CHAPTER 2

DESCRIPTION OF THE INSTRUMENTS EMPLOYED
AND
GENERAL EXPERIMENTAL PROCEDURES
A BRIEF DESCRIPTION OF THE INSTRUMENTS EMPLOYED

1. **MANUAL POLAROGRAPH**:

   The complete set up manufactured by Toshniwal Bros. (Pvt.) Limited used in the polarographic measurements consists of the following:


   The voltage applied to the electrodes of the cell can be varied in steps of 10 and 100 mV's, to give a voltage range of 0 to ±2.39 volts. There are nine sensitivity factors ranging from 1/1 to 1/500. A universal shunt provided in the instrument controls the sensitivity of the galvanometer. A built-in standard cell enables accurate calibration of the potentiometer. The standardization may be carried out at any time during the analysis without any disturbance to the circuit.

   A stout metal pillar fixed to a heavy base constitutes the electrode stand. A sliding clamp for the dropping mercury electrode capillary tube and a sliding carrier for the mercury reservoir, allow the adjustment of the reservoir in relation
to the capillary. The connection between capillary and reservoir is made with a polythene tubing.

The cell employed in the investigation is placed into a holder on the base of the stand. A platinum wire sealed through the bottom of the cell makes contact between the mercury pool in the cell and the positive lead of the applied EMF. Hydrogen is passed through a side tube provided to the cell.

The polarograph is coupled with an "OSAM" Reflecting Spot Galvanometer No.6G73-26 and is operated at 220 volt A.C. mains supply. The maximum sensitivity of the galvanometer is $4.8 \times 10^{-2}$A per division.

2. SPECTROCOL:

Photometric studies were carried out with Spectrocol model CL-23 manufactured by ELICO Private Limited, Hyderabad, India. Transistor stabilized light source ensuring constant intensity even during mains fluctuation is used as a light source in the colorimeter. The instrument has a gradient density interference filter having a narrow band-width. The use of a narrow slit facilitates in a better scanning width of the spectrum. The gradient intensity filter enables the wave length to change continuously from 400 to 700 nm of the entire visible range. A photocell acts as a detector. A panel meter with antiparallax
mirror scale calibrated in optical density and percentage transduction is used to read out the absorbance. The sample solution can be placed in matched test tubes. The wave length can be changed to a desired value by a thumb operated knurled circular drum on a calibrated scale. An automatic shutter arrangement in the sample chamber protects the photocell from direct illumination from the lamp in the absence of the test solution.

3. **pH METER**

The pH measurements were made using MLCO pH Meter, model MI-10 manufactured by MLCO Pvt. Ltd., Hyderabad, India. The instrument operates on A.C. mains and is provided with two scales – one reading from 0.0 to 7.0 and the other from 7.0 to 14.0 pH. Provision is made for temperature compensation from 0 to 100°C with a built-in temperature control in the instrument. A glass electrode EM-60, Beckman type in the range 0-13 pH and a saturated calomel reference electrode are employed. A buffer of pH 4.0 (sodium acetate and acetic acid) in the acid range and a buffer of pH 9.0 (Borax buffer) in the alkaline pH range are used to calibrate the instrument. The instrument records the pH to the second decimal. The pH is read directly on a panel meter with antiparallax scale calibrated with pH and potential.
After switching on the instrument the pointer of the meter is set to 0 mV. or 7 pH position by means of the control. This control is used only when the "Selector" switch is in 'Zero position'. By means of this selector switch the function of the instrument is selected, i.e., to correct zero drift, pH measurement or mV measurement. "Set buffer" control introduces a potential to cancel the asymmetry potential of the glass electrode. The control is operated while calibrating or standardizing the instrument with a standard buffer solution.

GENERAL PROCEDURES EMPLOYED IN THE STUDIES:

1. Extraction Studies:

Requisite volumes of the metal ion solution and the reagent solution are added to the buffer solution taken in a 50 ml separating funnel. The volume of the buffer solution is always more than fifty per cent of total volume of the reaction mixture. A known volume of the reaction mixture is shaken well with equal volume of the reaction mixture n-butanol for about 10 minutes for equilibration. In such cases where the two layers (organic and aqueous) are not separated quickly or homogeneous solution without the separation of layers is formed, addition of salting out agent such as a saturated solution of magnesium sulphate is used for quick and clear separation of the two layers.
2. **Optical Studies:**

The absorbance of the organic and aqueous layers are measured in the visible range 400-700 nm by taking the solutions in the glass test tubes provided with the instrument. Water and n-butanol are used as solvent blanks for aqueous and organic layers respectively. The measurements are repeated in each case three or four times for the sake of reproducibility. From the results the optimum pH required for the extraction polarographic study is decided.

3. **Polarographic Studies:**

6 ml of the organic layer is taken and is mixed with 5 ml of aq. supporting electrolyte directly in the polarographic cell and 1.5 ml of methanol are added to get a homogeneous solution. After degasifying the solution by passing pure hydrogen gas for about 30 minutes the current-voltage readings are recorded manually. Polarographic behaviour of the aqueous layer is also examined at each pH of the extraction after adding the same aqueous supporting electrolyte. From the data obtained in the studies optimum pH required for extraction polarograph is confirmed.

4. **Extraction Polarography:**

To a known volume of the optimum buffer solution,
different aliquots of the metal solution, and excess of the resacetophenononeoxine solution are added. The total volume of the reaction mixture is kept constant by adding requisite volume of conductivity water. Known volumes of solutions of salting out agent and the synergistic agent (if necessary) are added to the reaction mixture. n-Butanol of volume equal to that of the reaction mixture is added to the contents of the separating funnel and shaken for five minutes for equilibration. The layers are allowed to separate clearly. Known volume of the organic layer is taken and polarogram recorded as described in procedure 3.

A graph is drawn between the amounts of the metal ion taken and the limiting current of the polarogram to examine the linear proportionality between the two. The above procedure is repeated in presence of large amounts of ferric iron, zinc and nickel to study their influence on the extraction polarographic determination of the metal ion under study.