# Chapter III

## Chemicals and Instruments

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CHEMICALS AND INSTRUMENTS

CHEMICALS:

Cobalt (II) and Nickel (II) sulphates (CoSO₄·7H₂O & NiSO₄·7H₂O) of Analar quality were utilised for the present investigations. All the ligands were of B.D.H., Analar, E. Merck or equivalent reagent quality. In order to confirm the purity of the metal salts, they were estimated in the following manner:

(a) Estimation of Cobalt:

Cobalt was estimated gravimetrically by the usual method¹ using \( \Lambda^-\text{nitroso} - \beta^-\text{naphthol} \) as the precipitating agent.

(b) Estimation of Nickel:

The nickel content of the salt was estimated gravimetrically using dimethylglyoxime² as the precipitating agent.

The solutions of the above metal salts were prepared by dissolving the required quantity of the analysed samples in conductivity water. Solutions of the desired concentrations were then prepared from the stock solutions by diluting them with conductivity water.

Preparation of Cobalt and Nickel perchlorates:

Accurately weighed amounts of the cobalt and nickel chlorides were dissolved in a calculated quantity of perchloric acid and the solutions were
heated till complete removal of chloride ions. On cooling to room temperature, the volume was made up to the mark. The concentrations of the metal ions in the cobalt and nickel perchlorate solutions were estimated as cited above. The amount of free perchloric acid present was also determined and was neutralized by adding the required quantity of standard KOH (Carbonate free) solution.

**KOH Solution:**

Carbonate free KOH solution was preferred to sodium hydroxide in pH titrations, because when a high ratio of sodium to hydrogen ions exists, the sodium ions penetrate the glass membrane and give a false reading, while a higher ratio of potassium to hydrogen ions can exist before accuracy was affected.

The KOH pellets used for preparation of solution usually have the carbonate deposited on the outer surface in minute quantity. Therefore, to obtain carbonate free KOH solution, Ba(OH)$_2$ was added to the potassium hydroxide solution. The precipitate of BaCO$_3$ was allowed to settle and the excess of Ba$^{++}$ was removed by passing the solution through a column of ion exchange resin (Amberlite IR 120) which was quantitatively in the potassium form. The eluate was a solution of pure KOH free from carbonate. A decinormal solution of it was prepared and stocked in polythene bottle.

**Nitrogen:**

Nitrogen was freed from oxygen by bubbling the gas through pyrogallol solution.
Sodium perchlorate solution:

The sodium perchlorate solution required for maintaining the ionic strength constant was obtained by dissolving 39.635 gms. of sodium perchlorate (risted) in 250 ml. distilled water.

Dioxane:

The dioxane of R. D. H. quality was purified by the procedure described by Weisberger. ³

Metal salts and ligand solutions:

Standard solutions of the cobalt and nickel salts were prepared by dissolving the required quantity in double distilled water, and the standard was checked by estimating their metal contents as described earlier.

Standard solutions of the ligands were prepared by dissolving the definite quantity or definite volume (in the case of amines) in double distilled water.

The sodium salt solutions of organic acids were prepared by dissolving the required amounts of organic acids in distilled water and mixing equivalent quantities of A.R. sodium carbonate to it. The solutions were then heated for some time to remove carbon dioxide. On cooling to room temperature the volume was made up to the mark.

Freshly prepared solutions of metal salts and ligands were used in all the experiments to avoid hydrolysis.
Buffer solutions:

Standard buffer solutions of pH 4.1, pH 7.0 and pH 9.2 were prepared by dissolving the "Beckman buffer tablets" in distilled water.

INSTRUMENTS:

Conductometer:

Conductometric measurements were made on a Dornan high precision conductivity bridge with a WTW 1000 cycles Oscillator and a compact diptype conductivity cell together with an "Omega" type headphone. Before starting the experiments, the cell was cleaned with chromic acid and then plated² from a solution of chloroplatinic acid.

pH-meter:

An "Lico pH-meter, model LI-10, with glass and reference electrode assembly was used for pH measurements. The current supply was stabilized by A.C. Voltage stabilizer with a frequency of 1000 cycles/second. The pH-meter was readable up to 0.05 pH and this could be extended to 0.01 by approximation. Before starting the experiments, the pH-meter was standardized with the standard buffer solutions.

Spectrophotometer:

Beckman DU quartz spectrophotometer was used for absorption measurements in the ultra violet region for the range 200 - 400 millimicrones (m.μ), the source of light being a hydrogen lamp, operated by means of A.C. power supply, connected in series with a constant voltage transformer. Four matched rectangular silica cells of 1.0 cm. light path were used for containing reference
(solvent) and the specimen solutions. The cells were numbered so that they could always be placed in the same order in the metallic adapting during the experiments. The cells were washed thoroughly and dried every time before introducing a new sample in them.

At first, keeping the solvent cell in the beam at the selected wavelength indicated on the dial, the selector switch, the sensitivity control knob, and the dark current control knob were adjusted for zero of the meter reading. Then the specimen cell was brought in the light path by sliding the platform. Now operating the transmission knob, the pointer was again brought to zero position. The reading on the dial, then, indicated the optical density at the particular wavelength.
REFERENCES

1. Vogel, A.I.,

2. Vogel, A.I.,

3. Weissberger, A. and Proskauer, E.S.,

4. Vogel, A. I.,