CHAPTER 3
METHODS OF STUDY

In this chapter all techniques employed for obtaining the palaeomagnetic results are described.

Preparation of specimens

Oriented block samples collected in the field are brought to the laboratory. Each block sample is oriented in the laboratory with the help of level lines on the sides and is fixed in that position by using a mixture of cement, sand and water. A number of parallel lines to the magnetic north direction are drawn in waterproof ink on the top of the sample and the north direction of each line marked. In this position the vertical of the specimen is the vertical in the field and horizontal lines indicate the horizontal plane in the field. The samples after cementing are cured for about a week. They are now ready for drilling and obtaining cylindrical specimens. This is done by mounting on a drilling machine table beneath a diamond impregnated coring bit with a facility to run water through the bit for cooling the coring bit and the sample. The sample is then drilled slowly to obtain a straight cylinder of rock of 2.2 cm diameter and length varying between 4 to 10 cms depending on the thickness of the sample. The core is broken loose from the sample and the bottom and top cut smooth by using cutting machine with a diamond saw. Depending on the length of the core it is cut into one, two or three specimens. Each specimen is numbered and the north line transferred onto the top of it. The axis of the cylindrical specimen is the vertical in the field and the top or the bottom surface which are parallel to each other represent the horizontal plane in the field. If the cylindrical specimen is rotated with its north direction pointing to the geographic north, then the specimen is oriented in the laboratory as it was in the field. The specimens are suitably numbered to identify the site and the sample from which the particular specimen was derived. For example NLP1-1a number indicates that the specimen ‘a’ has come from core 1 of the sample 1 from NLP site- Nallalingayapalli site. The specimens are now ready for measurement of
magnetic direction in terms of declination (D), inclination (I), intensity of magnetization (J_n) and susceptibility (χ).

**Measurement of preliminary directions**

The first measurement on a specimen is made for its Natural Remnant Magnetization (NRM) comprising of D, I and the NRM intensity (J_n). This is called the preliminary measurement. Measurement of these parameters over a batch of specimens usually suggests the nature of the magnetization in the rock unit.

These preliminary measurements are made using a Minispin magnetometer (Fig 6A) interfaced with a personal computer. For the measurement of quartzite and Limestone specimens, each specimen is measured in six positions using a long spin in each position. For the specimens from the dykes measurements are made in four positions after giving a short spin in each position. This procedure is followed because of the weak magnetization in the quartzite and Limestone specimens compared with the strong magnetization in the dykes specimens. All specimens prepared from the rock types were measured adopting the procedure outlined above.

**Thermal demagnetization**

Rocks may acquire magnetization at the time of their formation during cooling in the case of igneous rocks and during deposition in the case of sedimentary rocks. The magnetization in igneous rocks is called Thermo Remnant Magnetization (TRM). The magnetization in the sedimentary rocks (and sometimes in igneous and metamorphic rocks) may be acquired by a chemical process such as the precipitation of a ferromagnetic mineral from solution; the magnetization is called Chemical Remnant Magnetization (CRM). Alternatively Detrital or Depositional Remnant Magnetization (DRM) may be acquired by a sediment during deposition and lithification. This magnetization is acquired by the aligning influence of Earth's magnetic field on ferromagnetic particles at the moment of settling. All these types of
magnetizations are called Natural Remnant Magnetizations (NRM) because they are acquired by natural processes although by different methods.

In addition to NRM the rocks usually acquire a secondary magnetization subsequent to their formation. This can result from viscous acquisition under the long-term influence of the ambient geomagnetic field. Other causes of acquisition of secondary magnetization may be during heating due to deep burial or by coming into proximity of an igneous event, lightning strikes or by weathering.

The preliminary directions are the NRM directions measured after a very long period of time during which any of the above mentioned processes may have modified the original magnetization acquired at the time of the formation of the rock. Therefore the measured NRM is the total magnetization acquired by the rock up to the time of the measurement. Hence it is necessary to remove any secondary magnetization that may be present in the specimens to recover the original magnetization. This is best done by a technique called thermal demagnetization. The specimen is initially heated to a temperature say 100°C in a field free space. It is then cooled in a magnetic field-free environment and its magnetization measured. The specimen then heated again to a higher temperature and measured for the direction and intensity after cooling again in the field-free environment. This procedure is repeated until the intensity of the specimen is very weak and is likely to have been near to the Curie temperature. This procedure is called progressive or stepwise thermal demagnetization and the results of this procedure are best shown as orthogonal projections of the remanence vector component and are called Vector component or Zijderveld diagrams. Observation of an initial linear trend of points indicates successful removal of the low blocking temperature component of magnetization and permits isolation of a high blocking temperature Characteristic Remnant Magnetization (ChRM), which is more likely to have been acquired by the rock at the time of its formation.
Thermal demagnetization of the specimens has been carried out using a Schonstedt Thermal Demagnetization apparatus (Fig. 6B) at the IIT-TIFR palaeomagnetism laboratory, Mumbai. This instrument permits progressive thermal demagnetization of rock specimens by heating to any required temperature up to 800°C and cooling in a zero field environment. There are two separate chambers in the instrument, one for heating and the other for cooling arranged coaxially so that as soon as specimens in the furnace chamber reach thermal equilibrium at the required temperature the specimen holder can be pushed directly into the cooling chamber. There is complete chamber isolation permitting one batch of specimens to be blower cooled while another batch is being heated. Each batch of specimens of ten requires 30 minutes for heating and then 15 minutes for cooling.

Stepwise thermal demagnetization has been carried out on specimens of Quartzite, Limestone and dykes up to 680°C up to the Curie temperature of the specimens when the intensity of the specimen is small or negligible.

Alternating Field (AF) demagnetizations

Alternating magnetic field (AF) demagnetization randomises the remanance according to the coercivity spectra of the magnetic particles in the specimen. The demagnetizing apparatus (Fig 6C) consists of a coil through which an alternating current is passed creating an alternating field along the axis of the coil. Specimens are introduced into the coil in a rotating state. After holding the specimen in the rotating state in the centre of the coil until the demagnetization at the set-demagnetizing field is completed. Then the specimen is withdrawn from the coil in the same rotating state. Then the rotation is stopped and the specimen is measured for the direction. The procedure is repeated at successively increasing strengths of alternating magnetic field, up to 600 Oe. This procedure was adopted on the selected specimens from different sites of the Banganapalli Quartzite, Narji Limestone and dykes.
Fig. 6A: Minispin magnetometer of Molspin make London
B: Thermal demagnetization equipment of Schonstedt make
Fig. 6C: Af demagnetization equipment of Molspin make
D: Susceptibility apparatus of Arun electronics, Mumbai.
Statistical treatment of Data

When a set of stable magnetization directions for samples from a particular formation have been determined by demagnetization tests and component analysis, a set of mean declinations ($D_m$) and inclinations ($I_m$) are determined. Fisher statistics are used to treat the data, and consider them to be points distributed on a sphere. The mean direction is calculated by finding the direction of the vector sum of the individual magnetic vectors.

The precision of the calculated mean direction is represented by the precision parameter 'k' and a circle of 95% confidence ($\alpha_{95}$) is a measure of the confidence with which the mean direction is defined. Since the remnant magnetization is a vector the standard vector algebra is used to obtain the mean direction. Each vector is given a unit weight so that D and I of each direction can be expressed in the Cartesian coordinates by the direction cosines.

$$X = \cos D \cos I$$
$$Y = \sin D \cos I$$
$$Z = \sin I$$

The cosines are then summed for a number (N) vectors to obtain the length of the resultant vector $R$, and its mean direction, $D_m$, $I_m$:

$$R^2 = (\Sigma X^2) + (\Sigma Y^2) + (\Sigma Z^2)$$
$$X = \Sigma X/N, \quad Y = \Sigma Y/N, \quad Z = \Sigma Z/N$$
$$D_m = \tan^{-1} Y/X$$
$$I_m = \sin^{-1} Z$$

The precision parameter is calculated using the Gaussian distribution in three dimensions whereby points on a sphere can be described in terms of a precision parameter, 'k', which is approximated as:
where N is the number of individual magnetization directions and R is the length of vector sum of the individual magnetic vectors.

A value of zero for 'k' means that the data points are randomly scattered. As 'k' increases, the points cluster more closely about a mean direction, with higher 'k' values indicating greater precision in defining this mean.

The accuracy of the mean direction derived from N vectors with resultant R can be expressed as the semi-angle of a cone about the observed mean within which the true mean lies with any given probability (1-P).

\[ \cos \alpha_{(1-P)} = 1-(N-R)/R \cdot [1/P^{(N-1)} - 1] \]

P is normally taken to be 0.05, i.e., there is a 95% probability that the true mean direction lies within the cone of confidence of radius \( \alpha_{0.95} \) around the observed mean.

The most reliable results therefore have the largest 'k' and smallest \( \alpha_{0.95} \).

**Calculation of pole position**

If the latitude of the site is \( \lambda \) (relative to the equator and defined by dipole axis) the inclination I of the dipole field at the site is given by

\[ \tan I = 2 \tan \lambda \]

Conversely if I is known the palaeolatitude \( \lambda_1 \) can be calculated. An NRM direction with D and I observed at a site corresponds to a magnetic pole at an angular distance P from the site along the great circle defined by the declination D, where,
\[ P = 90 - \tan^{-1}(1/2 \tan I_m) \]

\( P \) is the apparent or ancient magnetic colatitude (palaeolatitude, same as \( \lambda_i \) mentioned above). For plotting pole positions to derive Apparent Polar Wandering Paths (APWPs) it is convenient to refer the ancient pole positions to present geographical reference axes i.e., to calculate the present latitudes and longitudes of the ancient poles. If the location of the sampling site is in normal geographic latitude \( (\lambda_n) \) and longitude \( (\phi_n) \) then the latitude \( (\lambda_p) \) and longitude \( (\phi_p) \) of the corresponding pole is given by

\[
\sin \lambda_p = \sin \lambda_n \cos P + \cos \lambda_n \sin P \cos D_m
\]

\[
\phi_p = \phi_n + \beta \quad (\text{for } \cos P > \sin \lambda_n \sin \lambda_p)
\]

\[
\text{or } \phi_p = \phi + (180 - \beta) \quad (\text{for } \cos P < \sin \lambda_n \sin \lambda_p)
\]

where \( \sin \beta = \sin P \sin D_m / \cos \lambda_n \)

latitude is measured between 0 and 90°, positive in the northern hemisphere and longitude is measured from Greenwich 0° to 360°.

The pole calculated above uses \( D_m \) and \( I_m \), which is a mean direction, derived from several specimens. Such a pole is called a ‘Palaeomagnetic Pole’. On the other hand if a pole is determined from one direction of a sample or a dyke, or a lava flow in which the magnetization is likely to have been acquired over a few years or less we have an instantaneous record of the field called ‘Virtual Geomagnetic Pole’ (VGP). VGP gives a spot pole. In rocks such as sedimentary or metamorphic rocks in which the magnetization is acquired by digenesis or slow cooling we may have a long-term average of the geomagnetic field even within a single sample. This can yield a palaeomagnetic pole representative of the time-averaged field.
Rock magnetism

The rock magnetic studies of the samples include the determination of various parameters of Susceptibility ($\chi$), Saturation magnetization ($J_s$) and Saturation remanence ($J_r$). Rock samples comprise of a few percent of magnetic minerals distributed in a largely nonmagnetic matrix. Igneous rocks are magnetically stronger than sedimentary rocks because igneous rocks contain magnetite, which is strongly magnetic possibly with some titanium impurity while sedimentary rocks contain hematite. The physical properties of the rock magnetism have been discussed in detail by Radhakrishnamurty (1993). Rock magnetism is the basis of palaeomagnetism. Single domain and multi domain behaviour and the nature of ferromagnetic minerals contribute a variety of interesting phenomena, in the rocks.

Magnetic Mineralogy

In rock magnetic studies the most important minerals are chiefly composed of iron oxides and titanium. The Primary examples of these are the titanomagnetites (TM), which forms a solid solution series ($xFe_2Ti_4 \cdot (1-x)Fe_3O_4$) and can be represented by using FeO-TiO$_2$-Fe$_2$O$_3$ ternary diagram Fig.7 (Tarling, 1983).

In the magnetic studies of the sediments the most important minerals are magnetite, maghemite and hematite. A brief description of each of the minerals is given below.

*Magnetite* ($Fe_3O_4$)

Magnetite is a common ferromagnetic mineral present in large number of rocks mainly igneous rocks. It forms a part of Magnetite-Ulvospinel series with Ti composition of zero (TM0) to hundred (TM100). The curie temperature ($T_c$) of magnetite is about $580^\circ$C, and with increase of Ti content the Curie points reduces, in the titanomagnetite series. The Coercive force of Single Domain magnetite is 10 Oe.
Whereas for Multi Domain magnetite it is 2 Oe. The $J_s$ for SD and MD magnetite are the same (480 k Am$^{-1}$), but $J_f$ is ten times greater for SD grains (50 k Am$^{-1}$) than for MD grains (50 k A$^{-1}$) (Thompson and Oldfield, 1986).

*Maghaemite (γ Fe$_2$O$_3$)*

Maghaemite has a cation deficient spinel structure and is fully oxidized form of magnetite. It has the same crystal structure of magnetite with composition of haematite (Tarling, 1983). It is a low temperature oxidation product of magnetite and forms readily in the presence of water in a natural pH environment. Maghaemite is having cure temperature of about 345°C.

*Haematite (αFe$_2$O$_3$)*

Haematite is a part of titanohaematite series. All the members of this series are the oxidized equivalents of the titanomagnetite series. It is an antiferromagnetite mineral. The curie temperature of the haematite is about 680°C.

*Susceptibility ($\chi$)*

Magnetic susceptibility is the ratio of the intensity of magnetization produced in a substance to the intensity of the magnetic field to which it is subjected. Susceptibility ($\chi$) is a bulk property dependent on the ferromagnetic material present in the specimen. Therefore the property of the magnetic susceptibility is a useful parameter for comparing with the $J_n$ of the rock specimens in palaeomagnetic research. Since rocks acquire magnetization in the Earth’s magnetic field. Which is of the order of 0.5 Oe, the magnetic susceptibility of rock specimens is usually measured in the some field of 0.5 Oe. A susceptibility apparatus developed by Likhite and Radhakrishnamurty (1965) – Fig.6D and Bartington instruments Ltd., England were used to measure the susceptibility at room temperature, at -196°C and at -150°C. The Susceptibility ($\chi$), Relative Susceptibility -RS ($\chi$-196/\$\chi$25), Peak susceptibility
Fig. 7: Ternary diagram representing iron - titanium solid solution series
(after Tarling, 1983)

(\chi_1/\chi_2), Q_n ratio of dykes and susceptibility and Qn ratios of Quartzites were determined/calculated and given in Appendix V A and V B respectively.

Domain character

Magnetic domain is a region in a magnetized grain with in which the spontaneous magnetization has a constant value characteristic of mineral composition and temperature. The domains are separated from each other by domain walls in which spontaneous magnetization is in different directions.
Radhakrishnamurty and Deutsch (1974) and Radhakrishnamurty et al. (1975, 1977, 1978 and 1982) have identified three domain states, - Single domain (SD), Multi domain (MD), and super paramagnetic (SP) behaviour and one special oxidation state of single domain magnetite referred to as cation deficient (CD) state, - based on the hysteresis behaviour and susceptibility measurements on synthetic materials and single phased magnetic minerals (pure magnetite) and natural rock samples. The CD is single domain and having a composition close to $\gamma$Fe$_2$O$_3$, but showing anomalous hysteresis and susceptibility variations at low temperatures (Radhakrishnamurty et al., 1988).

According to Radhakrishnamurty (1993), Pure MD grains of magnetite can be identified by the presence of a sharp peak at $\chi^{-150}$, and samples with dominantly SD grains will have $\chi$ ($\chi^{-196, \chi_{22}}$) values around 0.4. These SD grains show usually the composition of TM 60 and relative remanence ($J_r/J_s$) values of about 0.5 and 0.8 at room temperature and at $-196^\circ$C respectively. SP grains are essentially SD grains and show Rayleigh (low field hysteresis) loops, with $J_r/J_s$ values less than 0.5 at room temperature and attain 0.8 at $-196^\circ$C. They will have RS values around 0.1 and show the composition of TM 70. The CD grains show increase in susceptibility and decrease in $J_r/J_s$ with decreasing temperature from room temperature to $-196^\circ$C, with RS values of about 1.5.

Radhakrishnamurty et al. (1977) summerised a scheme for determining the domain character of the magnetic materials in rocks and their stability of magnetization. Rocks with dominantly MD or SP grains or combination of them possibly contain unstable magnetic direction while those with SD or CD or mixtures of them are stable. Other combinations like SD+SP, CD+SP, CD+MD and SD+MD are likely to be partially stable. Based on the susceptibility and relative susceptibilities, domain characters of the rock specimens can be determined, with a view to understand the stability of magnetic direction.
Hysteresis

Hysteresis loop is a graph of the induced magnetization against applied field, by which most of the fundamental magnetic properties can be defined. Magnetic properties of a sample depend on its chemical composition and the domain character of the magnetic grains present in it. Hysteresis studies for representative samples are carried out and Coercive force ($H_c$), $J_r/J_s$ ratio (relative remanence) for each sample is given in Fig. 10A,B and in 11A,B.