CHAPTER IV

MATERIALS AND EXPERIMENTAL PROCEDURE

4.1 ^{57}Co - Pd Source and Absorber

The source is ^{57}Co diffused into a palladium matrix (6 μm thick), which was supplied by the Radiochemical Centre, Amersham, having an activity of about 5 mCi. The source foil is rigidly cemented in an aluminium alloy holder. The holder is provided with a female thread to allow attachment to the transducer (see Fig. 4.1). The active area is about 0.503 cm^2 (8 mm diameter). The 14.41 KeV Mössbauer gamma rays are produced by decay of ^{57}Co which is transformed by K capture into 136.32 KeV state of ^{57}Fe. After 8.9×10^{-9} seconds this level decays with the gamma emission of 122 KeV to the 14.41 KeV level. After 9.8×10^{-8} seconds the 14.41 KeV level decays through internal conversion or gamma emission of 14.41 KeV to the ground state of the ^{57}Fe nucleus. The decay scheme of ^{57}Co is shown in Fig. 4.2. By the laboratory measurements, the line width Γ of the source and the recoilless fraction are 0.097 mm/sec and 0.65 respectively.

The natural iron absorber (0.025 mm thick) which was supplied with the source, was used for velocity calibration of the spectrometer. The procedure of calibration will be mentioned later.

The absorber used in our study is made from $CoFe_2O_4$ ferrimagnetic compound, which was grown in the Solid-State Physics Division,

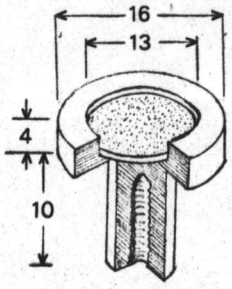

Fig. 4.1 Source holder (dimension in millimeter).

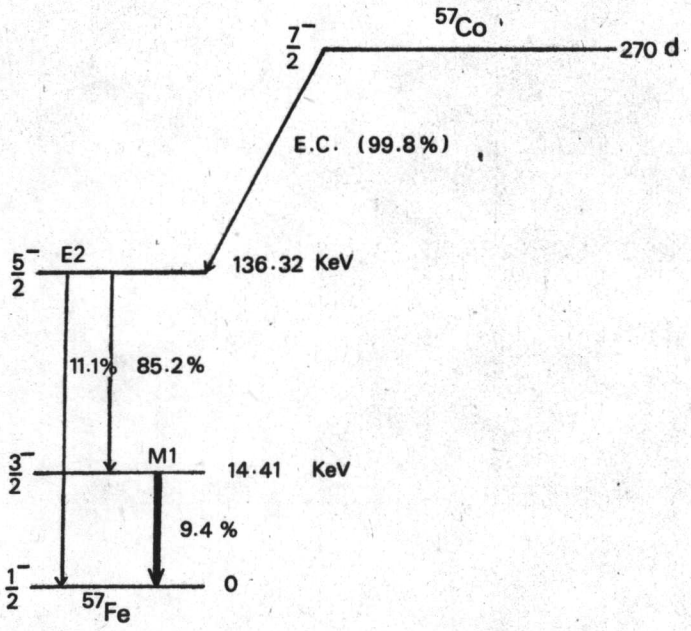

Fig. 4.2 The decay scheme of ^{57}Co.

Bhabha Atomic Research Centre, Trombay, Bombay. It was crushed to a fine powder by using an agate mortar and pestle and spread on self adhesive tape. In this way a suitable Mössbauer absorber was obtained with a thickness of about 200 mg/cm^2.

4.2 Detection System

As mentioned in chapter I, the gamma rays of the radioactive Mössbauer source were measured by the CANBERRA 1702 X-Ray detector. It is a Monoline crystal assembly which includes a high resolution NaI(Tl) crystal, a photomultiplier tube and voltage divider network. A low noise, charge sensitive preamplifier is built into the model 1702. Featuring a thin window (0.005" Beryllium) the detector is useful in low energy x-ray counting and spectroscopy applications. Transmission at 3.5 KeV is 50 % and at 6.0 KeV is 88 %. A removable brass collimator with a $\frac{1}{8}$" x $\frac{1}{2}$" slit is also provided. The equipment used for the system are :

ORTEC 401 A	Bin and Power Supply,
ND IM 88-0297	Bin and Power Supply,
CANBERRA 1702	X-Ray Detector,
ORTEC 456	High Voltage Power Supply,
CANBERRA 2010	Spectroscopy Amplifier,
ORTEC 406 A.	Single Channel Analyzer (SCA),
ND 560	Analog to Digital Convertor (ADC),
ND 2400	Multichannel Analyzer (MCA),

ND Type Writer Drive,

ND 316 Auto Finger,

IBM Selectric Type Writer,

TEKTRONIX T 935 Oscilloscope,

TELEQUIPMENT D 83 Oscilloscope,

Connecting Cables,

AMERSHAM CTD 123 Mössbauer Source (^{57}Co - Pd),

AMC 2084 Variable Energy X-Ray Source,

Aluminium Table with 5 Wooden Shelves (14" x 14" x 24").

The arrangement of the system is shown in Fig. 4.3.

ND 2400 MCA operated in pulse height analyzer mode, and incorporated with the nuclear pulse handling system, as shown in Fig. 4.3 of path 1, were used to detect and analyze the gamma spectrum of the Mössbauer source. The distances from source to absorber and absorber to detector are 6 cm and 5 cm respectively. The gamma rays passes through 4 mm. diameter hole of Pb collimator and absorber to the detector. A 29.95 mg/cm^2 aluminum foil was placed between the source and detector to reduce the intensity of the low energy X-rays. Using the AMC 2084 as standard sources, the calibration (energy/channel) was obtained. In path 2, the 406 A SCA was used to selected only the 14.41 KeV. Mössbauer gamma rays by adjusting its upper and lower discriminator. The Mössbauer spectra were obtained using path 3 with the velocity spectrometer described in chapter III. MCA operated in time mode and using 1024 channel was used for data accumulation. The analyzer was stepped from channel to channel at a constant rate

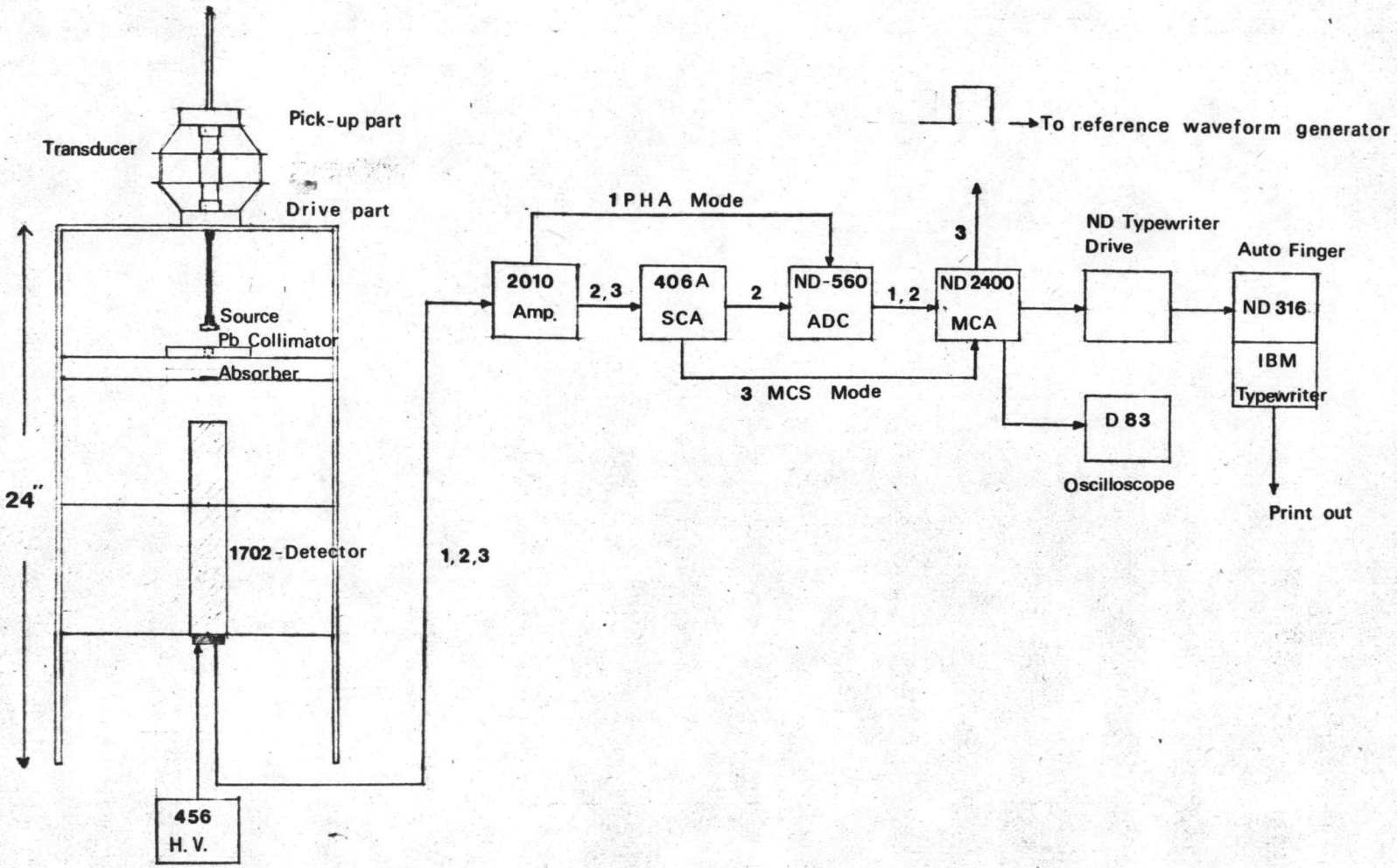

Fig. 4.3. Electronic block diagram for low energy gamma-ray spectroscopy used in Mössbauer study.

of 40 μsec/channel by an internal quartz-controlled oscillator. The velocity reference waveform, a triangular wave, is about 24.4 Hz having the amplitude of 300 mV_{p-p}.

Both the source and absorber were kept at room temperature. The velocity calibration (mm/sec channel) was done by looking at the magnetic hyperfine splitting in a natural iron absorber. The distance between absorption peaks 1 and 2, 2 and 3, 4 and 5, and 5 and 6 should be equal in a thin absorber of natural iron absorber and correspond to 2.244 mm/sec at 298°K (Preston, et al., 1962, Brafman, et al., 1966; Kalvious, et al., 1972). While the distance between the peaks 2 and 4, and 3 and 5 correspond to 3.924 mm/sec, and the magnitude of the total hyperfine spectrum splitting (peaks 1 - 6) corresponds to 10.657 mm/sec. A typical spectrum using a 0.025 mm natural iron absorber is shown in Fig. 5.5. Measuring times were 24 and 50 hours respectively for natural iron and $CoFe_2O_4$ absorbers.

The data were typed out by an IBM selectric typewriter driven by a Nuclear Data Autofinger. As mentioned in chapter III, the spectrum in the second half is the mirror image of the spectrum in the first half. To obtain the true velocity spectrum the contents of the appropriate channels have to be added. In this spectrometer, the velocities represented by channels 515-n equal to those of channels 516 + n ; n = 0, 1, 2, ...,507, and channels 7 - n equal to those of channels n ; n = 0, 1, ..., 4. This allows one to add the spectra obtained in the two halves of the analyzer without line broadening.

After the "folding procedure" has been applied, the Mössbauer spectra were computer analyzed by a least-squares fit to a Lorentzian line shape. Curve fitting was done on the IBM 370/145 computer at the Ministry of Finance with a LGFIT 2 program (von Meerwall, 1975). All of the results will be presented in the next chapter.