CHAPTER - II

EXPERIMENTAL TECHNIQUE

INTRODUCTION :

The spin-lattice and spin-spin relaxation times (T_1 and T_2) are determined by using commercially available Bruker PC 120 NMR process analyser. The block diagram of the instrument are as shown in the figure (2.1) . This is a compact and easy-to-operate instrument that can provide with variety of information. Its common application includes, automatic measurement of solid/liquid ratios in foodstuffs, measurement and calculation of nuclear-magnetic resonance (NMR) relaxation times (T_1 & T_2) in liquids and organic tissues, and the determination of water content of various substances. The major components of the instruments are magnet module and control module.

MAGNET MODULE

The magnet module houses the magnet unit, the radio frequency preamplifier, and the probe head. The latter includes the transmitter, receiver coil and associated tuning circuitry. The magnet unit consists of a permanent magnet and a magnet-temperature-control unit. The magnet has a field strength of 4.7 Kilogauss ,an air gap of 20 millimeter , and a weight of 45 kilograms. The temperature control unit employs multiple temperature sensors and dual heater / fan units to thermostat the magnet.

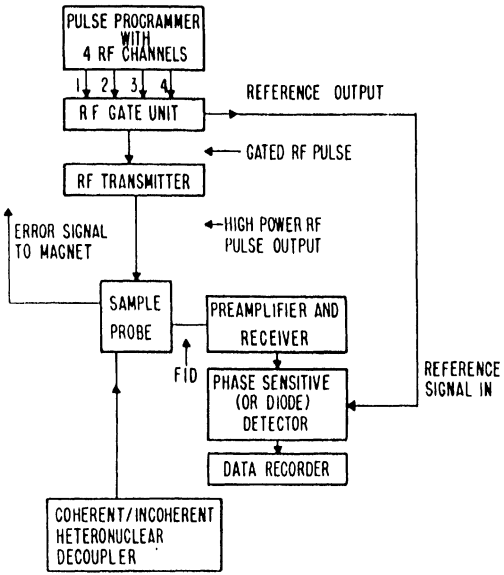

FIG 2.1 Block diagram of a versatile bruker PC-120 NMR Process Analyser.

CONTROL MODULE

The control module contains three main sections ; the power supply, the radio frequency circuitry, and the microprocessor unit.

POWER SUPPLY

The power supply provides all necessary DC voltages for operation of the microprocessor unit and the radio frequency pulse-generator circuits. It is powered by a normal 117-Volt AC power source and requires approximately 200 Watts.

RADIO FREQUENCY CIRCUITRY

The radio frequency consists of those components necessary to generate the correct radio frequency pulse for irradiating the sample (20 MHz in the case of H protons, less for other nuclei). In addition, it includes the amplification units that boost the pulse to the power levels needed to include an NMR response from the sample being analysed.

MICROPROCESSOR UNIT

The microprocessor unit is the "brain" of the instrument, monitoring and controlling the operation of the instrument. It is the most complex portion and consists of four major sub assemblies:

(1) The central processing unit (CPU):

It contains the microprocessor itself, which decodes and carries out the commands of the instrument control program, the read only memory (ROM), and the random access memory (RAM), the random access memory (RAM), a temporary or "working" memory for storing various numbers and calculation results. This subassembly also contains several components that support the operations of the microprocessor.

(2) The interface control consists of those components that enable the microprocessor to communicate with various peripheral devices. They include the digital balance etc.

(3) The programmable pulse sequence generator (PSG) produces the sequences of radio frequency pulses that are beamed into the sample being analysed.

(4) The analog-to-digital converter (ADC) converts the NMR signal (a signal voltage level) into a number that can be interpreted by the microprocessor.

THERMAL PRINTER

The thermal printer consists of the print mechanism and interfacing electronics. It uses a 5-by-7 dot matrix and prints 15 characters per line. The printer provides a record of results, parameter values used in the analysis.

KEY BOARD

This is used to change the various parameters of the pulse sequences, mode of detection of the signal (diode or

phase sensitive detector) gain of the amplifier etc.

Measurement Of Density (ρ)

The density (ρ) of the solutions at various temperatures are determined using a pyknometer of capacity of 10 ml. The pyknometer containing the solution is allowed to attain the temperature at which the density is to be measured and it is weighed with a single pan balance accurate to fourth place. By knowing the volume and weight of the solution the density is calculated at various temperature.

Measurement Of Shear Viscosity (η_s)

The viscosity (η_s) of the liquid mixtures was measured by an Ostwald's Viscometer. By measuring the time of flow of the solutions between the viscometer marks, the viscosities were determined using the relation

$$\eta_s = \left[\frac{\rho}{\rho_o}\right]\left[\frac{t}{t_o}\right]\eta_o \quad \ldots\ldots\ldots (2.1)$$

where ρ , η_s and t are respectively the density, shear viscosity and time of flow for liquid mixtures, ρ_o , η_o , t_o are the corresponding quantities for water. The measurements were made at different temperatures is kept immersed in the thermostatically controlled water bath.

CHEMICAL SHIFT :

The high resolution NMR spectra are recorded using a Varian EM 390 NMR spectrometer in the concentration range 0.01 to 0.13 mole fraction of the acid. The RF field was kept sufficiently low to avoid saturation effects. The chemical shift δ for protons in these solutions is calculated with reference to CH_2 signal of dioxan .