CHAPTER II

EXPERIMENTAL

3.1 Reagents

3.1.1 Solids

These reagents were purchased from Merck-Schuchardt:

Chromium (III) acetylacetonate

Cobalt (II) acetylacetonate

Cobalt (III) acetylacetonate

Copper (II) acetylacetonate

Iron (III) acetylacetonate

Manganese (II) acetylacetonate

Manganese (III) acetylacetonate

Nickel (II) acetylacetonate

Palladium (II) acetylacetonate

Zinc (II) acetylacetonate

3.2.1 Liquids

The reagents from Merck and BDH which were not analar grade were double redistilled prior to use. See Table of Solvents. (p.17)

3.2 Procedure

A Unicam SP 200 G grating infrared spectrophotometer was used throughout this work. Resolution: $\pm$ 1 $cm.^{-1}$ (2000 - 650 $cm.^{-1}$).

3.2.1 Solid spectra

The solid sample were prepared by employing the KBr disk method and recorded in the range between 4000- 650 $cm.^{-1}$ Calibration

of the frequency reading was made with polystyrene film.

2.2.2 Solution spectra

The acetylacetonate metal complexes were dissolved in various organic solvents until solution becomes saturated. The solution is then filtered off and put into a liquid variable path cell and then recorded immediately in order to minimize the effect of any possible reactions between the complexes and the solvents in the range 1750-1250 $cm.^{-1}$ The path lengths used were about 0.1-0.3 mm. depending on the solubility of complexes.

Since only one liquid variable path length cell is available in this laboratory, the solvent absorptions cannot be removed and in some cases, $\Delta\nu$ is difficult to read. However, by running a series of solutions with different concentrations; saturated, diluted 3 times, 5 times and 15 times, $\Delta\nu$'s can be read. The concentrations used were about 0.03-0.001 M.