CHAPTER III

EQUIPMENT AND PROCEDURE

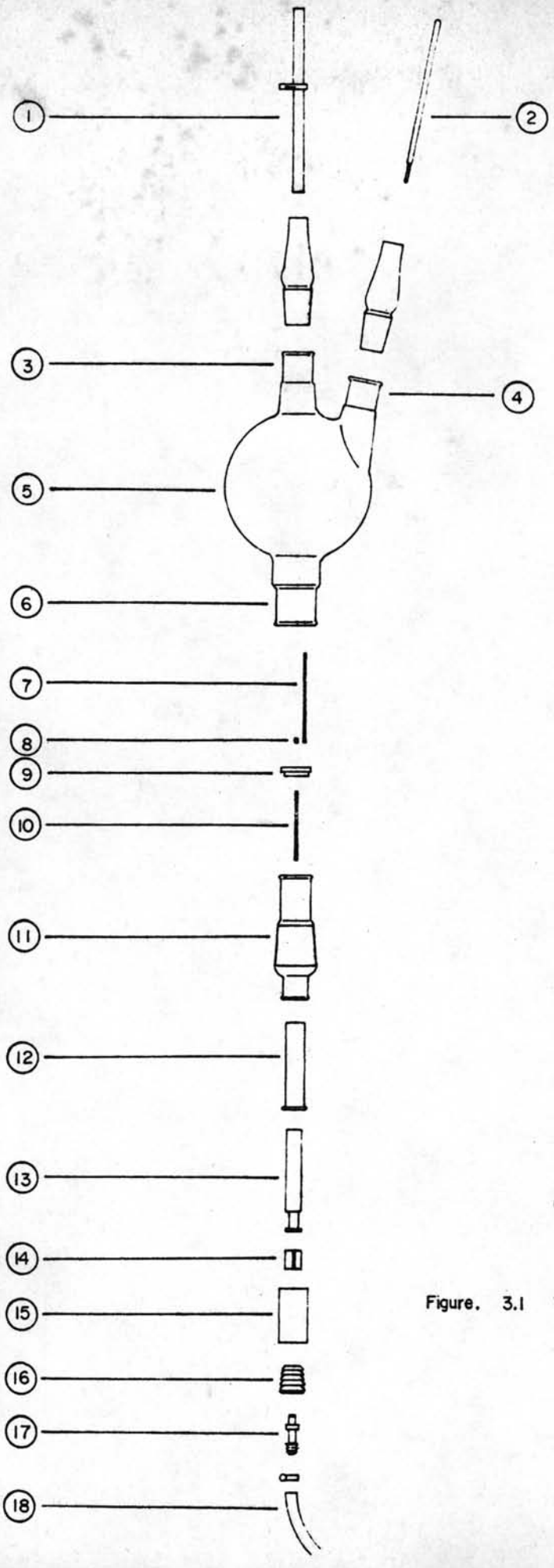
3.1 Equipment

The equipment used in this work consisted of the followings:

1. Diffusion cell
2. Constant temperature bath of model number MR 32104 manufactured by Blue M Electric Company, Blue Island Illinois U.S.A.
3. Conductometer. The portable electrolytic conductivity meter model CM-1F was used for measuring the conductivity of KCl solutions.
4. Gamma ray spectrometer of model number 8100/e 1024 Channels was manufactured by Canberra Industries, Meriden, Connecticut.
5. Thermometer. It's range is from -10.0° to 50.0°C and is able to read in 0.1°C.
6. IBM 370 computer at Nation Statistical Office
7. 50 Microliter hypodermic syringe of model number 2933087 was manufactured by Hamilton Company, Nevada, U.S.A.

3.1.1 Diffusion Cell

A low cost and simple diffusion cell was developed in this work. It consists of three main parts: the chamber unit, the capillary unit and the capillary moving unit, as shown in Fig. 3.1 and Fig. 3.2.



① GLASS ROD
② THERMO-METER
③ CENTER OPENING (19/26)
④ SIDE OPENING (19/26)
⑤ TWO-NECKED FLASK
⑥ BOTTOM OPENING (34/35)
⑦ GLASS VENT TUBE
⑧ CAPILLARY TUBE CAP
⑨ CAPILLARY TUBE HOLDER
⑩ CAPILLARY TUBE
⑪ CAPILLARY TUBE STAND
⑫ SYRINGE CYLINDER
⑬ SYRINGE PLUNGER
⑭ A PIECE OF RUBBER RING
⑮ RUBBER TUBE
⑯ PLASTIC TUBE
⑰ METAL SCREW TUBE
⑱ RUBBER TUBE

Figure. 3.1 The detailed of diffusion cell.

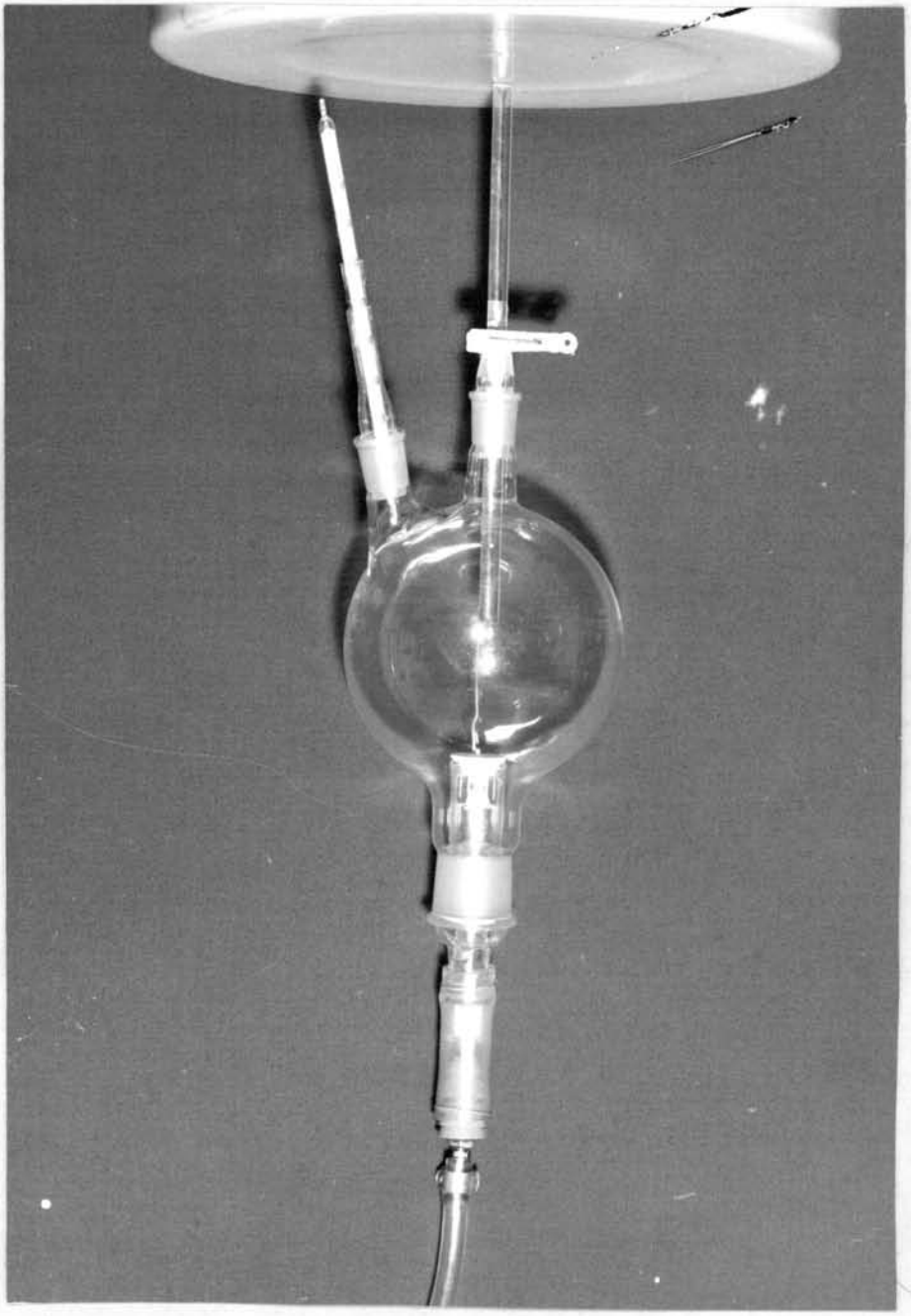


Figure 3.2 Photograph of diffusion cell

The Chamber Unit

The chamber unit is a two-necked flask with bottom opening, made of glass,as shown in Fig. 3.1. The center opening is a ground joint of 19/26 through which a glass rod is placed. The side opening, at an angle of 30 degree to the central line, is also a ground joint of 19/26 through which a thermometer is placed. It is also used for charging and draining solution in the chamber. The bottom opening is a ground joint of 34/35 where the capillary unit is placed.

The Capillary Unit

The capillary unit consists of capillary tube stand, capillary tube, capillary tube holder, and capillary tube cap, as shown in Fig. 3.1. The capillary tube has an inside diameter of 1.1 - 1.2 mm., and a height of 4.5 cm.. Its bottom end is sealed. This capillary tube provides the study of solute diffusion which takes place along its length. The capillary tube holder is made of plastic having two small holes; one for placing the glass vent tube and the other for the capillary tube cap. The capillary tube cap is made of Teflon. It is used for preventing the solution in the chamber and that in the capillary tube from contacting. The capillary tube stand is made of glass and whose outer surface is ground joint of 34/35. It is used to connect the capillary tube holder, the capillary moving unit and the chamber unit.

The Capillary Moving Unit

The capillary moving unit consists of syringe set, rubber tube, plastic tube and metal screw tube. The syringe set comprises of

syringe cylinder, syringe plunger,and a piece of rubber ring, as shown in Fig. 3.1, used to control the level of syringe plunger. The rubber tube is used to connect the lower end of the capillary tube stand and plastic tube. The metal screw tube is used to connect the plastic tube and the rubber tube from the compressed gas tank.

3.2 Chemicals

The chemicals used in this work were as follows:

1N KCl solution

4N HNO_3 solution

Thorium nitrate solution of concentration: 0.8M, 0.6M, 0.4M, 0.1M and 0.05M.

40 per cent Tributyl phosphate-60 per cent Kerosene solution

3.3 Procedure

The capillary unit was first tightly clamped to the stand holder. The capillary tube containing the desired solution with diffusing solute was fitted into the capillary tube holder. The teflon cap was carefully placed adjacent to the mouth of capillary tube. To provide better heat transfer, the space in the capillary tube stand was charged with liquid solution, leaving only a small volume of air in it. This air would escape through the vent tube when the syringe plunger was raised.

After the capillary tube had been placed in the capillary unit, the chamber unit was placed above the capillary unit, and then clamped to the stand in the temperature bath. The chamber was charged with liquid solution without diffusing solute to 3/4 of its height.(about 700 ml. in

this experiment). The thermometer was placed to the proper depth into the liquid solution. And the glass rod was placed into the center opening and held above the surface of the solution with the clip.

The desired temperature of the bath was controlled. The arrangement of the constant temperature bath was as shown in Fig. 3.3. Until the temperature of the solution in the chamber approached that of the bath, the experiment was then started by releasing the compressed gas from the gas cylinder to move the syringe plunger which in turn pushed the capillary tube upward. Consequently the cap was removed from the hole, and the solution in the capillary came into contact with that in the chamber. And solute diffusion started. The full displacement of the plunger was controlled by the rubber ring so that when the plunger ceased to move the mouth of the capillary tube would be at the same level as the surface of the capillary tube holder.

After the 3 hours of diffusion, the experiment was ended by lowering the glass rod to cover the mouth of the capillary tube, to terminate the diffusion. The thermometer was removed. Solution in the chamber was drained. Then the glass rod was removed and the whole diffusion cell was dismembered. The solution in the capillary tube was analysed.

3.3.1 Diffusion of KCl in Deionized Water

The 1N KCl sample was injected into four capillary tubes by using a 50 µl hypodermic syringe and the volume were recored. One capillary tube was kept for reference as the initial concentration the others were used in diffusion experiment. After 3 hours testing period, all samples were analysed by conductometer, as shown in Table 3.1.

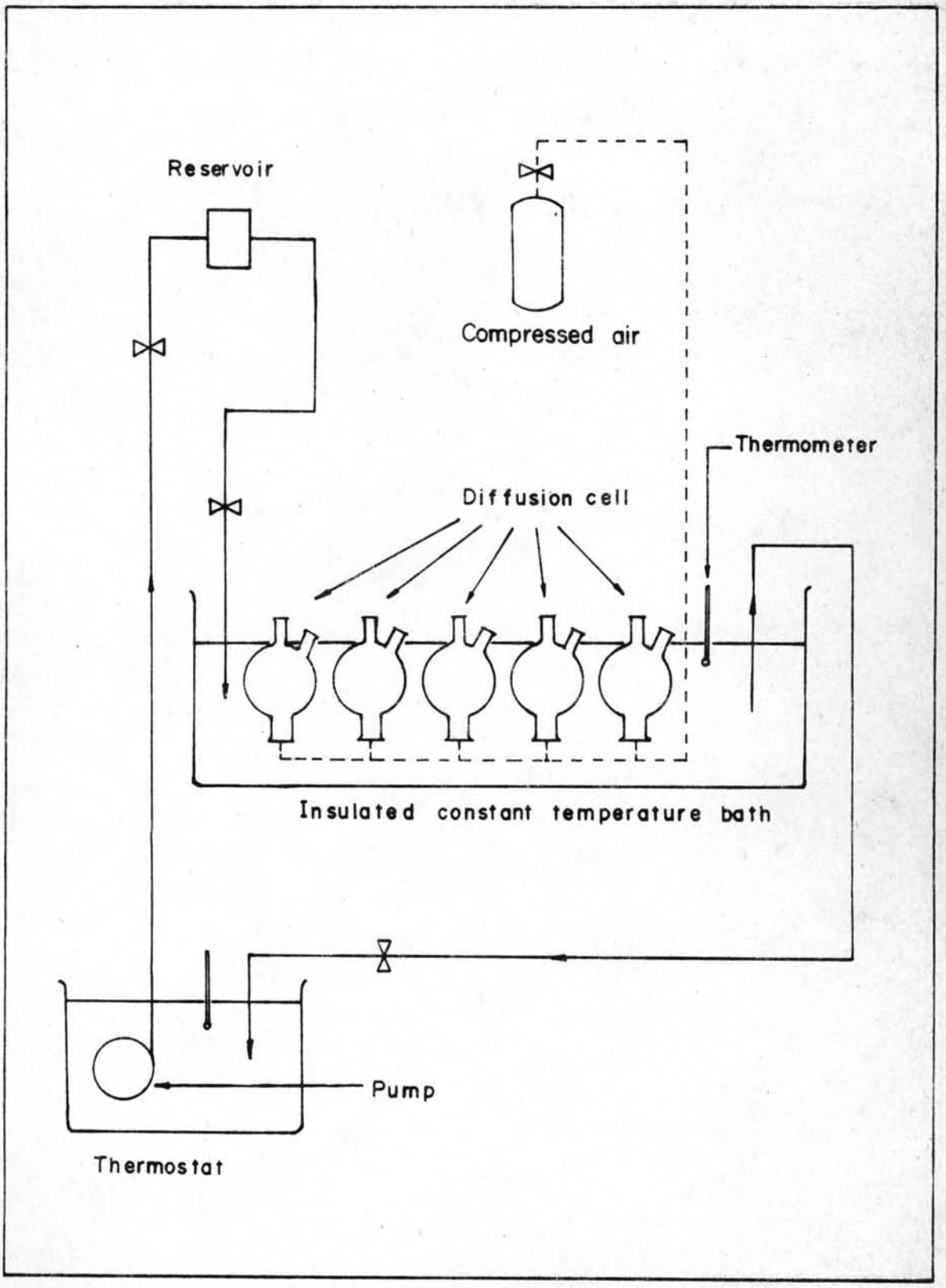


Figure 3.3 Arrangement of experimental apparatus.

Table 3.1 Experimental Data of KCl at 27.8°C

Experimental time 10800 sec.

Run Number	Capillary Volume (μl)	Conductance (μ℧)	Amount of KCl Solution (gm.)	Concentration (gm/ml)
1	45.5	25.5	7.00×10^{-4}	0.0153846
2	46.2	25.7	7.05×10^{-4}	0.0152597
3	46.5	25.9	7.10×10^{-4}	0.0152688
Reference	45.2	27.9	7.65×10^{-4}	0.0169247

3.3.2 Diffusion of $Th(NO_3)_4$ in 4N HNO_3

$Th(NO_3)_4$ in 4N HNO_3 solution of known concentration was injected into a set of two capillary tubes; one was used as the reference for the initial concentration, and the other was used in the diffusion experiment. $Th(NO_3)_4$ of the following concentrations were used: 0.8M, 0.6M, 0.4M, 0.1M and 0.05M. Experiments were carried out at the following temperatures: 10.5°C, 20.0°C, 22.0°C, 28.5°C and 30.5°C. After 3 hours testing period, all samples were analysed by Gamma ray spectrometer. All data were shown in Table 3.2, Table 3.3, Table 3.4, Table 3.5, Table 3.6.

Table 3.2 Experimental Data of $Th(NO_3)_4$ in 4N HNO_3 at 10.5°C

Experimental time 10800 sec.

$Th(NO_3)_4$ Concentration (M)	0.05	0.1	0.4	0.6	0.8
Initial Solution (Count/capillary vol., μl)	164/46	224/46.5	264/46.5	368/45.5	460/46
Final Solution (Count/capillary vol., μl)	147/45	208/45	249/46	360/46	446/45.25

Table 3.3 Experimental Data of $Th(NO_3)_4$ in 4N HNO_3 at 20.0°C

Experimental time 10800 sec.

$Th(NO_3)_4$ Concentration (M)	0.05	0.1	0.4	0.6	0.8
Initial Solution (Count/capillary vol., μl)	131/45	168/45	235/45.2	381/45.5	486/45
Final Solution (Count/capillary vol., μl)	118/44.5	154/45.3	222/45.5	365/46	465/44.5

Table 3.4 Experimental Data of $Th(NO_3)_4$ in 4N HNO_3 at 22.0°C

Experimental time 10800 sec.

$Th(NO_3)_4$ Concentration (M)	0.05	0.1	0.4	0.6	0.8
Initial Solution (Count/capillary vol., μl)	164/46	224/45	264/46.5	368/45.5	460/46
Final Solution (Count/capillary vol., μl)	146/45	205/45.3	244/45.5	346/45.2	434/45

Table 3.5 Experimental Data of $Th(NO_3)_4$ in 4N HNO_3 at 28.5°C

Experimental time 10800 sec.

$Th(NO_3)_4$ Concentration (M)	0.05	0.1	0.4	0.6	0.8
Initial Solution (Count/capillary vol., μl)	131/45	168/45	235/45.5	381/45.5	486/45.3
Final Solution (Count/capillary vol., μl)	119/45.5	152/45.3	216/46	349/45.3	452/45

Table 3.6 Experimental Data of $Th(NO_3)_4$ in 4N HNO_3 at 30.5°C

Experimental time 10800 sec.

$Th(NO_3)_4$ Concentration (M)	0.05	0.1	0.4	0.6	0.8
Initial Solution (Count/capillary vol.,μl)	131/47	158/45	277/46.5	384/47	510/46
Final Solution (Count/capillary vol.,μl)	110/45	160/46	246/46.5	342/46	463/45

3.3.3 Diffusion of $Th(NO_3)_4$ in 40% Tributyl phosphate-Kerosene Solution

The experimental procedure was the same as diffusion in 4N HNO_3. The desired solution in capillary tube was 0.8M $Th(NO_3)_4$ in 40% tributyl phosphate-kerosene solution. The experimental data was shown in Table 3.7

Table 3.7 Experimental Data of 0.8M $Th(NO_3)_4$ in 40% Tributyl phosphate-Kerosene Solution at 30.5°C

Experimental time 10800 sec.

$Th(NO_3)_4$ Concentration (M)	0.8
Initial Solution (Count/capillary vol.,μl)	7913/46
Final Solution (Count/capillary vol.,μl)	7380/45.8

3.4 Analysis of Solutions

3.4.1 KCl Solution

Concentration of KCl solutions were measured by the conductometer. A calibration curve (see Appendix 1) was first constructed. It was a plot of concentration versus conductivity. The concentration of KCl solution in a capillary tube was determined as follows.

The capillary tube was broken in a beaker. 100 ml. of deionized water was then added and stirred. The conductivity of the dilute solution was measured and concentration was read from the calibration curve.

3.4.2 $Th(NO_3)_4$ Solution

The gamma ray spectrometer (see Appendix 2) was started up by setting of the following: high voltage power supply 900 volts, amplifier had coarse gain 20, fine gain 0.5, discriminator had LLD 0.1 volt ULD 10 volts. The capillary tube which contained $Th(NO_3)_4$ solution was placed into the detector's chamber. The time was set up and then the ray was counted at channel number 132-181 which was $^{208}_{81}Tl$ spectrum (energy 2.62 MeV). The back-ground count was also determined.