained at 147°C for 30 minutes and the decays were recorded on the strip chart recorder at the speed of 5 cm per minute. The light detecting assembly has been described in section D-1. Fresh sample was taken for each of the two decay temperatures.