CHAPTER 4

EXPERIMENTALS

4.1 Materials

The starting materials of reagent grade were used for this research work. Hygroscopic compounds were kept in a desiccator over the anhydrous silica gel. Some chemical compounds with their important properties were shown as an example in the following table, but the whole lot is in Appendix I.

Table 4.1 Collection of chemical reagents with their properties (36).

Chemical reagents	Physical properties in solid crystal
$AgNO_2$	white,
$AgNO_3$	colorless,
Ag_2SO_4	white,
$Al(C_2H_3O_2)_3$	white
$Al(NO_3)_3 \cdot 9H_2O$	colorless, deliquescent,
$Al(OH)_3$	white,
$AlPO_4$	white,

4.2 Preliminary investigation of solid reactions

From 170 reagents, two solids were placed in contact with each other in plastic plate in circular form as in the figure 4.1

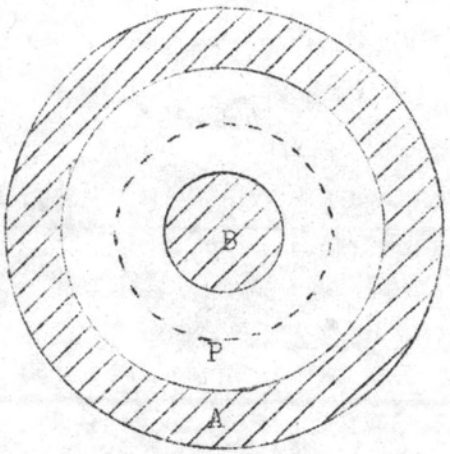

Fig 4.1 Reaction of solids A and B to give product P.

The changes of colour in contact area were observed. The product P was formed with its colour different from reactants when solid A reacts with solid B. For each set of experiment, solid A was kept constant and B was changed by permutation method.

4.3 Study of kinetic and mechanism of interesting reactions

The interesting reactions selected for mechanism study were carried out in semi-micro test tubes and the length of each species was measured at various times.

4.4 The comparison study with instrumental method.

The pettri dish of 5 cm. diameter was used to cellect the reaction products for instrumental analysis. The selected methods of analysis were X-ray fluorescence technique which could measure only percentage of heavy elements, infrared spectrophotometry and X-ray powder techniques the later two methods being used to show the difference between reactants and products with respect to structures. The experimental data indicated explicitly the occurence of products by solid-solid reactions.

4.4.1 X-ray fluorescence study.

The quantity of heavy atoms was analyzed by X-ray fluorescence techniques with gamma ray source (Pm^{147}/Al isotope) and Si (Li) detector were used. Diagram of the instrument was shown in the following.

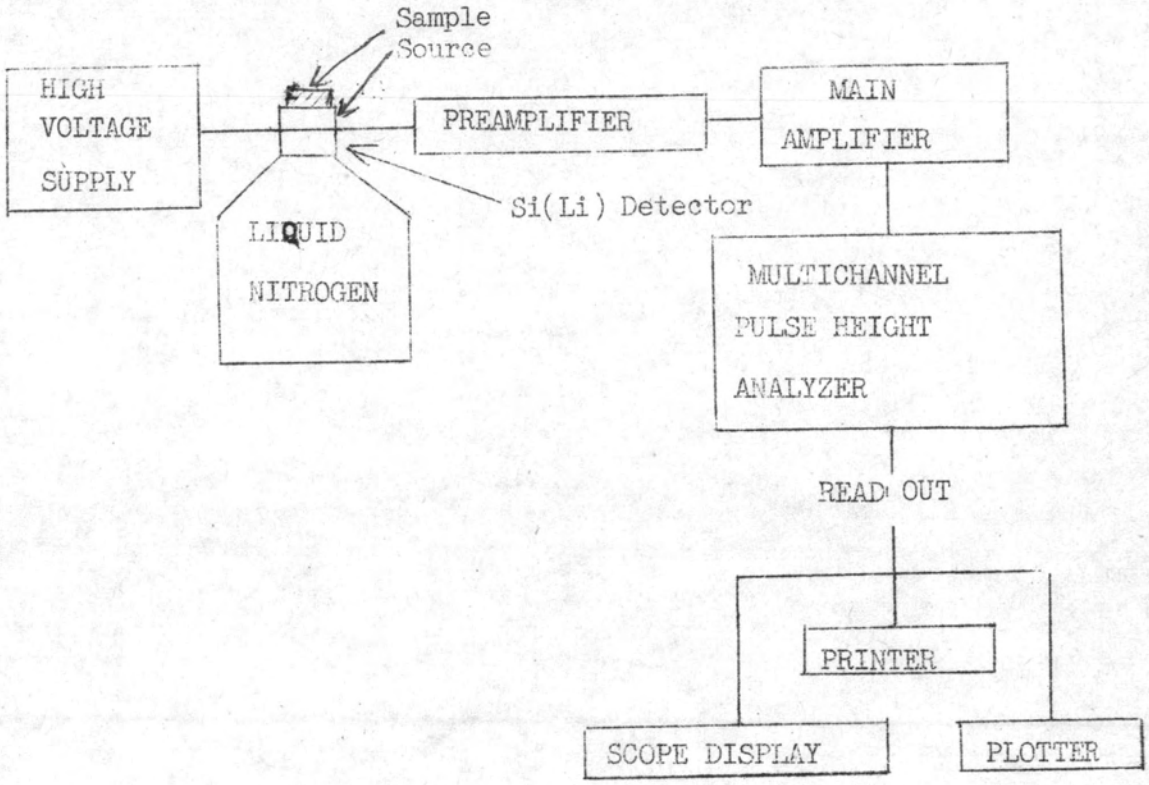

Fig 4.2 Block diagram of the X-ray fluorescence.

4.4.1.1 Preparation of standard sample.

Internal standard (same element) method was used, standard sample was prepared by varying weight of reactants, solid A and B, to give totally 5 gm. Solid A and B was mixed thoroughly in motar to give a homogeneous mixture. Then the percentage of known heavy element could be calculated.

4.4.1.2 Percentage determination

The measuring solid was contained in the plastic container of 15 mm. diameter and 20 mm in depth, where the sample thickness of 10 mm

was the optimum one for eliminating the absorption enhancement effect. The container was placed at fixed position on Si(Li) detector and irradiated with Pm^{147}/Al isotope gamma source about 400 seconds then multichannel pulse height analyzer was switched on and the printer was used.

The energy of K_α or L_α or etc. band, which was used to give a maximum peak that refered to maximum yield of scattering, depend on the nature of element. Each element was observed at the different range of channel.

4.4.2 Infrared spectrophotometric studies.

Infrared spectra were obtained with a Pye Unicam Sp 200 G Grating Infracord Spectrophotometer. The dried KBr was well mixed with dried sample. The mixture of KBr with any entity, reactant or product, was grounded thoroughly to achieve homogeneous mixture which was transferred evenly into a standard Pye Unicam die (13 mm). The Blackhawk enerpac model P - 39, for 10,000 Psi, was used in making pellet of mixture under pressure of 3,000 Psi. The optimum wave number of infrared spectrophotometer was scanned between the range of 4000 cm^{-1} to 667 cm^{-1}.

4.4.3 X-ray powder diffraction studies

The principle of the identification of substance by X-ray powder diffraction is based on the fact that each crystalline substance produces its own characteristic pattern which can be used to compare and identify the type and structure of compound.

The powder method seems to be convenient and provides many useful information. The X-ray powder photographs were recorded at 26°C with the Guinier-Hagg XDC-700 focusing powder camera using CuK α_1 - radiation (λ = 1.54051 Å), 34 KV, 21 mA exposed for two hours. Silicon was used as the internal calibration standard. The result pattern was used to compare between the products and reactants.

4.5 Conductivity measurement

4.5.1 Preparation of sample

The pellet of 2 mm thickness of the exact quantity of dried solid compound was made under pressure of 3,000 Psi by the same Blackhawk onerpac.

4.5.2 Apparatus

The Yew microammeter, milliammeter with scale 0-3 0-30,0-10, 0-100, the Yew voltmeter with scale 0-30, 0-100, 0-300, 0-1000 and Unicam power supply with 0-100 volt were connected together with two carbon electrodes of 1 cm. diameter and 10 cm. length with one of their end were tipped with copper plates which joined with copper wire. Both electrode and sample were held together within the 15 cm. long glass-tube as in the Figure 4.4:

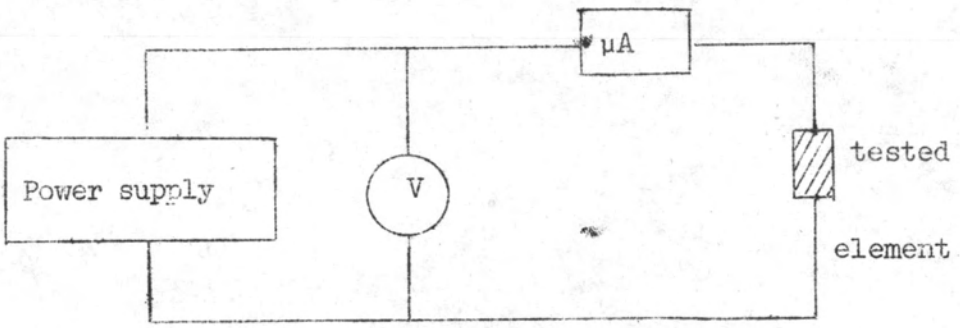

Fig. 4.3 The electric circuit for conductivity measurement in solid state.

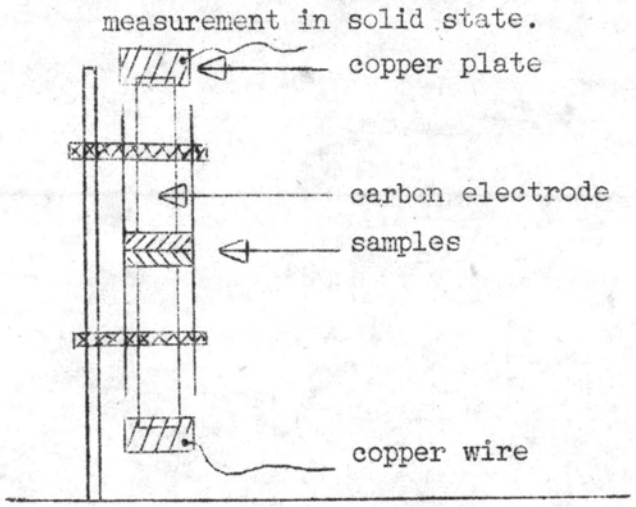

Fig. 4.4 The enlargement of tested element part.

4.5.3 Conductivity measurement of reactants and reactions in solid state.

Pellet samples were kept dry before use in desiccator over the anhydrous silica gel. To study the conductivity behaviour of reactants, its pellet was placed each time between two carbon electrodes. The electric current was measured when the voltage power supply was varied.

To study the characteristic conductivity of reactions, two pellets of material were assembled between carbon electrodes. The voltmeter was scanned for the appropriate value and then kept to be constant for each pair of reaction. The electrical current was noted immediately and carried on at every given time through the whole experiment.

The plot curve of current against time at constant voltage displayed the characteristic diffusion while each reaction was in progress.